Copper(I)-Catalyzed Borylative *Exo*-Cyclization of Alkenyl Halides Containing Unactivated Double Bond

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Table of Contents.

1. General and Materials.

Materials were obtained from commercial suppliers and purified by standard procedures unless otherwise noted. Solvents were also purchased from commercial suppliers, degassed via three freeze-pump-thaw cycles, and further dried over molecular sieves (MS 4A). NMR spectra were recorded on JEOL JNM-ECX400P spectrometer (^{1}H: 400 MHz and ^{13}C: 100 MHz). Tetramethylsilane (^{1}H) and $CDCl_3$ (^{31}C) were employed as external standards, respectively. CuCl (ReagentPlus® grade, 224332-25G, ≥99%) and K(O-*t*-Bu)/THF (1.0 M, 328650-50ML) were purchased from Sigma-Aldrich Co. and used as received. 1,4-Diisopropylbenzene was used as an internal standard to determine GC yield. GLC analyses were conducted with a Shimadzu GC-2014 or GC-2025 equipped with ULBON HR-1 glass capillary column (Shinwa Chemical Industries) and a FID detector. HPLC analyses with chiral stationary phase were carried out using a Hitachi LaChrome Elite HPLC system with a L-2400 UV detector. Recycle preparative gel permeation chromatography was conducted with a JAI LC-9101 using $CHCl_3$ as the eluent. Elemental analyses and high-resolution mass spectra were recorded at the Center for Instrumental Analysis, Hokkaido University.

2. General Experimental Procedures.

A Representative Procedure for the Copper(I)-Catalyzed Hydroboration of 1a (Table 1):

Copper chloride (2.5 mg, 0.025 mmol) and bis(pinacolato)diboron (152.4 mg, 0.6 mmol), Xantphos (14.5 mg, 0.025 mmol) were placed in an oven-dried reaction vial. After the vial was sealed with a screw cap containing a teflon-coated rubber septum, the vial was connected to a vacuum/nitrogen manifold through a needle. It was evacuated and then backfilled with nitrogen. This cycle was repeated three times. THF (0.4 mL) and K(O-*t*-Bu)/THF (1.0 M, 0.6 mL, 0.6 mmol) were added in the vial through the rubber septum. After 1-hexene (**1a**, 42 mg, 0.5 mmol) was added to the mixture at 30 °C, MeOH (415 μL, 1.0 mmol) was added dropwise. After the reaction was complete, the reaction mixture was passed through a short silica gel column eluting with Et_2O/hexane (20:80). The crude mixture was further purified by flash column chromatography (SiO_2, Et_2O/hexane, 0:100–4:96) to give the corresponding alkylboronate **3a** as a colorless oil. The flash column chromatography should be done within 5 min after the crude mixture was applied on the silica gel surface; otherwise the products are obtained in low yield.

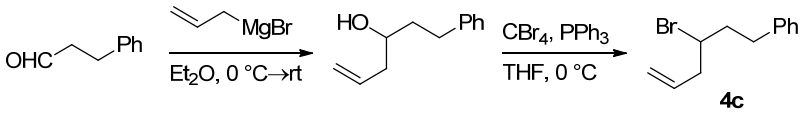
A Representative Procedure for the Copper(I)-Catalyzed Borylative Cyclization of Alkenyl Halides 4.

Copper chloride (2.5 mg, 0.025 mmol) and bis(pinacolato)diboron (152.4 mg, 0.6 mmol), Xantphos (14.5 mg, 0.025 mmol) were placed in an oven-dried reaction vial. The vial was sealed with a screw cap containing a teflon-coated rubber septum. The vial was connected to a vacuum/nitrogen manifold through a needle, evacuated and backfilled with nitrogen. THF (0.4 mL) and K(O-*t*-Bu)/THF (1.0 M, 0.6 mL, 0.6 mmol) were added in the vial through the rubber septum. Then alkenyl halide **4** (0.5 mmol) was added dropwise at 30 °C. After the reaction was complete, the reaction mixture was passed through a short silica gel column eluting with Et_2O/hexane (20:80). The crude mixture was further purified by flash column chromathography (SiO_2, Et_2O/hexane, 0:100–4:96) to give the corresponding cyclization product **5**.

3. Preparation of Substrates.

The starting materials (**1a**, **4a**, **4b**, **4m**, and **4p**) were purchased from commercial suppliers. (*E*)-1-Bromohex-3-ene (**4p**) was prepared by bromination of (*E*)-hex-3-en-1-ol with CBr_4/PPh_3 reagents.[1] The starting materials were dried over MS4A before use, without further purification.

Preparation of (3-bromohex-5-en-1-yl)benzene (4c).

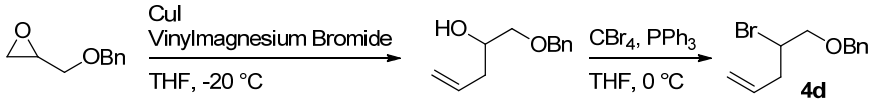


In a vacuum dried 100 mL round bottomed flask, 3-phenylpropanal (1.3 mL, 10 mmol) was dissolved in dry Et_2O (10 mL) and was cooled to 0 °C under nitrogen atmosphere. A Et_2O solution of allyl magnesium bromide (1.3 M, 11.5 mL, 15 mmol) was then added dropwise for 10 min. After stirred for 3 h, the reaction mixture was quenched by addition of saturated NH_4Cl aq. and extracted with Et_2O three times. The combined organic layer was then dried over $MgSO_4$. After filtration, the solvents were removed by evaporation. The crude product was purified by flash column chromatography to obtain the corresponding homoallylic alcohol (1.058 g, 6.0 mmol, 60%) as a colorless oil.

In a 50 mL round bottomed flask, CBr_4 (1.094 g, 3.3 mmol) and the homoallylic alcohol (529 mg, 3.0 mmol) were dissolved in dry THF (12 mL) and mixture was cooled to 0 °C. PPh_3 (866 mg, 3.3 mmol) was then added portion wise and the reaction mixture was stirred for 20 min. The reaction mixture was quenched by addition of water and extracted three times with Et_2O. The combined organic layer was dried over $MgSO_4$. After filtration, the solvents were removed by evaporation. The hexane suspension of the crude mixture was filtered and concentrated by evaporation. The crude product was purified by silica gel chromatography and bulb-to-bulb distillation (20 Pa, 90 °C) to obtain **4c** (395 mg, 1.65 mmol, 55%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 2.08–2.13 (m, 2H), 2.64 (t, J = 6.8 Hz, 2H), 2.75 (dt, J = 8.2, 13.9 Hz, 1H), 2.91 (dt, J = 7.0, 13.9 Hz, 1H), 3.99 (quin, J = 6.7 Hz, 1H), 5.10–5.14 (m, 2H), 5.78–5.88 (m, 1H), 7.19–7.31 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 33.6 (CH_2), 39.9 (CH_2), 43.3 (CH_2), 55.3 (*C*H), 117.9 (CH_2), 126.0 (*C*H), 128.4 (*C*H), 128.5 (*C*H), 134.6 (*C*H), 140.8 (*C*). HRMS–EI (m/z): $[M–Br]^+$ calcd for $C_{12}H_{15}$, 159.11737; found, 159.11655. Anal. Calcd for $C_{12}H_{15}B_r$: C, 60.27; H, 6.32. Found: C, 60.16; H, 6.37.

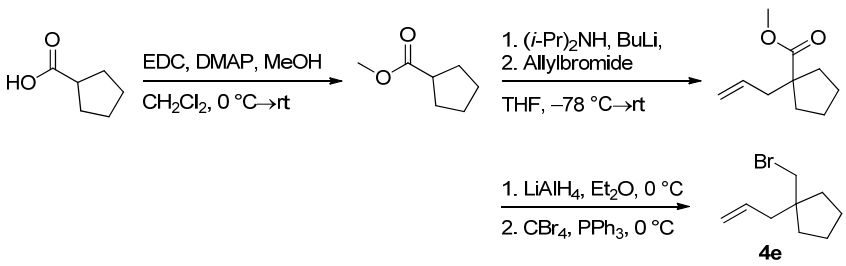
Preparation of {[(2-bromopent-4-en-1-yl)oxy]methyl}benzene (4d).



Anhydrous copper(I) iodide (285.7 mg, 1.5 mmol) was placed into a 100 mL round-bottomed flask equipped with a mechanical stirrer and a low-temperature thermometer. Then 4 mL of THF were added and the flask was cooled to –20 °C. Vinylmagnesium bromide (1.0 M in THF, 30 mL, 30 mmol) was added dropwise. After stirring for 20 min at –20 °C, benzyl glycidyl ether (2.3 mL, 15 mmol) in 2 mL of THF was added dropwise. After stirring for 12 h at –20 °C, the reaction mixture was quenched by addition of saturated NH_4Cl aq. and extracted with Et_2O three times. The combined organic layer was dried over Na_2SO_4. After filtration, the solvents were removed by evaporation. The crude product was purified by flash column chromatography to obtain the homoallylic alcohol (2.556 g, 13.3 mmol, 89%) as a colorless oil. **4d** was then synthesized by bromination with CBr4/PPh3 reagents according to the same procedure described above (445 mg, 1.7 mmol, 35%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 2.54–2.62 (m, 1H), 2.74–2.81 (m, 1H), 3.65–3.75 (m, 2H), 4.09–4.16 (m, 1H), 4.58 (s, 2H), 5.12–5.17 (m, 2H), 5.78–5.88 (m, 1H), 7.28–7.39 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 37.8 (*C*H_2), 69.6 (*C*H), 73.3 (*C*H_2), 73.8 (*C*H_2), 117.6 (*C*H_2), 127.66 (*C*H), 127.71 (*C*H), 128.4 (*C*H), 134.2 (*C*H), 137.9 (*C*). HRMS–EI (*m*/*z*): $[M+Na]^+$ calcd for $C_{12}H_{15}BrO$, 277.02040; found, 277.02013. Anal. Calcd for $C_{12}H_{15}BrO$: C, 56.49; H, 5.93. Found: C, 56.25; H, 5.86.

Preparation of 1-allyl-1-(bromomethyl)cyclopentane (4e).



In a vacuum dried 300 mL of a round bottomed flask, cyclopentanecarboxylic acid (2.17 mL, 20 mmol) and MeOH (971 μL, 24 mmol) were dissolved in dry CH_2Cl_2 (110 mL) and the flask was cooled to 0 °C under nitrogen atmosphere. 1-Ethyl-3-(3-dimethylaminopropyl)carbodiimide (EDC) (4.60 g, 24 mmol) and *N*,*N*-dimethyl-4-aminopyridine (DMAP) (2.44 g, 20 mmol) were then added portion wise. After stirred for 4 h at room temperature, the reaction mixture was quenched by addition of saturated NH_4Cl aq. and extracted with CH_2Cl_2 three times. The combined organic layer was then dried over $MgSO_4$. After filtration, the solvents were removed by evaporation. The crude

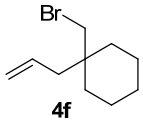
product was purified by flash column chromatography to obtain the corresponding methyl ester (1.839 g, 14.3 mmol, 72%) as a colorless oil.

To a 0 °C solution of *i*-Pr_2NH (2.0 mL, 14.5 mmol) in THF (15 mL) was added a hexane solution of *n*-BuLi (1.62 M, 6.8 mL, 11 mmol) dropwise. The reaction mixture was stirred for 20 min and then cooled to –78 °C. The methyl ester (1.282 g, 10 mmol) was added dropwise and the reaction mixture was stirred for 1 h at –78 °C. Allylbromide (1.3 mL, 15 mmol) was then added dropwise into the mixture. It was warmed to room temperature and stirred for 1 h. The reaction mixture was quenched by addition of saturated NH_4Cl aq. and extracted with Et_2O three times. The combined organic layer was then dried over $MgSO_4$. After filtration, the solvents were removed by evaporation. The crude product was purified by flash column chromatography to obtain the corresponding methyl ester (1.009 g, 6.0 mmol, 60%) as a colorless oil.

To a slurry of $LiAlH_4$ (342 mg, 9 mmol) in Et_2O (10 mL) was added a solution of the methyl ester (1.001g, 6 mmol) in Et_2O (6 mL) dropwise at 0 °C. After stirred for 2 h, the reaction mixture was quenched by addition of water and stirred until a white solid was formed. The mixture was filtered and dried over $MgSO_4$. The solvents were removed by evaporation under reduced pressure to obtain the alcohol (789 mg, 5.6 mmol, 94%) with acceptable purity. **4e** was then synthesized by bromination with CBr4/PPh3 reagents according to the same procedure described for **4d** (224 mg, 1.1 mmol, 22%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.49–1.69 (m, 8H), 2.23 (dt, *J* = 1.1, 6.7 Hz, 2H), 3.37 (s, 2H), 5.76 (ddt, *J* = 7.7, 10.2, 17.6 Hz, 2H), 5.70–5.81 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 25.0 (CH_2), 36.2 (CH_2), 42.1 (CH_2), 44.1 (CH_2), 46.6 (*C*), 118.0 (CH_2), 134.6 (*C*H). HRMS–EI (*m*/*z*): $[M–C_3H_6]^+$ calcd for C_6H_9Br, 159.98875; found, 159.98845.

Preparation of 1-allyl-1-(bromomethyl)cyclohexane (4f).

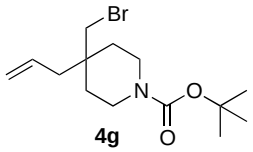


4f

4f was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.36–1.48 (m, 10H), 2.16 (d, *J* = 7.7 Hz, 2H), 3.37 (s, 2H), 5.09–5.16 (m, 2H), 5.75 (ddt, *J* = 7.7, 10.2, 17.6 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 21.5 (CH_2), 26.0 (CH_2), 33.9 (CH_2), 36.7 (*C*), 40.1 (CH_2), 43.8 (CH_2), 118.2 (CH_2), 133.5 (*C*H). HRMS–EI (*m*/*z*): $[M–C_3H_5]^+$ calcd for $C_7H_{12}Br$, 175.01223; found, 175.01211. Anal. Calcd for $C_{10}H_{17}Br$: C, 55.31; H, 7.89. Found: C, 55.09; H, 7.87.

Preparation of *tert*-butyl 4-allyl-4-(bromomethyl)piperidine-1-carboxylate (4g).

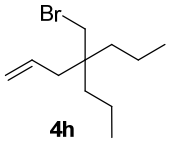


4g

4g was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.46 (s, 9H), 1.48–1.56 (m, 4H), 2.23 (d, J = 7.8 Hz, 2H), 3.29–3.51 (m, 6H), 5.12–5.19 (m, 2H), 5.66–5.78 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 28.4 (CH_3), 33.2 (CH_2), 35.5 (C), 39.0 (CH_2), 41.9 (CH_2), 79.5 (C), 119.2 (CH_2), 132.4 (CH), 154.8 (C). HRMS–ESI (m/z): $[M+Na]^+$ calcd for $C_{14}H_{24}BrNO_2Na$, 340.08826; found, 340.08825.

Preparation of 4-(bromomethyl)-4-propylhept-1-ene (4h).

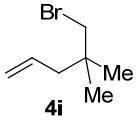


4h

4h was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.87–0.93 (m, 6H), 1.18–1.30 (m, 8H), 2.07 (dt, J = 1.1, 7.7 Hz, 2H), 3.27 (s, 2H), 5.72 (ddt, J = 7.6, 10.2, 17.6 Hz, 2H), 5.67–5.77 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 14.7 (CH_3), 16.3 (CH_2), 37.1 (CH_2), 39.4 (CH_2), 39.5 (C), 42.4 (CH_2), 118.1 (CH_2), 133.7 (CH). HRMS–EI (m/z): $[M–C_3H_5]^+$ calcd for $C_8H_{16}Br$, 191.04353; found, 191.04340.

Preparation of 5-bromo-4,4-dimethylpent-1-ene (4i).

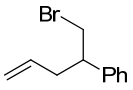


4i

4i was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.02 (s, 6H), 2.10 (d, J = 7.7 Hz, 2H), 3.28 (s, 2H), 5.07–5.12 (m, 2H), 5.72–5.83 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 25.6 (CH_3), 34.8 (C), 44.1 (CH_2), 46.2 (CH_2), 118.1 (CH_2), 134.1 (CH). HRMS–EI (m/z): $[M]^+$ calcd for $C_7H_{13}Br$, 176.02006; found, 176.01966.

Preparation of (1-bromopent-4-en-2-yl)benzene (4j).

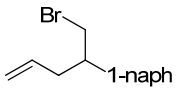


4j

4j was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 2.42–2.50 (m, 1H), 2.61–2.68 (m, 1H), 3.02–3.09 (m, 1H), 3.56–3.64 (m, 2H), 4.98–5.08 (m, 2H), 5.65 (ddt, *J* = 7.2, 10.3, 17.5 Hz, 1H), 7.18–7.36 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 38.0 (CH_2), 38.3 (CH_2), 47.4 (*C*H), 117.2 (CH_2), 127.0 (*C*H), 127.6 (*C*H), 128.4 (*C*H), 135.3 (*C*H), 141.7 (*C*). HRMS–EI (*m*/*z*): $[M–C_3H_5]^+$ calcd for C_8H_8Br, 182.98093; found, 182.98085. Anal. Calcd for $C_{11}H_{13}Br$: C, 58.69; H, 5.82. Found: C, 58.42; H, 5.83.

Preparation of 1-(1-bromopent-4-en-2-yl)naphthalene (4k).

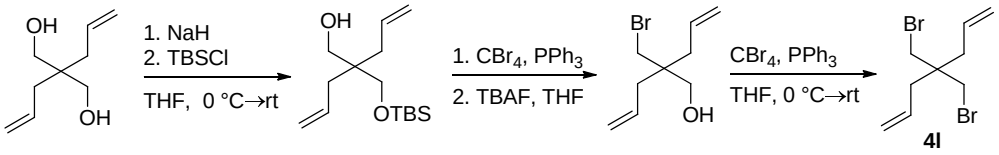


4k

4k was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 2.63–2.71 (m, 1H), 2.81–2.89 (m, 1H), 3.68–3.78 (m, 2H), 3.99 (quin, *J* = 7.7 Hz, 1H), 4.98–5.14 (m, 2H), 5.69 (ddt, *J* = 7.1, 10.3, 17.4 Hz, 1H), 7.37 (dd, *J* = 0.7, 7.3 Hz, 1H), 7.45–7.57 (m, 3H), 7.78 (d, *J* = 8.4 Hz, 1H), 7.87–7.89 (m, 1H), 8.06 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 37.5 (CH_2), 37.6 (CH_2), 41.0 (*C*H), 117.4 (CH_2), 122.6 (*C*H), 123.8 (*C*H), 125.2 (*C*H), 125.5 (*C*H), 126.3 (*C*H), 127.5 (*C*H), 129.1 (*C*H), 131.6 (*C*), 134.0 (*C*), 135.2 (*C*H), 137.3 (*C*). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{15}H_{15}Br$, 274.03571; found, 274.03529. Anal. Calcd for $C_{15}H_{15}Br$: C, 65.47; H, 5.49. Found: C, 65.47; H, 5.54.

Preparation of 4,4-bis(bromomethyl)hepta-1,6-diene (4l).



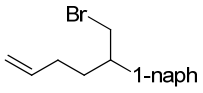
4l

The starting material, (2,2-diallylpropane-1,3-diol), was prepared by $LiAlH_4$ reduction of the diethyl diallylmalonate. To a suspension of NaH (60wt.%, 400 mg, 10 mmol) in THF (20 mL) was added dropwise the diol (1.562 g, 10 mmol) at room temperature, TBSCl (1.73 mL, 10 mmol) was then added and the reaction mixture was stirred for 6 h. The resulting suspension was diluted with ether and quenched with saturated aqueous Na_2CO_3. The mixture was extracted with Et_2O three times and dried over $MgSO_4$, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to afford the silyl protected product (2.587 g, 9.6 mmol, 96%) as a colorless oil. The monobromo compound was synthesized by bromination with CBr_4/PPh_3 reagents. The TBS group was then removed with TBAF (1.0 M in THF) to obtain the alcohol (701 mg, 3.2 mmol, 32%, 2 step) as a colorless oil. **4l** was also prepared by bromination of

the alcohol with CBr4/PPh3 reagents according to the same procedure described above (279 mg, 1.0 mmol, 50%).

^{1}H NMR (400 MHz, $CDCl_3$, δ): 2.19 (d, *J* = 7.7 Hz, 4H), 3.38 (s, 4H), 5.18–5.26 (m, 4H), 5.75 (ddt, *J* = 7.6, 10.0, 17.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 37.0 (*C*H_2), 39.2 (*C*H_2), 41.1 (*C*), 119.9 (*C*H_2), 132.0 (*C*H). HRMS–EI (*m*/*z*): $[M–CH_2Br]^+$ calcd for $C_8H_{12}Br$, 187.01223; found, 187.01154. Anal. Calcd for $C_9H_{14}Br_2$: C, 38.33; H, 5.00. Found: C, 38.06; H, 4.89.

Preparation of 1-(1-bromohex-5-en-2-yl)naphthalene (4n).

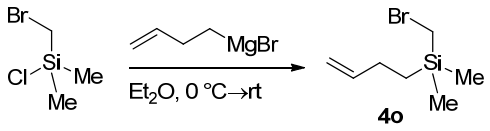


4n

4n was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.92–2.05 (m, 3H), 2.20–2.31 (m, 1H), 3.61–3.72 (m, 2H), 3.92 (brs, 1H), 4.90–4.97 (m, 2H), 5.72–5.83 (m, 1H), 7.39 (d, *J* = 7.3 Hz, 1H), 7.46–7.56 (m, 3H), 7.77 (d, *J* = 8.0 Hz, 1H), 7.86–7.89 (m, 1H), 8.07 (d, *J* = 8.8 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 31.1 (*C*H_2), 32.6 (*C*H_2), 38.5 (*C*H_2), 40.6 (*C*H), 115.1 (*C*H_2), 122.6 (*C*H), 123.4 (*C*H), 125.3 (*C*H), 125.5 (*C*H), 126.1 (*C*H), 127.5 (*C*H), 129.0 (*C*H), 131.9 (*C*), 133.9 (*C*), 137.8 (*C*H), 137.8 (*C*). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{16}H_{17}Br$, 288.05136; found, 288.05068. Anal. Calcd for $C_{16}H_{17}Br$: C, 66.45; H, 5.92. Found: C, 66.26; H, 5.94.

Preparation of (bromomethyl)(but-3-en-1-yl)dimethylsilane (4o).



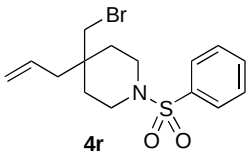
4o

In a vacuum dried 100 mL of a round bottomed flask, (bromomethyl)chlorodimethylsilane (1.364 ml, 10 mmol) was dissolved in dry Et_2O (10 mL) and was cooled to 0 °C under nitrogen atmosphere. A Et_2O solution of homoallyl magnesium bromide (1.3 M, 10 mL, 13 mmol) was then added dropwise for 10 min. After stirred overnight, the reaction mixture was quenched by addition of saturated NH_4Cl aq. and extracted with Et_2O three times. The combined organic layer was then dried over $MgSO_4$. After filtration, the solvents were removed by evaporation. Because the purity of the product was not enough even after the flash column chromatography, the product was then subjected to a recycle gel permeation chromatography to obtain the pure homoallylsilane **4o** (255 mg, 1.2 mmol, 12%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.14 (s, 6H), 0.74–0.80 (m, 2H), 2.07–2.14 (m, 2H), 2.48 (s, 2H), 4.92 (dq, *J* = 1.6, 10.3 Hz, 1H), 5.02 (dq, *J* = 1.8, 17.4 Hz, 1H), 5.87 (ddt, *J* = 6.5, 10.5, 17.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): –4.0 (*C*H_3), 13.2 (*C*H_2), 17.0 (*C*H_2), 27.6 (*C*H_2), 113.2 (*C*H_2), 140.9

(*C*H). HRMS–EI (*m*/*z*): $[M–C_4H_7]^+$ calcd for C_3H_8BrSi, 150.95786; found, 150.95753. Anal. Calcd for $C_7H_{15}BrSi$: C, 40.58; H, 7.30. Found: C, 40.44; H, 7.17.

Preparation of 4-allyl-4-(bromomethyl)-1-(phenylsulfonyl)piperidine (4r).

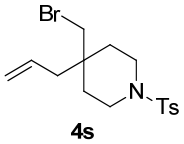


4r

4r was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.60–1.71 (m, 4H), 2.08 (d, *J* = 7.7 Hz, 2H), 2.98–3.09 (m, 4H), 3.22 (s, 2H), 5.07–5.14 (m, 2H), 5.64 (ddt, *J* = 7.6, 10.2, 17.9 Hz, 1H), 7.54–7.58 (m, 2H) 7.61–7.65 (m, 1H) 7.76–7.78 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 32.5 (*C*H_2), 34.7 (*C*), 38.9 (*C*H_2), 41.0 (*C*H_2), 41.6 (*C*H_2), 119.3 (*C*H_2), 127.3 (*C*H), 129.0 (*C*H), 131.7 (*C*H), 132.7 (*C*H), 135.9 (*C*). HRMS–ESI (*m*/*z*): $[M+Na]^+$ calcd for $C_{15}H_{20}BrNO_2SNa$, 380.02903; found, 380.02911.

Preparation of 4-allyl-4-(bromomethyl)-1-tosylpiperidine (4s).

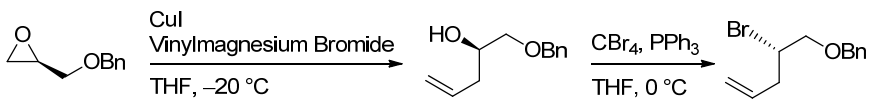


4s

4g was prepared from the corresponding methyl ester according to the procedure described above.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.60–1.71 (m, 4H), 2.08 (d, *J* = 7.8 Hz, 2H), 2.45 (s, 4H), 2.94–3.07 (m, 4H), 3.22 (s, 1H), 5.07–5.14 (m, 2H), 5.65 (ddt, *J* = 7.5, 10.0, 17.3 Hz, 1H), 7.34 (d, *J* =8.2 Hz, 2H), 7.65 (d, *J* = 8.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 21.5 (*C*H_3), 32.6 (*C*H_2), 34.8 (*C*), 39.1 (*C*H_2), 41.0 (*C*H_2), 41.7 (*C*H_2), 119.4 (*C*H_2), 127.5 (*C*H), 129.7 (*C*H), 131.9 (*C*H), 132.9 (*C*), 143.6 (*C*). HRMS–EI (*m*/*z*): $[M+Na]^+$ calcd for $C_{16}H_{22}BrNO_2SNa$, 394.04523; found, 394.04481.

Preparation of optically active (*S*)-{[(2-bromopent-4-en-1-yl)oxy]methyl}benzene [(*S*)-4d].

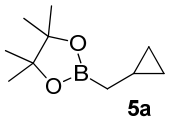


(*S*)-**4d**, >99% ee

(*S*)-**4d** was prepared from the corresponding (*R*)-benzyl glycidyl ether according to the procedure described above. The enantiomeric purity was determined by HPLC analysis on a chiral phase of a *p*-nitorobenzoate derivative of the alcohol obtained by Pd/C catalyzed hydrogenation of (*S*)-**4d** (Daicel CHIRALPAK® OJ-3, 2-PrOH/Hexane = 1/99, 0.5 mL/min, 40 °C). (*R*)-**4d**: tR = 29.29 min, (*S*)-**4d**: tR = 31.28 min. $[\alpha]_D^{25.0}$ –7.70 (deg cm^3 g^{-1} dm^{-1}) (*c* 1.0 in CHCl3).

4. Characterization of Borylation Products.

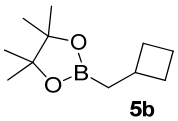
2-(Cyclopropylmethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5a).



5a

^{1}H NMR (400 MHz, $CDCl_3$, δ): –0.01–0.03 (m, 2H), 0.40–0.45 (m, 2H), 0.74–0.78 (m, 3H), 1.23 (s, 12H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 5.7 (*C*H_2), 5.9 (*C*H), 16.0 (br, B–*C*H_2), 24.8 (*C*H_3), 82.9 (*C*). HRMS–EI (*m*/*z*): $[M–CH_3]^+$ calcd for $C_9H_{16}BO_2$, 167.12433; found, 167.12411.

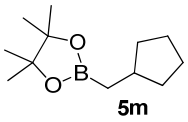
2-(Cyclobutylmethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5b).



5b

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.94 (d, *J* = 7.8 Hz, 2H), 1.23 (s, 12H), 1.54–1.64 (m, 2H), 1.72–1.85 (m, 2H), 2.04–2.11 (m, 2H), 2.47 (sep, *J*= 8.1 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 18.3 (*C*H_2), 20.0 (br, B–CH_2), 24.8 (*C*H_3), 30.8 (*C*H_2), 32.4 (*C*H), 82.8 (*C*). HRMS–EI (*m*/*z*): $[M–CH_3]^+$ calcd for $C_{10}H_{17}BO_2$, 181.14017; found, 181.13968.

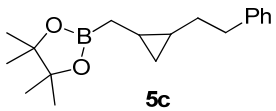
2-(Cyclopentylmethyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5m).



5m

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.84 (d, *J* = 7.3 Hz, 2H), 1.01–1.11 (m, 2H), 1.25 (s, 12H), 1.47–1.65 (m, 4H), 1.74–1.82 (m, 2H), 1.89–2.02 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 24.8 (*C*H_3), 25.1 (*C*H_2), 35.0 (*C*H_2), 36.1 (*C*H), 82.8 (*C*). The carbon directly attached to the boron atom was not detected, likely due to quadropolar relaxation. HRMS–EI (*m*/*z*): $[M–CH_3]^+$ calcd for $C_{10}H_{20}BO_2$, 195.15563; found, 196.15926. Anal. Calcd for $C_{12}H_{23}BO_2$: C, 68.59; H, 11.03. Found: C, 68.63; H, 11.12.

4,4,5,5-Tetramethyl-2-[(2-phenethylcyclopropyl)methyl)]-1,3,2-dioxaborolane (5c).

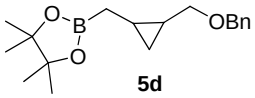


5c

The product (**5c)** was obtained as a diastereomeric mixture (1:1). The stereoselectivity was determined by ^{1}H NMR analysis of the alcohol derived from H_2O_2/NaOH oxidation of **5c.**

^{1}H NMR (400 MHz, $CDCl_3$, δ): –0.29 (q, *J* = 4.9 Hz, 0.5H), 0.17–0.27 (m, 1H), 0.42–0.57 (m, 1H), 0.61–0.90 (m, 3.5H), 1.26 (s, 12H), 1.40–1.68 (m, 2H), 2.68–2.73 (m, 2H), 7.14–7.29 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 9.8 (br, B–*C*H_2), 10.7 (*C*H), 11.9 (*C*H_2), 13.1 (*C*H_2), 13.8 (*C*H), 15.4 (*C*H), 15.7 (br, B–*C*H_2), 19.6 (*C*H), 24.7 (*C*H_3), 30.8 (*C*H_2), 35.9 (*C*H_2), 36.2 (*C*H), 36.5 (*C*H_2), 82.8 (*C*), 82.9 (*C*), 125.39 (*C*H), 125.42 (*C*H), 128.1 (*C*H), 128.33 (*C*H), 128.35 (*C*H), 142.71 (*C*), 142.73 (*C*). HRMS–EI (*m*/z): $[M]^+$ calcd for $C_{18}H_{27}BO_2$, 286.21041; found, 286.21040.

2-({2-[(Benzyloxy)methyl]cyclopropyl}methyl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5d).

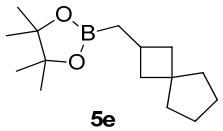


5d

The product (**5d)** was obtained as a diastereomeric mixture (1:1). The stereoselectivity was determined by GC analysis of the crude reaction mixture.

^{1}H NMR (400 MHz, $CDCl_3$, δ): –0.1 (q, *J* = 5.3 Hz, 0.5H), 0.31–0.42 (m, 1H), 0.66–1.13 (m, 4.5H), 1.24 (s, 12H), 3.23–3.52 (m, 2H), 4.49–4.56 (m, 2H), 7.24–7.37 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 10.7 (*C*H), 10.9 (*C*H), 11.5 (*C*H_2), 12.3 (*C*H), 15.2 (*C*H), 19.3 (*C*H), 24.69 (*C*H_3), 24.73 (CH_3), 70.5 (*C*H_2), 72.2 (*C*H_2), 72.6 (*C*H_2), 74.3 (*C*H_2), 83.0 (*C*), 127.3 (*C*H), 127.4 (*C*H), 127.6 (*C*H), 127.7 (*C*H), 128.2 (*C*H), 138.6 (*C*), 138.7 (*C*). HRMS–EI (*m*/*z*): $[M+Na]^+$ calcd for $C_{18}H_{27}BO_3Na$, 325.19510; found, 325.19433. Anal. Calcd for $C_{18}H_{27}BO_2$: C, 71.54; H, 9.00. Found: C, 71.56; H, 9.26.

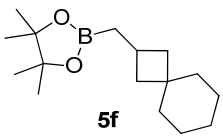
4,4,5,5-Tetramethyl-2-(spiro[3.4]octan-2-ylmethyl)-1,3,2-dioxaborolane (5e).



5e

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.93 (d, *J* = 7.3 Hz, 2H), 1.23 (s, 12H), 1.46–1.61 (m, 10H), 1.99 (dt, *J* = 2.8, 8.8 Hz, 2H), 2.35 (sep, *J*= 8.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 20.0 (br, B–*C*H_2), 23.6 (*C*H_2), 23.9 (*C*H_2), 24.7 (*C*H_3), 25.9 (*C*H), 39.1 (*C*H_2), 40.5 (*C*H_2), 42.2 (*C*H_2), 42.4 (*C*), 82.7 (*C*). HRMS–EI (*m*/z): $[M–CH_3]^+$ calcd for $C_{15}H_{24}BO_2$, 235.18720; found, 235.18696. Anal. Calcd for $C_{15}H_{27}BO_2$: C, 72.01; H, 10.88. Found: C, 71.78; H, 11.00.

4,4,5,5-Tetramethyl-2-(spiro[3.5]nonan-2-ylmethyl)-1,3,2-dioxaborolane (5f).

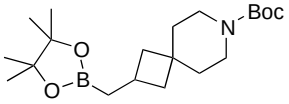


5f

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.93 (d, *J* = 8.1 Hz, 2H), 1.23 (s, 12H), 1.27–1.45 (m, 12H), 1.93

(dt, *J* = 2.6, 8.8 Hz, 2H), 2.34 (sep, *J* = 8.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 20.4 (br, B–*C*H_2), 22.8 (*C*H_2), 23.0 (*C*H_2), 24.7 (*C*H_3), 25.0 (*C*H), 26.1 (*C*H_2), 35.4 (*C*), 37.1 (*C*H_2), 41.0 (*C*H_2), 41.2 (*C*H_2), 82.7 (*C*). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{16}H_{29}BO_2$, 264.22606; found, 264.22586. Anal. Calcd for $C_{16}H_{29}BO_2$: C, 72.73; H, 11.06. Found: C, 72.46; H, 11.12.

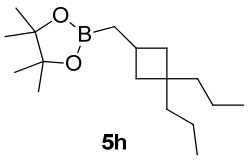
7-(Phenylsulfonyl)-2-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)-7 azaspiro[3.5]nonane (5g).



5g

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.96 (d, *J* = 8.0 Hz, 2H), 1.23 (s, 12H), 1.34–1.41 (m, 4H), 1.44 (s, 9H), 1.54 (t, *J* = 5.1 Hz, 2H), 1.98–2.03 (m, 2H), 2.41 (sep, *J* = 8.4 Hz, 1H), 2.87 (t, *J* = 5.7 Hz, 2H), 2.96 (t, *J* = 5.7 Hz, 2H), 7.50–7.54 (m, 2H) 7.57–7.61 (m, 1H) 7.74–7.76 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 20.0 (br, B–CH_2), 24.8 (*C*H_3), 25.0 (*C*H), 28/4 (*C*H_3), 33.7 (*C*), 36.1 (*C*H_2), 39.7 (*C*H_2), 40.2 (*C*H_2), 79.0 (*C*), 82.9 (*C*) 155.0 (*C*). HRMS–ESI (*m*/*z*): $[M+Na]^+$ calcd for $C_{20}H_{36}BNO_4Na$, 388.26296; found, 388.26309.

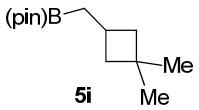
2-[(3,3-Dipropylcyclobutyl)methyl]-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5h).



5h

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.83–0.93 (m, 8H), 1.06–1.41 (m, 10H), 1.23 (s, 12H), 1.86–1.92 (m, 2H), 2.34 (sep, *J* = 8.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 14.8 (*C*H_3), 14.9 (*C*H_3), 17.0 (*C*H_2), 17.3 (*C*H_2), 20.3 (br, B–*C*H_2), 24.7 (*C*H_3), 25.0 (*C*H), 37.2 (*C*), 39.9 (*C*H_2), 40.8 (*C*H_2), 43.2 (*C*H_2), 82.7 (*C*). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{17}H_{33}BO_2$, 280.25736; found, 264.25722. Anal. Calcd for $C_{17}H_{33}BO_2$: C, 72.86; H, 11.87. Found: C, 72.82; H, 11.98.

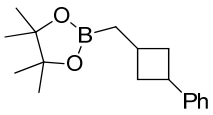
2-[(3,3-Dimethylcyclobutyl)methyl]-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (5i).



5i

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.92 (d, *J* = 8.0 Hz, 2H), 1.02 (s, 3H), 1.10 (s, 3H), 1.23 (s, 12H), 1.40 (dt, *J* = 2.6, 9.3 Hz, 2H), 1.90 (dt, *J* = 2.7, 9.0 Hz, 2H), 2.37 (sep, *J* = 8.5 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 20.0 (br, B–*C*H_2), 24.5 (*C*H), 24.8 (*C*H_3), 28.5 (*C*H_3), 31.3 (*C*H_3), 43.4 (*C*H_2), 82.7 (*C*). HRMS–EI (*m*/z): $[M–CH_3]^+$ calcd for $C_{12}H_{22}BO_2$, 209.17151; found, 209.17127.

4,4,5,5-Tetramethyl-2-((3-phenylcyclobutyl)methyl)-1,3,2-dioxaborolane (5j).

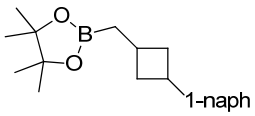


5j

The product (**5j**) was obtained as a diastereomeric mixture (1.3:1). The stereoselectivity was determined by ^{1}H NMR analysis of the crude reaction mixture.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.98 (d, *J* = 7.7 Hz, 2H), 1.16 (d, *J* = 8.1 Hz, 2H), 1.24 (s, 12H), 1.25 (s, 24H), 1.69–1.78 (m, 2H), 2.04–2.10 (m, 2H), 2.31–2.59 (m, 6H), 3.24–3.34 (m, 1H), 3.62 (quin, *J* = 8.3 Hz, 1H), 7.13–7.32 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 19.0 (br, B–*C*H_2), 24.5 (*C*H_3), 24.7 (*C*H_3), 27.4 (*C*H), 28.0 (*C*H), 35.9 (*C*H), 36.35 (*C*H), 36.41 (*C*H_2), 82.77 (*C*), 82.80 (*C*), 125.36 (*C*H), 125.44 (*C*H), 126.3 (*C*H), 128.0 (*C*H), 128.1 (*C*H), 146.0 (*C*), 146.6 (*C*). HRMS–EI (*m*/*z*): $[M–CH_3]^+$ calcd for $C_{16}H_{22}BO_2$, 257.17128; found, 257.17105.

4,4,5,5-Tetramethyl-2-{[3-(naphthalen-1-yl)cyclobutyl]methyl}-1,3,2-dioxaborolane (5k).

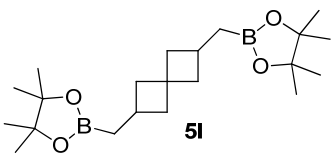


5k

The product (**5k)** was obtained as a diastereomeric mixture (1.4:1). The stereoselectivity was determined by GC analysis of the crude reaction mixture.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.99 (d, *J* = 7.7 Hz, 2H), 1.22–1.27 (m, 2H), 1.23 (s, 12H), 1.25 (s, 12H), 1.84–1.92 (m, 2H), 2.22–2.28 (m, 2H), 2.46–2.65 (m, 5H), 2.71–2.78 (m, 1H), 3.84–3.93 (m, 1H), 4.23 (quint, *J* = 7.9 Hz, 1H), 7.33 (d, *J* = 7.4 Hz, 1H), 7.40–7.51 (m, 8H), 7.67–7.71 (m, 2H), 7.81–7.91 (m, 3H), 7.97–8.00 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 19.0 (br, B–*C*H_2), 24.7 (*C*H_3), 27.7 (*C*H), 28.5 (*C*H), 33.3 (*C*H), 34.1 (*C*H), 35.4 (*C*H_2), 37.7 (*C*H_2), 82.78 (*C*), 82.82 (*C*), 121.9 (*C*H), 122.5 (*C*H), 124.15 (*C*H), 124.25 (*C*H), 125.2 (*C*H), 125.3 (*C*H), 125.4 (*C*H), 126.1 (*C*H), 126.2 (*C*H), 128.45 (*C*H), 128.54 (*C*H), 131.50 (*C*), 131.54 (*C*), 133.6 (*C*), 133.7 (*C*), 141.5 (*C*), 141.6 (*C*). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{21}H_{27}BO_2$, 322.21041; found, 322.21022.

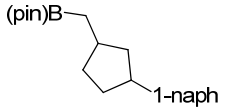
2,6-Bis[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]spiro[3.3]heptane (5l).



5l

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.88 (d, *J* = 7.7 Hz, 4H), 1.22 (s, 24H), 1.47–1.60 (m, 4H), 1.97–2.03 (m, 2H), 2.16–2.33 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 19.5 (br, B–*C*H_2), 24.7 (*C*H_3), 26.4 (*C*H), 35.8 (*C*), 43.3 (*C*H_2), 43.9 (*C*H_2), 82.7 (*C*). HRMS–EI (*m*/*z*): $[M+Na]^+$ calcd for $C_{12}H_{22}BO_2Na$, 399.28539; found, 399.28515.

4,4,5,5-Tetramethyl-2-{[3-(naphthalen-1-yl)cyclopentyl]methyl}-1,3,2-dioxaborolane (5n).

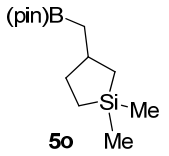


5n

The product (**5n)** was obtained as a diastereomeric mixture (1.1:1). The stereoselectivity was determined by ^{1}H NMR analysis of the crude reaction mixture.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.99–1.02 (m, 4H), 1.18–1.22 (m, 1H), 1.25 (s, 24H), 1.33–1.54 (m, 3H), 1.75–2.14 (m, 6H), 2.19–2.46 (m, 4H), 3.79–3.88 (m, 1H), 3.95 (quin, *J* = 8.2 Hz, 1H), 7.39–7.52 (m, 8H), 7.67–7.69 (m, 2H), 7.83–7.85 (m, 2H), 8.14 (t, *J* = 8.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 18.5 (br, B–*C*H_2), 24.7 (*C*H_3), 33.1 (*C*H_2), 33.9 (*C*H), 34.1 (*C*H_2), 35.0 (*C*H), 35.4 (*C*H_2), 36.1 (*C*H), 39.7 (*C*H), 41.1 (*C*H), 42.0 (*C*H_2), 43.0 (*C*H_2), 82.8 (*C*), 121.9 (*C*H), 123.8 (*C*H), 124.0 (*C*H), 125.1 (*C*H), 125.36 (*C*H), 125.48 (*C*H), 125.52 (*C*H), 126.0 (*C*H), 126.1 (*C*H), 128.6 (*C*H), 132.0 (*C*), 132.1 (*C*), 133.8 (*C*), 142.2 (*C*), 142.7 (*C*). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{22}H_{29}BO_2$, 336.22606; found, 336.22559.

1,1-Dimethyl-3-[(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]silolane (5o).

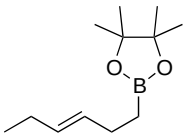


5o

This compound was obtained as a mixture with *endo*-cylization product (exo/endo 91:9).

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.08 (s, 6H), 0.42–0.53 (m, 1H), 0.72 (ddd, *J* = 2.5, 7.1, 14.8 Hz, 1H), 0.81–0.93 (m, 3H), 0.99–1.10 (m, 1H), 1.25 (s, 12H), 1.76–1.95 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): –1.23 (*C*H_3), –1.12 (*C*H_3), 13.2 (*C*H_2), 20.5 (br, B–*C*H_2), 23.0 (*C*H_2), 24.76 (*C*H_3), 24.79 (*C*H_3), 36.2 (*C*H_2), 37.1 (*C*H), 82.7 (*C*). HRMS–EI (*m*/*z*): $[M–CH_3]^+$ calcd for $C_{12}H_{24}BO_2Si$, 239.16386; found, 238.16649.

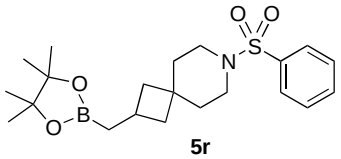
(*E*)-2-(Hex-3-en-1-yl)-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (6q)



6q

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.86 (t, *J* = 7.9 Hz, 2H), 0.95 (t, *J* = 7.7 Hz, 3H), 1.24 (s, 12H), 1.95–2.03 (m, 2H), 2.05–2.17 (m, 2H), 5.44 (dt, *J* = 2.56, 5.12 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 11.0 (br, B–*C*H_2), 14.0 (*C*H_3), 24.9 (*C*H_3), 25.6 (*C*H_2), 26.9 (*C*H_2), 83.0 (*C*), 130.9 (*C*H). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{12}H_{23}BO_2$, 210.17934; found, 210.17924.

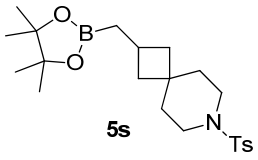
7-(Phenylsulfonyl)-2-((4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)methyl)-7 azaspiro[3.5] nonane (5r).



5r

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.89 (d, *J* = 7.7 Hz, 2H), 1.20 (s, 12H), 1.25–1.30 (m, 2H), 1.55–1.58 (m, 2H), 1.68 (t, *J* = 5.7 Hz, 2H), 1.85–1.90 (m, 2H), 2.34 (sep, *J* = 8.5 Hz, 1H), 2.87 (t, *J* = 2.9, 5.7 Hz, 2H), 2.96 (t, *J* = 3.0, 5.7 Hz, 2H), 7.50–7.54 (m, 2H), 7.57–7.61 (m, 1H), 7.74–7.76 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 21.0 (br, B–CH_2), 24.7 (*C*H_3), 24.8 (*C*H), 32.8 (*C*), 35.4 (*C*H_2), 38.8 (*C*H_2), 39.9 (*C*H_2), 43.0 (*C*H_2), 43.2 (*C*H_2), 82.8 (*C*), 127.5 (*C*H), 128.8 (*C*H), 132.5 (*C*H), 136.3 (*C*). HRMS–ESI (*m*/*z*): $[M+H]^+$ calcd for $C_{21}H_{33}BNO_4S$, 406.22179; found, 406.22174.

2-[(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)methyl]-7-tosyl-7-azaspiro[3.5]nonane (5s).

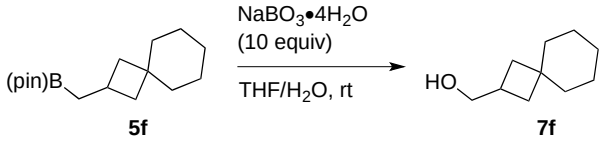


5s

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.89 (d, *J* = 7.7 Hz, 2H), 1.20 (s, 12H), 1.25–1.30 (m, 2H), 1.54–1.58 (m, 2H), 1.67 (t, *J* = 5.7 Hz, 2H), 1.85–1.90 (m, 2H), 2.33 (sep, *J* = 8.4 Hz, 1H), 2.43 (s, 3H), 2.86 (t, *J* = 5.5 Hz, 2H), 2.94 (t, *J* = 5.5 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 7.63 (d, *J* = 8.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 19.9 (br, B–*C*H_2), 21.3 (*C*H), 24.6 (*C*H_3), 24.7 (*C*H_3), 32.7 (*C*), 35.3 (*C*H_2), 38.8 (*C*H_2), 39.8 (*C*H_2), 42.9 (*C*H_2), 43.1 (*C*H_2), 82.6 (*C*), 127.4 (*C*H), 129.4 (*C*H), 133.2 (*C*), 143.0 (*C*). HRMS–EI (*m*/*z*): $[M+Na]^+$ calcd for $C_{22}H_{34}BNO_4SNa$, 442.21993; found, 442.21989.

5. Derivatization of Borylation Products.

Experimental Procedure for the $NaBO_3$ Oxidation of 5f.

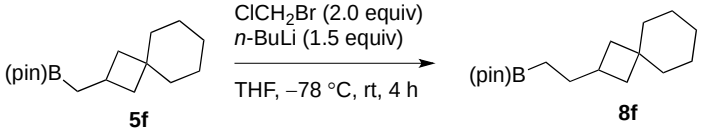


The oxidation was performed according to the literature procedure.[2]

In a reaction vial, $NaBO_3 \cdot 4H_2O$ (384.7 mg, 2.5 mmol) was dissolved in THF/H_2O (3:2, 5 mL). **5f** (66.1 mg, 0.25 mmol) was then added at room temperature. After stirred for 1.5 h, the reaction mixture was extracted three times with EtOAc, dried over $MgSO_4$, and filtered. The crude material was purified by flash column chromatography to obtain the corresponding alcohol **7f** (33.9 mg, 0.22 mmol, 88%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.19–1.58 (m, 12H), 1.81–1.86 (m, 2H), 2.40 (sep, J = 8.1 Hz, 1H), 3.58 (d, J = 7.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 22.6 (*C*H_2), 22.8 (*C*H_2), 26.0 (*C*H_2), 30.5 (*C*H), 35.1 (*C*H_2), 35.7 (*C*), 37.7 (*C*H_2), 40.5 (*C*H_2), 68.4 (*C*H_2). HRMS–EI (*m*/*z*): $[M–OH_2]^+$ calcd for $C_{10}H_{16}$, 136.12520; found, 136.12493.

Experimental Procedure for the One Carbon-Homologation of 5f.

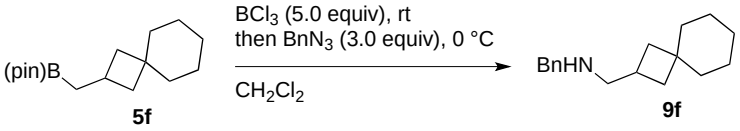


The homologation was performed according to the literature procedure.[3]

In an oven-dried reaction vial, **5f** (66.2 mg, 0.25 mmol) and bromochloromethane (64.7 mg, 0.5 mmol) were dissolved in dry THF (2 mL). After the mixture was cooled to –78 °C, *n*-BuLi in hexane (1.64 M, 0.23 mL, 0.38 mmol) was added dropwise. The mixture was stirred at –78 °C for 10 min, and then stirred at room temperature for 4 h. The reaction was quenched with NH_4Cl aq., extracted three times with Et_2O, dried over $MgSO_4$, and filtered. The crude material was then purified by flash column chromatography to obtain the corresponding homologation product **8f** (62.7 mg, 0.225 mmol, 90%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 0.66 (t, J = 8.2 Hz, 2H), 1.22–1.49 (m, 14H), 1.24 (s, 12H), 1.80–1.85 (m, 2H), 2.06 (sep, J = 8.2 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 9.0 (br, B–*C*H_2), 22.8 (*C*H_2), 23.0 (*C*H_2), 24.8 (*C*H_3), 26.1 (*C*H_2), 31.0 (*C*H_2), 32.1 (*C*H_2), 35.2 (*C*), 37.6 (*C*H_2), 38.5 (*C*H_2), 40.9 (*C*H_2), 82.8 (*C*). HRMS–EI (*m*/*z*): $[M]^+$ calcd for $C_{17}H_{31}BO_2$, 278.24202; found, 278.24193.

Experimental Procedure for the Amination of 5f.

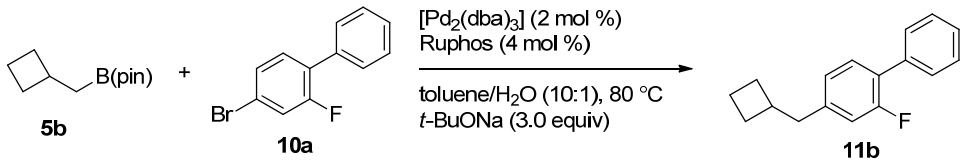


The amination was performed according to the literature procedure.[4]

In an oven-dried reaction vial, CH_2Cl_2 solution of BCl_3 (1.0 M, 1.25 mL, 1.25 mmol) was added under nitrogen atmosphere. **5f** (66.1 mg, 0.25 mmol) was added to the reaction vial with stirring at room temperature. After 4 h, the volatile materials were removed under reduced pressure, and dry CH_2Cl_2 (1.5 mL) was added to the resultant product. The reaction vial was cooled to 0 °C, and benzyl azide (100 mg, 0.75 mmol) was added to the mixture. After stirred for 16 h at 0 °C, the reaction mixture was quenched by adding aqueous NaOH (2.0 M), extracted three times with EtOAc, dried over Na_2SO_4, and filtered. The crude material was then purified by flash column chromatography to obtain the corresponding amine **9f** (36.5 mg, 0.15 mmol, 60%) as a yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.22–1.47 (m, 12H), 1.83–1.89 (m, 2H), 2.37 (sep, J = 8.2 Hz, 1H), 2.64 (d, J = 7.3 Hz, 2H), 3.77 (s, 2H), 7.22–7.35 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 22.7 (*C*H_2), 22.8 (*C*H_2), 26.0 (*C*H_2), 28.6 (*C*H), 36.0 (*C*), 37.1 (*C*H_2), 37.5 (*C*H_2), 40.7 (*C*H_2), 54.0 (*C*H_2), 56.5 (*C*H_2), 126.9 (*C*H), 128.1 (*C*H), 128.3 (*C*H), 140.3 (*C*). HRMS–ESI (*m*/*z*): $[M+H]^+$ calcd for $C_{17}H_{26}N$, 244.20598; found, 244.20593.

Procedure for the Suzuki-Miyaura Cross-Coupling Reaction of 5b with 4-Bromo-2-fluoro-1,1'-biphenyl (10a):

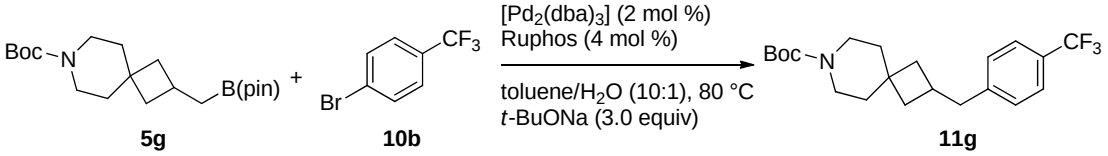


Suzuki-Miyaura cross-coupling reaction was performed according to the literature procedure.[5]

$Pd_2(dba)_3$ (4.6 mg, 0.005 mmol), Na(O-*t*-Bu) (72 mg, 0.75 mmol), Ruphos (4.7 mg, 0.01 mmol), and 4-bromo-2-fluoro-1,1'-biphenyl (62.7 mg, 0.25 mmol) were placed in an oven-dried reaction vial. After the vial was sealed with a screw cap containing a teflon-coated rubber septum, the vial was connected to a vacuum/nitrogen manifold through a needle. It was evacuated and then backfilled with nitrogen. This cycle was repeated three times. Toluene (0.5 mL) and H_2O (0.05 mL) and **5b** (58.8 mg, 0.30 mmol) were added in the vial through the rubber septum. The resulting mixture was stirred at 80 °C for 48 h. The reaction mixture was passed through a short silica gel column eluting with Et_2O. The crude mixture was further purified by flash column chromatography to give the corresponding coupling product **11b** (43.1 mg, 73%) as a colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.70–1.79 (m, 2H), 1.82–1.94 (m, 2H), 2.04–2.12 (m, 2H), 2.59 (sep, *J* = 7.9 Hz, 1H), 2.72 (d, *J* = 7.8 Hz, 2H), 6.92–6.99 (m, 2H), 7.24–7.36 (m, 2H), 7.41–7.44 (m, 2H), 7.53–7.55 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 18.3 (*C*H_2), 28.2 (*C*H_2), 36.9 (*C*H), 42.4 (*C*H_2), 115.9 (d, *J* = 22.0 Hz, *C*H), 124.5 (d, *J* = 2.8 Hz, *C*H), 126.2 (d, *J* = 13.4 Hz, C), 127.3 (*C*H), 128.4 (*C*H), 128.9 (d, *J* = 2.9 Hz, *C*H), 130.3 (d, *J* = 3.8 Hz, *C*H), 135.9 (*C*), 143.0 (d, *J* = 6.7 Hz, *C*), 159.6 (d, *J* = 248.1 Hz, *C*). ^{19}F NMR ($CDCl_3$, 372.5 MHz): –118.9. HRMS–ESI (*m*/*z*): $[M]^+$ calcd for $C_{17}H_{17}F_1$, 240.13143; found, 240.13090.

Procedure for the Suzuki-Miyaura Cross-Coupling Reaction of 5g between 1-Bromo-4-(trifluoromethyl)benzene (10b):



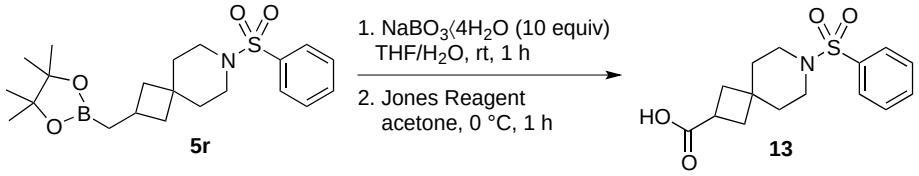
5g **10b** **11g**

Suzuki-Miyaura cross-coupling reaction was performed according to the literature procedure with slight modification.[5]

$Pd_2(dba)_3$ (4.6 mg, 0.005 mmol), Na(O-*t*-Bu) (72 mg, 0.75 mmol), Ruphos (4.7 mg, 0,01 mmol), and **5g** (91.3 mg, 0.25 mmol) were placed in an oven-dried reaction vial. After the vial was sealed with a screw cap containing a teflon-coated rubber septum, the vial was connected to a vacuum/nitrogen manifold through a needle. It was evacuated and then backfilled with nitrogen. This cycle was repeated three times. Toluene (0.5 mL) and H_2O (0.05 mL) and bromo-4-(trifluoromethyl)benzene (**10b**) (112.5 mg, 0.5 mmol) were added in the vial through the rubber septum. The resulting mixture was stirred at 80 °C for 48 h. The reaction mixture was passed through a short silica gel column eluting with Et_2O. The crude mixture was further purified by flash column chromatography to give the corresponding coupling product **11g** (62.3 mg, 65%) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.44–1.60 (m, 15H), 1.93–1.98 (m, 2H), 2.52 (sep, *J* = 8.2 Hz, 1H), 2.77 (d, *J* = 7.7 Hz, 2H), 3.27 (t, *J* = 5.3 Hz, 2H) 3.32 (t, *J* = 5.3 Hz, 2H), 7.22–7.27 (m, 2H), 7.52 (d, J = 7.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 28.4 (*C*H_3), 30.0 (*C*H), 34.0 (*C*), 36.1 (*C*H_2), 37.8 (*C*H_2), 39.6 (*C*H_2), 40.5 (br, *C*H_2), 43.4 (*C*H_2), 79.2 (*C*), 125.1 (q, *J* = 3.5 Hz, *C*H), 128.7 (*C*H), 145.0 (*C*), 154.9 (*C*). ^{19}F NMR ($CDCl_3$, 372.5 MHz): –62.2. HRMS–ESI (*m*/*z*): $[M+Na]^+$ calcd for $C_{21}H_{28}O_2NF_3Na$, 406.19643; found, 406.19645.

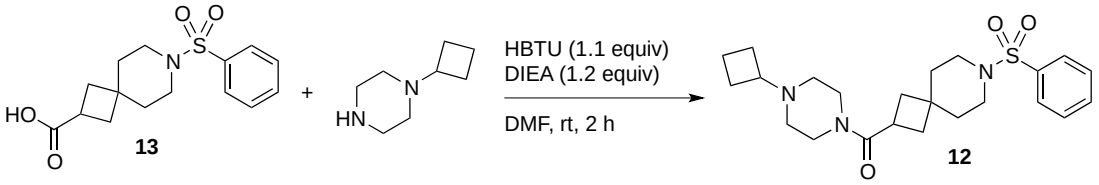
Preparation of 7-(phenylsulfonyl)-7-azaspiro[3.5]nonane-2-carboxylic acid (13).



In a reaction vial, $NaBO_3 \cdot 4H_2O$ (769.3 mg, 5 mmol) and **5r** (202.4 mg, 0.5 mmol) was dissolved in THF/H_2O (3:2, 5 mL) at room temperature. After stirred for 1 h, the reaction mixture was extracted three times with Et_2O, dried over $MgSO_4$, and filtered. The crude material was purified by flash column chromatography to give the corresponding alcohol (128.6 mg) as a white solid. The obtained alcohol was subjected to the oxidation reaction using Jones reagent (ca. 2.5 M, 0.25 mL, 0.625 mmol) in acetone at 0 °C. After 1 h, the reaction was quenched by saturated aqueous NH_4Cl, extracted three times with Et_2O. The combined organic layer was washed with aqueous NaOH (0.1 M) and then aqueous layer was acidified with saturated aqueous NH_4Cl, extracted six times with Et_2O. The combined organic layer was dried over $MgSO_4$ followed by evaporation to obtain the corresponding carboxylic acid **13** (100.1 mg, 64%, 2 steps) as a white solid.

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.67 (t, *J* = 5.9 Hz, 2H), 1.71 (t, *J* = 5.7 Hz, 2H), 1.97 (d, *J* = 8.8 Hz, 4H), 2.91 (t, *J* = 5.7 Hz, 2H), 2.97 (t, *J* = 5.7 Hz, 2H), 3.32 (quint, *J* = 8.9 Hz, 1H), 7.51–7.55 (m, 2H), 7.58–7.62 (m, 1H) 7.74–7.77 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$, δ): 31.4 (*C*H), 33.5 (*C*), 34.1 (CH_2), 35.6 (CH_2), 37.2 (CH_2), 42.6 (CH_2), 42.9 (CH_2), 127.4 (*C*H), 128.9 (*C*H), 132.7 (*C*H), 136.0 (*C*), 181.8 (*C*). HRMS–ESI (*m*/*z*): $[M–H]^+$ calcd for $C_{15}H_{18}NO_4S$, 308.09620; found, 308.09676.

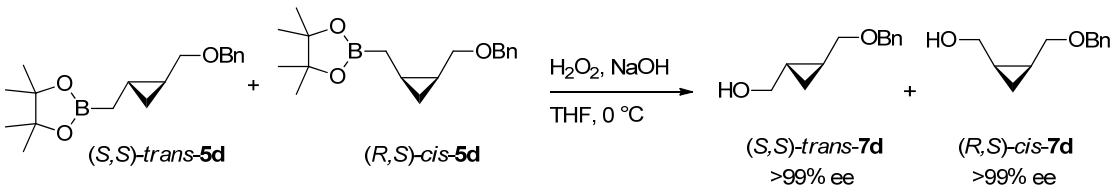
Preparation of (4-cyclobutylpiperazin-1-yl)(7-(phenylsulfonyl)-7-azaspiro[3.5]nonan-2-yl)methanone (12).



The condensation was performed according to the literature procedure.[6] In a vacuum dried 20 mL of a round bottomed flask, carboxylic acid (61.8 mg, 0.2 mmol) and *N*,*N*-diisopropylethylamine (DIEA) (41 μL) were dissolved in DMF (3 mL) under nitrogen atmosphere. A solution of *O*-benzotriazole-*N*,*N*,*N*',*N*'-tetramethyluronium hexafluorophosphate (HBTU) (83.4 mg, 0.22 mmol) and 1-cyclobutylpiperazine (28.1 mg, 0.2 mmol) in DMF (3 mL) was then added dropwise. After stirred for 2 h at room temperature, the solvent was removed under reduced pressure. The crude product was then purified by flash column chromatography to obtain the corresponding condensation product **12** (78.4 mg, 91%) as a white solid. ^{1}H NMR was in agreement with those in the literature.[6]

^{1}H NMR (400 MHz, $CDCl_3$, δ): 1.62–1.76 (m, 6H), 1.80–1.90 (m, 4H), 1.99–2.04 (m, 4H), 2.24 (dt, *J* = 5.7, 11.7 Hz, 4H), 2.68 (quint, *J* = 8.0 Hz, 1 H), 2.91 (t, *J* = 5.3 Hz, 2 H), 2.98 (t, *J* = 5.1 Hz, 2 H), 3.10 (quint, *J* = 8.8 Hz, 1 H), 3.29 (t, *J* = 4.9 Hz, 2H), 3.59 (t, *J* = 4.8 Hz, 2H), 7.52–7.56 (m, 2H), 7.59–7.63 (m, 1H), 7.74–7.76 (m, 2H).

6. Determination of Absolute Configuration of *trans*-5d and *cis*-5d.



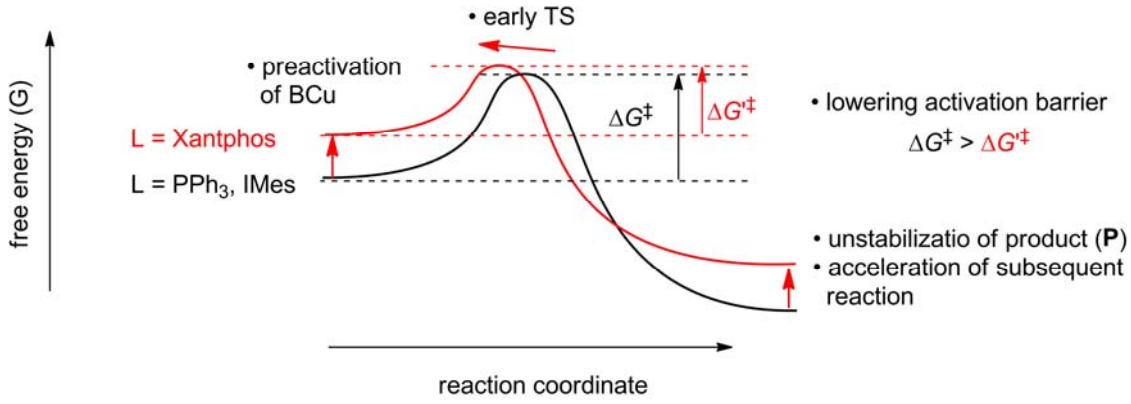
The absolute configuration of *trans*-**5d** and *cis*-**5d** were determined by comparing the optical rotation of the cyclopropylmethyl alcohol obtained by H_2O_2 oxidation.[7] The ee values were determined by HPLC analysis (Daicel CHIRALPAK® OJ-3, 2-PrOH/Hexane = 5/95, 0.5 mL/min, 40 °C). (*S*,*S*)-**7d**: tR = 36.71 min., (*R*,*R*)-**7d**: tR = 40.92 min, (*R*,*S*)-**7d**: t_R = 23.17 min., (*S*,*R*)-**7d**: t_R = 25.43 min. (*S*,*S*)-**7d**: $[\alpha]_D^{25.1}$ +10.56 (deg cm^3 g^{-1} dm^{-1}) (*c* 0.9 in CHCl3), (*R*,*S*)-**7d**: $[\alpha]_D^{23.9}$ –38.75 (deg cm^3 g^{-1} dm^{-1}) (*c* 0.8 in $CHCl_3$).

7. Details of DFT calculations

All calculations were performed with the Gaussian 09W (revision C.01) program package.[8] Geometry optimizations were performed with B3PW91/cc-pVDZ in the gas-phase. Xantphos (4,5-bis(diphenylphosphino)-9,9-dimethylxanthene) ligand was modeled by 4,5-bis(diphenylphosphino)-9,9-xanthene. Molecular orbitals were drawn by the GaussView 5.0 program. Frequency calculations were conducted on gas-phase optimized geometries to check the all the stationary points as either minima or transition states. Fragment distortion and interaction energies were calculated with B3PW91/cc-pVDZ in the gas-phase.

To understand the ligand effect observed in the borylative *exo*-cyclization reaction, we conducted distortion/interaction analysis and structure change analysis of stationary points. Comparison of the calculation results of ligands indicate that the low activation barrier for the reaction with Xantphos is due to the preactivation effect on the starting borylcopper(I)/Xantphos complex as summarized in Figure S1.

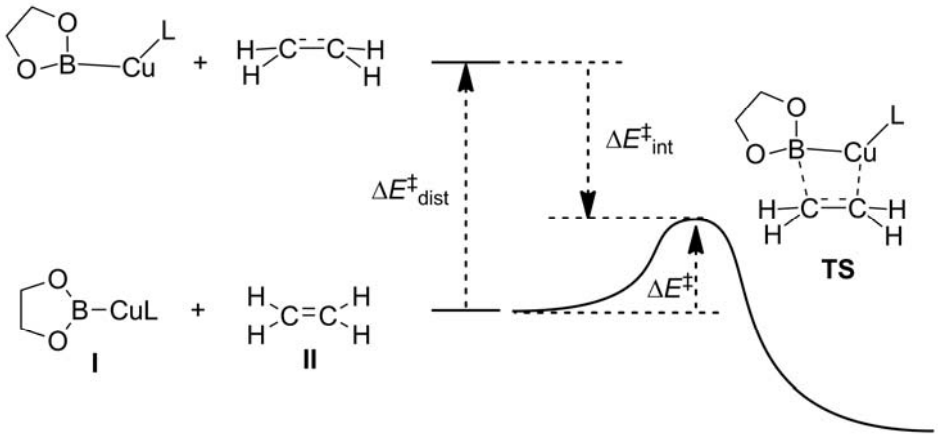
Figure S1. Summary of Ligand Effect of Xantphos on the Energy Profile in Addition of Borylcopper(I) to Alkene



7.1. Distortion/Interaction Analysis

The distortion/interaction analysis for the transition states in addition reaction of borylcopper(I) to ethylene indicated the preactivation nature of the Xantphos ligated borylcopper(I) complex. The energies to deform the isolated reactants to the transition geometry ($\Delta E^{\ddagger}_{dist}$) and the energy of interaction between these deformed reactants are summarized in Table S1. The π-complex (**III**) was omitted because ΔG_{III} values (complexation) were positive. The deformation energy for borylcopper(I) complex [$\Delta E^{\ddagger}_{dist}$ (Cu–B)] with Xantphos ligand (11.7 kcal/mol) is significantly smaller than those of PPh_3 and IMes (16.2 and 18.6 kcal/mol, respectively). The deformation energy for ethylene [$\Delta E^{\ddagger}_{dist}$ ($H_2C{=}CH_2$)] shows smaller difference. The interaction energy ($\Delta E^{\ddagger}_{int}$) of Xantphos complex (–38.8 kcal/mol) is largely smaller than those of PPh_3 and IMes (–44.8 and –49.1 kcal/mol, respectively). This analysis indicates that a major factor for the low activation barrier of Xantphos complex at the addition transition state is attributable to the preactivation nature of borylcopper(I)/Xantphos complex and the resultant early transition state. In other words, Xantphos ligand make the borylcopper(I) complex more close to the TS.

Table S1. Distortion/Interaction Analysis for Addition TS



	energy difference (kcal/mol)			
ligand (L)	$\Delta E^{\ddagger}$	$\Delta E^{\ddagger}_{dist}$ (Cu–B)	$\Delta E^{\ddagger}_{dist}$ ($H_2C{=}CH_2$)	$\Delta E^{\ddagger}_{int}$
Xantphos	2.1	**11.7**	**29.2**	**–38.8**
PPh_3	3.6	16.2	32.1	–44.8
IMes	3.0	18.6	33.5	–49.1

7.2. Structure Analysis of Stationary Points for Various Ligands

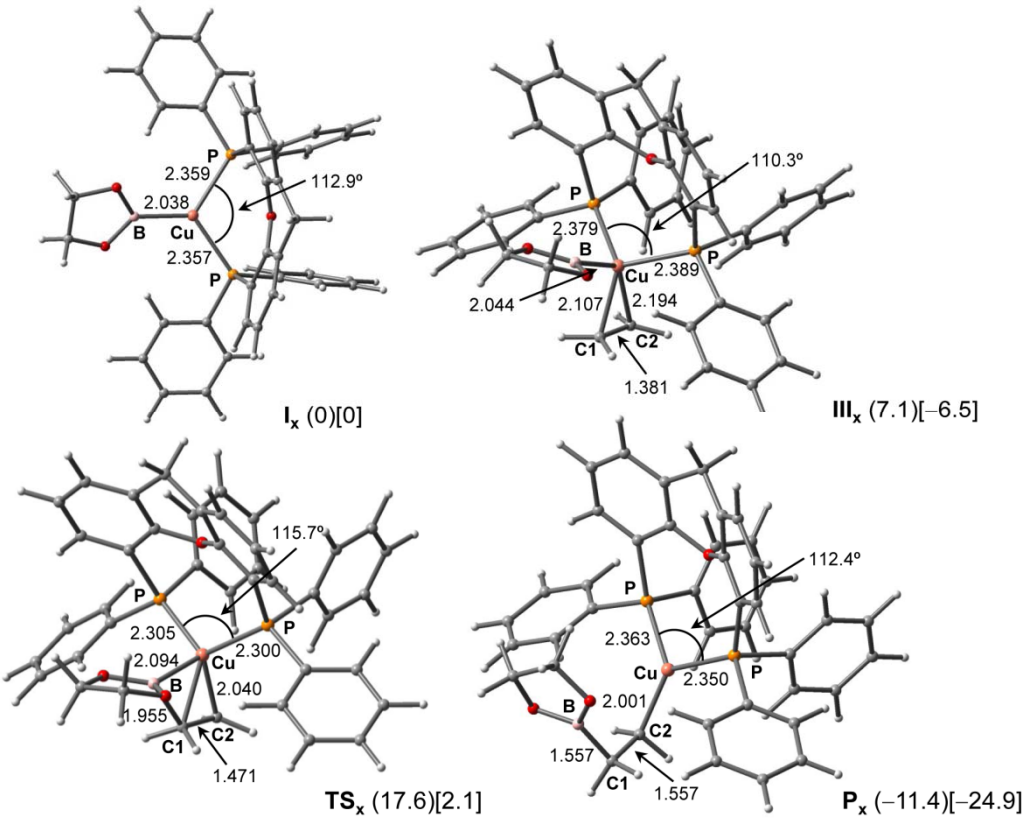
Comparison of optimization structures of Xantphos (**I_x**, **III_x**, **TS_x**, **P_x**), PPh_3 (**I_t**, **III_t**, **TS_t**, **P_t**) and IMes (**I_i**, **III_i**, **TS_i**, **P_i**) are consistent with the preactivation nature of Xantphos ligand. Summary of structural parameter and optimized structures are shown in Table S2 and Figure S2. Among the starting borylcopper(I) complexes, **I_x** (L = Xantphos) has the longest Cu–B bond, indicating the higher degree of activation in Cu–B as compared to **I_t** (X = PPh_3) and **I_i** (X = IMes). The longest Cu–B is also observed in **III_x**; on the contrary, the coordinated carbon-carbon double bond (C1–C2) is most weakened in **III_i**. **TS_x** has the longest Cu–B bond and the shortest C1–C2 bond length among structures with other ligands. This indicated that, in Xantphos complexes, the Cu–B bonds are activated throughout the reaction and that the transition state (**TS_x**) lies early in terms of the π bond scission of C1–C2 bond as compared to the reaction with other ligands.

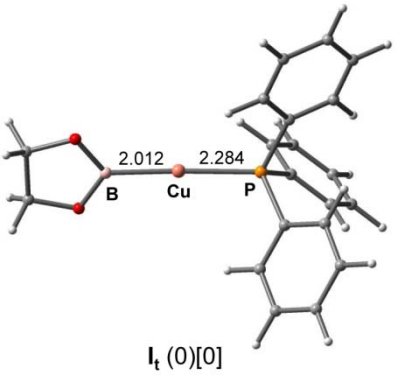
Table S2. Selected Bond Lengths of Optimized Structures

		bond length (Å)		
	ligand	Cu–B[a]	Cu–P	C1–C2[b]
ethylene	-	-	-	1.332
I_x	Xantphos	**2.038**	2.358, 2.357	-
I_t	PPh_3	2.012	2.238	-
I_i	IMes	2.011	-	-
III_x	Xantphos	**2.044**	2.379, 2.389	**1.381**
III_t	PPh_3	2.029	2.286	1.390
III_i	IMes	2.034	-	1.397
TS_x	Xantphos	**2.094**	2.305, 2.300	**1.471**
TS_t	PPh_3	2.067	2.181	1.490
TSi_t	IMes	2.068	-	1.492

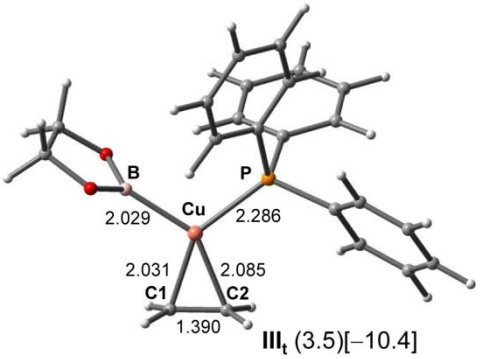
[a]The longest Cu–B bond length in the comparative stationary points were indicated by boldface. [b]The shortest C1–C2 bond length in the comparative stationary points were indicated by boldface.

Figure S2. Optimized Structures (I, III, TS, P) for Xantphos, PPh_3 and IMes with Structural Parameters and Free (in parentheses) and Electronic (in bracket) Energies

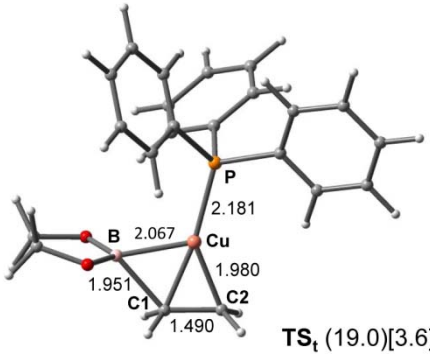




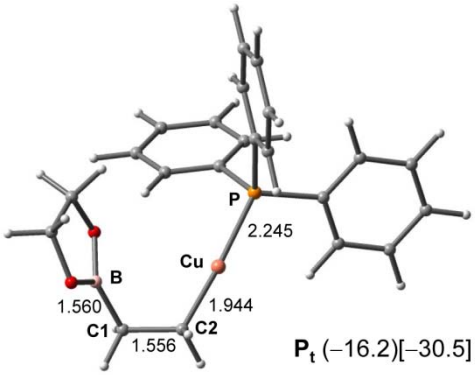
$\mathbf{I_t}$ (0)[0]



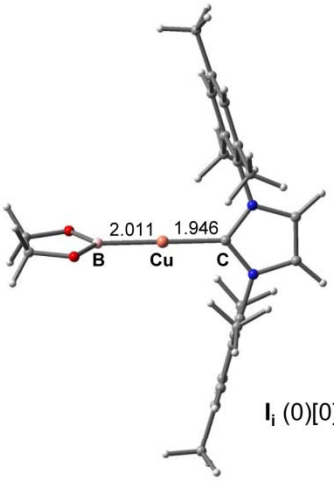
$\mathbf{III_t}$ (3.5)[–10.4]



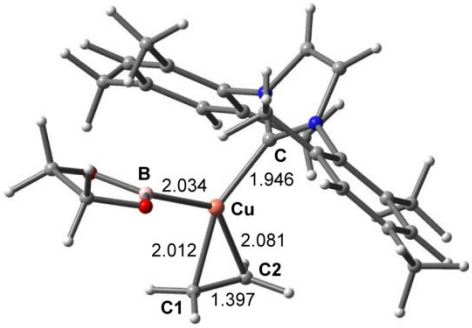
$\mathbf{TS_t}$ (19.0)[3.6]



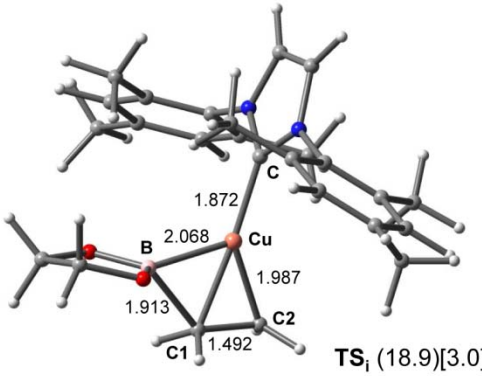
$\mathbf{P_t}$ (–16.2)[–30.5]



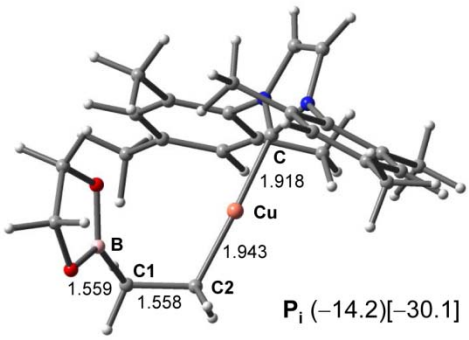
$\mathbf{I_i}$ (0)[0]



$\mathbf{III_i}$ (7.3)[–8.1]



$\mathbf{TS_i}$ (18.9)[3.0]



$\mathbf{P_i}$ (–14.2)[–30.1]

7.3. Structure Analysis of Reaction Pathways in the Reaction of Propene.

Comparison of the optimized structures in two different pathways, A and B gives important information on the regioselectivity (Figures S2, 3 and Table S3). The distance between C2 and Cu of $\mathbf{III_{PA}}$ is larger than that of $\mathbf{III_{PB}}$. This distortion in $\mathbf{III_{PA}}$ would be caused by the steric congestion between C2-CH_3 moiety and the bulky Xantphos ligand. This could destabilize the $\mathbf{III_{PA}}$ as compared to $\mathbf{III_{PB}}$. In the transition states, both boron atoms and copper centers are placed at much closer to the C1 atom as compared to those in the π-complexes. In addition, C1 atom took congested five-coordinated structure containing the newly forming C1-B and the breaking C1-Cu bonds. These structure alternation make the steric congestion around C1 atom more important than those of C2 atom in the transition states. Furthermore, C2 atoms take more sp^3-like configuration in transition sates. This could decrease the steric interaction between the substituents around C2 atoms and Xantphos ligand. These reversal structural features of the transition state against the π-complex can cause the destabilization in $\mathbf{TS_{PB}}$ as compared to $\mathbf{TS_{PA}}$. In the products ($\mathbf{P_{PA}}$ and $\mathbf{P_{PB}}$), interactions between C2 and the bulky Cu moiety become an important factor again, causing destabilization of $\mathbf{P_{PA}}$. The difference in electronic properties between -CH_2 and -$CHCH_3$ may influence the energy profiles in path A and B, although further analysis is required to clear this.

Figure S3. Two Diastereomeric Pathways for Addition Step of Borylcopper(I)(xantphos) Intermediate (I) to Propene ($\mathbf{II_P}$)

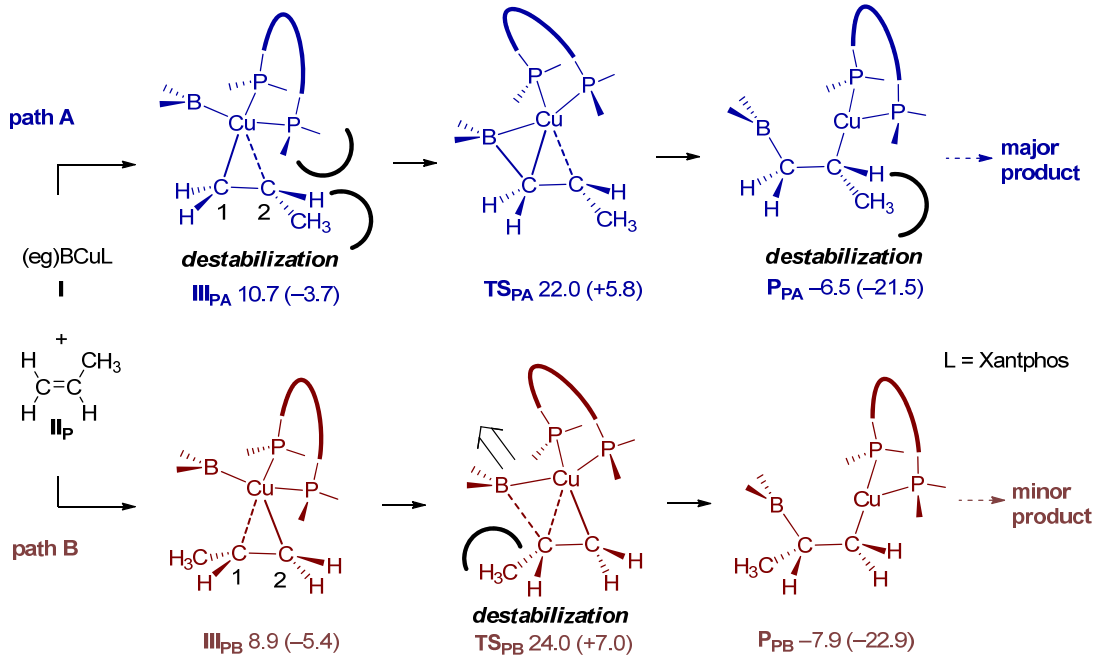
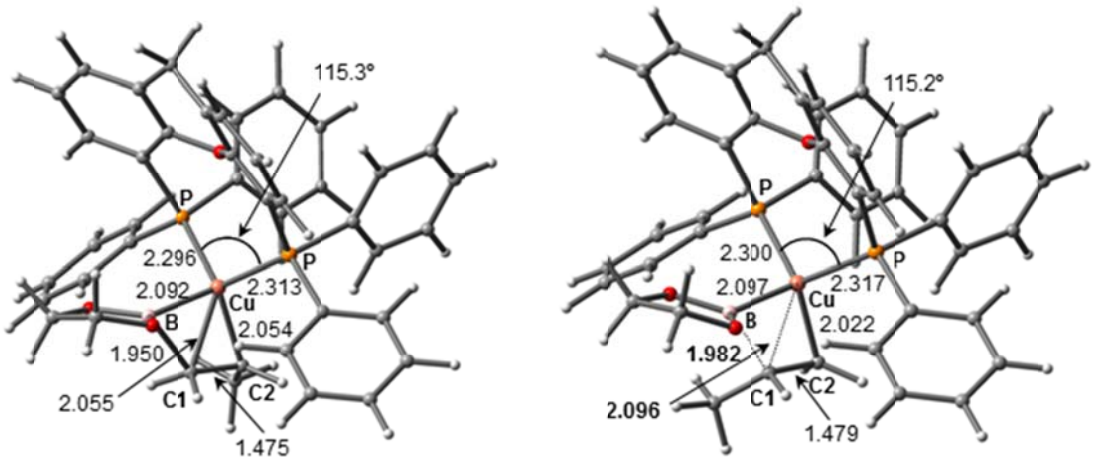


Table S3. Selected Atom Distances of Calculated TS for Path A and Path B

compound	atom distances (Å)					
	Cu–C[1]	B–C[1]	Cu–C[2]	B–Cu	Cu–P[1]	Cu–P[2]
III_{PA}	2.125	3.027	**2.267**	2.053	2.396	2.375
III_{PB}	**2.158**	**3.112**	2.189	2.049	2.380	**2.433**
Δ_{A-B}	+0.033	+0.085	–0.078	–0.004	–0.014	+0.058
TS_{PA}	2.055	1.950	**2.054**	2.092	2.296	2.313
TS_{PB}	**2.096**	**1.982**	2.022	2.09	2.300	2.317
Δ_{A-B}	+0.041	+0.032	–0.032	–0.002	+0.004	+0.004

Figure S4. Optimized Structures for the Transition States (TSx, TS_{PA}, TS_{PB}) with Structural Parameters and Free (in parentheses) and Electronic (in bracket) Energies



TS_{PA} (+22.0)[+5.8]

TS_{PB} (+24.0)[+7.0]

I_x

C	2.27490100	-0.17080700	1.45157000
C	1.19514900	-0.40824900	2.31312900
C	1.28618000	-0.28394800	3.70078000
C	2.51938400	0.08572200	4.24231900
C	3.62093200	0.30802800	3.41584300
C	3.49936600	0.18332700	2.03097300
H	2.61476000	0.19897200	5.32542700
H	4.58066400	0.59282200	3.85152800
H	4.35733100	0.38567800	1.38777000
C	0.05094300	-0.56337600	4.52156900
H	0.02828100	-1.63545400	4.79766900
H	0.07870100	-0.00122900	5.46721600
C	-1.19287800	-0.22288000	3.73756300
C	-1.15049400	-0.35606600	2.34827300
C	-2.38914000	0.21021800	4.31390300
C	-2.24628400	-0.07998000	1.51866600
C	-3.50286000	0.48329100	3.51944400
H	-2.44571000	0.33319800	5.39867200
C	-3.43234700	0.33997000	2.13275000
H	-4.43283700	0.81992700	3.98192400
H	-4.30020900	0.57724600	1.51538000
O	0.00446500	-0.77254000	1.72732800
P	1.95055300	-0.20934500	-0.37073800
P	-1.97952700	-0.15387400	-0.31224400
Cu	-0.00243300	1.08219000	-0.65854100
B	0.04791800	3.11937700	-0.70935500
O	1.06193000	3.92018800	-0.16503100
O	-0.91867800	3.94858500	-1.29592000
C	0.81003700	5.29835200	-0.45379900
H	0.88633900	5.88803100	0.47413600
H	1.57760000	5.66834700	-1.15642700
C	-0.59781000	5.32288500	-1.06361500
H	-0.64427400	5.87722000	-2.01511600
H	-1.34502900	5.75902700	-0.37733700
C	2.10138700	-2.00274600	-0.79478600
C	1.75559700	-2.39082600	-2.09843900
C	2.53464700	-2.97997600	0.11052400
C	1.86442000	-3.72261000	-2.49418900
H	1.40148800	-1.64078200	-2.81081800
C	2.62749800	-4.31637900	-0.28302900
H	2.80659200	-2.69558400	1.12907300
C	2.29899100	-4.69028700	-1.58599500
H	1.59851200	-4.00802900	-3.51454400
H	2.96474800	-5.06849100	0.43440500
H	2.37596300	-5.73578500	-1.89324400
C	-2.18838200	-1.94965300	-0.70916200
C	-2.10699600	-2.32462600	-2.06004400
C	-2.38948200	-2.94252800	0.25716100
C	-2.24383500	-3.65922100	-2.43482700
H	-1.94518600	-1.56108500	-2.82601400
C	-2.51234200	-4.28182600	-0.11960400
H	-2.46014000	-2.67038000	1.31197800
C	-2.44403700	-4.64372100	-1.46417500

H	-2.18871500	-3.93361700	-3.49096600
H	-2.66854200	-5.04535100	0.64633700
H	-2.54476900	-5.69118000	-1.75735900
C	-3.54226200	0.56253500	-0.99238000
C	-3.53429100	1.90571100	-1.39642000
C	-4.72099900	-0.18983600	-1.12027700
C	-4.69705400	2.48465300	-1.90970800
H	-2.62008000	2.50118900	-1.31721700
C	-5.87684800	0.39511400	-1.63682700
H	-4.73362900	-1.24017300	-0.82083600
C	-5.86698900	1.73493600	-2.03152300
H	-4.67937000	3.53075100	-2.22433300
H	-6.78834800	-0.19978600	-1.73377900
H	-6.77212000	2.19129600	-2.43988700
C	3.51130300	0.49562800	-1.06874500
C	3.60899700	1.89315700	-1.16723600
C	4.57989500	-0.30107000	-1.50478600
C	4.76696400	2.47572800	-1.68380200
H	2.77940000	2.52537800	-0.83715100
C	5.73018600	0.28960600	-2.03046400
H	4.51406600	-1.38873000	-1.43827500
C	5.82706600	1.67883600	-2.11965700
H	4.83494500	3.56405300	-1.75278100
H	6.55445200	-0.34183900	-2.37104200
H	6.72787200	2.13986800	-2.53207300

Zero-point correction= 0.609707 (Hartree/Particle)
Thermal correction to Energy= 0.650564
Thermal correction to Enthalpy= 0.651508
Thermal correction to Gibbs Free Energy= 0.531178
Sum of electronic and zero-point Energies= -4078.110659
Sum of electronic and thermal Energies= -4078.069802
Sum of electronic and thermal Enthalpies= -4078.068858
Sum of electronic and thermal Free Energies= -4078.189189

III_x

Cu	-0.00273200	-0.54431600	-1.31162600
C	0.08766600	-0.92311500	-3.38245600
H	-0.79873000	-1.51843000	-3.61261000
H	1.05054100	-1.41574200	-3.53606600
C	0.00696100	0.45090100	-3.26685700
H	-0.95080100	0.95959200	-3.39693900
H	0.90567600	1.06879800	-3.32475800
P	1.95438100	0.34607900	-0.27055800
P	-1.95782400	0.31915700	-0.26588000
B	0.03531600	-2.54163500	-0.87825200
O	-1.08873000	-3.36382800	-0.73709300
O	1.18910800	-3.32073500	-0.73030700
C	-0.68710100	-4.72553300	-0.55985300
H	-0.98992900	-5.31092400	-1.44574300
H	-1.19961500	-5.14710900	0.31961900
C	0.83719300	-4.66555100	-0.39118900
H	1.36987300	-5.36398400	-1.05671900
H	1.15459500	-4.87108800	0.64567200
C	-2.24442300	2.12315600	0.05951200

C	-2.64967300	4.88329600	0.42395400
C	-2.60485900	2.63192000	1.31373100
C	-2.07999900	3.02002600	-1.00818800
C	-2.29361300	4.38586000	-0.83172100
C	-2.79878800	4.00321900	1.49498900
H	-1.78967600	2.64206500	-1.99114900
H	-2.17487700	5.06649400	-1.67829700
H	-2.80827400	5.95477900	0.56606200
C	2.19290900	2.13594000	0.15054500
C	2.51824700	4.87877900	0.65706300
C	2.98674900	2.57256300	1.22137900
C	1.55666100	3.08989300	-0.65375400
C	1.72401300	4.45307600	-0.40776400
C	3.14544400	3.93546400	1.47344700
H	0.91716500	2.75429100	-1.47268300
H	1.21969200	5.18437500	-1.04317400
H	2.64310000	5.94576200	0.85636600
C	2.27967300	-0.46037000	1.36177000
C	2.49165500	-1.78880300	3.83203700
C	3.48531000	-1.05553000	1.74928400
C	1.20233800	-0.53695800	2.25320000
C	1.27785800	-1.19097000	3.48413900
C	3.59032400	-1.71540600	2.97571300
H	4.34220400	-1.01974800	1.07429000
H	4.53575400	-2.18155300	3.26074200
H	2.57479700	-2.31266100	4.78803500
C	-2.23302000	-0.43294600	1.40420800
C	-2.41461300	-1.74213700	3.88813900
C	-3.44065000	-1.00277000	1.82479800
C	-1.13805500	-0.52038600	2.27372400
C	-1.19885600	-1.16847400	3.50997400
C	-3.53135700	-1.65196600	3.05716200
H	-4.31127000	-0.95742100	1.16853100
H	-4.47867000	-2.09878000	3.36569100
H	-2.48487300	-2.26080000	4.84803000
C	-3.50613800	-0.15775700	-1.16682300
C	-5.77374500	-0.98088100	-2.60485100
C	-3.56061200	-1.44337500	-1.73196600
C	-4.59903600	0.70598600	-1.32752000
C	-5.72408500	0.29620800	-2.04595000
C	-4.69226100	-1.84863100	-2.44026400
H	-2.72242600	-2.13251500	-1.59619400
H	-4.57484900	1.70606000	-0.89109200
H	-6.56686500	0.98149000	-2.16557300
H	-4.72519600	-2.85264900	-2.87039800
H	-6.65446900	-1.29996000	-3.16745400
C	3.48527200	-0.04397100	-1.23624800
C	5.69963800	-0.72040600	-2.82762500
C	4.43704200	0.92589300	-1.58077500
C	3.64883300	-1.36017300	-1.70508400
C	4.75532400	-1.69155100	-2.48716400
C	5.53550300	0.58806500	-2.37455900
H	4.32349900	1.95312300	-1.23001800
H	2.90988400	-2.12544200	-1.44932300

H	4.87524300	-2.71917000	-2.83919600
H	6.26743800	1.35600500	-2.63671700
H	6.56030700	-0.98325000	-3.44740900
H	3.48285100	1.84196300	1.86379300
H	-2.74121200	1.95251500	2.15704500
H	-3.07498700	4.38267200	2.48175700
H	3.76385500	4.26211600	2.31313000
O	0.03204800	0.07112400	1.86185200
C	0.04861100	-1.19381700	4.35864600
H	0.06408900	-0.30224000	5.01523000
H	0.04701300	-2.06893500	5.02605100

Zero-point correction= 0.663296 (Hartree/Particle)
Thermal correction to Energy= 0.707467
Thermal correction to Enthalpy= 0.708412
Thermal correction to Gibbs Free Energy= 0.582151
Sum of electronic and zero-point Energies= -4156.628149
Sum of electronic and thermal Energies= -4156.583978
Sum of electronic and thermal Enthalpies= -4156.583034
Sum of electronic and thermal Free Energies= -4156.709294

TS_x

Cu	0.00245600	-0.41226400	-1.26853600
C	-0.01990800	-1.54036600	-2.98820100
H	-0.92564500	-2.08720800	-3.28383600
H	0.89046500	-2.06660400	-3.30286000
C	-0.03709800	-0.09877100	-3.28231200
H	-0.96684300	0.30237300	-3.69679000
H	0.86858900	0.31434800	-3.73618100
P	1.94990800	0.25870900	-0.24678100
P	-1.94817700	0.25965200	-0.23964500
B	-0.00629300	-2.50646400	-1.28833200
O	-1.15270100	-3.24890900	-0.98199800
O	1.12729500	-3.31038600	-1.11404200
C	-0.78342800	-4.59761400	-0.69128000
H	-1.09191800	-5.24754300	-1.52994200
H	-1.30640800	-4.93450100	0.21705500
C	0.74024900	-4.55074200	-0.52361100
H	1.25512300	-5.37915300	-1.03531600
H	1.04308600	-4.55708900	0.53793900
C	-2.10527200	2.08480000	0.02350300
C	-2.38484100	4.86669800	0.24852200
C	-1.76377500	2.92165100	-1.04831300
C	-2.57759800	2.65521300	1.21190500
C	-2.70872200	4.04083200	1.32510300
C	-1.91545800	4.30311400	-0.93973900
H	-2.84917800	2.01474100	2.05351100
H	-3.07191800	4.47500300	2.25981800
H	-2.49304700	5.95034600	0.33674100
C	2.11901900	2.08338200	0.01118700
C	2.41754300	4.86384300	0.22639400
C	2.60121800	2.65406300	1.19545900
C	1.77692500	2.91912800	-1.06122200
C	1.93815600	4.29989700	-0.95769400
C	2.74176700	4.03913200	1.30379300

H	1.38075200	2.47188100	-1.97699200
H	1.67745300	4.94064900	-1.80332500
H	2.53318600	5.94702300	0.31082200
C	2.25658000	-0.42340700	1.44887700
C	2.45686400	-1.65195800	3.97416000
C	3.46445900	-0.99009300	1.87457000
C	1.17216700	-0.47224200	2.33552900
C	1.24160600	-1.07884700	3.59154100
C	3.56357300	-1.60014400	3.12666700
H	4.32870500	-0.97185600	1.20858700
H	4.51102800	-2.04404700	3.43915500
H	2.53489600	-2.13759300	4.95055300
C	-2.25466300	-0.42368500	1.45475200
C	-2.44884200	-1.65313400	3.97977400
C	-3.46112700	-0.99143900	1.88224000
C	-1.16845200	-0.47276800	2.33879900
C	-1.23466300	-1.07929400	3.59474600
C	-3.55714800	-1.60201500	3.13436200
H	-4.32654600	-0.97360700	1.21777200
H	-4.50358100	-2.04685700	3.44863500
H	-2.52466500	-2.13901000	4.95622000
C	-3.53792200	-0.12690300	-1.11444600
C	-5.89400500	-0.78897900	-2.49583700
C	-3.71071300	-1.41339000	-1.65103700
C	-4.55830900	0.82094300	-1.27840800
C	-5.72720900	0.49070700	-1.96687600
C	-4.88431800	-1.73885000	-2.33165700
H	-2.92863200	-2.16543000	-1.52261100
H	-4.44160700	1.82624000	-0.87069700
H	-6.51108800	1.24221600	-2.08886600
H	-5.00618600	-2.74431000	-2.74206500
H	-6.80826800	-1.04513000	-3.03662800
C	3.53570200	-0.14243100	-1.12128800
C	5.88481200	-0.83152400	-2.50031500
C	4.55890500	0.79944900	-1.30019600
C	3.70168600	-1.43658900	-1.64123100
C	4.87207800	-1.77543800	-2.32080500
C	5.72443500	0.45568700	-1.98784800
H	4.44718800	1.81020200	-0.90477700
H	2.91663200	-2.18398800	-1.50357500
H	4.98852400	-2.78656900	-2.71859000
H	6.51081100	1.20248800	-2.12208800
H	6.79650300	-1.09816500	-3.04038400
H	-1.37564200	2.47478000	-1.96766500
H	2.87285100	2.01431200	2.03761300
H	-1.65517700	4.94467500	-1.78488900
H	3.11252800	4.47380000	2.23529300
O	0.00143300	0.11249200	1.91384600
C	0.00456300	-1.06043800	4.45521700
H	0.00572100	-1.90970300	5.15518400
H	0.00536300	-0.14461200	5.07763000

Zero-point correction= 0.663615 (Hartree/Particle)
Thermal correction to Energy= 0.706412
Thermal correction to Enthalpy= 0.707356

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Thermal correction to Gibbs Free Energy=        0.585209
Sum of electronic and zero-point Energies=         -4156.614123
Sum of electronic and thermal Energies=           -4156.571326
Sum of electronic and thermal Enthalpies=          -4156.570382
Sum of electronic and thermal Free Energies=       -4156.692529
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P_x

Cu	-0.01708000	-0.85601600	-0.97627800
C	0.34402500	-3.64873900	-2.11353400
H	0.03242000	-4.38710100	-2.87951400
H	1.44707700	-3.64265700	-2.11254800
C	-0.18672100	-2.21954900	-2.43099000
H	-1.25121100	-2.30194000	-2.72254600
H	0.33022200	-1.85142800	-3.34030200
P	1.98596400	0.10479400	-0.20994500
P	-1.92615000	0.29880700	-0.19672900
B	-0.14439300	-4.07335000	-0.69728900
O	-1.41763500	-4.53648800	-0.39931300
O	0.63951800	-3.98609100	0.44670000
C	-1.47632800	-4.84998600	0.99177600
H	-1.55471500	-5.94460800	1.10944300
H	-2.37461200	-4.39221400	1.43547100
C	-0.16999700	-4.28933400	1.58217700
H	0.35510600	-5.01282500	2.22538800
H	-0.33576900	-3.36628700	2.16341600
C	-2.02319800	1.93473900	-1.04918300
C	-2.15321700	4.33786800	-2.49238300
C	-1.84794000	1.93910400	-2.44257000
C	-2.25416000	3.14676600	-0.38754900
C	-2.31553900	4.34186000	-1.10717900
C	-1.92039100	3.13254600	-3.15816200
H	-2.39494500	3.15955700	0.69499200
H	-2.49680800	5.28118200	-0.57911100
H	-2.20577400	5.27356900	-3.05374400
C	2.27759100	1.67097700	-1.14247700
C	2.68926600	3.97195400	-2.69504900
C	2.72965200	2.85552900	-0.54762900
C	2.02342600	1.65324100	-2.52331800
C	2.23707700	2.79451500	-3.29434000
C	2.92998300	4.00044800	-1.32136000
H	1.65946800	0.73539000	-2.99290400
H	2.04199600	2.76527500	-4.36880500
H	2.85033400	4.86806000	-3.29884600
C	2.29615100	0.58279100	1.55589500
C	2.52980500	1.09568000	4.31811400
C	3.49336700	0.32167500	2.23764500
C	1.24047300	1.12867100	2.29917500
C	1.32711000	1.38366100	3.67053000
C	3.61073300	0.57846600	3.60402900
H	4.33469800	-0.10873300	1.69256600
H	4.55034900	0.36221900	4.11645000
H	2.61794400	1.28220400	5.39162300
C	-2.19360800	0.71812800	1.58715800
C	-2.34155800	1.17890700	4.36455800

C	-3.38411700	0.48848600	2.29100000
C	-1.09442600	1.18734100	2.32032900
C	-1.14184300	1.42910300	3.69541900
C	-3.45680500	0.71513600	3.66629900
H	-4.25735300	0.11231200	1.75611400
H	-4.39180500	0.52437400	4.19705100
H	-2.40029700	1.35333700	5.44209000
C	-3.54260800	-0.46769900	-0.66353900
C	-5.94807400	-1.73240500	-1.35825000
C	-3.60185800	-1.86302600	-0.78052400
C	-4.69705200	0.29044900	-0.91391100
C	-5.89264400	-0.34011200	-1.25794500
C	-4.80181900	-2.49085900	-1.12046000
H	-2.70381400	-2.46646500	-0.63166000
H	-4.65933800	1.38026700	-0.84783700
H	-6.78470700	0.25996700	-1.45348000
H	-4.82826500	-3.57877300	-1.21449800
H	-6.88441200	-2.22401700	-1.63321700
C	3.50672000	-0.87926200	-0.58649900
C	5.75457500	-2.47565400	-1.10038000
C	4.67301500	-0.31109400	-1.11794400
C	3.47179400	-2.25699800	-0.32250300
C	4.59520800	-3.04615400	-0.57177300
C	5.78969000	-1.10816500	-1.37626500
H	4.70943400	0.75805100	-1.33721400
H	2.56127200	-2.72026800	0.06709900
H	4.55576100	-4.11777800	-0.36309600
H	6.69126600	-0.65567900	-1.79643700
H	6.62876900	-3.09839400	-1.30512900
H	-1.65869700	0.99836500	-2.96681900
H	2.93100700	2.88497200	0.52516600
H	-1.78920900	3.12117500	-4.24261200
H	3.28113500	4.91920500	-0.84534900
O	0.07295300	1.40798600	1.62486000
C	0.10939700	1.95307800	4.35509100
H	0.11656800	1.71357500	5.42882300
H	0.12735400	3.05756000	4.27973400

Zero-point correction= 0.664537 (Hartree/Particle)
Thermal correction to Energy= 0.708151
Thermal correction to Enthalpy= 0.709096
Thermal correction to Gibbs Free Energy= 0.581897
Sum of electronic and zero-point Energies= -4156.656120
Sum of electronic and thermal Energies= -4156.612505
Sum of electronic and thermal Enthalpies= -4156.611561
Sum of electronic and thermal Free Energies= -4156.738759

I_t

P	-0.57310800	0.02068700	0.01043600
C	-1.20346800	-0.97492800	-1.40500900
C	-2.33823300	-0.62442800	-2.14846700
C	-0.48344100	-2.12964800	-1.74985900
C	-2.75321400	-1.42680800	-3.21280500
C	-0.90616900	-2.93163100	-2.80829500
C	-2.04185800	-2.58141200	-3.54187400

C	-1.39941900	1.65769500	-0.15687200
C	-2.63184800	1.95442000	0.44070100
C	-0.74362100	2.64427700	-0.90969100
C	-3.20465200	3.21636500	0.27602300
C	-1.32267200	3.90156200	-1.07643700
C	-2.55353700	4.18931600	-0.48388200
C	-1.35557800	-0.73614900	1.49713200
C	-0.82766700	-0.39190500	2.75137900
C	-2.42608500	-1.63709900	1.42605500
C	-1.37771500	-0.92427300	3.91614800
C	-2.96788700	-2.17481900	2.59504900
C	-2.44864600	-1.81726600	3.83967900
Cu	1.70854800	0.08592100	0.07694100
B	3.72065800	0.08487800	0.08131600
O	4.50549000	-1.05112900	0.26108400
O	4.52376100	1.20675500	-0.10348500
C	5.88820700	-0.69830700	0.16175000
H	6.41465200	-1.02761000	1.07307000
H	6.33583000	-1.22708300	-0.69746000
C	5.90085200	0.82957400	-0.01390900
H	6.42644800	1.14935500	-0.92910600
H	6.36380400	1.35046500	0.84193700
H	-2.36772400	-3.20533300	-4.37697900
H	-0.33888300	-3.82820400	-3.06830500
H	0.41778600	-2.39484100	-1.19020000
H	-2.89527000	0.28182500	-1.90170900
H	-3.63725400	-1.14470200	-3.78919600
H	-2.83433700	-1.92507400	0.45506900
H	-3.80062200	-2.87896600	2.53035100
H	-3.14150200	1.20011300	1.04403400
H	-4.16417200	3.44054600	0.74769800
H	-3.00244900	5.17758200	-0.60763900
H	-0.80350300	4.66325400	-1.66237400
H	-0.96013700	-0.64890300	4.88708200
H	-2.87372800	-2.24109100	4.75230400
H	0.02267000	0.29263800	2.81369700
H	0.22973700	2.42740100	-1.35836500

Zero-point correction= 0.344167 (Hartree/Particle)
Thermal correction to Energy= 0.368201
Thermal correction to Enthalpy= 0.369146
Thermal correction to Gibbs Free Energy= 0.283121
Sum of electronic and zero-point Energies= -2930.212235
Sum of electronic and thermal Energies= -2930.188201
Sum of electronic and thermal Enthalpies= -2930.187257
Sum of electronic and thermal Free Energies= -2930.273281

III_t

C	1.41996800	-3.22480000	-1.72034500
C	0.03883000	-3.10071100	-1.62407900
B	2.85095100	-0.65325300	-0.44071800
O	3.65576100	0.02165500	-1.35767200
O	3.47527800	-0.68519600	0.81016500
C	4.88110500	0.41269200	-0.72903700
C	4.70926700	0.03914800	0.75182700

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H                1.94641800   -2.98401300   -2.64794800
H               -0.50900800   -3.60417800   -0.82242400
H                1.98392500   -3.82686200   -1.00298600
H                5.52500500   -0.59883000    1.12902600
H                4.63877800    0.92563900    1.40527900
H                5.71879000   -0.12547600   -1.20509700
H                5.04081300    1.49278800   -0.88030500
Cu               1.02249200   -1.44415100   -0.82773700
P               -0.61386300   -0.04109700   -0.06556500
H               -0.55948200   -2.75644100   -2.47236700
C               -2.34759000   -0.21557500   -0.68485000
C               -3.01654400   -1.43161600   -0.46405800
C               -2.97944300    0.77301000   -1.45068800
C               -4.29472800   -1.64156800   -0.97629600
C               -4.25588100    0.55416000   -1.97478100
C               -4.91856800   -0.64866800   -1.73643300
C               -0.24040700    1.74774100   -0.32627500
C                0.70054400    2.10120400   -1.30183400
C               -0.87286600    2.75599700    0.41657300
C                0.98844700    3.44576800   -1.54618500
C               -0.57712200    4.09681100    0.17531000
C                0.35045900    4.44388700   -0.81020600
C               -0.76702500   -0.19982100    1.76810500
C                0.40891500   -0.44405300    2.49688900
C               -1.98752700   -0.08087100    2.44834900
C                0.35542300   -0.56094300    3.88611300
C               -2.03195600   -0.20226600    3.83807900
C               -0.86164700   -0.44248700    4.55924500
H               -1.07078600    4.87481800    0.76237600
H                1.27533300   -0.75173700    4.44347500
H                1.36801000   -0.53861600    1.97847200
H                0.58174200    5.49531500   -0.99682900
H                1.72476400    3.70940900   -2.30870900
H                1.23327200    1.32041700   -1.84993000
H               -2.98826500   -0.10876200    4.35828400
H               -0.89911100   -0.53968700    5.64684200
H               -1.59434100    2.49327200    1.19347400
H               -2.47449800    1.72151200   -1.64126700
H               -2.90983200    0.09965100    1.89218000
H               -2.53255800   -2.21850400    0.11938000
H               -4.80457900   -2.58904200   -0.78663400
H               -5.91799600   -0.81642800   -2.14407300
H               -4.73369800    1.33493100   -2.57124400
 Zero-point correction=                          0.397569 (Hartree/Particle)
 Thermal correction to Energy=                    0.424725
 Thermal correction to Enthalpy=                  0.425670
 Thermal correction to Gibbs Free Energy=         0.334560
 Sum of electronic and zero-point Energies=          -3008.736119
 Sum of electronic and thermal Energies=             -3008.708962
 Sum of electronic and thermal Enthalpies=           -3008.708018
 Sum of electronic and thermal Free Energies=        -3008.799128
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TS_t

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C                2.57392600   -2.55676200   -1.05328800
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C	1.30851300	-3.30824100	-1.28250400
B	2.86753500	-0.72726600	-0.44112500
O	3.49484200	0.09390300	-1.37187600
O	3.41920100	-0.56190600	0.82895300
C	4.40902300	0.94811200	-0.67975100
C	4.52329300	0.34396800	0.72864700
H	3.17046700	-2.35718200	-1.95339500
H	1.10155200	-4.12450400	-0.58300100
H	3.20020100	-2.92944000	-0.23076600
H	5.46299200	-0.21890500	0.86865500
H	4.45271400	1.09947800	1.52642900
H	5.37030100	0.96762500	-1.21698600
H	4.00110200	1.97338000	-0.66136800
Cu	0.94839500	-1.45843900	-0.67459700
P	-0.62435900	-0.06759900	-0.08513800
H	1.08396700	-3.56062900	-2.32394100
C	-0.90454200	-0.23080200	1.73034100
C	0.22714500	-0.26108400	2.56187500
C	-2.18015900	-0.35088200	2.29612600
C	0.07620300	-0.39361400	3.94138100
C	-2.32294900	-0.48805700	3.67847900
C	-1.19775200	-0.50648600	4.50261500
C	-2.28096500	-0.34908300	-0.85497800
C	-2.57193700	-1.64644000	-1.29906800
C	-3.23821200	0.66392800	-1.00869900
C	-3.81358100	-1.93098800	-1.86778500
C	-4.47566900	0.37597500	-1.58592600
C	-4.76670400	-0.92141800	-2.01182200
C	-0.32430800	1.73615900	-0.34067000
C	0.38440200	2.12845600	-1.48522400
C	-0.77020400	2.71405000	0.55896100
C	0.62142900	3.47904600	-1.73811900
C	-0.52118800	4.06494300	0.30839700
C	0.16930500	4.44972600	-0.84155200
H	-0.86790500	4.81969800	1.01826400
H	0.36298700	5.50725400	-1.03564800
H	1.17177500	3.77320300	-2.63473200
H	0.76838400	1.36949300	-2.17119300
H	-1.30554200	2.41947600	1.46449500
H	-3.01626200	1.68386500	-0.68768500
H	-5.21451100	1.17173200	-1.70688700
H	-5.73536200	-1.14287700	-2.46617800
H	-3.06534100	-0.34599100	1.65712100
H	-4.03174600	-2.94498000	-2.21088200
H	-1.80348100	-2.42103200	-1.20678600
H	1.22747400	-0.19328200	2.12459600
H	0.96190100	-0.41871500	4.58029300
H	-1.31258300	-0.61685100	5.58344500
H	-3.32157800	-0.58477800	4.11093900

Zero-point correction= 0.397388 (Hartree/Particle)
Thermal correction to Energy= 0.423411
Thermal correction to Enthalpy= 0.424355
Thermal correction to Gibbs Free Energy= 0.336980
Sum of electronic and zero-point Energies= -3008.713931

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Sum of electronic and thermal Energies=           -3008.687908
Sum of electronic and thermal Enthalpies=         -3008.686964
Sum of electronic and thermal Free Energies=      -3008.774339
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P_t

C	3.57001200	-2.24129300	-0.99498000
C	2.38072400	-2.03115900	-1.97528900
B	4.04480400	-0.87527500	-0.40970900
O	5.08574500	-0.12963200	-0.92250500
O	3.44021400	-0.23789200	0.66858300
C	5.23121300	1.05740400	-0.14778200
C	4.03668100	1.04971800	0.82708700
H	4.41982000	-2.75007200	-1.49050400
H	2.06469400	-3.01323100	-2.37828600
H	3.25519100	-2.89369700	-0.16099700
H	4.34183400	1.18538400	1.87707700
H	3.29124000	1.82490400	0.58210400
H	6.20196200	1.02982900	0.37609200
H	5.22856700	1.93404900	-0.81507300
Cu	0.84301100	-1.13271500	-1.19585500
P	-0.86091700	-0.10764100	-0.15398200
H	2.73573500	-1.46110800	-2.85631600
C	-0.88844100	-0.49074600	1.64721700
C	0.35001200	-0.64214600	2.29196000
C	-2.07404200	-0.65106800	2.37732100
C	0.39307400	-0.92957900	3.65630800
C	-2.02184600	-0.94626000	3.74057400
C	-0.78984500	-1.08250900	4.38200000
C	-2.55029600	-0.53770600	-0.75144400
C	-2.77882500	-1.86414200	-1.14891700
C	-3.59408100	0.39349000	-0.83570600
C	-4.03687600	-2.25602100	-1.60400300
C	-4.85009300	-0.00129100	-1.29992000
C	-5.07449300	-1.32474800	-1.68081900
C	-0.80291700	1.73298100	-0.24130400
C	-0.44164100	2.31464900	-1.46661600
C	-1.08475600	2.56003800	0.85389600
C	-0.38385400	3.70135800	-1.59815400
C	-1.01710600	3.94826900	0.72039500
C	-0.67097600	4.52059400	-0.50414900
H	-1.23573900	4.58503400	1.58082400
H	-0.61780500	5.60703800	-0.60513700
H	-0.10365400	4.14335400	-2.55694200
H	-0.19889500	1.67506900	-2.31963600
H	-1.35131500	2.11835400	1.81631500
H	-3.42491600	1.43252300	-0.54510300
H	-5.65698700	0.73230600	-1.36556700
H	-6.05779400	-1.62992700	-2.04590500
H	-3.04137800	-0.55457700	1.87955800
H	-4.20375200	-3.29105800	-1.91034700
H	-1.96244800	-2.59032600	-1.10674300
H	1.28270800	-0.54359800	1.72698800
H	1.35999400	-1.04749500	4.15061300
H	-0.75186300	-1.31770700	5.44824300

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H              -2.95022800   -1.07412800    4.30214100
Zero-point correction=                          0.399353 (Hartree/Particle)
Thermal correction to Energy=                    0.425972
Thermal correction to Enthalpy=                  0.426916
Thermal correction to Gibbs Free Energy=         0.335296
Sum of electronic and zero-point Energies=          -3008.766362
Sum of electronic and thermal Energies=             -3008.739743
Sum of electronic and thermal Enthalpies=           -3008.738799
Sum of electronic and thermal Free Energies=        -3008.830419
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I_i

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C              -0.67926100    3.08930200   -0.00532200
C               0.67897000    3.08936600    0.00178200
C              -0.00005300    0.91502200   -0.00029400
N              -1.07223300    1.75867900   -0.00563200
H              -1.39480500    3.90510500   -0.01187400
H               1.39444100    3.90524000    0.00722500
N               1.07205500    1.75877700    0.00391200
C              -2.44317800    1.32539900   -0.00692300
C              -3.12735800    1.24308900    1.21502500
C              -3.05760300    1.01284100   -1.22994200
C              -4.46845800    0.84837400    1.18644100
C              -4.39896700    0.62256400   -1.20265800
C              -5.12179000    0.53369400   -0.00841900
H              -5.01464400    0.77931500    2.13111800
H              -4.89133200    0.37584300   -2.14733700
C               2.44303200    1.32560400    0.00605600
C               3.05731100    1.01505700    1.22965200
C               3.12739100    1.24137700   -1.21567200
C               4.39871600    0.62484200    1.20318600
C               4.46850100    0.84678800   -1.18626700
C               5.12169300    0.53406400    0.00919300
H               4.89097500    0.37971600    2.14833400
H               5.01482500    0.77626300   -2.13075700
C              -2.43841300    1.53551000    2.51906900
H              -1.61179000    0.82710900    2.68987900
H              -2.00525200    2.54792400    2.54249300
H              -3.14036900    1.44752900    3.35923700
C              -2.29580500    1.06155200   -2.52459500
H              -1.75789500    2.01367900   -2.65200900
H              -1.54314500    0.25641700   -2.55948900
H              -2.97173700    0.93151600   -3.38060900
C               2.43860000    1.53172400   -2.52025800
H               1.61180100    0.82324900   -2.68989900
H               2.00570300    2.54421100   -2.54544000
H               3.14058800    1.44212300   -3.36022800
C               2.29535000    1.06576700    2.52413700
H               1.75716700    2.01795800    2.64988900
H               1.54290400    0.26048900    2.56032200
H               2.97121600    0.93736400    3.38045100
C               6.55580600    0.07902100    0.01323500
H               7.07737200    0.37415700   -0.90855900
H               7.10914800    0.49527800    0.86867100
H               6.61649100   -1.01973200    0.09032400
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C	-6.55594000	0.07875800	-0.01160200
H	-6.61681800	-1.01994600	-0.08909600
H	-7.07681800	0.37363100	0.91066200
H	-7.10984700	0.49542300	-0.86648700
Cu	0.00005200	-1.03096700	0.00045600
B	0.00017600	-3.04205000	0.00066700
O	-0.22832100	-3.84679000	-1.11939100
O	0.22879000	-3.84664000	1.12080000
C	-0.10977300	-5.22389900	-0.75999200
H	-1.02589300	-5.76182100	-1.05791200
H	0.73727600	-5.67251700	-1.30885600
C	0.11046100	-5.22380500	0.76153800
H	-0.73651500	-5.67250200	1.31044700
H	1.02666800	-5.76154800	1.05951300

Zero-point correction= 0.466373 (Hartree/Particle)
Thermal correction to Energy= 0.498030
Thermal correction to Enthalpy= 0.498974
Thermal correction to Gibbs Free Energy= 0.395969
Sum of electronic and zero-point Energies= -2817.951584
Sum of electronic and thermal Energies= -2817.919928
Sum of electronic and thermal Enthalpies= -2817.918983
Sum of electronic and thermal Free Energies= -2818.021989

III_i

C	-0.68772800	-2.09031400	-2.03520300
C	0.66798600	-2.10007000	-2.02912900
C	-0.00733900	-0.73993600	-0.32584000
N	-1.08213900	-1.25962400	-0.99292800
H	-1.40515100	-2.58548000	-2.68131600
H	1.38396000	-2.60613700	-2.66835900
N	1.06552600	-1.27443100	-0.98380400
C	-2.45418400	-1.03561400	-0.63222300
C	-3.14393800	0.05852500	-1.17346000
C	-3.07495400	-1.95312500	0.23533900
C	-4.48840700	0.22120900	-0.81395000
C	-4.41497900	-1.74258400	0.56369100
C	-5.13918600	-0.65879900	0.05165800
H	-5.03909600	1.07044800	-1.22757700
H	-4.90795900	-2.44781400	1.23886400
C	2.43881800	-1.06848800	-0.61657000
C	3.15345000	0.00001800	-1.17625800
C	3.03646400	-1.98057300	0.27244100
C	4.49950500	0.14176400	-0.81447500
C	4.37930800	-1.79144300	0.60251100
C	5.12828700	-0.73404900	0.07145900
H	5.06939300	0.97088900	-1.24266700
H	4.85472300	-2.49307200	1.29383400
C	-2.47743800	1.05335100	-2.07982100
H	-1.96167200	1.81918700	-1.47648500
H	-1.72834300	0.58311100	-2.73226400
H	-3.22078200	1.55969200	-2.71219700
C	-2.32025100	-3.12646300	0.79742300
H	-1.96870700	-3.80486500	0.00323100
H	-1.42877400	-2.79444600	1.35161600

H	-2.95472800	-3.70733300	1.48082600
C	2.25472600	-3.12645900	0.85374400
H	1.37035800	-2.76428700	1.40046500
H	1.88871100	-3.81014500	0.07066500
H	2.87496000	-3.70967200	1.54812400
C	2.51117700	0.99152600	-2.10335500
H	1.75015300	0.52743300	-2.74614400
H	2.01544600	1.78082200	-1.51395300
H	3.26616100	1.46723600	-2.74567100
C	6.56839700	-0.54332000	0.46438600
H	7.11997700	-1.49616800	0.44944200
H	7.08159000	0.15891100	-0.20792300
H	6.64629800	-0.13962000	1.48789800
C	-6.57697900	-0.44686000	0.44225800
H	-6.65321800	-0.08926200	1.48282400
H	-7.06520600	0.29906500	-0.20086900
H	-7.15378200	-1.38276000	0.37893200
Cu	-0.01663900	0.60958100	1.07609000
B	0.04797500	2.53133200	0.41246200
O	1.21430400	3.27649000	0.19422800
O	-1.07069500	3.33257600	0.14601000
C	0.87422800	4.63025000	-0.12163700
H	1.19476200	5.28575300	0.70688800
H	1.41439700	4.94280600	-1.03027300
C	-0.64980300	4.62371500	-0.30541500
H	-1.15586600	5.40231800	0.28818200
H	-0.94760600	4.75404100	-1.36093600
C	-0.11262800	-0.30426700	2.94291000
H	-1.05837500	-0.84673900	3.02516300
H	0.79570400	-0.89067500	3.10583800
C	-0.08290300	1.08972500	3.02873900
H	0.84328400	1.61359600	3.28060900
H	-1.00030700	1.65821000	3.20420000

Zero-point correction= 0.520272 (Hartree/Particle)
Thermal correction to Energy= 0.554637
Thermal correction to Enthalpy= 0.555582
Thermal correction to Gibbs Free Energy= 0.449802
Sum of electronic and zero-point Energies= -2896.471211
Sum of electronic and thermal Energies= -2896.436845
Sum of electronic and thermal Enthalpies= -2896.435901
Sum of electronic and thermal Free Energies= -2896.541681

TS_i

Cu	-0.00321500	0.45735700	1.09170000
C	-0.01312500	1.92586400	2.44755000
H	-0.91827600	2.52431000	2.62149500
H	0.89506400	2.51062200	2.64798800
C	-0.03117300	0.57306600	3.07539300
H	-0.95584100	0.29842800	3.59356100
H	0.87264200	0.29002800	3.62484000
B	0.01892700	2.46902700	0.61378800
O	-1.11698100	3.12738200	0.13685400
O	1.17885900	3.14189100	0.22490100
C	-0.70762100	4.35444200	-0.46914300

H	-0.97845300	5.19479100	0.19551800
H	-1.23669200	4.49140700	-1.42556200
C	0.81265200	4.21769100	-0.63788800
H	1.36090600	5.12781000	-0.34668400
H	1.09448600	3.96617200	-1.67638400
C	0.66835100	-2.36416400	-1.81203200
C	-0.68755400	-2.35733600	-1.81405400
C	-0.00440900	-0.80638000	-0.28885000
N	1.07080800	-1.41384900	-0.88061000
H	1.38189700	-2.95438400	-2.37739700
H	-1.40543100	-2.93980600	-2.38197200
N	-1.08353400	-1.40300200	-0.88399800
C	2.43935300	-1.12515100	-0.56156200
C	3.04288300	-1.79263400	0.51668600
C	3.13735500	-0.19412000	-1.34559100
C	4.38543800	-1.51224600	0.78712200
C	4.47830400	0.04963000	-1.03413900
C	5.11995800	-0.59682100	0.02630300
H	4.86901000	-2.02634700	1.62257300
H	5.03359700	0.77353600	-1.63687700
C	-2.45103900	-1.10709100	-0.56579900
C	-3.06313100	-1.78788400	0.49726900
C	-3.13893900	-0.15425500	-1.33546300
C	-4.40666700	-1.50348800	0.76675900
C	-4.47871200	0.09106300	-1.02674900
C	-5.13130500	-0.57269600	0.01829100
H	-4.89728000	-2.02723300	1.59180700
H	-5.02542100	0.83112500	-1.61801600
C	2.26588900	-2.75490400	1.37014500
H	1.46663400	-2.22244400	1.91314400
H	1.78252600	-3.53993800	0.76750000
H	2.92095200	-3.24135700	2.10580100
C	2.45703400	0.54105900	-2.46622200
H	2.03525800	-0.14629100	-3.21660300
H	1.62475000	1.14666900	-2.07469200
H	3.16108800	1.21236500	-2.97657000
C	-2.44520900	0.61020100	-2.42777800
H	-1.69135800	1.28531700	-1.99222600
H	-1.92400600	-0.05400700	-3.13426600
H	-3.16315100	1.21882500	-2.99458000
C	-2.29549400	-2.76522600	1.34202100
H	-1.81157100	-3.54453100	0.73248300
H	-1.49773700	-2.24238700	1.89651900
H	-2.95684000	-3.25838400	2.06751000
C	-6.57117500	-0.27013000	0.33456600
H	-7.20059600	-0.32399100	-0.56777200
H	-6.97898600	-0.97198900	1.07583400
H	-6.67910100	0.74812100	0.74369300
C	6.55423500	-0.29127500	0.36430400
H	6.61502600	0.47633300	1.15451600
H	7.08099100	-1.18293100	0.73607700
H	7.10317200	0.09215700	-0.50813300

Zero-point correction= 0.519741 (Hartree/Particle)
Thermal correction to Energy= 0.553230

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Thermal correction to Enthalpy=                0.554174
Thermal correction to Gibbs Free Energy=       0.450696
Sum of electronic and zero-point Energies=         -2896.454126
Sum of electronic and thermal Energies=            -2896.420638
Sum of electronic and thermal Enthalpies=          -2896.419693
Sum of electronic and thermal Free Energies=       -2896.523171
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P_i

Cu	0.04461200	0.32964600	-1.13507700
C	0.87121900	3.07527500	-1.91220600
H	1.90261300	2.80788100	-1.61786000
H	0.96252500	3.83230100	-2.71663000
C	0.11904000	1.80188500	-2.40063200
H	0.60942600	1.44641600	-3.32999500
H	-0.90105700	2.09467900	-2.72278500
B	0.16310100	3.73475800	-0.68986700
O	0.23485400	3.26534400	0.62001100
O	-0.63069500	4.86366800	-0.77638900
C	-0.45559000	4.18520600	1.46399000
H	0.28022900	4.73485300	2.07748500
H	-1.12505200	3.63558100	2.14458900
C	-1.21532600	5.11042400	0.49801200
H	-1.11022100	6.17654700	0.75456600
H	-2.29289200	4.87299100	0.45386300
C	-0.60889800	-2.66258800	1.67146100
C	0.74793000	-2.60804100	1.69852300
C	0.03860200	-1.08846100	0.15624100
N	-1.02099400	-1.73321400	0.72687000
H	-1.31234800	-3.26793700	2.23385800
H	1.47436000	-3.15560600	2.29012200
N	1.12191200	-1.64693500	0.77086200
C	-2.39696200	-1.46748600	0.40978600
C	-3.03304800	-2.24578600	-0.56890800
C	-3.06697900	-0.44893200	1.10487900
C	-4.38132200	-1.98751400	-0.83333300
C	-4.41433800	-0.23130200	0.80305500
C	-5.08943300	-0.98688700	-0.16068500
H	-4.88986500	-2.58653800	-1.59380500
H	-4.95017800	0.55786500	1.33775400
C	2.48511300	-1.28618600	0.49040600
C	3.19940400	-2.03744200	-0.45841500
C	3.06264300	-0.21028200	1.17935900
C	4.52994000	-1.69027000	-0.69867600
C	4.40085700	0.09402900	0.90193600
C	5.14962300	-0.62825300	-0.02941600
H	5.09721200	-2.26516300	-1.43603000
H	4.86678900	0.92905100	1.43221500
C	-2.28688900	-3.30540200	-1.33075600
H	-1.46564100	-2.85914100	-1.91437200
H	-1.83578800	-4.05562800	-0.66215900
H	-2.95557200	-3.82901900	-2.02722500
C	-2.35438600	0.40127200	2.11884700
H	-1.85841300	-0.20723600	2.89129700
H	-1.57131100	1.00749800	1.63427600

H	-3.05738600	1.08061200	2.62025100
C	2.27639700	0.62165600	2.15335300
H	1.60956700	1.32040800	1.61948700
H	1.64654600	0.00406500	2.81113300
H	2.95060000	1.21698400	2.78493200
C	2.54594500	-3.15936300	-1.21660700
H	2.12979200	-3.92694500	-0.54502400
H	1.71127700	-2.78107600	-1.82877600
H	3.26558400	-3.64875200	-1.88685500
C	6.57893600	-0.26557800	-0.32878400
H	6.98269600	0.43647800	0.41450900
H	7.22582900	-1.15656300	-0.34438400
H	6.66302600	0.21332000	-1.31865800
C	-6.53051000	-0.70614100	-0.49003200
H	-6.60828900	0.06099100	-1.27917000
H	-7.04492000	-1.60626900	-0.85724700
H	-7.07988800	-0.33021100	0.38572800

Zero-point correction= 0.522036 (Hartree/Particle)
Thermal correction to Energy= 0.555899
Thermal correction to Enthalpy= 0.556844
Thermal correction to Gibbs Free Energy= 0.450570
Sum of electronic and zero-point Energies= -2896.504585
Sum of electronic and thermal Energies= -2896.470722
Sum of electronic and thermal Enthalpies= -2896.469778
Sum of electronic and thermal Free Energies= -2896.576052

TS_{PA}

Cu	-0.00599600	-0.37167000	-1.20943900
C	0.06767100	-1.43846300	-2.96424900
H	-0.83856100	-1.93587100	-3.33715700
H	0.97493200	-1.99061200	-3.24386100
C	0.11069100	0.01239700	-3.22369600
H	1.10251500	0.36831500	-3.53015300
P	1.96547400	0.26215600	-0.17984300
P	-1.92660400	0.24914000	-0.11405800
B	-0.00624900	-2.46188600	-1.30638300
O	-1.15355200	-3.24829600	-1.14543800
O	1.13006800	-3.24353500	-1.05558500
C	-0.77285700	-4.59809900	-0.87708400
H	-0.97952400	-5.21678800	-1.76893200
H	-1.36969900	-4.98974800	-0.03884200
C	0.72568500	-4.51899500	-0.56030600
H	1.31174700	-5.30993600	-1.05456400
H	0.92507400	-4.56649600	0.52442600
C	-2.09137100	2.05289400	0.26981600
C	-2.38570700	4.81287200	0.67983500
C	-1.77559800	2.96075800	-0.75055100
C	-2.54588300	2.54196300	1.50091400
C	-2.68430800	3.91620700	1.70585000
C	-1.93482000	4.33119900	-0.55059900
H	-2.79806700	1.84745200	2.30469500
H	-3.03325200	4.28593100	2.67313600
H	-2.49940500	5.88760500	0.84024900
C	2.13140400	2.07822500	0.13518100

C	2.41892500	4.85235000	0.43447900
C	2.64205900	2.61287400	1.32438500
C	1.75479100	2.94693100	-0.89884200
C	1.91074600	4.32469600	-0.75428000
C	2.77688500	3.99459700	1.47478100
H	1.33573400	2.52598700	-1.81692100
H	1.62385100	4.99119300	-1.57106000
H	2.53065100	5.93289000	0.55174800
C	2.31835800	-0.47310100	1.48336500
C	2.57243900	-1.78914700	3.95916800
C	3.54027600	-1.03732200	1.87038500
C	1.24895100	-0.56858400	2.38373000
C	1.34361400	-1.22053600	3.61482000
C	3.66590400	-1.68985900	3.09856500
H	4.39404000	-0.98326700	1.19275100
H	4.62394600	-2.13077000	3.38171200
H	2.67159800	-2.30966400	4.91545100
C	-2.18889300	-0.53460300	1.54577400
C	-2.33223900	-1.87102500	4.01952700
C	-3.38125800	-1.13754200	1.96422000
C	-1.09039200	-0.60512800	2.41281600
C	-1.13197500	-1.26252300	3.64421200
C	-3.45173200	-1.80228300	3.19027700
H	-4.25632500	-1.10180600	1.31343300
H	-4.38750300	-2.27400400	3.49715200
H	-2.38852800	-2.39742500	4.97607100
C	-3.53990900	-0.09932700	-0.96197500
C	-5.93609500	-0.70629100	-2.29892900
C	-3.72677700	-1.36110300	-1.55020300
C	-4.56566700	0.85244200	-1.05352500
C	-5.75428400	0.54973200	-1.72067900
C	-4.92056700	-1.65977700	-2.20756400
H	-2.93876800	-2.11485200	-1.48365700
H	-4.43832100	1.83979000	-0.60707200
H	-6.54185400	1.30433200	-1.78674000
H	-5.05305800	-2.64644700	-2.65824300
H	-6.86621500	-0.94089800	-2.82213600
C	3.52969100	-0.10130400	-1.10811400
C	5.84107100	-0.73678500	-2.57445000
C	4.52936200	0.85996800	-1.31392300
C	3.70011300	-1.38771500	-1.64611800
C	4.85179100	-1.70012200	-2.36876700
C	5.67599300	0.54281800	-2.04491400
H	4.41412300	1.86508200	-0.90540700
H	2.93205700	-2.14879400	-1.48838900
H	4.97215500	-2.70557100	-2.77962600
H	6.44410100	1.30457600	-2.19939400
H	6.73803800	-0.98251700	-3.14804600
H	-1.40477200	2.58046500	-1.70528500
H	2.93995700	1.94774200	2.13753100
H	-1.69482600	5.02779700	-1.35717600
H	3.16992700	4.40087700	2.40996100
O	0.06452600	0.01524200	1.99903900
C	0.11645100	-1.25556800	4.49183300

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H               0.13916600   -2.13060300    5.15893600
H               0.10868100   -0.36416600    5.14869700
C              -1.02364700    0.63448000   -4.01011300
H              -2.01078600    0.32924900   -3.62537800
H              -0.99209200    1.73670200   -3.97147600
H              -1.00034900    0.36217200   -5.08669700
Zero-point correction=                          0.691422 (Hartree/Particle)
Thermal correction to Energy=                    0.735785
Thermal correction to Enthalpy=                  0.736729
Thermal correction to Gibbs Free Energy=         0.611320
Sum of electronic and zero-point Energies=          -4195.884897
Sum of electronic and thermal Energies=             -4195.840534
Sum of electronic and thermal Enthalpies=           -4195.839590
Sum of electronic and thermal Free Energies=        -4195.964998
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TS_{PB}

```
Cu             -0.01520600   -0.60292100   -1.11875100
C               0.11254000   -2.05158100   -2.62818200
H               1.11443100   -2.49326300   -2.71604100
C               0.07402900   -0.66341600   -3.13821900
H              -0.83091600   -0.39444500   -3.69602200
H               0.99574900   -0.30336400   -3.60692600
P               1.96635400    0.21323500   -0.23713400
P              -1.93115300    0.28062100   -0.20322000
B              -0.00054000   -2.66705900   -0.74790700
O              -1.15513600   -3.26927100   -0.23952000
O               1.11296900   -3.44858500   -0.42046200
C              -0.81449300   -4.52501500    0.35429300
H              -1.16206000   -5.34264400   -0.30216800
H              -1.32220100   -4.62078800    1.32610300
C               0.71249400   -4.48707300    0.47408600
H               1.19261900   -5.43276300    0.17805100
H               1.04401500   -4.23268500    1.49577700
C              -2.05915000    2.12121600   -0.35918900
C              -2.32009700    4.88366600   -0.77223000
C              -1.71705800    2.68982700   -1.59420500
C              -2.52253900    2.95083200    0.66961900
C              -2.64429500    4.32662000    0.46506800
C              -1.85945600    4.06108500   -1.80209300
H              -2.79542900    2.52146300    1.63581100
H              -3.00070600    4.96462400    1.27746800
H              -2.42152100    5.95964800   -0.93274200
C               2.15985700    2.05006100   -0.36948900
C               2.51234300    4.80736100   -0.74884500
C               2.70121900    2.84242100    0.65088200
C               1.78573800    2.65404100   -1.57801100
C               1.97298200    4.02234900   -1.76958700
C               2.86925300    4.21567200    0.46319700
H               1.34499600    2.03671200   -2.36529600
H               1.68638200    4.48020600   -2.71920800
H               2.64996400    5.88117700   -0.89642800
C               2.28836500   -0.09129600    1.56170700
C               2.50182700   -0.71784300    4.29877300
C               3.48857600   -0.58155300    2.09137000
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C	1.21931700	0.09334600	2.44848900
C	1.29565000	-0.21096300	3.80912800
C	3.59367100	-0.89388600	3.44833600
H	4.34137900	-0.73809100	1.42880800
H	4.53485200	-1.28236500	3.84296900
H	2.58517800	-0.96964800	5.35934600
C	-2.21403300	0.00895100	1.61015700
C	-2.39544200	-0.59836700	4.35521400
C	-3.42380100	-0.42038000	2.17070500
C	-1.11845400	0.14691700	2.47267800
C	-1.17836100	-0.15132900	3.83595000
C	-3.51375200	-0.72212200	3.53088300
H	-4.29871900	-0.53910000	1.52980700
H	-4.46374800	-1.06292900	3.94774700
H	-2.46584600	-0.84404100	5.41816400
C	-3.55962300	-0.25756400	-0.91575400
C	-5.98958000	-1.16961700	-1.99440400
C	-3.79532400	-1.63251400	-1.07768900
C	-4.55336400	0.65242400	-1.30314900
C	-5.75853200	0.19716600	-1.84221100
C	-5.00545700	-2.08120900	-1.60754700
H	-3.02561000	-2.34994400	-0.78387800
H	-4.39012200	1.72500500	-1.18814400
H	-6.52071400	0.92007700	-2.14312400
H	-5.17565900	-3.15422300	-1.72532000
H	-6.93309600	-1.52401100	-2.41644800
C	3.54501000	-0.37674200	-1.01560100
C	5.88763000	-1.36176700	-2.21802200
C	4.55613600	0.50106800	-1.43232800
C	3.71914900	-1.75665000	-1.21217700
C	4.88606900	-2.24139300	-1.80374300
C	5.71769100	0.00987000	-2.03163100
H	4.44000000	1.57689600	-1.29329800
H	2.94010500	-2.45436700	-0.89641100
H	5.00763300	-3.31782700	-1.94768500
H	6.49400500	0.70842800	-2.35325400
H	6.79688900	-1.74423900	-2.68799800
H	-1.33674500	2.04249400	-2.38910000
H	2.99890200	2.38601900	1.59714400
H	-1.59970700	4.49145500	-2.77215100
H	3.28743100	4.82427100	1.26870100
O	0.05577600	0.60101100	1.91897400
C	0.07323900	0.04301700	4.65612700
H	0.06664100	-0.61324100	5.53954000
H	0.10281900	1.08134500	5.03967800
C	-0.95871100	-2.98432100	-3.19414400
H	-0.77308700	-3.10727000	-4.27467200
H	-0.96548800	-3.98572000	-2.73610800
H	-1.96341400	-2.55380300	-3.07528600

Zero-point correction= 0.691847 (Hartree/Particle)
Thermal correction to Energy= 0.735892
Thermal correction to Enthalpy= 0.736836
Thermal correction to Gibbs Free Energy= 0.612462
Sum of electronic and zero-point Energies= -4195.882519

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Sum of electronic and thermal Energies=           -4195.838474
Sum of electronic and thermal Enthalpies=         -4195.837529
Sum of electronic and thermal Free Energies=      -4195.961904
```
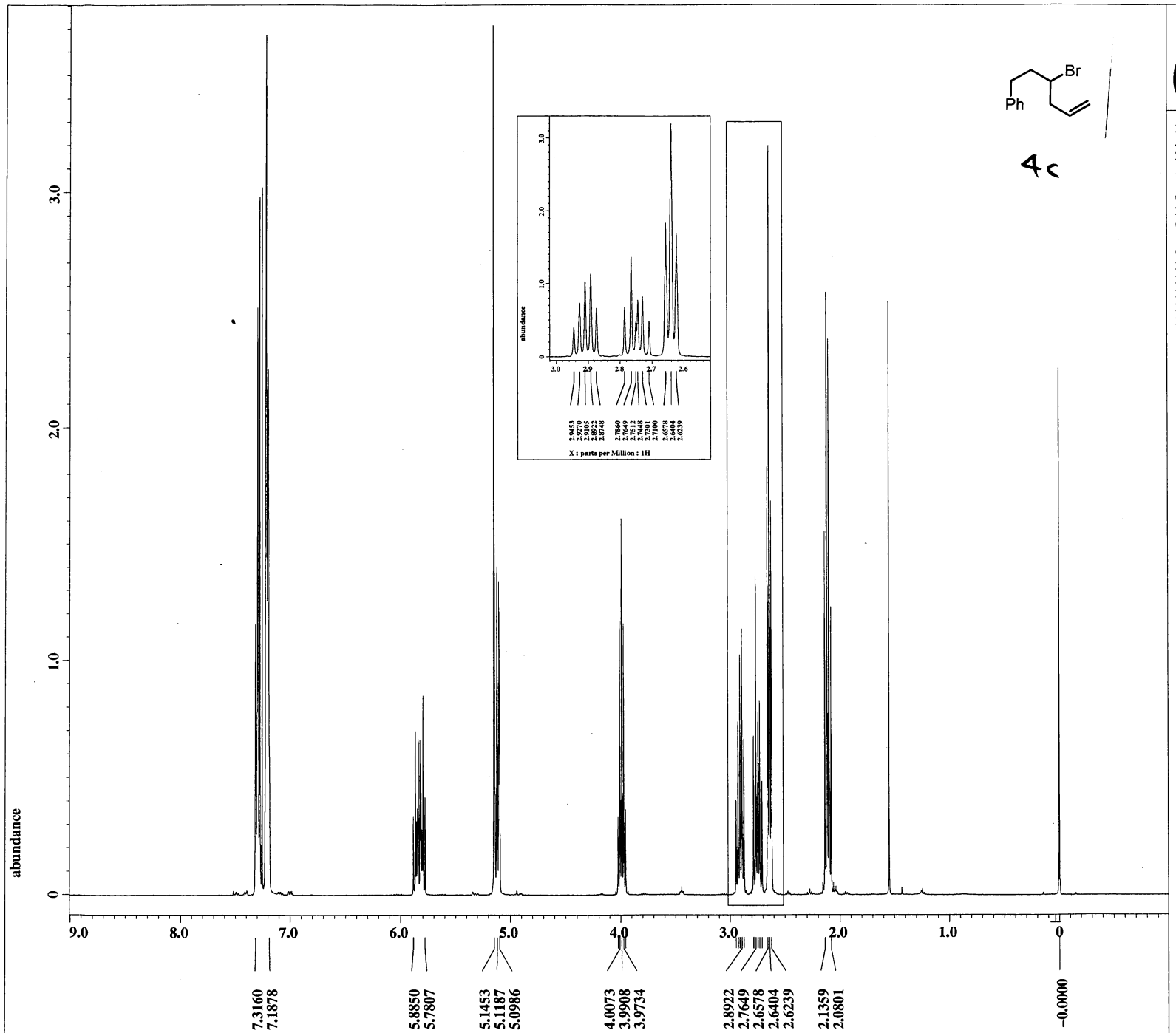

7. References

1. Kozlov, M. V.; Zhu, J.; Philipp, P.; Francke, W.; Zvereva, E. L.; Hansson, B. S.; Lofstedt, C. *J. of Chem. Ecol.* **1996**, *22*, 431.
2. Kabalka, G. W.; Shoup, T.M.; Goudgaon, N. M. *J. Org. Chem.* **1989**, *54*, 5930.
3. Sadhu, K. M.; Matteson, D. S. *Organometallics* **1985**, *4*, 1687.
4. Hupe, E.; Marek, I.; Knochel, P. *Org*. *Lett*. **2002**, *4*, 2861.
5. Yang, C.; Zhang, Z.; Tajuddin, H.; Wu, C.; Liang, J.; Liu, J.; Fu, Y.; Czyzewska, M.; Steel, P.; Marder, T.; Liu, L. *Angew*. *Chem*., *Int. Ed*. **2012**, *51*, 528.
6. Bernstein, P.; Brown, D.; Griffin, A.; Tremblay, M. C.; Wesolowski, S. U. S. Patent US 2010/0113465 A1, May 27, **2010**.
7. Takahashi, H.; Yoshioka, M.; Shibasaki, M.; Ohno, M.; Imai, N.; Kobayashi, S. *Tetrahedron*. **1995**, *51*, 12013.
8. Gaussian 09, Revision C.01, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K. N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, J. M.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, 2009.



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Solvent	= CHLOROFORM-D
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Revision_time	= 21-SEP-2012 18:18:55
Current_time	= 21-SEP-2012 18:19:22
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Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 44
Relaxation_delay	= 5[s]
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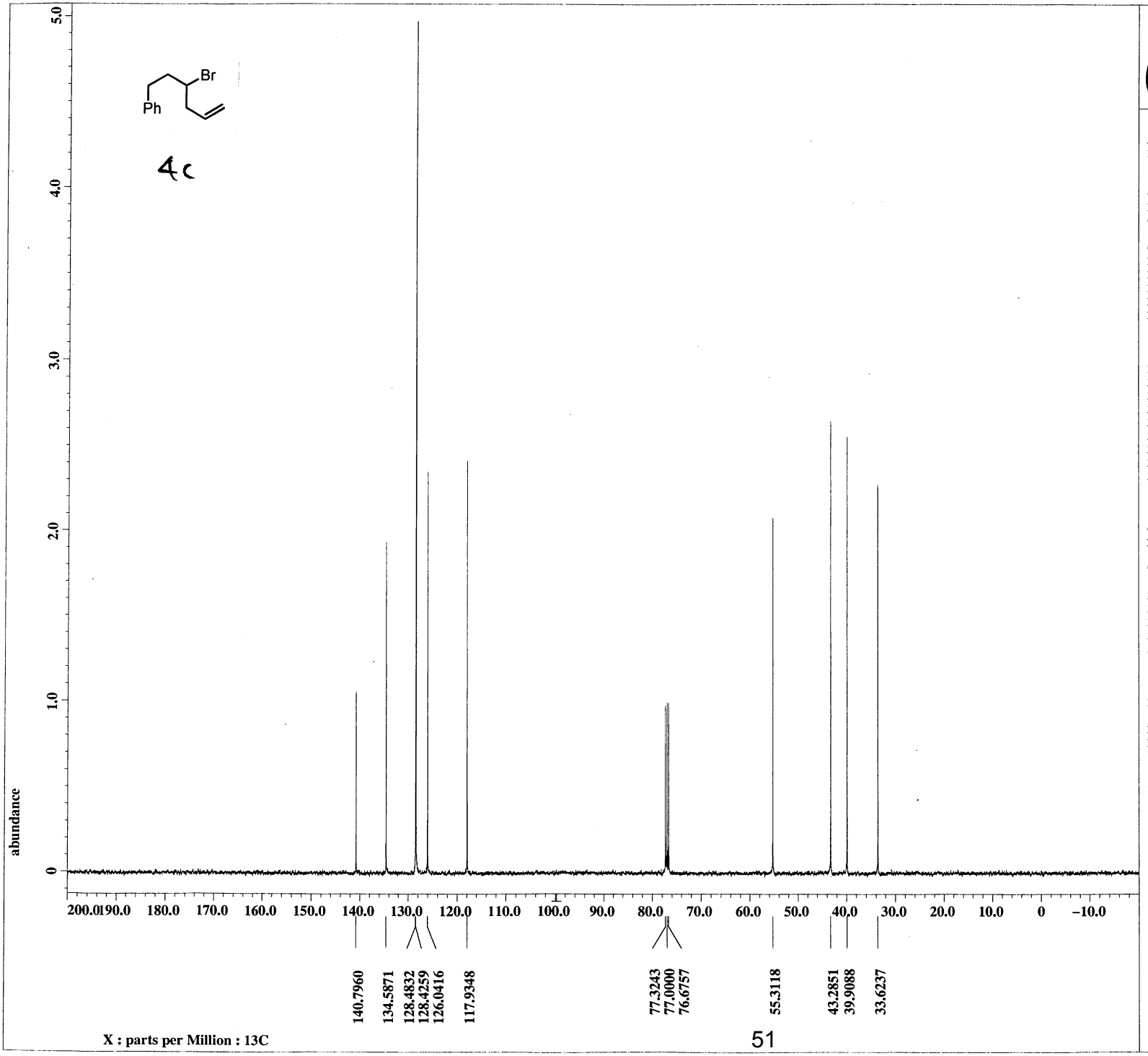
Temp_get	= 17.7[dC]



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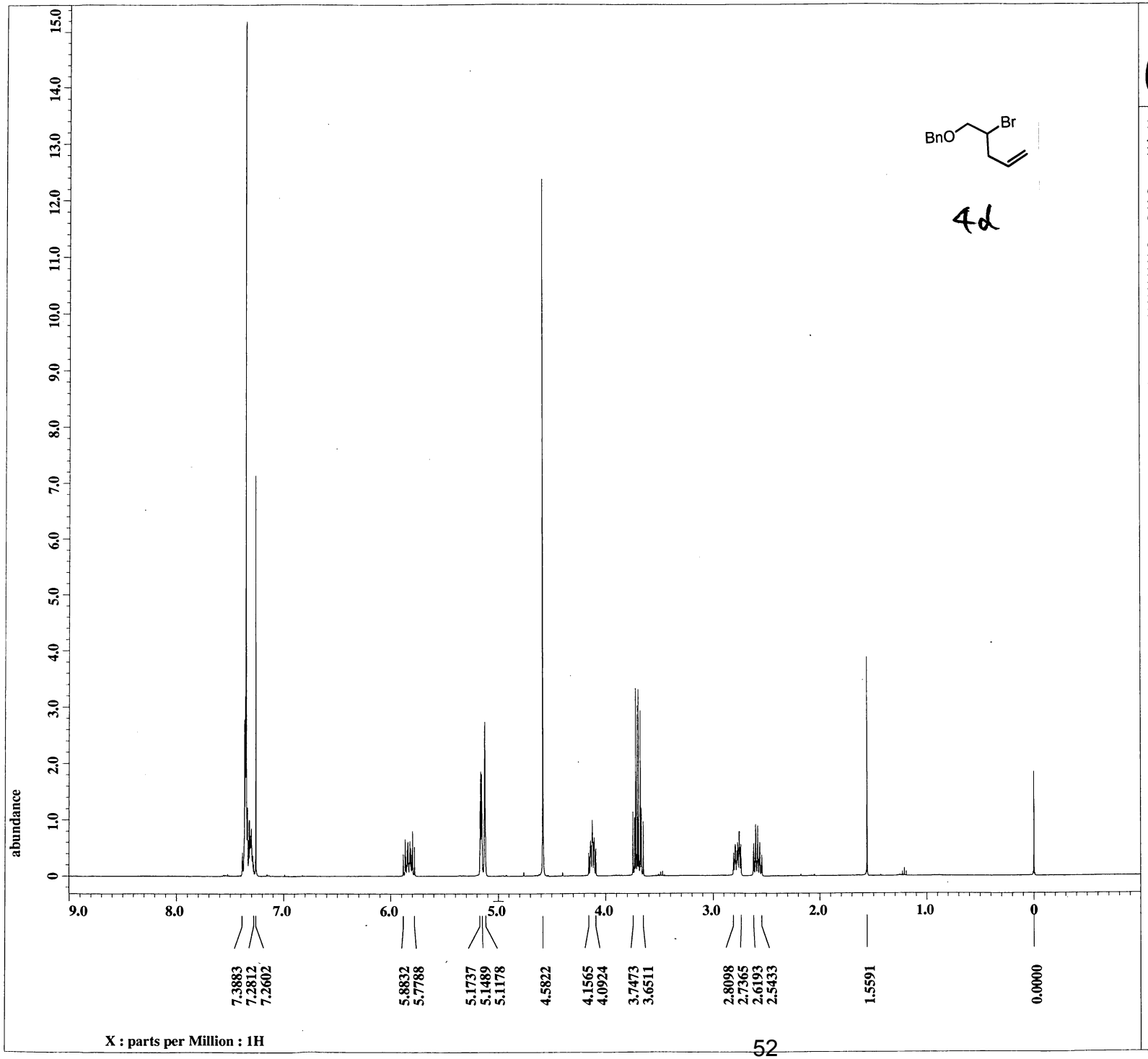
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 100
Total_scans	= 100
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 18.3[dC]



4d

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Dim_title	= 1H
Dim_units	= [ppm]
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Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
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X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
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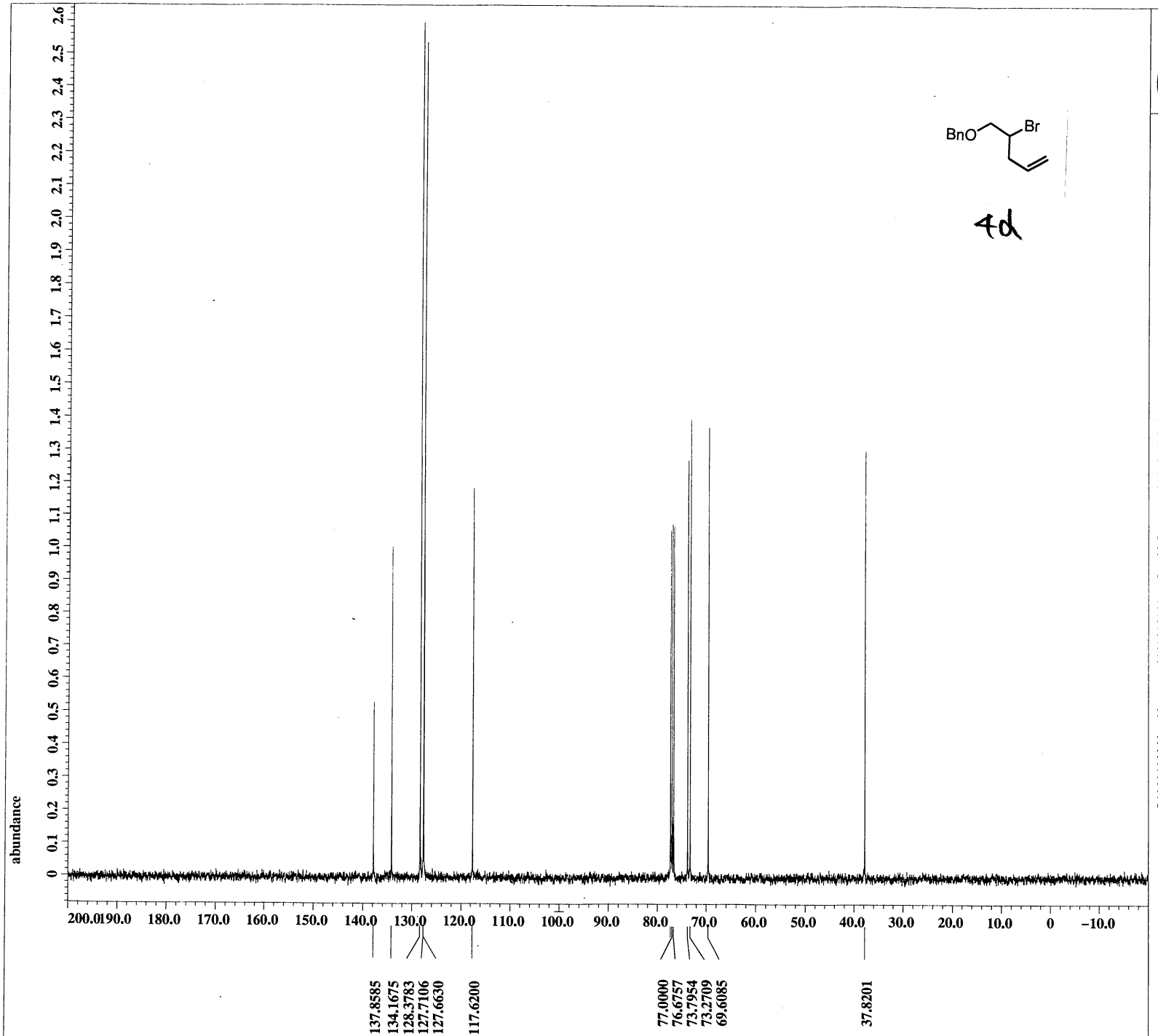
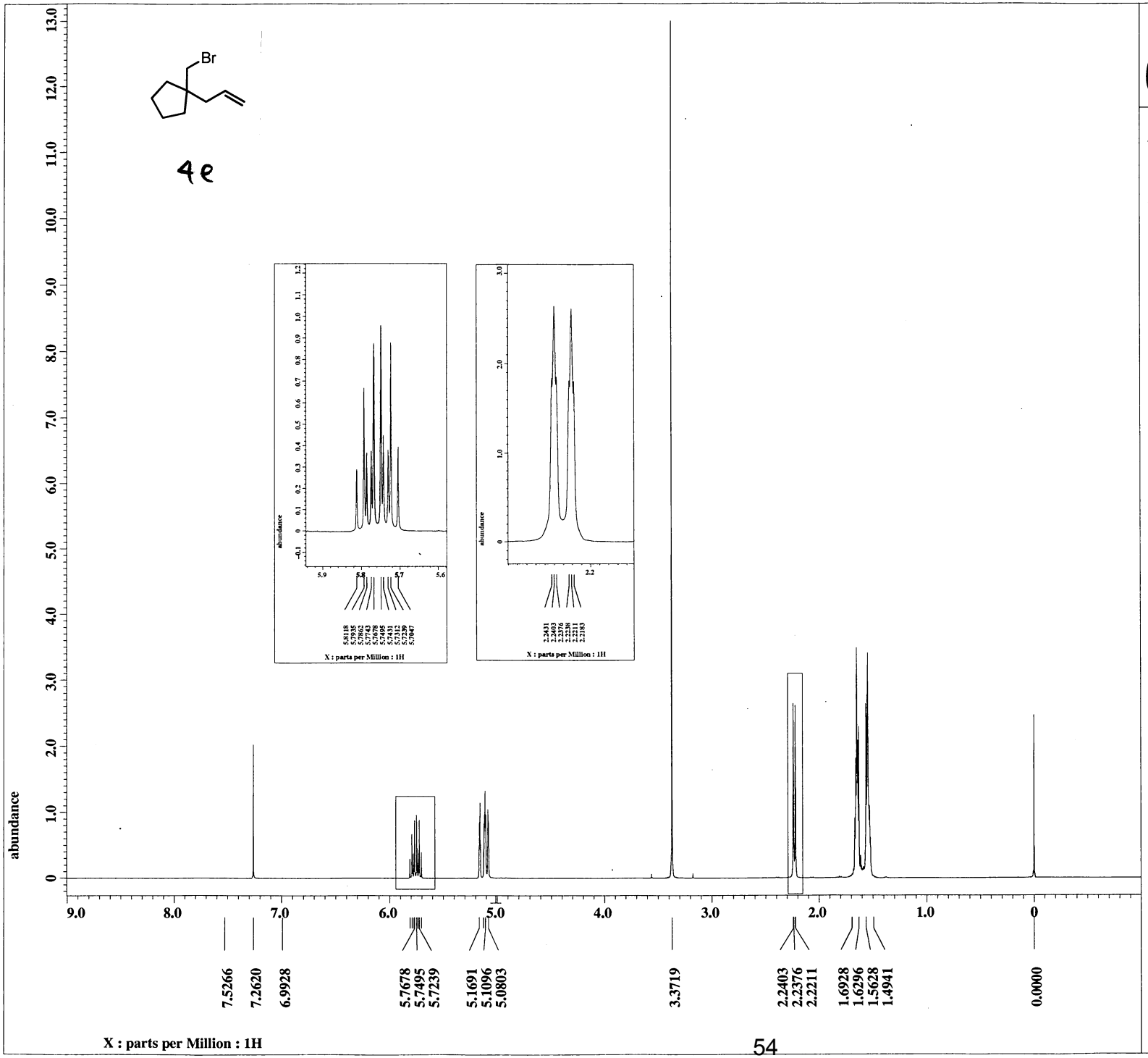
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 50
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 17.1[dC]



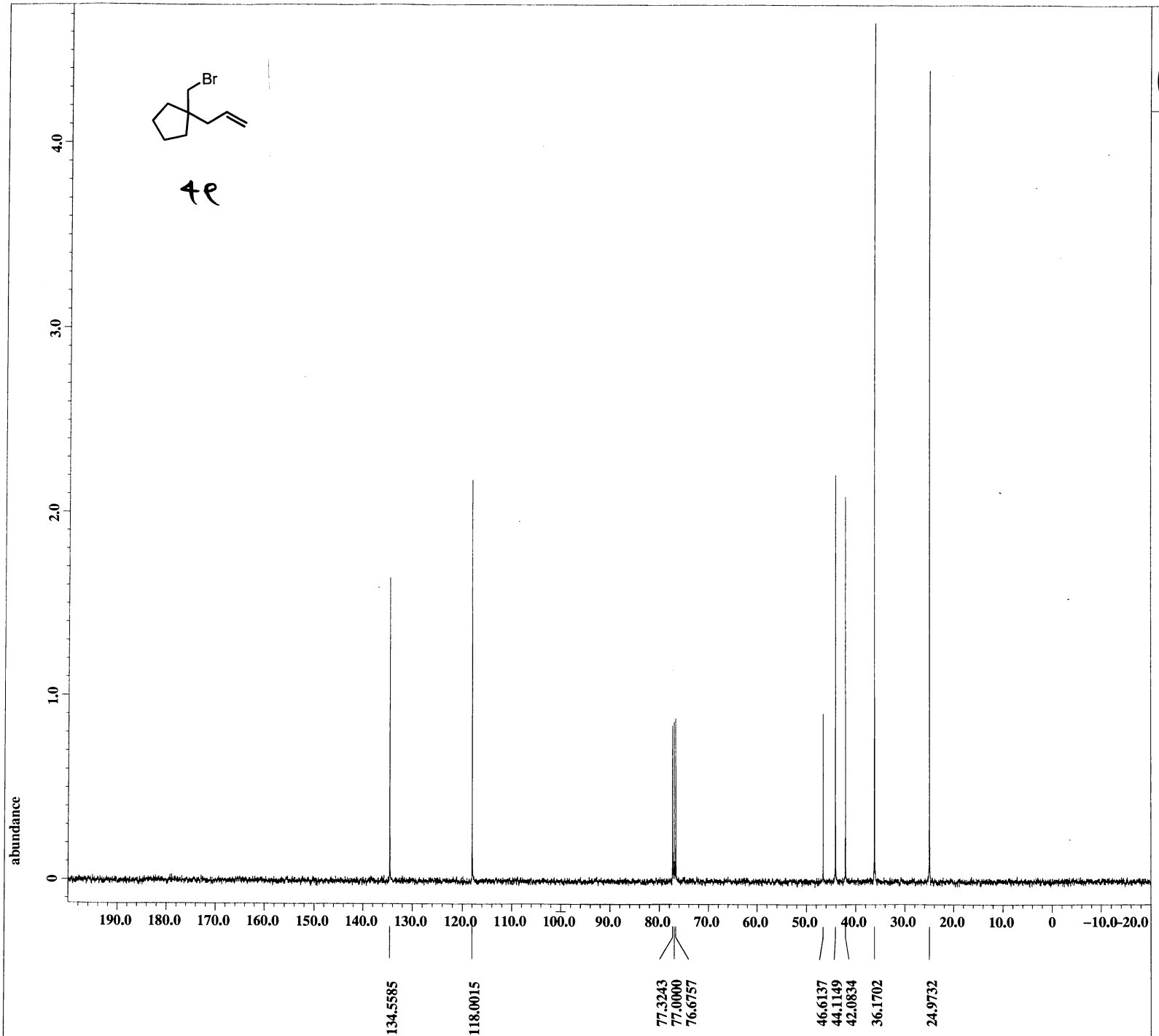
JEOL

Filename	= KUBO-355-13C-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#472677
Solvent	= CHLOROFORM-D
Creation_time	= 6-FEB-2012 12:19:31
Revision_time	= 20-AUG-2012 20:53:36
Current_time	= 20-AUG-2012 20:53:45
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 18.1[dC]



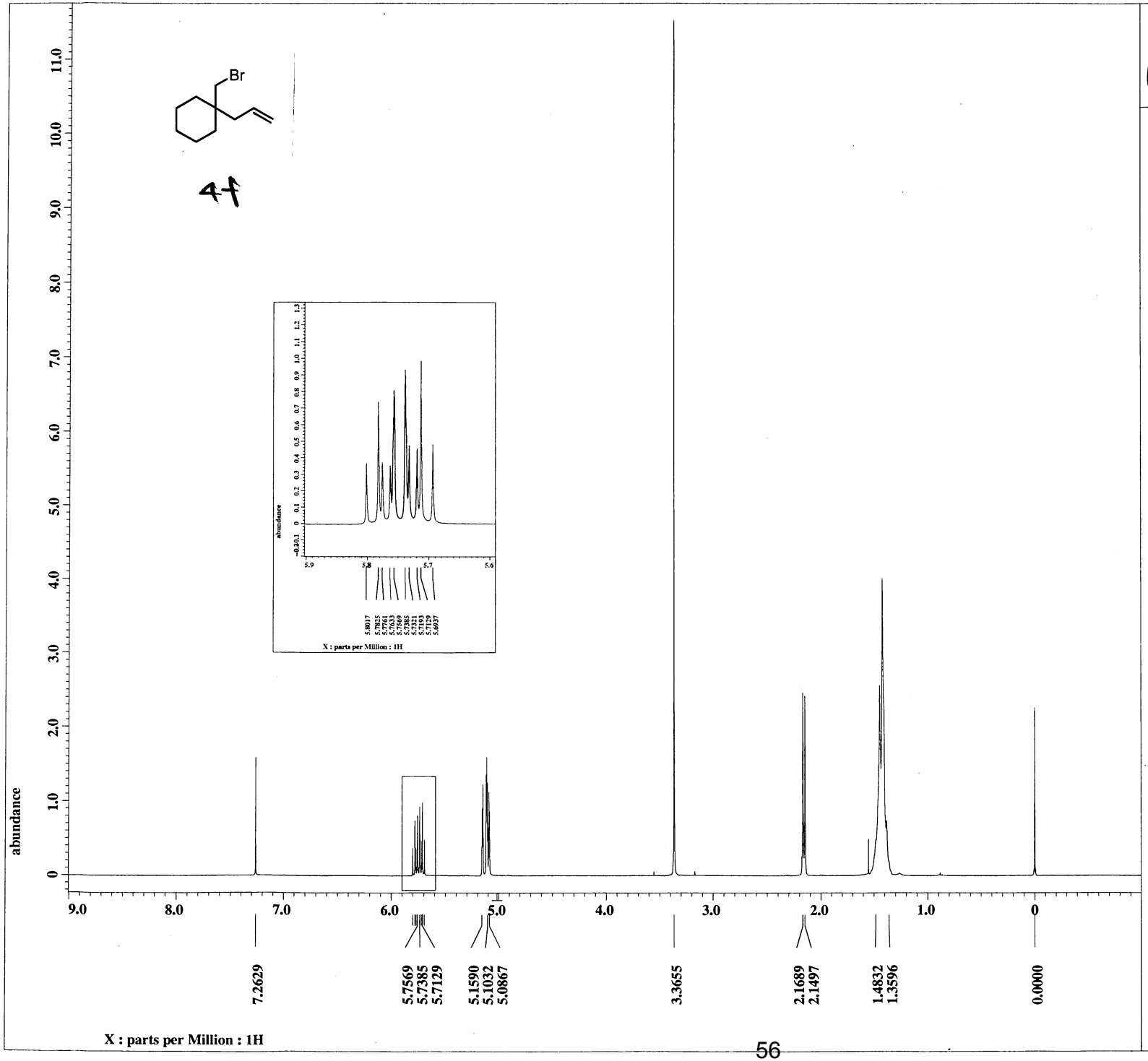
JEOL

Filename	= KUBO-444-gpc-7.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#56206
Solvent	= CHLOROFORM-D
Creation_time	= 23-APR-2012 00:32:42
Revision_time	= 21-SEP-2012 18:24:48
Current_time	= 21-SEP-2012 18:25:49
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.1[dC]



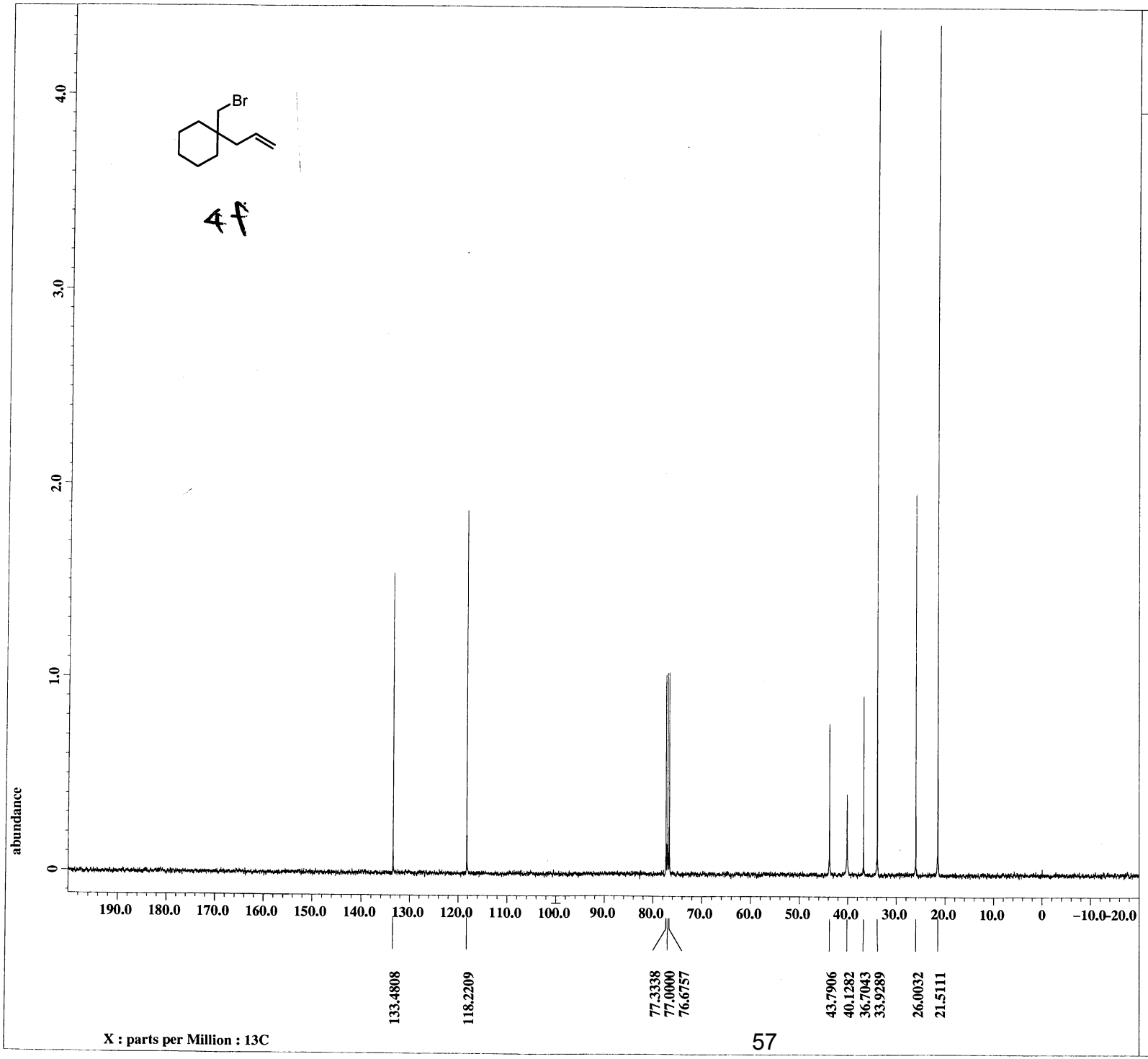
JEOL

Filename	= KUBO-444-13c-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#660287
Solvent	= CHLOROFORM-D
Creation_time	= 28-APR-2012 17:19:27
Revision_time	= 20-AUG-2012 20:50:00
Current_time	= 20-AUG-2012 20:50:12
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 50
Total_scans	= 50
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.9[dC]



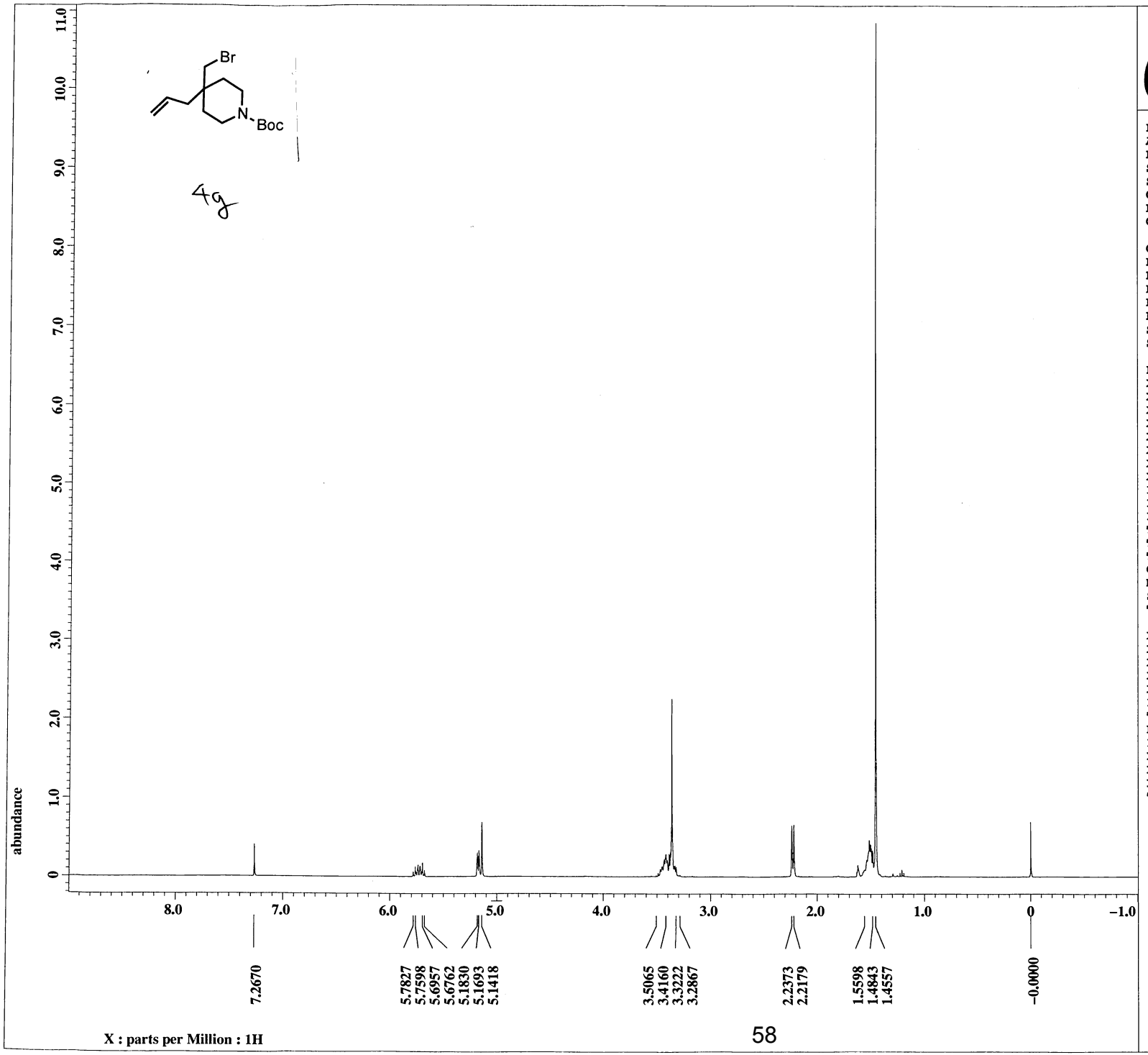
JEOL

```
Filename          = KUBO-400-gpc-5.jdf
Author            = element
Experiment        = single_pulse.ex2
Sample_id         = S#411597
Solvent           = CHLOROFORM-D
Creation_time     =  5-MAR-2012 10:31:39
Revision_time     = 21-SEP-2012 18:30:51
Current_time      = 21-SEP-2012 18:30:55

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.78790144[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.35869274[Hz]
X_sweep           = 5.87682181[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.8[us]
X_acq_time        = 2.78790144[s]
X_angle           = 45[deg]
X_atn             = 1.9[dB]
X_pulse           = 5.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 38
Relaxation_delay  = 5[s]
Repetition_time   = 7.78790144[s]
Temp_get          = 17.7[dC]
```



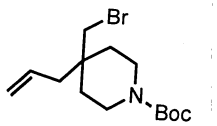
JEOL

Filename	= KUBO-400-13C-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#476754
Solvent	= CHLOROFORM-D
Creation_time	= 5-MAR-2012 12:23:30
Revision_time	= 20-AUG-2012 20:42:28
Current_time	= 20-AUG-2012 20:42:37
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 100
Total_scans	= 100
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 18.1[dC]



JEOL

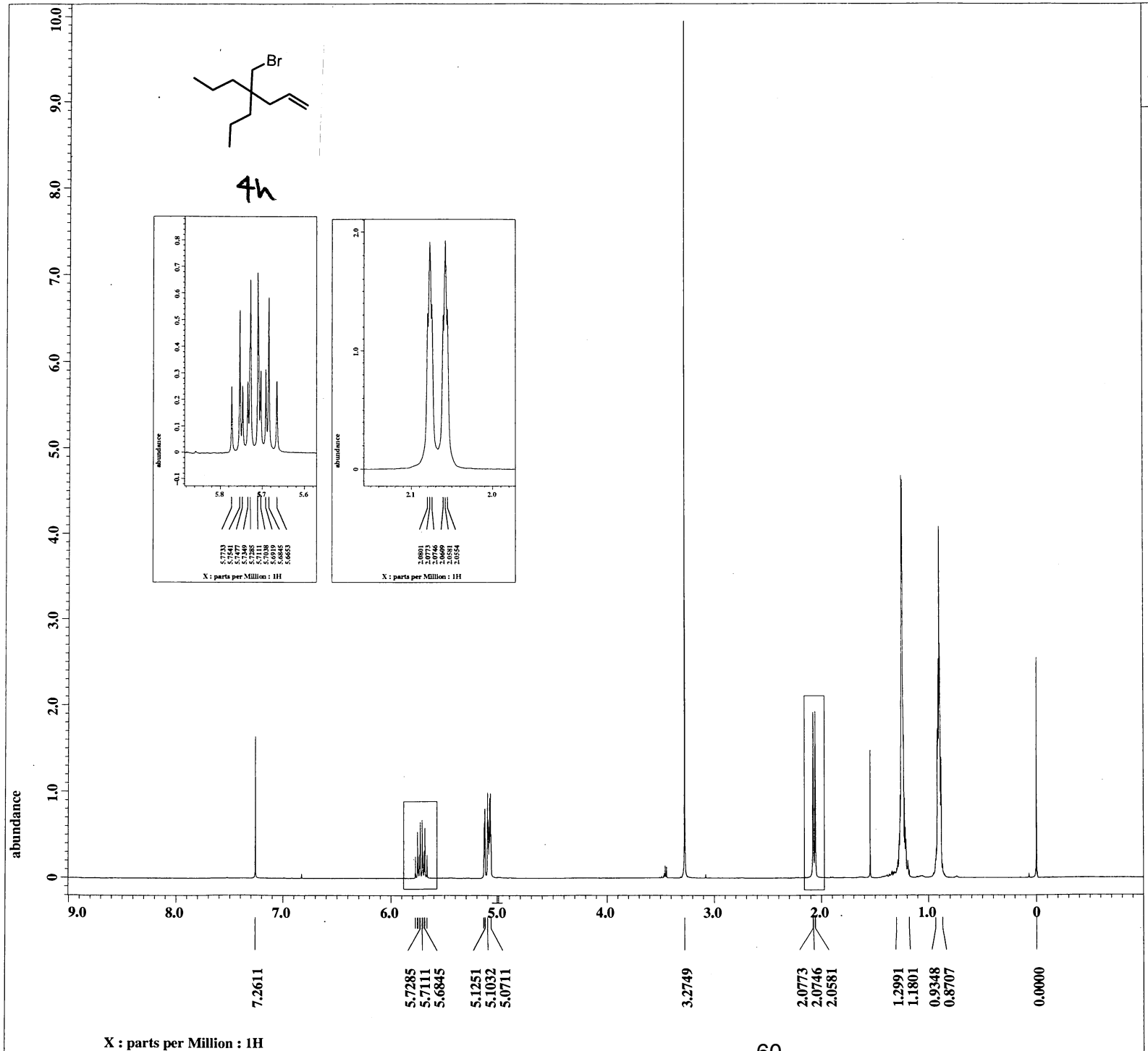
```
Filename          = KUBO-735-pure-2-5.jdf
Author            = element
Experiment        = single_pulse.ex2
Sample_id         = S#536314
Solvent           = CHLOROFORM-D
Creation_time     = 27-NOV-2012 14:26:38
Revision_time     = 13-JAN-2013 18:23:41
Current_time      = 13-JAN-2013 18:23:43

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 13107
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 2.20725248[s]
X_domain          = 1H
X_freq            = 395.88430144[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.45305193[Hz]
X_sweep           = 7.42280285[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 395.88430144[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 13.5[us]
X_acq_time        = 2.20725248[s]
X_angle           = 45[deg]
X_atn             = 1[dB]
X_pulse           = 6.75[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 34
Relaxation_delay  = 5[s]
Repetition_time   = 7.20725248[s]
Temp_get          = 402.2[dC]
```



4g

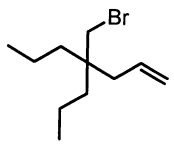
JEOL

Filename	= KUBO-735-13c-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#549276
Solvent	= CHLOROFORM-D
Creation_time	= 27-NOV-2012 14:51:04
Revision_time	= 13-JAN-2013 18:24:34
Current_time	= 13-JAN-2013 18:24:45
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 1.048576[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95367432[Hz]
X_sweep	= 31.25[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 9.5[us]
X_acq_time	= 1.048576[s]
X_angle	= 30[deg]
X_atn	= 3.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 19.607[dB]
Irr_atn_noe	= 19.607[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.048576[s]
Temp_get	= 402.2[dC]



JEOL

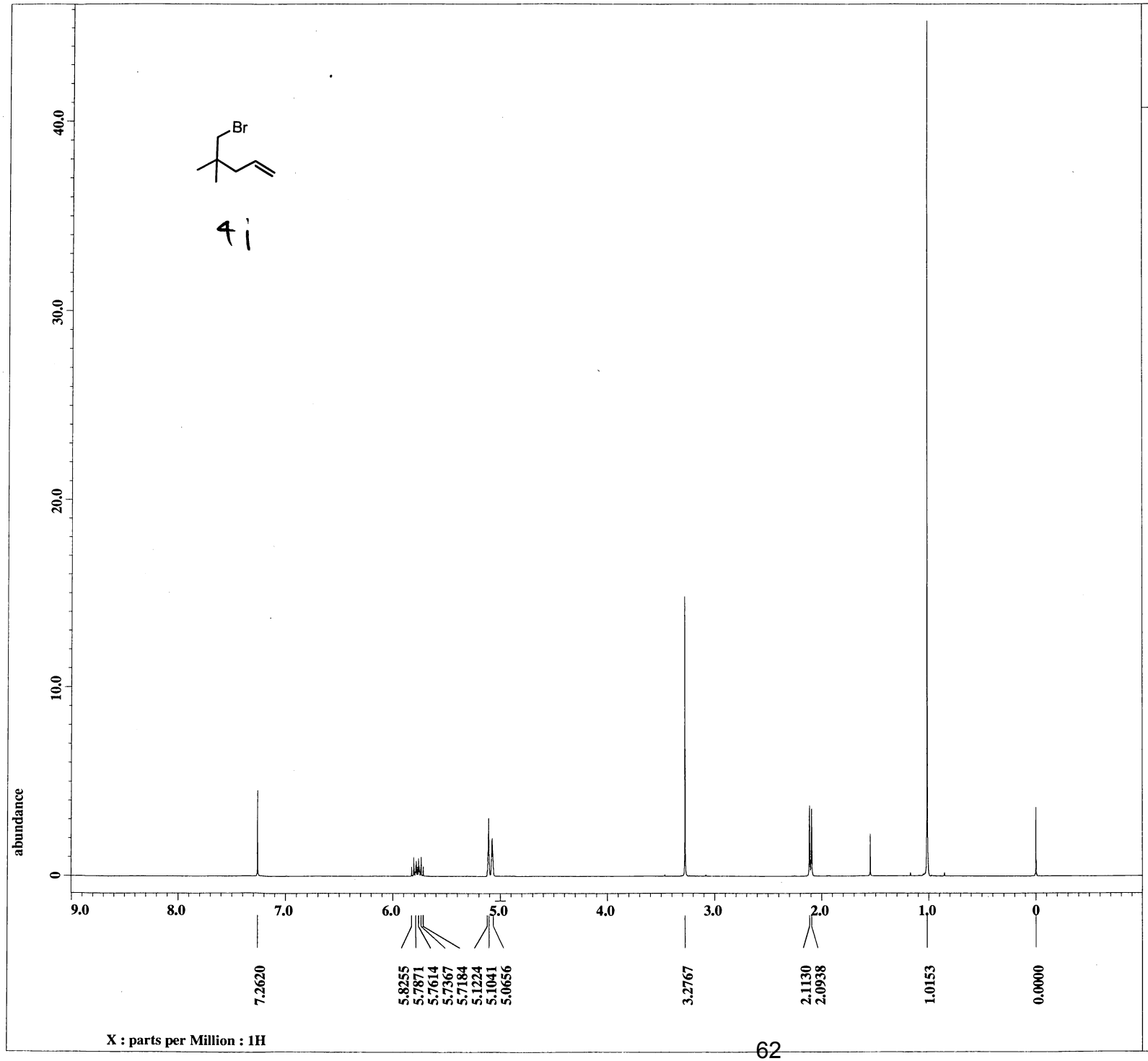
```
Filename          = KUBO-441-pure-distill
Author            = element
Experiment        = single_pulse.ex2
Sample_id         = S#12493
Solvent           = CHLOROFORM-D
Creation_time     = 19-APR-2012 23:20:15
Revision_time     = 21-SEP-2012 18:38:54
Current_time      = 21-SEP-2012 18:40:02

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.78790144[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.35869274[Hz]
X_sweep           = 5.87682181[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.8[us]
X_acq_time        = 2.78790144[s]
X_angle           = 45[deg]
X_atn             = 1.9[dB]
X_pulse           = 5.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 7.78790144[s]
Temp_get          = 20.8[dC]
```



4h

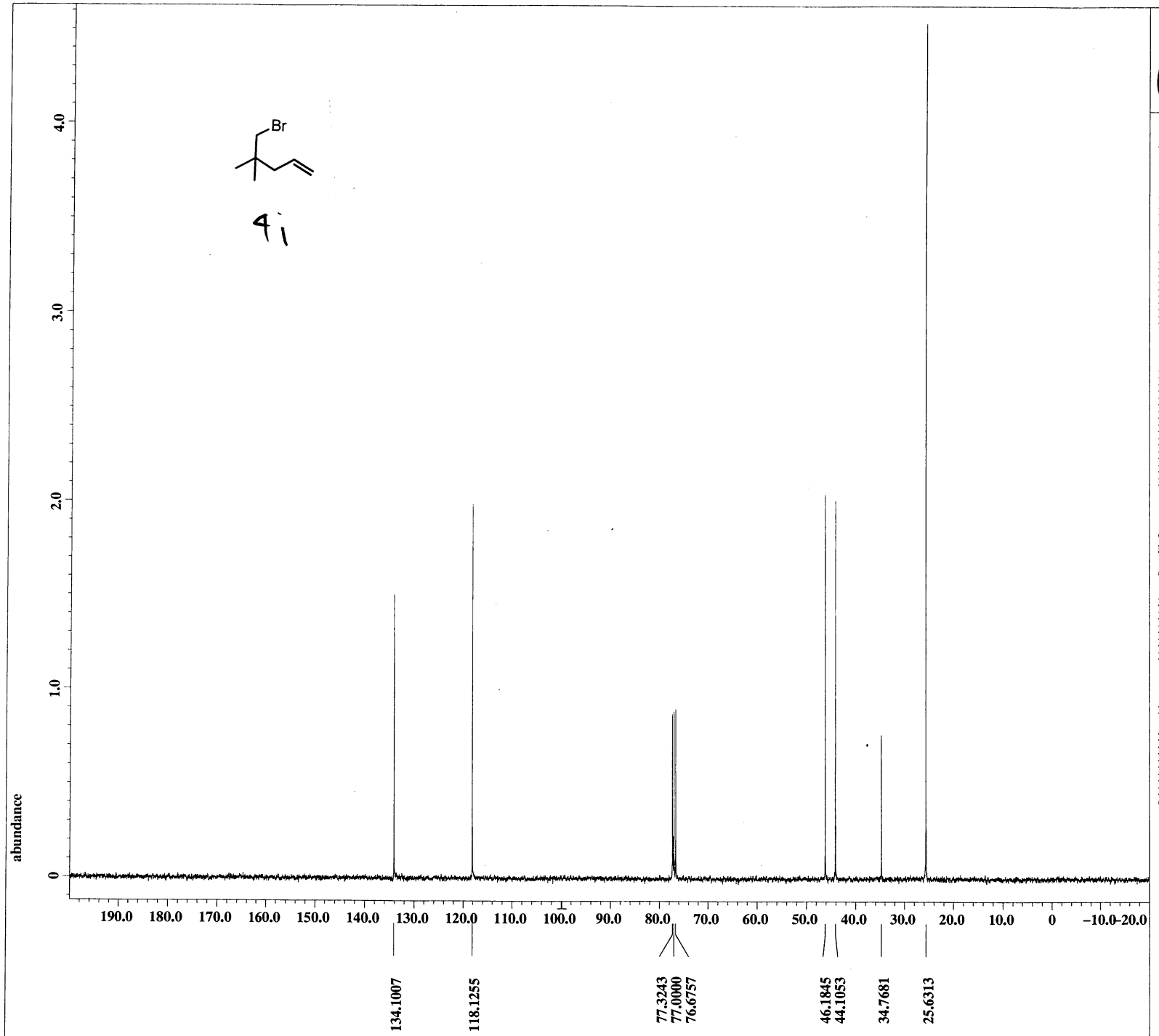
JEOL

Filename	= KUBO-441-13C-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#6067
Solvent	= CHLOROFORM-D
Creation_time	= 26-APR-2012 23:08:48
Revision_time	= 20-AUG-2012 20:45:55
Current_time	= 20-AUG-2012 20:46:08
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 40
Total_scans	= 40
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.9[dC]



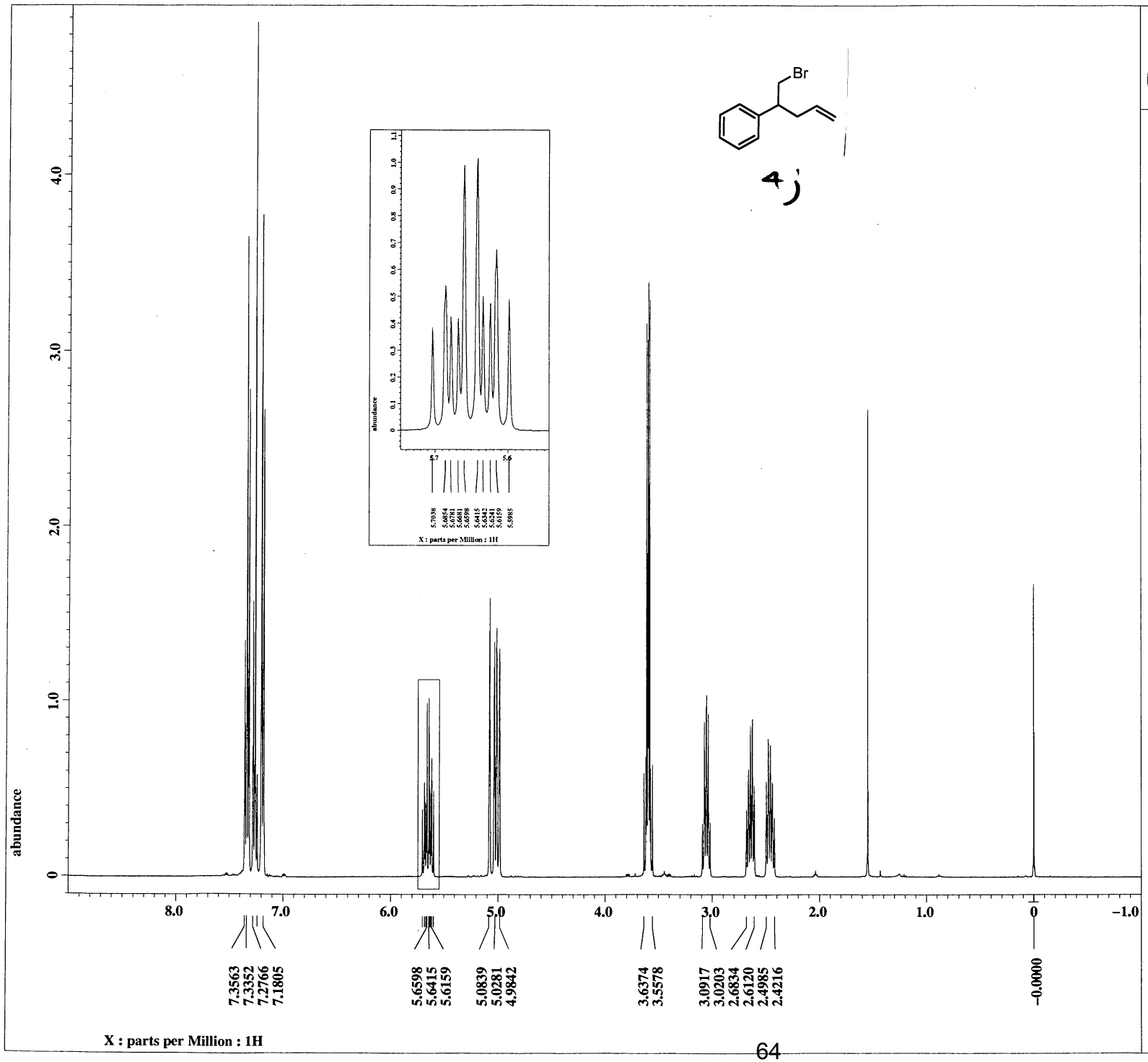
JEOL

```
Filename          = KUBO-431-gpc-2-6.jdf
Author            = element
Experiment        = single_pulse.ex2
Sample_id         = S#20225
Solvent           = CHLOROFORM-D
Creation_time     = 23-APR-2012 23:32:20
Revision_time     = 21-SEP-2012 18:45:01
Current_time      = 21-SEP-2012 18:45:06

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.78790144[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.35869274[Hz]
X_sweep           = 5.87682181[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.8[us]
X_acq_time        = 2.78790144[s]
X_angle           = 45[deg]
X_atn             = 1.9[dB]
X_pulse           = 5.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 44
Relaxation_delay  = 5[s]
Repetition_time   = 7.78790144[s]
Temp_get          = 20.6[dC]
```



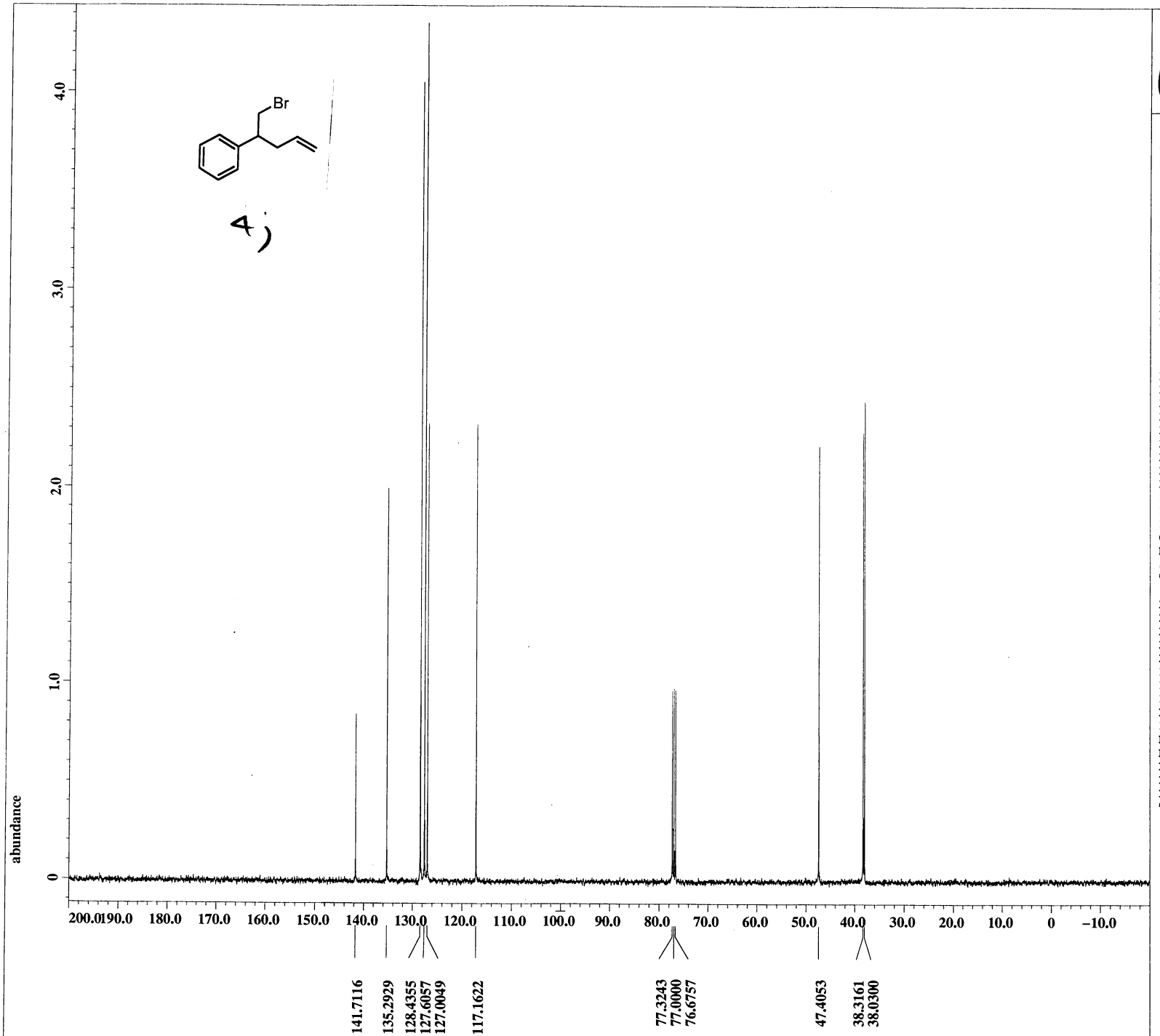
JEOL

Filename	= KUBO-431-13c-7.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#691729
Solvent	= CHLOROFORM-D
Creation_time	= 28-AUG-2012 17:57:25
Revision_time	= 28-AUG-2012 19:56:18
Current_time	= 28-AUG-2012 19:56:27
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 21[dC]



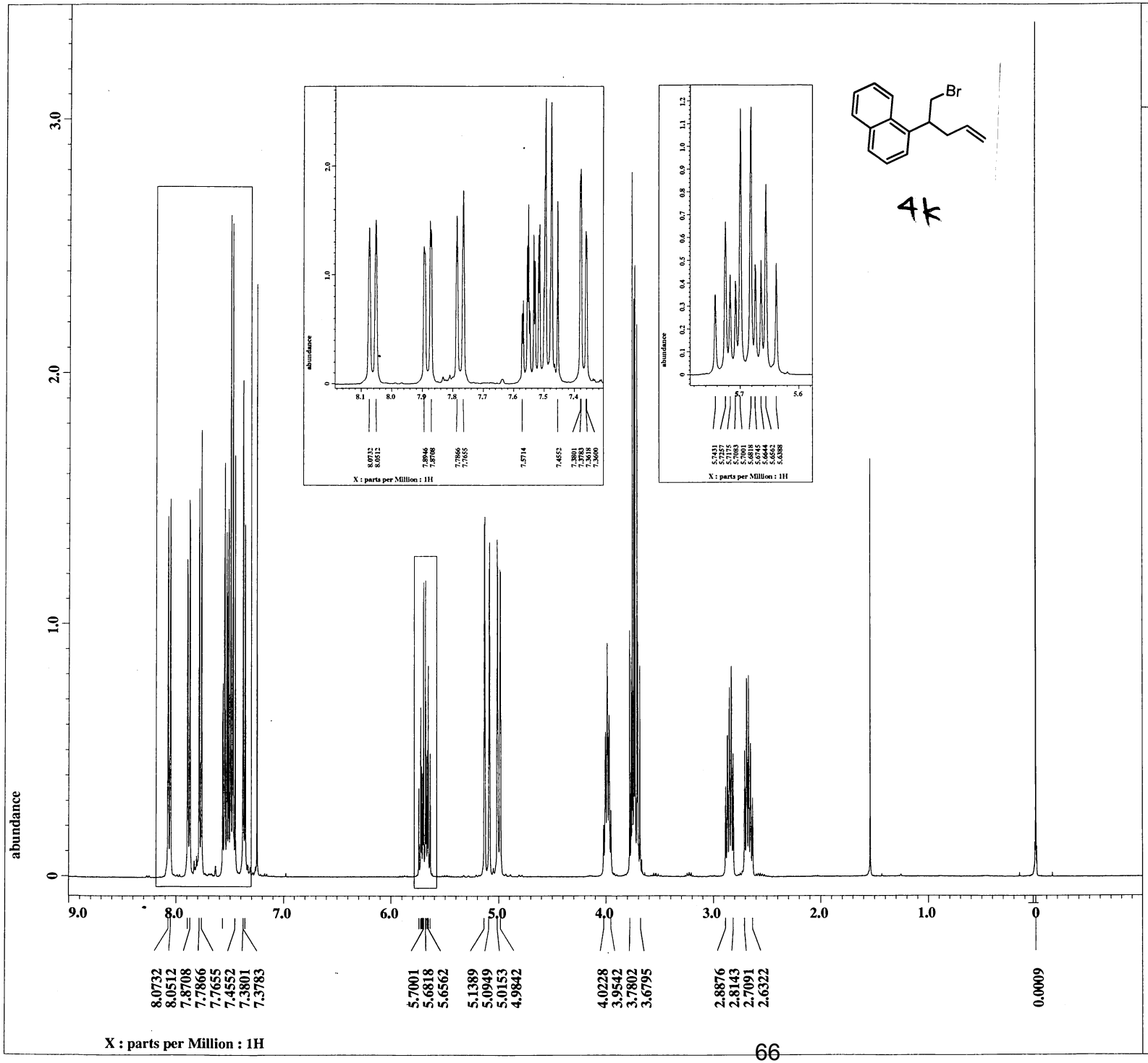
JEOL

Filename	= KUBO-389-pure-2-9.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#464150
Solvent	= CHLOROFORM-D
Creation_time	= 29-FEB-2012 11:59:40
Revision_time	= 21-SEP-2012 18:50:13
Current_time	= 21-SEP-2012 18:50:17
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 46
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 17.4[dC]



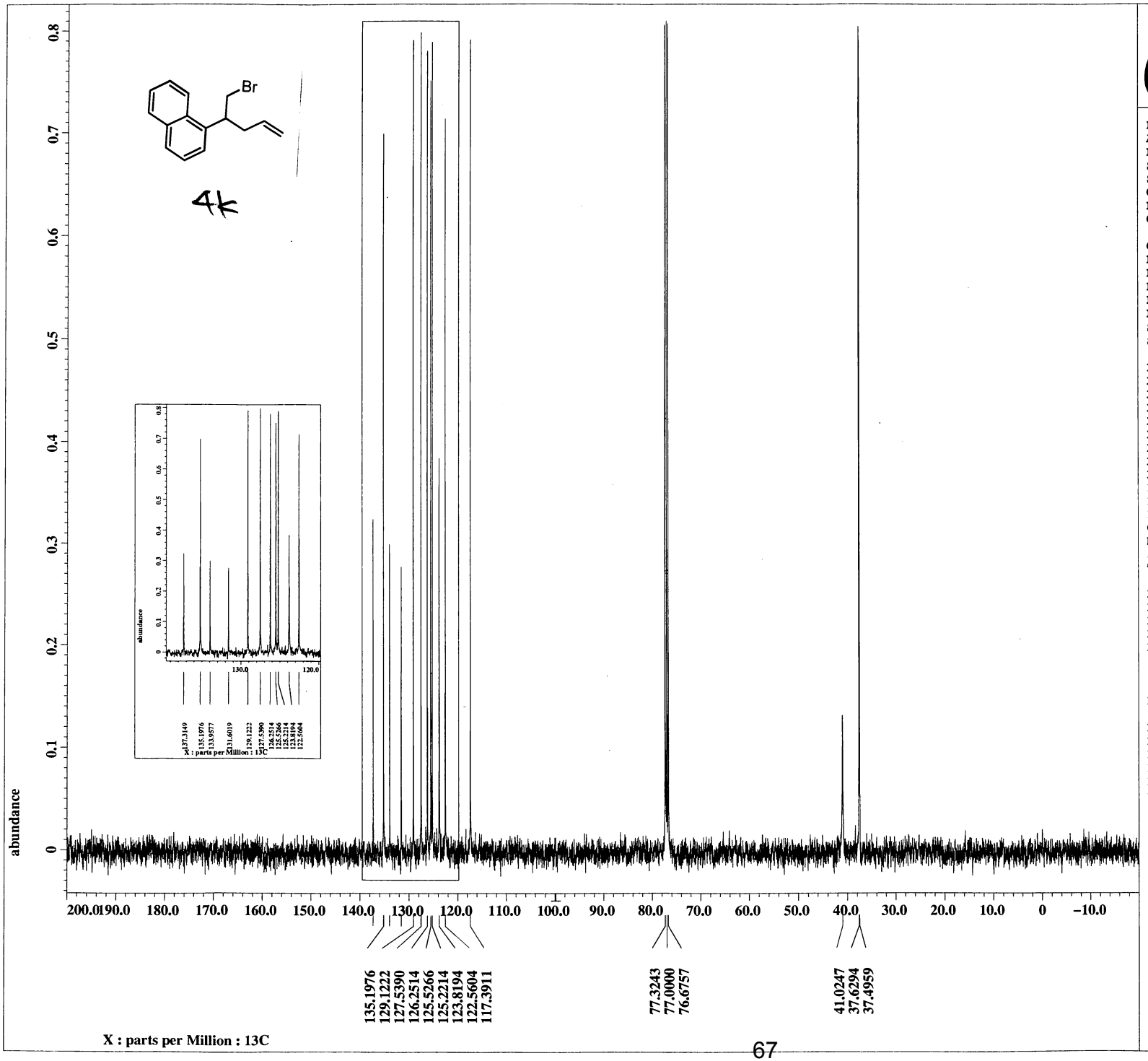
JEOL

Filename	= KUBO-389-13C-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#814293
Solvent	= CHLOROFORM-D
Creation_time	= 9-MAR-2012 21:44:27
Revision_time	= 20-AUG-2012 21:07:52
Current_time	= 20-AUG-2012 21:08:02
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 18.5[dC]



4k

Filename	= KUBO-405-pure-3-13.jd
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#391159
Solvent	= CHLOROFORM-D
Creation_time	= 7-MAR-2012 09:57:04
Revision_time	= 21-SEP-2012 18:56:08
Current_time	= 21-SEP-2012 18:58:03
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 44
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 17.8[dC]

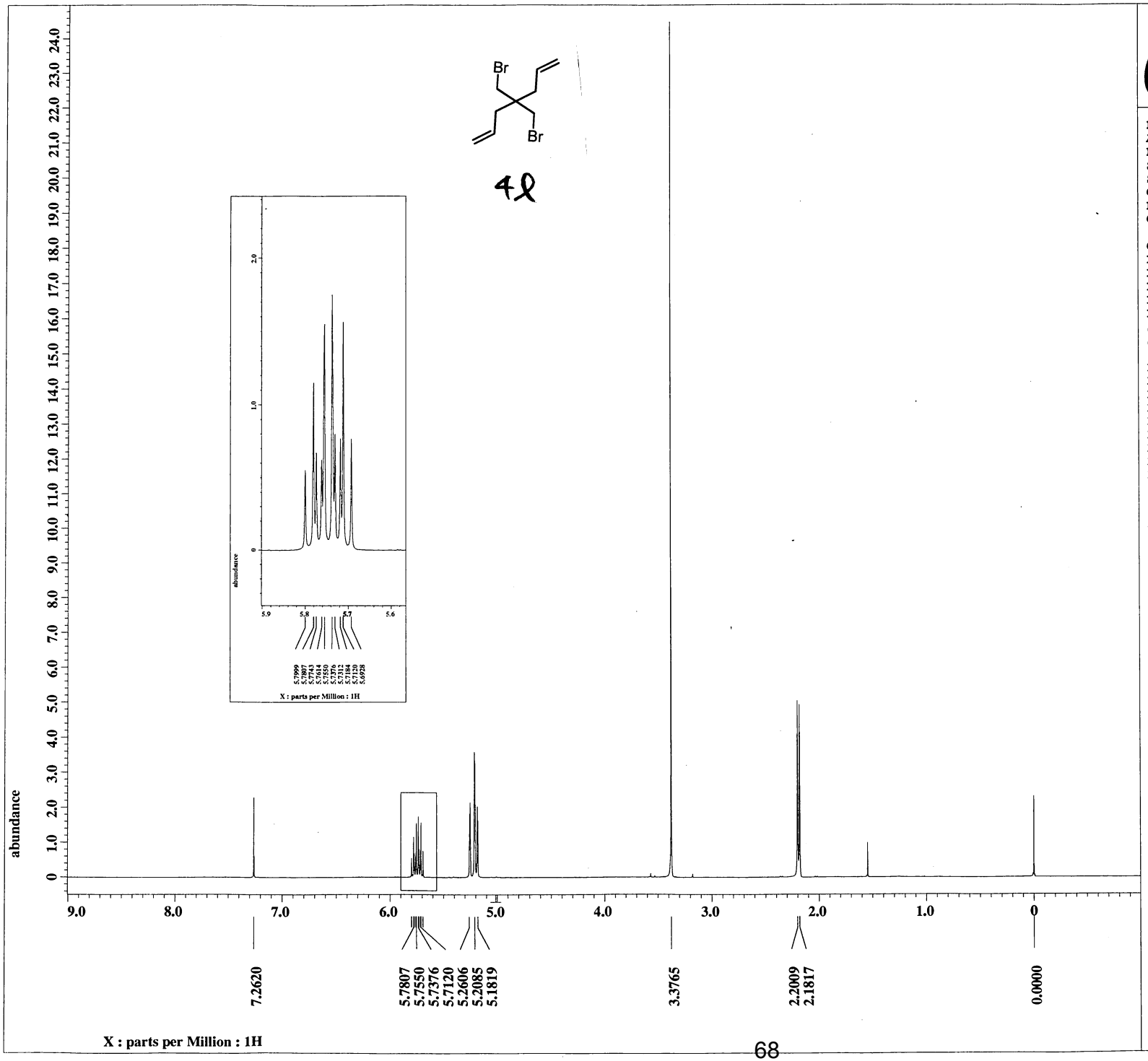


Filename = KUBO-405-13c-5.jdf
Author = element
Experiment = single_pulse_dec
Sample_id = S#401309
Solvent = CHLOROFORM-D
Creation_time = 7-MAR-2012 10:17:29
Revision_time = 21-SEP-2012 18:59:14
Current_time = 21-SEP-2012 19:01:03

Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400

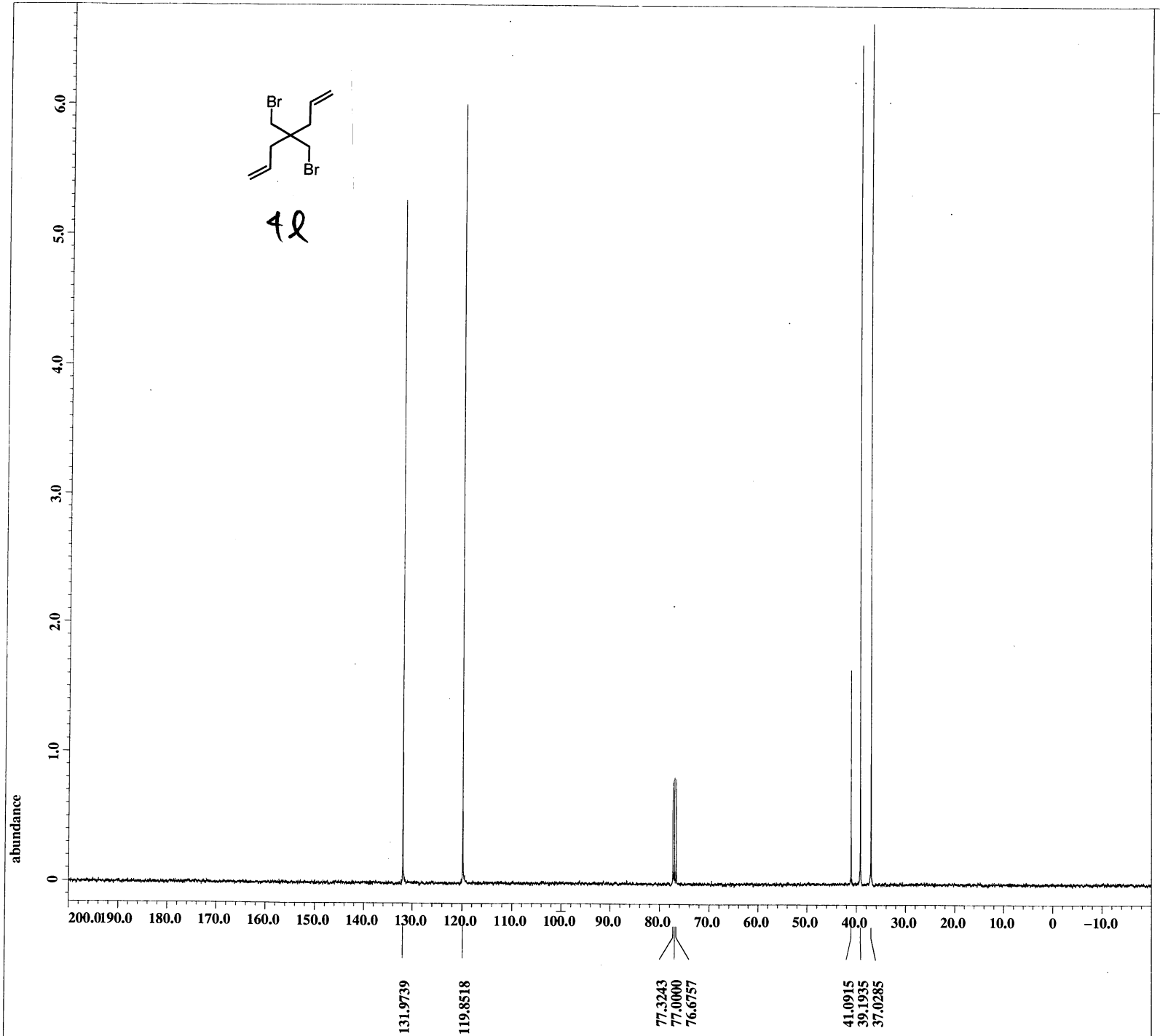
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 1.06430464[s]
X_domain = 13C
X_freq = 98.51479726[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.93958061[Hz]
X_sweep = 30.78817734[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 100
Total_scans = 100

X_90_width = 8.15[us]
X_acq_time = 1.06430464[s]
X_angle = 30[deg]
X_atn = 4.9[dB]
X_pulse = 2.71666667[us]
Irr_atn_dec = 22.445[dB]
Irr_atn_noe = 22.445[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.06430464[s]
Temp_get = 18.1[dC]



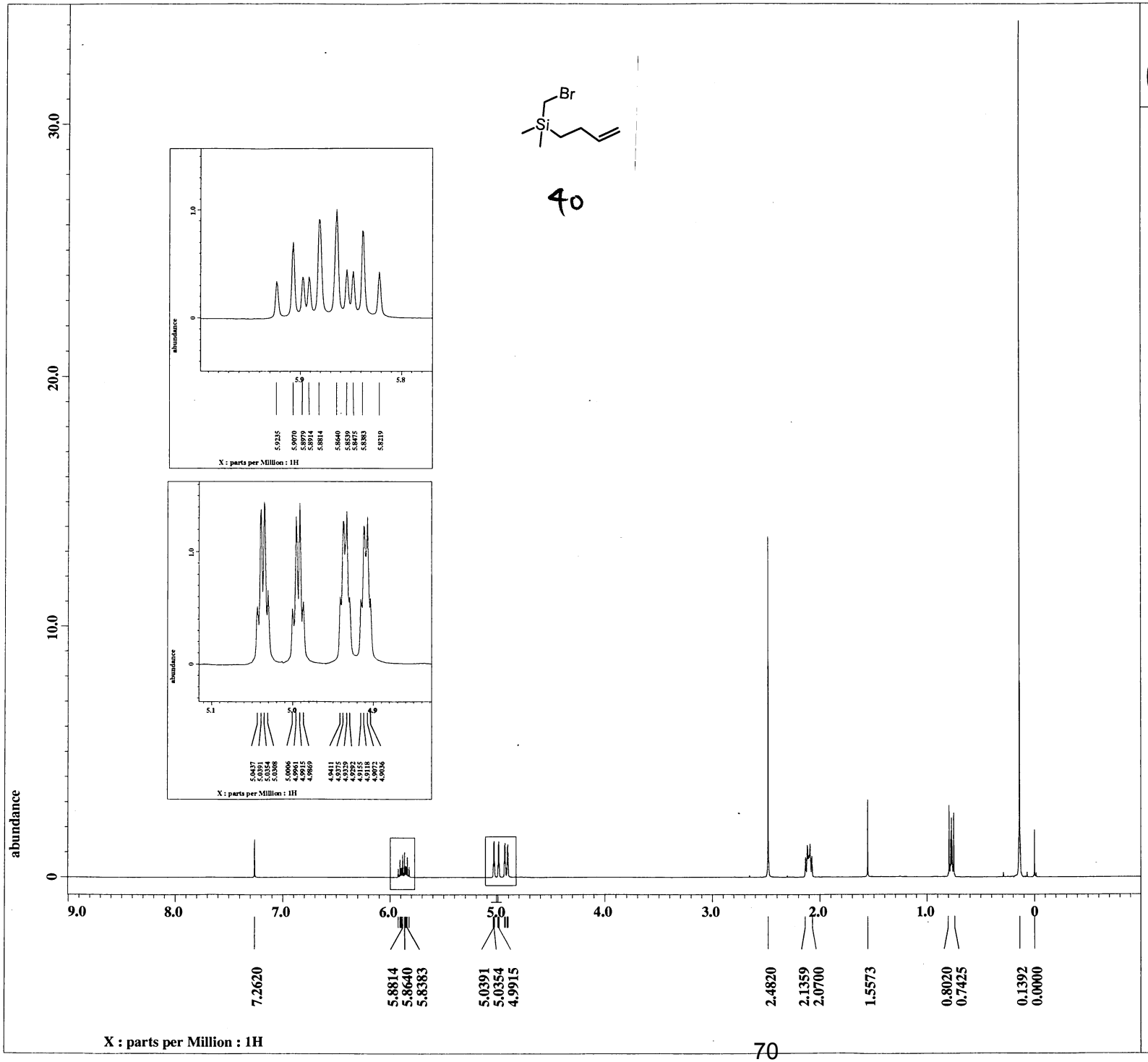
JEOL

```
Filename          = KUBO-556-pure-8.jdf
Author            = element
Experiment        = single_pulse.ex2
Sample_id         = S#611238
Solvent           = CHLOROFORM-D
Creation_time     = 30-JUN-2012 15:48:29
Revision_time     = 21-SEP-2012 19:22:20
Current_time      = 21-SEP-2012 19:22:38

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.78790144[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.35869274[Hz]
X_sweep           = 5.87682181[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.8[us]
X_acq_time        = 2.78790144[s]
X_angle           = 45[deg]
X_atn             = 1.9[dB]
X_pulse           = 5.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 7.78790144[s]
Temp_get          = 20.2[dC]
```



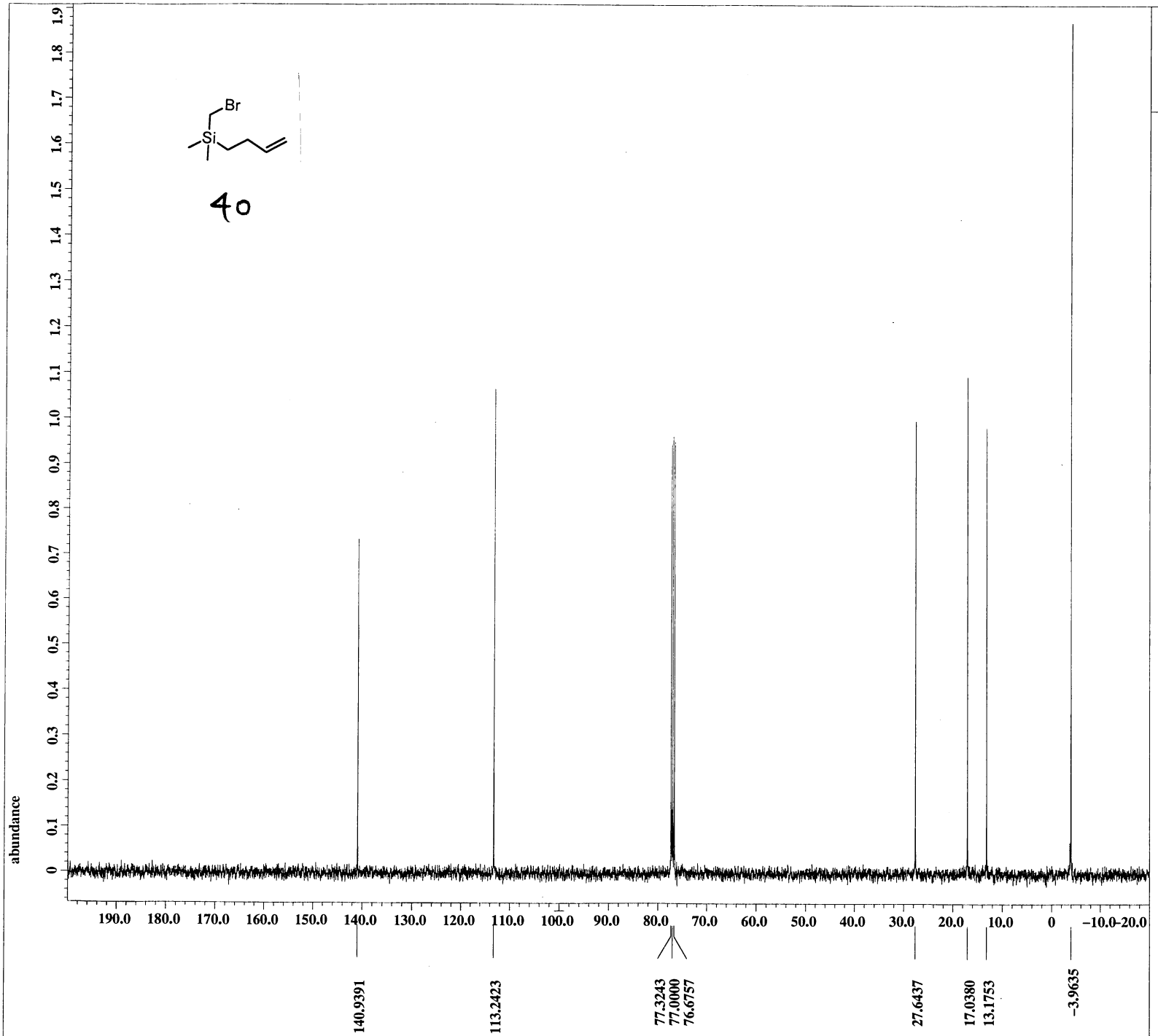
JEOL

Filename	= KUBO-556-13c-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#267763
Solvent	= CHLOROFORM-D
Creation_time	= 5-JUL-2012 06:18:09
Revision_time	= 20-AUG-2012 21:15:48
Current_time	= 20-AUG-2012 21:15:57
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 21[dC]



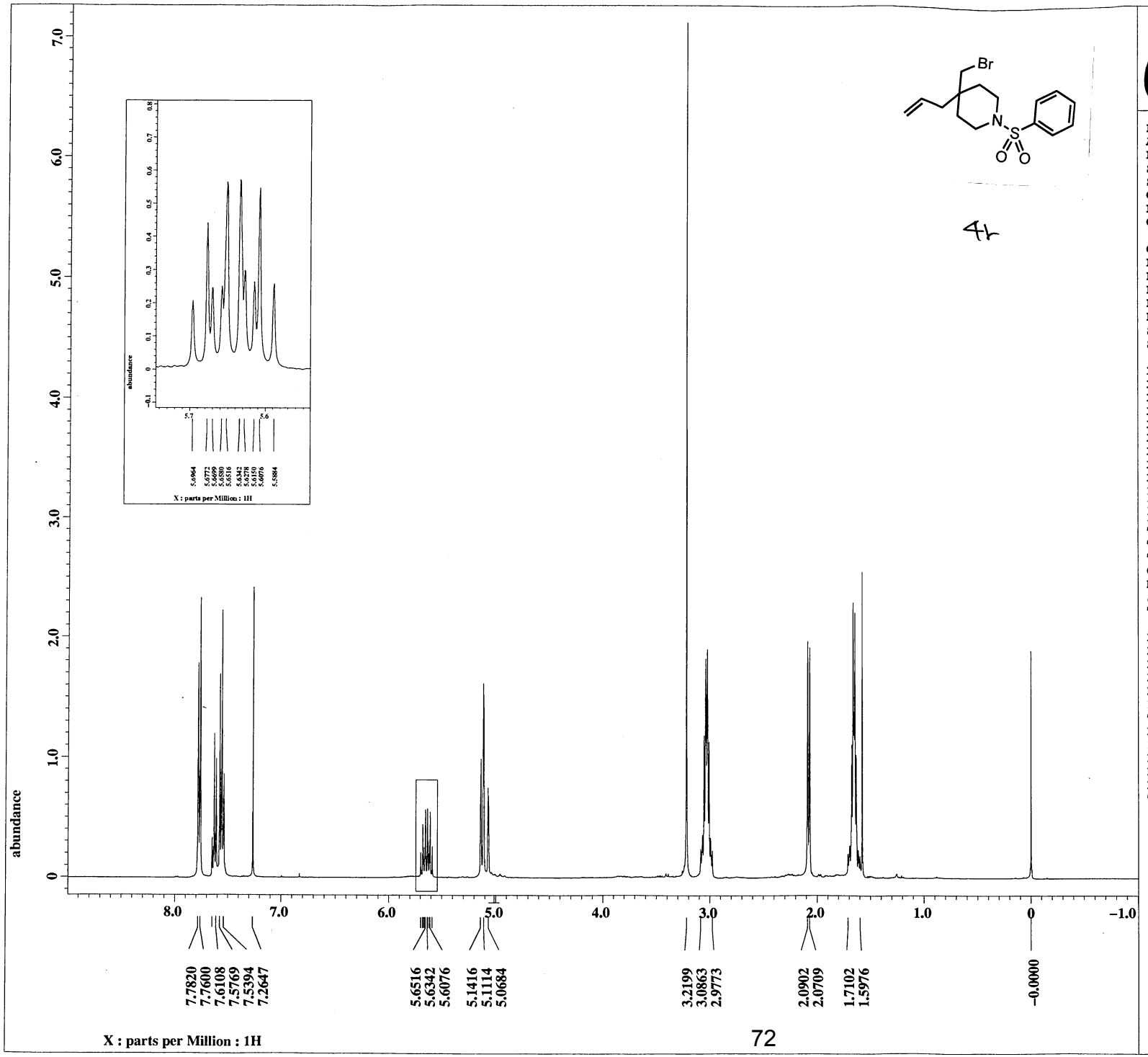
JEOL

Filename	= KUBO-487-pure-3-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#38707
Solvent	= CHLOROFORM-D
Creation_time	= 29-MAY-2012 23:58:33
Revision_time	= 21-SEP-2012 19:26:31
Current_time	= 21-SEP-2012 19:28:05
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 42
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.4[dC]



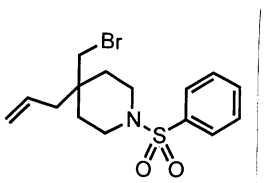
JEOL

Filename	= KUBO-487-13c-6.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#5578
Solvent	= CHLOROFORM-D
Creation_time	= 29-MAY-2012 23:05:34
Revision_time	= 23-AUG-2012 11:25:57
Current_time	= 23-AUG-2012 11:26:10
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.7[dC]



JEOL

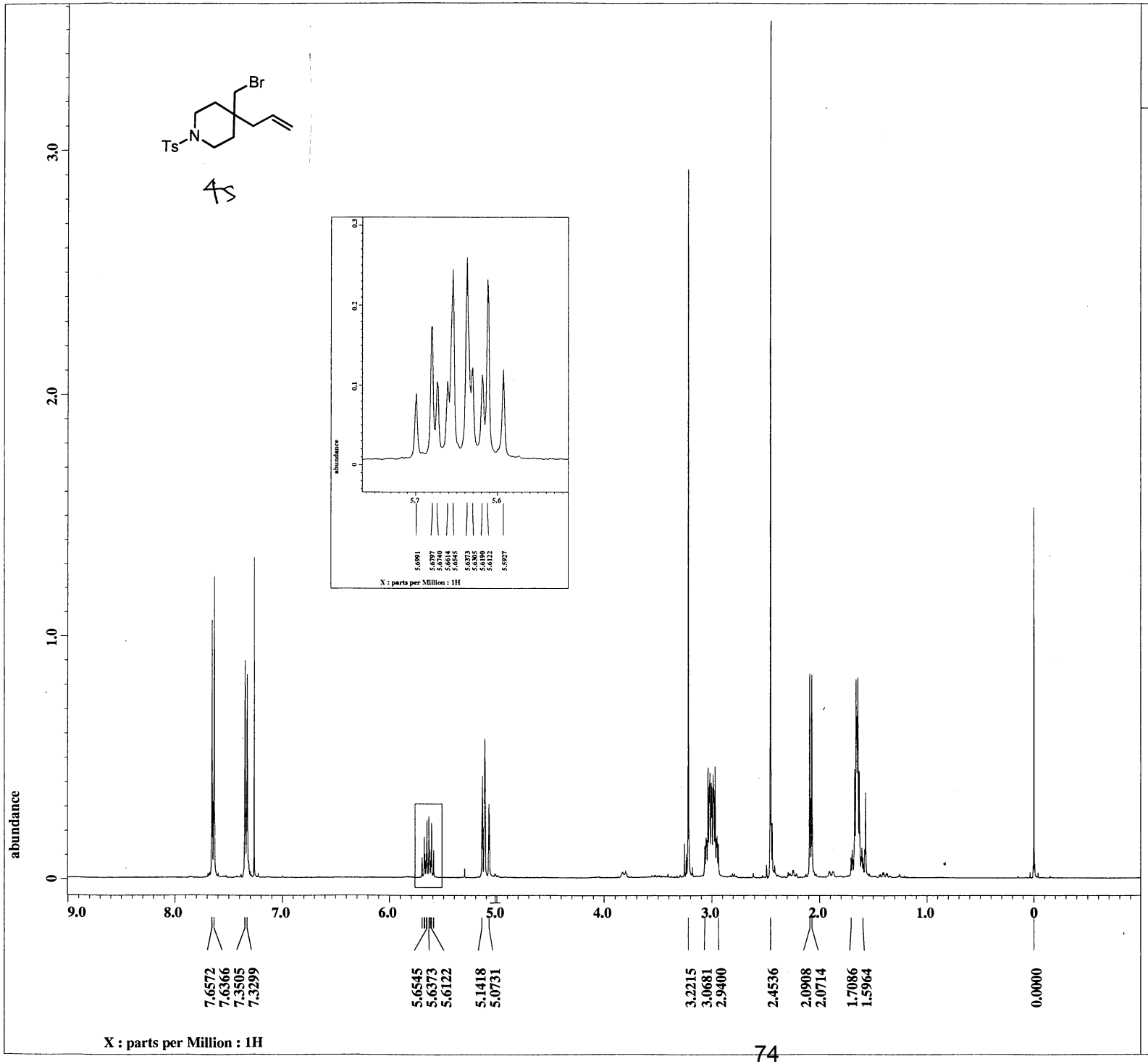
Filename	= KUBO-750-pure-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#601348
Solvent	= CHLOROFORM-D
Creation_time	= 4-DEC-2012 15:36:46
Revision_time	= 13-JAN-2013 18:16:44
Current_time	= 13-JAN-2013 18:17:07
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 46
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 19.4[dC]



4r

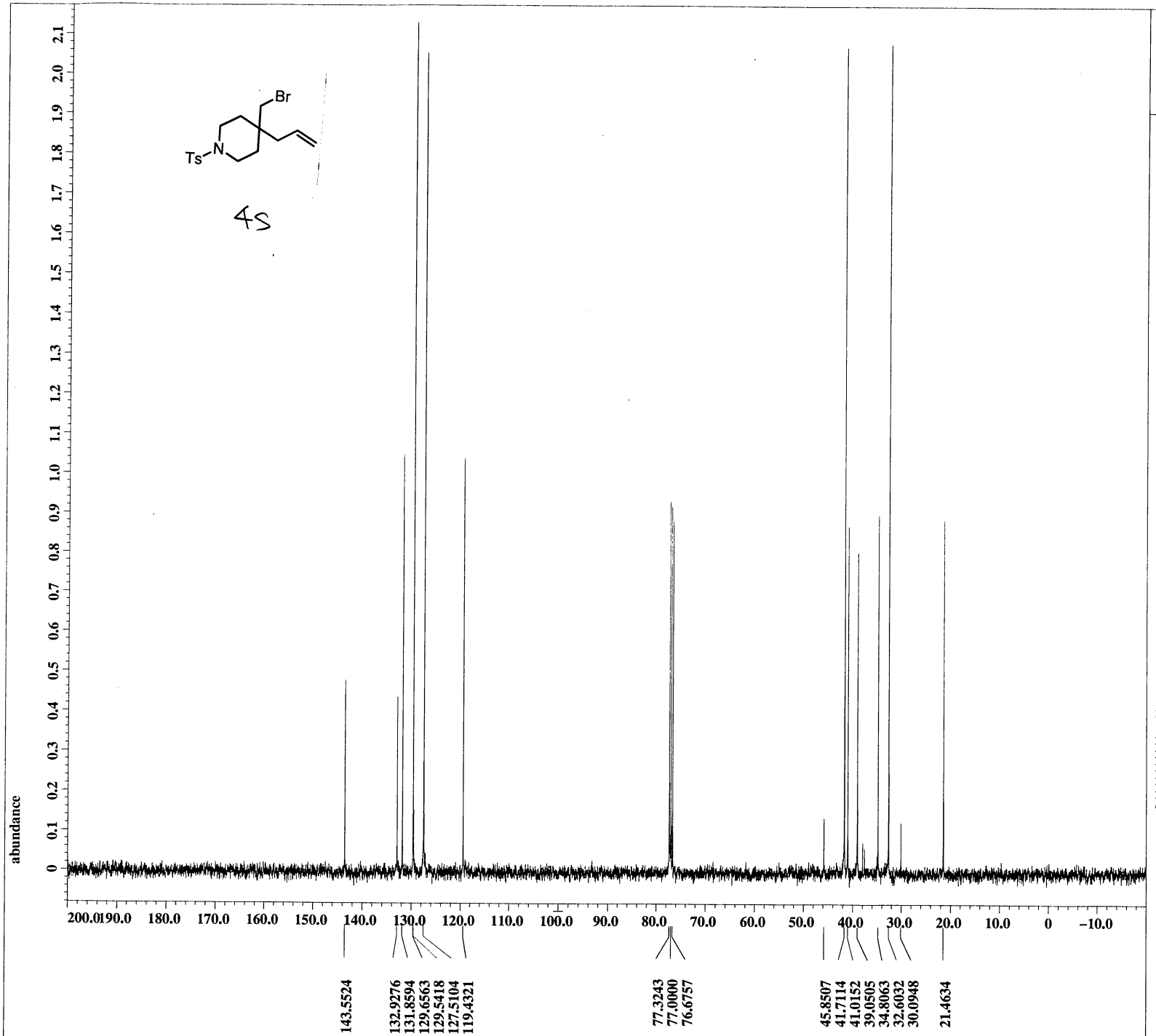
JEOL

Filename	= KUBO-750-13c-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#681910
Solvent	= CHLOROFORM-D
Creation_time	= 4-DEC-2012 17:53:02
Revision_time	= 13-JAN-2013 18:11:19
Current_time	= 13-JAN-2013 18:11:28
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 60
Total_scans	= 60
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20[dC]



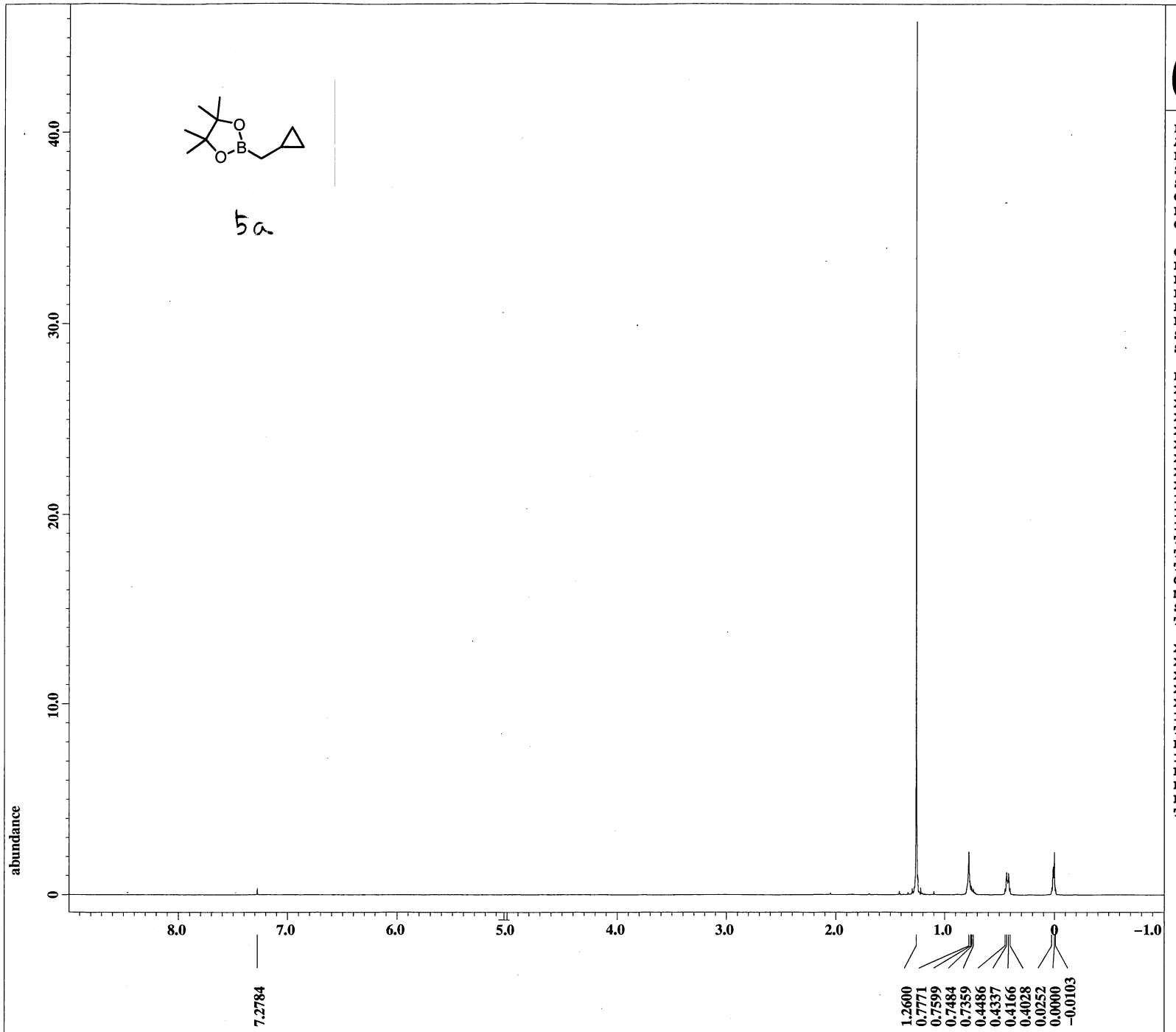
JEOL

Filename	= KUBO-557-crude-7.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#332021
Solvent	= CHLOROFORM-D
Creation_time	= 30-JUN-2012 08:53:57
Revision_time	= 21-SEP-2012 18:35:06
Current_time	= 21-SEP-2012 18:35:34
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 2.20725248[s]
X_domain	= 1H
X_freq	= 395.88430144[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.45305193[Hz]
X_sweep	= 7.42280285[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 395.88430144[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13.5[us]
X_acq_time	= 2.20725248[s]
X_angle	= 45[deg]
X_atn	= 1[dB]
X_pulse	= 6.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 5[s]
Repetition_time	= 7.20725248[s]
Temp_get	= 401.2[dC]



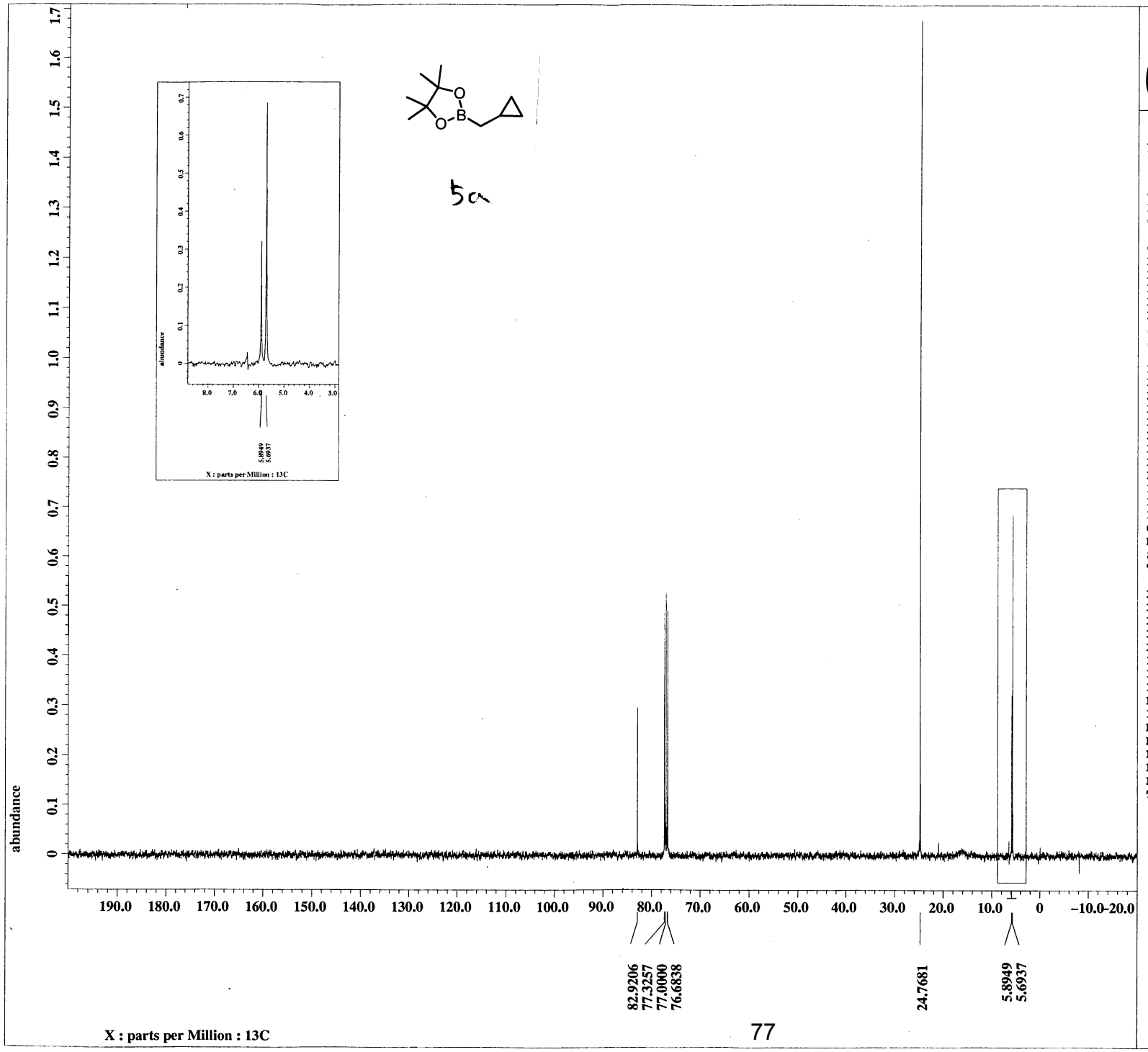
JEOL

Filename	= KUBO-557-13-6.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#267184
Solvent	= CHLOROFORM-D
Creation_time	= 4-JUL-2012 06:15:48
Revision_time	= 20-AUG-2012 21:20:11
Current_time	= 20-AUG-2012 21:20:19
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 50
Total_scans	= 50
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 21[dC]



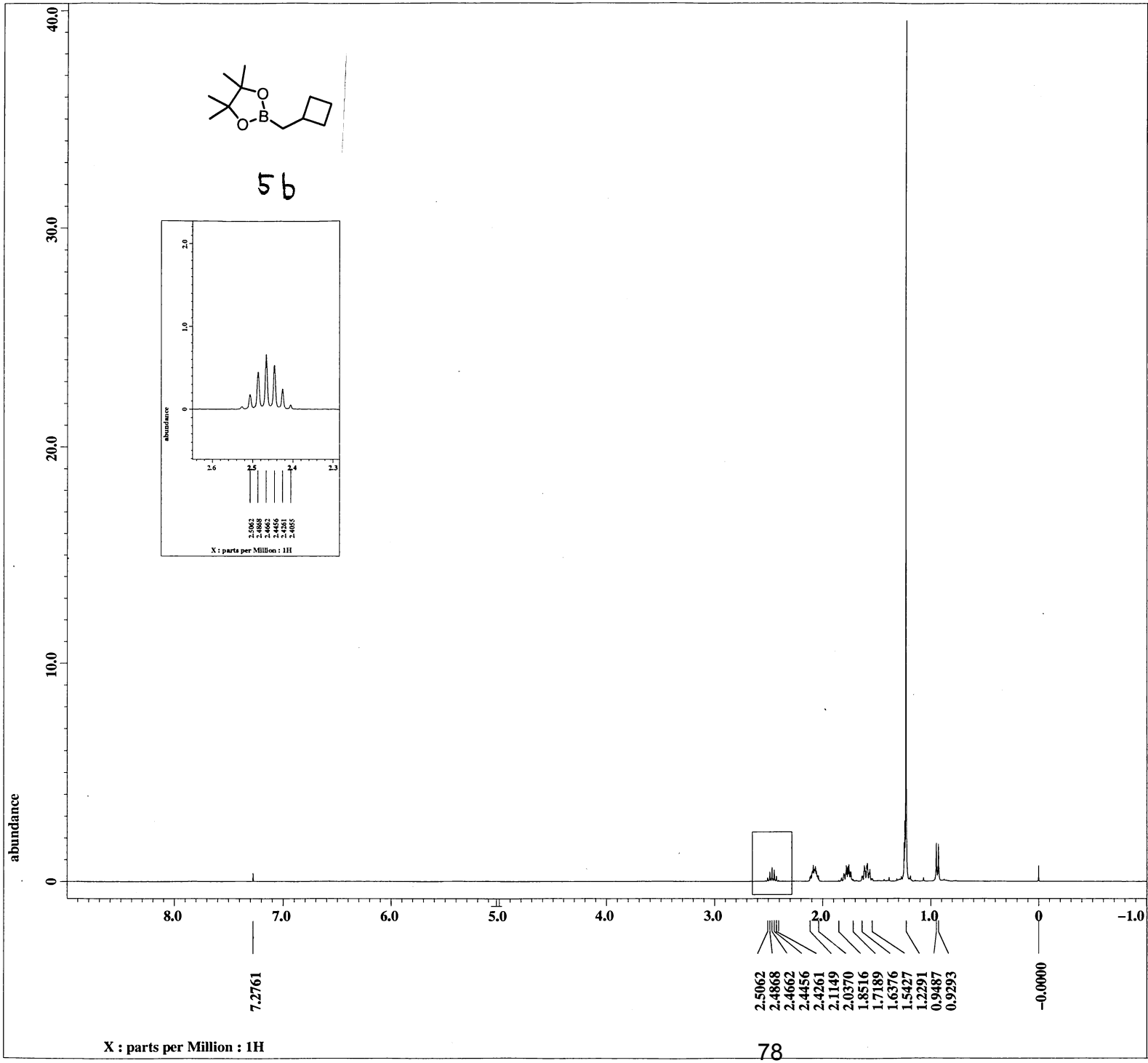
JEOL

Filename	= KUBO-239-pure-10.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#672152
Solvent	= CHLOROFORM-D
Creation_time	= 5-FEB-2012 18:22:59
Revision_time	= 20-SEP-2012 22:10:33
Current_time	= 20-SEP-2012 22:10:54
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 2.20725248[s]
X_domain	= 1H
X_freq	= 395.88430144[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.45305193[Hz]
X_sweep	= 7.42280285[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 395.88430144[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13.5[us]
X_acq_time	= 2.20725248[s]
X_angle	= 45[deg]
X_atn	= 1[dB]
X_pulse	= 6.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 7.20725248[s]
Temp_get	= 19.2[dC]

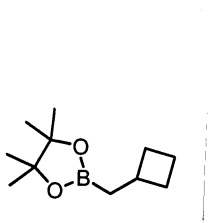


JEOL

Filename	= KUBO-239-13C-6.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#673167
Solvent	= CHLOROFORM-D
Creation_time	= 5-FEB-2012 18:27:51
Revision_time	= 20-AUG-2012 17:15:02
Current_time	= 20-AUG-2012 17:38:20
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 1.048576[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95367432[Hz]
X_sweep	= 31.25[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= TRUE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 9.5[us]
X_acq_time	= 1.048576[s]
X_angle	= 30[deg]
X_atn	= 3.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 19.607[dB]
Irr_atn_noe	= 19.607[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 52
Relaxation_delay	= 2[s]
Repetition_time	= 3.048576[s]
Temp_get	= 19.6[dC]



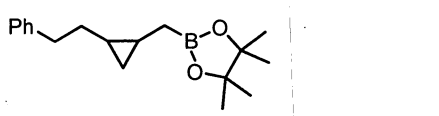
Filename	= KUBO-271-data-7.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#660737
Solvent	= CHLOROFORM-D
Creation_time	= 5-FEB-2012 18:03:57
Revision_time	= 20-SEP-2012 22:19:10
Current_time	= 20-SEP-2012 22:19:15
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 2.20725248[s]
X_domain	= 1H
X_freq	= 395.88430144[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.45305193[Hz]
X_sweep	= 7.42280285[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 395.88430144[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13.5[us]
X_acq_time	= 2.20725248[s]
X_angle	= 45[deg]
X_atn	= 1[dB]
X_pulse	= 6.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 30
Relaxation_delay	= 5[s]
Repetition_time	= 7.20725248[s]
Temp_get	= 19.2[dC]



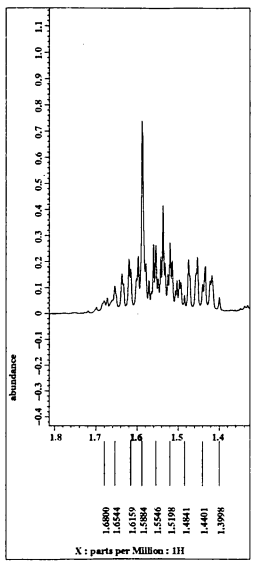
5b

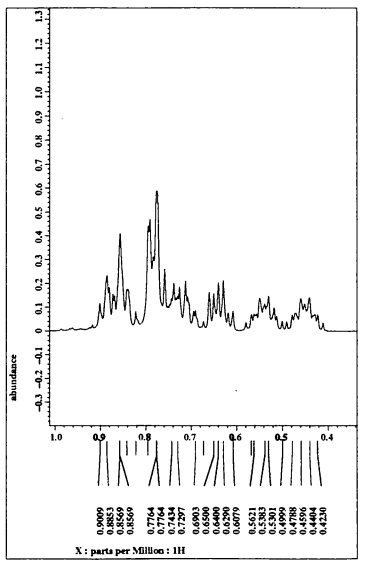
JEOL

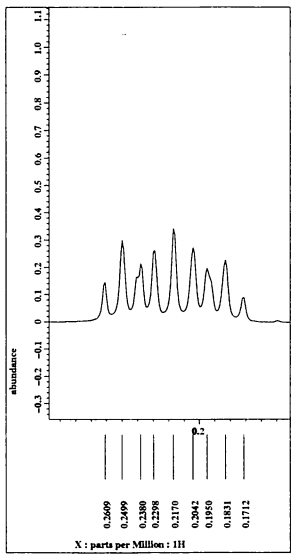
Filename	= KUBO-271-13C-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#662255
Solvent	= CHLOROFORM-D
Creation_time	= 5-FEB-2012 18:10:34
Revision_time	= 20-AUG-2012 17:44:16
Current_time	= 20-AUG-2012 17:46:22
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 1.048576[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95367432[Hz]
X_sweep	= 31.25[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 100
Total_scans	= 100
X_90_width	= 9.5[us]
X_acq_time	= 1.048576[s]
X_angle	= 30[deg]
X_atn	= 3.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 19.607[dB]
Irr_atn_noe	= 19.607[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.048576[s]
Temp_get	= 19.7[dC]

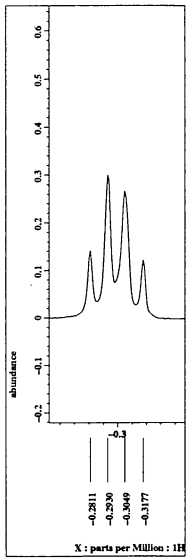


5c



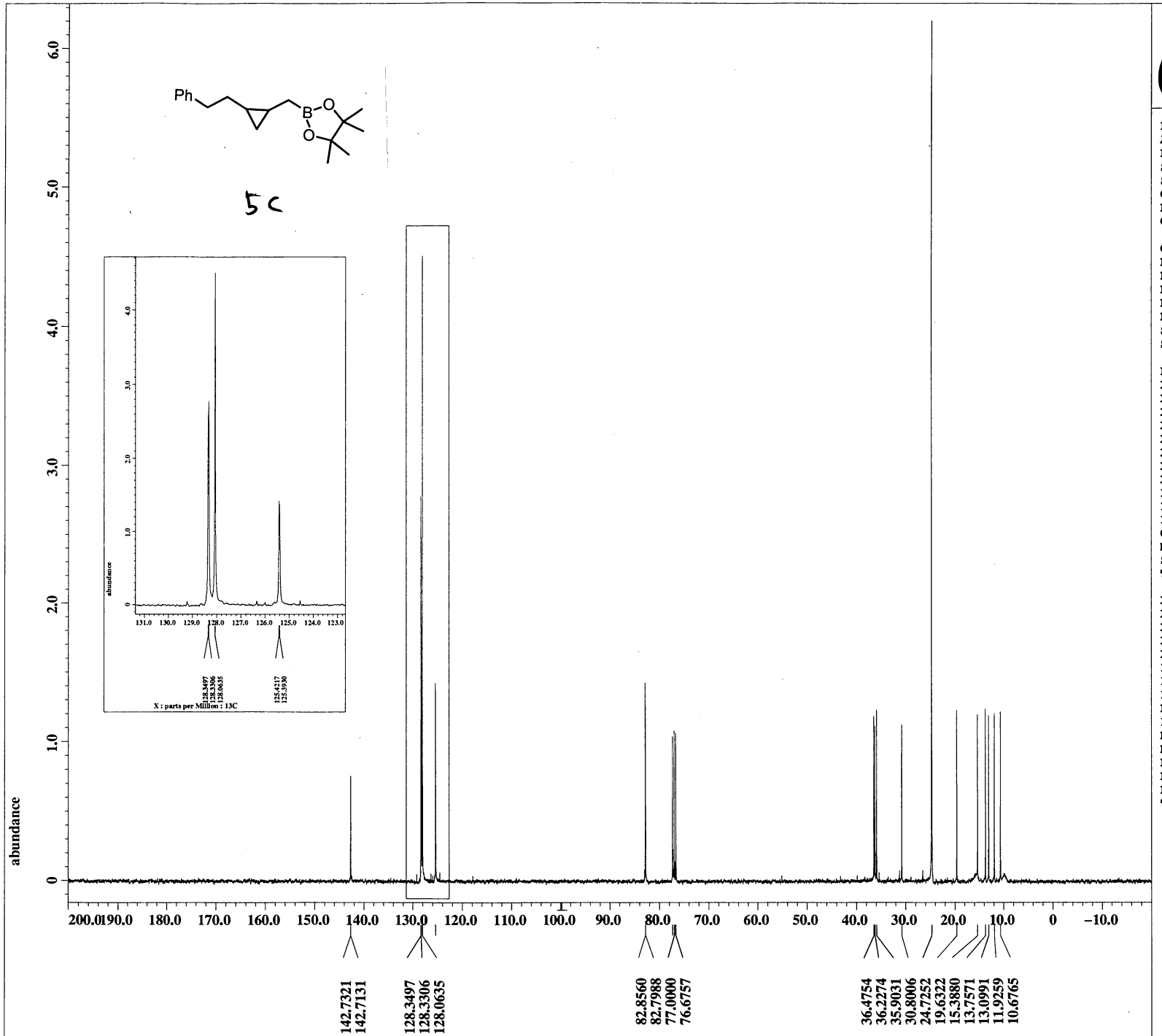






JEOL

Filename	= KUBO-418-pure-6.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#325158
Solvent	= CHLOROFORM-D
Creation_time	= 15-MAR-2012 08:06:14
Revision_time	= 20-SEP-2012 23:33:43
Current_time	= 20-SEP-2012 23:34:03
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 15.6[dC]

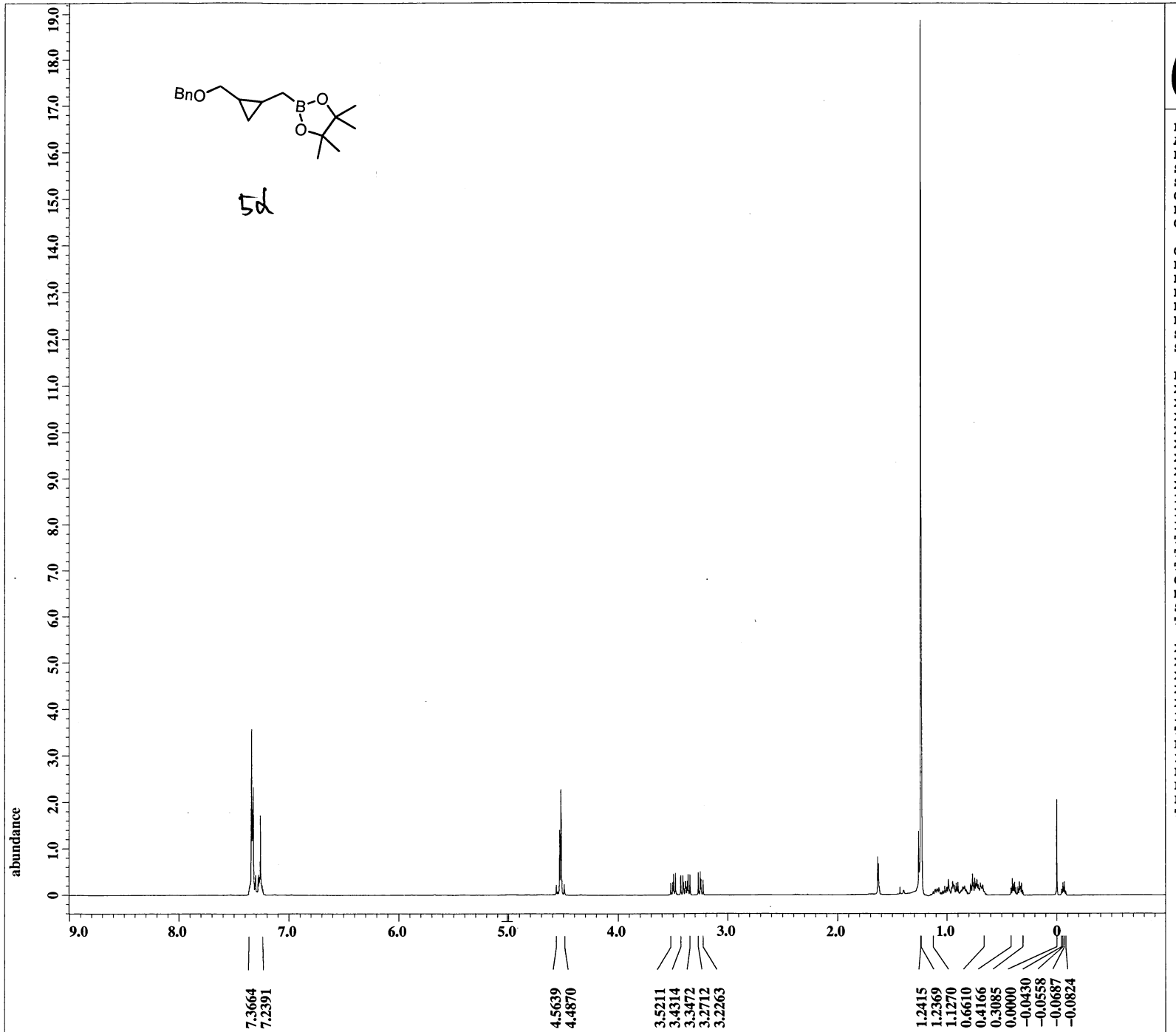


Filename = KUBO-418-13C-5.jdf
Author = element
Experiment = single_pulse_dec
Sample_id = S#355623
Solvent = CHLOROFORM-D
Creation_time = 15-MAR-2012 08:59:14
Revision_time = 20-SEP-2012 23:36:29
Current_time = 20-SEP-2012 23:37:27

Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400

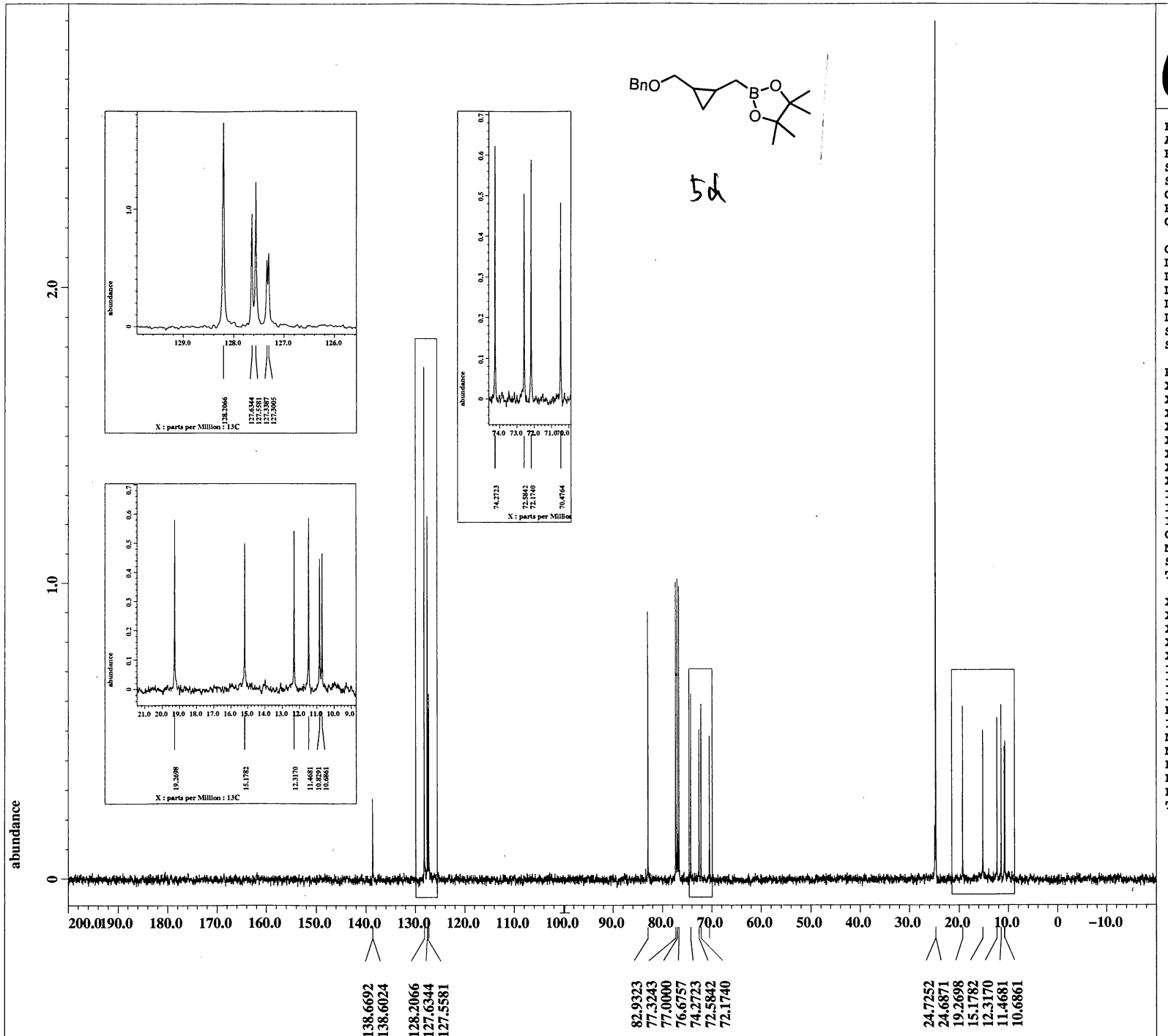
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 1.06430464[s]
X_domain = 13C
X_freq = 98.51479726[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.93958061[Hz]
X_sweep = 30.78817734[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 80
Total_scans = 80

X_90_width = 8.15[us]
X_acq_time = 1.06430464[s]
X_angle = 30[deg]
X_atn = 4.9[dB]
X_pulse = 2.71666667[us]
Irr_atn_dec = 22.445[dB]
Irr_atn_noe = 22.445[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.06430464[s]
Temp_get = 16.9[dC]



JEOL

Filename	= KUBO-563-pure-3-4.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#834383
Solvent	= CHLOROFORM-D
Creation_time	= 2-SEP-2012 21:51:39
Revision_time	= 21-SEP-2012 09:44:33
Current_time	= 21-SEP-2012 09:44:37
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.6[dC]

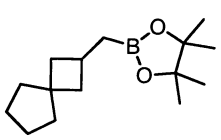


Filename = KUBO-563-13c-2-3.jdf
Author = element
Experiment = single_pulse_dec
Sample_id = S#846268
Solvent = CHLOROFORM-D
Creation_time = 2-SEP-2012 22:13:43
Revision_time = 21-SEP-2012 09:46:00
Current_time = 21-SEP-2012 09:47:44

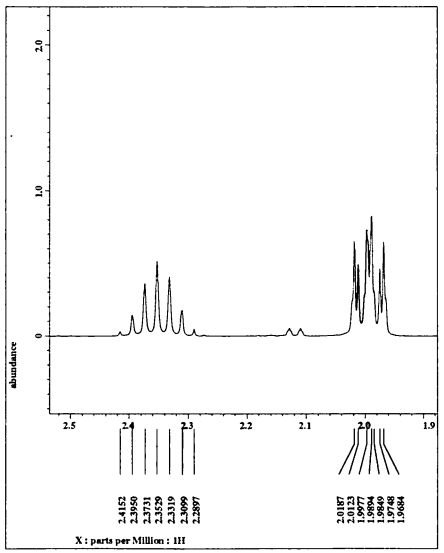
Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400

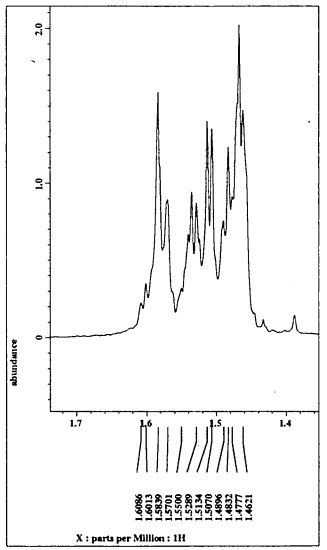
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 1.06430464[s]
X_domain = 13C
X_freq = 98.51479726[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.93958061[Hz]
X_sweep = 30.78817734[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 70
Total_scans = 70

X_90_width = 8.15[us]
X_acq_time = 1.06430464[s]
X_angle = 30[deg]
X_atn = 4.9[dB]
X_pulse = 2.71666667[us]
Irr_atn_dec = 22.445[dB]
Irr_atn_noe = 22.445[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 60
Relaxation_delay = 2[s]
Repetition_time = 3.06430464[s]
Temp_get = 20.8[dC]

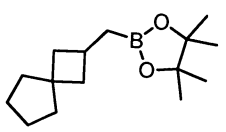


5e





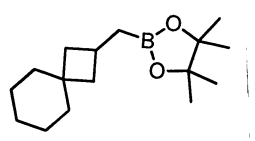
Filename	= KUBO-445-pure-7.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#629672
Solvent	= CHLOROFORM-D
Creation_time	= 23-APR-2012 16:28:23
Revision_time	= 21-SEP-2012 00:50:18
Current_time	= 21-SEP-2012 00:50:44
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.6[dC]



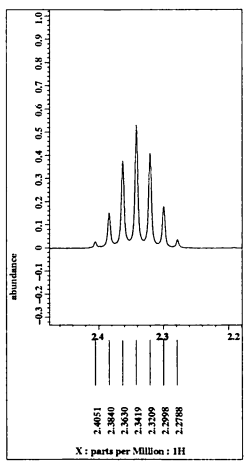
5e

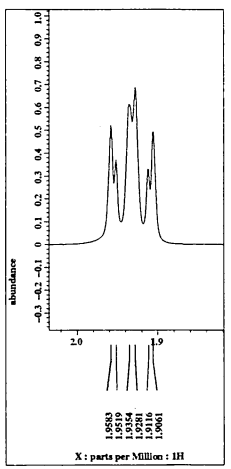
JEOL

Filename	= KUBO-445-13C-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#709906
Solvent	= CHLOROFORM-D
Creation_time	= 23-APR-2012 18:41:31
Revision_time	= 20-AUG-2012 19:46:54
Current_time	= 20-AUG-2012 19:47:00
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 25
Total_scans	= 25
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.8[dC]



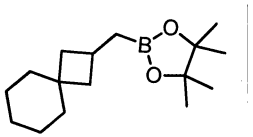
5f



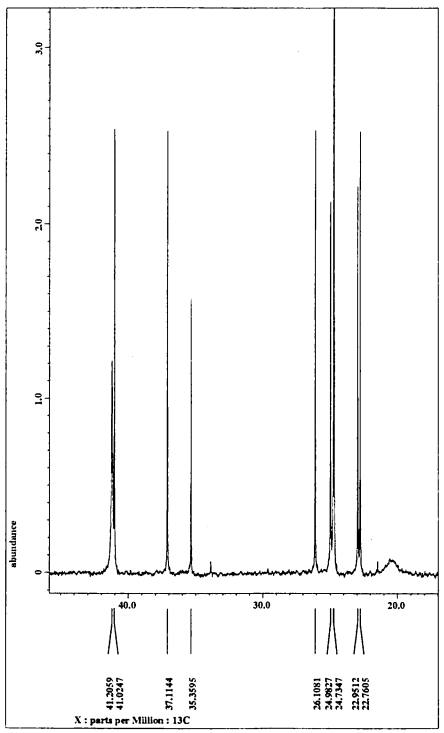


JEOL

Filename	= KUBO-401-pure-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#649730
Solvent	= CHLOROFORM-D
Creation_time	= 5-MAR-2012 17:08:29
Revision_time	= 21-SEP-2012 00:13:33
Current_time	= 21-SEP-2012 00:13:41
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 17.6[dC]



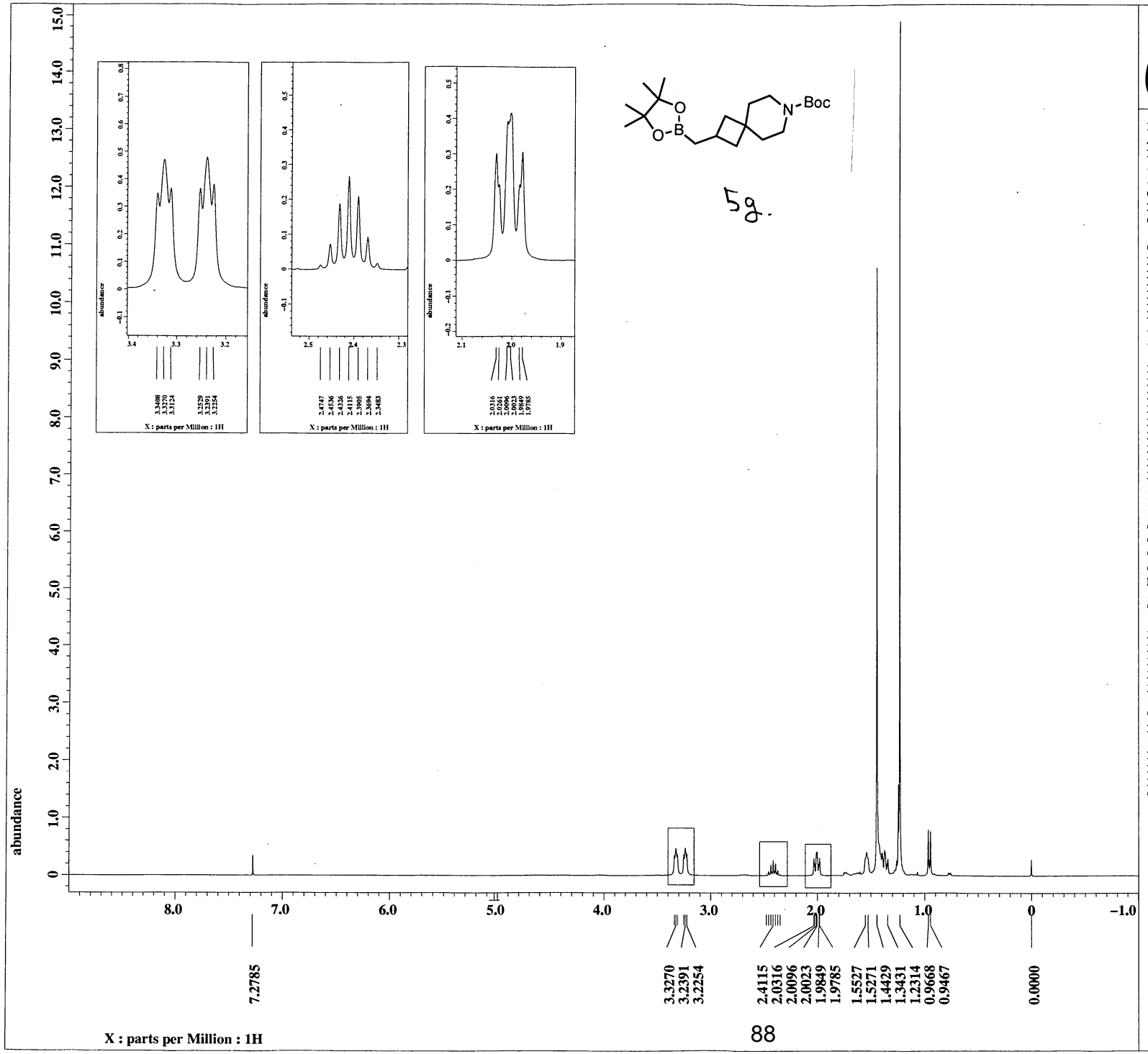
5f



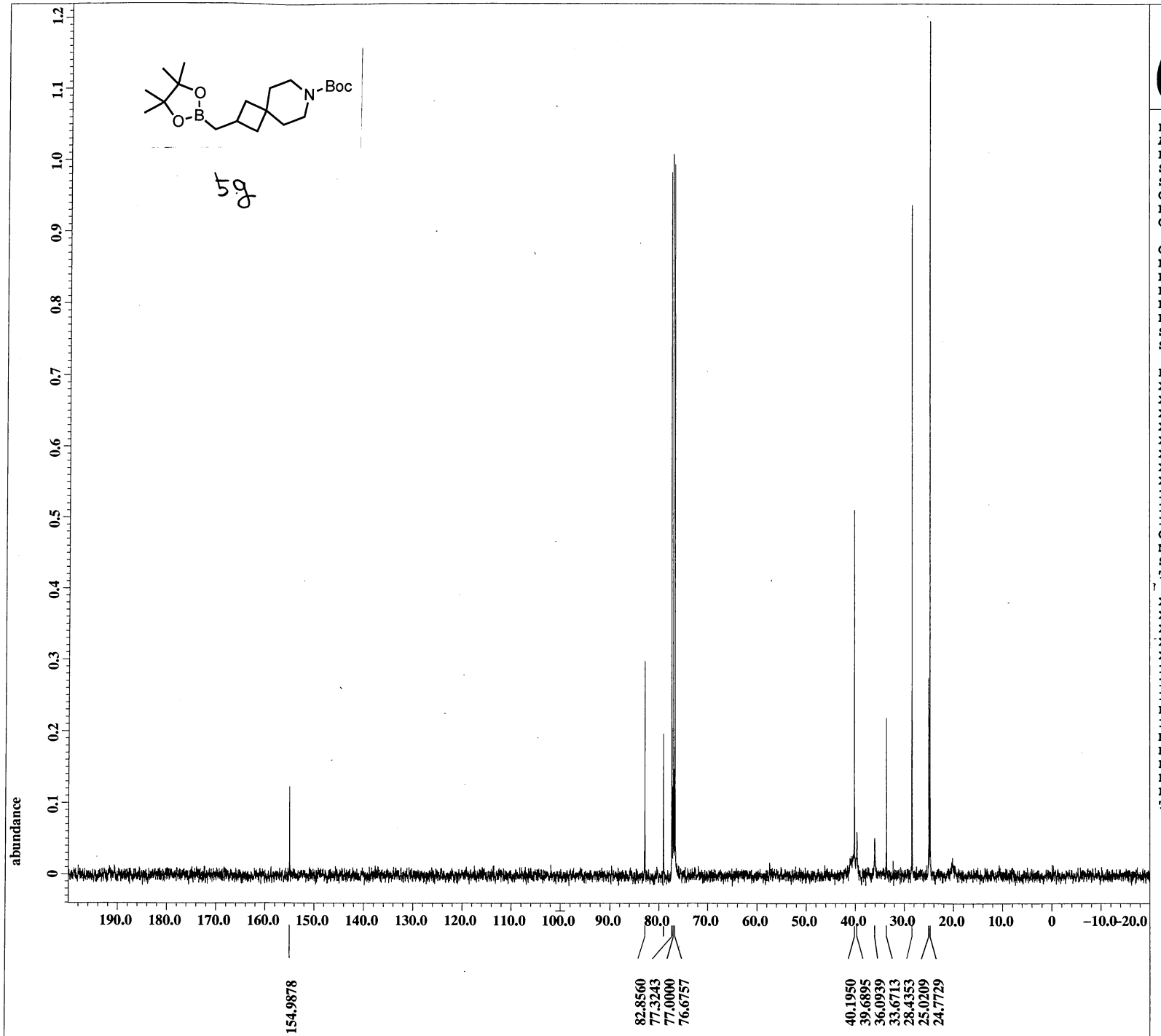
JEOL

Filename	= KUBO-401-13c-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#685994
Solvent	= CHLOROFORM-D
Creation_time	= 5-MAR-2012 18:11:11
Revision_time	= 20-AUG-2012 18:05:45
Current_time	= 20-AUG-2012 18:08:50
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 17.8[dC]

X : parts per Million : 13C

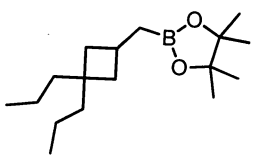


Filename	= KUBO-740-pure-3.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#624616
Solvent	= CHLOROFORM-D
Creation_time	= 2-DEC-2012 16:16:04
Revision_time	= 13-JAN-2013 17:50:42
Current_time	= 13-JAN-2013 17:50:46
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 32
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 18.8[dC]

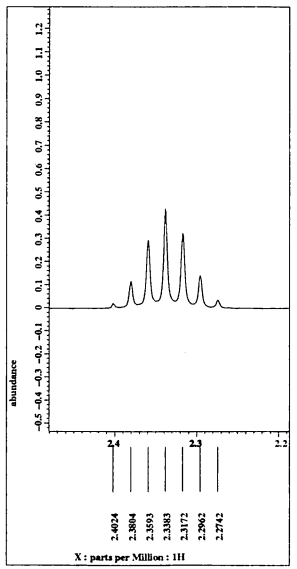


JEOL

Filename	= KUBO-740-13c-4.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#626829
Solvent	= CHLOROFORM-D
Creation_time	= 2-DEC-2012 16:28:08
Revision_time	= 13-JAN-2013 17:51:52
Current_time	= 13-JAN-2013 17:52:25
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 200
Total_scans	= 200
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 19[dC]

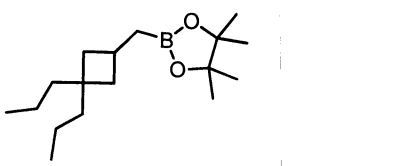


5h

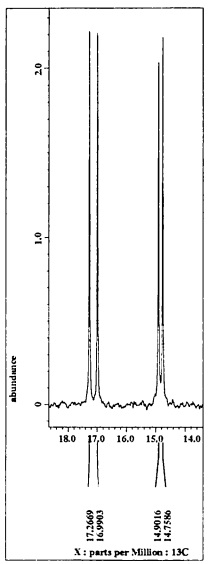


JEOL

Filename	= KUBO-442-pure-6.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#775622
Solvent	= CHLOROFORM-D
Creation_time	= 20-APR-2012 20:32:00
Revision_time	= 21-SEP-2012 00:35:01
Current_time	= 21-SEP-2012 00:35:31
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 21[dC]



5h

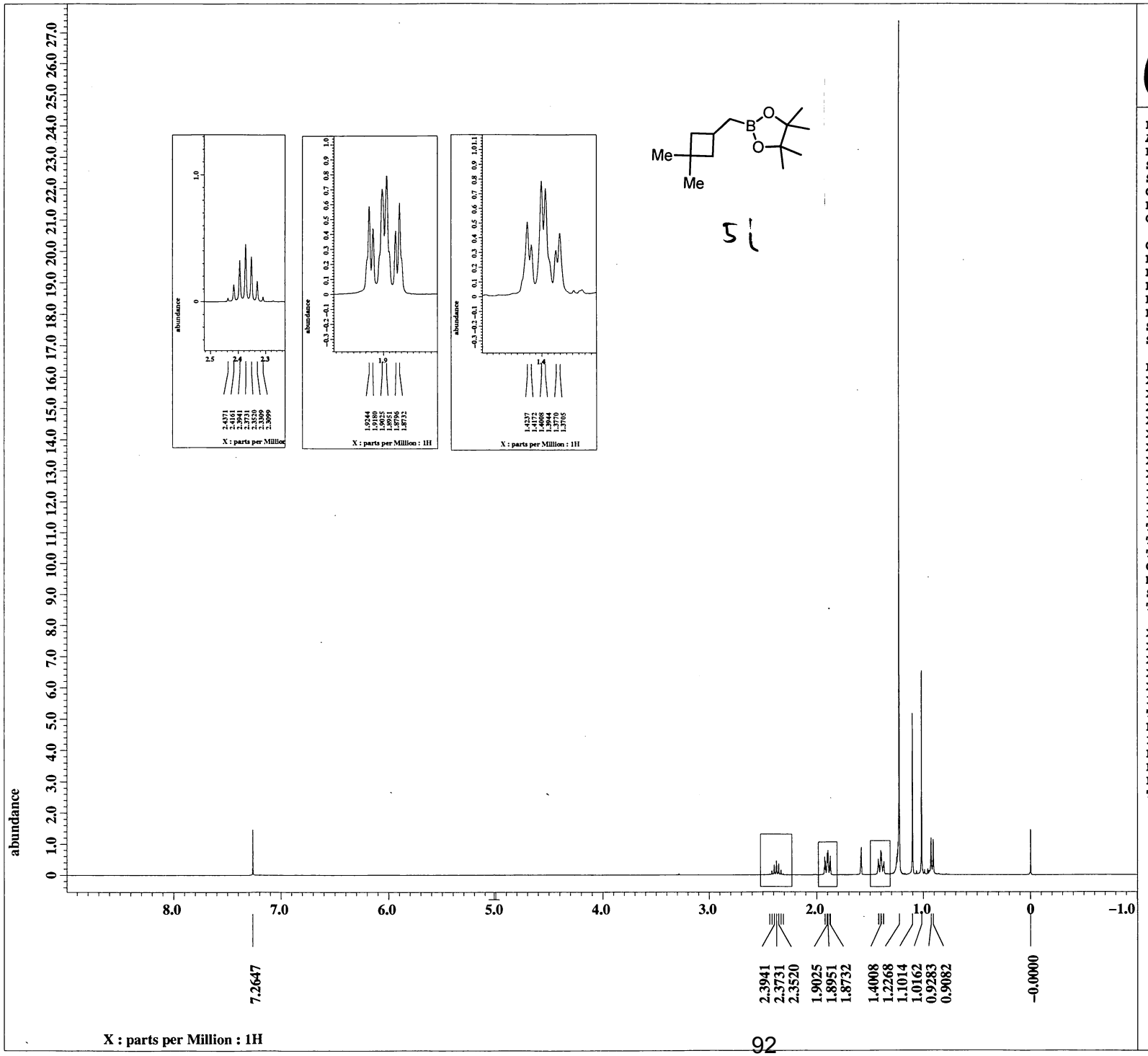


abundance

X : parts per Million : 13C

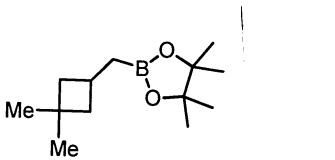
82.6748
77.3243
77.0000
76.6757
43.2279
40.7863
39.8993
37.1716
25.0113
24.7347
17.2669
16.9903
14.9016
14.7586

Filename	= KUBO-442-13c-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#838843
Solvent	= CHLOROFORM-D
Creation_time	= 20-APR-2012 22:17:35
Revision_time	= 20-AUG-2012 19:40:33
Current_time	= 20-AUG-2012 19:41:15
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 40
Total_scans	= 40
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 21.1[dC]



JEOL

Filename	= KUBO-448-pure-7.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#675389
Solvent	= CHLOROFORM-D
Creation_time	= 25-APR-2012 17:44:17
Revision_time	= 21-SEP-2012 00:54:39
Current_time	= 21-SEP-2012 00:54:47
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.9[dC]



5 i

abundance

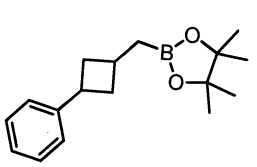
7.0 6.0 5.0 4.0 3.0 2.0 1.0 0

190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0 −10.0−20.0

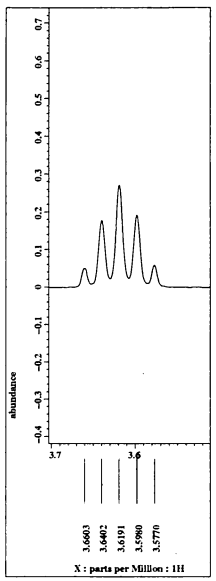
82.7129
77.3243
77.0000
76.6757
43.3519
31.2584
28.4830
24.7538
24.4582

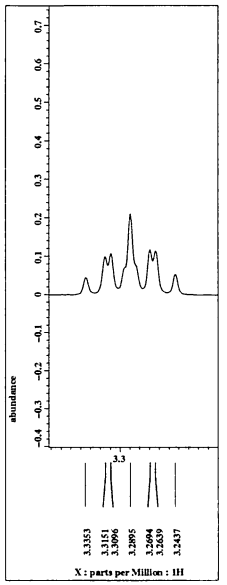
X : parts per Million : 13C

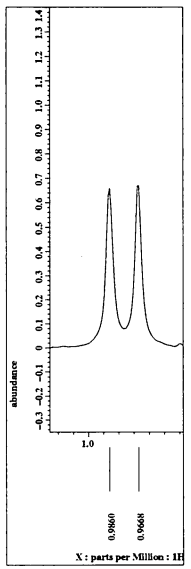
Filename	= KUBO-448-13C-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#708723
Solvent	= CHLOROFORM-D
Creation_time	= 25-APR-2012 18:40:03
Revision_time	= 20-AUG-2012 19:49:08
Current_time	= 20-AUG-2012 19:49:21
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 40
Total_scans	= 40
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 21.1[dC]



5j

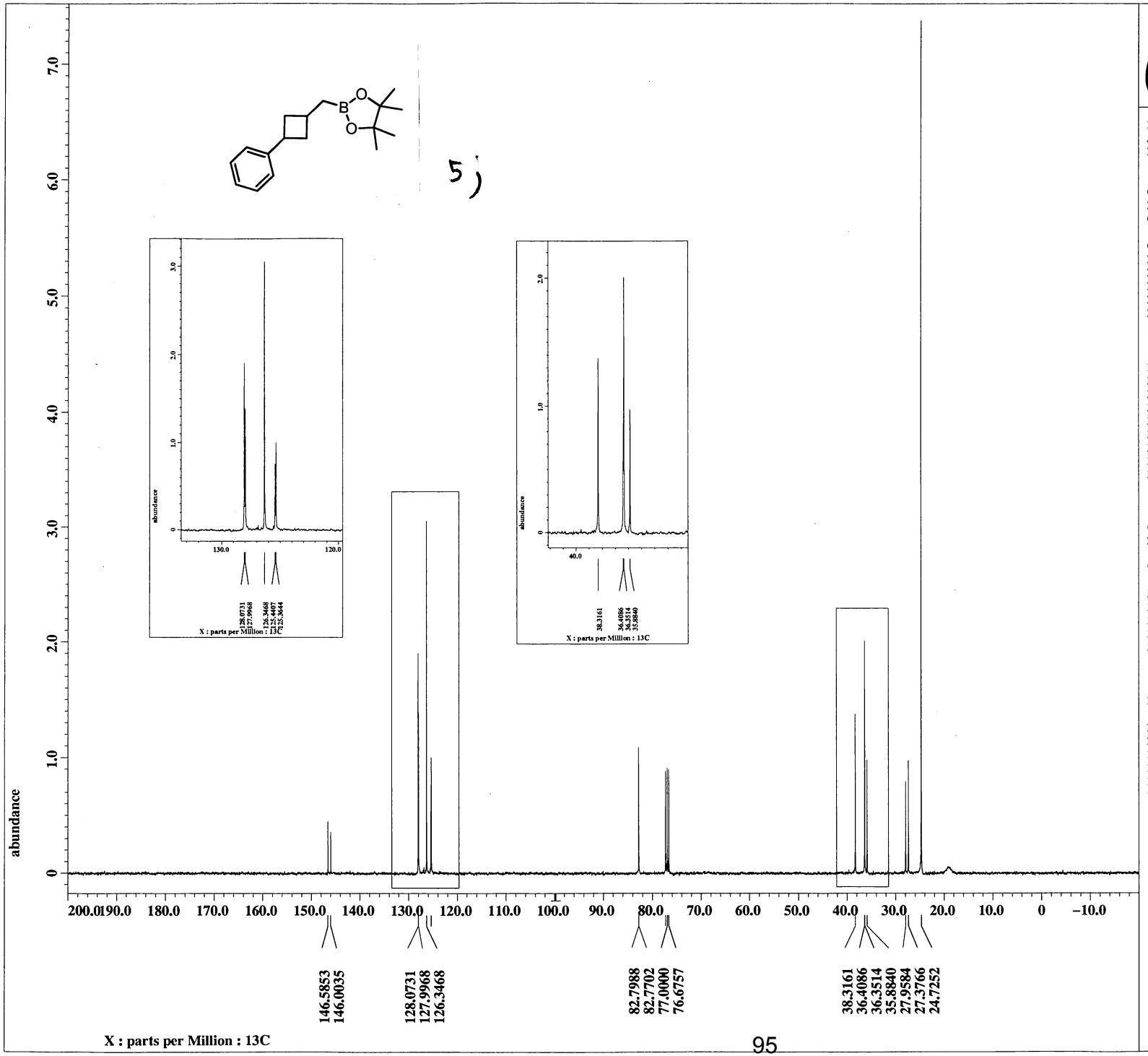






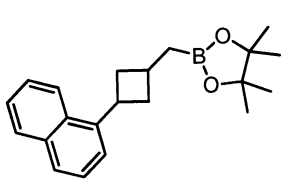
JEOL

Filename	= KUBO-391-pure-8.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#468385
Solvent	= CHLOROFORM-D
Creation_time	= 1-MAR-2012 12:06:51
Revision_time	= 20-SEP-2012 23:16:35
Current_time	= 20-SEP-2012 23:18:55
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 17.7[dC]

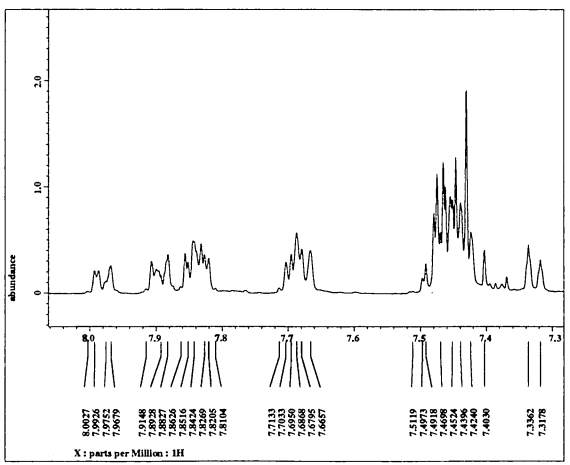


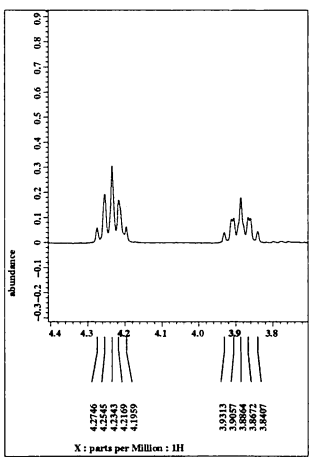
JEOL

Filename	= KUBO-391-13C-7.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#476037
Solvent	= CHLOROFORM-D
Creation_time	= 1-MAR-2012 12:21:26
Revision_time	= 20-SEP-2012 23:20:20
Current_time	= 20-SEP-2012 23:21:22
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 70
Total_scans	= 70
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 58
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 18[dC]



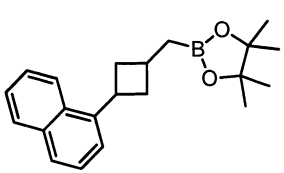
5k



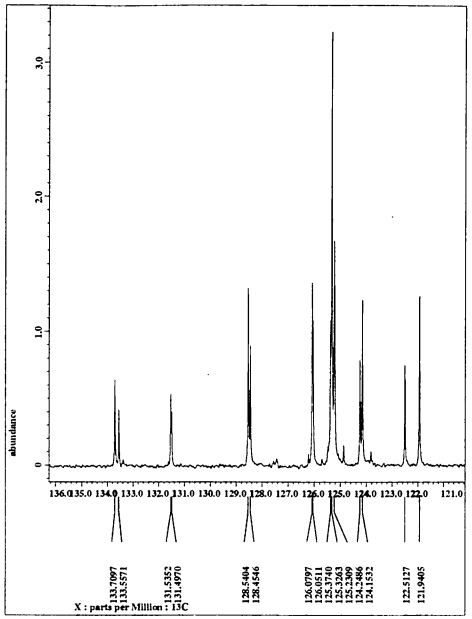


JEOL

Filename	= KUBO-407-pure-2-3.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#606201
Solvent	= CHLOROFORM-D
Creation_time	= 9-MAR-2012 15:55:24
Revision_time	= 21-SEP-2012 00:29:48
Current_time	= 21-SEP-2012 00:31:35
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 42
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 18.2[dC]



5k

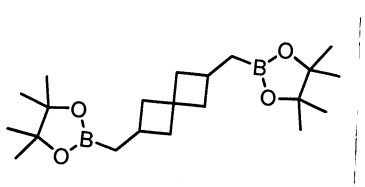


abundance

X : parts per Million : 13C

141.6067
141.5018
128.5404
126.0797
125.3263
125.2309
124.1532
121.9405
82.8178
82.7797
77.3243
77.0000
37.6771
35.4358
34.0719
33.3280
28.4544
27.6628
24.7252

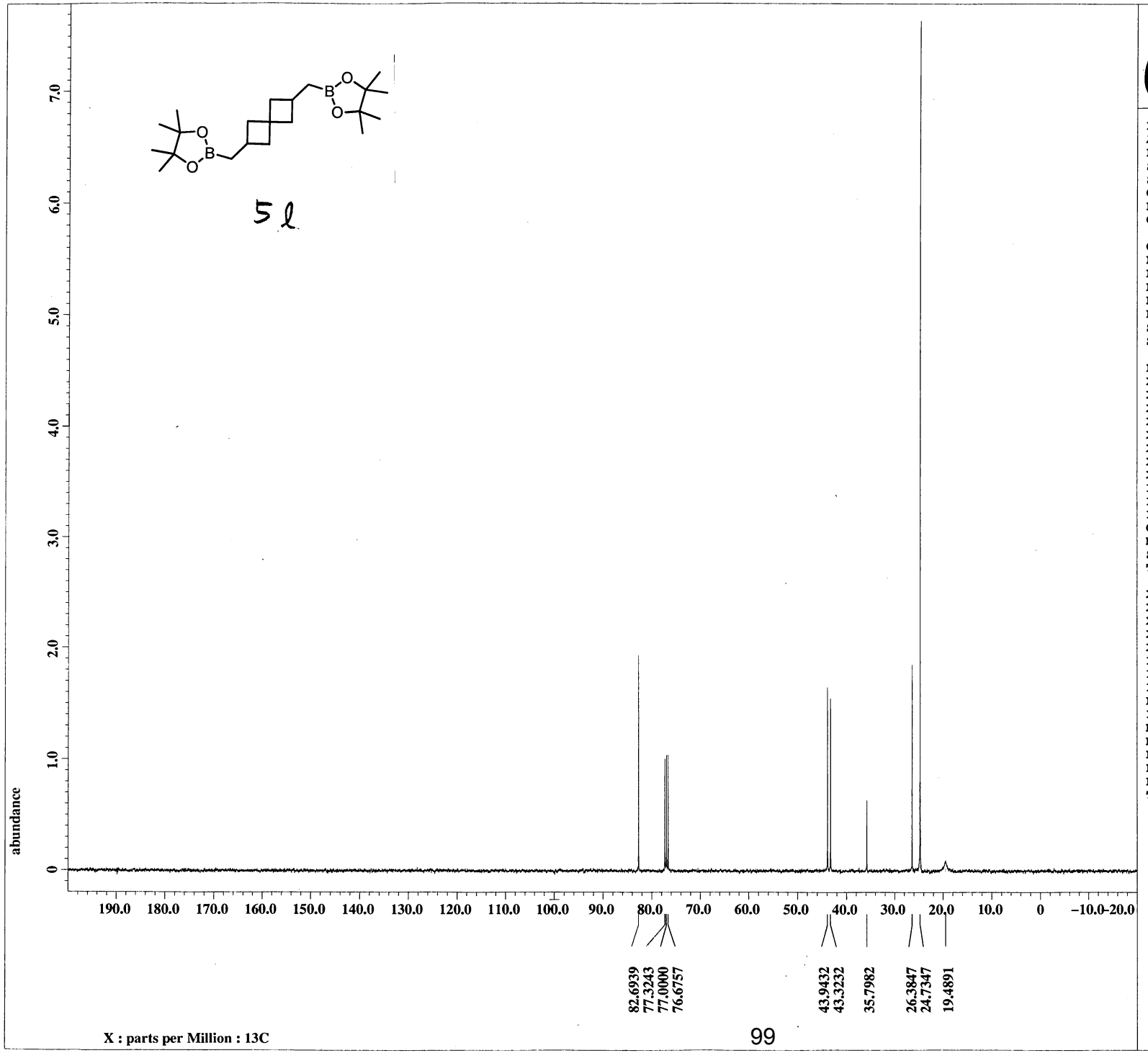
Filename	= KUBO-407-13c-4.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#637944
Solvent	= CHLOROFORM-D
Creation_time	= 9-MAR-2012 16:51:05
Revision_time	= 20-AUG-2012 18:15:42
Current_time	= 20-AUG-2012 18:16:07
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 18.5[dC]



5l

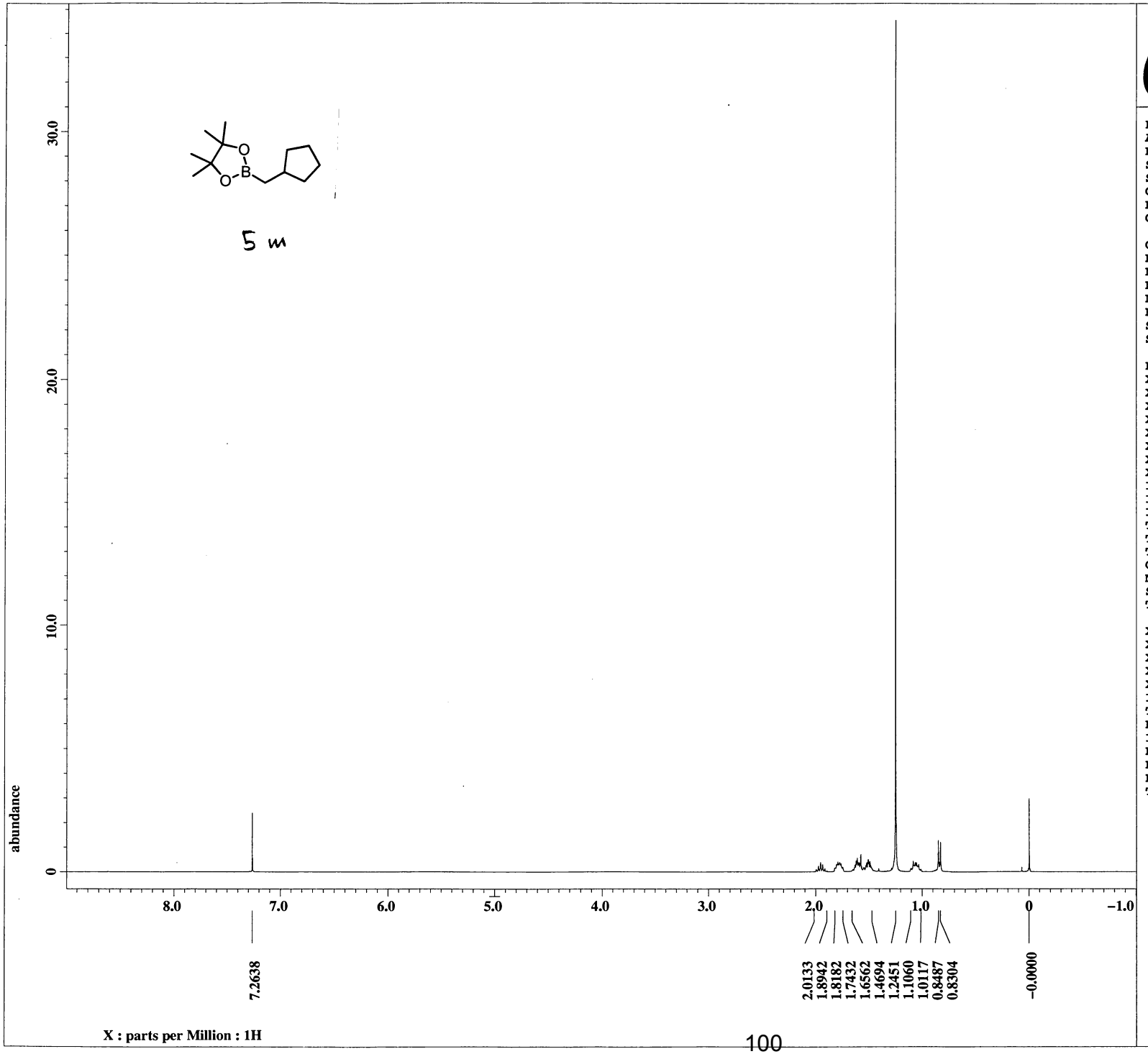
JEOL

Filename	= KUBO-559-PURE-2-3.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#499581
Solvent	= CHLOROFORM-D
Creation_time	= 4-JUL-2012 12:41:57
Revision_time	= 21-SEP-2012 09:34:26
Current_time	= 21-SEP-2012 09:34:40
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 38
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.6[dC]



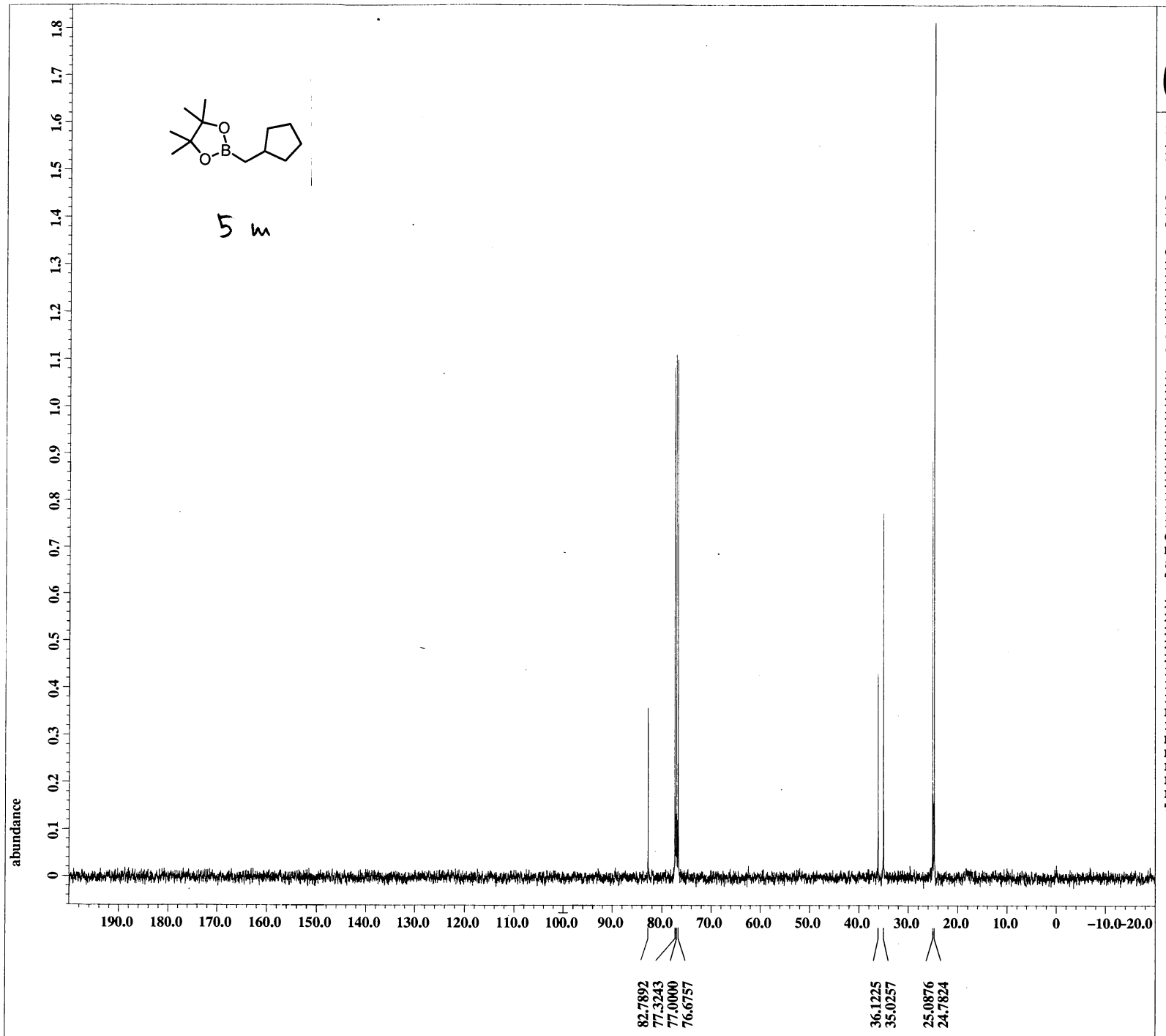
JEOL

Filename	= KUBO-559-13c-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#675503
Solvent	= CHLOROFORM-D
Creation_time	= 4-JUL-2012 17:36:32
Revision_time	= 20-AUG-2012 20:39:46
Current_time	= 20-AUG-2012 20:39:53
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 60
Total_scans	= 60
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.3[dC]

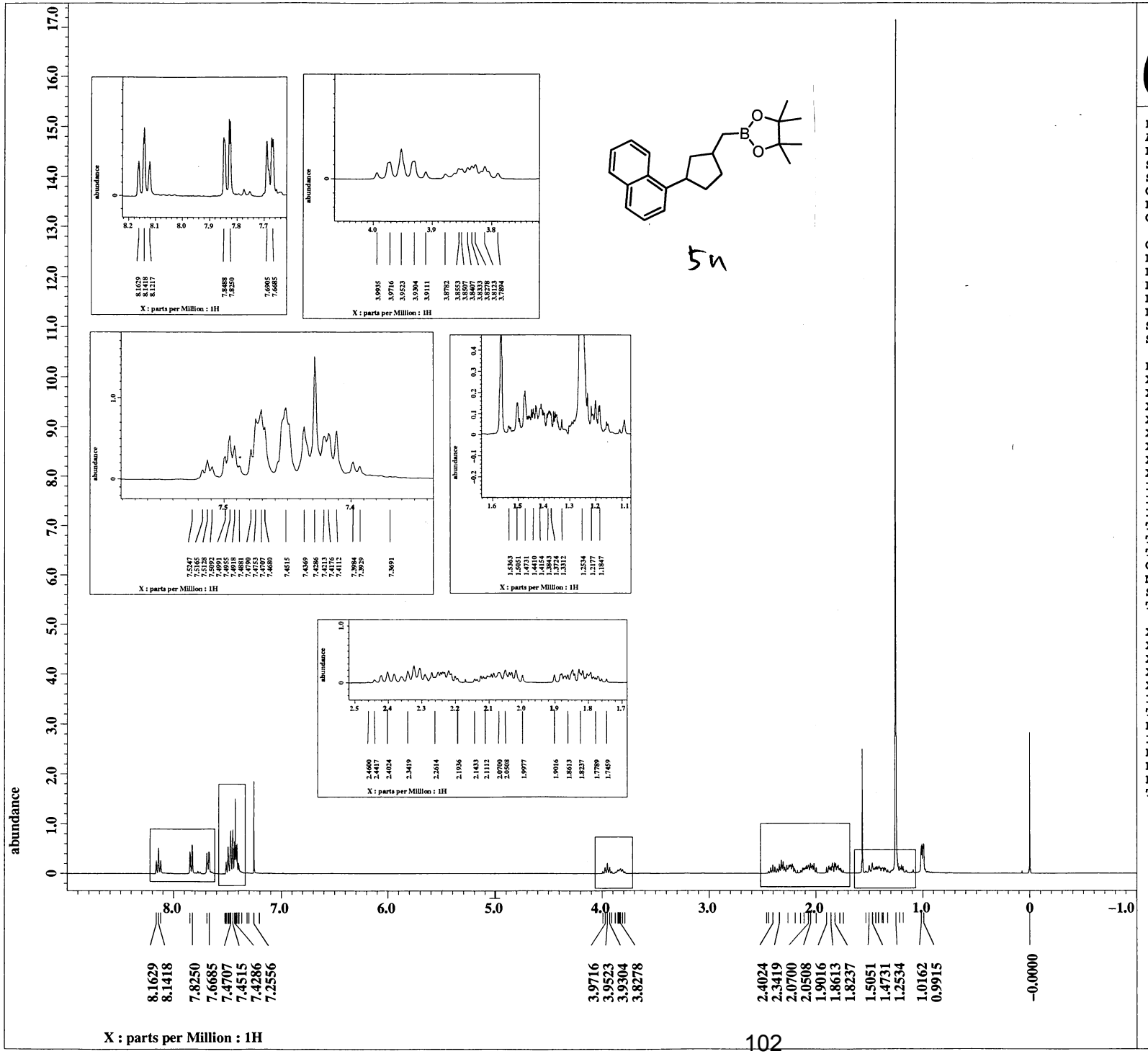


JEOL

Filename	= KUBO-246-pure-11.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#498981
Solvent	= CHLOROFORM-D
Creation_time	= 16-NOV-2011 13:05:07
Revision_time	= 20-SEP-2012 22:28:49
Current_time	= 20-SEP-2012 22:29:02
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 42
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 19.6[dC]



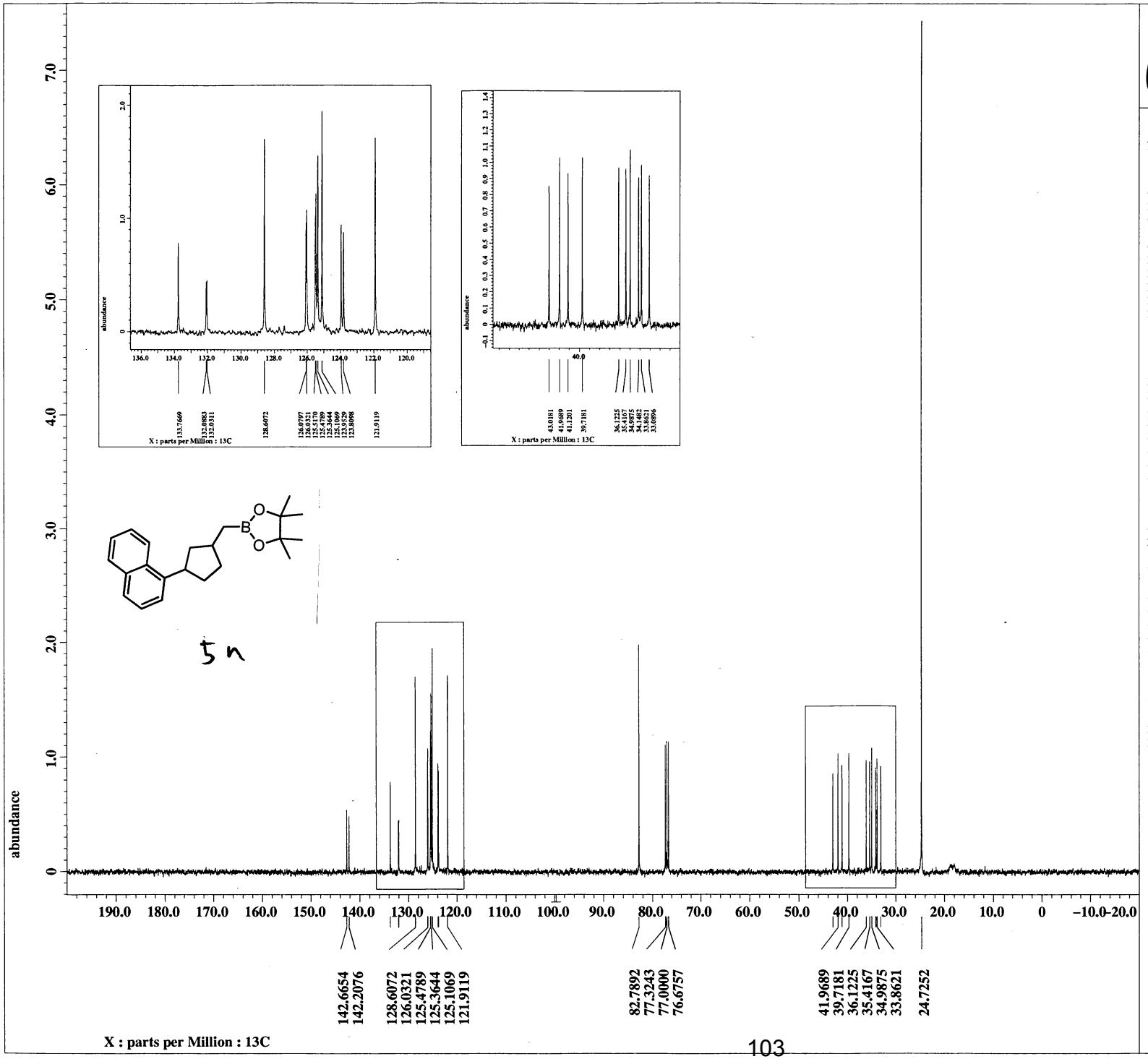
Filename	= KUBO-246-13C-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#628390
Solvent	= CHLOROFORM-D
Creation_time	= 5-FEB-2012 16:40:10
Revision_time	= 20-AUG-2012 17:51:10
Current_time	= 20-AUG-2012 17:51:37
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 100
Total_scans	= 100
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 18[dC]



5n

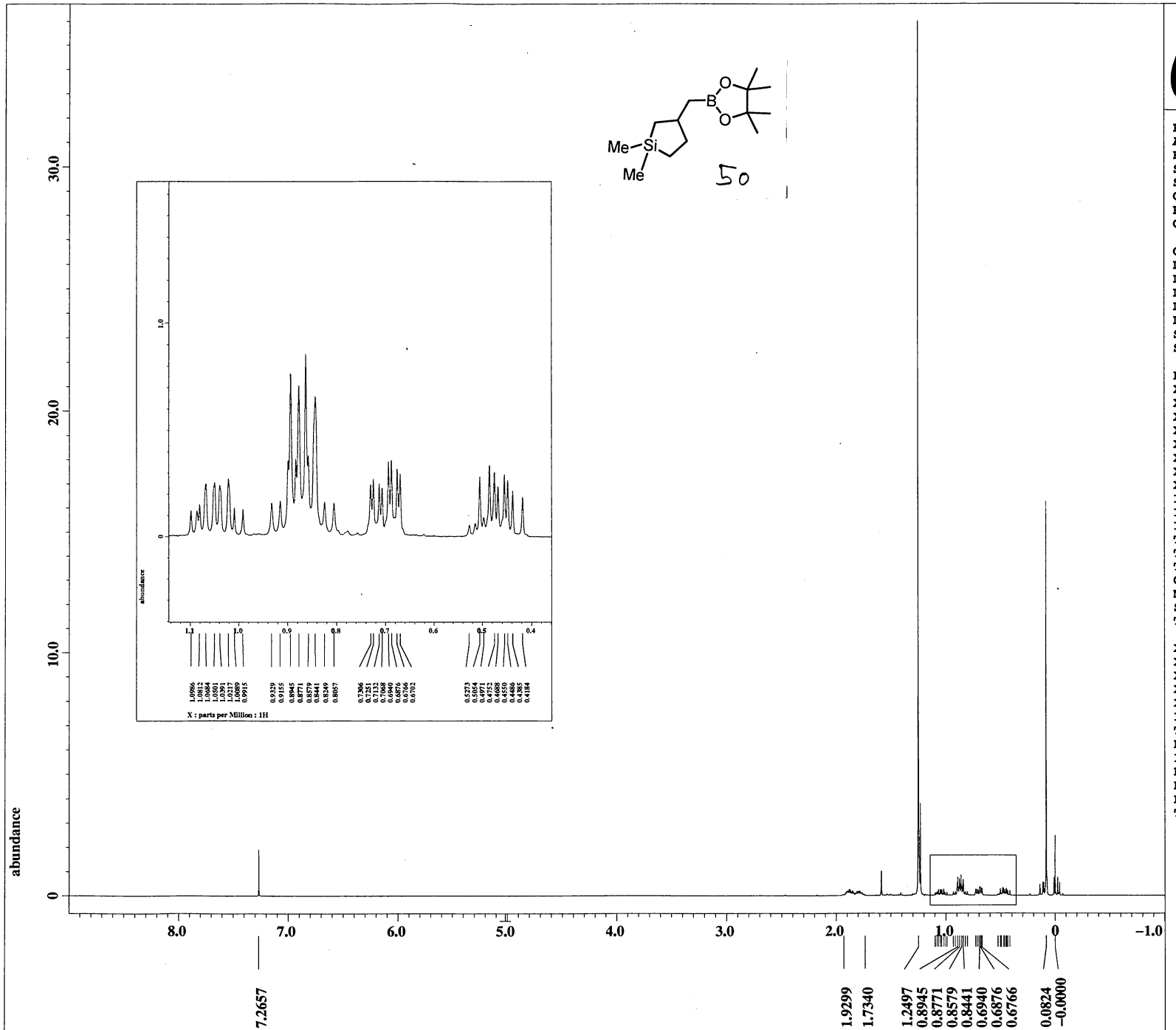
JEOL

Filename	= KUBO-450-pure-2-7.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#331487
Solvent	= CHLOROFORM-D
Creation_time	= 27-APR-2012 08:10:55
Revision_time	= 21-SEP-2012 01:19:01
Current_time	= 21-SEP-2012 01:19:09
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 42
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.6[dC]

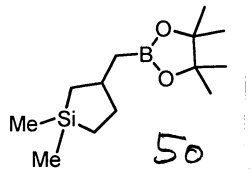


JEOL

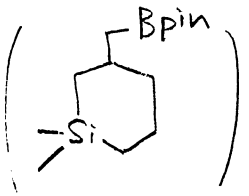
Filename	= KUBO-450-13c-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#340700
Solvent	= CHLOROFORM-D
Creation_time	= 27-APR-2012 08:25:57
Revision_time	= 21-SEP-2012 01:20:04
Current_time	= 21-SEP-2012 01:20:57
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 30
Total_scans	= 30
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.9[dC]



```
Filename          = KUBO-489-pure-2-5.jdf
Author            = element
Experiment        = single_pulse.ex2
Sample_id         = S#728587
Solvent           = CHLOROFORM-D
Creation_time     = 20-MAY-2012 19:09:34
Revision_time     = 21-SEP-2012 09:30:36
Current_time      = 21-SEP-2012 09:31:11

Comment           = single_pulse
Data_format       = 1D COMPLEX
Dim_size          = 16384
Dim_title         = 1H
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 2.78790144[s]
X_domain          = 1H
X_freq            = 391.78655441[MHz]
X_offset          = 5[ppm]
X_points          = 16384
X_prescans        = 1
X_resolution      = 0.35869274[Hz]
X_sweep           = 5.87682181[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Tri_domain        = 1H
Tri_freq          = 391.78655441[MHz]
Tri_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 8
Total_scans       = 8

X_90_width        = 10.8[us]
X_acq_time        = 2.78790144[s]
X_angle           = 45[deg]
X_atn             = 1.9[dB]
X_pulse           = 5.4[us]
Irr_mode          = Off
Tri_mode          = Off
Dante_presat      = FALSE
Initial_wait      = 1[s]
Recvr_gain        = 40
Relaxation_delay  = 5[s]
Repetition_time   = 7.78790144[s]
Temp_get          = 19.5[dC]
```



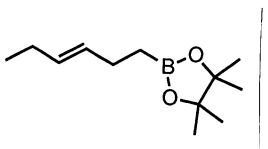
50



x endo-50

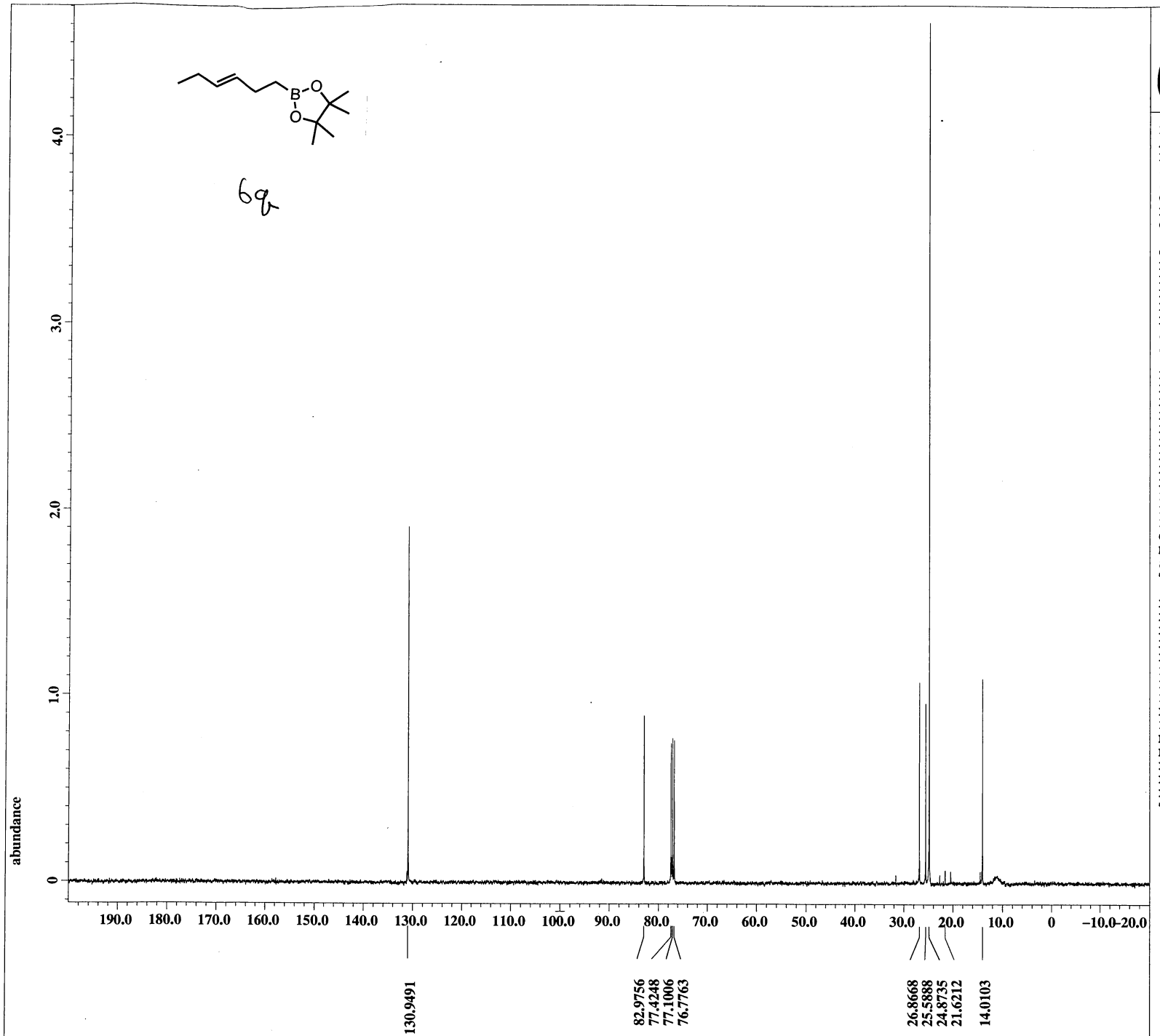
JEOL

Filename	= KUBO-489-13C-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#753444
Solvent	= CHLOROFORM-D
Creation_time	= 20-MAY-2012 19:50:42
Revision_time	= 20-AUG-2012 20:34:21
Current_time	= 20-AUG-2012 20:34:36
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 30
Total_scans	= 30
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 19.7[dC]



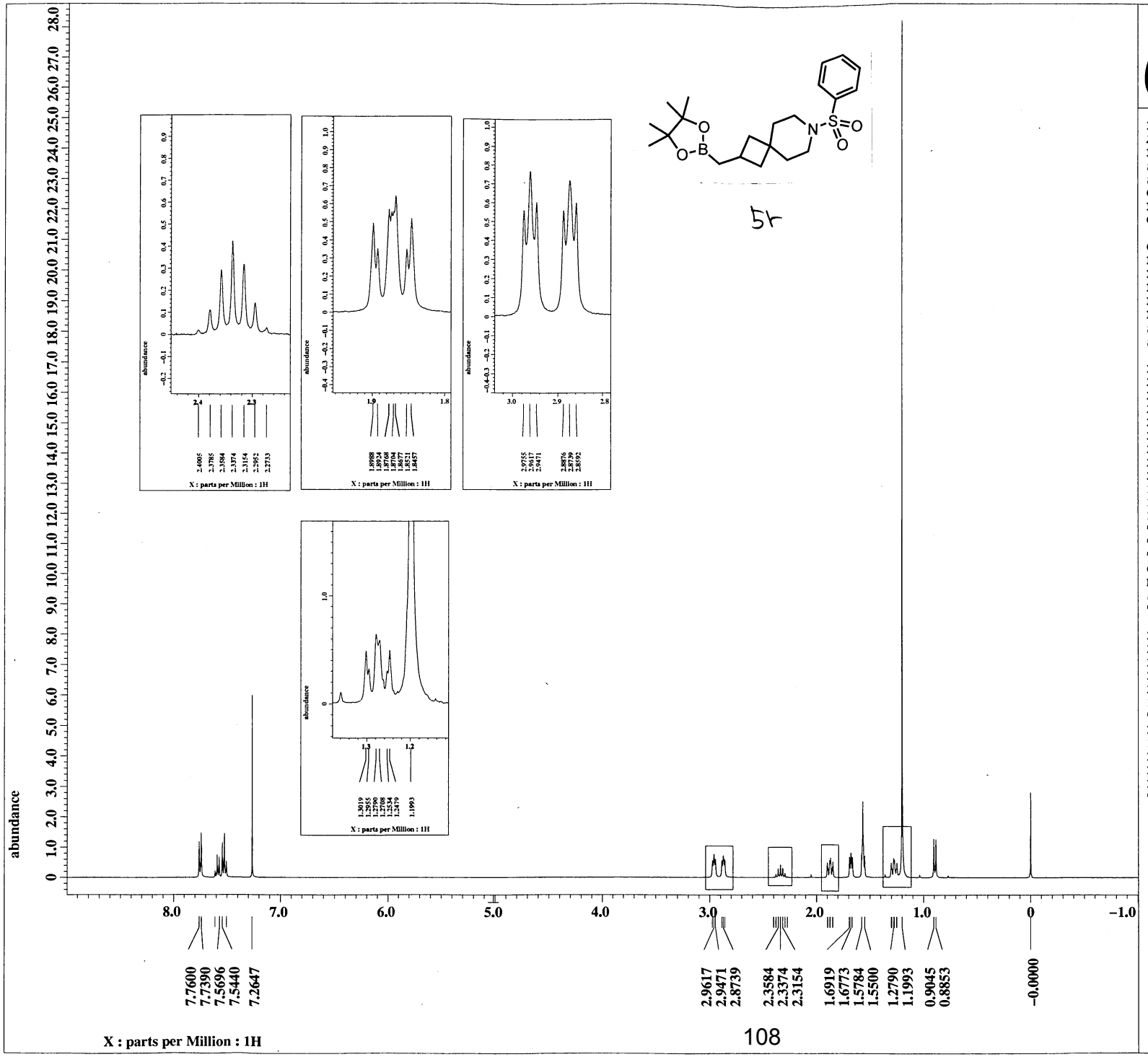
69

Filename	= KUBO-739-pure-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#597581
Solvent	= CHLOROFORM-D
Creation_time	= 29-NOV-2012 15:31:26
Revision_time	= 13-JAN-2013 18:30:31
Current_time	= 13-JAN-2013 18:30:43
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 20.2[dC]



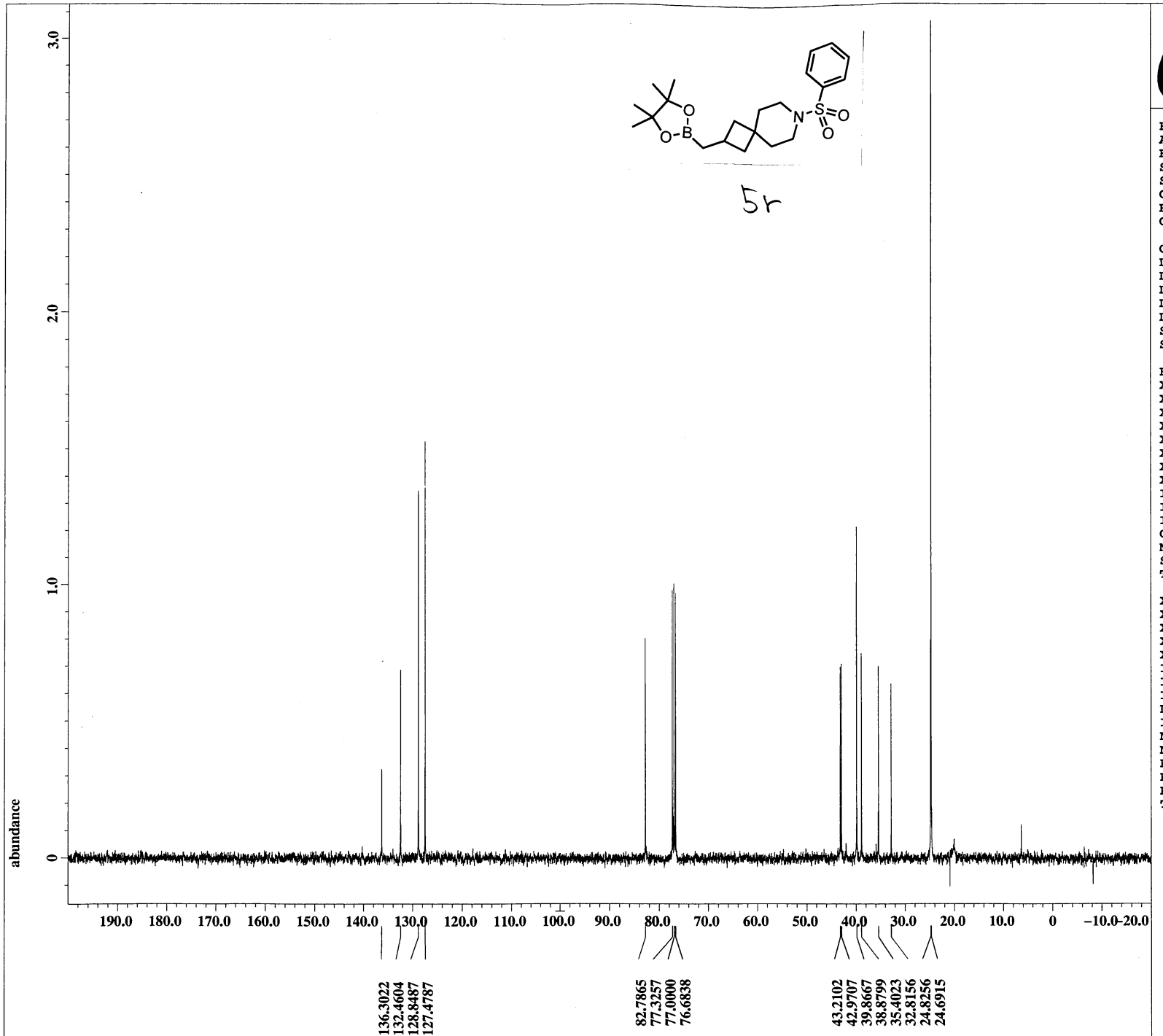
JEOL

Filename	= KUBO-739-13c-2.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#761894
Solvent	= CHLOROFORM-D
Creation_time	= 29-NOV-2012 20:11:04
Revision_time	= 13-JAN-2013 18:32:45
Current_time	= 13-JAN-2013 18:32:59
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 150
Total_scans	= 150
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.5[dC]



5r

Filename	= KUBO-762-pure-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#725013
Solvent	= CHLOROFORM-D
Creation_time	= 10-DEC-2012 19:02:03
Revision_time	= 13-JAN-2013 18:06:42
Current_time	= 13-JAN-2013 18:06:46
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 52
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 19.2[dC]

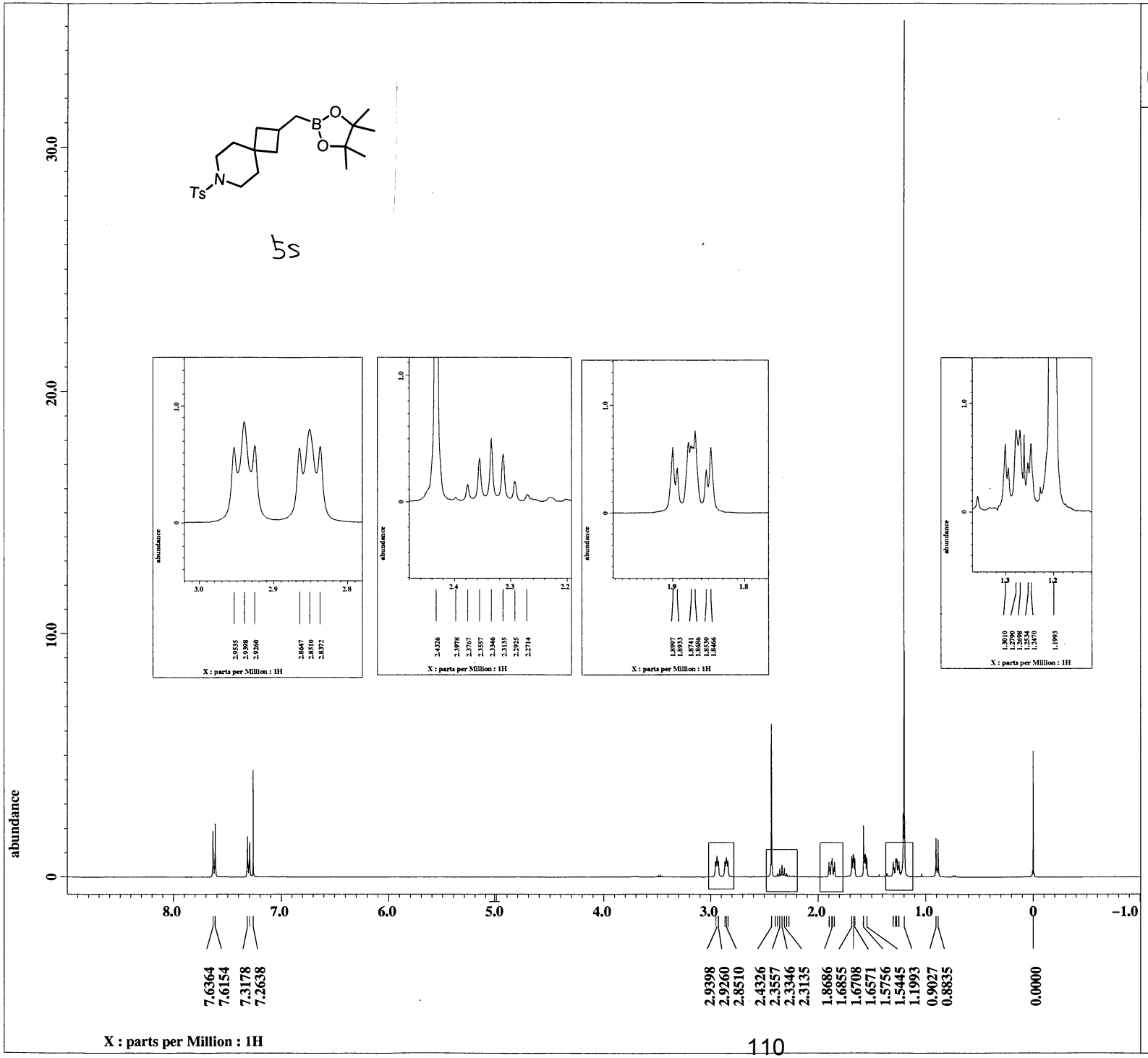


Filename = KUBO-762-13c-3.jdf
Author = element
Experiment = single_pulse_dec
Sample_id = S#394427
Solvent = CHLOROFORM-D
Creation_time = 15-JAN-2013 10:31:37
Revision_time = 15-JAN-2013 11:08:54
Current_time = 15-JAN-2013 11:09:02

Comment = single pulse decouple
Data_format = 1D COMPLEX
Dim_size = 26214
Dim_title = 13C
Dim_units = [ppm]
Dimensions = X
Site = ECX 400P
Spectrometer = DELTA2_NMR

Field_strength = 9.2982153[T] (400[MHz
X_acq_duration = 1.048576[s]
X_domain = 13C
X_freq = 99.54517646[MHz]
X_offset = 100[ppm]
X_points = 32768
X_prescans = 4
X_resolution = 0.95367432[Hz]
X_sweep = 31.25[kHz]
Irr_domain = 1H
Irr_freq = 395.88430144[MHz]
Irr_offset = 5[ppm]
Clipped = TRUE
Mod_return = 1
Scans = 50
Total_scans = 50

X_90_width = 9.5[us]
X_acq_time = 1.048576[s]
X_angle = 30[deg]
X_atn = 3.4[dB]
X_pulse = 3.16666667[us]
Irr_atn_dec = 19.607[dB]
Irr_atn_noe = 19.607[dB]
Irr_noise = WALTZ
Decoupling = TRUE
Initial_wait = 1[s]
Noe = TRUE
Noe_time = 2[s]
Recvr_gain = 58
Relaxation_delay = 2[s]
Repetition_time = 3.048576[s]
Temp_get = 400.9[dC]

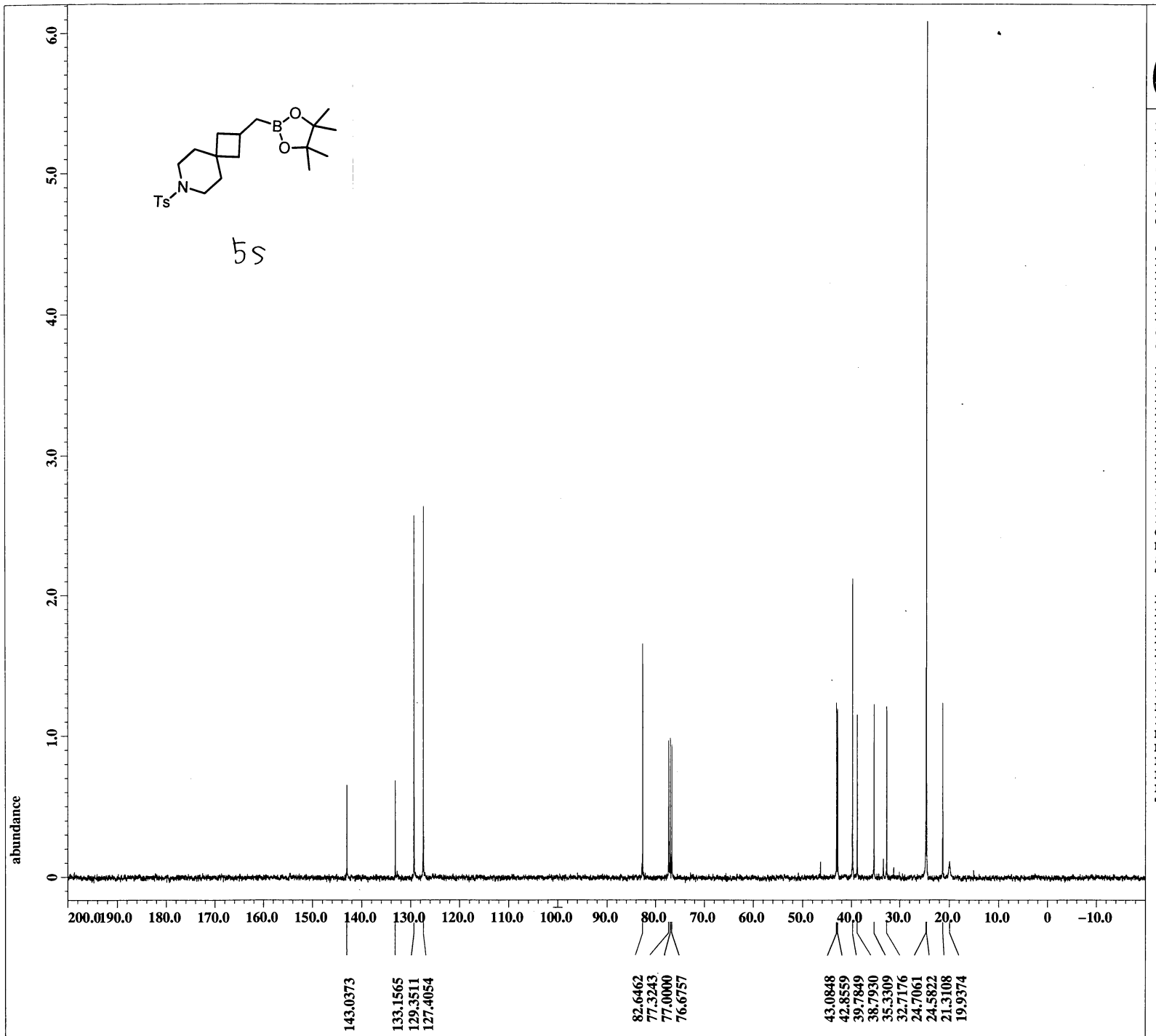


Filename = KUBO-558-pure-5.jdf
Author = element
Experiment = single_pulse.ex2
Sample_id = S#748987
Solvent = CHLOROFORM-D
Creation_time = 1-JUL-2012 19:37:42
Revision_time = 21-SEP-2012 10:05:06
Current_time = 21-SEP-2012 10:05:13

Comment = single_pulse
Data_format = 1D COMPLEX
Dim_size = 16384
Dim_title = 1H
Dim_units = [ppm]
Dimensions = X
Site = ECS 400
Spectrometer = JNM-ECS400

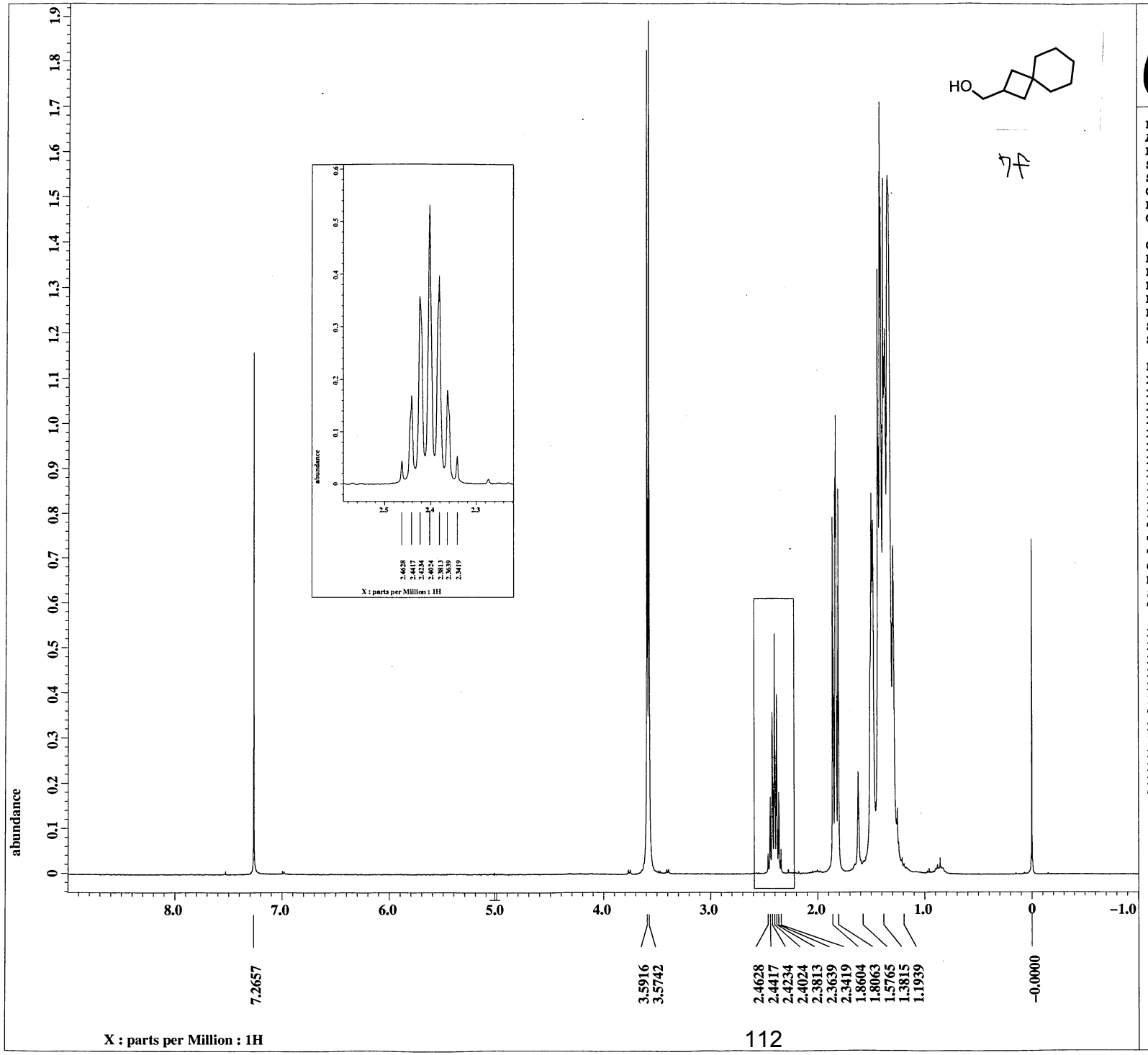
Field_strength = 9.20197068[T] (390[MH
X_acq_duration = 2.78790144[s]
X_domain = 1H
X_freq = 391.78655441[MHz]
X_offset = 5[ppm]
X_points = 16384
X_prescans = 1
X_resolution = 0.35869274[Hz]
X_sweep = 5.87682181[kHz]
Irr_domain = 1H
Irr_freq = 391.78655441[MHz]
Irr_offset = 5[ppm]
Tri_domain = 1H
Tri_freq = 391.78655441[MHz]
Tri_offset = 5[ppm]
Clipped = FALSE
Mod_return = 1
Scans = 8
Total_scans = 8

X_90_width = 10.8[us]
X_acq_time = 2.78790144[s]
X_angle = 45[deg]
X_atn = 1.9[dB]
X_pulse = 5.4[us]
Irr_mode = Off
Tri_mode = Off
Dante_presat = FALSE
Initial_wait = 1[s]
Recvr_gain = 48
Relaxation_delay = 5[s]
Repetition_time = 7.78790144[s]
Temp_get = 20.6[dC]



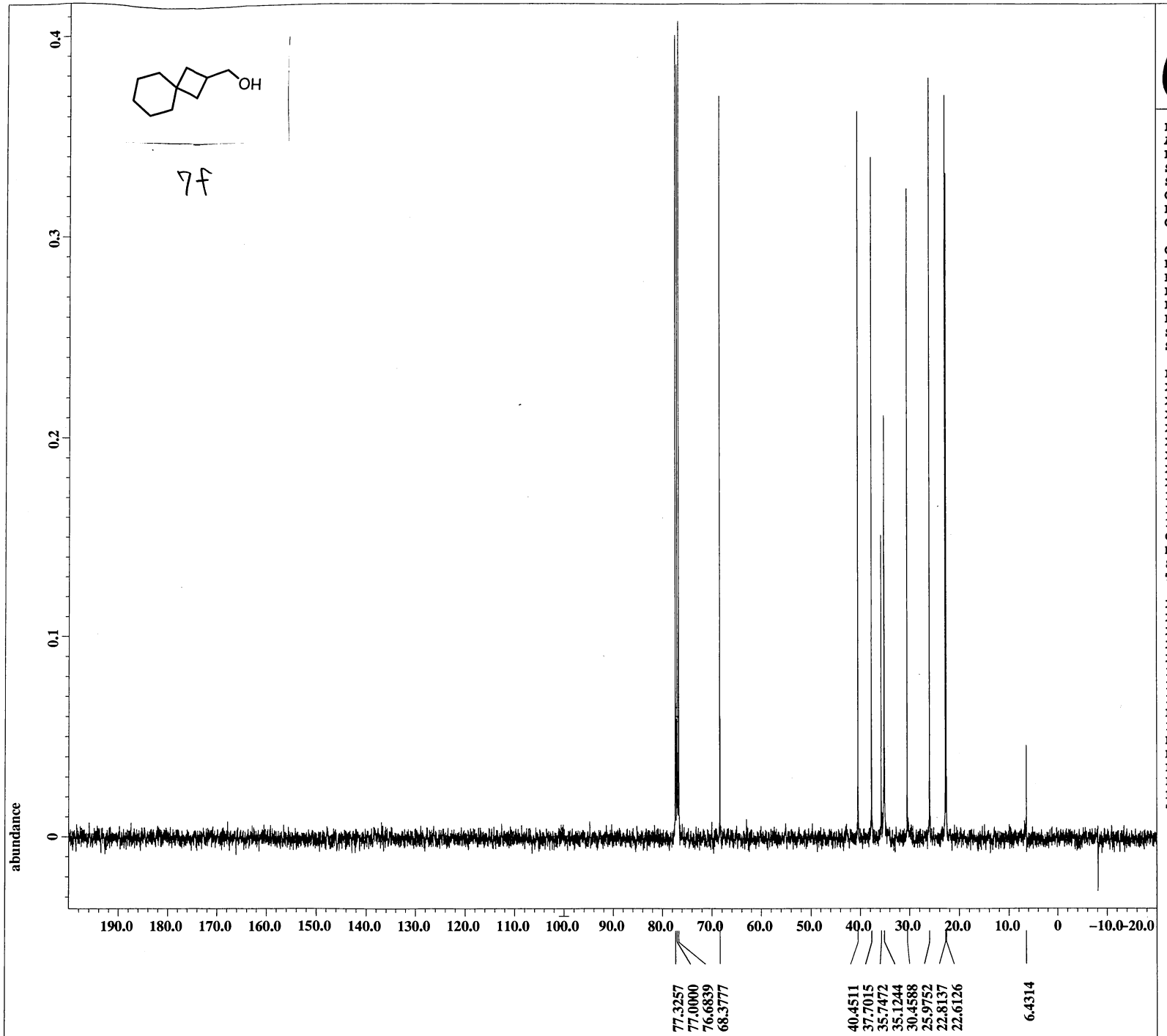
JEOL

Filename	= KUBO-558-13c-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#809288
Solvent	= CHLOROFORM-D
Creation_time	= 1-JUL-2012 21:19:08
Revision_time	= 21-SEP-2012 09:19:00
Current_time	= 21-SEP-2012 09:19:22
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 40
Total_scans	= 40
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 20.8[dC]



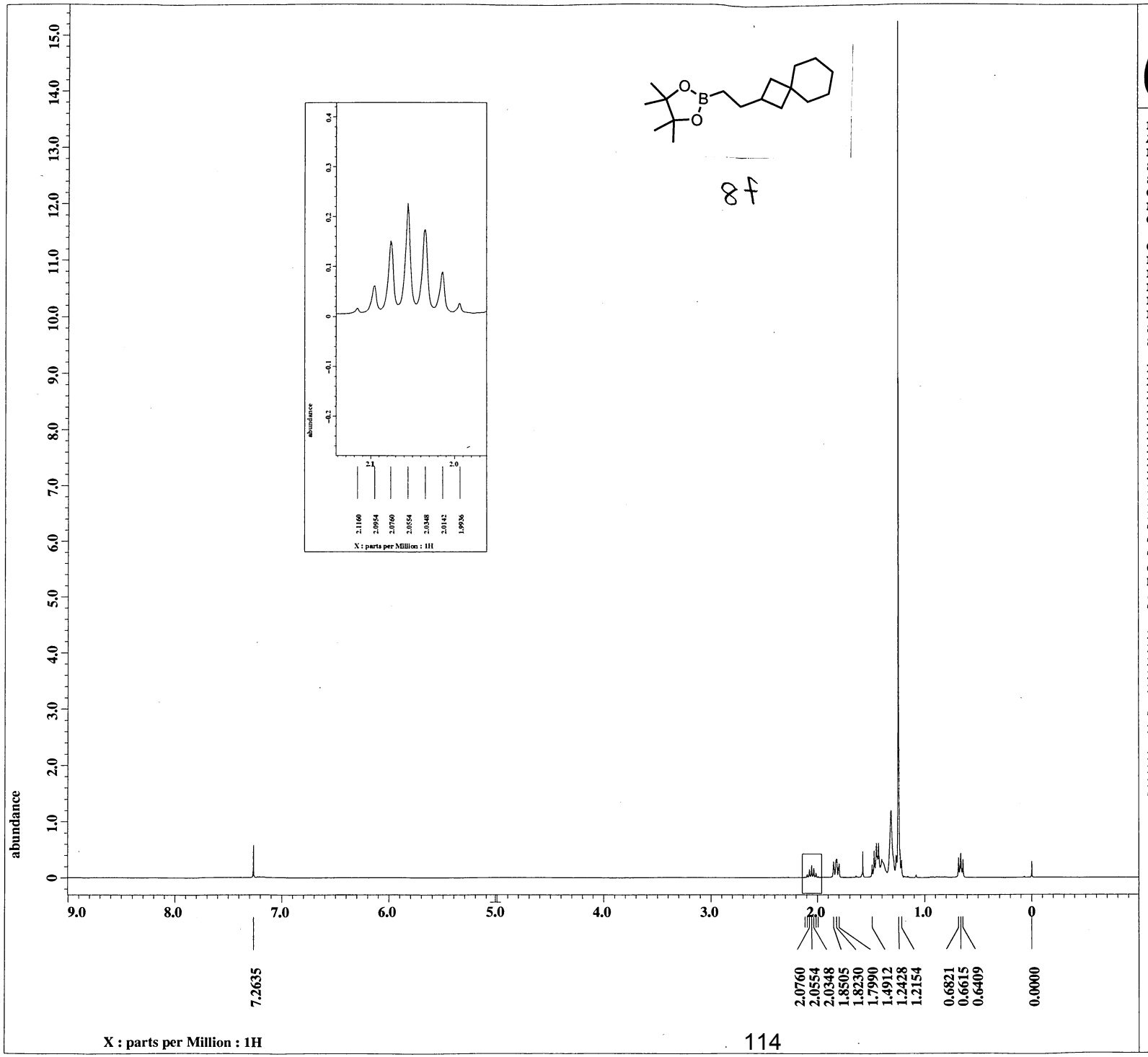
JEOL

Filename	= KUBO-758-pure-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#332425
Solvent	= CHLOROFORM-D
Creation_time	= 8-DEC-2012 08:08:17
Revision_time	= 13-JAN-2013 17:31:13
Current_time	= 13-JAN-2013 17:31:15
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 40
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 19.2[dC]



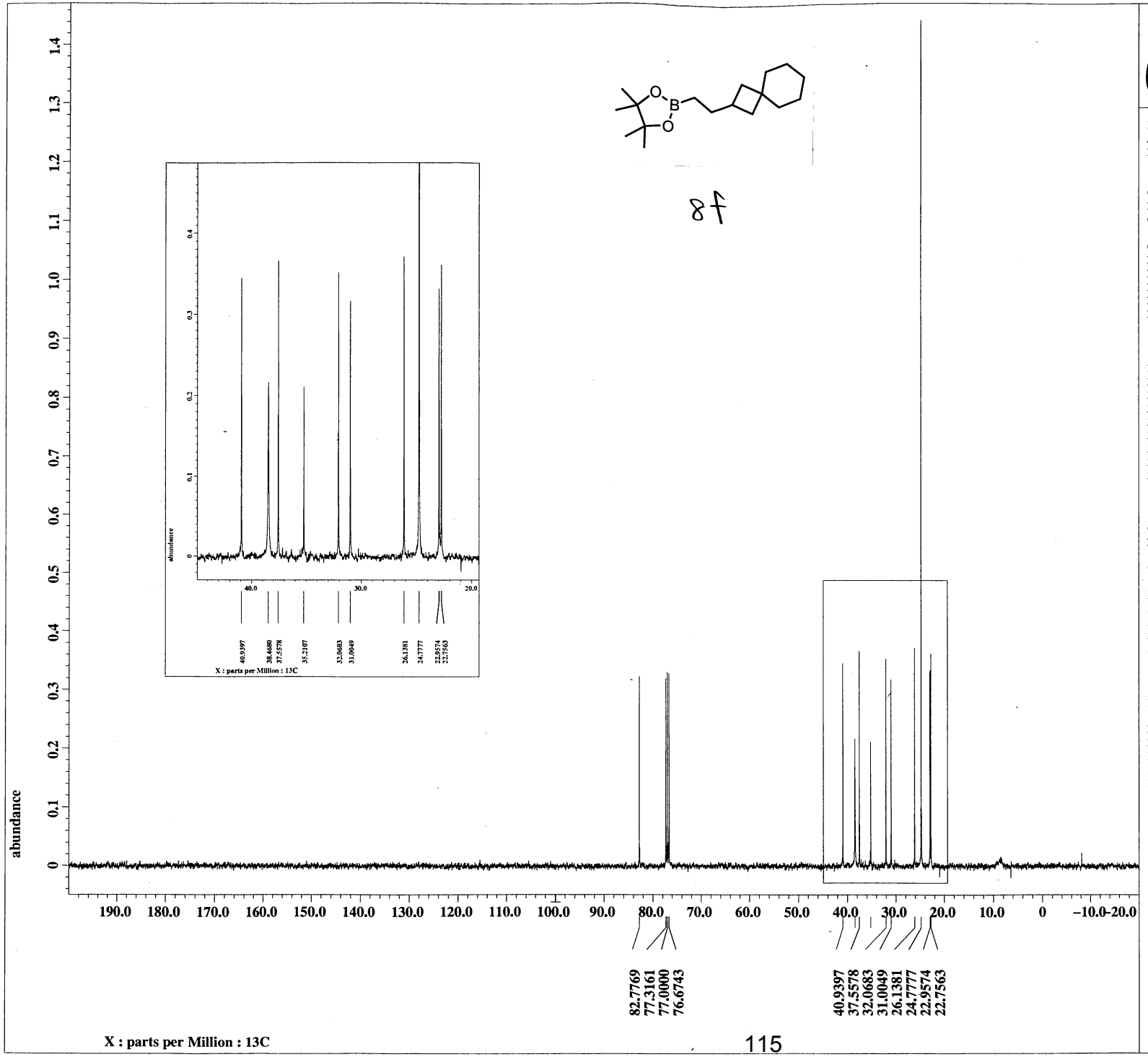
JEOL

```
Filename          = KUBO-758-13c-5.jdf
Author            = element
Experiment        = single_pulse_dec
Sample_id         = S#343619
Solvent           = CHLOROFORM-D
Creation_time     =  8-DEC-2012 09:13:00
Revision_time     = 13-JAN-2013 17:32:37
Current_time      = 13-JAN-2013 17:32:47

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECX 400P
Spectrometer      = DELTA2_NMR

Field_strength    = 9.2982153[T] (400[MHz
X_acq_duration    = 1.048576[s]
X_domain          = 13C
X_freq            = 99.54517646[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.95367432[Hz]
X_sweep           = 31.25[kHz]
Irr_domain        = 1H
Irr_freq          = 395.88430144[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 150
Total_scans       = 150

X_90_width        = 9.5[us]
X_acq_time        = 1.048576[s]
X_angle           = 30[deg]
X_atn             = 3.4[dB]
X_pulse           = 3.16666667[us]
Irr_atn_dec       = 19.607[dB]
Irr_atn_noe       = 19.607[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 50
Relaxation_delay  = 2[s]
Repetition_time   = 3.048576[s]
Temp_get          = 401.6[dC]
```



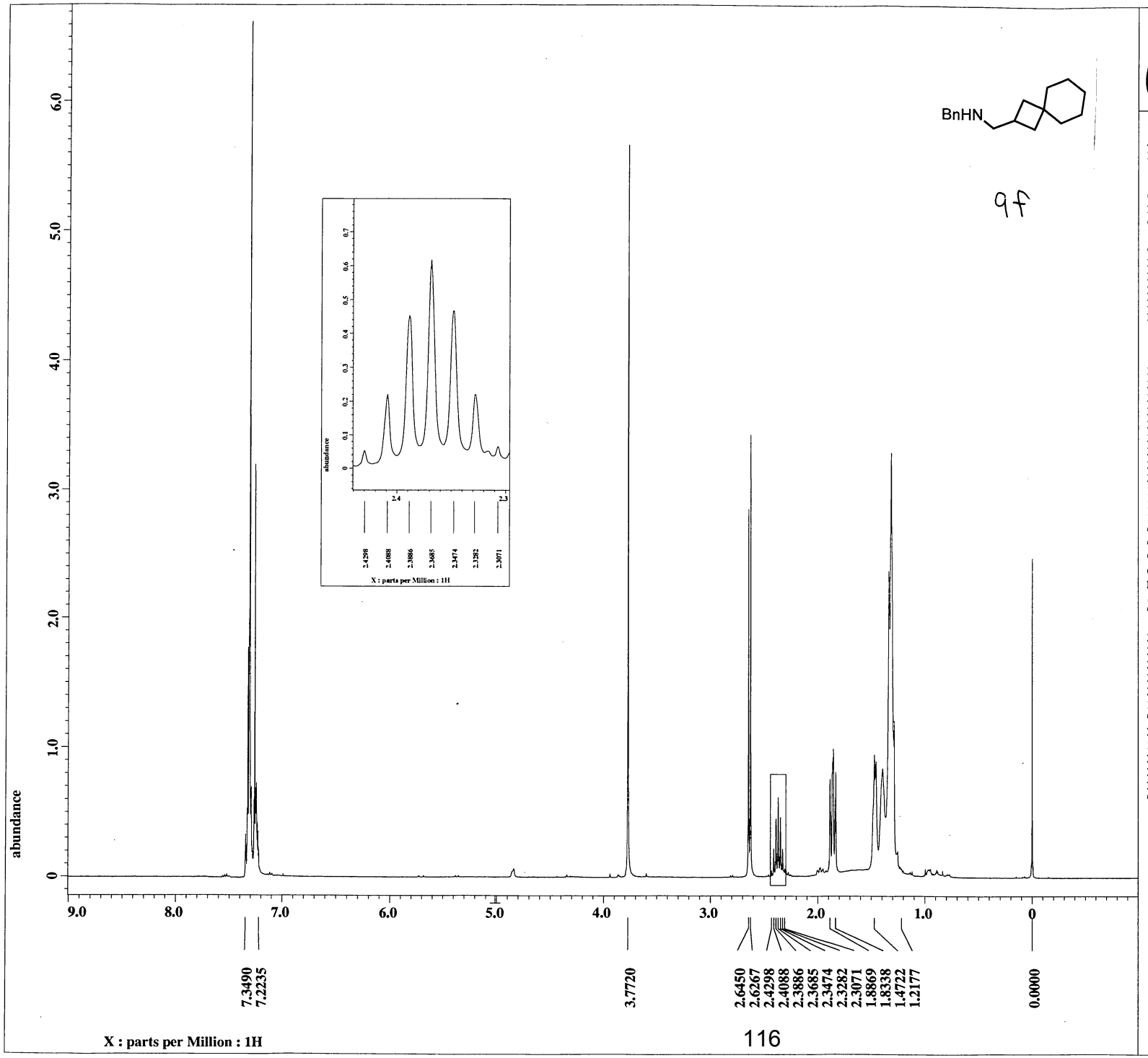
8f

Filename	= KUBO-765-pure-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#684404
Solvent	= CHLOROFORM-D
Creation_time	= 13-DEC-2012 18:33:31
Revision_time	= 13-JAN-2013 17:38:51
Current_time	= 13-JAN-2013 17:39:41
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 2.20725248[s]
X_domain	= 1H
X_freq	= 395.88430144[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.45305193[Hz]
X_sweep	= 7.42280285[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 395.88430144[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13.5[us]
X_acq_time	= 2.20725248[s]
X_angle	= 45[deg]
X_atn	= 1[dB]
X_pulse	= 6.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 34
Relaxation_delay	= 5[s]
Repetition_time	= 7.20725248[s]
Temp_get	= 402.6[dC]



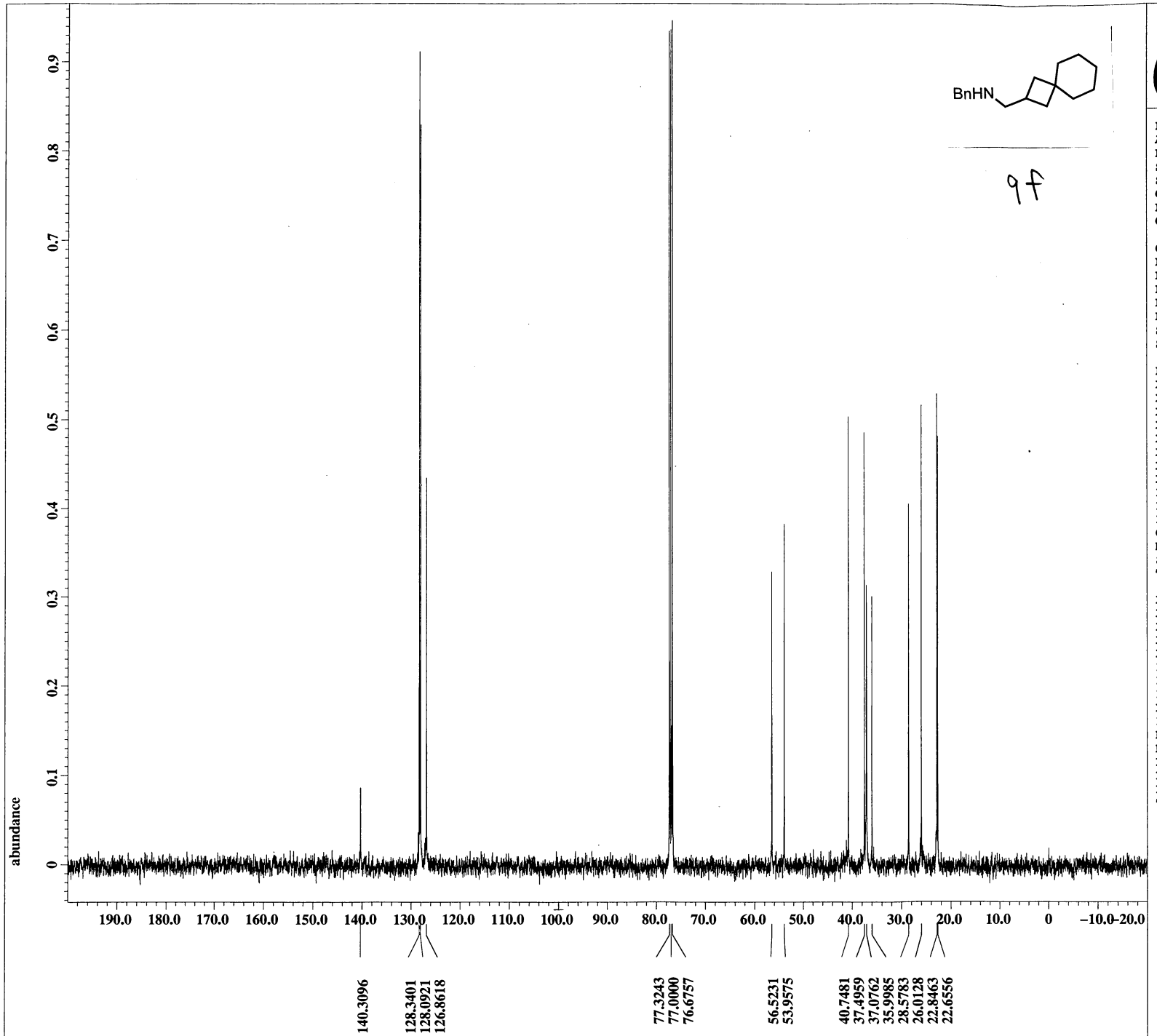
81

Filename	= KUBO-765-13c-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#691118
Solvent	= CHLOROFORM-D
Creation_time	= 13-DEC-2012 18:47:33
Revision_time	= 14-JAN-2013 17:40:29
Current_time	= 14-JAN-2013 17:40:58
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 1.048576[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95367432[Hz]
X_sweep	= 31.25[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 80
Total_scans	= 80
X_90_width	= 9.5[us]
X_acq_time	= 1.048576[s]
X_angle	= 30[deg]
X_atn	= 3.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 19.607[dB]
Irr_atn_noe	= 19.607[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 48
Relaxation_delay	= 2[s]
Repetition_time	= 3.048576[s]
Temp_get	= 402.5[dC]



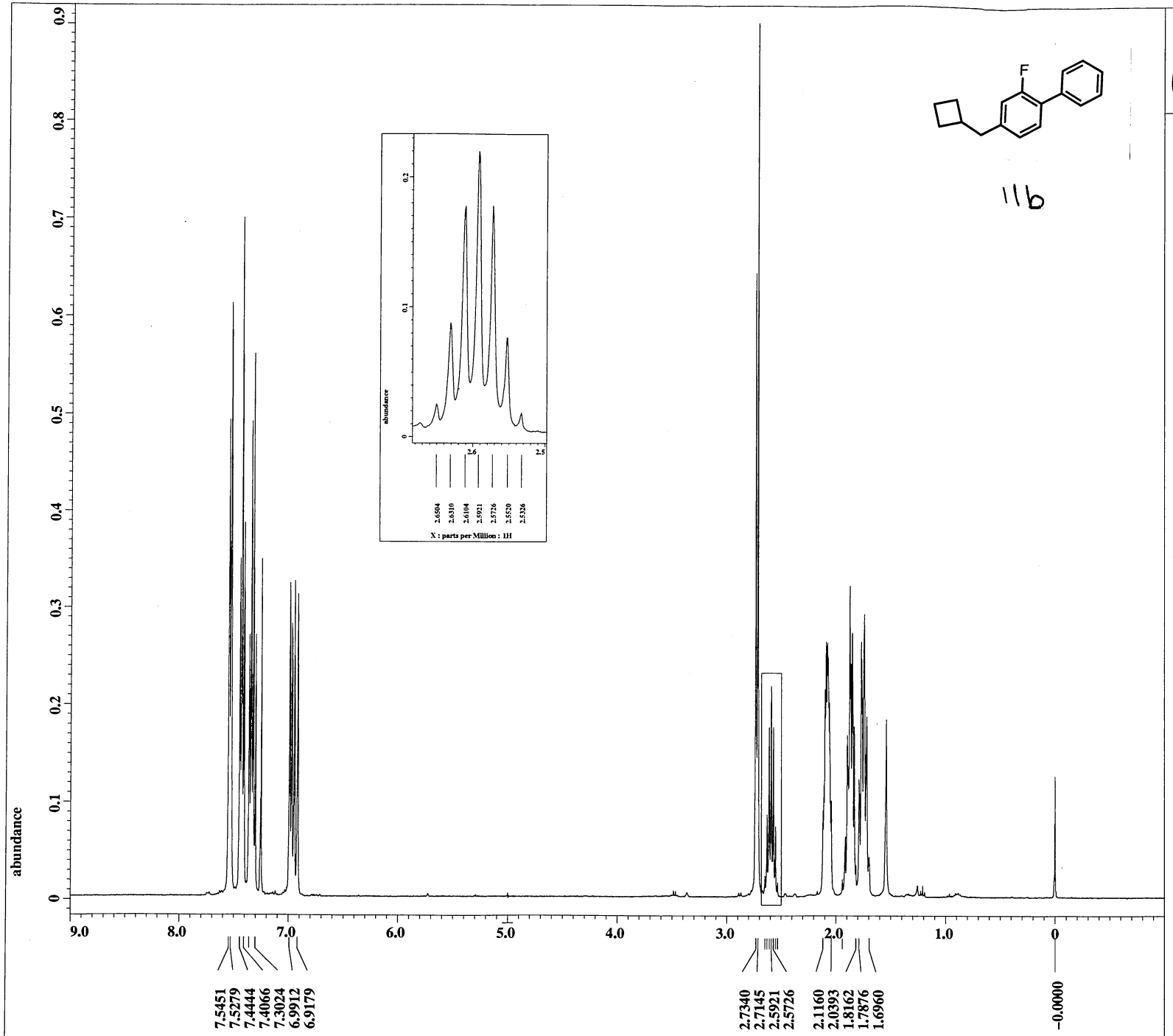
JEOL

Filename	= KUBO-774-pure-2-8.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#816235
Solvent	= CHLOROFORM-D
Creation_time	= 30-DEC-2012 21:31:18
Revision_time	= 16-JAN-2013 09:59:44
Current_time	= 16-JAN-2013 10:00:47
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 46
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 18.1[dC]



JEOL

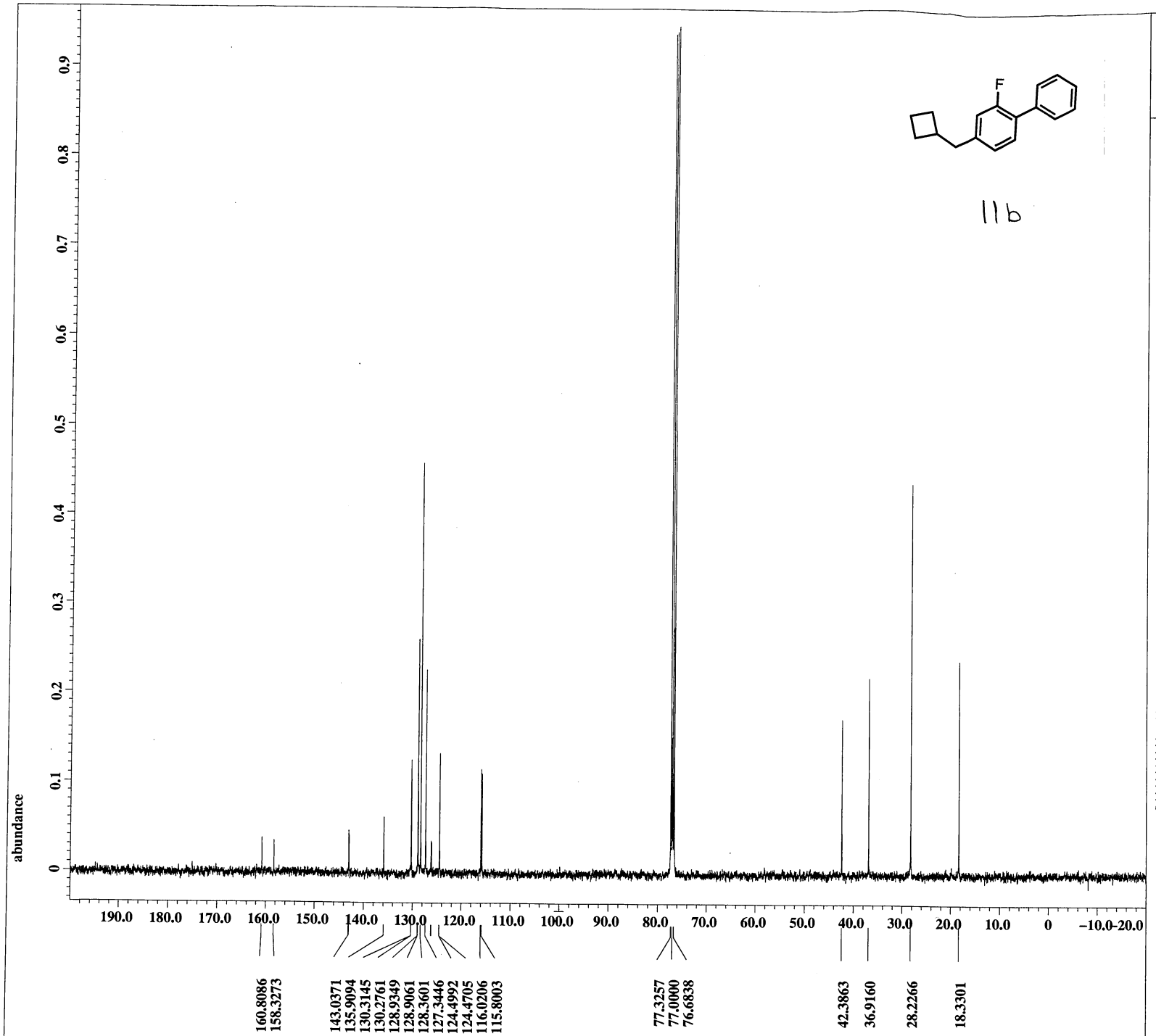
```
Filename          = KUBO-774-13C-3.jdf
Author            = element
Experiment        = single_pulse_dec
Sample_id         = S#823335
Solvent           = CHLOROFORM-D
Creation_time     = 30-DEC-2012 21:49:13
Revision_time     = 14-JAN-2013 16:42:57
Current_time      = 14-JAN-2013 16:43:11

Comment           = single pulse decouple
Data_format       = 1D COMPLEX
Dim_size          = 26214
Dim_title         = 13C
Dim_units         = [ppm]
Dimensions        = X
Site              = ECS 400
Spectrometer      = JNM-ECS400

Field_strength    = 9.20197068[T] (390[MH
X_acq_duration    = 1.06430464[s]
X_domain          = 13C
X_freq            = 98.51479726[MHz]
X_offset          = 100[ppm]
X_points          = 32768
X_prescans        = 4
X_resolution      = 0.93958061[Hz]
X_sweep           = 30.78817734[kHz]
Irr_domain        = 1H
Irr_freq          = 391.78655441[MHz]
Irr_offset        = 5[ppm]
Clipped           = FALSE
Mod_return        = 1
Scans             = 150
Total_scans       = 150

X_90_width        = 8.15[us]
X_acq_time        = 1.06430464[s]
X_angle           = 30[deg]
X_atn             = 4.9[dB]
X_pulse           = 2.71666667[us]
Irr_atn_dec       = 22.445[dB]
Irr_atn_noe       = 22.445[dB]
Irr_noise         = WALTZ
Decoupling        = TRUE
Initial_wait      = 1[s]
Noe               = TRUE
Noe_time          = 2[s]
Recvr_gain        = 60
Relaxation_delay  = 2[s]
Repetition_time   = 3.06430464[s]
Temp_get          = 18.4[dC]
```



11b

JEOL

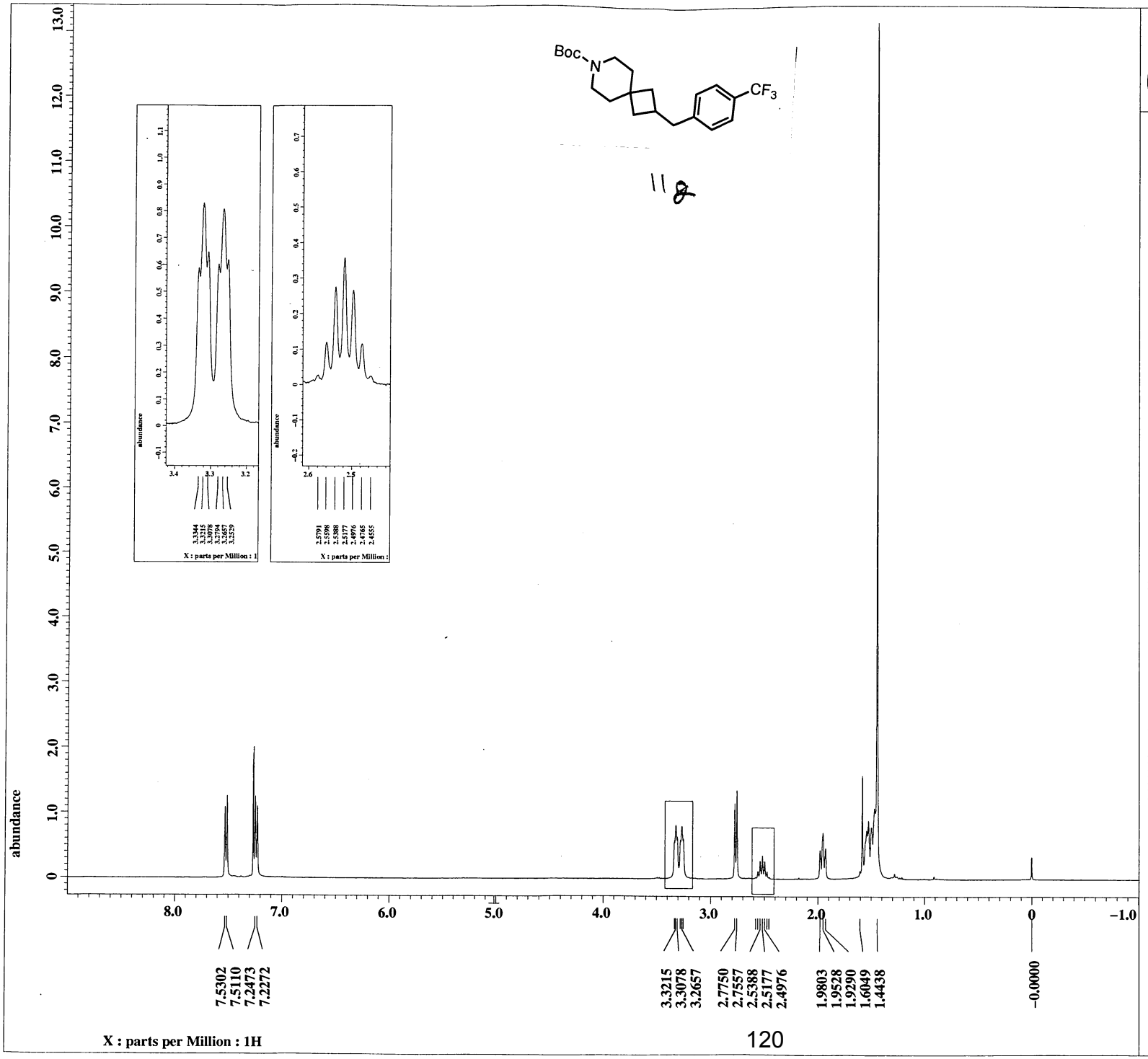
Filename	= KUBO-766-pure-2-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#690189
Solvent	= CHLOROFORM-D
Creation_time	= 16-DEC-2012 18:43:10
Revision_time	= 14-JAN-2013 15:35:47
Current_time	= 14-JAN-2013 15:37:06
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 13107
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 2.20725248[s]
X_domain	= 1H
X_freq	= 395.88430144[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.45305193[Hz]
X_sweep	= 7.42280285[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 395.88430144[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 13.5[us]
X_acq_time	= 2.20725248[s]
X_angle	= 45[deg]
X_atn	= 1[dB]
X_pulse	= 6.75[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 34
Relaxation_delay	= 5[s]
Repetition_time	= 7.20725248[s]
Temp_get	= 402.2[dC]



11b

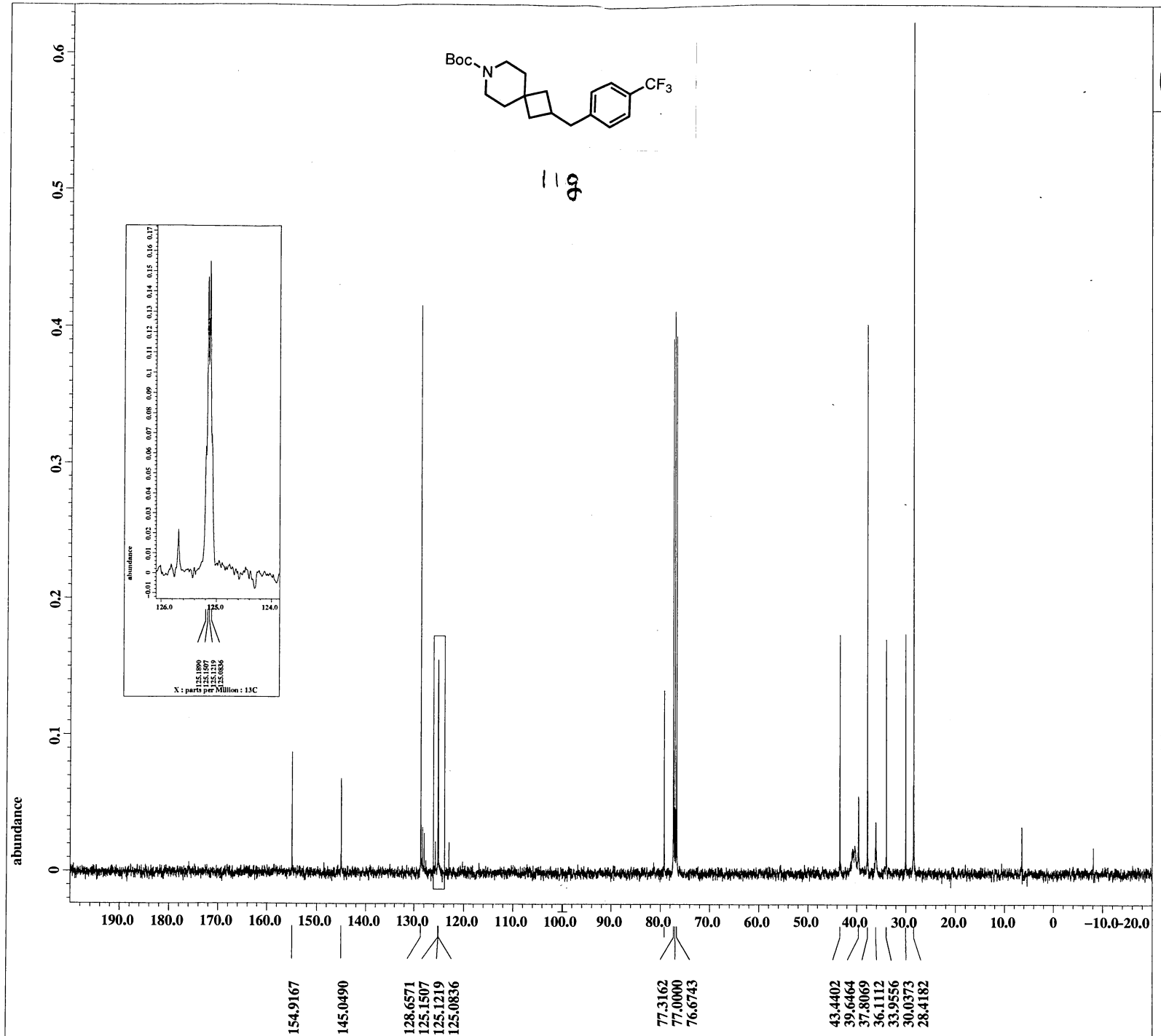
JEOL

Filename	= KUBO-766-13c-3.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#691829
Solvent	= CHLOROFORM-D
Creation_time	= 16-DEC-2012 19:25:26
Revision_time	= 14-JAN-2013 15:37:56
Current_time	= 14-JAN-2013 15:38:10
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 1.048576[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95367432[Hz]
X_sweep	= 31.25[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= TRUE
Mod_return	= 1
Scans	= 800
Total_scans	= 800
X_90_width	= 9.5[us]
X_acq_time	= 1.048576[s]
X_angle	= 30[deg]
X_atn	= 3.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 19.607[dB]
Irr_atn_noe	= 19.607[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 58
Relaxation_delay	= 2[s]
Repetition_time	= 3.048576[s]
Temp_get	= 402.2[dC]



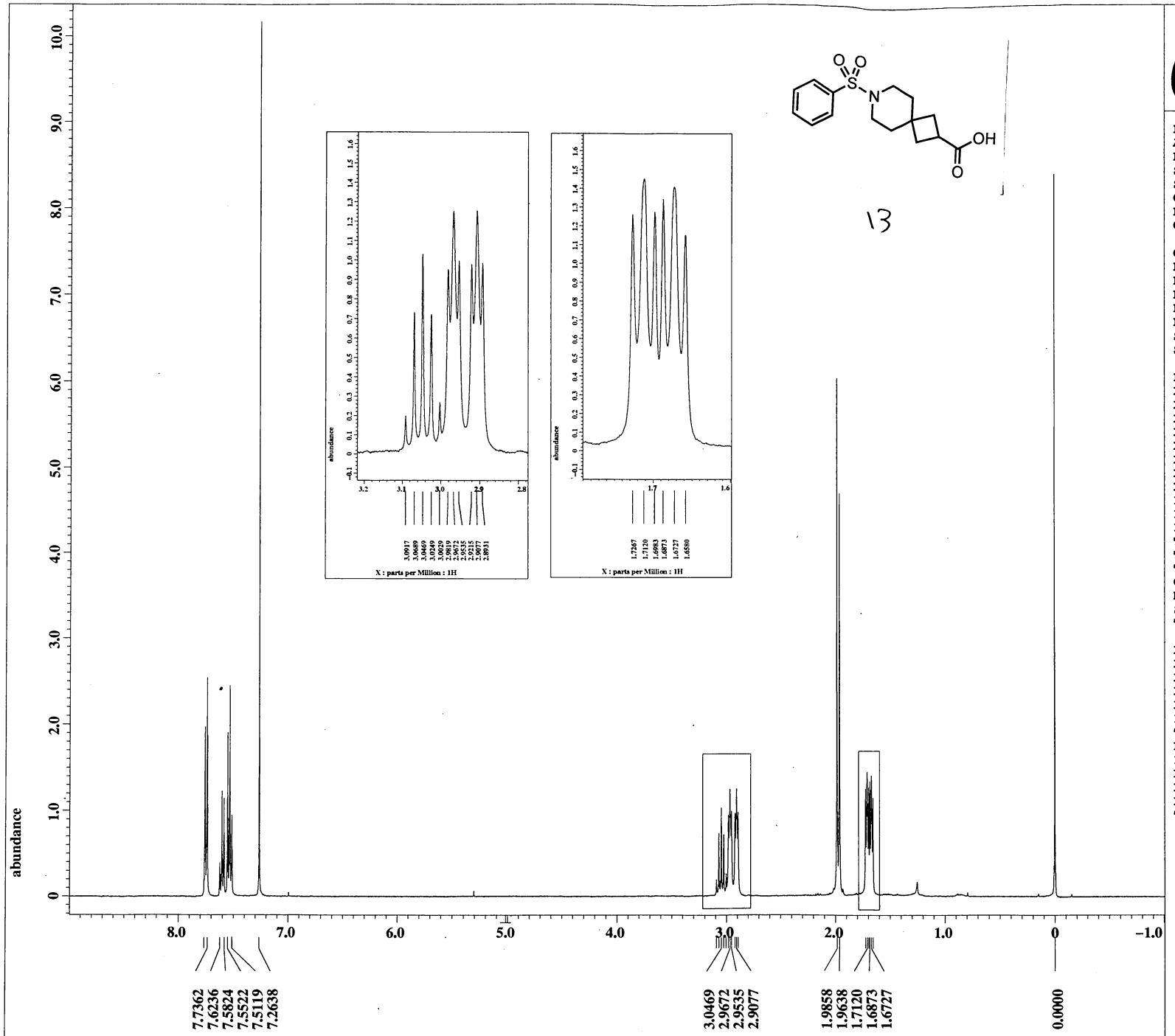
JEOL

Filename	= KUBO-767-pure-3-5.jdf
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#582364
Solvent	= CHLOROFORM-D
Creation_time	= 17-DEC-2012 15:03:05
Revision_time	= 13-JAN-2013 18:38:06
Current_time	= 13-JAN-2013 18:39:01
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 50
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 19.3[dC]

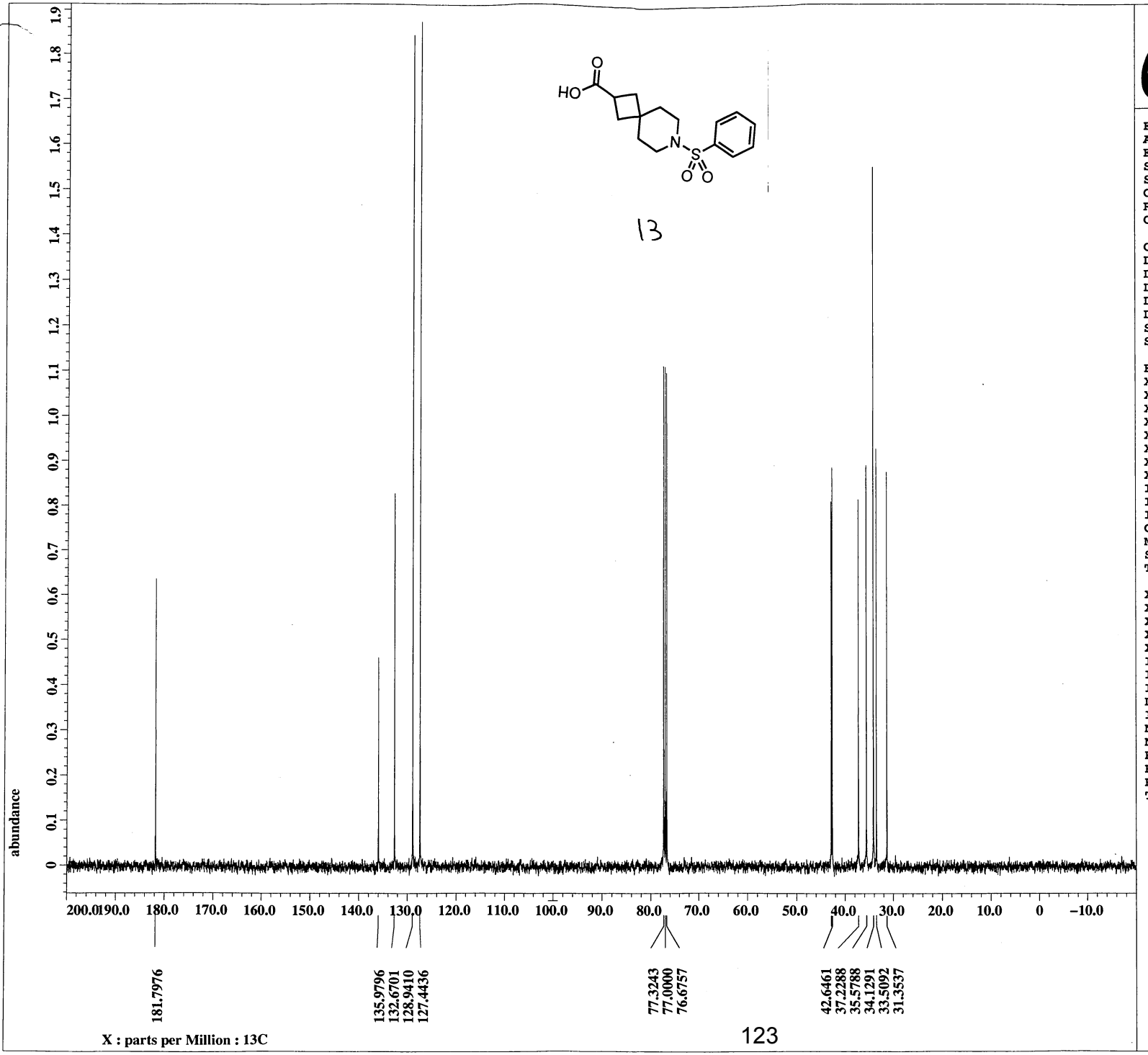


JEOL

Filename	= KUBO-767-13c-5.jdf
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#597370
Solvent	= CHLOROFORM-D
Creation_time	= 17-DEC-2012 16:17:18
Revision_time	= 13-JAN-2013 18:41:29
Current_time	= 13-JAN-2013 18:41:56
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECX 400P
Spectrometer	= DELTA2_NMR
Field_strength	= 9.2982153[T] (400[MHz
X_acq_duration	= 1.048576[s]
X_domain	= 13C
X_freq	= 99.54517646[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.95367432[Hz]
X_sweep	= 31.25[kHz]
Irr_domain	= 1H
Irr_freq	= 395.88430144[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 200
Total_scans	= 200
X_90_width	= 9.5[us]
X_acq_time	= 1.048576[s]
X_angle	= 30[deg]
X_atn	= 3.4[dB]
X_pulse	= 3.16666667[us]
Irr_atn_dec	= 19.607[dB]
Irr_atn_noe	= 19.607[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 50
Relaxation_delay	= 2[s]
Repetition_time	= 3.048576[s]
Temp_get	= 402.2[dC]



Filename	= KUBO-764-date-1h-5.jd
Author	= element
Experiment	= single_pulse.ex2
Sample_id	= S#22598
Solvent	= CHLOROFORM-D
Creation_time	= 11-JAN-2013 23:26:56
Revision_time	= 14-JAN-2013 15:26:47
Current_time	= 14-JAN-2013 15:27:24
Comment	= single_pulse
Data_format	= 1D COMPLEX
Dim_size	= 16384
Dim_title	= 1H
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 2.78790144[s]
X_domain	= 1H
X_freq	= 391.78655441[MHz]
X_offset	= 5[ppm]
X_points	= 16384
X_prescans	= 1
X_resolution	= 0.35869274[Hz]
X_sweep	= 5.87682181[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Tri_domain	= 1H
Tri_freq	= 391.78655441[MHz]
Tri_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 8
Total_scans	= 8
X_90_width	= 10.8[us]
X_acq_time	= 2.78790144[s]
X_angle	= 45[deg]
X_atn	= 1.9[dB]
X_pulse	= 5.4[us]
Irr_mode	= Off
Tri_mode	= Off
Dante_presat	= FALSE
Initial_wait	= 1[s]
Recvr_gain	= 56
Relaxation_delay	= 5[s]
Repetition_time	= 7.78790144[s]
Temp_get	= 18.8[dC]



JEOL

Filename	= KUBO-764-date-13C-3.j
Author	= element
Experiment	= single_pulse_dec
Sample_id	= S#36005
Solvent	= CHLOROFORM-D
Creation_time	= 11-JAN-2013 23:52:46
Revision_time	= 14-JAN-2013 15:28:18
Current_time	= 14-JAN-2013 15:28:25
Comment	= single pulse decouple
Data_format	= 1D COMPLEX
Dim_size	= 26214
Dim_title	= 13C
Dim_units	= [ppm]
Dimensions	= X
Site	= ECS 400
Spectrometer	= JNM-ECS400
Field_strength	= 9.20197068[T] (390[MH
X_acq_duration	= 1.06430464[s]
X_domain	= 13C
X_freq	= 98.51479726[MHz]
X_offset	= 100[ppm]
X_points	= 32768
X_prescans	= 4
X_resolution	= 0.93958061[Hz]
X_sweep	= 30.78817734[kHz]
Irr_domain	= 1H
Irr_freq	= 391.78655441[MHz]
Irr_offset	= 5[ppm]
Clipped	= FALSE
Mod_return	= 1
Scans	= 100
Total_scans	= 100
X_90_width	= 8.15[us]
X_acq_time	= 1.06430464[s]
X_angle	= 30[deg]
X_atn	= 4.9[dB]
X_pulse	= 2.71666667[us]
Irr_atn_dec	= 22.445[dB]
Irr_atn_noe	= 22.445[dB]
Irr_noise	= WALTZ
Decoupling	= TRUE
Initial_wait	= 1[s]
Noe	= TRUE
Noe_time	= 2[s]
Recvr_gain	= 60
Relaxation_delay	= 2[s]
Repetition_time	= 3.06430464[s]
Temp_get	= 19[dC]