Assembling Polycyclic Bisguanidine Motifs Resembling Batzelladine Alkaloids by Double Tethered Biginelli Condensations

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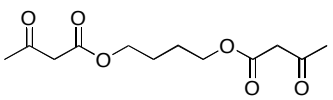
Supporting Information:

Experimental procedures and characterization data for bis-β-ketoesters **6**, **10–15**; double Biginelli products **9**, **16–21**, **25** and **27**; copies of ^{1}H and ^{13}C spectra for these new compounds, selected minor double Biginelli products and **23** (R = *n*-C_7H_{15}) (33 pages).

General Experimental. All reactions were carried out under a N_2 or Ar atmosphere in oven-dried and base-washed glassware. Tetrahydrofuran (THF), diethyl ether, and dichloromethane were degassed with argon and then passed through two 4 x 36 inch columns of anhydrous neutral A-2 alumina (8 x 14 mesh; activated under a flow of Ar at 350 °C for 3h) to remove water.[1] Toluene (PhMe) was degassed with argon, passed through one 4 x 36 inch column of Q-5 reactant (activated under a flow of 5% hydrogen/nitrogen at 250 °C for 3h) to remove oxygen and then through one 4 x 36 inch column of anhydrous alumina to remove water.[1] Silica gel (0.040–0.063 mesh) was used for flash chromatography unless otherwise stated. Molarities of organolithium reagents were established by titration with diphenylacetic acid. Trifluoroethanol (TFE) was purchased from Aldrich and used without further purification. ^{1}H NMR chemical shifts are reported as δ values in ppm relative to $CHCl_3$ or MeOH. ^{1}H NMR coupling constants are reported in Hz and refer to apparent multiplicities and not true coupling constants. Multiplicity is indicated as follows: s (singlet); d (doublet); q (quartet); m (multiplet); app d (apparent doublet); app t (apparent triplet); dd (doublet of doublets); ddd (doublet, doublet of doublets); dddd (doublet, doublet, doublet of doublets); br s (broad singlet). ^{1}H NMR spectra of guanidine double Biginelli products show few resolved signals; these spectra are not tabulated, but the original spectra are reproduced. The IR spectra absorptions are recorded as wavenumbers (cm^{-1}).



3-Oxobutyric acid 4-(3-oxobutyryloxy)butyl ester (10). General procedure for preparing bis-β-ketoesters using the Taber protocol.[2] A solution of DMAP (677 mg, 5.55 mmol), methyl

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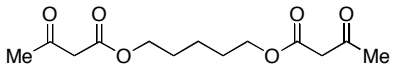
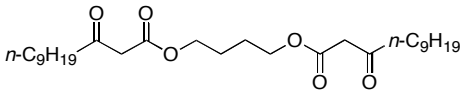
[1] Pangborn, A. B.; Giardello, M. A.; Grubbs, R. H.; Rosen, R. K.; Timmers, F. J. *Organometallics* **1996**, *15*, 1518.
[2] Taber, D. F.; Amedio, J.C.; Patel, Y. K. *J. Org. Chem.* **1985**, *50*, 3618.

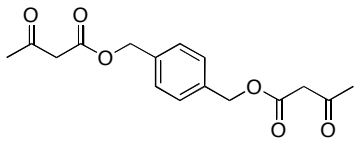
acetoacetate (1.8 mL, 17 mmol), 1,4-butanediol (500 mg, 5.5 mmol) and toluene (10 mL) was heated to 110 °C for 18 h. The reaction was allowed to cool to room temperature and then was concentrated in vacuo. The residue was purified by chromatography (1:4 ethyl acetate:hexanes) to afford 1.29 g (90%) of **10** as a yellow oil: ^{1}H NMR (500 MHz, $CDCl_3$) 4.04–4.02 (m, 4H), 3.36 (s, 4H), 2.13 (s, 6H), 1.62–1.59 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) 200.25, 166.6, 64.2, 49.42, 29.65, 24.6; IR (thin film) 2964, 1737, 1713, 1648, 1412, 1316, 1175, 1038, 955 cm^{-1}; HRMS (CI$^+$) *m/z* 259.1180 (259.1181 calcd for $C_{12}H_{19}O_6$ $[M+H]^+$).



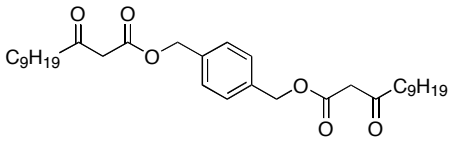
3-Oxododecanoic acid 4-(3-oxododecanoyloxy)butyl ester (6). Purification by chromatography (5:1–1:1 hexanes:EtOAc) afforded 85% of **6** as a colorless oil: ^{1}H NMR (400 MHz, $CDCl_3$) 4.2–4.1 (m 4H), 3.46 (s, 4H), 2.27 (s, 6H), 1.72–1.65 (m, 4H), 1.47–1.41 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) 200.5, 167.1, 65.0, 50.0, 30.2, 28.0, 22.2; IR (thin film) 2957, 1741, 1717, 1319, 1152 cm^{-1}; HRMS (CI$^+$) *m/z* 273.1339 (273.1340 calcd for $C_{13}H_{21}O_6$, MH^+).



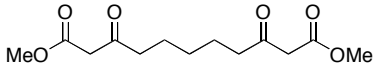
3-Oxododecanoic acid 4-(3-oxododecanoyloxy)butyl ester (11). Purification by chromatography (10:1 hexanes:EtOAc) afforded 48% of **11** as a colorless oil: ^{1}H NMR (400 MHz, $CDCl_3$) 4.20–4.18 (m, 4H), 3.43 (s, 4H), 2.52 (dt, J = 10.0, 1.8 Hz, 4H), 1.71–1.63 (m, 2H), 1.61–1.56 (m, 4H), 1.45–1.39 (m, 2H), 1.30–1.18 (m, 24H), 0.90 (t, J = 8.0 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) 202.6, 167.06, 64.4, 48.9, 42.9, 31.7, 29.25, 29.2, 29.1, 28.9, 23.3, 22.5, 13.9; IR (thin film) 2920, 1738, 1413, 1321, 1267, 1159, 1050, 965, 733 cm^{-1}; HRMS (CI$^+$) *m/z* 483.3686 (483.3685 calcd for $C_{29}H_{50}O_6$, MH^+).



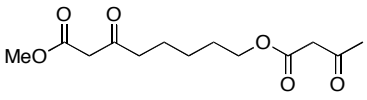
3-Oxobutyric acid 4-(3-oxobutyryloxymethyl)benzyl ester (12). Purification by chromatography (5:1 hexanes:EtOAc) afforded 70% of **12** as a colorless powder: ^{1}H NMR (500 MHz, $CDCl_3$) 7.42 (s, 4H), 5.23 (s, 4H), 3.56 (s, 4H), 2.31 (s, 6H); ^{13}C (125 MHz, $CDCl_3$,) 200.5, 167.1, 135.8, 128.8, 66.9, 50.21, 30.4; IR (thin film) 2960, 1742, 1713, 1410, 1316, 1268, 1148, 1032, 808 cm^{-1}; HRMS (CI$^+$) *m/z* 324.1447 (324.1447 calcd for $C_{16}H_{18}O_6$, $[M+NH_4]^+$).



3-Oxododecanoic acid 4-(3-oxododecanoyloxymethyl)benzyl ester (13). Purification by chromatography (4:1 hexanes:EtOAc) afforded 61% of **13** as a colorless oil that solidifies at 0 °C: ^{1}H NMR (500 MHz, $CDCl_3$) 7.35 (s, 4H), 5.16 (s, 4H), 3.47 (s, 4H), 2.50 (t, J = 7.3 Hz, 4H), 1.58–1.56 (m, 4H), 1.28–1.24 (m, 24H), 0.87 (t, J = 5.8 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) 202.6, 167.0, 135.5, 128.4, 88.6, 66.5, 49.1, 43.1, 31.8, 29.3, 29.3, 29.2, 28.9, 23.3, 22.6, 14.0; IR (thin film) 2927, 2858, 1746, 1406, 1220, 911 cm^{-1}; HRMS (ESI) *m/z* 553.3497 (553.3505 calcd for $C_{32}H_{50}O_6$, $[M+Na]^+$).



3,9-Dioxoundecanedioic acid dimethyl ester (14). Following the general procedure of Weiler,[3] NaH (413 mg, 60% dispersion, 10.4 mmol) was added slowly to a solution of methyl acetoacetate (1.0 g, 8.62 mmol) and THF (15 mL) at 0 °C. The opaque reaction mixture was stirred for 10 min at 0 °C, then *n*-BuLi (3.1 mL, 9.5 mmol) was added dropwise. The resulting yellow reaction mixture was stirred for an additional 10 minutes at 0 °C before 1,3-diiodopropane (500 uL, 4.3 mmol) was added dropwise. The reaction mixture was allowed to warm to room temperature over 30 min and then was quenched by the addition of 0.1 N HCl (~2 mL). Silica gel was added, the reaction mixture was concentrated and the residue purified by chromatography (4:1 hexanes:EtOAc) to afford 1.82 g (77 %) of **14** as a pale yellow liquid: ^{1}H NMR (400 MHz, $CDCl_3$) 3.48 (s, 6H), 3.24 (s, 4H), 2.33 (t, J = 7.1 Hz, 4H), 1.39–1.32 (m, 4H), 1.11–1.04 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) 202.1, 167.1, 51.5, 48.3. 41.9, 27.6, 22.4; IR (thin film) 2954, 2923, 2854, 1744, 1717, 1457, 1320, 1156, 908 cm^{-1}; HRMS (CI$^+$) *m/z* 273.1333 (273.1338 calcd for $C_{13}H_{21}O_6$, MH^+).



3-Oxo-8-(3-oxobutyryloxy)octanoic acid methyl ester (15). Following the general procedure of Weiler,[3] NaH (413 mg, 60% dispersion, 10.43 mmol) was added slowly to a solution of methyl acetoacetate (1.00 g, 8.62 mmol) and THF (15 mL) at 0 °C. The opaque reaction mixture was stirred for 10 min at 0 °C, then *n*-BuLi (3.1 mL, 9.5 mmol) was added dropwise. The resulting yellow reaction mixture was stirred for an additional 10 min at 0 °C before *tert*-butyl(4-iodobutoxy)dimethylsilane (2.2

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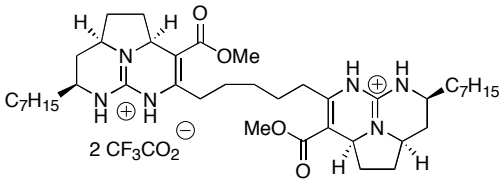
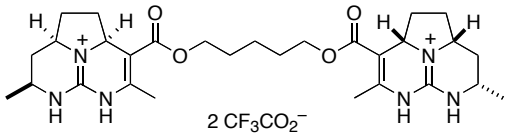
[3] Weiler, L. *J. Am. Chem. Soc.* **1970**, *92*, 6702–6704.

mL, 8.62 mmol) was added dropwise. The reaction was allowed to warm to room temperature over 30 min and then was quenched by the addition of 0.1 N HCl (~2 mL). Silica gel was added, the reaction mixture was concentrated and the residue was purified by chromatography to yield 3-oxo-8-(*tert*-butyldimethylsiloxy)octanoic acid methyl ester as a colorless liquid (2.41 g, 93 %).

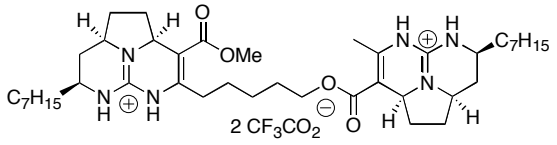
A portion of this liquid (150 mg, 0.49 mmol) was dissolved in MeOH (2 mL) and DOWEX-50 (300 mg) was added. This mixture was stirred at room temperature for 2 d, after which it was filtered and concentrated. The resulting residue was azeotroped with benzene (3x, 25 mL) and the residue was dissolved in Et_2O (20 mL). Diketene was added (60 uL, 7.4 mmol) and the reaction mixture was stirred for 18 h at room temperature. Silica gel was added, the reaction mixture was concentrated and the residue was purified by chromatography (4:1 hexanes:EtOAc) to afford 81 mg (60%) of **15** as a yellow liquid: ^{1}H NMR (400 MHz, $CDCl_3$) □4.13 (app t, *J* = 6.4 Hz, 2H), 3.73 (s, 3H), 2.55 (app t, *J* = 4.0 Hz, 2H), 2.26 (s, 3H), 1.67–1.58 (m, 8H), 1.38–1.34 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) □202.4, 167.6, 167.1, 65.1, 52.3, 50.0, 49.0. 42.7, 30.1, 28.2, 25.2, 22.9; IR (thin film) 2941, 1744. 1713, 1360, 1150, 1017, 999 cm^{-1}; HRMS (CI$^+$) *m/z* 273.1333 (273.1338 calcd for $C_{13}H_{21}O_6$, MH$^+$).



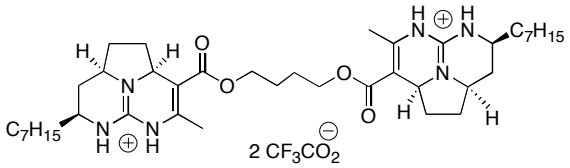
General procedure for double tethered Biginelli cyclizations. Preparation of (2a*S*,7*S*,8a*R*,2a'*S*,7*S*',8a'*R*)-7-heptyl-4-[5-(7'-heptyl-1',2',2a',5',6',7',8',8a'-octahydro-5',6',8b'-triazaacenaphthylene-3'-carboxylic acid methyl ester 4'-yl)pentyl]-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triazaacenaphthylene-3-carboxylic acid methyl ester bistrifluoroacetate (16). Bis-□-ketoester **14** (15 mg, 0.06 mmol) was added to a stirring mixture of guanidine aldehyde **4** (65 mg, 0.21 mmol), morpholinium acetate (80 mg, 0.6 mmol), Na_2SO_4 (80 mg, 0.5 mmol) and MeOH (2 mL, 0.1 M) (In general, morpholine and AcOH can be added as liquids or as the morpholinium acetate salt). The reaction mixture was heated to 60 °C and stirred for 72 h. Concentration and purification of the residue by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10% MeCN: 0.1% TFA in H_2O to 90% MeCN: 0.1% TFA in H_2O over 20 min) gave 18 mg (35%) of **16** and 12 mg (23%) of C_1-symmetric isomer **22**, both as nearly colorless oils. Characterization data for **16**: ^{13}C NMR (125 MHz, CD_3OD) □166.7, 148.1, 147.5, 102.9, 58.3, 57.2, 51.9, 51.5, 34.9, 34.0, 33.9, 32.9, 31.4, 30.4, 30.2, 29.9, 28.8, 27.5, 26.2, 23.7, 14.4; IR (thin film) 2921, 1686, 1457, 1185, 899 cm^{-1}; HRMS (ESI$^+$) *m/z* 707.5226 (707.5224 calcd for $C_{41}H_{68}O_4N_6$, M$^+$); [□]$^{27}_{589}$ -43.2, [□]$^{27}_{577}$ -42.1, [□]$^{27}_{546}$ -46.6, [□]$^{27}_{435}$ -83.0, [□]$^{27}_{405}$ -98.9 (*c* 1.0, MeOH). Characterization data for **22**: ^{13}C NMR (125 MHz, CD_3OD) 166.7, 148.0, 147.5, 58.2, 57.2, 56.7, 56.1, 53.5, 51.9, 51.8, 51.5, 48.5, 36.0, 34.9, 34.0, 33.9, 33.6, 32.9, 32.8, 32.5, 31.5, 31.4, 30.5, 30.4, 30.3, 29.9, 29.0, 28.8, 27.5, 26.3, 26.2, 23.7, 14.4; HRMS (ESI$^+$) *m/z* 707.5224 (707.5224 calcd for $C_{41}H_{68}O_4N_6$).



Preparation of (2a*S*,7*S*,8a*S*,2a'*S*,7*S*',8a'*S*)-7-Methyl-4-methyl-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triazaacenaphthylene-3-carboxylic acid 4-(7'-methyl-4'-methyl-1',2',2a',5',6',7',8',8a'-octahydro-5',6',8b'-triazaacenaphthylene-3'-carboxy)butyl ester bistrifluoroacetate (9). Purification by preparative HPLC (Phenomenex C-18, 210 nm, 30% MeCN: 60% 0.1% TFA in H_2O) gave 24 mg (41%) of the C_2-symmetric product **9** as a nearly colorless oil: ^{13}C NMR (125 MHz, CD_3OD) 166.2, 146.8, 143.9, 102.9, 65.3, 58.2, 57.1, 47.2, 36.1, 34.0, 29.4, 27.6, 23.9, 19.9, 17.8; HRMS (FAB) *m/z* 539.3217 (539.3346 calcd for $C_{29}H_{43}O_4N_6$); $[]^{25}_{D}$ -93.6, $[]^{25}_{546}$ -112, $[]^{25}_{435}$ -207, $[]^{25}_{405}$ -246 (*c* 0.7, MeOH).

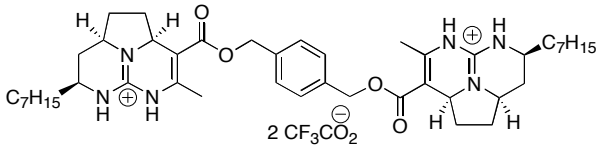


(2a*S*,7*S*,8a*R*,2a'*S*,7*S*',8a'*R*)-4-(5-(7'-Heptyl-4-methyl-1',2',2a',5',6',7',8',8a'-octahydro-5',6',8b'-triaza-acenaphthylene-3'-carboxy)pentyl)-7-heptyl-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triaza-acenaphthylene-3-carboxylic acid methyl ester bistrifluoroacetate (17). Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10 % MeCN: 0.1% TFA in H_2O to 90% MeCN: 0.1% TFA in H_2O over 20 min) gave 26 mg (39%) of **17** and 24 mg (37%) of a stereoisomer that was contaminated with **17**, both as nearly colorless oils. Characterization data for **17**: ^{13}C NMR (125 MHz, CD_3OD) 166.7, 166.6, 148.1, 147.4, 147.3, 144.7, 103.5, 102.6, 65.4 (2C), 58.3, 58.25, 57.21, 57.2, 51.9, 51.5, 34.9, 33.9, 33.9, 32.9, 31.4, 30.5, 30.3, 29.3, 28.7, 27.5, 27.4, 26.8, 26.3, 26.2, 23.7, 17.8, 14.4; HRMS (ESI^+) *m/z* 707.5224 (707.5202 calcd for $C_{41}H_{68}O_4N_6$); $[]^{27}_{589}$ -14.7, $[]^{27}_{577}$ -15.9, $[]^{27}_{546}$ -17.9, $[]^{27}_{435}$ -44.1, $[]^{27}_{405}$ -57.2 (*c* 1.2, MeOH).

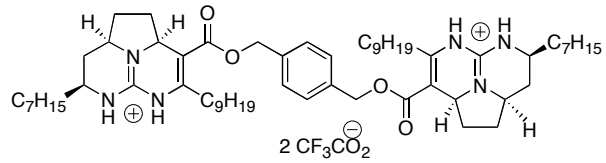


(2a*S*,7*S*,8a*R*,2a'*S*,7*S*',8a'*R*)-7-Heptyl-4-methyl-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triaza-acenaphthylene-3-carboxylic acid 4-(7'-heptyl-4'-methyl-1',2',2a',5',6',7',8',8a'-octahydro-5',6',8b'-triazaacenaphthylene-3'-carboxy)-butyl ester bistrifluoroacetate (18) Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10% MeCN: 0.1% TFA in H_2O to 90%

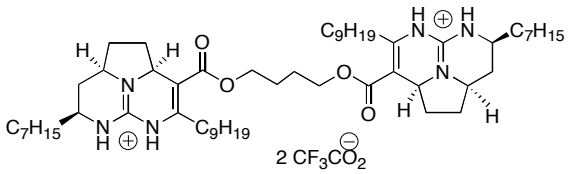
MeCN: 0.1% TFA in H_2O over 20 min) gave 20 mg (42%) of the C_2-symmetric product **18** and 10 mg (22%) of a C_1-symmetric product that was contaminated with **18**, both as nearly colorless oils. Characterization data for **18**: ^{13}C NMR (125 MHz, CD_3OD) 166.5, 147.4, 144.2, 103.2, 65.2, 58.4, 57.2, 51.2, 34.9, 33.9, 32.9, 30.4, 30.3, 27.5, 26.6, 26.2, 23.7, 17.8, 14.4; IR (thin film) 2929, 1686, 1200, 1077, 721 cm^{-1}; HRMS (ESI$^+$) *m/z* 693.5056 (693.5067 calcd for $C_{40}H_{66}O_4N_6$); $[]^{27}_{589}$ -55.5, $[]^{27}_{577}$ -58.0, $[]^{27}_{546}$ -61.9, $[]^{27}_{435}$ -86.3, $[]^{27}_{405}$ -95.8 (*c* 0.5, MeOH).



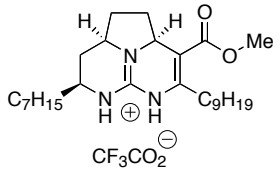
(2a*S*,7*S*,8a*R*,2a'*S*,7*S*',8a'*R*)-7-Heptyl-4-methyl-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triaza-acenaphthylene-3-carboxylic acid 4-(7'-heptyl-4'-methyl-1',2',2a',5',6',7',8',8a'-octahydro-5',6',8b'-triazaacenaphthylene-3'-carboxy)methylbenzyl ester bistrifluoroacetate (19). Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10% MeCN: 0.1% TFA in H_2O to 90% MeCN: 0.1% TFA in H_2O over 20 min) gave 13 mg (25%) of the C_2-symmetric product **19** and 7 mg (13%) of a C_1-symmetric product that was contaminated with some **19**, both as nearly colorless oils. Characterization data for **19**: ^{13}C NMR (125 MHz, CD_3OD) 163.2, 144.7,141.2, 135.9, 129.8, 103.0, 67.0, 58.3, 57.2, 51.5, 35.0, 33.9, 33.0, 33.4, 30.3, 27.5, 26.2, 23.8, 17.9, 14.5; IR (thin film) 2927, 2858, 1684, 1197, 1135, 718 cm^{-1}; HRMS (ESI $^+$) *m/z* 741.5063 (741.5067 calcd for $C_{44}H_{66}O_4N_6$).



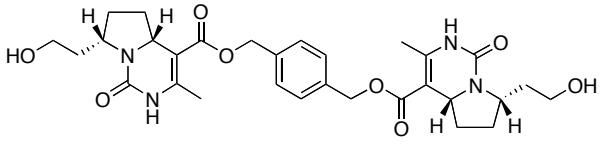
(2a*S*,7*S*,8a*R*,2a'*S*,7*S*',8a'*R*)-7-Heptyl-4-nonyl-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triaza-acenaphthylene-3-carboxylic acid 4-(7'-heptyl-4'-nonyl-1',2',2a',5',6',7',8',8a'-octahydro-5',6',8b'-triazaacenaphthylene-3'-carboxy)methylbenzyl ester bistrifluoroacetate (20). This reaction was carried out by the general procedure using trifluoroethanol as the solvent. Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10% MeCN: 0.1% TFA in H_2O to 90% MeCN: 0.1% TFA in H_2O over 20 min) gave 17 mg (34%) of the C_2-symmetric product **20** and 12 mg (25%) of a C_1-symmetric product that was contaminated with some **20**, both as nearly colorless oils. Characterization data for **20**: ^{13}C NMR (125 MHz, CD_3OD) 166.0, 148.2, 147.3, 137.6, 129.9, 103.0, 67.1, 58.3, 57.2, 51.5, 35.0, 34.0, 33.9, 33.0, 32.9, 32.0, 30.6, 30.4 (4C), 30.3, 29.3, 27.5, 26.3, 23.7, 23.7, 14.5, 14.4; IR (thin film) 2932, 2856, 1583, 1479, 1438, 1250 cm^{-1}; HRMS (ESI) *m/z* 483.3817 (483.3819 calcd for $C_{60}H_{98}O_4N_6$); $[]^{27}_{589}$ -38.8, $[]^{27}_{577}$ -37.6, $[]^{27}_{546}$ -43.6, $[]^{27}_{435}$ -96.1, $[]^{27}_{405}$ -120 (*c* 0.5, MeOH).



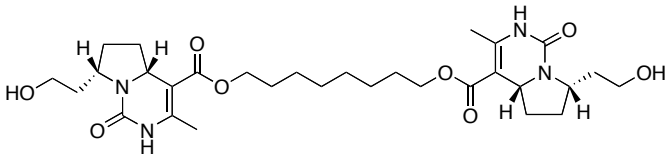
(2a*S*,7*S*,8a*R*,2a'*S*,7*S*',8a'*R*)-7-Heptyl-4-nonyl-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triaza-acenaphthylene-3-carboxylic acid 4-(7'-heptyl-4'-nonyl-1',2',2a',5',6',7',8',8a'-octahydro-5',6',8b'-triazaacenaphthylene-3'-carboxy)butyl ester bistrifluoroacetate (21). This reaction was carried out by the general procedure using trifluoroethanol as the solvent. Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10% MeCN: 0.1% TFA in H_2O to 90% MeCN: 0.1% TFA in H_2O over 20 min) gave 16 mg (33%) of the C_2-symmetric product **21** and 5 mg (11%) of a C_1-symmetric product that was contaminated with some **21**, both as nearly colorless oils. Characterization data for **21**: ^{13}C NMR (125 MHz, CD_3OD) □165.8, 147.6, 146.6, 102.3, 67.2, 57.2, 56.3, 51.5, 43.4, 35.0, 34.0, 33.0, 32.9, 31.8, 30.6, 30.5, 30.4, 30.44, 30.3, 29.2, 27.5, 26.6, 26.3, 23.8, 23.7, 14.4; IR (thin film) 3352, 2927, 2495, 1671, 1686, 1459, 1202 cm^{-1}; HRMS (ESI$^+$) *m/z* 459.3835 (459.3819 calcd for $C_{56}H_{98}O_4N_6$ $[M]^{2+}$); $[□]^{27}_{589}$ -41.3, $[□]^{27}_{577}$ -50.7, $[□]^{27}_{546}$ -72.4, $[□]^{27}_{435}$ -123.1, $[□]^{27}_{405}$ -159.2 (*c* 0.5, MeOH).



(2a*S*,7*S*,8a*R*)-7-Heptyl-4-nonyl-1,2,2a,5,6,7,8,8a-octahydro-5,6,8b-triazaacenaphthylene-3-carboxylic acid methyl ester trifluoroacetate (23 R = *n*-C_7H_{15}**).** Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10% MeCN: 0.1% TFA in H_2O to 90% MeCN: 0.1% TFA in H_2O over 20 min) gave 45 mg (77%) of **23** as a nearly colorless oil: ^{1}H NMR (500 MHz, CD_3OD) □4.59–4.55 (m, 1H), 3.86–3.82 (m, 1H), 3.77 (s, 3H), 3.58–3.49 (m, 1H), 2.84–2.76 (m, 1H), 2.75–2.66 (m, 1H), 2.61–2.53 (m, 1H), 2.49–2.44 (m, 1H), 2.22–2.17 (m, 1H), 1.74–1.55 (m, 6H), 1.53–1.38 (m, 25H), 0.96–0.92 (m, 6H); ^{13}C NMR (125 MHz, CD_3OD) □166.7, 148.2, 147.5, 102.8, 58.3, 57.2, 51.9, 51.5, 34.9, 34.0, 33.9, 33.0, 32.9, 31.6, 30.6, 30.4, 30.4, 30.3 (2C), 30.3, 29.2, 27.5, 26.2, 23.7, 23.7, 14.4, 14.3; IR (thin film) 2927, 2858, 1686, 1436, 1202, 1140, 906, 727 cm^{-1}; HRMS (ESI) *m/z* 446.3764 (446.3747 calcd for $C_{27}H_{48}O_2N_3$); $[□]^{27}_{589}$ -6.75, $[□]^{27}_{577}$ -6.92, $[□]^{27}_{546}$ -7.91, $[□]^{27}_{435}$ -17.0, $[□]^{27}_{405}$ -20.1 (*c* 3.8, MeOH).



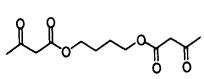
(2*S*,4a*R*,2'*S*,4a'*R*)-7-(2-Hydroxy-ethyl)-3-methyl-1-oxo-1,2,4a,5,6,7-hexahydro-pyrrolo[1,2-*c*]pyrimidine-4-carboxylic acid 4-(7'-(2'-hydroxyethyl)-3'-methyl-1'-oxo-1',2',4a',5',6',7'-hexahydropyrrolo[1',2'-*c*]pyrimidine-4'-carboxy)-methylbenzyl ester (25). Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10% MeCN: 0.1% TFA in H_2O to 90% MeCN: 0.1% TFA in H_2O over 20 min) gave 21 mg (69%) of the C_2-symmetric product **25** and 7 mg (23%) of a less pure sample of a C_1-symmetric isomer, both as pale yellow films. Characterization data for **25**: ^{1}H NMR (500 MHz, $CDCl_3$) (trace amounts of MeOH added to solubilize **25**) δ 7.61 (br s, 1H), 7.41 (s, 2H), 5.25 (d, J = 2.5 Hz, 1H), 5.18 (d, J = 2.5 Hz, 1H), 4.33–4.30 (m, 1H), 4.21–4.17 (m, 1H), 3.65–3.55 (m, 1H), 2.25 (s, 3H), 2.13–2.08 (m, 1H), 1.93–1.65 (m, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.5, 154.4, 148.8, 136.2, 128.7, 102.5, 65.7, 59.2, 58.5, 52.4, 50.9, 39.5, 30.8, 29.9, 18.4; IR (thin film) 2950, 1676, 1630, 1437, 1313, 1251, 1112, 1074 cm^{-1}; HRMS (ESI) m/z 605.2572 (605.2587 calcd for $C_{30}H_{38}O_8N_4$); $[\alpha]^{27}_{589}$ -39.7, $[\alpha]^{27}_{577}$ -40.5, $[\alpha]^{27}_{546}$ -46.0, $[\alpha]^{27}_{435}$ -98.9, $[\alpha]^{27}_{405}$ -110.1 (c 1, MeOH).



(2*S*,4a*R*,2'*S*,4a'*R*)-7-(2-Hydroxyethyl)-3-methyl-1-oxo-1,2,4a,5,6,7-hexahydro-pyrrolo[1,2-*c*]pyrimidine-4-carboxylic acid 4-(7'-(2'-hydroxyethyl)-3'-methyl-1'-oxo-1',2',4a',5',6',7'-hexahydropyrrolo[1',2'-*c*]pyrimidine-4'-carboxy)octyl ester (27). Purification by preparative HPLC (Phenomenex C-18, 16 mL/min, 210 nm, 10 % MeCN: 0.1% TFA in H_2O to 90 % MeCN: 0.1% TFA in H_2O over 20 min) gave 20 mg (61%) of the C_2-symmetric product **27** and 7 mg (20%) of a less pure sample of a C_1-symmetric isomer, both as pale yellow films the former of which slowly solidified upon standing at 0 °C. Characterization data for **27**: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (m, 1H), 4.31 (ddd, J = 11.2, 5.0, 1.2 Hz, 1H), 4.23–4.16 (m, 2H), 4.16-4.11 (m, 1H), 3.70–3.58 (m, 2H), 2.58–2.53 (m, 1H), 2.28 (s, 3H), 2.19–2.10 (m, 1H), 1.95–1.87 (m, 1H), 1.82–1.76 (m, 3H), 1.76–1.68 (m, 4H), 1.39–1.37 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.9, 154.5, 148.1, 103.0, 64.2, 59.2, 58.2, 52.4, 39.6, 30.9, 29.9, 29.1, 28.7, 26.0, 18.2; IR (thin film) 3236, 2927, 2858, 1738, 1645, 1452, 1321, 1251, 1081 cm^{-1}; HRMS (ESI$^+$) m/z 613.3217 (613.3214 calcd for $C_{30}H_{46}O_8N_4$); $[\alpha]^{27}_{589}$ -57.3, $[\alpha]^{27}_{577}$ -57.6, $[\alpha]^{27}_{546}$ -64.0, $[\alpha]^{27}_{435}$ -145.4, $[\alpha]^{27}_{405}$ -188.7 (c 0.5, MeOH).

^{1}H and ^{13}C NMR Spectra

□. □-Ketoesters

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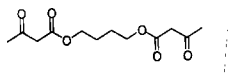


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1D NMR plot parameters
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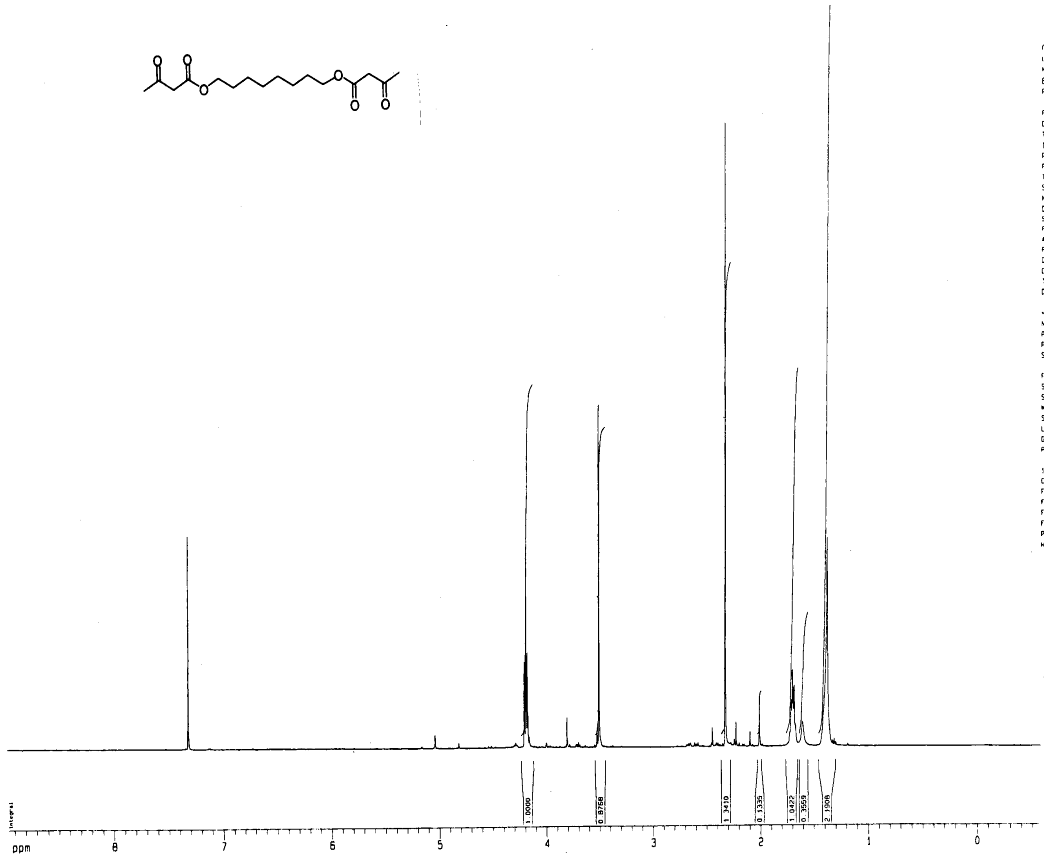

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SOLVENT CDCl3
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DS 2
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FIDRES 0 122266 Hz
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RG 406 4
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DE 6 00 usec
TE 300 0 K
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F2 - Processing parameters
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1D NMR plot parameters
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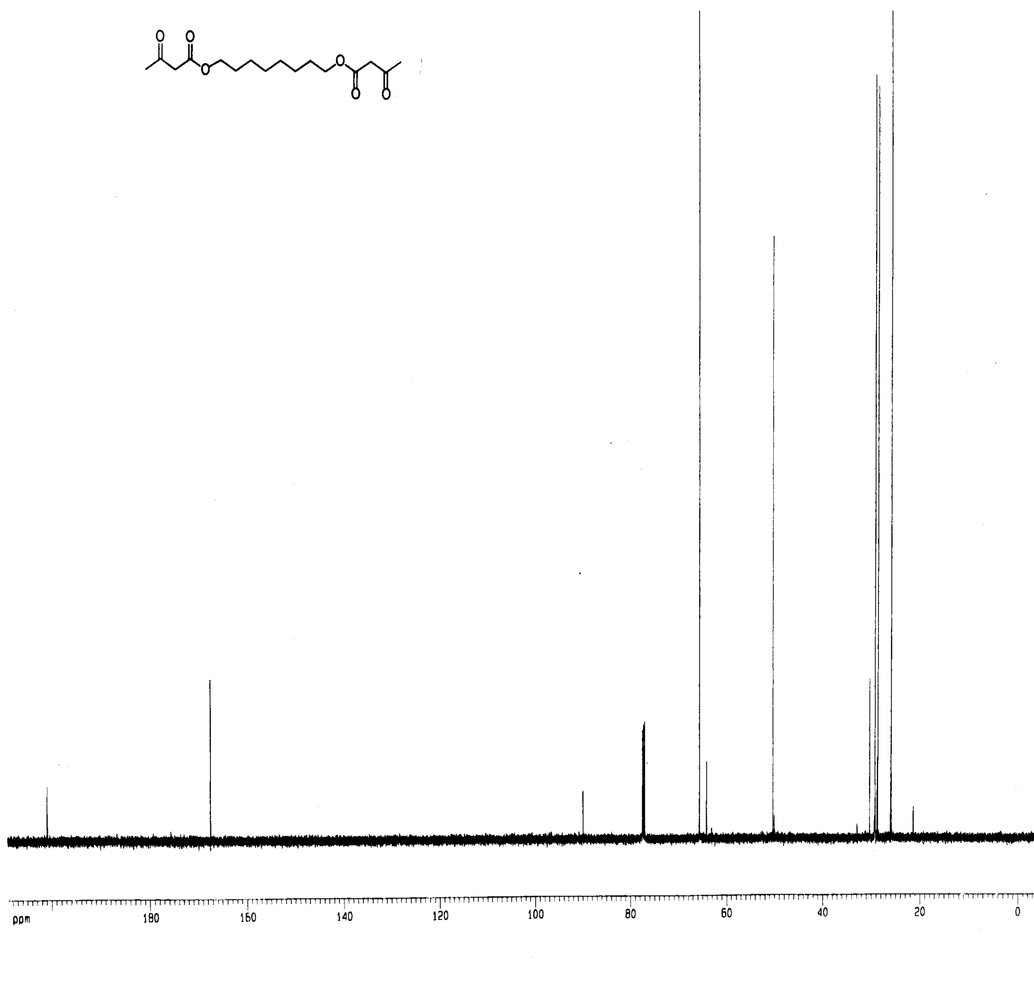

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RG 4096
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TE 300 0 K
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PL1 0 00 dB
SFO1 125 7213038 MHz

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PCPD2 80 00 usec
PL2 -3 00 dB
PL12 15 10 dB
SFO2 499 9334995 MHz

F2 - Processing parameters
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SF 125 7074760 MHz
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SSB 0
LB 0 00 Hz
GB 0
PC 2 00

1D NMR plot parameters
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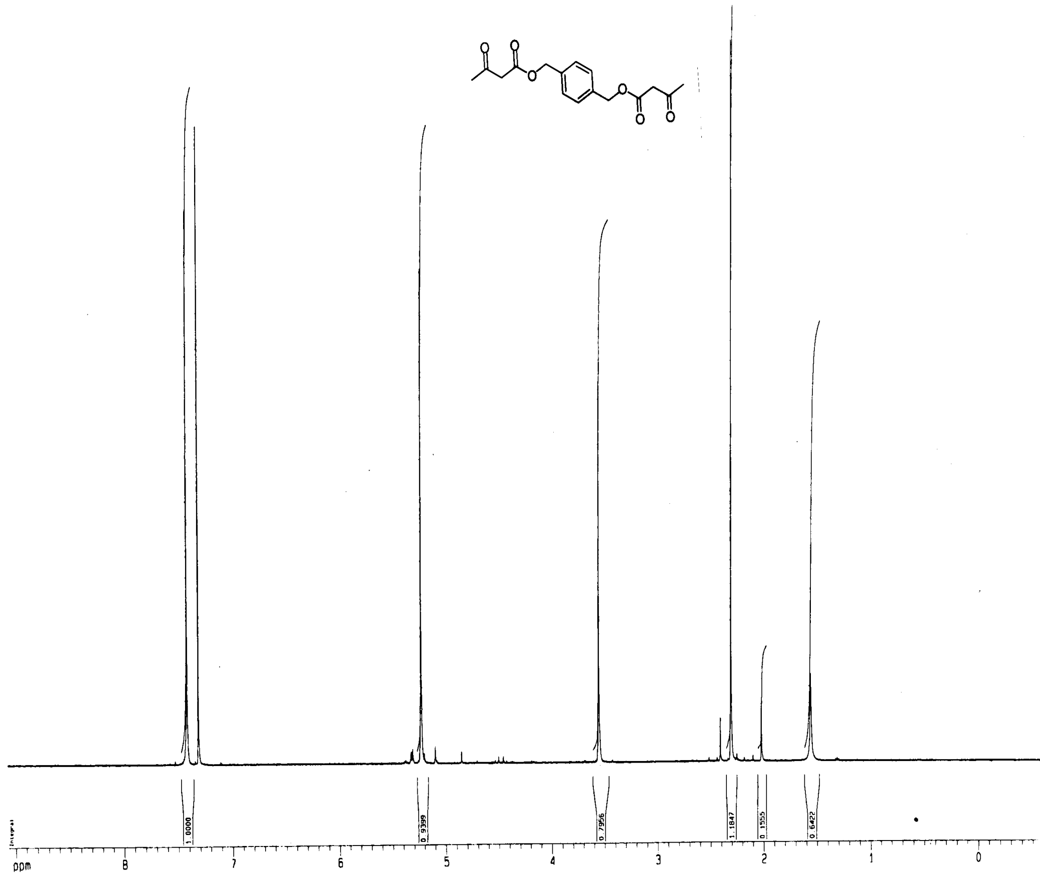

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============ CHANNEL f1 ========
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SFO1 499 9334995 MHz

F2 - Processing parameters
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WDW no
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PC 4 00

1D NMR plot parameters
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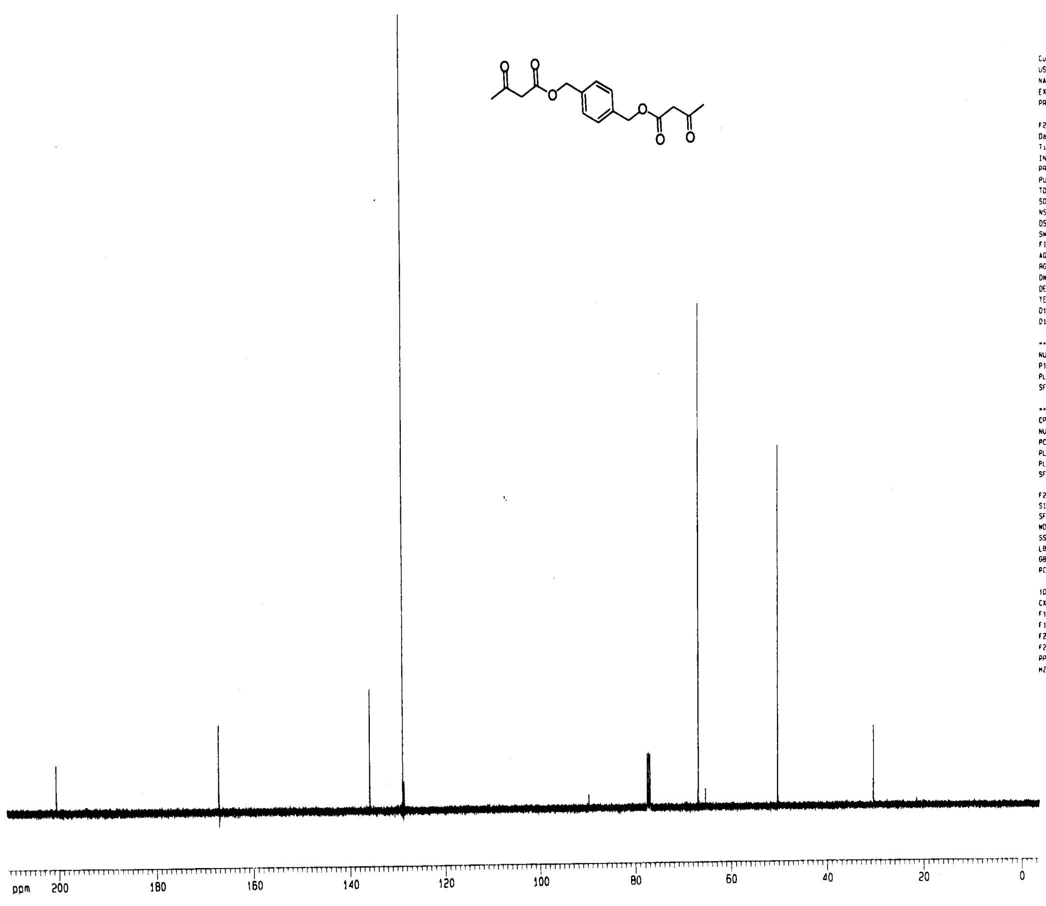

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PROCNO 1

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RG 2896 3
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DE 4 50 usec
TE 300 0 K
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D11 0 03000000 sec

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PL1 0 00 dB
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============ CHANNEL f2 ========
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PCPD2 80 00 usec
PL2 -3 00 dB
PL12 15 10 dB
SFO2 499 9334995 MHz

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1D NMR plot parameters
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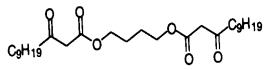


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1D NMR plot parameters
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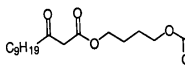


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PROCNO               1

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PULPROG        zgdc30
TD               65536
SOLVENT          CDCl3
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DS                   4
SWH          30303.031 Hz
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AQ            1.0813940 sec
RG                4096
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D11           0.03000000 sec

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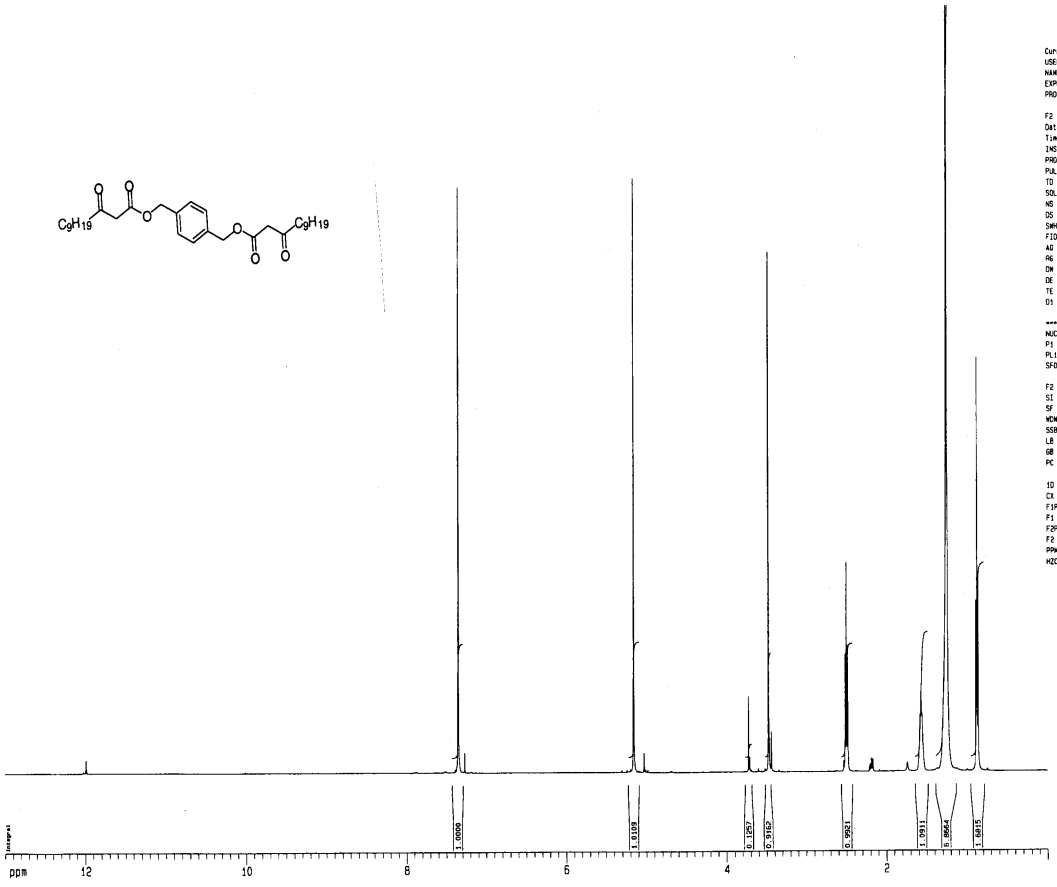


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DS                      2
SWH              8012.820 Hz
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SFO1           499.8334988 MHz

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HZCM           284.99081 Hz/cm
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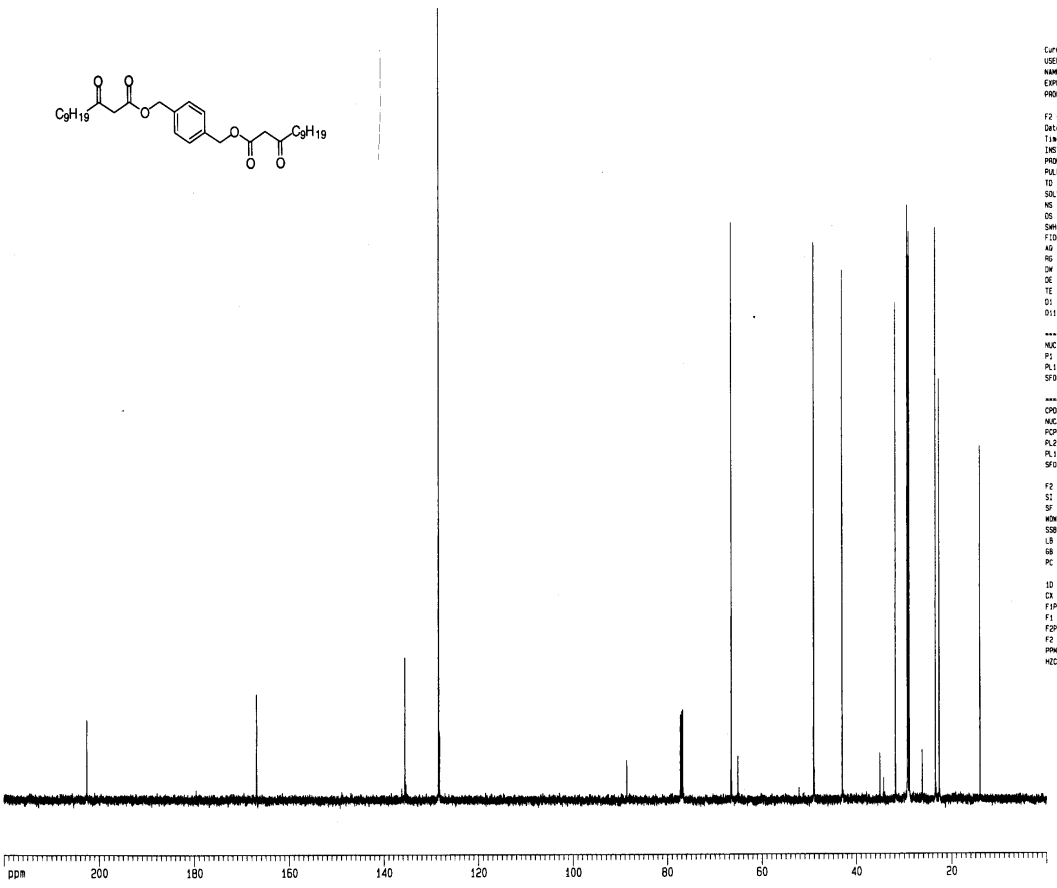
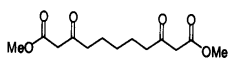
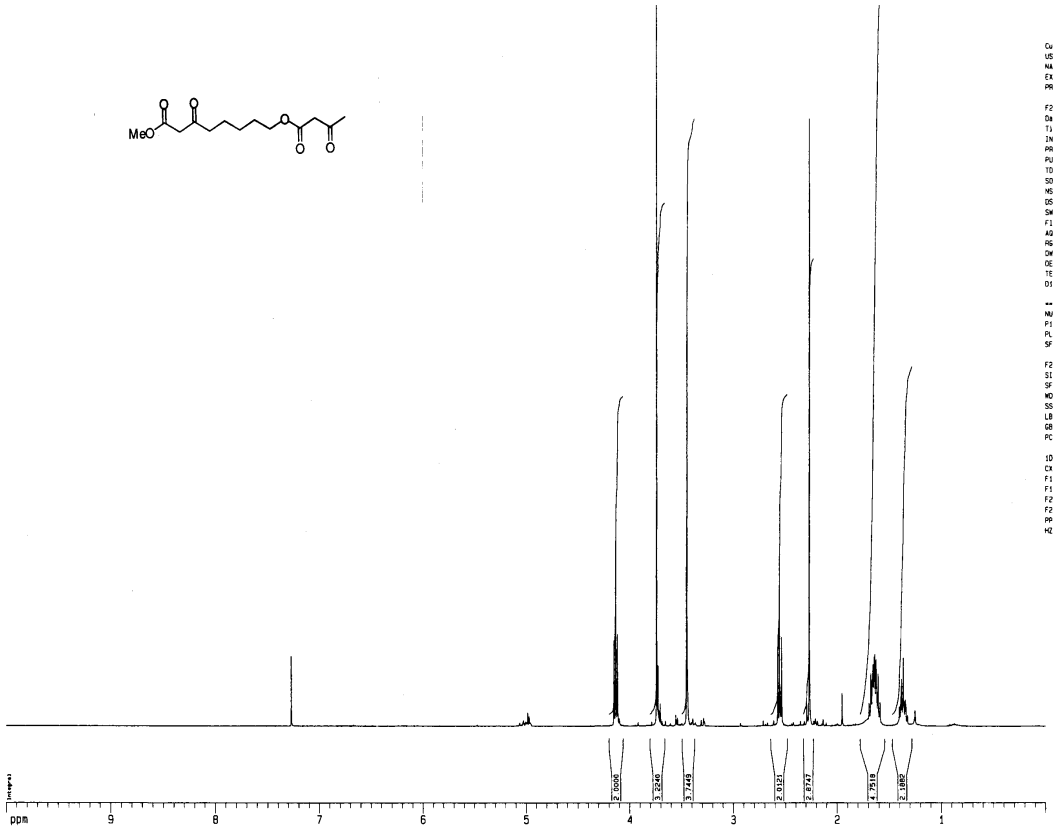



```
Current Data Parameters
USER                shawnc
NAME             sc-III-93a
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_            20030523
Time                 9.43
INSTRUM             gn500
PROBHD    5 mm broadban
PULPROG          zgdc30
TD                  65536
SOLVENT             CDCl3
NS                    101
DS                      4
SWH             30303.031 Hz
FIDRES           0.462388 Hz
AQ               1.0813940 sec
RG                 3649.1
DW                16.500 usec
DE                 4.50 usec
TE                 300.0 K
D1              0.25000000 sec
D11             0.03000000 sec

========== CHANNEL f1 =======
NUC1                   13C
P1                  15.00 usec
PL1                  0.00 dB
SFO1           125.6961791 MHz

========== CHANNEL f2 =======
CPDPRG2             waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -3.00 dB
PL12                15.10 dB
SFO2           499.8334988 MHz

F2 - Processing parameters
SI                  65536
SF             125.6823668 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
F1P                220.000 ppm
F1               27650.12 Hz
F2P                 0.000 ppm
F2                  0.00 Hz
PPMCM            9.64912 ppm/cm
HZCM           1212.72461 Hz/cm
```




```
Current Data Parameters
USER            shawnc
NAME         sc-III-115a
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_         20030609
Time              14.42
INSTRUM         drx400
PROBHD   5 mm QNP H/F
PULPROG           zg30
TD               65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            6410.256 Hz
FIDRES         0.097813 Hz
AQ             5.1118579 sec
RG                    8
DW               78.000 usec
DE                4.50 usec
TE              300.0 K
D1             0.10000000 sec

========== CHANNEL f1 =======
NUC1                 1H
P1                12.00 usec
PL1               0.00 dB
SFO1        400.1328009 MHz

F2 - Processing parameters
SI                65536
SF          400.1300175 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 2.00

1D NMR plot parameters
CX               22.80 cm
F1P             10.000 ppm
F1              4001.30 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          0.43860 ppm/cm
HZCM         175.49562 Hz/cm
```


```
Current Data Parameters
USER            shawnc
NAME         sc-III-115a
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_         20030609
Time              14.44
INSTRUM         drx400
PROBHD   5 mm QNP H/F
PULPROG         zgpg30
TD               65536
SOLVENT           CDCl3
NS                   77
DS                    4
SWH           24154.590 Hz
FIDRES         0.368570 Hz
AQ             1.3566452 sec
RG                1280.2
DW               20.700 usec
DE                4.50 usec
TE              300.0 K
D1             0.10000000 sec
D11            0.03000000 sec

========== CHANNEL f1 =======
NUC1                 13C
P1                10.12 usec
PL1               0.00 dB
SFO1        100.6237964 MHz

========== CHANNEL f2 =======
CPDPRG2          waltz16
NUC2                 1H
PCPD2             90.00 usec
PL2              17.00 dB
PL12             17.92 dB
SFO2        400.1328009 MHz

F2 - Processing parameters
SI                65536
SF          100.6128110 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX               22.80 cm
F1P             220.000 ppm
F1              22134.62 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM          9.64912 ppm/cm
HZCM         970.82538 Hz/cm
```

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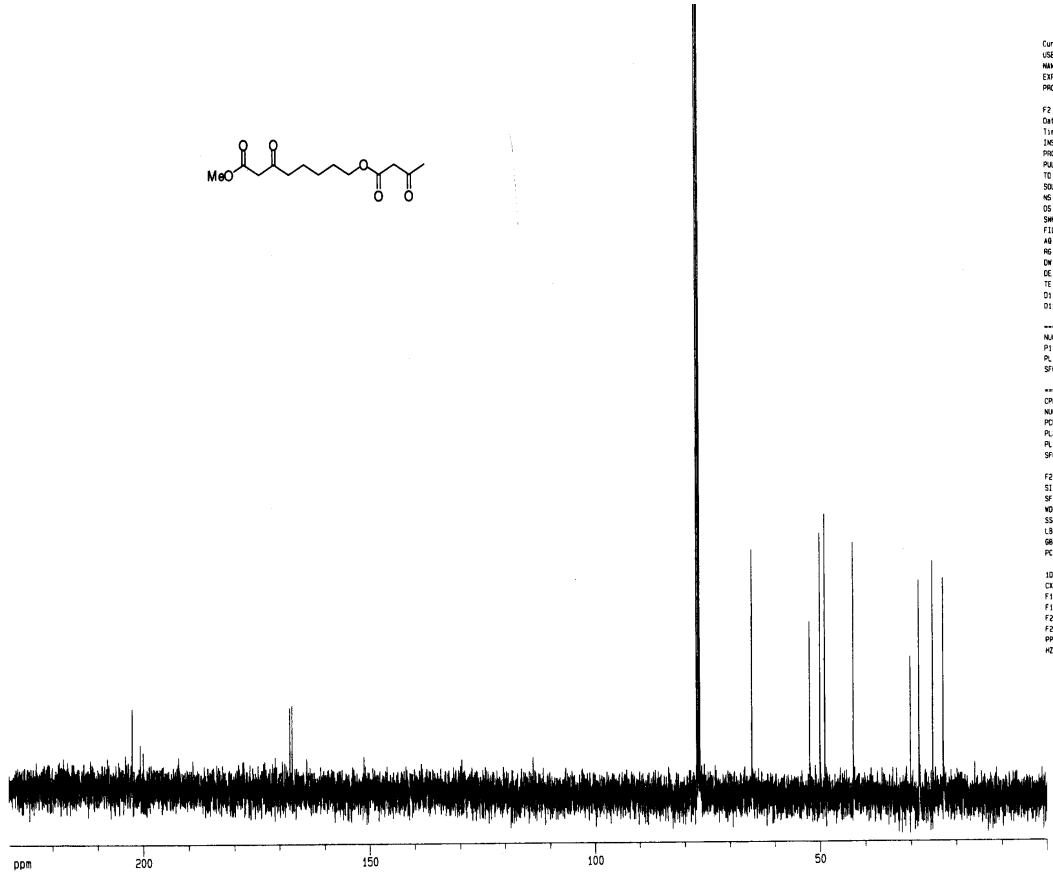
Current Data Parameters
USER                shawnc
NAME             sc-III-123a
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_            20030621
Time                12.00
INSTRUM             drx400
PROBHD     5 mm QNP H/F
PULPROG             zg30
TD                 65536
SOLVENT             CDCl3
NS                      8
DS                      2
SWH              6410.256 Hz
FIDRES           0.097813 Hz
AQ               5.1118579 sec
RG                  161.3
DW                 78.000 usec
DE                  4.50 usec
TE                 300.0 K
D1               0.10000000 sec

========== CHANNEL f1 =======
NUC1                   1H
P1                  12.00 usec
PL1                 0.00 dB
SFO1          400.1320009 MHz

F2 - Processing parameters
SI                 65536
SF            400.1300175 MHz
WDW                    no
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   2.00

1D NMR plot parameters
CX                  22.80 cm
F1P                10.000 ppm
F1               4001.30 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM            0.43860 ppm/cm
HZCM           175.49562 Hz/cm
```

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Current Data Parameters
USER                shawnc
NAME             sc-III-123a
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_            20030621
Time                12.04
INSTRUM             drx400
PROBHD     5 mm QNP H/F
PULPROG          zgdc30
TD                 65536
SOLVENT             CDCl3
NS                    282
DS                      4
SWH             24154.590 Hz
FIDRES           0.368570 Hz
AQ               1.3566452 sec
RG                 1624.6
DW                 20.700 usec
DE                  4.50 usec
TE                 300.0 K
D1               0.10000000 sec
D11              0.03000000 sec

========== CHANNEL f1 =======
NUC1                   13C
P1                  10.12 usec
PL1                 0.00 dB
SFO1          100.6239964 MHz

========== CHANNEL f2 =======
CPDPRG2          waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 17.00 dB
PL12                17.90 dB
SFO2          400.1320009 MHz

F2 - Processing parameters
SI                 65536
SF            100.6127756 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                  22.80 cm
F1P               230.000 ppm
F1              23140.94 Hz
F2P                 0.000 ppm
F2                   0.00 Hz
PPMCM            10.06772 ppm/cm
HZCM           1014.95349 Hz/cm
```
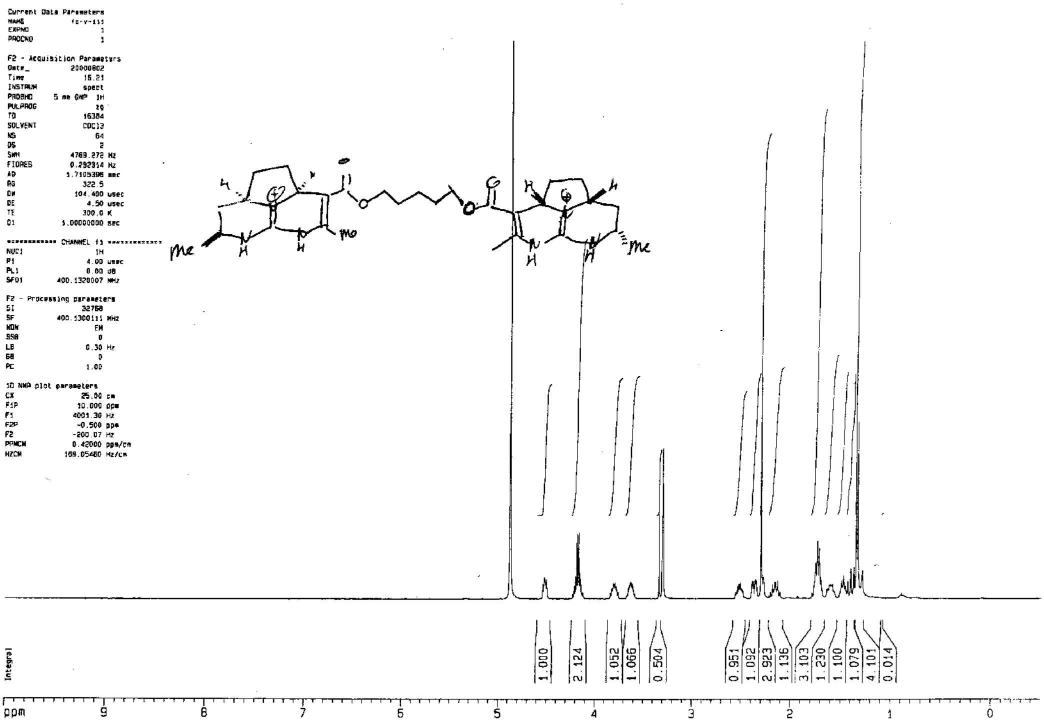
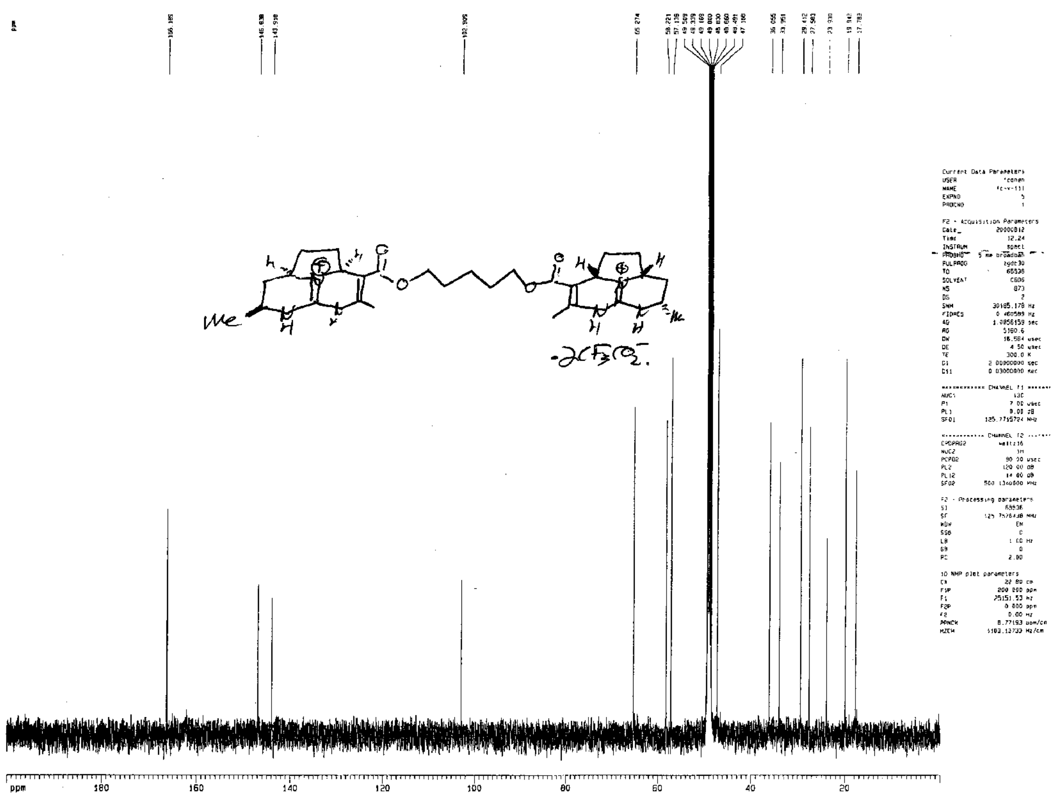
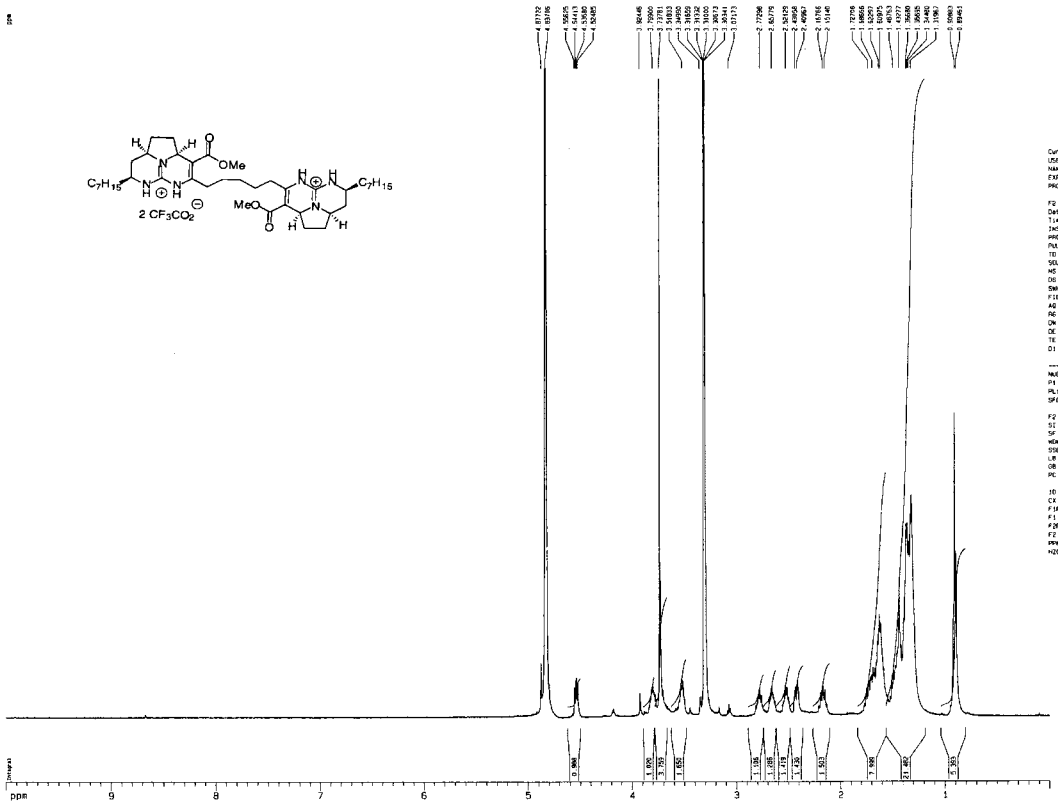

^{1}H and ^{13}C NMR Spectra

. C_2-Symmetric Bisguanidine and Bisureas


Current Data Parameters
NAME fc-v-111
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20000802
Time 15.21
INSTRUM spect
PROBHD 5 mm DNP 1H
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 64
DS 2
SWH 4789.272 Hz
FIDRES 0.292314 Hz
AQ 1.7105396 sec
RG 322.5
DW 104.400 usec
DE 4.50 usec
TE 300.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 4.00 usec
PL1 0.00 dB
SFO1 400.1320007 MHz
F2 - Processing parameters
SI 32768
SF 400.1300111 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 25.00 cm
F1P 10.000 ppm
F1 4001.30 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.42000 ppm/cm
HZCM 168.05460 Hz/cm
Me
Me
Integral
1.000
2.124
1.052
1.066
0.504
0.951
1.092
2.923
1.136
3.103
1.230
1.100
1.079
4.101
0.014
ppm
9
8
7
6
5
4
3
2
1
0


ppm
166.185
145.638
143.918
102.995
65.274
58.221
57.138
49.509
49.339
49.169
49.000
48.830
48.660
48.491
47.160
36.055
33.951
29.412
27.583
23.930
18.912
17.783
Me
Me
-2CF3CO2-
Current Data Parameters
USER cohen
NAME fc-v-111
EXPNO 5
PROCNO 1
F2 - Acquisition Parameters
Date_ 20000812
Time 12.24
INSTRUM spect
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT C6D6
NS 873
DS 2
SWH 30185.170 Hz
FIDRES 0.460589 Hz
AQ 1.0856159 sec
RG 5160.6
DW 16.564 usec
DE 4.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 7.00 usec
PL1 0.00 dB
SFO1 125.7715724 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 120.00 dB
PL12 14.60 dB
SFO2 500.1340800 MHz
F2 - Processing parameters
SI 65536
SF 125.7576438 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.80
1D NMR plot parameters
CX 22.80 cm
F1P 200.000 ppm
F1 25151.53 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.77193 ppm/cm
HZCM 1103.13733 Hz/cm
ppm
180
160
140
120
100
80
60
40
20

1H spectrum



Current Data Parameters
USER shaenc
NAME sc-III-122a
EXPNO 1
PROCNO 1

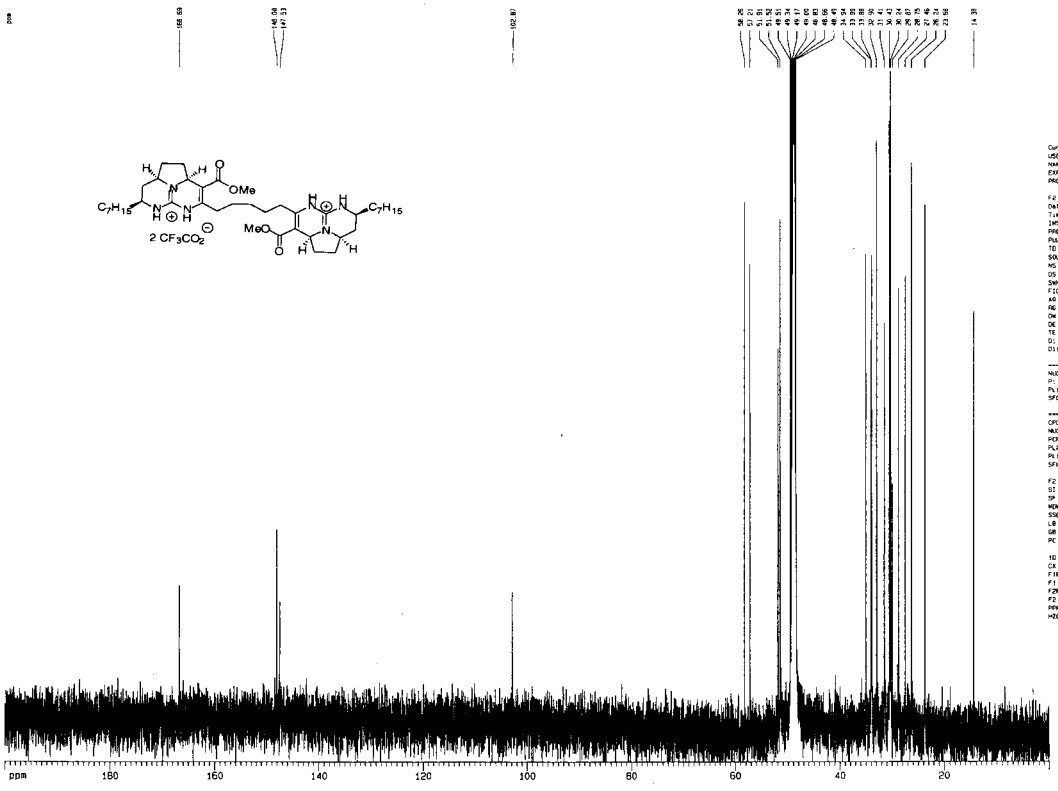
F2 - Acquisition Parameters
Date_ 20030621
Time 11 12
INSTRUM qnp500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CD3OD
NS 64
DS 2
SWH 8012.820 Hz
FIDRES 0.122256 Hz
AQ 4.0894566 sec
RG 287.4
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

---------- CHANNEL f1 -------
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8334988 MHz

F2 - Processing parameters
SI 65536
SF 499.8300145 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 4998.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43850 ppm/cm
HZCM 219.22369 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER shaenc
NAME sc-III-122a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030621
Time 11 16
INSTRUM qnp500
PROBHD 5 mm broadban
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4547
DS 4
SWH 30303.031 Hz
FIDRES 0.462398 Hz
AQ 1.0813940 sec
RG 2568.3
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec

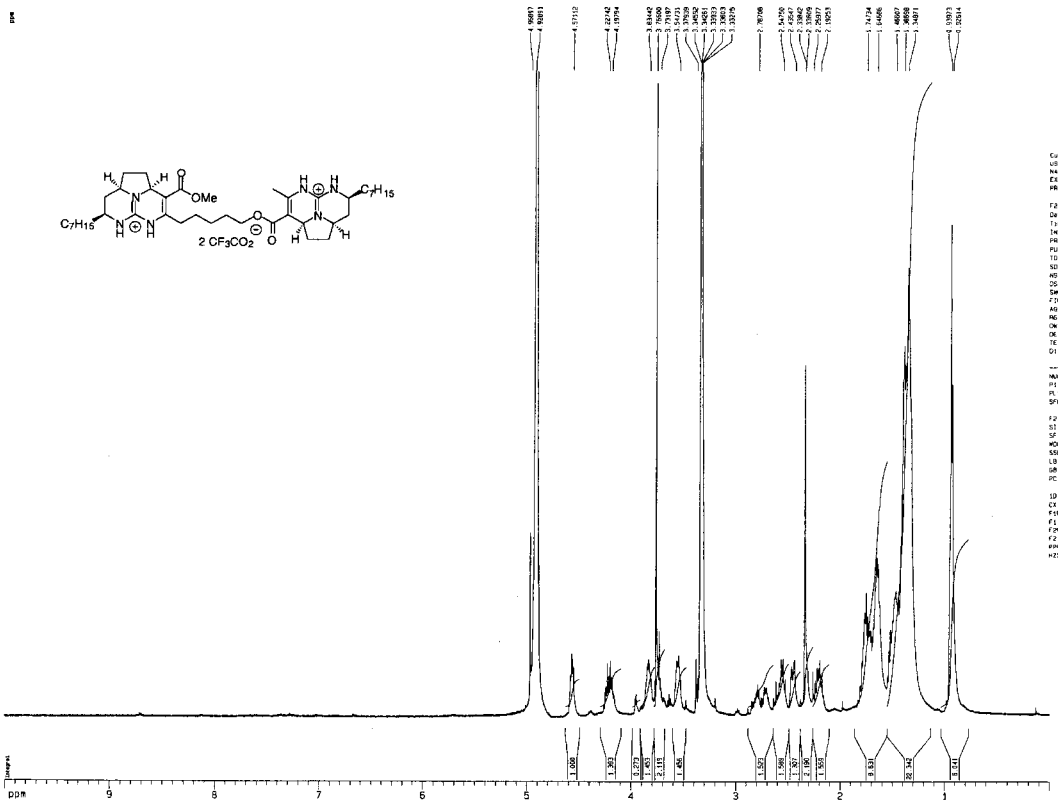
---------- CHANNEL f1 -------
NUC1 13C
P1 15.00 usec
PL1 0.00 dB
SFO1 125.6961791 MHz

---------- CHANNEL f2 -------
CPDPRG2 waltz16
NUC2 1H
PCPD2 86.00 usec
PL2 -3.00 dB
PL12 15.10 dB
SFO2 499.8334988 MHz

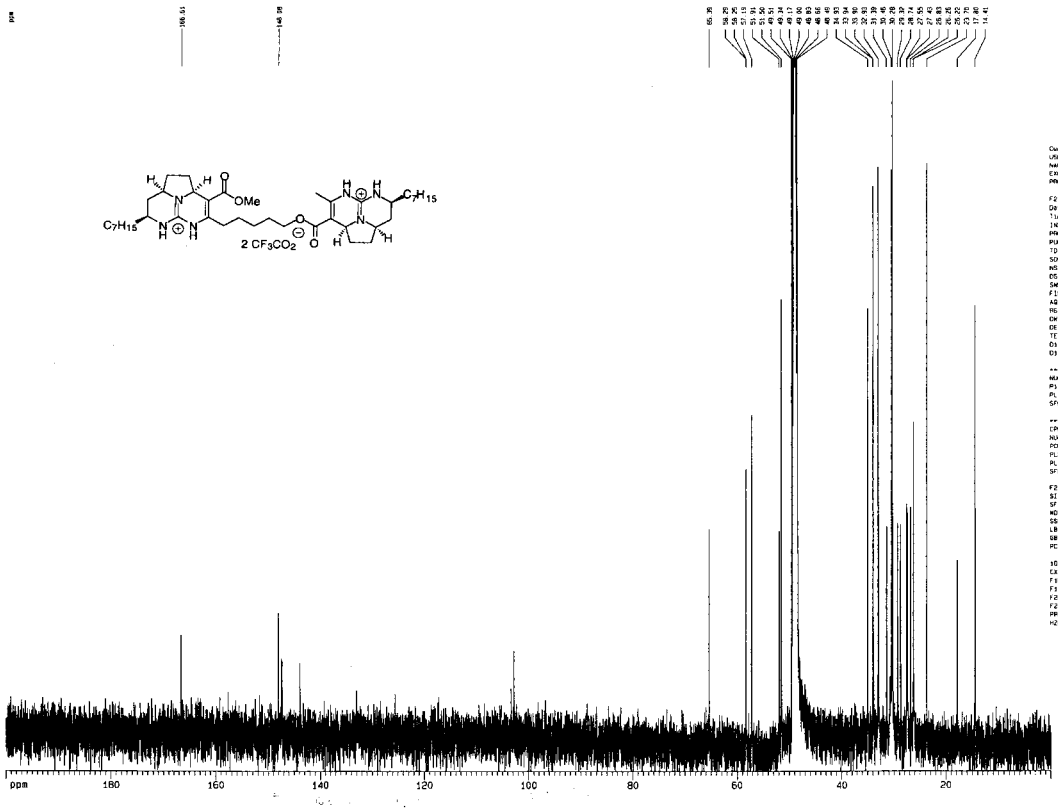
F2 - Processing parameters
SI 65536
SF 125.6821781 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
F1P 200.000 ppm
F1 25136.44 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.77193 ppm/cm
HZCM 1102.47522 Hz/cm

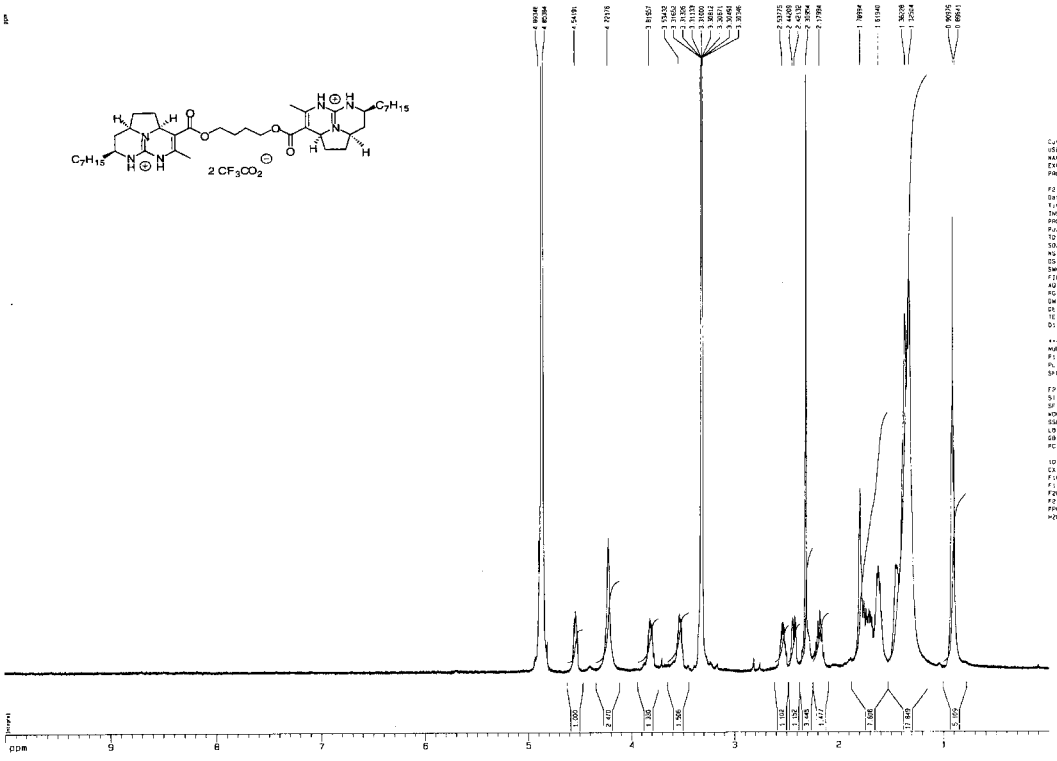
1H spectrum



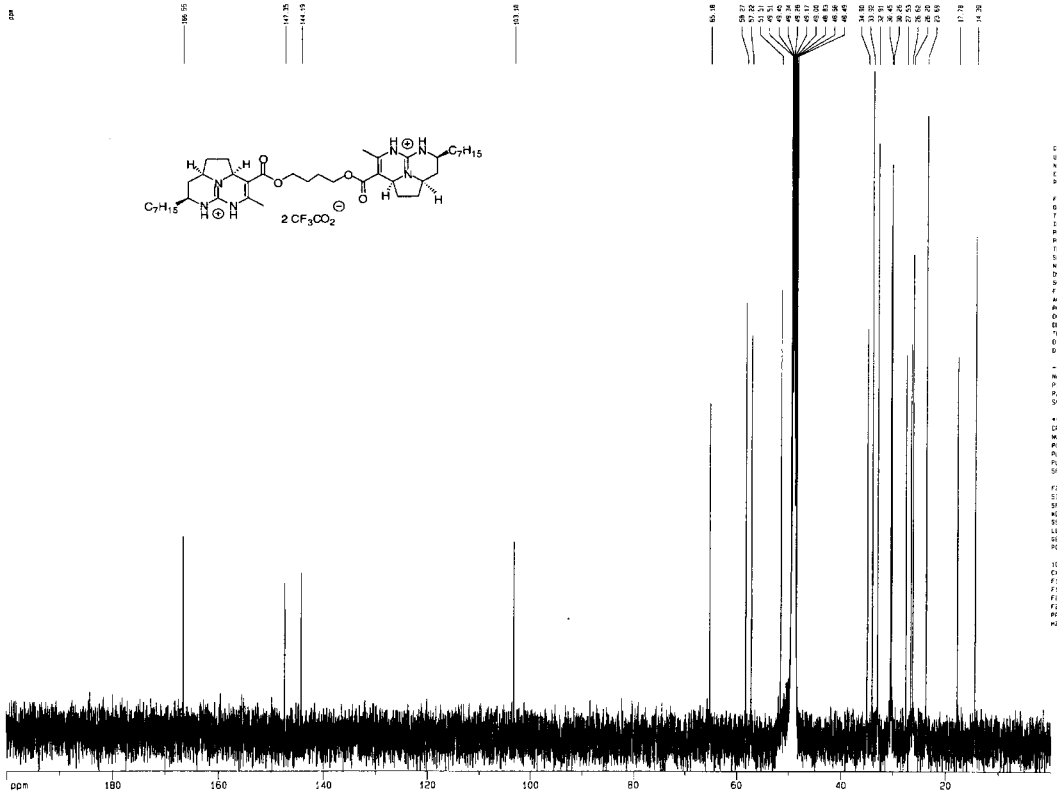
13C spectrum with 1H decoupling



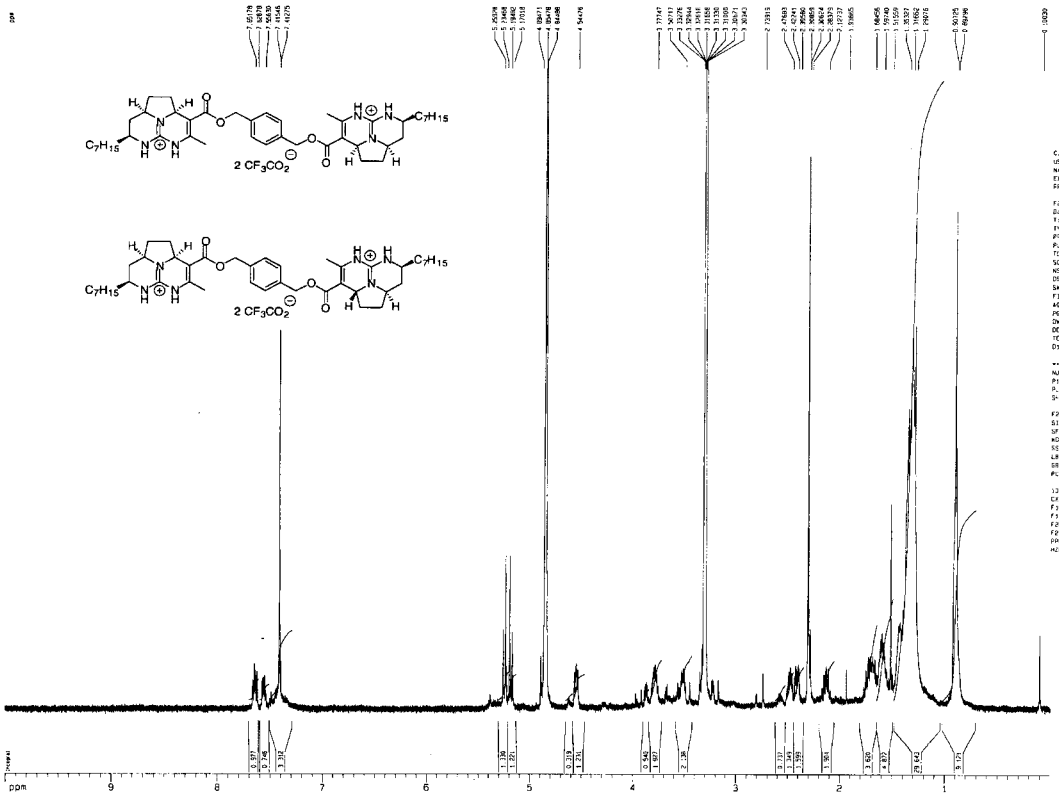
1H spectrum



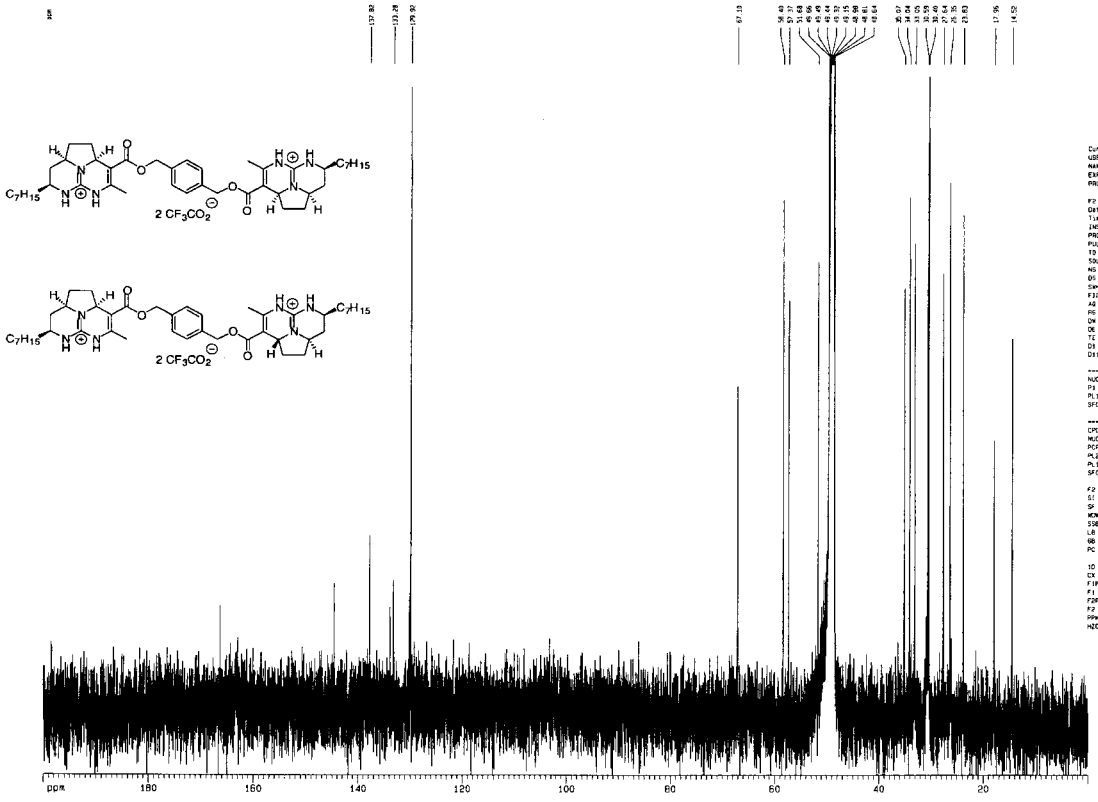
13C spectrum with 1H decoupling



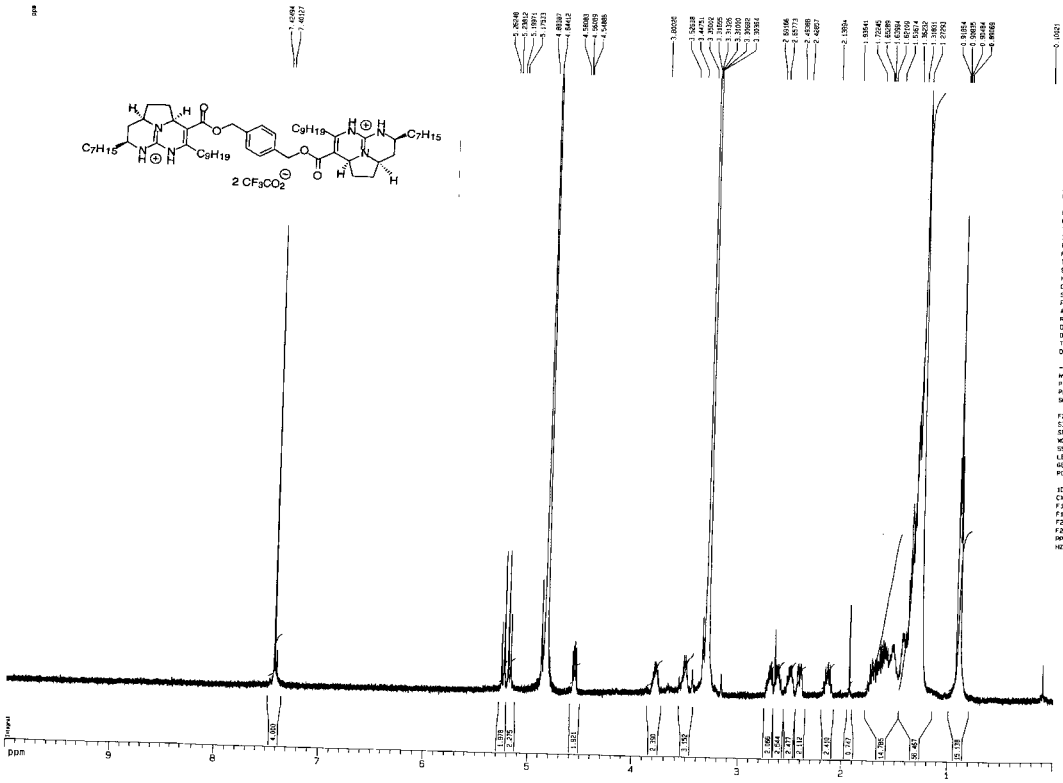
1H spectrum



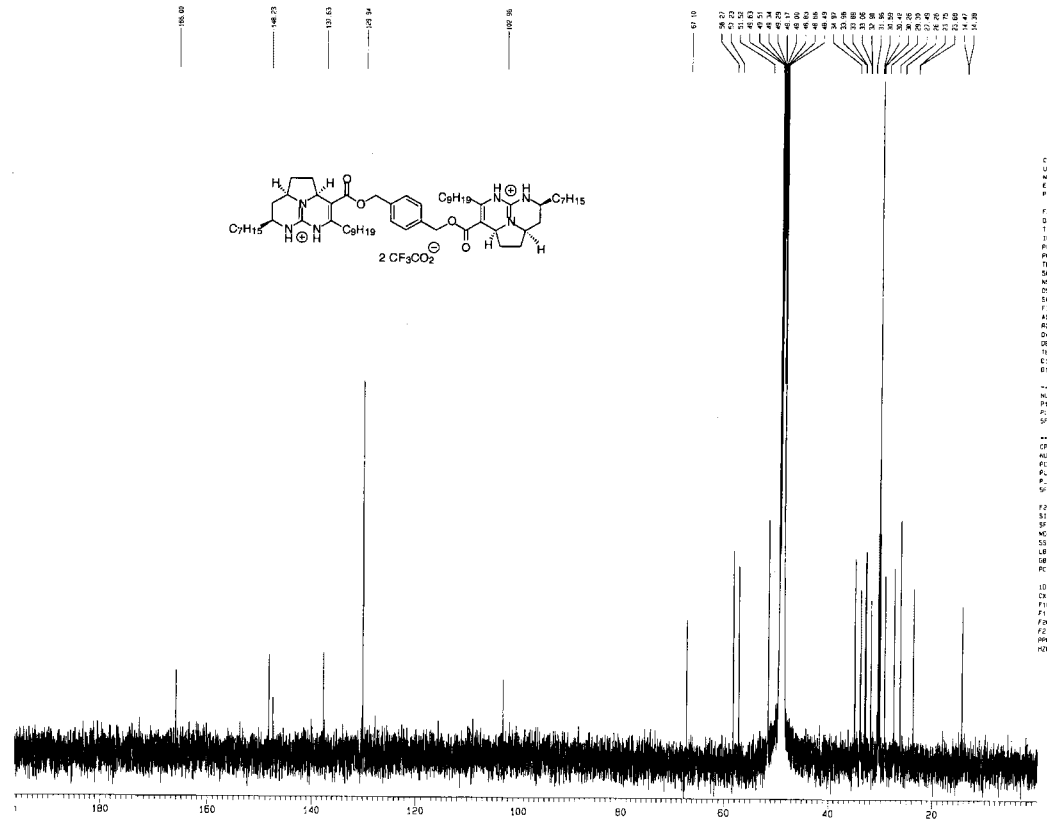
13C spectrum with 1H decoupling



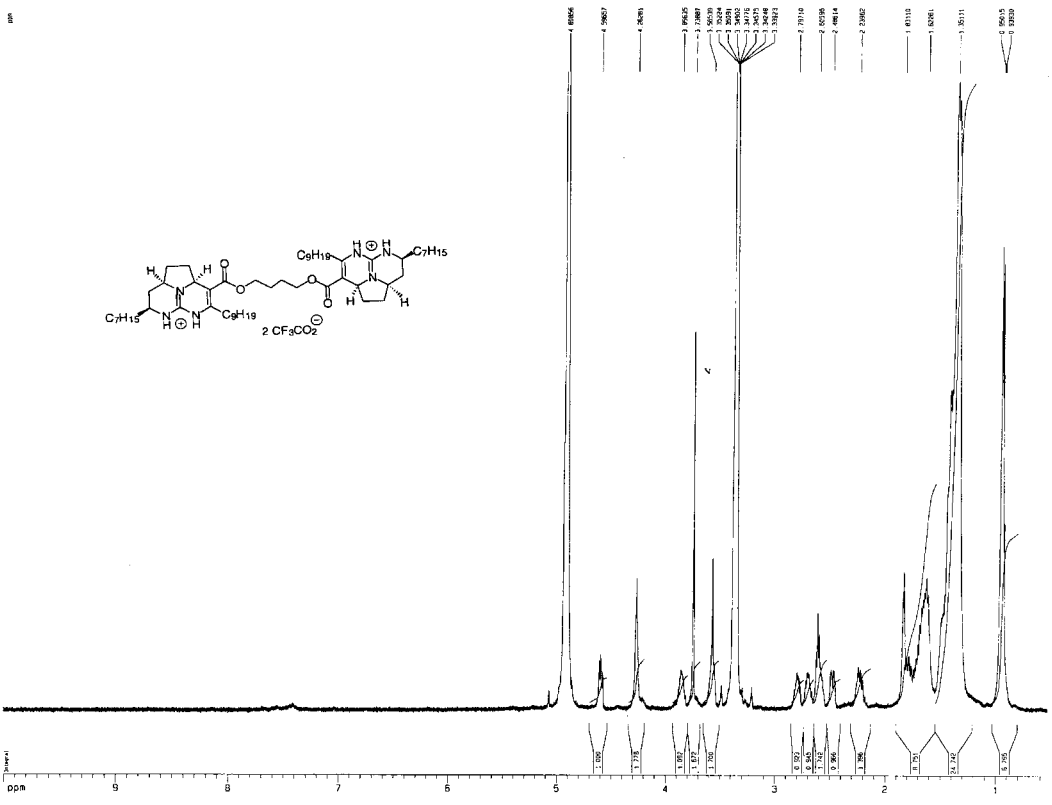
1H spectrum



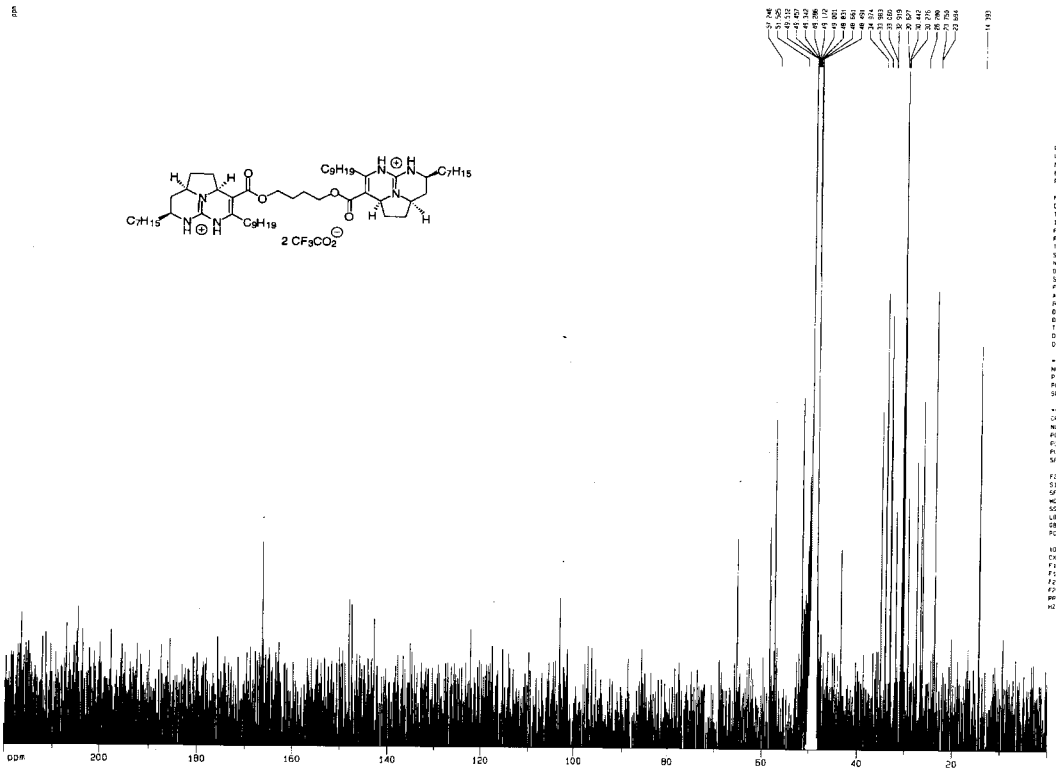
13C spectrum with 1H decoupling



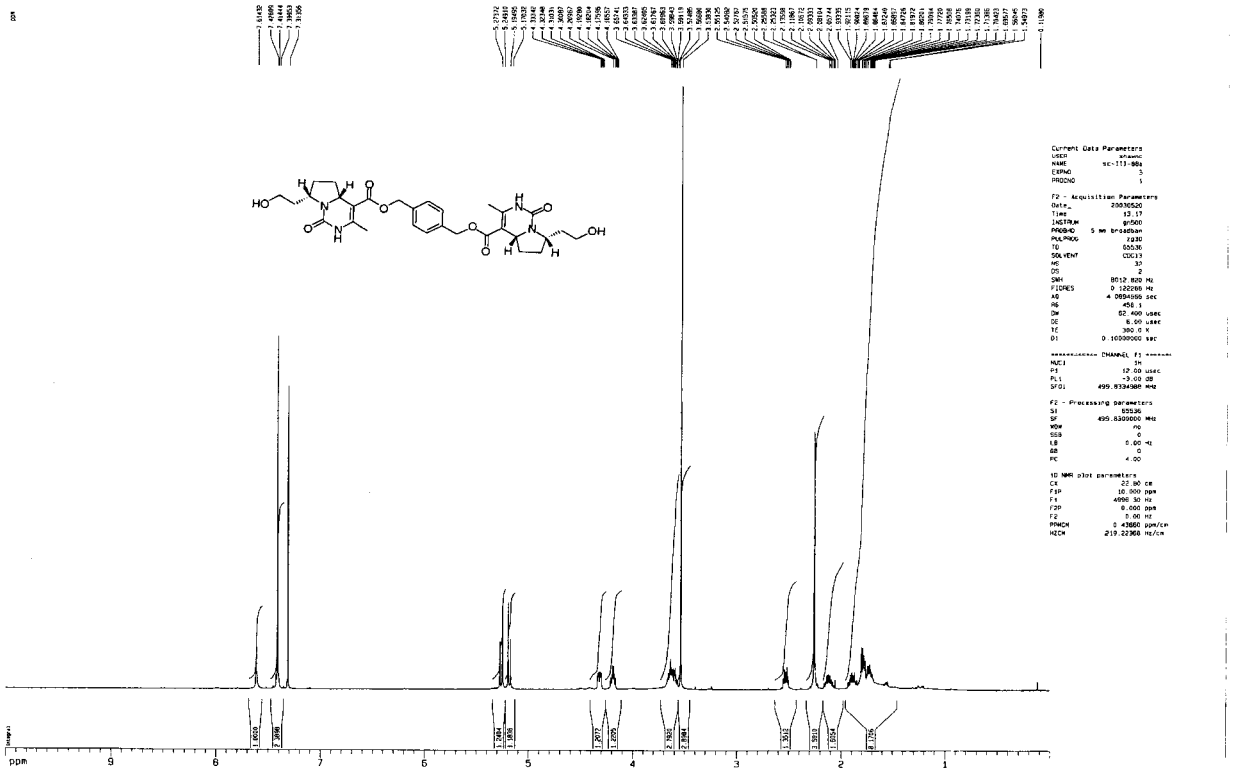
1H spectrum



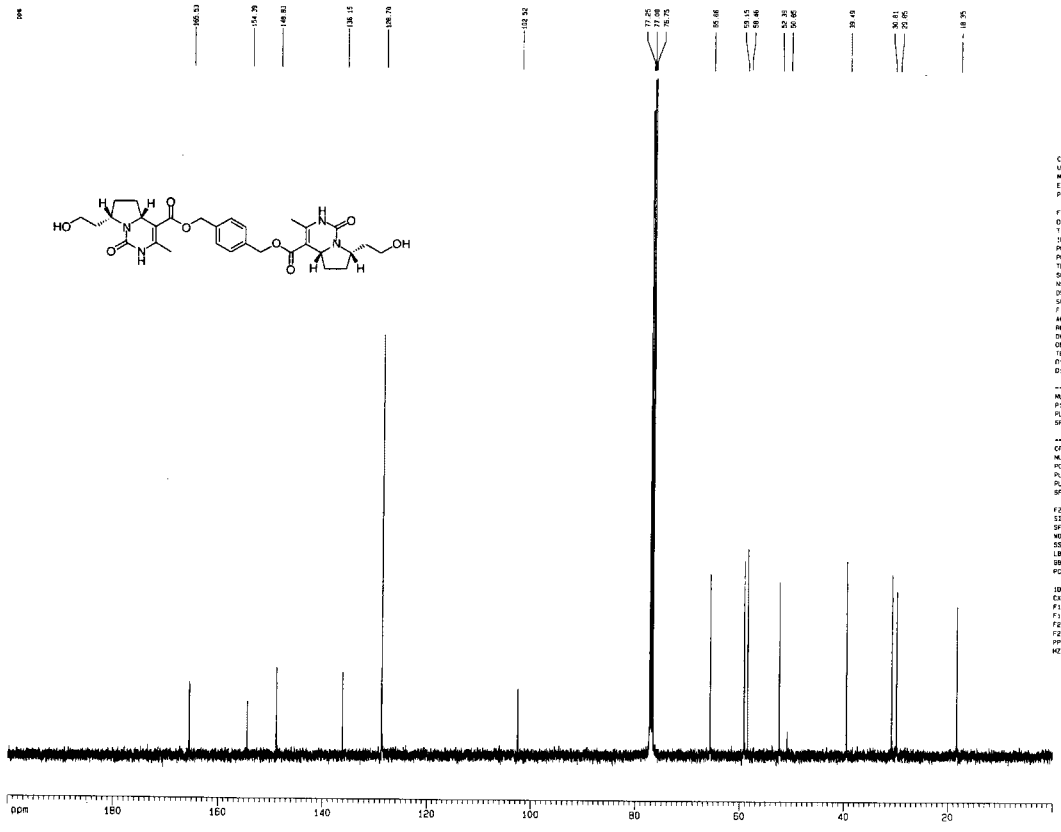
13C spectrum with 1H decoupling



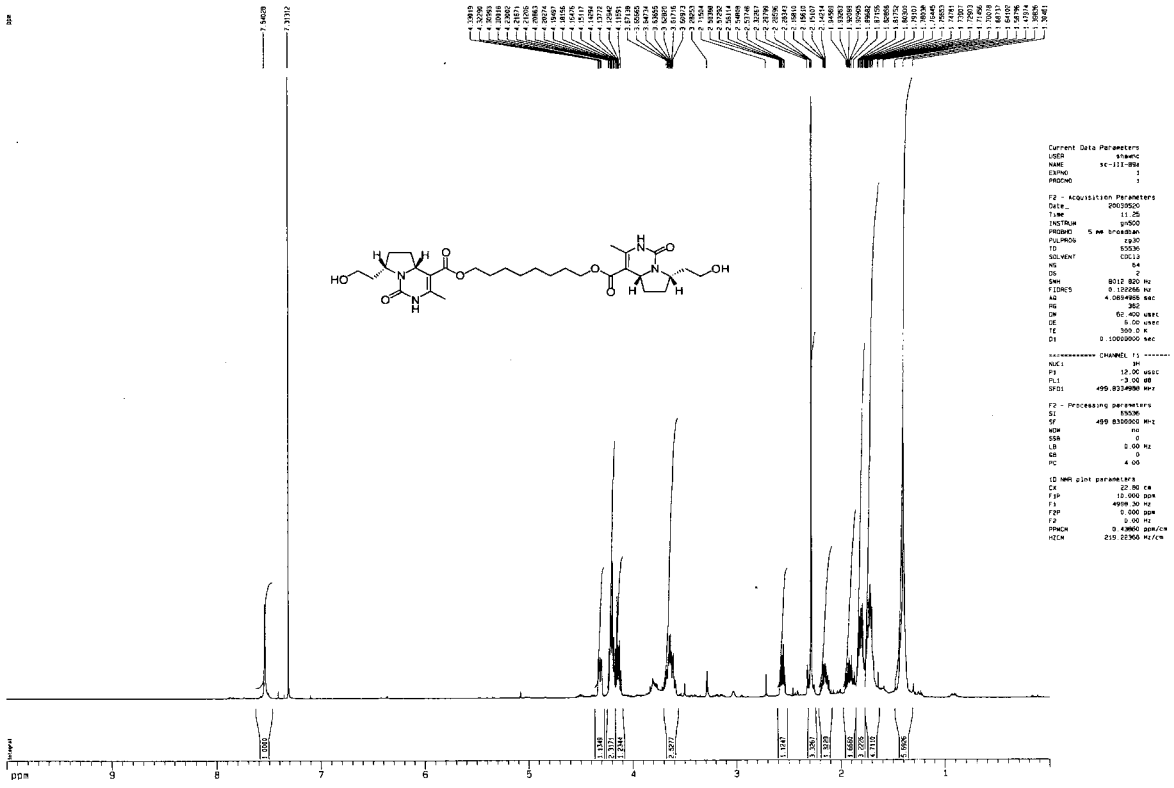
1H spectrum



13C spectrum with 1H decoupling



1H spectrum



Current Data Parameters
USER shawnc
NAME sc-III-89d
EXPNO 1
PROCNO 1

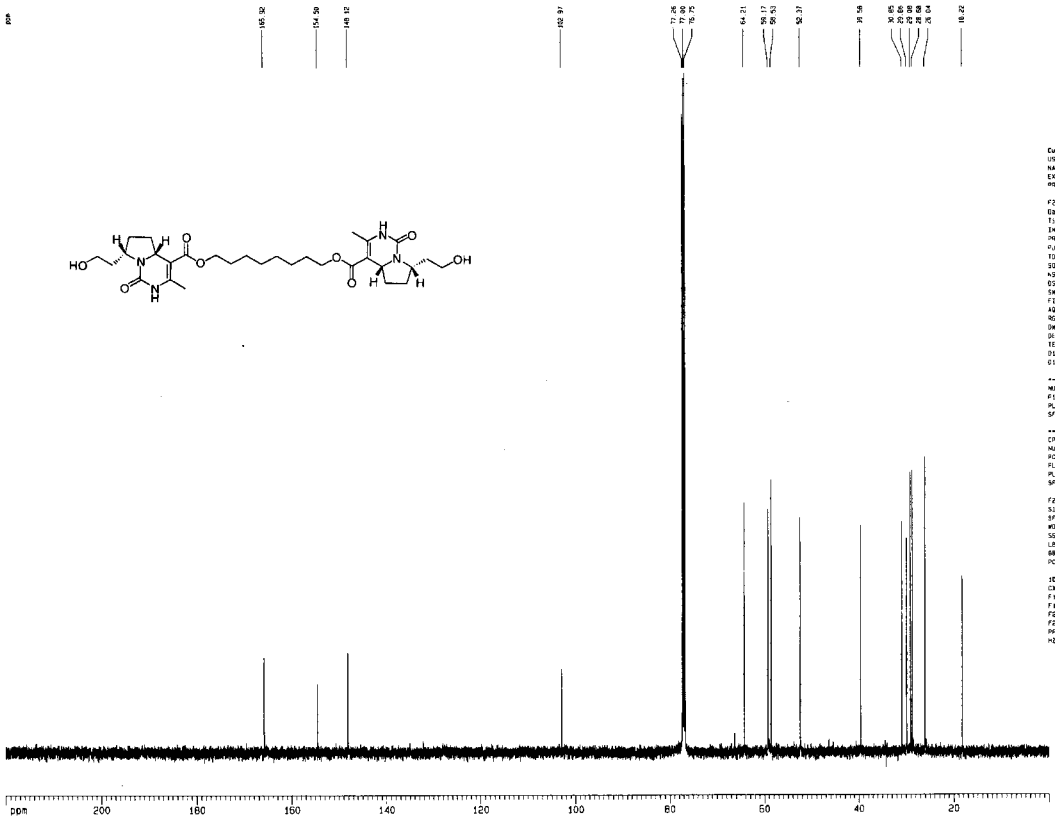
F2 - Acquisition Parameters
Date_ 20030520
Time 11.25
INSTRUM qn500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894988 sec
RG 362
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

========== CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8334988 MHz

F2 - Processing parameters
SI 65536
SF 499.8300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 4998.30 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43860 ppm/cm
HZCM 219.23360 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER shawnc
NAME sc-III-89a
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030520
Time 11.30
INSTRUM qn500
PROBHD 5 mm broadban
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3420
DS 4
SWH 30303.031 Hz
FIDRES 0.462398 Hz
AQ 1.0813946 sec
RG 2896.3
DW 16.500 usec
DE 6.50 usec
TE 303.0 K
D1 0.25000000 sec
D11 0.03000000 sec

========== CHANNEL f1 =======
NUC1 13C
P1 15.00 usec
PL1 0.00 dB
SFO1 125.6961791 MHz

========== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 60.00 usec
PL2 -3.00 dB
PL12 15.10 dB
SFO2 499.8334988 MHz

F2 - Processing parameters
SI 65536
SF 125.6823594 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

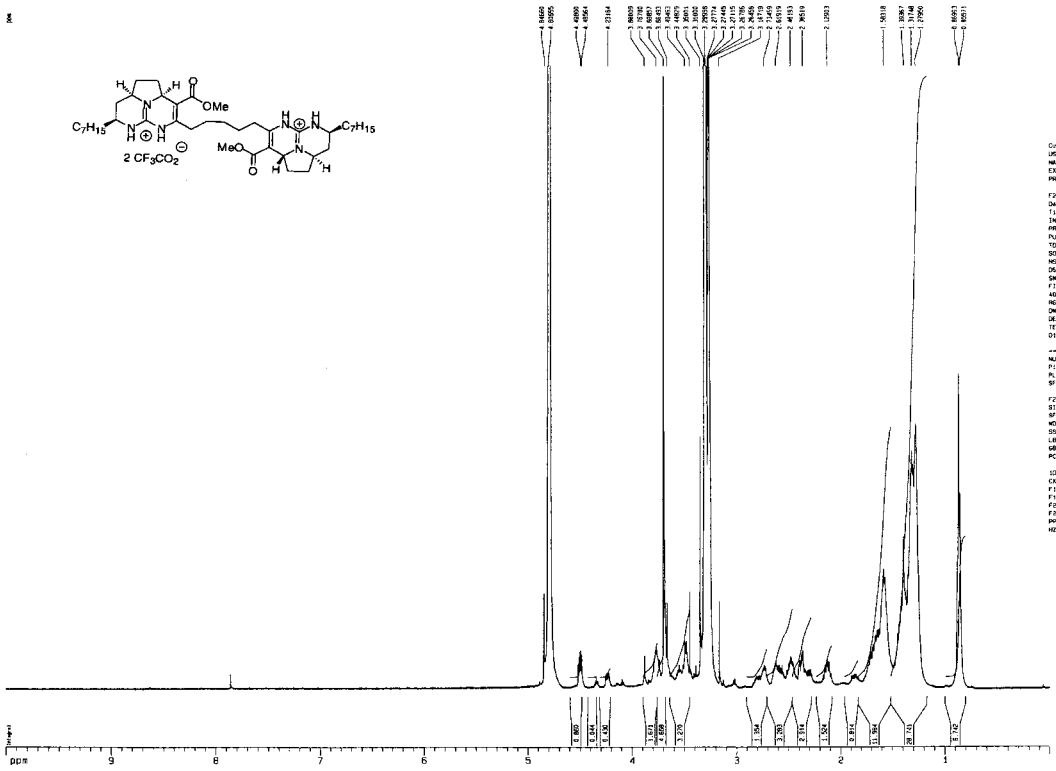
1D NMR plot parameters
CX 22.80 cm
F1P 220.000 ppm
F1 27650.12 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 9.64912 ppm/cm
HZCM 1212.72449 Hz/cm

^{1}H and ^{13}C NMR Spectra

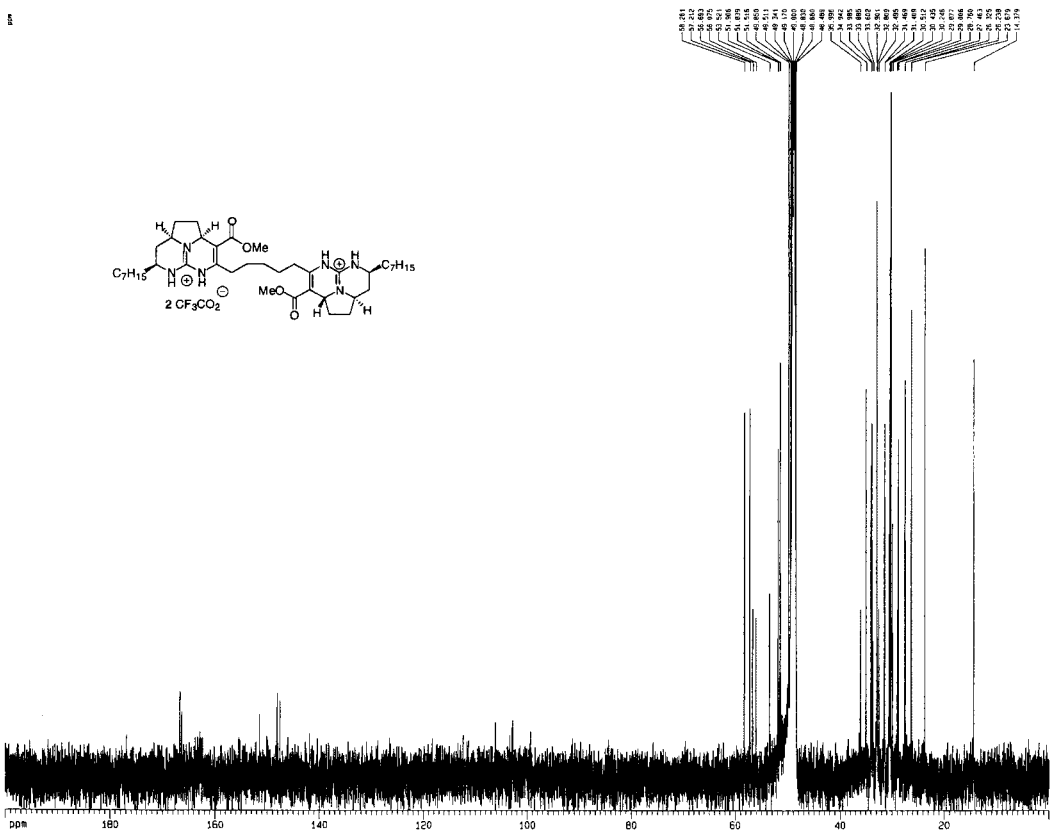
C. Minor Stereoisomers of Selected Double Biginelli Products

These samples are typically contaminated with some of the major C_2-symmetric isomers.

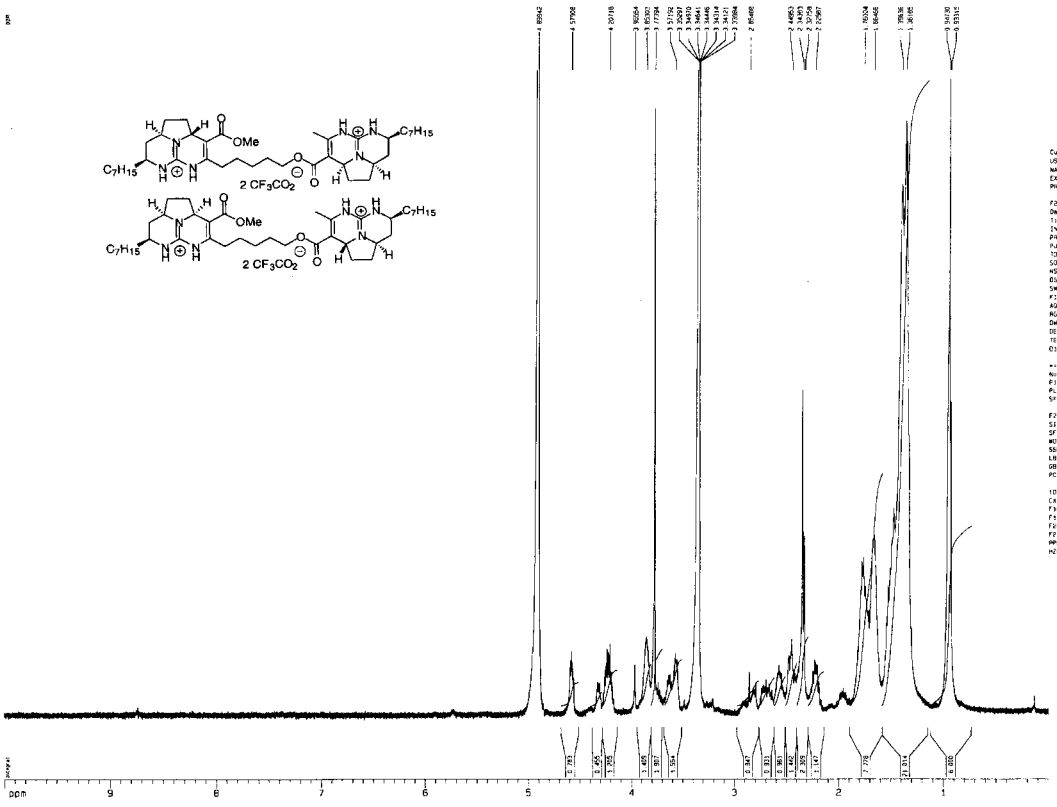
1H spectrum



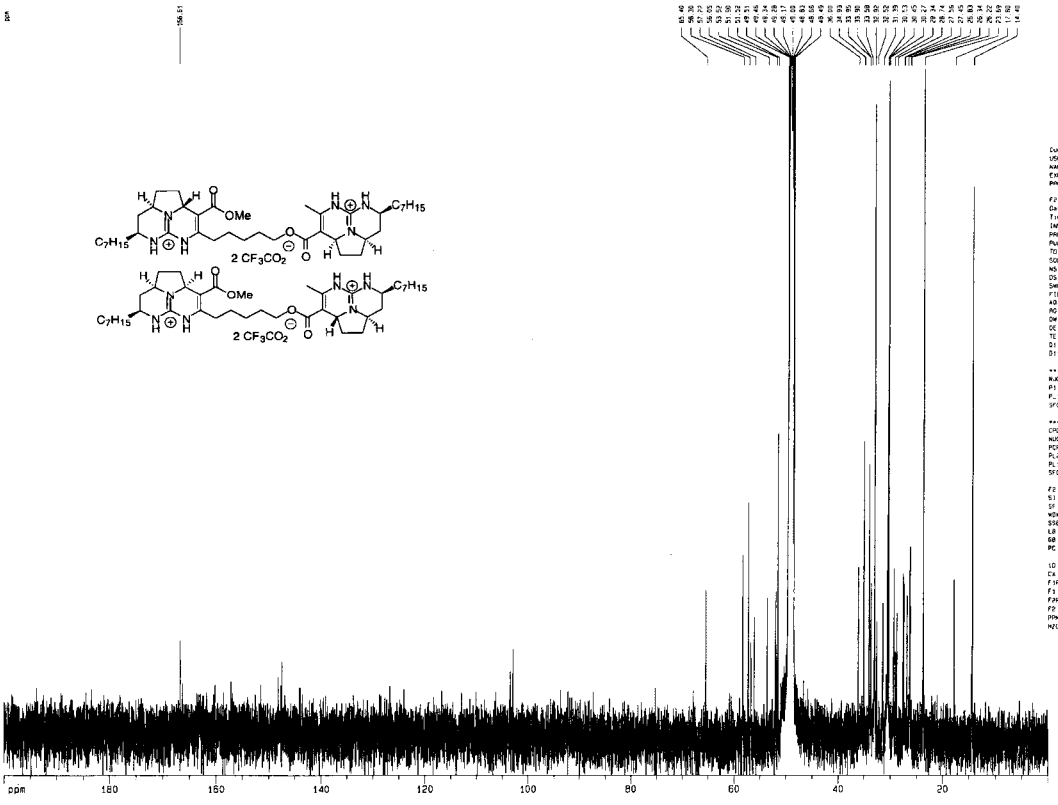
13C spectrum with 1H decoupling



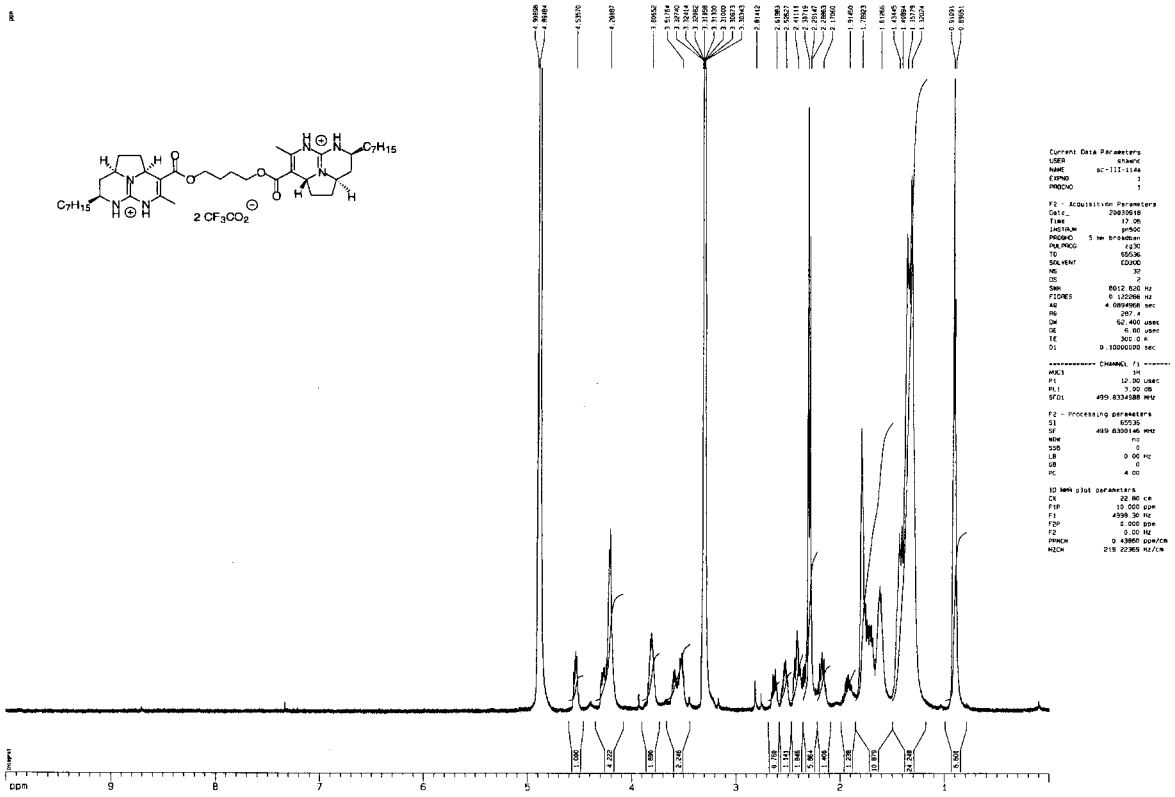
1H spectrum



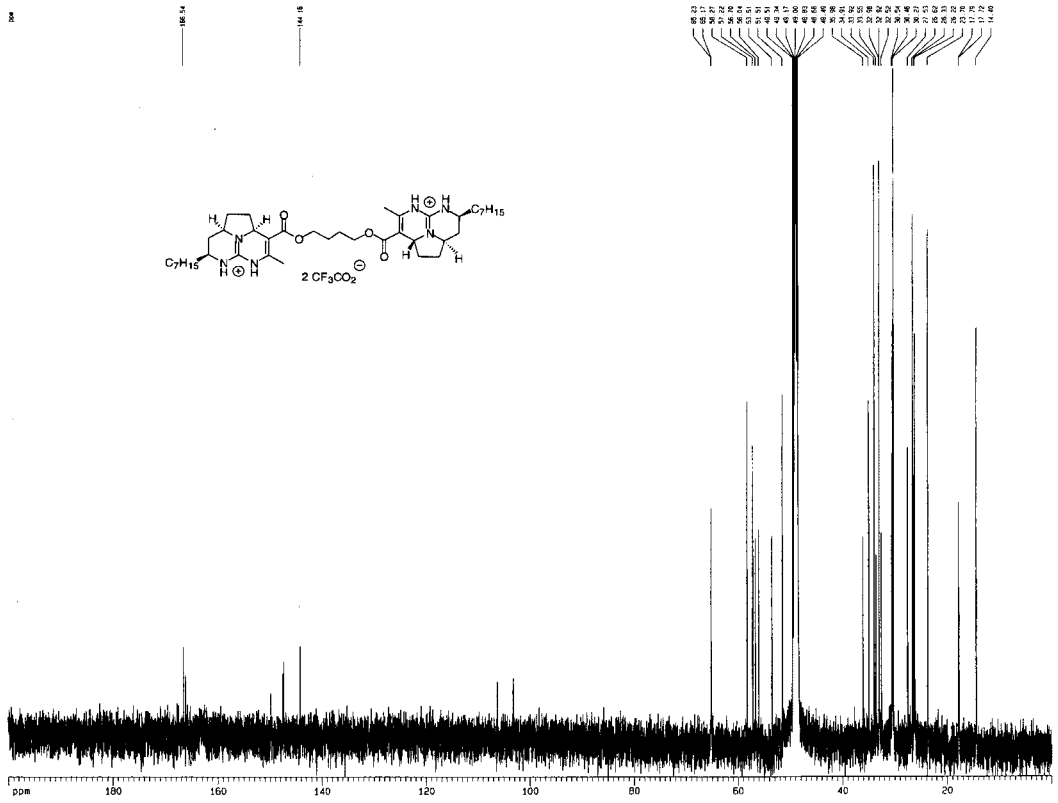
13C spectrum with 1H decoupling



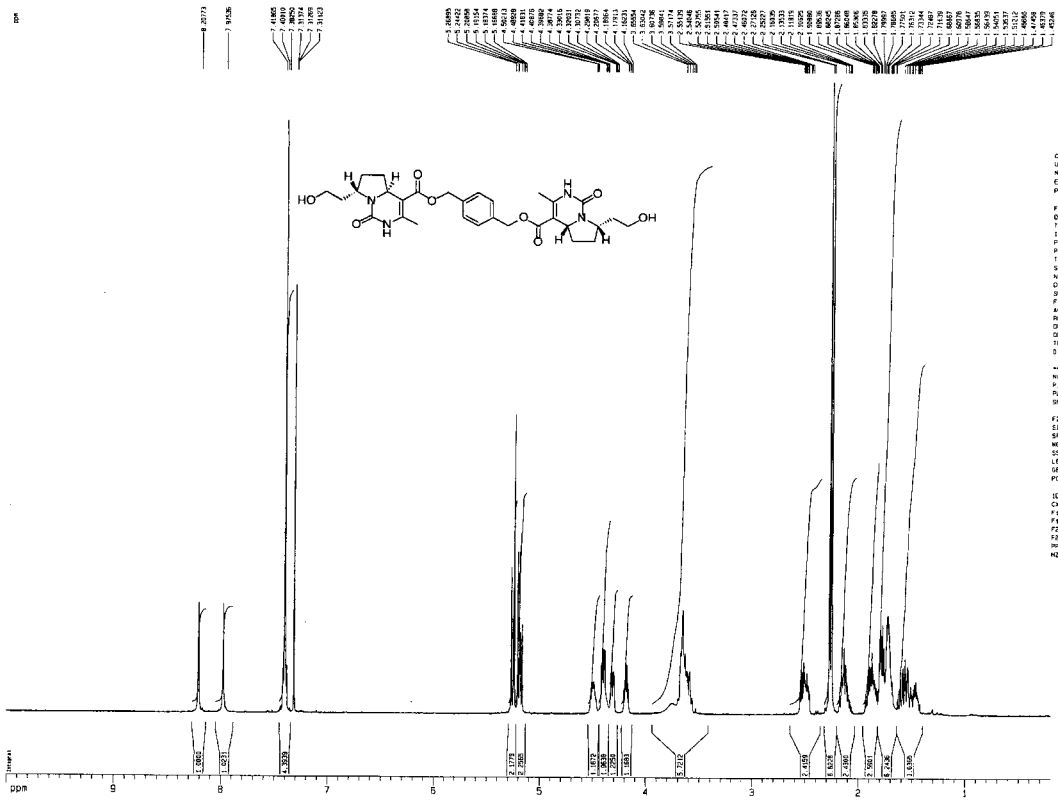
1H spectrum



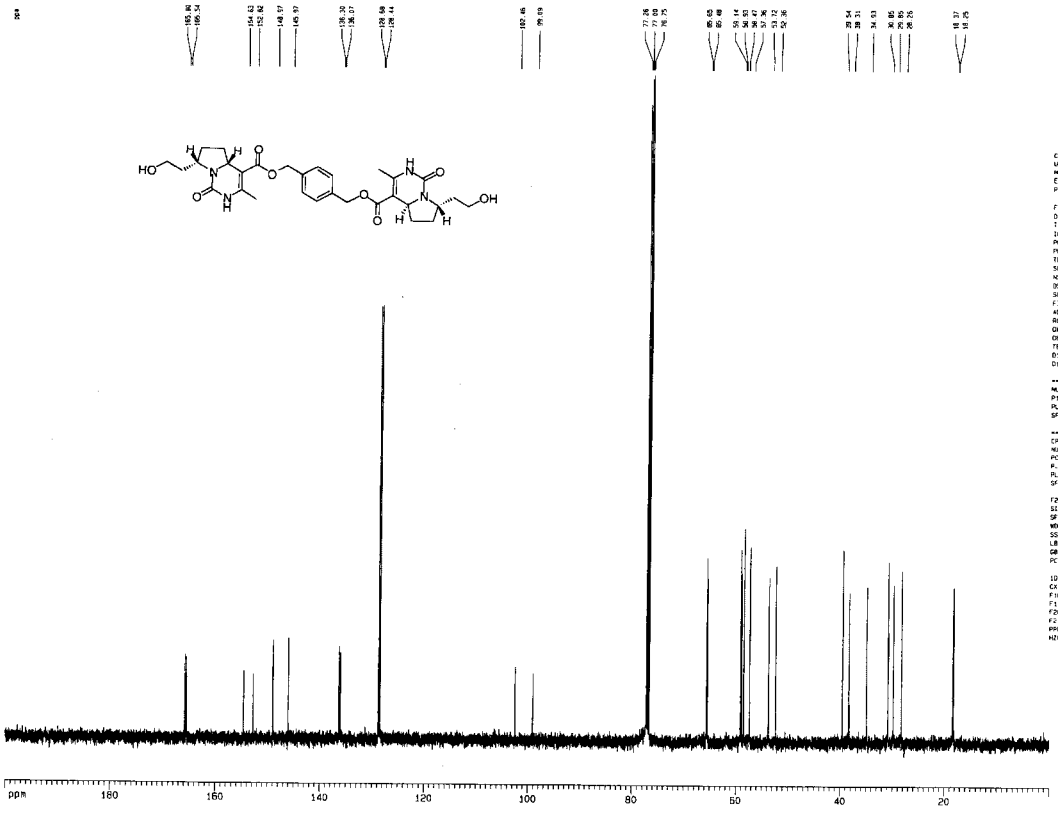
13C spectrum with 1H decoupling



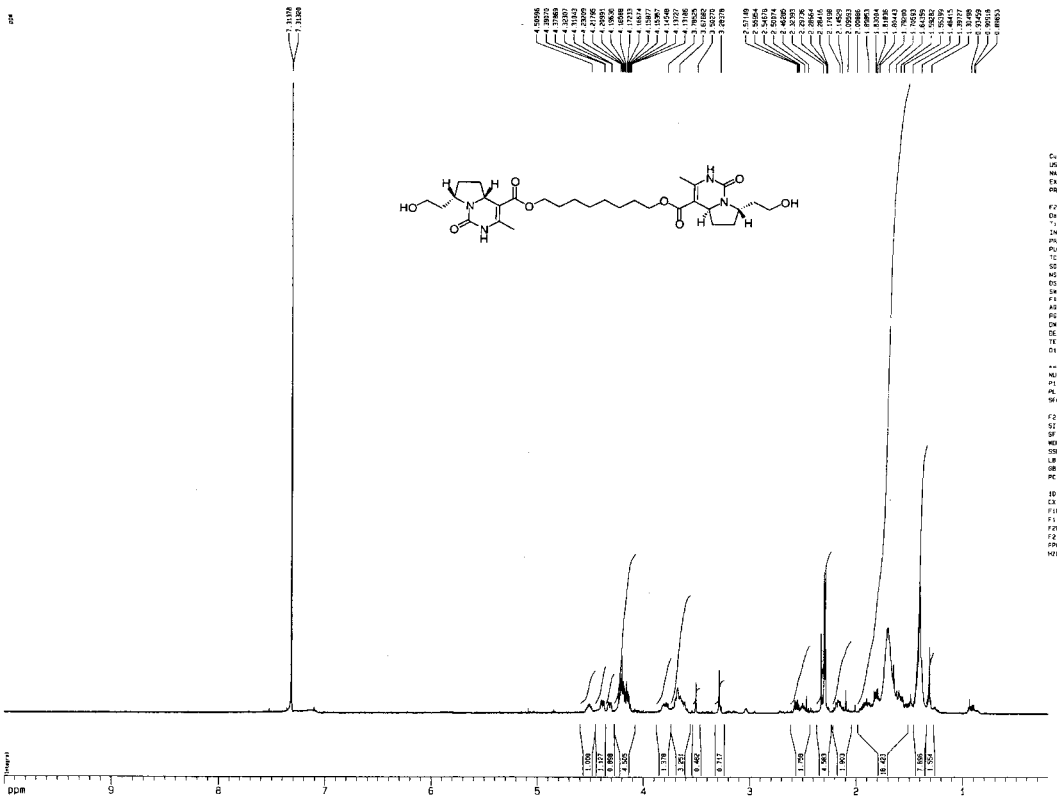
1H spectrum



13C spectrum with 1H decoupling



1H spectrum



Current Data Parameters
USER shawnc
NAME sc-III-86a
EXPNO 4
PROCNO 1

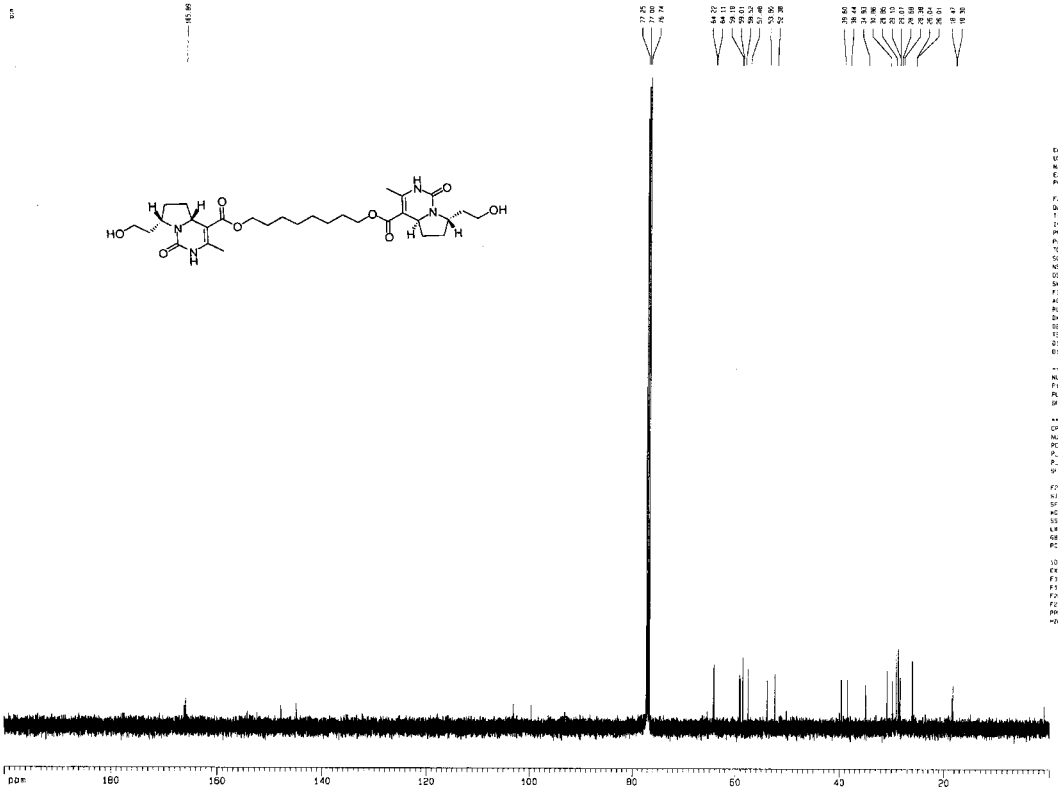
F2 - Acquisition Parameters
Date_ 20030522
Time 17.05
INSTRUM gn500
PROBHD 5 mm Broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 512
DW 62.400 usec
DE 6.00 usec
TE 303.0 K
D1 0.10000000 sec

========== CHANNEL f1 =======
NUC1 1H
P1 12.00 usec
PL1 -3.00 dB
SFO1 499.8334980 MHz

F2 - Processing parameters
SI 65536
SF 499.8300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.50

1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 4998.30 Hz
F2P 0.000 ppm
F2 99.97 Hz
PPMCM 0.42982 ppm/cm
HZCM 214.83920 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
USER shawnc
NAME sc-III-86a
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date 20030522
Time 17.08
INSTRUM gn500
PROBHD 5 mm Broadban
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5799
DS 4
SWH 30303.031 Hz
FIDRES 0.462388 Hz
AQ 1.0813940 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 302.0 K
D1 0.25000000 sec
D11 0.03000000 sec

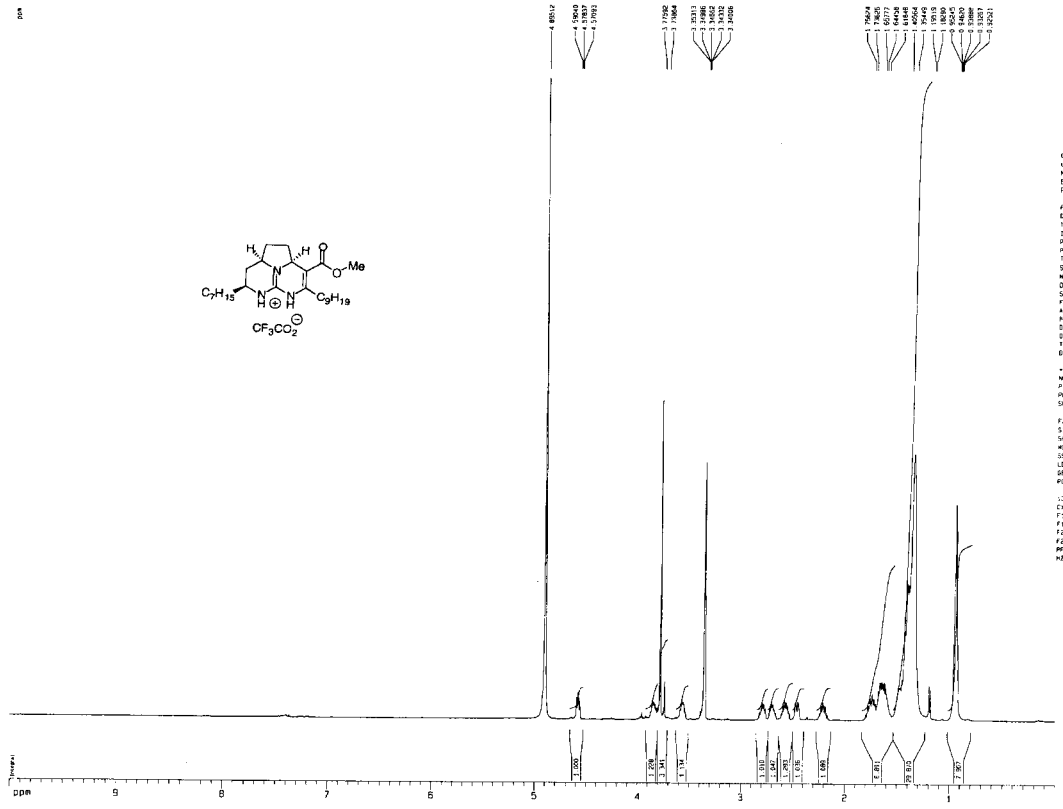
========== CHANNEL f1 =======
NUC1 13C
P1 15.00 usec
PL1 0.00 dB
SFO1 125.6961751 MHz

========== CHANNEL f2 =======
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 3.00 dB
PL12 15.10 dB
SFO2 499.8334988 MHz

F2 - Processing parameters
SI 65536
SF 125.6903994 MHz
WDW no
SSB 0
LB 0.90 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
F1P 250.000 ppm
F1 25136.47 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.77153 ppm/cm
HZCM 1102.47681 Hz/cm

1H spectrum



Current Data Parameters
NAME sc-III-105a
EXPNO 2
PROCNO 1

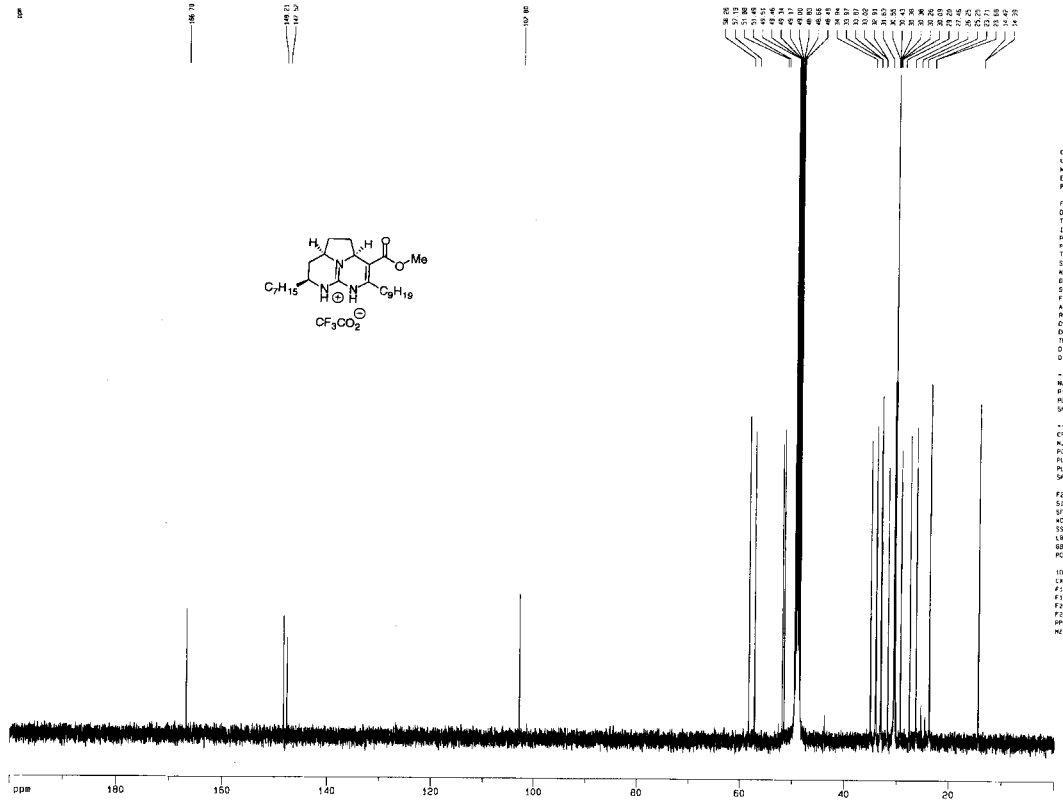
F2 - Acquisition Parameters
Date_ 20030610
Time 16.09
INSTRUM omega500
PROBHD 5 mm Broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 9012.620 Hz
FIDRES 0.137506 Hz
AQ 4.0804561 sec
RG 128
DW 62.400 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 -1.00 dB
SFO1 500.2235015 MHz

F2 - Processing parameters
SI 65536
SF 500.2200000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 4.00

1D NMR plot parameters
CX 22.80 cm
F1P 10.000 ppm
F1 5002.20 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.43800 ppm/cm
HZCM 219.05474 Hz/cm

13C spectrum with 1H decoupling



Current Data Parameters
NAME sc-III-105a
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20030610
Time 16.52
INSTRUM omega500
PROBHD 5 mm Broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 4471
DS 4
SWH 30303.031 Hz
FIDRES 0.462390 Hz
AQ 1.0813946 sec
RG 5160.6
DW 16.500 usec
DE 4.50 usec
TE 300.0 K
D1 0.25000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 22.20 usec
PL1 6.00 dB
SFO1 125.7942048 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 1.00 dB
PL12 14.40 dB
SFO2 500.2230013 MHz

F2 - Processing parameters
SI 65536
SF 125.7802458 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 2.00

1D NMR plot parameters
CX 22.80 cm
F1P 200.000 ppm
F1 25156.05 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 8.77193 ppm/cm
HZCM 1103.33545 Hz/cm