Electronic Supplementary Material (ESI) for Chemical Communications

A regioselective synthesis of poly-substituted aryl triflones through self-promoting three component reaction

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Electronic Supplementary Information

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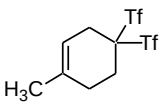
1. General and materials

All reactions were carried out under Ar atmosphere. Melting points were uncorrected. ^{1}H and ^{13}C NMR spectra were taken on a 400 MHz spectrometer, and chemical shifts were reported in parts per million (ppm) using $CHCl_3$ (7.26 ppm) in $CDCl_3$ for ^{1}H NMR, and $CDCl_3$ (77.01 ppm) for ^{13}C NMR as an internal standard, respectively. ^{19}F NMR spectra were taken on a 300 MHz spectrometer, and chemical shifts were reported in parts per million using trifluoromethylbenzene (0 ppm) as a standard. Mass spectra were recorded by an electrospray ionization-time of flight (ESI-TOF) mass spectrometer or an EI mass spectrometer. Column chromatography was performed on neutral silica gel (75-150 μm). Medium pressure liquid chromatography (MPLC) was performed using 40 x 300 mm i. d. pre-packed column (silica gel, 50 μm) with UV or RI detector. Tf_2CH_2 **1** was supplied from Central Glass Co. and this material can be also prepared by the Waller's procedure in the laboratory.[1] $Tf_2CHCH_2CHTf_2$ **2** was prepared from Tf_2CH_2 by the reported procedure.[2] Paraformaldehyde was purchased from Tokyo Chemical Industry, Co.

2. Three component synthesis of *gem*-bis(triflyl)cyclohexenes

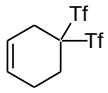
1-Methyl-4,4-bis(trifluoromethylsulfonyl)cyclohex-1-ene (3aa)



To a solution of Tf_2CH_2 **1** (150.1 mg, 0.54 mmol) in acetonitrile (0.4 mL), paraformaldehyde (90% purity, 17.9

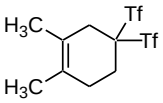
mg, 0.54 mmol) and isoprene (107.4 μL, 1.07 mmol) were added at room temperature. After being stirred at the same temperature for 2.5 h, the reaction mixture was evaporated. The resultant residue was purified by column chromatography on neutral silica gel (hexane/EtOAc = 20 : 1) to give the Diels–Alder adduct **3aa** in 76% yield (147.2 mg, 0.41 mmol). Likewise, the reaction of Tf_2CH_2 **1** (128.9 mg, 0.46 mmol), paraformaldehyde (90% purity, 15.3 mg, 0.46 mmol) and isoprene (92.2 μL, 0.92 mmol) in 1,2-dichloroethane (1.0 mL) was completed within 13 h at 40 °C to give the adduct **3aa** in 84% yield (139.1 mg, 0.39 mmol). Compared to the reaction in acetonitrile, the reaction in 1,2-dichloroethane resulted in the smooth conversion of starting materials without the formation of side products. Colorless oil; IR (neat) ν 3034, 2978, 2923, 2862, 1452, 1385, 1212, 1100 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.74 (3H, s), 2.34 (2H, brt, J = 6.4 Hz), 2.73 (2H, t, J = 6.4 Hz), 3.13 (2H, brs), 5.35-5.38 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.8, 23.0, 25.6, 25.8, 93.7, 113.4, 120.4 (q, $J_{C\text{-}F}$ = 332.9 Hz), 134.6; ^{19}F NMR (282 Hz, $CDCl_3$) δ –4.9 (6F, s); MS (ESI-TOF) m/z 383 $[M+Na]^+$; HRMS calcd for $C_9H_{10}F_6NaO_4S_2$ $[M+Na]^+$, 382.9822; found, 382.9827. Anal. Calcd for $C_9H_{10}F_6O_4S_2$: C, 30.00; H, 2.80. Found: C, 30.12; H, 2.89.

4,4-Bis(trifluoromethylsulfonyl)cyclohex-1-ene (3ab)



A mixture of Tf_2CH_2 **1** (1.42 g, 5.05 mmol) and paraformaldehyde (90% purity, 252.5 mg, 7.58 mmol) in 1,2-dichloroethane (10 mL) was stirred at 40 °C for 4.5 h under buta-1,3-diene atmosphere. After concentration of the reaction mixture under reduced pressure, column chromatography on neutral silica gel (hexane/EtOAc = 10 : 1) of the resultant residue gave the Diels–Alder adduct **3ab** in 99.6% yield (1.74 g, 5.03 mmol). Colorless crystals (Et_2O); Mp. 33.0-33.9 °C; IR (KBr) ν 3050, 2932, 2864, 1442, 1384, 1197, 1104 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.43-2.49 (2H, m), 2.71 (2H, t, J = 6.4 Hz), 3.15 (1H, brs), 5.68-5.72 (1H, m), 5.91-5.94 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.9, 22.3, 25.4, 93.6, 119.5, 120.4 (q, $J_{C\text{-}F}$ = 332.4 Hz), 126.6; ^{19}F NMR (282 Hz, $CDCl_3$) δ –4.7 (6F, s); MS (ESI-TOF) m/z 369 $[M+Na]^+$; HRMS calcd for $C_8H_8F_6NaO_4S_2$ $[M+Na]^+$, 368.9666; found, 368.9649. Anal. Calcd for $C_8H_8F_6O_4S_2$: C, 27.75; H, 2.33. Found: C, 27.85; H, 2.47.

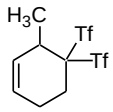
1,2-Dimethyl-4,4-bis(trifluoromethylsulfonyl)cyclohex-1-ene (3ac)



According to the synthetic procedure for **3aa**, this compound was prepared in 88% yield (149.2 mg, 0.40 mmol) by the reaction of Tf_2CH_2 **1** (127.4 mg, 0.45 mmol), paraformaldehyde (90% purity, 18.2 mg, 0.45 mmol) and 2,3-dimethylbuta-1,3-diene (102.9 μL, 0.91 mmol) in 1,2-dichloroethane (1.0 mL) at 40 °C for 6 h. Colorless oil; IR (neat) ν 2997, 2924, 2865, 1448, 1384, 1200, 1101 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.69 (3H, s), 1.71 (3H, s), 2.31-2.37 (2H, m), 2.67 (2H, t, J = 6.5 Hz), 3.00 (2H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 18.5, 18.6, 23.2, 27.1, 30.7, 95.1, 119.0, 120.4 (q, $J_{C\text{-}F}$ = 332.5 Hz), 126.7; ^{19}F NMR (282 Hz, $CDCl_3$) δ –5.1 (6F, s); MS (ESI-TOF) m/z 397 $[M+Na]^+$; HRMS calcd for $C_{10}H_{12}F_6NaO_4S_2$ $[M+Na]^+$, 396.9979; found,

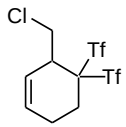
396.9952. Anal. Calcd for $C_{10}H_{12}F_6O_4S_2$: C, 32.09; H, 3.23. Found: C, 32.38; H, 3.30.

3-Methyl-4,4-bis(trifluoromethylsulfonyl)cyclohex-1-ene (3ad)



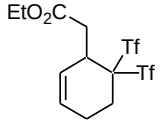
According to the synthetic procedure for **3aa**, this compound was prepared in 81% yield (127.3 mg, 0.35 mmol) by the reaction of Tf_2CH_2 **1** (122.7 mg, 0.44 mmol), paraformaldehyde (90% purity, 21.9 mg, 0.66 mmol) and (*E*)-penta-1.3-diene (87.8 μL, 0.88 mmol) in 1,2-dichloroethane (2.0 mL) at 40 °C for 8 h. Colorless oil; IR (neat) ν 3047, 2990, 2931, 2868, 1378, 1196, 1101 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.58 (3H, d, J = 7.2 Hz), 2.36-2.47 (1H, m), 2.47-2.64 (2H, m), 2.81 (1H, dt, J = 14.0, 5.0 Hz), 3.42-3.52 (1H, m), 5.51-5.57 (1H, m), 5.79-5.84 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.8, 21.9, 23.7, 34.3, 99.9, 120.3 (q, J_{C-F} = 334.7 Hz), 120.4 (q, J_{C-F} = 333.6 Hz), 124.7, 128.0; ^{19}F NMR (282 Hz, $CDCl_3$) δ –4.8 (3F, s), –4.7 (3F, s); MS (ESI-TOF) *m/z* 383 $[M+Na]^+$; HRMS calcd for $C_9H_{10}F_6NaO_4S_2$ $[M+Na]^+$, 382.9822; found, 382.9859.

3-(Chloromethyl)-4,4-bis(trifluoromethylsulfonyl)cyclohex-1-ene (3ae)



According to the synthetic procedure for **3aa**, this compound was prepared in 91% yield (132.1 mg, 0.34 mmol) by the reaction of Tf_2CH_2 **1** (103.3 mg, 0.37 mmol), paraformaldehyde (18.5 mg, 0.55 mmol) and (*E*)-5-chloropenta-1,3-diene[3] (75.6 mg, 0.74 mmol) in 1,2-dichloroethane (1.0 mL) at 40 °C for 6.5 h. Colorless crystals (hexane-EtOAc); Mp. 68.5-72.0 °C; R (KBr) ν 3051, 2932, 1383, 1205, 1102 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.40-2.62 (3H, m), 2.82-2.93 (1H, m), 3.59 (1H, brd, J = 10.6 Hz), 3.73 (1H, t, J = 10.6 Hz), 4.53 (1H, dd, J = 10.6, 2.4 Hz), 5.97-6.04 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) 22.1, 24.8, 43.0 (2C), 97.4, 120.1 (q, J_{C-F} = 334.6 Hz), 120.3 (q, J_{C-F} = 333.6 Hz), 122.7, 126.9; ^{19}F NMR (282 Hz, $CDCl_3$) δ –4.6 (3F, s), –4.5 (3F, s); MS (ESI-TOF) *m/z* 417 $[M+Na]^+$, 419 $[M+2+Na]^+$; HRMS calcd for $C_9H_9ClF_6NaO_4S_2$ $[M+Na]^+$, 416.9466; found, 416.9435. Anal. Calcd for $C_9H_9ClF_6O_4S_2$: C, 27.38; H, 2.30. Found: C, 27.38; H, 2.49.

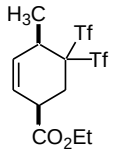
Ethyl 2-(6,6-bis(trifluoromethylsulfonyl)cyclohex-2-enyl)acetate (3af)



According to the synthetic procedure for **3aa**, this compound was prepared in 78% yield (1.71 g, 3.97 mmol) by the reaction of Tf_2CH_2 **1** (1.42 g, 5.06 mmol), paraformaldehyde (90% purity, 253 mg, 7.59 mmol) and (*E*)-ethyl hexa-3,5-dienoate[4] (851 mg, 6.07 mmol) in 1,2-dichloroethane (15 mL) at 40 °C for 3 h. The structure of this compound was also confirmed by an X-ray crystallographic analysis (See, Table S1). Colorless crystals (hexane-EtOAc); Mp. 89.0-90.8 °C; IR (KBr) ν 3047, 2987, 2942, 1738, 1379, 1196,

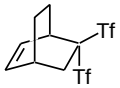
1103 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.29 (3H, t, J = 7.2 Hz), 2.38-2.61 (3H, m), 2.77 (1H, dd, J = 16.8, 11.3 Hz), 2.82-2.91 (1H, m), 3.54 (1H, dd, J = 16.8, 1.7 Hz), 3.99 (1H, brd, J = 11.3 Hz), 4.20 (2H, q), 5.51 (1H, dd, J = 10.2, 1.7 Hz), 5.81-5.89 (1H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 22.0, 25.0, 34.8, 36.4, 61.4, 97.1, 120.2 (q, J_{C-F} = 335.1 Hz), 120.3 (q, J_{C-F} = 334.1 Hz), 125.4, 125.5, 170.6; ^{19}F NMR (282 Hz, $CDCl_3$) δ –4.61 (3F, s), –4.58 (3F, s); MS (ESI-TOF) m/z 433 $[M+H]^+$; HRMS calcd for $C_{12}H_{15}F_6O_6S_2$ $[M+H]^+$, 433.0214; found, 433.0193. Anal. Calcd for $C_{12}H_{14}F_6O_4S_2$: C, 33.34; H, 3.26. Found: C, 33.52; H, 3.54.

(1*R,4*R**)-Ethyl 4-methyl-5,5-bis(trifluoromethylsulfonyl)cyclohex-2-enecarboxylate (3ag)**



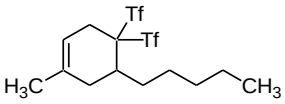
According to the synthetic procedure for **3aa**, this compound was prepared in 98% yield (212.7 mg, 0.492 mmol) by the reaction of Tf_2CH_2 **1** (141.1 mg, 0.50 mmol), paraformaldehyde (90% purity, 20.1 mg, 0.60 mmol) and ethyl sorbate (90.1 μL, 0.60 mmol) in 1,2-dichloroethane (0.5 mL) at 40 °C for 2.5 h. The relative stereochemistry was determined by NOESY spectra. Colorless oil; IR (neat) ν 2988, 2951, 1739, 1385, 1196, 1096 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.24 (3H, t, J = 7.1 Hz), 1.38 (3H, d, J = 6.9 Hz), 2.87 (1H, dd, J = 15.0, 11.1 Hz), 2.97 (1H, dd, J = 15.0, 6.3 Hz), 3.44-3.52 (1H, m), 4.16 (2H, q, J = 7.1 Hz), 5.70 (1H, ddd, J = 10.2, 4.8, 2.8 Hz), 5.86 (1H, brd, J = 10.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 19.4, 22.7, 31.8, 39.3, 62.0, 101.4, 120.56 (q, J_{C-F} = 333.9 Hz), 120.62 (q, J_{C-F} = 334.1 Hz), 123.3, 129.6, 170.5; ^{19}F NMR (282 Hz, $CDCl_3$) δ –4.5 (3F, s), –2.8 (3F, s); MS (ESI-TOF) m/z 433 $[M+H]^+$; HRMS calcd for $C_{12}H_{15}F_6O_6S_2$ $[M+H]^+$, 433.0214; found, 433.0156. Anal. Calcd for $C_{12}H_{14}F_6O_6S_2$: C, 33.34; H, 3.26. Found: C, 33.54; H, 3.33.

(1*R,4*R**)-5,5-Bis(trifluoromethylsulfonyl)bicyclo[2.2.2]oct-2-ene (3ah)**



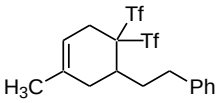
According to the synthetic procedure for **3aa**, this compound was prepared in 60% yield (111.6 mg, 0.30 mmol) by the reaction of Tf_2CH_2 **1** (139.4 mg, 0.50 mmol), paraformaldehyde (90% purity, 33.2 mg, 1.00 mmol) and cyclohexa-1,3-diene (71.2 μL, 0.75 mmol) in 1,2-dichloroethane (2.0 mL) at 80 °C for 8.5 h. Colorless crystals; Mp. 36.0-38.2 °C; IR (KBr) ν 3064, 2958, 2893, 1382, 1192, 1097 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.33-1.38 (2H, m), 1.80-1.83 (1H, m) 2.33-2.40 (2H, m), 2.62 (1H, brd, J = 15.6 Hz), 2.94-3.03 (1H, m), 3.66-3.74 (1H, m), 6.35 (1H, t, J = 7.2 Hz), 6.45 (1H, t, J = 7.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 23.6, 29.1, 31.6, 33.0, 100.6, 120.3, 120.6 (q, J_{C-F} = 333.6 Hz), 130.3, 134.8; ^{19}F NMR (282 Hz, $CDCl_3$) δ –4.3 (3F, s), –4.2 (3F, s); MS (ESI-TOF) m/z 395 $[M+Na]^+$; HRMS calcd for $C_{10}H_{10}F_6NaO_4S_2$ $[M+Na]^+$, 394.9822; found, 394.99843. Anal. Calcd for $C_{10}H_{10}F_6O_4S_2$: C, 32.26; H, 2.71. Found: C, 32.48; H, 2.77.

1-Methyl-5-pentyl-4,4-bis(trifluoromethylsulfonyl)cyclohex-1-ene (3ba)



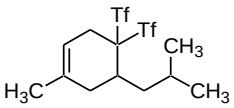
According to the synthetic procedure for **3aa**, this compound was prepared in 88% yield (178.4 mg, 0.41 mmol) by the reaction of Tf_2CH_2 **1** (130.9 mg, 0.47 mmol), hexanal (58.0 μL, 0.47 mmol) and isoprene (94.2 μL, 0.94 mmol) in 1,2-dichloroethane (1.0 mL) at room temperature for 20 h. Colorless oil; IR (neat) ν 2959, 2931, 2962, 1383, 1200, 1099 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.90 (3H, t, J = 6.7 Hz), 1.22-1.40 (5H, m), 1.47-1.58 (1H, m), 1.73 (3H, s), 1.74-1.81 (1H, m), 2.22-2.36 (2H, m), 2.51 (1H, dd, J = 18.1, 6.4 Hz), 2.73-2.85 (1H, m), 3.07 (1H, d, J = 18.5 Hz), 3.18 (1H, dd, J = 18.5, 4.2 Hz), 5.32 (1H, brs); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 22.4 (2C), 27.7, 28.6, 30.8, 31.4, 33.9, 39.9, 100.8, 112.5, 119.9 (q, $J_{C\text{-}F}$ = 333.1 Hz), 120.4 (q, $J_{C\text{-}F}$ = 334.8 Hz), 136.2; ^{19}F NMR (282 Hz, $CDCl_3$) δ –8.4 (3F, s), –3.9 (3F, s); MS (ESI-TOF) m/z 431 $[M+H]^+$; HRMS calcd for $C_{14}H_{21}F_6O_4S_2$ $[M+H]^+$, 431.0785; found, 431.0741.

(2-(3-Methyl-6,6-bis(trifluoromethylsulfonyl)cyclohex-3-enyl)ethyl)benzene (3ca)



According to the synthetic procedure for **3aa**, this compound was prepared in 89% yield (235.1 mg, 0.51 mmol) by the reaction of Tf_2CH_2 **1** (159.1 mg, 0.57 mmol), 3-phenylpropionaldehyde (74.7 μL, 0.57 mmol) and isoprene (114 μL, 1.14 mmol) in 1,2-dichloroethane (1.0 mL) at room temperature for 24 h. Colorless oil; IR (neat) ν 3027, 2920, 1381, 1206, 1100 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.73 (3H, s), 2.03-2.15 (1H, m), 2.36 (1H, d, J = 17.9 Hz), 2.52 (1H, dd, J = 17.9, 6.4 Hz), 2.61-2.92 (4H, m), 3.05 (1H, d, J = 18.2 Hz), 3.16 (1H, dd, J = 18.2, 4.6 Hz), 5.32 (1H, brs), 7.19-7.25 (3H, m), 7.28-7.39 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.5, 28.4, 32.2, 33.9, 34.0, 39.1, 100.5 (m), 112.6, 119.9 (q, $J_{C\text{-}F}$ = 333.3 Hz), 120.3 (q, $J_{C\text{-}F}$ = 334.8 Hz), 126.4, 128.4, 128.6, 136.1, 140.0; ^{19}F NMR (282 Hz, $CDCl_3$) δ –8.3 (3F, s), –3.8 (3F, s); MS (ESI-TOF) m/z 487 $[M+Na]^+$; HRMS calcd for $C_{17}H_{18}F_6NaO_4S_2$ $[M+Na]^+$, 487.0448; found, 487.0387.

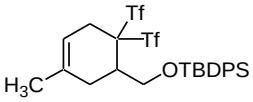
5-Isobutyl-1-methyl-4,4-bis(trifluoromethylsulfonyl)cyclohex-1-ene (3da)



According to the synthetic procedure for **3aa**, this compound was prepared in 89% yield (369.1 mg, 0.89 mmol) by the reaction of Tf_2CH_2 **1** (283.8 mg, 1.01 mmol), 3-methylbutanal (87 mg, 1.01 mmol) and isoprene (200 μL, 2.00 mmol) in 1,2-dichloroethane (2.0 mL) at room temperature for 17 h. Colorless crystals (Et_2O); Mp. 36.0-37.5 °C; IR (KBr) ν 2962, 2874, 1382, 1200, 1098 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.92 (3H, d, J = 6.6 Hz), 0.99 (3H, d, J = 6.5 Hz), 1.72 (3H, s), 1.70-1.84 (2H, m), 2.00-2.09 (1H, m), 2.29 (1H, dd, J = 18.1, 10.4 Hz), 2.47 (1H, dd, J = 18.1, 6.5 Hz), 2.90-3.00 (1H, m), 3.08 (1H, d, J = 18.4 Hz), 3.17 (1H, dd, J = 18.4, 4.3 Hz), 5.32 (1H, brs); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.4, 22.5, 24.0, 25.6, 28.5, 34.1, 37.8, 39.6, 101.1,

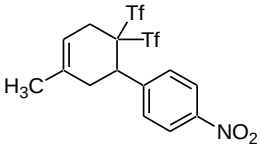
112.4, 119.9 (q, $J_{C\text{-}F}$ = 333.3 Hz), 120.4 (q, $J_{C\text{-}F}$ = 334.8 Hz), 136.2; ^{19}F NMR (282 Hz, $CDCl_3$) δ –8.4 (3F, s), –3.8 (3F, s); MS (ESI-TOF) *m*/*z* 439 $[M+Na]^+$; HRMS calcd for $C_{13}H_{18}F_6NaO_4S_2$ $[M+Na]^+$, 439.0448; found, 439.0455.

tert-Butyl((3-methyl-6,6-bis(trifluoromethylsulfonyl)cyclohex-3-enyl)methoxy)diphenylsilane (3ea)



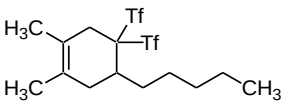
According to the synthetic procedure for **3aa**, this compound was prepared in 78% yield (244.4 mg, 0.39 mmol) by the reaction of Tf_2CH_2 **1** (139.6 mg, 0.50 mmol), $TBDPSOCH_2CHO$ (223.9 mg, 0.75 mmol) and isoprene (100 μL, 1.0 mmol) in 1,2-dichloroethane (1.0 mL) at room temperature for 20 h. Colorless oil; IR (neat) ν 3073, 2933, 2859, 1382, 1200, 1103, 702 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.16 (9H, s), 1.76 (3H, s), 2.46 (1H, dd, *J* = 18.0, 9.0 Hz), 2.84 (1H, dd, *J* = 18.0, 6.0 Hz), 3.10-3.28 (3H, m), 4.05 (1H, dd, *J* = 10.3, 9.6 Hz), 4.52 (1H, dd, *J* = 10.3, 3.3 Hz), 5.36 (1H, brs), 7.41-7.53 (6H, m), 7.69-7.77 (4H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.3, 22.6, 26.8, 28.0, 31.6, 41.4, 63.1, 99.5, 112.6, 120.0 (q, $J_{C\text{-}F}$ = 333.3 Hz), 120.4 (q, $J_{C\text{-}F}$ = 334.6 Hz), 127.8, 129.90 and 129.93, 132.8 and 133.0, 135.5, 135.8; ^{19}F NMR (282 Hz, $CDCl_3$) δ –7.8 (3F, s), –3.7 (3F, s); MS (ESI-TOF) *m*/*z* 651 $[M+Na]^+$; HRMS calcd for $C_{26}H_{30}F_6NaO_5S_2Si$ $[M+Na]^+$, 651.1106; found, 651.1059.

1-(3-Methyl-6,6-bis(trifluoromethylsulfonyl)cyclohex-3-enyl)-4-nitrobenzene (3fa)



According to the synthetic procedure for **3aa**, this compound was prepared in 55% yield (187.0 mg, 0.39 mmol) by the reaction of Tf_2CH_2 **1** (199.5 mg, 0.71 mmol), *p*-nitrobenzaldehyde (107.8 mg, 0.71 mmol) and isoprene (142.7 μL, 1.42 mmol) in 1,2-dichloroethane (1.0 mL) at room temperature for 16 h. Yellow crystals (hexane-EtOAc); Mp. 75.5-76.5 °C; IR (KBr) ν 3077, 2920, 2859, 1525, 1381, 1350, 1207, 1095 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.80 (3H, s), 2.70 (1H, dd, *J* = 18.5, 6.5 Hz), 2.80 (1H, dd, *J* = 18.5, 6.5 Hz), 3.34 (2H, s), 4.28 (1H, t, J = 6.5 Hz), 5.56 (1H, brs), 7.69 (2H, d, *J* = 8.8 Hz), 8.17 (2H, d, *J* = 8.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 22.5, 27.0, 36.8, 43.5, 100.3, 113.3, 120.1 (q, $J_{C\text{-}F}$ = 333.9 Hz), 120.4 (q, $J_{C\text{-}F}$ = 334.0 Hz), 123.8, 130.9, 135.9, 144.5, 147.9; ^{19}F NMR (282 Hz, $CDCl_3$) δ –5.7 (3F, s), –4.2 (3F, s); MS (ESI-TOF) *m*/*z* 504 $[M+Na]^+$; HRMS calcd for $C_{15}H_{13}F_6NNaO_6S_2$ $[M+Na]^+$, 503.9986; found, 503.9991. Anal. Calcd for $C_{15}H_{13}F_6NO_6S_2$: C, 37.43; H, 2.73; N, 2.91. Found: C, 37.48; H, 2.94; N, 3.01.

1,2-Dimethyl-5-pentyl-4,4-bis(trifluoromethylsulfonyl)cyclohex-1-ene (3bc)

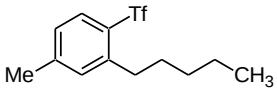


According to the synthetic procedure for **3aa**, this compound was prepared in 87% yield (630 mg, 1.41 mmol)

by the reaction of Tf_2CH_2 **1** (454.7 mg, 1.62 mmol), hexanal (198.2 μL, 1.62 mmol) and 2,3-dimethylbuta-1,3-diene (0.37 mL, 3.24 mmol) in 1,2-dichloroethane (3.0 mL) at room temperature for 16 h. Colorless oil; IR (neat) ν 2928, 2862, 1382, 1198, 1102 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.90 (3H, t, J = 6.7 Hz), 1.22-1.39 (5H, m), 1.45-1.58 (1H, m), 1.67 (3H, s), 1.68 (3H, s), 1.67-1.82 (1H, m), 2.22-2.34 (2H, m), 2.50 (1H, dd, J = 18.0, 6.1 Hz), 2.68-2.77 (1H, m), 2.94 (1H, d, J = 18.2 Hz), 3.05 (1H, d, J = 18.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 18.2, 18.3, 22.4, 27.8, 30.5, 31.4, 33.7, 35.1, 39.9, 102.0, 118.2, 119.9 (q, J_{C-F} = 333.2 Hz), 120.4 (q, J_{C-F} = 334.8 Hz), 128.0; ^{19}F NMR (282 Hz, $CDCl_3$) δ –9.3 (3F, s), –3.6 (3F, s); MS (ESI-TOF) m/z 467 $[M+Na]^+$; HRMS calcd for $C_{15}H_{22}F_6NaO_4S_2$ $[M+Na]^+$, 467.0761; found, 467.0709.

3. Synthesis of poly-substituted aryl triflones

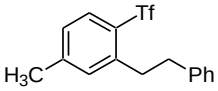
4-Methyl-2-pentyl-1-(trifluoromethylsulfonyl)benzene (5ba)



A solution of **3ba** (215.6 mg, 0.50 mmol) in degassed xylenes (2.0 mL) was stirred at 140 °C for 4 h. The reaction mixture was directly purified by column chromatography on silica gel (hexane/EtOAc = 20 : 1) to give 1-methyl-5-pentyl-4-(trifluoromethylsulfonyl)cyclohexa-1,3-diene **4ba** in 89% yield (132.8 mg, 0.45 mmol). Colorless oil; IR (neat) ν 2960, 2930, 2861, 1450, 1384, 1207, 1100 cm^{-1}; ^1H NMR (400 MHz, $CHCl_3$) δ 0.87 (3H, t, J = 7.0 Hz), 1.18-1.40 (6H, m), 1.46-1.62 (2H, m), 1.96 (3H, s), 2.36 (1H, dd, J = 18.1, 1.2 Hz), 2.55 (1H, dd, J = 18.1, 8.4 Hz), 2.65-2.75 (1H, m), 5.95-6.02 (1H, m), 7.17 (1H, d, J = 5.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 22.5, 24.2, 25.8, 30.7, 31.5, 31.9, 33.4, 118.3, 120.1 (q, J_{C-F} = 326.9 Hz), 128.9, 143.3, 149.3; ^{19}F NMR (282 Hz, $CDCl_3$) δ –16.0 (3F, s); MS (ESI-TOF) m/z 297 $[M+H]^+$; HRMS calcd for $C_{13}H_{20}F_3O_3S$ $[M+H]^+$, 297.1136; found, 297.1153.

To a solution of diene **4ba** (30.2 mg, 101 μmol) in methylcyclohexane (2.0 mL), activated MnO_2 (Aldrich, azeotropic with toluene, 88.2 mg) was added. After being stirred at 70 °C for 4 h, the mixture was filtrated through celite pad. The filtrate was evaporated and purified by column chromatography on silica gel (hexane/EtOAc = 30 : 1) to give aryl triflone **5ba** in 80% yield (23.9 mg, 81 μmol). Colorless oil; IR (neat) ν 2958, 2931, 2862, 1601, 1362, 1213, 1152, 1127, 1045, 666 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ 0.69 (3H, t, J = 8.0 Hz), 1.08-1.23 (4H, m), 1.38-1.50 (2H, m), 2.22 (3H, s), 2.75 (2H, dd, J = 8.1, 7.8 Hz), 7.14 (d, J = 8.2 Hz), 7.21 (1H, s), 7.73 (1H, d, J = 8.2 Hz); ^{13}C NMR (100 MHz, CD_3CN) δ 14.3, 21.7, 23.1, 32.5, 33.1, 34.0, 121.1 (q, J_{C-F} = 325.9 Hz), 126.5, 129.3, 134.3, 134.7, 148.2, 150.0; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.9 (3F, s); MS (ESI-TOF) m/z 317 $[M+Na]^+$; HRMS calcd for $C_{13}H_{17}F_3NaO_2S$ $[M+Na]^+$, 317.0799; found, 317.0790.

4-Methyl-2-phenethyl-1-(trifluoromethylsulfonyl)benzene (5ca)

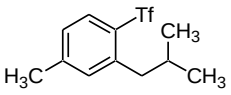


According to the synthetic procedure for **5ba**, 1-methyl-5-phenethylcyclohexa-4-(trifluoromethylsulfonyl)-1,3-diene **4ca** was obtained in 95% yield (124.5 mg, 0.38 mmol) by the thermolysis of **3ca** (183.5 mg, 0.40

mmol) in degassed xylenes (3.0 mL) for 2 h at 140 °C. Pale yellow oil; IR (neat) ν 2929, 1568, 1360, 1211, 1118 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.89-2.08 (2H, m), 2.02 (3H, s), 2.49 (1H, d, J = 18.1 Hz), 260-2.72 (2H, m), 2.78 (1H, ddd, J = 13.5, 9.9, 5.8 Hz), 2.84-2.92 (1H, m), 6.09 (1H, brs), 7.20-7.31 (4H, m), 7.31-7.42 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.2, 31.8, 32.2, 32.5, 33.5, 118.5, 120.1 (q, J_{C-F} = 326.8 Hz), 126.0, 128.3 (2C), 128.4, 141.2, 143.6, 149.5; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.9 (3F, s); MS (ESI-TOF) m/z 353 $[M+Na]^+$; HRMS calcd for $C_{16}H_{17}F_3NaO_2S$ $[M+Na]^+$, 353.0799; found, 353.0761.

The above cyclohexadiene **4ca** (66.1 mg, 0.20 mmol) was oxidized to aryl triflone **5ca** in 82% yield (53.9 mg, 0.16 mmol) by treating with activated MnO_2 (Aldrich, azeotropic with toluene, 175 mg) in methylcyclohexane (2.0 mL) for 4 h at 70 °C. Colorless crystals (Et_2O); Mp. 68.5-70.0 °C; IR (KBr) ν 3033, 2926, 1600, 1360, 1200, 1116, 1045, 655 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.49 (1H, s), 3.01 (1H, dd, J = 8.6, 7.9 Hz), 3.33 (1H, dd, J = 8.6, 7.9 Hz), 7.24-7.60 (7H, m), 8.04 (1H, d, J = 8.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.7, 36.1, 39.0, 120.1 (q, J_{C-F} = 326.8 Hz), 126.3, 126.4, 128.3, 128.5, 128.6, 133.6, 133.8, 141.0, 145.8, 148.0; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.9 (3F, s); MS (ESI-TOF) m/z 351 $[M+Na]^+$; HRMS calcd for $C_{16}H_{15}F_3NaO_2S$ $[M+Na]^+$, 351.0643; found, 351.0638.

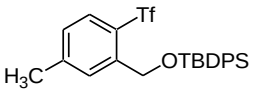
2-Isobutyl-4-methyl-1-(trifluoromethylsulfonyl)benzene (5da)



According to the synthetic procedure for **5ba**, 5-isobutyl-1-methyl-4-(trifluoromethylsulfonyl)cyclohexa-1,3-diene **4da** was obtained in 98% yield (141.9 mg, 0.50 mmol) by thermolysis of **3da** (212.4 mg, 0.51 mmol) in degassed xylenes (3.0 mL) for 3 h at 140 °C. Pale yellow oil; IR (neat) ν 2959, 2872, 1568, 1359, 1214, 1129 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.89 (6H, d, J = 6.5 Hz), 1.26 (1H, ddd, J = 13.4, 10.1, 3.5 Hz), 1.54 (1H, ddd, J = 13.4, 10.9, 3.9 Hz), 1.56-1.68 (1H, m), 1.95 (3H, s), 2.34 (1H, d, J = 17.9 Hz), 2.52 (1H, dd, J = 17.9, 7.9 Hz), 2.73-2.81 (1H, m), 5.99 (1H, brs), 7.15 (1H, d, J = 5.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.9, 23.7, 24.3, 24.6, 29.9, 33.1, 38.9, 118.4, 120.1 (q, J_{C-F} = 326.7 Hz), 129.3, 143.2, 149.1; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.9 (3F, s); MS (ESI-TOF) m/z 283 $[M+H]^+$; HRMS calcd for $C_{12}H_{18}F_3O_2S$ $[M+H]^+$, 283.0980; found, 283.0951.

The above cyclohexadiene **4da** (54.2 mg, 192 μmol) was oxidized to aryl triflone **5da** in 83% yield (44.7 mg, 159 μmol) by treating with activated MnO_2 (Aldrich, azeotropic with toluene, 200 mg) in methylcyclohexane (2.0 mL) for 5 h at 70 °C. Colorless crystals (hexane); Mp. 55.0-58.5 °C; IR (neat) ν 3054, 2962, 2872, 1600, 1362, 1212, 1128, cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.94 (6H, d, J = 6.6 Hz), 1.96-2.09 (1H, m), 2.46 (3H, s), 2.86 (2H, d, J = 7.2 Hz), 7.23 (1H, s), 7.26 (1H, d, J = 8.2 Hz), 7.96 (1H, d, J = 8.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.7, 22.2, 30.8, 41.9, 120.0 (q, J_{C-F} = 326.6 Hz), 126.8, 128.0, 133.6, 133.9, 145.9, 147.4; ^{19}F NMR (282 Hz, $CDCl_3$) δ –16.0 (3F, s); MS (EI) m/z 280 $[M]^+$. Anal. Calcd for $C_{12}H_{15}F_3O_2S$: C, 51.42; H, 5.39. Found: C, 51.50; H, 5.38.

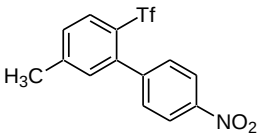
tert-Butyl(5-methyl-2-(trifluoromethylsulfonyl)benzyloxy)diphenylsilane (5ea)



According to the synthetic procedure for **5ba**, *tert*-butyl((5-methyl-2-(trifluoromethylsulfonyl)cyclohexa-2,4-dienyl)methoxy)diphenylsilane **4ea** was obtained in 85% yield (138.8 mg, 0.28 mmol) by thermolysis of **3ea** (207.5 mg, 0.33 mmol) in degassed xylenes (3.0 mL) for 4 h at 140 °C. Pale yellow oil; IR (neat) ν 3072, 2932, 2859, 1567, 1360, 1214, 1129, 703, 670 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.07 (9H, s), 1.92 (3H, s), 2.54 (1H, dd, J = 18.3, 8.6 Hz), 2.88 (1H, d, J = 18.3 Hz), 2.85-3.02 (1H, m), 3.57 (1H, t, J = 9.7 Hz), 3.78 (1H, dd, J = 9.7, 4.3 Hz), 5.92-5.97 (1H, m), 7.21 (1H, d, J = 5.9 Hz), 7.35-7.47 (6H, m), 7.60-7.66 (4H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.3, 24.0, 26.8, 31.2, 34.6, 61.9, 118.3, 120.0 (q, J_{C-F} = 326.7 Hz), 124.4, 127.71 and 127.76, 129.77 and 129.80, 133.1 and 133.3, 135.49 and 135.51, 145.5, 150.4; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.8 (3F, s); MS (ESI-TOF) m/z 495 $[M+H]^+$; HRMS calcd for $C_{25}H_{30}F_3O_3SSi$ $[M+H]^+$, 495.1637; found, 495.1592.

The above cyclohexadiene **4ea** (38.6 mg, 78 μmol) was oxidized to aryl triflone **5ea** in 80% yield (30.9 mg, 63 μmol) by treating with activated MnO_2 (Aldrich, azeotropic with toluene, 120 mg) in methylcyclohexane (1.0 mL) for 5 h at 70 °C. Colorless oil; IR (neat) ν 3072, 2959, 2932, 2859, 1598, 1364, 1214, 1125, 822, 702 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.15 (9H, s), 2.52 (3H, s), 5.20 (2H, s), 7.32 (1H, d, J = 8.1 Hz), 7.35-7.47 (6H, m), 7.64-7.69 (4H, m), 7.88 (1H, d, J = 8.1 Hz), 8.01 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.4, 22.2, 26.9, 62.1, 119.9 (q, J_{C-F} = 326.9 Hz), 124.0, 127.8, 128.4, 129.1, 129.9, 132.8, 133.2, 135.4, 144.9, 148.5; ^{19}F NMR (282 Hz, $CDCl_3$) δ –16.1 (3F, s); MS (ESI-TOF) m/z 493 $[M+H]^+$; HRMS calcd for $C_{25}H_{28}F_3O_3SSi$ $[M+H]^+$, 493.1481; found, 493.1529.

5-Methyl-4'-nitro-2-(trifluoromethylsulfonyl)biphenyl (5fa)

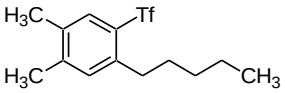


According to the synthetic procedure for **5ba**, 1-(5-methyl-2-(trifluoromethylsulfonyl)cyclohexa-2,4-dienyl)-4-nitrobenzene **4fa** was obtained in 98% yield (96.0 mg, 276 μmol) by thermolysis of **3fa** (135.2 mg, 281 μmol) in degassed xylenes (3.0 mL) for 4 h at 140 °C. Yellow crystals (hexane-Et_2O); Mp. 119–120 °C; IR (KBr) ν 3080, 2935, 1566, 1522, 1348, 1209, 1127, 855 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.87 (3H, s), 2.50 (1H, d, J = 18.3 Hz), 3.12 (1H, dd, J = 10.2 Hz), 4.12 (1H, d, J = 10.2 Hz), 6.13-6.20 (1H, m), 7.40 (2H, d, J = 8.7 Hz), 7.55 (1H, d, J = 5.9 Hz), 8.14 (2H, d, J = 8.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.0, 36.7, 38.3, 118.9, 119.8 (q, J_{C-F} = 326.4 Hz), 124.0, 126.2, 127.9, 145.7, 147.41, 147.44, 149.3; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.6 (3F, s); MS (ESI-TOF) m/z 348 $[M+H]^+$; HRMS calcd for $C_{14}H_{13}F_3NO_4S$ $[M+H]^+$, 348.0517; found, 348.0544.

The above cyclohexadiene **4fa** (95.8 mg, 276 μmol) was oxidized to aryl triflone **5fa** in 97% yield (93.3 mg, 270 μmol) by treating with activated MnO_2 (Aldrich, azeotropic with toluene, 300 mg) in methylcyclohexane (2.0 mL) for 4 h at 70 °C. Colorless crystals (acetone); Mp. 157.5-159.5 °C; IR (KBr) ν 3113, 2923, 2854, 1593, 1518, 1365, 1347, 1213, 1132 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.54 (3H, s), 7.23 (1H, s), 7.28 (2H,

d, J = 8.6 Hz), 7.54 (1H, d, J = 8.3 Hz), 8.11 (1H, d, J = 8.3 Hz), 8.25 (2H, d, J = 8.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.7, 118.6 (q, $J_{C\text{-}F}$ = 326.9 Hz), 121.6, 125.4, 129.4, 129.5, 132.5, 132.8, 142.1, 143.4, 146.8, 147.1; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.4 (3F, s); MS (ESI-TOF) m/z 368 $[M+Na]^+$; HRMS calcd for $C_{14}H_{10}F_3NNaO_4S$ $[M+Na]^+$, 368.0180; found, 368.0183.

1,2-Dimethyl-4-pentyl-5-(trifluoromethylsulfonyl)benzene (5bc)

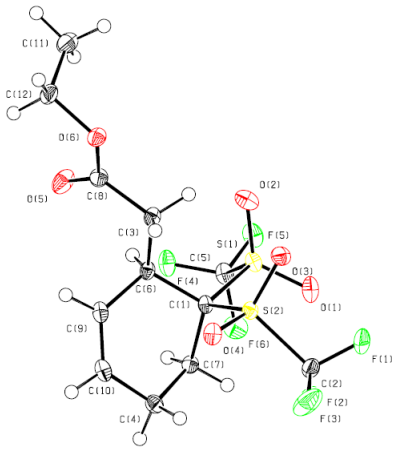


According to the synthetic procedure for **5ba**, cyclohexadiene **4bc** was obtained in 93% yield (148.2 mg, 0.48 mmol) as an inseparable mixture of 1,2-dimethyl-5-pentyl-4-(trifluoromethylsulfonyl)cyclohexa-1,3-diene and cyclohexa-1,4-diene in a ratio of 12 : 1 by thermolysis of **3bc** (228.2 mg, 0.51 mmol) in degassed xylenes (3.0 mL) for 2 h at 140 °C. Pale yellow oil; IR (neat) ν 2954, 2932, 2861, 1568, 1360, 1211, 1149, 1118 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.86 (3H, t, J = 7.0 Hz), 1.14-1.37 (6H, m), 1.44-1.54 (2H, m), 1.84 (3H, s), 1.87 (3H, s), 2.33 (1H, d, J = 17.1 Hz), 2.50-2.66 (2H, m), 7.04 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 16.7, 20.4, 22.4, 25.9, 30.6, 31.5, 31.9, 34.9, 120.1 (q, $J_{C\text{-}F}$ = 326.8 Hz), 124.1, 128.9, 141.2, 147.6; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.9 (3F, s); MS (ESI-TOF) m/z 311 $[M+H]^+$; HRMS calcd for $C_{14}H_{22}F_3O_2S$ $[M+H]^+$, 311.1293; found, 311.1306.

The above cyclohexadiene **4bc** (69.4 mg, 224 μmol) was oxidized to aryl triflone **5bc** in 81% yield (55.9 mg, 181 μmol) by treating with activated MnO_2 (Aldrich, azeotropic with toluene, 220 mg) in methylcyclohexane (2.0 mL) for 12 h at 70 °C. Colorless oil; IR (neat) ν 2956, 2929, 1490, 1215, 1200, 1119 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.91 (3H, t, J = 6.8 Hz), 1.30-1.43 (4H, m), 1.59-1.68 (2H, m), 2.31 (3H, s), 2.35 (3H, s), 2.90-2.98 (2H, m), 7.32 (1H, s), 7.79 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 19.2, 20.1, 22.4, 31.8, 32.3, 33.0, 120.1 (q, $J_{C\text{-}F}$ = 326.8 Hz), 126.1, 133.7, 133.8, 136.0, 144.7, 147.6; ^{19}F NMR (282 Hz, $CDCl_3$) δ –15.9 (3F, s); MS (EI) m/z 308 $[M]^+$, 239 $[M–CF_3]^+$. Anal. Calcd for $C_{14}H_{19}F_3O_2S$: C, 54.53; H, 6.21. Found: C, 54.65; H, 6.31.

4. X-ray crystallographic data of 3af

Crystallographic data for the X-ray crystal structure analysis of **3af** has been deposited with Cambridge Crystallographic Data Center (CCDC) as supplementary publication No. CCDC 821320. This data can be obtained free of charge from the CCDC *via* www.ccdc.cam.ac.uk/data_request/cif.



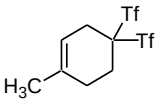
ORTEP diagram of **3af**

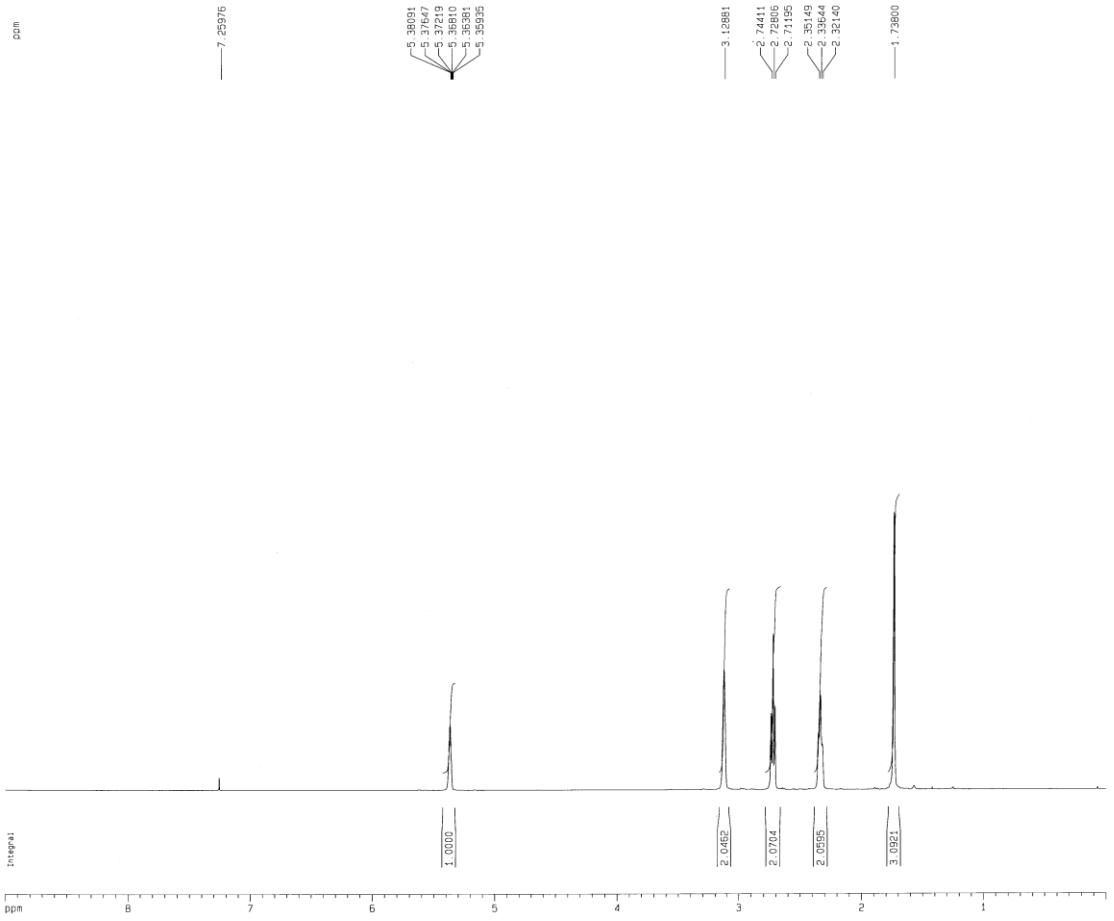
Table S1. Crystal data and structure refinement for **3af**.

Empirical formula	$C_{12}H_{14}F_6O_6S_2$	
Formula weight	432.35	
Temperature	90 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 8.1916(10) Å	α= 90°.
	b = 12.7099(16) Å	β= 97.767(2)°.
	c = 16.097(2) Å	γ = 90°.
Volume	1660.5(4) $Å^3$	
Z	4	
Density (calculated)	1.729 Mg/m^3	
Absorption coefficient	0.413 mm^{-1}	
F(000)	880	
Crystal size	0.26 x 0.23 x 0.17 mm^3	
Theta range for data collection	2.05 to 27.64°.	
Index ranges	-10<=h<=8, -13<=k<=16, -20<=l<=20	
Reflections collected	9303	
Independent reflections	3765 [R(int) = 0.0213]	
Completeness to theta = 27.64°	97.6 %	
Absorption correction	Analytical	

Max. and min. transmission	0.9331 and 0.9002
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3765 / 0 / 236
Goodness-of-fit on F^2	1.000
Final R indices [I>2sigma(I)]	R1 = 0.0317, wR2 = 0.0757
R indices (all data)	R1 = 0.0385, wR2 = 0.0798
Largest diff. peak and hole	0.647 and -0.458 e.Å^{-3}

5. ^{1}H and ^{13}C NMR spectrum



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PROCNO                     1

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PULPROG                zg30
TD                    65536
SOLVENT                CDCl3
NS                         6
DS                         2
SWH                8278.146 Hz
FIDRES             0.126314 Hz
AQ                 3.9584243 sec
RG                      40.3
DW                    60.400 usec
DE                     6.00 usec
TE                    198.2 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

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P1                     11.50 usec
PL1                    -4.00 dB
SFO1           400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                 400.0300067 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        32.00 cm
CY                         8.00 cm
F1P                    9.000 ppm
F1                  3600.27 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                 0.28125 ppm/cm
HZCM                112.50844 Hz/cm
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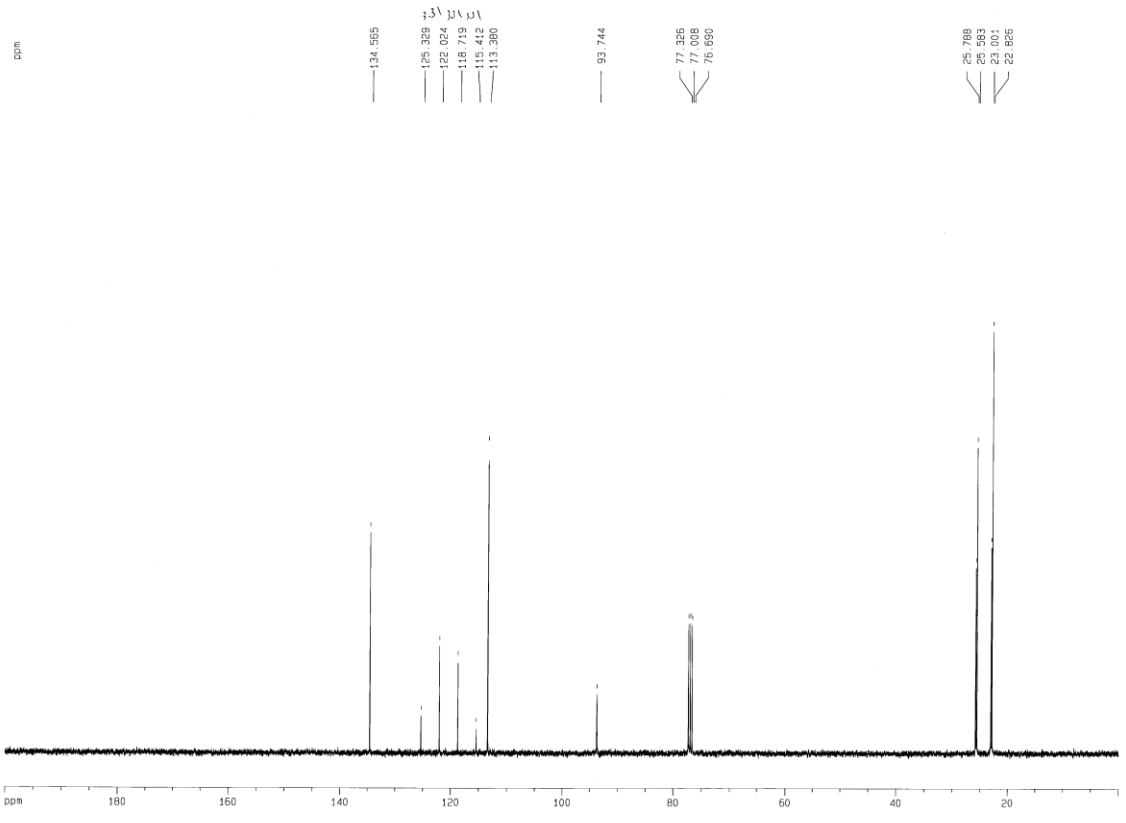


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Current Data Parameters
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EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
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PULPROG             zgpg30
TD                    65536
SOLVENT                CDCl3
NS                        54
DS                         4
SWH                26178.010 Hz
FIDRES             0.399445 Hz
AQ                 1.2517875 sec
RG                   11585.2
DW                    19.100 usec
DE                     6.00 usec
TE                    202.2 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                    -3.50 dB
SFO1           100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                    -4.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
SFO2           400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF                 100.5876240 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        32.00 cm
CY                        12.00 cm
F1P                   200.000 ppm
F1                  20117.53 Hz
F2P                   -0.000 ppm
F2                    -0.00 Hz
PPMCM                 6.25000 ppm/cm
HZCM                628.67267 Hz/cm
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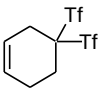

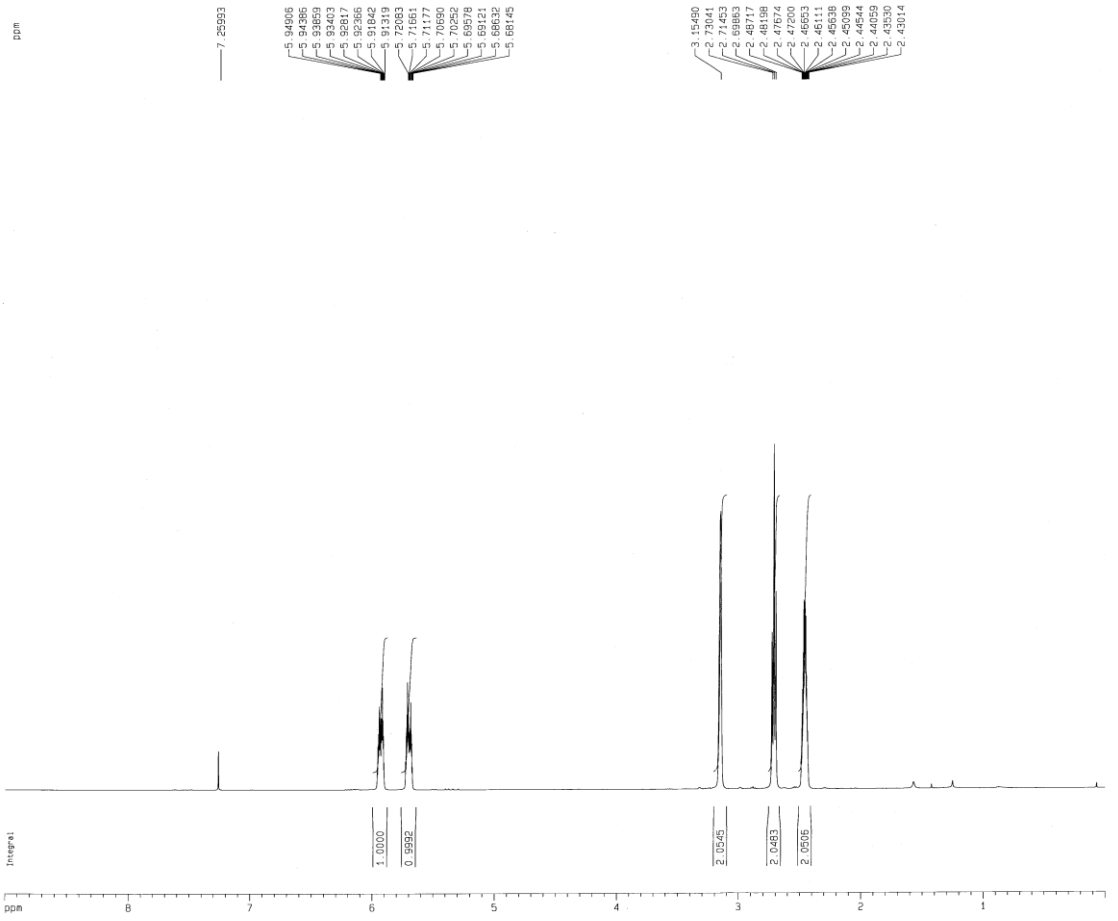

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NAME MF-119
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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Time 16.38
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 13
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 90.5
DW 60.400 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
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P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
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CY 10.00 cm
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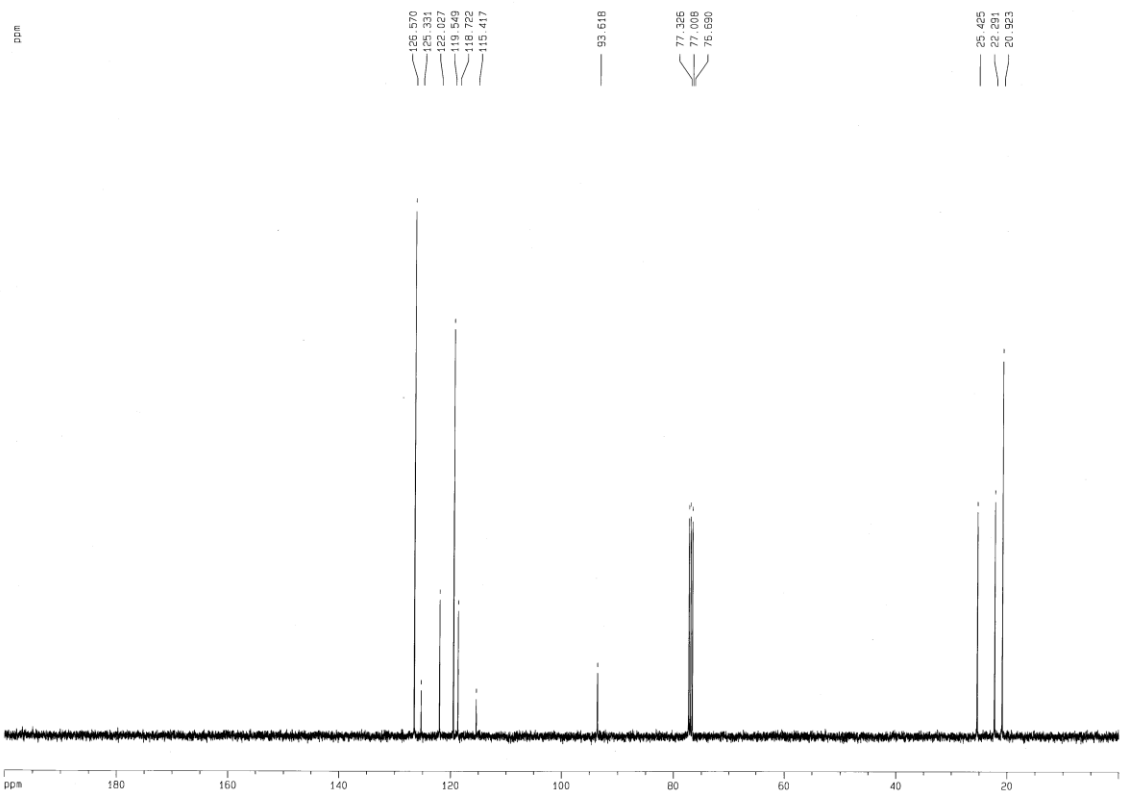
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



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EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
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Time 16.42
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 61
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876246 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
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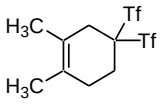
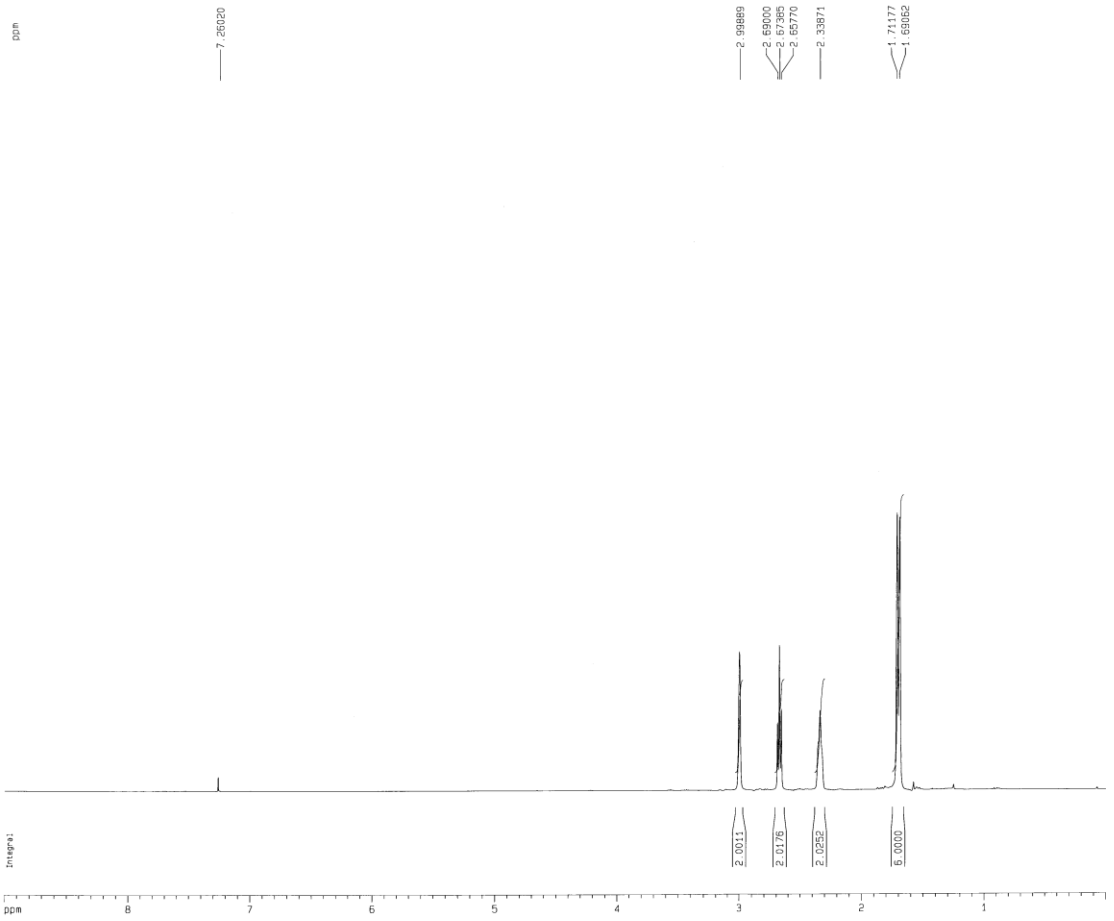
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





Current Data Parameters
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EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 40.3
DW 60.400 usec
DE 6.00 usec
TE 199.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 8.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
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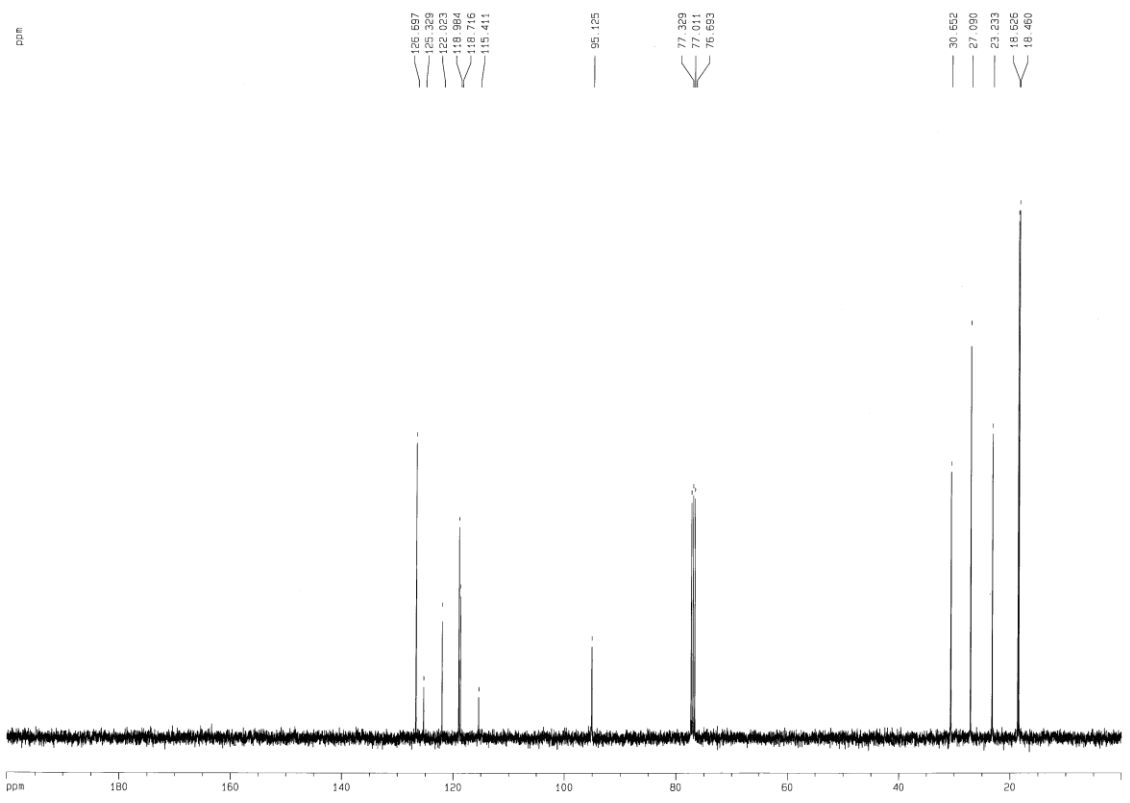

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EXPNO 2
PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 34
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 14596.5
DW 19.100 usec
DE 6.00 usec
TE 201.2 K
D1 2.00000000 sec
d11 0.03000000 sec
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MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876238 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
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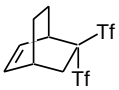
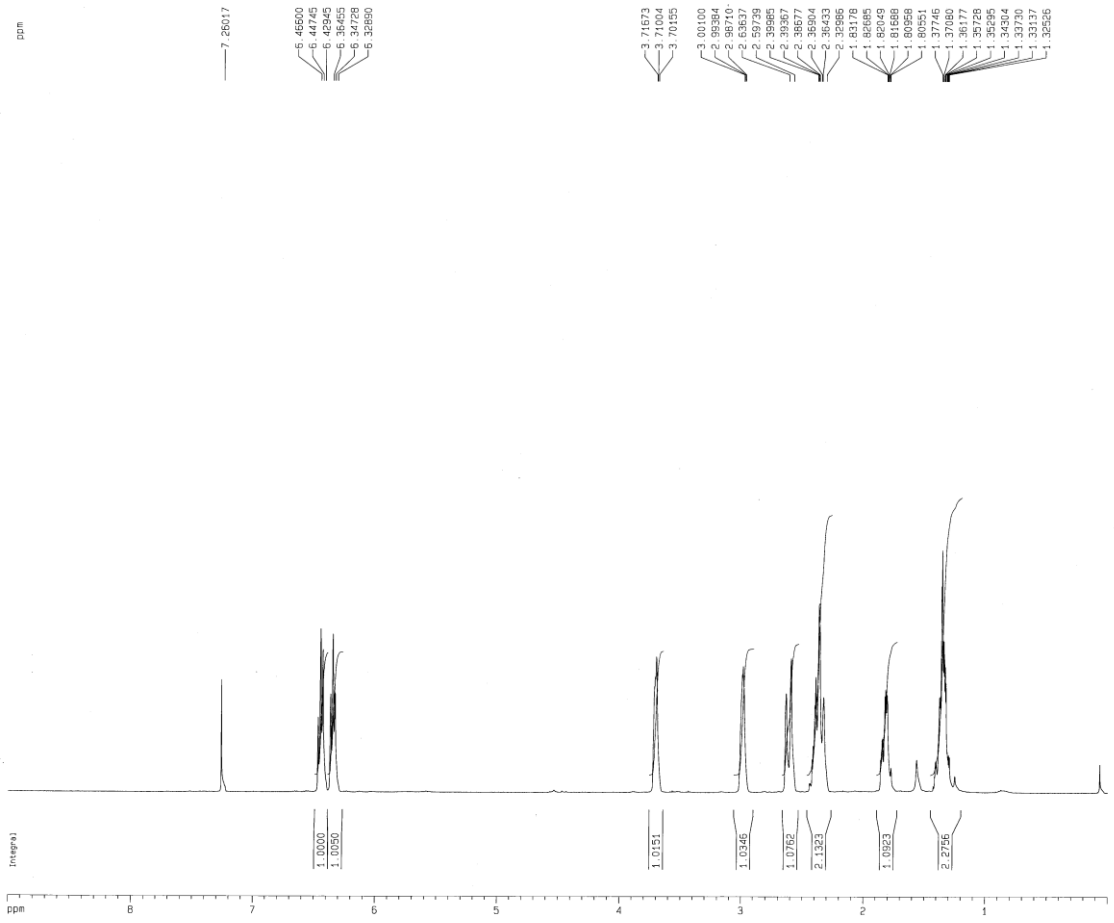
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





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EXPNO 1
PROCNO 1

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Time 16.20
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 199.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SF01 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 7.00 cm
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F1 3600.27 Hz
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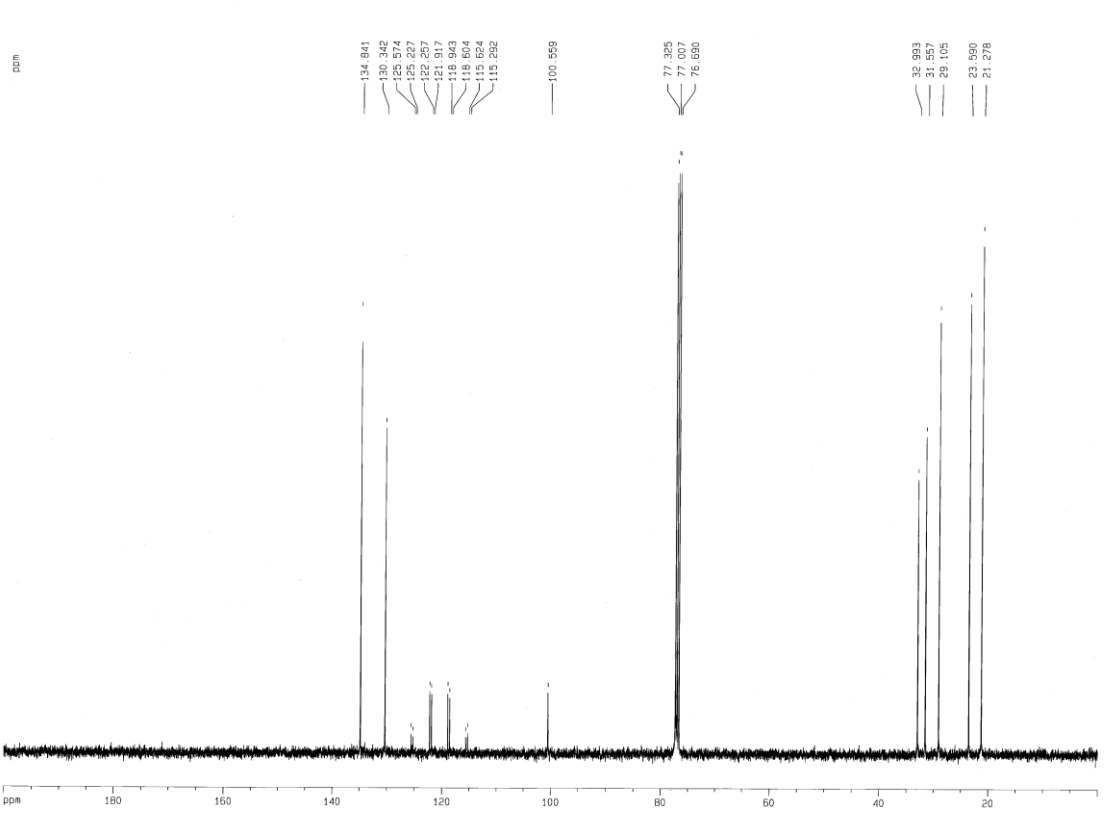
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME MF-120
EXPNO 2
PROCNO 1

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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 692
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 7298.2
DW 19.100 usec
DE 6.00 usec
TE 198.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SF01 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SF02 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876246 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
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F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
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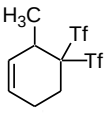

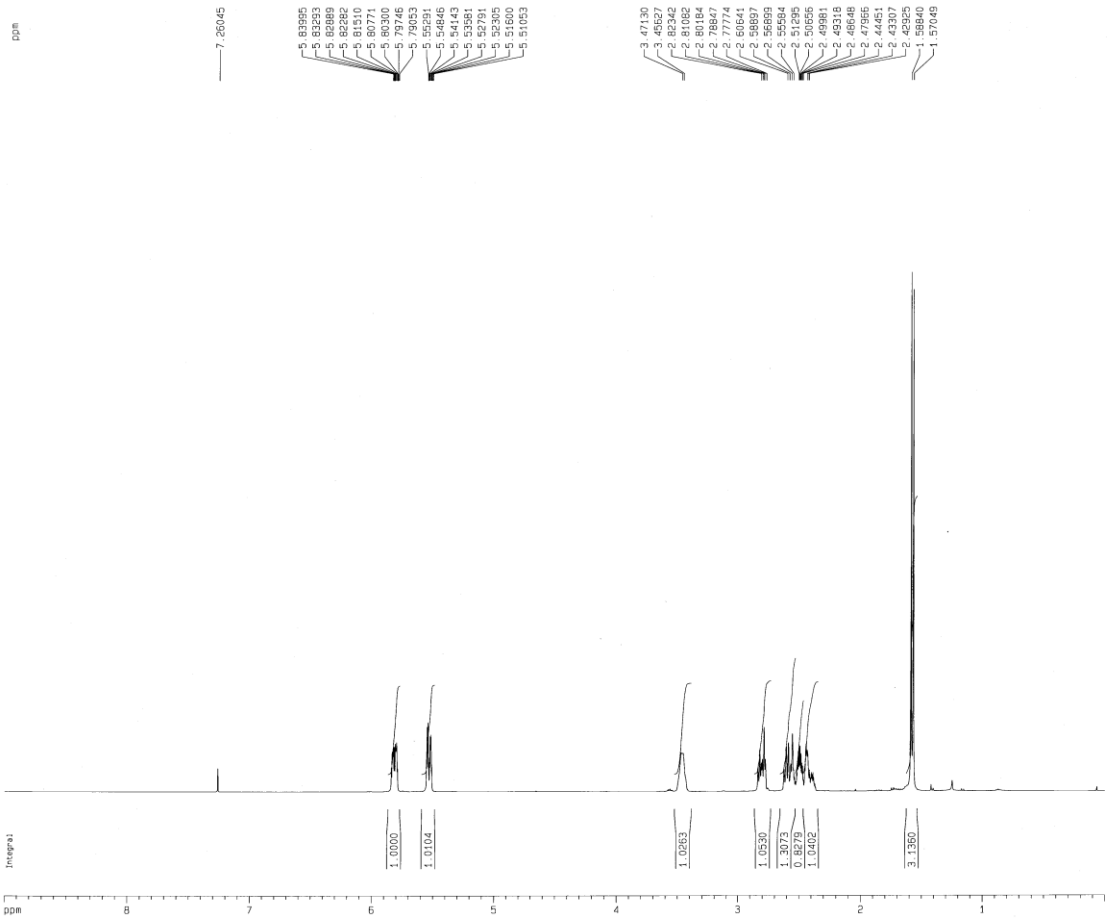


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EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
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Time                  16.08
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PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       10
DS                        2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                      80.6
DW                   60.400 usec
DE                    6.00 usec
TE                   199.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                    11.50 usec
PL1                   -4.00 dB
SFO1           400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                400.0300065 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                    32.00 cm
CY                    15.00 cm
F1P                 9.000 ppm
F1                  3600.27 Hz
F2P                 0.000 ppm
F2                    0.00 Hz
PPMCM             0.28125 ppm/cm
HZCM            112.50844 Hz/cm
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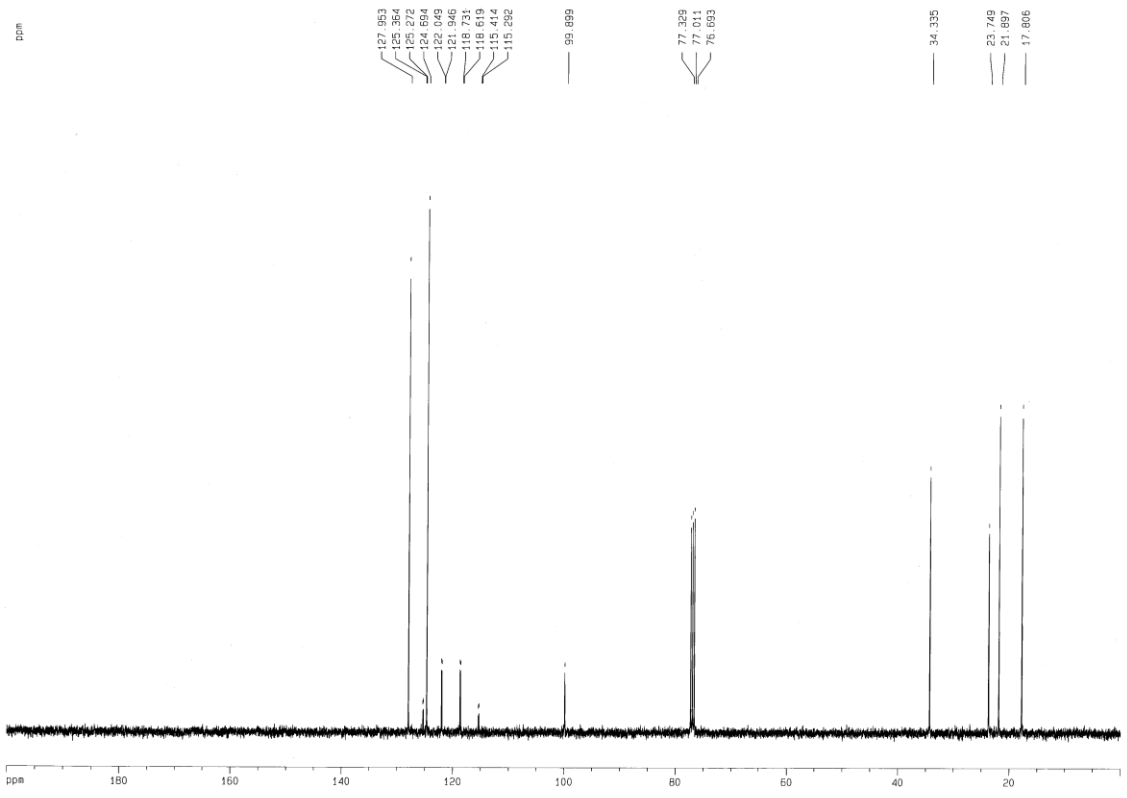


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Current Data Parameters
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EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
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Time                  16.13
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PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       62
DS                        4
SWH               26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                     13004
DW                   19.100 usec
DE                    6.00 usec
TE                   199.2 K
D1                2.00000000 sec
d11               0.03000000 sec
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MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                    10.00 usec
PL1                   -3.50 dB
SFO1           100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -4.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
SFO2           400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF             100.5876246 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                    32.00 cm
CY                    15.00 cm
F1P                 200.000 ppm
F1                  20117.53 Hz
F2P                 -0.000 ppm
F2                    -0.00 Hz
PPMCM              6.25000 ppm/cm
HZCM            628.67267 Hz/cm
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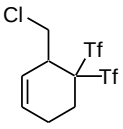

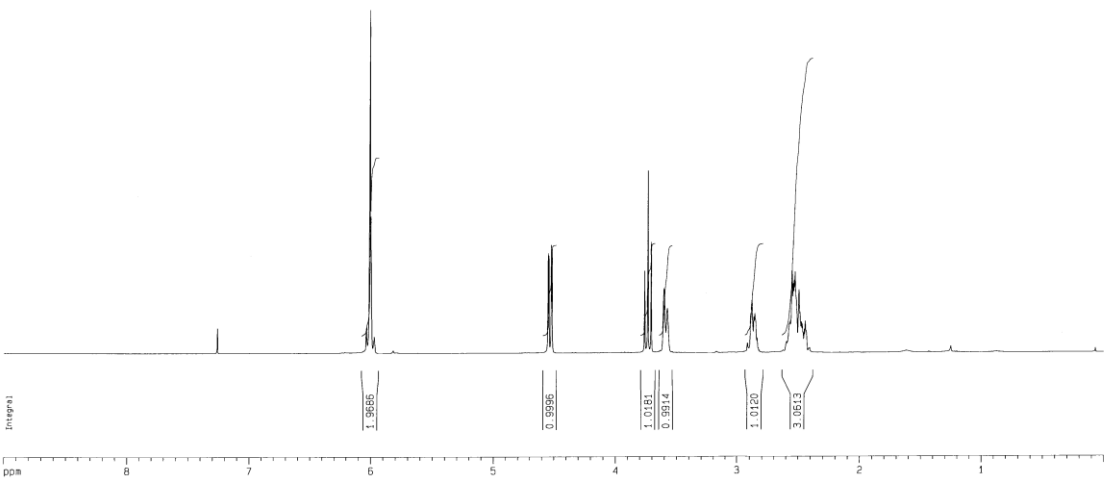


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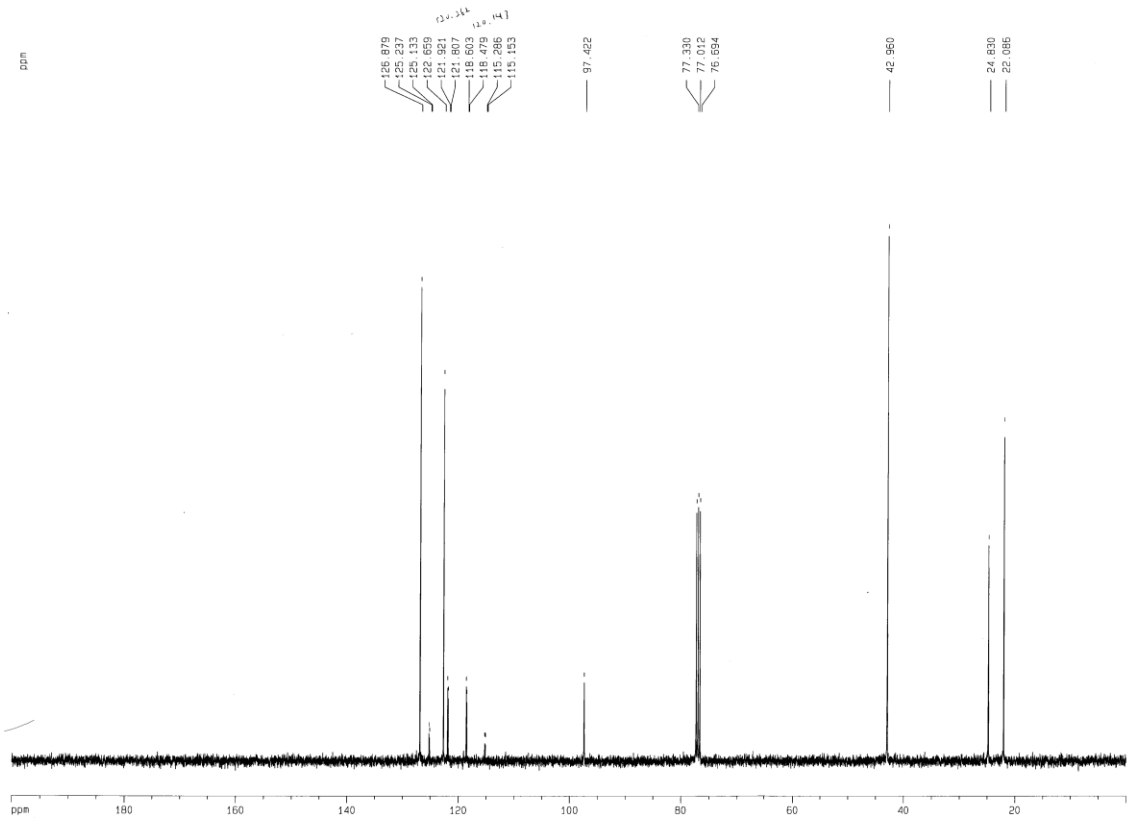
Current Data Parameters
NAME                  MF-170
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100727
Time                  16.19
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        32
DS                         2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                     101.6
DW                  60.400 usec
DE                   6.00 usec
TE                   295.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     11.50 usec
PL1                    -4.00 dB
SFO1            400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                400.0300067 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     32.00 cm
CY                     10.00 cm
F1P                    9.000 ppm
F1                   3600.27 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM              0.28125 ppm/cm
HZCM             112.50844 Hz/cm
```




```
Current Data Parameters
NAME                  MF-170
EXPNO                      2
PROCNO                     1

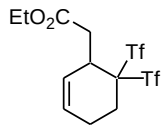
F2 - Acquisition Parameters
Date_              20100727
Time                  16.26
INSTRUM               spect
PROBHD   5 mm QNP 1H/13
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                        77
DS                         4
SWH              26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                    1448.2
DW                  19.100 usec
DE                   6.00 usec
TE                   295.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

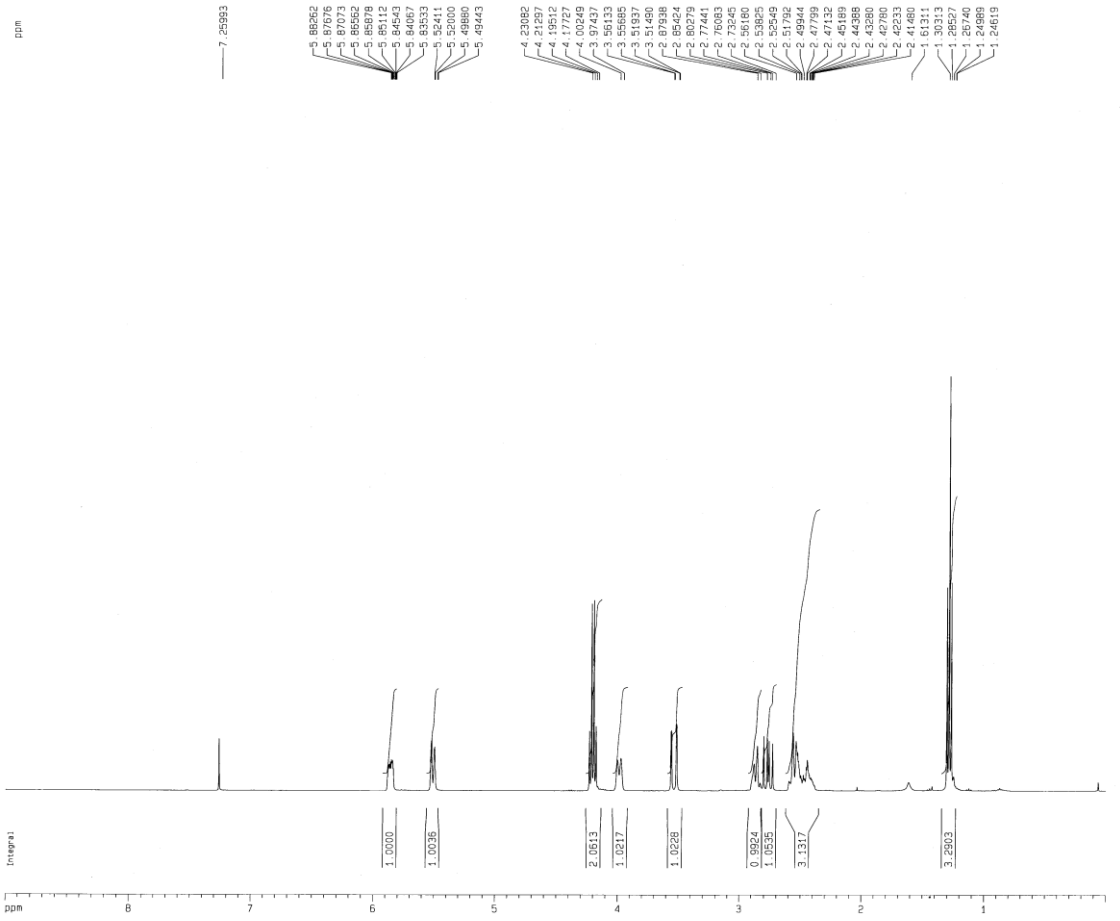
======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PL1                    -3.50 dB
SFO1            100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                 100.00 usec
PL2                    -4.00 dB
PL12                   17.00 dB
PL13                   17.00 dB
SFO2            400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF              100.5876246 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     32.00 cm
CY                     15.00 cm
F1P                   200.000 ppm
F1                  20117.53 Hz
F2P                   -0.000 ppm
F2                    -0.00 Hz
PPMCM              6.25000 ppm/cm
HZCM             628.67267 Hz/cm
```





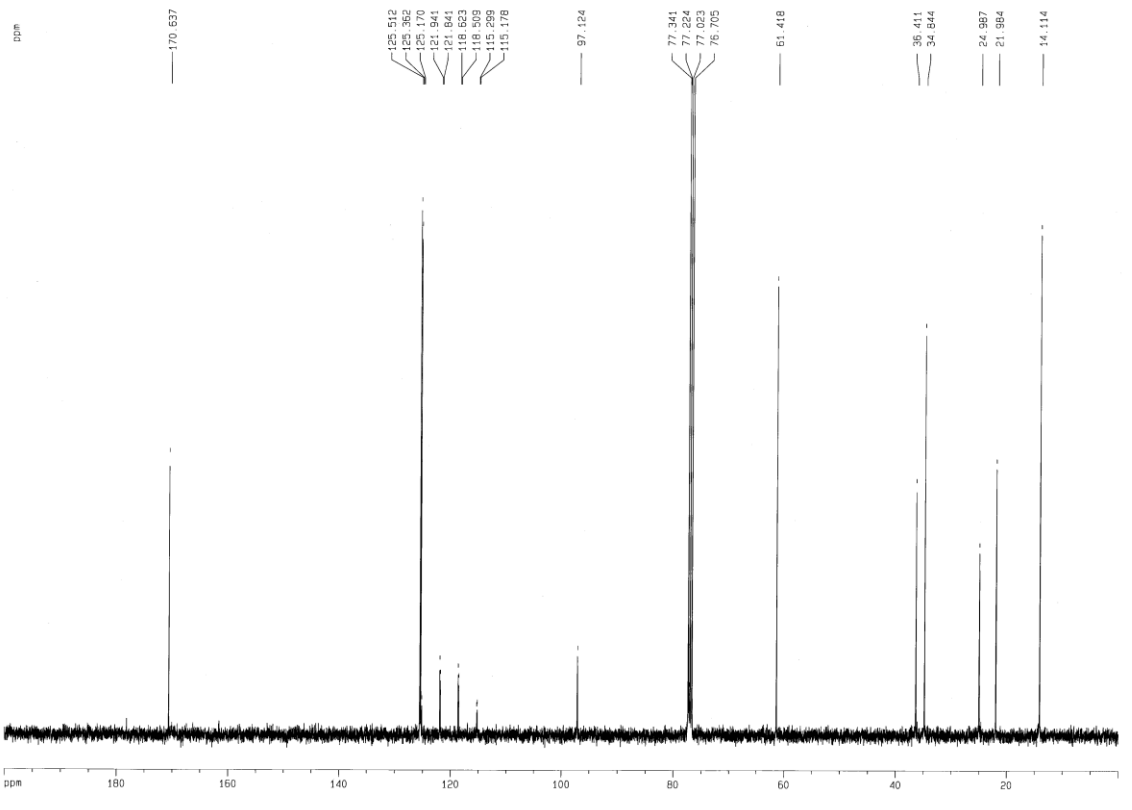

```
Current Data Parameters
NAME                  MF-150
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100708
Time                  16.27
INSTRUM               spect
PROBHD   5 mm GNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        13
DS                         2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                     143.7
DW                  60.400 usec
DE                   6.00 usec
TE                   295.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                   11.50 usec
PL1                   -4.00 dB
SFO1           400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                400.0300067 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     32.00 cm
CY                     12.00 cm
F1P                   9.000 ppm
F1                  3600.27 Hz
F2P                   0.000 ppm
F2                    0.00 Hz
PPMCM             0.28125 ppm/cm
HZCM             112.50844 Hz/cm
```




```
Current Data Parameters
NAME                  MF-150
EXPNO                      2
PROCNO                     1

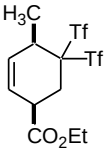
F2 - Acquisition Parameters
Date_              20100708
Time                  16.34
INSTRUM               spect
PROBHD   5 mm GNP 1H/13
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       252
DS                         4
SWH               26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                      6502
DW                  19.100 usec
DE                   6.00 usec
TE                   295.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

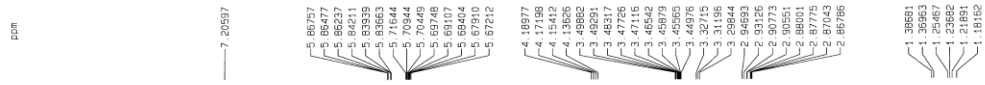
======== CHANNEL f1 ========
NUC1                      13C
P1                   10.00 usec
PL1                   -3.50 dB
SFO1           100.5986886 MHz

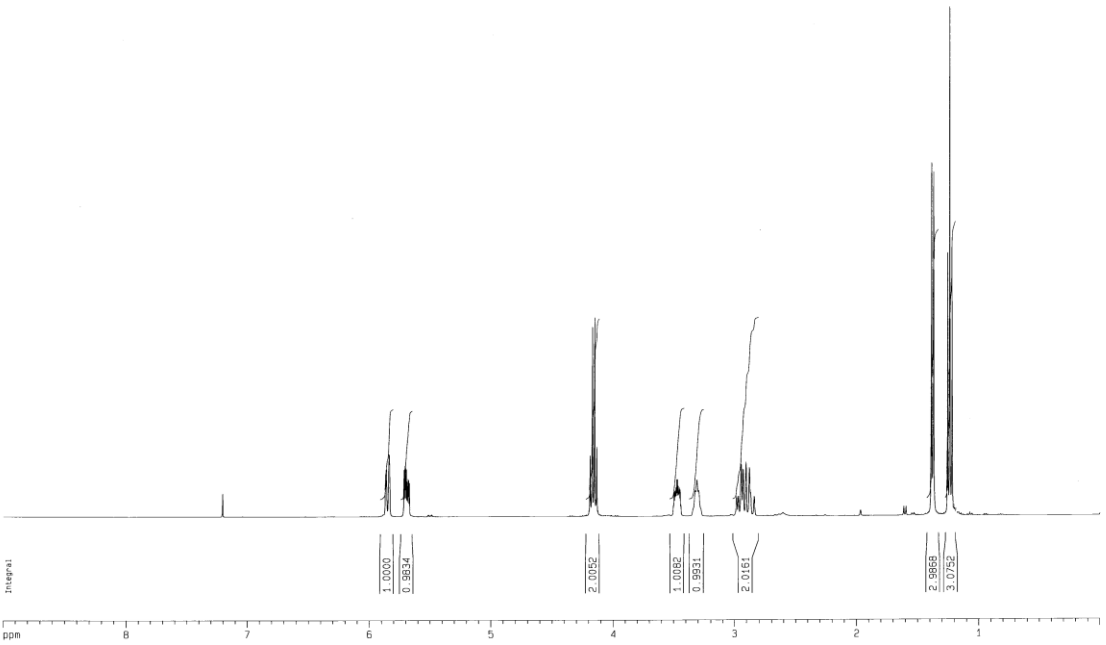
======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   -4.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
SFO2           400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF                100.5876240 MHz
WDW                       EM
SSB                        0
LB                     1.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     32.00 cm
CY                     15.00 cm
F1P                  200.000 ppm
F1                  20117.53 Hz
F2P                   -0.000 ppm
F2                    -0.00 Hz
PPMCM             6.25000 ppm/cm
HZCM             628.67267 Hz/cm
```







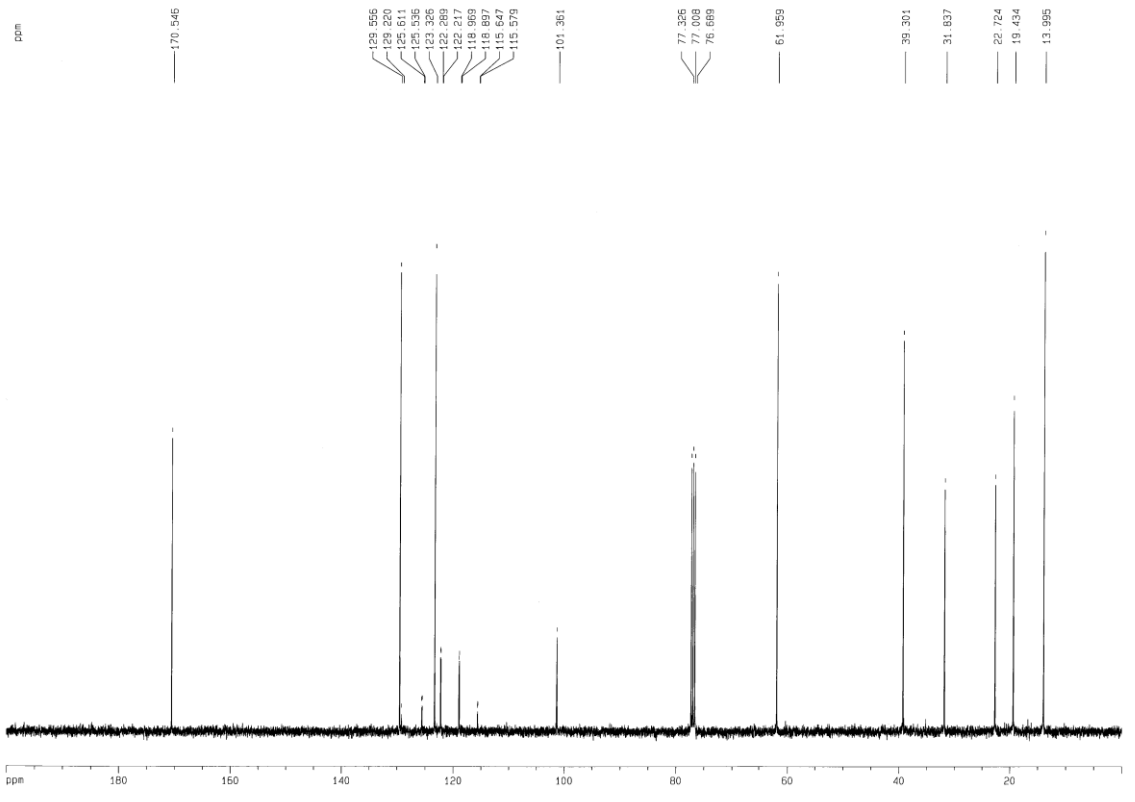
Current Data Parameters
NAME MF-178
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100727
Time 15.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 294.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300280 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50845 Hz/cm



Current Data Parameters
NAME MF-178
EXPNO 2
PROCNO 1

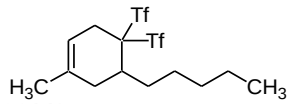
F2 - Acquisition Parameters
Date_ 20100727
Time 15.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 94
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 3251
DW 19.100 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

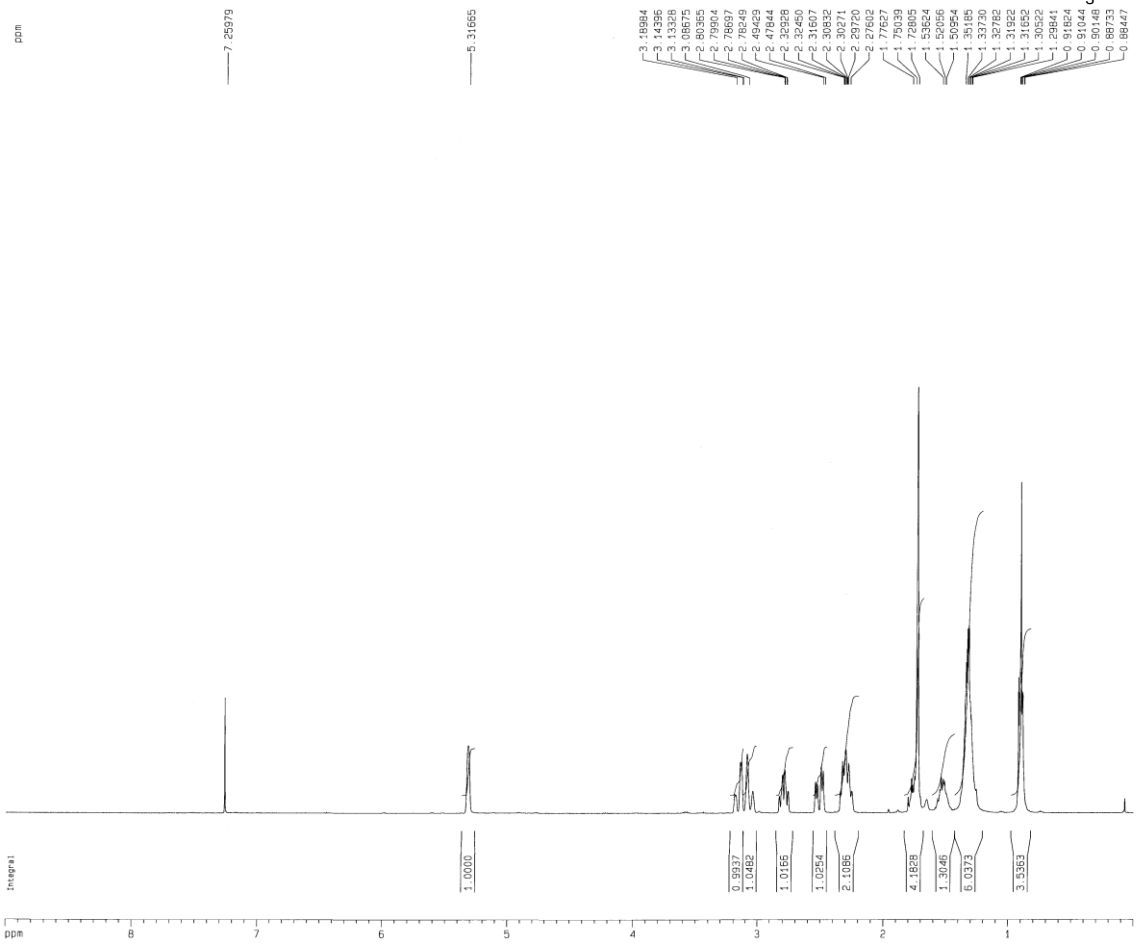
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876262 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 14.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





HF-615

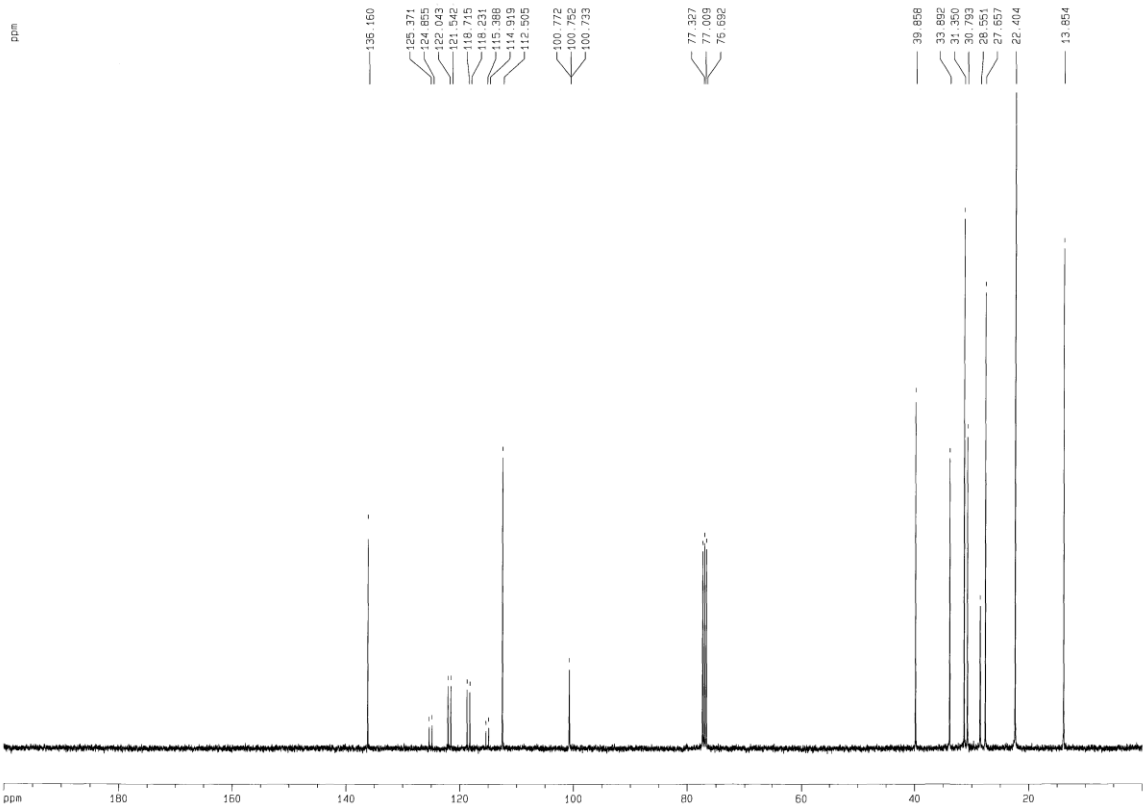
Current Data Parameters
NAME ~~HF-618~~
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100831
Time 18.48
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME HF-618
EXPNO 2
PROCNO 1

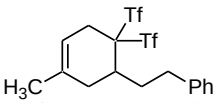
F2 - Acquisition Parameters
Date_ 20100831
Time 18.57
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 155
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 7298.2
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

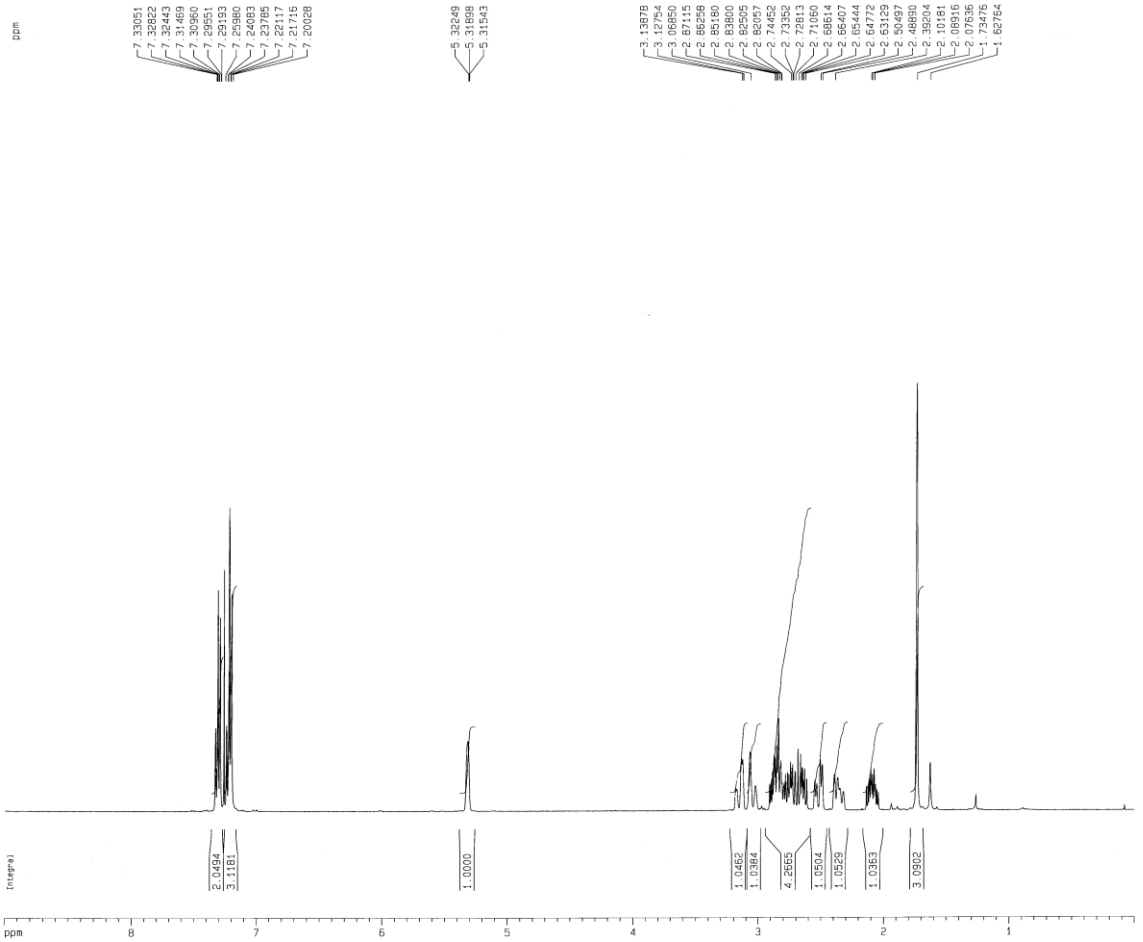
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876222 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





MF-198

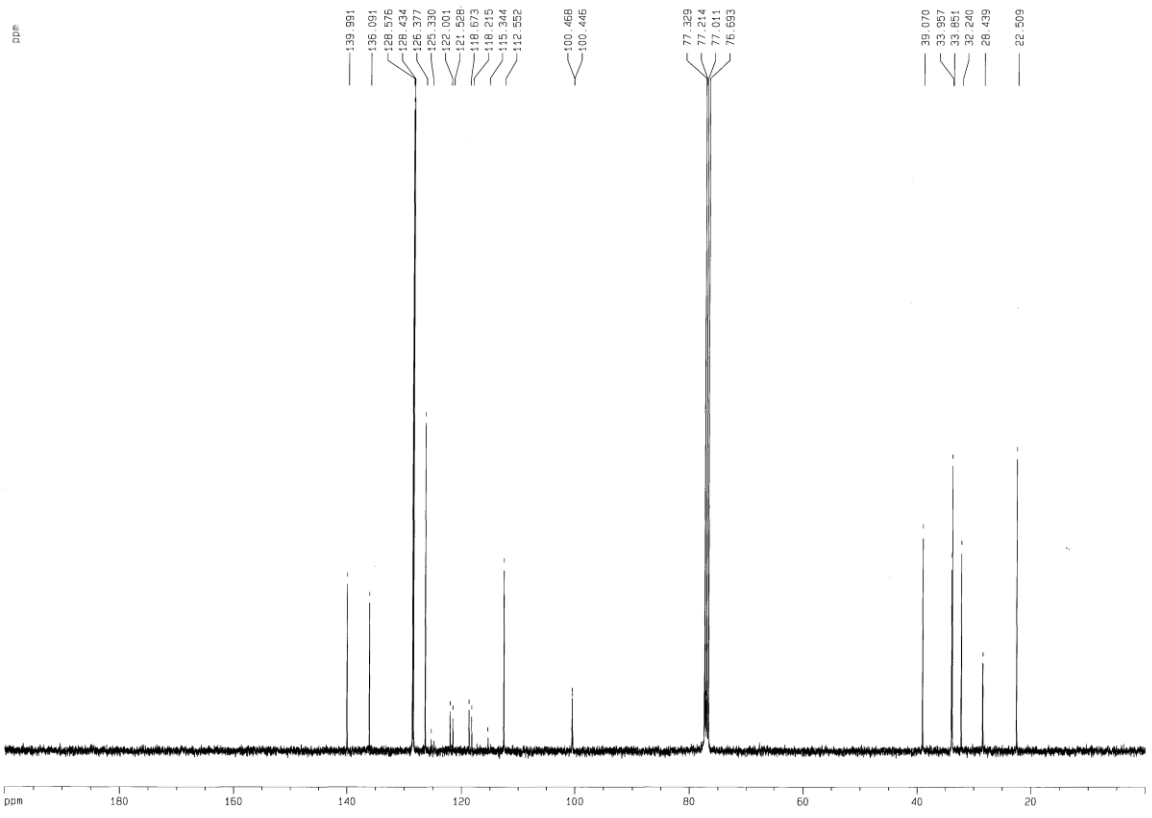
Current Data Parameters
NAME ~~MF-196~~
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100831
Time 17.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME MF-196
EXPNO 2
PROCNO 1

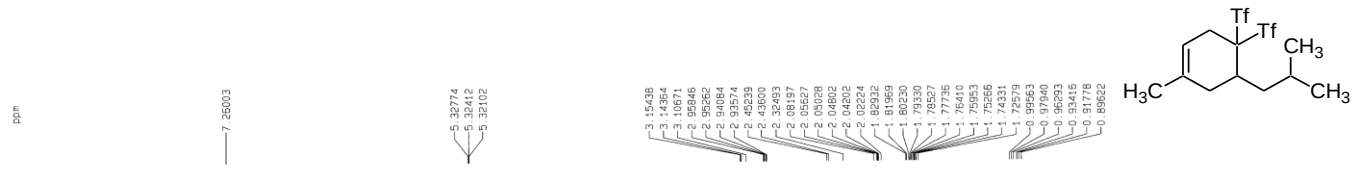
F2 - Acquisition Parameters
Date_ 20100831
Time 17.49
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1082
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

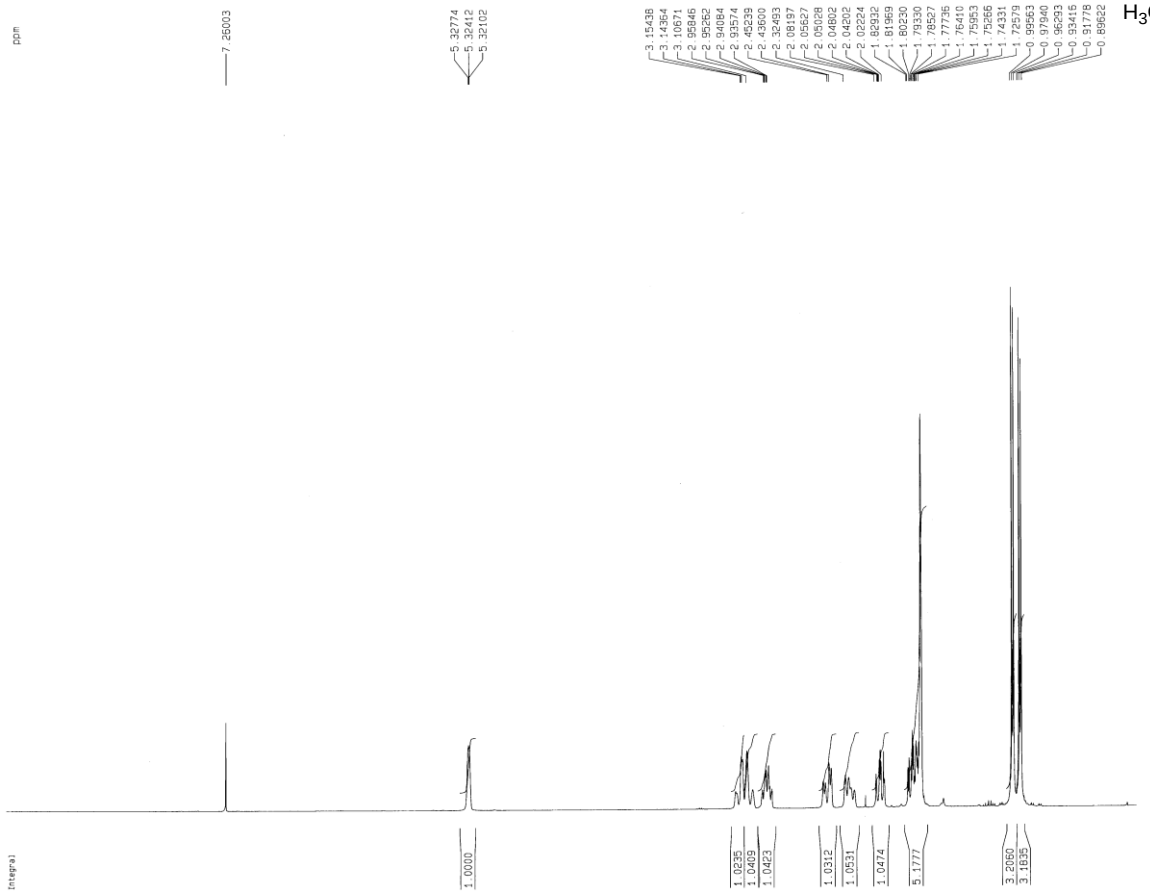
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876240 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





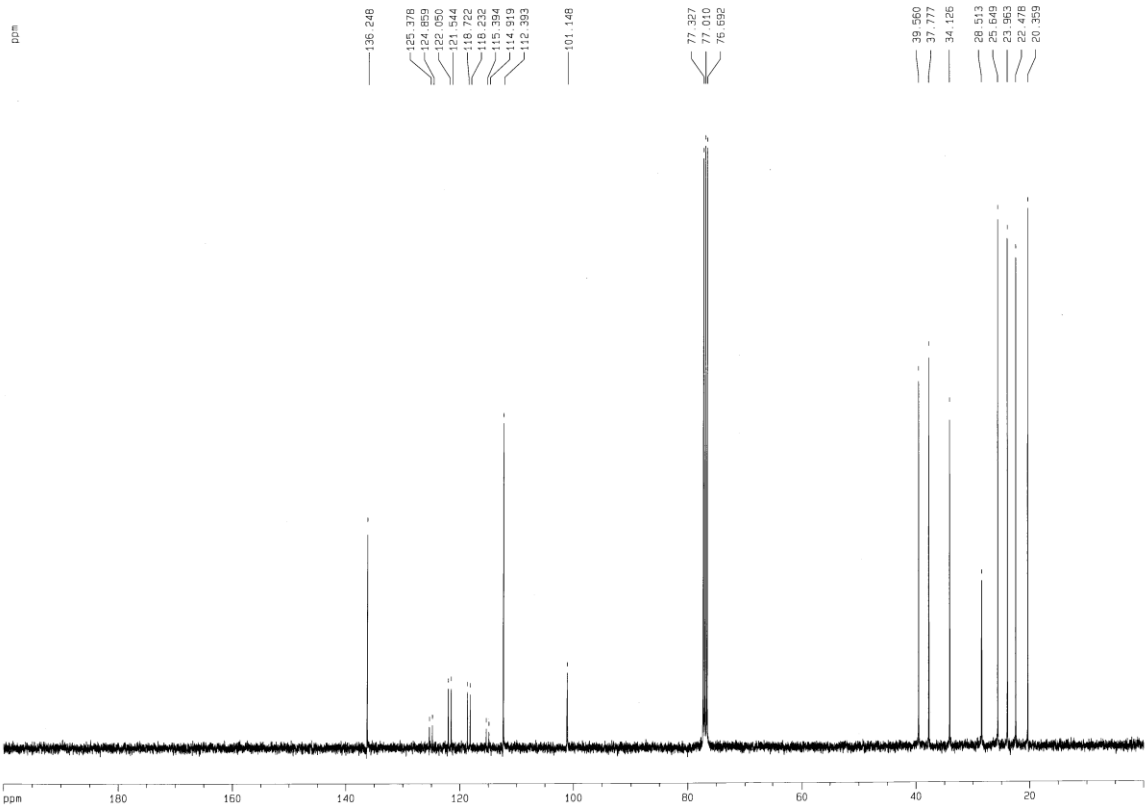
Current Data Parameters
NAME CBA06-iBu
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110131
Time 16.28
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA06-iBu
EXPNO 2
PROCNO 1

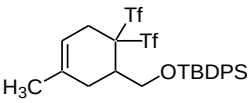
F2 - Acquisition Parameters
Date_ 20110131
Time 16.30
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 569
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

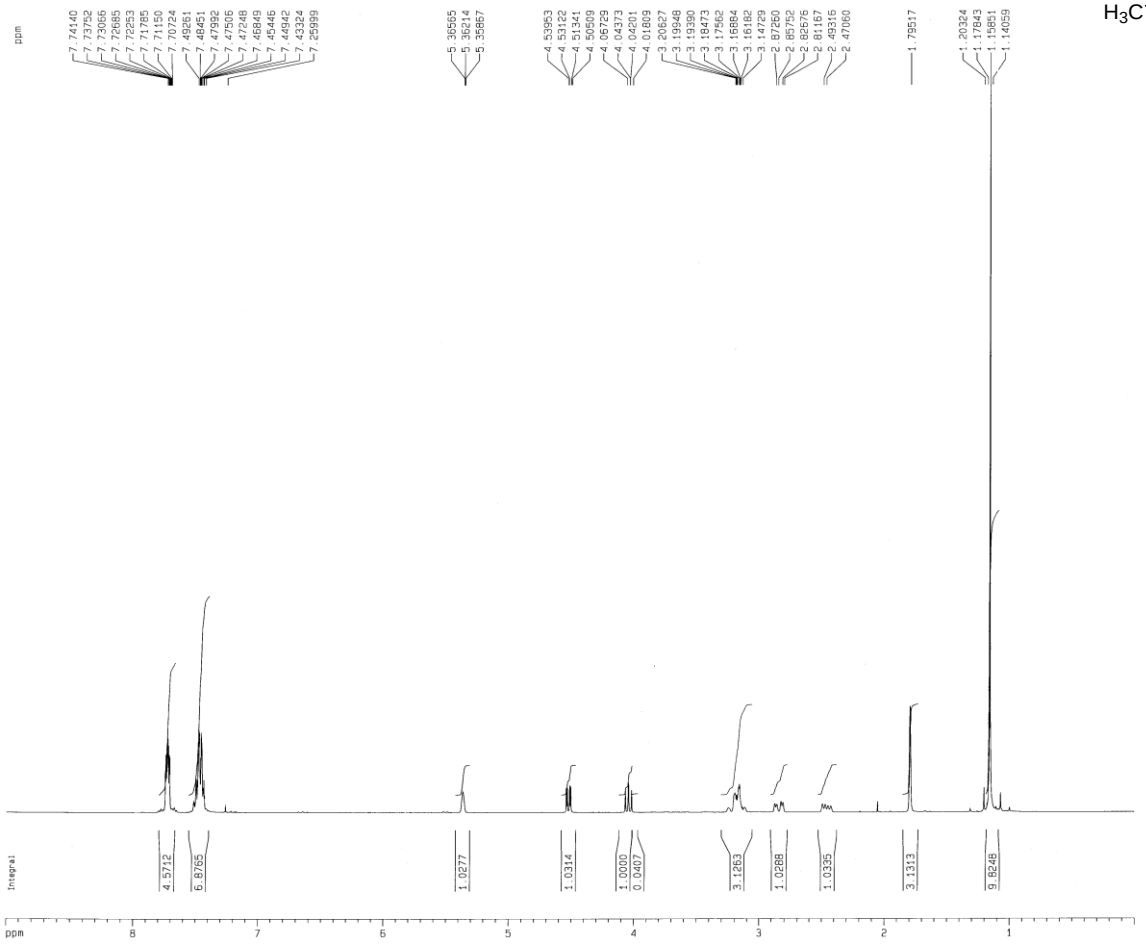
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876222 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





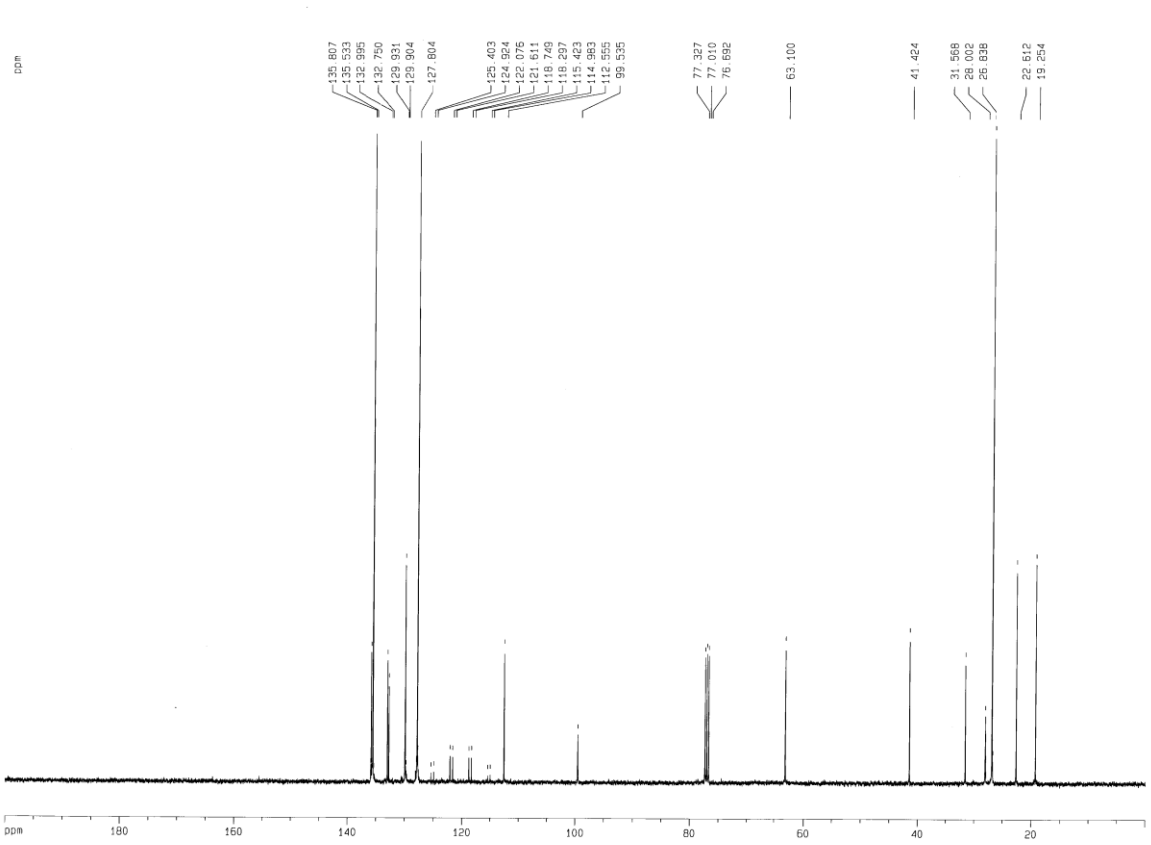

```
Current Data Parameters
NAME          CBA6-OTBDPS
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_          20110214
Time              11.05
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   4
DS                   2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                40.3
DW              60.400 usec
DE               6.00 usec
TE              300.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                11.50 usec
PL1               -4.00 dB
SFO1        400.0324703 MHz

F2 - Processing parameters
SI                  32768
SF          400.0300062 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00

1D NMR plot parameters
CX                 32.00 cm
CY                 21.00 cm
F1P                9.000 ppm
F1               3600.27 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          0.28125 ppm/cm
HZCM          112.50844 Hz/cm
```




```
Current Data Parameters
NAME          CBA6-OTBDPS
EXPNO                   2
PROCNO                  1

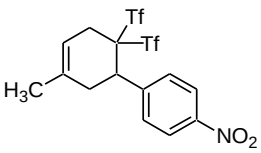
F2 - Acquisition Parameters
Date_          20110214
Time              11.08
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                 104
DS                   4
SWH          26178.010 Hz
FIDRES        0.399445 Hz
AQ            1.2517875 sec
RG                8192
DW              19.100 usec
DE               6.00 usec
TE              300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

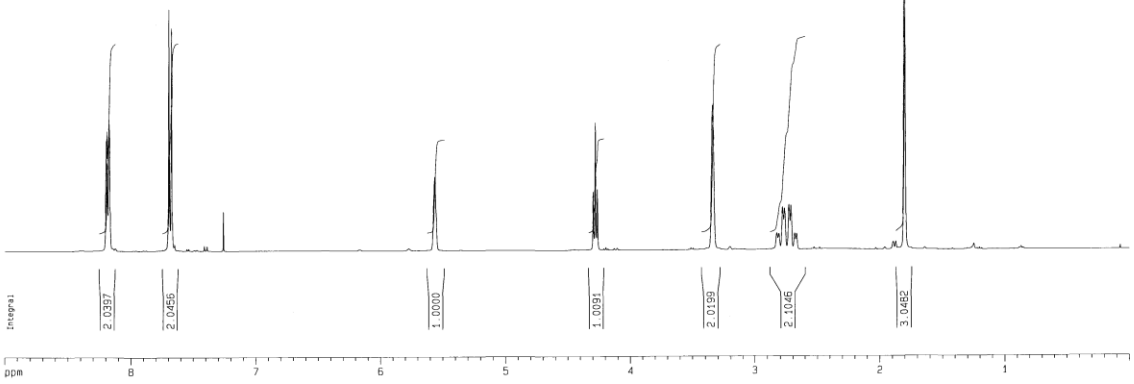
======== CHANNEL f1 ========
NUC1                13C
P1               10.00 usec
PL1               -3.50 dB
SFO1        100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2               -4.00 dB
PL12              17.00 dB
PL13              17.00 dB
SFO2        400.0316001 MHz

F2 - Processing parameters
SI                  32768
SF          100.5876294 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                 32.00 cm
CY                 19.00 cm
F1P              200.000 ppm
F1             20117.53 Hz
F2P              -0.000 ppm
F2                -0.00 Hz
PPMCM          6.25000 ppm/cm
HZCM          628.67273 Hz/cm
```





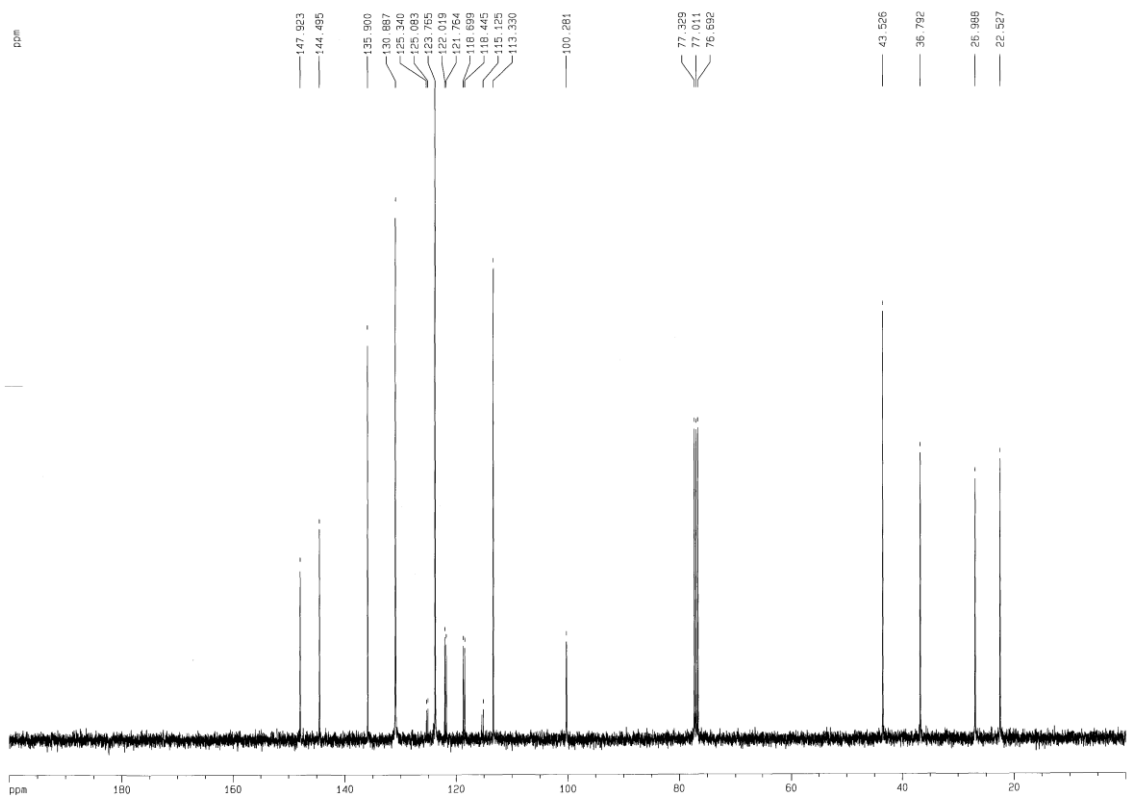

```
Current Data Parameters
NAME                  MF-189
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100823
Time                  10.51
INSTRUM               spect
PROBHD   5 mm GNP 1H/13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                         7
DS                         2
SWH               8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                     101.6
DW                  60.400 usec
DE                   6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                    11.50 usec
PL1                   -4.00 dB
SFO1             400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF               400.0300067 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                     1.00

1D NMR plot parameters
CX                    32.00 cm
CY                    10.00 cm
F1P                  9.000 ppm
F1                3600.27 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             0.28125 ppm/cm
HZCM            112.50844 Hz/cm
```




```
Current Data Parameters
NAME                  MF-189
EXPNO                      2
PROCNO                     1

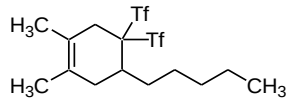
F2 - Acquisition Parameters
Date_              20100823
Time                  10.54
INSTRUM               spect
PROBHD   5 mm GNP 1H/13
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                        98
DS                         4
SWH              26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                      8192
DW                  19.100 usec
DE                   6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

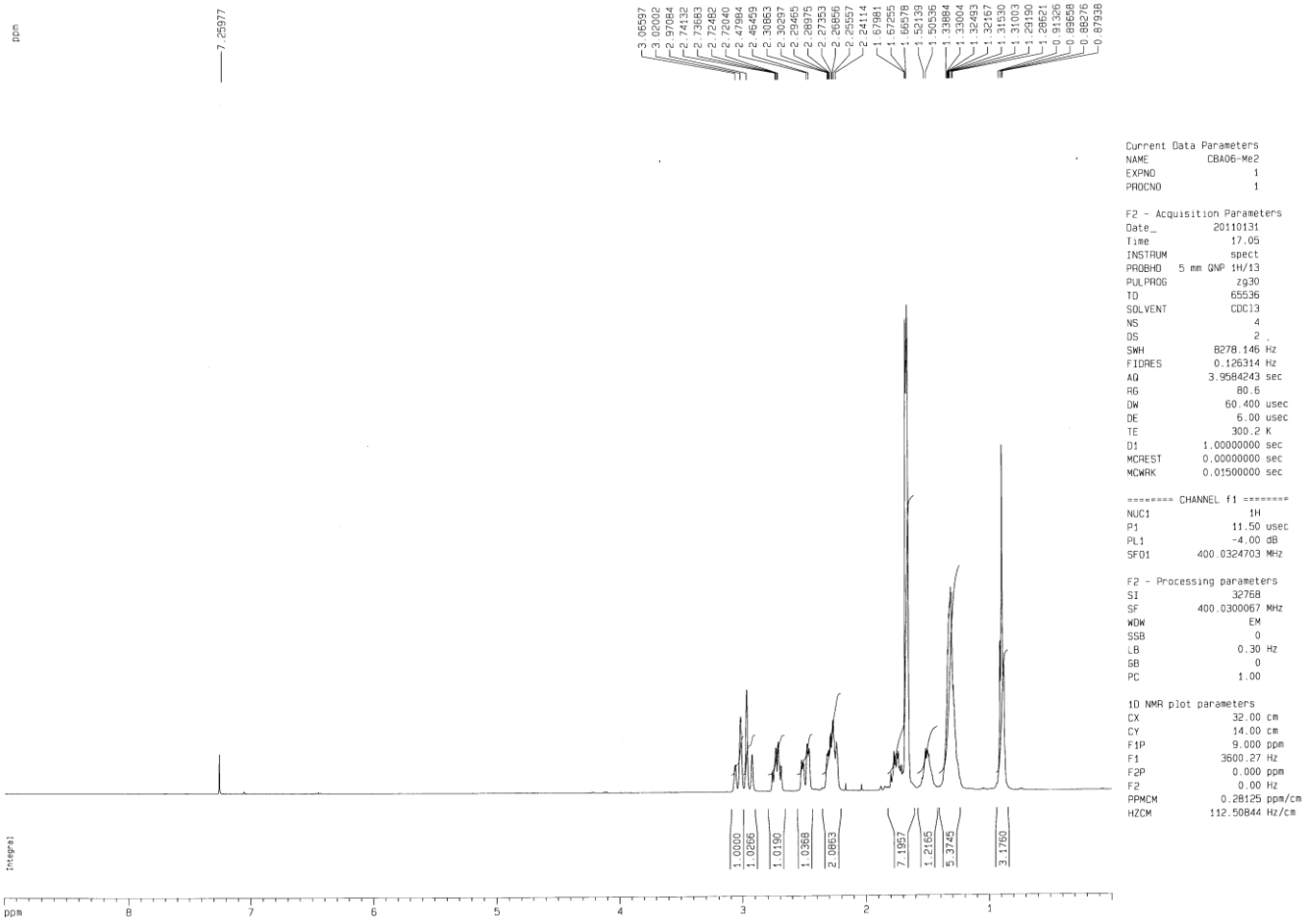
======== CHANNEL f1 ========
NUC1                      13C
P1                    10.00 usec
PL1                   -3.50 dB
SFO1             100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                       1H
PCPD2                100.00 usec
PL2                   -4.00 dB
PL12                  17.00 dB
PL13                  17.00 dB
SFO2             400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF               100.5876270 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                     1.40

1D NMR plot parameters
CX                    32.00 cm
CY                    19.00 cm
F1P                 200.000 ppm
F1                20117.53 Hz
F2P                  -0.000 ppm
F2                   -0.00 Hz
PPMCM             6.25000 ppm/cm
HZCM            628.67267 Hz/cm
```





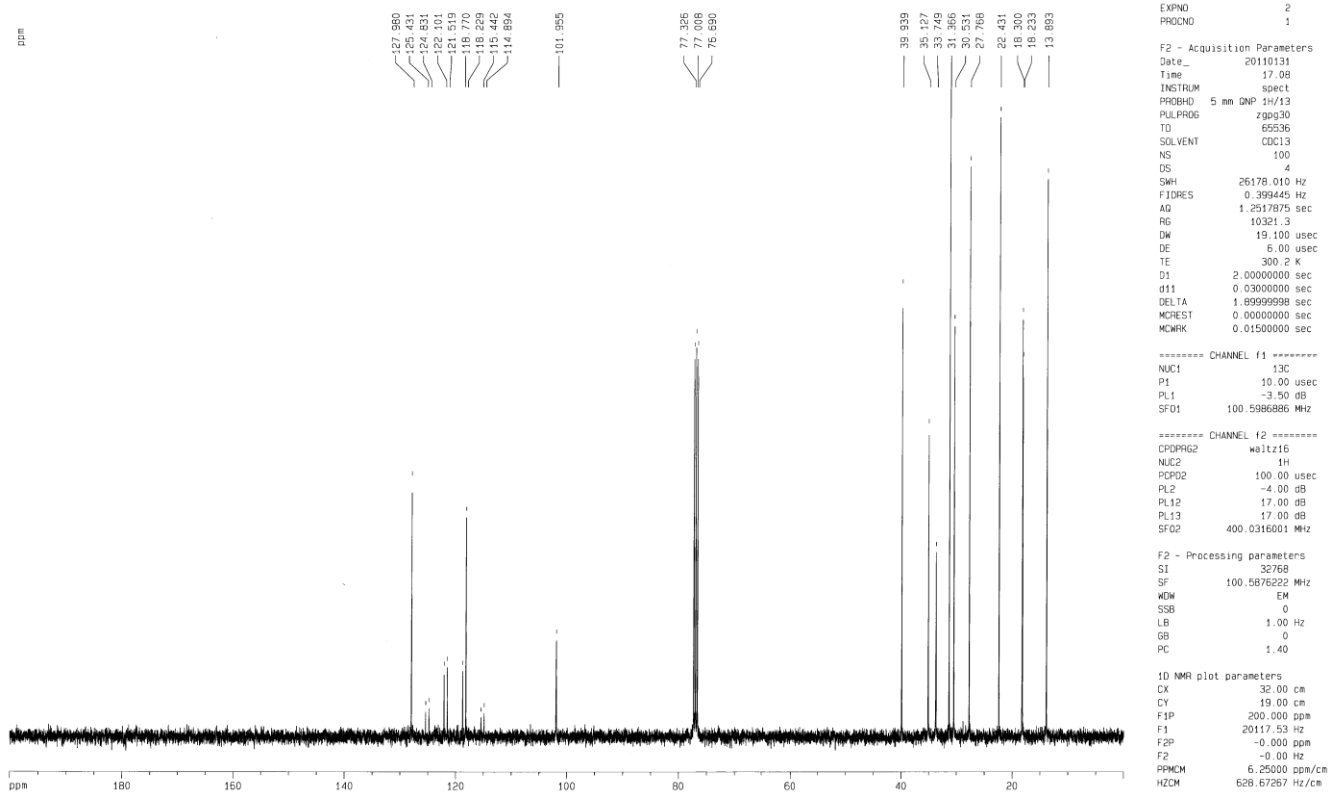
Current Data Parameters
NAME CBA06-Me2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110131
Time 17.05
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 80.6
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 14.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA06-Me2
EXPNO 2
PROCNO 1

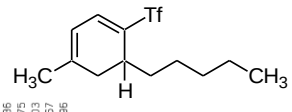
F2 - Acquisition Parameters
Date_ 20110131
Time 17.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 10321.3
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

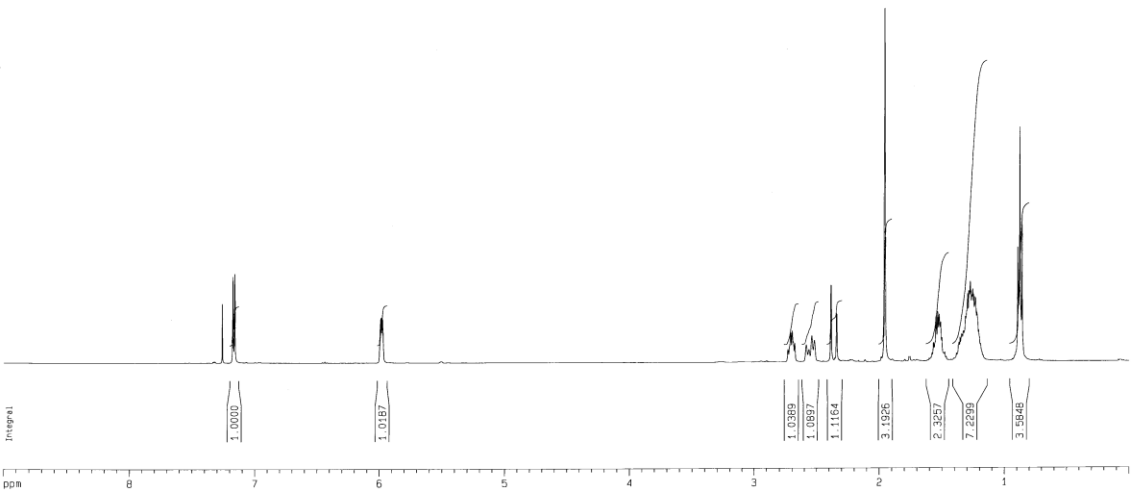
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876222 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





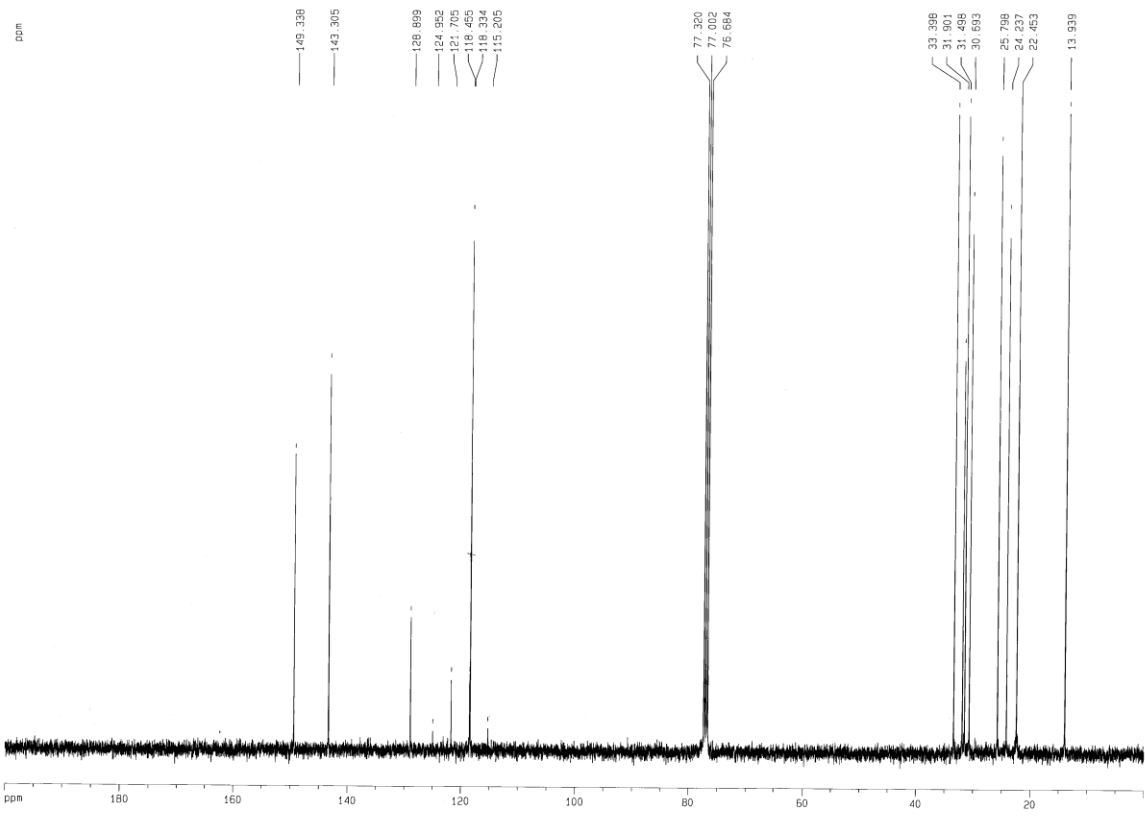
Current Data Parameters
NAME MF-218
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101210
Time 15.43
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 161.3
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME MF-218
EXPNO 2
PROCNO 1

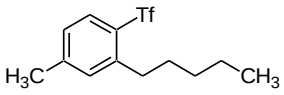
F2 - Acquisition Parameters
Date_ 20101210
Time 16.08
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 479
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 16384
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

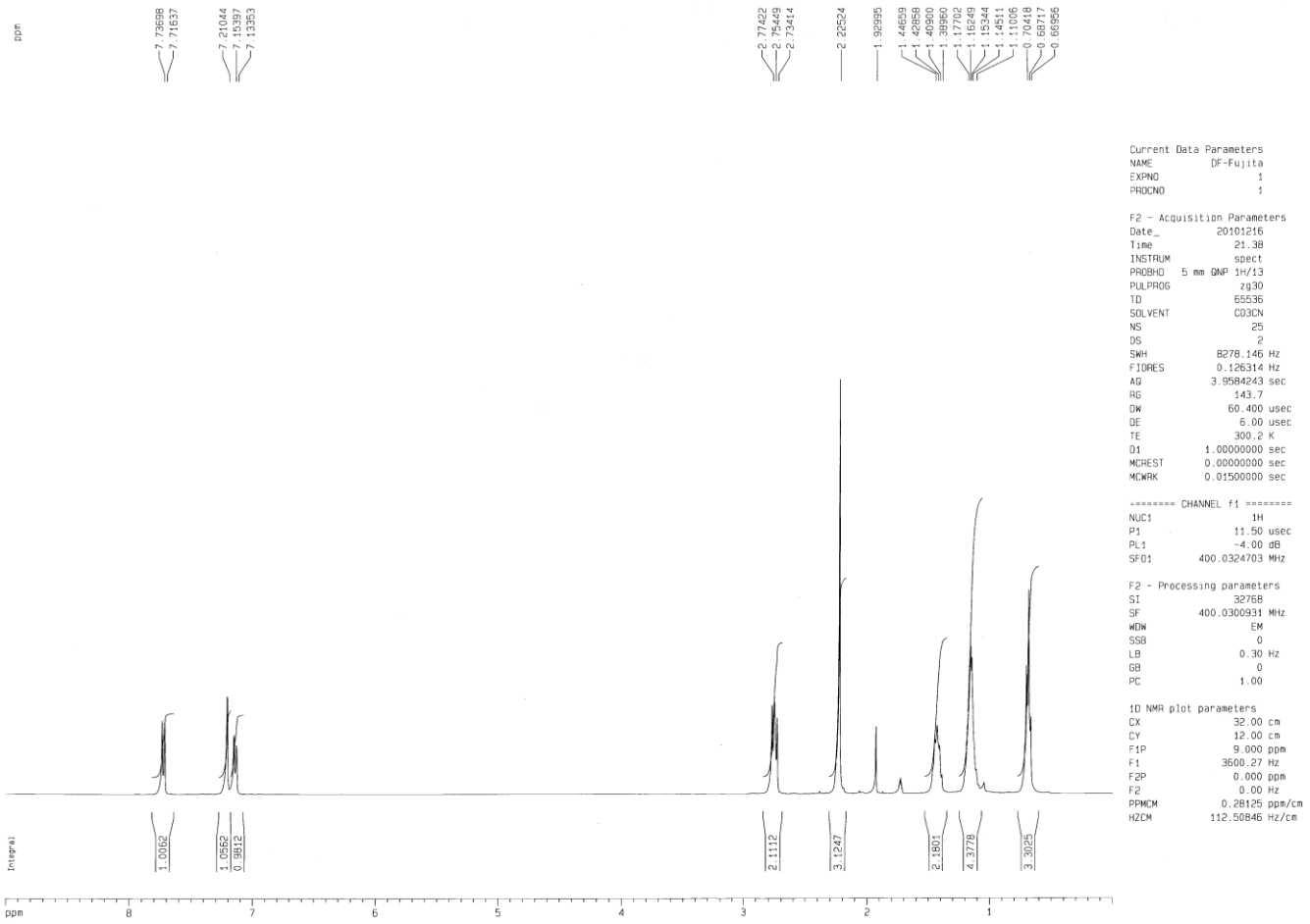
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876240 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





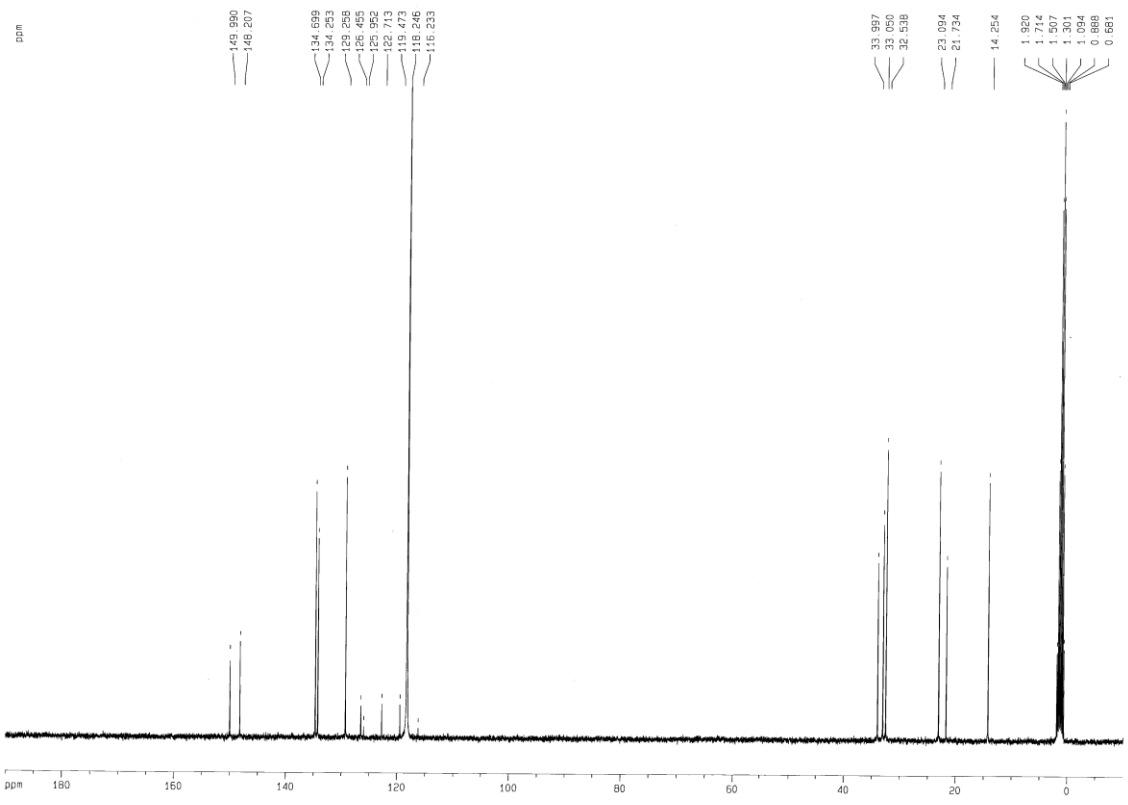
Current Data Parameters
NAME DF-Fujita
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20101216
Time 21.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 25
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300931 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50846 Hz/cm



Current Data Parameters
NAME DF-Fujita
EXPNO 2
PROCNO 1

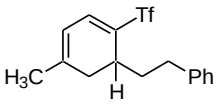
F2 - Acquisition Parameters
Date_ 20101216
Time 21.40
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 336
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 4597.6
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

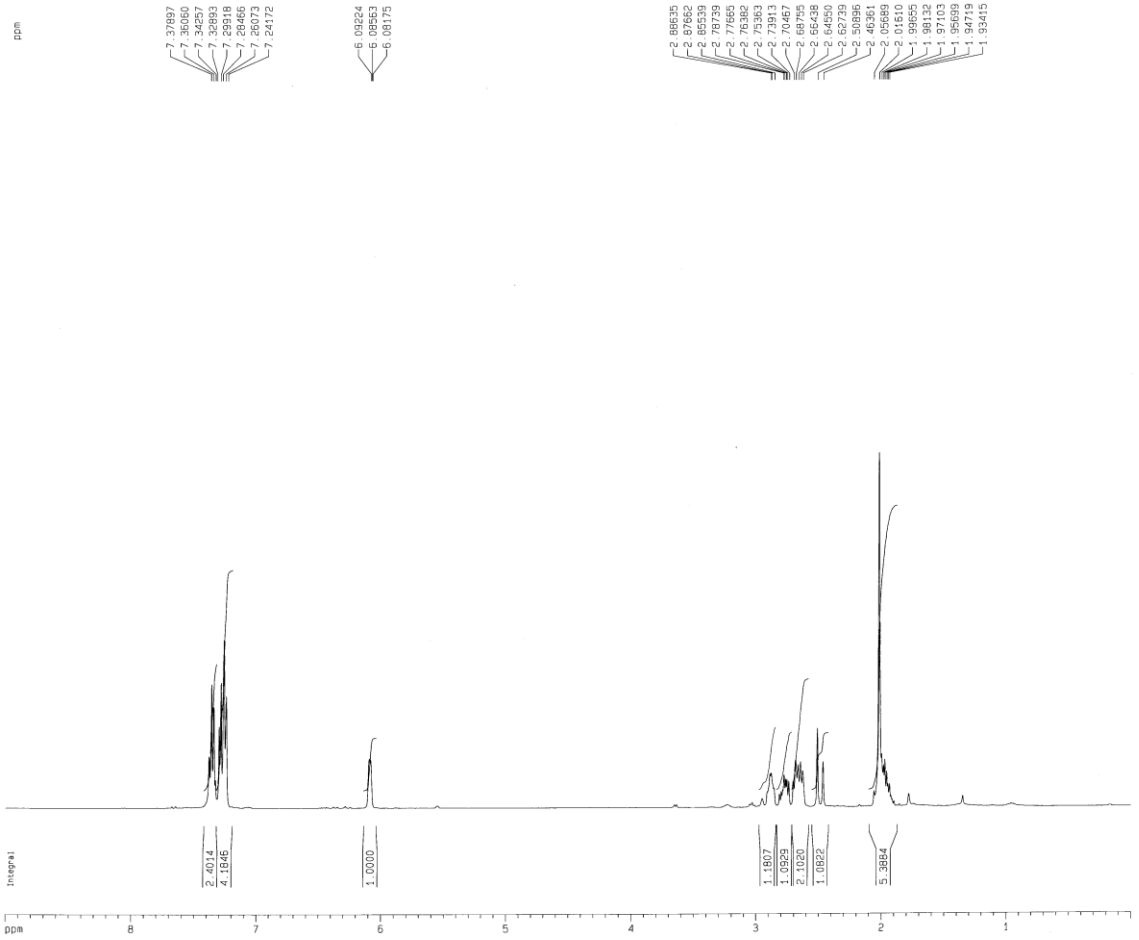
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5875236 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 190.000 ppm
F1 19111.63 Hz
F2P -10.000 ppm
F2 -1005.87 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67206 Hz/cm





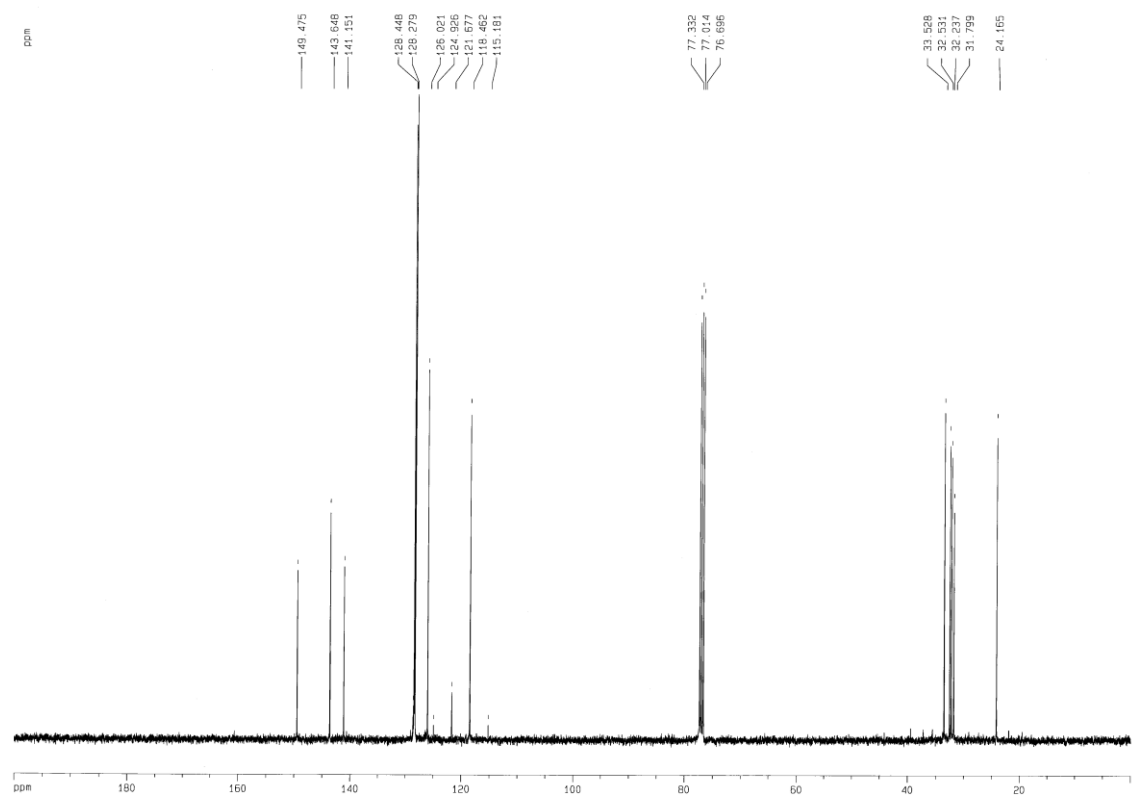

```
Current Data Parameters
NAME                  CBA06-D2P
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20110203
Time                      16.44
INSTRUM                   spect
PROBHD    5 mm QNP 1H/13
PULPROG                   zg30
TD                       65536
SOLVENT                   CDCl3
NS                            9
DS                            2
SWH                  8278.146 Hz
FIDRES               0.126314 Hz
AQ                   3.9584243 sec
RG                          128
DW                     60.400 usec
DE                      6.00 usec
TE                      300.2 K
D1                   1.00000000 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                        1H
P1                      11.50 usec
PL1                     -4.00 dB
SFO1                400.0324703 MHz

F2 - Processing parameters
SI                        32768
SF                  400.0305673 MHz
WDW                          EM
SSB                           0
LB                         0.30 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                        32.00 cm
CY                        10.00 cm
F1P                        9.000 ppm
F1                      3600.28 Hz
F2P                        0.000 ppm
F2                         0.00 Hz
PPMCM                  0.28125 ppm/cm
HZCM                 112.50860 Hz/cm
```




```
Current Data Parameters
NAME                  CBA06-D2P
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20110203
Time                      16.48
INSTRUM                   spect
PROBHD    5 mm QNP 1H/13
PULPROG                 zgpg30
TD                       65536
SOLVENT                   CDCl3
NS                          238
DS                            4
SWH                  26178.010 Hz
FIDRES               0.399445 Hz
AQ                   1.2517875 sec
RG                         8192
DW                     19.100 usec
DE                      6.00 usec
TE                      300.2 K
D1                   2.00000000 sec
d11                  0.03000000 sec
DELTA                1.89999998 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                      10.00 usec
PL1                     -3.50 dB
SFO1                100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                  100.00 usec
PL2                     -4.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
SFO2                400.0316001 MHz

F2 - Processing parameters
SI                        32768
SF                  100.5877756 MHz
WDW                          EM
SSB                           0
LB                         1.00 Hz
GB                            0
PC                         1.40

1D NMR plot parameters
CX                        32.00 cm
CY                        19.00 cm
F1P                      200.000 ppm
F1                     20117.55 Hz
F2P                       -0.000 ppm
F2                        -0.00 Hz
PPMCM                  6.25000 ppm/cm
HZCM                 628.67365 Hz/cm
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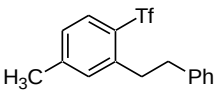
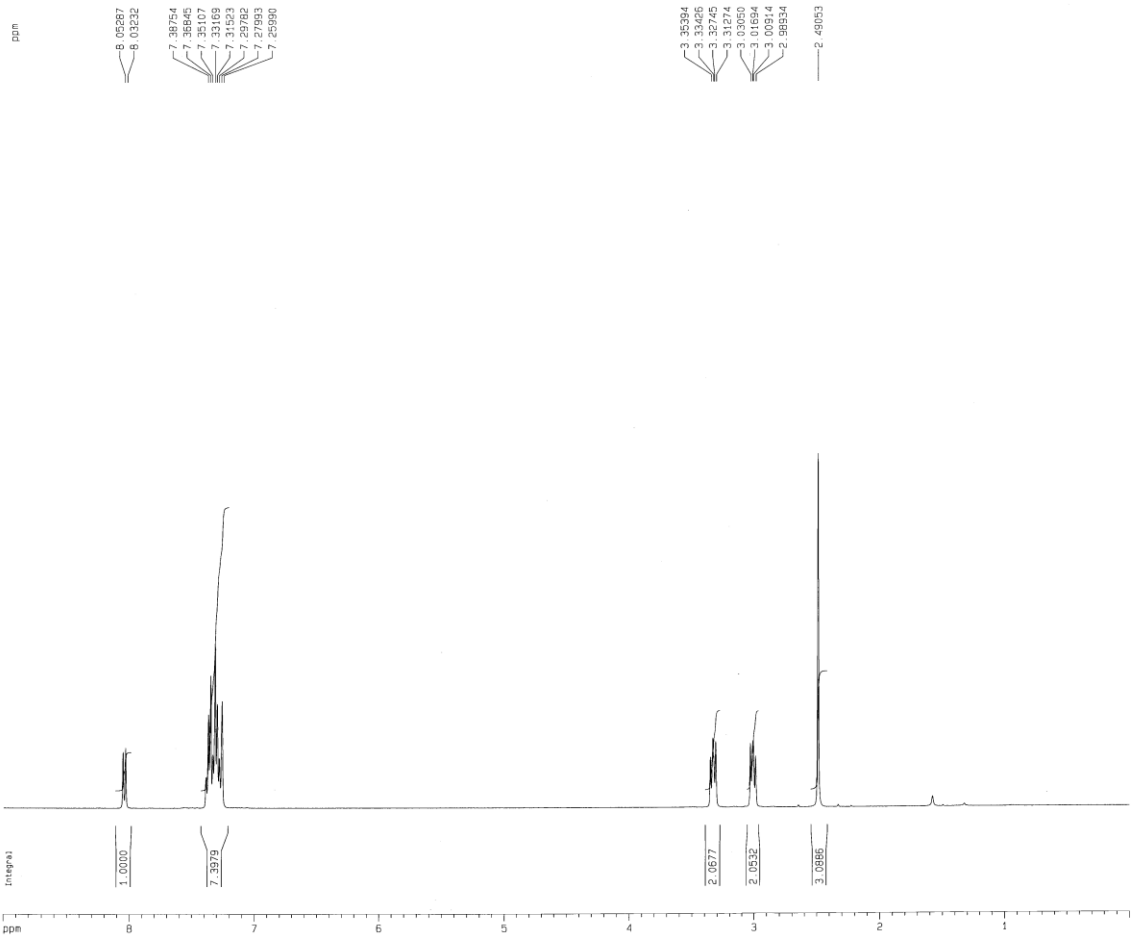




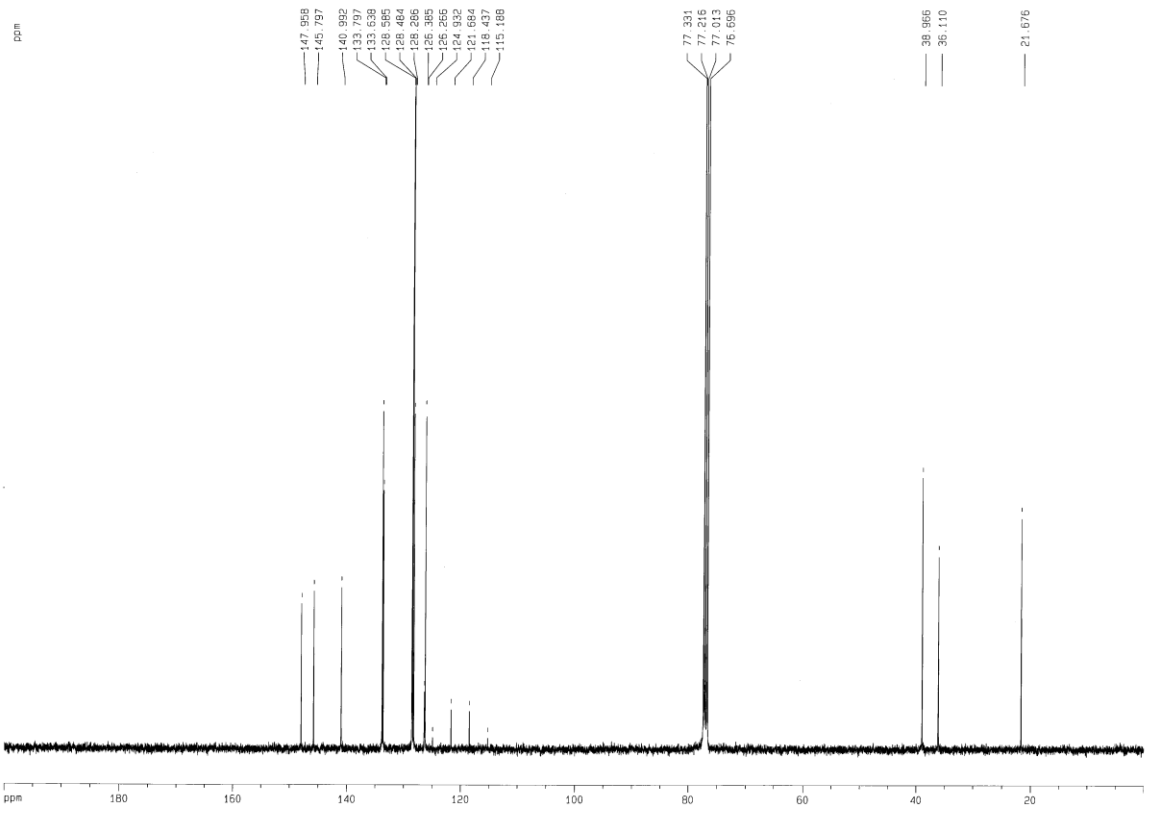
Current Data Parameters
NAME CBA06-B2P
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110205
Time 12.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 362
DW 60.400 usec
DE 6.00 usec
TE 303.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0299850 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA06-B2P
EXPNO 2
PROCNO 1

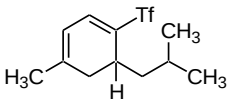
F2 - Acquisition Parameters
Date_ 20110205
Time 12.45
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1703
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 9195.2
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

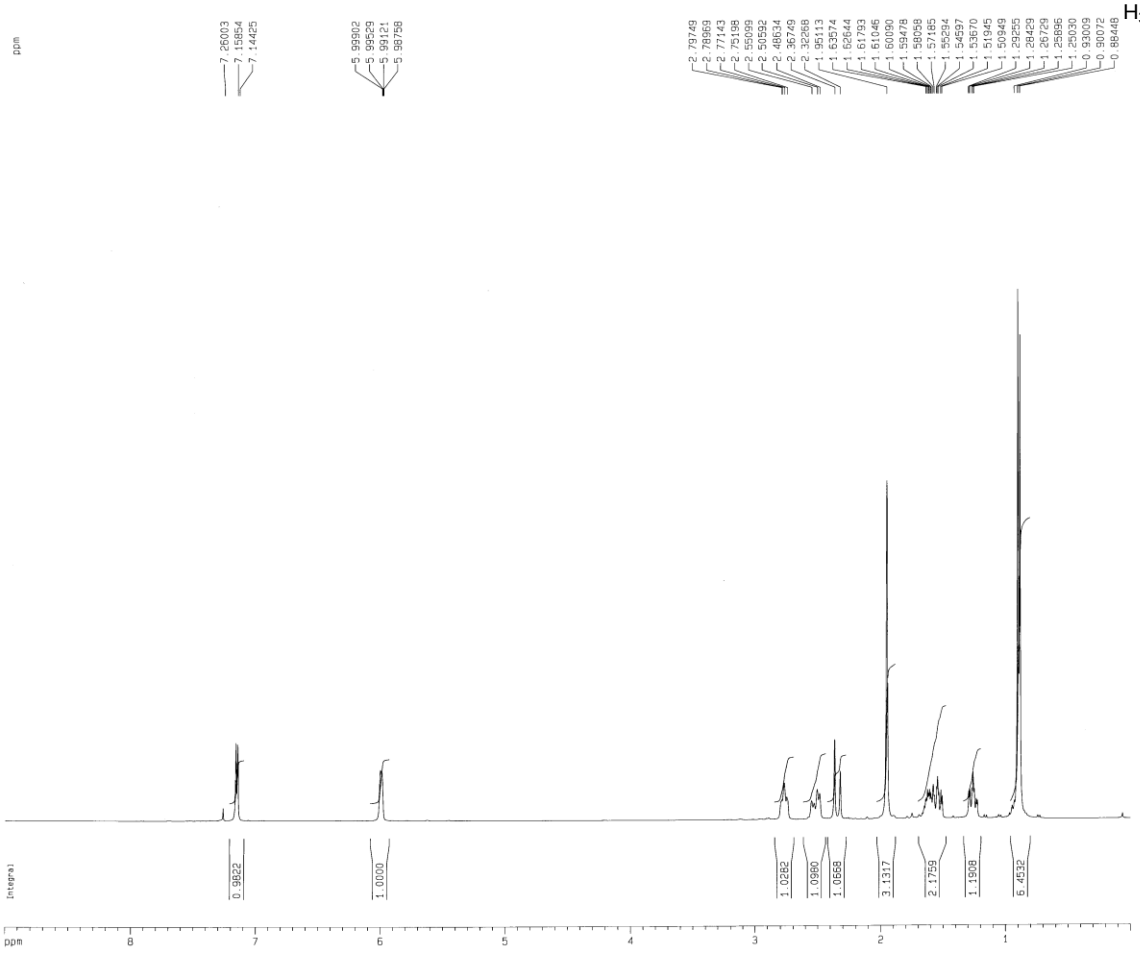
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876238 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





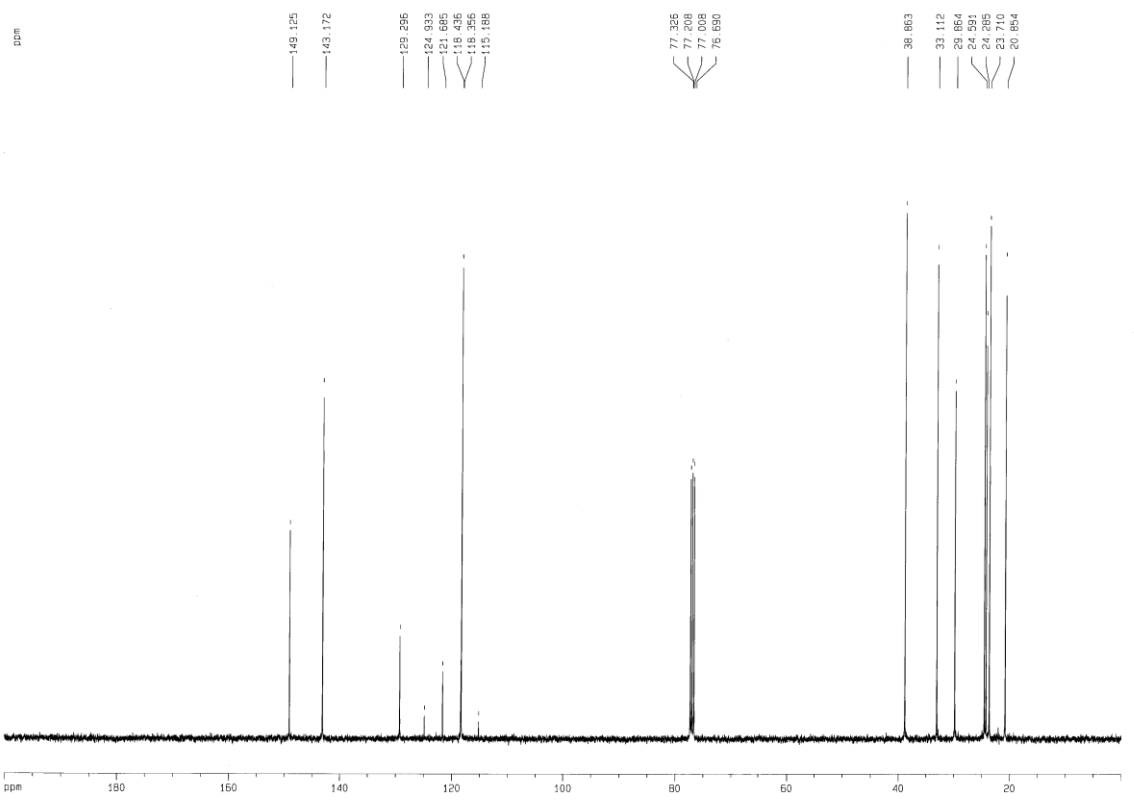
Current Data Parameters
NAME CBA06-DiBu
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110203
Time 17.04
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 71.8
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA06-DiBu
EXPNO 2
PROCNO 1

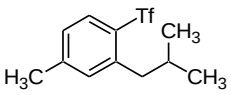
F2 - Acquisition Parameters
Date_ 20110203
Time 17.07
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 289
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 16384
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

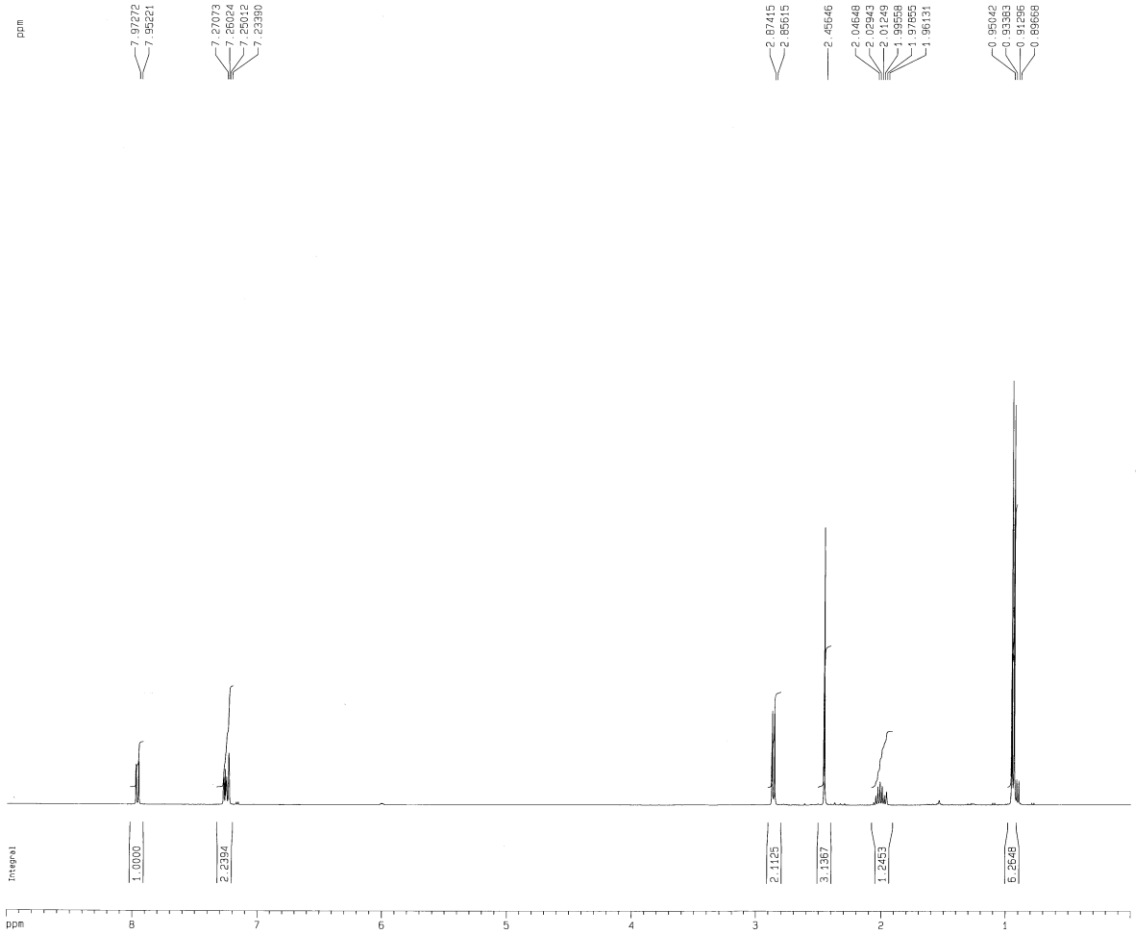
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876246 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





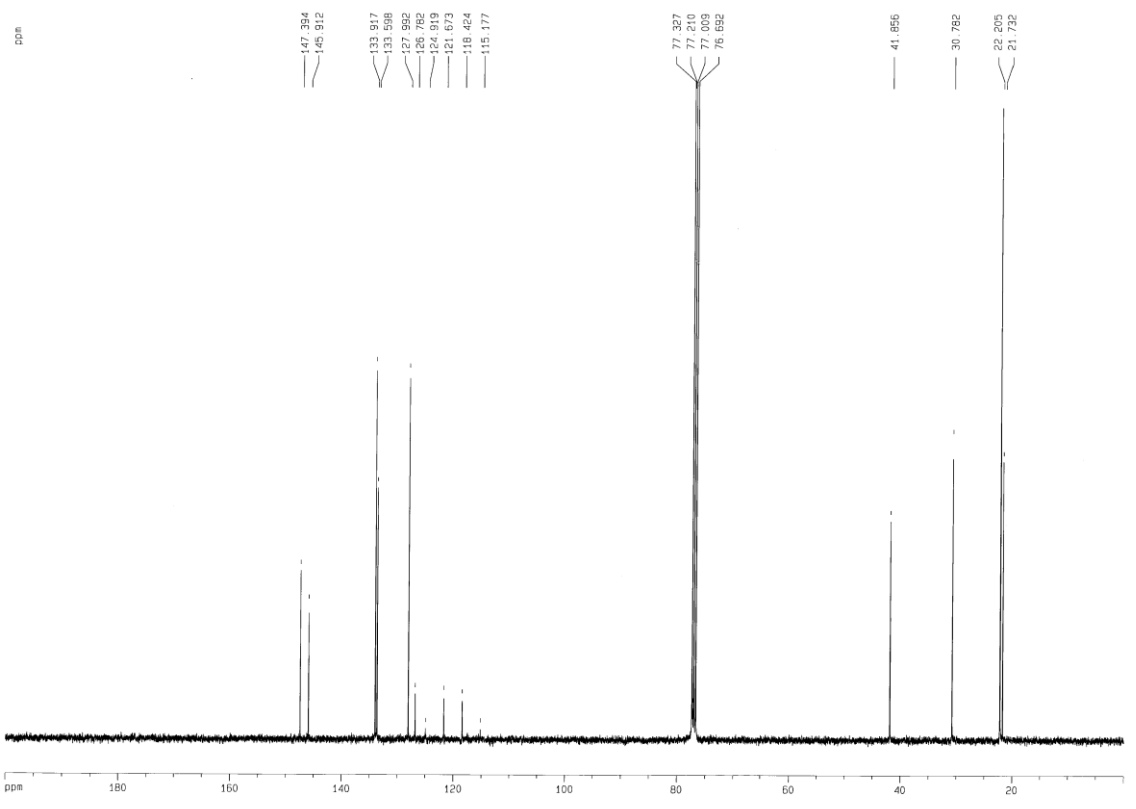
Current Data Parameters
NAME CBA06-BiBu
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110205
Time 14.21
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 256
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 12.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA06-BiBu
EXPNO 2
PROCNO 1

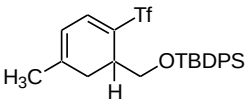
F2 - Acquisition Parameters
Date_ 20110205
Time 14.24
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1063
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 8192
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

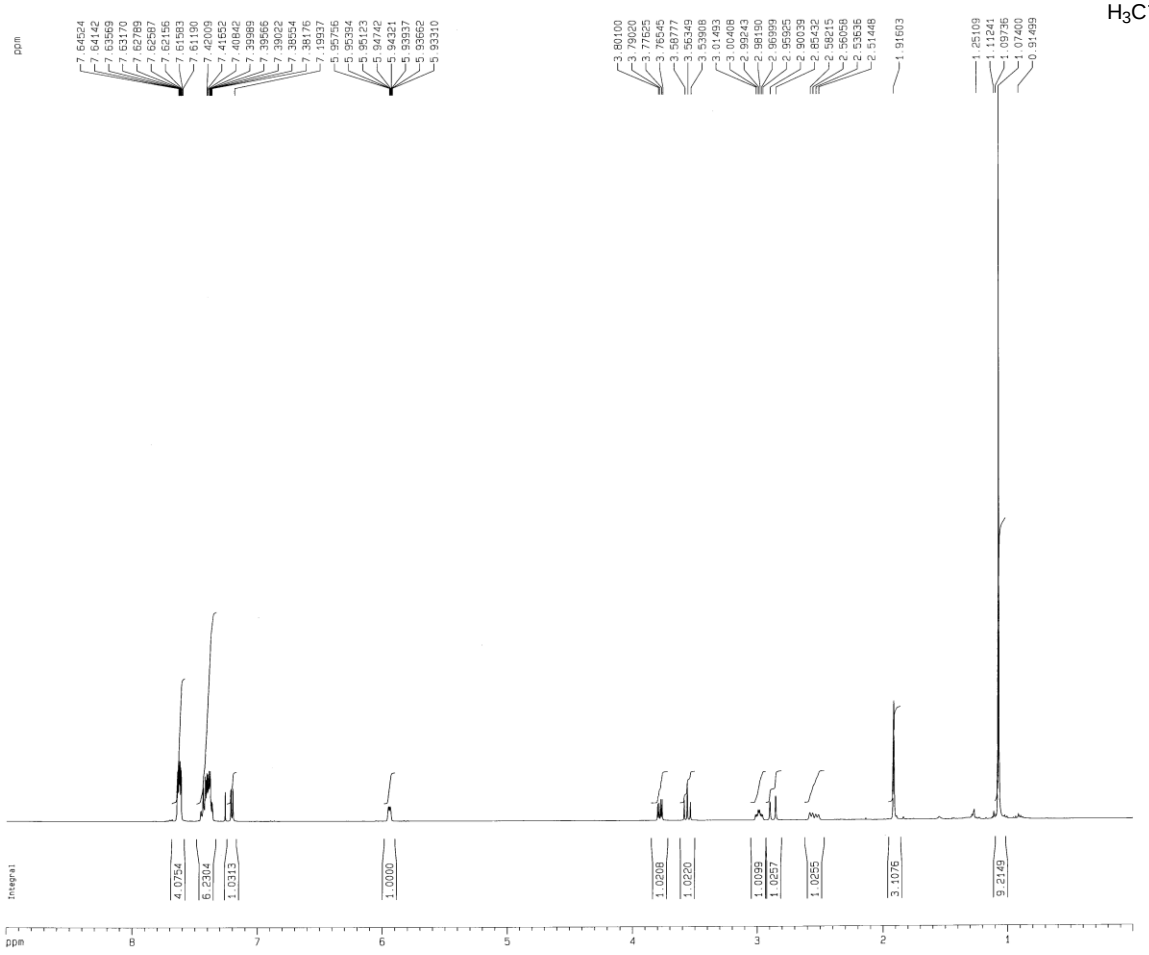
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

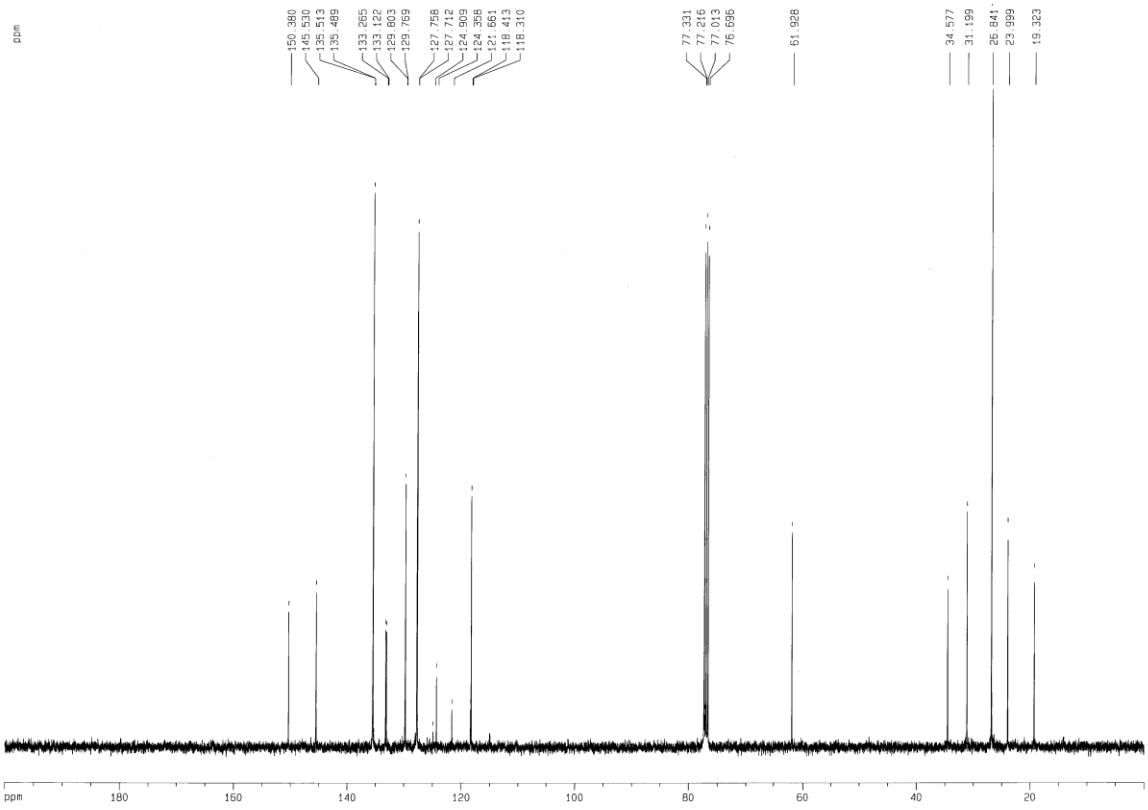
F2 - Processing parameters
SI 32768
SF 100.5876230 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm

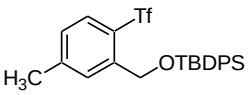


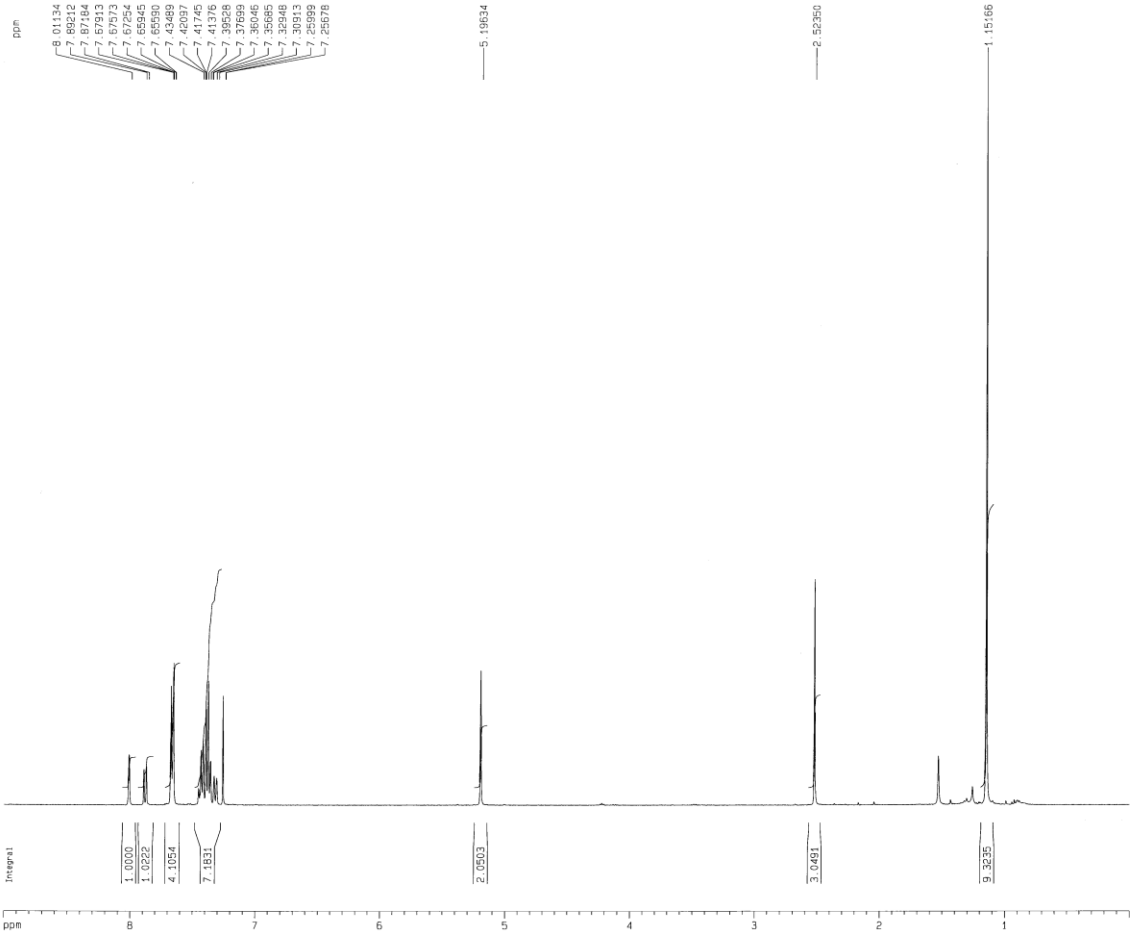



```
Current Data Parameters
NAME       CBA6-D-OTBDPS
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20110215
Time              23.00
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   11
DS                    2
SWH           8278.146 Hz
FIDRES        0.126314 Hz
AQ            3.9584243 sec
RG                161.3
DW               60.400 usec
DE                6.00 usec
TE               300.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                11.50 usec
PL1               -4.00 dB
SFO1        400.0324703 MHz

F2 - Processing parameters
SI                32768
SF        400.0300065 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                32.00 cm
CY                21.00 cm
F1P               9.000 ppm
F1              3600.27 Hz
F2P               0.000 ppm
F2                0.00 Hz
PPMCM         0.28125 ppm/cm
HZCM         112.50844 Hz/cm
```




```
Current Data Parameters
NAME       CBA6-D-OTBDPS
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20110215
Time              23.06
INSTRUM           spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                  272
DS                    4
SWH          26178.010 Hz
FIDRES        0.399445 Hz
AQ            1.2517875 sec
RG                 8192
DW               19.100 usec
DE                6.00 usec
TE               300.2 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               10.00 usec
PL1               -3.50 dB
SFO1        100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2               -4.00 dB
PL12              17.00 dB
PL13              17.00 dB
SFO2        400.0316001 MHz

F2 - Processing parameters
SI                32768
SF        100.5876246 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40

1D NMR plot parameters
CX                32.00 cm
CY                19.00 cm
F1P             200.000 ppm
F1            20117.53 Hz
F2P              -0.000 ppm
F2               -0.00 Hz
PPMCM         6.25000 ppm/cm
HZCM         628.67267 Hz/cm
```





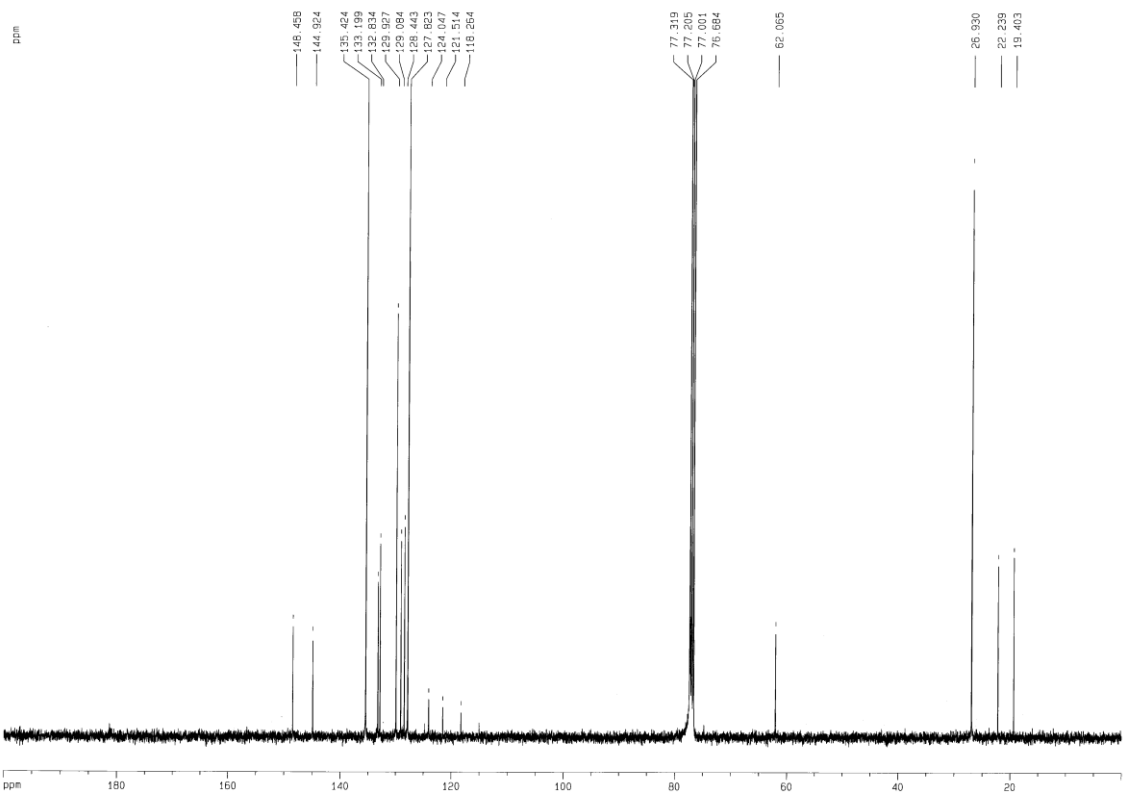
Current Data Parameters
NAME CBA6-OTBDPS
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110215
Time 12.33
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 574.7
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 21.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA6-OTBDPS
EXPNO 2
PROCNO 1

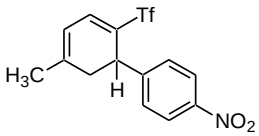
F2 - Acquisition Parameters
Date_ 20110215
Time 12.37
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 4004
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 6502
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

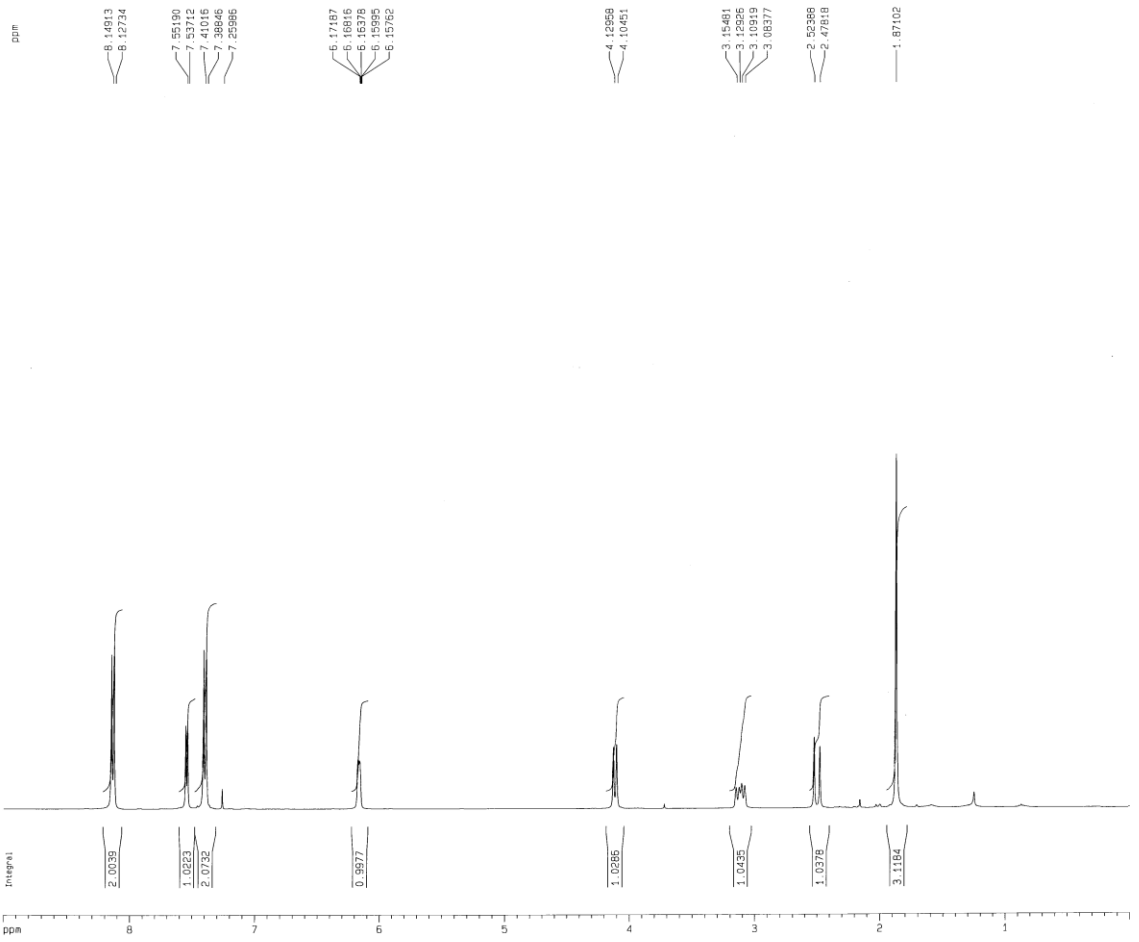
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876240 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 25.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm





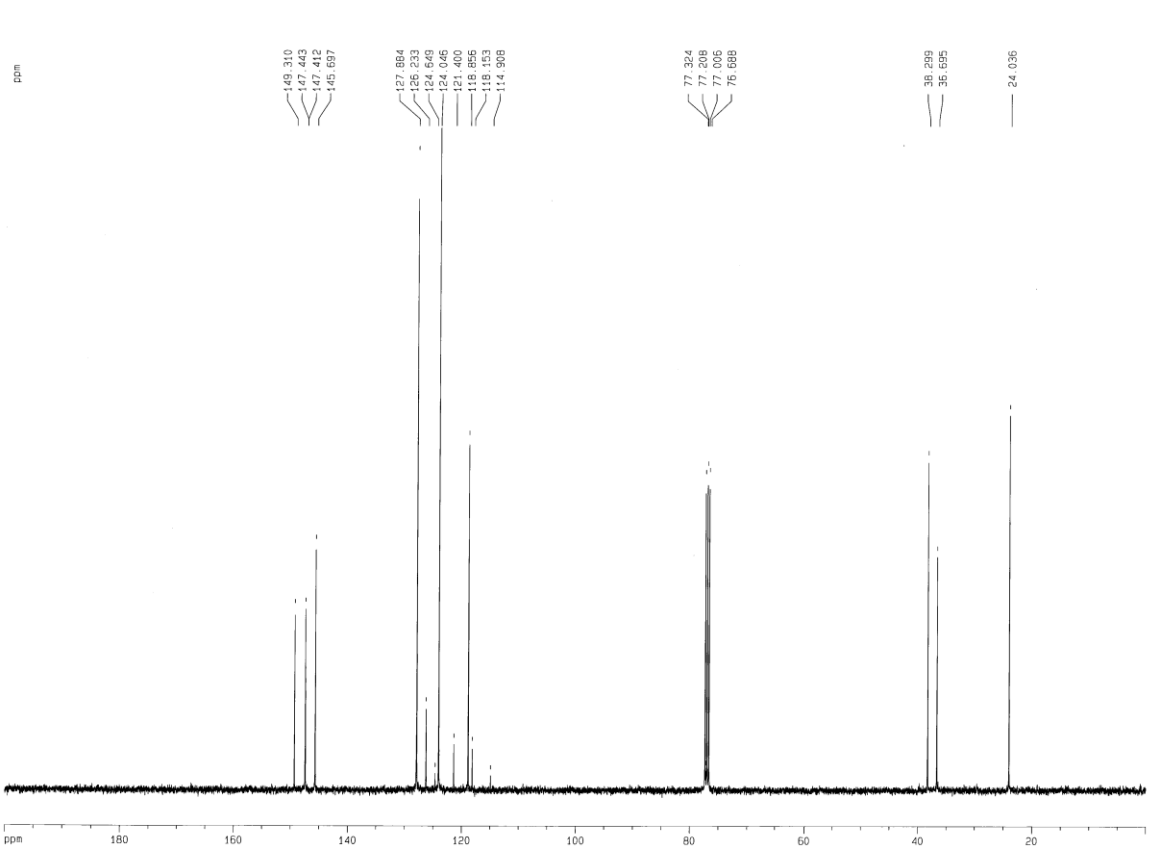
Current Data Parameters
NAME CBA06-DNO2
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110209
Time 12.17
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 15
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 143.7
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 10.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA06-DNO2
EXPNO 2
PROCNO 1

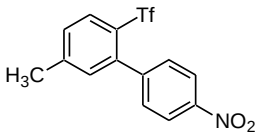
F2 - Acquisition Parameters
Date_ 20110209
Time 12.21
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 354
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 6502
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

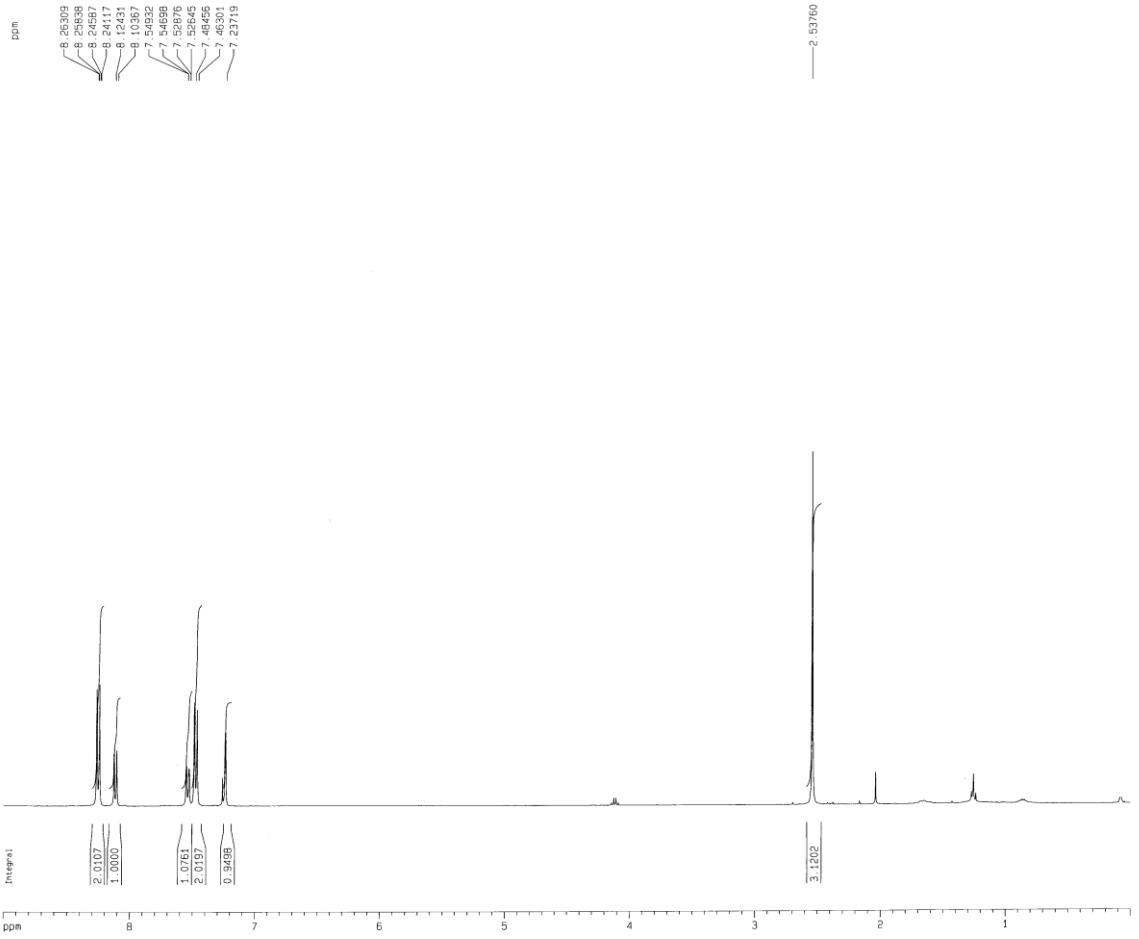
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

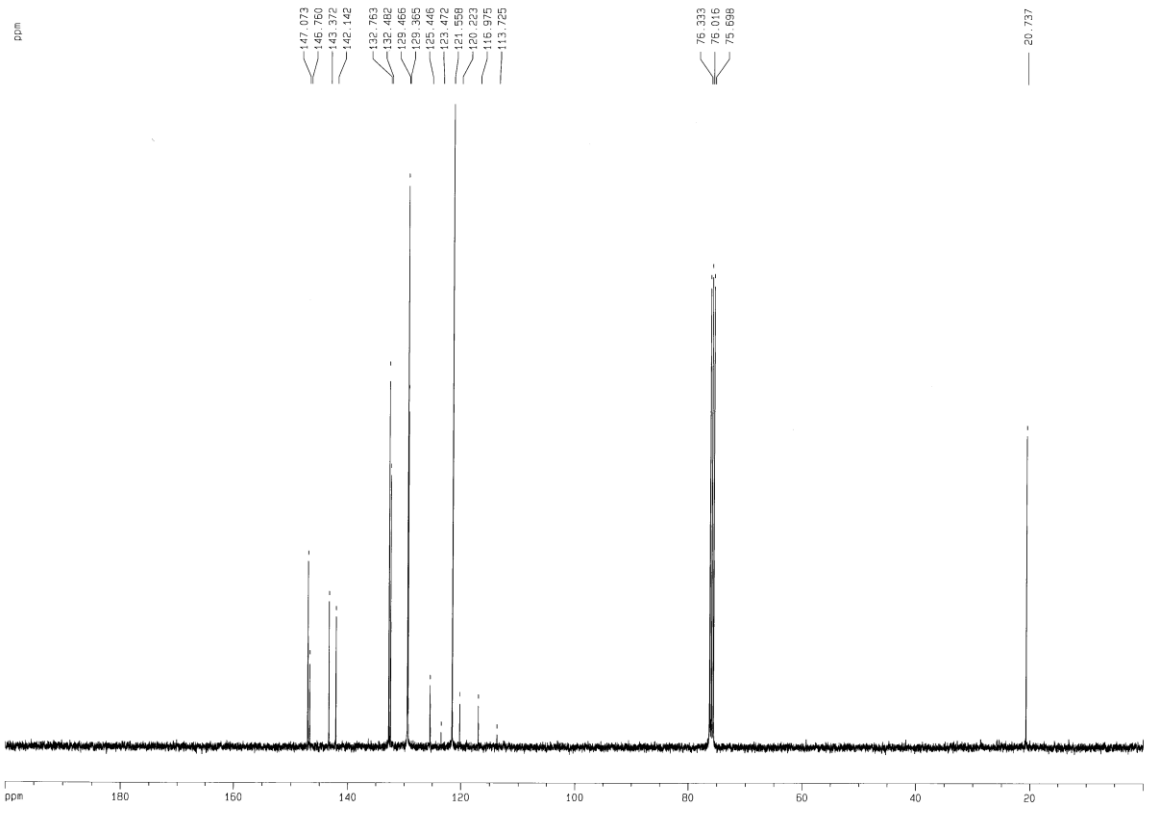
F2 - Processing parameters
SI 32768
SF 100.5876278 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 19.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67273 Hz/cm

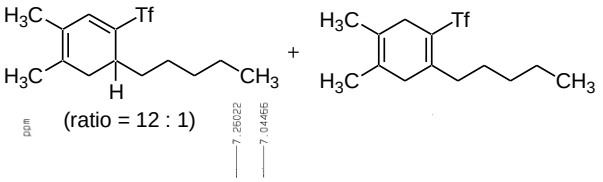


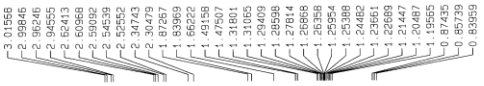


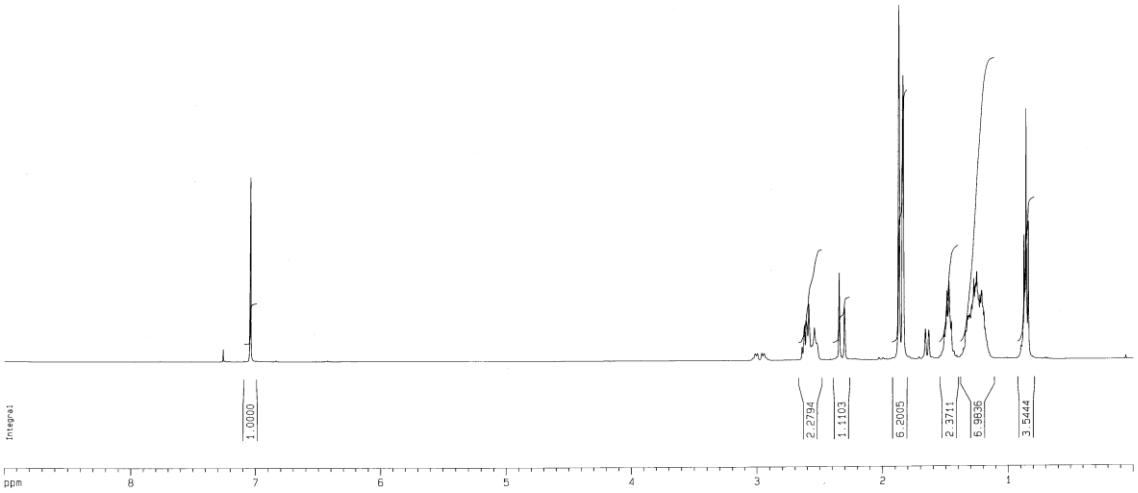

```
Current Data Parameters
NAME                  CBA6-BNO2
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_               20110210
Time                    13.54
INSTRUM                 spect
PROBHD   5 mm QNP 1H/13
PULPROG                 zg30
TD                     65536
SOLVENT                 CDCl3
NS                          5
DS                          2
SWH                8278.146 Hz
FIDRES            0.126314 Hz
AQ                3.9584243 sec
RG                      228.1
DW                   60.400 usec
DE                    6.00 usec
TE                   300.2 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      11.50 usec
PL1                     -4.00 dB
SFO1              400.0324703 MHz

F2 - Processing parameters
SI                         32768
SF                400.0300065 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00

1D NMR plot parameters
CX                         32.00 cm
CY                         10.00 cm
F1P                        9.000 ppm
F1                      3600.27 Hz
F2P                        0.000 ppm
F2                         0.00 Hz
PPMCM                  0.28125 ppm/cm
HZCM                 112.50844 Hz/cm
```



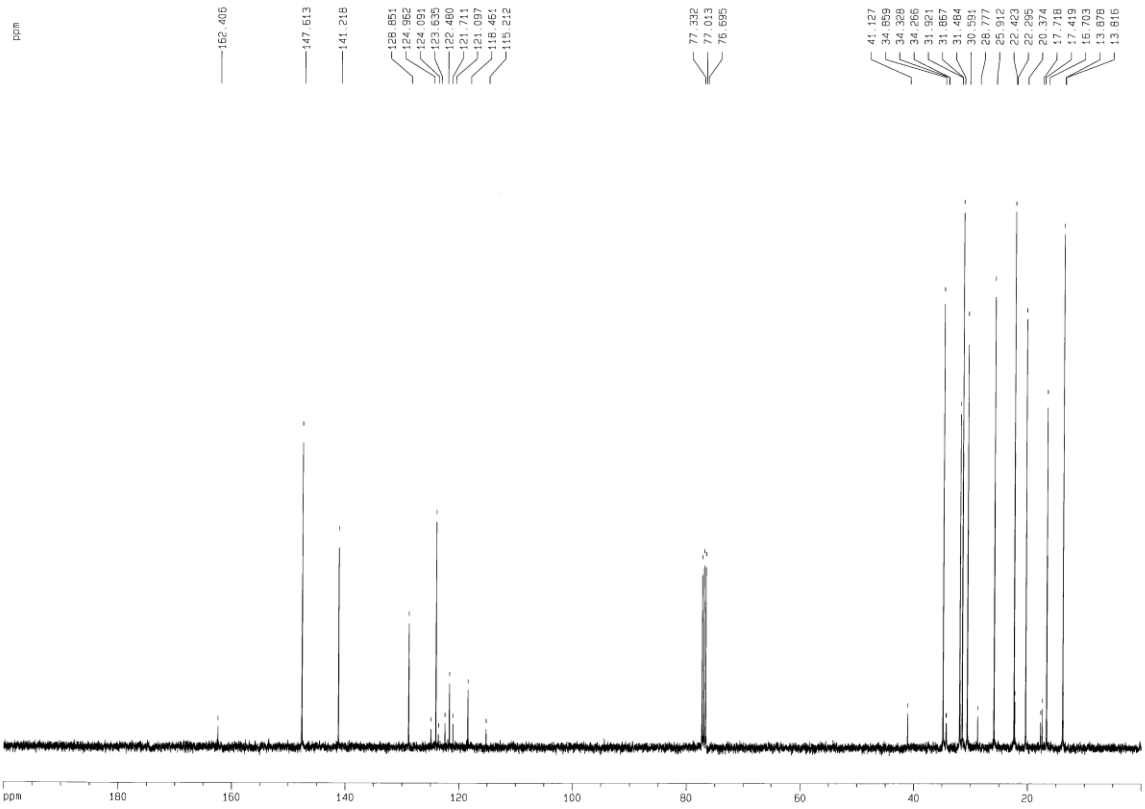

```
Current Data Parameters
NAME                  CBA6-BNO2
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_               20110210
Time                    13.56
INSTRUM                 spect
PROBHD   5 mm QNP 1H/13
PULPROG               zgpg30
TD                     65536
SOLVENT                 CDCl3
NS                        566
DS                          4
SWH               26178.010 Hz
FIDRES            0.399445 Hz
AQ                1.2517875 sec
RG                      8192
DW                   19.100 usec
DE                    6.00 usec
TE                   300.2 K
D1                2.00000000 sec
d11               0.03000000 sec
DELTA             1.89999998 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.00 usec
PL1                     -3.50 dB
SFO1              100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                     -4.00 dB
PL12                    17.00 dB
PL13                    17.00 dB
SFO2              400.0316001 MHz

F2 - Processing parameters
SI                         32768
SF                100.5877255 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40

1D NMR plot parameters
CX                         32.00 cm
CY                         19.00 cm
F1P                      200.000 ppm
F1                     20117.54 Hz
F2P                       -0.000 ppm
F2                        -0.00 Hz
PPMCM                  6.25000 ppm/cm
HZCM                 628.67328 Hz/cm
```



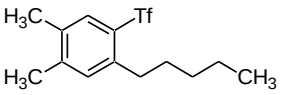


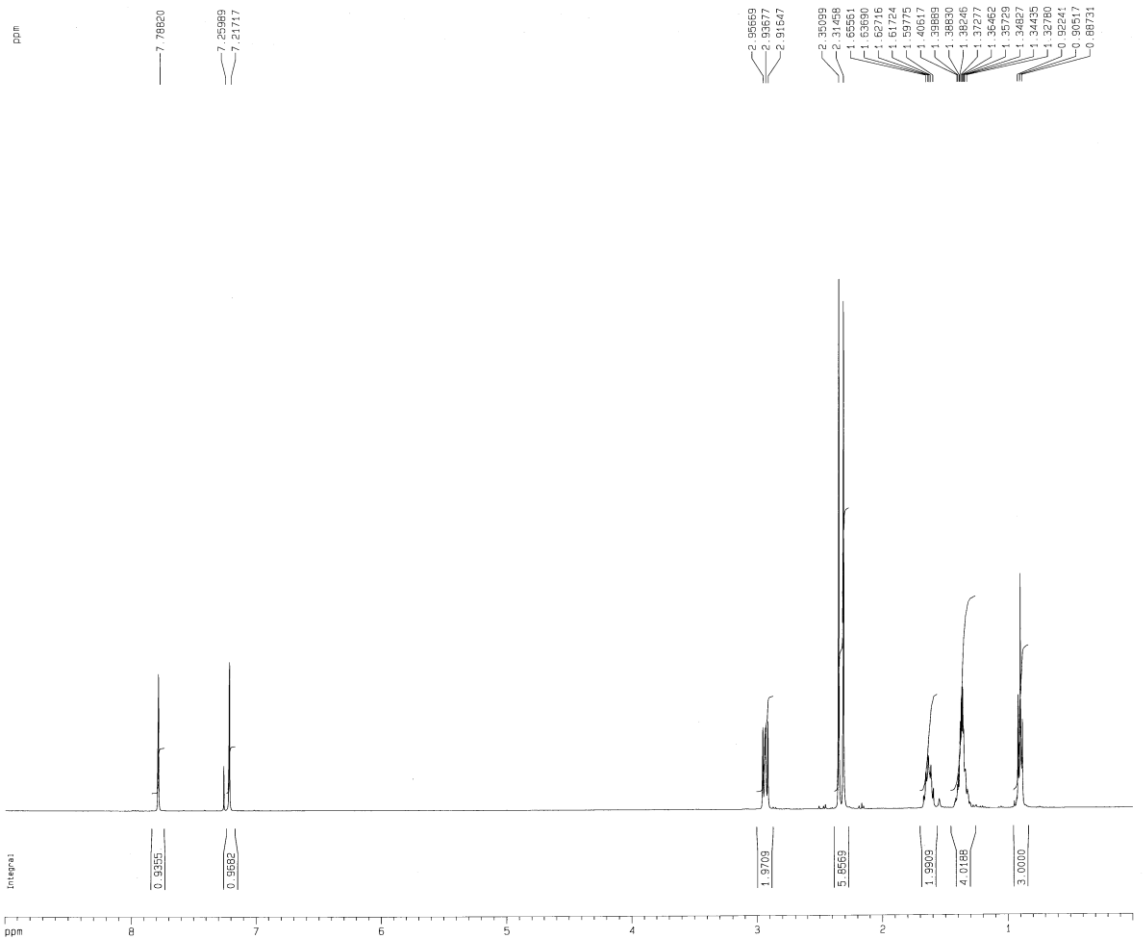



```
Current Data Parameters
NAME          CBA06-DMe2
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_        20110203
Time             17.25
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                 3
DS                 2
SWH          8278.146 Hz
FIDRES       0.126314 Hz
AQ           3.9584243 sec
RG                40.3
DW              60.400 usec
DE               6.00 usec
TE              300.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1              11.50 usec
PL1             -4.00 dB
SFO1       400.0324703 MHz

F2 - Processing parameters
SI             32768
SF         400.0300065 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00

1D NMR plot parameters
CX             32.00 cm
CY             10.00 cm
F1P          9.000 ppm
F1           3600.27 Hz
F2P          0.000 ppm
F2             0.00 Hz
PPMCM        0.28125 ppm/cm
HZCM         112.50844 Hz/cm
```




```
Current Data Parameters
NAME          CBA06-DMe2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_        20110203
Time             17.28
INSTRUM          spect
PROBHD   5 mm QNP 1H/13
PULPROG          zgpg30
TD              65536
SOLVENT          CDCl3
NS                53
DS                 4
SWH          26178.010 Hz
FIDRES       0.399445 Hz
AQ           1.2517875 sec
RG              2580.3
DW              19.100 usec
DE               6.00 usec
TE              300.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1              10.00 usec
PL1             -3.50 dB
SFO1       100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2           100.00 usec
PL2             -4.00 dB
PL12            17.00 dB
PL13            17.00 dB
SFO2       400.0316001 MHz

F2 - Processing parameters
SI             32768
SF         100.5876246 MHz
WDW               EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40

1D NMR plot parameters
CX             32.00 cm
CY             15.00 cm
F1P          200.000 ppm
F1           20117.53 Hz
F2P          -0.000 ppm
F2             -0.00 Hz
PPMCM        6.25000 ppm/cm
HZCM         628.67267 Hz/cm
```





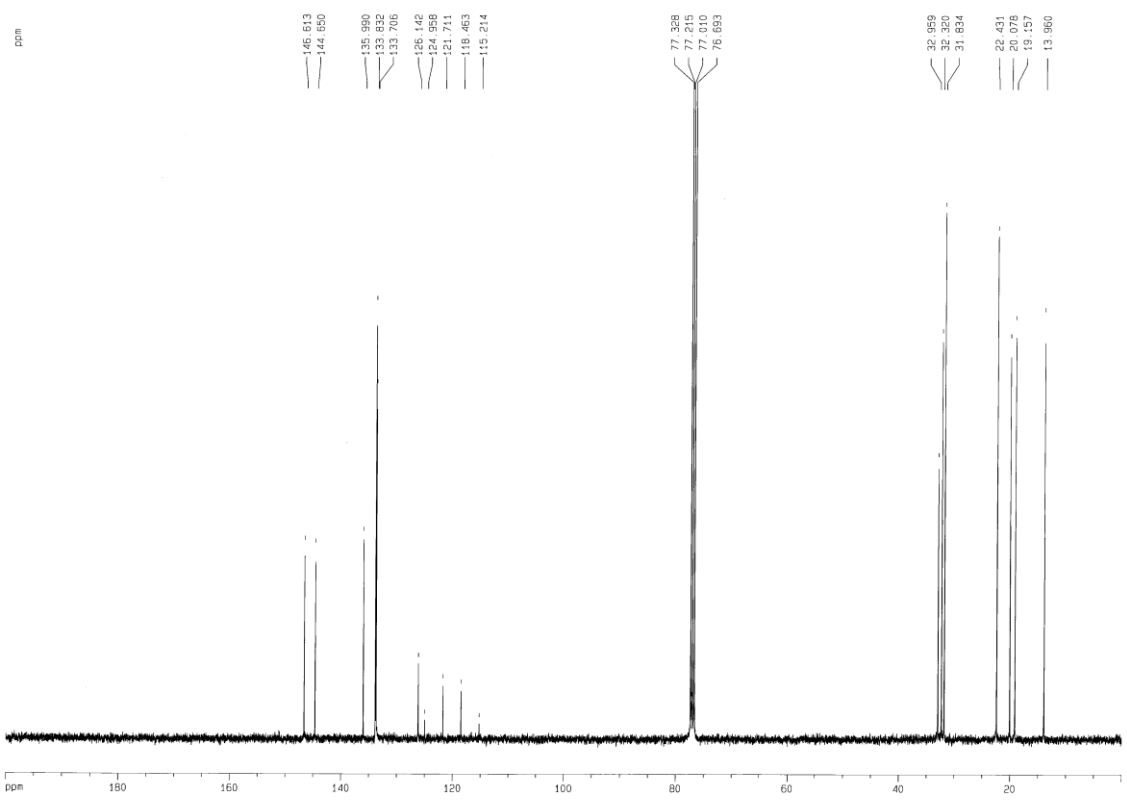
Current Data Parameters
NAME CBA06-B2Me
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110208
Time 17.50
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8278.146 Hz
FIDRES 0.126314 Hz
AQ 3.9584243 sec
RG 181
DW 60.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.50 usec
PL1 -4.00 dB
SFO1 400.0324703 MHz

F2 - Processing parameters
SI 32768
SF 400.0300067 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 9.000 ppm
F1 3600.27 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 0.28125 ppm/cm
HZCM 112.50844 Hz/cm



Current Data Parameters
NAME CBA06-B2Me
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110208
Time 17.52
INSTRUM spect
PROBHD 5 mm GNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 996
DS 4
SWH 26178.010 Hz
FIDRES 0.399445 Hz
AQ 1.2517875 sec
RG 9195.2
DW 19.100 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 -3.50 dB
SFO1 100.5986886 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -4.00 dB
PL12 17.00 dB
PL13 17.00 dB
SFO2 400.0316001 MHz

F2 - Processing parameters
SI 32768
SF 100.5876230 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 32.00 cm
CY 15.00 cm
F1P 200.000 ppm
F1 20117.53 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 6.25000 ppm/cm
HZCM 628.67267 Hz/cm

6. References

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