

Supporting Information for:

Synthesis of Cyclohexanes via [3+3] Hexannulation of Cyclopropanes and 2-Chloromethyl Allylsilanes

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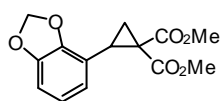
General Considerations

Melting points were determined using a Gallenkamp melting point apparatus and are uncorrected. Infrared spectra were obtained as thin films on NaCl plates using a Bruker Vector 33 FT-IR instrument. NMR experiments were performed on Varian Mercury 400, Varian Inova 600 and Inova 400 instruments and samples were obtained in CDCl₃ (referenced to residual CHCl₃ at 7.26 ppm for ¹H and 77.0 ppm for ¹³C) or DMSO-*d*₆ (referenced to residual DMSO at 2.50 ppm for ¹H and 39.5 ppm for ¹³C). 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, m = multiplet, br = broad. High resolution mass spectra (HRMS) were obtained on a Finnigan MAT 8200 spectrometer at 70 eV. Microwave reactions were performed in a 400 W Biotage Initiator 2.0 microwave reactor. All reactions were performed under an atmosphere of argon unless otherwise indicated.

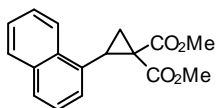
Toluene, tetrahydrofuran (THF), *N,N*-dimethylformamide (DMF), and methylene chloride were dried and deoxygenated by passing the nitrogen purged solvents through activated alumina columns. 2-(Chloromethyl)-3-trimethylsilyl-1-propene **2a** (97%, Aldrich) was used as received. All other reagents and solvents were used as purchased from Aldrich, Strem, Caledon or VWR. Reaction progress was monitored by thin layer chromatography (TLC) (EM Science, silica gel 60 F₂₅₄), visualizing with UV light, and the plates developed using *p*-anisaldehyde or basic potassium permanganate stains. Flash chromatography was performed using silica gel purchased from Silicycle Chemical Division Inc. (230-400 mesh).

Experimental Procedure, Spectral Data for Cyclopropanediesters 3a-h

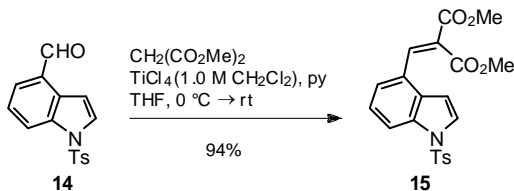
Cyclopropanediesters **3a-f, h** were prepared from the respective arylidenemalonates according to the procedure of Corey and Chaykovsky:¹ for a general procedure, see preparation of cyclopropanediester **3f** (p. S3). Vinyl cyclopropanediester **3g** was prepared by a one-step literature procedure.² Cyclopropanediesters **3a**³, **b**⁴, **e**⁵ and **h**⁶ are known compounds and were prepared with spectral properties identical to those previously reported.



3c Dimethyl 2-(piperon-2-yl)cyclopropane-1,1-dicarboxylate was prepared according to the procedure above: 1.19 g (4.42 mmol) dimethyl 2(piperon-2-ylmethylene)malonate, 1.07 g (4.86 mmol) trimethylsulfoxonium iodide and 0.23 g (5.75 mmol) NaH in DMSO reacted to yield 1.14 g **3c** in 90% yield (1.14 g, 4.1mmol) as a cream-coloured solid; m.p. 58-60 °C; $R_f = 0.33$ (30 % EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃): δ 6.65-6.71 (m, 3H), 5.92 (s, 2H), 3.78 (s, 3H), 3.45 (s, 3H), 3.15 (t, $J = 8.8$ Hz, 1H), 2.11 (dd, $J = 8.0$, 5.2 Hz, 1H), 1.70 (dd, $J = 9.6$, 5.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 170.3, 167.2, 147.7, 147.1, 128.5, 122.1, 53.0, 52.5, 37.2, 32.6, 29.9, 19.6; IR (thin film, cm⁻¹): 3002, 2953, 2903, 1724, 1506, 1492, 1437, 1333, 1285, 1213, 1128, 1038, 933, 815, 769, 703; HRMS calculated for C₁₄H₁₄O₆ 278.0790; found 278.0784.



3d Dimethyl 2-(naphthalen-1-yl)cyclopropane-1,1-dicarboxylate was prepared according to the aforementioned procedure: 1.19 g (4.42 mmol) dimethyl 2(naphthalene-1-ylmethylene)malonate, 1.07 g (4.86 mmol) trimethylsulfoxonium iodide and 0.23 g (5.75 mmol) NaH in DMSO reacted to yield 1.14 g **3d** (90% yield, 16 h) as a cream-coloured solid, m.p. 76-78 °C; $R_f = 0.41$ (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 8.17 (d, $J = 7.8$ Hz, 1H), 7.82 (d, $J = 7.2$ Hz, 1H), 7.75 (d, $J = 8.4$ Hz, 1H), 7.54 (t, $J = 7.2$ Hz, 1H), 7.49 (t, $J = 6.6$ Hz, 1H), 7.39 (t, $J = 7.2$ Hz, 1H), 7.30 (d, $J = 7.2$ Hz, 1H), 3.88 (s, 3H), 3.68-3.71 (m, 1H), 3.05 (s, 3H), 2.43 (dd, $J = 6.4$, 4.8 Hz, 1H), 1.85 (dd, $J = 9.0$, 4.8 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 170.6, 167.3, 133.6, 133.2, 131.0, 128.6, 128.0, 126.4, 126.1, 125.9, 125.2, 124.5, 53.2, 52.1, 37.0, 30.6, 19.1; IR (thin film, cm⁻¹): 3048, 3011, 2954, 2849, 1732, 1597, 1510, 1436, 1404, 1342, 1287, 1209, 1131, 1105, 999, 986, 969, 894, 805, 783, 757; HRMS calculated for C₁₇H₁₆O₄ 284.1049; found 284.0153.



15 Dimethyl 2-(1-tosyl-1H-indol-4-ylmethylene)-malonate was prepared as follows: to a flask with THF (30 mL, anhydrous) cooled to 0 °C was added dropwise TiCl₄ (9.0 mL, 1.0 M solution in CH₂Cl₂). Aldehyde **14**⁷ (1.34 g, 4.48 mmol) in THF (25 mL) was added via cannula, followed by dimethylmalonate (0.51 mL, 4.48 mmol). Pyridine (1.45 mL, 17.9 mmol) was then added dropwise, and the mixture was allowed to warm to rt over 3 h. The reaction was

¹ Corey, E. J.; Chaykovsky, M. *J. Am. Chem. Soc.* **1965**, *87*, 1353.

² Kierstead, R. W.; Linstead, R. P.; Weedon, B. C. L. *J. Chem. Soc.* **1952**, 3610.

³ Ashraf, G.; Aboul-Enein, H. Y.; Mueller, P. *Chirality* **2004**, *17*, 44.

⁴ Mueller, P.; Ghanem, A. *Org. Lett.* **2004**, *6*, 4347.

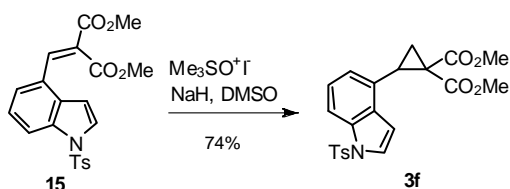
⁵ Shostakovskii, V. M.; *Izvestiya Akademii Nauk SSSR, Seriya Khimicheskaya* **1982**, *9*, P2126.

⁶ Verhe, R.; De Kimpe, N.; De Buyck, L.; Courtheyn, D.; Schamp, N. *Synthesis* **1978**, *7*, 530.

⁷ Using procedure established by Batcho and Leimgruber: *Org. Synth.* **1985**, *63*, 214-220.

Preparation of indole-4-carboxaldehyde: Kozikowski, A. P.; Ishida, H.; Chen, Y.-Y. *J. Org. Chem.* **1980**, *45*, 3350.

quenched with aqueous NH_4Cl , and mixture extracted 3x with Et_2O . Extracts were further washed with water, brine, and dried over MgSO_4 . After filtration and concentration *in vacuo*, the residue was purified by flash column chromatography on silica gel (1:4 EtOAc/hexanes) to afford **15** in 94% yield (1.73 g, 4.18 mmol) as a cream-coloured solid; m.p. 138-140 °C; $R_f = 0.15$ (30% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3): δ 8.07 (s, 1H), 8.03 (d, $J = 7.6$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 2H), 7.66 (d, $J = 4.0$ Hz, 1H), 7.32-7.29 (m, 2H), 7.23 (d, $J = 8.4$ Hz, 2H), 6.82 (d, $J = 3.6$ Hz, 1H), 3.86 (s, 3H), 3.77 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3): δ 167.2, 164.6, 145.6, 139.6, 135.2, 135.1, 131.2, 130.2, 127.7, 127.1, 126.7, 126.0, 124.9, 122.7, 115.9, 106.9, 53.0, 52.9, 21.8; IR (thin film, cm^{-1}): 3144, 2954, 2918, 2849, 1733, 1628, 1596, 1437, 1377, 1364, 1250, 1226, 1204, 1189, 1140, 1089, 1061, 987, 860, 813, 737, 704, 574; HRMS calculated for $\text{C}_{21}\text{H}_{19}\text{NO}_6\text{S}$ 413.0933; found 413.0925.

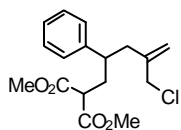


3f Dimethyl 2-(1-tosyl-1H-indol-4-yl)cyclopropane-1,1-dicarboxylate was prepared as follows: to a flask charged with a magnetic stirrer, NaH (0.218 g, 5.44 mmol, 60% dispersion in oil) and trimethylsulfoxonium iodide (1.01 g, 4.60 mmol) was added DMSO dropwise (6 mL). After gas evolution had subsided and the mixture became clear, a solution of

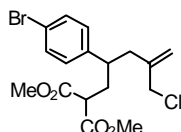
malonate **15** (1.73 g, 4.18 mmol) in DMSO (6 mL) was added via cannula, and the reaction stirred 15 h under argon. The mixture was then diluted with water, and extracted with EtOAc x4. Combined extracts were washed with water, brine, and dried over MgSO_4 . After filtration and concentration *in vacuo*, the residue was purified by flash column chromatography on silica gel (1:4 EtOAc/hexanes) to afford cyclopropane **3f** in 74% yield (1.30 g, 3.04 mmol) as a pale yellow solid; m.p. 126-128 °C; $R_f = 0.15$ (30% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3): δ 7.88 (d, $J = 8.4$ Hz, 1H), 7.72 (d, $J = 8.4$ Hz, 2H), 7.56 (d, $J = 3.6$ Hz, 1H), 7.23-7.19 (m, 3H), 6.94 (d, $J = 7.2$ Hz, 1H), 6.80 (d, $J = 3.6$ Hz, 1H), 3.81 (s, 3H), 3.38 (t, $J = 8.8$ Hz, 1H), 3.00 (s, 3H), 2.32 (s, 3H), 2.27 (dd, $J = 8.0, 4.8$ Hz, 1H), 1.76 (dd, $J = 9.2, 4.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 170.3, 167.2, 145.2, 135.4, 134.8, 131.7, 130.0, 127.7, 127.0, 126.6, 124.6, 122.2, 113.2, 107.9, 53.2, 52.0, 36.8, 30.1, 21.7, 18.9; IR (thin film, cm^{-1}): 3144, 3001, 1729, 1597, 1527, 1493, 1437, 1373, 1334, 1282, 1212, 1179, 1131, 1090, 1041, 990, 888, 844, 762, 704, 685, 646; HRMS calc'd for $\text{C}_{22}\text{H}_{21}\text{NO}_6\text{S}$ 427.1090; found 427.1097.

Experimental Procedure, Spectral Data for Allylated Products 4a-h

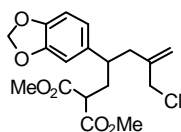
Compounds **4a-h** were prepared by the procedure as follows: to a solution of cyclopropanediester **3** (1.0 equiv.) in anhydrous methylene chloride under an atmosphere of argon, cooled to -78 °C was added TiCl_4 dropwise (1.0 M solution in methylene chloride, 1.0 equiv.). The mixture was stirred for the time indicated under an argon atmosphere, at which time a deep red colour was observed. 2-(Chloromethyl)-3-trimethylsilyl-1-propene **2a** (1.2 equiv.) was then added dropwise, and the reaction mixture was stirred under argon for the time indicated, allowing the cooling bath to warm to 0 °C. When consumption of starting material was observed by TLC, the reaction mixture was quenched by dropwise addition of a saturated solution of NH_4Cl , immediately removed from the cooling bath and warmed to room temperature. The mixture was further diluted and extracted twice with EtOAc, and the organics washed successively with NH_4Cl , water and brine. The organic extracts were dried over MgSO_4 , filtered, concentrated, and the residue purified by flash column chromatography to provide **4**.



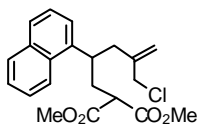
4a *Dimethyl 2-(4-(chloromethyl)-2-phenylpent-4-enyl)malonate*: 2 h, 92% yield, colourless oil; $R_f = 0.53$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.30 (t, $J = 8.4$ Hz, 2H), 7.22 (t, $J = 7.2$ Hz, 1H), 7.14 (d, $J = 7.2$ Hz, 2H), 5.11 (s, 1H), 4.91 (s, 1H), 3.96-3.89 (m, 2H), 3.74 (s, 3H), 3.61 (s, 3H), 3.14 (dd, $J = 10.2, 4.8$ Hz, 1H), 2.78-2.83 (m, 1H), 2.53 (m, 2H), 2.34 (ddd, $J = 13.8, 10.2, 4.2$ Hz, 1H), 2.08 (ddd, $J = 13.8, 10.8, 4.8$ Hz, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 170.0, 169.9, 142.8, 128.9, 127.9, 127.2, 117.2, 52.7, 49.8, 48.2, 41.8, 40.7, 35.0; IR (thin film, cm^{-1}): 2953, 1734, 1700, 1684, 1653, 1540, 1506, 1495, 1436, 1340, 1262, 1151, 1016, 914, 754, 749, 702; HRMS calc'd for $\text{C}_{17}\text{H}_{21}\text{ClO}_4$ 324.1128; found 324.1130.



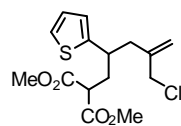
4b *Dimethyl 2-(4-(chloromethyl)-2-(4-bromophenyl)pent-4-enyl)malonate*: 2.5 h, 88% yield, colourless oil; $R_f = 0.43$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.43 (d, $J = 8.4$ Hz, 2H), 7.03 (d, $J = 8.4$ Hz, 2H), 5.11 (s, 1H), 4.89 (s, 1H), 3.97-3.89 (m, 2H), 3.742 (s, 3H), 3.62 (s, 3H), 3.12 (dd, $J = 10.4, 4.8$ Hz, 1H), 2.80 (m, 1H), 2.50 (d, $J = 7.6$ Hz, 2H), 2.35 (ddd, $J = 14.0, 10.4, 4.0$ Hz, 1H), 2.04 (ddd, $J = 14.0, 11.2, 4.8$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.0, 169.9, 148.2, 146.6, 142.7, 136.6, 121.2, 117.2, 108.6, 107.7, 101.2, 52.8, 49.7, 48.2, 41.5, 40.8, 35.2; IR (thin film, cm^{-1}): 2952, 1733, 1653, 1490, 1378, 1333, 1284, 1233, 1214, 1200, 1045, 897, 833, 819, 709, 673; HRMS calc'd for $\text{C}_{17}\text{H}_{20}\text{BrClO}_4$ 402.0233; found 402.0228.



4c *Dimethyl 2-(4-(chloromethyl)-2-(piperon-2-yl)pent-4-enyl)malonate*: 30 min, 74% yield, colourless oil, $R_f = 0.36$ (30% EtOAc/Hexanes), $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 6.73 (d, $J = 7.8$ Hz, 1H), 6.64 (d, $J = 1.8$ Hz, 1H), 6.57, (dd, $J = 7.8, 1.8$ Hz, 1H), 5.94 (s, 2H), 5.11 (s, 1H), 4.91 (s, 1H), 3.93 (m, 2H), 3.75 (s, 3H), 3.65 (s, 3H), 3.18 (dd, $J = 10.2, 4.8$ Hz, 1H), 2.71-2.77 (m, 1H), 2.47 (dd, $J = 7.8, 3.0$ Hz, 2H), 2.32 (ddd, $J = 14.9, 10.8, 3.6$ Hz, 1H), 1.98 (ddd, $J = 14.4, 11.4, 4.8$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 170.0, 169.9, 148.2, 146.6, 142.7, 136.6, 121.2, 117.2, 108.6, 107.7, 101.2, 52.8, 49.7, 48.2, 41.5, 40.8, 35.2; IR (thin film, cm^{-1}): 2954, 2849, 1751, 1734, 1636, 1505, 1488, 1437, 1328, 1246, 1101, 935, 859, 811, 749, 676; HRMS calc'd for $\text{C}_{18}\text{H}_{21}\text{ClO}_6$ 368.1027; found 368.1030.

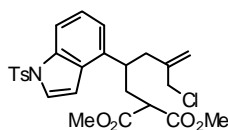


4d *Dimethyl 2-(4-(chloromethyl)-2-(naphthalen-1-yl)pent-4-enyl)malonate*: 2 h, 88% yield, colourless oil; $R_f = 0.44$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, $\text{DMSO}-d_6$, 65 °C): δ 8.09 (d, $J = 8.4$ Hz, 1H), 7.93 (dd, $J = 8.4, 1.2$ Hz, 1H), 7.80 (dd, $J = 6.8, 2.0$ Hz, 1H), 7.58-7.49 (m, 4H), 5.14 (s, 1H), 4.97 (s, 1H), 4.13 (s, 2H), 3.86-3.81 (m, 1H), 3.56 (s, 3H), 3.50 (s, 3H), 3.18 (dd, $J = 9.6, 6.0$ Hz, 1H), 2.62 (d, $J = 6.4, 2\text{H}$), 2.38 (dq, $J = 14, 5.2$ Hz, 1H), 2.31-2.20 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 169.8, 142.7, 139.0, 136.6, 134.0, 132.2, 129.1, 127.2, 126.1, 125.6, 123.1, 122.5, 117.1, 52.5, 52.4, 49.2, 48.0, 40.5, 34.3, 33.7; IR (thin film, cm^{-1}): 3071, 2954, 2921, 2850, 1734, 1636, 1462, 1377, 1264, 1075, 743, 720; HRMS calc'd for $\text{C}_{21}\text{H}_{23}\text{ClO}_4$ 374.1285; found 374.1284.

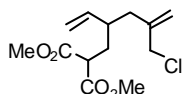


4e *Dimethyl 2-(4-(chloromethyl)-2-(thien-2-yl)pent-4-enyl)malonate*: 30 min, 62% yield, colourless oil; $R_f = 0.44$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.17 (d, $J = 5.4$ Hz, 1H), 6.92 (dd, $J = 5.4, 3.6$ Hz, 1H), 6.79 (d, $J = 2.4$ Hz, 1H), 5.15 (s, 1H), 4.96 (s, 1H), 4.00-3.93 (m, 2H), 3.75 (s, 3H), 3.66 (s, 3H), 3.29 (dd, $J = 10.8, 4.8$ Hz, 1H), 3.21-3.16 (m, 1H), 2.56 (dd, $J = 7.8, 3.6$ Hz, 2H), 2.41 (ddd, $J = 13.8,$

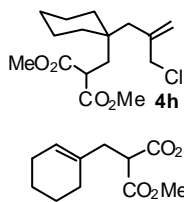
10.8, 4.2 Hz, 1H), 2.02 (dddd, $J = 15.6, 14.4, 11.4, 4.2$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.9, 169.8, 146.7, 142.4, 126.9, 125.2, 124.0, 117.5, 52.8, 49.7, 48.1, 42.1, 37.4, 36.2; IR (thin film, cm^{-1}): 2954, 2927, 1734, 1653, 1436, 1349, 1260, 1206, 1152, 1054, 1031, 918, 847, 701; HRMS calc'd for $\text{C}_{15}\text{H}_{19}\text{ClO}_4\text{S}$ 330.0693; found 330.0690.



4f Dimethyl 2-(4-(chloromethyl)-2-(1-tosyl-1H-indol-4-yl)pent-4-enyl)malonate: 2h, 81% yield, colourless oil; $R_f = 0.48$ (30% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3): δ 7.84 (d, $J = 8.4$ Hz, 1H), 7.76 (d, $J = 8.4$ Hz, 2H), 7.57 (d, $J = 4.0$ Hz, 1H), 7.29-7.22 (m, 3H), 7.05 (d, $J = 7.6$ Hz, 1H), 6.71 (d, $J = 4.0$ Hz, 1H), 5.03 (s, 1H), 4.87 (s, 1H), 3.89 (s, 2H), 3.64 (s, 3H), 3.52 (s, 3H), 3.31-3.23 (m, 1H), 3.10 (q, $J = 5.2$ Hz, 1H), 2.64-2.49 (m, 2H), 2.42-2.35 (m, 1H), 2.35 (s, 3H), 2.20 (ddd, $J = 16, 10.8, 4.8$ Hz, 1H); ^{13}C NMR (125 MHz, CDCl_3): δ 169.7, 169.6, 145.0, 142.4, 135.6, 135.2, 134.8, 129.9, 129.8, 126.4, 126.1, 124.9, 117.0, 111.9, 110.0, 107.7, 106.7, 52.4, 49.4, 47.9, 39.9, 37.8, 33.8, 21.6; IR (thin film, cm^{-1}): 2951, 1751, 1734, 1555, 1473, 1451, 1191, 1147, 1091, 1068, 910, 813, 751, 704, 588; HRMS calc'd for $\text{C}_{26}\text{H}_{28}\text{ClNO}_6\text{S}$ 517.1326; found 517.1320.



4g Dimethyl 2-(4-(chloromethyl)-2-(vinyl)pent-4-enyl)malonate: 16 h, 83% yield, colourless oil; $R_f = 0.50$ (30% EtOAc/hexanes); ^1H NMR (600 MHz, CDCl_3): δ 5.51-5.45 (m, 1H), 5.17 (s, 1H), 5.06 (dd, $J = 10.8, 1.8$ Hz, 1H), 4.99 (dd, $J = 16.8, 1.2$ Hz, 1H), 4.96 (s, 1H), 4.04-3.99 (m, 2H), 3.75 (s, 3H), 3.70 (s, 3H), 3.45 (dd, $J = 9.6, 4.8$ Hz, 1H), 2.25-2.20 (m, 3H), 2.11 (ddd, $J = 13.2, 9.6, 2.4$ Hz, 1H), 1.71 (ddd, $J = 15, 10.2, 4.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3): δ 169.4, 169.3, 137.5, 130.8, 129.5, 117.8, 60.3, 53.0, 52.0, 51.4, 49.1, 37.0, 31.5, 20.8; IR (thin film, cm^{-1}): 3082, 3000, 2955, 2849, 1735, 1642, 1437, 1349, 1273, 1273, 1243, 1210, 1153, 1013, 995, 920, 819, 786, 750, 686; HRMS calc'd for $\text{C}_{13}\text{H}_{19}\text{ClO}_4$ 274.0972; found 274.0971.



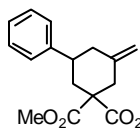
4h Dimethyl 2-(4-(chloromethyl)-2-(spiro-cyclohexyl)pent-4-enyl)malonate: 16 h, accompanied by **6** as an inseparable 2:1 mixture (72%) $R_f = 0.52$ (30% EtOAc/hexanes); ^1H NMR (400 MHz, CDCl_3): δ 5.16 (s, 1H), 5.00 (s, 1H), 4.13-4.00 (m, 2H), 3.76 (s, 3H), 3.66-2.47 (d, $J = 8.8$ Hz, 2H), 2.37-2.31 (m, 1H), 2.21-2.15 (m, 1H), (s, 3H) IR (thin film, cm^{-1}): 2930, 2854, 1756, 1738, 1436, 1340, 1241, 1152, 1026, 917; HRMS calc'd for $\text{C}_{16}\text{H}_{25}\text{ClO}_4$ 316.1441; found 316.1430. Carrying the reaction mixture onwards to the annulation step allowed for the separation of **6** from the reaction mixture. Spectral data for **6** was in accordance with previously reported data.⁸

Experimental procedure, spectral data for annulation products 5a-h

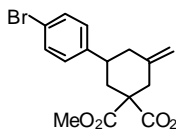
Compounds **5a-h** were prepared by the procedure as follows: a round-bottom flask was charged with NaH (1.1 equivalents, 60% oil dispersion). DMF was added via cannula and the flask cooled to 0 °C. Allyl chloride **4** was then added via cannula as a solution in DMF, and the flask purged with argon. The reaction was allowed to warm to room temperature and stirred overnight. Addition of allyl chloride was accompanied by a change from colourless to a yellow solution. On consumption of starting material as indicated by TLC, the flask was cooled once again to 0 °C and the

⁸ Trost, B. M.; Verhoeven, T. R. *J. Am. Chem. Soc.* **1978**, *100*, 3435.

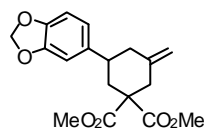
reaction slowly quenched dropwise with water. The mixture was extracted 5x with EtOAc, and the combined organics washed with a 5% aqueous solution of LiCl and water. The organic extracts were dried over MgSO₄, filtered, concentrated, and the residue purified by flash column chromatography on silica (eluent: EtOAc/hexanes) to provide **5**.



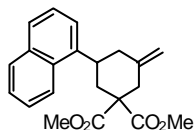
5a *Dimethyl 3-methylene-5-phenylcyclohexane-1,1-dicarboxylate*: 16 h, 95% yield, colourless oil; $R_f = 0.58$ (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 7.31 (t, $J = 7.8$ Hz, 2H), 7.24 (d, $J = 7.2$ Hz, 2H), 7.22 (t, $J = 7.2$ Hz, 1H), 4.85 (m, 1H), 4.83 (m, 1H), 3.77 (s, 3H), 3.72 (s, 3H), 3.01 (dt, $J = 13.8$, 1.8 Hz, 1H), 2.89 (tt, $J = 12.0$, 4.2 Hz, 1H), 2.60 (d, $J = 13.2$ Hz, 1H), 2.54 (dd, $J = 13.8$, 1.2 Hz, 1H), 2.47 (dt, $J = 12.6$, 2.4 Hz, 1H), 2.21 (t, $J = 12.6$ Hz, 1H), 1.89 (t, $J = 12.6$ Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 172.0, 170.7, 144.9, 143.3, 128.5, 126.8, 126.5, 111.6, 56.8, 52.8, 52.5, 41.6, 41.3, 39.2, 38.4; IR (thin film, cm⁻¹): 3064, 3030, 2851, 1734, 1604, 1603, 1495, 1454, 1435, 1313, 1289, 1254, 1234, 1197, 1166, 1071, 1045, 1008, 958, 900, 871, 766, 700; HRMS calculated for C₁₇H₂₀O₄ 288.1362; found 288.1360.



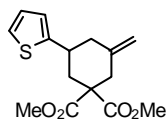
5b *Dimethyl 3-methylene-5-(4-bromophenyl)cyclohexane-1,1-dicarboxylate*: 16 h, 95% yield, white solid; m.p. 85-87 °C; R_f 0.63 (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 7.42 (d, $J = 8.4$ Hz, 2H), 7.11 (d, $J = 8.4$ Hz, 2H), 4.85 (d, $J = 1.8$ Hz, 1H), 4.83 (d, $J = 1.8$ Hz, 1H), 3.76 (s, 3H), 3.72 (s, 3H), 2.99 (d, $J = 15.6$ Hz, 1H), 2.86 (tt, $J = 12.6$, 3.6 Hz, 1H), 2.57-2.52 (m, 2H), 2.45-2.42 (m, 1H), 2.16 (t, $J = 12.6$ Hz, 1H), 1.83 ($J = 13.2$, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 172.1, 170.9, 144.1, 143.2, 131.8, 128.8, 120.5, 112.1, 56.9, 53.1, 52.8, 41.4, 41.3, 39.4, 38.5; IR (thin film, cm⁻¹): 2952, 1733, 1653, 1490, 1436, 1371, 1333, 1284, 1233, 1214, 1196, 1045, 897, 872, 835, 819, 709, 673; HRMS calc'd for C₁₇H₁₉BrO₄ 366.0467; found 366.0458.



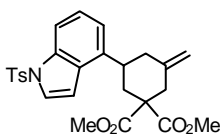
5c *Dimethyl 3-methylene-5-(piperon-2-yl)cyclohexane-1,1-dicarboxylate*: 15 h, 97% yield, white solid; m.p. 117-120 °C; $R_f = 0.51$ (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 6.72 (d, $J = 8.4$ Hz, 1H), 6.63 (d, $J = 1.8$ Hz, 1H), 6.56 (dd, $J = 7.8$, 1.8 Hz, 1H), 5.93 (s, 2H), 5.10 (s, 1H), 4.91 (s, 1H), 3.96-3.89 (m, 2H), 3.74 (s, 3H), 3.64 (s, 3H), 3.17 (dd, $J = 10.8$, 4.2 Hz, 1H), 2.76-2.71 (m, 1H), 2.50-2.44 (m, 1H), 2.31 (ddd, $J = 15$, 11.4, 3 Hz, 1H), 1.97 (ddd, $J = 16.2$, 11.4, 4.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 172.2, 171.0, 147.9, 146.3, 143.5, 139.3, 119.9, 111.8, 108.5, 107.5, 101.0, 57.0, 53.1, 52.7, 41.8, 41.6, 39.4, 38.9; IR (thin film, cm⁻¹): 2955, 2926, 2854, 1733, 1655, 1505, 1488, 1439, 1313, 1245, 1210, 1127, 1090, 1040, 935, 902, 871, 805, 700, 657; HRMS calc'd for C₁₈H₂₀O₆ 332.1260, found 332.1258.



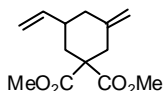
5d *Dimethyl 3-methylene-5-(naphthalen-1-yl)cyclohexane-1,1-dicarboxylate*: 16 h, 91% yield, cream-coloured solid; m.p. 90-93 °C; $R_f = 0.54$ (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 8.37 (d, $J = 8.4$ Hz, 1H), 7.86 (d, $J = 8.4$ Hz, 1H), 7.74 (d, $J = 7.8$ Hz, 1H), 7.58 (t, $J = 8.4$ Hz, 1H), 7.49 (t, $J = 7.2$ Hz, 1H), 7.41-7.46 (m, 2H), 4.91 (d, $J = 1.2$ Hz, 1H), 4.89 (d, $J = 1.2$ Hz, 1H), 3.86 (s, 3H), 3.78 (tt, $J = 12.0$, 3.6 Hz, 1H), 3.69 (s, 3H), 3.09 (dt, $J = 7.8$, 1.2 Hz, 1H), 2.82 (d, $J = 13.2$ Hz, 1H), 2.68 (d, $J = 13.8$ Hz, 1H), 2.58 (dt, $J = 13.2$, 1.8 Hz, 1H), 2.51 (t, $J = 13.2$ Hz, 1H), 1.85 (dd, $J = 13.2$, 1.2 Hz, 1H); ¹³C NMR (125 MHz, CDCl₃): δ 171.9, 171.2, 143.7, 140.9, 133.9, 131.5, 128.8, 127.1, 126.2, 125.4, 123.4, 122.0, 111.6, 110.0, 57.1, 52.8, 40.1, 39.6, 38.8, 36.5, 31.4; IR (thin film, cm⁻¹): 3074, 3050, 2953, 2848, 1732, 1655, 1598, 1512, 1434, 1399, 1291, 1262, 1207, 1109, 1044, 999, 902, 872, 798, 779, 701, 651, 640; HRMS calc'd for C₂₁H₂₂O₄: 338.1518; found 338.1513.



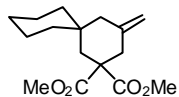
5e Dimethyl 3-methylene-5-(thiophen-2-yl)cyclohexane-1,1-dicarboxylate: 16 h, 90% yield, colourless oil; $R_f = 0.49$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 7.15 (dd, $J = 4.8, 1.2$ Hz, 1H), 6.94 (dd, $J = 4.8, 3.0$ Hz, 1H), 6.81 (d, $J = 3.6$ Hz, 1H), 4.85 (m, 2H), 3.75 (s, 3H), 3.73 (s, 3H), 3.22 (tt, $J = 13.2, 3.0$ Hz, 1H), 2.98 (dt, $J = 13.8, 1.8$ Hz, 1H), 2.75 (dt, $J = 13.2, 1.8$ Hz, 1H), 2.63 (dt, $J = 12.0, 2.4$ Hz, 1H), 2.53 (dd, $J = 13.2, 1.8$ Hz, 1H), 2.20 (t, $J = 12$ Hz, 1H), 1.88 (t, $J = 13.8$ Hz, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 172.0, 170.9, 149.3, 142.8, 126.8, 123.1, 122.9, 112.3, 56.8, 53.1, 52.8, 42.8, 39.5, 39.4, 37.2; IR (thin film, cm^{-1}): 3056, 2954, 2923, 2850, 1734, 1653, 1475, 1435, 1377, 1312, 1292, 1265, 1208, 1169, 1090, 902, 739, 702, 613; HRMS calc'd for $\text{C}_{15}\text{H}_{18}\text{O}_4\text{S}$: 294.0926 found 294.0920.



5f Dimethyl 3-methylene-5-(1-tosyl-1H-indol-4-yl)cyclohexane-1,1-dicarboxylate: 16h, 91% yield, colourless oil; $R_f = 0.47$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 7.85 (d, $J = 8.4$ Hz, 1H), 7.77 (d, $J = 8.4$ Hz, 2H), 7.58 (d, $J = 4.0$ Hz, 1H), 7.26 (t, $J = 8.8$ Hz, 1H), 7.22 (d, $J = 8.8$ Hz, 2H), 7.08 (d, $J = 7.6$ Hz, 1H), 6.94 (d, $J = 4.0$ Hz, 1H), 4.85 (s, 1H), 4.82 (s, 1H), 3.80 (s, 3H), 3.67 (s, 3H), 3.27 (tt, $J = 12.8, 4.0$ Hz, 1H), 3.02 (d, $J = 13.6$ Hz, 1H), 2.65 (d, $J = 13.2$ Hz, 1H), 2.56 (d, $J = 13.2$ Hz, 1H), 2.48 (d, $J = 10.8$ Hz, 1H), 2.40 (d, $J = 12.8$ Hz, 1H), 2.35 (s, 3H), 1.76 (t, $J = 13.2$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 171.7, 170.9, 144.8, 143.3, 137.7, 135.3, 134.7, 129.8, 129.7, 126.8, 125.9, 124.7, 118.9, 111.8, 111.7, 107.2, 56.9, 52.8, 52.5, 39.30, 39.26, 38.5, 38.2, 21.5; IR (thin film, cm^{-1}): 2954, 2927, 1733, 1656, 1598, 1494, 1434, 1425, 1373, 1288, 1209, 1188, 1132, 1090, 1043, 1017, 962, 902, 871, 762, 704, 679, 646, 579; HRMS calc'd for $\text{C}_{26}\text{H}_{27}\text{NO}_6\text{S}$: 481.1559; found 481.1554.

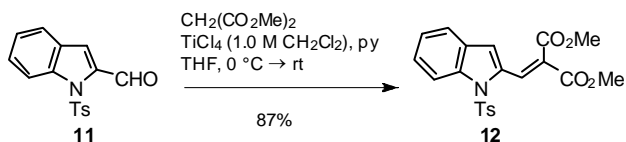
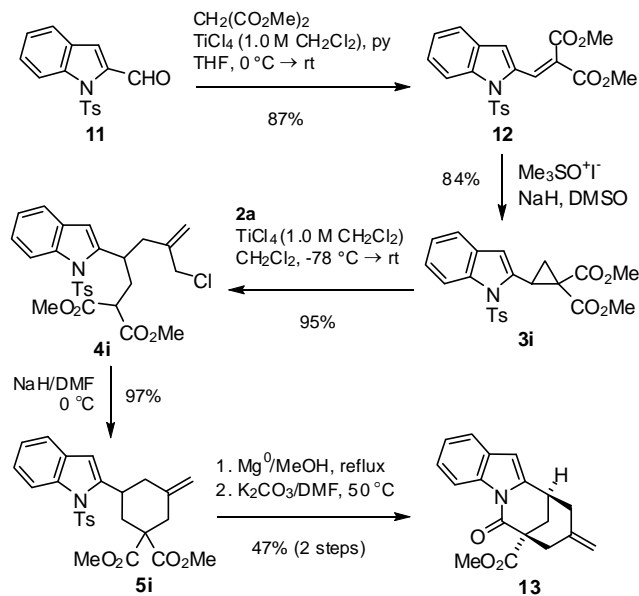


5g Dimethyl 3-methylene-5-(vinyl)cyclohexane-1,1-dicarboxylate: 16h, 79% yield; colourless oil; $R_f = 0.52$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (400 MHz, CDCl_3): δ 5.74 (ddd, $J = 17.2, 9.2, 9.0$ Hz, 1H), 5.05 (dt, $J = 17.2, 1.6$ Hz, 1H), 4.98 (dt, $J = 10.8, 1.2$ Hz, 1H), 4.79 (t, $J = 1.6$ Hz, 2H), 3.72 (s, 6H), 2.93 (dt, $J = 13.6, 1.6$ Hz, 1H), 2.48-2.39 (m, 2H), 2.35-2.31 (m, 2H), 1.83 (t, $J = 14$ Hz, 1H), 1.54 (t, $J = 12.8$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 169.9, 169.7, 142.4, 140.2, 116.7, 52.5, 49.5, 47.9, 40.1, 38.9, 33.0; IR (thin film, cm^{-1}): 3079, 2954, 1735, 1636, 1507, 1472, 1291, 1252, 1169, 1138, 1093, 913, 820, 558; HRMS calc'd for $\text{C}_{13}\text{H}_{18}\text{O}_4$: 238.1205; found 238.1210.



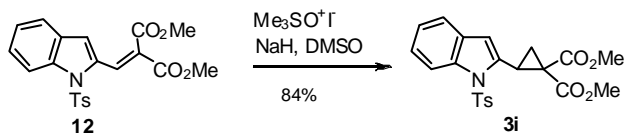
5h Dimethyl 3-methylene-5-(spiro-cyclohexyl)cyclohexane-1,1-dicarboxylate: 16 h, 75% yield, colourless oil; $R_f = 0.56$ (30% EtOAc/hexanes); $^1\text{H NMR}$ (600 MHz, CDCl_3): δ 4.86 (s, 1H), 4.85 (s, 1H), 3.73 (s, 3H), 3.72 (s, 3H), 3.17 (d, $J = 16.8$ Hz, 1H), 2.61 (dq, $J = 16.8, 2.4$ Hz, 1H), 2.59-2.54 (m, 2H), 2.24-2.18 (m, 1H), 1.76 (d, $J = 13.2$ Hz, 1H), 1.69 (d, $J = 13.2$ Hz, 2H), 1.63-1.57 (m, 2H), 1.40-1.32 (m, 1H), 1.22-1.10 (m, 3H), 1.03 (dq, $J = 12.6, 3.0$ Hz, 1H), 0.95 (dq, $J = 12.0, 3.0$ Hz, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3): δ 172.9, 171.3, 147.5, 106.5, 62.3, 52.5, 52.1, 51.7, 43.1, 39.8, 35.3, 32.7, 31.5, 30.8, 26.8, 26.7; IR (thin film, cm^{-1}): 3074, 3050, 2953, 2848, 1732, 1653, 1598, 1512, 1454, 1434, 1399, 1291, 1261, 1207, 1169, 1109, 1044, 1018, 999, 943, 902, 873, 853, 797, 736, 701, 650; HRMS calc'd for $\text{C}_{16}\text{H}_{24}\text{O}_4$: 280.1675; found 280.1680.

Synthesis of a Pyrido[1,2-*a*]Indole Model Core (Scheme 3):



12 Dimethyl 2-(1-tosyl-1H-indol-2-ylmethylene)-malonate was prepared in the same manner as **15**. *N*-Tosyl indole-2-carboxaldehyde **11**⁹ (1.0 g, 3.34 mmol) was reacted with dimethylmalonate (0.44 g, 3.34 mmol), TiCl₄ (1.0 M solution

in CH₂Cl₂, 6.7 mmol, 6.7 mL) and pyridine (1.1 mL, 13.4 mmol) in THF (30 mL) to give malonate **12** in 87% yield (1.2 g, 2.9 mmol) as a pale yellow solid; m.p.114-116 °C; R_f = 0.45 (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 8.36 (s, 1H), 8.14 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 1H), 7.37 (t, *J* = 9 Hz, 1H), 7.24 (t, *J* = 7.2 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.91 (s, 1H), 3.91 (s, 3H), 3.74 (s, 3H), 2.32 (s, 3H); ¹³C NMR (100 MHz, CDCl₃): δ 166.7, 164.0, 145.4, 137.9, 134.7, 133.8, 133.0, 129.9, 129.7, 127.3, 126.9, 124.6, 122.2, 115.7, 115.3, 53.1, 53.0, 21.8; IR (thin film, cm⁻¹): 2955, 1734, 1718, 1576, 1541, 1399, 1369, 1231, 1207, 1161, 1071, 1050, 1022, 981, 912, 845, 813, 755, 706, 668; HRMS calculated for C₂₁H₁₉NO₆S: 413.0933; found 413.0933.

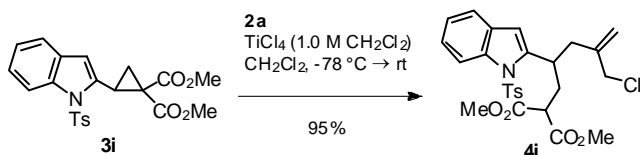


3i Dimethyl 2-(1-tosyl-1H-indol-2-yl)cyclopropane-1,1-dicarboxylate was prepared in the same manner as **3f**. Malonate **12** (0.29 g, 0.69 mmol) was reacted with sodium

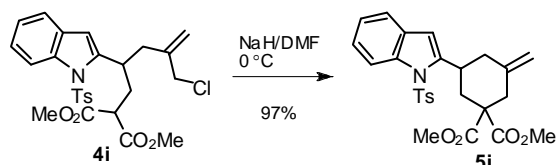
hydride (60% oil dispersion, 30 mg, 0.76 mmol), trimethylsulfoxonium iodide (0.168 g, 0.76 mmol) to provide **3i** in 84% yield (0.25 g, 0.58 mmol) as a cream-coloured solid; m.p.104-108 °C; R_f = 0.45 (30% EtOAc/hexanes); ¹H NMR (400 MHz, CDCl₃): δ 8.10 (d, *J* = 8.4 Hz, 1H), 7.74 (d, *J* = 8.4 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 1H), 7.30-7.28 (m, 1H), 7.22-7.17 (m, 3H), 6.35 (s, 1H), 3.85 (s, 3H), 3.64 (t, *J* = 8.8 Hz, 1H), 3.36 (s, 3H), 2.35 (s, 3H), 2.12 (dd, *J* = 8.0, 5.2 Hz, 1H), 1.90 (dd, *J* = 9.2, 5.2 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 169.8, 167.1, 145.0, 137.5, 136.4, 135.6, 130.0, 128.7, 126.9, 124.9, 123.6, 121.1, 114.5, 109.7, 53.2, 52.8, 38.1, 26.7, 21.8, 20.4; IR (thin film, cm⁻¹): 2953, 2926, 1734, 1594, 1451,

⁹ Riu, A.; Harrison-Marchand, A.; Maddaluno, J.; Gulea, M.; Albadri, H.; Masson, S. *Eur. J. Org. Chem.* **2007**, 29, 4948.

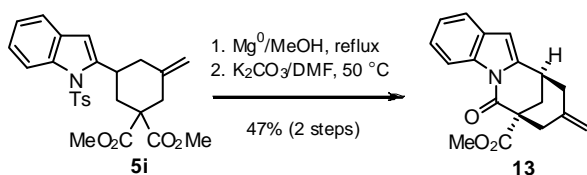
1437, 1363, 1303, 1272, 1221, 1152, 1090, 988, 970, 898, 834, 748, 704, 667, 624, 576; HRMS calculated for C₂₂H₂₁NO₆S: 427.1090; found: 427.1097.



4i Dimethyl 2-(4-(chloromethyl)-2-(1-tosyl-1H-indol-4-yl)pent-4-enyl)malonate was prepared according to the allylation procedure described above: cyclopropane **3i** (152 mg, 0.356 mmol) was reacted with 2-(chloromethyl)-3-trimethylsilyl-1-propene **2a** (77 μL, 0.427 mmol) and TiCl₄ (1.0 M solution, 0.356 mmol, 0.36 mL) in CH₂Cl₂ (3 mL) at -78 °C for 3 h, then warmed to rt overnight, to give allyl chloride **4i** after workup of reaction in 95% yield (175 mg, 0.338 mmol) as a clear colourless oil; R_f = 0.55 (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 8.18 (d, *J* = 8.4 Hz, 1H), 7.59 (d, *J* = 8.4 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.29 (t, *J* = 9.0 Hz, 1H), 7.23 (t, *J* = 7.8 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.53 (s, 1H), 5.26 (s, 1H), 5.09 (s, 1H), 4.07-4.02 (m, 2H), 3.97-3.94 (m, 1H), 3.72 (s, 3H), 3.66 (s, 3H), 3.43 (dd, *J* = 9.6, 4.2 Hz, 1H), 2.65 (dd, *J* = 14.4, 4.2 Hz, 1H), 2.54 (ddd, *J* = 15, 10.2, 4.8 Hz, 1H), 2.36-2.31 (m, 1H), 2.32 (s, 3H), 2.11 (ddd, *J* = 13.8, 9.6, 3.6 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 170.1, 169.7, 145.1, 144.4, 142.3, 137.5, 136.1, 130.1, 129.7, 126.6, 124.7, 124.0, 120.7, 117.8, 115.5, 109.2, 52.9 (x2 C), 49.6, 48.0, 40.9, 33.8, 33.5, 21.8; IR (thin film, cm⁻¹): 2954, 1751, 1734, 1559, 1473, 1451, 1191, 1147, 1091, 1068, 910, 813, 751, 704, 579; HRMS calculated for C₂₆H₂₈ClNO₆S: 517.1326; found: 517.1325.



5i Dimethyl 3-methylene-5-(1-tosyl-1H-indol-4-yl) cyclohexane-1,1-dicarboxylate was prepared according to the annulation procedure described above: **4i** (175 mg, 0.356 mmol) was treated with NaH (60% oil dispersion, 15 mg, 0.375 mmol) in DMF (2 mL) at 0 °C, and the reaction mixture stirred 16 h. After work-up and purification by column chromatography (eluent: 15% EtOAc/hexanes), cyclohexene **5i** was isolated in 97% yield (166 mg, 0.345 mmol) as a clear colourless oil; R_f = 0.62 (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 8.22 (d, *J* = 7.8 Hz, 1H), 7.63 (d, *J* = 9 Hz, 2H), 7.39 (d, *J* = 7.2 Hz, 1H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 2H), 6.47 (s, 1H), 4.91 (s, 2H), 3.82 (s, 3H), 3.77 (tt, *J* = 12, 3 Hz, 1H), 3.74 (s, 3H), 3.04 (d, *J* = 13.2 Hz, 1H), 2.79-2.75 (m, 1H), 2.52 (d, *J* = 13.8, 1H), 2.31 (s, 3H), 2.00 (t, *J* = 13.2 Hz, 1H), 1.99 (t, *J* = 12 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 172.2, 170.9, 145.7, 144.9, 142.4, 137.4, 135.9, 130.1, 130.0, 126.6, 124.5, 123.9, 120.5, 115.7, 112.5, 108.3, 56.6, 53.1, 52.8, 42.6, 39.6, 37.2, 34.7, 21.7; IR (thin film, cm⁻¹): 2954, 1734, 1653, 1369, 1291, 1198, 1174, 1147, 1089, 1040, 916, 751, 655, 640, 579; HRMS calculated for C₂₆H₂₇NO₆S: 481.1559; found: 481.1558.

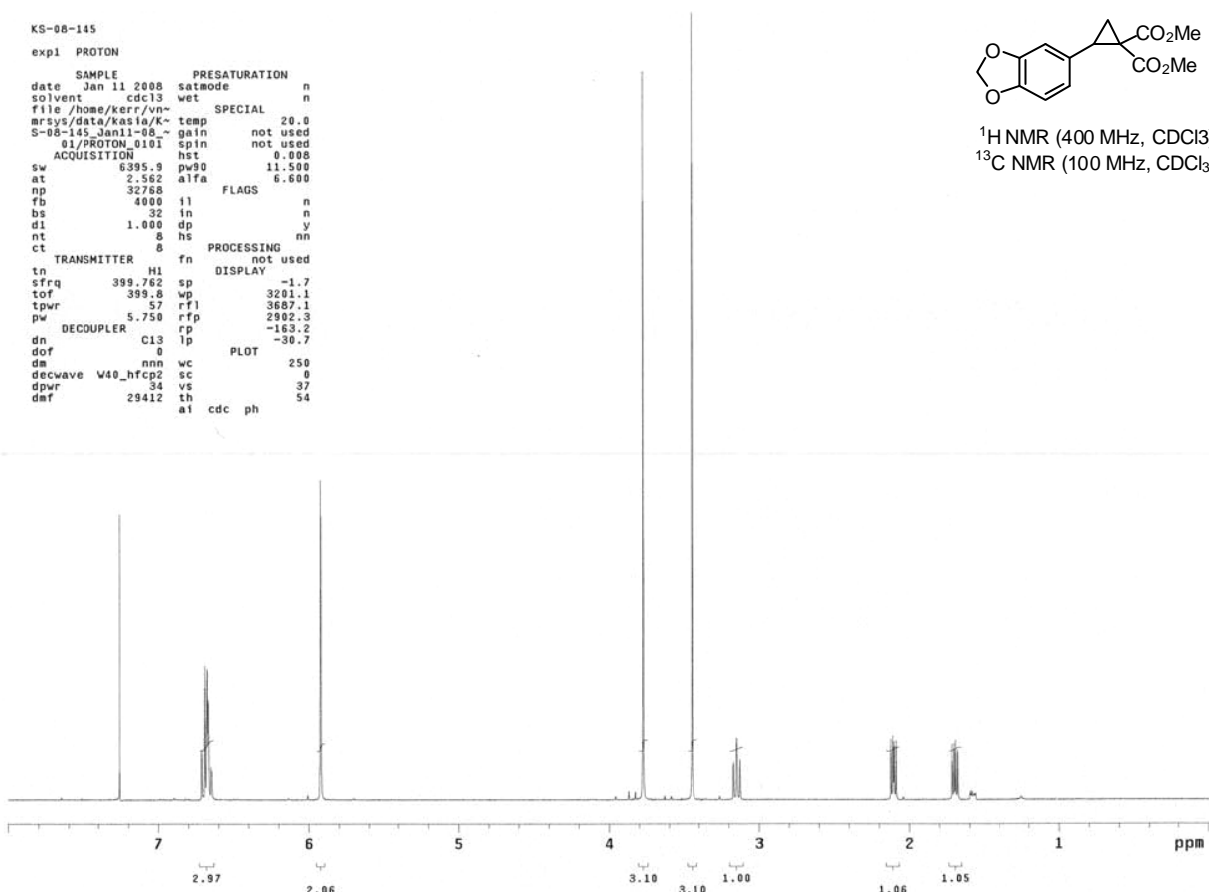
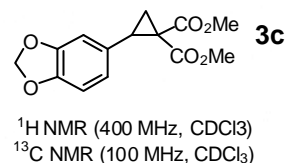


13: Indole **5i** (166 mg, 0.34 mmol) was dissolved in dry MeOH (3 mL). Magnesium turnings (85 mg, 3.5 mmol) were added and the mixture was brought to reflux over 16 h. The reaction was quenched slowly with a saturated ammonium chloride solution and extracted with methylene chloride. The organic extracts were dried over MgSO₄ and concentrated; the subsequent residue was dissolved in DMF (2 mL) and the solution cooled to 0 °C. K₂CO₃ (46 mg, 0.35 mmol) was added portionwise and the solution was then warmed to 50 °C and stirred for 16 h. The reaction was slowly quenched with water, extracted with methylene chloride, and the combined organics washed with water and sat.d. LiCl. Compound **13** was isolated in 47% over the two steps (47 mg, 0.16 mmol) as a white solid; decomp. 70 °C; R_f = 0.55 (30% EtOAc/hexanes); ¹H NMR (600 MHz, CDCl₃): δ 8.38 (d, *J* = 8.4 Hz, 1H), 7.47 (d, *J* = 6.6 Hz, 1H), 7.29-7.24 (m, 2H), 6.34 (s, 1H), 4.72 (d, *J* = 1.8 Hz, 1H), 4.57 (d, *J* = 1.8 Hz, 1H), 3.82 (s, 3H), 3.59-3.57 (m, 1H), 3.00 (dt, *J* = 13.8, 1.8 Hz, 1H), 2.83 (m, 1H), 2.73 (dq, *J* = 13.8, 1.8 Hz, 1H), 2.55 (m, 1H), 2.44-2.41 (m, 1H), 2.12 (dd, *J* = 12.6, 2.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃): δ 171.7, 168.7, 141.1, 139.8, 134.9, 130.2, 124.6, 124.5, 120.3, 116.8, 114.8, 104.8, 55.0, 53.1, 41.6, 40.9, 35.1, 31.4; IR (thin film, cm⁻¹): 2952, 1735, 1700, 1599, 1473, 1456, 1379, 1321, 1303, 1182, 1046, 1035, 894, 810, 757, 652, 621; HRMS calculated for C₁₈H₁₇NO₃: 295.1208; found: 295.1209.

```

KS-08-145
expl  PROTON
      SAMPLE      PRESATURATION
date  Jan 11 2000 satmode      n
solvent  cdcl3      wet
file /home/kerr/vn- SPECIAL
mrsys/data/kasia/K- temp      20.0
S-08-145 Jan11-08- gain      not used
01/PROTON_0101 spin      not used
ACQUISITION hst      0.008
sw      6395.9      pw90      11.500
at      2.582      alfa      6.600
np      32768      FLAGS
fb      4000      f1      n
bs      32      in      n
d1      1.000      dp      y
nt      8      hs      nn
ct      8      PROCESSING
      TRANSMITTER      fn      not used
tn      H1      DISPLAY
sfrq    399.762      sp      -1.7
tof     399.8      wp      3201.1
tpwr    57      rfl      3887.1
pw      5.750      rfp      2902.3
      DECOUPLER      rp      -163.2
dn      C13      lp      -30.7
dof     0      PLOT
dm      nnn      wc      250
decwave W40_hfcp2 sc      0
dpr     34      vs      37
dmf     29412      th      54
      ai      cdc ph

```



```

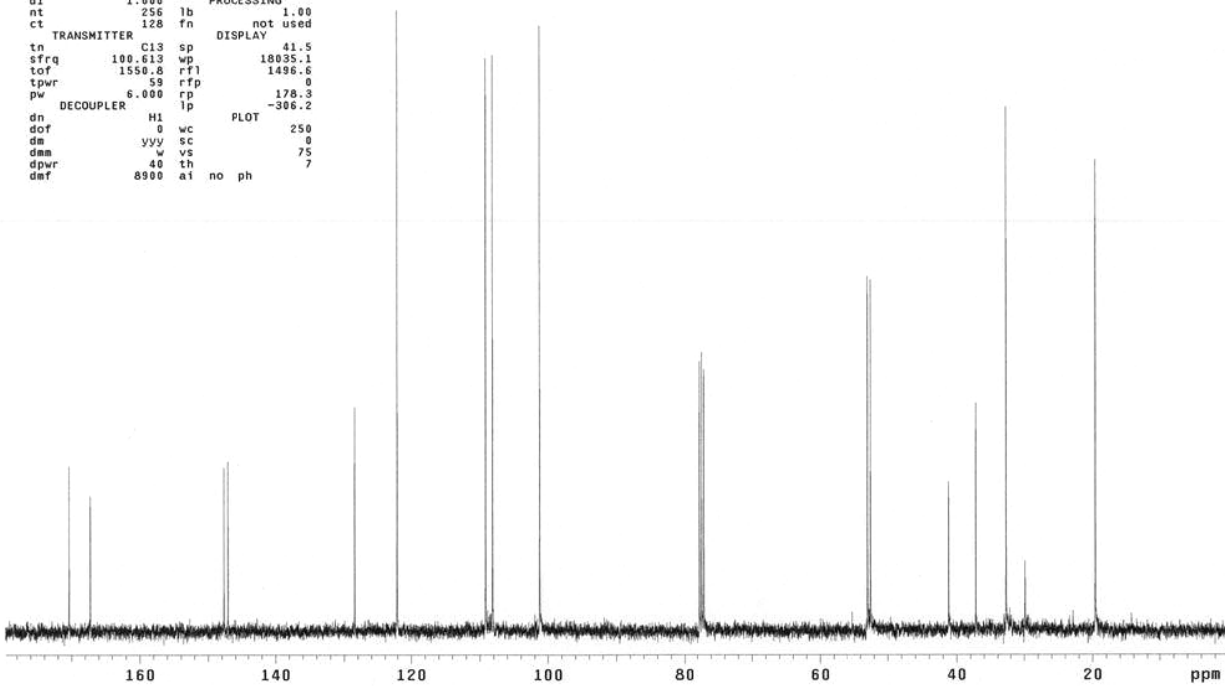
KS-08-145
STANDARD 1H OBSERVE

```

```

exp2  s2pu1
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date  Dec 19 2007 temp      not used
solvent  cdcl3      gain      not used
file /export/home/~ spin      not used
vnari/walkup/auto- hst      0.008
19.12.07/0505      pw90      12.000
ACQUISITION alfa      20.000
sw      25125.6      FLAGS
at      1.199      f1      n
np      60270      in      n
fb      13800      dp      y
bs      64      hs      nn
d1      1.000      PROCESSING
nt      256      lb      1.00
ct      128      fn      not used
      TRANSMITTER      DISPLAY
tn      C13      sp      41.5
sfrq    100.613      wp      18035.1
tof     1550.6      rfl      1496.6
tpwr    59      rfp      0
pw      6.000      rp      178.3
      DECOUPLER      lp      -306.2
dn      H1      PLOT
dof     0      wc      250
dm      yyy      sc      0
dmm     w      vs      75
dpr     40      th      7
dmf     8900      ai      no ph

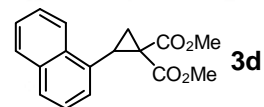
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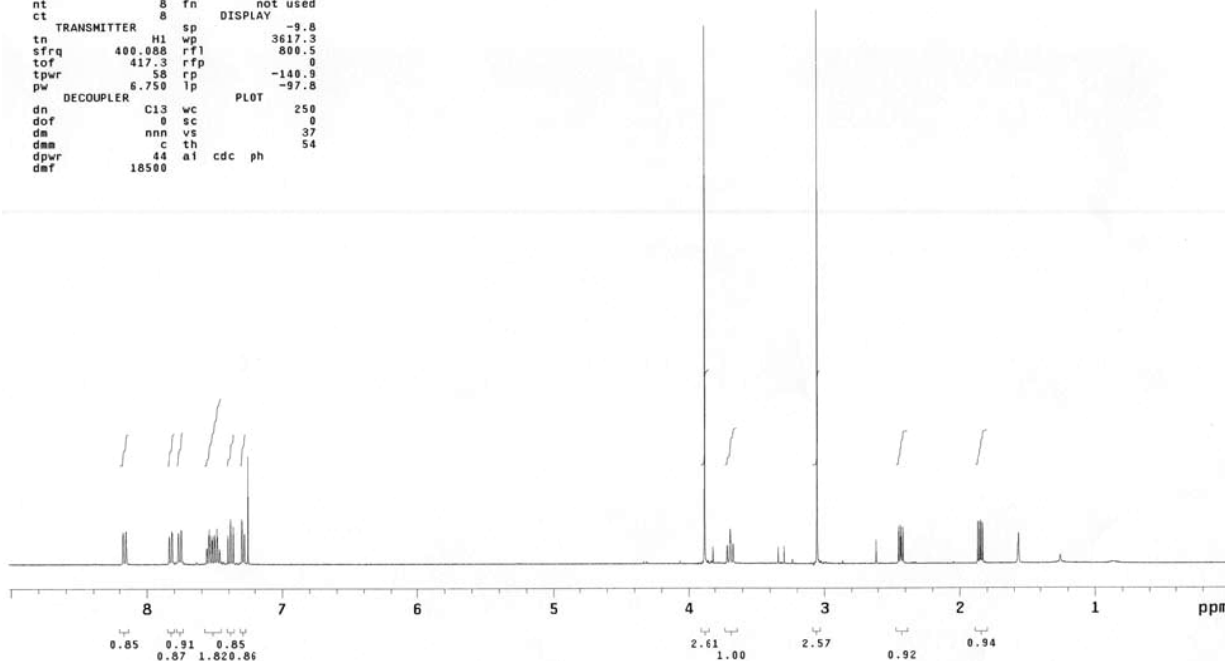
KS-08-143
STANDARD 1H OBSERVE

exp3 s2pu1

```
SAMPLE          SPECIAL
date   May 9 2008 temp      not used
solvent cdc13 gain      not used
file   /export/home/~ spin      28
vnmr1/walkup/auto_~ hst      0.008
09.05.08/0123 pw90      13.500
ACQUISITION     alfa      20.000
sw       6402.0         FLAGS
at       4.000         i1      n
np       51216        in      n
fb       not used     dp      y
bs       16          hs      nn
d1       1.000        h1      nn
nt       8           fn      not used
ct       8           DISPLAY
TRANSMITTER     sp      -9.8
tn          H1      wp      3617.3
sfrq       400.088  rfl     800.5
tof        417.3    rfp     0
tpwr       58      rp     -140.9
pw         6.750    lp     -97.8
DECOUPLER      C13    wc      250
dof        0       sc      0
dm         nnn     vs      37
dmm        C      th      54
dpwr       44     ai      cdc ph
dmf        18500
```



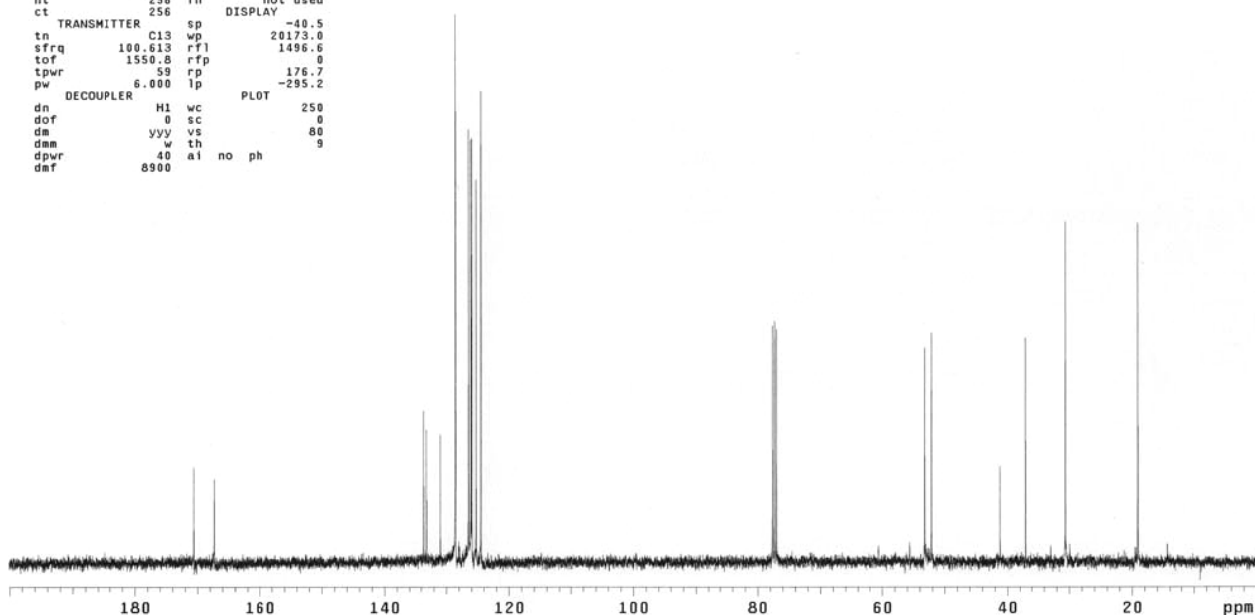
¹H NMR (400 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-08-143
STANDARD 1H OBSERVE

exp2 s2pu1

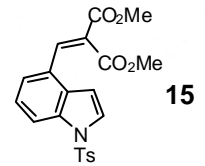
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SAMPLE          SPECIAL
date   Dec 19 2007 temp      not used
solvent cdc13 gain      not used
file   /export/home/~ spin      not used
vnmr1/vnmr/sys/data~ hst      0.008
/Kerr/KS-08-143/CA~ pw90      12.000
RBON.fid alfa      20.000
ACQUISITION     FLAGS
sw       25125.6      i1      n
at       1.199       in      n
np       60270       dp      y
fb       13800      hs      nn
bs       84         hs      nn
d1       1.000       lb      1.00
nt       256        fn      not used
ct       256        DISPLAY
TRANSMITTER     sp      -40.5
tn          C13     wp      20173.0
sfrq       100.613  rfl     1496.6
tof        1550.8   rfp     0
tpwr       58      rp     176.7
pw         6.000    lp     -295.2
DECOUPLER      H1    wc      250
dof        0       sc      0
dm         yyy     vs      80
dmm        w      th      9
dpwr       40     ai      no ph
dmf        8900
```



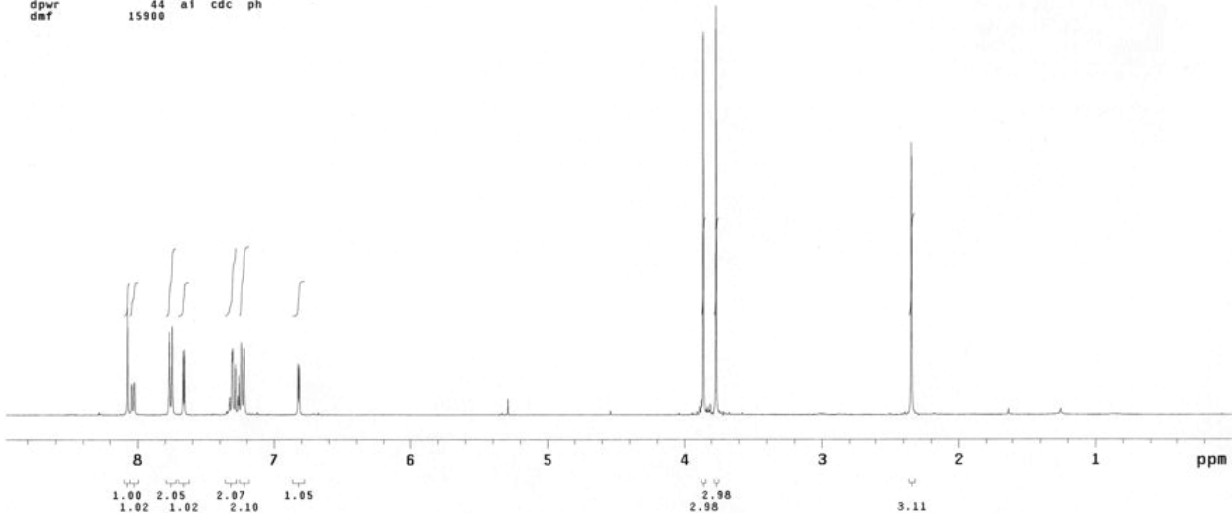
KS-09-033-2
STANDARD 1H OBSERVE

exp10 s2pul

```
SAMPLE SPECIAL
date Feb 8 2008 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin 20
vnmr1/walrup/auto_~ hst 0.008
08.02.08/0402 pw90 14.800
ACQUISITION alfa 20.000
sw 6402.0 FLAGS
at 4.000 f1 n
np 51216 in n
fb not used dp y
bs 16 hs nn
d1 1.000 fn PROCESSING
nt 8 fn not used
ct 8 DISPLAY
TRANSMITTER sp 0.1
tn H1 wp 3583.7
sfrq 400.988 rf1 3703.6
tof 417.3 rfp 2884.6
tpwr 57 rp -144.2
pw 7.400 lp -97.8
DECOUPLER C13 wc PLOT 250
dn 0 sc 0
dm nnn vs 37
dmm c th 5
dpwr 44 a1 cdc ph
daf 15900
```



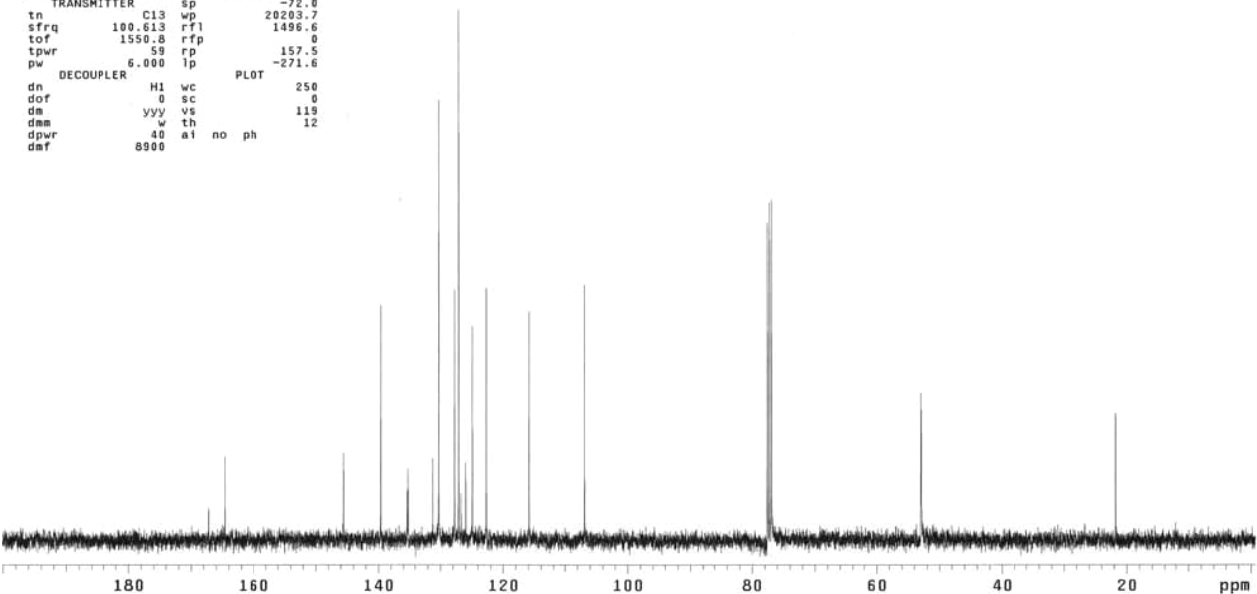
¹H NMR (400 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-09-033-2
STANDARD 13C OBSERVE

exp2 s2pul

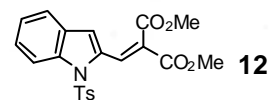
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SAMPLE SPECIAL
date Feb 8 2008 temp not used
solvent cdcl3 gain not used
file /export/home/~ spin not used
vnmr1/vnmrsvs/data_~ hst 0.008
/Kerr/KS-09-033-2/~ pw90 12.000
CARBON.f1d alfa 20.000
ACQUISITION FLAGS
sw 25125.6 f1 n
at 1.193 in n
np 80270 dp y
fb 13800 hs PROCESSING
bs 64 hs nn
d1 1.000 lb 1.00
nt 256 fn not used
ct 256 DISPLAY
TRANSMITTER sp -72.0
tn C13 wp 20203.7
sfrq 100.613 rf1 1496.6
tof 1550.8 rfp 0
tpwr 59 rp 157.5
pw 6.000 lp -271.6
DECOUPLER H1 wc PLOT 250
dn 0 sc 0
dm yyy vs 119
dmm w th 12
dpwr 40 a1 no ph
daf 8900
```



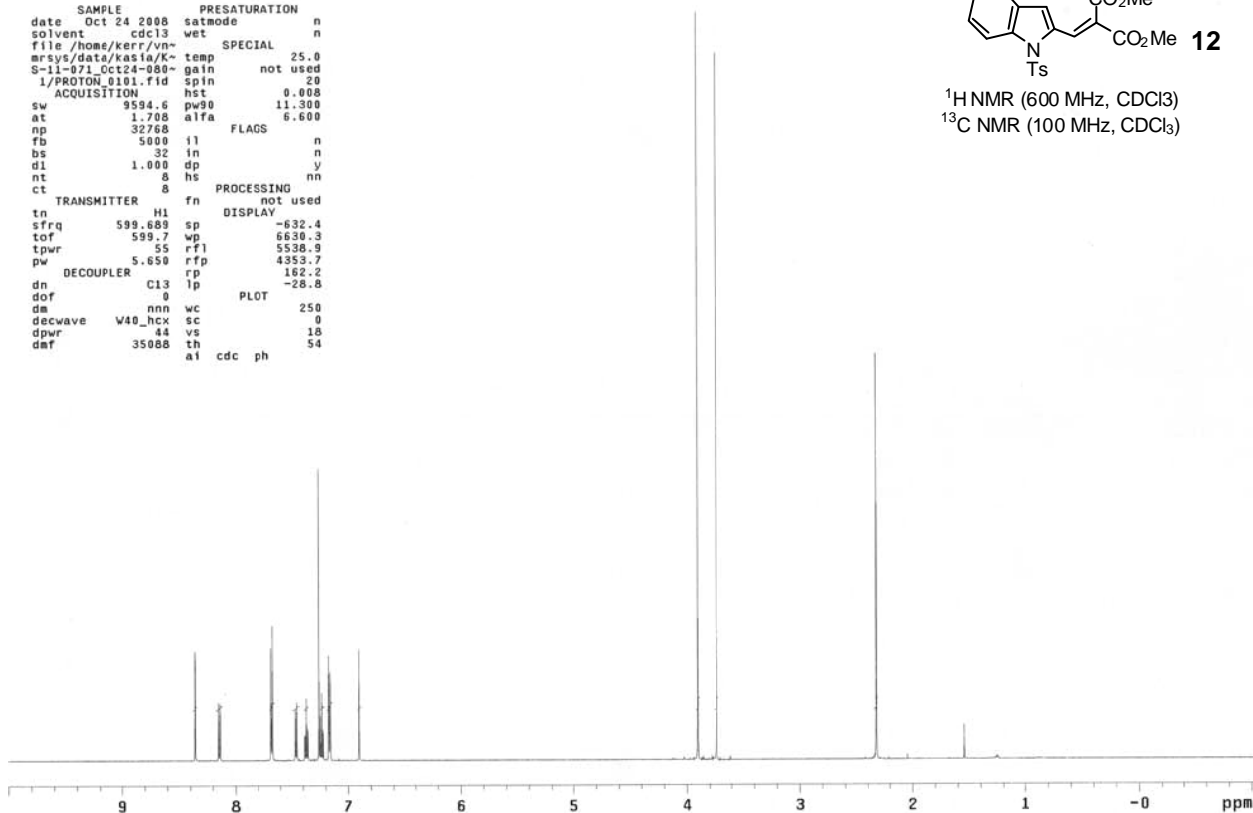
KS-11-071

expl PROTON

```
SAMPLE          PRESATURATION
date Oct 24 2008 satmode      n
solvent cdcl3    wet         n
file /home/kerr/vn- SPECIAL
arsys/data/kas/k~ temp      25.0
S-11-071_Oct24-080 gain    not used
1/PROTON_0101.fid spin      20
ACQUISITION     hst         0.008
sw 9594.6       pw90      11.300
at 1.708        alfa      6.600
np 32768        FLAGS
fb 5000         i1        n
bs 32           in        n
d1 1.000       dp         y
nt 8           hs        nn
ct 8           fn        not used
TRANSMITTER     fn        not used
tn H1          DISPLAY
sfrq 599.689   sp         -632.4
tof 599.7      wp         6630.3
tpwr 55        rf1        5538.9
pw 5.650      rfp        4353.7
DECOUPLER      C13      rp         162.2
dn 0          lp         -28.8
dof 0         PLOT
dm nnn        wc         250
decwave W40_hcx sc         0
dpwr 44       vs         18
daf 35088     th         54
a1 cdc ph
```



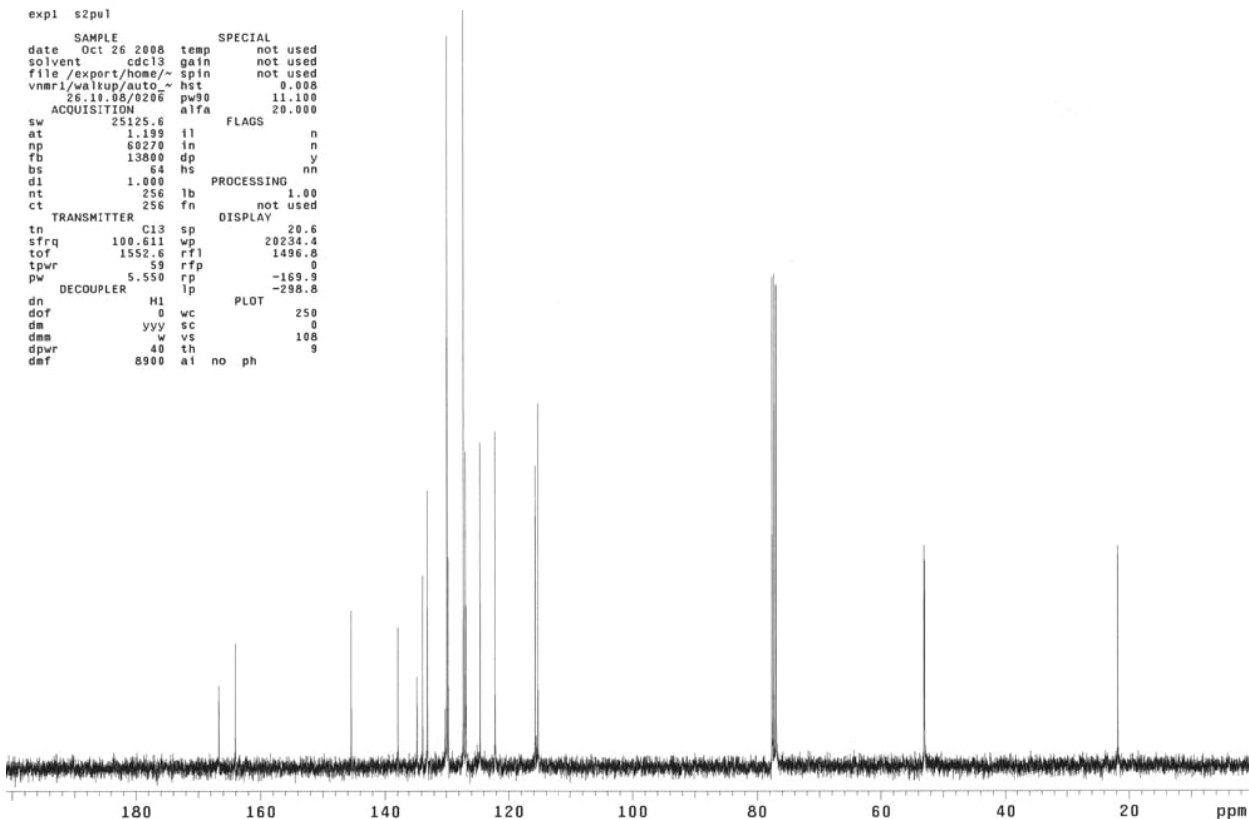
¹H NMR (600 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-11-071
STANDARD 1H OBSERVE

expl s2pul

```
SAMPLE          SPECIAL
date Oct 26 2008 temp      not used
solvent cdcl3    gain    not used
file /export/home/vn- spin    not used
nmr1/va1kup/auto~ hst      0.008
26.10.08/0205     pw90    11.100
ACQUISITION     alfa     20.000
sw 25125.6       FLAGS
at 1.199        i1        n
np 60270        in        n
fb 13800       dp         y
bs 64          hs        nn
d1 1.000       dp         y
nt 256        hs        nn
ct 256        fn        not used
TRANSMITTER     C13      DISPLAY
tn H1          sp         20.6
sfrq 100.611   wp         20234.4
tof 1552.6    rf1        1496.8
tpwr 59        rfp        0
pw 5.550     rp         -169.9
DECOUPLER      H1      lp         -288.8
dn 0          wc         250
dof 0         yy        sc         0
dm w         vs         108
dpwr 40       th         9
daf 8900     a1      no ph
```

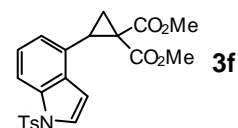


KS-09-035
STANDARD 1H OBSERVE

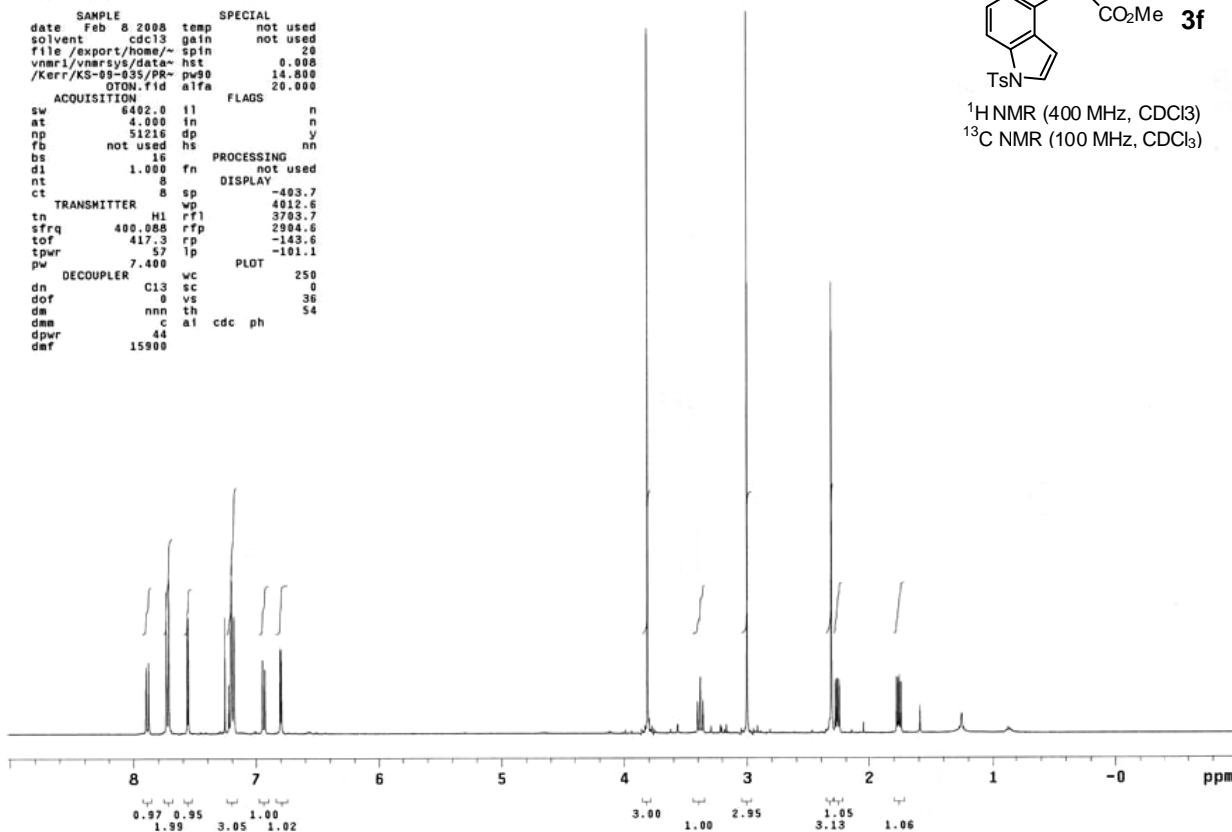
expl s2pu1

```
SAMPLE SPECIAL
date Feb 8 2008 temp not used
solvent cdcl3 gain not used
file /export/home/~ sp1n not used
vnmr1/vnmrsys/data/~ hst 0.008
/Kerr/KS-09-035/PR~ pw90 14.800
OTON.fid alfa 20.000

ACQUISITION FLAGS
sw 6402.0 f1 n
at 4.000 f0 n
np 51216 dp y
fb not used hs nn
bs 16 fn PROCESSING
d1 1.000 f0 not used
nt 8 DISPLAY
ct 8 sp -493.7
TRANSMITTER wp 4012.6
tn H1 rf1 3793.7
sfrq 400.088 rfp 2994.6
tof 417.3 rp -143.6
tpwr 57 lp -101.1
pw 7.400 PLOT
DECOUPLER wc 250
dn C13 sc 0
dof 0 vs 36
dm nnn th 54
dmm c al cdc ph
dpwr 44
dmf 15900
```



¹H NMR (400 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)

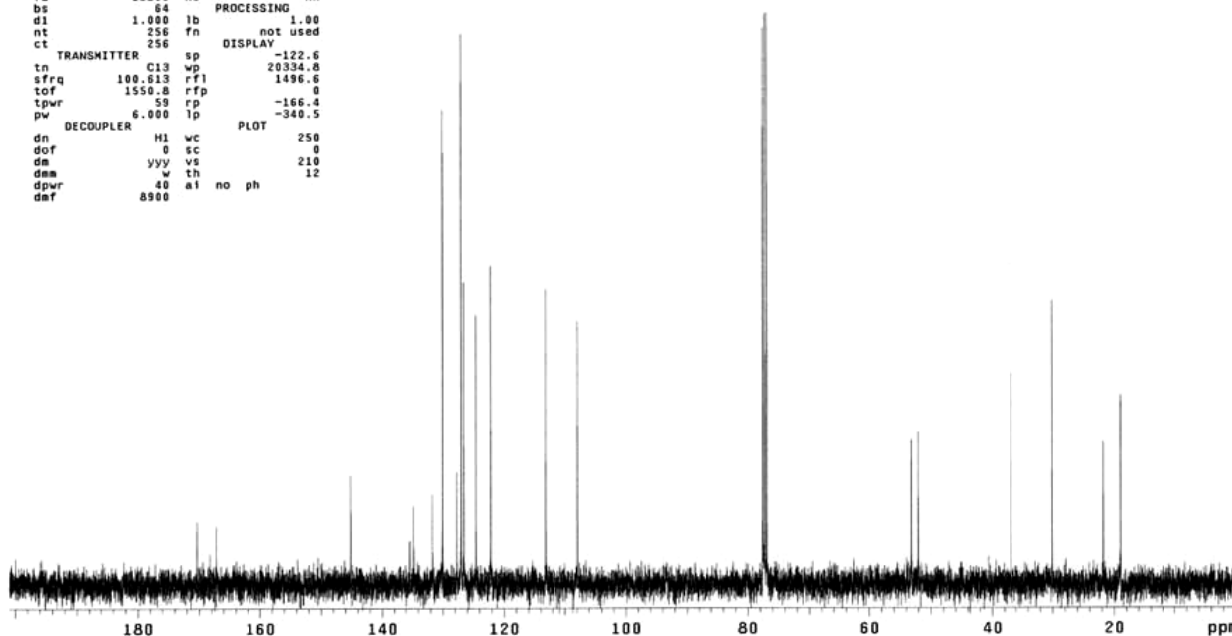


KS-09-035
STANDARD 1H OBSERVE

expl s2pu1

```
SAMPLE SPECIAL
date Feb 8 2008 temp not used
solvent cdcl3 gain not used
file /export/home/~ sp1n not used
vnmr1/vnmrsys/data/~ hst 0.008
/Kerr/KS-09-035/CA~ pw90 12.000
R8ON.fid alfa 20.000

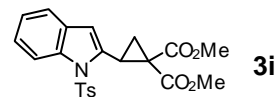
ACQUISITION FLAGS
sw 25125.6 f1 n
at 1.189 f0 n
np 60270 dp y
fb 13800 hs PROCESSING
bs 84 fn 1.00
d1 1.000 f0 not used
nt 256 f0 DISPLAY
ct 256 sp -122.6
TRANSMITTER C13 wp 20334.8
tn H1 rf1 1496.6
sfrq 100.613 rfp 0
tof 1550.8 rp -166.4
tpwr 59 lp -340.5
pw 6.000 PLOT
DECOUPLER H1 wc 250
dn 0 sc 0
dof 0 vs 210
dm yyy th 12
dmm w th
dpwr 40 al no ph
dmf 8980
```



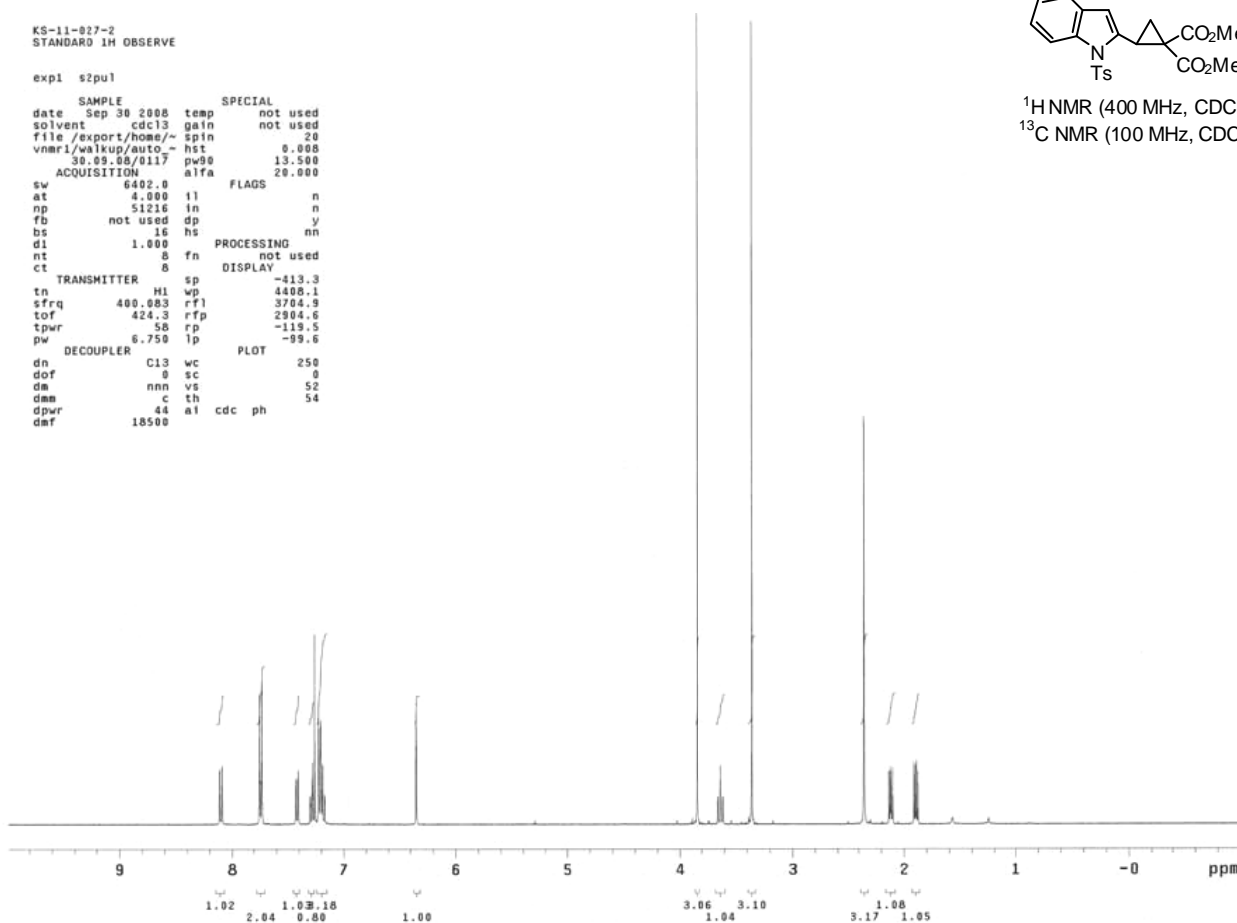
KS-11-027-2
STANDARD 1H OBSERVE

expl s2pul

SAMPLE		SPECIAL	
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solvent	cdcl3	gain	not used
file	/export/home/~	spin	20
vnmr1/walkup/auto_	hst	0.008	
30.09.08/0117	pw90	13.500	
ACQUISITION	alfa	20.000	
sw	6402.0	FLAGS	
at	4.000	l1	n
np	51216	in	n
fb	not used	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	8	fn	not used
ct	8	DISPLAY	
TRANSMITTER		sp	-413.3
tn	H1	wp	4406.1
sfrq	400.083	rfl	3704.9
tof	424.3	rfp	2904.6
tpwr	50	rp	-119.5
pw	8.750	lp	-99.6
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
da	nnn	vs	52
dmm	c	th	54
dpwr	44	ai	cdc ph
dmf	18500		



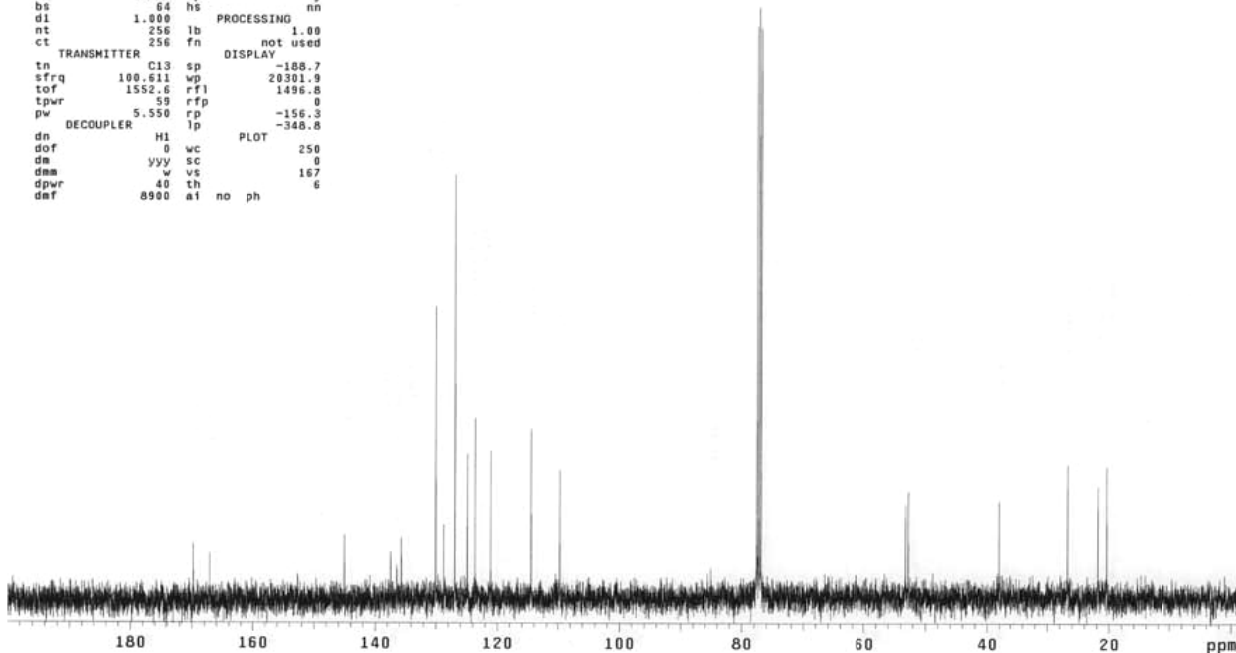
¹H NMR (400 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-11-027-2
STANDARD 1H OBSERVE

expl s2pul

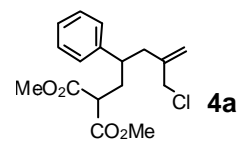
SAMPLE		SPECIAL	
date	Sep 30 2008	temp	not used
solvent	cdcl3	gain	not used
file	/export/home/~	spin	not used
vnmr1/walkup/auto_	hst	0.008	
30.09.08/0118	pw90	11.100	
ACQUISITION	alfa	20.000	
sw	25125.6	FLAGS	
at	1.199	l1	n
np	60270	in	n
fb	13800	dp	y
bs	64	hs	nn
d1	1.000	PROCESSING	
nt	256	lb	1.00
ct	256	fn	not used
TRANSMITTER		sp	-188.7
tn	C13	wp	20301.9
sfrq	100.611	rfl	1496.8
tof	1552.6	rfp	0
tpwr	50	rp	-156.3
pw	5.550	lp	-348.8
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
da	yyy	vs	167
dmm	w	th	6
dpwr	40	ai	no ph
dmf	8900		



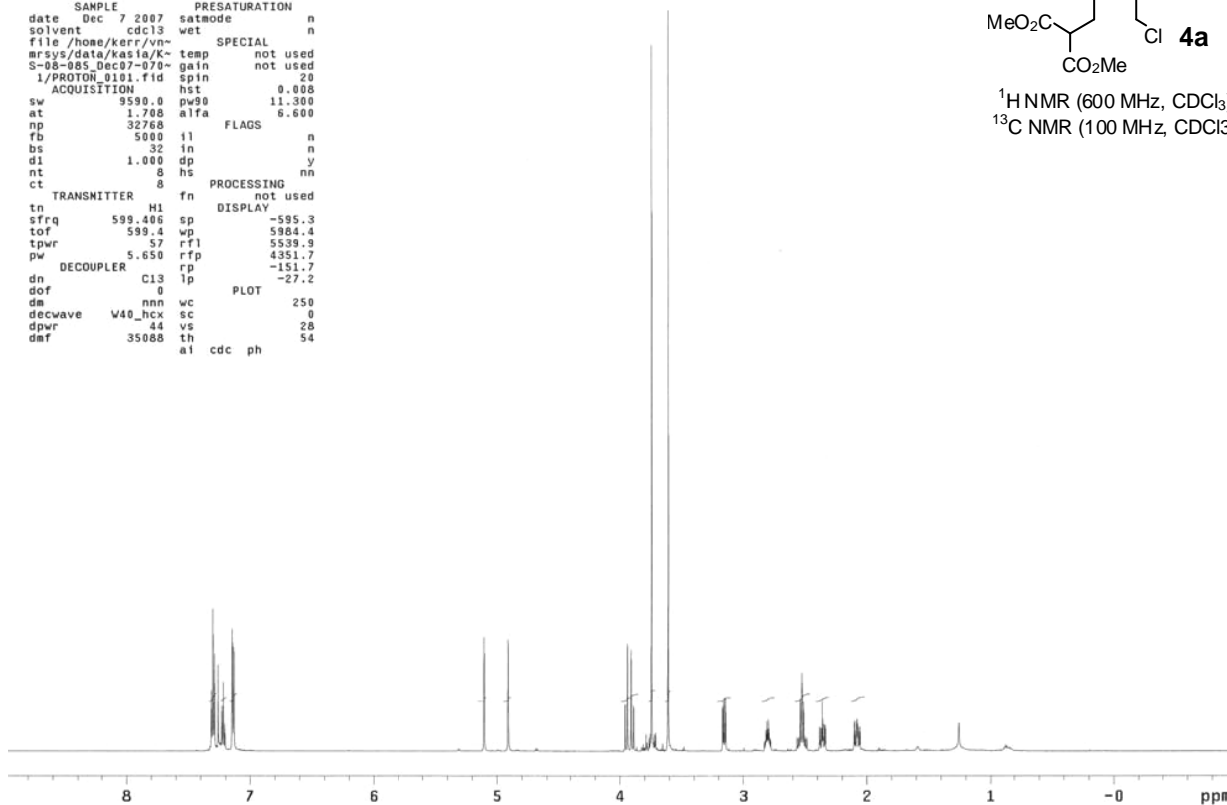
KS-08-085

exp1 PROTON

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SAMPLE      PRESATURATION
date Dec 7 2007 satmode n
solvent cdc13 wet n
file /home/kerr/vnmr-
mrsys/data/kas10/k- SPECIAL
S-08-085_Dec07-070- gain not used
1/PROTON_0101.fid spin 20
ACQUISITION hst 0.008
sw 9590.0 pw90 11.300
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5080 il n
bs 32 in n
d1 1.000 dp y
nt 8 hs PROCESSING
ct 8 fn not used
tn H1 DISPLAY
sfrq 599.406 sp -595.3
tof 599.4 wp 5984.4
tpwr 57 rfl 5539.9
pw 5.650 rfp 4351.7
DECOUPLER C13 rp -151.7
dn C13 lp -27.2
dof 0 PLOT
dm nnn wc 250
decwave W40_hcx sc 0
dpr 44 vs 28
dmf 35088 th 54
ai cdc ph
```



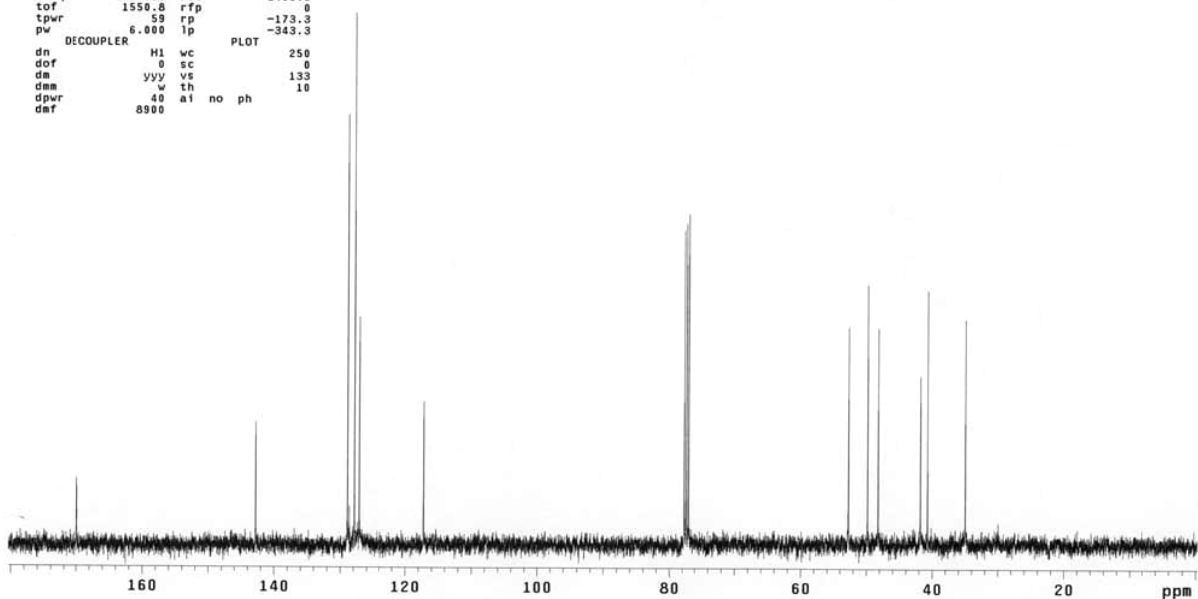
¹H NMR (600 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-08-085
STANDARD 1H OBSERVE

exp2 s2pu1

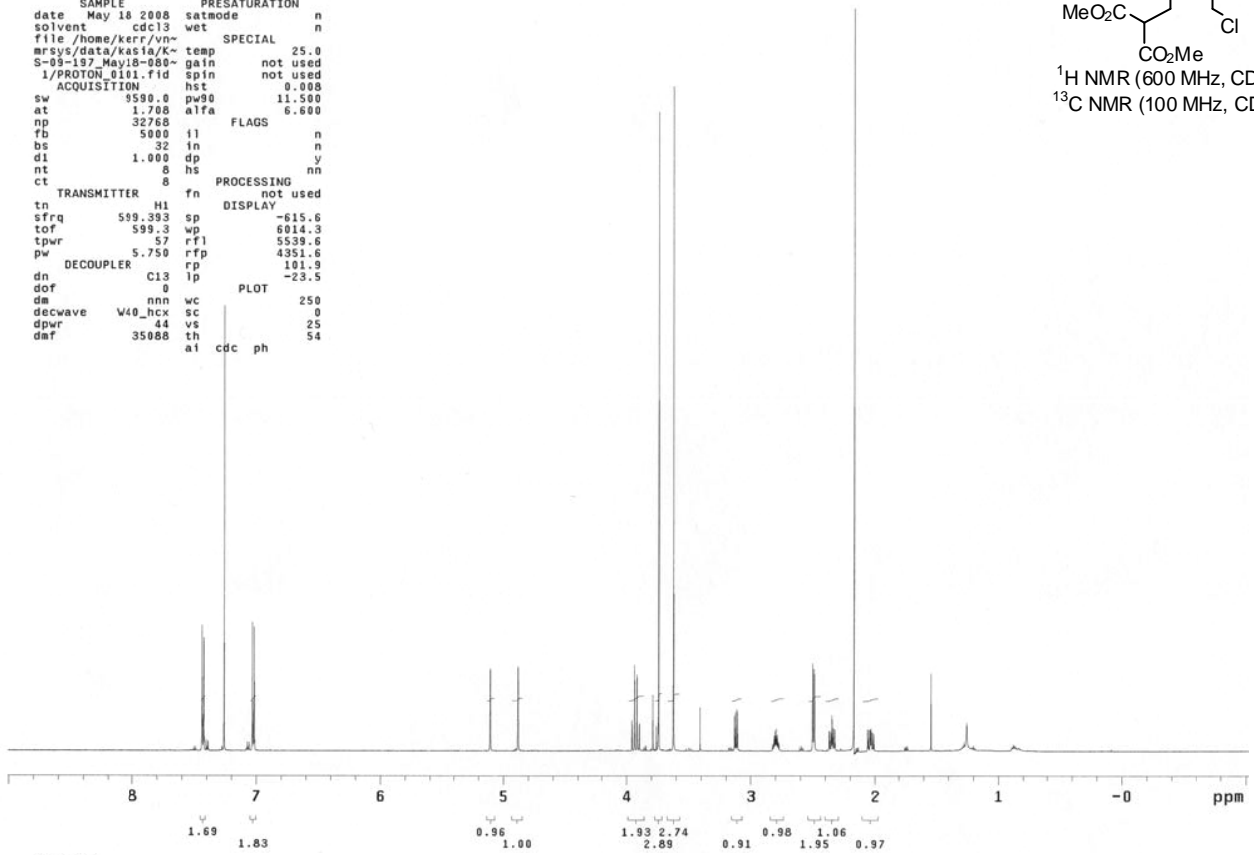
```
SAMPLE      SPECIAL
date Dec 7 2007 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/vnmr- hst 0.008
/Kerr/KS-08-085/CA- pw90 12.000
RBDN.fid alfa 28.000
ACQUISITION FLAGS
sw 25125.6 il n
at 1.199 in n
np 60270 dp y
fb 13800 hs PROCESSING
bs 64 hs nn
d1 1.000 lb 1.00
nt 256 fn not used
ct 256 DISPLAY
tn C13 wp -24.4
sfrq 100.613 rfl 18166.2
tof 1550.8 rfp 1496.6
tpwr 59 rp -173.3
pw 6.000 lp -343.3
DECOUPLER H1 wc PLOT
dn H1 wc 250
dof 0 sc 0
dm yyy vs 133
dms w th 10
dpr 40 ai no ph
dmf 8900
```



KS-09-197

exp1 PROTON

```
SAMPLE      PRESATURATION
date May 18 2008  satmode n
solvent cdc13  wet n
file /home/kerr/vn-  SPECIAL
mrsys/data/kasia/K-  temp 25.0
S-09-197_May18-060- gain not used
1/PROTON_0181.fid  spin not used
ACQUISITION  hst 0.008
sw 9590.0  pw90 11.500
at 1.708  alfa 6.600
np 32768  FLAGS
fb 5000  il n
bs 32  in n
dl 1.000  dp y
nt 8  hs nn
ct 8  PROCESSING not used
TRANSMITTER  fn DISPLAY
tn H1  sp -615.6
sfrq 599.393  wp 6014.3
tof 599.3  rfl 5539.6
tpwr 57  rfp 4351.6
pw 5.750  rp 101.9
DECOUPLER  C13  lp -23.5
dn C13  PLOT
dof 0  wc 250
dm nnn  sc 0
decwave W40_hcx  vs 25
dpwr 44  th 54
daf 35088  al cdc ph
```

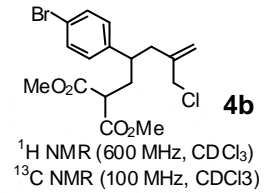
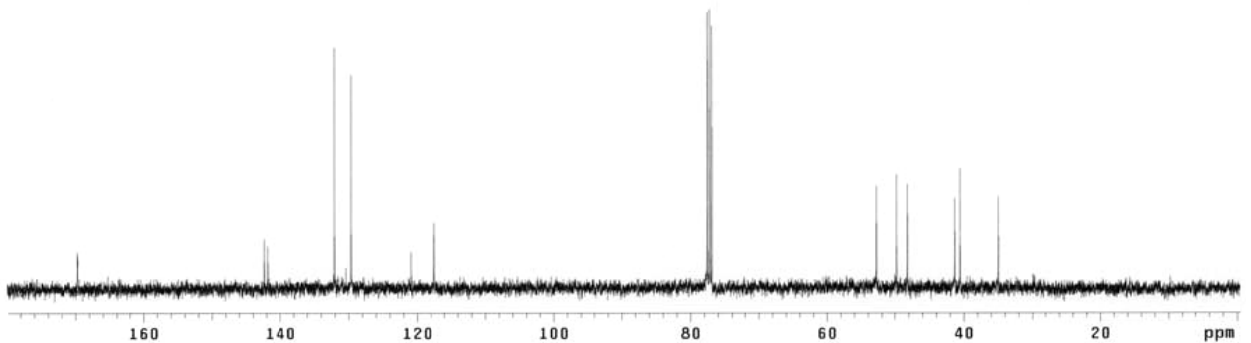


KS-09-197

STANDARD 1H OBSERVE

exp2 s2pu1

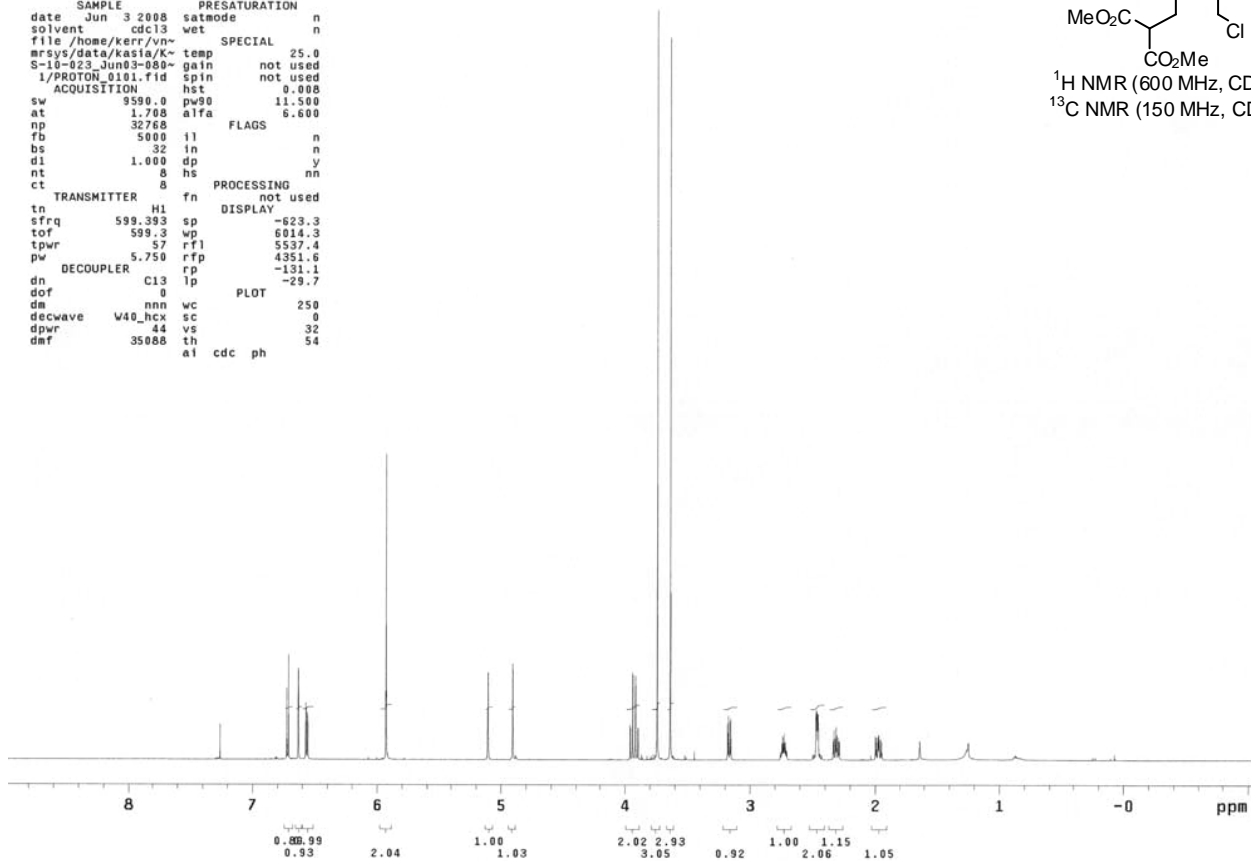
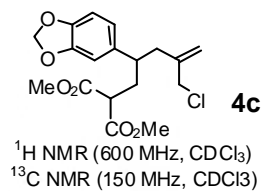
```
SAMPLE      SPECIAL
date May 18 2008  temp not used
solvent cdc13  gain not used
file /export/home/~  spin not used
vnmr1/wakeup/auto_  hst 0.008
18.05.09/9205  pw90 11.100
ACQUISITION  alfa 20.000
sw 25125.6  FLAGS
at 1.199  il n
np 68270  in n
fb 13800  dp y
bs 64  hs nn
dl 1.000  PROCESSING 1.00
nt 256  lb fn not used
ct 256  DISPLAY
TRANSMITTER  C13  sp -40.5
tn H1  wp 18140.1
sfrq 100.613  rfl 1496.6
tof 1550.8  rfp 0
tpwr 59  rp -150.2
pw 5.550  lp -369.3
DECOUPLER  H1  PLOT
dn H1  wc 250
dof 0  sc 0
dm yyy  vs 98
dmm w  th 4
dpwr 40  al no ph
daf 8900
```



KS-10-023

expl PROTON

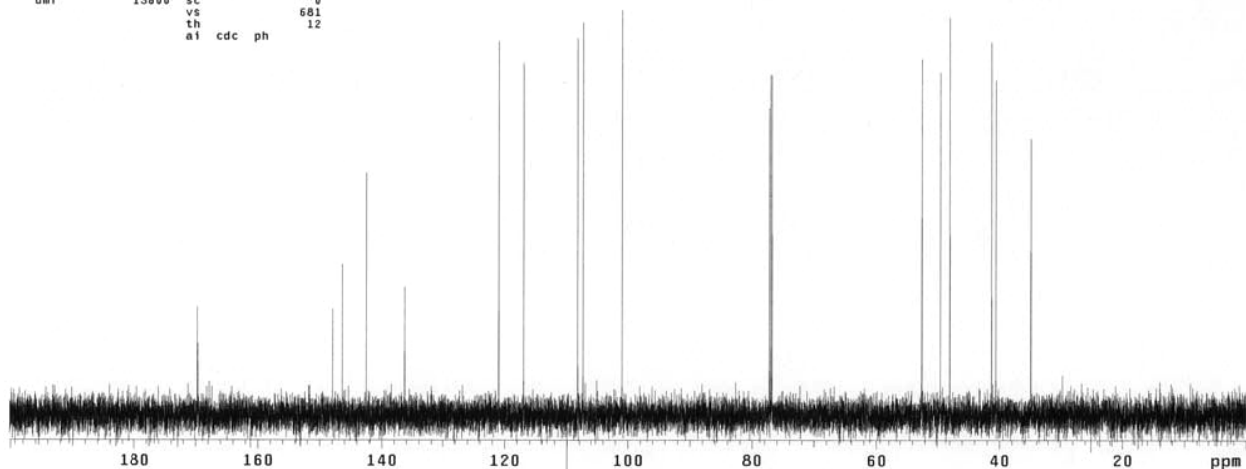
```
SAMPLE      PRESATURATION
date Jun 3 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
mrsys/data/kasia/K- temp 25.0
S-10-023_Jun03-080- gain not used
1/PROTON_0101.fid spin not used
ACQUISITION hst 0.008
sw 9590.0 pw90 11.500
at 1.788 alfa 6.600
np 32768 FLAGS
fb 5000 i1 n
bs 32 in n
d1 1.000 dp y
nt 8 hs nn
ct 8 fn PROCESSING
TRANSMITTER H1 fn not used
tn H1 DISPLAY
sfrq 599.393 sp -623.3
tof 599.3 wp 6014.3
tpwr 57 rfl 5537.4
pw 5.750 rfp 4351.6
DECOUPLER rp -131.1
dn C13 lp -29.7
dof 8 PLOT
dm nnn wc 250
decwave W40_hcx sc 0
dpwr 44 vs 32
dmf 35088 th cdc ph 54
```



KS-10-023

expl CARBON

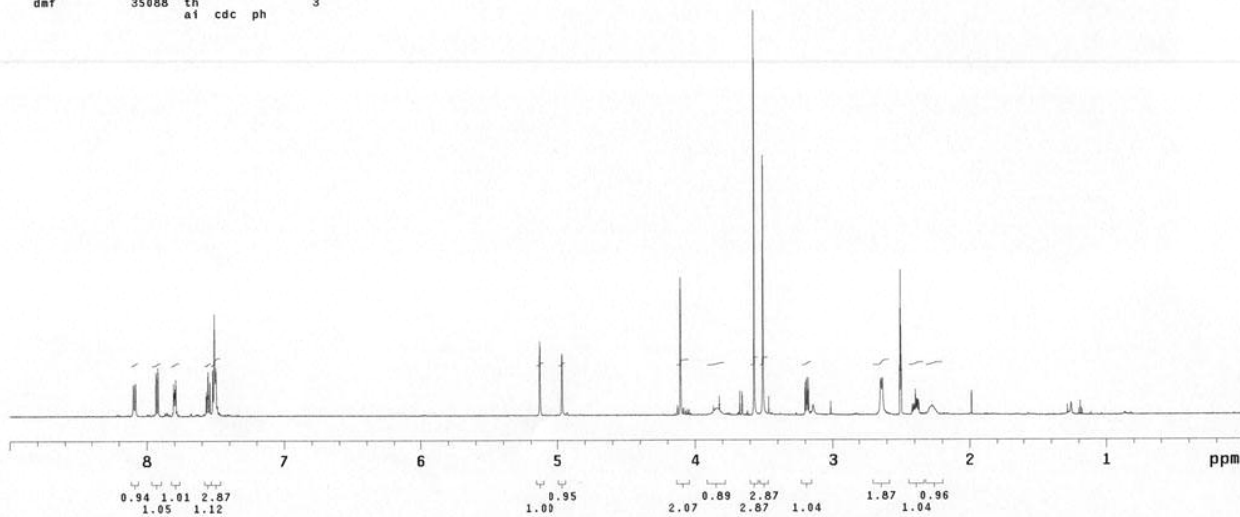
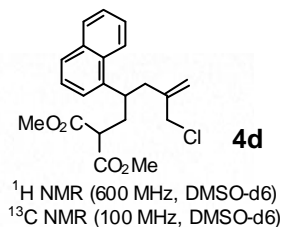
```
SAMPLE      PRESATURATION
date Jun 3 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
mrsys/data/kasia/K- temp 25.0
S-10-023_Jun03-080- gain not used
2/CARBON_0101.fid spin not used
ACQUISITION hst 0.008
sw 37682.5 pw90 23.300
at 0.870 alfa 10.000
np 65536 FLAGS
fb 21000 i1 n
bs 64 in n
d1 1.000 dp y
nt 128 hs nn
ct 128 fn PROCESSING
TRANSMITTER lb 0.50
tn C13 lsfid -2
sfrq 150.733 fn not used
tof 2295.0 DISPLAY
tpwr 55 sp -13.1
pw 11.650 wp 30183.5
DECOUPLER H1 rfp 2262.4
dn 0 rp -160.8
dof yyy lp -52.8
dm w PLOT
decwave 42 wc 250
dpwr 13600 sc 0
dmf vs 681
th cdc ph 12
```



KS-09-051

expl PROTON

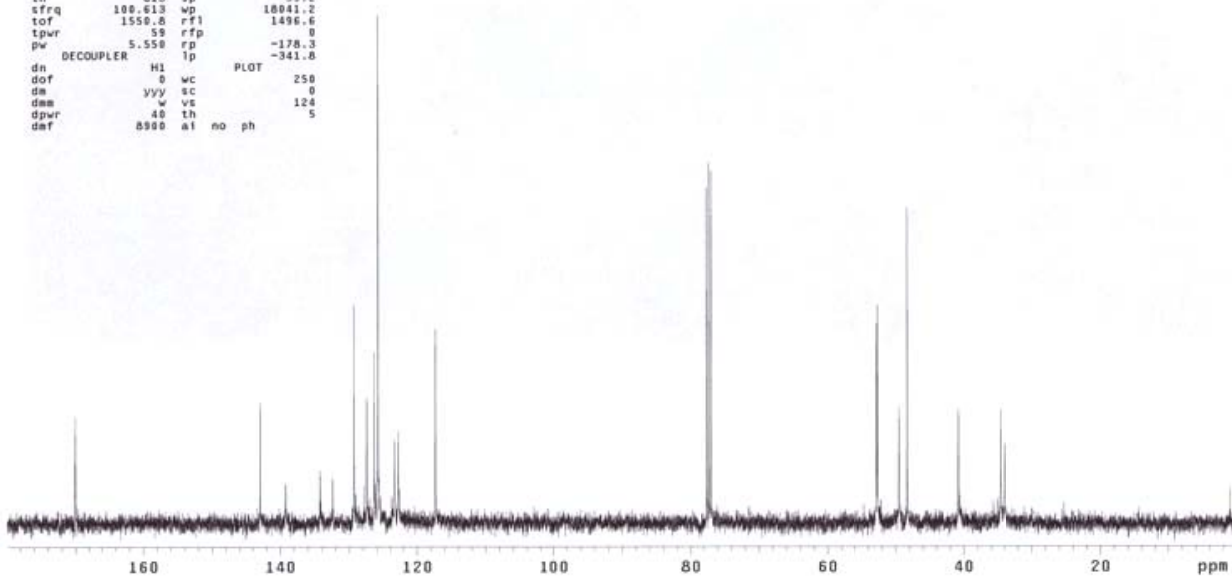
```
SAMPLE          PRESATURATION
date Feb 16 2008 satmode n
solvent dmsc wet n
file /home/kerr/vnr- SPECIAL
mrsys/data/kasia/K- temp 66.0
S-09-051-2_Feb16-0- gain not used
801/PROTON_0101 spin 20
ACQUISITION      hst 0.008
sw 9590.0 pw90 11.300
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5000 i1 n
bs 32 in n
d1 1.000 dp y
nt 8 hs nn
ct 8 PROCESSING
tn H1 fn not used
sfrq 599.409 sp DISPLAY -3.1
tof 599.4 wp 5397.9
tpwr 57 rfl 2677.0
pw 5.650 rfp 1498.5
DECOUPLER rp 174.4
dn C13 p -19.6
dof 0 PLOT
da nnn wc 250
decwave W40_hcx sc 0
dpwr 44 vs 24
dmf 35088 th 3
ai cdc ph
```



KS-10-033
STANDARD 1H OBSERVE

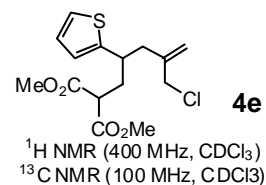
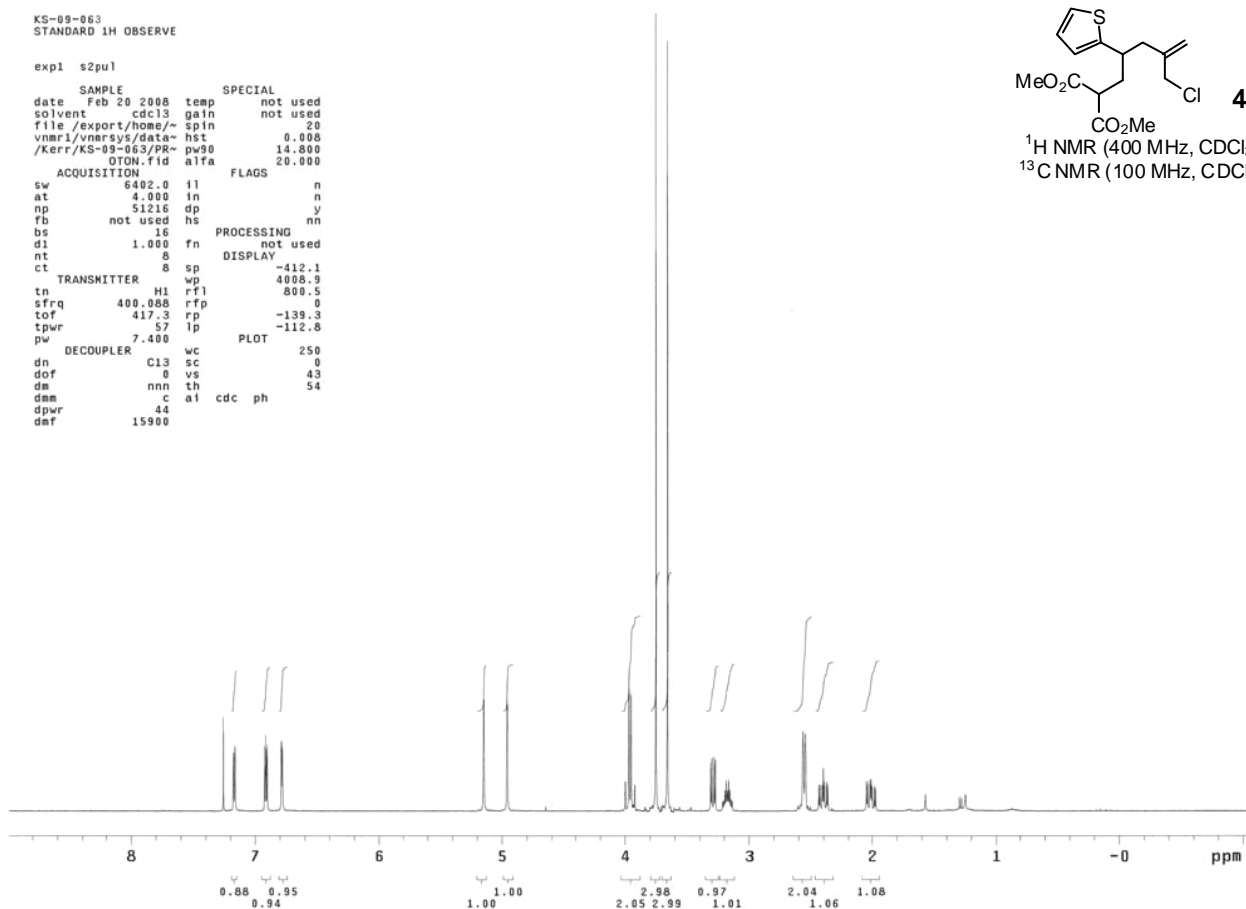
expl1 s2pu1

```
SAMPLE          SPECIAL
date May 24 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/wakeup/auto_ hst 0.008
24.05.08/0103 pw90 11.100
ACQUISITION      alfa 20.000
sw 25125.6 FLAGS
at 1.188 i1 n
np 60270 in n
fb 13800 dp y
bs 64 hs nn
d1 1.000 PROCESSING
nt 256 lb fn 1.00
ct 256 PLOT
tn C13 sp DISPLAY 59.2
sfrq 100.613 wp 10041.2
tof 1550.8 rfl 1496.6
tpwr 59 rfp 0
pw 5.550 rp -178.3
DECOUPLER 1p -341.8
dn H1 PLOT
dof 0 wc 250
da vvy sc 0
dms w vs 124
dpwr 40 th 5
dmf 8900 ai no ph
```



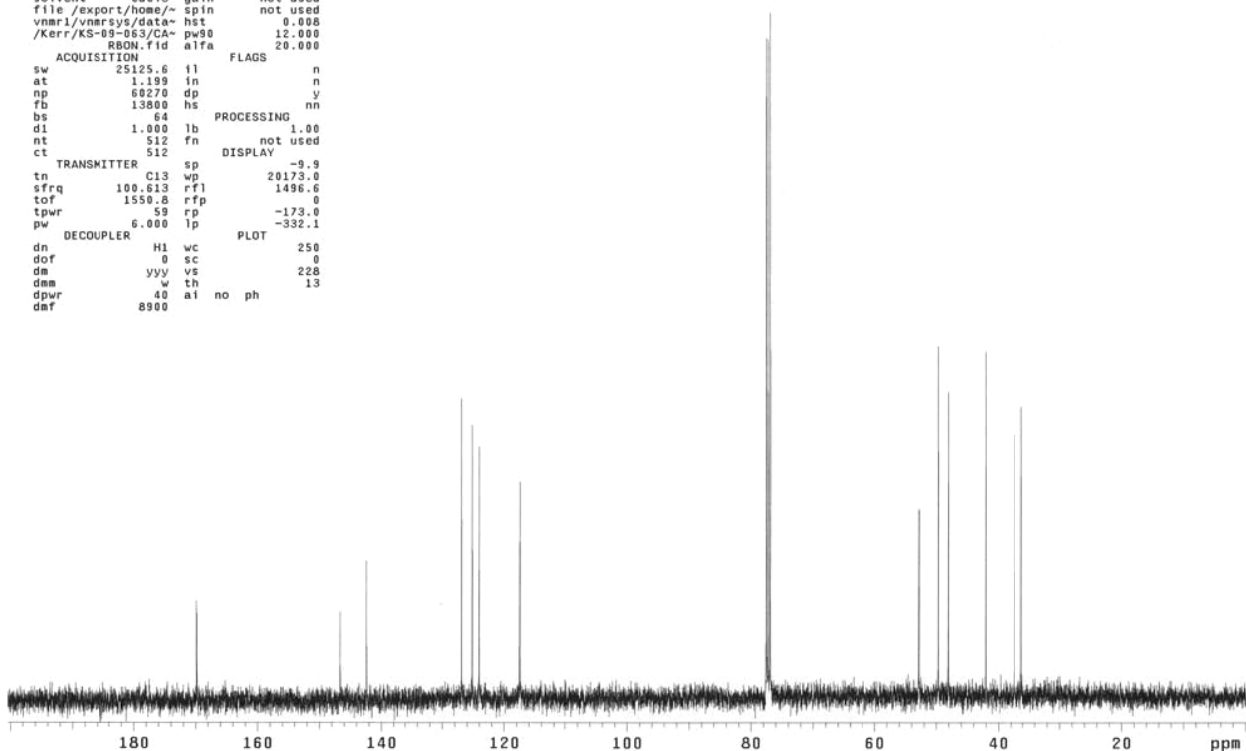
KS-09-063
STANDARD 1H OBSERVE

```
exp1 s2pu1
SAMPLE SPECIAL
date Feb 20 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ spin 20
vnmr1/vnmrSYS/data~ hst 0.008
/Kerr/KS-09-063/PR~ pw90 14.800
OTON.fid a1fa 20.000
ACQUISITION FLAGS
sw 6402.0 f1 n
at 4.000 in n
np 51216 dp y
fb not used hs nn
bs 16 PROCESSING
d1 1.000 fn not used
nt 8 DISPLAY
ct 8 sp -412.1
TRANSMITTER wp 4808.9
tn H1 rF1 800.5
sfrq 400.088 rfp 0
tof 417.3 rp -139.3
tpwr 57 lp -112.8
pw 7.400 PLOT
DECOUPLER wc 250
dn C13 sc 0
dof 0 vs 43
dm nnn th 54
dmm c a1 cdc ph
dpwr 44
dmf 15900
```



KS-09-063
STANDARD 1H OBSERVE

```
exp1 s2pu1
SAMPLE SPECIAL
date Feb 20 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/vnmrSYS/data~ hst 0.008
/Kerr/KS-09-063/CA~ pw90 12.000
RBN.fid a1fa 20.000
ACQUISITION FLAGS
sw 25125.6 f1 n
at 1.199 in n
np 68270 dp y
fb 13600 hs nn
bs 64 PROCESSING
d1 1.000 fb 1.00
nt 512 fn not used
ct 512 DISPLAY
TRANSMITTER sp -9.9
tn C13 wp 20173.0
sfrq 100.613 rF1 1496.6
tof 1550.8 rfp 0
tpwr 59 rp -173.0
pw 6.000 lp -332.1
DECOUPLER H1 wc 250
dn 0 sc 0
dof yyy vs 228
dm w th 15
dmm 40 a1 no ph
dpwr 8900
dmf
```



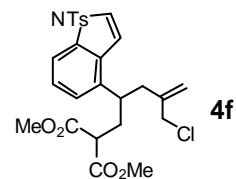
KS-09-037
STANDARD 1H OBSERVE

exp8 s2pu1

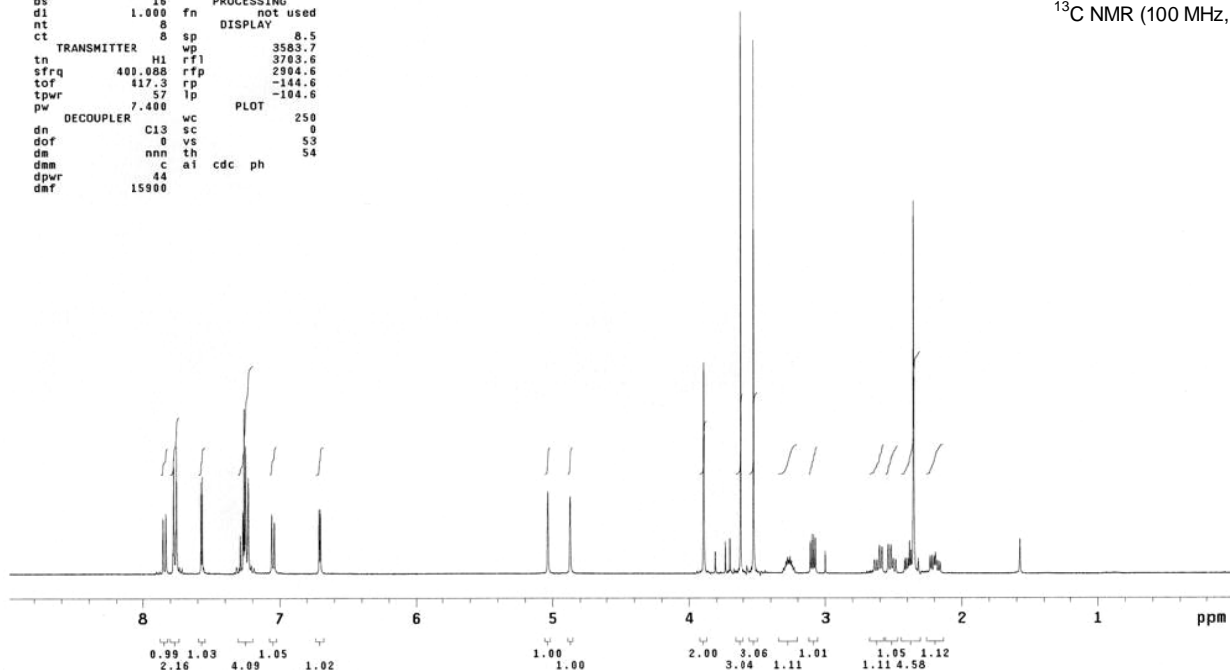
```
SAMPLE SPECIAL
date Feb 9 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/vnmrSYS/data~ hst 0.008
/Kerr/KS-09-037/PR~ pw90 14.000
      OTOM.fid a1fa 20.000

ACQUISITION FLAGS
sw 6402.0 f1 n
at 1.000 in n
np 51216 dp y
fb not used hs nn
bs 16 PROCESSING
d1 1.000 fn not used
nt 8 DISPLAY
ct 8 sp 8.5
TRANSMITTER wp 3583.7
tn H1 rf1 3703.6
sfrq 400.088 rfp 2904.6
tof 417.3 rp -144.6
tpwr 57 lp -104.6
pw 7.400 PLOT

DECOUPLER wc 250
dn C13 sc 0
dof 0 vs 53
dm nnn th 54
dmm c a1 cdc ph
dpwr 44
dmf 15900
```



¹H NMR (400 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



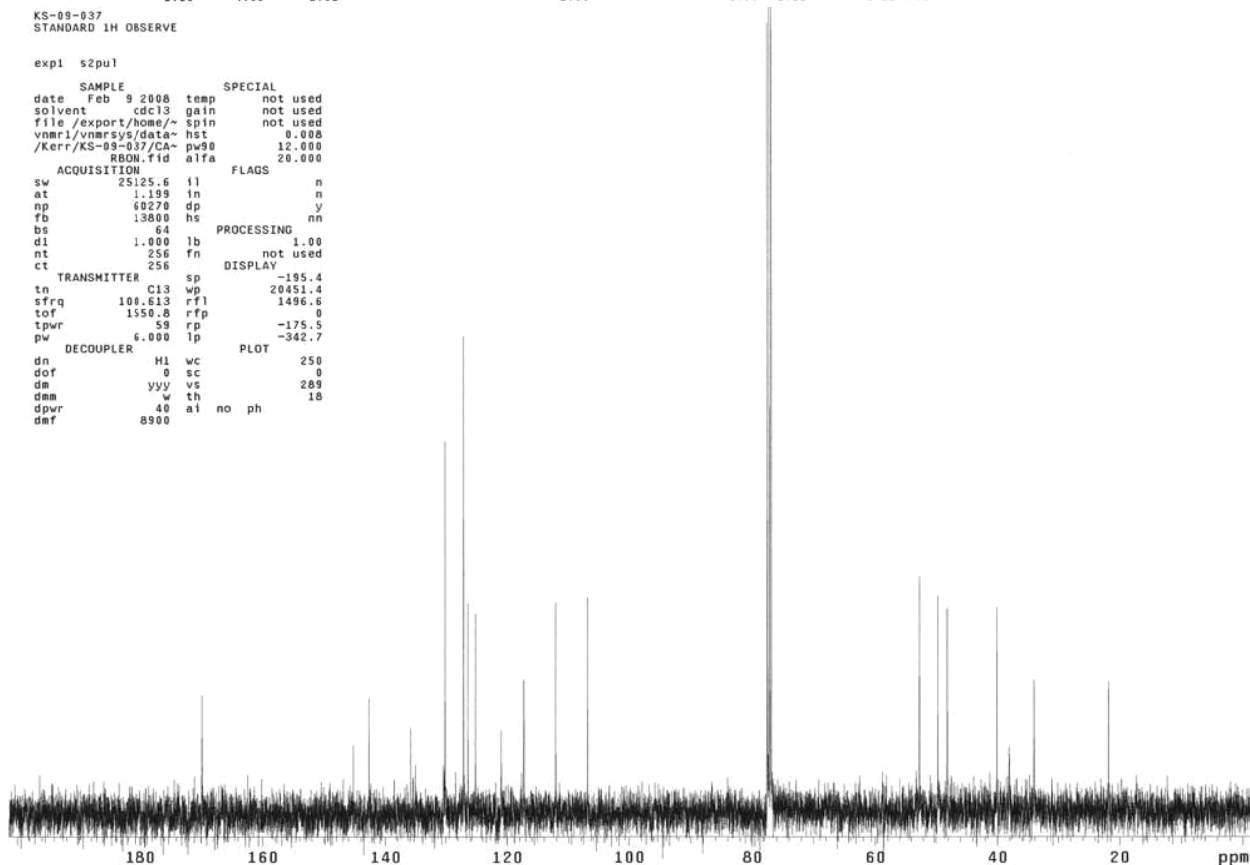
KS-09-037
STANDARD 1H OBSERVE

exp1 s2pu1

```
SAMPLE SPECIAL
date Feb 9 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/vnmrSYS/data~ hst 0.008
/Kerr/KS-09-037/CA~ pw90 12.000
      RSON.fid a1fa 20.000

ACQUISITION FLAGS
sw 25125.6 f1 n
at 1.199 in n
np 68270 dp y
fb 13800 hs nn
bs 64 PROCESSING
d1 1.000 lb 1.00
nt 256 fn not used
ct 256 DISPLAY
TRANSMITTER sp -195.4
tn C13 wp 20451.4
sfrq 101.613 rf1 1496.6
tof 1550.8 rfp 0
tpwr 59 rp -175.5
pw 6.000 lp -342.7

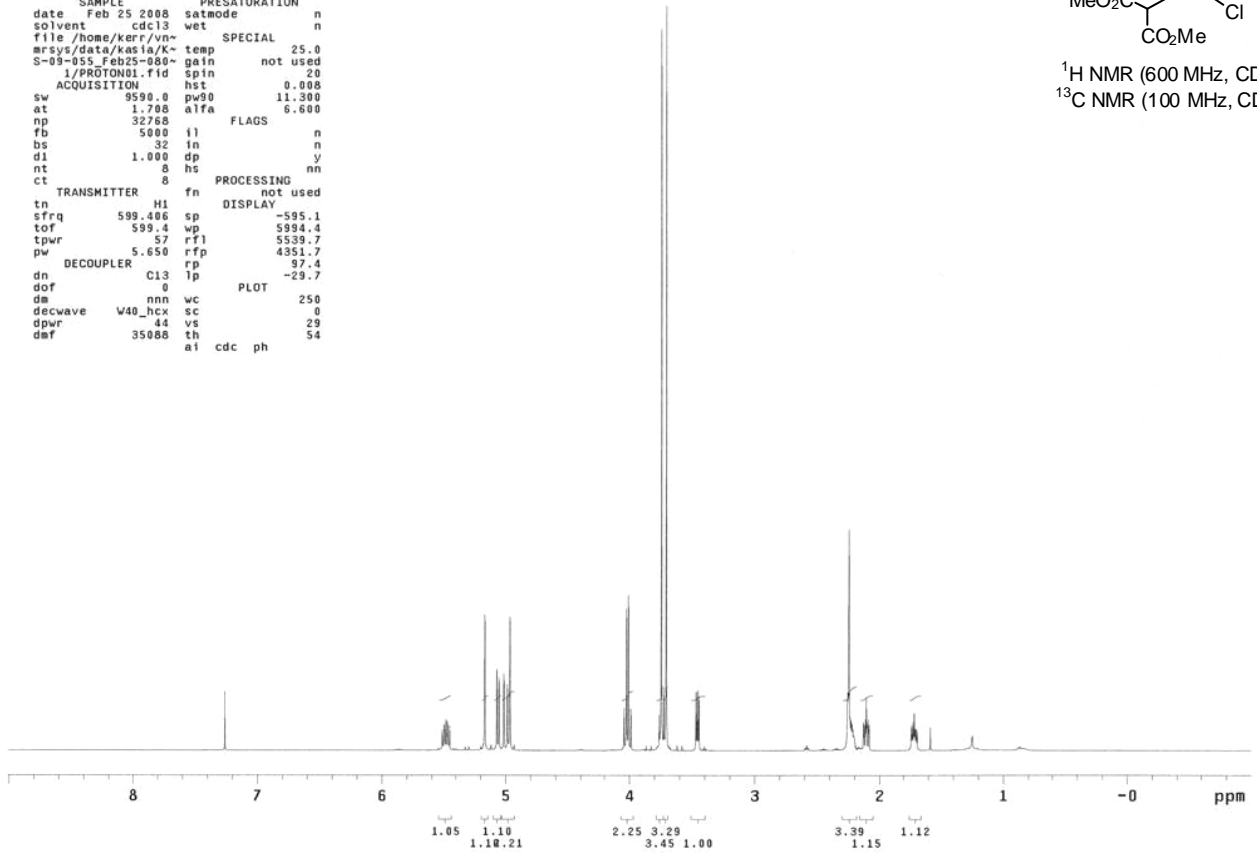
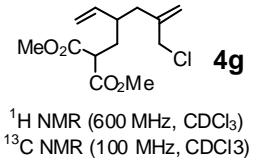
DECOUPLER H1 wc 250
dn H1 sc 0
dof yyv vs 289
dm w th 18
dpwr 40 a1 no ph
dmf 8900
```



KS-09-055

exp1 PROTON

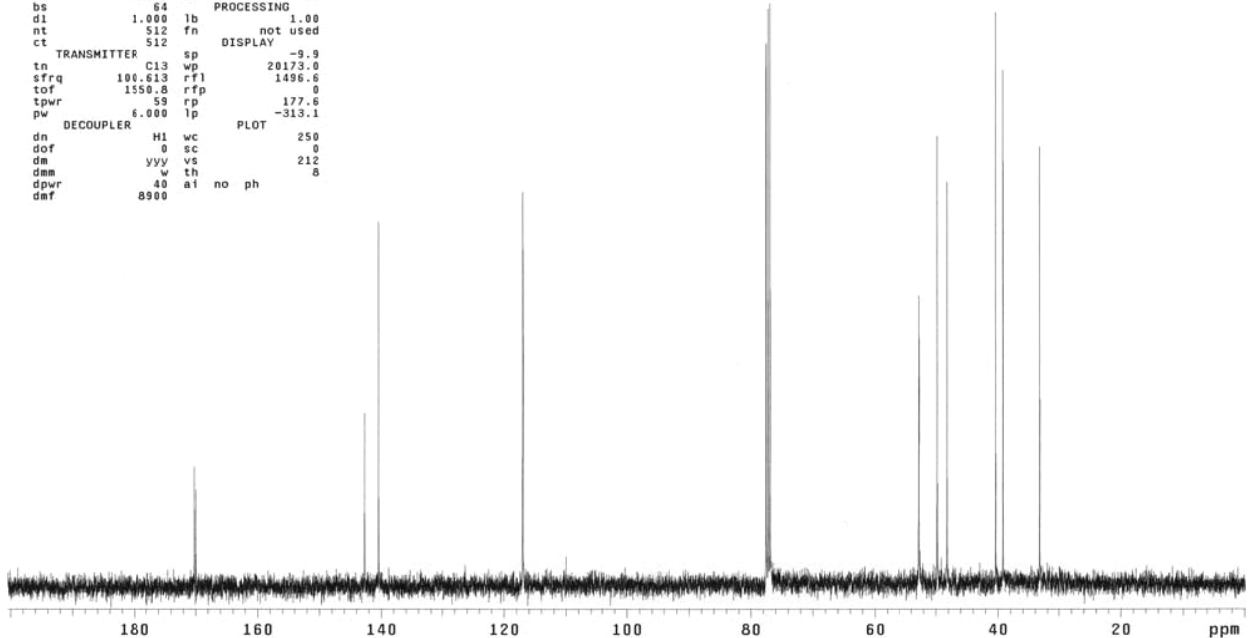
```
SAMPLE      PRESATURATION
date Feb 25 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
mrssys/data/kasia/k- temp 25.0
S-09-055_Feb25-060 gain not used
1/PROTON01.fid spin 20
ACQUISITION wc hst 0.008
sw 9590.0 pv90 11.300
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5000 il n
bs 32 in n
d1 1.000 dp y
nt 8 hs nn
ct 8 PROCESSING
tn H1 fn not used
sfrq 599.406 sp -595.1
tof 599.4 wp 5994.4
tpwr 57 rfl 5599.7
pw 5.650 rfp 4351.7
DECOUPLER rp 97.4
dn C13 lp -29.7
dof 0 PLOT
dm nnn wc 250
decwave W40_hcx sc 0
dpwr 44 vs 29
dof 35088 th 54
dof al cdc ph
```



KS-09-055
STANDARD 1H DESERVE

exp1 s2pu1

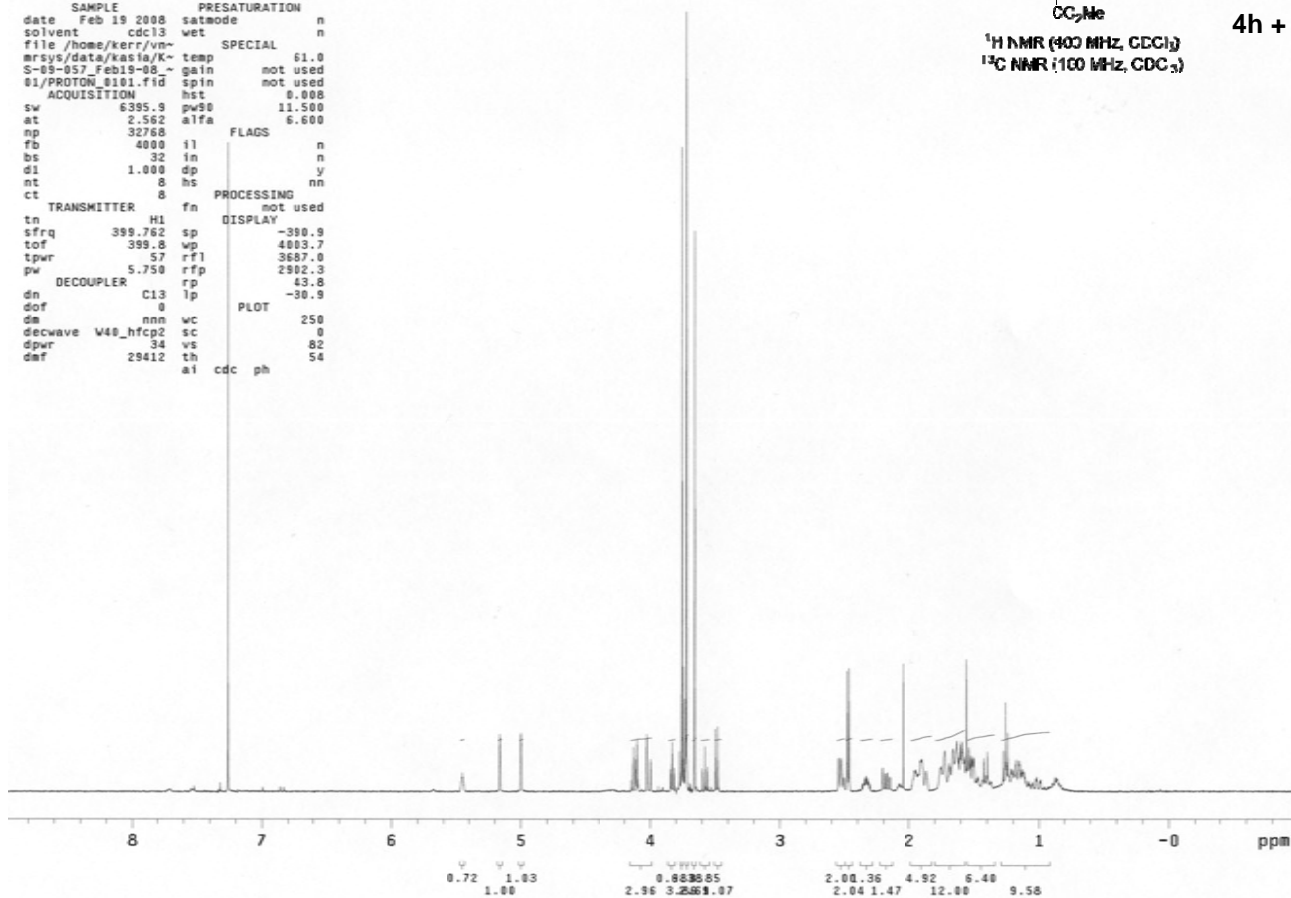
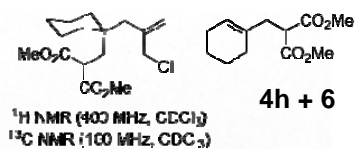
```
SAMPLE      SPECIAL
date Feb 25 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ not used
vnmr1/vnmrsys/data~ hst 0.008
/Kerr/KS-09-055/CA- pv90 12.000
RBON.fid alfa 20.000
ACQUISITION FLAGS
sw 25125.6 il n
at 1.199 in n
np 60270 dp y
fb 13800 hs
bs 64 PROCESSING
d1 1.000 lb 1.00
nt 512 fn not used
ct 512 DISPLAY
tn C13 sp -9.9
sfrq 100.613 wp 20173.0
tof 1550.8 rfl 1496.6
tpwr 59 rfp 0
pw 6.000 rp 177.6
DECOUPLER lp -313.1
dn H1 wc 250
dof 0 sc 0
dm yy vs 212
dmm w th 8
dpwr 40 ai no ph
dof 8900
```



KS-09-057

exp2 PROTON

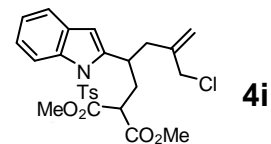
```
SAMPLE          PRESATURATION
date    Feb 19 2008  satmode      n
solvent  cdcl3      wet          n
file    /home/kerr/vn-          SPECIAL
mrssys/data/kasla/K-          temp      61.0
S-09-057_Feb19-06_-          gain      not used
01/PROTON_0101.fid          spin      not used
ACQUISITION          hst          0.008
sw       6395.9      pw50        11.500
at       2.562      alfa         6.600
np       32768
fb       4000      il          n
bs       32        in          n
d1       1.000     dp          y
nt       8         hs          nn
ct       8         PROCESSING
TRANSMITTER          fn          not used
tn       H1        DISPLAY
sfrq    399.762    sp          -399.9
tof     399.8     wp          4003.7
tpwr    57        rf1         3687.0
pw       5.750    rfp         2902.3
DECOUPLER          C13        rp          43.8
dn       0        lp          -30.9
dof     0         PLOT
dm       nnn      wc          250
decwave W40_hfcp2  sc          0
dpr     34        vs          82
dnt     29412    th          54
a1      cdc      ph
```



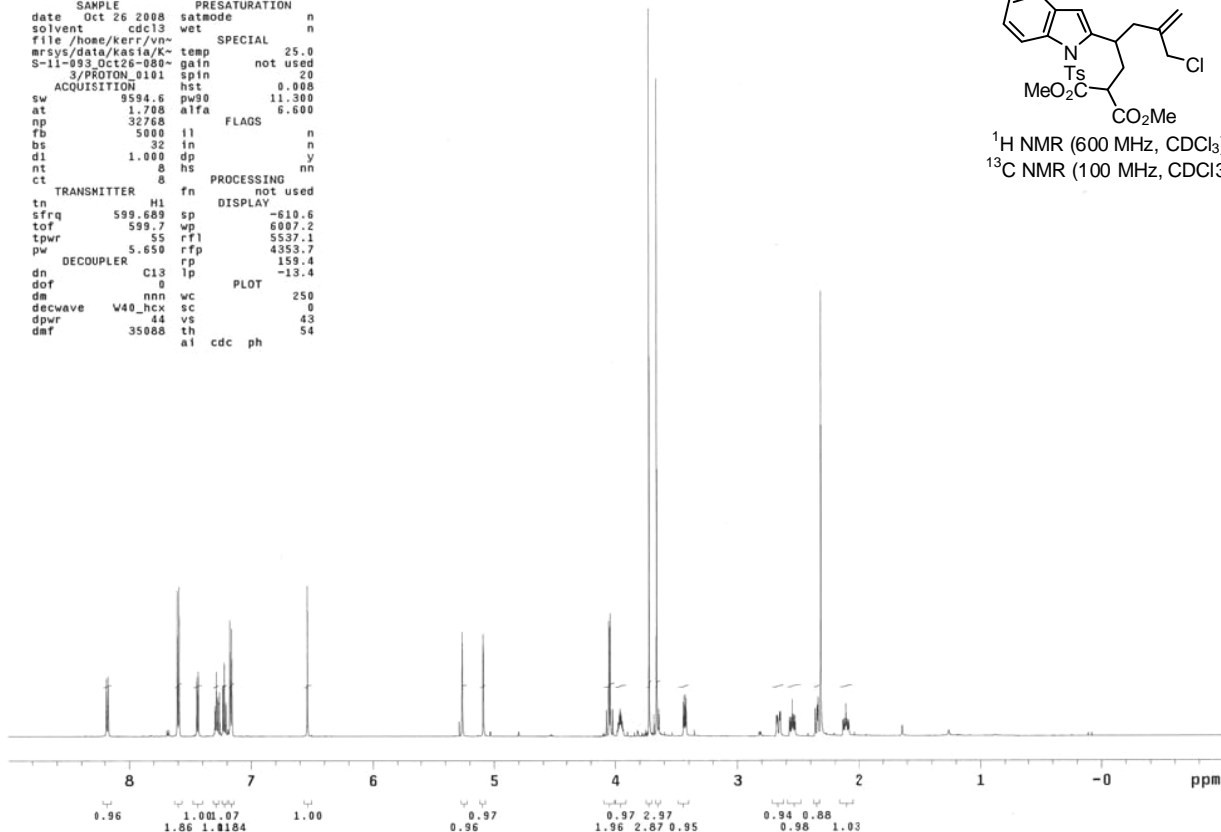
KS-11-093

exp1 PROTON

```
SAMPLE      PRESATURATION
date Oct 26 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
mrsys/data/kas1a/K- temp 25.0
S-11-093_Oct26-080- gain not used
3/PROTON_0101 spin 20
ACQUISITION hst 0.008
sw 9594.6 pw90 11.300
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5000 f1 n
bs 32 in n
d1 1.000 dp y
nt 8 hs nn
ct 8 PROCESSING
TRANSMITTER H1 fn not used
tn 0 DISPLAY
sfrq 599.689 sp -610.6
tof 599.7 wp 6007.2
tpwr 55 rfp 5537.1
pw 5.650 rfp 4353.7
DECOUPLER rp 159.4
dn C13 lp -13.4
dof 0 PLOT
dm nnn wc 250
decwave W40_hcx sc 0
dpwr 44 vs 43
dmf 35068 a1 cdc ph 54
```



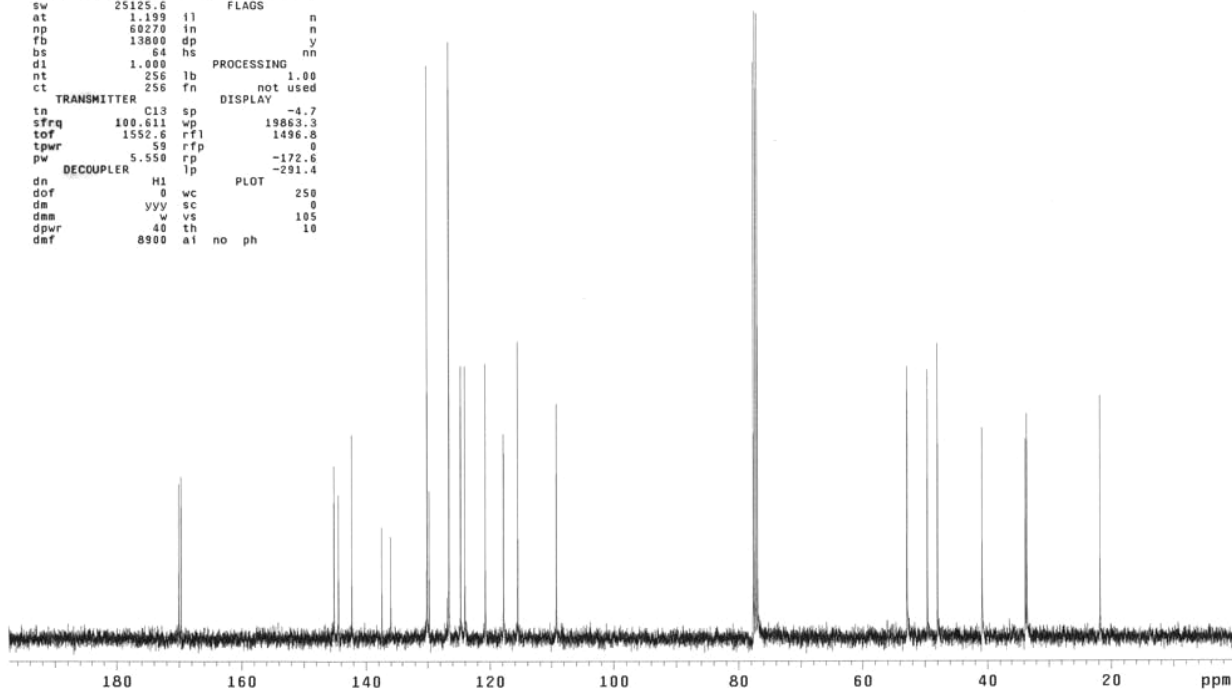
¹H NMR (600 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-11-093
STANDARD 1H OBSERVE

exp1 s2pu1

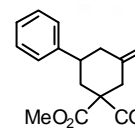
```
SAMPLE      SPECIAL
date Oct 26 2008 temp not used
solvent cdc13 gain not used
file /export/home/ spin not used
vnmr1/walkup/auto_ hst 0.008
26.10.08/0106 pw90 11.100
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 f1 n
np 60270 in n
fb 13800 dp y
bs 64 hs nn
d1 1.000 dp y
nt 256 lb PROCESSING 1.00
ct 256 fn not used
TRANSMITTER H1 DISPLAY
tn C13 sp -4.7
sfrq 100.611 wp 19883.3
tof 1552.6 rfp 1496.8
tpwr 59 rfp 0
pw 5.550 rp -172.6
DECOUPLER H1 lp -291.4
dn 0 wc PLOT 250
dm yyy sc 0
dmm w vs 105
dpwr 40 th 10
dmf 8900 a1 no ph
```



```

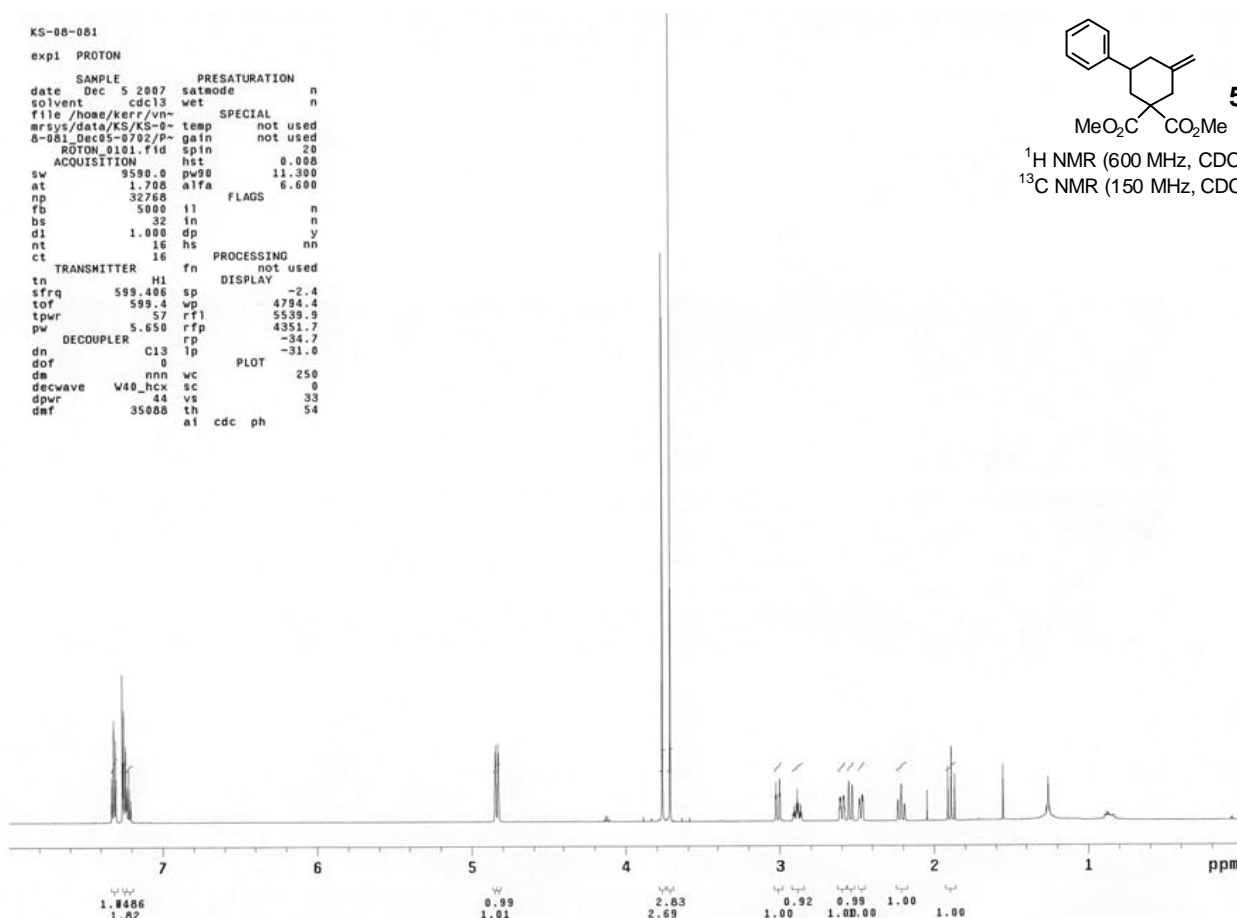
KS-08-081
exp1  PROTON
SAMPLE      PRESATURATION
date Dec 5 2007 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
mrsys/data/KS/KS-0- temp not used
8-081_Dec05-0702/P- gain not used
ARBON_0101.fid spin 20
ACQUISITION hst 0.008
sw 9590.0 pw90 11.300
at 1.708 alfa 6.600
np 32768 FLAGS n
fb 5000 f1 n
bs 32 in n
dl 1.000 dp y
nt 16 hs nn
ct 16 PROCESSING
TRANSMITTER fn not used
tn H1 DISPLAY
sfrq 599.408 sp -2.4
tof 599.4 wp 4794.4
tpwr 57 rfl 5539.9
pw 5.650 rfp 4351.7
DECOUPLER rp -34.7
dn C13 lp -31.0
dof 0 PLOT
dm nnn wc 250
decwave V40_hcx sc 0
dpwr 44 vs 33
dmf 35008 th cdc ph 54

```



5a

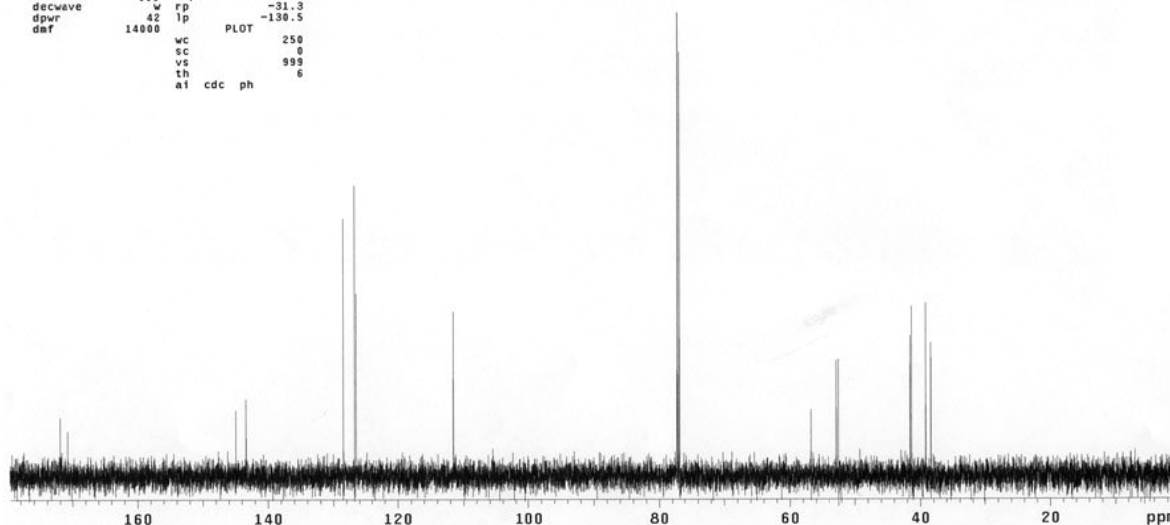
¹H NMR (600 MHz, CDCl₃)
¹³C NMR (150 MHz, CDCl₃)



```

KS-08-081
exp1  CARBON
SAMPLE      PRESATURATION
date Dec 5 2007 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
mrsys/data/KS/KS-0- temp not used
8-081_Dec05-0703/C- gain not used
ARBON_0101 spin 20
ACQUISITION hst 0.008
sw 37682.5 pw90 23.300
at 0.870 alfa 10.000
np 65536 FLAGS n
fb 21000 f1 n
bs 64 in n
dl 1.000 dp y
nt 256 hs nn
ct 256 PROCESSING
TRANSMITTER lb -0.59
tn C13 gf 8.261
sfrq 150.736 gfs not used
tof 2295.0 lsfid -2
tpwr 55 fn not used
pw 11.650 DISPLAY
DECOUPLER sp 1.1
dn H1 wp 27070.5
dof 0 rfl 2262.1
dm yyy rfp 0
decwave w rp -31.3
dpwr 42 lp -130.5
dmf 14000 PLOT
wc 250
sc 0
vs 999
th 6
al cdc ph

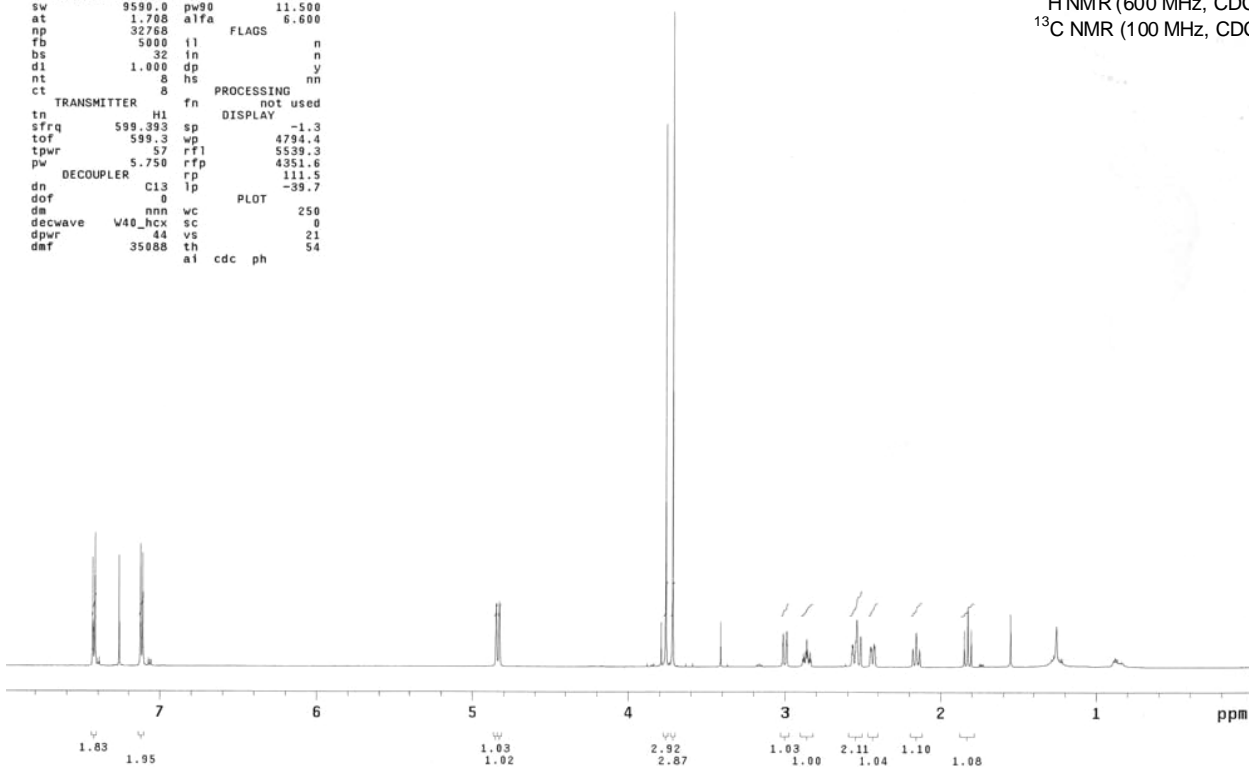
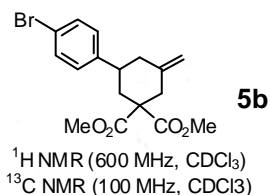
```



```

KS-09-201
expl  PROTON
SAMPLE      PRESATURATION
date May 20 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vnmr-
mrsys/data/kasia/k-
S-09-201_May20-080-
1/PROTON_0101 gain not used
ACQUISITION 0 hst 0.008
sw 9590.0 pw90 11.500
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5000 il n
bs 32 in n
d1 1.000 dp y
nt 8 hs nn
ct 8
TRANSMITTER 8 fn PROCESSING not used
tn H1 DISPLAY
sfrq 599.393 sp -1.3
tof 599.3 wp 4794.4
tpwr 57 rfl 5539.3
pw 5.750 rfp 4351.6
DECOUPLER rp 111.5
dn C13 lp -39.7
dof 0 PLOT
dm nnn wc 250
decwave V40_hcx sc 0
dpwr 44 vs 21
dmf 35088 th 54
al cdc ph

```

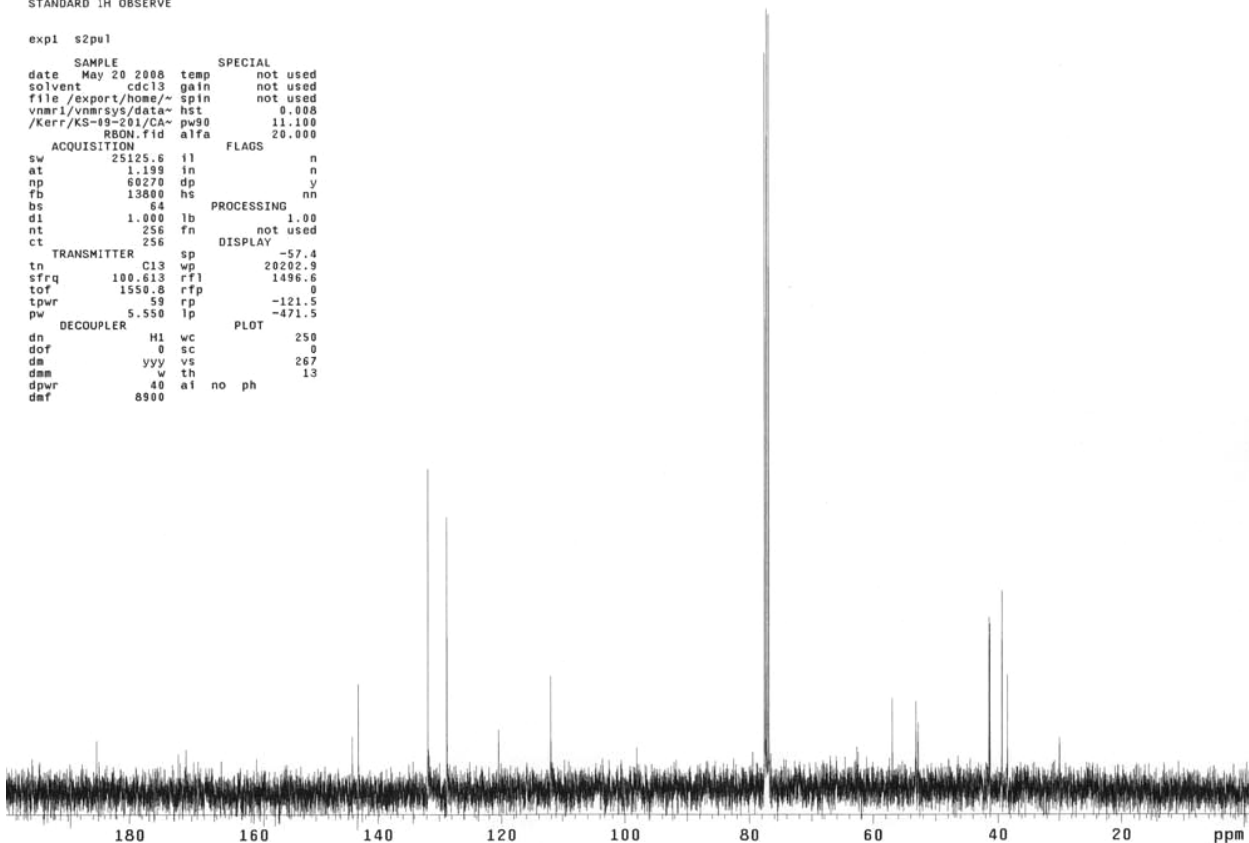


KS-09-201
STANDARD 1H OBSERVE

```

expl s2pu1
SAMPLE      SPECIAL
date May 20 2008 temp not used
solvent cdc13 gain not used
file /export/home/
vnmr1/vnmr-
mrsys/data/
Kerr/KS-09-201/CA-
RBON.fid hst 0.008
ACQUISITION 0 pw90 11.100
sw 25125.6 alfa 20.000
at 1.199 in n
np 80270 dp y
fb 13800 hs nn
bs 64 PROCESSING
d1 1.000 lb 1.00
nt 256 fn not used
ct 256 DISPLAY
TRANSMITTER 256 sp -57.4
tn C13 wp 20202.9
sfrq 100.613 rfl 1496.6
tof 1550.8 rfp 0
tpwr 59 rp -121.5
pw 5.550 lp -471.5
DECOUPLER H1 wc 250
dof 0 sc 0
dm yyy vs 267
dmw w th 13
dpwr 40 al no ph
dmf 8900

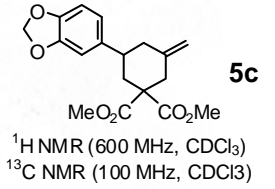
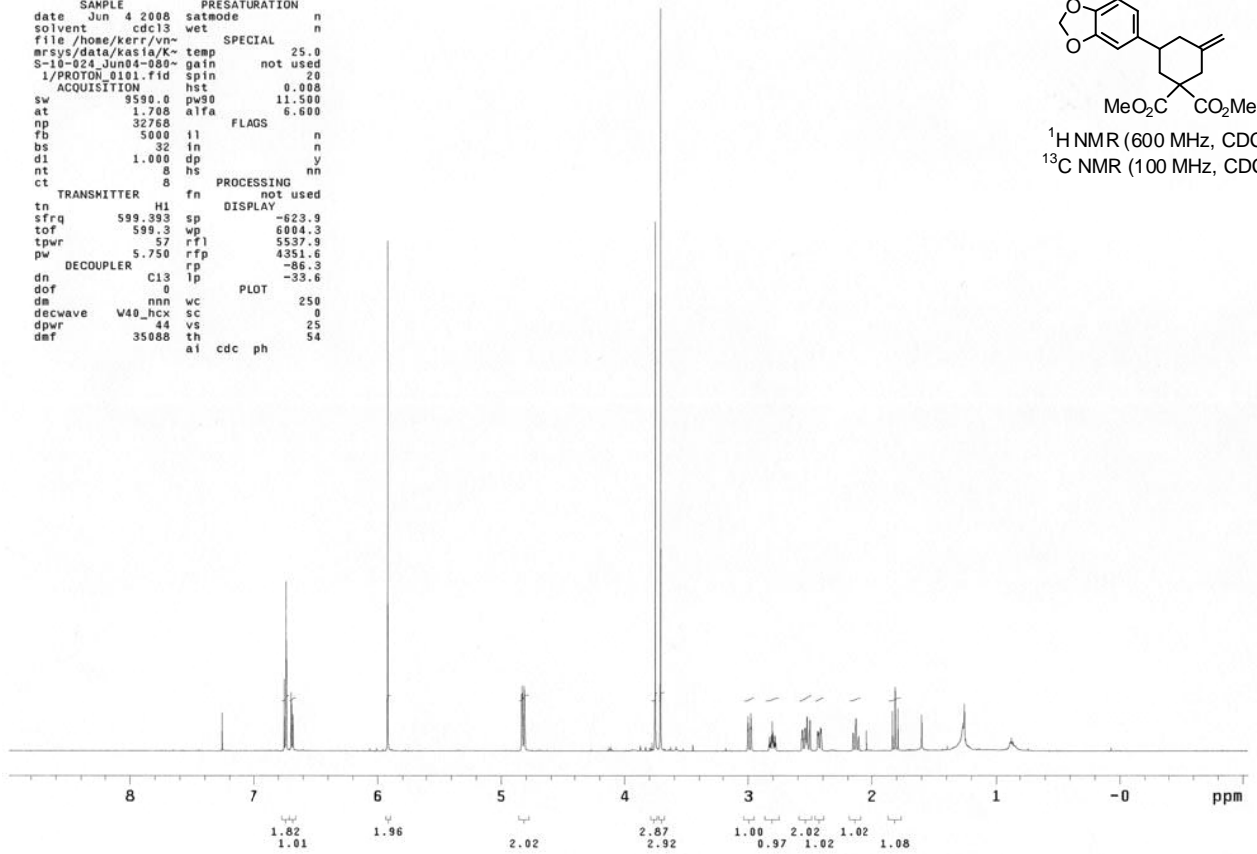
```



KS-10-024

exp1 PROTON

```
SAMPLE          PRESATURATION
date Jun 4 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
  er/sys/data/kasio/k- temp 25.0
S-10-024_Jun04-080~ gain not used
1/PROTON_0101.fid spin 20
ACQUISITION hst 0.008
sw 9500.0 pw90 11.500
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5000 il n
bs 32 in n
d1 1.000 dp y
nt 8 hs nn
ct 8 PROCESSING
tn H1 fn not used
sfrq 599.393 sp DISPLAY
tof 599.3 wp 6004.3
tpwr 57 rfl 5537.9
pw 5.750 rfp 4351.6
DECOUPLER C13 rp -86.3
dn C13 lp -33.6
dof 0 PLOT
dm nnn wc 250
decwave W40_hcx sc 0
dpwr 44 vs 25
dmf 35088 th 54
ai cdc ph
```

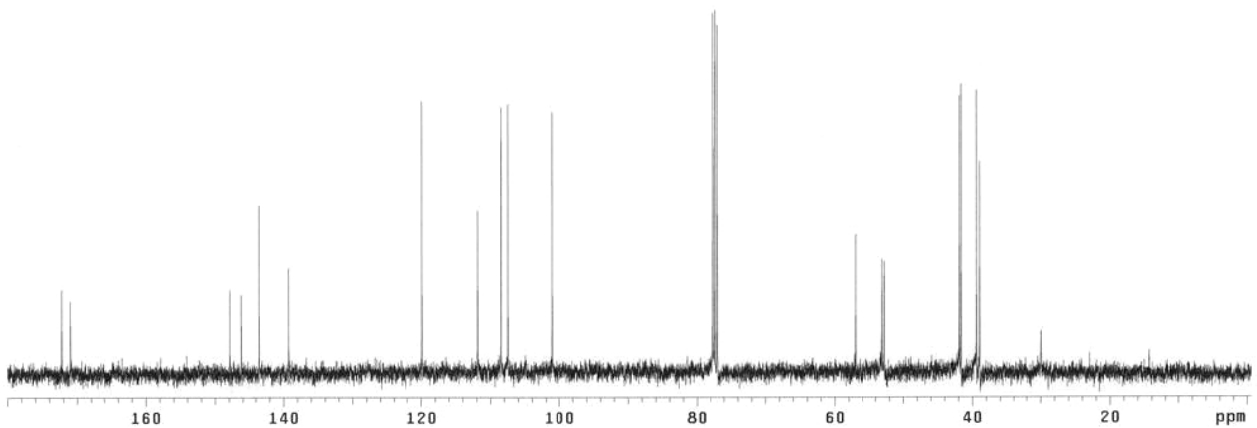


KS-10-024

STANDARD 1H OBSERVE

exp7 s2pu1

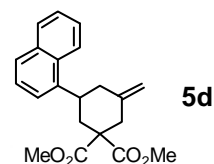
```
SAMPLE          SPECIAL
date Jun 4 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/walkup/auto~ hst 0.008
04.06.08/0103 pw90 11.100
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 64 hs nn
d1 1.000 PROCESSING
nt 256 lb 1.00
ct 256 fn not used
tn C13 sp DISPLAY
sfrq 100.613 wp 18166.2
tof 1550.8 rfl 1496.6
tpwr 59 rfp 0
pw 5.550 rp -135.7
DECOUPLER H1 lp -400.8
dn H1 wc 250
dof 0 sc 0
dm yyy vs 125
dpwr 40 th 10
dmf 8900 ai no ph
```



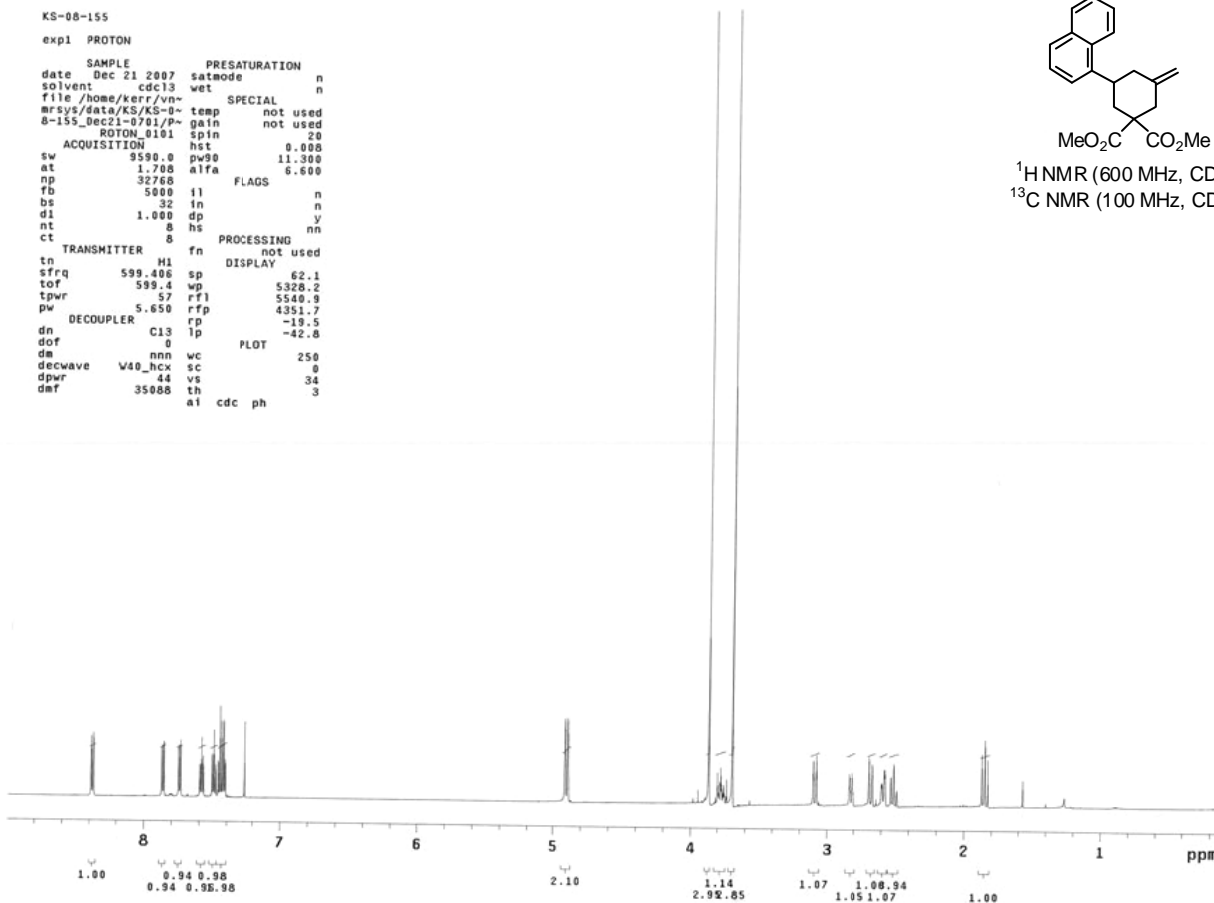
```

KS-08-155
expl  PROTON
SAMPLE          PRESATURATION
date   Dec 21 2007  satmode      n
solvent cdc13      wet           n
file /home/kerr/vn- SPECIAL
mrsvs/data/KS/KS-0- temp      not used
8-155_Dec21-0701/p- gain      not used
      ROTON_0101  spin         20
ACQUISITION    hst           0.008
sw           9590.0  pw90      11.300
at           1.708  alfa       6.600
np           32765  FLAGS
fb           5000  i1           n
bs           32    in           n
d1           1.000  dp           y
nt           8     hs           nn
ct           8     PROCESSING
TRANSMITTER    H1    fn      not used
tn             0     DISPLAY
sfrq          599.406 sp        62.1
tof           599.4  wp        5328.2
tpwr          57    rf1        5540.9
pw           5.650  rfp        4351.7
DECOUPLER      C13  lp        -19.5
dn            0     PLOT
dof           0     wc        250
dm            nnn  sc         0
decwave       V40_hcx vs         34
dpwr          44  th         3
dof           35088  ai      cdc ph

```



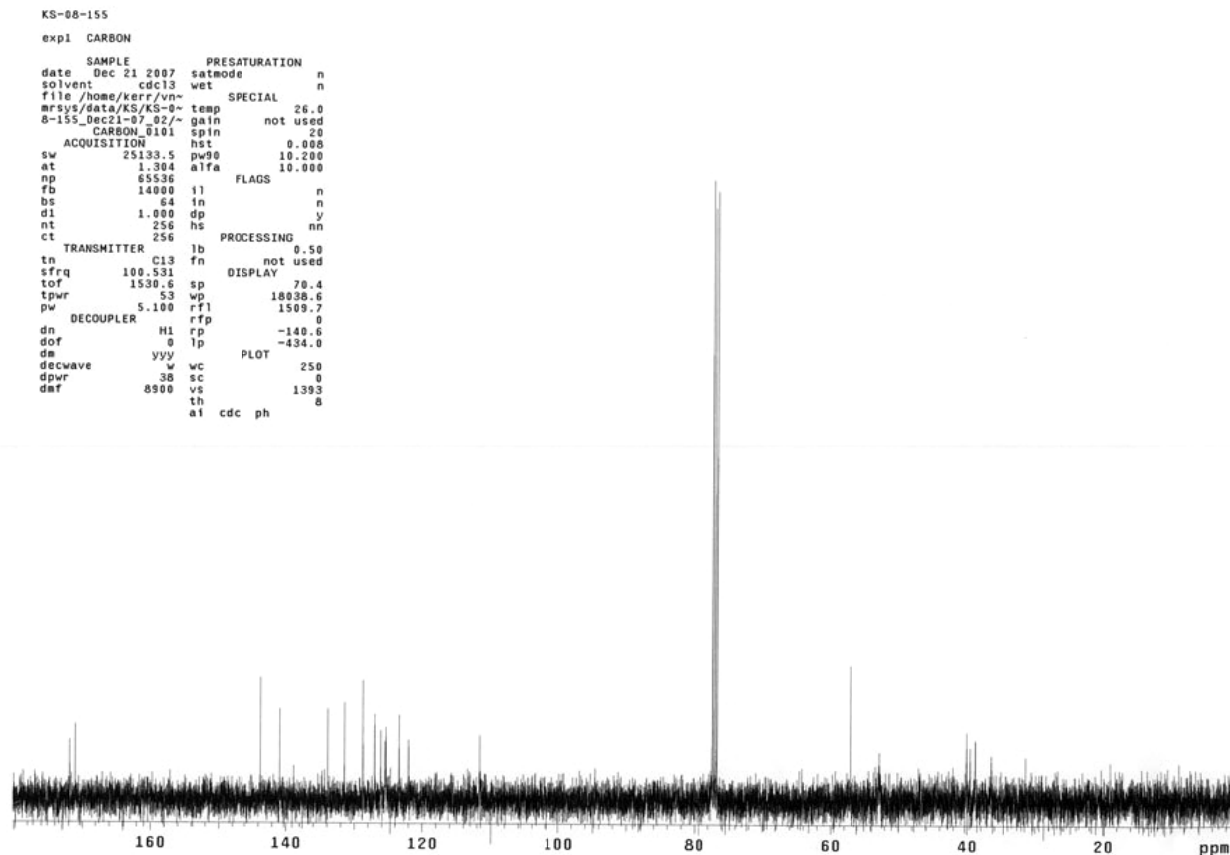
¹H NMR (600 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



```

KS-08-155
expl  CARBON
SAMPLE          PRESATURATION
date   Dec 21 2007  satmode      n
solvent cdc13      wet           n
file /home/kerr/vn- SPECIAL
mrsvs/data/KS/KS-0- temp      26.0
8-155_Dec21-07_02/- gain      not used
      CARBON_0101  spin         20
ACQUISITION    hst           0.008
sw           25133.5  pw90      10.200
at           1.304  alfa       10.000
np           65536  FLAGS
fb           14000  i1           n
bs           64    in           n
d1           1.000  dp           y
nt           256   hs           nn
ct           256   PROCESSING
TRANSMITTER    C13  fb      0.50
tn             0     fn      not used
sfrq          100.531 sp        70.4
tof           1530.6  wp        18038.6
tpwr          53    rf1        1509.7
pw           5.100  rfp         0
DECOUPLER      H1    rp        -140.6
dn            0     lp        -434.0
dof           0     PLOT
dm            yyy  wc        250
decwave       w   sc         0
dpwr          30  vs         1393
dof           8900  th         8
dof           ai      cdc ph

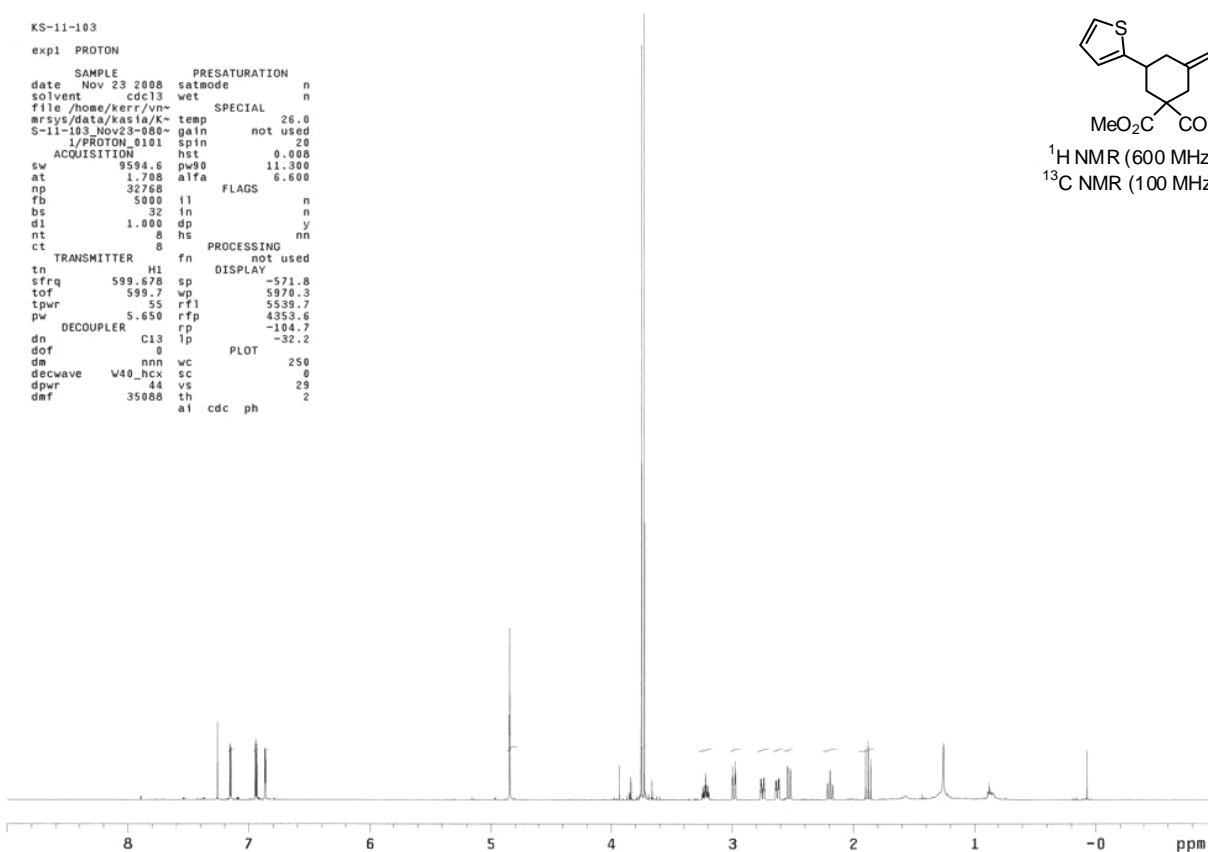
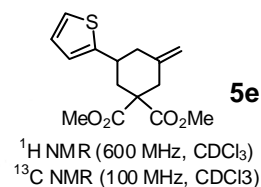
```



```

KS-11-103
expl  PROTON
      SAMPLE      PRESATURATION
date  Nov 23 2008  satmode      n
solvent  cdcl3  wet          n
file  /home/kerr/vn~ SPECIAL  26.0
mrsys/data/kas1a/K~ gain      not used
S-11-103_Nov23-080- 1/PROTON_0101 spin      20
ACQUISITION  hst      0.008
sw  9594.6  pw90     11.300
at  1.708  alfa     6.600
np  32768  FLAGS
fb  5000  il      n
bs  32  in      n
dl  1.000  dp      y
nt  8  hs      nn
ct  6  fn      not used
      TRANSMITTER  H1  DISPLAY
tn  599.678  sp      -571.8
sfrq  599.7  wp      5970.3
tpwr  55  rfl      5599.7
pw  5.650  rfp      4353.6
      DECOUPLER  C13  lp      -104.7
dn  C13  lp      -32.2
dof  0  PLOT
dm  nnn  wc      250
decave  V40_hcx  sc      0
dpwr  44  vs      29
dmf  35088  th      2
      ai  cdc  ph

```

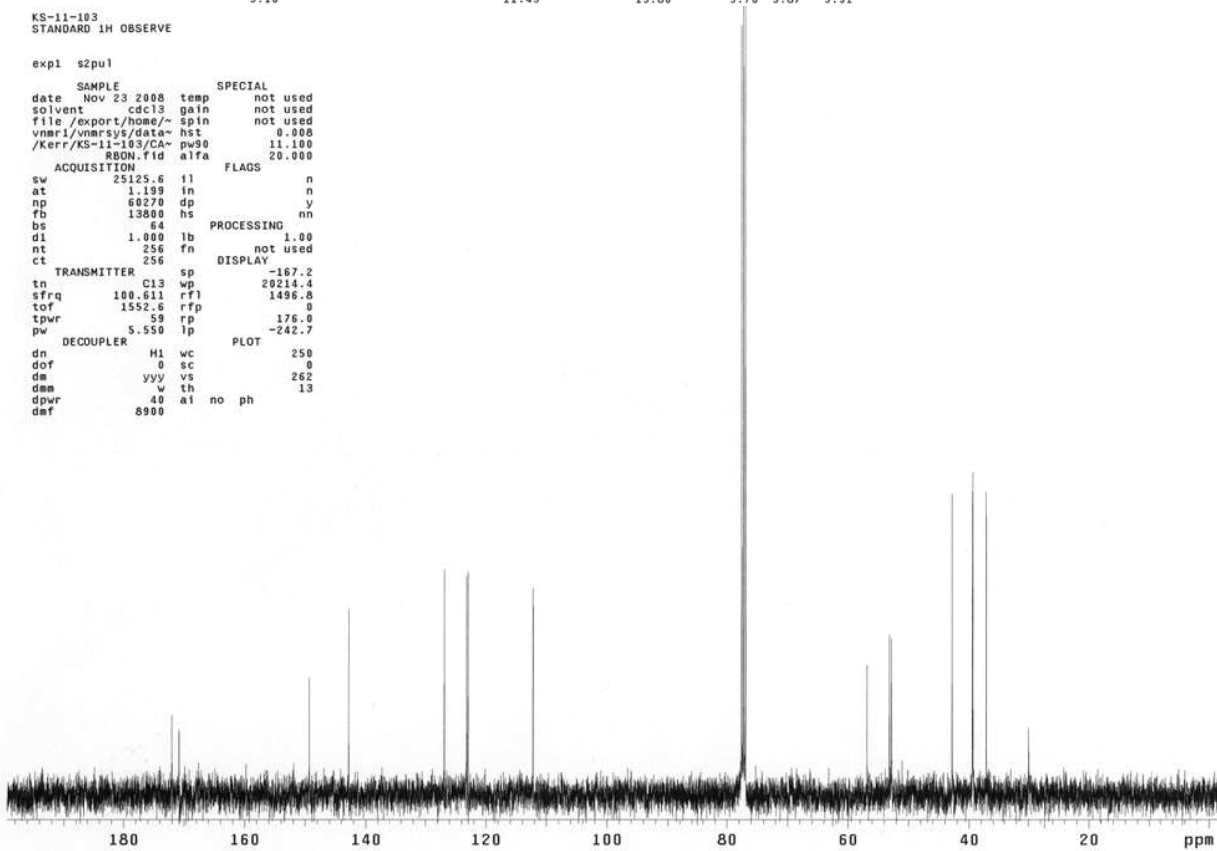


KS-11-103
STANDARD 1H OBSERVE

```

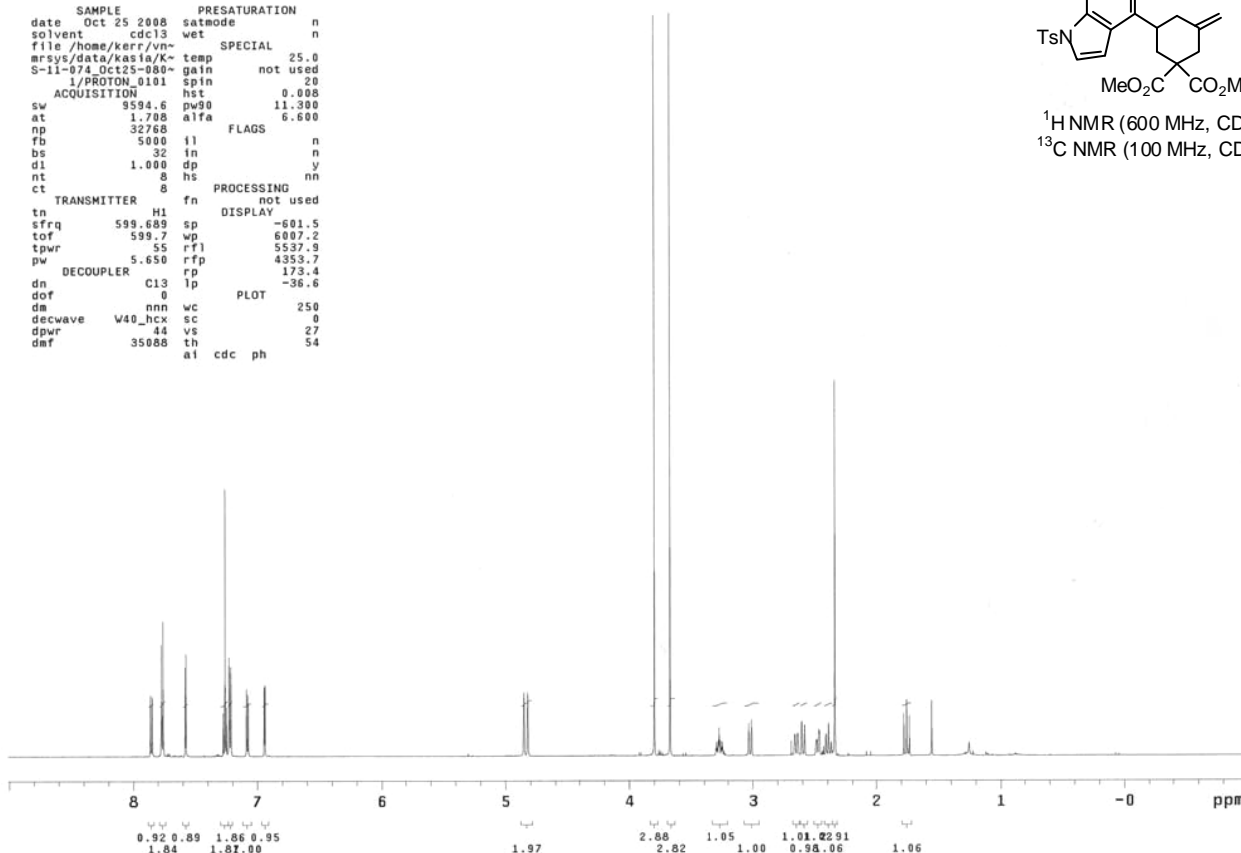
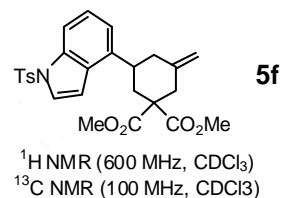
expl  s2pul
      SAMPLE      SPECIAL
date  Nov 23 2008  temp      not used
solvent  cdcl3  gain      not used
file  /export/home/~ vnmr1/vnmrsys/data~ hst      0.008
/Kerr/KS-11-103/CA~ /Kerr/KS-11-103/CA~ pw90     11.100
      RBN.fid  alfa     20.000
ACQUISITION  FLAGS
sw  25125.6  fl      n
at  1.199  in      n
np  60276  dp      y
fb  13800  hs      nn
bs  64  PROCESSING
dl  1.000  lb      1.00
nt  256  fn      not used
ct  256  DISPLAY
      TRANSMITTER  C13  sp      -167.2
tn  C13  wp      20214.4
sfrq  100.611  rfl      1496.8
tof  1552.6  rfp      0
tpwr  59  rfp      176.0
pw  5.550  lp      -242.7
      DECOUPLER  H1  wc      250
dn  H1  wc      0
dof  0  sc      262
dm  w  vs      13
dwa  w  th
dpwr  40  ai  no  ph
dmf  8900

```



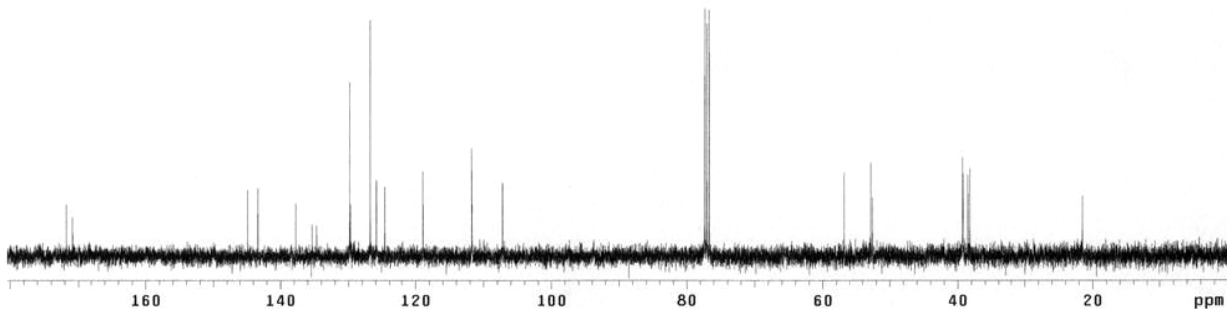
KS-11-074

```
expl PROTON
SAMPLE PRESATURATION
date Oct 25 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn~ SPECIAL 25.0
mrsys/data/kas1a/K~ temp not used
S-11-074_Oct25-080~ gain not used
1/PROTON_0101 spin 20
ACQUISITION hst 0.008
sw 9594.6 pw90 11.300
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5000 il n
bs 32 in n
d1 1.000 dp y
nt 8 hs nn
ct 8
TRANSMITTER H1 fn not used
tn H1 DISPLAY
sfrq 599.689 sp -601.5
tof 599.7 wp 6007.2
tpwr 55 rfl 5537.9
pw 5.650 rfp 4353.7
DECOUPLER rp 173.4
dn C13 lp -36.6
dof 0 PLOT
dm nnn wc 250
decwave W40_hcx sc 0
dpr 44 vs 27
dmf 35088 th 54
ai cdc ph
```



KS-09-049

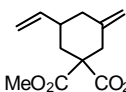
```
expl CARBON
SAMPLE PRESATURATION
date Feb 29 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn~ SPECIAL 26.0
mrsys/data/kas1a/K~ temp not used
S-09-049_Feb29-08~ gain not used
02/CARBON_0101 spin not used
ACQUISITION hst 0.008
sw 25133.5 pw90 9.900
at 1.304 alfa 10.000
np 65536 FLAGS
fb 14000 il n
bs 64 in n
d1 1.000 dp y
nt 256 hs nn
ct 256
TRANSMITTER H1 fn 0.50
tn C13 DISPLAY
sfrq 100.531 sp -17.8
tof 1530.6 wp 18152.9
tpwr 53 rfl 1509.7
pw 4.950 rfp 35.0
DECOUPLER H1 rp -358.6
dn 0 lp
dof 0 PLOT
dm yyy wc 250
decwave 38 sc 0
dpr 8800 vs 670
dmf th 3
ai cdc ph
```



KS-08-201

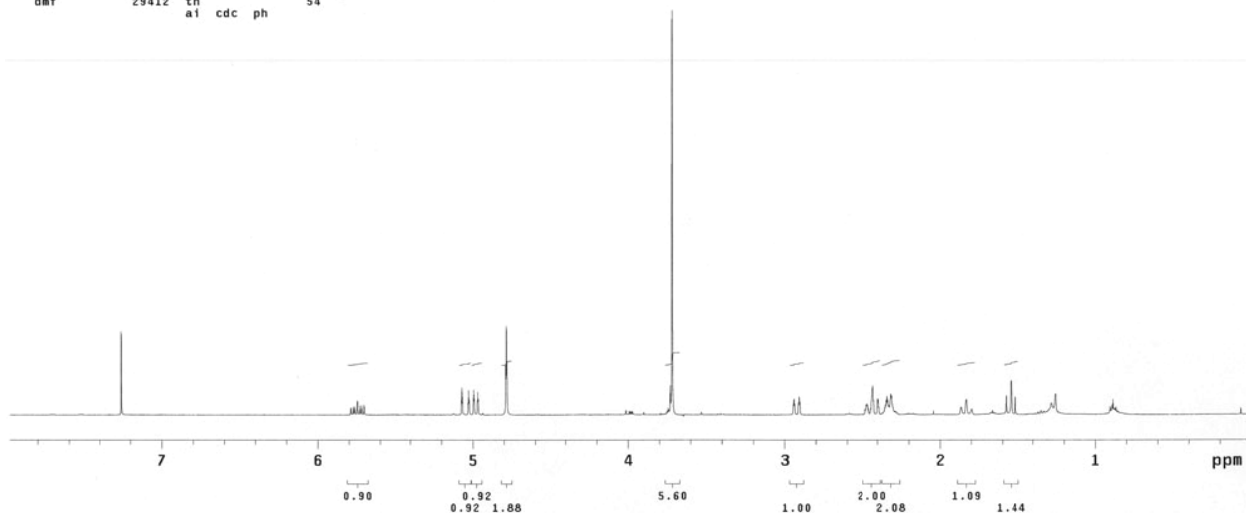
expl PROTON

```
SAMPLE      PRESATURATION
date Jan 21 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn-
mrsvs/data/kasia/k- SPECIAL 26.0
S-08-201_Jan21-08_ gain not used
01/PROTON_0101.fid spin not used
ACQUISITION hst 0.000
sw 6395.9 pw90 11.500
at 2.562 alfa 6.600
np 32768 FLAGS
fb 4000 il n
bs 32 in n
d1 1.000 dp y
nt 16 hs PROCESSING nn
ct 16 fn not used
TRANSMITTER H1 DISPLAY
tn 399.762 sp 3.6
tof 399.8 wp 3180.4
tpwr 57 rf1 3686.3
pw 5.750 rfp 2902.3
DECOUPLER C13 rp 79.6
dn 0 lp PLOT -34.1
dof 0 wc 250
dm nnn sc 0
decwave W40_hfcp2 vs 24
dpwr 34 vs 24
dmf 29412 th 54
ai cdc ph
```



5g

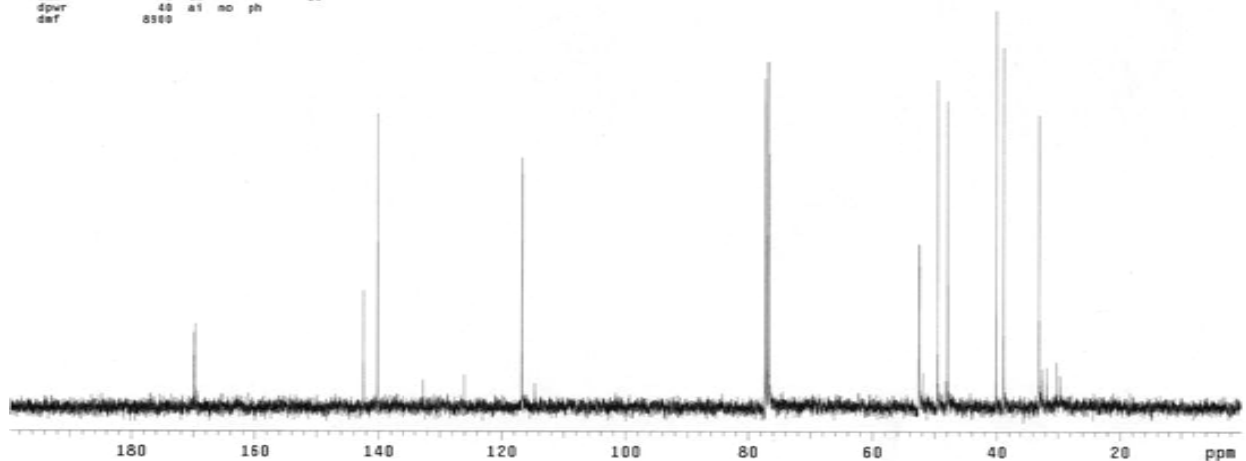
¹H NMR (400 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-2
STANDARD 1H OBSERVE

expl s2pu1

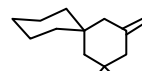
```
SAMPLE      SPECIAL
date Dec 18 2008 temp not used
solvent cdc13 gain not used
file /export/home/ vn- spin not used
vnmr1/vnmrsvs/data- hst1 8.000
/Kerr/KS-12-2/CARS- pw90 11.100
0N.fid alfa 28.000
ACQUISITION FLAGS
sw 25125.6 il n
at 1.199 sm n
np 68270 dp y
fb 13800 hs
bs 64 PROCESSING
d1 1.800 lb 1.00
nt 256 fn not used
ct 256 DISPLAY
TRANSMITTER sp 37.1
tn C13 wp 20812.8
sfrq 100.611 rf1 9258.8
tof 1552.6 rfp 7746.2
tpwr 59 rp 102.3
pw 5.550 lp PLOT -295.6
DECOUPLER H1 wc 250
dof 0 sc 0
dm 337 vs 98
dnn w th 13
dpwr 40 ai no ph
dmf 8980
```



KS-09-061A

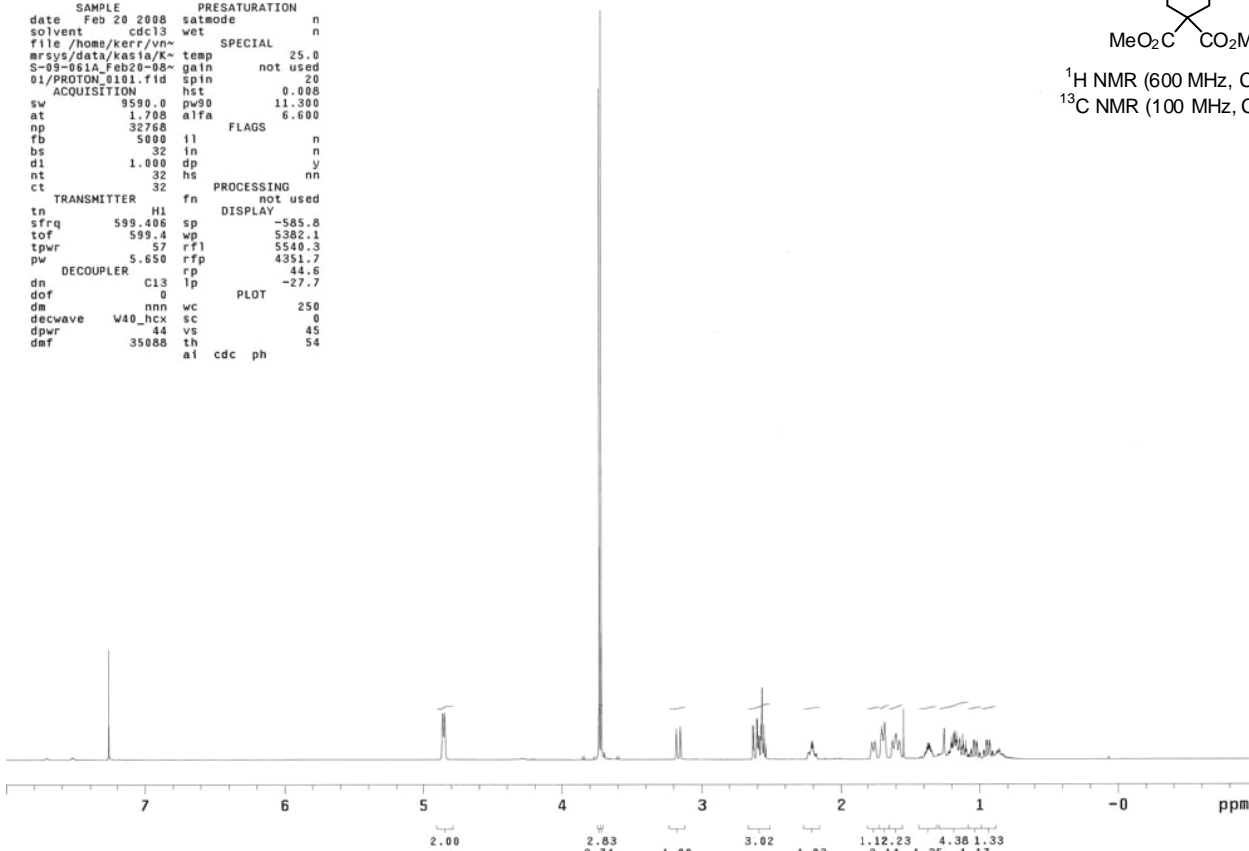
expl PROTON

```
SAMPLE          PRESATURATION
date Feb 20 2008 satmode n
solvent cdc13    wet      n
file /home/kerr/vn~ SPECIAL
mrsys/data/kerr/k~ temp 25.0
S-09-061A_Feb20-08~ gain not used
01/PROTON_0101.fid spin 20
ACQUISITION     hst      0.008
sw 9590.0       pw90     11.300
at 1.708        alfa     6.600
np 32768        FLAGS
fb 5000         il       n
bs 32           in       n
d1 1.000        dp       y
nt 32           hs       nn
ct 32           fn       not used
TRANSMITTER H1      DISPLAY
tn H1          sp       -585.8
sfrq 599.406  wp       5382.1
tpwr 57        rf1     5540.3
pw 5.650      rfp     4351.7
DECOUPLER C13    rp       44.6
dn C13        lp       -27.7
dof 0         PLOT
dm nnn        wc       250
decwave W40_hcx sc      0
dpwr 44       vs       45
dmf 35088    th       54
a1 cdc ph
```



MeO₂C CO₂Me **5h**

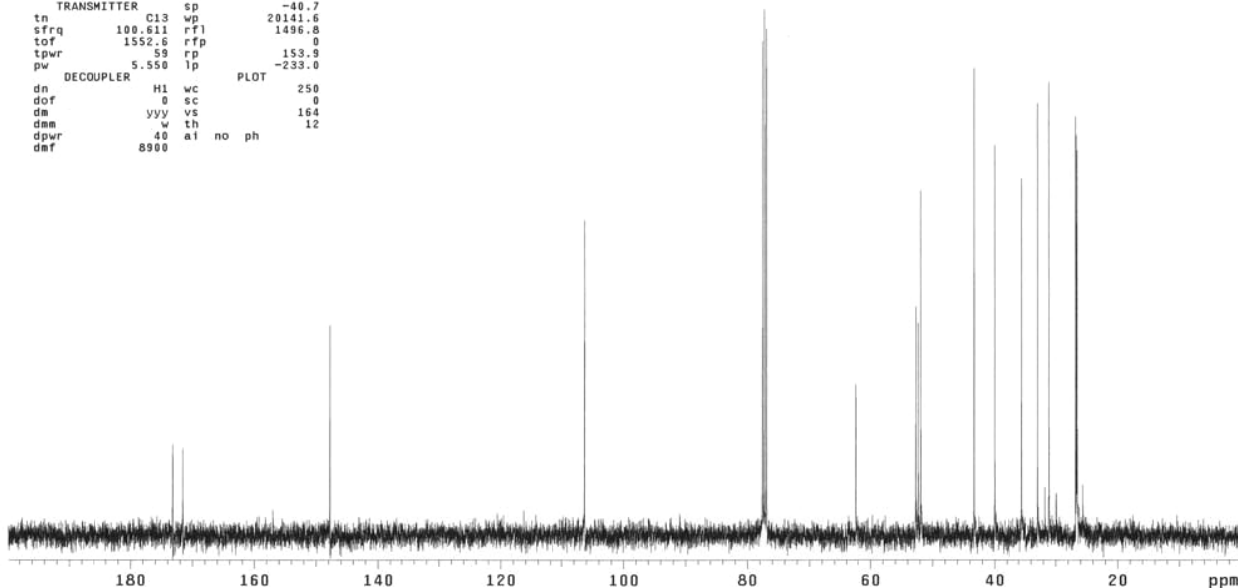
¹H NMR (600 MHz, CDCl₃)
¹³C NMR (100 MHz, CDCl₃)



KS-10-030
STANDARD 1H OBSERVE

expl s2pu1

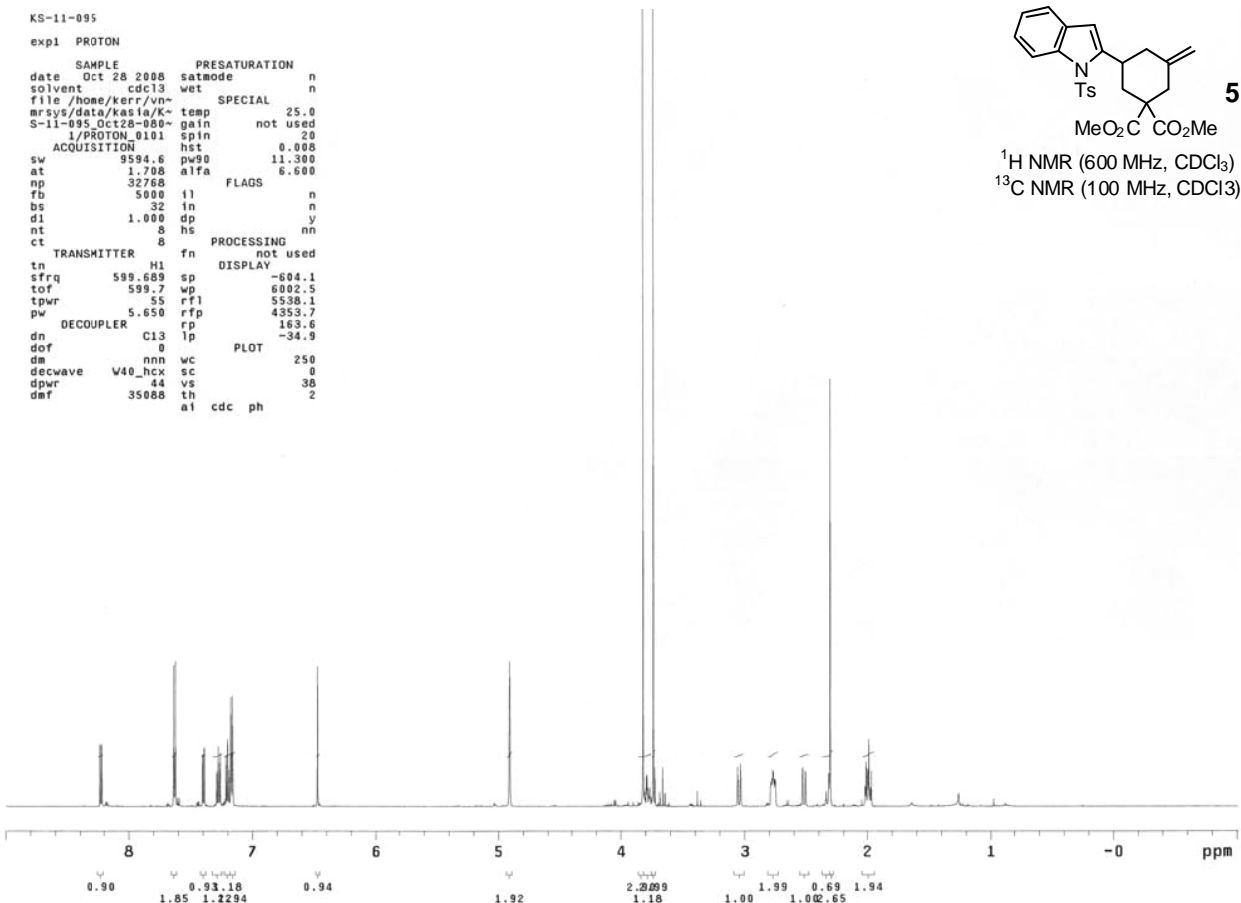
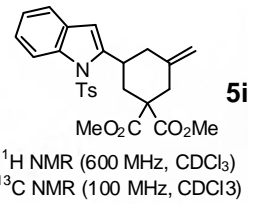
```
SAMPLE          SPECIAL
date Aug 22 2008 temp not used
solvent cdc13    gain not used
file /export/home/~ spin not used
vnmr1/vnmr/sy/data~ hst      0.008
/Kerr/KS-10-030A/C~ pw90    11.100
ARBON.fid alfa   20.000
ACQUISITION     FLAGS
sw 25125.6       il       n
at 1.199         in       n
np 60270         dp       y
fb 13800         hs       nn
bs 64           PROCESSING
d1 1.000         lb       1.00
nt 256          fn       not used
ct 256          DISPLAY
TRANSMITTER C13    sp       -40.7
tn C13         wp       20141.6
sfrq 100.611   rf1     1496.8
tof 1552.6     rfp     0
tpwr 59        rp     153.9
pw 5.550      lp     -233.0
DECOUPLER H1     wc       250
dn H1         sc       0
dof 0         dm       164
dm yyy        vs       12
dmm w         th
dpwr 40       a1 no ph
dmf 8900
```



KS-11-095

expl PROTON

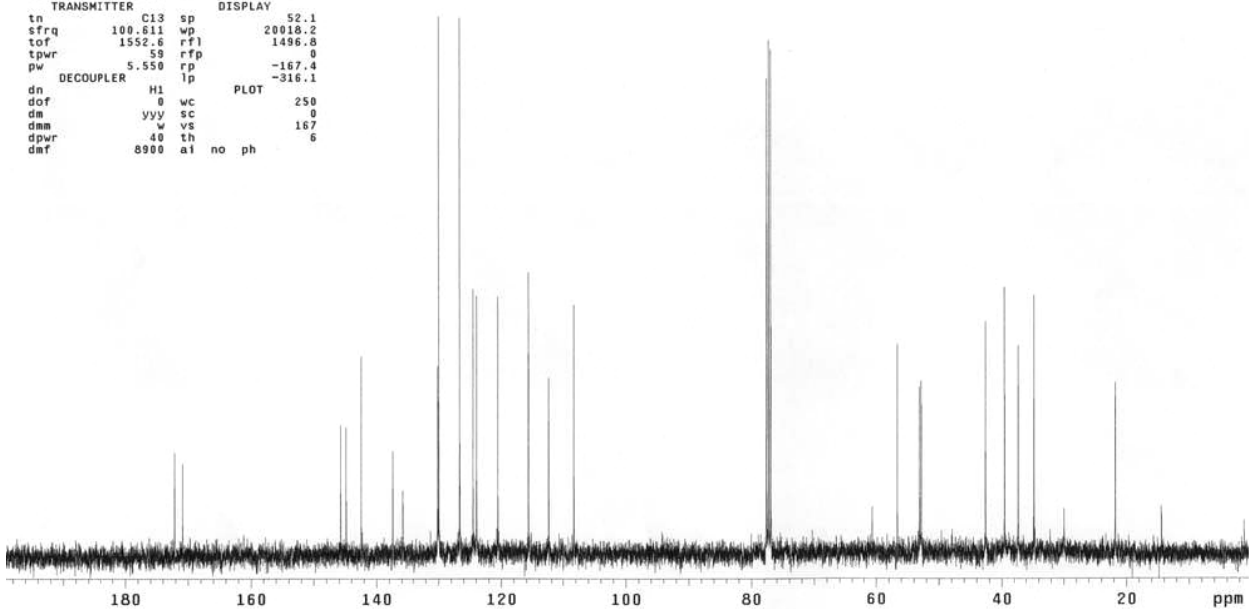
```
SAMPLE      PRESATURATION
date Oct 28 2008 satmode n
solvent cdc13 wet n
file /home/kerr/vn- SPECIAL
arsys/data/kasia/K- 25.0
S-11-095_Oct28-060- gain not used
1/PROTON_0101 spin 20
ACQUISITION hst 0.008
sw 9594.6 pw90 11.300
at 1.708 alfa 6.600
np 32768 FLAGS
fb 5000 il n
bs 32 in n
d1 1.000 dp y
nt 8 hs PROCESSING nn
ct 8 fn not used
tn H1 DISPLAY
sfrq 599.689 sp -604.1
tof 599.7 wp 6002.5
tpwr 55 rfl 5538.1
pw 5.650 rfp 4353.7
dn DECOUPLER C13 rp 163.6
dof 0 lp PLOT -34.9
dm nnn wc 250
decwave V40_hcx sc 0
dpwr 44 vs 36
dmf 35088 th 2
ai cdc ph
```



KS-11-095
STANDARD 1H OBSERVE

expl s2pu1

```
SAMPLE      SPECIAL
date Oct 27 2008 temp not used
solvent cdc13 gain not used
file /export/home/~ spin not used
vnmr1/walkup/auto_~ hst 0.008
27.18.08/0110 pw90 11.100
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 64 hs PROCESSING nn
d1 1.000 dp y
nt 256 lb not used
ct 256 fn not used
tn C13 sp DISPLAY 52.1
sfrq 100.611 wp 20018.2
tof 1552.6 rfl 1496.8
tpwr 59 rfp 0
pw 5.550 rp -167.4
dn DECOUPLER H1 lp PLOT -316.1
dof 0 wc 250
dm yyy sc 0
dmm w vs 167
dpwr 40 th 6
dmf 8900 ai no ph
```

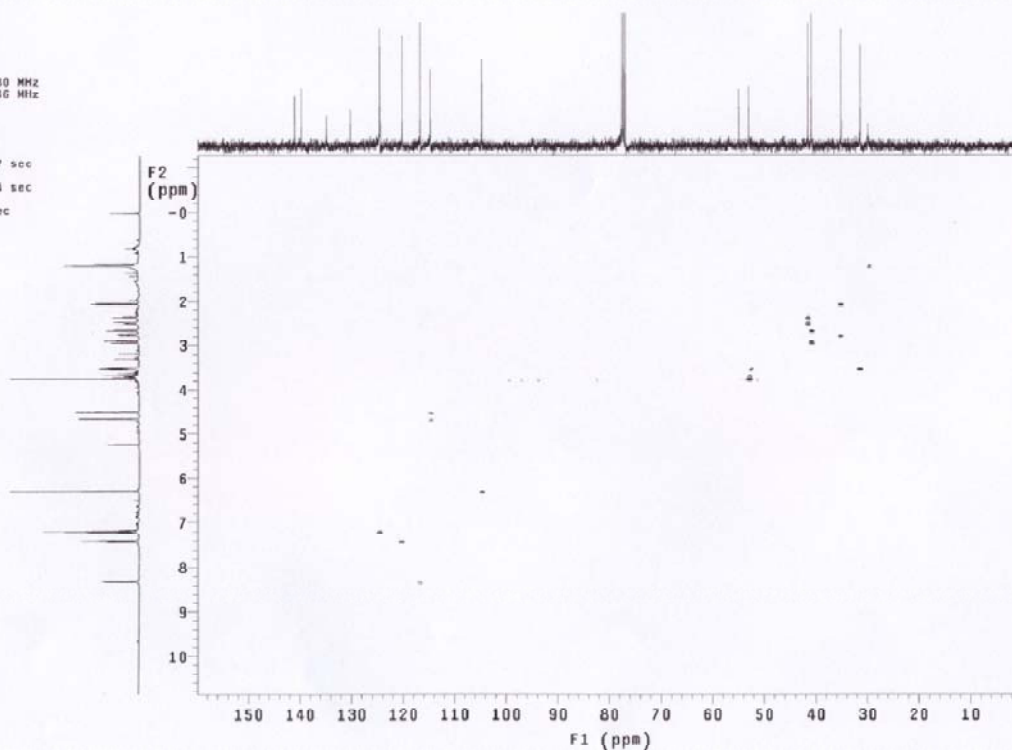


KS-11-113-2
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory: auto_11Nov2008

Pulse Sequence: gHSQC
Solvent: cdcl3
Ambient temperature
Sample #2
File: gHSQC
Mercury-400BB "nara400"

Relax. delay 1.000 sec
Acq. time 0.209 sec
Width 4992.0 Hz
2D Width 17094.0 Hz
4 repetitions
2 x 128 increments
OBSERVE M1, 400.0802880 MHz
DECOUPLE C13, 100.6077646 MHz
Power 44 dB
on during acquisition
off during delay
GARP-1 modulated
DATA PROCESSING
Gauss apodization 0.057 sec
F1 DATA PROCESSING
Gauss apodization 0.014 sec
FT size 2048 x 2048
Total time 24 min, 39 sec



KS-11-113
STANDARD 1H OBSERVE

Archive directory: /export/home/vnmr1/vnmrSYS/data
Sample directory: auto_11Nov2008

Pulse Sequence: gCOSY
Solvent: cdcl3
Ambient temperature
Sample #2
File: gCOSY
Mercury-400BB "nara400"

Relax. delay 1.000 sec
Acq. time 0.244 sec
Width 4201.7 Hz
2D Width 4201.7 Hz
Single scan
128 increments
OBSERVE M1, 400.0802884 MHz
DATA PROCESSING
Sq. sine bell 0.122 sec
F1 DATA PROCESSING
Sq. sine bell 0.020 sec
FT size 2048 x 2048
Total time 3 min, 57 sec

