Supporting Information

Total Synthesis of (+)-Isatisine A

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General Considerations

Melting points were determined using a Gallenkamp melting point apparatus and are uncorrected. Infrared spectra were obtained as thin films on NaCl plates. HPLCs were conducted with a dual λ -absorbance detector set to 256 nm. NMR experiments were conducted in CDCl_3 (referenced to 7.26 ppm for ^1H and 77.0 for ^{13}C) or d_4 -MeOD (referenced to 3.31 ppm for ^1H or 49.15 ppm for ^{13}C). Coupling constants (J) are in Hz. The multiplicities of the signals are described using the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, m = multiplet, br = broad. High-resolution mass spectra (HRMS) were obtained at 70 eV. Toluene, THF, ether, DMF and methylene chloride were dried and deoxygenated by passing the nitrogen-purged solvents through activated alumina columns. All other reagents and solvents were used as purchased from the supplier. The progress of reactions was followed by thin layer chromatography (TLC) (silica gel 60 F₂₅₄) and the developed plates stained using acidic anisaldehyde, phosphomolybdic acid, or basic potassium permanganate. Flash column chromatography was performed using silica gel (230-400 mesh).

Experimental Procedures

Oxidation of pyrroloindole 19: Compound **19** (0.15 g, 0.88 mmol) in MeOH (10 mL) was treated with $\text{MoO}_5 \cdot \text{P} \cdot \text{H}$ (0.45 g, 1.05 mmol) and stirred for 7 d. Upon removal of MeOH the remaining residue was dissolved in Et_2O , filtered through Celite, pre-adsorbed onto silica and purified *via* flash column chromatography (18-35% EtOAc/Hexanes) to yield compound **23** (0.029 g, 17%) as a yellow solid, compound **24** (0.047 g, 25%) as a white solid and compound **25** (0.014 g, 7.6%) as a yellow oil.

Alternatively, if compound **19** (0.05 g, 0.9 mmol) was treated with $\text{MoO}_5 \cdot (\text{OPPh}_3)_2$ (0.26 g, 0.35 mmol) in MeOH/ CH_2Cl_2 (3 mL : 3mL) for 16 h a 48% yield of **24** (0.031 g) could be obtained. Treatment of **24** (0.01g, 0.045 mmol) with IBX (0.028 g, 0.055 mmol) in DMSO (2 mL) for 6 h yields compound **25** (0.008 g, 80%); **23**: ^1H -NMR (400 MHz, CDCl_3): δ = 7.82 (d, J = 8.0 Hz, 1H), 7.73-7.70 (m, 2H), 7.28-7.24 (m, 2H), 3.31-3.22 (m, 2H), 2.61 (dd, J = 16.8, 8.8 Hz, 1H), 2.42 (dd, J = 13.6, 8.0 Hz, 1H), 2.21 (ddd, J = 13.2, 12.4, 8.8 Hz, 1H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 197.2, 174.3, 149.1, 138.0, 125.4, 125.2, 123.7, 116.7, 92.2, 33.7, 29.3; IR (thin film, cm^{-1}): ν_{max} = 3380, 2926, 2855, 1723, 1604, 1466, 1388, 1314, 1302, 1271, 1232, 1213, 1173, 1150, 1092, 1050, 984, 905, 736; HRMS calc. for $\text{C}_{11}\text{H}_9\text{NO}_3$

= 203.0582, found 203.0583; R_f = 0.33, 50% EtOAc/hexanes. **24**: $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.57 (d, J = 7.6 Hz, 1H), 7.51 (d, J = 8.0 Hz, 1H), 7.36 (t, J = 7.6 Hz, 1H), 7.19 (t, J = 7.6 Hz, 1H), 4.59 (d, J = 9.2 Hz, 1H), 3.35 (d, J = 9.6 Hz, 1H), 3.21 (s, 3H), 2.82-2.62 (m, 2H), 2.10-1.96 (m, 2H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 193.9, 138.3, 135.8, 129.9, 126.6, 125.5, 115.8, 104.1, 74.8, 50.1, 34.3, 22.8; ; IR (thin film, cm^{-1}): ν_{max} = 3383, 1991, 2963, 2937, 2831, 1692, 1606, 1483, 1467, 1399, 1319, 1181, 1168, 1100, 1071, 926, 909, 758, 736; HRMS calc. for $\text{C}_{12}\text{H}_{13}\text{NO}_3$ = 219.0895, found 219.0902; R_f = 0.20, 50% EtOAc/hexanes. **25**: $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.87 (d, J = 8.4 Hz, 1H), 7.73-7.70 (m, 2H), 7.28-7.24 (m, 2H), 3.25 (s, 3H), 3.18 (ddd, J = 16.8, 12.4, 7.6 Hz, 1H), 2.59 (dd, J = 16.8, 8.8 Hz, 1H), 2.47 (dd, J = 13.6, 7.6 Hz, 1H), 2.12 (ddd, J = 13.6, 12.4, 8.8 Hz, 1H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 197.5, 175.2, 150.5, 138.1, 125.1, 124.8, 124.6, 116.5, 96.0, 51.9, 34.0, 28.6; IR (thin film, cm^{-1}): ν_{max} = 2992, 2953, 2933, 2832, 1724, 1605, 1466, 1347, 1311, 1171, 1149, 1100, 1072, 879, 759, 696; HRMS calc. for $\text{C}_{12}\text{H}_{11}\text{NO}_3$ = 217.0739, found 217.0746 R_f = 0.62, 50% EtOAc/hexanes.

Compound **C**

Compound **C** and **20** was obtained through a modification of a literature procedure.¹

To a solution of enamine **A** (3.83 g, 19.9 mmol), and pyridine (1.61 mL, 19.9 mmol) in THF (25 mL) was added a solution of acid chloride **B** (3.00 g, 19.9 mmol) in THF (25 mL). After heating to reflux for 16 h the reaction was transferred to a separatory funnel containing H_2O . The solution was extracted with EtOAc (3x), and the combined organic extract were washed with brine (1x), dried over MgSO_4 and concentrated under reduced pressure. The crude red oil (8.22 g) was dissolved in a mixture of 1,4-dioxane (90 mL) and H_2O (30 mL) then heated to reflux for 16 h. The volatile organic solvent was removed under reduced pressure and the remaining solution transferred to a separatory funnel, extracted with EtOAc (3x), the combined organic extracts were washed with brine (1x), dried over MgSO_4 and concentrated under reduced pressure. Purification by flash column chromatography (16-24% EtOAc/Hexanes) yielded the title compound (2.58 g, 52%) as a red liquid. (R_f = 0.43, 50% EtOAc/hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 8.09 (dd, J = 8.4, 1.2 Hz, 1H), 7.57 (dt, J = 7.8, 1.2 Hz, 1H), 7.44 (dt, J = 7.8, 1.2 Hz, 1H), 7.29 (dd, J = 7.8, 1.2 Hz, 1H), 4.14 (s, 2H), 3.65 (s, 3H), 2.91 (t, J = 6.6 Hz, 2H), 2.63 (t, J = 6.6 Hz, 2H);

^{13}C -NMR (100 MHz, CDCl_3): δ = 204.0, 173.1, 148.4, 133.6, 133.6, 130.1, 128.4, 125.1, 51.8, 51.8, 47.7, 37.0, 27.7; IR (thin film, cm^{-1}): ν_{max} = 3071, 3000, 2955, 2855, 1737, 1724, 1526, 1349, 1200, 1180, 1088, 790, 737; HRMS calc. for $\text{C}_{12}\text{H}_{14}\text{NO}_5^{1+}$ = 252.0865, found 252.0872.

Compound **20**

Compound **C** (1.5 g, 5.97 mmol) was dissolved in a solution of MeOH (120 mL) and 1 M aqueous NH_4Cl (34 mL). Zinc dust (13.3 g, 203 mmol) was added to the mixture and stirred for 21 h after which the solution was filtered through celite and the MeOH removed under reduced pressure. The remaining liquid was transferred to a separatory funnel, extracted with EtOAc (3x), the combined organic extracts were washed with brine (1x), dried over MgSO_4 and concentrated under reduced pressure. Purification *via* flash column chromatography (15-20% EtOAc/Hexanes) yielded compound **20** (1.11 g, 91%) as a yellow/white solid. (R_f = 0.49, 50% EtOAc/hexanes); ^1H -NMR (600 MHz, CDCl_3): δ = 8.47 (broad s, 1H), 7.53 (d, J = 7.8 Hz, 1H), 7.31 (d, J = 8.4 Hz, 1H), 7.13 (t, J = 7.8 Hz, 1H), 7.07 (t, J = 7.8 Hz, 1H), 6.23 (s, 1H), 3.73 (s, 3H), 3.08 (t, J = 6.6 Hz, 2H), 2.74 (t, J = 6.6 Hz, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 174.3, 138.2, 136.0, 128.5, 121.3, 119.9, 119.6, 110.5, 99.9, 52.0, 33.9, 23.1; IR (thin film, cm^{-1}): ν_{max} = 3358, 1720, 1459, 1421, 1293, 1198, 796, 770, 746, 647; HRMS calc. for $\text{C}_{12}\text{H}_{13}\text{NO}_2$ = 203.0953, found 203.0946; mp = 98-100 °C.

Compound **26**

Compound **20** in a MeOH: CH_2Cl_2 (11 mL:11 mL) was treated with $\text{MoO}_5 \cdot \text{P} \cdot \text{H}$ and stirred for 48 h under an argon atmosphere. The reaction was diluted with Et_2O , filtered through Celite, pre-adsorbed onto silica and purified *via* flash column chromatography (13-16% EtOAc/Hexanes) to yield **26** (0.157 g, 57%) as a red oil. (R_f = 0.38, 50% EtOAc/hexanes); ^1H -NMR (600 MHz, CDCl_3): δ = 7.56 (d, J = 7.8 Hz, 1H), 7.47 (t, J = 7.8 Hz, 1H), 6.85 (d, J = 8.4 Hz, 1H), 6.83 (t, J = 7.2 Hz, 1H), 5.28 (broad s, 1H), 3.65 (s, 3H), 3.15 (s, 3H), 2.63-2.58 (m, 1H), 2.90-2.44 (m, 1H), 2.18-2.14 (m, 1H), 2.08-2.03 (m, 1H); ^{13}C -NMR

(100 MHz, CDCl₃): δ = 200.7, 174.4, 160.1, 138.3, 124.6, 120.1, 119.5, 112.3, 91.2, 51.8, 50.9, 31.1, 27.5; IR (thin film, cm⁻¹): ν_{max} = 33.57, 2953, 1717, 1618, 1488, 1471, 1457, 1321, 1199, 1079, 905, 755; HRMS for C₁₃H₁₅NO₄ could not be obtained on multiple attempts.

Compound **28**

A mixture of compound **26** (0.33 g, 1.32 mmol), indole (0.17 g, 1.46 mmol), and acetic acid (5.4 mL) in MeOH (75 mL) was heated to reflux for 16 h after which time the MeOH was removed under reduced pressure. The remaining solution was transferred to a separatory funnel containing EtOAc. The mixture was washed with a saturated aqueous solution of NaHCO₃ (2x), H₂O (1x), brine (1x), dried over MgSO₄ and concentrated under reduced pressure. Purification *via* flash column chromatography (15-22% EtOAc/Hexanes) yielded compound **28** (0.36 g, 81%) as a yellow/brown solid. (R_f = 0.31, 50% EtOAc/hexanes); ¹H-NMR (600 MHz, CDCl₃): δ = 8.34 (br s, 1H), 7.65 (d, J = 7.2 Hz, 1H), 7.51-7.48 (m, 2H), 7.32 (d, J = 7.8 Hz, 1H), 7.16 (t, J = 7.2 Hz, 1H), 7.11 (s, 1H), 7.04 (t, J = 7.8 Hz, 1H), 6.89-6.85 (m, 2H), 5.23 (broad s, 1H), 3.62 (s, 3H), 2.71-2.66 (m, 1H), 2.57-2.52 (m, 1H), 2.42-2.36 (m, 1H), 2.30-2.25 (m, 1H); ¹³C-NMR (100 MHz, CDCl₃): δ = 202.4, 174.2, 160.8, 137.7, 136.9, 125.0, 124.9, 122.6, 122.4, 120.6, 120.1, 119.8, 119.3, 114.2, 112.6, 111.6, 68.5, 51.7, 31.0, 28.7; IR (thin film, cm⁻¹): ν_{max} = 3366, 2951, 2926, 2854, 1722, 1685, 1617, 1489, 1467, 1323, 1279, 1197, 1176, 1151, 1104, 1014, 887, 749; HRMS calc. for C₂₀H₁₈N₂O₃ = 334.1317, found 334.1316.

Compound **21**

Ester **28** (0.14 g, 0.42 mmol) was hydrolyzed with LiOH (0.035 g, 0.84 mmol) in a 3:1 mixture of MeOH and H₂O (10 mL total volume). After stirring for 16 h the MeOH was removed under reduced

pressure and the remaining aqueous solution transferred to a separatory funnel containing EtOAc and saturated aqueous NaHCO₃. The organic layer was removed and the aqueous layer was acidified with 5% aqueous HCl. The aqueous layer was extracted with EtOAc (2x), the combined organic extracts were washed with brine (1x), dried over MgSO₄ and concentrated to yield acid **29** (0.14 g, quant.) as a yellow foam which was carried onto the next step without any further purification. To acid **29** (0.05 g, 0.15 mmol) in THF (120 mL) was added EDC (0.082 mL, 0.47 mmol) and the mixture heated to reflux. After 20 h the THF was removed under reduced pressure, the remaining residue dissolved in Et₂O and transferred to a separatory funnel. The organic layer was washed with 5% aqueous HCl (3x), saturated aqueous NaHCO₃ (1x), brine (1x), dried over MgSO₄ and concentrated under reduced pressure. Purification *via* flash column chromatography (EtOAc/Hexanes) yielded compound **21** (0.03 g, 66% over 2 steps) as a yellow foam. (*R*_f = 0.32, 50% EtOAc/hexanes); ¹H-NMR (600 MHz, CDCl₃): δ = 8.22 (broad s, 1H), 8.02 (d, *J* = 7.8 Hz, 1H), 7.98 (d, *J* = 7.8 Hz, 1H), 7.68 (t, *J* = 7.8 Hz, 1H), 7.63 (d, *J* = 7.2 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.24-7.18 (m, 4H), 2.98 (ddd, *J* = 16.2, 12.0, 7.8 Hz, 1H), 2.89 (dd, *J* = 12.0, 7.8 Hz, 1H), 2.65-2.54 (m, 2H); ¹³C-NMR (100 MHz, CDCl₃): δ = 198.8, 173.8, 149.4, 137.3, 136.8, 125.4, 125.1, 125.0, 124.6, 122.9, 122.0, 120.8, 120.5, 116.5, 112.6, 111.6, 74.0, 34.6, 29.3; IR (thin film, cm⁻¹): ν_{max} = 3401, 3062, 2991, 2928, 1708, 1654, 1466, 1343, 1216, 1137, 744; HRMS calc. for C₁₉H₁₄N₂O₂ = 302.1066, found 302.1055.

Compound **D**

To a slurry of sodium hydride (2.24 g 60 % in oil, 56.1 mmol) in DMF (80 mL) under argon was added methyl 2-indole carboxylate (7.56 g, 43.16 mmol) in DMF (80 mL) and the solution stirred until evolution of gas ceased. A solution of tosyl chloride (10.70 g, 56.1 mmol) in DMF (40 mL) was then added and the mixture stirred for 16 h at room temperature. The reaction was added to H₂O and extracted with EtOAc (4x), the combined organic extracts were washed with H₂O (2x), brine, dried over MgSO₄ and filtered. The crude ¹H-NMR showed a 1:0.15 ratio of product to starting material which were separated *via* flash column chromatography (5-8% EtOAc/Hexanes) to yield 12.03 g (85%, 93% brsm) **D**. (*R*_f = 0.38, 30% EtOAc/hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 8.12 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 2H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.45 – 7.41 (m, 1 H), 7.30 – 7.25 (m, 3H), 7.16 (d, *J* = 0.8 Hz, 1H), 3.94 (s, 3H), 2.38 (s, 3H); ¹³C-NMR (100 MHz, CDCl₃): δ = 161.7, 144.9, 138.2, 135.7, 131.4, 129.5, 128.1, 127.3, 127.0, 124.0, 122.5, 116.8, 115.4, 52.7, 21.6; IR (thin film, cm⁻¹): ν_{max} = 2952, 1734, 1434,

1374, 1341, 1309, 1202, 677, 578; HRMS calc. for $C_{17}H_{15}NO_4S$ = 329.0721, found 329.0722; mp = 81-83 °C, lit. mp = 83-84 °C.²

Compound **E**

A slurry of $LiAlH_4$ (3.33 g, 87.7 mmol) in THF (300 mL) was cooled to 0 °C and **D** (12.0 g, 36.5 mmol) in THF (50 mL) was added *via* canula. An additional 50 mL of THF was used to quantify the transfer. The reaction was monitored by TLC and upon consumption of the starting material 3.3 mL of H_2O was added, followed by 3.3 mL of 15% aqueous NaOH, then another 10 mL of H_2O . The solution was filtered through Celite using 100 mL of THF to rinse the reaction flask and filter cake, concentrated and taken onto the next reaction. A small amount of material was purified by flash column chromatography (15% EtOAc/Hexanes) and characterized. (R_f = 0.20, 30% EtOAc/hexanes); 1H -NMR (400 MHz, $CDCl_3$): δ = 8.08 (d, J = 8.0 Hz, 1H), 7.75 (d, J = 8.0 Hz, 2H), 7.51 (d, J = 8.0 Hz, 1H), 7.35 – 7.23 (m, 4H), 6.67 (s, 1H), 4.94 (s, 1H), 3.15 (bs, 1H), 2.37 (s, 3H); ^{13}C -NMR (100 MHz, $CDCl_3$): δ = 145.1, 140.2, 137.0, 135.6, 129.9, 129.1, 126.4, 125.0, 123.7, 121.1, 114.3, 111.2, 58.5, 21.5; IR (thin film, cm^{-1}): ν_{max} = 3400, 3068, 2928, 2678, 1598, 1452, 1369, 1188, 1174, 1091, 1061, 912, 813, 749, 581; HRMS calc. for $C_{16}H_{15}NO_3S$ = 301.1079, found 301.0773; mp = 92-94 °C.

Compound **31b**

DMSO (5.2 mL, 73.0 mmol) in CH_2Cl_2 (50 mL) was added *via* canula to a solution of oxalyl chloride (4.14 mL, 47.5 mmol) in CH_2Cl_2 (250 mL) at -78 °C.¹¹⁰ Crude **4-E** was dissolved in CH_2Cl_2 (50 mL) and added to the solution. After stirring for 30 min NEt_3 (25 mL, 183 mmol) was added and the reaction was warmed to room temperature while stirring overnight. The reaction was then added to H_2O and extracted with CH_2Cl_2 (2x), the combined organic extracts were washed with 5% aqueous HCl, brine, dried over $MgSO_4$, filtered and concentrated. Purification by flash column chromatography (3-11% EtOAc/Hexanes) yielded 8.98 g (82% over 2 steps) of **31b** as a white solid. (R_f = 0.37, 30% EtOAc/hexanes); 1H -NMR

(400 MHz, CDCl₃): δ = 10.53 (s, 1H), 8.23 (dd, J = 0.4, 8.4 Hz, 1H), 7.65 (d, 8.4 Hz, 2H), 7.62 (dd, J = 1.2, 8.0 Hz, 1H), 7.52 (ddd, J = 1.2, 7.2, 7.2 Hz, 1H), 7.31 (ddd, J = 0.8, 7.2, 7.2 Hz, 1H), 7.18 (d, J = 8.4 Hz, 2H), 2.32 (s, 3H); ¹³C-NMR (100 MHz, CDCl₃): δ = 183.3, 145.6, 138.4, 137.7, 134.6, 129.9, 128.8, 128.1, 126.6, 124.8, 123.6, 118.8, 115.3, 21.6; IR (thin film, cm⁻¹): ν_{max} = 3123, 3172, 3922, 2903, 1696, 1605, 1438, 1237, 1124, 1051, 913, 813, 669, 578; HRMS calc. for C₁₆H₁₃NO₃S = 299.0626, found 299.0616; mp = 136-137 °C.

Compound **30a**

To a solution of aldehyde **31b** (0.1 g, 0.33 mmol) and vinylcyclopropane **32c** (0.09 g, 0.49 mmol) in CH₂Cl₂ (1 mL) was added SnCl₄ (0.13 mL of 1 M solution, 0.13 mmol) and stirred for 16 h. The solution was passed through a plug of silica, rinsed with CH₂Cl₂ and concentrated. The ¹H-NMR spectra of the crude reaction mixture showed a 1:0.33 of 2,5-*cis* to 2,5-*trans* isomers. Purification by flash column chromatography (4-7% EtOAc/Hexanes) yielded a mixture of **30a** (0.150g, 95%) as a white foam. (R_f = 0.28, eluted twice in 20% EtOAc/hexanes); (Minor isomer denoted by *) ¹H-NMR (400 MHz, CDCl₃): δ = 8.08 (dq, J = 7.6, 0.8 Hz, 1H*), 8.05 (dq, J = 8.0, 0.8 Hz, 1H), 7.70-7.66 (m, 2H*, 2H), 7.34-7.42 (m, 1H*, 1H), 7.29-7.71 (m, 4H*, 4H), 6.86 (d, J = 0.8 Hz, 1H*), 6.77 (t, J = 0.8 Hz, 1H), 6.73 (d, J = 0.8 Hz, 1H), 6.69 (t, J = 0.8 Hz, 1H*), 6.04 (ddd, J = 16.8, 10.4, 6.4 Hz, 1H), 5.88 (ddd, J = 16.8, 10.0, 6.0 Hz, 1H*), 5.45 (dt, J = 17.2, 1.2 Hz, 1H), 5.35 (dt, J = 17.2, 1.2 Hz, 1H*), 5.31 (dt, J = 10.4, 1.2 Hz, 1H), 5.19 (dt, J = 10.4, 1.2 Hz, 1H*), 4.96-4.91 (m, 1H*), 4.53-4.48 (m, 1H), 3.89 (s, 3H), 3.84 (s, 3H*), 3.45 (s, 3H*), 3.39 (s, 3H), 3.13 (dd, J = 13.2, 8.0 Hz, 1H*), 2.76 (dd, J = 13.2, 11.6 Hz, 1H), 2.48 (dd, J = 13.2, 4.8 Hz, 1H), 2.46 (dd, J = 13.6, 7.2 Hz, 1H*), 2.30 (s, 1H*, 1H); ¹³C-NMR (100 MHz, CDCl₃): δ = 170.8, 168.4, 144.8, 139.2, 137.0, 136.0, 135.2, 129.7, 129.6, 129.2, 126.7, 126.6, 124.7, 123.7, 121.1, 118.3, 114.7, 111.9, 79.3, 78.0, 66.0, 53.3, 52.7, 40.3, 21.5; HRMS calc. for C₂₅H₂₅NO₇S = 483.1352, found 483.1338.

Compound **37**

To a mixture of alcohols **35** and **36** (0.317 g, 0.74 mmol, 0.57 mmol desired isomer) in DMF (1 mL) was added imidazole (0.111 g, 1.63 mmol) and TBDPSCl (0.21 mL, 0.82 mmol). The reaction was stirred for 16 h after which time it was added to a separatory funnel containing water and Et₂O and extracted with Et₂O (3x). The combined organic extracts were washed with brine, dried over MgSO₄, pre-adsorbed onto silica and purified by flash column chromatography (5-10% EtOAc/Hexanes) to yield the title compound (0.305 g, 80% from desired isomer) as a white foam. (*R*_f = 0.48, 30% EtOAc/hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 8.12 (d, *J* = 8.2 Hz, 1H), 7.71-7.67 (m, 6H), 7.44-7.28 (m, 8H), 7.19 (dt, *J* = 7.6, 0.8 Hz, 1H), 7.15 (d, *J* = 8.8 Hz, 1H), 6.93 (s, 1H), 5.89 (dd, *J* = 4.0, 1.2 Hz, 1H), 4.36-4.30 (m, 1H), 3.96 (dd, *J* = 11.2, 3.6 Hz, 1H), 3.80 (s, 3H), 3.76 (dd, *J* = 11.8, 3.6 Hz, 1H), 3.42 (dt, *J* = 9.2, 3.6 Hz, 1H), 2.39-2.31 (m, 1H), 2.30 (s, 3H), 2.14 (ddd, *J* = 12.8, 5.6, 3.2 Hz, 1H), 1.09 (s, 9H); ¹³C-NMR (100 MHz, CDCl₃): δ = 174.26, 144.83, 141.6, 137.2, 135.6, 135.5, 135.5, 133.2, 133.0, 129.8, 129.7, 129.2, 127.7, 126.6, 124.4, 123.5, 120.9, 114.5, 109.7, 79.8, 77.1, 64.6, 52.2, 50.9, 31.1, 26.8, 21.5, 19.2; IR (thin film, cm⁻¹): ν_{max} = 3070, 3049, 2954, 2931, 2858, 1735, 1473, 1451, 1387, 1210, 1105, 1055, 823, 744; HRMS calc. for C₃₈H₄₁NO₆SSi = 667.2424, found 667.2439.

Compound **43**

To a mixture of alcohols **42** (0.619 g, 1.27 mmol, 10:1 *cis:trans*) in CH₂Cl₂ (50 mL) was added imidazole (0.190 g, 2.79 mmol) and TBDPSCl (0.36 mL, 1.40 mmol). The reaction was stirred for 16 h after which time it was added to a separatory funnel containing water and CH₂Cl₂ and extracted with CH₂Cl₂ (3x). The combined organic extracts were washed with brine, dried over MgSO₄, pre-adsorbed onto silica and purified by flash column chromatography (10% EtOAc/Hexanes) to yield a mixture of **43** (0.874 g, 95%, 10:1 10:1 *cis:trans*) as a white foam. (*R*_f = 0.40, 30% EtOAc/hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 8.05 (d, *J* = 8.4 Hz, 1H), 7.73-7.66 (m, 7H), 7.44-7.13 (m, 10H), 6.97 (s, 1H), 6.77 (s,

1H), 4.16 (X of ABX, $J = 12.0, 4.0$ Hz, 1H), 4.03 (A/B of ABX, $J = 11.6, 3.6$ Hz, 1H), 3.88 (s, 3H), 3.83 (A/B of ABX, $J = 11.6, 3.6$ Hz, 1H), 3.1 (s, 3H), 3.03 (t, $J = 12.4$ Hz, 1H), 3.33 (dd, $J = 12.8, 4.8$ Hz, 1H), 2.29 (s, 3H), 1.12 (s, 9H); ^{13}C -NMR (100 MHz, CDCl_3): $\delta = 170.9, 168.3, 144.6, 139.4, 137.1, 135.7, 135.6, 135.2, 133.3, 133.0, 129.8, 129.7, 129.7, 129.6, 129.4, 127.7, 126.6, 124.6, 123.6, 121.1, 114.7, 112.4, 78.9, 77.8, 66.0, 64.3, 53.3, 52.7, 36.0, 26.9, 26.8, 21.5, 19.3$; IR (thin film, cm^{-1}): $\nu_{\text{max}} = 3072, 3051, 2954, 2932, 2889, 2859, 2259, 1735, 1473, 1451, 1376, 1279, 1152, 912, 823, 811, 737$; HRMS calc. for $\text{C}_{40}\text{H}_{43}\text{NO}_8\text{SSi} = 725.2479$, found 725.2505.

Compound **32d** (5:1 dr)

To a stirring solution of racemic dimethyl vinylcyclopropane-1,1-diester²² (**32**) (2.84 g, 15.44 mmol) in methanol (12 mL) was added 11.8 mL of an aqueous 1.7 N NaOH solution. The resultant solution was stirred for 1.5 h after which time TLC analysis showed complete consumption of the starting material. The reaction was poured into a separatory funnel containing 5% aqueous HCl and extracted with EtOAc (3x). The combined organic extracts were washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo* to yield 2.89 g of a clear colorless oil. The resultant acid was dissolved in DMF (50 mL) and K_2CO_3 (2.35 g, 17.0 mmol) was added followed by 1.88 mL (15.75 mmol) of allyl bromide. The solution was stirred under argon for 48 h then added to H_2O and extracted with Et_2O (4x). The combined organic extracts were washed with H_2O (2x), brine, dried over MgSO_4 , and filtered. Concentration *in vacuo* yielded 3.08 g (77% over 2 steps) of **32d** as a clear colorless oil which was used without further purification. ($R_f = 0.54$, 30% EtOAc/hexanes); ^1H -NMR (400 MHz, CDCl_3): $\delta = 5.88$ (X of ABX, $J = 17.2, 11.2, 6.0, 6.0$ Hz, 1H), 5.46-5.21 (m, 4H), 5.13 (dd, $J = 10.0, 1.6$ Hz, 1H), 4.66 (A/B of ABX, $J = 13.2, 5.2, 1.6, 1.6$ Hz, 1H), 4.59 (A/B of ABX, $J = 13.2, 5.2, 1.6, 1.6$ Hz, 1H), 3.73 (s, 3H), 2.58 (apparent q, $J = 8.4$ Hz, 1H), 1.72 (dd, $J = 7.6, 4.8$ Hz, 1H), 1.58 (dd, $J = 7.6, 4.8$ Hz, 1H); ^{13}C -NMR (100 MHz, CDCl_3): $\delta = 169.1, 167.7, 132.9, 131.6, 118.7, 118.2, 66.0, 52.5, 35.8, 31.4, 20.6$; IR (thin film, cm^{-1}): $\nu_{\text{max}} = 3089, 3015, 2992, 2954, 1730, 1457, 1367, 1208, 1131, 920, 748$; HRMS calc. for $\text{C}_{11}\text{H}_{14}\text{O}_4 = 210.0892$, found 210.0885.

Compound **30c**

To a solution of aldehyde **32b** (0.7 g, 2.35 mmol) and vinylcyclopropane **31d** (0.45 g, 2.14 mmol) in CH_2Cl_2 (30 mL) was added SnCl_4 (0.43 mL of 1 M solution, 0.43 mmol) and stirred for 3.3 h. The solution was passed through a plug of silica, rinsed with CH_2Cl_2 and concentrated. ^1H -NMR of the crude reaction mixture showed a 1:0.51 of 2,5-*cis* to 2,5-*trans* isomers. Purification by flash column chromatography (4-10% EtOAc/Hexanes) yielded a mixture of **30c** (0.889 g, 81%) as a white foam. (R_f = 0.31, 30% EtOAc/hexanes); For ^1H -NMR (400 MHz, CDCl_3) and ^{13}C -NMR (100 MHz, CDCl_3) see spectra in supporting information; HRMS calc. for $\text{C}_{27}\text{H}_{27}\text{NO}_7\text{S}$ = 509.1508, found 509.1522.

Compound **30d**

$\text{Sn}(\text{OTf})_2$ (0.18 g, 0.44 mmol) was placed in a flask equipped with a stir bar and purged with argon. Cyclopropane **32e** (0.77 g, 2.94 mmol) and aldehyde **31e** (0.77 g, 3.78 mmol) were dissolved in 3 mL CH_2Cl_2 and added to the flask.^{13c} An additional 1 mL of CH_2Cl_2 was used to quantify the transfer of cyclopropane and aldehyde. After stirring for 49 h the reaction was passed through a small plug of silica gel and the resultant solution was concentrated *in vacuo*. Purification by flash column chromatography (5-8% EtOAc/Hexanes) yielded 0.23 g (3 diastereomers, 2:1-*cis:trans*) as a white foam and 0.81 g (major diastereomer shown above) of **30d**. (R_f = 0.31 (mixture) and 0.43 (major isomer), 30% EtOAc/hexanes); Major diastereomer: ^1H -NMR (600 MHz, CDCl_3): δ = 8.02 (d, J = 7.8 Hz, 1H), 7.49 (d, J = 7.2 Hz, 1H), 7.29-7.18 (m, 5H), 6.89 (d, J = 7.2 Hz, 2H), 6.79 (s, 1H), 6.53 (d, J = 1.2 Hz, 1H), 6.06 (ddd, J = 16.8, 10.2, 6.6 Hz, 1H), 5.44 (dt, J = 16.8, 1.2 Hz, 1H), 5.30 (dt, J = 16.8, 1.2 Hz, 1H), 4.85 (d, J = 12.0 Hz, 1H), 4.48-4.44 (m, 1H), 4.31 (d, J = 12.0 Hz, 1H), 3.82 (s, 3H), 3.73 (s, 3H), 2.78 (dd, J = 13.2,

12.0 Hz, 1H), 2.46 (dd, $J = 13.2, 4.8$ Hz, 1H); ^{13}C -NMR (100 MHz, CDCl_3): $\delta = 171.1, 168.0, 151.7, 138.4, 136.7, 135.9, 134.4, 128.7, 128.2$ (multiple C's), 124.4, 123.0, 120.7, 118.1, 115.6, 109.9, 79.3, 79.0, 67.7, 65.7, 53.2, 52.9, 41.0; IR (thin film, cm^{-1}): $\nu_{\text{max}} = 3068, 2954, 2855, 1734, 1456, 1440, 1332, 1260, 1218, 1086, 1051, 766$; HRMS calc. for $\text{C}_{26}\text{H}_{25}\text{NO}_7 = 463.1631$, found 463.1524.

Compound **47a**

To the major diastereomer of **30d** (0.77 g, 1.66 mmol) dissolved in THF (5 mL), H_2O (5 mL), and acetone (1 mL) was added methanesulfonamide (0.16 g, 1.66 mmol), NMO (0.20 g, 1.74 mmol) and a large crystal of OsO_4 . The reaction was monitored by TLC and upon completion sodium sulphite was added to the reaction and stirred for a further 30 min. The reaction was then filtered through Celite and the volatile components were removed *in vacuo*. The remaining solution was added to brine and extracted with EtOAc (4x), dried over MgSO_4 , filtered and concentrated under reduced pressure to yield a white foam ($R_f = 0.37$, 100% EtOAc). The resultant foam was dissolved in THF (10 mL) and H_2O (10 mL), and NaIO_4 (7.63 g, 35.7 mmol) added. The reaction was monitored by TLC and upon completion 50 mL of EtOH was added and the solution filtered through Celite rinsing the filter cake with 10 mL of EtOH ($R_f = 0.67$, 100% EtOAc). NaBH_4 (0.10 g, 2.6 mmol) was added to the filtrate and the solution stirred at room temperature. TLC monitoring showed consumption of the aldehyde after 30 min at which time the reaction was quenched with drop wise addition of a saturated aqueous solution of NH_4Cl . The organic solvents were removed under reduced pressure and the remaining solution added to H_2O and extracted with EtOAc (4x). The combined organic extracts were washed with brine, dried over MgSO_4 , filtered and concentrated *in vacuo*. Purification by flash column chromatography (15-20% EtOAc/Hexanes) yielded 0.46 g of the primary alcohol **47a** as a white foam (61% over 3 steps). ($R_f = 0.26$, 50% EtOAc/hexanes); ^1H -NMR (600 MHz, CDCl_3): $\delta = 8.00$ (d, $J = 9.0$ Hz, 1H), 7.48 (d, $J = 7.8$ Hz, 1H), 7.30-7.17 (m, 5H), 6.87 (d, $J = 7.8$ Hz, 2H),

Alcohol **47a** (0.41 g, 0.88 mmol) were dissolved in CH₂Cl₂ (5 mL) followed by the successive addition of TBSCl (0.20 g, 1.32 mmol) and imidazole (0.09 g, 1.32 mmol). The reaction was complete after 15 min at which point it was added to H₂O and extracted with CH₂Cl₂ (2x). The combined organic extracts washed with brine, dried over MgSO₄, and filtered. Purification *via* flash column chromatography (6-15% EtOAc/Hexanes) yielded 0.48 g (94%) of **48a** as a white foam. (R_f = 0.34, 30% EtOAc/Hexanes); ¹H-NMR (600 MHz, CDCl₃): δ = 8.05 (d, J = 9.0 Hz, 1H), 7.46 (d, J = 7.8 Hz, 1H), 7.28-7.17 (m, 5H), 6.97 (s, 1H), 6.87 (d, J = 7.8 Hz, 2H), 6.57 (s, 1H), 4.80 (d, J = 12.6 Hz, 1H), 4.37 (d, J = 12.6 Hz, 1H), 4.07 (apparent dq, J = 12.0, 4.2 Hz, 1H), 3.98 (dd, J = 10.8, 3.6 Hz, 1H), 3.86 (dd, J = 10.8, 4.2 Hz, 1H), 3.81 (s, 3H), 3.75 (s, 3H), 2.92 (t, J = 12.0 Hz, 1H), 2.33 (dd, J = 13.2, 4.8 Hz, 1H), 0.95 (s, 9H), 0.14 (s, 3H), 0.11 (s, 3H); ¹³C-NMR (100 MHz, CDCl₃): δ = 177.2, 168.1, 151.8, 138.7, 136.8, 134.4, 128.9, 128.2, 128.2, 124.3, 123.0, 120.7, 115.6, 110.2, 79.1, 78.8, 67.7, 65.8, 63.6, 53.3, 52.9, 36.6, 25.9, 18.4, -5.3, -5.4; IR (thin film, cm⁻¹): ν_{max} = 2955, 2930, 2889, 2857, 1753, 1728, 1454, 1440, 1329, 1267, 1224, 1134, 1085, 913, 840, 735; HRMS calc. for C₃₁H₃₉NO₈Si = 581.2448, found 581.2460.

Compound **49a**

To compound **48a** (0.10 g, 0.17 mmol) in THF (3 mL) was added 0.01 g of 10% Pd/C. The reaction vessel was purged with hydrogen gas (4x) then placed under 1 atmosphere of hydrogen for 3 h after which time TLC analysis showed consumption of the diester. The solution was filtered through a pad of Celite and the Celite was rinsed with THF (3 mL). Triphenylphosphine (0.046g, 0.17 mmol) and allyl alcohol (0.012 mL, 0.17 mmol) were added to the solution along with a magnetic stir bar. With stirring DIAD (0.034 mL, 0.17 mmol) was added to the solution and stirring was continued for 50 min after which time a further 2 equiv. of all the reagents were added. After 40 min TLC analysis showed consumption of the acid substrate. Purification by flash column chromatography (4-6% EtOAc/Hexanes) yielded 0.083 g (91%) of **49a** as a white foam. (R_f = 0.42, 30% EtOAc/Hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 8.13 (d, J = 8.4 Hz, 1H), 7.46 (d, J = 7.8 Hz, 1H), 7.26 (t, J = 7.8 Hz, 1H), 7.19 (d, J = 7.2 Hz, 1H), 6.97 (s, 1H), 6.59 (s, 1H), 5.38-5.32 (m, 1H), 4.94-4.91 (m, 2H), 4.21 (dd, J = 13.2, 6.0 Hz, 1H), 4.07 (apparent dq, J = 11.4, 4.2 Hz, 1H), 3.99-3.93 (m, 5H), 3.87 (dd, J = 10.8, 3.0 Hz, 1H), 3.84 (s, 3H), 2.91 (t, J = 12.0 Hz, 1H), 2.33 (dd, J = 12.6, 4.2 Hz, 1H), 0.97 (s, 9H), 0.15 (s, 3H), 0.13 (s, 3H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 171.2, 167.9, 151.9, 18.6, 136.9, 130.6, 128.8, 124.3, 123.0, 120.6, 119.0, 115.5, 110.3, 79.1, 78.8, 66.4, 65.8, 63.7, 53.4, 53.0, 36.6, 26.0, 18.4, -5.3, -5.4; IR (thin film, cm^{-1}): ν_{max} = 2954, 2929, 2857, 1739, 1456, 1440, 1334, 1258, 1222, 1140, 1121, 1088, 1059, 837, 779, 766, 748; HRMS calc. for $\text{C}_{27}\text{H}_{37}\text{NO}_8\text{Si}$ = 531.2288, found 531.2290.

Compound **31f**

Indole-2-carboxaldehyde (0.118 g, 0.81 mmol), was placed into a slurry of NaH (60% in oil, 0.04 g, 0.977 mmol) in DMF (5 mL). To this solution was added benzoyl chloride (0.14 mL, 0.977 mmol) and stirred for 16 h. The reaction was added to H_2O and extracted with EtOAc (4x),

the combined organic extracts were washed with H₂O (2x), brine, dried over MgSO₄ and filtered. Purification by flash column chromatography (5% EtOAc/Hexanes) yielded 0.15 g (60%) of **31f** as a white foam. (*R*_f = 0.49, 30% EtOAc/Hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 10.43 (s, 1H), 8.16 (d, *J* = 8.0 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.52-7.40 (m, 7H), 7.31 (t, *J* = 7.6 Hz, 1H), 5.53 (s, 2H); ¹³C-NMR (100 MHz, CDCl₃): δ = 184.0, 151.2, 137.7, 137.7, 134.2, 129.1, 128.9, 128.8, 128.5, 127.6, 124.1, 123.3, 117.2, 116.2, 69.8; IR (thin film, cm⁻¹): ν_{max} = 3125, 3033, 1740, 1670, 1535, 1417, 1326, 1219, 1126, 1088, 743; HRMS calc. for C₁₇H₁₃NO₃ = 279.0895, found 279.0901.

Compound **30e**

Sn(OTf)₂ (0.18 g, 0.44 mmol) was placed in a flask equipped with a stir bar and purged with argon. Cyclopropane **32e** (0.20 g, 0.75 mmol) and aldehyde **31f** (0.19 g, 0.68 mmol) were dissolved in 1 mL CH₂Cl₂ and added to the flask.^{13c} After stirring for 27 h the reaction was passed through a small plug of silica gel and the resultant solution was concentrated *in vacuo*. Purified by flash column chromatography (4-8% EtOAc/Hexanes) yielded 0.18 of **30e** (54%) as the only diastereomer isolated. Other minor isomers were also present but were separated and discarded. (*R*_f = 0.38, 20% EtOAc/hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 7.96 (d, *J* = 8.0 Hz, 1H), 7.49-7.47 (m, 1H), 7.43-7.34 (m, 5H), 7.24-7.16 (m, 5H), 6.88 (d, *J* = 7.6 Hz, 2H), 6.80 (s, 1H), 6.61 (s, 1H), 6.06 (ddd, *J* = 16.8, 10.4, 6.4 Hz, 1H), 5.45 (d, *J* = 17.2 Hz, 1H), 5.31 (d, *J* = 10.0 Hz, 1H), 5.19 (s, 2H), 4.83 (d, *J* = 12.0 Hz, 1H), 4.48 (apparent q, *J* = 10.0 Hz, 1H), 4.32 (d, *J* = 12.4 Hz, 1H), 3.79 (s, 3H), 2.80 (t, *J* = 12.4 Hz, 1H), 2.48 (dd, *J* = 13.6, 4.8 Hz, 1H); ¹³C-NMR (100 MHz, CDCl₃): δ = 171.1, 168.1, 151.3, 138.7, 136.5, 136.0, 135.0, 134.6, 128.8, 128.6, 128.5, 128.2, 128.2, 128.1, 124.4, 123.0, 120.7, 118.1, 115.8, 110.0, 79.3, 79.0, 68.5, 67.6, 65.7, 52.9, 41.0; IR (thin film, cm⁻¹): ν_{max} = 3115, 3034, 2953, 1734, 1454, 1400, 1325, 1268, 1215, 1079, 748; HRMS calc. for C₃₂H₂₉NO₇ = 539.1944, found 539.1957.

Compound **47b**

To **30e** (0.18 g, 0.33 mmol) dissolved in THF (2 mL), H₂O (2 mL), and acetone (1 mL) was added methanesulfonamide (0.031 g, 0.33 mmol), NMO (0.043 g, 0.37 mmol) and a large crystal of OsO₄. The reaction was monitored by TLC and upon completion sodium sulphite was added to the reaction and stirred for a further 30 min. The reaction was then filtered through Celite and the volatile components were removed *in vacuo*. The remaining solution was added to brine and extracted with EtOAc (4x), dried over MgSO₄, filtered and concentrated under reduced pressure to yield a white foam (*R*_f = 0.41, 100% EtOAc). The resultant foam was dissolved in THF (2 mL) and H₂O (2 mL) and NaIO₄ (0.078 g, 0.37 mmol) was added. The reaction was monitored by TLC and upon completion 8 mL of EtOH was added and the solution filtered through Celite rinsing the filter cake with 3 mL of EtOH (*R*_f = 0.77, 100% EtOAc). NaBH₄ (0.017 g, 0.44 mmol) was added to the filtrate and the solution stirred at room temperature. TLC monitoring showed consumption of the aldehyde after 45 min at which time the reaction was quenched with drop wise addition of a saturated aqueous solution of NH₄Cl. The organic solvents were removed under reduced pressure and the remaining solution added to H₂O and extracted with EtOAc (4x). The combined organic extracts were washed with brine, dried over MgSO₄, filtered and concentrated *in vacuo*. Purification by flash column chromatography (30-40% EtOAc/Hexanes) yielded 0.10 g of the primary alcohols **47b** as a white foam (70% over 3 steps). (*R*_f = 0.50, 80% EtOAc/hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 7.94 (d, *J* = 8.0 Hz, 1H), 7.48-7.46 (m, 1H), 7.43-7.35 (m, 5H), 7.25-7.14 (m, 5H), 6.86 (d, *J* = 7.2 Hz, 2H), 6.80 (s, 1H), 6.61 (s, 1H), 5.21 (AB, *J* = 12.4 Hz, 2H), 4.82 (d, *J* = 12.0 Hz, 1H), 4.30 (d, *J* = 12.0 Hz, 1H), 4.19 (dddd, *J* = 10.8, 5.6, 5.2, 3.2 Hz, 1H), 3.98-3.95 (m, 1H), 3.83-3.81 (m, 1H), 3.77 (s, 3H), 2.92 (dd, *J* = 8.8, 7.6 Hz, 1H), 2.32 (dd, *J* = 13.2, 4.8 Hz, 1H), 2.18 (broad s, 1H); ¹³C-NMR (100 MHz, CDCl₃): δ = 171.1, 168.2, 151.3, 138.3, 136.4, 134.9, 14.5, 128.6, 128.6, 128.5, 128.2, 128.1, 124.5, 123.0, 120.7, 115.8, 109.8, 79.3, 78.6, 68.6, 67.6, 65.7, 63.6, 52.9, 36.7; IR (thin film, cm⁻¹): ν_{max} = 3536, 3034, 2952, 2880, 2250, 1734, 1455, 1397, 1325, 1267, 1215, 1120, 1079, 910, 735; HRMS calc. for C₃₁H₂₉NO₈ = 543.1893, found 543.1883.

Compound **48b**

Alcohol **47b** (0.21 g, 0.39 mmol) was dissolved in CH₂Cl₂ (5 mL) followed by the successive addition of TBSCl (0.077 g, 0.50 mmol) and imidazole (0.034 g, 0.50 mmol). The reaction was complete after 6 h at which point it was added to H₂O and extracted with CH₂Cl₂ (2x). The combined organic extracts washed with brine, dried over MgSO₄, and filtered. Purification *via* flash column chromatography (5% EtOAc/Hexanes) yielded 0.25 g (98%) of **48b** as a white foam. (*R*_f = 0.50, 30% EtOAc/Hexanes); ¹H-NMR (400 MHz, CDCl₃): δ = 7.99 (d, *J* = 7.2 Hz, 1H), 7.47-7.33 (m, 6H), 7.24-7.15 (m, 5H), 6.99 (s, 1H), 6.87 (d, *J* = 6.8 Hz, 2H), 6.65 (s, 1H), 5.21 (AB, *J* = 12.0 Hz, 2H), 4.79 (d, *J* = 12.0 Hz, 1H), 4.37 (d, *J* = 12.0 Hz, 1H), 4.09 (apparent dq, *J* = 11.6, 4.0 Hz, 1H), 3.98 (dd, *J* = 11.2, 4.0 Hz, 1H), 3.86 (dd, *J* = 11.6, 4.0 Hz, 1H), 3.77 (s, 3H), 2.93 (dd, *J* = 8.0, 7.2 Hz, 1H), 2.32 (dd, *J* = 12.8, 4.4 Hz, 1H), 0.95 (s, 9H), 0.15 (s, 3H), 0.12 (s, 3H); ¹³C-NMR (100 MHz, CDCl₃): δ = 171.1, 168.1, 151.3, 139.0, 136.7, 135.1, 134.6, 128.9, 128.6, 128.6, 128.5, 128.2, 128.1, 124.3, 123.0, 120.7, 115.7, 110.3, 79.0, 78.8, 68.5, 67.5, 65.8, 63.7, 52.9, 36.5, 26.0, 18.4, -5.3, -5.4; IR (thin film, cm⁻¹): ν_{max} = 2953, 2928, 2857, 1736, 1455, 1325, 1259, 1214, 1119, 1079, 1029, 836, 747, 697; HRMS calc. for C₃₇H₄₃NO₈Si = 657.2758, found 657.2744.

Compound **49b**

To compound **48b** (0.23 g, 0.35 mmol) in THF (5 mL) was added 0.023 g of 10% Pd/C. The reaction vessel was purged with hydrogen gas (4x) then placed under 1 atmosphere of hydrogen for 3 h after which time TLC analysis showed consumption of the diester. The solution was filtered through a pad of Celite and the Celite was rinsed with THF (5 mL). Triphenylphosphine

(0.184g, 0.74 mmol) and allyl alcohol (0.048 mL, 0.70 mmol) were added to the solution along with a magnetic stir bar. With stirring DIAD (0.14 mL, 0.70 mmol) was added to the solution and stirring was continued for 50 min after which time a further 2 equiv. of all the reagents were added. After 40 min TLC analysis showed consumption of the acid substrate. Purification by flash column chromatography (5% EtOAc/Hexanes) yielded 0.144 g of a 3:1 mixture of **49b** (67%) and **F** (22%) as a white foam. (R_f = 0.50, 30% EtOAc/Hexanes) This reaction was conducted 3 times and the ratio of the two products ranged from 3:1-1:1.2, **49b**:**F**. The 1:1.2 mixture of products was used for characterization; ^1H -NMR (400 MHz, CDCl_3), **49b** denoted by *; δ = 9.23 (br s, 1H*), 8.06 (d, J = 8.0 Hz, 1H), 7.58 (d, J = 7.6 Hz, 1H), 7.50 (d, J = 8.0 Hz, 1H*), 7.39-7.30 (m, 2H, 1H*), 7.12 (t, J = 7.6 Hz, 1H*), 7.03 (t, J = 7.6 Hz, 1H*), 6.63 (d, J = 0.8 Hz, 1H), 6.33 (d, J = 1.2 Hz, 1H*), 5.93 (s, 1H*), 5.50 (s, 1H), 5.47-5.37 (m, 1H*), 5.00 (s, 1H*), 4.96 (d, J = 6.0 Hz, 1H*), 4.42-4.36 (m, 1H), 4.24-4.04 (m, 4H*), 3.88-3.84 (m, 1H*), 3.84 (s, 3H*), 3.78 (s, 3H), 3.63 (A/B of ABX, J = 10.8, 4.4 Hz, 1H), 3.57 (A/B of ABX, J = 10.8, 4.4 Hz, 1H), 3.04 (dd, J = 13.2, 11.2 Hz, 1H*), 2.92 (dd, J = 13.2, 6.4 Hz, 1H), 2.38-2.31 (m, 1H, 1H*), 1.00 (s, 9H*), 0.75 (s, 9H), 0.24 (s, 3H*), 0.23 (s, 3H*), -0.4 (s, 3H), -0.11 (s, 3H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 170.9, 169.3, 167.5, 167.2, 140.0, 135.8, 135.7, 134.9, 130.9, 120.6, 128.1, 124.9, 124.7, 121.7, 121.6, 120.4, 119.4, 118.7, 114.3, 111.0, 104.1, 100.6, 84.4, 78.9, 78.8, 69.3, 66.5, 66.3, 64.2, 63.9, 53.5, 53.2, 34.7, 33.5, 26.1, 25.7, 18.8, 18.2, -5.1 (2C), -5.4, -5.6; HRMS calc. for $\text{C}_{22}\text{H}_{30}\text{NO}_5\text{Si}^{1+}$ = 416.1893, found 416.1889; HRMS calc. for $\text{C}_{25}\text{H}_{35}\text{NO}_6\text{Si}$ = 473.2234, found 473.2244.

Compound **I**

Compound 51: To compound **49c** (0.098 g, 0.16 mmol) in THF (4 mL) was added TBAF (1 M in THF, 0.17 mmol, 0.17 mmol) with stirring under argon. The reaction was monitored by TLC and upon completion the solution was concentrated *in vacuo*, the clear residue was pre-adsorbed onto silica and purified by flash column chromatography (20-50% EtOAc/Hexanes) to yield alcohol **I** (0.072 g, 90%) as a clear colorless oil. (R_f = 0.17, 50% EtOAc/Hexanes); ^1H -

NMR (600 MHz, CDCl₃): δ = 8.09 (d, J = 8.4 Hz, 1H), 7.70 (d, J = 8.4 Hz, 2H), 7.41 (d, J = 8.0 Hz, 1H), 7.27 (ddd, J = 7.2, 7.2, 1.2 Hz, 1H), 7.20-7.15 (m, 3H), 6.80 (s, 1H), 6.74 (s, 1H), 5.45 (dddd, J = 6.0, 6.0, 10.4, 16.4 Hz, 1H), 4.98 (dq, J = 17.2 Hz, 1.2 Hz, 1H), 4.89 (dd, J = 10.4 Hz, 0.8 Hz, 1H), 4.28 (m, 1H), 4.21 (dddd, J = 11.2, 4.4, 4.4, 2.8 Hz, 1H), 4.10-4.05 (m, 1H), 3.95 (dd, J = 12.4, 2.8 Hz, 1H), 3.87 (s, 3H), 3.77 (dd, J = 12.0, 4.8 Hz, 1H), 2.92 (dd, J = 13.2, 11.6 Hz, 1H), 2.37 (dd, J = 13.2, 4.8 Hz, 1H), 2.30 (s, 3H); ¹³C-NMR (100 MHz, CDCl₃): δ = 170.7, 167.7, 144.8, 138.8, 136.9, 135.4, 130.8, 129.7, 129.1, 126.6, 124.8, 123.6, 121.1, 118.9, 114.9, 111.7, 78.7, 77.6, 66.6, 66.2, 63.3, 53.3, 35.9, 21.5; IR (thin film, cm⁻¹): ν_{max} = 3450, 2954, 2928, 2881, 1734, 1597, 1451, 1372, 1276, 1173, 914, 733, 672, 579; HRMS calc. for C₂₆H₂₇NO₈S = 513.1445, found 513.1457.

Compound **51**

Alcohol **I** (0.072 g, 0.14 mmol) and triethylamine (0.021 mL, 0.15 mmol) were dissolved in THF (4 mL) followed by the addition of 4-bromobenzoyl chloride (0.034 g, 0.15 mmol). The solution was stirred at room temperature for 16 hr then added to H₂O and extracted with EtOAc (3x). The combined organic extracts were washed with brine and dried over MgSO₄. Purification by flash column chromatography (20-50% EtOAc/Hexanes) yielded 0.055 g (56%) of **51** as a white solids as well as 0.023 g of alcohol **I**. (R_f = 0.61, 50% EtOAc/Hexanes) The solid was recrystallized from CH₂Cl₂ and hexanes to obtain crystals suitable for X-ray crystallography; **51**: ¹H-NMR (600 MHz, CDCl₃): δ = 8.06 (d, J = 8.0 Hz, 1H), 7.93-7.90 (m, 2H), 7.68 (d, J = 8.4 Hz, 2H), 7.58-7.55 (m, 2H), 7.28-7.24 (m, 2H), 7.19-7.15 (m, 1H), 7.12 (d, J = 8.4 Hz, 2H), 6.77 (s, 1H), 6.75 (s, 1H), 5.51 (dddd, J = 16.4, 10.8, 6.0, 6.0 Hz, 1H), 5.02 (dq, J = 17.2, 1.2 Hz, 1H), 4.95-4.92 (m, 1H), 4.68 (dd, J = 12.0, 3.2 Hz, 1H), 4.52 (dd, J = 12.0, 4.8 Hz, 1H), 4.46-4.40 (m, 1H), 4.33 (ddt, J = 12.8, 6.0, 1.2 Hz, 1H), 4.14 (ddt, J = 12.8, 6.0, 1.2 Hz, 1H), 3.89 (s, 3H), 2.96 (dd, J = 12.8, 11.6 Hz, 1H), 2.51 (dd, J = 13.2, 4.8 Hz, 1H), 2.27 (s, 3H); ¹³C-NMR (100 MHz, CDCl₃): δ = 170.6, 167.4, 165.6, 144.8, 138.6, 137.0, 135.3, 131.8, 131.3, 129.7, 129.1, 128.7, 128.4, 126.6, 124.9, 123.7, 121.0, 118.9, 114.9, 111.8, 79.0, 76.1, 66.7, 65.9, 65.0, 53.4, 36.7,

21.5; IR (thin film, cm^{-1}): ν_{max} = 2955, 2927, 2888, 1734, 1591, 1451, 1375, 1269, 1175, 1101, 1069, 1012, 755, 580; HRMS calc. for $\text{C}_{33}\text{H}_{30}\text{BrNO}_9\text{S}$ = 695.0812, found 695.0825; mp = 150-153 °C.

Compound **54**

Alcohol **53** (0.27 g, 0.53 mmol) was dissolved in CH_2Cl_2 (6 mL) followed by the successive addition of TBDPSCl (0.15 mL, 0.59 mmol) and imidazole (0.04 g, 0.59 mmol). The reaction was complete after 28 h at which point it was added to H_2O and extracted with CH_2Cl_2 (2x). The combined organic extracts was washed with brine, dried over MgSO_4 , and filtered. Purification *via* flash column chromatography (5% EtOAc/Hexanes) yielded 0.38 g (97%) of **54** as a white foam. (R_f = 0.50, 30% EtOAc/Hexanes); ^1H -NMR (600 MHz, CDCl_3): δ = 8.07 (d, J = 8.4 (Hz, 1H), 7.75 (dt, J = 8.4, 1.2 Hz, 4H), 7.67 (d, J = 7.8 Hz, 2H), 7.45-7.42 (m, 2H), 7.40-7.38 (m, 4H), 7.34 (d, J = 7.8 Hz, 1H), 7.25 (dt, J = 8.4, 1.2 Hz, 1), 7.17 (t, J = 7.8 Hz, 1H), 7.13 (d, J = 7.8 Hz, 2H), 6.80 (s, 1H), 6.14 (s, 1H), 5.09 (d, J = 4.8 Hz, 1H), 4.29 (X of ABX, apparent q, J = 5.4 Hz, 1H), 4.02 (AB of ABX, J = 10.8, 5.4 Hz, 1H), 3.29 (s, 3H), 2.29 (s, 3H), 1.73 (s, 3H), 1.38 (s, 3H), 1.12 (s, 9H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 170.2, 144.6, 137.4, 136.9, 135.7, 135.6, 133.4, 129.7, 129.6, 129.5, 127.7, 126.8, 124.8, 123.8, 121.0, 116.7, 115.1, 112.5, 94.9, 85.1, 84.5, 82.8, 63.4, 52.2, 27.8, 26.8, 26.0, 21.5, 19.3; IR (thin film, cm^{-1}): ν_{max} = 3072, 2994, 2933, 2858, 1757, 1735, 1376, 1217, 1188, 1151, 1091, 733; HRMS (TOF MS ES+) calc. for $\text{C}_{41}\text{H}_{45}\text{NNaO}_8\text{SSi}$ = 762.2533, found 762.2556; $[\alpha]^{25}_{\text{D}}$ = +68 ° (c 2.5, MeOH).

Compound **J**

To compound **54** (0.33 g, 0.48 mmol) in MeOH (16 mL) was added NH₄Cl (0.11 g, 1.91 mmol) and magnesium turnings (0.24 g, 9.56 mmol). The reaction was monitored by TLC (20% EtOAc/Hexanes) and upon completion was transferred to a separatory funnel containing saturated aqueous NH₄Cl. Extraction with EtOAc (3x), followed by washing the combined organic extracts with brine (1x), drying over MgSO₄ and concentration under reduced pressure yielded 0.27 g of crude oil. Purification *via* flash column chromatography (3% EtOAc/Hexanes) yielded 0.20 g of **J** (71%) as a white foam. (*R*_f = 0.32, 20% EtOAc/Hexanes); ¹H-NMR (600 MHz, CDCl₃): δ = 8.36 (broad s, 1H), 7.77-7.74 (m, 4H), 7.53 (d, *J* = 7.2 Hz, 1H), 7.47-7.45 (m, 2H), 7.42-7.39 (m, 4H), 7.14 (d, *J* = 7.8 Hz, 1H), 7.12-7.09 (m, 1H), 7.06-7.03 (m, 1H), 6.43 (d, *J* = 1.8 Hz, 1H), 5.33 (s, 1H), 5.03 (d, *J* = 3.6 Hz, 1H), 4.29 (X of ABX, apparent q, *J* = 4.8 Hz, 1H), 4.05 (AB of ABX, *J* = 10.8, 5.4 Hz, 2H), 3.25 (s, 3H), 1.68 (s, 3H), 1.35 (s, 3H), 1.14 (s, 9H); ¹³C-NMR (100 MHz, CDCl₃): δ = 170.8, 135.9, 135.9, 135.8, 133.6, 133.4, 133.0, 130.1, 128.3, 128.1, 128.0, 122.2, 120.8, 119.9, 117.3, 111.1, 100.7, 94.0, 85.3, 85.3, 84.9, 63.7, 52.7, 27.9, 27.1, 25.9, 19.6; IR (thin film, cm⁻¹): ν_{max} = 3453, 3050, 2932, 2858, 1735, 1473, 1457, 1377, 1338, 1213, 1113, 869, 824, 789, 739; HRMS calc. for C₃₄H₃₉NO₆Si = 585.2647, found 585.2568; [α]_D²⁵ = + 32 ° (c 2.3, MeOH).

Compound **55**

To indole **J** (0.14 g, 0.25 mmol) in DMF (18 mL) was added DBU (0.11 mL, 0.74 mmol) and stirred for 3 h after which the reaction was added to a separatory funnel containing saturated aqueous NH₄Cl. This solution was extracted with Et₂O (4x), the combined organic extracts were washed with brine (1x), dried over MgSO₄ and concentrated under reduced pressure. Purification *via* flash column chromatography (3% EtOAc/Hexanes) yielded compound **55** (0.12 g, 84%). The racemate yielded crystals suitable for single crystal X-ray diffraction crystallography (slow evaporation of CH₂Cl₂ from hexanes). (*R*_f = 0.50, 20% EtOAc/Hexanes); **55**: ¹H-NMR (600 MHz, CDCl₃): δ = 8.02 (d, *J* = 7.8 Hz, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.46 (dd, *J* = 8.4, 1.8 Hz, 2H), 7.37-7.24 (m, 8H), 6.50 (s, 1H), 5.34 (s, 1H), 5.00 (d, *J* = 1.2 Hz, 1H), 4.51 (ddd, *J* = 7.8,

5.4, 1.8 Hz, 1H), 3.44 (dd, $J = 10.8, 5.4$ Hz, 1H), 3.04 (dd, $J = 10.8, 7.8$ Hz, 1H), 1.69 (s, 3H), 1.66 (s, 3H), 0.88 (s, 9H); ^{13}C -NMR (100 MHz, CDCl_3): $\delta = 167.0, 139.1, 135.4, 135.2, 134.6, 132.8, 132.4, 130.7, 129.7, 129.7, 127.6, 127.6, 124.9, 124.9, 121.7, 116.9, 114.6, 104.2, 98.0, 87.3, 86.2, 79.7, 63.0, 26.9, 26.5, 25.0, 19.0$; IR (thin film, cm^{-1}): $\nu_{\text{max}} = 3073, 2933, 2859, 1753, 1473, 1453, 1385, 1114, 1069, 824, 750$; HRMS calc. for $\text{C}_{33}\text{H}_{35}\text{NO}_5\text{Si} = 553.2284$, found 553.2296; $[\alpha]_{\text{D}}^{25} = -38^\circ$ (c 2.1, MeOH).

Compound **56**

A DMDO solution in acetone (0.1 M, 2.16 mL, 0.22 mmol) was added to a solution of compound **55** (0.012g, 0.022 mmol) in acetone and stirred. The reaction was monitored by TLC (EtOAc/Hexanes) and upon completion the solvent was removed under reduced pressure with light heating to yield compound **56** (0.013 g, quant.) as a clear oil. This compound was not stable to purification *via* flash column chromatography and was characterized in its crude form. ($R_f = 0.41$, 20% EtOAc/Hexanes); ^1H -NMR (600 MHz, CDCl_3): $\delta = 7.79$ (d, $J = 7.2$ Hz, 1H), 7.52-7.50 (m, 3H), 7.44 (d, $J = 7.8$ Hz, 2H), 7.40-7.35 (m, 3H), 7.31 (t, $J = 7.8$ Hz, 2H), 7.26 (t, $J = 7.8$ Hz, 2H), 7.15 (t, $J = 7.8$ Hz, 1H), 5.02 (d, $J = 3.0$ Hz, 1H), 4.86 (s, 1H), 4.82 (s, 1H), 4.34-4.33 (X of ABX, m, 1H), 3.59 (A/B of ABX, $J = 12.0, 4.8$ Hz, 1H), 3.46 (A/B of ABX, $J = 12.0, 4.8$ Hz, 1H), 1.67 (s, 3H), 1.65 (s, 3H), 0.81 (s, 9H); ^{13}C -NMR (100 MHz, CDCl_3): $\delta = 167.4, 139.7, 135.5, 135.4, 133.0, 132.6, 130.7, 130.2, 129.8, 129.7, 127.7, 127.6, 126.2, 124.8, 118.0, 116.3, 97.2, 87.2, 85.4, 82.8, 82.3, 62.8, 57.5, 27.2, 26.4, 26.2, 18.9$; IR (thin film, cm^{-1}): $\nu_{\text{max}} = 3060, 3028, 2880, 1734, 1452, 1436, 1261, 1212, 1121, 1029, 970, 752$; HRMS could not be obtained on this compound on multiple attempts.

Compound **57**Compound **58**

A DMDO solution in acetone (0.065 M, 18 mL, 1.17 mmol) was added to a solution of compound **55** (64.9 mg, 0.117 mmol) in acetone and stirred. The reaction was monitored by TLC (EtOAc/Hexanes) and upon consumption of **55**, CSA (26.5 mg, 0.117 mmol) was added to the reaction and stirred for a further 1.25 h. The solvent was removed under reduced pressure with light heating and the resultant residue dissolved in EtOAc, added to H₂O and extracted with EtOAc (3x). The combined organic extracts were washed with brine (1x), dried over MgSO₄ and concentrated to yield a mixture of compound **57** and compound **58**. The crude extract was taken up in EtOAc (3.5 mL) followed by the addition of IBX (66 mg, 0.24 mmol) and heated to reflux for 16 h. The reaction was filtered through Celite, and purified *via* flash column chromatography (8-12% EtOAc/Hexanes) to yield compound **58** (41.3 mg, 60%) as a colorless oil; **57**: ¹H-NMR (400 MHz, CDCl₃): δ = 7.61 (d, J = 8.0 Hz, 1H), 7.55 (d, J = 7.2 Hz, 2H), 7.48 (d, J = 7.6 Hz, 2H), 7.40-7.29 (m, 8H), 7.16 (t, J = 7.6 Hz, 1H), 5.58 (broad s, 1H), 4.97 (d, J = 4.4 Hz, 1H), 4.24 (s, 1H), 4.17 (X of ABX, apparent q, J = 4.0 Hz, 1H), 4.09, (broad s, 1H), 3.74 (A/B of ABX, J = 11.6, 3.2 Hz, 1H), 3.68 (A/B of ABX, J = 11.6, 3.6 Hz, 1H), 2.86 (br s, 1H), 1.63 (s, 6H), 0.80 (s, 9H); ¹³C-NMR (100 MHz, CDCl₃): δ = 166.9, 136.7, 135.5, 134.5, 133.1, 132.7, 129.8, 129.7, 127.6, 127.6, 127.6, 125.8, 125.4, 118.9, 115.9, 97.4, 94.7, 86.6, 84.7, 84.1, 70.5, 62.9, 29.7, 27.2, 26.4, 26.2, 18.9; R_f = 0.05, 20% EtOAc/Hexanes. **58**: ¹H-NMR (400 MHz, CDCl₃): δ = 7.80 (d, J = 8.0 Hz, 1H), 7.69 (d, J = 7.6 Hz, 1H), 7.57-7.49 (m, 3H), 7.39-7.28 (m, 8H), 7.15 (t, J = 7.6 Hz, 1H), 5.12 (d, J = 4.4 Hz, 1H), 4.46 (s, 1H), 4.07 (X of ABX, m, 1H), 3.74 (broad s, 1H), 3.68-3.60 (AB of ABX, m, 2H), 1.63 (s, 6H), 0.70 (s, 9H); ¹³C-NMR (100 MHz, CDCl₃): δ = 193.3, 169.7, 149.6, 137.6, 135.4, 135.4, 132.8, 132.4, 129.7, 129.5, 127.7, 127.5, 125.8, 125.5, 124.7, 119.6, 117.2, 97.3, 89.8, 86.6, 84.5, 84.0, 91.9, 29.7, 27.1, 26.8, 26.4, 26.1, 18.8; IR (thin film, cm⁻¹): ν_{max} = 3395, 3072, 3052, 2993, 2932, 2893, 2858, 1734, 1605, 1472, 1429, 1385, 1376, 1363, 1303, 1265, 1215, 1170, 1150, 1112, 1106, 1073, 975, 945, 868; HRMS (TOS MS ES⁺) calc. for C₃₃H₃₅NNaO₇Si = 608.2081, found 608.2053; R_f = 0.31, 30% EtOAc/Hexanes.

AL-9-83
STANDARD IN OBSERVE

exp7 s2pu1

[illegible]

20

AK-9-205-pure
STANDARD 1H ONSERVE

exp1 a2pul

[illegible]

1H-10-15
STANDARD IN OBSERVE

```

exp1 s2jml
=====
SAMPLE
date Feb 26 2008 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
pr/medda/1antisl-hat 0.008
e A file/1H-10-15/- p050 14.800
PROTON.FID alfa 20.000
=====
ACQUISITION
nu 6403.0 il n
at 4.000 in n
ap 51214 dp y
rs not used ha
ha 16 PROCESSING
al 1.000 fa not used
st 8 DISPLAY
=====
PARAMETER
tu 8 ap -0.5
tu 3600.2 wp
tu 800.5
sfreq 600.088 xfp 0
tofr 417.3 xfp -139.4
tpr 87 lp -116.4
pw 7.400 PL02
=====
DECOUPLER
an wc 370
an C13 ac 0
an 0 vs 63
an nm th 73
an c al cdc ph
an 44
anr 15900

```

AK-10-15
STANDARD IN OBSERVE

exp1 s2jeol

SAMPLE		SPECIAL	
date	Feb 26 2008	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
rr/avedia/instain-hat	0.008		
s A fidh/AK-10-15/- p90	12.000		
CARBON.fid	alifa	20.000	
ACQUISITION		FLAGS	
sw	25123.6 il	n	
at	1.199 in	n	
ap	60270 dp	y	
ps	13800 ha	nm	
ha	64	PROCESSING	
al	1.000 1b	not used	
st	256 fa	not used	
ct	256	DISPLAY	
TRANSMITTER		SP	
tn	C13 wp	20133.9	
sfreq	100.613 xrl	9268.0	
toif	1500.8 xfp	7746.3	
tpwr	59 rp	169.2	
pw	6.000 lp	-318.7	
DECOUPLER		PLOT	
dn	kl wc	370	
dcf	0 sc	0	
dm	YY vs	169	
dms	w th	12	
dpr	40 ai	no ph	
dnt	8900

MS-10-67
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE		SPECIAL	
date	Mar 10 2008	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	20	
rr/aveda/instain-hat		0.008	
n A fids/AR-10-67/-	pd90	13.500	
PROTON FID	alifa	20.000	
ACQUISITION			
sw	6402.0	il	n
at	4.000	in	n
ap	51216	dp	y
fs	not used	ha	nm
he	16	PROCESSING	
hl	1.000	fa	not used
st	8	DISPLAY	
ct	TRANSMITTER	wp	3602.9
ln	kl xel	wp	3706.1
sfq	400.088	xfp	2904.6
tof	417.3	xp	-144.4
tpw	58	lp	-102.3
pw	6.750	PILOT	
DECOUPLER			
dc	C13	sc	0
dcf	0	vs	143
dm	nm	th	38
dms	c	ai	cdc ph
dpr	44		
dmf	18500

EX-10-67
STANDARD IN OBSERVE

exp1 s2jml

```

SAMPLE
date Mar 10 2008 temp not used
solvent cdcl3 gain not used
file /home/data/mw-spin not used
pr/medda/Isatin-hat 0.008
s 2 file/EX-10-67/- p090 11.100
CARDS: f1d s1fa 20.000

ACQUISITION
ns 25128.4 il n
st 1.199 in n
sp 60370 dp y
ss 13800 ha
sa 64 1b PROCESSING 1.00
sl 2.000 1b not used
sc 2000 fa
ct 2000 display -4.8

TRANSMITTER
tm C13 wp 25139.8
sfreq 100.613 xrl 9368.4
tofr 1590.8 xrp 7746.3
tpr -168.8
pw 5.950 lp -348.3

DECOUPLER
dm el wc 370
dof 0 ac 0
dm Y77 vs 245
dms w th 17
dpr 40 sl no ph
dmf 8900

```

AK-9-185 pure

exp1 PROTON

```

SAMPLE      PREPARATION
date  Jan 18 2008  antmode  n
solvent  cdcl3  wet  n
file  /home/data/ha-  SPECIAL
xr/Avddia/Isatisin- temp  25.0
e A fidu/AK-9-185-- gain  not used
pure-01/PROTON_0101- spin  20
-fid  hat  0.008
ACQUISITION  pps0  11.300
sw  9590.0  alfa  6.600
at  1.708
ap  32768  il  FLAG
B  5000  in  n
ha  31  dp  n
dl  1.000  ha  PROCESSING
at  8  fa  DEC used
CT TRANSMITTER  xl  ap  DISPLAY
ln  4.2
afreq  599.406  wp  5396.1
toif  599.4  xrl  5540.0
tpr  57  xfp  4351.7
pw  5.650  rp  -57.4
pc  DISCOUPLER  lp  -24.0
dn  CL3  PL0Z
dof  0  wc  370
dm  nm  sc  0
decwave  W60_hox  vs  74
dpr  44  th  56
def  30088  al  cdc  ph

```

NAME: 1S
STANDARD IN OBSERVE

exp1 s2jeu1

SAMPLE		SPECIAL	
date	Jan 18 2008	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
rr/avedia/1satiaia-hat		0.008	
s A fidu/AE-9-185/-	pp90	12.000	
CARBON.fid	alfa	20.000	
ACQUISITION			
sw	25123.6	il	n
at	1.199	in	n
sp	60270	dp	y
fs	13800	hs	nm
ha	64	hb	PROCESSING
sl	2.000	lb	not used
st	1.000	fn	not used
ct	1000	fn	DISPLAY
TRANSMITTER			
tn	C13	wp	22128.3
sfreq	100.613	xrl	9268.0
toif	1560.8	xrp	7746.3
tpwr	59	rp	-173.9
pw	6.000	lp	-334.9
DECOUPLER			
dn	kl	wc	370
dcf	0	sc	0
dm	YY	vs	267
dms	w	th	13
dpr	40	ai	no ph
dai	8900

MS-179-46-40
STANDARD IN OBSERVE

exp1 s2jw1

SAMPLE		SPECIAL	
date	Oct 7 2007	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	spin	20
rr/meds/spectra	- hat	0.008	
for PSAL-8-179-F6	- p40	14.800	
8-80/PROTON.fid	alfa	20.000	
ACQUISITION			
ns	6402.0	il	n
st	4.000	in	n
sp	51216	dp	y
fs	not used	ha	nm
hs	16	PROCESSING	
sl	1.000	fa	not used
st	8	DISPLAY	
ct	TRANSMITTER	8	8P
tn	3589.2	wp	3589.2
sfreq	400.088	xcp	3705.0
tozf	417.3	xp	2904.6
tpwr	57	lp	-146.8
pw	7.400	PILOT	-102.7
DECOUPLER			
dn	C13	ac	370
dozf	0	vs	0
dm	nm	th	79
dms	c	ai	2
dpr	44		
dmf	15900

23

MS-179-F66-80
STANDARD IN OBSERVE

exp1 s2jeol

SAMPLE		SPECIAL	
date	Oct 7 2007	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
rr/avedia/spectra	- bat	0.008	
for PS/AL-8-179-F6- p490		12.000	
8-80/CARBON.fid	alfa	20.000	
ACQUISITION			
ns	25123.6	il	n
at	1.199	in	n
sp	60270	dp	y
rs	13800	hs	nm
ha	64	lb	PROCESSING
sl	1.000	lb	1.00
st	256	fn	not used
ct	256	fn	DISPLAY
TRANSMITTER			
tn	C13	wp	22130.6
sfreq	100.613	xrl	9301.9
toif	1500.8	xrp	7746.3
tpwr	59	rp	168.0
pw	6.000	lp	-335.9
DECOUPLER			
dn	81	wc	370
dof	0	sc	0
dm	YY	vs	150
dms	w	th	20
dpr	40	ai	no ph
dai	8900

MS-179-FS-45
STANDARD IN OBSERVE

MS-179-FS1-54
STANDARD IN OBSERVE

exp1 s2jeu1

SAMPLE		SPECIAL	
date	Oct 7 2007	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	spin	not used
rr/avedia/spectra	- bat		0.008
for FS/AL-8-179-FS	- p40		12.000
2-56/CARBON-13	alfa		20.000
ACQUISITION			
ns	25123.6	il	n
at	1.199	in	n
ap	60270	dp	y
rs	13800	hs	nm
ha	64	lb	PROCESSING
sl	1.000	lb	not used
st	256	fn	not used
ct	256	fn	DISPLAY
TRANSMITTER			
tn	C13	wp	20134.7
sfreq	100.613	xrl	9271.2
toif	1250.8	xrp	7746.3
tpwr	59	rp	169.5
pw	6.000	lp	-329.5
DECOUPLER			
dn	h1	wc	370
dcf	0	sc	0
dm	YY	vs	146
dms	w	th	12
dpr	40	ai	no ph
dai	8900

MS-179-F2B-35
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE SPECIAL
date Oct 8 2007 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
rr/avedia/spectra-hat 0.008
for PS/AE-8-179-F2-pd0 14.800
8-35/PROTON.fid alfa 20.000
ACQUISITION FLAG
ms 6402.0 il n
at 4.000 in n
ap 51216 dp y
fs not used ha PROCESSING
hs 16
sl 1.000 fa not used
st 8
ct TRANSMITTER 8 4p 0.3
tx 3589.2
tx 81 xfl 3705.3
sfreq 400.088 xfp 2904.6
tofr 417.3 xpr -142.1
tpr 57 lp -97.6
pw 7.400 PLOT
pc DECOUPLER wc 370
dn C13 ac 0
dof 0 vs 81
dm nm th 4
dms c al cdc ph
dpr 44
dif 15900

MS-179-F26-35
STANDARD IN OBSERVE

exp1 s2jeol

SAMPLE		SPECIAL	
date	Oct 8 2007	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ms-spin	spin	not used
rr/avedia/spectra	- bat	0.008	
for PS/MS-8-179-F2	- p490	12.000	
9-35/autoc_090oct200-	alfa	20.000	
7/CARBON.fid	FLAG		
ACQUISITION			
sw	25123.6	in	n
at	1.199	dp	y
ap	60270	ha	um
as	13800	hb	PROCESSING
ba	1.00	ib	not used
bi	2.000	fa	not used
bt	2000	display	2.2
TRANSMITTER			
tu	C13	xrl	22130.6
tf	100.613	xfp	9266.2
to	1560.8	rfp	7746.3
tp	59	lp	163.2
pw	6.000	plot	-261.0
DECOUPLER			
dw	81	sc	370
do	0	vs	0
de	Y77	th	581
ds	w	ai	16
dsr	40		
dsf	89.00

AK-16-127 1mva 400

exp1 1H NMR

date Mar 14 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/Avedis/Isatisin- temp 25.0
 e A file/AK-16-127- gain not used
 _02/PROC01_01.fl- spin 20
 d bat 0.008
 ACQUISITION pps 9.500
 sw 6395.9 alfa 6.600
 at 2.562
 ap 32768 il FLAG
 fs 4000 in n
 ha 31 dp y
 di 1.000 ha PROCESSING
 at 8 fa
 ct TRANSMITTER 8 fa DISPLAY 1.3
 ts 395.762 wp 3586.5
 tof 395.8 xrl 3686.4
 tpr 55 xfp 2902.3
 pr 4.750 rp -20.2
 ps DECOUPLER 1p -20.1
 dn CL3 PLOT
 dof 0 wc 370
 dm nm sc 0
 deconvolve H40_hcp2 vs 87
 dpr 34 th 76
 def 29412 ai cdc ph

AE-16-127 1mva 400

exp1 CANNON

```

SAMPLE      PREPARATION
date Mar 14 2009 antmode n
solvent cdcl3 wet n
file /home/data/ha- SPECIAL
rr/Avddia/Instlin- temp 25.0
e A file/AE-16-127- gain not used
_02/CANNON_0101.fl- spin 20
d hat 0.008
ACQUISITION d hat 0.008
pw 2513.5 alfa 10.000
at 1.304
sp 6536 il FLAG
B 14000 in n
ha 4 dp Y
dl 1.000 ha nm
ct 256 lb PROCESSING 0.50
ts TRANSMITTER fn not used
in C13 DISPLAY
sfrq 100.531 sp 8.5
toF 1520.6 wp 20105.7
tpr 54 xrl 9247.1
pw 4.400 rfp 7740.0
DECOUPLER rp 76.0
dn xl lp -393.1
dof 0
dm YYY wc 370
decwave w ac 0
qpr 39 vs 1205
def 9500 th 12
ai cdc ph

```

AK-16-169 nova 400

exp1 proton

```

SAMPLE
date Mar 14 2009
solvent cdcl3
file /home/data/ha-
xr/Avddia/Isatinin-
e A file/AK-16_169-
_02/PROC01_01.f1-
ACQUISITION
nu 6395.9
at 2.562
rg 32768
Bb 4000
ha 31
dl 1.000
ct TRANSMITTER
in 8
fa 8
DISPLAY
1.1
afsq 399.762
tof 399.8
tpr 59
pw 4.750
ds DISCOUPLER
ds 1p
dsf 0
ds nm
ds wave M40_Mcp2
ds pr 34
dsf 29412
  
```

AE-16-169 1mva 400

exp1 CANNON

```

SAMPLE PREPARATION
date Mar 14 2009 antmode n
solvent cdcl3 wet n
file /home/data/ha- SPECIAL
r/Avella/Instalin- temp 25.0
s A file/AE-16-169- gain not used
_02/CANON_0101.fl- spin 20
d hat 0.008
ACQUISITION pps 8.800
sw 2513.5 alfa 10.000
at 1.304
sp 6536 il FLAG
B 14000 in n
ha 4 dp Y
sl 1.000 ha nm
ct 256 lb PROCESSING 0.50
ct TRANSMITTER fa not used
in 100.531 sp DISPLAY
sfreq 1530.6 wp 20105.7
toF 54 xrl 9348.8
tpr 4.400 rfp 7740.0
pw DECOUPLER rp 43.6
dn el lp -336.8
dof 0 PLOT
dm YYY wc 370
de wave w ac 0
qpr 39 vs 1096
def 9500 th il
ai cdc ph

```

AK-16-171
STANDARD IN OBSERVE

exp1 s2pul

SAMPLE SPECIAL
date Mar 16 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
rr/aveda/1astislin-hat 0.008
e A fids/AK-16-171- p40 13.500
/PROTON.fid alfa 20.000

ACQUISITION FLAG
aw 6402.0 il n
at 4.000 in n
ap 51216 dp y
as not used ha m
ba 16 PROCESSING
bl 1.000 fa not used
bt 8 DISPLAY -1.1
ct TRANSMITTER 8 4804.5
in xl xfl 3704.4
stfq 400.083 xfp 2904.6
toz 624.3 xp -145.6
tpr 58 lp -94.9
pw 6.750 PLOT
pc DECOUPLER wc 370
an cl3 ac 0
dof 0 vs 28
dm nm th 28
dms c al cdc ph
dpr 44
dnt 18500

AK-16-171
STANDARD IN OBSERVE

exp1 s2pul

SAMPLE		SPECIAL	
date	Mar 16 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
rr/avedia/instalin-hat		0.008	
n A fidm/AE-16-171- p40		11.100	
/CARBOR.fid	alpha	20.000	
ACQUISITION			
sw	25125.6	il	n
st	1.199	in	n
sp	60270	dp	y
fs	13800	ha	um
ba	64	PROCESSING	
sl	1.000	lb	not used
sc	1000	fs	not used
ct	128	DISPLAY	
TRANSMITTER			
tu	C13	wp	20124.7
sfreq	100.611	xrl	9372.5
tofq	1522.6	xrp	7746.2
tpwr	59	rp	127.7
pw	5.550	lp	-2396.9
DECOUPLER			
du	h1	wc	370
dczf	0	sc	0
du	YY	vs	167
duw	w	th	11
qpr	40	sl	no
chf	8900	ph

MS-12-187-F79-104
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE SPECIAL
date Sep 24 2008 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
rr/avedia/issatlin-hat 0.008
n A fids/AR-12-197-pd0 13.500
-779-104/PROTON.F1-alfa 20.000
FLAOS d

ACQUISITION il n
aw 6402.0 in n
at 4.000 dp y
ap 51216 ha y
ba not used ha PROCESSING m
bb not used fa not used
bl 1.000 not used
bm 3.3
bt 8 ap 3.3
ct 8 wp 3585.9
td 3705.1
tm xl xfp 2904.6
tn xl xfp -125.2
afiq 400.083 xp -125.2
tofi 424.3 lp -93.9
tpr 58 PLOT
pw 6.750 wc 370
ps 0
dn C13 vs 130
dof 0 th 44
dm nm al cdc ph
dmc c
dpr c
dof 44
dof 13500

MS-12-187-F74-28
STANDARD IN ORNERYE

exp1 s2jml

SAMPLE SPECIAL
date Sep 23 2008 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin not used
rr/avedia/issalin-hat 0.008
n A fids/AR-12-197- p40 11.100
-774-78/CARBON.fid alfa 20.000
ACQUISITION FLAG
ms 25125.6 il n
st 1.199 in n
sp 60270 dp y
fs 13800 ha PROCESSING
ha 64 1b
sl 2.000 1b not used
sc 1000 fs not used
ct 1000 fs DISPLAY
TRANSMITTER sp -23.5
tx C13 wp 20130.9
sfreq 100.611 xrl 9268.0
toif 1522.6 xfp 7746.2
tpr 59 fp -150.0
pw 5.550 lp -378.6
DISCOUPLER PLOT
dn 81 wc 370
dof 0 sc 0
dm YYY vs 380
dms w th 14
dpr 40 al no ph
dif 8900

AK-13-93-448-63
STANDARD IN OBSERVE

exp1 s2jeu1

SAMPLE		SPECIAL	
date	Oct 17 2008	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
rr/aveda/instain-hat		0.008	
s A fidu/AK-13-93--	pp90	11.100	
F48-62/CARBON.fid	alfa	20.000	
ACQUISITION		FLAGS	
sc	25123.6 il	n	
st	1.199 in	n	
sp	60270 dp	y	
ss	13800 ha	nm	
sa	64	PROCESSING	
sl	1.000 lb	not used	
st	266 fa	not used	
ct	64	DISPLAY	
TRANSMITTER		sp	-7.4
tn	C13 wp	20130.9	
stfz	100.611 xrl	9278.0	
tozf	1522.6 xfp	7746.2	
tpwr	59 rp	170.3	
pw	5.550 lp	-288.7	
DECOUPLER		PLAT	
dn	kl wc	370	
doz	0 sc	0	
dm	YY vs	76	
dms	w th	11	
dpr	40 ai		
daz	8900

```

AC-13-129
=====
SAMPLE PREPARATION
date Jan 27 2010 satmode n
solvent cdcl3 wet n
file /home/data/ha- SPECIAL
rr/Avddia/Instain- temp 25.0
s A fide/AC-13-129- gain not used
-O2/CANON 01.fid spin 20
ACQUISITION bat 0.008
sw 25125.6 pps0 12.000
at 1.304 alfa 10.000
rg 65536
zb 12800 il FLAG
ba 64 in n
dl 2.000 dp y
st 512 ha nm
ct TRANSMITTER S12 1b PROCESSING 0.50
tn TRANSMITTER C13 1b not used
sfrq 100.611 DISPLAY
tofr 1538.0 sp -0.7
tqpr 57 wp 20130.9
pw 6.000 xrl 9245.2
dc DECOUPLER xrl 7746.2
dn xrl rp -162.7
dof 0 1p -489.0
dm YYY PLOT
decwave w wc 370
dpr 40 sc 0
def 8900 vs 263
th ai cdc ph 19

```

AK-20-85 carbon

exp1 CARBON

```

SAMPLE      PREPARATION
date Feb 5 2010 antmode n
solvent cdcl3 wet n
file /home/data/ha-
rr/Arveda/Instalin- temp 25.0
e A fids/AK-20-85-- gain not used
carbon_01/CARBON_0- spin 20
1.fid bat 0.008
ACQUISITION pps0 12.000
sw 25125.6 alfa 10.000
at 1.304
rg 69336 il FLAG
B 13800 ia n
ha 64 dp y
dl 2.000 ha
at 256 lb PROCESSING 0.50
CT TRANSMITTER f1 not used
in CL3 DISPLAY
sfreq 100.611 sp -1.6
toif 1518.0 wp 20130.9
tpr 57 r1l 9246.1
pw 6.000 rfp 7746.2
DCOUPLEAN rp 151.0
dn 81 lp -379.1
dof 0 PLOT
dm YYY wc 370
de wave w sc 0
dpr 40 vs 186
def 8900 th 12
al cdc ph
  
```

AK-16-79 Inova 400

exp1 PR10N

SAMPLE PREPARATION
 date Mar 14 2009 antmode n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/Aveda/Instalin- temp 25.0
 e A file/AK-16-79 - gain not used
 02/PROTON_0101.fid spin 20
 ACQUISITION bat 0.008
 sw 6395.9 pps 9.500
 at 2.562 alfa 6.600
 np 32768
 fb 4000 il FLAG 0
 ha 31 in n
 al 1.000 dp y
 nt 8 ha PROCESSING
 ct TRANSMITTEN 8 fa PROC used
 to TRANSMITTEN 81 fa DISPLAY 2.2
 afreq 399.762 ap 3596.5
 tof 399.8 wp 3685.5
 tpr 59 xrl 2902.3
 pw 4.750 xfp -17.7
 dc DISCOUPLER rp -27.3
 dn C13 lp PLOT
 dof 0
 dm nm wc 370
 decwave W40_hcp2 sc 0
 dpr 34 vs 53
 def 29412 th 38
 al cdc ph

AK-16-79 Inova 400

exp1 CARMON

```

SAMPLE PREPARATION
date Mar 14 2009 satmode n
solvent cdcl3 wet n
file /home/data/ha-
rr/Avddia/instain- temp 25.0
s A file/AK-16-79 - gain not used
02/CARMON_0101.fid spin 20
ACQUISITION bat 0.008
sw 2513.5 pps 8.800
at 1.304 alfa 10.000
sp 65336
zb 14000 il FLAG
ba 4 in n
dl 1.000 dp y
at 256 ha
ct TRANSMITTER 256 lb PROCESSING 0.50
tn TRANSMITTER C13 fm not used
sfreq 100.531 DISPLAY
toif 1530.6 sp 8.5
tprc 54 wp 20105.7
pw 4.400 rrl 9350.2
DECOUPLER rf 7740.0
dn 81 rf 38.0
dof 0 lp -325.4
ds YYY PLOT
ds w wc 370
ds ac 0
dpr 9500 vs 930
def th ai cdc ph il
  
```

AK-16-73-F21-35
STANDARD IN OBSERVE

exp1 s2jeol

SAMPLE SPECIAL
date Feb 18 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin not used
rr/aveda/instalin-hat 0.008
s A fids/AK-16-73-- p490 11.100
F21-35/auco_18Feb2- alfa 20.000
009/CARBON.fid FLAG

ACQUISITION il n
aw 25125.6 in n
at 1.199 dp y
ap 60270 ha
as 13860 lb PROCESSING
ba 2.000 fb not used
bl 2.000 fa not used
cl 1000 fm not used
ct 640 sp -2.0
td 20130.9
tn C13 xfl 2372.6
uf 100.611 xfp 7746.2
tof 1502.6 rp 91.0
tpr 59 lp -348.4
pw 5.550 PLOT

DECOUPLER wc 370
dn El ac 0
dof 0 vs 240
dm YYY th 15
dun w al no ph
dpr 40
dmf 8900

AK-16-05-09-30 12000 600

exp1 1H NMR

date Feb 17 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/AveDa/Spectra - temp 25.0
 for PS/AK-16-05-09- gain not used
 -3001/MGTON 0101. - spin 20
 f1d hat 0.008
 ACQUISITION pps0 11.600
 sr 9594.6 alfa 6.600
 at 1.708
 ap 32768 11 FLAG
 B 5000 1a n
 ha 31 dp n
 dl 1.000 ha PROCESSING
 at 8 s fa
 CT TRANSMITTER 81 9P DISPLAY 1.3
 ts 595.664 wp 5382.3
 tof 595.6 xrl 5552.7
 tpr 57 xfp 4353.5
 pr 5.800 rp 154.5
 DECOUPLER 1p -25.4
 dn CL3 PLOT
 dof 0 wc 370
 dm nm sc 0
 deconvolve W0_hm vs 54
 dpr 45 th 76
 def 31088 al cdc ph

MS-16-SF-P3-30
STANDARD IN OBSERVE

```

exp1 s2jeu1
=====
SAMPLE          SPECIAL
date   Feb 18 2009 temp not used
solvent cdcl3 gain not used
file   /home/data/ha-spin not used
rr/Avddia/Spectra - bat 0.008
for PS/AL-16-SF-P3- p490 11.100
-30/CARBON.fid alfa 20.000
=====
ACQUISITION
=====
ms 25123.6 il n
st 1.199 in n
sp 60270 dp y
fs 13800 ha PROCESSING
ha 64 1b
al 2.000 1b not used
st 1000 fs not used
ct 192 DISPLAY
=====
TRANSMITTER sp -3.0
tn C13 wp 20109.4
sfreq 100.611 xrl 9271.2
tof 1522.6 xfp 7746.2
tpr 59 rp 93.1
pw 5.550 lp -277.0
=====
DECOUPLER PLOT
dn el wc 370
dof 0 sc 0
dm YYY vs BS
dms w th 12
dpr 40 al no ph
dmf 8900

```

MS-16-PT-PS-30
STANDARD IN OBSERVE

exp1 s2jeu1

SAMPLE		SPECIAL	
date	Feb 18 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	spin	not used
rr/avedia/spectra	- bat	0.008	
for PS/AL-16-37-PS	p40	11.100	
-30/CARBON.fid	alfa	20.000	
ACQUISITION		FLAGS	
sc	25123.6	il	n
at	1.199	in	n
ap	60270	dp	y
bs	13800	hs	m
ba	64	1b	PROCESSING
cl	2.000	1b	not used
ct	1000	fn	not used
ct	192	fn	DISPLAY
TRANSMITTER		SP	
tn	C13	wp	21107.7
sfreq	100.611	xrl	9269.0
toif	1522.6	xrp	7746.2
tpwr	59	rp	52.3
pw	5.550	lp	-284.7
DECOUPLER		PLOT	
dn	kl	wc	370
doz	0	sc	0
dm	YY	vs	174
dms	w	th	7
dpr	40	ai	no ph
dai	8900

EX-16-97
STANDARD IN OBSERVE

```

exp1 s2jeu1
=====
SAMPLE      SPECIAL
date Feb 20 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin not used
rr/Avddia/Spectra - hat 0.008
for PS/AL-16-97/mn-pd90 11.100
to_20Feb2009/CARB-alfa 20.000
=====
N.fid
=====
ACQUISITION il n
aw 25123.6 in n
at 1.199 dp y
ap 60270 ha
as 13800 lb PROCESSING
at 2.000 fa not used
at 1000 4p DISPLAY -996.6
=====
TRANSMITTER wp 21107.7
tu C13 xrl 9267.3
sfrq 100.611 xfp 7746.2
toF 1562.6 rp 92.2
tpwr 59 lp -378.1
=====
PLOT
=====
DECOUPLER wc 370
dn el ac 0
doF 0 vs 226
dm YYY th 10
dms w ai no ph
dper 40
dnt 8900

```

AK-20-35-carbon

exp1 PROTON

```

SAMPLE
date Dec 11 2009 PREPARATION n
solvent cdcl3 wet n
file /home/data/ke-
xr/avedia/spectra - temp 25.0
for PS/AK-20-35-car gain not used
rbon_01/PROTON_01 - spin 20
fid bat 0.008
ACQUISITION pps0 13.100
sw 6402.0 alfa 10.000
at 2.599
rg 32768 il FLAG
rs 3600 la n
hs 31 dp y
dl 1.000 ha PROCESSING
at 8 s fa
ct TRANSMITTER 8 fa DISPLAY
ls 81 sp -0.8
sfreq 400.083 wp 4399.1
toF 628.7 xrl 3706.4
tpr 58 xfp 2904.6
pw 6.550 rp -159.7
pc DECOUPLE 1p -65.0
dn CL3
dof 0 wc 370
dm nm sc 0
decwave g vs 51
dpr 43 th 38
def 17100 ai cdc ph

```

AK-20-35-carbon

exp1 CARBON

```

SAMPLE PREPARATION
date Dec 11 2009 antmode n
solvent cdcl3 wet n
file /home/data/ke-
xr/avedia/spectra - temp 25.0
for PS/AK-20-35-car gain not used
rbon_01/CARBON_01.- spin 20
fid bat 0.008
ACQUISITION pps0 13.200
sw 25125.6 alfa 10.000
at 1.304
rg 69336 il FLAG
rs 13800 in n
hs 64 dp y
dl 1.000 ha nm
nt 256 lb PROCESSING
ct TRANSMITTER fa not used
in TRANSMITTER CL3 DISPLAY
sfreq 100.611 sp -4.3
toif 1518.0 wp 20134.7
tprz 56 rrl 9248.0
pw 6.600 rfp 7746.2
ps DECOUPLER rp 168.3
dn 81 lp -433.7
dof 0
dm YYY wc 370
ds w sc 0
dpr 40 vs 88
def 9300 th 13
al cdc ph

```

AK-20-45-F32-33-carbon

exp1 CARBON

```

SAMPLE PREPARATION
date Dec 11 2009 antmode n
solvent cdcl3 wet n
file /home/data/ke-
  sr/Avadhi/spectra - temp 25.0
for PS/AK-20-45-F3- gain not used
2-33-carbon.02/CAR- spin 20
NON_01.fid hat 0.008
ACQUISITION pps0 13.200
sw 25125.6 alfa 10.000
at 1.304
ap 69336 il FLAG
B 13800 in n
ha 64 dp y
dl 1.000 ha
at 256 lb PROCESSING 0.50
CT TRANSMITTER fa not used
IN CL3 DISPLAY
sfreq 100.611 sp
toif 1518.0 wp 20109.4
tpr 56 rrl 9245.8
pw 6.600 rfp 7746.2
DCOUPLEAN rp 152.8
dn xl lp -410.2
dof 0 PLOT
da YYY wc 370
de wave w sc 0
dpr 40 vs 170
def 9300 th 13
ai cdc ph

```

```

NK-20-53
exp1 CAMION

date Dec 15 2009  PREPARATION  n
solvent cdcl3  wet
file /home/data/ha-  SPECIAL
xr/Avddia/Spectra - temp 25.0
for PS/NK-20-53_01- gain not used
/CAMION_01.fid spin 20
ACQUISITION bat 0.008
sw 25125.6 pps0 13.200
at 1.304 alfa 10.000
rg 65536
zb 12800 il FLAG
ba 64 ia n
dl 1.000 dp y
nt 256 ha
ct TRANSMITTER C13 lb PROCESSING 0.50
tn 100.611 fm not used
tofq 1538.0 ap -5.1
tqpr 56 wp 20134.7
pw 6.600 xrl 9348.8
dc DECOUPLER xl rp 7746.2
dof 0 lp 166.6
dm YYY PLAT -419.1
ds w wc 370
dpr 40 ac 0
dof 9300 vs 156
al cdc pb 12
  
```

```

NK-20-55
exp1  CANNON

date Dec 16 2009  antmode  n
solvent  cdcl3  wet
file /home/data/ha-
xr/Avddia/Spectra - temp 25.0
for FS/NK-20-55_01- gain not used
/CANNON_01.fid spin 20
ACQUISITION bat 0.008
sw 25125.6 pps0 13.200
at 1.304 alfa 10.000
rg 65536
zb 12800 il
ha 64 ia n
dl 1.000 dp y
at 256 ha
ct TRANSMITTER 256 lb PROCESSING 0.50
tn TRANSMITTER C13 fn not used
sfrq 100.611 DISPLAY
tofr 1538.0 ap -1000.2
tqpr 56 wp 21107.7
pw 6.600 xrl 9245.6
dc DECOUPLER xl rp 7746.2
dof 0 lp 133.7
dm YYY w wc -346.4
decwave 370
dpr 40 ac 0
def 9300 vs 183
ai cdc ph 7

```

AE-20-69 Inova 400

exp1 CANNON

```

date      Jan 8 2010  satmode  n
solvent   cdcl3  wet        n
file      /home/data/ha-
xr/Avetta/Instain- temp    not used
e A file/AE-20-690- gain    not used
2/CANNON_0101.fid spin     20
ACQUISITION bat          0.008
sw 2513.5 pps0          14.300
at 1.304 alfa          10.000
rg 65536
zb 14000 il
ha 4 in
dl 2.000 dp y
st 1000 ha
ct TRANSMITTER 772 lb PROCESSING 0.50
tn 100.531 fn not used
sfreq 100.531 DISPLAY
tofq 1530.6 ap -1009.0
tqpr 50 wp 2114.4
pw 7.150 xrl 9248.3
pc DECOUPLER rf0 7740.0
dn 0 rf0 -86.0
dof 0 lp -312.5
dm YYY PL0T
decwave w wc 370
dpr 38 ac 0
def 8700 vs 2054
ai cdc pb 7

```

AK-17-25 Inova 600

exp1 PR10N

date Apr 6 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/Aveda/Instalin- temp 25.0
 e A file/AK-17_250- gain not used
 1/PROTON_0101.fid spin 20
 ACQUISITION bat 0.008
 sw 9594.6 pps 12.000
 at 1.708 alfa 6.600
 rg 32768
 fb 5000 il FLAG5 n
 ha 31 in n
 dl 1.000 dp y
 nt 8 ha PROCESSING nm
 ct TRANSMITTER s fa PROCESSING nm
 to TRANSMITTER el fa DISPLAY nm
 afreq 599.664 ap -11.0
 tof 599.6 wp 5404.6
 tpr 57 xrl 5552.2
 pr 6.000 xfp 4353.5
 dc DECOUPLER rp 43.7
 dn C13 lp -32.4
 dof 0 PLOT
 dm nm wc 370
 de wave hmc_hmc sc 0
 dpr 44 va 114
 def 30088 th 76
 al cdc ph

AK-16-151
STANDARD IN OBSERVE

exp1 s2jeu1

SAMPLE		SPECIAL	
date	Mar 9 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	spin	not used
rr/avedia/instain-hat		0.008	
n	11.100		
a	16-151- p490		
/CARBOR.fid	alpha	20.000	
ACQUISITION			
sc	25123.6	il	n
at	1.199	in	n
ap	60270	dp	y
as	13800	ha	nm
ba	64		
bc	2.000	lb	not used
bd	1.000	fn	not used
be	640		
cf	TRANSMITTER	sp	-8.2
in	C13	wp	20124.7
sfreq	100.611	xrl	9270.3
toq	1522.6	xrp	7746.2
tpwr	59	rp	101.4
pw	5.550	lp	-271.4
DECOUPLER			
dn	kl	wc	370
doz	0	sc	0
dm	YY	vs	240
dms	w	th	26
dpr	40	ai	no ph
dai	8900

expl a2pul

[illegible]

AK-16-165-F12
STANDARD IN OBSERVE

```

exp1 s2jeol
=====
SAMPLE      SPECIAL
date Mar 16 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin not used
rr/Avddia/Instalin-hat 0.008
s 1 fids/AK-16-165-pd90 11.100
-F12/autot_16mar200-alfa 20.000
9-20-48-38/CAMRHS.- FLA08
=====
fid 11 n
ACQUISITION in n
aw 25125.6 dp y
at 1.199 ha PROCESSING
ap 60270 1b
as 13852 1b not used 1.00
ba 1.000 4a
cl 1.000 4a DISPLAY -9.7
ct 256 ap
ct TRANSMITTER xfil 20124.7
tn C13 xfp 9274.1
tn 7746.2
sfrq 100.611 rfp 129.0
tof 1592.6 lp -314.9
tpr 59 PLOT
pw 5.550 wc 370
DECOUPLER ac 0
dm 81 vs 151
dof 0 th 11
dm YYY ai no ph
dms w
dpr 40
dmf 8900

```

1H-16-181-F2H-48
STANDARD IN OBSERVE

```

exp1 s2p01
=====
SAMPLE          SPECIAL
Date Mar 19 2009 temp not used
solvent cdcl3 gain not used
file /home/data/mw-spin 20
pr/medda/1antia-in-hat 0.008
o A file/16-16-181- p040 13.500
-735-48/PROTON.FID alfa 20.000
=====
ACQUISITION
aw 6403.0 il n
at 4.000 in n
ap 51214 dp y
as not used ha
bs not used ha PROCESSING
br 1.000 fa not used
cl 1.000 fa not used
cr 1.000 fa not used
ct TRANSMITTER s ap -0.4
tm 3600.2 wp 3600.2
ln kl xtl 3703.9
sfreq 600.083 xfp 2904.6
toif 624.3 xfp -149.4
tprz 58 lp -97.0
pw 6.750 p102
=====
DECOUPLER wc 370
dn cl3 sc 0
dof 0 vs 84
dm nm th 2
dun c al cdc ph
dpr 44
dat 18500

```

1H-16-181-FIN-48
STANDARD IN OBSERVE

exp1 s2jeul

SAMPLE SPECIAL
Date Mar 19 2009 temp not used
solvent cdcl3 gain not used
File /home/data/ha-spin not used
pr/meda/16-181-hat 0.008
s 2 file/16-181-pd90 11.100
-725-48/mto_19Mar-alfa 20.000
2009/CARDOR.fid FID
ACQUISITION il n
aw 2513.6 in n
at 1.199 dp y
ap 60370 ha PROCESSING um
Bz 13800 lb
Bz 1.000 lb not used 1.00
Bz 1.000 fa not used
at 284 DISPLAY
et 128 ap -1003.9
tm TRANSMITTER wp 21119.2
tm C13 xrl 9373.0
sfrq 100.611 xfp 7746.2
toif 15823.6 xfp 123.0
tprc 99 lp -2394.8
pw 5.550 PLAT
pw DECOUPLER wc 370
dm el ac 0
dof 0 vs 183
dm 377 th
dm w al no ph
dpr 40
dmf 8900

MS-16-187-FM-70
STANDARD IN OBSERVE

```

exp1 s2jml
=====
SAMPLE          SPECIAL
Date Mar 20 2009 temp not used
solvent cdcl3 gain not used
file /home/data/mw-spin 20
pr/medda/1ant1in-hat 0.008
o A file/MS-16-187- p40 13.500
-726-70/PROTON.FID sifa 20.000
=====
ACQUISITION
aw 6403.0 il n
at 4.000 in n
ap 51214 dp y
rs not used ha
hs not used ha PROCESSING
il 16
sl 1.000 fa not used
st 8
=====
PARAMETER      SP
ts TRANSMITTER 8 3600.2
tu K1 XEL 3703.9
mfq 600.083 xfp 2904.6
toif 624.3 xp -149.4
tpr 58 lp -99.2
pw 6.750 PL02
=====
DISCOUPLAR wc 370
an C13 ac 0
dof 0 vs 60
da num th 2
dun c al cdc ph
dpr 44
dat 13500

```

1H-16-187-F26-70
STANDARD IN COMMENT

exp1 s2jeul

SAMPLE		SPECIAL	
date	Mar 20 1009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
tr/avedia/tautalin-hat		0.008	
n 2 file/AR-16-187- p090		11.100	
-726-70/CARDOR.fid	alifa	20.000	
ACQUISITION			
sw	25125.4	il	n
st	1.199	in	n
sp	60370	dp	y
rs	13800	ha	nm
ha	64	ib	PROCESSING
al	1.000	lb	1.00
st	264	fa	not used
ct	128	display	
TRANSMITTER			
in	cl3	wp	-1002.5
sfreq	100.611	xrl	2119.2
toif	1523.6	xrp	9371.6
tpwr	99	rp	7746.2
pw	5.590	lp	130.5
DECOUPLER			
dn	el	wc	-329.8
doif	0	sc	370
dm	Y77	vs	0
dms	w	th	127
dpr	40	sl	6
dmf	8900	no	ph

15-15-141
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE SPECIAL
Date Mar 19 2009 temp not used
solvent cdcl3 gain not used
File /home/data/ha-spin 20
ir/meda/15-15-141-hat 0.008
o 2 file/15-15-141- p490 13.500
/PROTON.fid alfa 20.000
ACQUISITION F1A08
sw 6403.0 il n
st 4.000 in n
sp 51216 dp y
rs not used ha PROCESSING um
ha 1.000 fa not used
st 8
ct TRANSMITTER 8 sp -0.5
in 3600.2
f1 xel 2703.9
sfreq 400.083 xfp 2904.6
tof 424.3 xfp -137.1
tpr 58 lp -122.7
pw 6.790 PLAT
dc DECOUPLER wc 370
dm C13 sc 0
dof 0 vs 60
dm num th 76
dm c al cdc ph
dpr 44
dat 13500

15-15-141
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE		SPECIAL	
date	Mar 19 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/mw-spin	not used	
pr/meds/instain-hat		0.008	
o 2 file/AR-15-141- p090		11.100	
/auto_19Mar2009/CN-alfa		20.000	
NAME: f1d			
ACQUISITION			
aw	2513.6	in	n
at	1.199	dp	y
ap	60370	ha	nm
PROCESSING			
as	13800	lb	not used
at	1.000	fn	not used
at	286	display	-5.9
TRANSMITTER			
tn	C13	xrl	20124.7
mfzq	100.611	xrp	7746.2
toif	1582.6	xp	115.8
tpwr	99	lp	-232.0
PLAT			
pw	5.550	wc	370
dm	kl	ac	0
dof	0	va	244
dm	777	th	13
dm	w	al	no ph
qpr	40		
dmf	8900

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AK-15-143
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE SPECIAL
date Mar 18 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
rr/aveda/1astislin-hat 0.008
n A fids/AK-15-143- p40 13.500
/PROTON.fid alfa 20.000
ACQUISITION FLAG
aw 6402.0 il n
at 4.000 in n
ap 51216 dp y
bs not used ha PROCESSING
he 16
hl 1.000 fa not used
st 8
ct TRANSMITTER 8 sp -0.8
in xl xfl wp 3600.2
sfreq 400.083 xfp 2904.6
tofr 624.3 xp -146.9
tprc 58 lp -98.8
pw 6.750 PLOT
pc DECOUPLER wc 370
dn C13 ac 0
dof 0 vs 78
dm nm th 76
dms c al cdc ph
dpr 44
dif 18500

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AK-15-143
STANDARD IN OBSERVE

exp1 s2jeu1

SAMPLE		SPECIAL	
date	Mar 18 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	spin	not used
rr/avedia/issatlin-hat		0.008	
s A fidu/AK-15-143- p490		11.100	
/CARBOR.fid	alfa	20.000	
ACQUISITION			
sc	25123.6	il	n
at	1.199	in	n
ap	60270	dp	y
bs	13800	hs	m
bs	64	lb	PROCESSING
cl	1.000	lb	1.00
ct	256	fn	not used
ct	256	fn	not used
ct	256	fn	not used
TRANSMITTER			
in	C13	wp	20124.7
sfreq	100.611	xrl	9269.7
tofr	1522.6	xrp	7746.2
tpwr	59	rp	110.4
pw	5.550	lp	-339.8
DECOUPLER			
dn	kl	wc	370
doz	0	sc	0
dm	YY	vs	224
dms	w	th	13
dpr	40	ai	no ph
dpr	8900

MS-15-RT-F50
STANDARD IN OBSERVE

```

exp1 s2jml
=====
SAMPLE              SPECIAL
date Jan 12 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
rr/aveda/1astislin-hat 0.008
n A fids/AR-15-97-- p40 13.500
F50/PROTON.fid alfa 20.000
=====
ACQUISITION
ms 6402.0 il n
st 4.000 in n
sp 51216 dp y
fs not used ha PROCESSING
hs 16
sl 1.000 fa not used
st not used
=====
TRANSMITTER 8 8P -82.2
tx xl xfl 3682.6
rfq 400.083 xfp 3703.2
tof 624.3 xp -155.5
tpr 58 lp -87.3
pw 6.750 PLOT
=====
DECOUPLER wc 370
dn C13 ac 0
dof 0 vs 115
dm nm th 2
dms c al cdc ph
dpr 44
dnt 18500

```

AK-15-07-050
STANDARD IN OBSERVE

exp1 s2jeol

SAMPLE		SPECIAL	
date	Jan 12 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
rr/aveda/instain-hat		0.008	
s A fids/AK-15-07--	pd90	11.100	
P50/aufo_12Jan009-	alfa	20.000	
/CARBOR.fid	FLAG		
ACQUISITION			
sw	25123.6	in	n
at	1.199	dp	y
ap	60270	ha	mm
bs	13800	lb	PROCESSING
br	2.000	fb	not used
cl	2.000	fn	not used
ct	2000	sp	DISPLAY
td	2000	wp	-997.8
tn	C13	xrl	21119.2
trf	100.611	xfp	9267.0
tof	1562.6	rfp	7746.2
tpwr	59	lp	80.1
pw	5.550	plot	-350.8
DECOUPLER			
dn	el	ac	370
doz	0	va	0
dm	Y77	th	563
dms	w	ai	9
dpr	40	no	ph
dif	8900

MS-15-07-0715-38
STANDARD IN OBSERVE

exp1 s2jml
SPECIAL
date Jan 12 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
rr/meda/spectra-hat 0.008
for PS/AL-15-07-P3-pd0 13.500
5-38/PROTON.fid alfa 20.000
ACQUISITION
ms 6402.0 il n
st 4.000 in n
sp 51216 dp y
fs not used ha PROCESSING
hs 1.000 fa not used
st 8 2.2
ct TRANSMITTER 8 3589.2
ts xl xfl 3703.5
stfq 400.083 xfp 2904.6
tofq 624.3 xfp -152.8
tpwr 58 lp -96.5
pw 6.750 PLOT
pc DISCOUPLER wc 370
ds cl3 ac 0
dsf 0 vs 45
ds nm th 3
dsu c al cdc ph
dpr 44
dnt 18500

MS-15-07-216-3H
STANDARD IN OBSERVE

exp1 s2jeol

SAMPLE		SPECIAL	
date	Jan 12 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	not used	
rr/avedia/spectra	- bat	0.008	
for PS/AK-15-07-P3	- p40	11.100	
5-18/autoc_12/mz100	- alfa	20.000	
9/CARBON.fid	FLAG		
ACQUISITION			
sc	25123.6	in	n
at	1.199	dp	y
ap	60270	ha	mm
as	13800	lb	PROCESSING
ba	1.000	fb	not used
bt	1.000	fn	not used
ct	448	sp	DISPLAY
tu	19111.8	wp	-999.2
tn	C13	xrl	9269.9
sfreq	100.611	xfp	7746.2
toif	1562.6	rfp	72.0
tpwr	59	lp	-271.4
pw	5.550	plot	
DECOUPLER			
dn	hl	sc	370
dof	0	va	0
dm	Y77	th	274
dms	w	ai	no ph
dpr	40		
daf	8900

MS-15-ST-244
STANDARD IN OBSERVE

```

exp1 s2jml
=====
SAMPLE              SPECIAL
date Jan 12 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin 20
rr/avedia/spectra-hat 0.008
for PS/AL-15-ST-P4-pdO 13.500
6/PROTON.fid alfa 20.000
=====
ACQUISITION
ms 6402.0 il n
st 4.000 in n
sp 51216 dp y
fs not used ha PROCESSING
hs 16
sl 1.000 fa not used
st 8
=====
TRANSMITTER 8 sp 1.9
hp 3588.2
ts xl xfl 3703.7
sfreq 400.083 xfp 2904.6
tofr 624.3 xpr -157.3
tpr -92.8
pw 6.750 PLOT
=====
DECOUPLER wc 370
ds cl3 ac 0
dsf 0 vs 84
ds nm th 2
dsu c al cdc ph
dspr 44
dsf 18500

```

EX-15-87-P44
STANDARD 1H OBSERVE

expl a2pul

[illegible]

MS-15-131-F25-30
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE		SPECIAL	
date	Jan 18 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	20	
rr/aveda/instain-hat	0.008		
n A fids/AE-15-131- p40	13.500		
-F25-30/PROTON.fid	20.000		
ACQUISITION			
sw	6402.0	il	n
st	4.000	in	n
sp	51216	dp	y
fs	not used	ha	nm
he	16	PROCESSING	
sl	1.000	fa	not used
st	8	DISPLAY	
ct	TRANSMITTER	wp	-0.1
tu	kl	xel	3600.2
stfz	400.083	xfo	3703.4
toz	624.3	xp	2904.6
tpw	58	lp	-156.7
pw	6.750	PILOT	-95.8
dc	DISCOUPLER	wc	370
dn	C13	ac	0
doz	0	vs	95
dm	nm	th	3
dms	c	ai	cdc
dpr	44		
dmf	18500

KE-15-131-F25-30
STANDARD IN OMSERVE

exp1 s2jw1

date Jan 19 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ke-15-131-
r/Avetia/Imetala-hat not used
e A file/AM-15-131- p000 0.008
-F25-30/mto.192m- alfa 11.100
2009/CARBON.fid 20.000
ACQUISITION il n
25125.6 in n
1.199 dp y
60270 hs PROCESSING m
13800 lb
2.000 fb not used
1.00
2.000 fa not used
1000 DISPLAY
1000 sp -998.9
ct TRANSMITTER wp 2119.2
tn C13 xfl 9392.6
sfrq 100.611 xfp 7746.2
tof 1552.6 rp 92.0
tpr 99 lp -359.1
pw 5.550 PLOT
pw DECOUPLER wc 370
dn H1 ac 0
dof 0 vs 435
dm Y77 th 9
dms w ai no ph
dpr 40
dmf 8900

AK-19-193-nov13-2 novm 600

exp1 PROTON

date Nov 13 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 rx/AveDia/AK-19-19- temp 25.0
 7-Nov13-201/PROTON- gain not used
 _0101 spin 20
 ACQUISITION bat 0.008
 9593.5 pps0 9.100
 at 1.708 alfa 6.600
 ng 32768
 fb 5000 il 11 FLAG08
 ha 31 in n
 al 1.000 dp y
 nt 8 ha
 ct TRANSMITTER S fn PROCESSING
 to TRANSMITTER xl fn DISPLAY
 afreq 599.624 ap -0.5
 tof 599.6 wp 5395.7
 tpr 58 xrl 5544.7
 pr 4.550 xfp 4353.2
 dc DISCOUPLER rp 141.7
 dn C13 lp -17.2
 dof 0 PLOT
 dm nm wc 370
 de wave hfc_hmc sc 0
 dpr 44 va 46
 def 35088 th 73
 al cdc ph

AK-19-197 nova 400

exp1 CARMON

```

SAMPLE PREPARATION
date Nov 8 2009 antmode n
solvent cdcl3 wet n
file /home/data/ha- SPECIAL
r/Avedia/Instain- temp 25.0
e A file/AK-19-197- gain not used
03/CARMON_0101.fid spin 20
ACQUISITION bat 0.008
sw 2513.5 pps 14.300
at 1.304 alfa 10.000
np 69536
fb 14000 il FLAG
ba 64 in n
dl 2.000 dp y
nt 256 ha nm
CT TRANSMITTER C13 lb not used
to 100.531
sfreq 150.631 DISPLAY 7.7
toif 1510.6 ap 20115.7
tpr 50 wp 9322.5
pw 7.150 rfil 7740.0
DCOUPLES rfp -25.7
dn 0 lp -292.9
dm YYY PLOT
de wave w wc 370
dpr 38 sc 0
def 8700 vs 307
th al cdc ph 9

```

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AC-15-157
NOTED IN RESERVE

2xpl 22pnt

[illegible]

MS-18-5
STANDARD IN OBSERVE

exp1 s2jw1

SAMPLE		SPECIAL	
date	Jul 7 2009	temp	not used
solvent	cdcl3	gain	not used
file	/home/data/ha-spin	spin	not used
rr/avedia/spectra	- bat	0.008	
for PSAL-18-5/CAN-	p40	12.600	
BON. fld		alpha	20.000
ACQUISITION			
sw	25135.6	il	n
st	1.199	in	n
sp	60270	dp	y
fs	13800	ha	nm
PROCESSING			
ss	64	lb	
sl	1.000	lb	1.00
st	1.024	fs	not used
ct	640	fs	DISPLAY
TRANSMITTER			
tu	C13	wp	20109.4
sfreq	100.611	xrl	9368.2
toif	1523.6	xrp	7746.2
tpwr	59	rp	97.2
pw	6.300	lp	-317.0
DECOUPLER			
du	E1	wc	370
dcif	0	sc	0
du	YTY	vs	333
du	w	th	14
qpr	42	ai	no ph
duif	11100

AK-17-191-F3-5 Inova 600

exp1 1H NMR

date Jun 10 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/AveDia/Spectra - temp 25.0
 for PS/AK-17-191-F- gain not used
 3-501/PROTON_0101.- spin 20
 f1d bat 0.008
 ACQUISITION pps0 11.900
 sr 9594.6 alfa 6.600
 at 1.708
 ap 32768 il FLAG
 B 5000 la n
 ha 31 dp n
 dl 1.000 ha PROCESSING
 at 8 fa
 CT TRANSMITTER 8 fa DISPLAY 0.0
 la 8p
 afreq 599.646 wp 5382.3
 tof 599.7 xrl 5544.5
 tpr 57 xfp 4353.4
 pr 5.950 rp -36.6
 DECOUPLER 1p -25.3
 dn CL3 PLOT
 doif 0 wc 370
 dm nm sc 0
 deconvolve W6_box vs 57
 dpr 44 th 38
 def 35088 al cdc ph

55

MS-17-191-F2-5
STANDARD IN OBSERVE

exp1 s2jml

SAMPLE SPECIAL
date Jun 10 2009 temp not used
solvent cdcl3 gain not used
file /home/data/ha-spin not used
rr/avedia/spectra-hat 0.008
for PS/AL-17-191-F- p40 12.600
3-5/CARBON.fid alfa 20.000
ACQUISITION FLAG
ms 25125.6 il n
st 1.199 in n
sp 60270 dp y
fs 11800 ha PROCESSING
ha 64 1b
al 2.000 1b not used
sc 1024 fs
ct 1024 DISPLAY
TRANSMITTER sp 0.8
tx cl3 wp 20109.4
sfreq 100.611 xrl 9367.5
toif 1522.6 xfp 7746.2
tpr 59 rp 125.6
pw 6.300 lp -332.5
DISCOUPLER PLOT
dn kl wc 370
dof 0 sc 0
dm YYY vs 443
dms w th 15
qpr 42 al no ph
dof 11100

56

57

AK-18-189-F55-68 invra 400

exp1 PROTON

SAMPLE PREPARATION
 date Aug 20 2009 antmode n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/avedia/spectra - temp 25.0
 for PS/AK-18-189-F- gain not used
 55-68_02/PROTON_01- spin 20
 01.fid hat 0.008
 ACQUISITION psd0 13.800
 sw 6395.9 alfa 6.600
 at 2.562
 ap 32768 il FLAG
 B 4000 in n
 ha 31 dp n
 di 1.000 ha PROCESSING
 at 8 fa
 CT TRANSMITTER xl 9p DISPLAY 1.4
 fa 395.762 wp 3594.6
 tof 399.8 xrl 3669.5
 tpr 56 xfp 2902.3
 pr 6.900 rp -114.1
 DECOUPLER lp -53.6
 dn CL3 PLOT
 doif 0 wc 370
 dn nm sc 0
 decoupe W40_hfcp va 99
 dpr 34 th 76
 def 29412 al cdc ph

MS-18-189-F55-68-13C Inova 400

exp1 CANNON

```

SAMPLE      PREPARATION
date Aug 20 2009 antide n
solvent cdcl3 wet n
file /home/data/ha- SPECIAL
xr/Avddia/Spectra - temp 25.0
for PS/AK-18_189-F- gain not used
55-68-13C_01/CANBO- spin 20
N_0101.fid hat 0.008
ACQUISITION pps0 10.000
ms 2513.5 alfa 10.000
at 1.304 FLAG
ap 6536 il n
B 14000 in n
ha 4 dp y
sl 2.000 ha nm
at 5000 lb PROCESSING 0.50
ct TRANSMITTER C13 not used
in TRANSMITTER C13 DISPLAY
sfrq 100.531 sp -9.9
toF 1510.6 wp 20115.7
tpr 53 xrl 9372.4
pw 5.000 rfp 7740.0
dn DECOUPLER xl lp 156.2
dof 0 PLAT -376.6
dm YYY wc 370
dm w ac 0
dpr 39 vs 4007
def 9300 th 12
ai cdc ph

```

expl a2pul

date	time	sample	temp	special
solvent	cdcl3	spin	not used	
file /home/data/ha/ha-	gln		not used	
rrr/vevdi/Spectra - ha			0.008	
for PS/AS-19-35-ch- pw0			13.400	
ac/ROTOM.F16	ala		20.000	
ACQUISITION				
sw	3974.6	11	n	n
as	3.998	in	n	n
fb	31784	dp	y	y
ac	ha		nm	nm
16				
1.000	fm	not used		
8				
0				
TRANSMITTER			0.9	
rf	3567.4	Hz	3567.4	
400.022	Hz		3567.6	
-371.2	Hz		218.5	
19.3	Hz		170.2	
6.900	Hz		-60.5	
DECOMPLEX				
C13	ac	0	370	
0	vs	110		
nm	th			
al	cdc	ph		
44				
18100

KE-19-35-CHW
STANDARD IN OBSERVE

sepl s2jw1

date Aug 31 2009 temp not used
 solvent cdcl3 gain not used
 file /home/data/ke-19-35-CHW not used
 nr/Avddia/Spectra - hst 0.008
 for PS/AK-19-35-CHW p00 12.600
 sr/CARBON-13 f1d alfa 20.000
 ACQUISITION
 sr 25125.6 f1 n
 at 1.199 in n
 sp 60270 dp y
 sh 13800 hs PROCESSING nm
 sm 64 lb
 sl 2.000 lb not used
 st 1024 fs not used
 ct 832 DISPLAY
 ct TRANSMITTER sp 0.9
 tn C13 wp 20109.4
 mfrq 100.611 xel 9267.4
 tof 1552.6 xfp 7746.2
 tper 99 rp 129.9
 pw 6.300 lp -341.1
 DECOUPLER
 dn B1 wc 370
 dof 0 ac 0
 dm Y77 vs 416
 dms w th 14
 dper 42 ai no ph
 dnf 11100

exp1 proton

SAMPLE		DATE		OCT 31, 2009		ANALYSIS		PREPARATION		ANALYSIS	
id	name	date	time	solvent	conc	temp	gain	temp	gain	temp	gain
1	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
2	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
3	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
4	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
5	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
6	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
7	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
8	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
9	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
10	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
11	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
12	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
13	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
14	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
15	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
16	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
17	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
18	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
19	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
20	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
21	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
22	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
23	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
24	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
25	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
26	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
27	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
28	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
29	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
30	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
31	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000	1000
32	1000	1000	1000

AE-19-165-F6-15

exp1 CARBON

date Oct 31 2009 PREPARATION n
solvent cdcl3 wet n
file /home/data/ha- SPECIAL
r/Avddia/Instalin- temp 25.0
s A fidr/AE-19-165- gain not used
-F6-1601/CARBON_01- spin 20
-fid bat 0.008
ACQUISITION pps 12.600
sw 25125.6 alfa 10.000
st 1.304
sp 6536 11 FLAG
B 13800 in n
ha 64 dp y
sl 2.000 ha nm
st 256 1b PROCESSING 0.50
ct TRANSMITTER fn not used
in CL3 DISPLAY
bfq 100.611 sp -8.2
tof 1538.0 wp 20134.7
tpr 59 xrl 9248.8
pw 6.300 rfp 7746.2
DCOUPLES
dn 81 1p 163.4
dof 0 -429.8
dm YYY wc 370
decwave w sc 0
dpr 42 vs 61
def 11100 th 9
al cdc ph

AK-19-171-F9-12

exp1 PROTON

date Oct 28 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/Avetia/Isatin- temp 25.0
 e A fidm/AK-19-171- gain not used
 -F9-1201/PROTON_01- spin 20
 .fid bat 0.008
 ACQUISITION pps 13.800
 sw 6402.0 alfa 10.000
 at 2.599
 ap 32768 il 11 FLAG
 B 3600 in n
 ha 31 dp n
 dl 1.000 ha PROCESSING
 at 8 fa
 ct TRANSMITTER 8 fa DISPLAY
 ls 81 9p -0.3
 afreq 400.083 wp 3600.8
 tof 424.7 xrl 800.5
 tpr 58 xfp 0
 pr 6.900 rp -174.4
 DECOUPLER 1p -68.0
 ch CL3 PLOT
 dof 0 wc 370
 dm nm sc 0
 deconvolve H4O_NCHSE va 54
 dpr 44 th 76
 def 18500 al cdc ph

61 major

AE-19-171-F9-12

exp1 CAMION

SAMPLE PREPARATION
 date Oct 28 2009 antmode n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/Avella/Instalin- temp 25.0
 e A fidr/AE-19-171- gain not used
 -F9-1201/CAMION_01- spin 20
 .fid hat 0.008
 ACQUISITION pps0 12.600
 sr 25125.6 alfa 10.000
 at 1.304
 sp 6536 il
 B 13800 in n
 ha 64 dp y
 sl 2.000 ha nm
 at 2000 lb PROCESSING 0.50
 ct TRANSMITTER C13 not used
 in DISPLAY
 bnfq 100.611 sp 7.2
 tof 1538.0 wp 22136.0
 tpr 59 xrl 9309.4
 pr 6.300 rfp 7746.2
 DECOUPLER
 dn 81 lp 119.5
 dof 0 -432.3
 PLG
 dn YYY wc 370
 de wave w ac 0
 dpr 42 vs 438
 def 11100 th 17
 al cdc ph

61 minor

AK-19-189-F42-47

exp1 PROTON

date Nov 4 2009 PREPARATION n
solvent cdcl3 wet n
file /home/data/ha- SPECIAL
xr/Avadha/Isatisin- temp 25.0
e A fids/AK-19-189- gain not used
-F42-4701/PROTON_0- spin 20
1.fid hat 0.008
ACQUISITION pps 13.800
sw 6402.0 alfa 10.000
at 2.599
np 32768 il FLAG
B 3600 in n
ha 31 dp n
dl 1.000 ha PROCESSING
at 8 fa
CT TRANSMITTER RI 49 DISPLAY 2.5
ls 400.083 wp 3600.8
toF 428.7 xrl 3702.4
tpr 58 xfp 2904.6
pw 6.900 rp -174.1
DECOUPLER lp -69.1
dn CL3 PLOT
dof 0 wc 370
dm nm sc 0
decoupled H4O_NCHSE va 69
dpr 44 th 76
def 18900 al cdc ph

61 minor

AK-19-189-F42-47

exp1 CAMION

date	Row	4	2009	antimode	n
solvent	cdcl3	wet			n
file	/home/data/ha-			SPECIAL	
rr/Avddia/Isatisin-	temp	25.0			
e A fids/AK-19-189-	gain	not used			
-F42-4701/CAMION_0-	spin	20			
1.fid	bat	0.008			
acq	psd0	12.600			
acq	25125.6	alfa	10.000		
at	1.304			FLAG	
sp	65536	il			n
fb	13800	ia			n
ba	64	dp			y
dl	2.000	ha			mm
at	512	lb		PROCESSING	0.50
ct	TRANSMITTER	cl3	fn	not used	
ln	100.611	sp		DISPLAY	
sfreq	1538.0	wp			-2.8
tofr	59	xrl			2136.0
tpwr	6.300	rfl			9343.5
pw	DISCOUPLER	rp			7746.2
dn	kl	lp			128.4
dof	0			PLOT	-427.3
dm	YTY	wc			370
decwave	w	sc			0
dpr	42	vs			155
def	11100	th			12
		ai	cdc	pb

AK-19-187-F44-S5 inova 600

exp1 1H NMR

date Nov 5 2009 PREPARATION n
 solvent cd3od wet n
 file /home/data/ha- SPECIAL
 sr/Aveda/AK-19-18- temp 25.0
 7-F44-S501/PROC01 - gain not used
 0101 spin 20
 ACQUISITION bat 0.008
 sr 9593.5 pps 9.100
 at 1.708 alfa 6.600
 ng 32768
 fb 5000 il FLAG
 ha 31 in n
 al 1.000 dp y
 nt 8 ha PROCESSING nm
 ct TRANSMITTER s fn PROCESSING nm
 to TRANSMITTER xl fn DISPLAY -27.5
 afreq 599.626 ap
 tof 599.6 wp 5395.7
 tprz 58 xrl 4095.2
 pr 4.550 xfp 2866.2
 dc DISCOUPLER rf -119.9
 dn C13 lp -22.6
 dof 0 PLOT
 dm nm wc 370
 de wave hmc_hmc ac 0
 dpr 44 va 76
 def 30088 th 73
 al cdc ph

AK-19-187-F44-55

exp1 CARMON

```

SAMPLE      PREPARATION
date  Row 5 2009  antinide  n
solvent  cd3od  wet  n
file  /home/data/ha-  SPECIAL
rr/Avedia/Isatinin- temp 25.0
e A fids/AK-19-187- gain not used
-F44-5502/CARMON_0- spin 20
1. fids  hat 0.008
ACQUISITION  pps0 12.600
sw 25125.6  alfa 10.000
at 1.304
sp 69536 11
Bb 13800 1a n
ha 64 dp
dl 2.000 ha
at 2000 1b PROCESSING 0.50
ct TRANSMITTER C13  not used
in DISPLAY 4.2
afsq 100.613 sp
toif 1518.0 wp 22136.0
tprz 59 xrl 6282.9
pw 6.300 rfp 4944.5
DECOUPLER  rf -133.6
dn 81 1p -316.6
dof 0
dm YYY wc 370
dm w ac 0
dprv 42 vs 323
def 11100 th 11
al cdc ph

```

AK-20-27-#46-58 Inova 600

exp1 1H NMR

date Dec 11 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/Avadha/Spectra - temp 25.0
 for PS/AK-20-27-#4 - gain not used
 6-5802/PROTON_0101 - spin 20
 .fid hat 0.008
 ACQUISITION pps 9.400
 9593.5 alfa 6.600
 at 1.708
 ap 32768 il FLAG
 B 5000 in n
 ha 32 dp y
 al 1.000 ha PROCESSING nm
 ct 8 fn
 TRANSMITTER xl ap DISPLAY 0.3
 freq 599.624 wp 5391.6
 tof 599.6 xrl 2543.9
 tpr 58 xfp 4353.2
 pw 4.700 rp 124.1
 DECOUPLER lp -28.9
 dn CL3 PLOT
 dof 0 wc 370
 dm nm ac 0
 decouple W60_hm vs 65
 dpr 44 th 76
 det 35088 al cdc ph

AK-20-27-271-80 Inova 600

exp1 1H NMR

date Dec 11 2009 PREPARATION n
 solvent cdcl3 wet n
 file /home/data/ha- SPECIAL
 sr/Avedia/Spectra - temp 25.0
 for PS/AK-20-27- gain not used
 1-8002/PROTON_0101- spin 20
 .fid hat 0.008
 ACQUISITION pre90 9.400
 sr 9593.5 alfa 6.600
 at 1.708
 ap 32768 il FLAG
 B 5000 in n
 ha 32 dp y
 al 1.000 ha PROCESSING nm
 ct 8 fn
 TRANSFORMER 81 8p DISPLAY -0.5
 afreq 599.624 wp 5401.0
 tof 599.6 xrl 2544.7
 tpr 58 xfp 4353.2
 pr 4.700 rp 119.4
 DECOUPLER 1p -23.5
 dn C13 PLOT
 dof 0 wc 370
 dm nm ac 0
 decouple W60_hmc vs 44
 dpr 44 th 76
 def 30088 al cdc ph

[illegible]

[illegible]

AK-19-201A Inova 600

exp7 PROTON

SAMPLE		DATE		PREPARATION		ANALYSIS		RESULTS		COMMENTS	
id	name	date	time	method	operator	temp	pressure	yield	quality	notes	actions
1	101-101	2009	10/17	101-101	101-101	101-101	101-101	101-101	101-101	101-101	101-101
2	101-102	2009	10/17	101-102	101-102	101-102	101-102	101-102	101-102	101-102	101-102
3	101-103	2009	10/17	101-103	101-103	101-103	101-103	101-103	101-103	101-103	101-103
4	101-104	2009	10/17	101-104	101-104	101-104	101-104	101-104	101-104	101-104	101-104
5	101-105	2009	10/17	101-105	101-105	101-105	101-105	101-105	101-105	101-105	101-105
6	101-106	2009	10/17	101-106	101-106	101-106	101-106	101-106	101-106	101-106	101-106
7	101-107	2009	10/17	101-107	101-107	101-107	101-107	101-107	101-107	101-107	101-107
8	101-108	2009	10/17	101-108	101-108	101-108	101-108	101-108	101-108	101-108	101-108
9	101-109	2009	10/17	101-109	101-109	101-109	101-109	101-109	101-109	101-109	101-109
10	101-110	2009	10/17	101-110	101-110	101-110	101-110	101-110	101-110	101-110	101-110
11	101-111	2009	10/17	101-111	101-111	101-111	101-111	101-111	101-111	101-111	101-111
12	101-112	2009	10/17	101-112	101-112	101-112	101-112	101-112	101-112	101-112	101-112
13	101-113	2009	10/17	101-113	101-113	101-113	101-113	101-113	101-113	101-113	101-113
14	101-114	2009	10/17	101-114	101-114	101-114	101-114	101-114	101-114	101-114	101-114
15	101-115	2009	10/17	101-115	101-115	101-115	101-115	101-115	101-115	101-115	101-115
16	101-116	2009	10/17	101-116	101-116	101-116	101-116	101-116	101-116	101-116	101-116
17	101-117	2009	10/17	101-117	101-117	101-117	101-117	101-117	101-117	101-117	101-117
18	101-118	2009	10/17	101-118	101-118	101-118	101-118	101-118	101-118	101-118	101-118
19	101-119	2009	10/17	101-119	101-119	101-119	101-119	101-119	101-119	101-119	101-119
20	101-120	2009	10/17	101-120	101-120	101-120	101-120	101-120	101-120	101-120	101-120
21	101-121	2009	10/17	101-121	101-121	101-121	101-121	101-121	101-121	101-121	101-121
22	101-122	2009	10/17	101-122	101-122	101-122	101-122	101-122	101-122	101-122	101-122
23	101-123	2009	10/17	101-123	101-123	101-123	101-123	101-123	101-123	101-123	101-123
24	101-124	2009	10/17	101-124	101-124	101-124	101-124	101-124	101-124	101-124	101-124
25	101-125	2009	10/17	101-125	101-125	101-125	101-125	101-125	101-125	101-125	101-125
26	101-126	2009	10/17	101-126	101-126	101-126	101-126	10

isatisine A acetonide (2)
in CD₃OD

```

AK-19-201-carbon.inova.400
exp7  CARMON

SAMPLE PREPARATION
date Nov 17 2009 antmode n
solvent cd3od wet n
file /home/data/ha- SPECIAL
rr/Avetta/isatisin- temp 25.0
e A fid/ak-19_201- gain not used
-carbon01/CARMON_0- spin 20
101.fid hat 0.008
ACQUISITION pps0 14.300
sw 2513.5 alfa 10.000
at 1.304
sp 69336 il FLAG
B 14000 in n
ha 64 dp y
dl 2.000 ha mm
at 1000 lb PROCESSING 0.50
ct TRANSMITTER C13 not used
in DISPLAY
sfreq 100.531 sp -7.5
toF 1510.7 wp 22117.6
tpr 50 rF1 6291.1
pw 7.150 rFp 4940.6
DISCOUPLER rp 67.4
dn R1 lp -334.9
dof 0 PLOT
da YYY wc 370
de wave w ac 0
dpr 38 vs 1056
def 8700 th 6
al cdc ph
  
```

Nε-20-isatinsine-A-acetone/de-CDC13

(+)-isatisine A (1)

AK-20-isatisine A Inova 600

exp1 PROTON

```

SAMPLE      PREPARATION
date  Nov 20 2009  antide n
solvent  cd3od  wet  n
file  /home/data/ha- SPECIAL
xr/Avddia/AK-20-is- temp 25.0
atlineda02/PROTON - gain not used
0101 spin 20
acq 0.008
ACQUISITION  bat
nu 9593.5 ppe0 9.100
at 1.708 alfa 6.600
rg 32768
zb 5000 il
ha 32 ia
dl 1.000 dp
at 8 ha
ct TRANSMITTER 8 fn PROCESSING
tn TRANSMITTER 8 fn DISPLAY
afreq 599.626 ap 3.3
toif 599.6 wp 5385.7
tpr 58 xrl 3171.3
pw 4.550 xfp 1984.8
dc DECOUPLER rf -120.4
dn C13 1p -25.5
dof 0
nm nm wc 370
decwave 140_hcm ac 0
dpr 44 vs 22
def 30088 th 1
al cdc ph

```

(+)-isatisine A (1)

AK-20-isatisine A Inova 400

exp7 CARMON

```

SAMPLE      PREPARATION
date  Nov 20 2009  antmode  n
solvent  cdcl3  wet  n
file  /home/data/ha-
rr/Avectra/isatisin- temp  25.0
e A fids/AK-20_isa- gain  not used
tisineA01-carbon/C- spin  20
ARBOR_0101.fid  bat  0.008
ACQUISITION  pps0  14.300
sw  2513.5  alfa  10.000
at  1.304
ap  69336  i1  FLAG
B  14000  ia  n
ba  64  dp  y
bl  2.000  ha  mm
ct  1000  lb  PROCESSING  0.50
cs  TRANSMITTER  f1  not used
in  TRANSMITTER  C13  DISPLAY
sfreq  100.531  ap  -6.7
toif  1510.7  wp  22137.6
tpr  50  r11  6290.3
pw  7.150  r1p  4940.6
pc  DECOUPLER  rp  60.1
dn  81  lp  -322.0
dof  0
dm  YYY  wc  370
ds  w  sc  0
dpr  38  vs  1713
def  8700  th  12
ai  cdc  pa

```

11.65	579128	PB	0.711	49.426
17.67	590720	PB	1.104	50.415

RT	AREA	TYPE	AR/HT	AREA%
17.63	688850	PB	1.118	100.000

Compound **51**

Compound 55

AD-19-207A Ltr to 604

10790 10791

[illegible]

Kerr: Synthetic material

Naturally derived material

tonide (**2**)

10/27/2017

[illegible]

² Jones, C. D. *J. Org. Chem.* **1972**, 37, 3624-3625.