Expanding the scope of Mn(OAc)₃ mediated cyclizations; Synthesis of the tetracyclic core of tronocarpine.

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Supporting Information

Contents:

Experimental

General Information	S2
Typical procedure for Mn(OAc) ₃ promoted cyclizations	S2
Compounds 15-19, 21, 25-28, 39 and 40	S2 - S6
Typical procedure for acylation of indoles and pyrroles	S6
Compounds 8-13, 22, 24	S7 - S9
Procedures for preparation of compounds 14, 23, 34-48 and 41	S10-S13
¹H and ¹³C Spectra of compounds 8-19, 21-28, and 34-41	S14-S40

Experimental

General Information

Melting points are uncorrected. Infrared spectra were obtained as thin films on NaCl plates. ^1H and ^{13}C NMR experiments were performed at 400 or 600 MHz spectra were recorded in CDCl_3 (referenced to 7.26 ppm for ^1H and 77.0 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, DMF, methylene chloride were dried and deoxygenated by passing the nitrogen purged solvents through activated alumina columns. $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ was prepared in large quantities (>60 grams) using a literature procedure¹ or purchased from Aldrich. All other reagents and solvents were used as purchased from Aldrich, Strem, Caledon or VWR unless otherwise stated. Reaction progress was followed by thin layer chromatography (TLC) (EM Science, silica gel 60 F254) and the developed plates stained using acidic anisaldehyde. Flash chromatography was performed using silica gel purchased from Silicycle Chemical Division Inc. (230-400 mesh)

Typical procedure for $\text{Mn}(\text{OAc})_3$ promoted cyclization of indoles, pyrroles, and indoliness. (compounds 15-19, 21, 25-28, 39 and 40)

The indole/pyrrole/indoline substrate was dissolved in MeOH in a round bottomed flask equipped with magnetic stir bar. $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ was added (3.0 eq. for indole and pyrrole substrates, 5.0 eq. for indoline substrates). The flask was then fitted with a condenser, rubber septum and Argon balloon. The dark brown reaction mixture was stirred at reflux temperature overnight (16-24 hours) at which point TLC analysis showed complete consumption of starting material. (In some cases TLC plates were inconclusive because the product could not be distinguished from the starting material). The solvent was removed *in vacuo* (usually after transfer of the reaction mixture to a larger round bottomed flask). The reaction mixture (a brown solid) was then partitioned between brine and EtOAc. The organic layer was removed and the aqueous layer (often together with some brown emulsion) was extracted with EtOAc three times. The organic layers were combined and washed with water three times, then dried with MgSO_4 , and solvent removed *in vacuo*. The crude reaction mixture was pre-adsorbed onto silica and purified by flash chromatography on silical gel using gradient elution with EtOAc in Hexanes to yield compounds 15-19, 21, 25-28, 39 and 40 which are fully characterized below.

Compound 15

The general procedure was followed using indole **8** (1.00 g, 3.30 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (2.65 g, 9.90 mmol), and MeOH (25 ml). Purification of the crude reaction mixture by flash chromatography yielded 737 mg (2.45 mmol, 74%) compound **15** as light yellow

¹ De Klein, W. J. In *Organic Syntheses by Oxidation with Metal Compounds*; Mijs, W. J., de Jonge, C. R. H., Eds.; Plenum Press: New York, 1986; pp 261-314.

solid. MP = 116-117°C; R_f = 0.38 (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 8.48 (d, J = 8.4 Hz, 1H), 7.53 (d, J = 8.4 Hz, 1H), 7.34-7.37 (m, 1H), 7.27-7.30 (m, 1H), 6.70 (s, 1H), 3.85 (s, 6H), 2.82 (t, J = 6.6, 2H), 2.67 (t, J = 6.6, 2H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 168.9, 167.5, 135.2, 132.7, 128.9, 125.5, 124.3, 120.6, 116.6, 110.0, 109.3, 55.3, 53.6, 30.7, 28.3; IR (thin film): ν_{max} = 2956, 1738, 1713, 1592, 1568, 1453, 1372, 1352, 1320, 1271, 1243, 1172, 1080, 1067, 1000, 754; HRMS calc'd for $\text{C}_{16}\text{H}_{15}\text{NO}_5$ = 301.0950, found 301.0941.

(Compound **15** was also prepared from indoline **36** in 67% yield using an analogous procedure to the one described above with 5.0 eq of $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$.)

Compound 16

The general procedure was followed using indole **9** (197 mg, 0.621 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (498 mg, 1.86 mmol), and MeOH (15 ml). Purification of the crude reaction mixture by flash chromatography yielded 160 mg (0.509 mmol, 82%) of **16** as a yellow solid. MP = 116-118°C; R_f = 0.25 (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 8.49 (d, J = 8.0 Hz, 1H), 7.51 (d, J = 8.0 Hz, 1H), 7.29-7.39 (m, 2H), 3.82 (s, 6H), 2.86 (t, J = 6.8 Hz, 2H), 2.69 (t, J = 6.8, 2H), 2.17 (s, 3H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 169.5, 167.4, 134.2, 130.7, 127.9, 125.6, 124.0, 118.5, 118.0, 116.7, 55.1, 53.4, 31.0, 29.4, 9.0; IR (thin film): ν_{max} = 2954, 1737, 1708, 1456, 1366, 1320, 1274, 1243, 1207, 1149, 1074, 753; HRMS calc'd for $\text{C}_{17}\text{H}_{17}\text{NO}_5$ = 315.1107, found 315.1108.

Compound 17

The general procedure was followed using indole **10** (175 mg, 0.525 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (422 mg, 1.58 mmol), and MeOH (15 ml). Purification of the crude reaction mixture by flash chromatography yielded 129 mg (0.389 mmol, 74 %) of compound **17** as an orange solid. MP = 138-141 °C; R_f = 0.23 (30% EtOAc in Hexanes) $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 8.35 (d, J = 8.8 Hz, 1H), 6.94-7.00 (m, 2H), 6.64 (s, 1H), 3.84 (apparent s, 9H), 2.78-2.81 (m, 2H), 2.67-2.70 (m, 2H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 168.9, 167.1, 156.9, 133.2, 129.9, 129.8, 117.4, 113.7, 109.2, 103.5, 55.6, 55.3, 53.6, 30.6, 28.4; IR (thin film): ν_{max} = 3001, 2956, 2838, 1738, 1709, 1615, 1593, 1477, 1443, 1376, 1319, 1272, 1249, 1212, 1175; HRMS calc'd for $\text{C}_{17}\text{H}_{17}\text{NO}_6$ = 331.1056, found 331.1059.

Compound 18

The general procedure was followed using indole **11** (213 mg, 0.557 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (448 mg, 1.67 mmol) and MeOH (15 mL). Purification of the crude reaction mixture by flash chromatography yielded 145 mg (0.381 mmol, 68%) of compound **18** as an orange solid. MP = 145-146 °C; R_f = 0.23 (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 8.34 (d, J = 8.8 Hz, 1H), 7.66 (d, J = 2.0 Hz, 1H), 7.44 (dd, J = 8.8, 2.2 Hz, 1H), 6.64 (s, 1H), 3.85 (s, 6H), 2.82 (t, J =

6.4 Hz, 2H), 2.70 (t, 6.4 Hz, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 168.6, 167.4, 134.0, 133.8, 130.7, 128.3, 123.4, 118.0, 117.6, 108.4, 55.2, 53.7, 30.6, 28.2; IR (thin film): ν_{max} = 2956, 1738, 1586, 1449, 1371, 1353, 1314, 1270, 1243, 1173, 1082, 1069, 1002, 811, 714; HRMS calc'd for $\text{C}_{16}\text{H}_{14}\text{BrNO}_5$ = 379.0055, found 379.0050.

Compound 19

The general procedure was followed using indole **12** (729 mg, 2.13 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (1.71 g, 6.39 mmol) and MeOH (25 mL). Purification of the crude reaction mixture by flash chromatography yielded 519 mg (1.53 mmol, 72%) compound **19** as an orange solid. MP = 125-130°C; R_f = 0.75 (70% EtOAc in Hexanes); ^1H -NMR (400 MHz, CDCl_3): δ = 8.53 (d, J = 8.0 Hz, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.38-7.47 (m, 2H), 3.88 (s, 6H), 3.77 (s, 2H), 2.95 (t, J = 6.4 Hz, 2H), 2.72 (t, J = 6.4 Hz, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 169.8, 169.3, 135.9, 128.3, 126.2, 124.1, 122.7, 118.1, 116.9, 116.8, 111.8, 52.7, 52.6, 50.0, 32.8, 23.3, 14.4; IR (thin film): ν_{max} = 3403, 3006, 2957, 1736, 1610, 1456, 1370, 1319, 1274, 1248, 1177, 1074, 752; HRMS calc'd for $\text{C}_{18}\text{H}_{16}\text{N}_2\text{O}_5$ = 340.1059, found 340.1049.

Compound 21

The general procedure was followed using N-alkyl indole **14** (215 mg (0.744 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (598 mg, 2.23 mmol) and MeOH (15 mL). Purification of the crude reaction mixture by flash chromatography yielded 144 mg (0.502 mmol, 67%) compound **21** as a yellow solid. MP = 121-123 °C; R_f = 0.38 (30% EtOAc in Hexanes) ^1H -NMR (400 MHz, CDCl_3): δ = 7.61 (d, J = 8.0 Hz, 1H), 7.30 (d, J = 8.0 Hz, 1H) 7.20-7.24 (m, 1H), 7.11-7.15 (m, 1H), 6.62 (s, 1H), 4.09 (t, J = 6.2 Hz, 1H), 3.82 (s, 6H), 2.53-2.57 (m, 2H), 2.10-2.16 (m, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 170.3, 136.3, 130.8, 127.5, 121.7, 120.7, 120.1, 109.1, 102.1, 56.1, 53.2, 41.9, 28.7, 19.8; IR (thin film): ν_{max} = 2954, 1735, 1735, 1458, 1436, 1285, 1259, 1200, 1111, 749; HRMS calc'd for $\text{C}_{16}\text{H}_{17}\text{NO}_4$ = 287.1158, found 287.1150.

(Compound **21** was also prepared from indoline **38** in 62% yield using an analogous procedure to the one above with 5.0 eq of $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ and glacial acetic acid as solvent.)

Compound 25

The general procedure was followed using pyrrole **22** (300 mg, 1.18 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (953 mg, 3.56 mmol) and MeOH (15 mL). Purification of the crude reaction mixture by flash chromatography yielded 207 mg (0.824 mmol, 70%) compound **25** as an off white solid. MP = 74-76°C; R_f = 0.27 (30% EtOAc in Hexanes); ^1H -NMR (600 MHz, CDCl_3): δ = 7.43-7.45 (m, 1H), 6.35-6.36 (m, 1H), 6.29-6.30 (m, 1H), 3.81 (s, 6H), 2.75 (t, J = 9.6 Hz, 2H), 2.63 (t, J = 9.6 Hz, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 169.2, 166.5, 127.5, 117.9, 113.4, 112.8, 54.3, 53.4, 29.8, 28.6; IR (thin film): ν_{max} = 2958, 1731, 1407, 1360, 1212,

1258, 1226, 1093, 1072, 1042, 992, 741; HRMS calc'd for $C_{12}H_{13}NO_5$ = 251.0794, found 251.0802.

Compound 26

The general procedure was followed using pyrrole **23** (166 mg, 0.69 mmol), $Mn(OAc)_3 \cdot 2H_2O$ (554 mg, 2.07 mmol), and MeOH (10 ml). Purification of the crude reaction mixture by flash chromatography yielded 91 mg (0.38 mmol, 56%) compound **26** as a yellow oil with a minor unknown impurity visible by 1H NMR. R_f = 0.40 (30% EtOAc in Hexanes) 1H -NMR (400 MHz, $CDCl_3$): δ = 6.60-6.61 (m, 1H), 6.23-6.24 (m, 1H), 6.17-6.18 (m, 1H), 3.95 (t, J = 6.0 Hz, 2H), 3.76 (s, 6H), 2.42-2.44 (m, 2H), 1.99-2.01 (m, 2H); ^{13}C -NMR (100 MHz, $CDCl_3$): δ = 170.9, 123.1, 120.5, 108.9, 108.1, 55.2, 53.0, 45.0, 29.7, 29.0, 20.5; IR (thin film): ν_{max} = 2954, 2918, 2850, 1735, 1718, 1457, 1437, 1254, 1110, 879, 621; HRMS calc'd for $C_{12}H_{15}NO_4$ = 237.1001, found 237.0997.

Compounds 28 and 29

The general procedure was followed using pyrrole **24** (48.0 mg, 0.146 mmol), $Mn(OAc)_3 \cdot 2H_2O$ (201 mg, 0.729 mmol), and MeOH (10 ml). Purification of the crude reaction mixture by flash chromatography yielded 32.0 mg (0.098 mmol, 67 %) of a yellow oil consisting of a mixture of regioisomers **28** and **29**, 2:1 ratio based on 1H NMR. R_f = 0.28 (30% EtOAc in Hexanes) 1H -NMR (400 MHz, $CDCl_3$): δ = 7.69 (d, J = 1.2 Hz, 1H, **29**), 7.46-7.51 (m), 7.20-7.36 (m), 6.66 (d, J = 1.2 Hz, 1H, **29**), 6.31 (d, J = 3.2 Hz, 1H, **28**), 3.87 (s, 6H, **29**), 3.48 (s, 6H, **28**), 2.68-2.83 (m). ^{13}C -NMR (100 MHz, $CDCl_3$): δ = 169.1, 166.7, 166.2, 134.2, 133.3, 129.1, 128.7, 128.5, 127.9, 127.3, 127.2, 125.7, 123.6, 117.2, 115.6, 113.4, 112.2, 55.7, 54.4, 53.5, 53.0, 30.9, 30.1, 29.7, 28.6; IR (thin film): ν_{max} = 3145, 3059, 3031, 3005, 2954, 2924, 2848, 1733, 1451, 1435, 1391, 1373, 1324, 1315, 1282, 1257, 1234, 1196, 1178, 1162, 1075, 757, 702; HRMS calc'd for $C_{12}H_{15}NO_4$ = 327.1107, found 327.1115.

Compound 39

The general procedure was followed using indole **37** (1.06g, 3.84 mmol), $Mn(OAc)_3 \cdot 2H_2O$ (5.15g, 19.2 mmol), and MeOH (20 ml). Purification of the crude reaction mixture by flash chromatography yielded 595 mg (2.18 mmol, 57%) of compound **39** as a white solid. MP = 121-123 °C; R_f = 0.39 (30% EtOAc in Hexanes) 1H -NMR (400 MHz, $CDCl_3$): δ = 7.59-7.62 (m, 1H), 7.26-7.28 (m, 1H), 7.16-7.20 (m, 1H), 7.08-7.12 (m, 1H), 7.55 (d, J = 0.8 Hz, 1H), 4.21 (t, J = 6.8 Hz, 2H), 3.80 (s, 6H), 3.19 (t, J = 6.8 Hz, 2H); ^{13}C -NMR (100 MHz, $CDCl_3$): δ = 169.4, 138.8, 132.7, 132.4, 121.6, 119.7, 109.7, 96.6, 58.8, 53.3, 42.5, 36.9; IR (thin film): ν_{max} = 3054, 2955, 2898, 2848, 1738, 1456, 1435, 1272, 1223, 1100, 778, 746; HRMS calc'd for $C_{15}H_{15}NO_4$ = 273.1001, found 273.1007.

Compound 40

The general procedure was followed using indole **38** (144 mg, 0.408 mmol), $\text{Mn}(\text{OAc})_3 \cdot 2\text{H}_2\text{O}$ (546mg, 2.04 mmol), and MeOH (10 ml). Purification of the crude reaction mixture by flash chromatography yielded 98 mg (0.28l, 69%) of compound **40** as a clear oil. R_f = 0.50 (30% EtOAc in Hexanes) $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 7.61 (d, J = 7.2 Hz, 1H), 7.33-7.38 (m, 3H), 7.25-7.27 (m, 2H), 7.05 (t, J = 7.8 Hz, 1H), 6.96 (t, J = 7.8 Hz, 1H), 6.61-6.64 (m, 2H), 5.52 (t, J = 7.2 Hz, 1H), 3.82 (s, 3H), 3.80 (s, 3H), 3.66 (dd, J = 13.2, 7.2 Hz, 1H), 3.02 (dd, J = 31.2, 7.2 Hz, 1H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 169.4, 169.3, 139.3, 132.9, 132.5, 129.0, 128.4, 126.8, 121.5, 121.3, 119.9, 110.7, 97.0, 60.2, 58.6, 53.5, 53.3, 47.6; IR (thin film): ν_{max} = 3052, 3031, 3007, 2953, 2844, 1735, 1453, 1436, 1261, 1247, 1159, 1103, 770, 747, 701; HRMS calc'd for $\text{C}_{12}\text{H}_{15}\text{NO}_4$ = 349.1314, found 349.1304.

Typical procedure preparation of N-acyl indoles and N-acyl pyrroles (compounds 8-13, 22 and 24)**Preparation of Acid Chloride 40****Compound 40**

2-Methoxycarbonyl-pentanedioic acid 1-methyl ester² was dissolved in dry toluene in an oven dried round bottomed flask equipped with stir bar, rubber septum, and Argon balloon. DMF (2-5 drops) was added. Oxalyl chloride (1.5 equivalents) was added via syringe. The reaction was stirred at room temperature for 30 minutes. The flask was then fitted with a reflux condenser and the reaction was warmed to 60 °C for 2 hours then allowed to cool to room temperature. The solvent was removed *in vacuo* and the crude product, a yellow oil, was dissolved in DMF and used immediately in the next reactions.

General acylation procedure (compounds 8-13, 2 and, 24)

The indole/pyrrole substrate was dissolved in dry DMF in an oven dried round bottomed flask equipped with a magnetic stir bar. NaH (1.2 - 1.3 eq), 60% dispersion in oil, was added and the flask was fitted with a rubber septum and Argon balloon. The reaction mixture was stirred for 30-60 minutes while H_2 was evolved. Acid chloride **40** (1.3-1.5 eq) in DMF was added via cannula. The reaction was allowed to stir at room temperature overnight (8-16 hours) at which point the mixture was poured into water and extracted three times with diethyl ether. The organic layers were combined, washed once with brine, dried with MgSO_4 , filtered and solvent removed *in vacuo*. The crude reaction mixture was pre-adsorbed on to silica and purified by flash column chromatography on silical gel

² Prabhu, K. R.; Pillarsetty, N.; Gali, H.; Katti, K. V. *J. Am. Chem. Soc.* **2000**, *122*, 1554.

using gradient elution with EtOAc in Hexanes to yield compounds **8-13**, **22** and **24** which are fully characterized below.

*Comment: The above described reactions did not proceed to completion. The isolated yields were poor and some starting material was always recovered. Various optimization strategies were attempted without improvement. DMF was the only useful solvent. Use of more NaH resulted in lower yields. Addition of a large excess of acid chloride **42** did increase yields however, for the purposes of this study, we found it preferable to conserve this reagent and accept modest yields in these reactions. It seems likely that under basic conditions acid chloride **42** may react intramolecularly to yield a cyclobutanone product. However, we were only able to isolate starting material and desired acylated products and the fate of the unused acid chloride was not confirmed.*

Compound 8

The general procedure was followed using indole (2.98 g, 25.5 mmol), NaH (1.22 g, 30.5 mmol), DMF (15 mL) and acid chloride **40** (38.2 mmol in 10 mL of DMF, from 7.79 g of the corresponding acid precursor). Purification by flash column chromatography yielded 1.20 g (3.96 mmol, 16%) of compound **8** as a clear oil. $R_f = 0.30$ (30% EtOAc in Hexanes) $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 8.45$ (d, $J = 8.4$ Hz, 1H), 7.56 (d, $J = 8.4$ Hz, 1H), 7.47 (d, $J = 4.0$ Hz, 1H), 7.33-7.37 (m, 1H), 7.25-7.29 (m, 1H), 6.65 (d, $J = 4.0$ Hz, 1H), 3.76 (s, 6H), 3.67 (t, $J = 7.2$ Hz, 1H), 3.05 (t, $J = 7.2$ Hz, 2H), 2.43 (t, $J = 7.2$ Hz, 2H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): $\delta = 170.1$, 169.4, 135.5, 130.3, 125.1, 124.4, 123.7, 120.8, 116.5, 109.4, 52.7, 50.1, 32.7, 23.4; IR (thin film): $\nu_{\text{max}} = 2955$, 1750, 1734, 1707, 1537, 1472, 1394, 1224, 1155, 753, 719; HRMS calc'd for $\text{C}_{16}\text{H}_{17}\text{NO}_5 = 303.1107$, found 303.1103.

Compound 9

The general procedure was followed using 3-methyl indole (752 mg, 5.73 mmol), NaH (297 mg, 7.45 mmol), DMF (15 mL) and acid chloride **40** (8.57 mmol in 5 mL of DMF from 1.75 g of acid precursor). Purification by flash column chromatography yielded 269 mg (0.848 mmol, 15%) compound **9** as a light yellow solid. MP = 83–85 °C; $R_f = 0.25$ (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): $\delta = 8.42$ (d, $J = 7.2$ Hz, 1H), 7.50 (d, $J = 8.0$ Hz, 1H), 7.27-7.37 (m, 2H), 7.22 (s, 1H), 3.77 (s, 6H), 3.67 (t, $J = 7.2$ Hz, 1H), 3.01 (t, $J = 7.2$ Hz, 2H), 2.43 (q, $J = 7.2$ Hz, 2H), 2.28 (s, 3H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): $\delta = 169.7$, 169.5, 135.9, 131.3, 125.2, 123.5, 121.3, 118.8, 118.7, 116.6, 52.7, 50.2, 32.8, 23.5, 9.7; IR (thin film): $\nu_{\text{max}} = 2955$, 2924, 1736, 1700, 1451, 1397, 1347, 1323, 1293, 1212, 1157, 1063, 749; HRMS calc'd for $\text{C}_{17}\text{H}_{19}\text{NO}_5 = 317.1263$, found 317.1272.

Compound 10

The general procedure was followed using 5-methoxyindole (500 mg, 3.40 mmol), NaH (177 mg, 4.42 mmol), DMF (15 mL) and acid chloride **40** (5.10 mmol in 5 mL of DMF from 1.04 g of acid precursor). Purification by flash column chromatography yielded 226 mg (0.678 mmol, 19%) compound **10** as a light blue oil (with minor unidentified impurities visible proton NMR). R_f = 0.20 (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 8.33 (d, J = 8.4 Hz, 1H), 7.44 (d, J = 3.6 Hz, 1H), 7.02 (d, J = 2.4 Hz, 1H), 6.95 (dd, J = 8.4, 2.4 Hz, 1H), 6.57 (d, J = 3.6 Hz, 1H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 169.7, 169.4, 156.5, 131.3, 130.2, 125.0, 117.3, 113.5, 109.3, 103.6, 55.6, 52.7, 50.2, 32.5, 23.5; IR (thin film): ν_{max} = 3002, 2955, 2838, 1735, 1703, 1614, 1588, 1475, 1442, 1394, 1318, 1259, 1207, 1152, 1108, 1031, 813, 719, 627; HRMS calc'd for $\text{C}_{17}\text{H}_{21}\text{NO}_5$ = 333.1212, found 331.1217.

Compound 11

The general procedure was followed using 5-bromo indole (500 mg, 2.55 mmol), NaH (133 mg, 3.32 mmol), DMF (15 mL) and acid chloride **40** (3.83 mmol in 5 mL of DMF from 781 mg of acid precursor). Purification by flash column chromatography yielded 373 mg (0.976 mmol, 38%) compound **11** as an off white solid. MP = 111-113 °C; R_f = 0.23 (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 8.33 (d, J = 9.6 Hz, 1H), 7.69 (d, J = 1.8 Hz, 1H), 7.48 (d, J = 4.2 Hz, 1H), 7.44 (dd, J = 9.0, 1.8 Hz, 1H), 6.59 (d, J = 4.2 Hz, 1H), 3.77 (s, 6H), 3.65 (t, J = 7.2 Hz, 1H), 3.05 (t, J = 7.2 Hz, 2H), 2.43 (q, J = 7.2 Hz, 2H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 170.0, 169.4, 134.3, 132.0, 128.0, 125.5, 123.5, 117.9, 117.0, 108.5, 52.7, 50.1, 32.7, 23.4; IR (thin film): ν_{max} = 2955, 1734, 1712, 1446, 1390, 1334, 1313, 1265, 1217, 1200, 1156, 1056, 894, 812, 761, 719; HRMS calc'd for $\text{C}_{16}\text{H}_{16}\text{BrNO}_5$ = 381.0212, found 381.0221.

Compound 12

The general procedure was followed using indole-3-acetonitrile (4.57 g, 29.3 mmol), NaH (1.52 g, 38.0 mmol), DMF (40 mL) and acid chloride **40** (41.0 mmol in 10 mL of DMF from 8.36 g of acid precursor). Purification by flash column chromatography yielded 3.27 g (9.55 mmol, 33%) compound **12** as a low melting orange solid. R_f = 0.75 (70% EtOAc in Hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 8.46 (d, J = 8.4 Hz, 1H), 7.54 (s, 1H), 7.51 (d, J = 8.4 Hz, 1H), 7.43 (t, J = 8.4 Hz, 1H), 7.35 (t, J = 8.4 Hz, 1H), 3.80 (s, 2H), 3.78 (s, 6H), 3.65 (t, J = 7.2 Hz, 1H), 3.06 (t, 7.2 Hz, 2H), 2.44 (q, J = 7.2 Hz, 2H). $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 169.8, 169.3, 135.9, 128.3, 126.2, 124.0, 122.7, 118.1, 116.8, 111.7, 52.7, 50.0, 32.7, 23.2, 14.4; IR (thin film): ν_{max} = 2957, 1733, 1608, 1453, 1394, 1355, 1205, 1160, 1048, 753; HRMS calc'd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_5$ = 342.1216, found 342.1207.

Compound 13

The general procedure was followed using 3-formyl indole (168 mg, 1.15 mmol), NaH (55 mg, 1.4 mmol), DMF (8 mL) and acid chloride **42** (1.5 mmol in 5 mL of DMF from 305 mg of acid precursor). Purification by flash column chromatography yielded 148 mg (0.418 mmol, 36 %) compound **13** as a yellow oil. R_f = 0.68 (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (600 MHz, CDCl_3): δ = 10.11 (s, 1H), 8.39 (d, J = 7.8 Hz, 1H), 8.26 (d, J = 7.8 Hz, 1H), 8.14 (s, 1H), 7.39-7.44 (m, 2H), 3.77 (s, 6H), 3.66 (t, J = 7.2 Hz, 1H), 3.15 (t, J = 7.2 Hz, 2H), 2.45 (q, J = 7.2, 2H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 185.6, 170.3, 169.3, 136.4, 134.5, 126.9, 125.9, 125.4, 122.8, 121.9, 116.4, 52.8, 49.8, 32.8, 23.2; IR (thin film): ν_{max} = 2956, 1732, 1676, 1608, 1552, 1448, 1401, 1343, 1313, 1262, 1250, 1163, 1128, 1061, 785, 755

Compound 22

The general procedure was followed using pyrrole (1.27 g, 18.9 mmol), NaH (983 mg, 22.7 mmol), DMF (20 mL) and acid chloride **40** (24.6 mmol in 10 mL of DMF from 5.01 g of acid precursor). Purification by flash column chromatography yielded 805 mg (3.18 mmol, 17%) compound **22** as a clear oil. R_f = 0.27 (30% EtOAc in Hexanes); $^1\text{H-NMR}$ (400 MHz, CDCl_3): δ = 7.30 (apparent broad s, 2H), 6.28 (t, J = 2.2 Hz, 2H), 3.75 (s, 6H), 3.59 (t, J = 7.2 Hz, 1H), 2.96 (t, J = 7.2 Hz, 2H), 2.36 (q, J = 7.2, 2H); $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 169.3, 169.3, 118.9, 113.2, 52.7, 50.1, 31.5, 23.2; IR (thin film): ν_{max} = 3150, 3004, 2957, 2849, 1734, 1471, 1438, 1284, 1206, 1158, 907, 747; HRMS calc'd for $\text{C}_{12}\text{H}_{15}\text{NO}_5$ = 253.0950, found 253.0941.

Compound 24

The general procedure was followed using 3-phenyl pyrrole³ (95 mg, 0.66 mmol), NaH (34 mg, 0.85 mmol), DMF (5 mL) and acid chloride **40** (0.99 mmol in 5 mL of DMF from 202 mg of acid precursor). Purification by flash column chromatography yielded 58 mg (0.18 mmol, 27%) compound **24** as a yellow solid. MP = 107-109 °C; R_f = 0.28 (30% EtOAc in Hexanes) $^1\text{H-NMR}$ (400 MHz, CDCl_3): 7.62 (s, 1H), 7.56 (d, J = 7.2 Hz, 2H), 7.37-7.42 (m, 3H), 7.26-7.30 (m, 1H), 6.64-6.66 (m, 1H), 3.79 (s, 6H), 3.64 (t, J = 7.2 Hz, 1H), 3.02 (t, J = 7.2 Hz, 2H), 2.42 (q, J = 7.2 Hz, 2H); δ = $^{13}\text{C-NMR}$ (100 MHz, CDCl_3): δ = 169.3, 169.2, 133.7, 129.0, 128.8, 127.0, 125.6, 120.0, 114.6, 112.0, 52.7, 50.1, 31.5, 23.3 IR (thin film): ν_{max} = 3126, 2959, 1746, 1734, 1711, 1444, 1352, 1346, 1275, 1236, 1200, 1179, 1149, 1085, 203, 759, 709, 694; HRMS calc'd for $\text{C}_{12}\text{H}_{15}\text{NO}_4$ = 329.1263, found 329.1268.

³ Smith N. D.; Huang, D.; Cosford, N. D. P. *Org. Lett.* **2002**, *4*, 3537.

Experimental procedures for preparation of compounds **14**, **23**, **34-38** and **41**.

Compound **14**

NaH (60% dispersion in oil, 253 mg, 6.33 mmol) was added to a round bottomed flask equipped with stir bar. The NaH was then washed with hexanes, and dried briefly *in vacuo*. The flask was equipped with a rubber septum and Argon balloon. DMF (20 mL) was added followed by dimethyl malonate (0.902 mL, 7.90 mmol) via syringe. The mixture was allowed to stir at room temperature for 30 minutes while H₂ was evolved. 1-(3-Bromo-propyl)-indole⁴ (378 mg, 1.58 mmol) in DMF (10 mL) was added via cannula. The reaction was allowed to stir at room temperature for 8 hours at which point TLC analysis showed complete consumption of starting material. The reaction was quenched by dropwise addition of water and then partitioned between water and diethyl ether. The organic layer was collected and the aqueous layer was extracted three times with diethyl ether. The organic layers were combined and washed once with brine, dried with MgSO₄, filtered, and solvent removed *in vacuo*. The crude reaction mixture was purified by flash chromatography on silica gel (EtOAc/Hexanes) to yield 377 mg (1.30 mmol, 82%) of compound **14** as a yellow oil. *R*_f = 0.36 (30% EtOAc in Hexanes) ¹H-NMR (600 MHz, CDCl₃): δ = 7.63 (d, *J* = 7.8 Hz, 1H), 7.33 (d, *J* = 7.8 Hz, 1H), 7.21 (t, *J* = 7.8 Hz, 1H), 7.09-7.12 (m, 2H), 6.50 (d, *J* = 3.0 Hz, 1H), 4.15 (t, *J* = 7.2 Hz, 1H), 3.71 (s, 6H), 3.34 (t, *J* = 7.2 Hz, 1H), 1.86-1.96 (m, 4H); ¹³C-NMR (100 MHz, CDCl₃): δ = 169.4, 135.8, 128.6, 127.6, 121.5, 121.0, 119.3, 109.2, 101.2, 52.5, 51.1, 45.8, 27.8, 26.1; IR (thin film): ν_{max} = 3052, 3028, 3005, 2953, 2875, 1751, 1735, 1512, 1465, 1337, 1315, 1298, 1278, 1157, 1013, 743; HRMS calc'd for C₁₂H₁₇NO₄ = 289.1314, found 289.1305.

Compound **23**

Compound **23** was prepared using the same procedure as compound **14** above. Quantities used were as follows: NaH (60% dispersion in oil, 715 mg, 28.6 mmol), dimethyl malonate (2.51g, 19.0 mmol), DMF (20 mL) and 1-(3-Bromo-propyl)-pyrrole³ (2.99g, 15.9 mmol) in DMF (10 mL). Purification by flash column chromatography yielded 2.07 g (8.65mmol, 54%) compound **23** as a yellow oil with a minor unknown impurity visible by ¹H NMR. *R*_f = 0.40 (30% EtOAc in Hexanes) ¹H-NMR (400 MHz, CDCl₃): δ = 6.14 (t, *J* = 1.6 Hz, 2H), 6.14 (t, *J* = 1.6 Hz, 2H), 3.90 (t, *J* = 6.8 Hz, 2H), 3.73 (s, 6H), 3.33 (t, *J* = 6.8 Hz, 1H), 1.87-1.91 (m, 2H), 1.78-1.83 (m, 2H); ¹³C-NMR (100 MHz, CDCl₃): δ = 169.5, 120.4, 108.2, 52.6, 51.1, 49.0, 29.2, 26.0; IR (thin film): ν_{max} = 3394, 2955, 2850, 1749, 1734, 1676, 1457, 1436, 1283, 1265, 1244, 1231, 1155, 1090, 746, 729; HRMS calc'd for C₁₂H₁₇NO₄ = 239.1158, found 239.1160.

⁴ Dehaen, W.; Hassner, A. *J. Org. Chem.* **1991**, *56*, 896.

Compound 34

Indoline (1.54 g, 12.9 mmol) was dissolved in CH_2Cl_2 (15 mL) in an oven dried round bottomed flask equipped with a magnetic stirrer, rubber septum and Argon balloon. Et_3N (2.15 mL 15.5 mmol), was added via syringe. Acid chloride **40** (14.2 mmol) in CH_2Cl_2 (10 mL) was added via cannula. (this acid chloride was prepared from 2.90 g of the corresponding acid as described above, then dissolved in CH_2Cl_2 and used immediately). The reaction mixture was stirred at room temperature for 1 hour at which point TLC analysis showed consumption of all starting material. The reaction mixture was poured into saturated aqueous NH_4Cl and extracted three times with CH_2Cl_2 . The organic layers were combined and washed with water and brine. The organic layer was then dried with anhydrous MgSO_4 , filtered, and solvent removed *in vacuo*. The resulting brown solid (>95% pure by ^1H NMR) purified by flash chromatography on silica gel (EtOAc/Hexanes) to yield 3.19 g (10.5 mmol, 81%) compound **34** as an off white solid. MP = 95-96°C; R_f = 0.18 (30% EtOAc in Hexanes); ^1H -NMR (600 MHz, CDCl_3): δ = 8.22 (d, J = 7.8 Hz, 1H), 7.17-7.20 (m, 2H), 7.01 (t, J = 7.8 Hz, 1H), 4.03 (t, J = 8.4 Hz, 2H), 3.75 (s, 6H), 3.65 (t, J = 7.2 Hz, 1H), 3.65 (t, J = 8.4 Hz, 2H), 2.53 (t, J = 7.2 Hz, 2H), 3.34 (q, J = 7.2 Hz, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 169.7, 142.9, 131.0, 127.5, 124.5, 123.7, 116.9, 52.6, 50.4, 47.8, 32.7, 28.0, 23.6; IR (thin film): ν_{max} = 1321, 2988, 2959, 2916, 2860, 1739, 1662, 1596, 1485, 1441, 1423, 1343, 1295, 1295, 1252, 1230, 1197, 1165, 1010, 891, 773; HRMS calc'd for $\text{C}_{16}\text{H}_{19}\text{NO}_5$ = 305.1263, found 305.1269.

Compound 35

Indoline (647 mg, 5.44 mmol) was dissolved in dry THF (15 mL) in an oven dried round bottomed flask equipped with a magnetic stir bar, condenser, rubber septum and Argon balloon. 1,3-dibromopropane (1.11 mL, 10.9 mmol) was added via syringe. The reaction mixture was stirred at reflux temperature overnight (14 hours) at which point TLC analysis indicated complete consumption of starting material. The reaction was cooled to room temperature and the solvent was removed *in vacuo*. The crude reaction mixture was partitioned between diethyl ether and 5% NaOH. The organic layer was collected and the aqueous layer was extracted twice with diethyl ether. The organic layers were combined and washed once with brine then dried with MgSO_4 , filtered, and solvent removed *in vacuo*. The crude reaction mixture was then purified by flash chromatography on silica gel (EtOAc/Hexanes) to yield 599 mg (3.48 mmol, 64%) compound **35** as a clear oil. R_f = 0.69 (30% EtOAc in Hexanes) ^1H -NMR (400 MHz, CDCl_3): δ = 7.06-7.10 (m, 2H), 6.67 (t, J = 7.2 Hz, 1H), 6.54 (d, J = 8.4 Hz, 1H), 3.55 (t, J = 6.4 Hz, 2H), 3.36 (t, J = 8.0 Hz, 2H), 3.25 (t, J = 6.4 Hz, 2H), 2.99 (t, J = 8.0 Hz, 2H), 2.16 (p, J = 6.4 Hz, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 152.4, 129.8, 127.3, 124.4, 117.6, 106.9, 53.5, 47.6, 31.4, 30.6, 28.6; IR (thin film): ν_{max} = 3047, 3026, 2960, 2823, 1607, 1489, 1471, 1460, 1388, 1362, 1304, 1254, 1216, 1156, 1023, 746, 713; HRMS calc'd for $\text{C}_{12}\text{H}_{15}\text{NO}_4$ = 239.0310, found 239.0308.

Compound 36

Compound **36** was prepared using the same procedure as compound **14** above. Quantities used were as follows: NaH (60% dispersion in oil, 800 mg, 20.0 mmol), dimethyl malonate (2.85 mL, 25.0 mmol), DMF (15 mL) and alkyl bromide **35** (1.20 g, 5.00 mmol) in DMF (8 mL). Purification by flash column chromatography yielded 1.12 g (3.85 mmol, 77%) of compound **36** as a yellow oil (with a minor unknown aliphatic impurity visible by ^1H and ^{13}C NMR). R_f = 0.71 (30% EtOAc in Hexanes) ^1H -NMR (400 MHz, CDCl_3): δ = 7.04-7.08 (m, 2H), 6.64 (t, J = 7.6 Hz, 1H), 6.45 (d, J = 7.6 Hz, 1H), 3.74 (s, 6H), 3.43 (t, J = 7.2 Hz, 1H), 3.31 (t, J = 8.4 Hz, 2H), 3.07 (t, J = 7.2 Hz, 2H), 2.95 (t, J = 8.4 Hz, 2H), 1.99-2.06 (m, 2H), 1.60-1.68 (m, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 169.7, 152.5, 130.0, 127.3, 124.4, 117.5, 106.8, 53.0, 52.5, 51.4, 48.8, 28.5, 26.5, 25.1; IR (thin film): ν_{max} = 3462, 3027, 3003, 2955, 2870, 2847, 1752, 1735, 1607, 1489, 1460, 1437, 1314, 1255, 1234, 1157, 1023, 745; HRMS calc'd for $\text{C}_{16}\text{H}_{21}\text{NO}_4$ = 291.1471, found 291.1478.

Compound 37

Indoline (1.19g 10.0 mmol) and clopropane-1,1-dicarboxylic acid dimethyl ester (1.58g, 10.0 mmol) were dissolved in toluene (15 mL) in an oven dried round bottomed flask equipped with magnetic stir bar. $\text{Yb}(\text{OTf})_3 \cdot x\text{H}_2\text{O}$ (311 mg, 0.5 mmol) was added, the flask was fitted with a condenser, rubber septum and argon balloon. The reaction was stirred at reflux temperature overnight. Toluene was removed *in vacuo* and the crude reaction mixture was redissolved in dichloromethane, pre-adsorbed onto silica gel, and purified by flash chromatography on silica gel (EtOAc/Hexanes) to yield 2.23 g (0.801 mmol, 80%) compound **37** as a yellow oil. R_f = 0.49 (30% EtOAc in Hexanes) ^1H -NMR (600 MHz, CDCl_3): δ = 7.04-7.07 (m, 2H), 6.66 (dd, J = 7.8, 7.2 Hz, 1H), 6.45 (d, J = 7.2 Hz, 1H), 3.72 (s, 6H), 3.58 (t, J = 7.2 Hz, 1H), 3.32 (t, J = 8.4 Hz, 2H), 3.13 (t, J = 7.2 Hz, 2H), 2.95 (t, J = 8.4 Hz, 2H), 2.23 (q, J = 7.2 Hz, 2H); ^{13}C -NMR (100 MHz, CDCl_3): δ = 169.8, 152.2, 129.8, 127.3, 124.4, 117.8, 107.0, 53.3, 52.5, 49.2, 47.3, 28.5, 27.0; IR (thin film): ν_{max} = 3470, 3027, 3001, 2954, 2845, 1751, 1736, 1608, 1490, 1437, 1354, 1237, 1197, 1157, 1023, 748; HRMS calc'd for $\text{C}_{15}\text{H}_{19}\text{NO}_4$ = 277.1314, found 277.1305.

Compound 38

Compound **38** was prepared using the same procedure as compound **37** above. Quantities used were as follows: indoline (227 mg, 1.91 mmol), 2-phenyl-cyclopropane-1,1- dimethyl ester⁵ (447 mg, 1.91 mmol), $\text{Yb}(\text{OTf})_3 \cdot x\text{H}_2\text{O}$ (59 mg, 0.096 mmol) and toluene (10 mL). Purification by flash column chromatography yielded 498 mg (1.27 mmol, 74 %) compound **38** as a clear oil. R_f = 0.56 (30% EtOAc in Hexanes) ^1H -NMR (600 MHz, CDCl_3): δ = 7.21-7.29 (m, 5H), 7.03 (t, J = 7.8 Hz, 1H), 6.98 (d, J = 7.8 Hz, 1H), 6.58 (t, J = 7.8 Hz, 1H), 6.53 (d, J = 7.8 Hz, 1H),

⁵ Corey, E. J.; Chaykovsky, M. *J. Am. Chem. Soc.* **1965**, 87, 1353

4.78 (dd, $J = 10.8, 5.4$ Hz, 1H), 3.69 (s, 3H), 3.64 (s, 3H), 3.58 (t, 7.2 Hz, 1H), 3.36-3.40 (m, 1H), 3.04 (q, $J = 9.0$ Hz, 1H), 2.82-2.93 (m, 2H), 2.65-2.71 (m 1H), 2.51-2.56 (m, 1H); ^{13}C -NMR (100 MHz, CDCl_3): $\delta = 169.9, 169.7, 151.1, 138.7, 129.5, 128.4, 127.7, 127.5, 127.2, 124.5, 117.1, 106.7, 56.5, 52.6, 52.4, 49.2, 46.2, 30.4, 28.0$; IR (thin film): $\nu_{\text{max}} = 3029, 2953, 2848, 1751, 1734, 1606, 1487, 1473, 1461, 1457, 1436, 1256, 1204, 1156, 745, 701$; HRMS calc'd for $\text{C}_{12}\text{H}_{15}\text{NO}_4 = 353.1627$, found 353.1624.

Compound 41

Compound **19** (578 mg, 1.70 mmol) was dissolved in anhydrous EtOH (75 mL) and dry THF (50 mL) in a 500 mL vessel. Raney-Nickel[®] (2400 slurry in water, approx. 0.5 g) was added using a spatula. The reaction vessel was then shaken for 48 hours in a Parr hydrogenator under an H_2 pressure of 4 atm. The reaction mixture was then filtered through celite and solvent removed *in vacuo* yielding a brown solid which was triturated with EtOAc to yield 460 mg (1.47 mmol, 87%) compound **41** as a fine white powder (very insoluble in common organic solvents). MP = 253-255°C; $R_f = 0.63$ (100% EtOAc) ^1H -NMR (400 MHz, CDCl_3): 8.53 (d, $J = 8.4$ Hz, 1H), 7.30-7.45 (m, 3H), 6.21 (t, $J = 7.2$ Hz, N-H), 3.83 (s, 3H), 3.55-3.60 (m, 1H), 3.45-3.51 (m, 1H), 2.96-2.99 (m, 2H), 2.85-2.91 (m, 1H), 2.60-2.74 (m, 3H); $\delta = ^{13}\text{C}$ -NMR (100 MHz, DMSO): 171.0, 168.2, 168.0, 133.7, 129.3, 127.4, 125.5, 123.9, 118.7, 115.8, 53.6, 53.0, 37.3, 29.7, 28.5, 25.6; $\delta =$ IR (thin film): $\nu_{\text{max}} = 3103, 2958, 1725, 1706, 1367, 1240, 1194, 1175, 1149, 796, 762, 751$ HRMS calc'd for $\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_4 = 312.1110$, found 312.1109.

^1H -NMR (400 MHz, DMSO) for comparison with previous literature⁶: $\delta = 8.34$ -8.37 (m, 2H), 7.54 (d, $J = 8.0$ Hz, 1H), 7.29-7.40 (m, 2H), 3.74 (s, 3H), 3.21-3.40 (m, 2H, plus large overlapping H_2O peak), 2.58-2.97 (m, 5H), 2.32-2.36 (m, 1H).

⁶ Compound **41** has been previously characterized: Mahboobi, S; Burgemeister, T. Kastner, F; *Archiv der Pharmazie*. **1995**, 328(1), 29-38. ^1H NMR data was provided as follows: ^1H -NMR (250 MHz, DMSO): $\delta = 2.38$ -3.51 (m, 9H, 4 CH_2 , NH), 3.75 (s, 3H, COOCH_3), 7.29-8.39 (m, 4 arom. H). In contrast to these authors, we see the amide NH fall in the aromatic region in this spectrum.

jw-11-23a
expi PROTON

SAMPLE		SPECIAL	
date	Dec 4 2005	temp	25.0
solvent	CDCl3	gain	not used
file	/export/home/~	spin	22
kerr/vnmr/sys/data/~	hst	0.000	
jw-11-23a_2005-12-~	pw90	10.300	
85/PROTON_01.fid	alpha	0	

ACQUISITION		FLAGS	
sv	9592.3	l1	n
at	1.000	in	n
np	36290	dp	y
fb	5800	hs	nn
bs	32	fn	not used
d1	1.000	fn	not used
nt	16	fn	not used
ct	16	sp	213.9

TRANSMITTER		PLOT	
tn	H1	rf1	5537.7
sfrq	599.495	rpf	4352.3
tof	599.3	rp	163.9
tpwr	57	lp	-32.9
pw	5.150	vc	250
dn	C13	sc	0
dof	0	vs	35
dm	nm	th	27
decwave	0	ai	cdc ph
dpwr	45		
daf	17094

Compound 12
¹H 600 MHz

jw-13-19bb
STANDARD 1H OBSERVE

expi s2pul

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/Kerr/jw-13-19bb/C-	pw90	10.400	
ARBON.fid	alpha	20.000	

ACQUISITION		FLAGS	
sv	25125.6	l1	n
at	1.199	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	fn	not used
d1	1.000	lb	1.00
nt	512	fn	not used
ct	512	fn	not used

TRANSMITTER		PLOT	
tn	C13	sp	-536.6
sfrq	100.613	rf1	21130.6
tof	1550.0	rpf	9272.9
tpwr	50	rp	7746.3
pw	5.200	lp	-179.1
dn	H1	vc	250
dof	0	sc	0
dm	yy	vs	125
dwa	w	th	9
dpwr	41	ai	no ph
daf	9900		

Compound 12
¹³C 100 MHz

jm-10-103a inova 600

exp1 PROTON

```

SAMPLE
date Oct 20 2005 temp 25.0
solvent CDCl3 gain not used
file /export/home/~ spin 22
kerr/vnmr/syst/data/~ hst 0.000
jm-10-103a_2005-10 pw9 10.900
-20/PROTON_01.fid alfa 0
ACQUISITION
sw 5592.3 f1 n
at 1.882 in n
np 36298 dp y
fb 5800 hs nn
bs 32
dl 1.800 fn not used
nt 16
ct 16
TRANSMITTER
tn H1 rf1 453.7
sfrq 599.495 rfp 5768.1
tof 599.3 rp 5537.4
tpwr 53 lp 4352.3
pw 5.450 PLOT 250
DECOUPLER C13 sc 0
dn 6 vs 12
dof 6 th 3
decwave V40_hcx al cdc ph
dpwr 38
daf 6570

```

Compound 13
 ^1H 400 MHz

jm-10-103a
STANDARD 1H OBSERVE

exp1 s2pu1

```

SAMPLE
date Oct 20 2005 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/vnmr/syst/data/~ hst 0.000
/Kerr/jm-10-103a/C- pw9 11.600
ARBON.fid alfa 20.000
ACQUISITION
sw 25125.6 f1 n
at 1.199 in n
np 68270 dp y
fb 13000 hs nn
bs 64
dl 1.000 lb 1.00
nt 512 fn not used
ct 512
TRANSMITTER
tn C13 wp 1864.2
sfrq 100.613 rf1 17165.5
tof 1550.8 rfp 9269.6
tpwr 57 rp 7746.3
pw 5.000 lp -144.6
DECOUPLER H1 vc 250
dn 0 sc 0
dof 0 vs 103
dsw 4 th 6
dpwr 40 al no ph
daf 9300

```

Compound 13
 ^{13}C 100 MHz

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jm-13-53a
exp2  PROTON

SAMPLE      PRESATURATION
date Jun 5 2006  satmode n
solvent CDC13  wet      n
file /export/home/~
kerr/vmarsys/data/~
jm-13-53a_2006-06-~
95/szpu1-01  spin      20
ACQUISITION  hst        0.000
sw 9501.2  pw90        10.000
at 1.786  alpha        6.000
np 32760  l1          n
fb 5000  l1          n
bs 32  fn          n
dl 1.000  dp          y
nt 16  hs          nn
ct 16  PROCESSING  nn

TRANSMITTER H1  fn  not used
tn          H1  DISPLAY -51.3
sfrq 599.463  sp          5330.6
tof 599.4  wp          5337.9
tpwr 56  rfp          4352.1
pw 5.150  rp          156.4
DECOUPLER C13  lp          -35.6
dn          PLOT 250
dof 0  wc          0
decwave V40_hcx  sc          0
dpwr 43  vs          24
dntf 35000  ch          54
a1 cdc ph

```

Compound 14
¹H 600 MHz

Compound 14
¹³C 100 MHz

```

jm-13-53a
STANDARD 1H OBSERVE

exp2  szpu1

SAMPLE      SPECIAL
date Jun 6 2006  temp  not used
solvent cdc13  gain  not used
file /export/home/~
vmarsys/data/~
jm-13-53a_2006-06-~
95/szpu1-01  spin      20
ACQUISITION  hst        0.000
sw 9501.2  pw90        10.000
at 1.786  alpha        6.000
np 32760  l1          n
fb 5000  l1          n
bs 32  fn          n
dl 1.000  dp          y
nt 16  hs          nn
ct 16  PROCESSING  nn

TRANSMITTER H1  fn  not used
tn          H1  DISPLAY -127.1
sfrq 599.463  sp          5330.6
tof 599.4  wp          5337.9
tpwr 56  rfp          4352.1
pw 5.150  rp          156.4
DECOUPLER C13  lp          -35.6
dn          PLOT 250
dof 0  wc          0
decwave V40_hcx  sc          0
dpwr 43  vs          24
dntf 35000  ch          54
a1 cdc ph

```

jm-9-195a
expt1 PROTON

```

SAMPLE
date Sep 12 2005 temp 25.0
solvent CDCl3 gain not used
file /export/home/~ spin 22
kerr/vnmrsys/data/~ hst 0.008
jm-9-195a_2005-09- pw9 10.900
12/PROTON_01.fid alfa 0

ACQUISITION
sw 9582.3 f1 n
at 1.882 in n
np 36298 dp y
fb 5000 hs nn
bs 32
dl 1.000 fn not used
nt 16
ct 16 sp DISPLAY
TRANSMITTER
tn H1 rf1 177.6
sfrq 599.495 rfp 5192.0
tof 599.3 rp 5548.0
tpwr 53 lp 4352.3
pw 5.450 plot -167.3
DECOUPLER
dn C13 wc 250
dof 0 vs 0
ds nnn th 22
decwave W40_hcx al cdc ph
dpr 39
def 6578

```

Compound 15
¹H 600 MHz

jm-9-195a carbon inova 600
expt1 CARBON

```

SAMPLE
date Sep 12 2005 temp 25.0
solvent CDCl3 gain not used
file /export/home/~ spin 16
kerr/vnmrsys/data/~ hst 0.008
jm-9-195a_2005-09- pw0 20.900
12/CARBON_01.fid alfa 10.000

ACQUISITION
sw 37682.5 f1 n
at 1.300 in n
np 96818 dp y
fb 21000 hs nn
bs 64
dl 1.000 lb PROCESSING
nt 256 fa 0.50
ct 256 DISPLAY
TRANSMITTER
tn C13 wp 3283.6
sfrq 150.759 rf1 23322.2
tof 3127.7 rfp 13869.9
tpwr 57 rp 11697.2
pw 10.450 lp -163.0
DECOUPLER
dn H1 wc 250
dof 0 sc 0
ds yyy vs 495
decwave w th
dpr 36 al cdc ph
def 13158

```

Compound 15
¹³C 100 MHz

jm-11-111a
STANDARD 1H OBSERVE

exp1 s2pul

SAMPLE		SPECIAL	
date	Jan 26 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
vmr1/vmrsys/data/~	hst	0.000	
/Kerr/jm-11-111a/p-	pw90	14.000	
ROTON	f1d	alpha	168.000

ACQUISITION		FLAGS	
sw	5599.1	l1	n
at	4.000	in	n
np	44792	dp	y
fb	not used	hs	nn
bs	16		
d1	2.000	lsfid	-1
nt	120	fn	not used
ct	120		

TRANSMITTER		PLOT	
tn	H1	wp	20.9
sfrq	400.007	rfl	3454.0
tof	17.2	rpf	3703.4
tpwr	57	rp	2904.6
pw	7.300	lp	172.0
			-24.6

DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	14
dms	c	th	54
dpwr	43	ai	cdc
daf	15000		ph

Compound 16
¹H 400 MHz

jm-11-111a
STANDARD 1H OBSERVE

exp1 s2pul

SAMPLE		SPECIAL	
date	Jan 26 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
vmr1/vmrsys/data/~	hst	0.000	
/Kerr/jm-11-111a/C-	pw90	11.000	
ARBON	f1d	alpha	20.000

ACQUISITION		FLAGS	
sw	25125.6	l1	n
at	1.100	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64		
d1	1.000	lb	1.00
nt	1000	fn	not used
ct	1000		

TRANSMITTER		PLOT	
tn	C13	wp	553.0
sfrq	100.613	rfl	16074.0
tof	1550.0	rpf	9270.6
tpwr	57	rp	7746.3
pw	5.800	lp	-123.0
			-320.0

DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yyy	vs	140
dms	v	th	3
dpwr	40	ai	no
daf	9300		ph

Compound 16
¹³C 100 MHz

jm-11-151a
expl s2pul

SAMPLE		SPECIAL	
date	Feb 16 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		SPECIAL	
sw	6395.3	hst	0.000
at	3.744	pw90	11.700
np	47892	alfa	6.600
fb	4800	FLAGS	
bs	16	in	n
d1	1.000	dp	y
nt	32	hs	nn
ct	32	PROCESSING	
TRANSMITTER		DISPLAY	
tn	H1	fn	not used
sfrq	399.762	sp	-3.0
tof	399.3	wp	3454.1
tpwr	57	rfl	3686.8
pw	5.650	rfp	2902.3
DECOUPLER		PLOT	
dn	C13	lp	-27.5
dof	0	nnn	250
dm	c	sc	0
dpr	37	vs	41
daf	17094	th	2
	ai	cdc	ph

Compound 18
 ^1H 400 MHz

jm-12-184ccc
STANDARD 1H OBSERVE

expl s2pul

SAMPLE		SPECIAL	
date	May 2 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~ spin	spin	not used
nmr1/vnmrsvs/data/~	hst	hst	0.000
/kerr/jm-12-184ccc-	pw90	pw90	10.400
/CARDON.fid	alfa	alfa	26.000
ACQUISITION		FLAGS	
sw	25125.6	il	n
at	1.199	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	PROCESSING	
d1	1.000	lb	1.00
nt	1000	fn	not used
ct	1000	DISPLAY	
TRANSMITTER		PLOT	
tn	C13	sp	-149.4
sfrq	100.613	wp	29420.2
tof	1550.6	rfl	9266.7
tpwr	59	rfp	7746.3
pw	5.200	lp	-379.3
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yyv	vs	312
dpr	41	th	0
daf	9900	ai	no ph

Compound 18
 ^{13}C 100 MHz

jm-10-135cr inova 400

expl s2pul

SAMPLE		SPECIAL	
date	Nov 2 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.000
sw	6395.9	pw90	11.700
at	3.744	alfa	6.600
np	47032	FLAGS	
fb	4000	il	n
bs	16	in	n
dl	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	
tn	TRANSMITTER	fn	not used
sfrq	399.762	sp	DISPLAY
tof	399.0	vp	-44.2
tpwr	57	rfl	3521.5
pw	5.850	rpf	3686.0
DECOUPLER	rp	2982.3	
dn	C13	lp	-155.1
dof	0	PLT	-3.5
dm	nnn	vc	250
dma	c	sc	0
dpwr	37	vs	39
daf	17094	th	2
	a1	cdc	ph

Compound 19
¹H 400 MHz

jm-10-104dd

STANDARD 1H OBSERVE

expl s2pul

SAMPLE		SPECIAL	
date	May 1 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/hoac/-	spin	not used
vnmr1/vnmrsys/data-	hst	0.000	
/Kerr/jm-12-104dd/-	pw90	10.400	
ACQUISITION		alfa	20.000
sw	25125.6	il	n
at	1.109	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	PROCESSING	
dl	1.000	lb	1.00
nt	512	fn	not used
ct	512	DISPLAY	
tn	TRANSMITTER	sp	-48.9
sfrq	C13	vp	20304.0
tof	100.613	rfl	9270.6
tpwr	1550.8	rpf	7746.3
pw	5.200	lp	-167.7
DECOUPLER	rp	PLT	-349.0
dn	H1	vc	250
dof	0	sc	0
dm	yvv	vs	222
dma	w	th	11
dpwr	41	a1	no
daf	9900	ph

Compound 19
¹³C 100 MHz

```

jm-12-179a
expl s2pu1

SAMPLE          SPECIAL
date May 2 2006 temp not used
solvent CDCl3   gain not used
file          exp spin    2.0
ACQUISITION    hst      0.000
sw 6395.9      pw90     11.700
at 3.744       alfa     6.600
np 47892       flags
fb 4000        il      n
bs 16          in      n
d1 1.000       dp      y
nt 16         hs      nn
ct 16         fn      not used

TRANSMITTER     H1      not used
tn              sp      DISPLAY -3.2
sfrq 399.762    vp      3925.0
tof 399.0       rfi      3687.0
tpwr 5.850     rfp      2992.3
pw          rp      89.1
DECOUPLER C13      ip      -7.6
dn          0      PLOT 250
dof         nnn    wc      0
dm          c      sc      0
dpwr        37    vs      23
dmf        17094  th      54
                  al cdc ph

```

Compound 21
¹H 400 MHz

```

jm-12-183aa
STANDARD 1H OBSERVE
expl s2pu1

SAMPLE          SPECIAL
date May 2 2006 temp not used
solvent cdcl3   gain not used
file /export/home/~ spin not used
vnmr1/vnmr5/data/~ hst 0.000
/Kerr/jm-12-183aa/~ pw90 10.400
CARBON.F1d alfa 20.000
ACQUISITION    flags
sw 25125.6     il      n
at 1.199       in      n
np 60270       dp      y
fb 13800       hs      nn
bs 64          flags
d1 1.000       lb      1.00
nt 512         fn      not used
ct 512         DISPLAY -67.3

TRANSMITTER     C13      not used
tn              sp      20324.7
sfrq 100.613    rfi      9200.3
tof 1550.8     rfp      7746.3
tpwr 5.200     rp      -160.5
pw          lp      -355.6
DECOUPLER H1      vc      250
dn          0      sc      0
dof         yyy    vs      210
dm          v      th      10
dpwr        41    al no ph
dmf        9980

```

Compound 21
¹³C 100 MHz

jm-18-99a inova 400

expt s2pu1

SAMPLE		SPECIAL	
date	Oct 18 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.000
sw	6395.3	pw90	11.700
at	5.744	alpha	6.600
np	47892	FLAGS	n
fb	4000	l1	n
bs	16	in	n
dl	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	not used
tn	TRANSMITTER	fn	DISPLAY
sfrq	399.762	sp	-4.2
tof	399.0	wp	3684.4
tpwr	57	rfl	3680.0
pw	5.850	rfl	2982.3
DECOUPLER		rp	154.9
dn	C13	lp	-19.8
dof	0	PLOT	258
da	nn	vc	0
dma	c	sc	0
dpwr	37	vs	25
dnt	17086	th	2
	a1	cdc	ph

Compound 22
¹H 600 MHz

jm-18-99a
STANDARD IN OBSERVE

expt s2pu1

SAMPLE		SPECIAL	
date	Oct 18 2005	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~ spin	spin	not used
nmr1/vnmrsvs/data/~	hst	hst	0.000
/Kerr/jm-18-99a/CA-	pw90	pw90	11.600
800N.fid	alpha	alpha	20.000
ACQUISITION		FLAGS	n
sw	25125.6	l1	n
at	1.199	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	PROCESSING	not used
dl	1.000	lb	1.00
nt	512	fn	not used
ct	512	DISPLAY	1407.2
tn	TRANSMITTER	sp	16684.2
sfrq	100.613	rfl	9270.6
tof	1556.8	rfl	7746.3
tpwr	57	rp	-122.3
pw	5.800	lp	-348.7
DECOUPLER		PLOT	250
dn	H1	vc	0
dof	0	sc	0
da	yyv	vs	107
dma	v	th	13
dpwr	40	a1	no
dnt	9999	ph

Compound 22
¹³C 100 MHz

je-12-184f Mn-2

exp2 szpu1

SAMPLE		SPECIAL	
date	May 1 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION			
sw	6395.9	hst	0.000
at	3.744	pufo	11.700
np	47892	alfa	6.600
fb	4000	FLAGS	
bs	16	il	n
dl	1.000	in	n
nt	16	hs	y
ct	16	nn	nn
PROCESSING			
tn	H1	fn	not used
sfrq	399.762	sp	88.9
toF	399.0	wp	3070.0
tpwr	57	rfl	3666.0
pw	5.850	rfp	2902.3
DECOUPLER			
dn	C13	lp	-10.5
dof	0	PLOT	250
dm	nnn	vc	0
dma	c	sc	0
dpwr	37	vs	44
daf	17000	th	3
	al	cdc	ph

Compound 23
 ^1H 400 MHz

je-12-184ff
STANDARD IN OBSERVE

exp1 szpu1

SAMPLE		SPECIAL	
date	May 1 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~ spin	spin	not used
vnarl/vnarsys/data~	hst	0.000	
/Kerr/je-12-184ff/~	pufo	10.400	
CARBON:fid			
sw	25105.6	il	n
at	1.199	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	PROCESSING	
dl	1.000	lb	1.00
nt	1000	fn	not used
ct	1000	DISPLAY	
TRANSMITTER			
tn	C13	sp	-211.5
sfrq	100.613	wp	20470.4
toF	1550.0	rfl	3266.7
tpwr	59	rfp	7746.3
pw	5.200	lp	-314.0
DECOUPLER			
dn	H1	vc	250
dof	0	sc	0
dm	yyy	vs	203
dma	w	th	12
dpwr	41	al	no ph
daf	9900

Compound 23
 ^{13}C 100 MHz

```

jm-12-191b
exp1 s2pu1

SAMPLE
date May 4 2006 temp not used
solvent CDCl3 gain not used
file exp spin 20
ACQUISITION
sw 6395.9 hst 0.000
at 3.744 pwr 11.700
np 47892 flad 6.600
fb 4000 i1 n
bs 16 in n
d1 1.000 dp y
nt 32 hs nm
ct 32

TRANSMITTER H1 fn not used
tn sfrq 399.762 sp DISPLAY -19.6
tof 399.6 wp 3584.7
tpwr 57 rfl 2902.9
pw DECOUPLER 5.650 rfp 2126.7
dn C13 lp -32.1
dof 0
dm nnn vc 250
dwa c sc 0
dpu 37 vs 40
def 17094 th 2
a1 cdc ph 2

```

Compound 24
 ^1H 400 MHz

jm-12-191bb
STANDARD IN OBSERVE

```

exp1 s2pu1

SAMPLE
date May 4 2006 temp not used
solvent cdc13 gain not used
file /export/home/ spin not used
vnmr1/vnmr5ys/data- hst 0.000
/Kerr/jm-12-191bb/ pwr 10.400
CARBON.fid alfa 20.000
ACQUISITION
sw 25125.6 i1 n
at 1.199 in n
np 60270 sp y
fb 13000 hs nm
bs 64
d1 1.000 th 1.00
nt 1000 fn not used
ct 1000 DISPLAY -364.3
TRANSMITTER C13 wp 20230.0
tn sfrq 100.613 rfl 9266.7
tof 1550.0 rfp 7746.3
tpwr 59 rfp -168.0
pw DECOUPLER 5.200 lp -342.6
dn H1 wc 250
dof 0 sc 0
dm yyy vs 367
dwa w th 10
dpu 41 a1 no ph 10
def 9900

```

Compound 24
 ^{13}C 100 MHz

```

jm-12-199a
expl s2pul

SAMPLE
date May 8 2006 temp not used
solvent CDCl3 gain not used
file 090 spin 20
ACQUISITION
sw 6395.9 hst 0.000
at 3.744 pps 11.700
np 47892 alfa 6.600
fb 4000
bs 16 in n
d1 1.000 dp y
nt 16 hs nn
ct 16

TRANSMITTER H1 fn not used
tn 399.762 sp DISPLAY 49.3
sfrq 399.8 wp 3429.4
tpwr 57 rfl 799.4
pw 5.850 rfp 0
DECOUPLER C13 lp -21.9
dn 9
dof 3 PLOT 250
ds 0
dss 0
dpr 37 vs 50
dnt 17094 th 54
a1 cdc ph

```

Compounds 27 and 28
2:1 ratio
¹H 400 MHz

```

jm-12-199a
STANDARD 1H OBSERVE
expl s2pul

SAMPLE
date May 8 2006 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin not used
nmr1/vnmrsys/data/~ hst 0.000
/Kerr/jm-12-199a/C- ARBON.fid alfa 20.000
ACQUISITION
sw 25125.6 f1 n
at 1.190 in n
np 60270 dp y
fb 13000 hs nn
bs 64
d1 1.000 lb 1.00
nt 512 fn not used
ct 512

TRANSMITTER C13 wp DISPLAY -67.3
tn 100.613 rfl 20345.4
sfrq 1550.9 rfp 9268.3
tpwr 59 rfp 7746.3
pw 5.200 lp -322.5
DECOUPLER H1 wc PLOT 250
dn 0
dof 0 sc 0
ds yyy vs 207
dss w th 8
dpr 41 a1 no ph
dnt 9900

```

Compounds 27 and 28
¹³C 100 MHz

jm-11-183a
expt1 PROTON

SAMPLE		SPECIAL	
date	Feb 24 2006	temp	25.9
solvent	CDCl3	gain	not used
file	/export/home/~	spin	22
kerr/vnmrsys/data/~	hst	0.000	
jm-11-183a_2006-02~	pw99	10.399	
-24/PROTON_01.fid	alpha	0	
ACQUISITION		FLAGS	
sv	9591.2	l1	n
at	1.882	in	n
np	36294	dp	y
rb	5806	hs	nn
bs	32	PROCESSING	
dl	1.600	fn	not used
nt	16	DISPLAY	
ct	16	sp	769.8
TRANSMITTER		vp	4411.8
tn	H1	rf1	5545.5
sfrq	599.479	rfp	4352.2
tof	599.3	rp	-10.8
tpwr	57	lp	-31.5
pw	5.150	PLOT	
dn	DECOUPLER	wc	250
dof	C13	sc	0
dm	0	vs	24
decwave	0	ai	cdc ph
dpr	45		
def	17094

Compound 34
 ^1H 600 MHz

jm-12-43cr
STANDARD 1H OBSERVE

expt1 s2pul

SAMPLE		SPECIAL	
date	Mar 15 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
vnmrs/vnmrsys/data/~	hst	0.000	
/Kerr/jm-12-43cr/C~	pw99	11.000	
ARBON.fid	alpha	20.000	
ACQUISITION		FLAGS	
sv	25125.6	l1	n
at	1.199	in	n
np	60270	dp	y
rb	13880	hs	nn
bs	64	PROCESSING	
dl	1.000	lb	1.00
nt	2000	fn	not used
ct	2000	DISPLAY	
TRANSMITTER		sp	439.3
tn	C13	vp	19741.2
sfrq	100.613	rf1	9267.5
tof	1550.8	rfp	7746.3
tpwr	50	rp	172.1
pw	5.500	lp	-327.3
dn	DECOUPLER	H1	wc
dof	0	sc	0
dm	yyv	vnc	419
dmm	w	tle	0
dpr	41	ai	no ph
def	9900

Compound 34
 ^{13}C 100 MHz

```

jm-13-23a
exp1 s2pu1

SAMPLE
date May 15 2008 temp SPECIAL
solvent cdc13 gain not used
file /export/home/~ sp1n not used
vnmr1/walkup/auto_ hst 8.000
ACQUISITION 15.85.06/0105 pw98 10.400
sw 25125.6 flags 28.000
at 1.198 i1 n
np 60270 in n
fb 13800 dp y
bs 64 hs
d1 1.000 PROCESSING
nt 512 tb 1.00
ct 512 fn not used

TRANSMITTER
tn c13 ep -140.2
sfrq 100.613 wp 18365.5
tof 1550.8 rfi 9271.3
tpwr 5.200 rfp 7746.3
pw 5.200 rp -146.1
DECOUPLER H1 lp -390.5
dn 8 wc PLOT
dof 8 vc 250
dm yyj sc 0
dwa w vs 144
dpwr 41 th 11
def 9909 al no ph
  
```

Compound 35
¹H 400 MHz

```

jm-13-23a
STANDARD 1H OBSERVE
  
```

```

exp1 s2pu1

SAMPLE
date May 15 2008 temp SPECIAL
solvent cdc13 gain not used
file /export/home/~ sp1n not used
vnmr1/walkup/auto_ hst 8.000
ACQUISITION 15.85.06/0105 pw98 10.400
sw 25125.6 flags 28.000
at 1.198 i1 n
np 60270 in n
fb 13800 dp y
bs 64 hs
d1 1.000 PROCESSING
nt 512 tb 1.00
ct 512 fn not used

TRANSMITTER
tn c13 ep -140.2
sfrq 100.613 wp 18365.5
tof 1550.8 rfi 9271.3
tpwr 5.200 rfp 7746.3
pw 5.200 rp -146.1
DECOUPLER H1 lp -390.5
dn 8 wc PLOT
dof 8 vc 250
dm yyj sc 0
dwa w vs 144
dpwr 41 th 11
def 9909 al no ph
  
```

Compound 35
¹³C 100 MHz

jm-12-197a
exp2 PROTON

SAMPLE		PRESATURATION	
date	May 8 2006	satmode	n
solvent	CDCl3	wet	n
file	/export/home/~	SPECIAL	
kerr/vnmrsys/data/~	temp	not used	
jm-12-197a_2006-05-	gain	not used	
-05/s2pu1-01	spin	22	
ACQUISITION		hst	0.000
sw	9591.2	pw90	10.300
at	1.700	alpha	0.000
np	32760	FLAGS	
fb	5000	il	n
bs	32	in	n
d1	1.000	dp	y
nt	16	hs	nn
ct	16	PROCESSING	
TRANSMITTER		fn	not used
tn	H1	DISPLAY	
sfreq	599.463	sp	47.9
tof	599.4	wp	5330.6
tpwr	56	rfl	2417.1
pw	5.150	rfp	1216.9
DECOUPLER		rp	-114.9
dn	C13	lp	-27.0
dof	0	PLOT	250
dm	nnn	wc	
decwave	W40_hcx	sc	0
dpwr	43	vs	30
daf	35000	th	54
	at	cdc ph

Compound 38
¹H 600 MHz

jm-12-197a
STANDARD 1H OBSERVE

exp1 s2pu1

SAMPLE		SPECIAL	
date	May 8 2006	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
vnmr/vnmrsys/data/~	hst	0.000	
/Kerr/jm-12-197a/C-	pw90	10.400	
ARBON.fid	alpha	20.000	
ACQUISITION		FLAGS	
sw	25125.6	il	n
at	1.199	in	n
np	60270	dp	y
fb	13000	hs	nn
bs	64	PROCESSING	
d1	1.000	lb	1.00
nt	512	fn	not used
ct	512	DISPLAY	
TRANSMITTER		sp	14.7
tn	C13	wp	20220.4
sfreq	100.613	rfl	9269.0
tof	1550.0	rfp	7746.3
tpwr	59	rp	177.0
pw	5.200	lp	-331.9
DECOUPLER		rp	
dn	H1	wc	250
dof	9	sc	0
dm	yyy	vs	176
dms	4	th	11
dpwr	41	at	no ph
dnt	9900

Compound 38
¹³C 100 MHz

Compound 40
¹H 600 MHz

Compound 40
 ^{13}C 100 MHz

je-13-amide
STANDARD 1H OBSERVE

exp1 s2pu1

SAMPLE		SPECIAL	
date	Aug 10 2006	temp	not used
solvent	dms	gain	not used
file	/export/home/~	spin	20
vnarl/vnmr/sys/data/~	hst	0.008	
/Kerr/je-13-amide/~	pw98	12.300	
PROTON.fid	alfa	168.600	
ACQUISITION		FLAGS	
sw	5599.1	fl	n
at	4.000	in	n
np	44792	dp	y
rb	not used	hs	nn
bs	16	PROCESSING	
dl	2.000	lsfid	-1
nt	128	fn	not used
ct	128	DISPLAY	
TRANSMITTER		PLOT	
tn	H1	wp	17.6
sfrq	400.089	rf1	3952.2
tof	17.2	rpf	1791.0
tpwr	59	rp	996.2
pw	6.150	lp	173.8
DECOUPLER			-28.5
dn	C13	wc	250
dof	0	sc	0
de	nnn	vs	106
dms	c	th	2
dpwr	44	cdc	ph
daf	18500

Compound 41
 ^1H 400 MHz in DMSO