Supporting Information

Gold-catalyzed Room-Temperature Isomerization of Unactivated Allenes to 1,3-Dienes

Ting Chun-Ming, Hsu Yi-Lin and Rai-Shung Liu*

Department of Chemistry, National Tsing-Hua University, Hsinchu, Taiwan, ROC
----------------------- e-mail:*rsliu@mx.nthu.edu.tw*

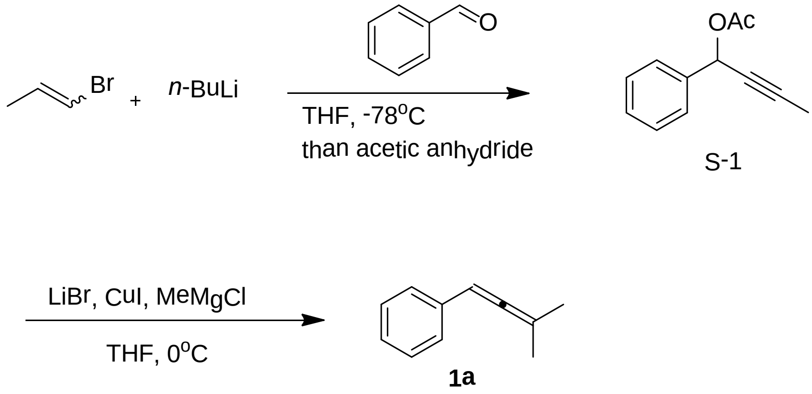
Table of contents:

(a)Experimental Procedures for Synthesis of the Substrates and Catalytic Operations

(1) General Sections

Unless otherwise noted, all reactions were carried out under a nitrogen atmosphere in oven-dried glassware using standard syringe, cannula and septa apparatus. Benzene, , tetrahydrofuran and hexane were dried with sodium benzophenone and distilled before use. Dichloromethane, diethyl ether and acetonitrile was dried over CaH_2 and distilled before use. NMR spectra were run at 400 MHz (^{1}H NMR) or 100 MHz (^{13}C NMR) in $CDCl_3$ or CD_2Cl_2.

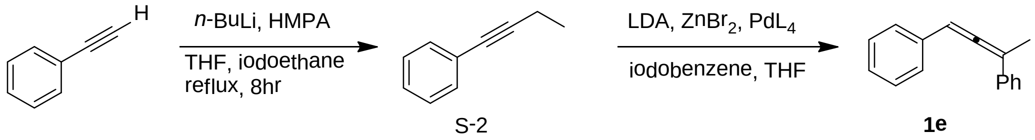
(2)Preparation of (3-methylbuta-1,2-dien-1-yl)benzene (1a)



To a THF (60 mL) solution of 1-bromo-1-propene (2.6 mL, 30.1 mmol) was added n-butyl-lithium (2.5 M, 22.6 mL, 56.5 mmol) at -78 °C, and the mixture was stirred for 30 min before it was treated with benzaldehyde (2g, 18.8 mmol in 5 mL THF).The solution was stirred for 1 hour at room temperature. Acetic anhydride (2.8 mL, 30.1 mmol) was add to this solution at 0 °C, and the solution was stirred for another 1 hour before addition water. The aqueous solution was extracted with EtOAc, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate S-1 (3.05 g, 86 %).

To a THF (50 mL) solution of CuI (5,87 g, 21.2 mmol) and LiBr (2.67 g, 21.1 mmol) was added MeMgCl (22 wt% in THF, 10.2 mL, 21.2 mmol) at 0 °C. The solution was stirred for 1 hour before added the S-1 (2.9 g, 15.4 mmol) and stirred 8 hour at room temperature. The solution was put into a beaker that contained 50 ml Sat.$Na_2CO_{3(aq)}$ and 50 mL hexane, then removed the solid by filter paper. The aqueous solution was extracted with EtOAc, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate **1a** (2 g, 92 %) as colorless oil. We used the same procedure to prepared substrate **1b~d** and **1k~n**.

(3) Preparation of buta-1,2-diene-1,3-diyldibenzene[s1] (1e)

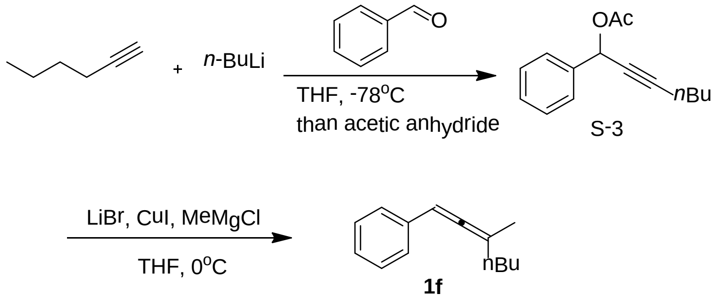


To a THF (30 mL) solution of phenylacetylene (2 g, 19.6 mmol) was added n-butyl-lithium (2.5 M, 9.4 mL, 23.5 mmol) at -40 °C. After stirring in this temperature for 30 min, HMPA (1 mL, 5.5 mmol) was added and the reaction was allowed to warm up to room temperature within 20 min. Then iodoethane (2.2 mL, 29.4 mmol) was added to the solution and the resulting solution was heated to reflux for 8 hours, The reaction was then quenched with a saturated aqueous NH_4Cl solution, The aqueous solution was extracted with ether, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate S-2 (1.9 g, 75 %).

To a THF (20 mL) solution of S-2 (500 mg, 3.8 mmol) was added LDA (6.9 mmol) at 0 °C, the reaction was allowed to warm up to room temperature within 1 h, Then a solution of dry $ZnBr_2$ (4.32 g, 19.0 mmol) in THF (10 mL) was added and stirred for 25 min. $Pd(PPh_3)_4$ (222 mg, 0.2 mmol) in THF(3 mL) and 4-bromoiodobenzene (0.44 mL, 3.4 mmol) were added subsequently, and the resulting mixture was stirred at room temperature. After the reaction was complete as monitored by TLC, it was quenched with saturated NH_4Cl solution. The aqueous solution was extracted with ether, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate **1e** (0.8 g, 80 %).

[s1]: Jinbo Zhao, Yu Liu,Qiwen He, Yuxue Li, Shengming Ma, *Chem. Eur. J.* **2009**, *15*, 11361

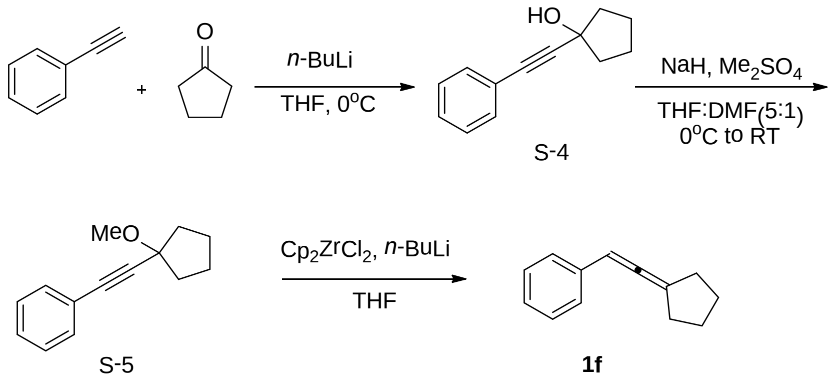
(4)Preparation of (3-methylhepta-1,2-dien-1-yl)benzene (1f)



To a THF (30 mL) solution of 1-hexyne (1.4 mL, 12.2 mmol) was added n-butyl-lithium (2.5 M, 4.5 mL, 11.4 mmol) at -78 °C, and the mixture was stirred for 30 min before it was treated with benzaldehyde (1g, 9.4 mmol in 5 mL THF).The solution was stirred for 1 hour at room temperature. Acetic anhydride (1.5 mL, 15.1 mmol) was add to this solution at 0 °C, and the solution was stirred for another 1 hour before addition water. The aqueous solution was extracted with EtOAc, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate S-3 (2.0 g, 92 %).

To a THF (50 mL) solution of CuI (3.6 g, 8.6 mmol) and LiBr (0.75 g, 8.6 mmol) was added MeMgCl (22 wt% in THF, 2.9 mL, 8.6 mmol) at 0 °C. The solution was stirred for 1 hour before added the S-3 (1 g, 4.3 mmol) and stirred 8 hour at room temperature. The solution was put into a beaker that contained 50 ml Sat.$Na_2CO_{3(aq)}$ and 50 mL hexane, then removed the solid by filter paper. The aqueous solution was extracted with EtOAc, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate **1f** (0.7 g, 86 %) as colorless oil.

(5) Preparation of (2-cyclopentylidenevinyl)benzene[s2] (1h)



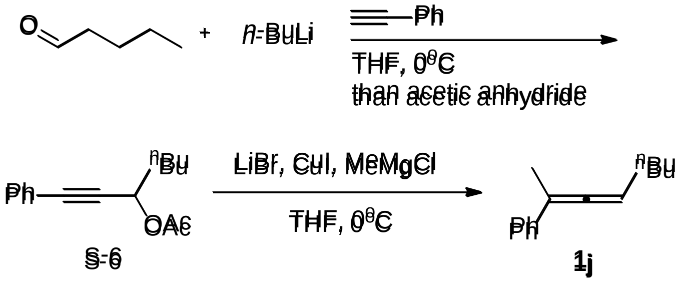
To a solution of THF (20 mL) of phenylacetylene (0.77 mL, 6.8 mmol) was added n-butyl-lithium (2.5 M, 2.6 mL, 6.5 mmol) at 0 °C. After stirring for 1 h at the same tempreture, Cyclopentanone (0.5 g, 5.9 mmol)was added and the mixture was warmed up to room tempreture and stirred for 3 h. The reaction mixture was quenched with with a saturated aqueous NH_4Cl solution, The aqueous solution was extracted with ether, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate S-4 (0.9 g, 83 %).

At 0 ^oC, to a suspension of NaH (0.3 g, 7.4 mmol) in THF and DMF (5:1) was added dropwise to a THF solution (5 mL) of S-4 (1.15 g, 6.2 mmol) with stirring for 1 hour, Me_2SO_4 (1.5 equiv) was added at 0 ^oC and stirred at room tempreture for another 2 h. The reaction mixture was quenched with with a saturated aqueous NH_4Cl solution, The aqueous solution was extracted with ether, and the organic phase was dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate S-5 (1.13 g, 92 %).

To a solution of Cp_2ZrCl_2 (1.16 g, 3.99 mmol) in THF (20 mL) was added *n*-BuLi (2.5 M, 3.2 mL, 7.98 mmol) at -78 ^oC. After the mixture was stirred for 1 h at the same temperature, S-5 (0,5 g, 2.5 mmol) was added, and then the mixture was warmed to room temperature and stirred for 3 h. The reaction mixture was quenched with 3 N HCl aqueous solution and extracted with ethyl ether. The extract was washed with saturated NaHCO3 solution and dried over $MgSO_4$, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate S-4 (0.36 g, 85 %). We used the same procedure to prepared substrate **1g**, **1i**. and **d_6-2a**.

[s2]: Hao Zhang, Xiaoping Fu, Jingjin Chen, Erjuan Wang, Yuanhong Liu,, Yuxue Li, *J. Org. Chem*. **2009**, *74*, 9351.

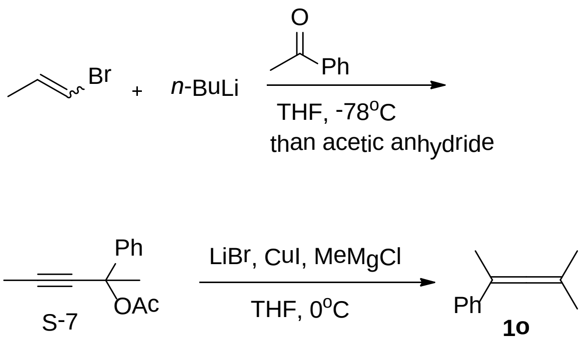
(6) Preparation of octa-2,3-dien-2-ylbenzene (1j)



To a stirred solution of phenylacetylene (2.37 g, 23.2 mmol) in THF (20 mL) at 0 °C was slowly added *n*-BuLi (2.5 M in hexane, 9.3 mL, 23.2 mmol). The reaction mixture was further stirred at 0 °C for 30 min before addition of n-Valeraldehyde (2.00 g, 23.2 mmole). The reaction was then warmed to room temperature for 30 mins. After the addition of acetic anhydride (3.5 mL, 37.2 mmol) at 0 °C, the reaction mixture was further stirred at room temperature for 2 hour. The reaction mixture was quenched with water followed by extraction with ethyl acetate. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, and concentrated. The resulting oily residue was purified by silica gel column chromatography (hexane:EA = 10:1) to give pure S-6 (4.80 g, 20.9 mmol, 90%).

Then a stirred solution of CuI (3.31 g, 17.4 mmol) and LiBr (1.51 g, 17.4 mmol) in THF at 0 °C was slowly added MeMgCl (22% in THF, 5.79 mL, 17.4 mmol). The reaction mixture was further stirred at room temperature for 30 min before addition of S-6 (2.00 g, 8.7 mmol) at 0 °C. The reaction mixture was then stirred at room temperature overnight. The solution was put into a beaker that contained 50 ml Sat.Na2CO3(aq) and 50 mL hexane, then removed the solid by filter paper. The aqueous solution was extracted with EtOAc, and the organic phase was dried over MgSO4, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate**1j** (1.33g, 7.1 mmol, 82%).

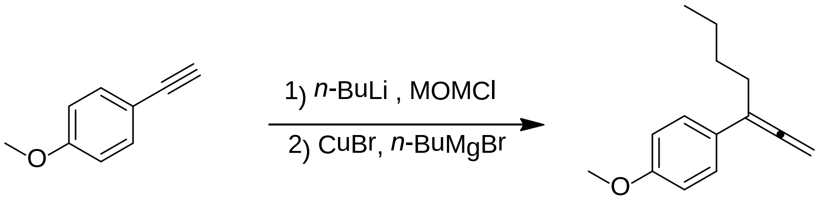
(7) Preparation of (4-methylpenta-2,3-dien-2-yl)benzene (1o)



To a stirred solution of 1-bromo-1-propene (2.3 mL, 26.6 mmol) in THF (80 mL) at -78 °C was slowly added *n*-BuLi (2.5 M in hexane, 20.0 mL, 50.0 mmol). The reaction mixture was further stirred for 30 min before addition of acetophenone (2.00 g, 16.6 mmole). The reaction was then warmed to room temperature for 30 mins. After the addition of acetic anhydride (3.1 mL, 33.3 mmol) at 0 °C, the reaction mixture was further stirred at room temperature for 2h. The reaction mixture was quenched with water followed by extraction with ethyl acetate. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, and concentrated. The resulting oily residue was purified by silica gel column chromatography (hexane:EA = 10:1) to give pure S-7 (2.96 g, 14.6 mmol, 88% yield).

Then a stirred solution of CuI (3.77 g, 19.8 mmol) and LiBr (1.73 g, 19.8 mmol) in THF at 0 °C was slowly added MeMgCl (22% in THF, 6.67 mL, 19.8 mmol). The reaction mixture was further stirred at room temperature for 30 min before addition of S-7 (2.00 g, 9.9 mmol) at 0 °C. The solution was put into a beaker that contained 50 ml Sat.Na2CO3(aq) and 50 mL hexane, then removed the solid by filter paper. The aqueous solution was extracted with EtOAc, and the organic phase was dried over MgSO4, and evaporated to dryness. The residues were eluted through a silica gel column to afford substrate **1o** (1.43 g, 8.3 mmol, 84%). Compound **1p** and **1q** were prepared by following this procedure.

(8) Preparation of 1-(hepta-1,2-dien-3-yl)-4-methoxybenzene[s3] (3a)

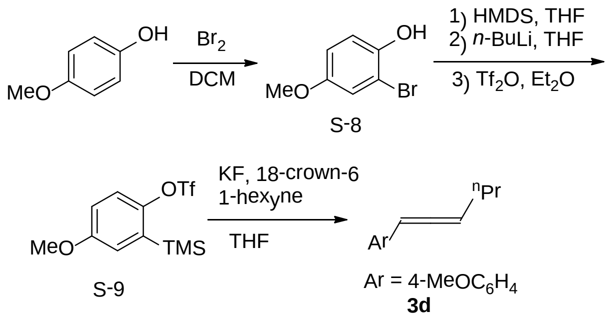


3a

To a solution of 1-ethynyl-4-methoxybenzene (0.7 g, 5.3 mmol) in anhydrous ether (15.0 mL) was added dropwise a solution of *n*-BuLi (2.5 M, 2.1 mL, 5.3 mmol) at -40 ^{o}C within 10 min. After being stirred for 30 min, MOMCl (0.42 mL, 5,3 mmol) was added dropwise at the same temperatue, which was followed by warming up to room temperature naturally for about 1.5 h. Then CuBr (0.152 g, 1.06 mmol) and a solution of *n*-C_4H_9MgBr in ether (10.6 mmol) were added dropwise sequentially to the reaction mixture for 0.5 h at room temperature. After being stirred for 1.5 hour as monitored by TLC, the reaction mixture was quenched with an aqueous solution of saturated ammonium chloride (5 mL), extracted with ether, washed with saturated brine, and dried over anhydrous sodium sulfate. The residues were eluted through a silica gel column to afford substrate **3a.** We used the same procedure to prepared substrate **3b** and **3c**.

[s3] Jing Li, Chao Zhou, Chunling Fu, Shengming Ma, *Tetrahedron*, **2009**, *65*, 3695.

(9) Preparation of 1-(hexa-1,2-dien-1-yl)-4-methoxybenzene (3d)



S-8

S-9

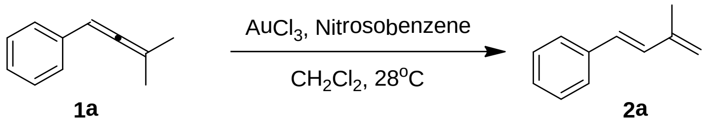
3d

To a stirred solution of 4-methoxyphenol (3.0 g, 24.2 mmol) in dichloromethane (160 mL) was slowly added bromine (1.37 mL, 26.6 mmol) to give S-8 (4.91 g, 24.2 mmol, 100% yield).

A mixture of S-8 (6.00 g, 29.6 mmol) and HMDS (5.72 g, 35.5mmol) in THF (96 mL) was refluxed at least 2 hour. The solvent was evaporated, and the residue was vacuumed to remove NH_3 and unreacted HMDS. The product was dissolved in THF (96 mL) and cooled to -78 °C. The *n*-BuLi (2.5 M in hexane, 13.0 mL, 32.5 mmol) then was added slowly at -78 °C. Stirring was kept up for 1 hour until the temperature reached -30 °C. Then Et_2O (96 mL) was added. The reaction mixture was cooled to -78 °C again, Tf_2O (10.4 g, 36.9 mmol) was added dropwise. Stirring was kept up for 1 hour until the temperature reached -30 °C. The reaction mixture was quenched with $NaHCO_3$ solution and extracted with ethyl acetate. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, and concentrated. The residue was purified by silica gel column chromatography (hexane:EA = 10:1) to give S-9 (7.0 g, 21.3 mmol, 72%).

A mixture of KF (0.39 g, 6.9 mmol) and 18-crown-6-ether (2.11 g, 8.0 mmol) was dissolved in THF (9 mL) before addition of S-9 (0.75 g, 2.3 mmol) and 1-hexyne (0.75 g, 9.1 mmol). The reaction mixture was stirred at room temperature for 8 hour. The reaction mixture was diluted with CH_2Cl_2, filtered through celite and silica gel. The residue was purified by silica gel column chromatography using hexane as eluent to give pure **3d** (0.27g, 1.4 mmol, 62%).

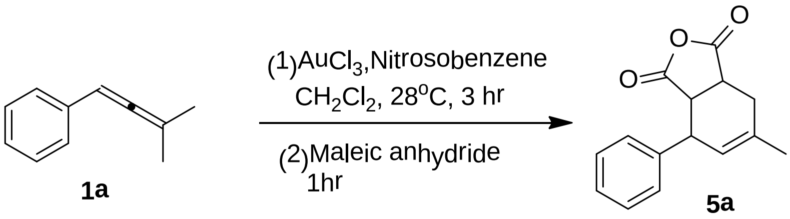
(c) Standard procedure for catalytic operations



1a

2a

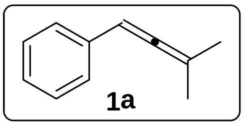
A 10-ml flask containing $AuCl_3$ (7.4 mg, 5 mole %) was dried in vacuum for 1 h before it was filled with N_2 using a N_2 balloon. To this flask was charged with dichloromethane (3 mL) , After 5 min **1a** (3-methylbuta-1,2-dien-1-yl)benzene (70 mg, 0.49 mmol) and nitrosobenzene (5 mg, 10 mol %) dissolved in dichloromethane was added to flask. After 3 hour, The solution was concentrated and eluted through a silica column (hexane) to afford compounds **2a** (64 mg, 92%).



A 10-ml flask containing $AuCl_3$ (7.4 mg, 5 mole %) was dried in vacuum for 1 h before it was filled with N_2 using a N_2 balloon. To this flask was charged with dichloromethane (3 mL) , After 5 min **1a** (3-methylbuta-1,2-dien-1-yl)benzene (70 mg, 0.49 mmol) and nitrosobenzene (5 mg, 10 mol %) dissolved in dichloromethane was added to flask. After 3 hour was added maleic anhydride(1.05eq, in DCM). After 1 hour the solution was concentrated and eluted through a silica column (hexane/ethyl acetate = 10:1) to afford compounds **5a** (105 mg, o.44 mol, 90%).

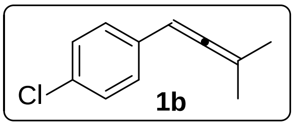
(c) Spectrum data

Spectra data for (3-methylbuta-1,2-dien-1-yl)benzene (1a)


1a

IR (neat, cm^{-1}): 3010 (s), 1900 (s), 1620 (m), 760 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.28~7.26 (m, 4 H), 7.28~7.26 (m, 1 H), 5.99~5.93 (m, 1 H), 1.82 (s, 3 H), 1.81 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.1, 136.0, 128.4(CH×2), 126.6(CH×2), 126.3, 99.1, 92.5, 20.2, (CH_3×2).; HRMS calcd for $C_{11}H_{12}$: 144.0939; Found : 144.0935.

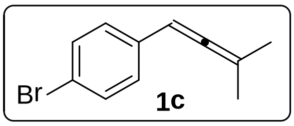
Spectra data for 1-chloro-4-(3-methylbuta-1,2-dien-1-yl)benzene (1b)



1b

IR (neat, cm^{-1}): 2980 (s), 1955 (s), 1570 (m), 790 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.23 (d, *J* = 10.8 Hz, 2 H), 7.16 (d, *J* = 8.8 Hz, 2 H), 5.94~5.91 (m, 1 H), 1.81 (s, 3 H), 1.80 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.2, 134.5, 131.8, 128.5(CH×2), 127.8(CH×2), 99.6, 91.7, 20.1(CH_3×2). ; HRMS calcd for $C_{11}H_{11}Cl$: 178.0549; Found : 178.0547.

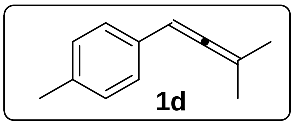
Spectra data for 1-bromo-4-(3-methylbuta-1,2-dien-1-yl)benzene (1c)



1c

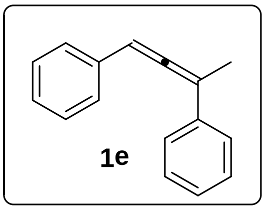
IR (neat, cm^{-1}): 2990 (s), 1950 (s), 1520 (m), 810 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.37 (d, J = 8.4 Hz, 2 H), 7.10 (d, J = 8.4 Hz, 2 H), 5.92~5.89 (m, 1 H), 1.80 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 203.2, 135.0, 131.5(CH×2), 128.1(CH×2), 119.9, 99.6, 91.7, 20.1(CH_3×2).; HRMS calcd for $C_{11}H_{11}Br$: 222.0044; Found : 222.0043.

Spectra data for 1-methyl-4-(3-methylbuta-1,2-dien-1-yl)benzene (1d)


1d

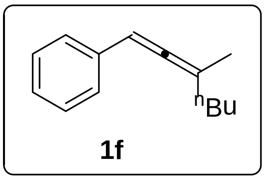
IR (neat, cm^{-1}): 3030 (s), 1945 (s), 1640 (m), 795 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.15 (d, J = 8.0 Hz, 2 H), 8.08 (d, J = 8.0 Hz, 2 H), 5.96~5.93 (m, 1 H), 2.31 (s, 3 H), 1.80 (s, 3 H), 1.79 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.8, 135.9, 132.9, 129.1(CH×2), 126.5(CH×2), 98.8, 92.3, 21.0, 20.2(CH_3×2).; HRMS calcd for $C_{11}H_{14}$: 158.1096; Found : 158.1094.

Spectra data for buta-1,2-diene-1,3-diyldibenzene (1e)


1e

IR (neat, cm^{-1}): 3025 (s), 1950 (s), 1620 (m), 780 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46~7.44 (m, 2 H), 7.33~7.27 (m, 4 H), 7.22~7.17 (m, 2 H), 6.47~6.45 (m, 1 H), 2.21 (d, J = 2.8 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 206.8, 136.3, 134.5,128.7(CH×2),128.4(CH×2), 127.0(CH×2), 126.9(CH×2), 125.8(CH×2), 104.5, 96.6, 16.7. ; HRMS calcd for $C_{16}H_{14}$: 206.1096; Found : 206.1094.

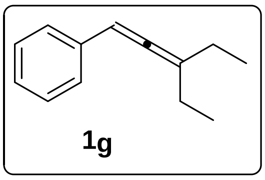
Spectra data for (3-methylhepta-1,2-dien-1-yl)benzene (1f)



1f

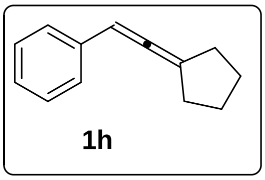
IR (neat, cm^{-1}): 3015 (s), 1940 (s), 1580 (m), 790 (s)^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27~7.24 (m, 4 H), 7.16~7.12 (m, 1 H), 6.04~6.01 (m, 1 H), 2.08~2.04 (m, 2 H), 1.78 (d, J = 2.8 Hz, 3 H), 1.49~1.41 (m, 2 H), 1.38~1.31 (m, 2 H), 0.87 (t, J = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.7, 136.1, 128.4(CH×2), 126.5(CH×2), 126.3, 103.7, 93.8, 33.8, 29.7, 22.5, 18.8, 13.9.; HRMS calcd for $C_{14}H_{18}$: 186.1406; Found : 186.1402.

Spectra data for (3-ethylpenta-1,2-dien-1-yl)benzene (1g)


1g

IR (neat, cm^{-1}): 2995 (s), 1920 (s), 1550 (m), 765 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27~7.26 (m, 4 H), 7.17~7.12 (m, 1 H), 6.18~6.15 (m, 1 H), 2.15~2.05 (m, 4 H), 1.04 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 201.6, 128.4(CH×2), 126.3(CH×3), 112.3, 96.5, 25.8(CH_2×2), 12.3(CH_3×2).; HRMS calcd for $C_{13}H_{16}$: 172.1252; Found : 172.1252.

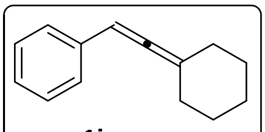
Spectra data for (2-cyclopentylidenevinyl)benzene (1h)



1h

IR (neat, cm^{-1}): 3030 (s), 1951 (s), 1598 (m), 693 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28~7.24 (m, 4 H), 7.16~7.12 (m, 1 H), 6.09~6.05 (m, 1 H), 2.54~2.41 (m, 4 H), 1.76~1.72 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.6, 136.0, 128.4(CH×2), 126.6(CH×2), 126.3, 107.5, 94.9, 31.1(CH_2×2), 27.1(CH_2×2). HRMS calcd for $C_{13}H_{14}$: 170.1096; Found : 170.1094.

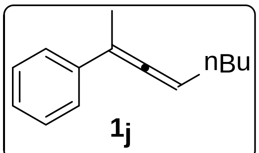
Spectra data for (2-cyclohexylidenevinyl)benzene (1i)



1i

IR (neat, cm^{-1}): 2928 (s), 1965 (s), 1446 (m), 851 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27~7.26 (m, 4 H), 7.16~7.12 (m, 1 H), 5.98~5.96 (m, 1 H), 2.28~2.26 (m, 2 H), 2.21~2.14 (m, 2 H), 1.71~1.57 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 199.6, 136.1, 128.4(CH×2), 126.5(CH×2), 126.2, 106.4, 92.4, 31.3(CH_2×2), 27.7(CH_2×2), 26.1.; HRMS calcd for $C_{14}H_{16}$: 184.1252; Found : 184.1251.

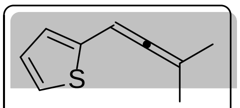
Spectra data for octa-2,3-dien-2-ylbenzene (1j)



1j

IR (neat, cm^{-1}): 1952, 1612, 3014, 2935; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 7.2 Hz, 2 H), 7.30 (t, *J* = 8.0 Hz, 2 H), 7.17 (t, *J* = 7.2 Hz, 1 H), 5.42 (s, 1 H), 2.07~2.12 (m, 5 H), 1.34~1.46 (m, 4 H), 0.89 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 204.1, 137.7, 128.2(CH×2), 126.2, 125.5(CH×2), 100.3, 93.0, 31.4, 28.7, 22.3, 17.1, 13.9; HRMS calcd. For $C_{14}H_{18}$: 186.2920; Found: 182.2920.

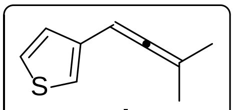
Spectra data for 2-(3-methylbuta-1,2-dien-1-yl)thiophene (1k)



1k

IR (neat, cm^{-1}): 3110 (m), 1890 (s), 1540 (m), 690 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.09 (d, *J* = 5.2 Hz, 1 H), 6.90 (dd, *J* = 5.2, 3.6 Hz, 1 H), 6.83 (d, *J* = 3.6 Hz, 1 H), 6.19~6.16 (m, 1 H), 1.79 (s, 3 H), 1.78 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 202.4, 140.9, 127.3, 123.9, 123.8, 99.6, 86.8, 20.3(CH_3×2).; HRMS calcd for $C_9H_{10}S$: 150.0503; Found : 150.0500.

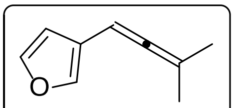
Spectra data for 3-(3-methylbuta-1,2-dien-1-yl)thiophene (1l)



1l

IR (neat, cm^{-1}): 2970 (m), 1820 (s), 1490 (m), 720 (s); ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.23~7.21 (m, 1 H), 7.02 (d, *J* = 4.8 Hz, 1 H), 6.99 (d, *J* = 3.2 Hz, 1 H), 6.05~6.02 (m, 1 H), 1.78 (s, 3 H), 1.77 (s, 3 H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 203.1, 137.5, 126.4, 125.5, 119.8, 89.2, 87.1, 20.4(CH$_3$×2).; HRMS calcd for $C_9H_{10}S$: 150.0503; Found : 150.0502.

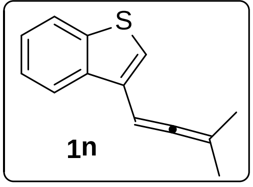
Spectra data for 3-(3-methylbuta-1,2-dien-1-yl)furan (1m)



1m

IR (neat, cm^{-1}): 3110 (m), 1880 (s), 1155 (s) , 869 (s); ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.32 (s, 2 H), 6.33 (s, 1 H), 5.88~5.85 (m, 1 H), 1.76 (s, 3 H), 1.75 (s, 3 H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 202.8, 143.1, 138.9, 121.6, 109.0, 98.0, 82.4, 20.3(CH$_3$×2). HRMS calcd for $C_9H_{10}O$: 134.0732; Found : 134.0732.

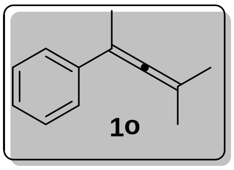
Spectra data for 3-(3-methylbuta-1,2-dien-1-yl)benzo[b]thiophene (1n)



1n

IR (neat, cm^{-1}): 3070 (m), 1910 (s), 1500 (s), 800 (s); ^{1}H NMR (400 MHz, CDCl$_3$): δ 8.04 (d, *J* = 7.6 Hz, 1 H), 7.82 (d, *J* = 7.6 Hz, 1 H), 7.39~7.31 (m, 2 H), 7.20 (s, 1 H), 6.35~6.32 (m, 1 H), 1.86 (s, 3 H), 1.85 (s, 3 H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 203.5, 140.7, 137.4, 130.5, 124.3, 124.1, 122.7, 122.6, 122.5, 98.0, 86.1, 20.7(CH$_3$×2); HRMS calcd for $C_{13}H_{12}S$: 200.0600; Found : 200.0598.

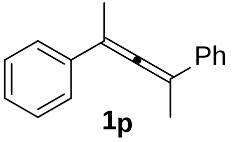
Spectra data for (4-methylpenta-2,3-dien-2-yl)benzene (1o)



1o

IR (neat, cm^{-1}): 1910, 1622, 3015, 2905; ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.36 (d, *J* = 7.6 Hz, 2 H), 7.29 (t, *J* = 7.6 Hz, 2 H), 7.15 (t, *J* = 7.2 Hz, 1 H), 2.04 (s, 3 H), 1.78 (s, 6 H); ^{13}C NMR (100 MHz, CDCl$_3$): δ 202.1, 138.7, 128.1(CH×2), 126.1, 125.7(CH×2), 98.1, 96.8, 20.4(CH$_3$×2), 17.2; HRMS calcd. For $C_{12}H_{14}$: 158.2390; Found: 158.2390

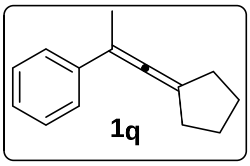
Spectra data for penta-2,3-diene-2,4-diyldibenzene (1p)



1p

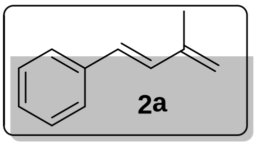
IR (neat, cm^{-1}): 1945, 1615, 3016, 2945; ^{1}H NMR (400 MHz, *d*^{2}-DCM): δ 7.42 (d, *J* = 6.4 Hz, 4 H), 7.31 (t, *J* = 6.8 Hz, 4 H), 7.20 (t, *J* = 7.6 Hz, 2 H), 2.20 (s, 6 H); ^{13}C NMR (100 MHz, *d*^{2}-DCM): δ 206.9, 137.6(C×2), 128.8 (CH×4), 127.1(CH×2), 126.1(CH×4), 102.9(C×2), 17.0(CH$_3$×2); HRMS calcd. For $C_{17}H_{16}$: 220.3083; Found: 220.3083.

Spectra data for (1-cyclopentylideneprop-1-en-2-yl)benzene (1q)


1q

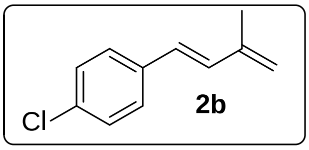
IR (neat, cm^{-1}): 1956, 1610, 3015, 2940; ^{1}H NMR (400 MHz, d^2-DCM): δ 7.36 (d, *J* = 6.8 Hz, 2 H), 7.28 (t, *J* = 7.6 Hz, 2 H), 7.15 (t, *J* = 7.6 Hz, 1 H), 2.05~2.46 (m, 4 H), 2.05 (s, 3H), 1.55~1.76 (m, 4 H); ^{13}C NMR (100 MHz, d^2-DCM): δ 208.1, 139.2, 128.6(CH×2), 126.4, 126.1(CH×2), 106.0, 101.0, 31.4(CH_2×2), 27.6 CH_2×2), 17.5; HRMS calcd. For $C_{14}H_{16}$: 184.2762; Found: 184.2762.

Spectra data for (*E*)-(3-methylbuta-1,3-dien-1-yl)benzene (2a)


2a

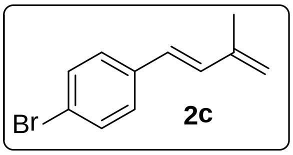
IR (neat, cm^{-1}): 2900(s), 1605 (s), 1375 (s), 753 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41 (d, *J* = 7.2 Hz, 2 H), 7.30 (t, *J* = 7.6 Hz, 2 H), 7.21 (t, *J* = 7.6 Hz, 1 H), 6.86 (d, *J* = 16.0 Hz, 1 H), 6.52 (d, *J* = 16.0 Hz, 1 H), 5.10 (s, 1 H), 5.06 (s, 1 H), 1.96 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.0, 137.4, 131.6, 128.7, 128.6(CH×2), 127.4, 126.4(CH×2), 117.3, 18.6.; HRMS calcd for $C_{11}H_{12}$: 144.0939; Found : 144.0937.

Spectra data for (*E*)-1-chloro-4-(3-methylbuta-1,3-dien-1-yl)benzene (2b)


2b

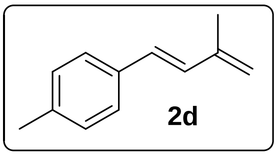
IR (neat, cm^{-1}): 2940 (s), 1620 (s), 1370 (s), 860 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 (d, *J* = 8.4 Hz, 2 H), 7.26 (d, *J* = 8.4 Hz, 2 H), 6.82 (d, *J* = 16.4 Hz, 1 H), 6.46 (d, *J* = 16.4 Hz, 1 H), 5.11 (s, 1 H), 5.08 (s, 1 H), 1.95 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.8, 135.9, 132.9, 132.3, 128.7(CH×2), 127.6(CH×2), 127.4, 117.9, 18.5.; HRMS calcd for $C_{11}H_{11}Cl$: 178.0549; Found : 178.0549.

Spectra data for (*E*)-1-bromo-4-(3-methylbuta-1,3-dien-1-yl)benzene (2c)


2c

IR (neat, cm^{-1}): 3050 (s), 1640 (s), 1350 (s), 855 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.4 Hz, 2 H), 7.27 (d, *J* = 8.4 Hz, 2 H), 6.84 (d, *J* = 16.0 Hz, 1 H), 6.44 (d, *J* = 16.0 Hz, 1 H), 5.11 (s, 1 H), 5.09 (s, 1 H), 1.95 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.8, 136.3, 132.4, 131.7(CH×2), 127.9(CH×2), 127.4, 121,1, 118.0, 18.5.; HRMS calcd for $C_{11}H_{11}Br$: 222.0044; Found : 222.0042.

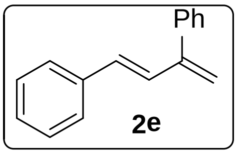
Spectra data for (E)-1-methyl-4-(3-methylbuta-1,3-dien-1-yl)benzene (2d)


2d

IR (neat, cm^{-1}): 2970 (s), 1600 (s), 1330 (m), 790 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (d, *J* = 8.0 Hz, 2 H), 7.11 (d, *J* = 8.0 Hz, 2 H), 6.82 (d, *J* =

16.4 Hz, 1 H), 6.49 (d, *J* = 16.4 Hz, 1 H), 5.07 (s, 3 H), 5.02 (s, 3 H), 2.32 (s, 3 H), 1.95 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.1, 137.2, 134.6, 130.7, 129.3(CH×2), 128.6, 126.4(CH×2), 116.8, 21.2, 18.6.; HRMS calcd for $C_{11}H_{14}$: 158.1096; Found : 158.1096.

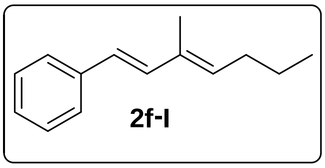
Spectra data for (*E*)-buta-1,3-diene-1,3-diyldibenzene (2e)



2e

IR (neat, cm^{-1}): 3010 (s), 1580 (s), 1395 (m), 760 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39~7.35 (m, 7 H), 7.31~7.28 (m, 2 H), 7.23~7.19 (m, 1 H), 7.03 (d, *J* = 16.4 Hz, 1 H), 6.48 (d, *J* = 16.4 Hz, 1 H), 5.40 (s, 1 H), 5.03 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.1, 140.2, 137.2, 131.9, 130.3, 128.6(CH×2), 128.4(CH×2), 128.2(CH×2), 127.6, 127.5, 126.5(CH×2), 117.3.; HRMS calcd for $C_{16}H_{14}$: 206.1096; Found : 206.1095.

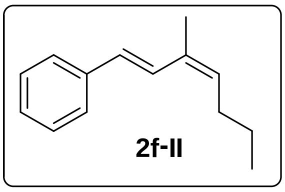
Spectra data for ((1*E*,3*E*)-3-methylhepta-1,3-dien-1-yl)benzene (2f-I)



2f-I

IR (neat, cm^{-1}): 2930 (s), 1560 (s), 1335 (m), 735 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 8.0 Hz, 2 H), 7.27 (t, *J* = 7.6 Hz, 2 H), 7.16 (t, *J* = 8.0 Hz, 1 H), 6.80 (d, *J* = 16.0 Hz, 1 H), 6.43 (d, *J* = 16.0 Hz, 1 H), 5.63 (t, *J* = 7.6 Hz, 1 H), 2.16 (q, *J* = 7.2 Hz, 2 H), 1.85 (s, 3 H), 1.46~1.40 (m, 2 H), 0.92 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.0, 134.3, 134.1, 133.8, 128.5(CH×2), 126.8, 126.1(CH×2), 125.5, 30.5, 22.8, 13.9, 12.4.; HRMS calcd for $C_{14}H_{18}$: 186.1406; Found : 186.1404.

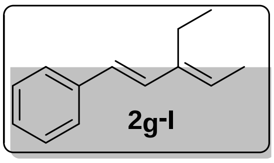
Spectra data for ((1*E*,3*Z*)-3-methylhepta-1,3-dien-1-yl)benzene (2f-Ⅱ)



2f-II

IR (neat, cm^{-1}): 2935 (s), 1560 (s), 1335 (m), 735 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 (d, *J* = 7.6 Hz, 2 H), 7.30 (t, *J* = 7.6 Hz, 2 H), 7.19 (t, *J* = 7.6 Hz, 1 H), 7.18 (d, *J* = 16.0 Hz, 1 H), 6.53 (d, *J* = 16.0 Hz, 1 H), 5.45 (t, *J* = 7.6 Hz, 1 H), 2.32 (q, *J* = 7.6 Hz, 2 H), 1.92 (s, 3 H), 1.47~1.38 (m, 2 H), 0.93 (t, *J*=7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.1, 132.3, 132.0, 128.6(CH×2), 128.0, 127.1, 126.3(CH×2), 126.2, 29.7, 23.1, 20.5, 13.9.; HRMS calcd for $C_{14}H_{18}$: 186.1406; Found : 186.1406.

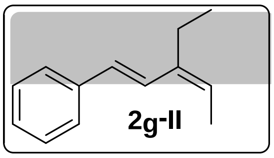
Spectra data for ((1*E*,3*E*)-3-ethylpenta-1,3-dien-1-yl)benzene (2g-I)



2g-I

IR (neat, cm^{-1}): 2910 (s), 1600 (s), 1355 (m), 900 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 7.6 Hz, 2 H), 7.28 (t, *J* = 7.6 Hz, 2 H), 7.17 (t, *J* = 7.6 Hz, 1 H), 6.68 (d, *J* = 16.4 Hz, 1 H), 6.45 (d, *J* = 16.4 Hz, 1 H), 5.64 (q, *J* = 7.2 Hz, 1 H), 2.35 (q, *J* = 7.6 Hz, 2 H), 1.77 (d, *J* = 7.2 Hz, 3 H), 1.05 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.0, 138.1, 132.4, 128.5(CH×2), 127.5, 126.8, 126.1(CH×2), 124.8, 19.5, 13.7, 13.3.; HRMS calcd for $C_{13}H_{16}$: 172.1252; Found : 172.1252.

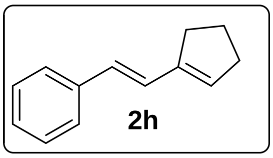
Spectra data for ((1*E*,3*Z*)-3-ethylpenta-1,3-dien-1-yl)benzene (2g-Ⅱ)



2g-II

IR (neat, cm^{-1}): 2915 (s), 1610 (s), 1355 (m), 720 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.43 (d, *J* = 7.6 Hz, 2 H), 7.31 (t, *J* = 7.6 Hz, 2 H), 7.20 (t, *J* = 7.6 Hz, 1 H), 7.13 (d, *J* = 16.4 Hz, 1 H), 6.57 (d, *J* = 16.4 Hz, 1 H), 5.53 (q, *J* = 7.2 Hz, 1 H), 2.31 (q, *J* = 7.6 Hz, 2 H), 1.83 (d, *J* = 7.2 Hz, 3 H), 1.10 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.6, 138.1, 128.6(CH×2), 127.4, 127.1, 126.3(CH×2), 124.9, 124.2, 26.5, 13.6, 13.1.; HRMS calcd for $C_{13}H_{16}$: 172.1252; Found : 172.1250.

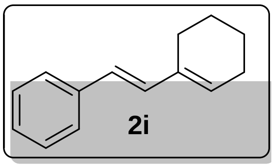
Spectra data for (*E*)-(2-(cyclopent-1-en-1-yl)vinyl)benzene (2h)



2h

IR (neat, cm^{-1}): 2920 (s), 1625 (s), 1340 (m), 920 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 (d, *J* = 7.6 Hz, 2 H), 7.29 (t, *J* = 7.6 Hz, 2 H), 7.18 (t, *J* = 7.6 Hz, 1 H), 7.00 (d, *J* = 16.4 Hz, 1 H), 6.39 (d, *J* = 16.4 Hz, 1 H), 5.84 (br, 1 H), 2.55~2.52 (m, 2 H), 2.48~2.44 (m, 2 H), 2.00~1.92 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.8, 137.8, 132.0, 128.6, 128.5(CH×2), 127.0, 126.2(CH×2), 125.7, 33.0, 31.2, 23.1.; HRMS calcd for $C_{13}H_{14}$: 170.1096; Found : 170.1096.

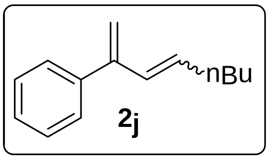
Spectra data for (*E*)-(2-(cyclohex-1-en-1-yl)vinyl)benzene (2i)



2i

IR (neat, cm^{-1}): 2905 (s), 1630 (s), 1330 (m), 960 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.38 (d, *J* = 7.6 Hz, 2 H), 7.28 (t, *J* = 7.6 Hz, 2 H), 7.17 (t, *J* = 7.2 Hz, 1 H), 6.76 (d, *J* = 16.4 Hz, 1 H), 6.42 (d, *J* = 16.4 Hz, 1 H), 5.89 (t, *J* = 4.0 Hz, 1 H), 2.26~2.24 (m, 2 H), 2.18~2.17 (m, 2 H), 1.74~1.68 (m, 2 H), 1.65~1.59 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.0, 135.8, 132.5, 130.8, 128.5(CH×2), 126.8, 126.1(CH×2), 124.6, 26.1, 24.5, 22.5(CH_2×2).; HRMS calcd for $C_{14}H_{16}$: 184.1252; Found : 184.1250.

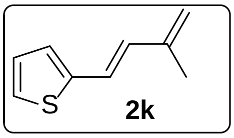
Spectra data for octa-1,3-dien-2-ylbenzene (2j)



2j

IR (neat, cm^{-1}): 2920, 1610, 1345, 910; ^{1}H NMR (400 MHz, $CDCl_3$) (*E* form is major isomer): δ 7.25~7.41 (m, 5 H), 6.28 (d, *J* = 16.0 Hz, 1 H), 5.61~5.72 (m, 1 H), 5.15 (s, 1 H), 5.03 (s, 1 H), 2.08~2.13 (m, 2 H), 1.22~1.35 (m, 4 H), 0.80~0.89 (m, 3 H); *Z* isomer selected peaks, δ 6.10 (d, *J* = 11.6 Hz, 1 H), 5.50 (s, 1 H), 5.12 (s, 1 H), the remaining peaks are overlapped; ^{13}C NMR (100 MHz, $CDCl_3$) (E+Z): δ 148.2, 148.6, 140.9, 134.6, 134.6, 131.3, 128.7, 128.2(CH×2), 128.2(CH×2), 128.0 (CH×2), 127.4, 127.2, 126.6(CH×2), 114.6, 114.4, 32.5, 32.0, 31.4, 28.3, 22.7, 22.3, 13.9, 13.9 ; HRMS calcd For $C_{14}H_{18}$: 186.2920; Found: 182.2920.

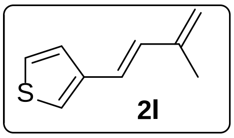
Spectra data for (*E*)-2-(3-methylbuta-1,3-dien-1-yl)thiophene (2k)



2k

IR (neat, cm^{-1}): 3100 (s), 1655 (s), 1420 (m), 670 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.14 (d, *J* = 4.8 Hz, 1 H), 6.98~6.94 (m, 2 H), 6.66 (dd, *J* = 16.0 Hz, 2 H), 5.07 (s, 1 H), 5.03 (s, 1 H), 1.92 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.9, 141.6, 131.5, 127.5, 125.8, 124.1, 121.8, 117.2, 18.4.; HRMS calcd for $C_9H_{10}S$: 150.0503; Found : 150.0503.

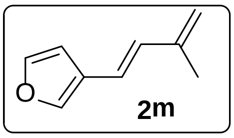
Spectra data for (*E*)-3-(3-methylbuta-1,3-dien-1-yl)thiophene (2l)



2l

IR (neat, cm^{-1}): 3020 (s), 1700 (s), 1425 (m), 610 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.27~7.23 (m, 2 H), 7.17~7.16 (m, 1 H), 6.71 (d, *J* = 16.0 Hz, 1 H), 6.53 (d, *J* = 16.0 Hz, 1 H), 5.06 (s, 1 H), 5.02 (s, 1 H), 1.93 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.0, 140.1, 131.7, 126.0, 124.9, 122.9, 122.0, 116.8, 18.5.; HRMS calcd for $C_9H_{10}S$: 150.0503; Found : 150.0502.

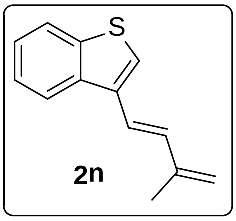
Spectra data for (*E*)-3-(3-methylbuta-1,3-dien-1-yl)furan (2m)



2m

IR (neat, cm^{-1}): 3120 (s), 1670 (s), 1450 (m), 830 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (s, 1 H), 7.36 (t, *J* = 1.6 Hz, 1 H), 6.57 (d, *J* = 16.0 Hz, 1 H), 6.55 (s, 1 H), 6.38 (d, *J* = 16.0 Hz, 1 H), 5.02 (s, 1 H), 4.99 (s, 1 H), 1.91 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.6, 141.9, 140.7, 131.5, 124.3, 118.4, 116.3, 107.4, 18.4.; HRMS calcd for $C_9H_{10}O$: 134.0732; Found : 134.0730.

Spcetra data for (*E*)-3-(3-methylbuta-1,3-dien-1-yl)benzo[b]thiophene (2n)

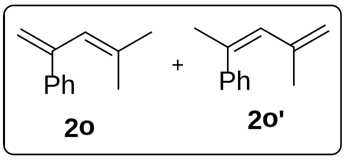


2n

IR (neat, cm^{-1}): 3080 (s), 1680 (s), 1490 (m), 820 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.92 (d, *J* = 7.6 Hz, 1 H), 7.85 (d, *J* = 7.6 Hz, 1 H), 7.46 (s, 1 H), 7.43~7.33 (m, 2 H), 6.95 (d, *J* = 16.0 Hz, 1 H), 6.79 (d, *J* = 16.0 Hz, 1 H), 5.14 (s, 1 H), 5.09 (s, 1 H), 2.00 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.0, 140.4, 137.8, 134.0, 133.2, 124.4, 124.2, 122.9, 121.8, 121.4, 120.5, 117.4, 18.5.; HRMS calcd for $C_{13}H_{12}S$:

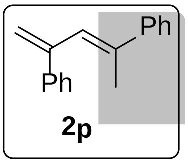
200.0600; Found : 200.0600.

Spectra data for (4-methylpenta-1,3-dien-2-yl)benzene (2o) and (Z)-(4-methylpenta-2,4-dien-2-yl)benzene (2o')



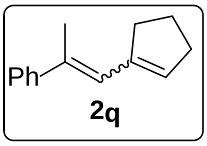
IR (neat, cm^{-1}): 2910, 1600, 1340, 905, 730**;** ^{1}H NMR (600 MHz, CDCl$_3$) (2o form is major isomer): 2o isomer, δ 7.16~7.42 (m, 5 H), 5.93 (s, 1 H), 5.50 (s, 1 H), 5.05 (s, 1 H), 1.86 (s, 3 H), 1.69(s, 3 H); 2o' isomer selected peaks, δ 6.20 (s, 1 H), 5.08 (s, 1 H), 4.92 (s, 1 H), 2.21 (s, 3 H), 1.93 (s, 1 H), the remaining peaks are overlapped; ^{13}C NMR (100 MHz, CDCl$_3$) (2o+2o'): δ 145.4, 144.3, 142.2, 141.4, 136.8, 136.2, 129.7, 128.2, 128.1(CH×2), 127.3(CH×2), 126.9, 126.6(CH×2), 125.9(CH×2), 125.0, 115.3, 114.3, 26.3, 23.7, 19.6, 17.5; HRMS calcd. For C$_{12}$H$_{14}$: 158.2390; Found: 158.2390.

Spectra data for (*E*)-penta-1,3-diene-2,4-diyldibenzene (2p)



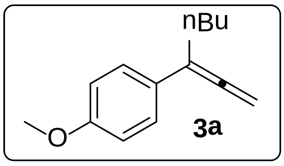
IR (neat, cm^{-1}): 2920, 1610, 1335, 910; ^{1}H NMR (400 MHz, d^2-DCM): δ 7.52 (d, *J* = 8.0 Hz, 2 H), 7.93 (t, *J* = 7.6 Hz, 2 H), 7.28~7.38 (m, 6 H), 6.56 (s, 1 H) , 5.69 (s, 1 H), 5.25 (s, 1 H), 2.12 (s, 3 H); ^{13}C NMR (100 MHz, d^2-DCM): δ 148.5, 143.8, 141.3, 139.3, 128.7(CH×2), 128.7(CH×2), 128.0, 127.7, 127.6, 126.9(CH×2), 126.3(CH×2), 115.6, 17.8; HRMS calcd. For C$_{17}$H$_{16}$: 220.3083; Found: 220.3083.

Spetra data for (1-(cyclopent-1-en-1-yl)prop-1-en-2-yl)benzene (2q)



IR (neat, cm^{-1}): 2925, 1615, 1340, 915. 730; ^{1}H NMR (400 MHz, d^2-DCM) (*E* form is major isomer): *E* isomer, δ 7.15~7.43 (m, 5 H), 6.42 (s, 1 H), 5.80 (s, 1 H), 2.65 (t, *J* = 6.8 Hz, 2 H), 2.43 (t, *J* = 6.8 Hz, 2 H), 2.24 (s, 3H), 1.91~1.98 (m, 2 H); *Z* isomer selected peaks, δ 6.25 (s, 1 H), 5.58 (s, 1 H), 2.22 (t, *J* = 6.8 Hz, 2 H), 2.07 (s, 3 H), 1.73 (t, *J* = 6.8 Hz, 2 H), 1.61~1.67 (m, 2 H), the remaining peaks are overlapped; ^{13}C NMR (100 MHz, d^2-DCM): *E* isomer, 145.1, 142.4, 135.6, 132.4, 128.6 (CH×2), 127.1, 126.2 (CH×2), 125.0, 35.9, 32.9, 24.4, 17.9; *Z* isomer, 143.8, 142.6, 137.5, 131.6, 128.7 (CH×2), 128.0 (CH×2), 127.0, 124.4, 34.1, 32.2, 27.8, 24.6; HRMS calcd. For C$_{14}$H$_{18}$: 184.2762; Found: 184.2762.

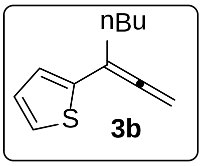
Spectra data for 1-(hepta-1,2-dien-3-yl)-4-methoxybenzene (3a)



IR (neat, cm^{-1}): 2995 (s), 1950 (s), 1690 (w), 875 (s); ^{1}H NMR (400 MHz, CDCl$_3$): δ 7.31 (d, *J* = 9.2 Hz, 2 H), 6.85 (d, *J* = 9.2 Hz, 2 H), 5.02~5.01 (m, 1 H), 3.79 (s, 3 H), 2.39~2.34

(m, 2H), 1.41~1.34 (m, 2 H), 0.91 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.2, 158.3, 128.6, 127.0(CH×2), 113.7(CH×2), 104.4, 77.9, 55.1, 30.0, 29.3, 22.5, 14.0. HRMS calcd for $C_{14}H_{18}O$: 202.1358; Found : 202.1356.

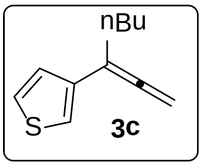
Spectra data for 2-(hepta-1,2-dien-3-yl)thiophene (3b)



3b

IR (neat, cm^{-1}): 3159 (s), 1945 (s), 1650 (w), 860 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.16~7.14 (m, 1 H), 6.96~6.94 (m, 1 H), 6.92~6.91 (m, 1 H), 5.08~5.06 (m, 2 H), 2.41~2.35 (m, 2 H), 1.58~1.51 (m, 2 H), 1.44~1.35 (m, 2 H), 0.92 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 207.7, 141.5, 127.2, 124.0, 122.7, 101.1, 78.7, 30.4, 29.9, 22.4, 13.9.; HRMS calcd for $C_{14}H_{11}S$: 178.0816; Found : 178.0810.

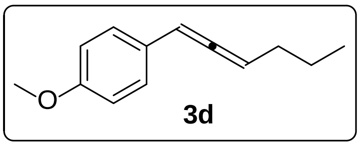
Spectra data for 3-(hepta-1,2-dien-3-yl)thiophene (3c)



3c

IR (neat, cm^{-1}): 3010 (s), 1845 (s), 1670 (w), 850 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.24~7.22 (m, 1 H), 7.11~7.10 (m, 1 H), 7.05~7.04 (m, 1 H), 5.02~5.00 (m, 2 H), 2.38~2.32 (m, 2 H), 1.57~1.49 (m, 2 H), 1.43~1.35 (m, 2 H), 0.92 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 208.8, 138.3, 126.8, 125.2, 118.5, 101.4, 77.8, 29.9(CH_2×2), 22.5, 14.0.; HRMS calcd for $C_{14}H_{11}S$: 178.0816; Found : 178.0814.

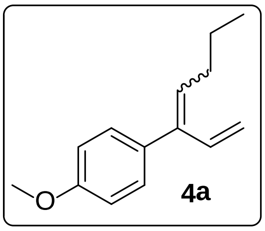
Spectra data for 1-(hexa-1,2-dien-1-yl)-4-methoxybenzene (3d)



3d

IR (neat, cm^{-1}): 1930, 1610, 3015, 2940; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20 (d, *J* = 7.6 Hz, 2 H), 6.85 (d, *J* = 7.2 Hz, 2 H), 6.05~6.10 (m, 1 H), 5.50~5.57 (m, 1 H), 2.05~2.11 (m, 2 H), 1.44~1.52 (m, 2 H), 0.95 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 204.5, 158.5, 127.6, 127.4, 114.0, 94.8, 93.9, 55.1, 30.8, 22.4, 13.7; HRMS calcd. For $C_{11}H_{12}O$: 160.2119; Found: 160.2119.

Spcetra data for 1-(hepta-1,3-dien-3-yl)-4-methoxybenzene (4a)

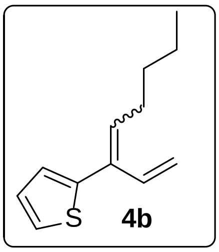


4a

IR (neat, cm^{-1}): 3000 (s), 1650 (s), 1405 (s), 780 (s); ^{1}H NMR (400 MHz, $CDCl_3$): (*E* form is major isomer): δ 7.19 (d, *J* = 8.4 Hz, 2 H), 6.84 (d, *J* = 8.4 Hz, 2 H), 6.86~6.79 (m, 1 H), 5.50 (t, *J* = 7.6 Hz, 1 H), 5.19 (dt, *J* = 10.8, 2.0 Hz, 1 H), 5.05 (dd, *J* = 19.2, 1.6 Hz, 1 H), 3.80 (s, 3 H), 2.43 (ABq, 2 H, Δ_{AB}=12.9 Hz, J_{AB}=7.2 Hz), 1.51~1.42 (m, 2 H), 0.94 (t, *J* = 7.2 Hz, 3 H); (*Z* form): δ 7.02 (d, *J* = 8.4 Hz, 2 H), 6.89 (d, *J* = 8.4 Hz, 2 H), 6.56~6.49 (m, 1 H), 5.68 (t, *J* = 7.6 Hz, 1 H), 4.95(d, *J* = 10.4 Hz, 1 H), 4.67 (d, *J* = 17.2 Hz, 1 H), 3.81 (s, 3 H), 1.91 (ABq, 2 H, Δ_{AB}=12.9 Hz, J_{AB}=7.2 Hz), 1.37~1.31 (m, 2 H), 0.82 (t, *J* = 7.2 Hz, 3

H); ^{13}C NMR (100 MHz, $CDCl_3$): (*E* form is major isomer): δ 158.5, 141.0, 134.1, 133.5, 132.4, 129.8, 116.9, 113.3, 55.2, 30.1, 22.9, 13.8.; (*Z* form): δ 158.3, 141.1, 134.1, 130.6, 113.4, 55.1, 31.1, 22.8, 13.9, the remaining peaks are overlapped.; HRMS calcd for $C_{14}H_{18}O$: 202.1358; Found : 202.13568.

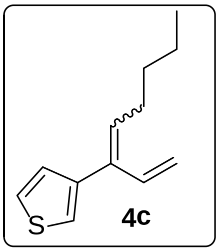
Spectra data for 2-(octa-1,3-dien-3-yl)thiophene (4b)



4b

IR (neat, cm^{-1}): 3105 (s), 1680 (s), 950 (s), 720 (s); ^{1}H NMR (400 MHz, $CDCl_3$): (*E* form is major isomer): δ 7.16 (dd, *J* = 4.8, 1.2 Hz, 1 H), 6.98~6.94 (m, 1 H), 6.78~6.71 (m, 1 H) , 5.81 (t, *J* = 7.2 Hz, 1 H), 5.39 (dd, *J* = 17.6, 1.1 Hz, 1 H), 5.30 (dt, *J* = 11.2, 1.2 Hz, 1 H), 2.25 (ABq, 2 H, Δ_{AB}=12.9 Hz, J_{AB}=7.2 Hz), 1.52~1.43 (m ,2 H), 0.94 (t, *J* = 7.2 Hz, 3 H); (*Z* form): δ7.63 (dd, *J* = 5.2, 1.2 Hz, 1 H), 7.05~7.03 (m, 1 H), 6.83~6.82 (m, 1 H), 6.57~6.50 (m, 1 H), 5.83 (t, *J* = 7.6 Hz, 1 H), 5.02 (d, *J* = 10.4 Hz, 1 H), 4.95 (d, *J* = 17.2 Hz, 1 H), 2.01 (ABq, 2 H, Δ_{AB}=12.9 Hz, J_{AB}=7.2 Hz), 1.42~1.36 (m, 2 H), 0.86 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): (*E* form is major isomer): δ 143.9, 133.3, 132.8, 132.5, 126.8, 124.9, 123.6, 117.9, 30.4, 22.7, 13.8.; (*Z* form): δ 140.4, 137.0, 126.9, 126.6, 125.0, 114.4, 31.4, 14.0, the remaining peaks are overlapped.; HRMS calcd for $C_{14}H_{11}S$: 178.0816; Found : 178.0816.

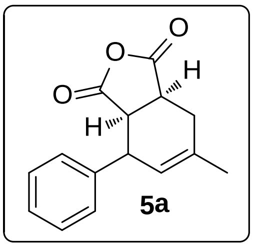
Spectra data for 3-(octa-1,3-dien-3-yl)thiophene (4c)



4c

IR (neat, cm^{-1}): 3050 (s), 1710 (s), 990 (s), 730 (s); ^{1}H NMR (400 MHz, $CDCl_3$): (*E* form is major isomer): δ 7.24 (d, *J* = 12.0 Hz, 1 H), 7.11 (d, *J* = 2.4 Hz, 1 H), 7.06 (d, *J* = 5.2 Hz, 1 H), 6.82~6.75 (m, 1 H), 5.68 (t, *J* = 7.6 Hz, 1 H), 5.25~5.18 (m, 2 H), 2.25 (ABq, 2 H, Δ_{AB}=12.9 Hz, J_{AB}=7.2 Hz), 1.51~1.42 (m ,2 H), 0.94 (t, *J* = 7.6 Hz, 3 H); (*Z* form): δ 7.31~7.29 (m, 1 H), 7.02 (m, 1 H), 6.90 (m, 1 H), 6.55~6.48 (m, 1 H), 5.71 (t, *J* = 7.6 Hz, 1 H), 4.97 (d, *J* = 10.8 Hz, 1 H), 4.80 (d, *J* = 16.8 Hz, 1 H), 1.98 (ABq, 2 H, Δ_{AB}=12.9 Hz, J_{AB}=7.2 Hz), 1.39~1.34 (m, 2 H), 0.84 (t, *J* = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): (*E* form is major isomer): δ 142.3, 135.1, 133.1, 132.5, 128.1, 124.6, 121.7, 117.0, 30.1, 22.8, 13.9,; (*Z* form): δ 140.5, 134.4, 129.1, 123.1, 113.9, 31.2, the remaining peaks are overlapped.; HRMS calcd for $C_{14}H_{11}S$: 178.0816; Found : 178.0815.

Spectrra data for (3aR,7aS)-6-methyl-4-phenyl-3a,4,7,7a-tetrahydroisobenzofuran-1,3-dione (5a)

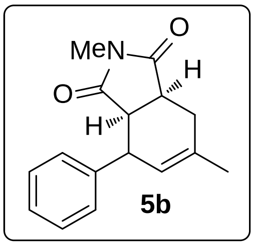


5a

IR (neat, cm^{-1}): 3010 (s), 1840 (s), 1350 (s), 1100 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.35~7.28 (m, 3 H), 7.17 (d, *J* = 8.0

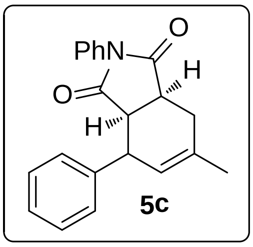
Hz, 2 H), 5.86 (br, 1 H), 3.74 (br, 1 H), 3.51~3.47 (m, 2 H), 2.76 (d, *J* = 13.6 Hz, 1 H), 2.41~2.36 (m, 1 H), 1.88 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 173.8, 170.7, 138.1, 136.7, 128.7(CH×2), 128.5(CH×2), 127.7, 123.1, 45.9, 41.0, 40.0, 27.5, 23.5.; HRMS calcd for $C_{15}H_{14}O_3$: 242.0943; Found : 242.0942.

Spectra data for (3aR,7aS)-2,6-dimethyl-4-phenyl-3a,4,7,7a-tetrahydro-1H-isoindole-1,3(2H)-dione (5b)



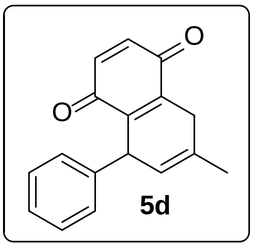
IR (neat, cm^{-1}): 30303 (s), 1760 (s), 1710 (s), 1050 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29~7.21 (m, 3 H), 7.12 (d, *J* = 8.4 Hz, 2 H), 5.80~5.79 (m, 1 H), 3.72 (br , 1 H), 3.27~3.23 (m, 1 H), 3.16 (td, *J* = 8.8, 2.4 Hz, 1 H), 2.77 (dd, *J* = 16.8, 2.4 Hz, 1 H), 2.37~2.31 (m, 1 H), 1.83 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 179.3, 177.2, 139.2, 135.8, 128.6(CH×2), 127.9(CH×2), 126.8 122.8, 45.1, 41.4, 38.9, 27.3, 24.0, 23.3.; HRMS calcd for $C_{16}H_{17}NO_2$: 222.1259; Found : 255.1257.

Spectra data for (3aR,7aS)-6-methyl-2,4-diphenyl-3a,4,7,7a-tetrahydro-1H-isoindole-1,3(2H)-dione (5c)



IR (neat, cm^{-1}): 3070 (s), 3020 (s). 1790 (s), 1070 ; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.28~7.23 (m, 6 H), 7.15 (d, *J* = 8.0 Hz, 2 H), 6.59 (d, *J* = 8.0 Hz, 2 H), 5.85~5.83 (m, 1 H), 3.92~3.89 (m, 1 H), 3.42~3.34 (m, 2 H), 2.91 (d, *J* = 18.4 Hz, 1 H), 2.50~2.43 (m, 1 H), 1.89 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.5, 176.5, 139.0, 135.0, 131.4, 129.1(CH×2), 128.7(CH×2), 128.3(CH×2), 128.2, 127.2, 126.1(CH×2), 122.7, 44.5, 41.4, 38.6, 26.5, 23.4.; HRMS calcd for $C_{21}H_{19}NO_2$: 317.1416; Found : 317.1416.

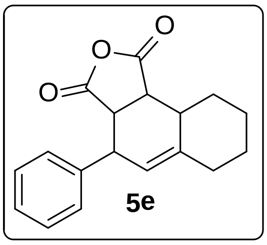
Spcetra data for 7-methyl-5-phenylnaphthalene-1,4(5H,8H)-dione (5d)



IR (neat, cm^{-1}): 3030 (w), 3010 (s), 1690 (s), 1150 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26~7.16 (m, 5 H), 6.70 (d, *J* = 10.4 Hz, 1 H), 6.60 (d, *J* = 10.4 Hz, 1 H), 5.58~5.57 (m, 1 H), 4.58~4.56 (m, 1 H), 3.12~3.09 (m, 2 H), 1.81 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 187.3, 186.1, 142.9, 141.8, 139.2, 136.6, 135.9, 128.9, 128.5(CH×2), 128.3(CH×2), 126.8, 122.9, 41.5, 28.9, 22.7.; HRMS calcd for $C_{17}H_{14}O_2$: 250.0994; Found : 250.0992.

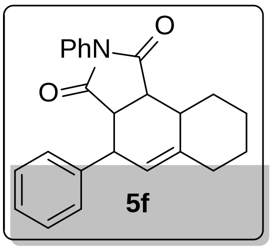
Spectra data for

4-phenyl-3a,4,7,8,9,9a-hexahydronaphtho[1,2-c]furan-1,3(6H,9bH)-dione (5e)


5e

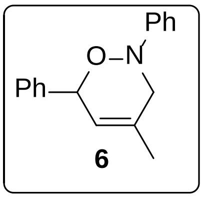
IR (neat, cm^{-1}): 3020 (s), 1800 (s), 1310 (s). 1000 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33~7.26 (m, 3 H), 7.21~7.19 (m, 2 H), 6.66~6.65 (m, 1 H), 3.85~3.82 (m, 1 H), 3.48 (t, *J* = 8.0 Hz, 1 H), 3.39 (t, *J* = 8.0 Hz, 1 H), 2.58 (br, 1 H), 2.42~2.38 (m, 1 H), 2.29~2.26 (m, 1 H), 2.15~2.08 (m, 1 H), 2.00~1.90 (m, 2 H), 1.85~1.80 (m, 1 H), 1.50~1.45 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 170.6, 170.5, 141.1, 137.8, 129.1(CH×2), 128.5(CH×2), 128.0, 119.0, 45.7, 42.1, 39.5, 34.5, 29.5, 26.2, 25.9.; HRMS calcd for $C_{18}H_{18}O_3$: 282.1256; Found : 282.1256.

Spectra data for 2,4-diphenyl-3a,4,6,7,8,9,9a,9b-octahydro-1H-benzo[e]isoindole-1,3(2H)-dione (5f)


5f

IR (neat, cm^{-1}): 3100 (s), 3010 (s), 1820 (s), 1150 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29~7.23 (m, 5 H), 7.22~7.19 (m, 3 H), 6.40~6.37 (m, 2 H), 5.65~5.64 (m, 1 H), 3.94~3.91 (m, 1 H), 3.44~3.34 (m, 2 H), 2.72~2.67 (m, 1 H), 2.62~2.59 (m, 1 H), 2.42~2.38 (m, 1 H), 2.15~2.10 (m, 1 H), 2.01~1.92 (m, 2 H), 1.86~1.83 (m, 1 H), 1.51~1.44 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.2, 176.0, 140.8, 138.8, 131.2, 129.7(CH×2), 128.6(CH×2), 128.3(CH×2), 128.1, 127.3, 126.3(CH×2), 119.1, 44.8, 40.9, 40.6, 36.1, 35.2, 30.3, 27.0, 26.6.; HRMS calcd for $C_{24}H_{23}NO_2$: 357.1729; Found : 357.1729.

Spectra data for 4-methyl-2,6-diphenyl-3,6-dihydro-2H-1,2-oxazine (6)


6

IR (neat, cm^{-1}): 3050 (s), 1350 (s), 1075 (s), 1050 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 (d, J = 6.4 Hz, 2 H), 7.37~7.23 (m, 5 H), 7.10 (d, J = 7.6 Hz, 2 H), 6.95 (t, J = 7.6 Hz, 1 H), 5.75~5.74 (m, 1 H), 5.53~5.20 (m, 1 H), 3.82~3.72 (m, 2 H), 1.88 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.2, 139.5, 131.5(CH×2), 128.3(CH×2), 128.2, 128.0(CH×2), 122.9, 122.1, 115.8(CH×2), 79.2, 55.3, 20.3; HRMS calcd for $C_{17}H_{17}NO$: 251.1310; Found : 251.1310.

(d) X-Ray data for compound (5c)

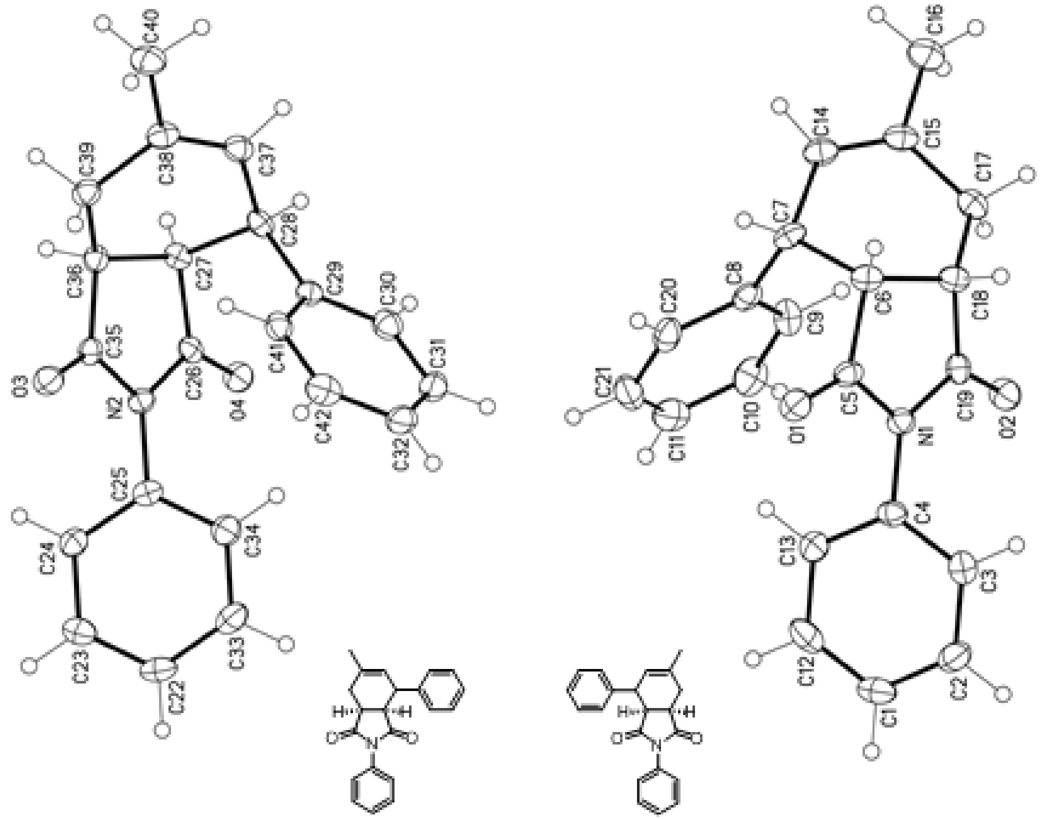


Table 1. Crystal data and structure refinement for twin5.

Identification code	twin5	
Empirical formula	C42 H38 N2 O4	
Formula weight	634.74	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21 1	
Unit cell dimensions	a = 9.7876(18) Å	α= 90°.
	b = 8.3007(14) Å	β= 94.442(4)°.
	c = 19.986(4) Å	γ = 90°.
Volume	1618.8(5) Å^3	
Z	2	
Density (calculated)	1.302 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	672	
Crystal size	0.28 x 0.25 x 0.22 mm^3	
Theta range for data collection	2.04 to 26.54°.	
Index ranges	-12<=h<=12, 0<=k<=10, 0<=l<=25	

Reflections collected	3637
Independent reflections	3637 [R(int) = 0.0399]
Completeness to theta = 26.54°	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7454 and 0.6525
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3637 / 1 / 436
Goodness-of-fit on F^2	1.031
Final R indices [I>2sigma(I)]	R1 = 0.0337, wR2 = 0.0704
R indices (all data)	R1 = 0.0395, wR2 = 0.0727
Absolute structure parameter	0.5(14)
Largest diff. peak and hole	0.153 and -0.179 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for twin5. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	7386(2)	7205(3)	3743(1)	23(1)
O(2)	10339(2)	4223(3)	5121(1)	24(1)
O(3)	5247(2)	8757(3)	-215(1)	22(1)
O(4)	2790(2)	5834(3)	1207(1)	21(1)
N(1)	8612(2)	5640(3)	4534(1)	18(1)
N(2)	3728(2)	7349(3)	391(1)	16(1)
C(1)	5374(3)	3924(4)	5602(2)	27(1)
C(2)	6402(3)	4834(4)	5921(2)	27(1)
C(3)	7467(3)	5424(4)	5570(1)	23(1)
C(4)	7497(3)	5067(4)	4895(1)	18(1)
C(5)	8451(3)	6604(4)	3962(1)	19(1)
C(6)	9826(3)	6677(4)	3662(1)	19(1)
C(7)	9791(3)	5607(4)	3019(1)	20(1)
C(8)	8929(3)	4087(4)	3059(1)	20(1)
C(9)	9374(3)	2763(4)	3448(1)	24(1)
C(10)	8539(3)	1443(4)	3509(2)	28(1)
C(11)	7253(3)	1388(5)	3170(2)	30(1)
C(12)	5413(3)	3574(4)	4926(2)	29(1)
C(13)	6480(3)	4139(4)	4570(1)	25(1)
C(14)	11245(3)	5231(4)	2864(1)	22(1)
C(15)	12288(3)	5051(4)	3319(1)	21(1)
C(16)	13695(3)	4560(4)	3135(2)	29(1)
C(17)	12151(3)	5266(4)	4061(1)	22(1)
C(18)	10839(3)	6117(4)	4238(1)	19(1)
C(19)	9968(3)	5180(4)	4684(1)	18(1)
C(20)	7644(3)	4002(5)	2717(1)	25(1)
C(21)	6822(3)	2661(4)	2766(2)	29(1)
C(22)	202(3)	9178(4)	-678(2)	25(1)
C(23)	1145(3)	8294(4)	-1009(1)	24(1)
C(24)	2305(3)	7670(4)	-656(1)	21(1)
C(25)	2502(3)	7933(4)	28(1)	17(1)
C(26)	3775(3)	6420(4)	972(1)	16(1)
C(27)	5251(3)	6394(4)	1268(1)	16(1)

C(28)	5391(3)	7487(4)	1903(1)	18(1)
C(29)	4410(3)	8928(4)	1879(1)	18(1)
C(30)	3295(3)	8930(4)	2271(1)	22(1)
C(31)	2404(3)	10234(4)	2264(1)	25(1)
C(32)	2588(3)	11538(4)	1854(2)	26(1)
C(33)	423(3)	9437(4)	8(2)	25(1)
C(34)	1569(3)	8811(4)	360(1)	22(1)
C(35)	5029(3)	7818(4)	230(1)	16(1)
C(36)	6071(3)	6946(4)	686(1)	17(1)
C(37)	6851(3)	8050(4)	2036(1)	19(1)
C(38)	7742(3)	8247(4)	1574(2)	20(1)
C(39)	7413(3)	7868(4)	849(1)	19(1)
C(40)	9169(3)	8881(4)	1753(2)	26(1)
C(41)	4592(3)	10271(4)	1474(1)	20(1)
C(42)	3688(3)	11553(4)	1455(2)	26(1)

Table 3. Bond lengths [Å] and angles [°] for twin5.

O(1)-C(5)	1.207(3)
O(2)-C(19)	1.215(3)
O(3)-C(35)	1.213(3)
O(4)-C(26)	1.207(3)
N(1)-C(19)	1.392(4)
N(1)-C(5)	1.394(4)
N(1)-C(4)	1.436(3)
N(2)-C(35)	1.393(4)
N(2)-C(26)	1.393(4)
N(2)-C(25)	1.437(3)
C(1)-C(2)	1.375(5)
C(1)-C(12)	1.385(4)
C(1)-H(1)	0.9500
C(2)-C(3)	1.390(4)
C(2)-H(2)	0.9500
C(3)-C(4)	1.383(4)
C(3)-H(3)	0.9500
C(4)-C(13)	1.381(4)
C(5)-C(6)	1.516(4)
C(6)-C(18)	1.533(4)
C(6)-C(7)	1.561(4)
C(6)-H(6)	1.0000
C(7)-C(14)	1.513(4)
C(7)-C(8)	1.523(4)
C(7)-H(7)	1.0000
C(8)-C(20)	1.387(4)
C(8)-C(9)	1.396(4)
C(9)-C(10)	1.378(4)
C(9)-H(9)	0.9500
C(10)-C(11)	1.384(4)
C(10)-H(10)	0.9500
C(11)-C(21)	1.377(5)
C(11)-H(11)	0.9500
C(12)-C(13)	1.390(4)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500

C(14)-C(15)	1.323(4)
C(14)-H(14)	0.9500
C(15)-C(16)	1.508(4)
C(15)-C(17)	1.509(4)
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-C(18)	1.531(4)
C(17)-H(17A)	0.9900
C(17)-H(17B)	0.9900
C(18)-C(19)	1.499(4)
C(18)-H(18)	1.0000
C(20)-C(21)	1.381(5)
C(20)-H(20)	0.9500
C(21)-H(21)	0.9500
C(22)-C(23)	1.386(4)
C(22)-C(33)	1.389(4)
C(22)-H(22)	0.9500
C(23)-C(24)	1.390(4)
C(23)-H(23)	0.9500
C(24)-C(25)	1.384(4)
C(24)-H(24)	0.9500
C(25)-C(34)	1.377(4)
C(26)-C(27)	1.518(4)
C(27)-C(36)	1.535(4)
C(27)-C(28)	1.557(4)
C(27)-H(27)	1.0000
C(28)-C(37)	1.508(4)
C(28)-C(29)	1.532(4)
C(28)-H(28)	1.0000
C(29)-C(30)	1.392(4)
C(29)-C(41)	1.397(4)
C(30)-C(31)	1.389(4)
C(30)-H(30)	0.9500
C(31)-C(32)	1.377(5)
C(31)-H(31)	0.9500
C(32)-C(42)	1.389(4)
C(32)-H(32)	0.9500

C(33)-C(34)	1.378(4)
C(33)-H(33)	0.9500
C(34)-H(34)	0.9500
C(35)-C(36)	1.500(4)
C(36)-C(39)	1.533(4)
C(36)-H(36)	1.0000
C(37)-C(38)	1.327(4)
C(37)-H(37)	0.9500
C(38)-C(39)	1.493(4)
C(38)-C(40)	1.510(4)
C(39)-H(39A)	0.9900
C(39)-H(39B)	0.9900
C(40)-H(40A)	0.9800
C(40)-H(40B)	0.9800
C(40)-H(40C)	0.9800
C(41)-C(42)	1.382(4)
C(41)-H(41)	0.9500
C(42)-H(42)	0.9500
C(19)-N(1)-C(5)	112.3(2)
C(19)-N(1)-C(4)	123.4(2)
C(5)-N(1)-C(4)	124.1(2)
C(35)-N(2)-C(26)	112.0(2)
C(35)-N(2)-C(25)	122.2(2)
C(26)-N(2)-C(25)	125.4(2)
C(2)-C(1)-C(12)	119.6(3)
C(2)-C(1)-H(1)	120.2
C(12)-C(1)-H(1)	120.2
C(1)-C(2)-C(3)	120.7(3)
C(1)-C(2)-H(2)	119.7
C(3)-C(2)-H(2)	119.7
C(4)-C(3)-C(2)	119.3(3)
C(4)-C(3)-H(3)	120.3
C(2)-C(3)-H(3)	120.3
C(13)-C(4)-C(3)	120.6(3)
C(13)-C(4)-N(1)	119.7(2)
C(3)-C(4)-N(1)	119.6(3)
O(1)-C(5)-N(1)	125.0(3)

O(1)-C(5)-C(6)	127.5(3)
N(1)-C(5)-C(6)	107.5(2)
C(5)-C(6)-C(18)	103.5(2)
C(5)-C(6)-C(7)	109.8(2)
C(18)-C(6)-C(7)	114.7(2)
C(5)-C(6)-H(6)	109.6
C(18)-C(6)-H(6)	109.6
C(7)-C(6)-H(6)	109.6
C(14)-C(7)-C(8)	111.9(2)
C(14)-C(7)-C(6)	109.0(2)
C(8)-C(7)-C(6)	113.9(2)
C(14)-C(7)-H(7)	107.2
C(8)-C(7)-H(7)	107.2
C(6)-C(7)-H(7)	107.2
C(20)-C(8)-C(9)	117.8(3)
C(20)-C(8)-C(7)	120.0(3)
C(9)-C(8)-C(7)	122.1(2)
C(10)-C(9)-C(8)	121.0(3)
C(10)-C(9)-H(9)	119.5
C(8)-C(9)-H(9)	119.5
C(9)-C(10)-C(11)	120.4(3)
C(9)-C(10)-H(10)	119.8
C(11)-C(10)-H(10)	119.8
C(21)-C(11)-C(10)	119.2(3)
C(21)-C(11)-H(11)	120.4
C(10)-C(11)-H(11)	120.4
C(1)-C(12)-C(13)	120.4(3)
C(1)-C(12)-H(12)	119.8
C(13)-C(12)-H(12)	119.8
C(4)-C(13)-C(12)	119.3(3)
C(4)-C(13)-H(13)	120.3
C(12)-C(13)-H(13)	120.3
C(15)-C(14)-C(7)	124.8(3)
C(15)-C(14)-H(14)	117.6
C(7)-C(14)-H(14)	117.6
C(14)-C(15)-C(16)	122.2(3)
C(14)-C(15)-C(17)	122.7(2)
C(16)-C(15)-C(17)	115.1(2)

C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(15)-C(17)-C(18)	114.9(2)
C(15)-C(17)-H(17A)	108.5
C(18)-C(17)-H(17A)	108.5
C(15)-C(17)-H(17B)	108.5
C(18)-C(17)-H(17B)	108.5
H(17A)-C(17)-H(17B)	107.5
C(19)-C(18)-C(17)	115.1(2)
C(19)-C(18)-C(6)	103.9(2)
C(17)-C(18)-C(6)	118.1(2)
C(19)-C(18)-H(18)	106.3
C(17)-C(18)-H(18)	106.3
C(6)-C(18)-H(18)	106.3
O(2)-C(19)-N(1)	124.2(3)
O(2)-C(19)-C(18)	127.9(2)
N(1)-C(19)-C(18)	107.8(2)
C(21)-C(20)-C(8)	121.2(3)
C(21)-C(20)-H(20)	119.4
C(8)-C(20)-H(20)	119.4
C(11)-C(21)-C(20)	120.4(3)
C(11)-C(21)-H(21)	119.8
C(20)-C(21)-H(21)	119.8
C(23)-C(22)-C(33)	119.6(3)
C(23)-C(22)-H(22)	120.2
C(33)-C(22)-H(22)	120.2
C(22)-C(23)-C(24)	120.2(3)
C(22)-C(23)-H(23)	119.9
C(24)-C(23)-H(23)	119.9
C(25)-C(24)-C(23)	119.2(3)
C(25)-C(24)-H(24)	120.4
C(23)-C(24)-H(24)	120.4
C(34)-C(25)-C(24)	120.8(3)
C(34)-C(25)-N(2)	119.7(2)

C(24)-C(25)-N(2)	119.4(2)
O(4)-C(26)-N(2)	124.9(2)
O(4)-C(26)-C(27)	127.4(2)
N(2)-C(26)-C(27)	107.6(2)
C(26)-C(27)-C(36)	103.8(2)
C(26)-C(27)-C(28)	109.3(2)
C(36)-C(27)-C(28)	115.2(2)
C(26)-C(27)-H(27)	109.5
C(36)-C(27)-H(27)	109.5
C(28)-C(27)-H(27)	109.5
C(37)-C(28)-C(29)	110.3(2)
C(37)-C(28)-C(27)	110.3(2)
C(29)-C(28)-C(27)	114.5(2)
C(37)-C(28)-H(28)	107.1
C(29)-C(28)-H(28)	107.1
C(27)-C(28)-H(28)	107.1
C(30)-C(29)-C(41)	117.7(3)
C(30)-C(29)-C(28)	120.0(3)
C(41)-C(29)-C(28)	122.3(2)
C(31)-C(30)-C(29)	120.9(3)
C(31)-C(30)-H(30)	119.5
C(29)-C(30)-H(30)	119.5
C(32)-C(31)-C(30)	120.5(3)
C(32)-C(31)-H(31)	119.7
C(30)-C(31)-H(31)	119.7
C(31)-C(32)-C(42)	119.4(3)
C(31)-C(32)-H(32)	120.3
C(42)-C(32)-H(32)	120.3
C(34)-C(33)-C(22)	120.2(3)
C(34)-C(33)-H(33)	119.9
C(22)-C(33)-H(33)	119.9
C(25)-C(34)-C(33)	119.9(3)
C(25)-C(34)-H(34)	120.1
C(33)-C(34)-H(34)	120.1
O(3)-C(35)-N(2)	124.4(3)
O(3)-C(35)-C(36)	127.2(3)
N(2)-C(35)-C(36)	108.4(2)
C(35)-C(36)-C(39)	114.8(2)

C(35)-C(36)-C(27)	103.5(2)
C(39)-C(36)-C(27)	118.5(2)
C(35)-C(36)-H(36)	106.4
C(39)-C(36)-H(36)	106.4
C(27)-C(36)-H(36)	106.4
C(38)-C(37)-C(28)	125.5(3)
C(38)-C(37)-H(37)	117.3
C(28)-C(37)-H(37)	117.3
C(37)-C(38)-C(39)	123.0(2)
C(37)-C(38)-C(40)	121.6(3)
C(39)-C(38)-C(40)	115.4(2)
C(38)-C(39)-C(36)	115.4(2)
C(38)-C(39)-H(39A)	108.4
C(36)-C(39)-H(39A)	108.4
C(38)-C(39)-H(39B)	108.4
C(36)-C(39)-H(39B)	108.4
H(39A)-C(39)-H(39B)	107.5
C(38)-C(40)-H(40A)	109.5
C(38)-C(40)-H(40B)	109.5
H(40A)-C(40)-H(40B)	109.5
C(38)-C(40)-H(40C)	109.5
H(40A)-C(40)-H(40C)	109.5
H(40B)-C(40)-H(40C)	109.5
C(42)-C(41)-C(29)	121.4(3)
C(42)-C(41)-H(41)	119.3
C(29)-C(41)-H(41)	119.3
C(41)-C(42)-C(32)	120.0(3)
C(41)-C(42)-H(42)	120.0
C(32)-C(42)-H(42)	120.0

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^3$) for twin5. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

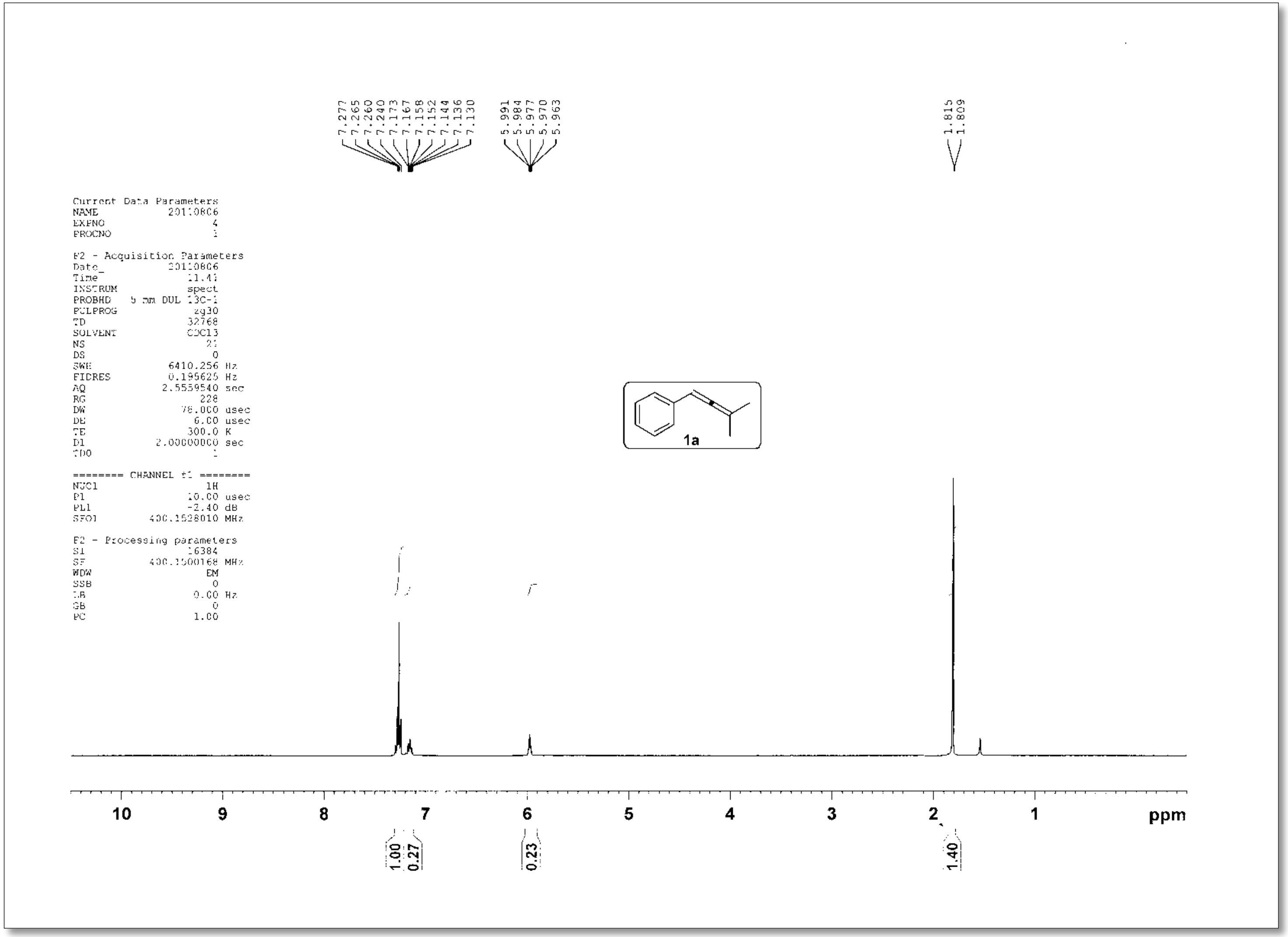
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	23(1)	25(1)	22(1)	2(1)	2(1)	8(1)
O(2)	22(1)	23(1)	26(1)	6(1)	-1(1)	1(1)
O(3)	21(1)	24(1)	23(1)	7(1)	4(1)	3(1)
O(4)	18(1)	23(1)	22(1)	2(1)	3(1)	-6(1)
N(1)	18(1)	19(1)	18(1)	-1(1)	3(1)	0(1)
N(2)	14(1)	15(1)	18(1)	1(1)	1(1)	-1(1)
C(1)	20(1)	30(2)	33(2)	8(2)	10(1)	1(1)
C(2)	28(2)	33(2)	19(1)	3(1)	5(1)	4(2)
C(3)	22(1)	22(2)	23(1)	-2(1)	-2(1)	1(1)
C(4)	16(1)	20(2)	20(1)	4(1)	4(1)	4(1)
C(5)	22(1)	19(2)	18(1)	-3(1)	6(1)	0(1)
C(6)	20(1)	15(2)	23(1)	0(1)	6(1)	2(1)
C(7)	24(1)	20(2)	16(1)	1(1)	7(1)	5(1)
C(8)	22(1)	21(2)	16(1)	-3(1)	5(1)	6(1)
C(9)	24(1)	20(2)	25(1)	-2(1)	-5(1)	2(1)
C(10)	38(2)	20(2)	26(2)	-2(2)	0(1)	2(2)
C(11)	34(2)	26(2)	32(2)	-15(2)	12(2)	-7(2)
C(12)	19(1)	28(2)	41(2)	-6(2)	0(1)	-6(1)
C(13)	23(1)	31(2)	21(1)	-5(1)	2(1)	-1(2)
C(14)	23(1)	19(2)	24(1)	0(1)	8(1)	3(1)
C(15)	20(1)	17(2)	26(1)	-1(1)	10(1)	0(1)
C(16)	23(2)	30(2)	34(2)	-1(2)	6(1)	2(1)
C(17)	17(1)	23(2)	26(1)	1(1)	0(1)	1(1)
C(18)	19(1)	16(2)	23(1)	-1(1)	5(1)	0(1)
C(19)	20(1)	15(2)	19(1)	-2(1)	-2(1)	-2(1)
C(20)	25(1)	31(2)	19(1)	-6(1)	-1(1)	8(1)
C(21)	18(1)	37(2)	31(2)	-15(2)	0(1)	1(2)
C(22)	15(1)	28(2)	31(2)	4(2)	-4(1)	1(1)
C(23)	22(1)	26(2)	22(1)	1(1)	-4(1)	-2(1)
C(24)	20(1)	20(2)	22(1)	-1(1)	3(1)	4(1)
C(25)	15(1)	14(2)	22(1)	2(1)	0(1)	-1(1)
C(26)	20(1)	12(2)	15(1)	-3(1)	0(1)	0(1)
C(27)	18(1)	13(2)	17(1)	2(1)	0(1)	-2(1)

C(28)	23(1)	18(2)	13(1)	2(1)	0(1)	-1(1)
C(29)	15(1)	21(2)	16(1)	-2(1)	-1(1)	-1(1)
C(30)	22(1)	24(2)	20(1)	0(1)	1(1)	-3(1)
C(31)	18(1)	33(2)	24(1)	-10(2)	1(1)	0(1)
C(32)	25(2)	27(2)	27(2)	-10(2)	-5(1)	9(1)
C(33)	20(1)	24(2)	31(2)	3(1)	6(1)	5(1)
C(34)	19(1)	25(2)	22(1)	1(1)	3(1)	-3(1)
C(35)	18(1)	14(2)	17(1)	-2(1)	2(1)	1(1)
C(36)	17(1)	18(2)	17(1)	-1(1)	3(1)	-1(1)
C(37)	20(1)	17(2)	20(1)	-2(1)	-2(1)	0(1)
C(38)	18(1)	17(2)	26(2)	0(1)	-2(1)	0(1)
C(39)	16(1)	21(2)	21(1)	3(1)	4(1)	0(1)
C(40)	20(1)	28(2)	28(1)	-1(2)	-2(1)	-6(1)
C(41)	21(1)	21(2)	18(1)	-3(1)	1(1)	-2(1)
C(42)	31(2)	18(2)	28(2)	-1(1)	-4(1)	0(2)

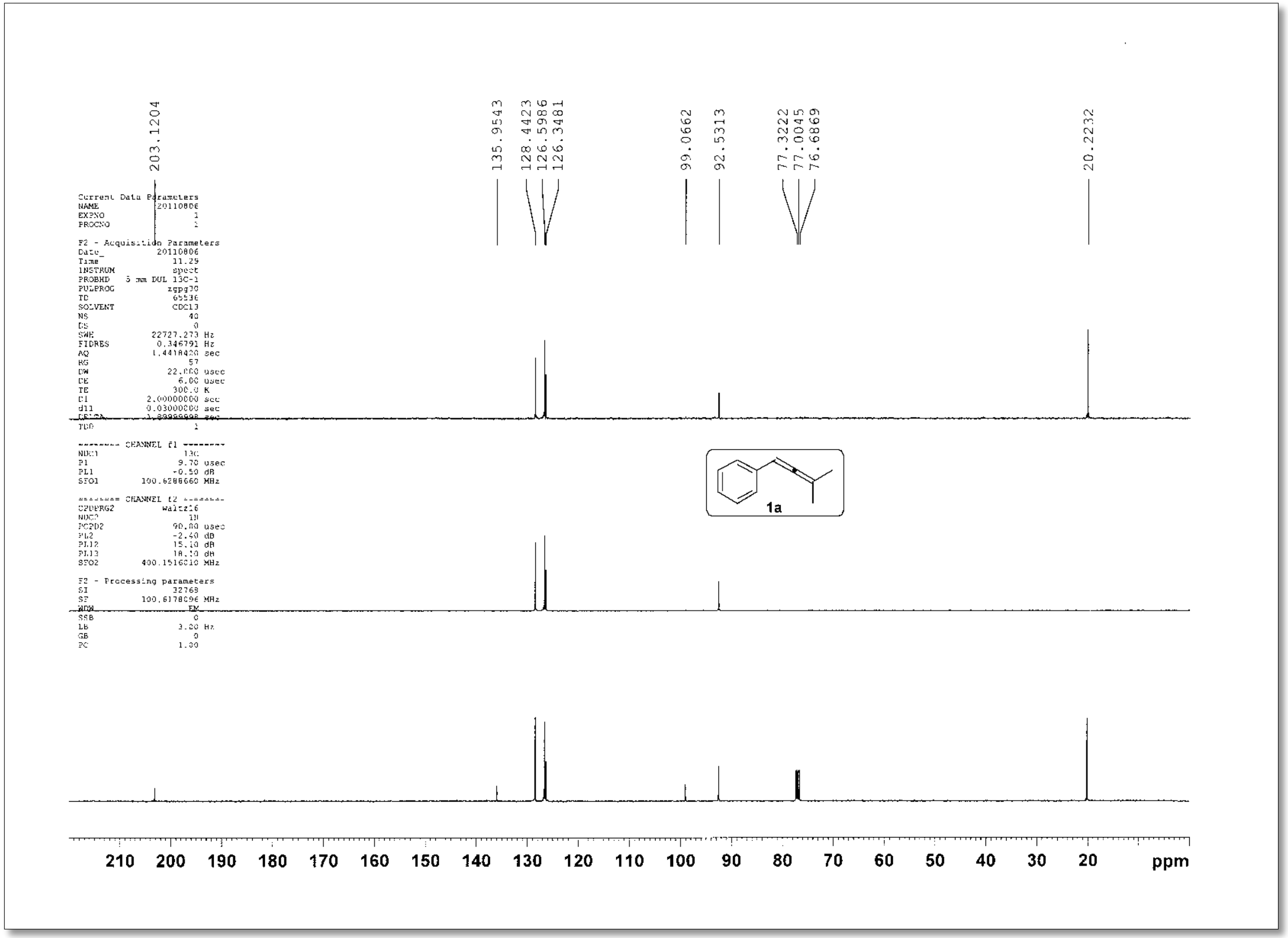
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10 ^{3}) for twin5.

	x	y	z	U(eq)
H(1)	4642	3537	5843	33
H(2)	6383	5062	6386	32
H(3)	8168	6065	5792	27
H(6)	10033	7817	3543	23
H(7)	9369	6266	2638	23
H(9)	10265	2771	3673	28
H(10)	8848	565	3786	34
H(11)	6674	482	3215	36
H(12)	4705	2943	4704	35
H(13)	6509	3890	4107	30
H(14)	11422	5115	2405	26
H(16A)	13700	4496	2646	43
H(16B)	13929	3505	3332	43
H(16C)	14369	5360	3308	43
H(17A)	12949	5890	4254	26
H(17B)	12188	4191	4276	26
H(18)	11138	7109	4494	23
H(20)	7324	4881	2444	30
H(21)	5953	2618	2519	35
H(22)	-591	9605	-920	30
H(23)	997	8114	-1479	29
H(24)	2956	7069	-882	25
H(27)	5516	5265	1396	19
H(28)	5173	6805	2293	22
H(30)	3141	8028	2548	27
H(31)	1662	10226	2543	30
H(32)	1969	12420	1845	32
H(33)	-218	10048	236	30
H(34)	1715	8985	829	27
H(36)	6318	5948	442	21
H(37)	7160	8282	2488	23
H(39A)	8174	7225	689	23

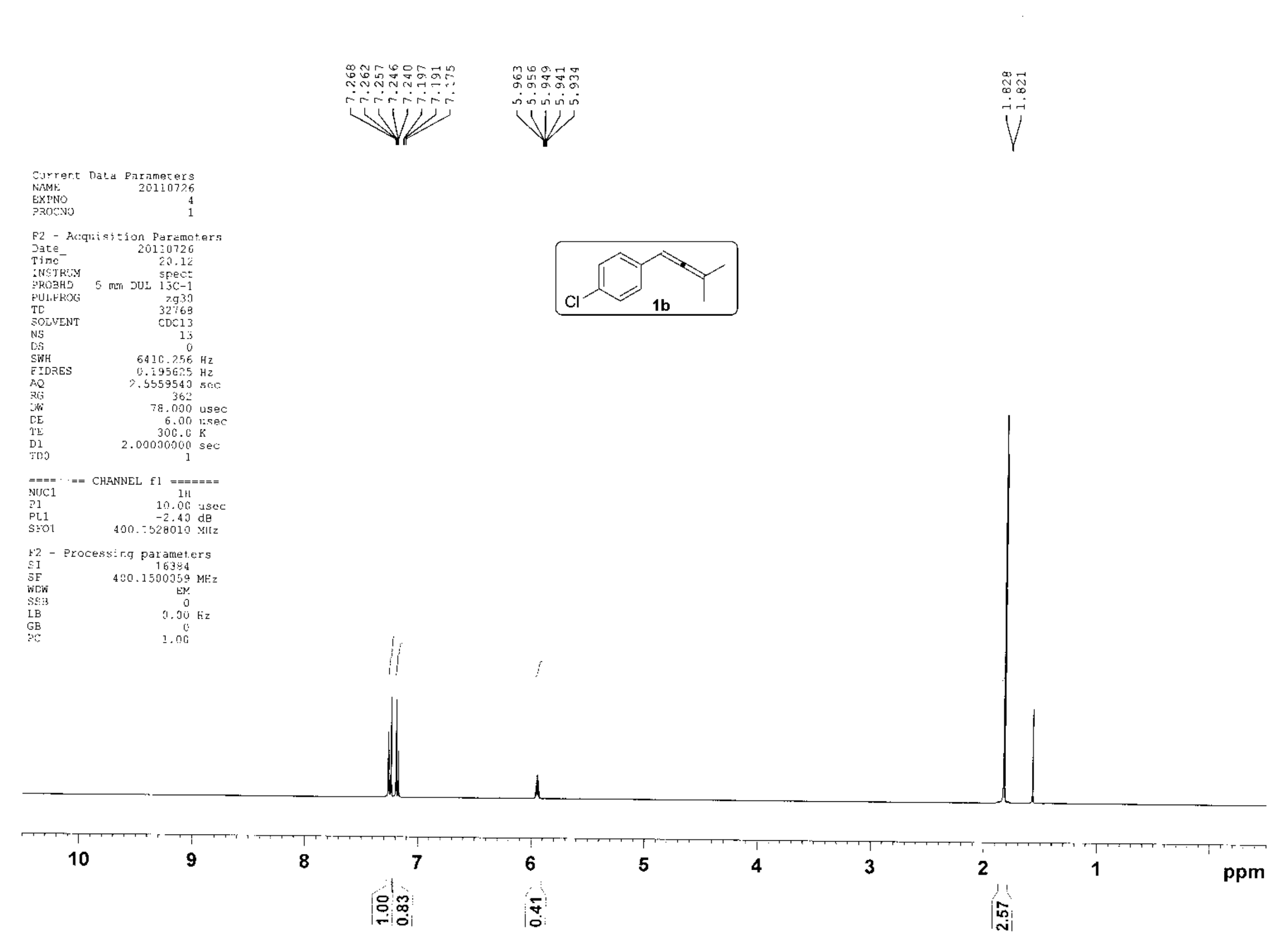
H(39B)	7369	8891	594	23
H(40A)	9284	9106	2236	38
H(40B)	9307	9874	1502	38
H(40C)	9843	8073	1638	38
H(41)	5352	10304	1207	24
H(42)	3820	12444	1169	31



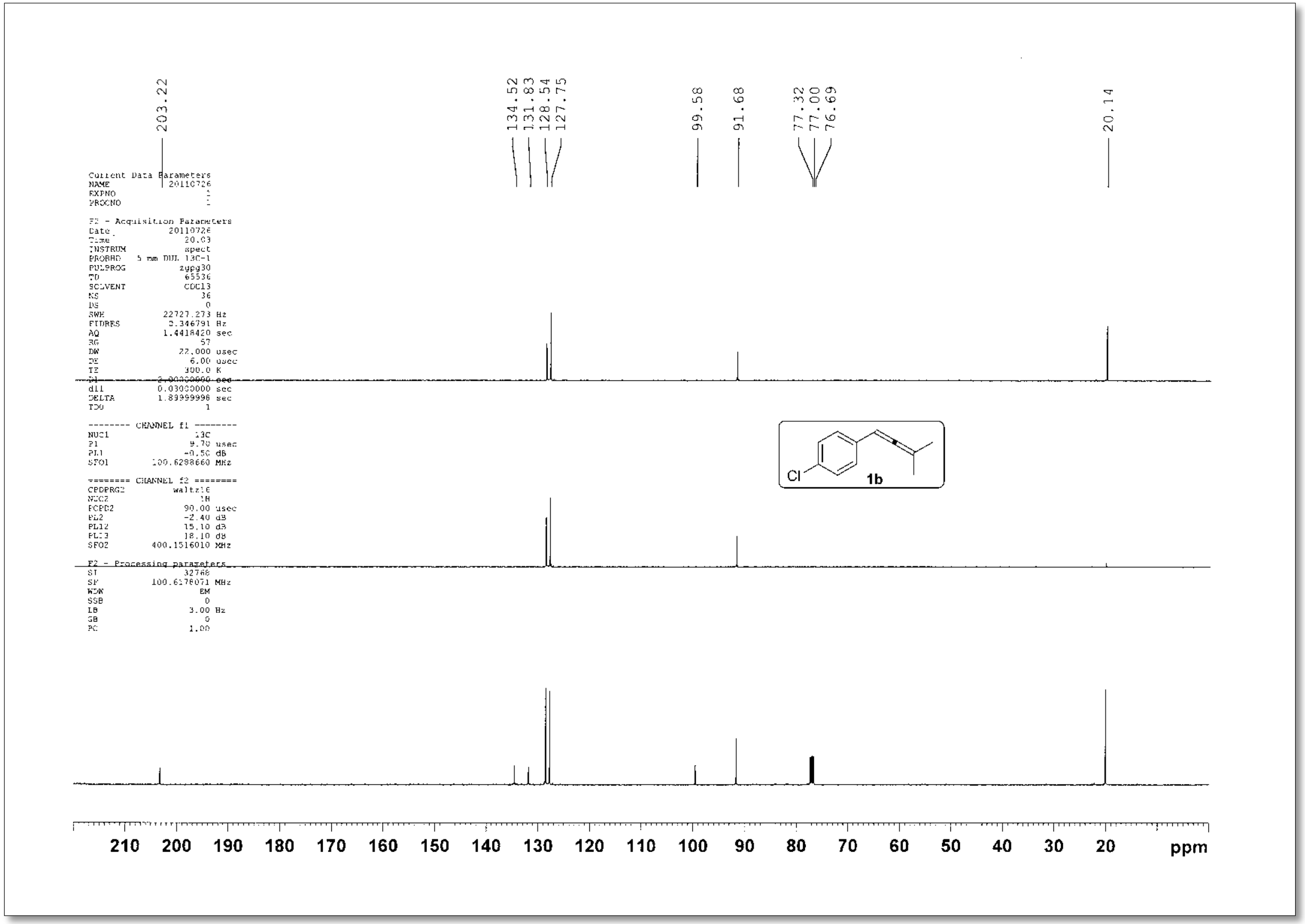
```
Current Data Parameters
NAME               20110806
EXPNO                     4
PROCNO                    1

F2 - Acquisition Parameters
Date_              20110806
Time                  11.41
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       21
DS                        0
SWH                6410.256 Hz
FIDRES             0.195625 Hz
AQ                2.5559540 sec
RG                      228
DW                   78.000 usec
DE                     6.00 usec
TE                    300.0 K
D1               2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.00 usec
PL1                   -2.40 dB
SFO1            400.1528010 MHz

F2 - Processing parameters
SI                    16384
SF              400.1500168 MHz
WDW                      EM
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```



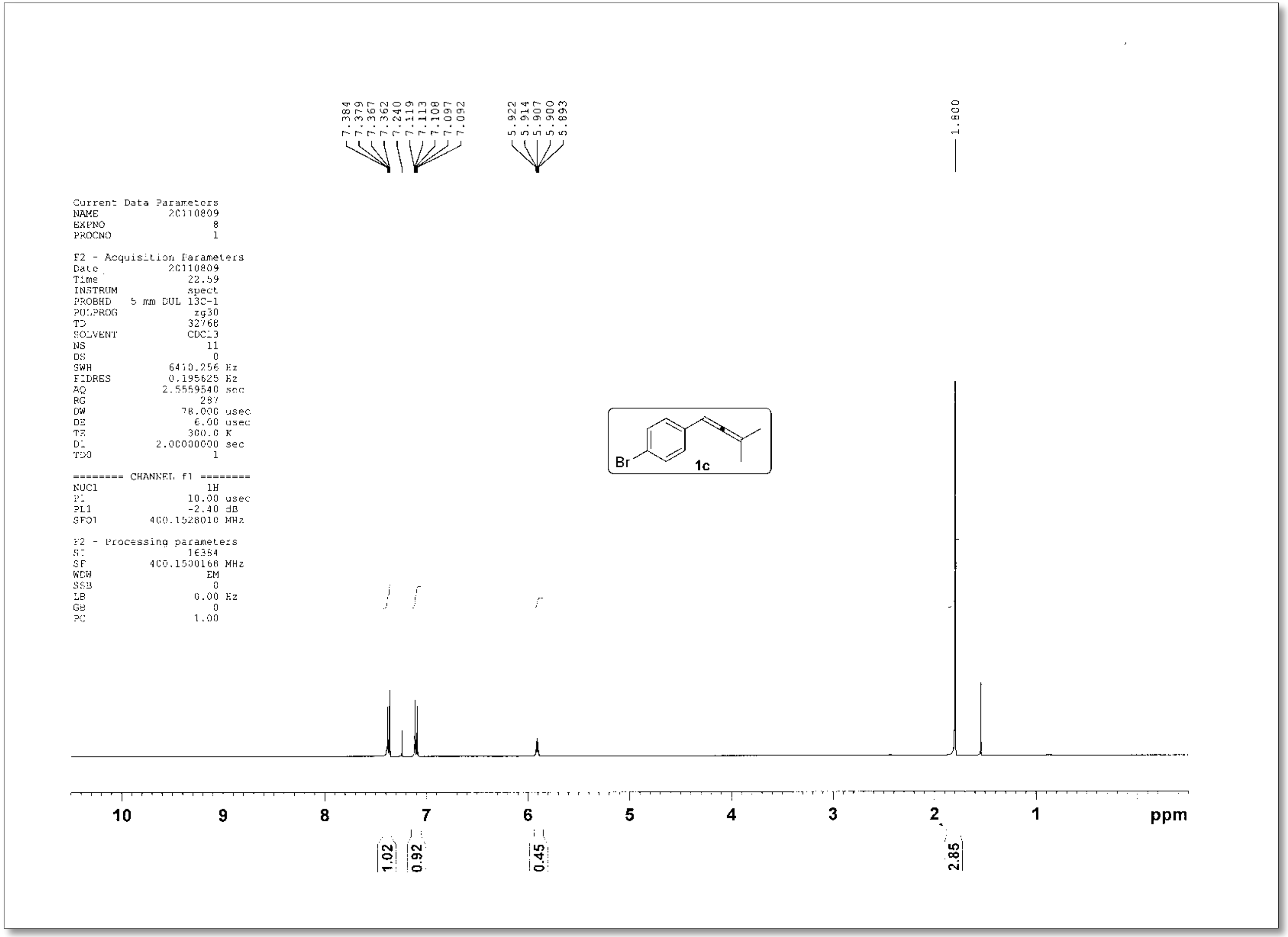
```
Current Data Parameters
NAME            20110806
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110806
Time               11.29
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    40
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178096 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME             20110726
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110726
Time                20.12
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     13
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    362
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 =======
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500059 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

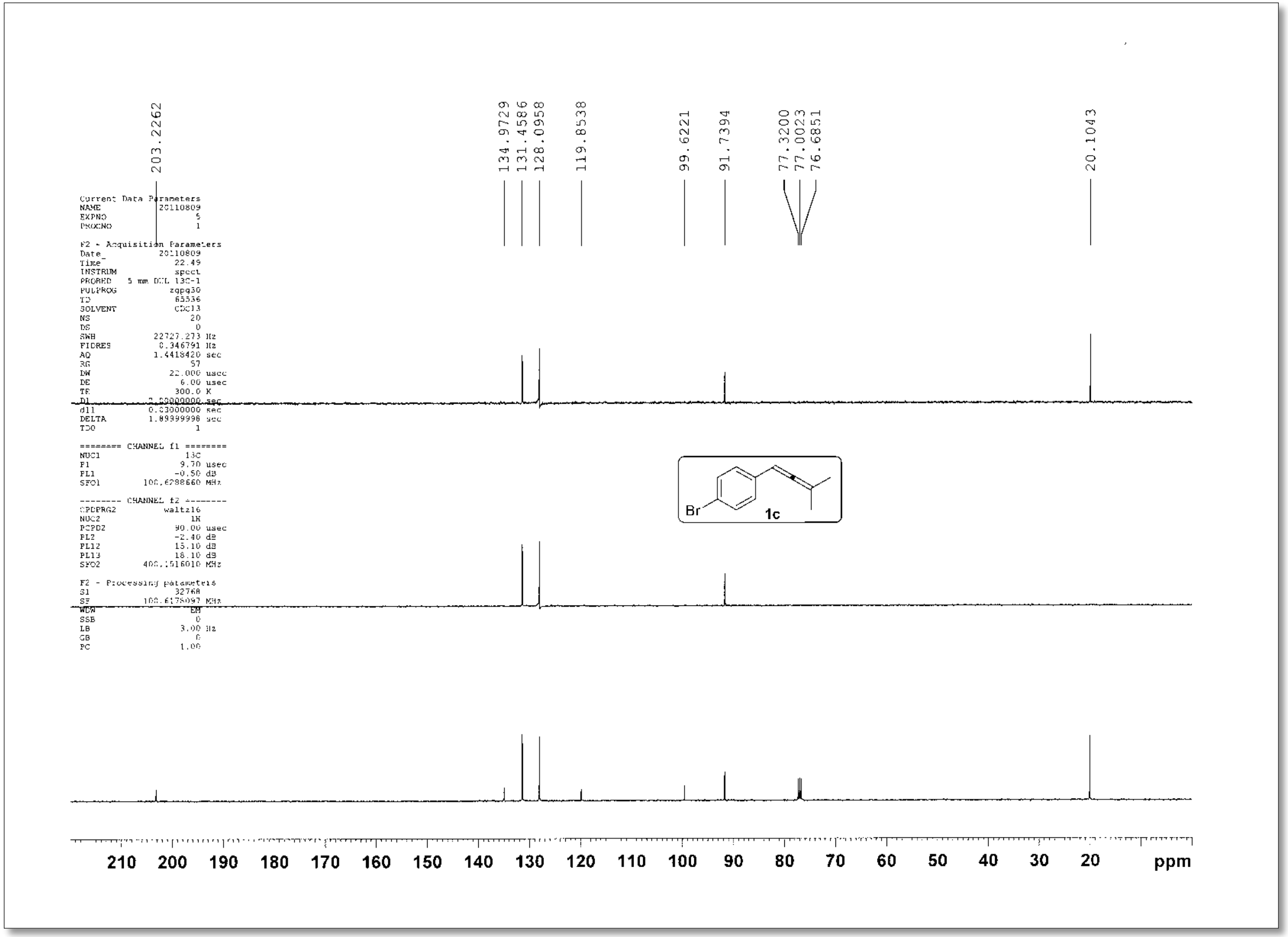


```
Current Data Parameters
NAME            20110726
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110726
Time               20.03
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    36
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999996 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6298660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178071 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```

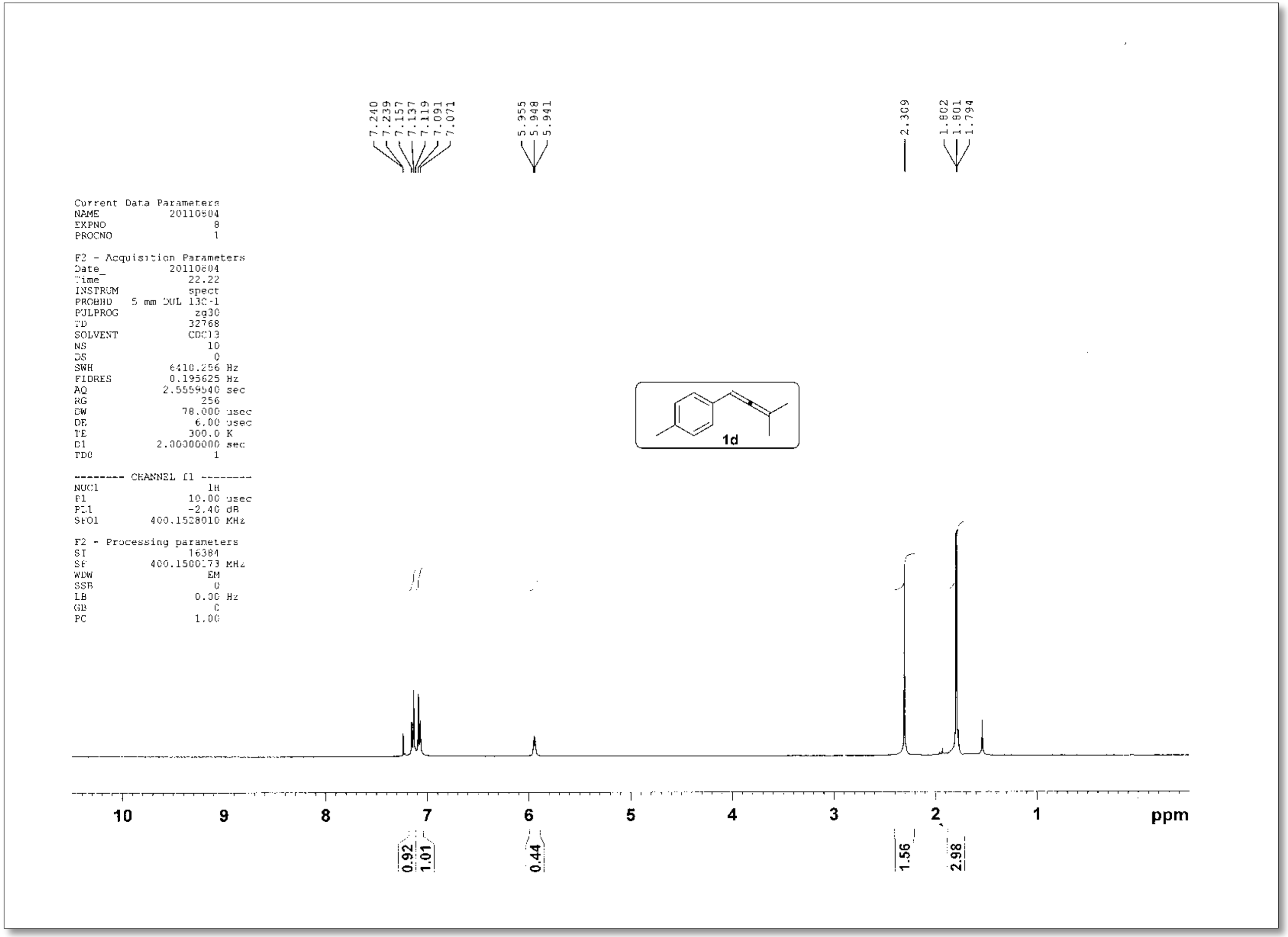


```
Current Data Parameters
NAME              20110809
EXPNO                    8
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110809
Time                 22.59
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      11
DS                       0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ               2.5559540 sec
RG                     287
DW                  78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1           400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF             400.1500168 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

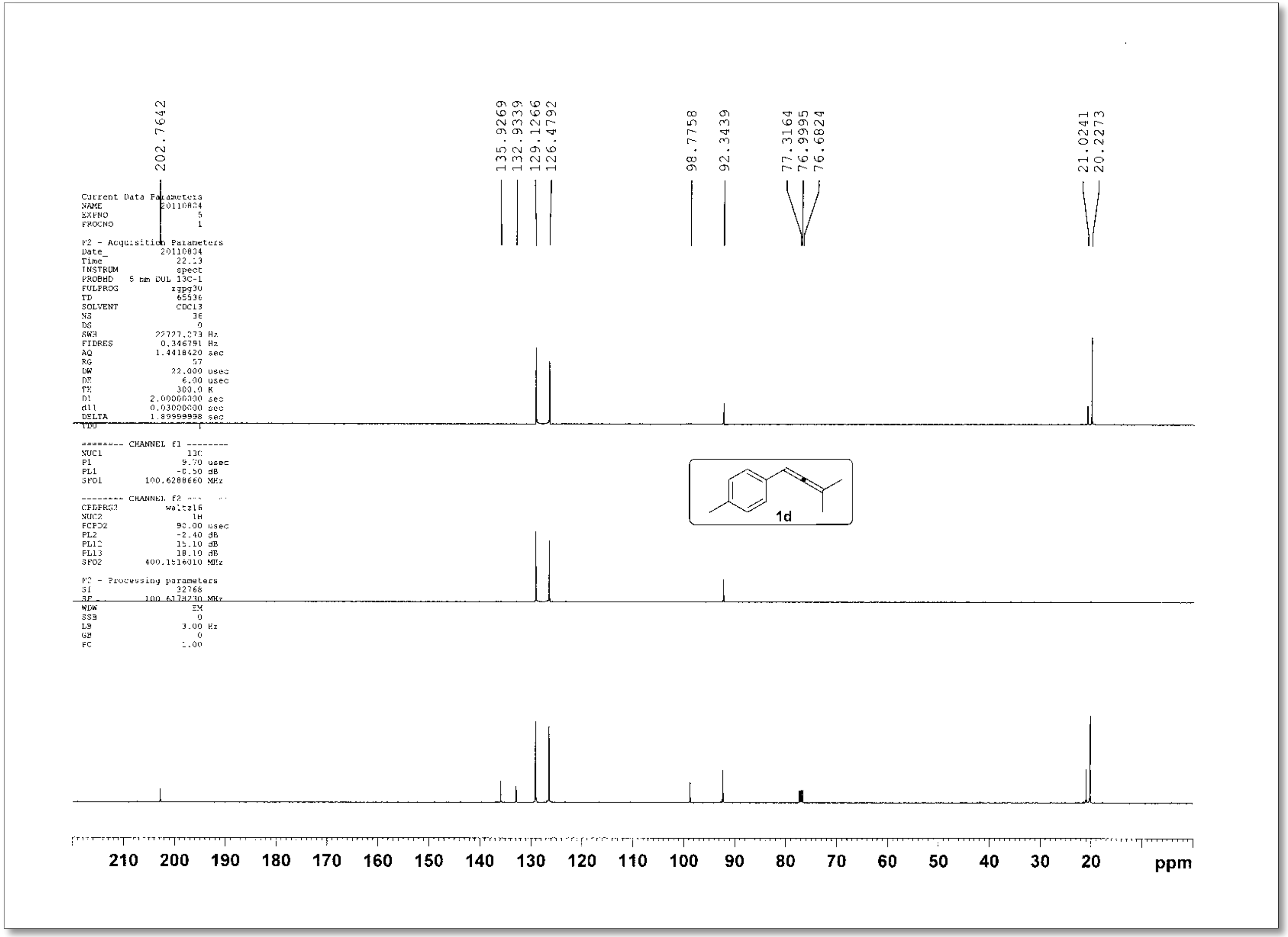
1c



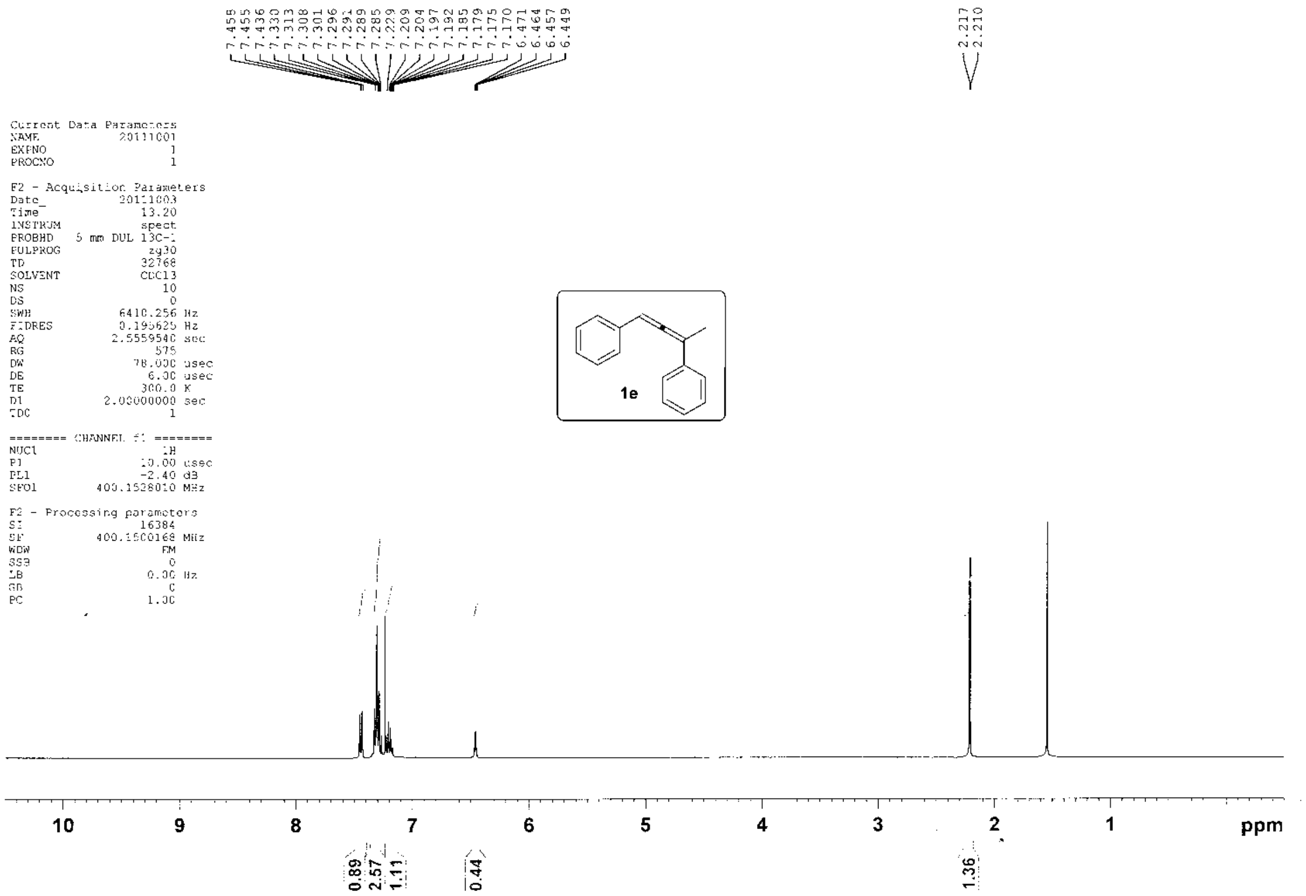
```
Current Data Parameters
NAME             20110809
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110809
Time                22.49
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6298660 MHz

-------- CHANNEL f2 --------
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178097 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



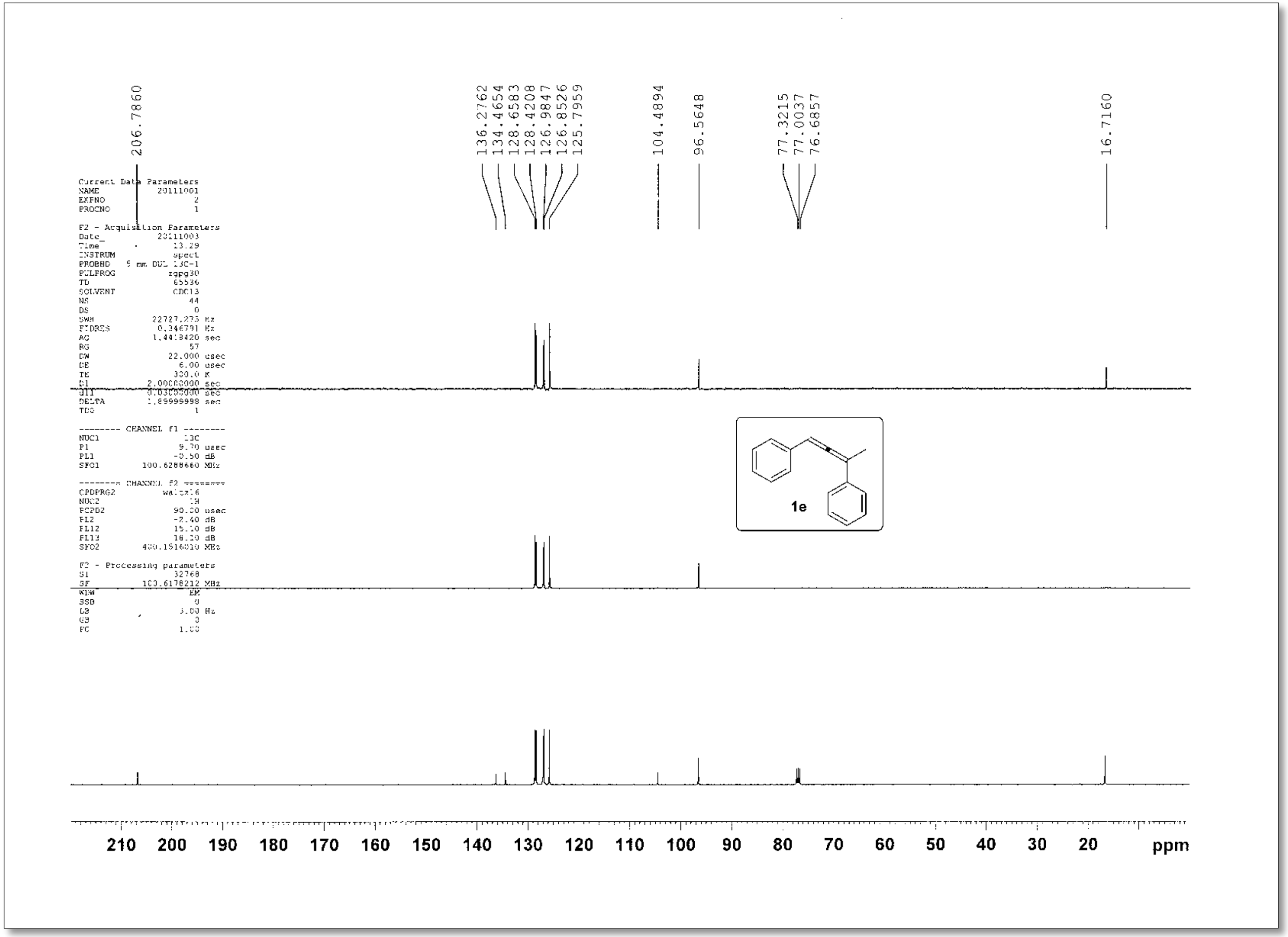
```
Current Data Parameters
NAME            20110604
EXPNO                  8
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110604
Time               22.22
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   256
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500173 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



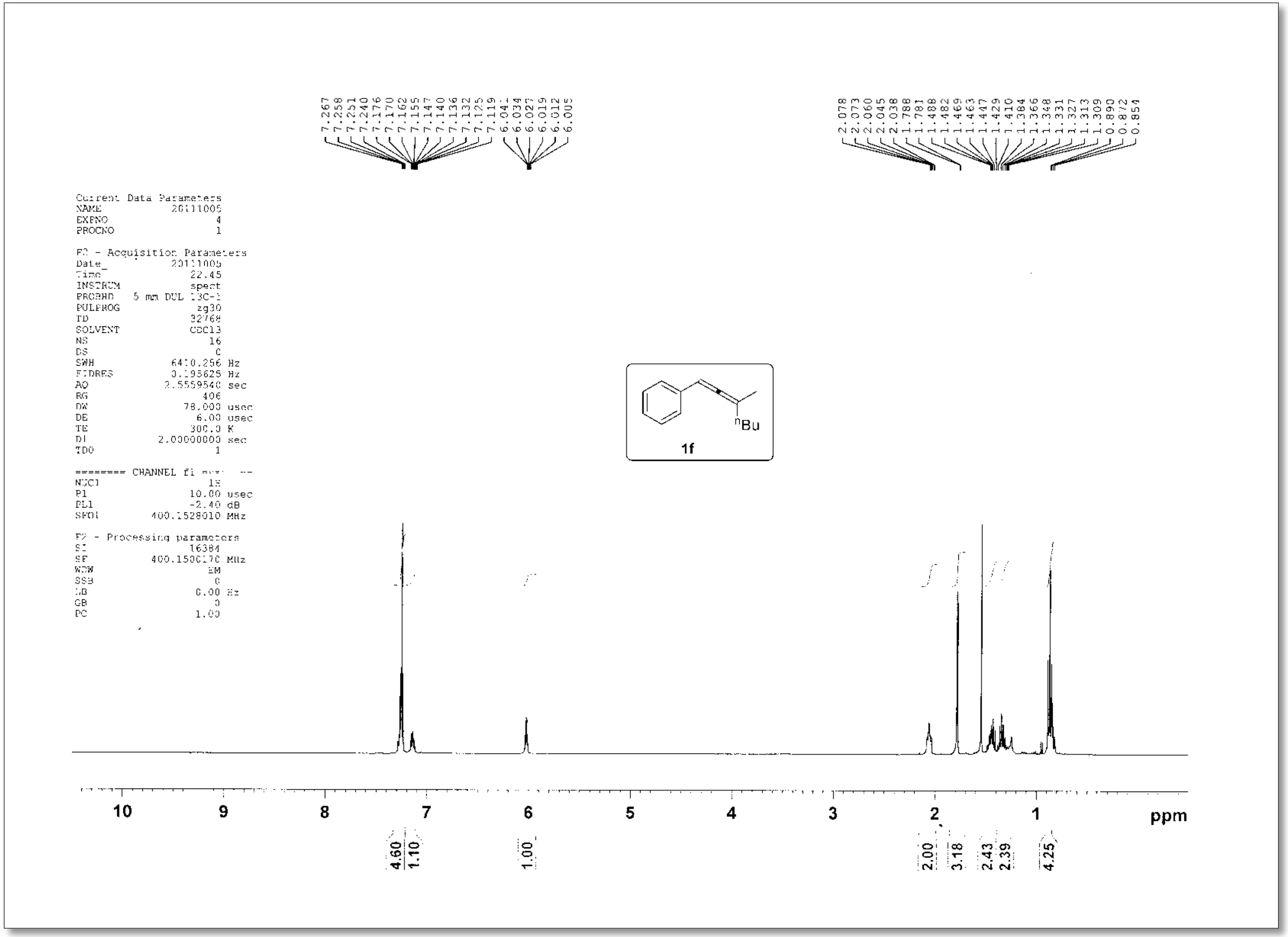
```
Current Data Parameters
NAME             20110824
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110824
Time                22.13
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     36
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178230 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



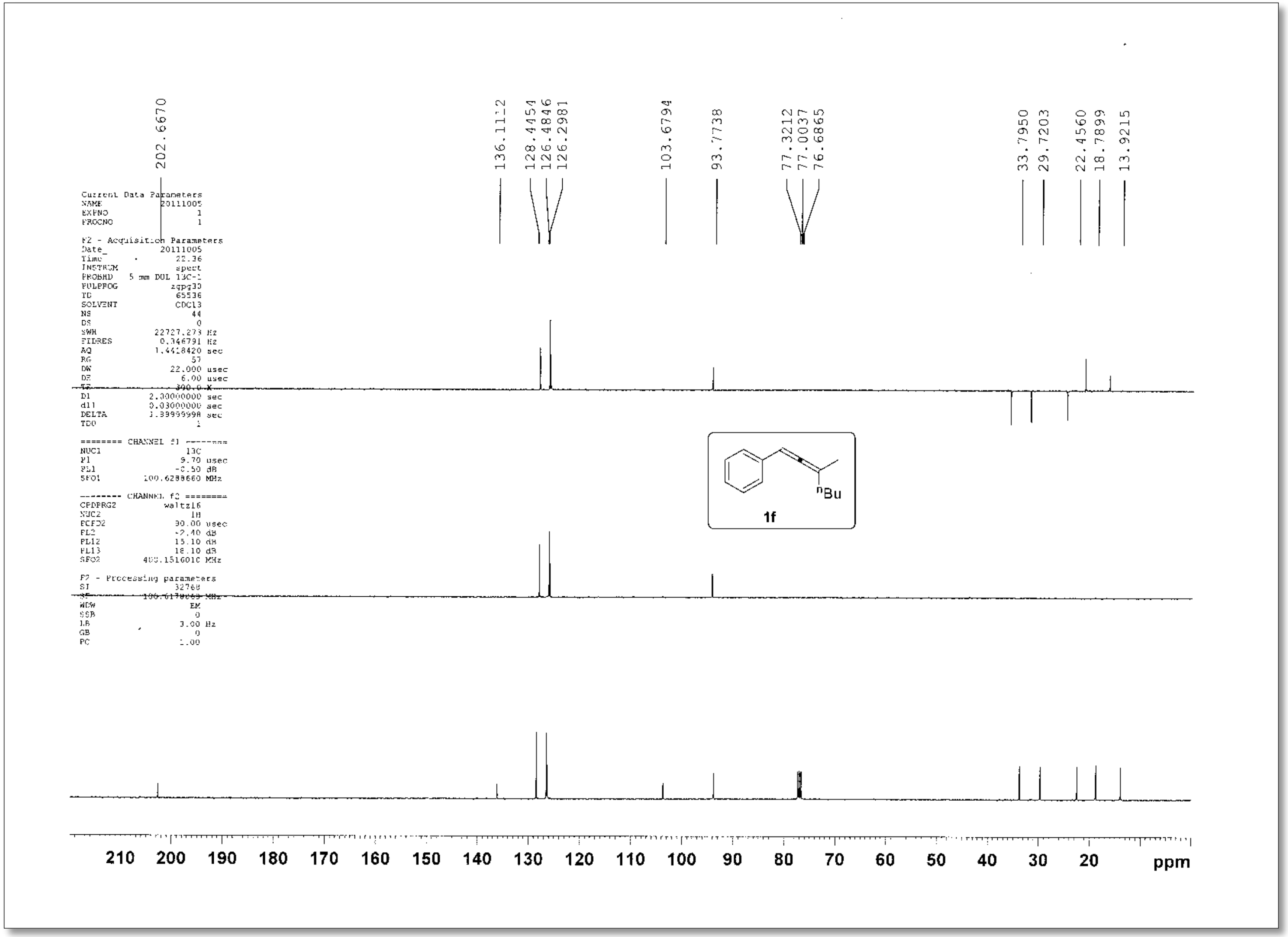
```
Current Data Parameters
NAME             20111001
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111003
Time                13.20
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     10
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    575
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



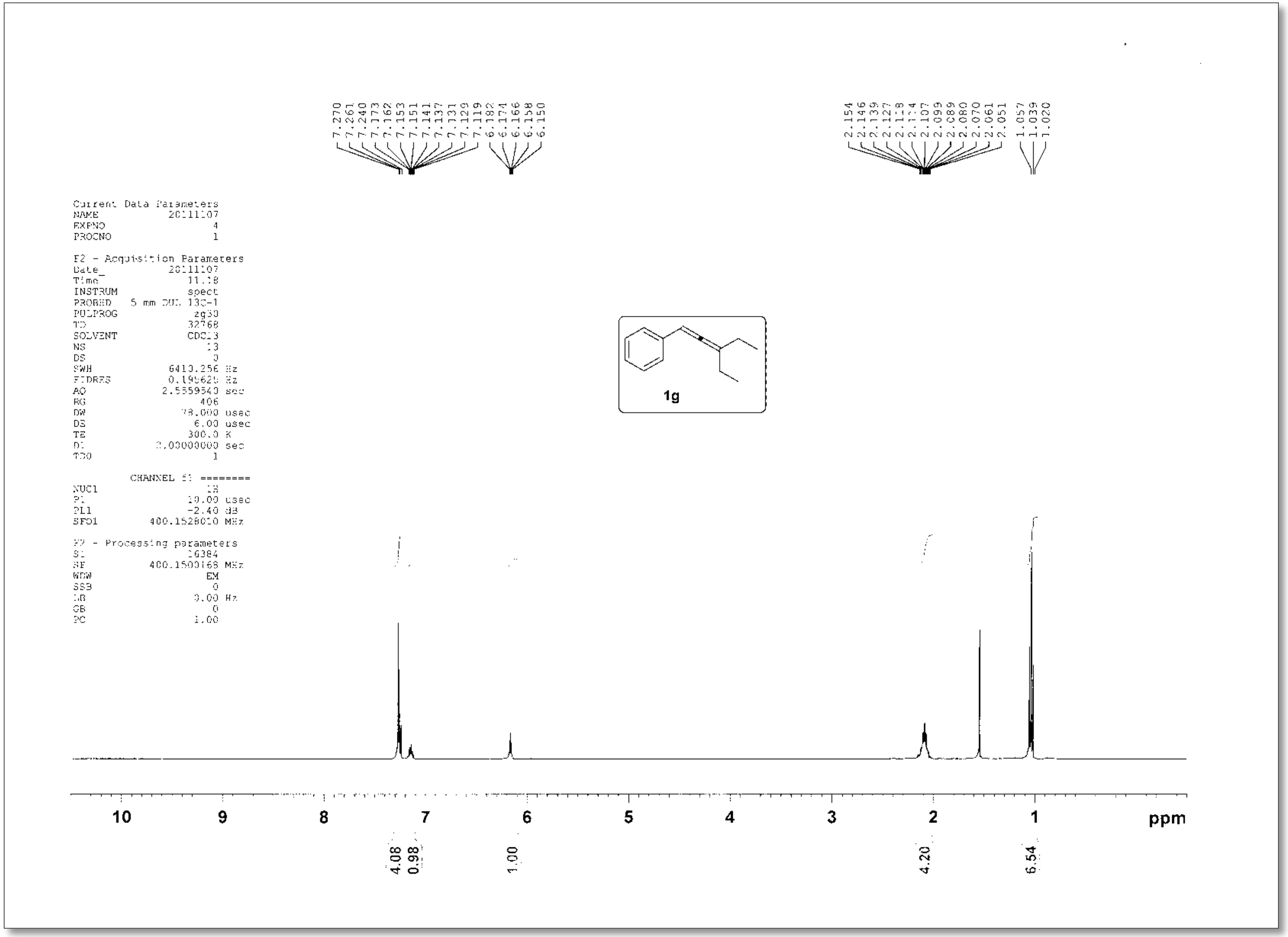
```
Current Data Parameters
NAME            20111001
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111003
Time               13.29
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    44
DS                     0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AC            1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1        100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               16.10 dB
SFO2        400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF          100.6178212 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```

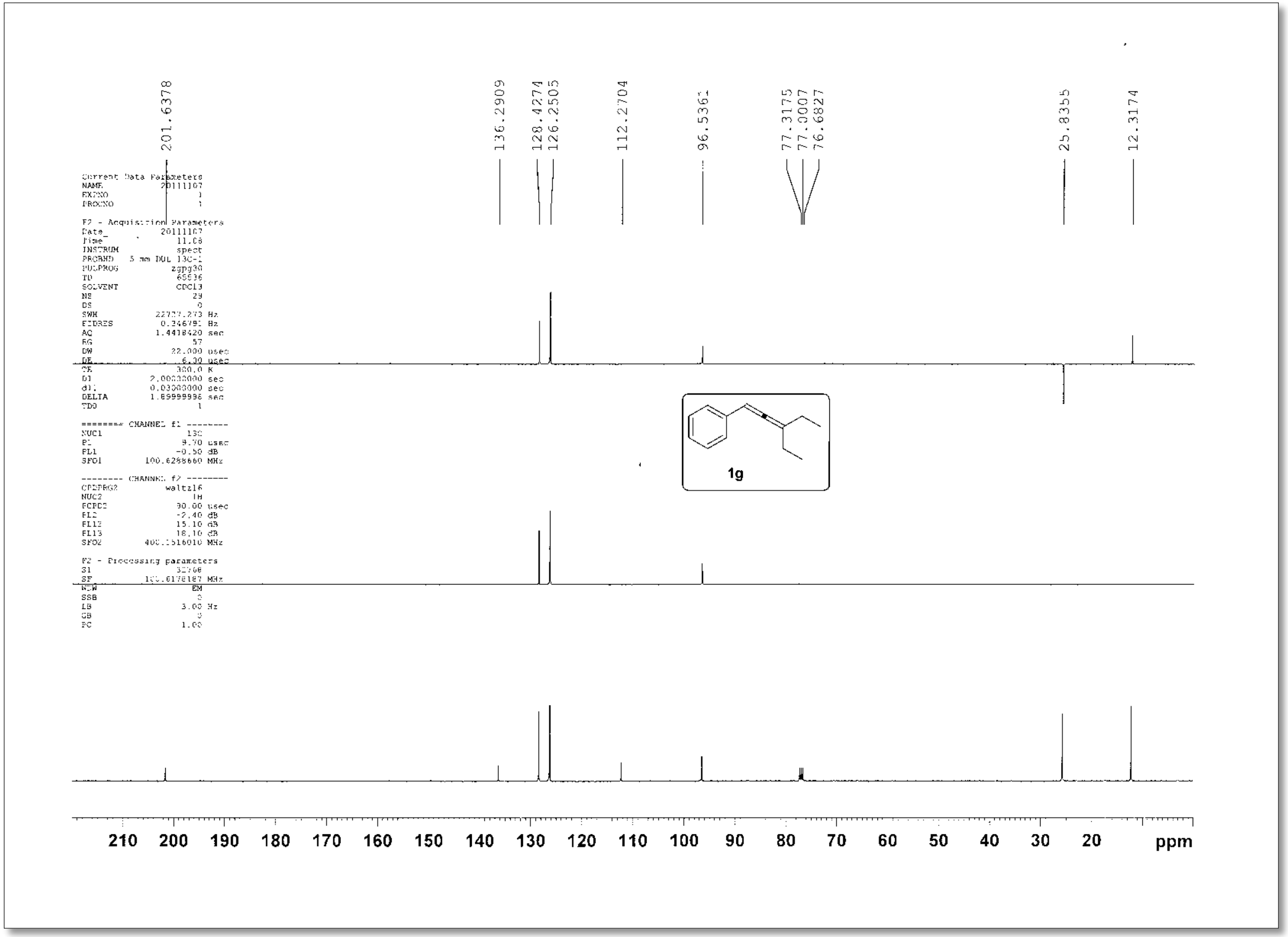


```
Current Data Parameters
NAME            20111005
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111005
Time               22.45
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   406
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500170 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

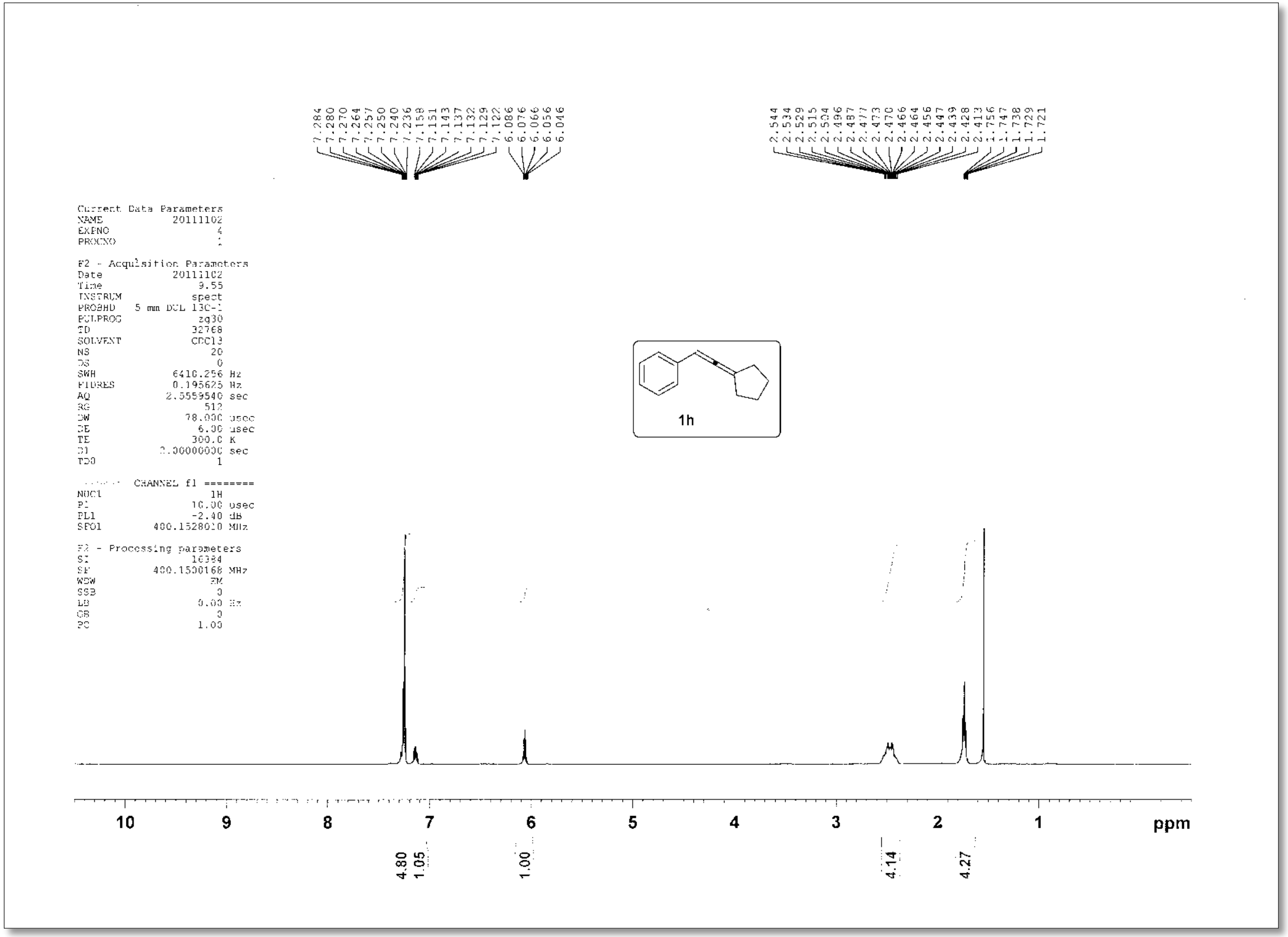


```
Current Data Parameters
NAME            20111005
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111005
Time               22.36
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    44
DS                     0
SWH           22727.273 Hz
FIDRES         0.346791 Hz
AQ            1.4418420 sec
RG                    57
DW               22.000 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178069 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```


7.270
7.261
7.240
7.173
7.162
7.153
7.151
7.141
7.137
7.131
7.129
7.119
6.182
6.174
6.166
6.158
6.150
2.154
2.146
2.139
2.127
2.118
2.114
2.107
2.099
2.089
2.080
2.070
2.061
2.051
1.057
1.039
1.020
Current Data Parameters
NAME 20111107
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111107
Time 11.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 13
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 406
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500166 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1g
10
9
8
7
6
5
4
3
2
1
ppm
4.08
0.98
1.00
4.20
6.54

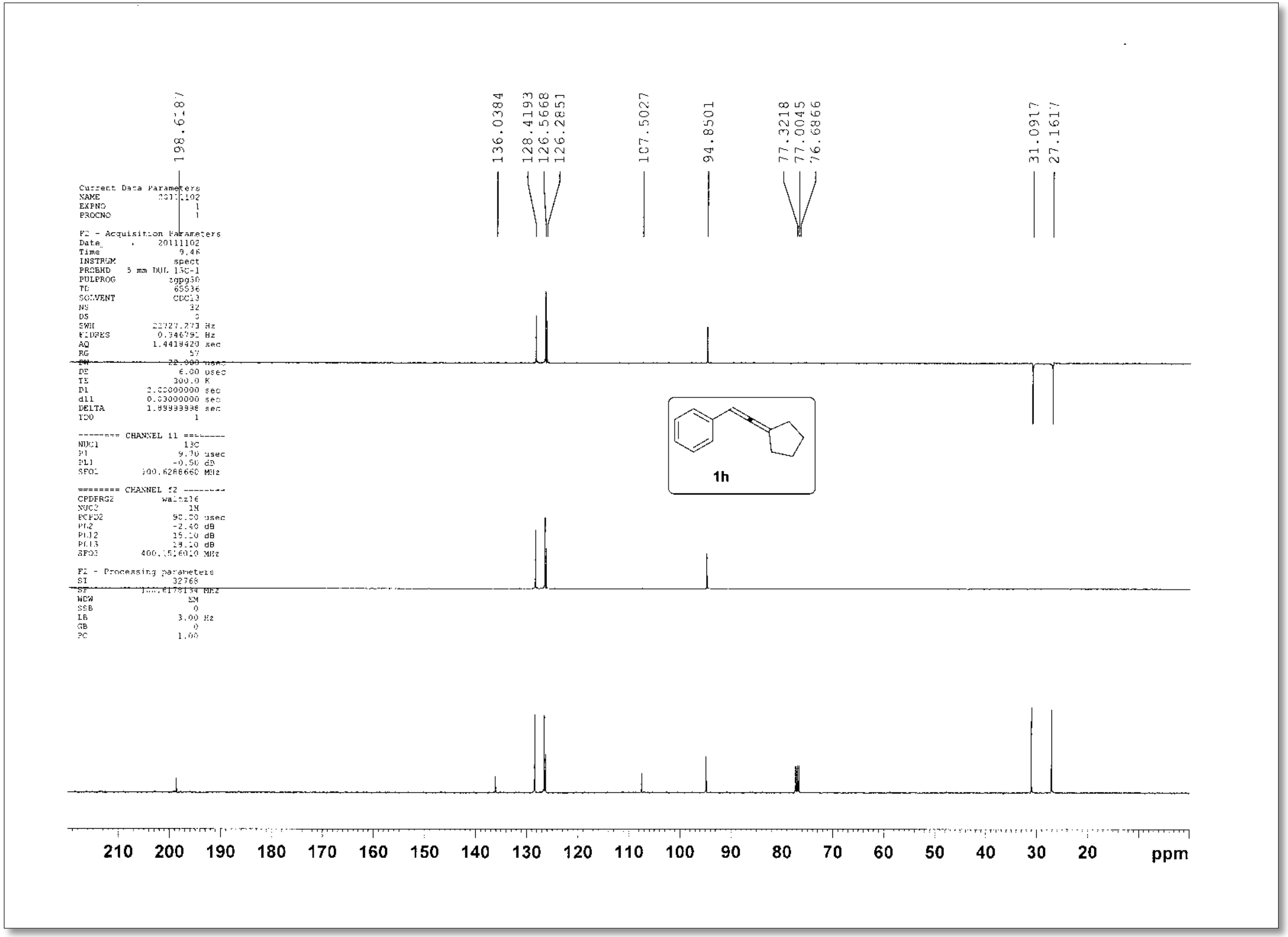


```
Current Data Parameters
NAME             20111107
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111107
Time                11.06
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    29
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 --------
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

-------- CHANNEL f2 --------
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178187 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



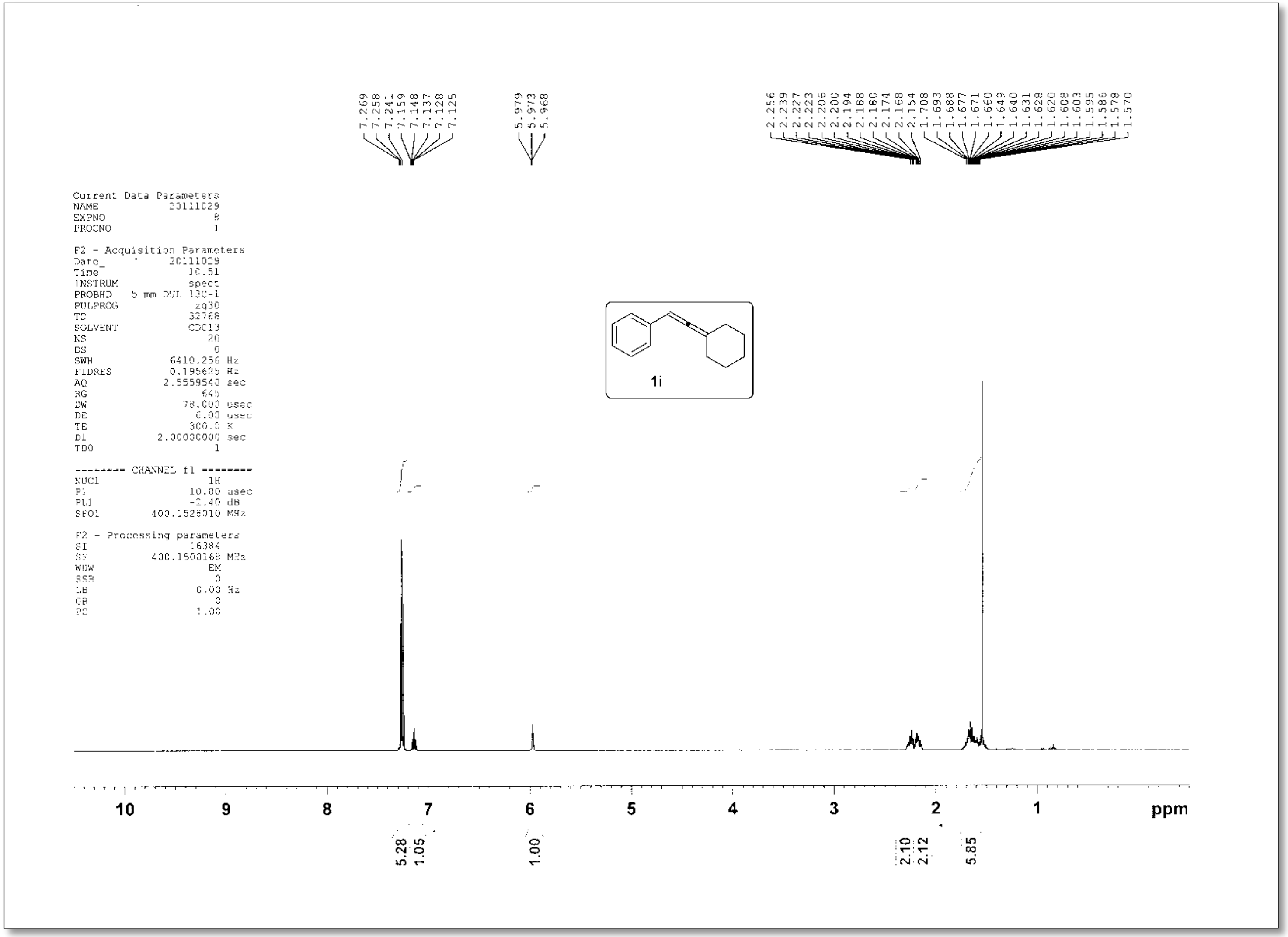
1h

```
Current Data Parameters
NAME              20111102
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date              20111102
Time                  9.55
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      20
DS                       0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ               2.5559540 sec
RG                     512
DW                  78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1           400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF             400.1500168 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

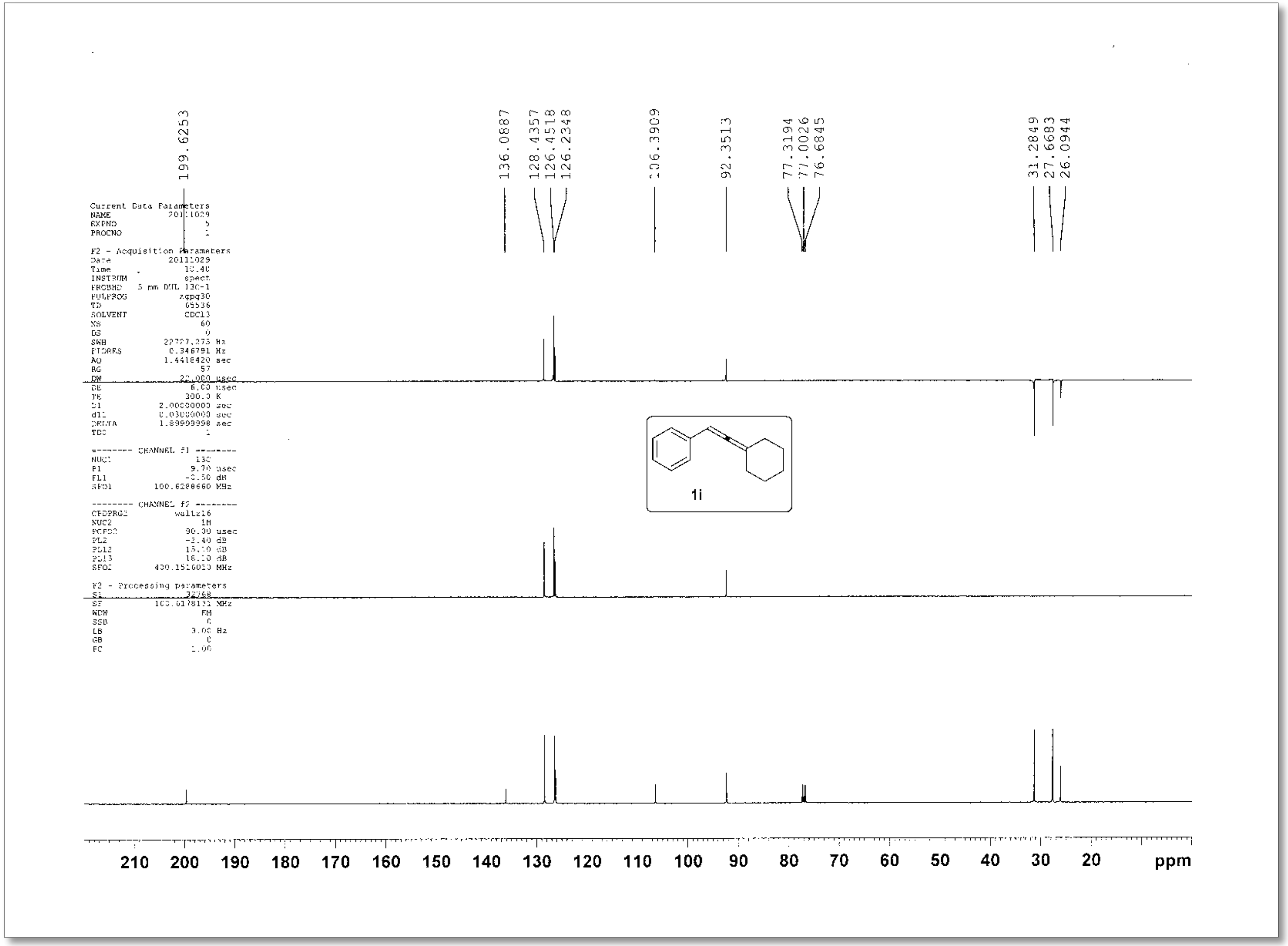


1h

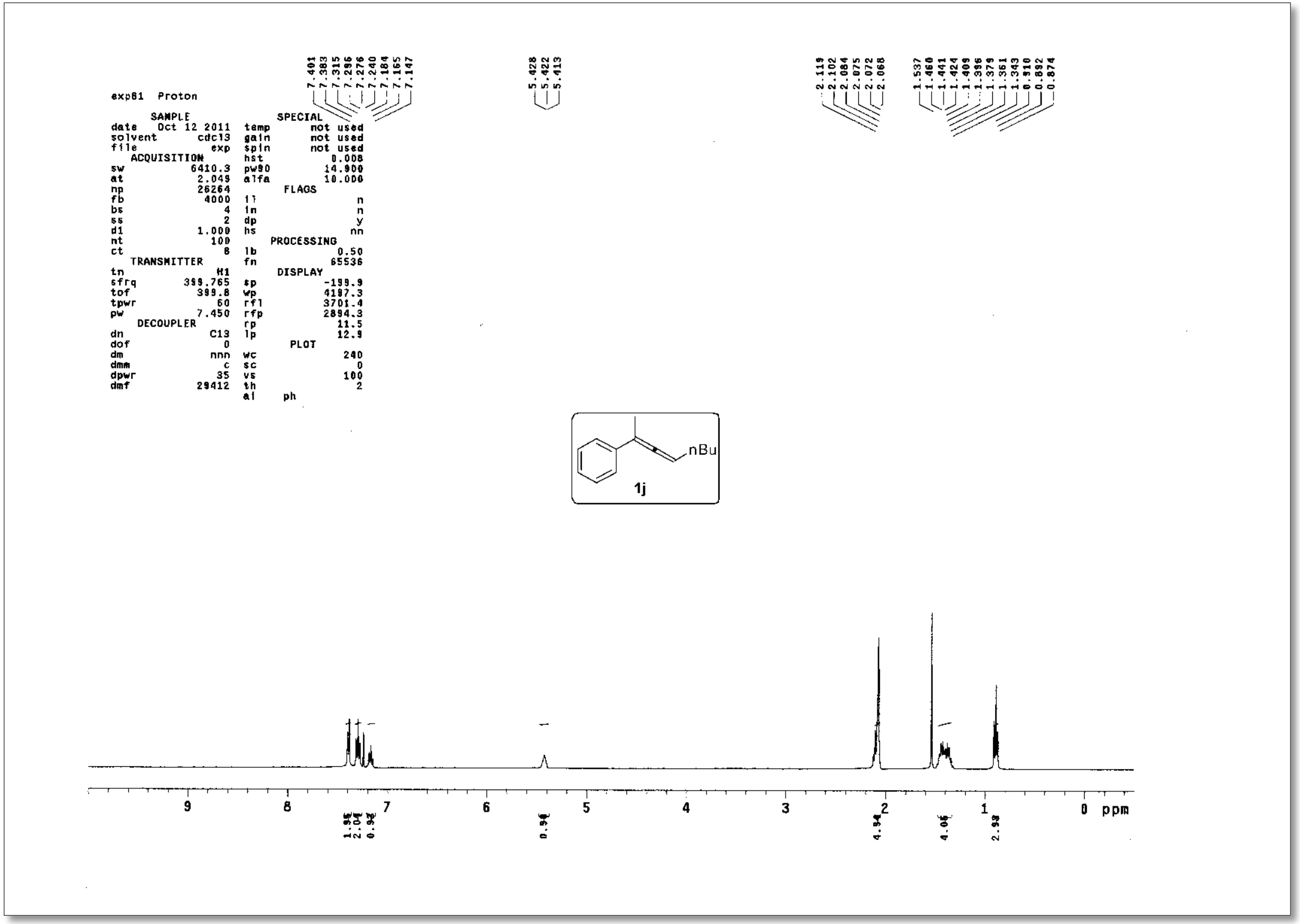
```
Current Data Parameters
NAME             20111102
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111102
Time                 9.46
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4419420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6179134 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME               20111029
EXPNO                     8
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111029
Time                  10.51
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       20
DS                        0
SWH                6410.256 Hz
FIDRES             0.195625 Hz
AQ                2.5559540 sec
RG                      645
DW                   78.000 usec
DE                     6.00 usec
TE                    300.0 K
D1               2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.00 usec
PL1                   -2.40 dB
SFO1            400.1528010 MHz

F2 - Processing parameters
SI                    16384
SF              400.1500168 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

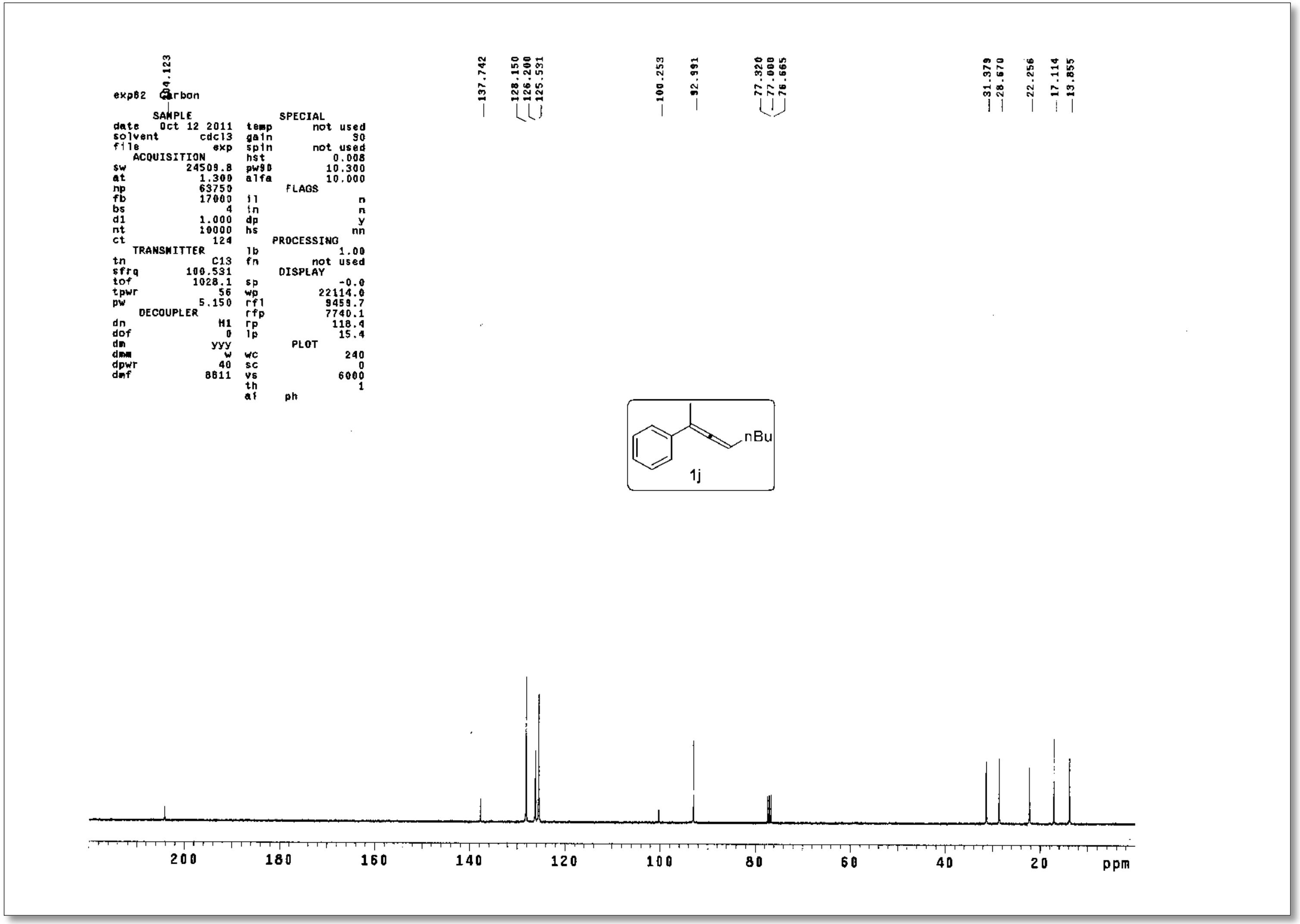


```
Current Data Parameters
NAME             20111029
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111029
Time                10.40
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     60
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -1.40 dB
PL12                15.10 dB
PL13                16.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178131 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```

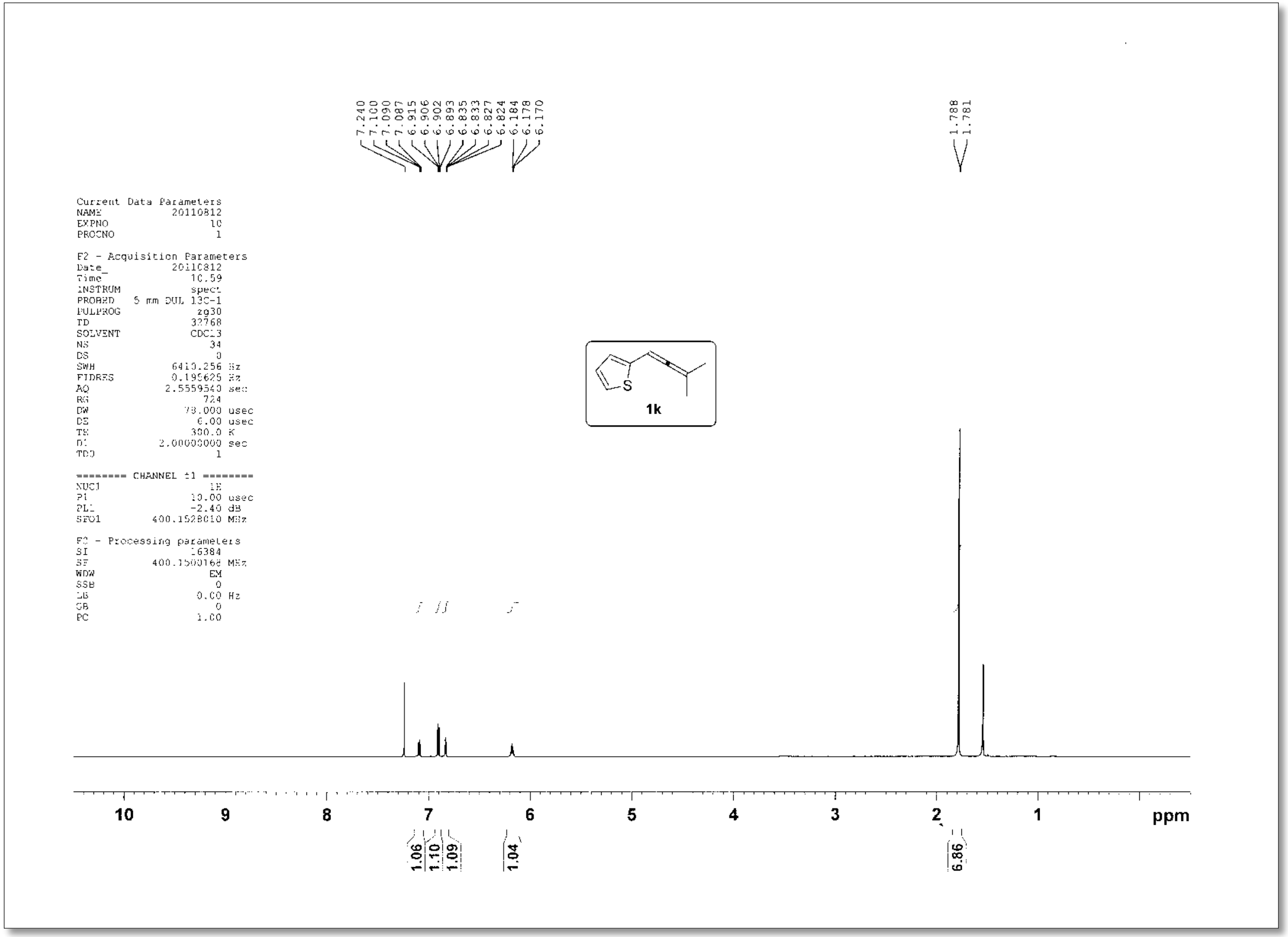


```
exp81  Proton

      SAMPLE                   SPECIAL
date   Oct 12 2011   temp        not used
solvent      cdcl3   gain        not used
file           exp   spin        not used
   ACQUISITION       hst            0.008
sw          6410.3   pw90          14.900
at           2.049   alfa          10.000
np           26264            FLAGS
fb            4000   il                 n
bs               4   in                 n
ss               2   dp                 y
d1           1.000   hs                nn
nt             100          PROCESSING
ct               8   lb              0.50
   TRANSMITTER       fn             65536
tn              H1            DISPLAY
sfrq       399.765   sp            -199.9
tof          399.8   wp            4197.3
tpwr            60   rfl           3701.4
pw           7.450   rfp           2894.3
    DECOUPLER        rp              11.5
dn             C13   lp              12.9
dof              0             PLOT
dm             nnn   wc               240
dmm              c   sc                 0
dpwr            35   vs               100
dmf          29412   th                 2
                     ai     ph
```

1j

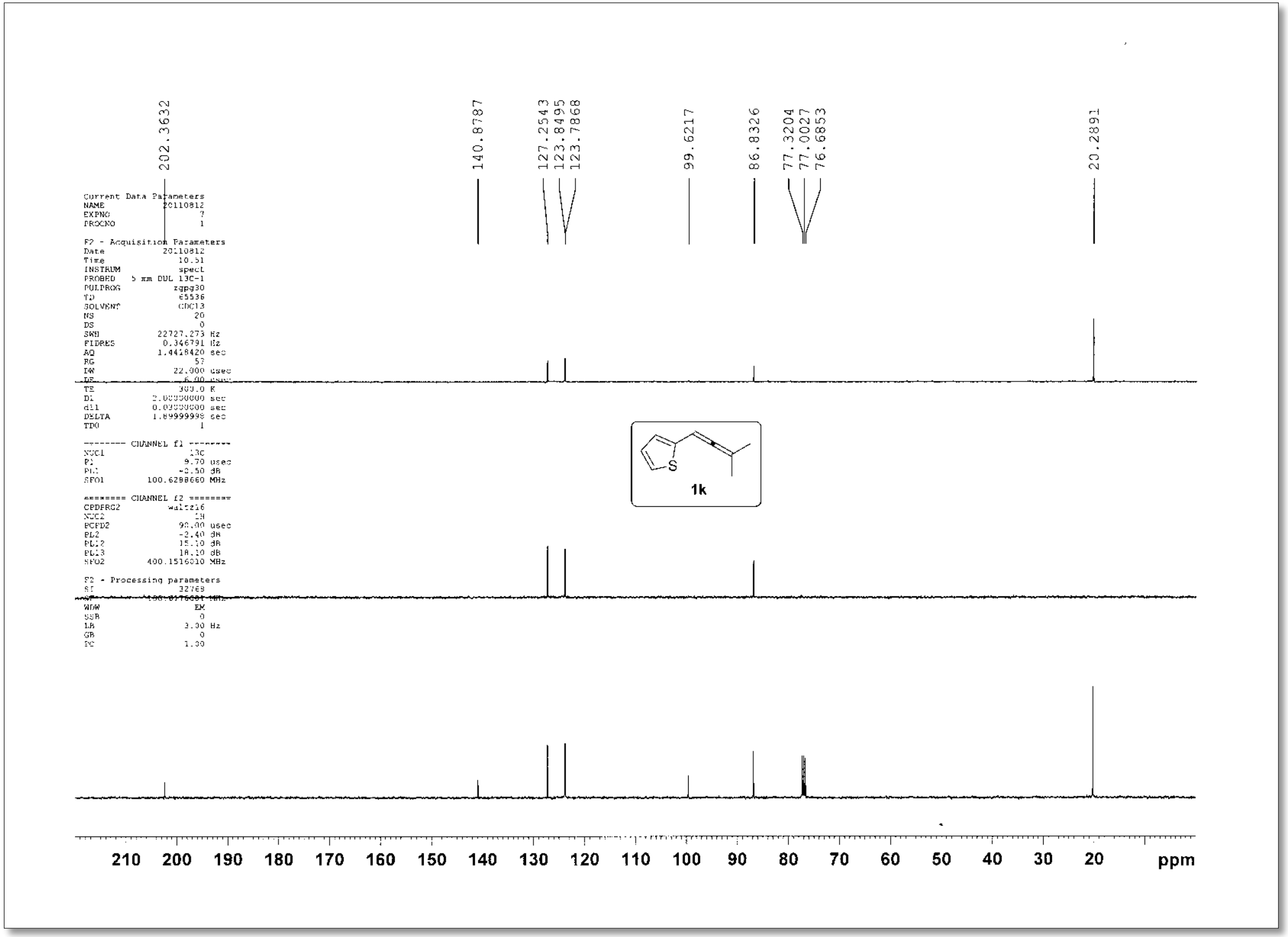


```
exp82  Carbon

      SAMPLE                  SPECIAL
date    Oct 12 2011   temp        not used
solvent       cdcl3   gain              90
file            exp   spin        not used
   ACQUISITION        hst            0.008
sw          24509.8   pw90          10.300
at            1.300   alfa          10.000
np            63750          FLAGS
fb            17000   il                 n
bs                4   in                 n
d1            1.000   dp                 y
nt            10000   hs                nn
ct              124         PROCESSING
   TRANSMITTER        lb              1.00
tn              C13   fn          not used
sfrq        100.531          DISPLAY
tof          1028.1   sp              -0.0
tpwr             56   wp           22114.0
pw            5.150   rfl           9459.7
    DECOUPLER         rfp           7740.1
dn               H1   rp             118.4
dof               0   lp              15.4
dm              yyy            PLOT
dmm               w   wc               240
dpwr             40   sc                 0
dmf            8811   vs              6000
                      th                 1
                      ai     ph
```

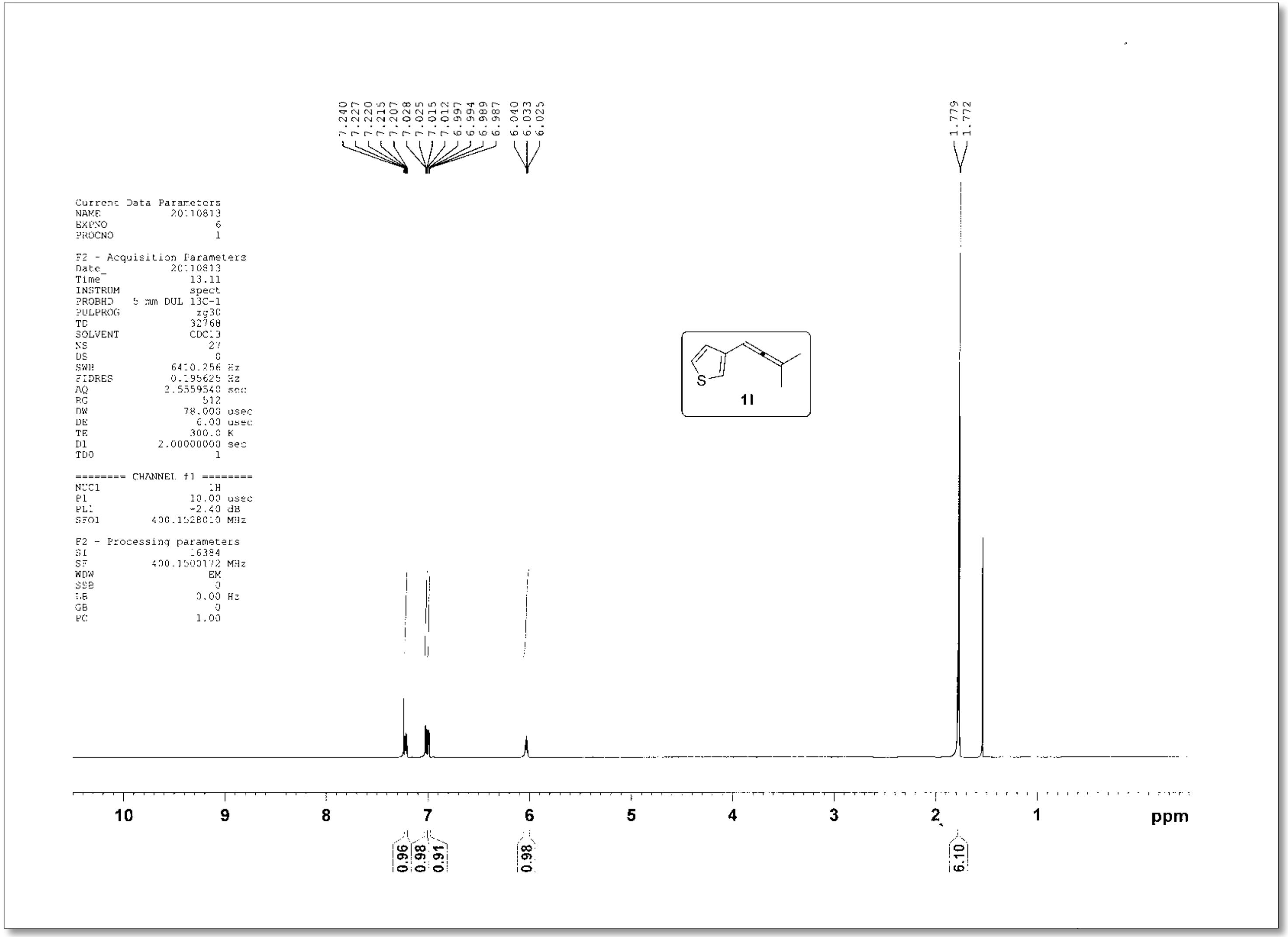


```
Current Data Parameters
NAME             20110812
EXPNO                  10
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110812
Time                10.59
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     34
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    724
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

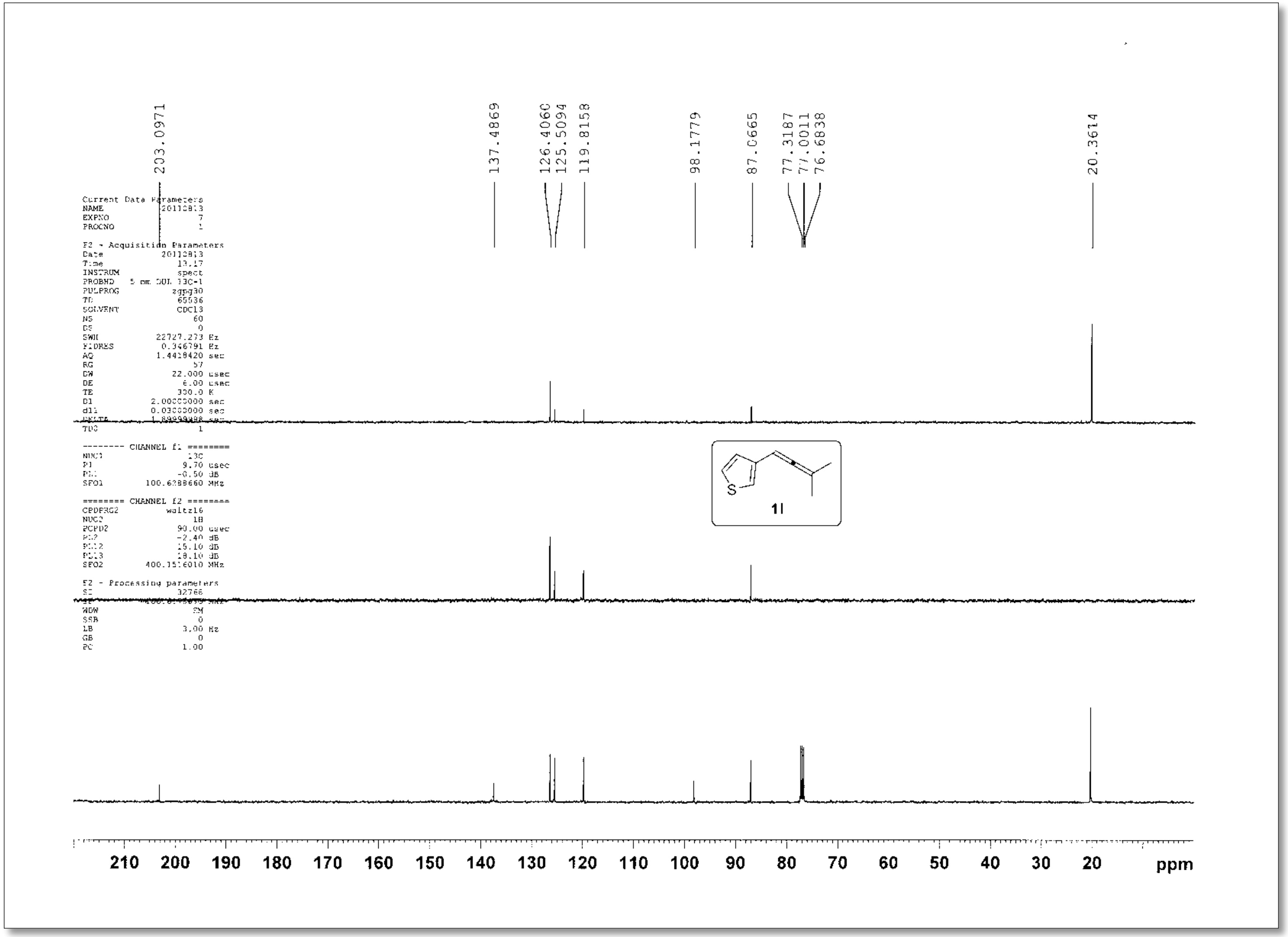
1k



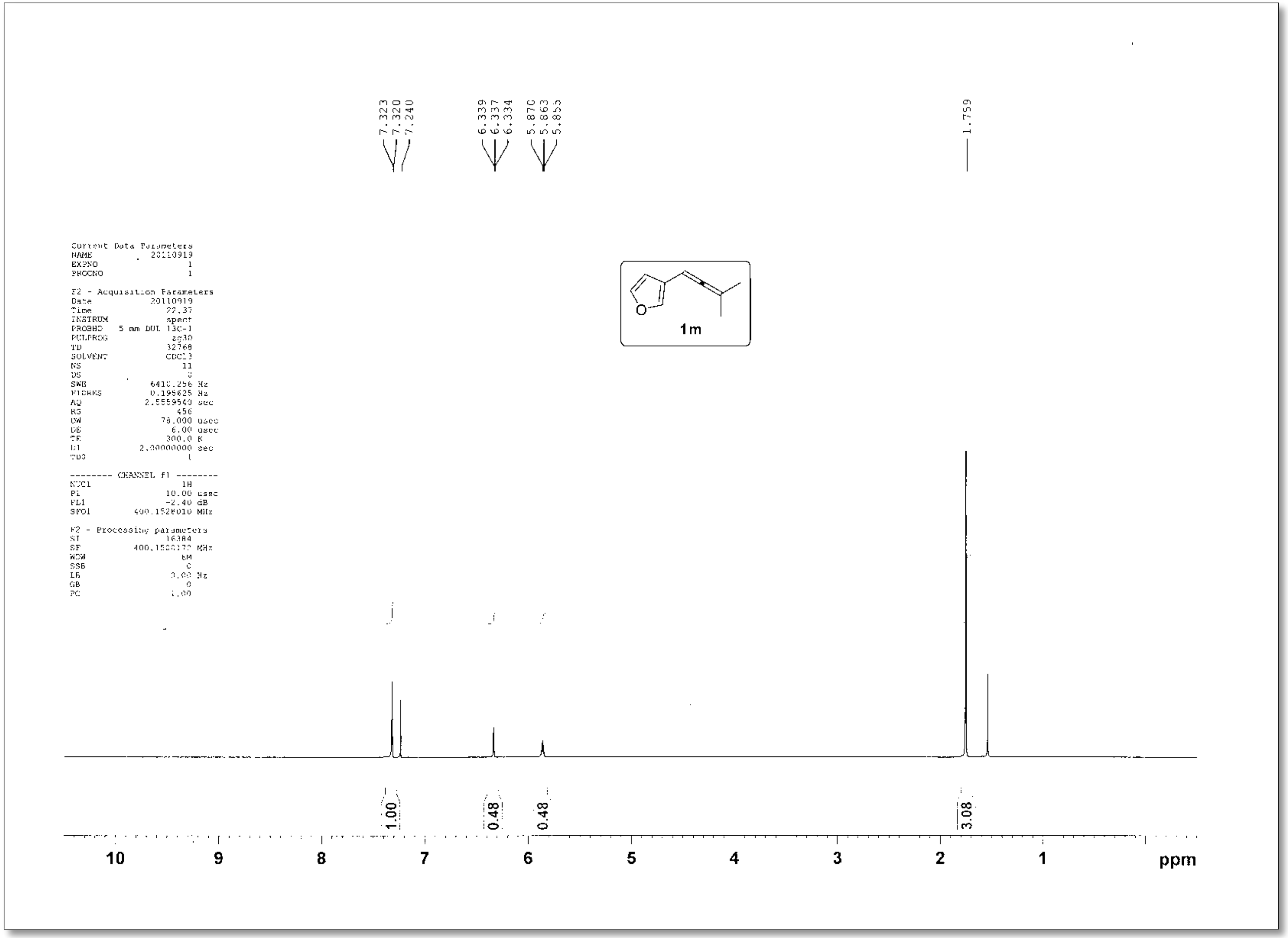
```
Current Data Parameters
NAME            20110812
EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
Date            20110812
Time               10.51
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    20
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -2.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6176691 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



```
Current Data Parameters
NAME             20110813
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110813
Time                13.11
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     27
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    512
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF           400.1500172 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

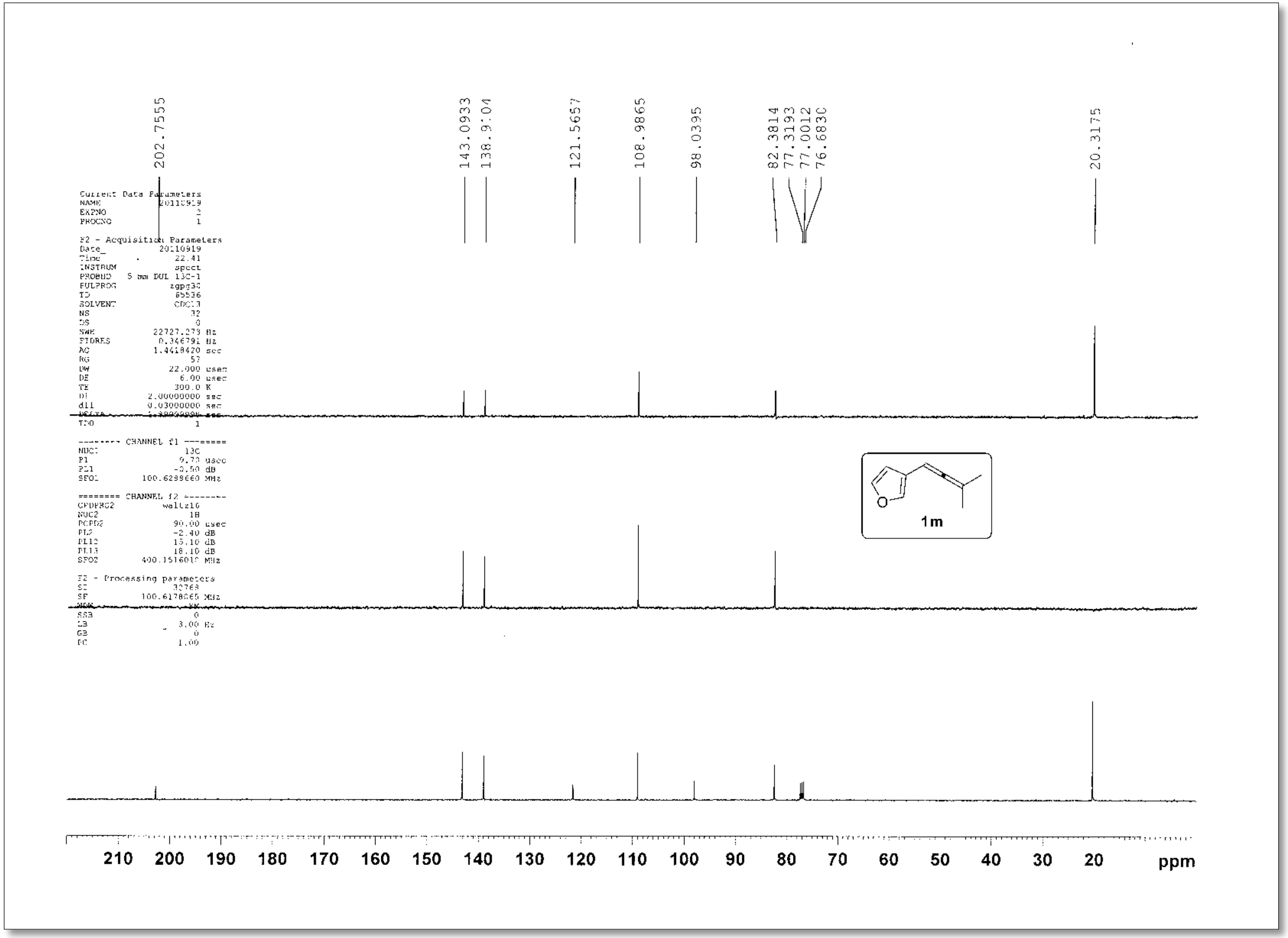


```
Current Data Parameters
NAME             20110813
EXPNO                   7
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110813
Time                13.17
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     60
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            [illegible] MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```

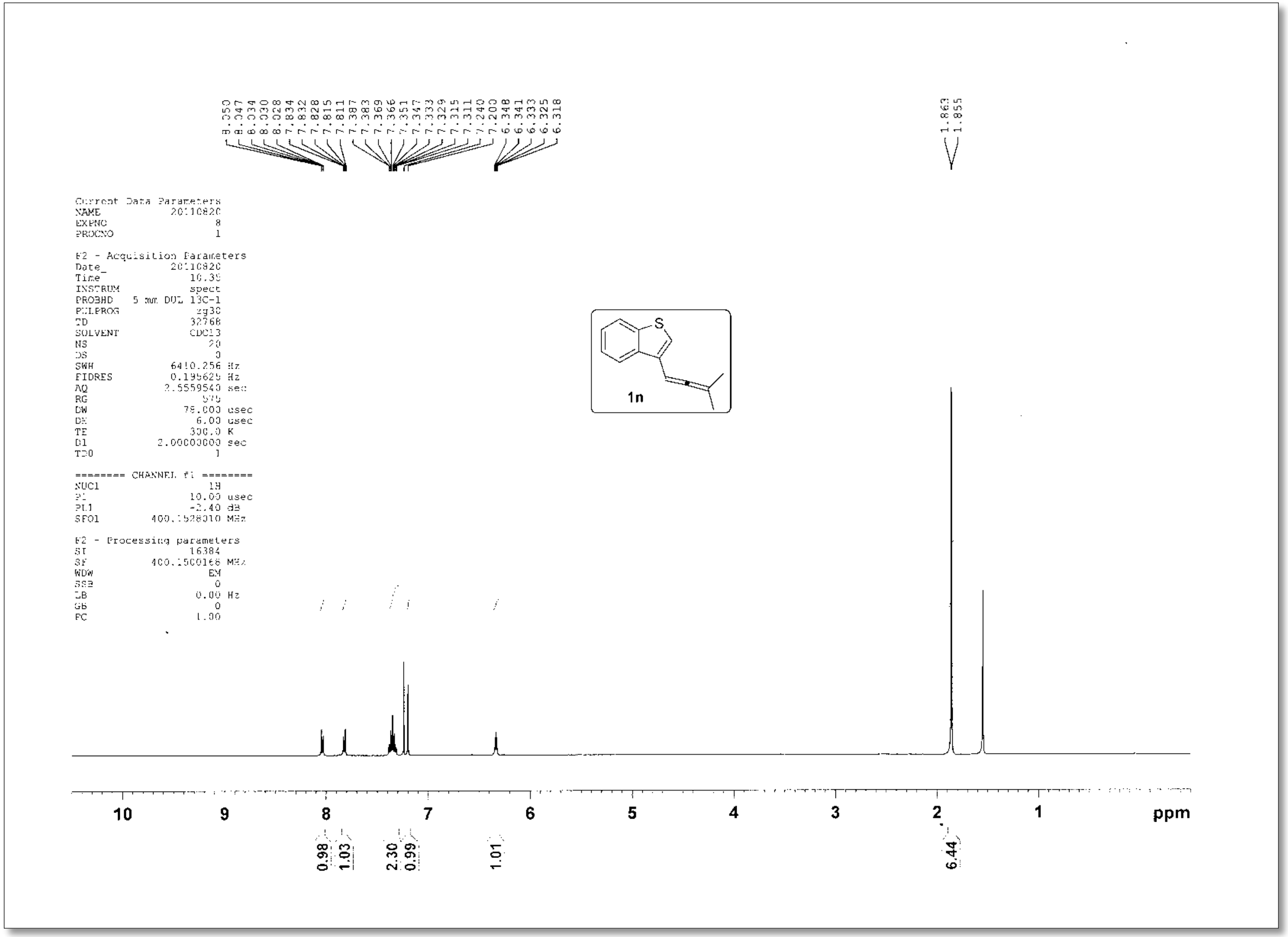


1m

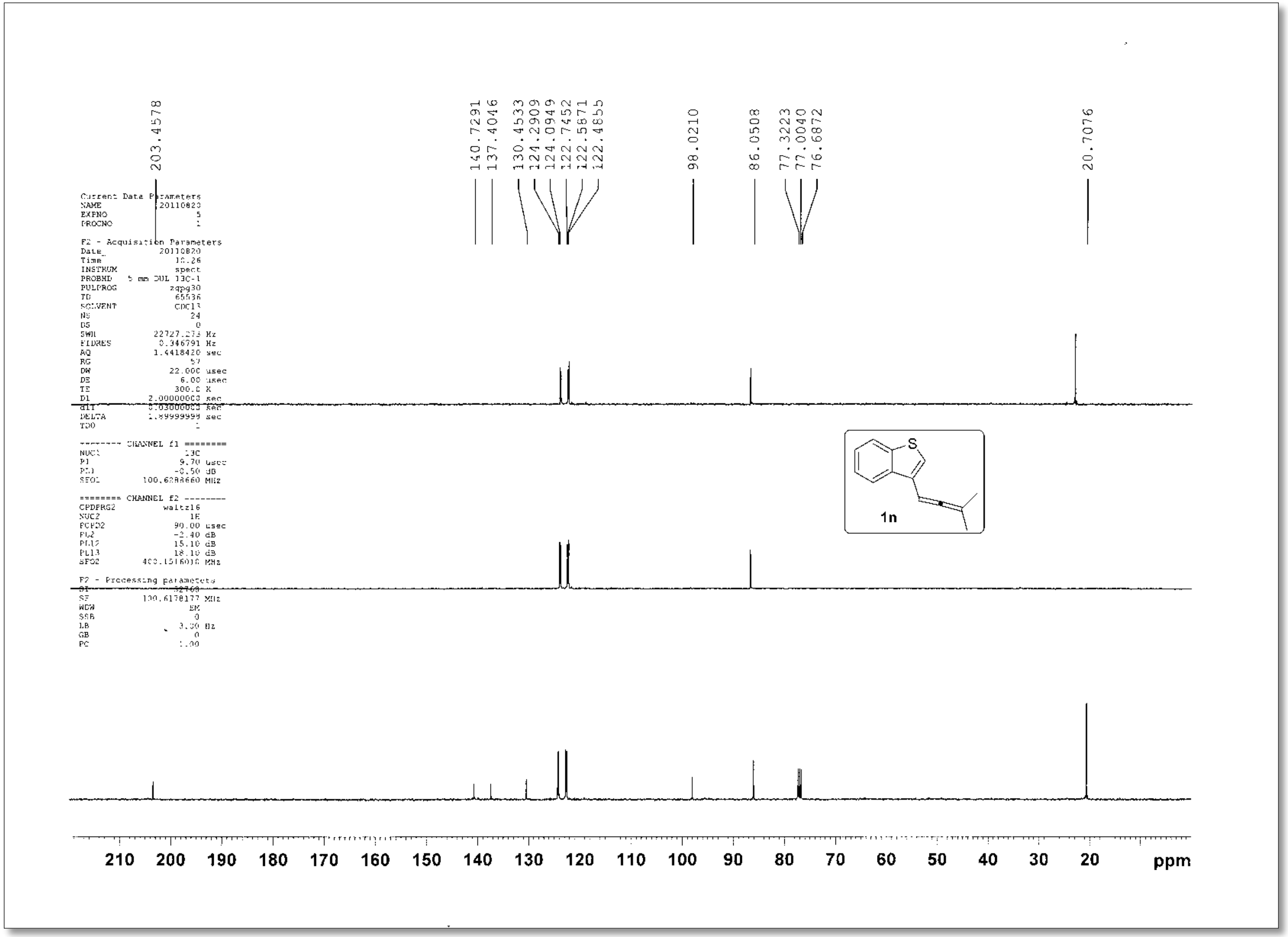
```
Current Data Parameters
NAME              20110919
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date              20110919
Time                 22.37
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      11
DS                       0
SWH            6410.256 Hz
FIDRES         0.195625 Hz
AQ            2.5559540 sec
RG                     456
DW              78.000 usec
DE                6.00 usec
TE                 300.0 K
D1          2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1               10.00 usec
PL1               -2.40 dB
SFO1        400.1526010 MHz

F2 - Processing parameters
SI                   16384
SF          400.1500170 MHz
WDW                     EM
SSB                      0
LB                 0.00 Hz
GB                       0
PC                    1.00
```



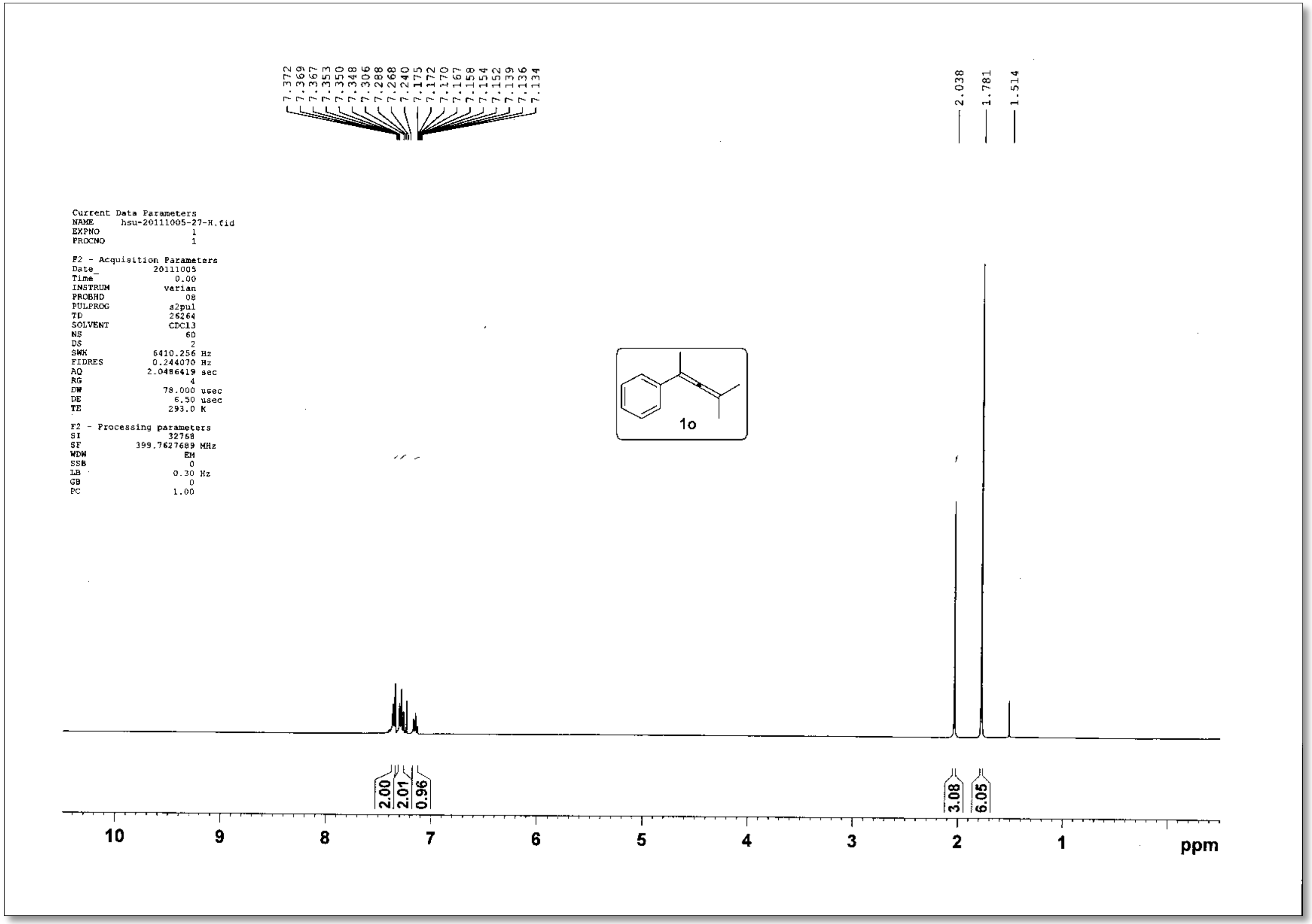
```
Current Data Parameters
NAME             20110919
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110919
Time                22.41
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          [illegible] sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6289660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178065 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



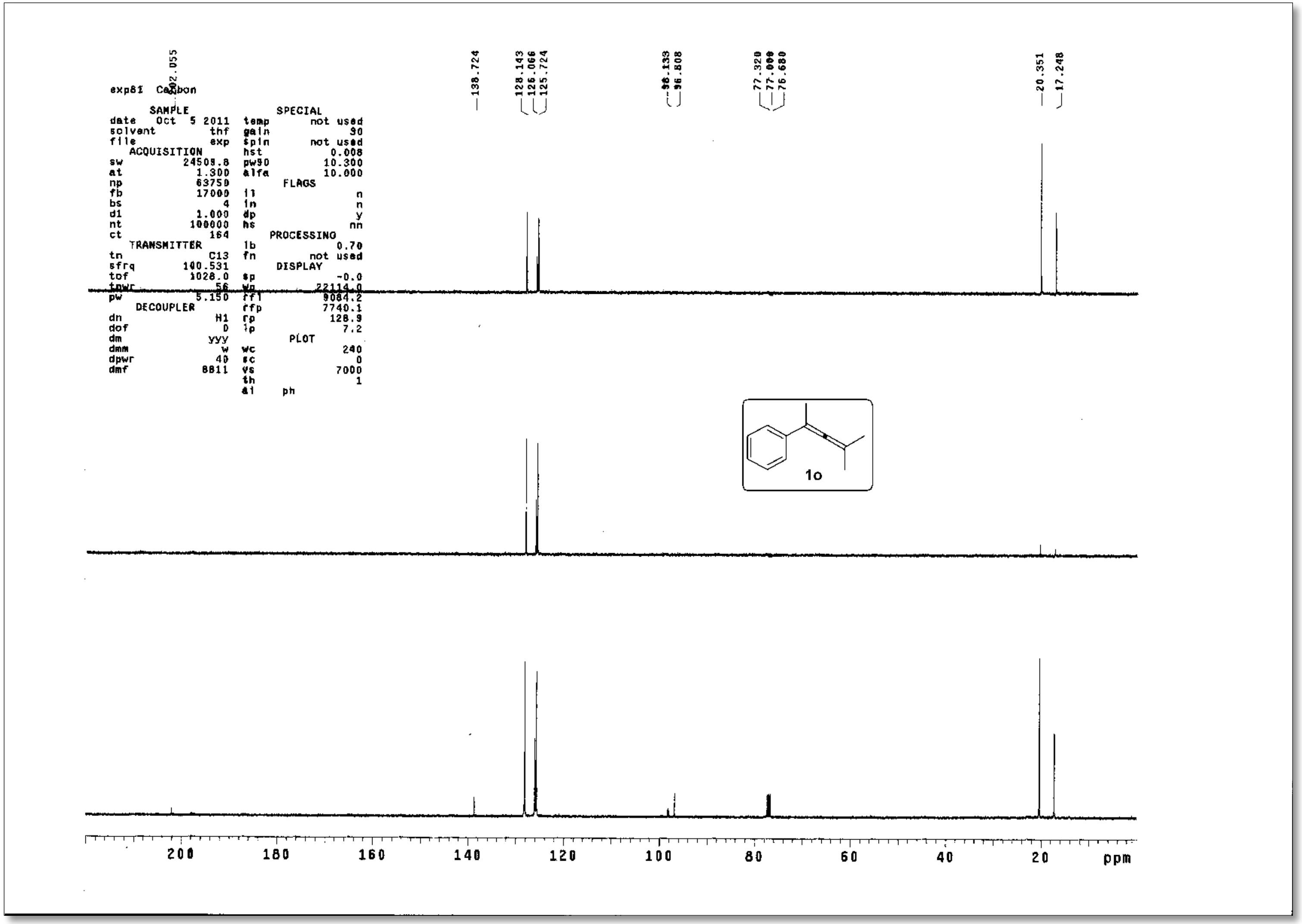
```
Current Data Parameters
NAME             20110820
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110820
Time                10.39
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     20
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    575
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500166 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



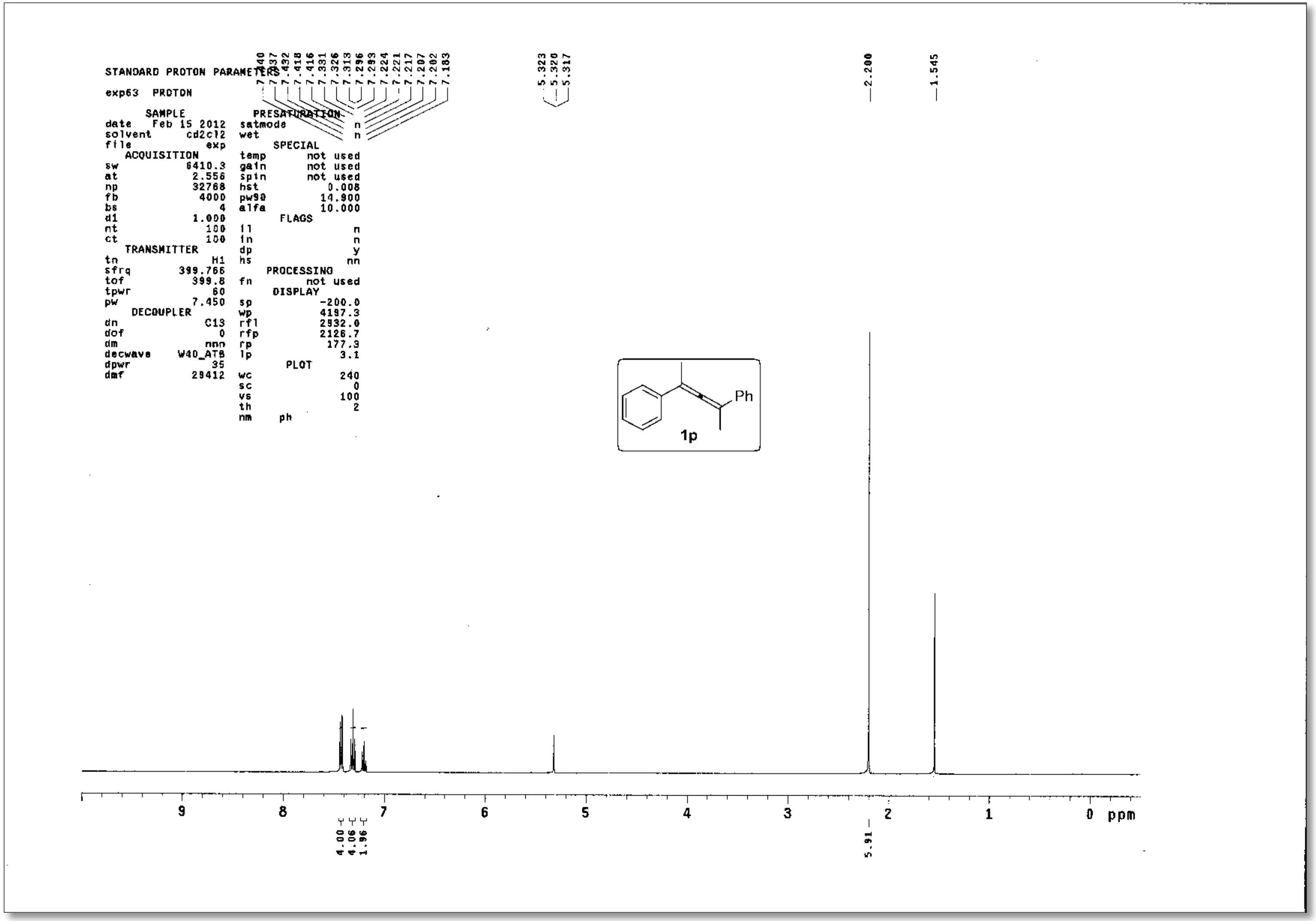
```
Current Data Parameters
NAME          20110820
EXPNO                5
PROCNO               1

F2 - Acquisition Parameters
Date_         20110820
Time             10.26
INSTRUM          spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  24
DS                   0
SWH          22727.273 Hz
FIDRES        0.346791 Hz
AQ           1.4418420 sec
RG                  57
DW              22.000 usec
DE                6.00 usec
TE               300.0 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.70 usec
PL1              -0.50 dB
SFO1       100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -2.40 dB
PL12             15.10 dB
PL13             18.10 dB
SFO2       400.1516010 MHz

F2 - Processing parameters
SI               32768
SF         100.6178177 MHz
WDW                 EM
SSB                  0
LB                3.00 Hz
GB                   0
PC                1.00
```



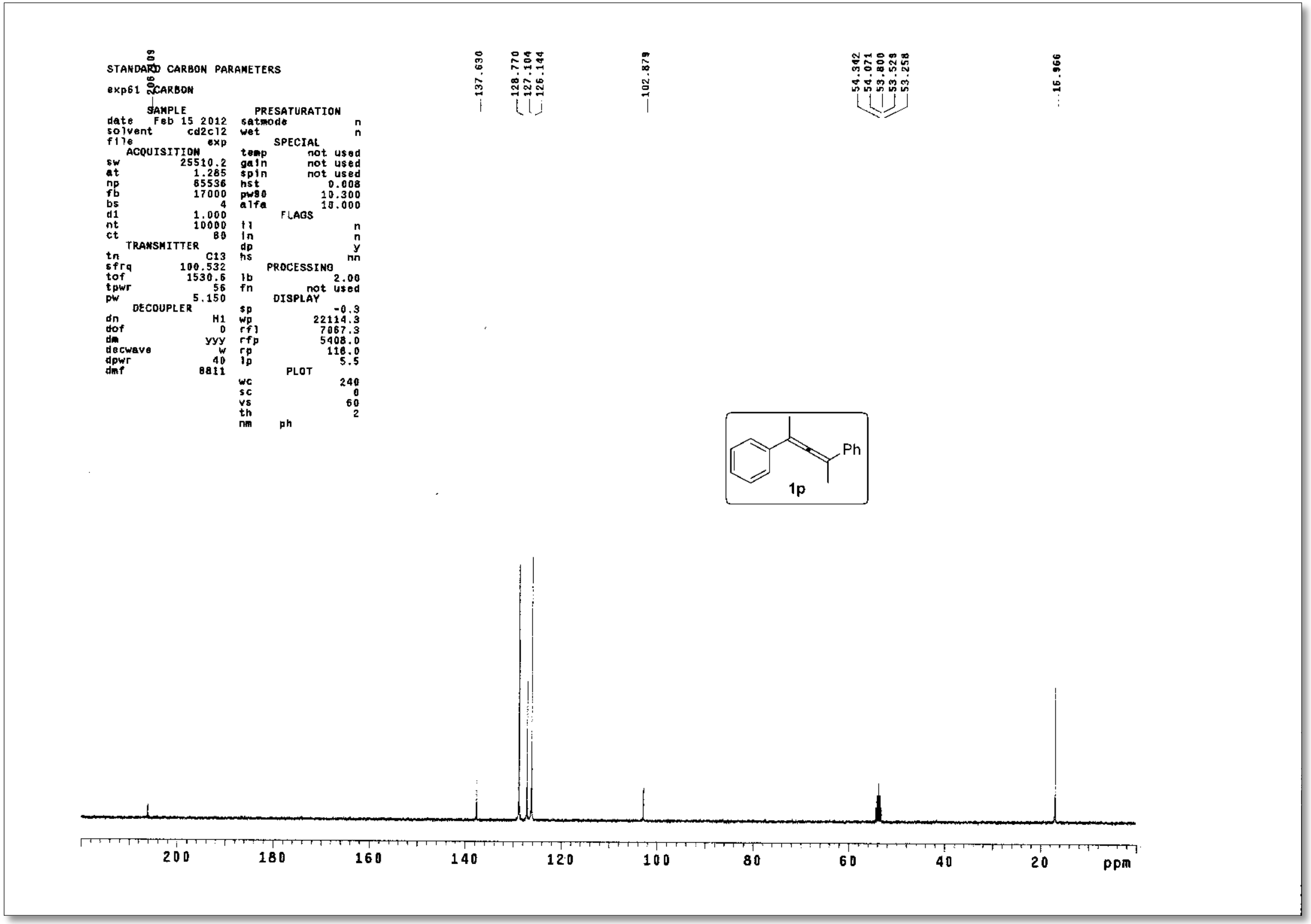
```
Current Data Parameters
NAME      hsu-20111005-27-H.fid
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20111005
Time               0.00
INSTRUM          varian
PROBHD               08
PULPROG           s2pul
TD                26264
SOLVENT           CDCl3
NS                   60
DS                    2
SWH            6410.256 Hz
FIDRES         0.244070 Hz
AQ            2.0486419 sec
RG                    4
DW               78.000 usec
DE                 6.50 usec
TE                293.0 K

F2 - Processing parameters
SI                32768
SF          399.7627689 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



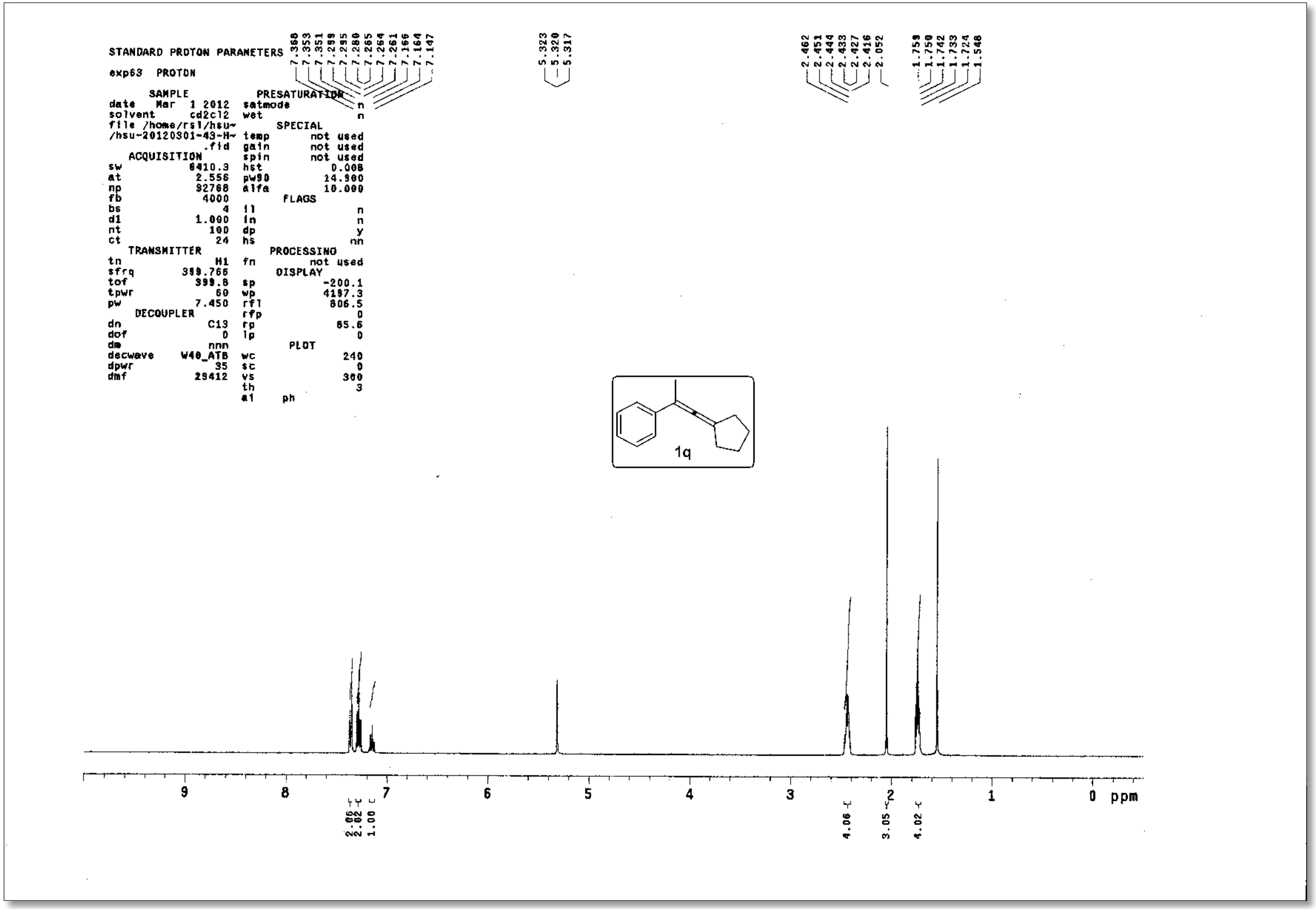
```
exp81  Carbon

      SAMPLE                  SPECIAL
date    Oct  5 2011   temp        not used
solvent          thf   gain              90
file             exp   spin        not used
   ACQUISITION         hst            0.008
sw           24509.8   pw90          10.300
at             1.300   alfa          10.000
np             63750           FLAGS
fb             17000   il                 n
bs                 4   in                 n
d1             1.000   dp                 y
nt            100000   hs                nn
ct               164        PROCESSING
   TRANSMITTER         lb              0.70
tn               C13   fn          not used
sfrq         100.531          DISPLAY
tof           1028.0   sp              -0.0
tpwr              56   wp           22114.0
pw             5.150   rfl           9084.2
    DECOUPLER          rfp           7740.1
dn                H1   rp             128.9
dof                0   lp               7.2
dm               yyy            PLOT
dmm                w   wc               240
dpwr              40   sc                 0
dmf             8811   vs              7000
                       th                 1
                       ai     ph
```



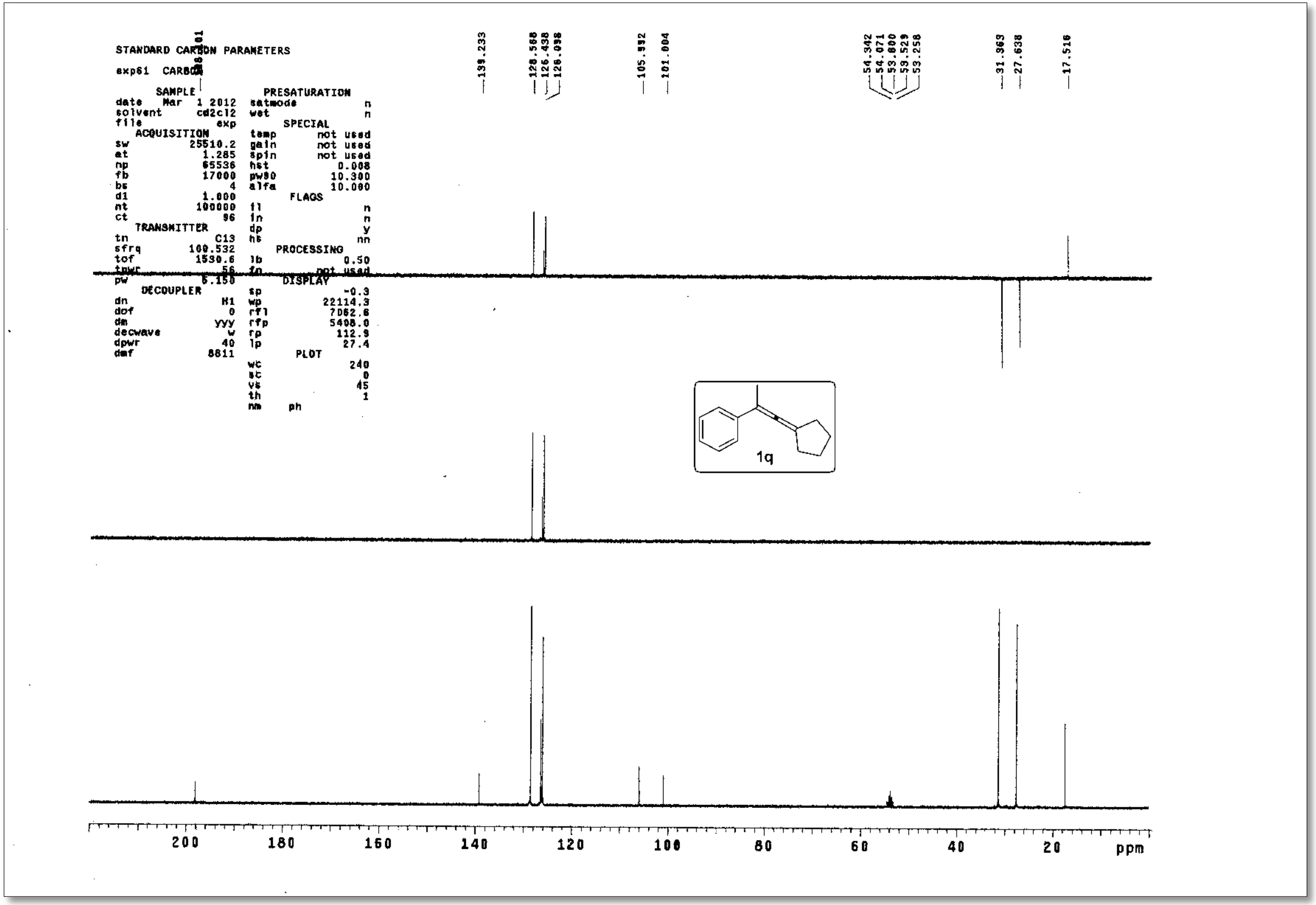
```
STANDARD PROTON PARAMETERS

exp63  PROTON

      SAMPLE               PRESATURATION
date   Feb 15 2012    satmode              n
solvent     cd2cl2    wet                  n
file           exp           SPECIAL
   ACQUISITION        temp          not used
sw          6410.3    gain          not used
at           2.556    spin          not used
np           32768    hst              0.008
fb            4000    pw90            14.900
bs               4    alfa            10.000
d1           1.000           FLAGS
nt             100    il                   n
ct             100    in                   n
   TRANSMITTER        dp                   y
tn              H1    hs                  nn
sfrq       399.766          PROCESSING
tof          399.8    fn            not used
tpwr            60           DISPLAY
pw           7.450    sp              -200.0
    DECOUPLER         wp              4197.3
dn             C13    rfl             2932.0
dof              0    rfp             2126.7
dm             nnn    rp               177.3
decwave    W40_ATB    lp                 3.1
dpwr            35             PLOT
dmf          29412    wc                 240
                      sc                   0
                      vs                 100
                      th                   2
                      nm     ph
```



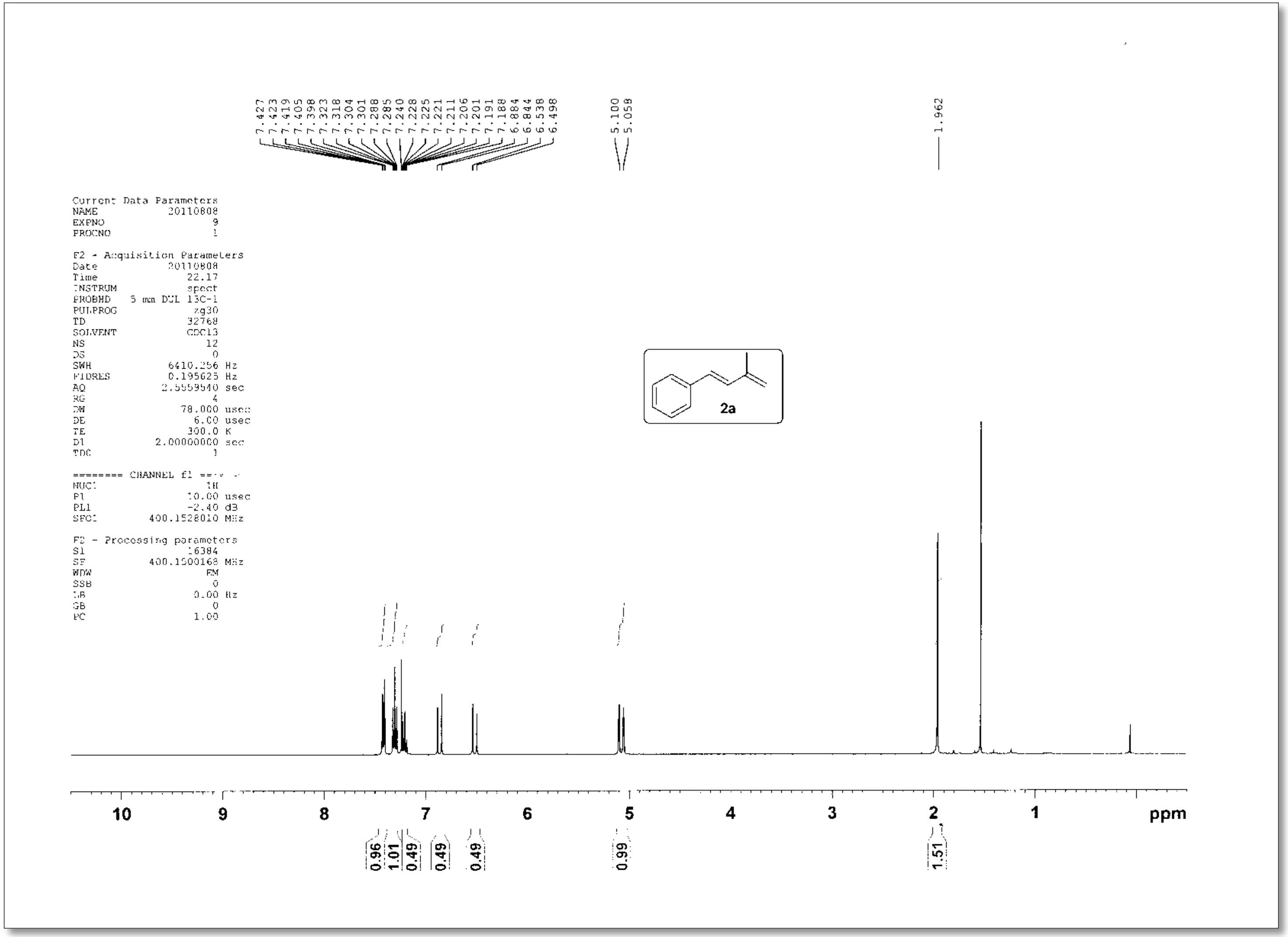
```
STANDARD CARBON PARAMETERS

exp61  CARBON

      SAMPLE               PRESATURATION
date   Feb 15 2012   satmode              n
solvent     cd2cl2   wet                  n
file           exp          SPECIAL
   ACQUISITION       temp          not used
sw         25510.2   gain          not used
at           1.285   spin          not used
np           65536   hst              0.008
fb           17000   pw90            10.300
bs               4   alfa            10.000
d1           1.000          FLAGS
nt           10000   il                   n
ct              80   in                   n
   TRANSMITTER       dp                   y
tn             C13   hs                  nn
sfrq       100.532        PROCESSING
tof         1530.6   lb                2.00
tpwr            56   fn            not used
pw           5.150         DISPLAY
    DECOUPLER        sp                -0.3
dn              H1   wp             22114.3
dof              0   rfl             7067.3
dm             yyy   rfp             5408.0
decwave          w   rp               118.0
dpwr            40   lp                 5.5
dmf           8811          PLOT
                     wc                 240
                     sc                   0
                     vs                  60
                     th                   2
                     nm      ph
```



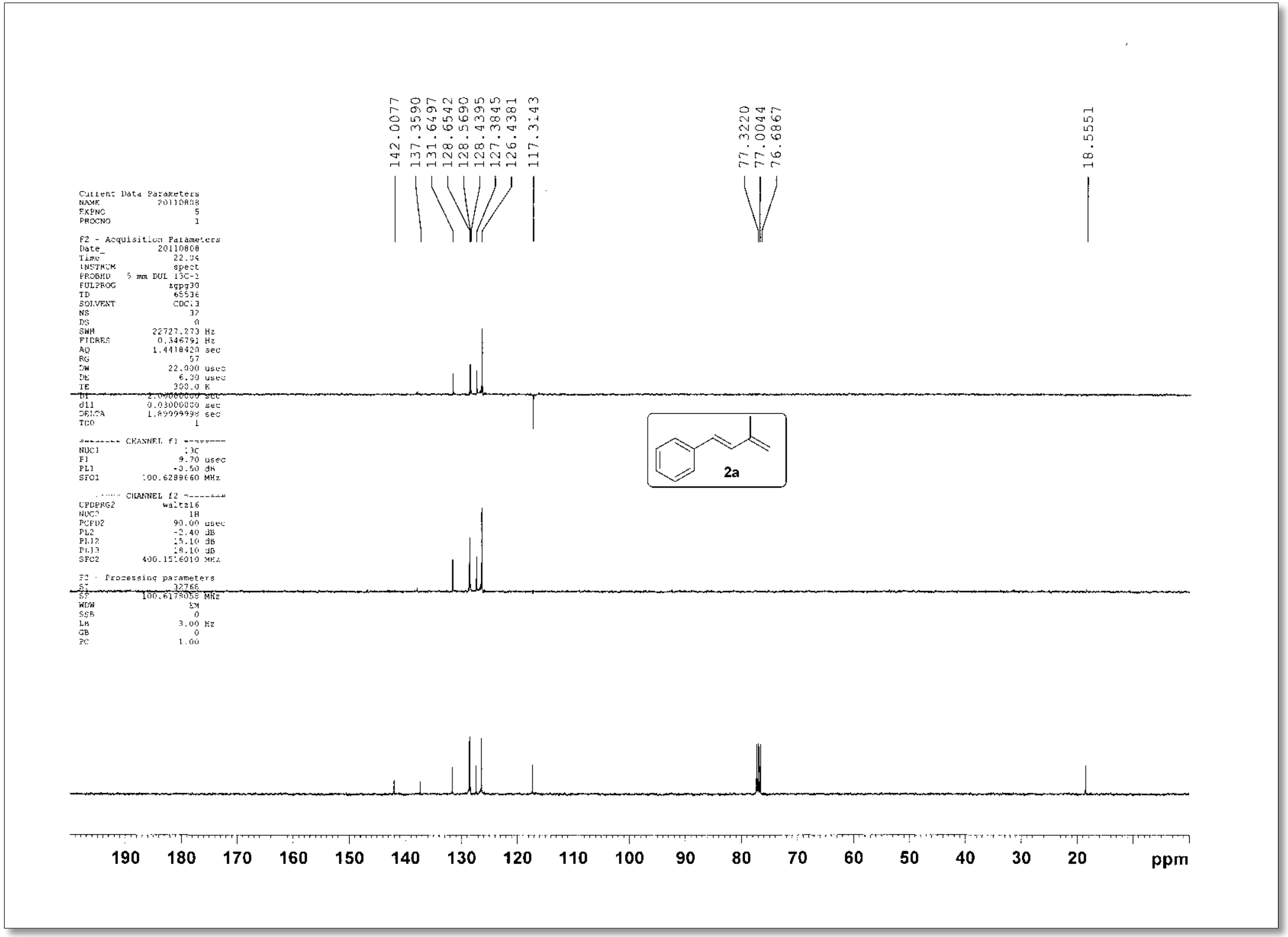
```
STANDARD PROTON PARAMETERS

exp63  PROTON

      SAMPLE              PRESATURATION
date   Mar  1 2012   satmode             n
solvent      cd2cl2  wet                 n
file /home/rsl/hsu~        SPECIAL
/hsu~20120301-43-H~  temp        not used
               .fid  gain        not used
    ACQUISITION      spin        not used
sw           6410.3  hst            0.008
at            2.556  pw90          14.900
np            32768  alfa          10.000
fb             4000          FLAGS
bs                4  il                  n
d1            1.000  in                  n
nt              100  dp                  y
ct               24  hs                 nn
    TRANSMITTER            PROCESSING
tn               H1  fn          not used
sfrq        399.766         DISPLAY
tof           399.8  sp            -200.1
tpwr             60  wp            4197.3
pw            7.450  rfl            806.5
     DECOUPLER       rfp                0
dn              C13  rp              65.6
dof               0  lp                 0
dm              nnn           PLOT
decwave     W40_ATB  wc               240
dpwr             35  sc                 0
dmf           29412  vs               360
                     th                 3
                     ai     ph
```



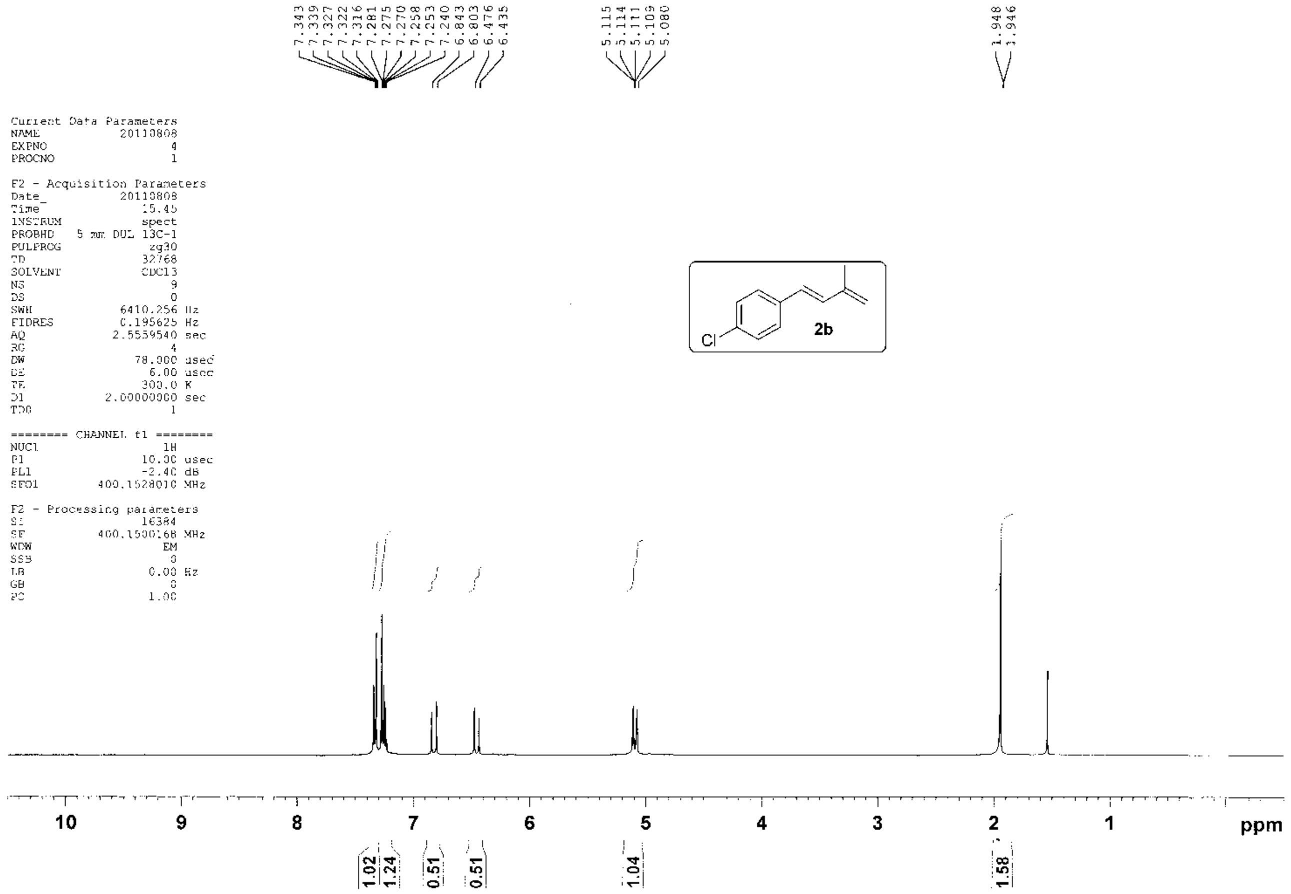
```
STANDARD CARBON PARAMETERS

exp61  CARBON

      SAMPLE              PRESATURATION
date   Mar  1 2012   satmode               n
solvent      cd2cl2   wet                   n
file            exp         SPECIAL
   ACQUISITION       temp          not used
sw          25510.2   gain          not used
at            1.285   spin          not used
np            65536   hst              0.008
fb            17000   pw90            10.300
bs                4   alfa            10.000
d1            1.000          FLAGS
nt           100000   il                    n
ct               96   in                    n
   TRANSMITTER        dp                    y
tn              C13   hs                   nn
sfrq        100.532        PROCESSING
tof          1530.6   lb                 0.50
tpwr             56   fn            not used
pw            5.150         DISPLAY
    DECOUPLER         sp                 -0.3
dn               H1   wp              22114.3
dof               0   rfl              7062.6
dm              yyy   rfp              5408.0
decwave           w   rp                112.9
dpwr             40   lp                 27.4
dmf            8811          PLOT
                      wc                  240
                      sc                    0
                      vs                   45
                      th                    1
                      nm       ph
```



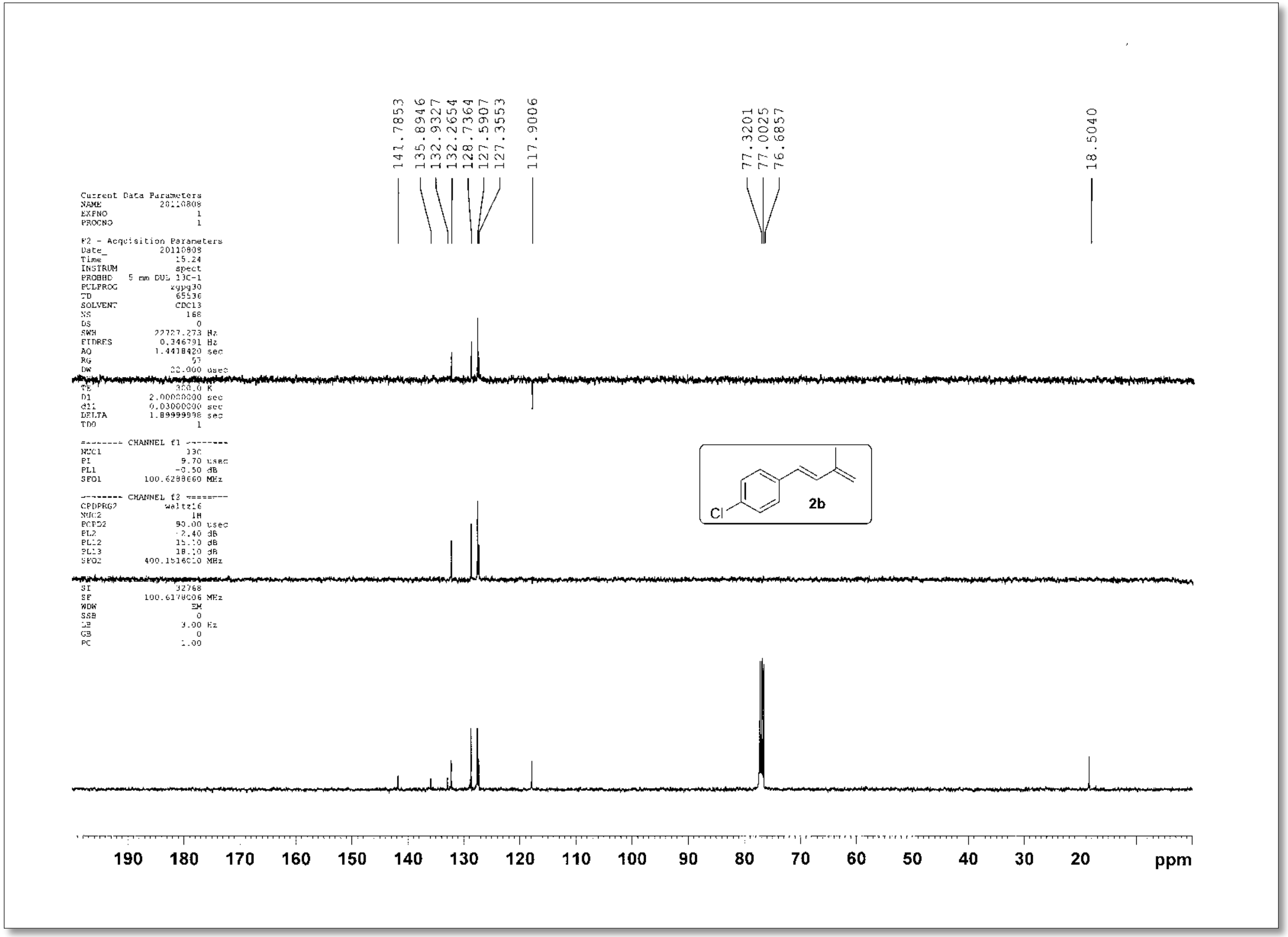
```
Current Data Parameters
NAME              20110808
EXPNO                    9
PROCNO                   1

F2 - Acquisition Parameters
Date              20110808
Time                 22.17
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      12
DS                       0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                       4
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF           400.1500168 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```



```
Current Data Parameters
NAME             20110808
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110808
Time                22.34
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178058 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```

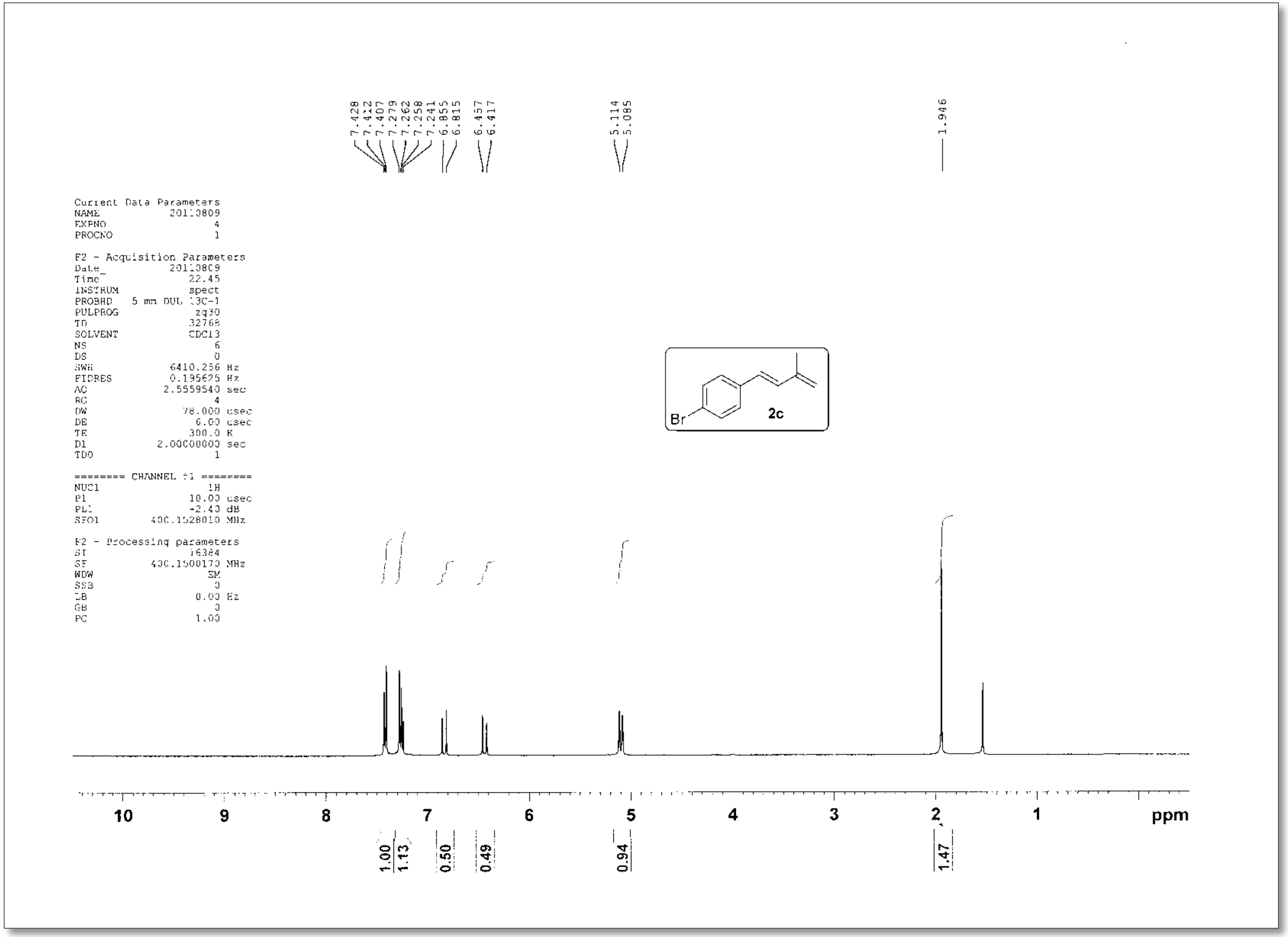


```
Current Data Parameters
NAME             20110808
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110808
Time                15.45
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      9
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                      4
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

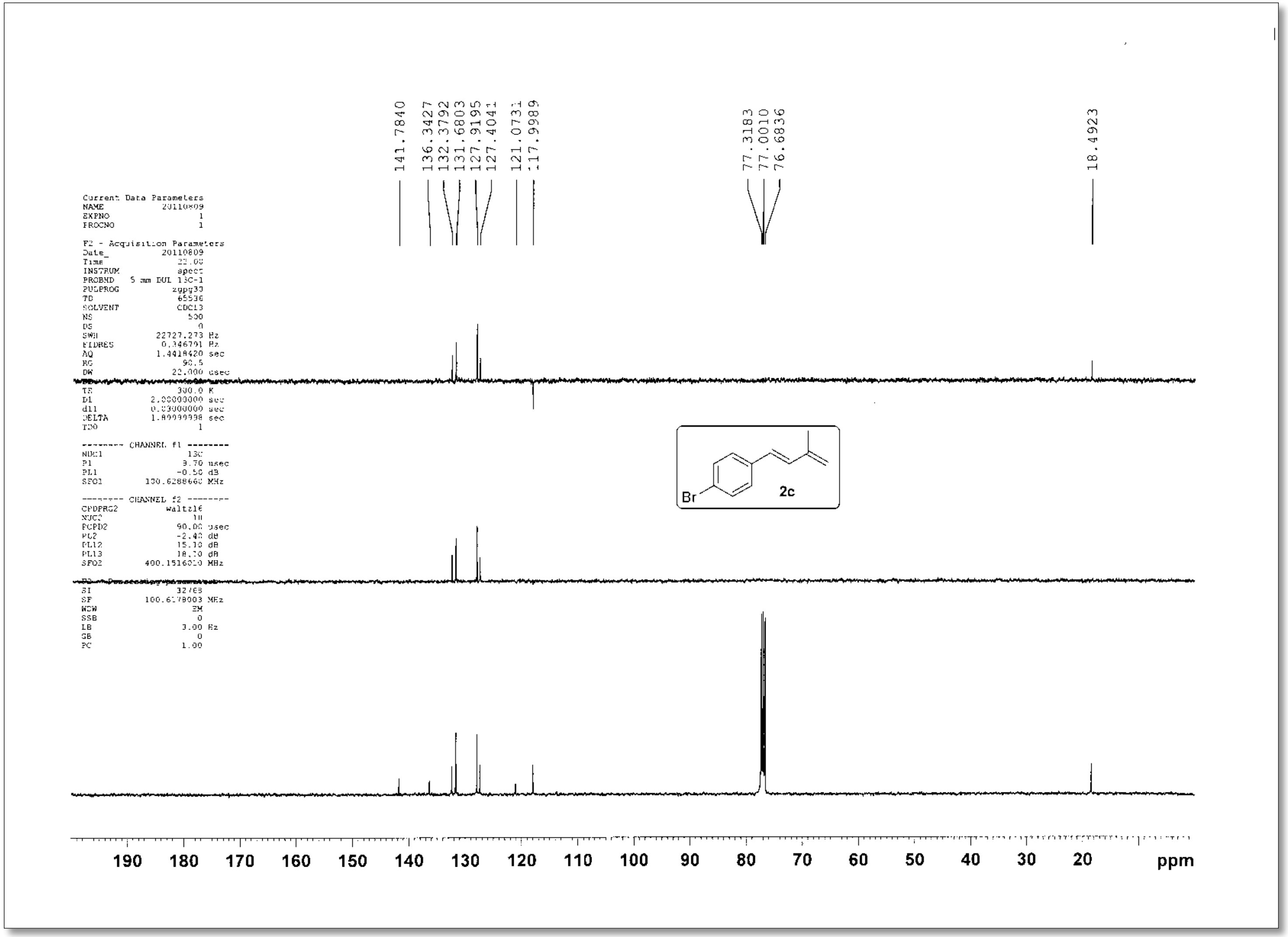


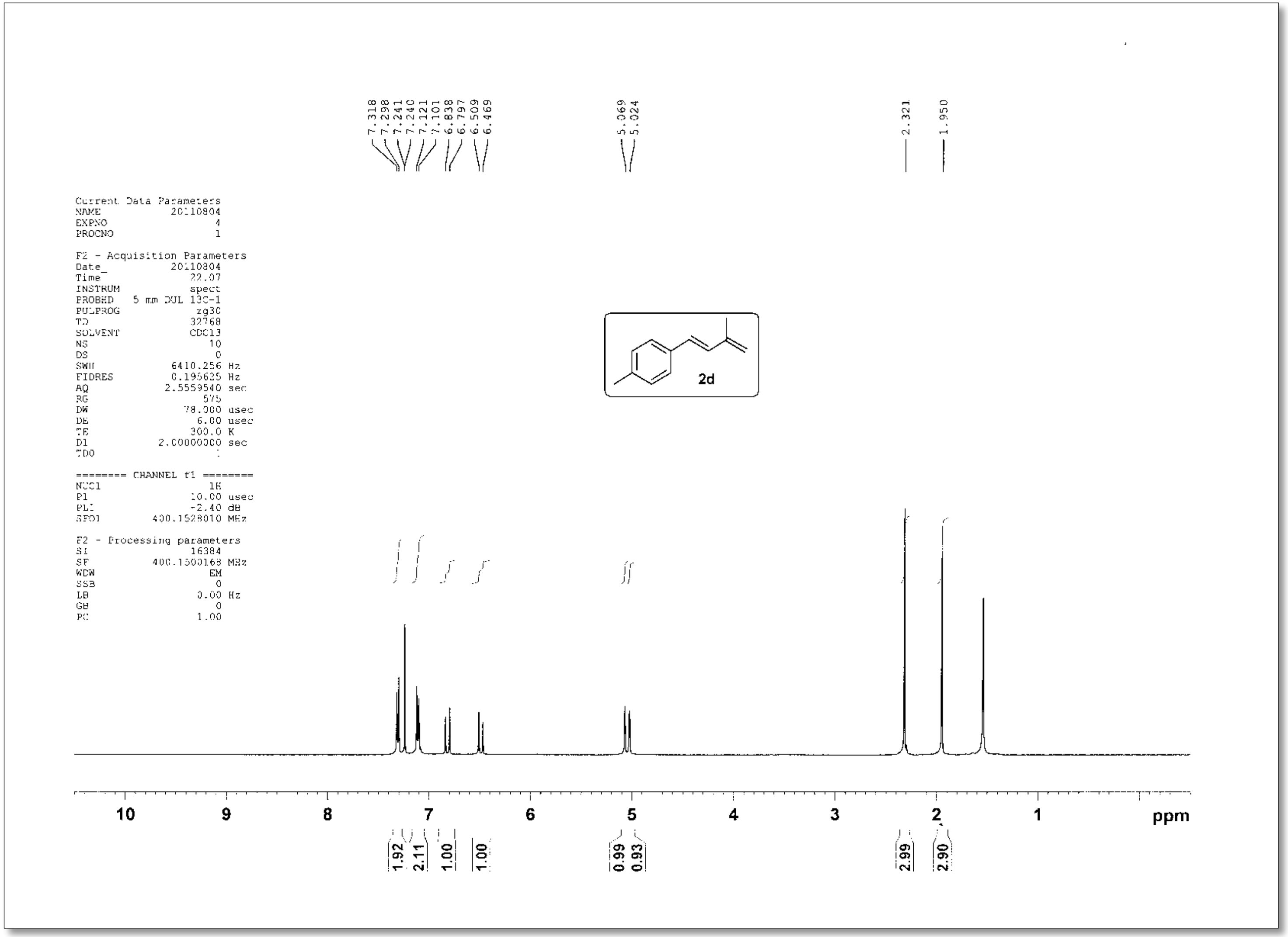
2b

```
Current Data Parameters
NAME              20110808
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110808
Time                 15.24
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     168
DS                       0
SWH              22727.273 Hz
FIDRES            0.346791 Hz
AO               1.4418420 sec
RG                      57
DW                  22.000 usec
TE                   300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                  -0.50 dB
SFO1           100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -2.40 dB
PL12                 15.10 dB
PL13                 18.10 dB
SFO2           400.1516010 MHz

SI                   32768
SF             100.6178006 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

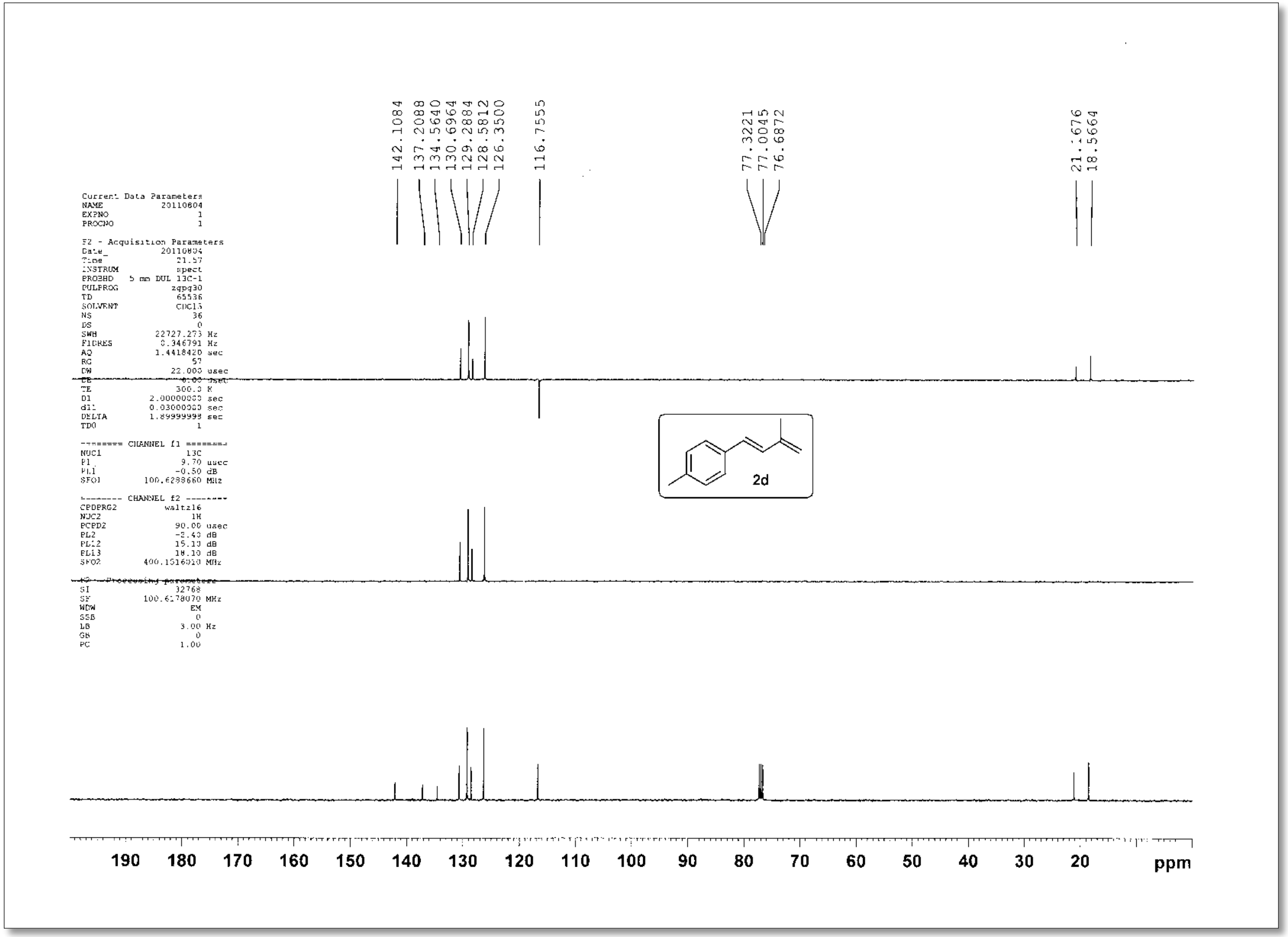


```
Current Data Parameters
NAME             20110809
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110809
Time_               22.45
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      6
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                      4
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500170 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

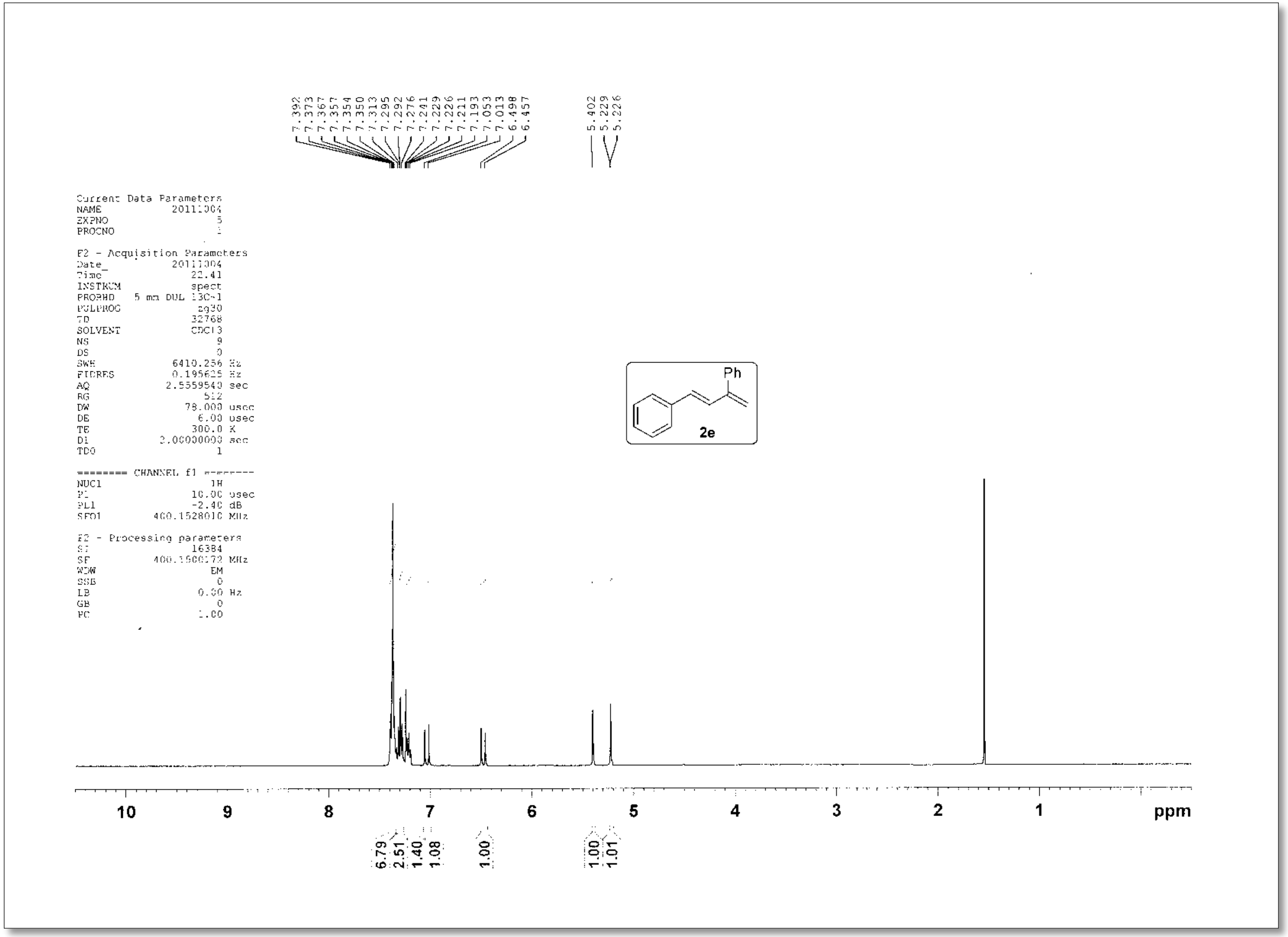

141.7840
136.3427
132.3792
131.6803
127.9195
127.4041
121.0731
117.9989
77.3183
77.0010
76.6836
18.4923
Current Data Parameters
NAME 20110809
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110809
Time 22.08
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 500
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 90.5
DW 22.000 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178003 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Br
2c
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



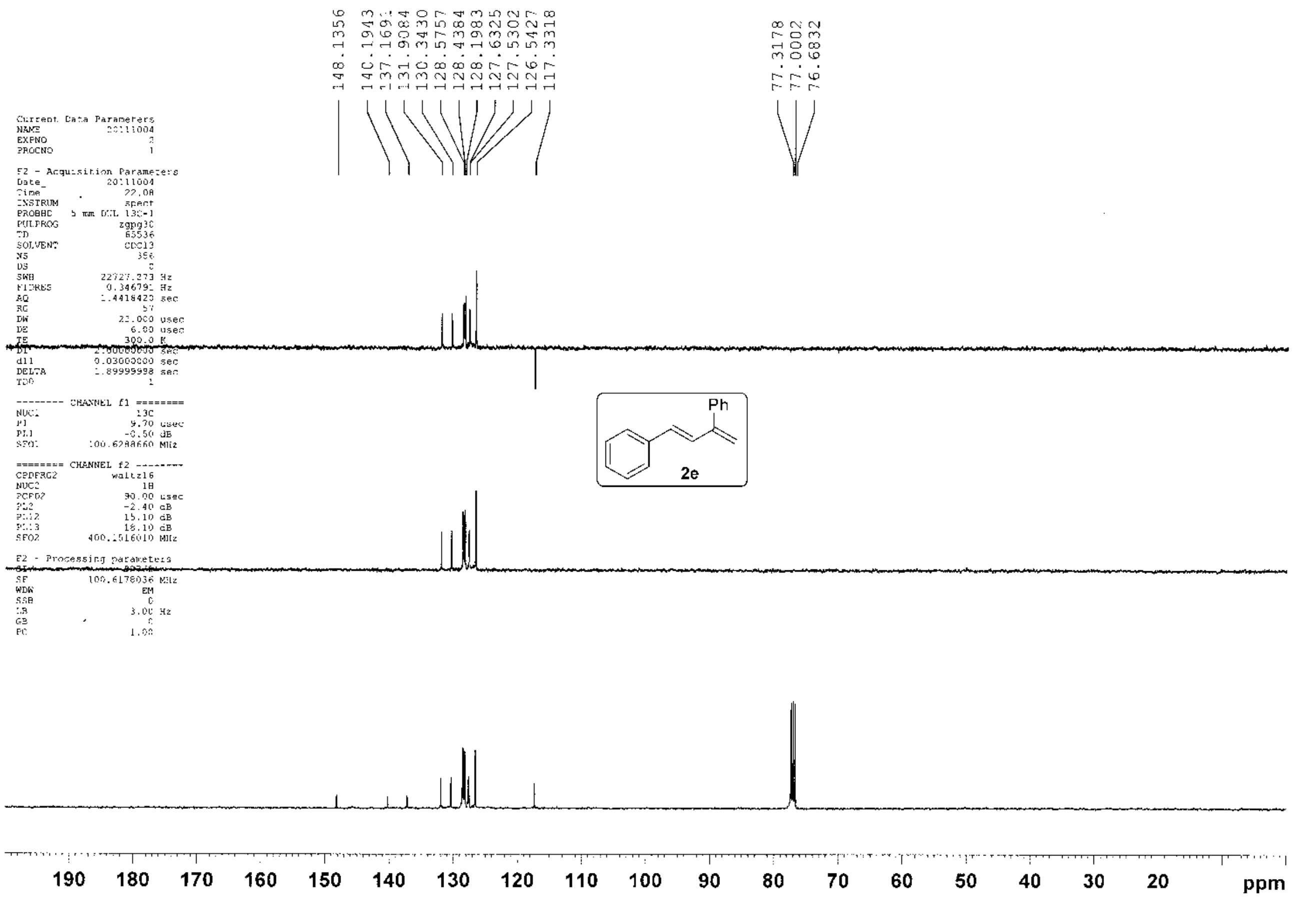
```
Current Data Parameters
NAME             20110804
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110804
Time                22.07
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     10
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    575
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



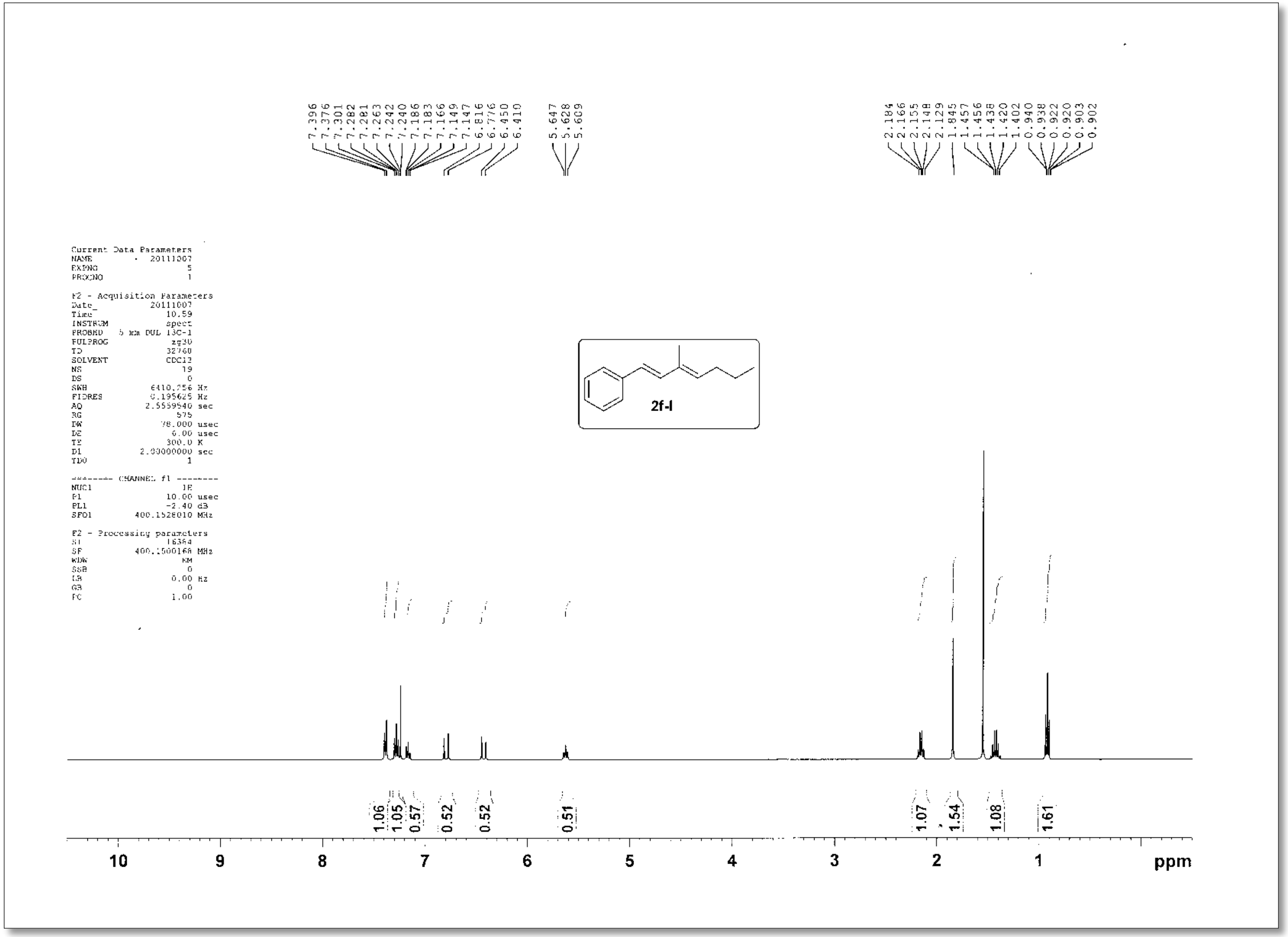
```
Current Data Parameters
NAME             20110604
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110604
Time                21.57
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    36
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178070 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



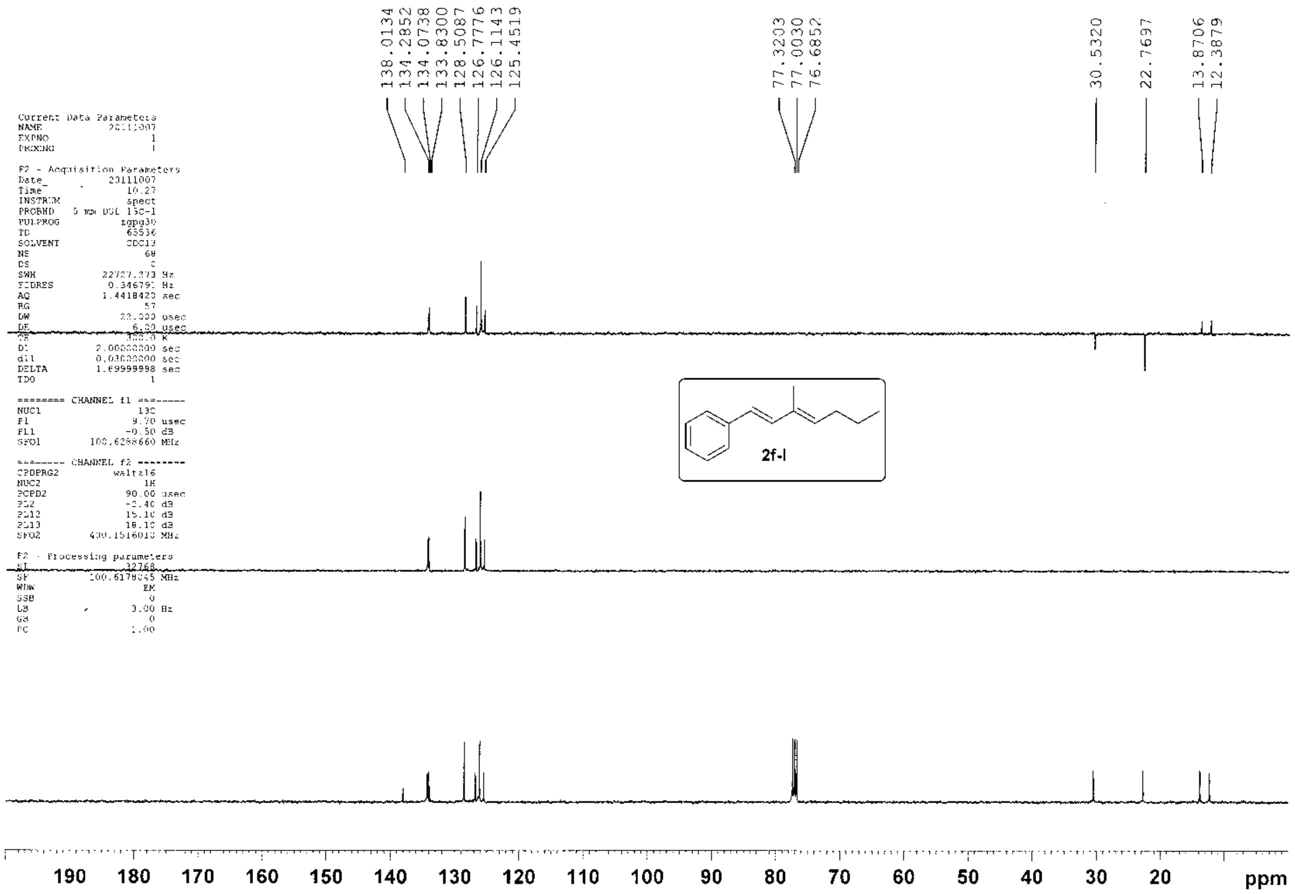
```
Current Data Parameters
NAME             20111004
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111004
Time                22.41
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                      9
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    512
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500172 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



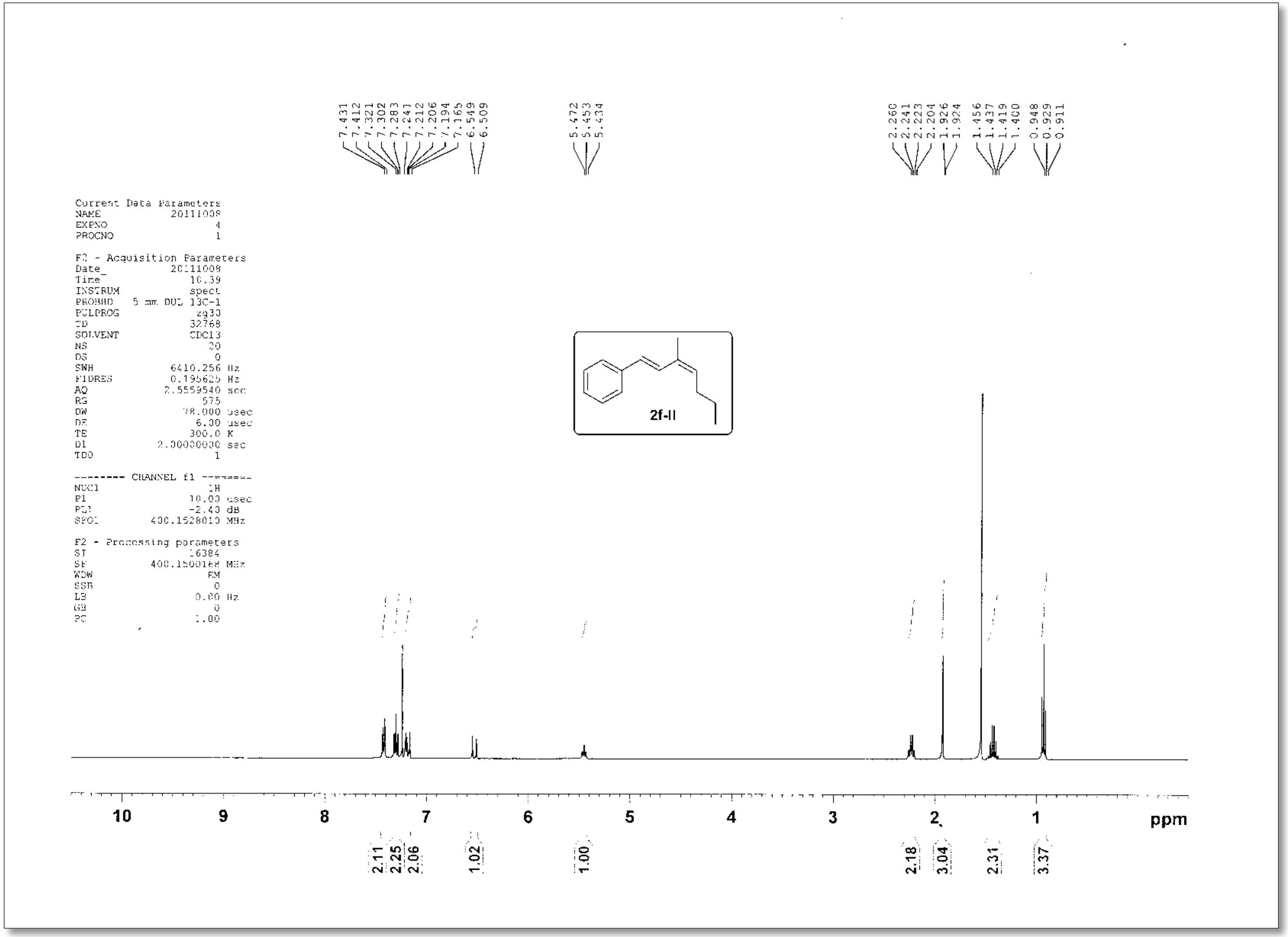
```
Current Data Parameters
NAME             20111004
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111004
Time                22.08
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    356
DS                      0
SWB          22727.273 Hz
FIDRES        0.346791 Hz
AQ           1.4418420 sec
RG                     57
DW              22.000 usec
DE                6.00 usec
TE               300.0 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                     1

-------- CHANNEL f1 ========
NUC1                  13C
P1                9.70 usec
PL1              -0.50 dB
SFO1       100.6288660 MHz

======== CHANNEL f2 --------
CPDPRG2           waltz16
NUC2                   1H
PCPD2            90.00 usec
PL2              -2.40 dB
PL12             15.10 dB
PL13             18.10 dB
SFO2       400.1516010 MHz

F2 - Processing parameters
SI                 [illegible]
SF         100.6178036 MHz
WDW                    EM
SSB                     0
LB                3.00 Hz
GB                      0
PC                   1.00
```



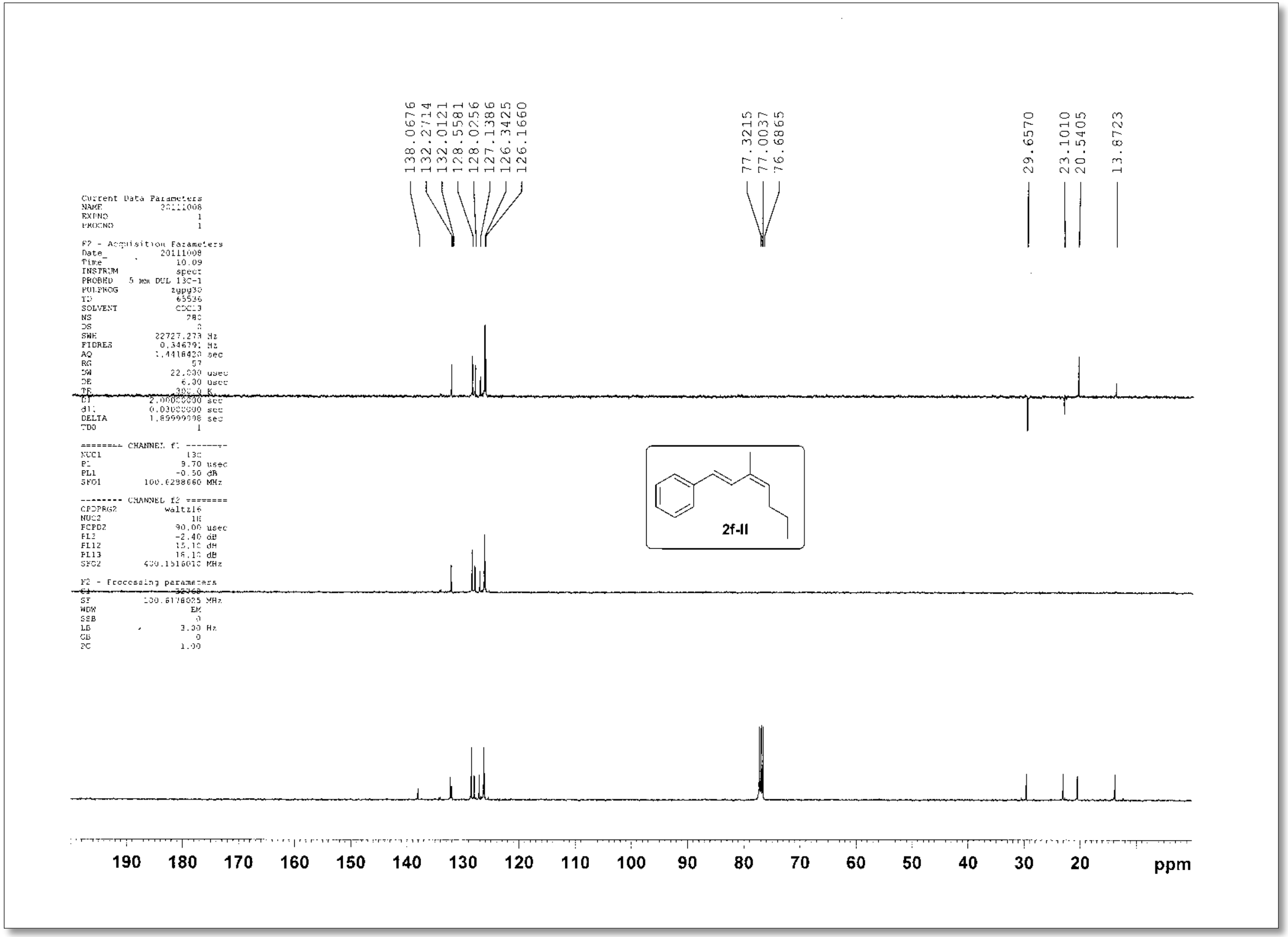
```
Current Data Parameters
NAME          .  20111007
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111007
Time                10.59
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     19
DS                      0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                    575
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

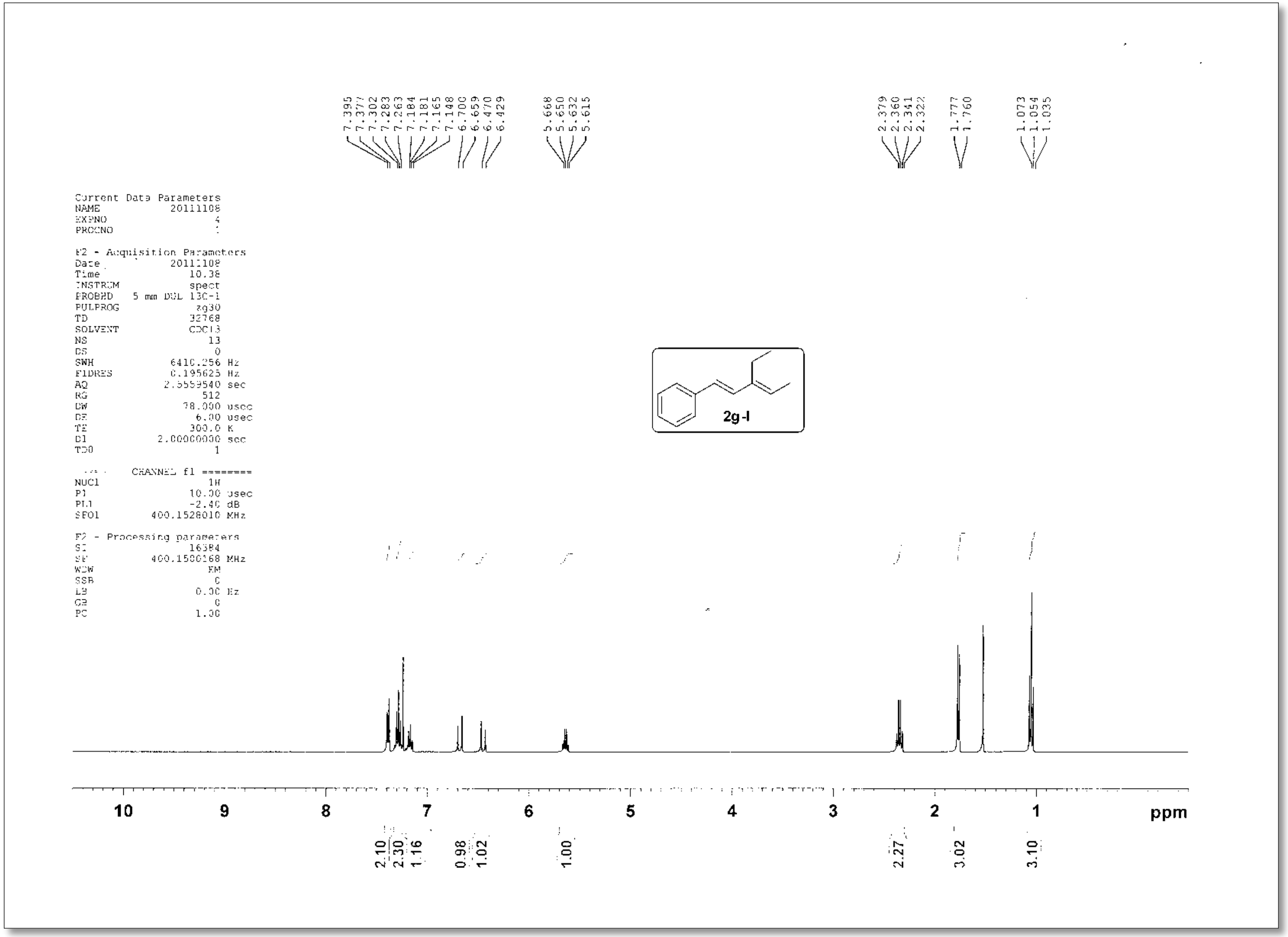


```
Current Data Parameters
NAME            20111007
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111007
Time               10.27
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    68
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.69999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178045 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```

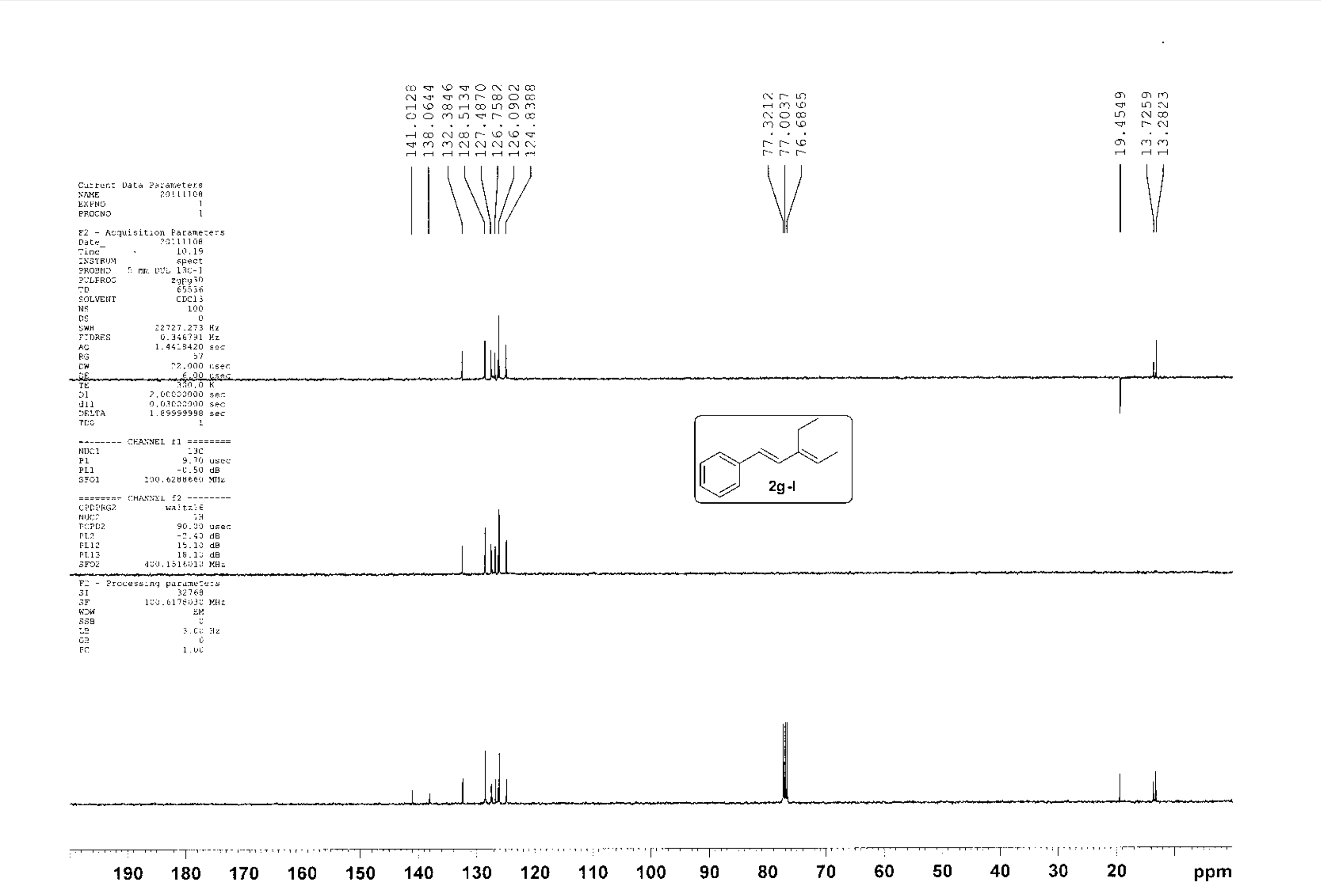


```
Current Data Parameters
NAME              20111008
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111008
Time                 10.39
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      20
DS                       0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ               2.5559540 sec
RG                     575
DW                  78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1           400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF             400.1500188 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

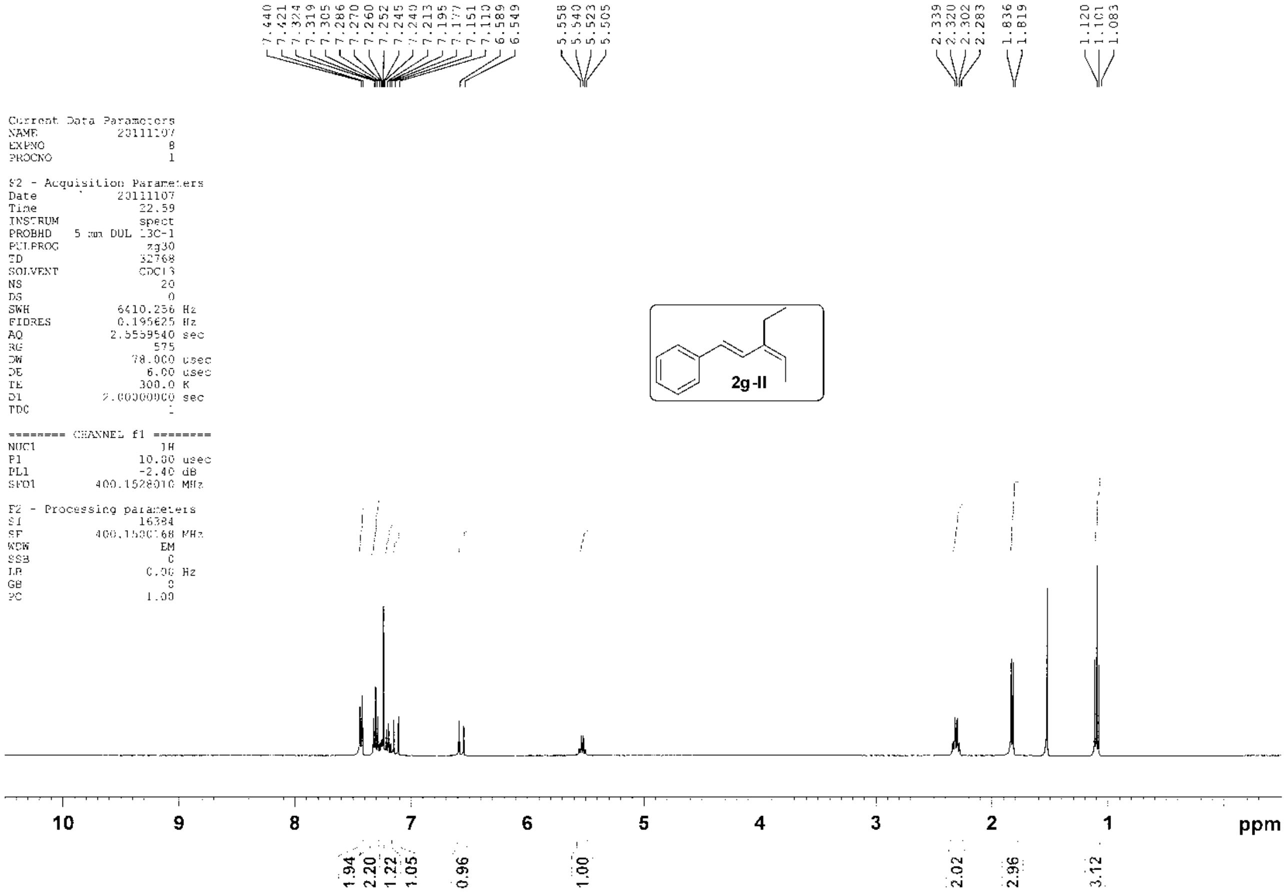

138.0676
132.2714
132.0121
128.5581
128.0256
127.1386
126.3425
126.1660
77.3215
77.0037
76.6865
29.6570
23.1010
20.5405
13.8723
Current Data Parameters
NAME 20111008
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111008
Time 10.09
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 780
DS 2
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.30 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6298660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 16.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178025 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
2f-II
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



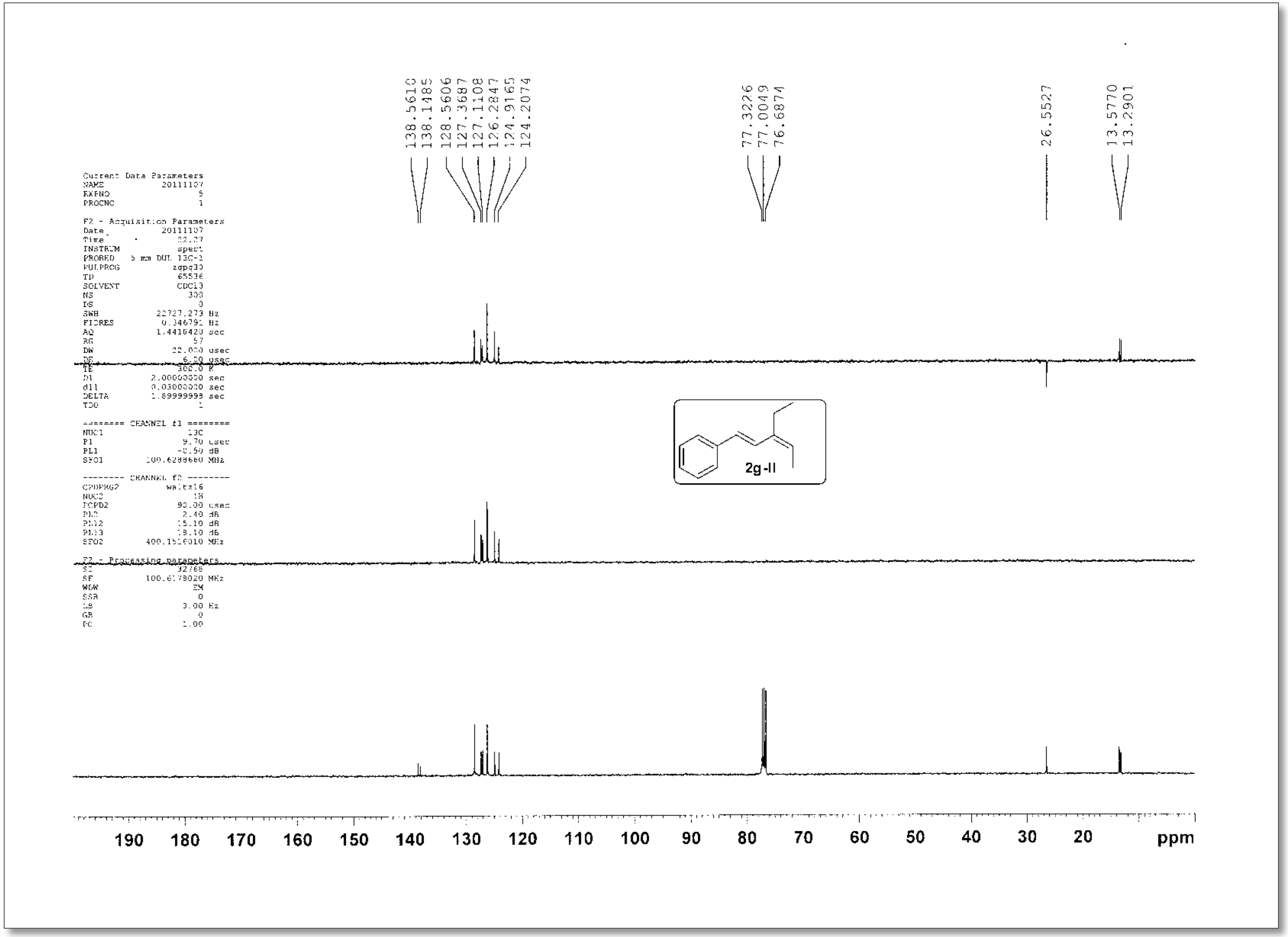
```
Current Data Parameters
NAME             20111108
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111108
Time                10.38
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     13
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    512
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



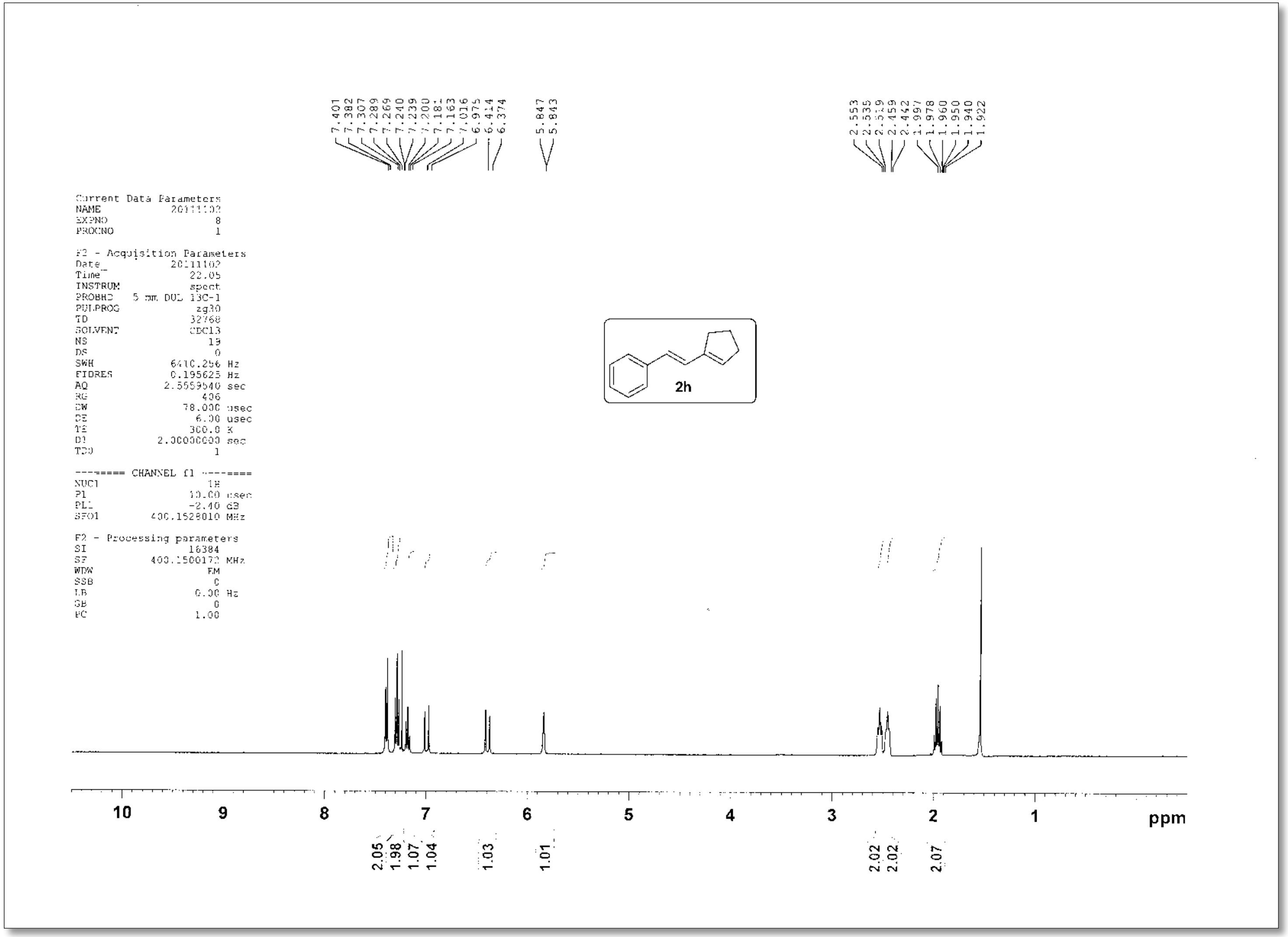
```
Current Data Parameters
NAME             20111108
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111108
Time                10.19
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    100
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178030 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



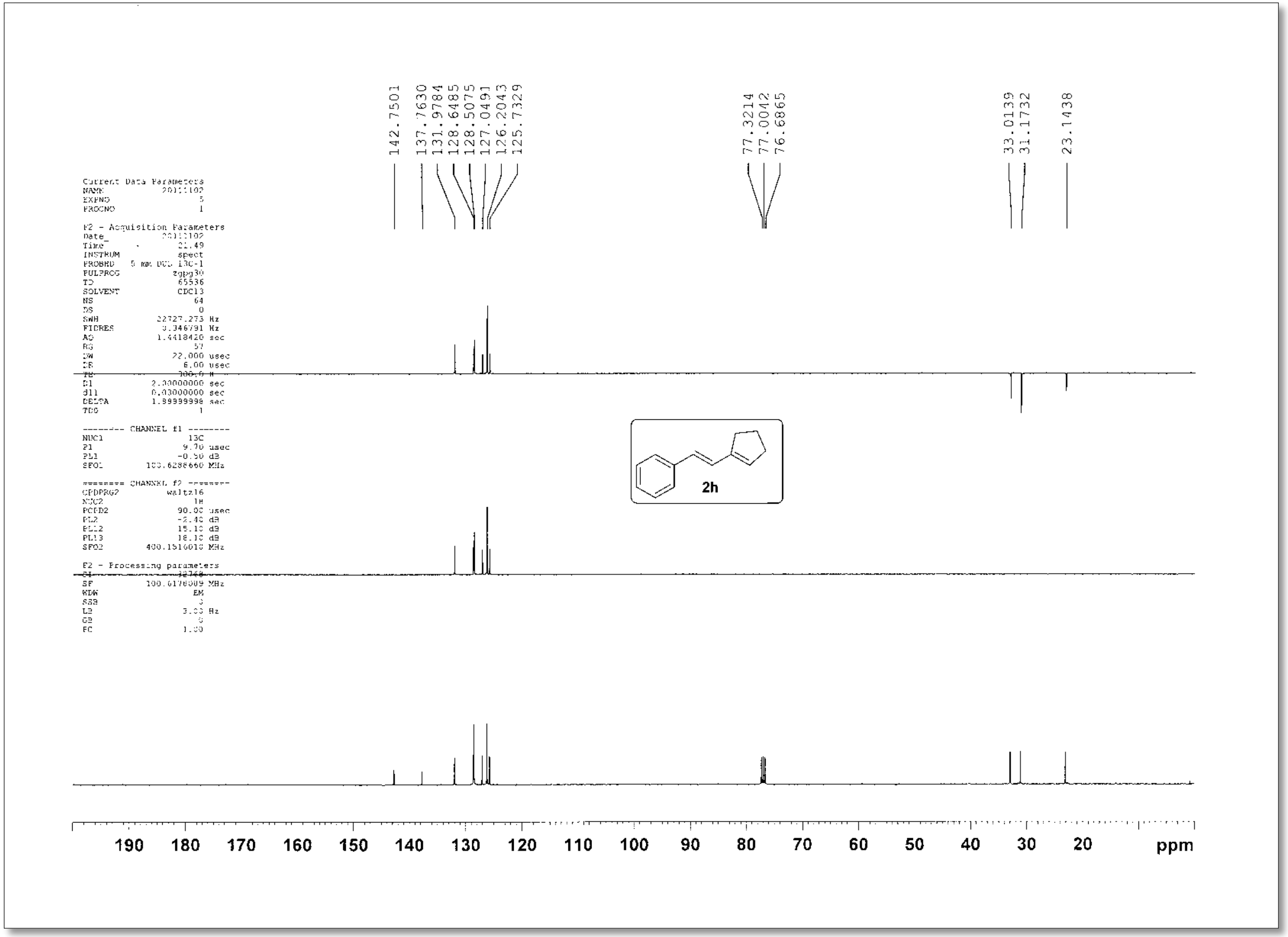
```
Current Data Parameters
NAME             20111107
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date             20111107
Time                22.59
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     20
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    575
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TDO                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



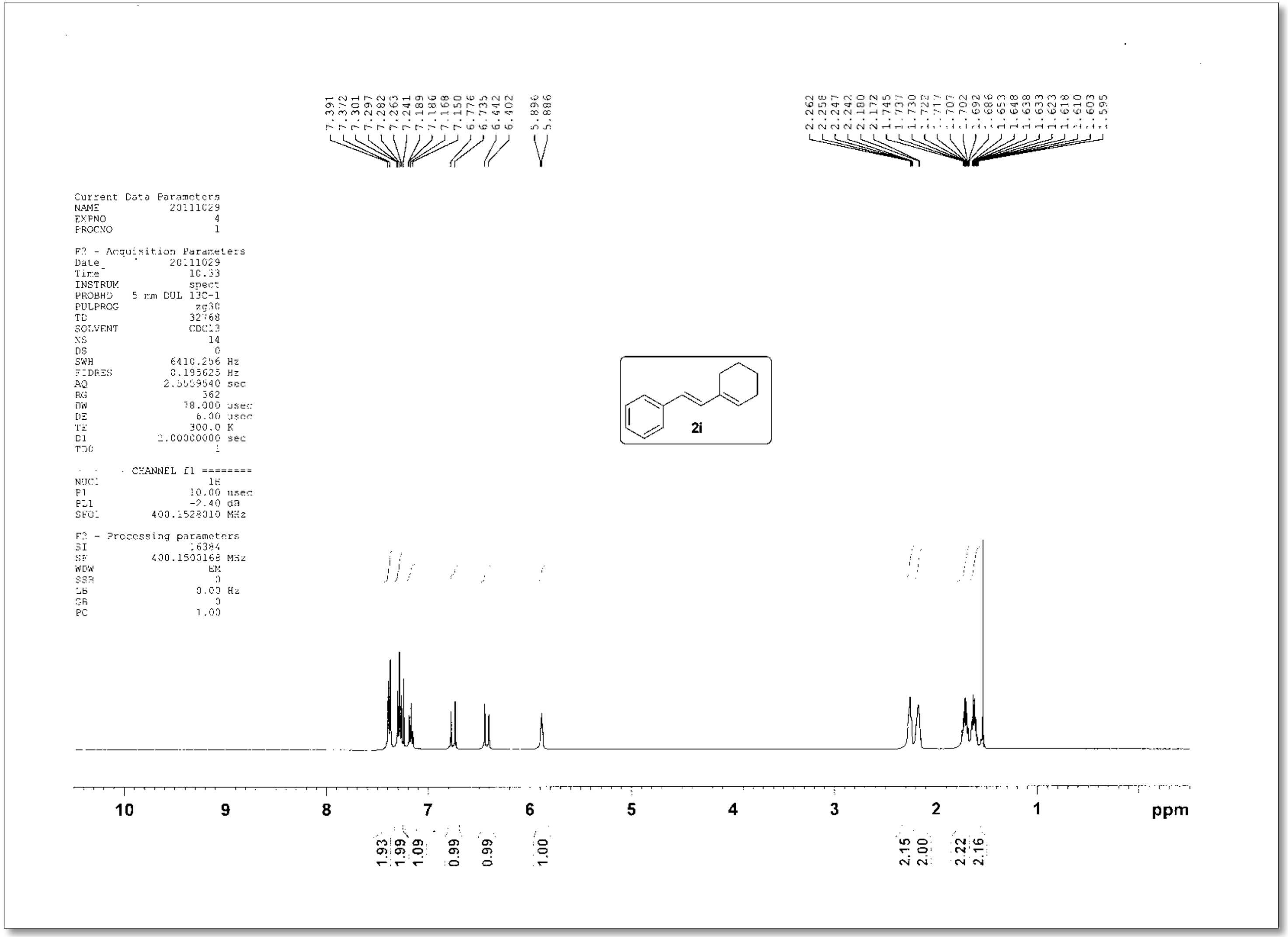
```
Current Data Parameters
NAME             20111107
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111107
Time                22.27
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

-------- CHANNEL f2 --------
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                 2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178020 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



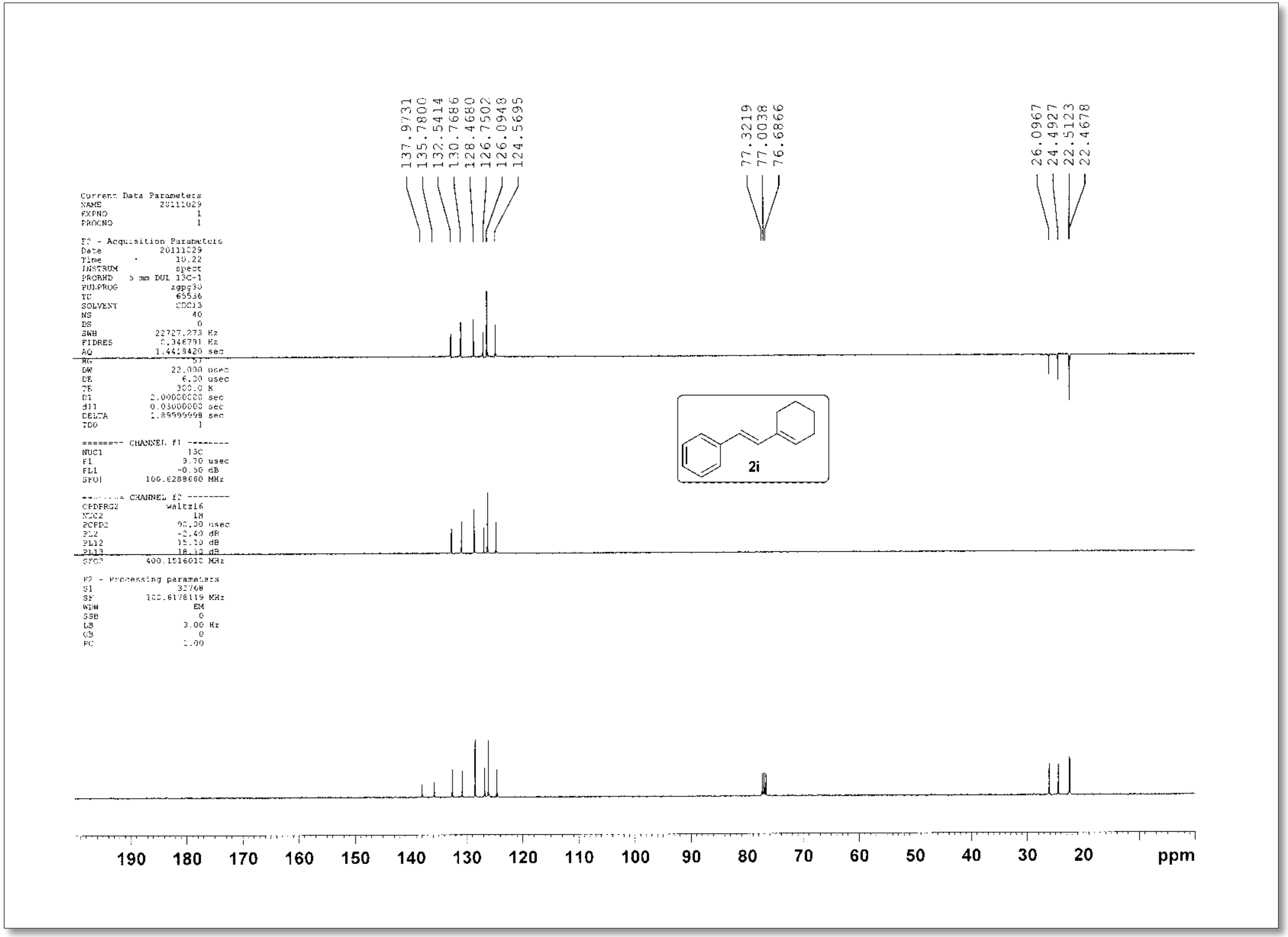
```
Current Data Parameters
NAME             20111102
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111102
Time                22.05
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     19
DS                      0
SWH           6410.256 Hz
FIDRES        0.195625 Hz
AQ           2.5559540 sec
RG                    406
DW             78.000 usec
DE               6.00 usec
TE              300.0 K
D1         2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1              10.00 usec
PL1             -2.40 dB
SFO1      400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF        400.1500172 MHz
WDW                    EM
SSB                     0
LB               0.00 Hz
GB                      0
PC                   1.00
```



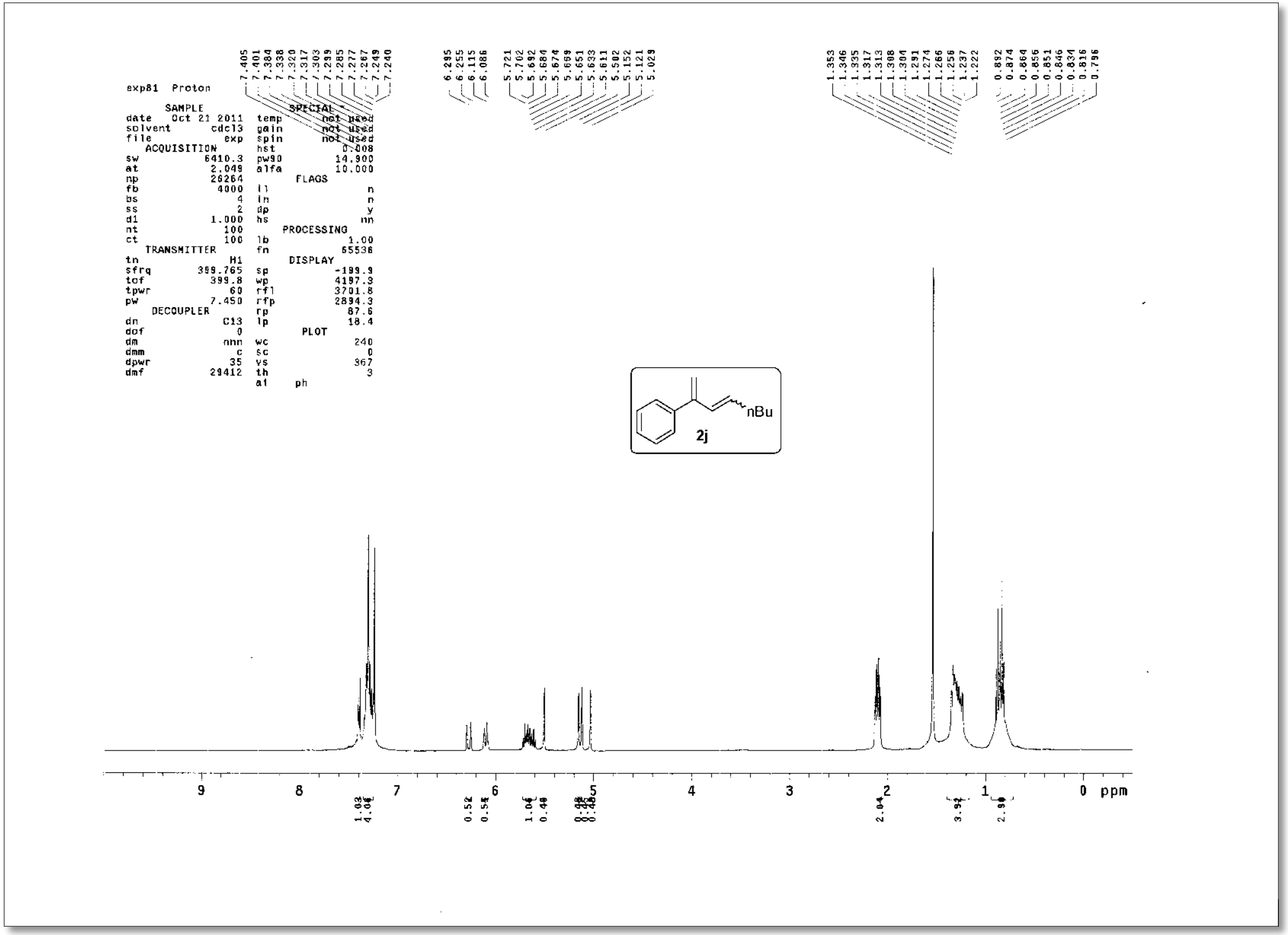
```
Current Data Parameters
NAME              20111102
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111102
Time      .          21.49
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     64
DS                      0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

-------- CHANNEL f1 --------
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF           100.6178009 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



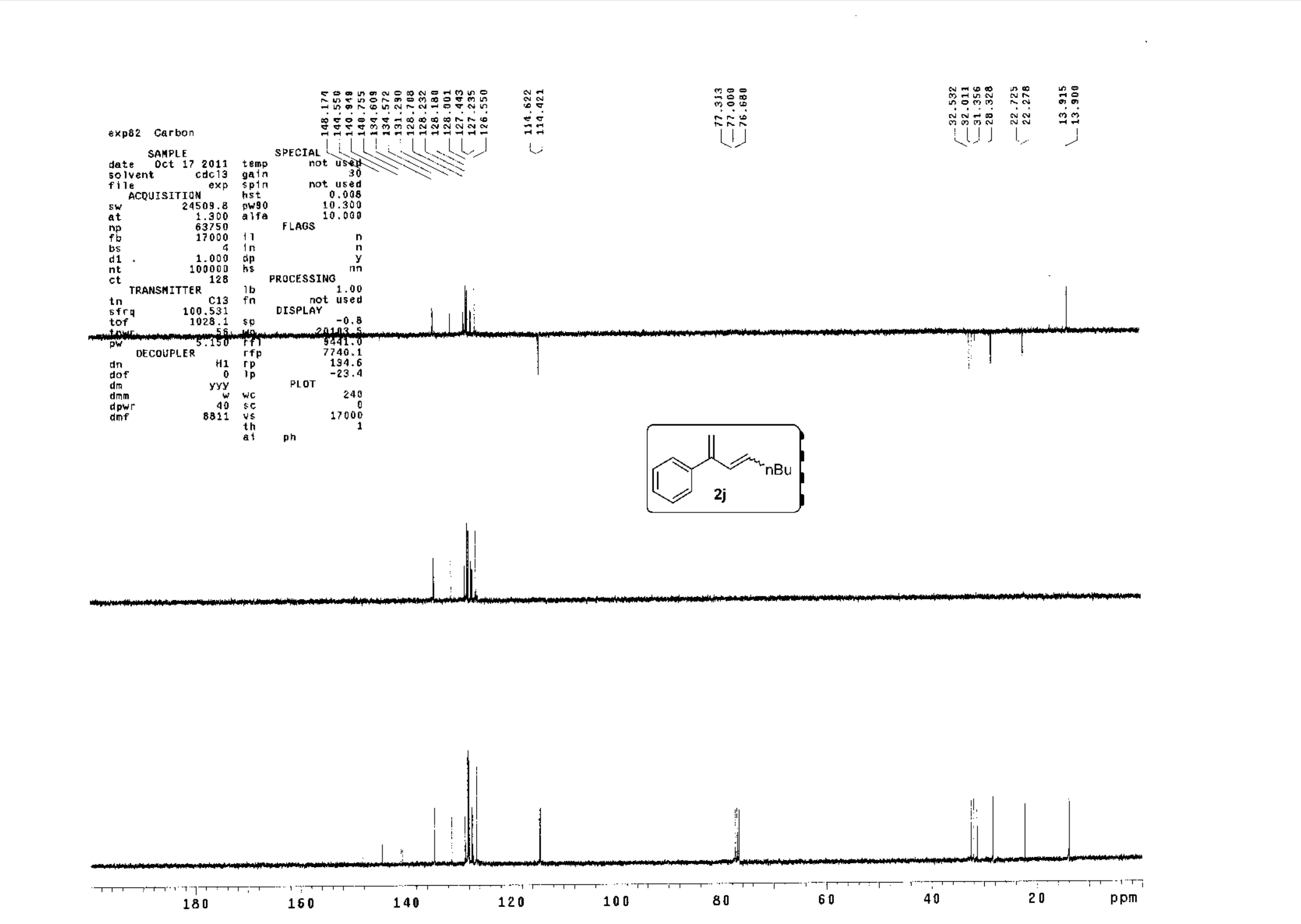
```
Current Data Parameters
NAME            20111029
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111029
Time               10.33
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    14
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   362
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500168 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

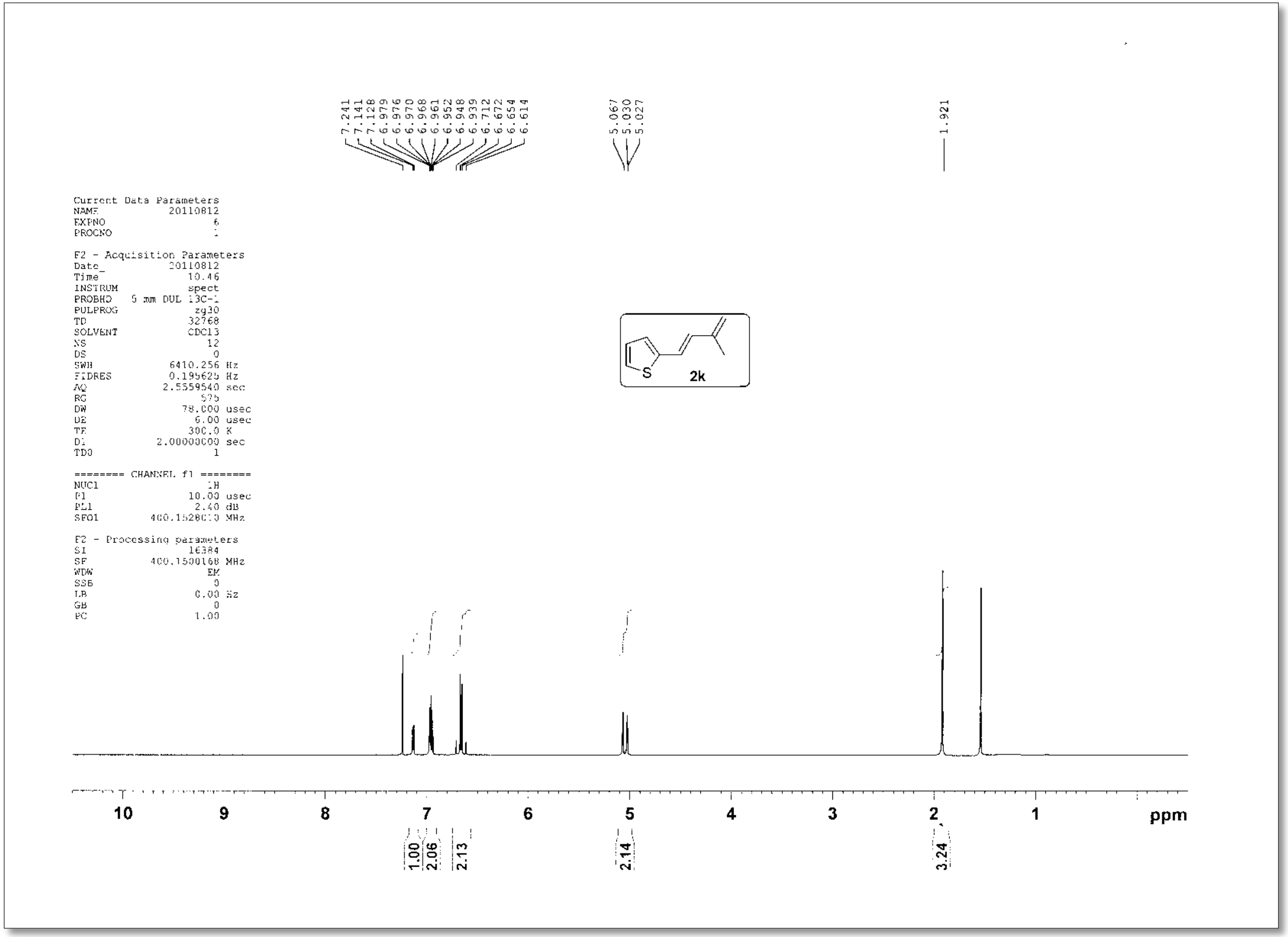


```
Current Data Parameters
NAME             20111029
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date             20111029
Time        .       10.22
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     40
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178119 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```

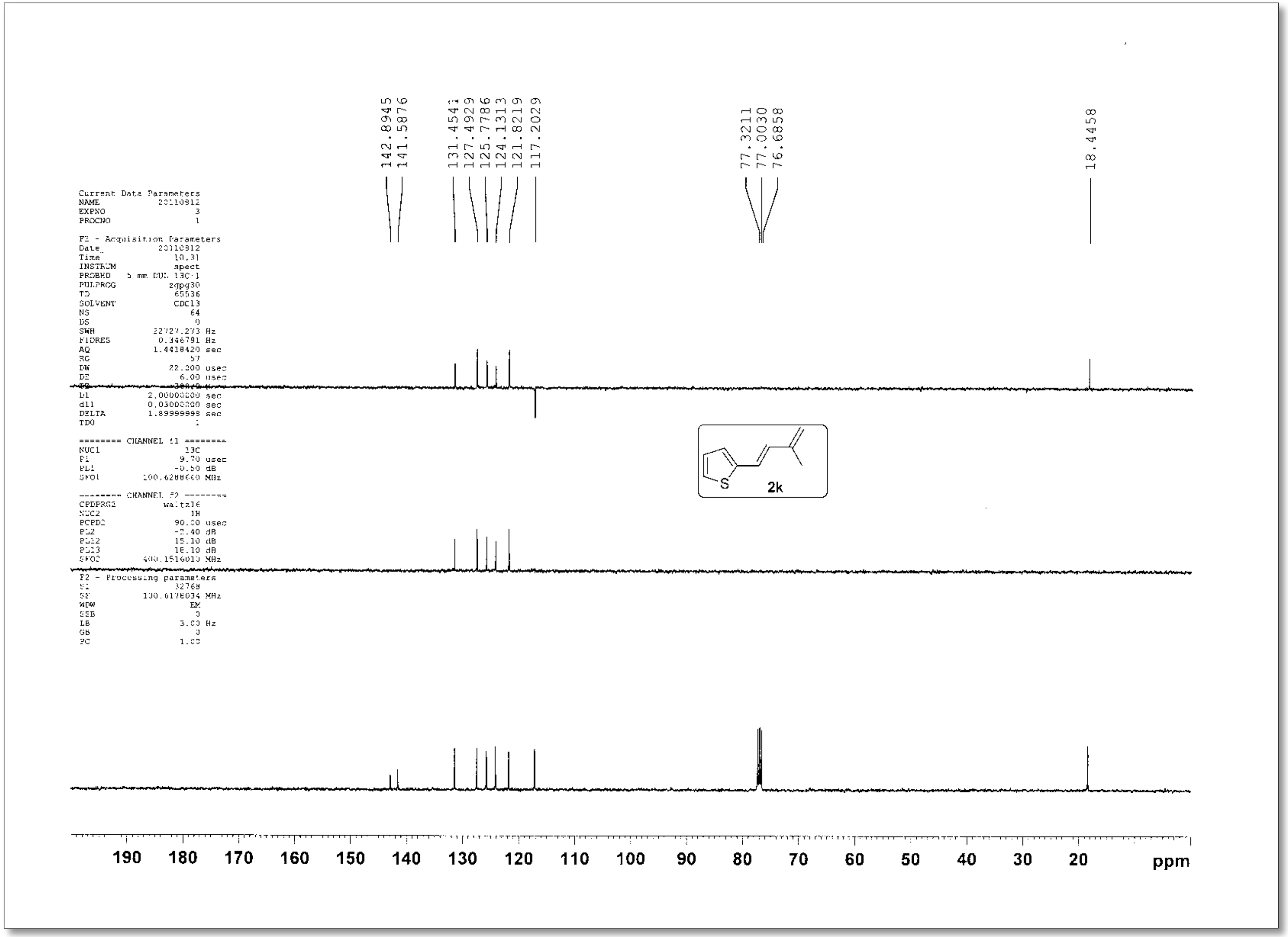


```
exp81  Proton

      SAMPLE                     SPECIAL
date    Oct 21 2011   temp          not used
solvent       cdcl3   gain          not used
file            exp   spin          not used
   ACQUISITION        hst              0.008
sw           6410.3   pw90            14.900
at            2.049   alfa            10.000
np            26264          FLAGS
fb             4000   il                   n
bs                4   in                   n
ss                2   dp                   y
d1            1.000   hs                  nn
nt              100        PROCESSING
ct              100   lb                1.00
   TRANSMITTER        fn               65536
tn               H1          DISPLAY
sfrq        399.765   sp              -199.9
tof           399.8   wp              4197.9
tpwr             60   rfl             3701.8
pw            7.450   rfp             2894.3
    DECOUPLER         rp                87.6
dn              C13   lp                18.4
dof               0           PLOT
dm              nnn   wc                 240
dmm               c   sc                   0
dpwr             35   vs                 367
dmf           29412   th                   3
                      ai     ph
```

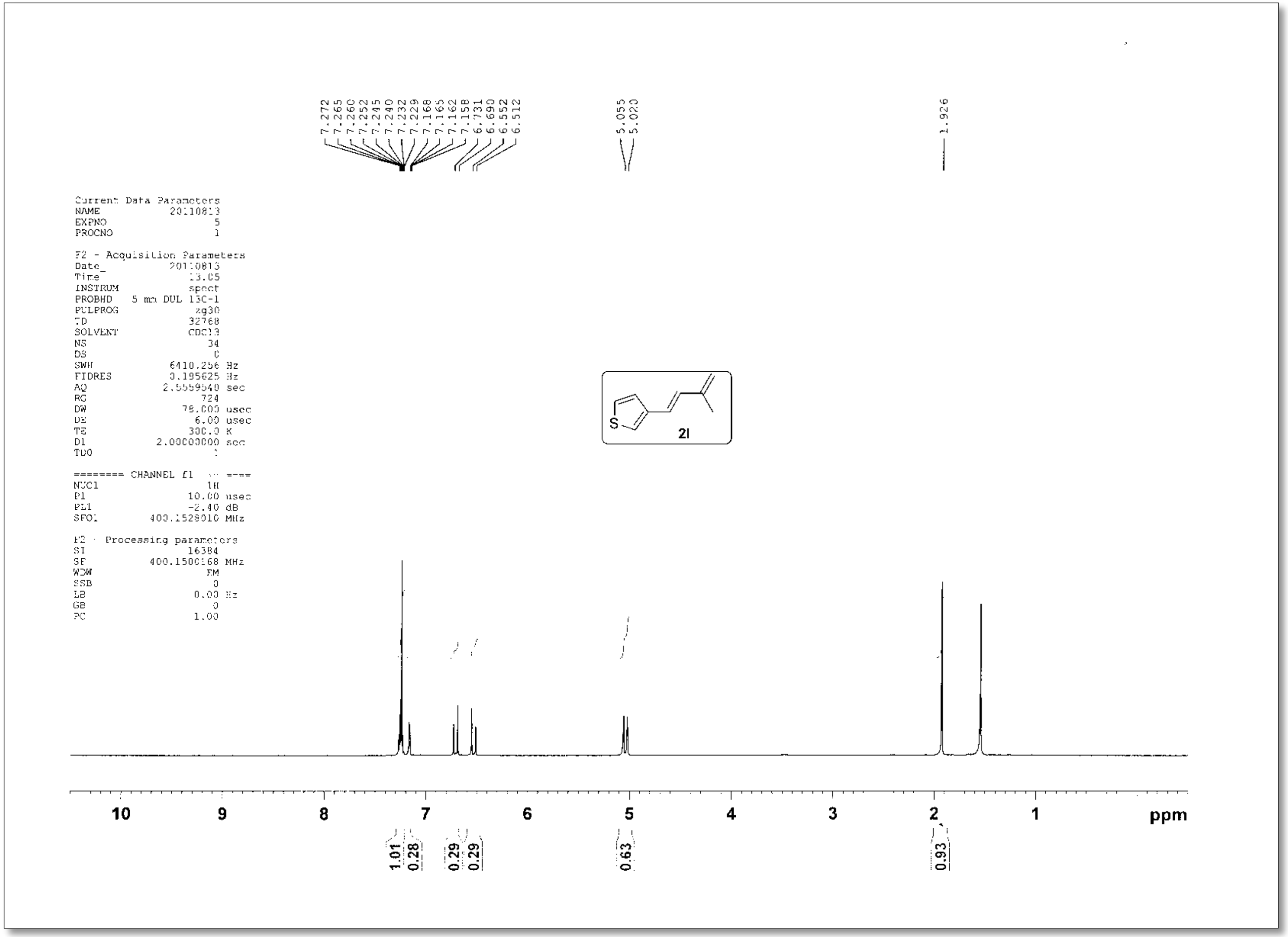

exp82 Carbon
SAMPLE
date Oct 17 2011
solvent cdcl3
file exp
ACQUISITION
sw 24509.8
at 1.300
np 63750
fb 17000
bs 4
d1 1.000
nt 100000
ct 128
TRANSMITTER
tn C13
sfrq 100.531
tof 1028.1
tpwr 56
pw 5.150
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 40
dmf 8811
SPECIAL
temp not used
gain 30
spin not used
hst 0.008
pw90 10.300
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -0.8
wp 20103.5
rfl 9441.0
rfp 7740.1
rp 134.6
lp -23.4
PLOT
wc 240
sc 0
vs 17000
th 1
ai ph
148.174
144.550
140.948
140.755
134.609
134.572
131.290
128.788
128.232
128.180
128.001
127.443
127.235
126.550
114.622
114.421
77.313
77.000
76.680
32.532
32.011
31.356
28.328
22.725
22.278
13.915
13.900
nBu
2j
180
160
140
120
100
80
60
40
20
ppm



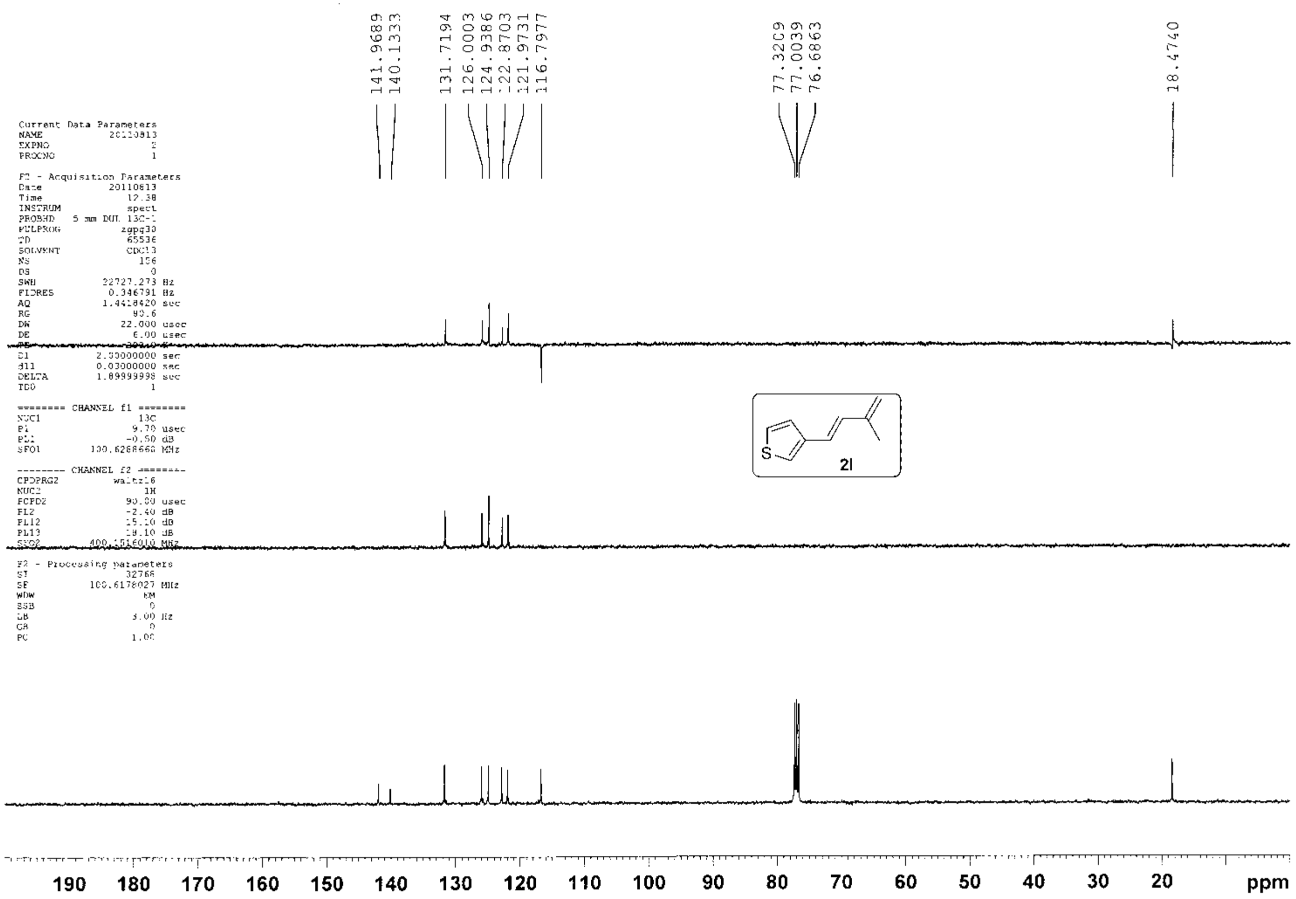
```
Current Data Parameters
NAME            20110812
EXPNO                  6
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110812
Time               10.46
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   575
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                 2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500168 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



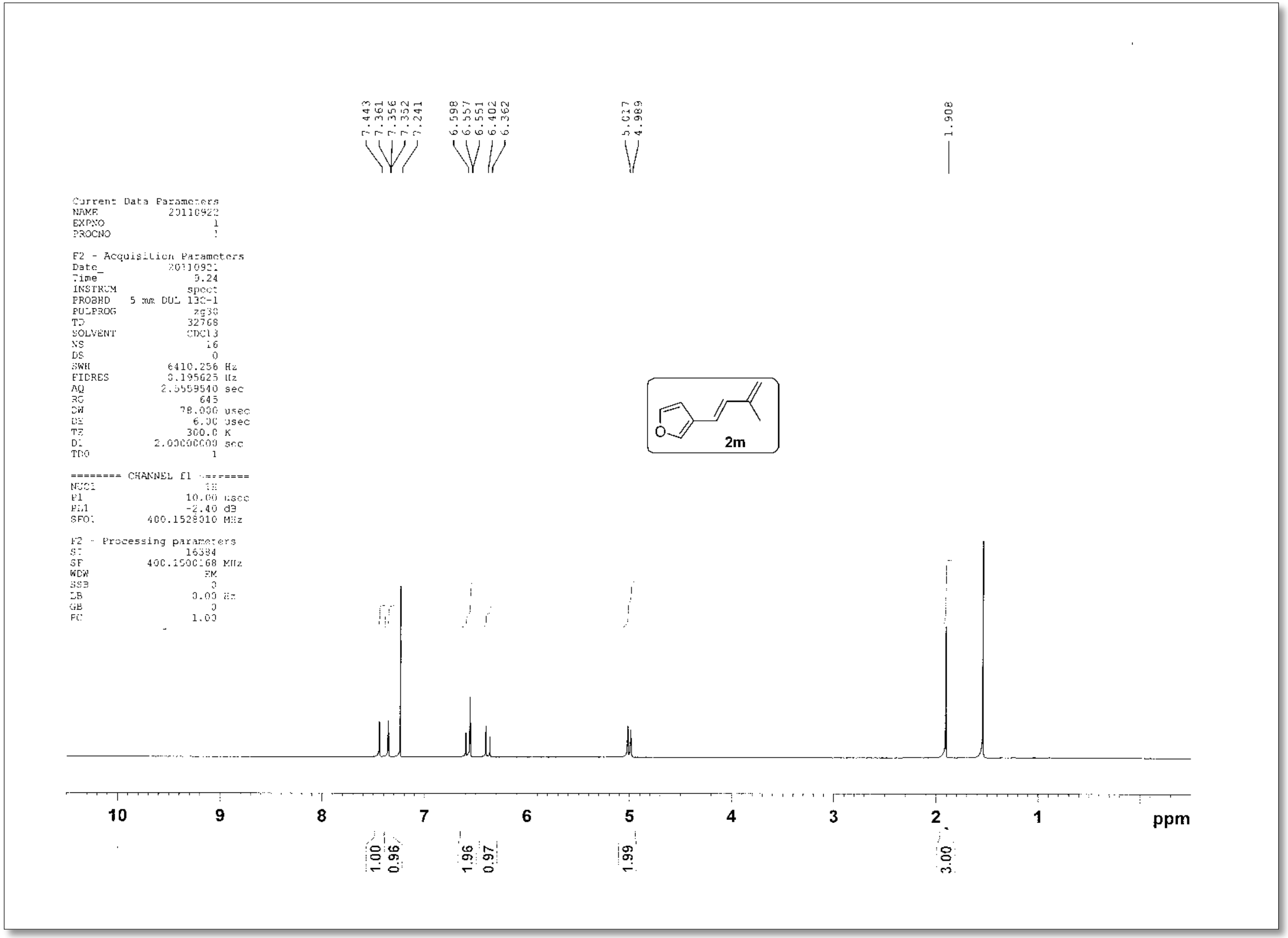
```
Current Data Parameters
NAME              20110912
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110912
Time                 10.31
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      64
DS                       0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                      57
DW                  22.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                  -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -2.40 dB
PL12                 15.10 dB
PL13                 18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                   32768
SF            100.6178034 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```



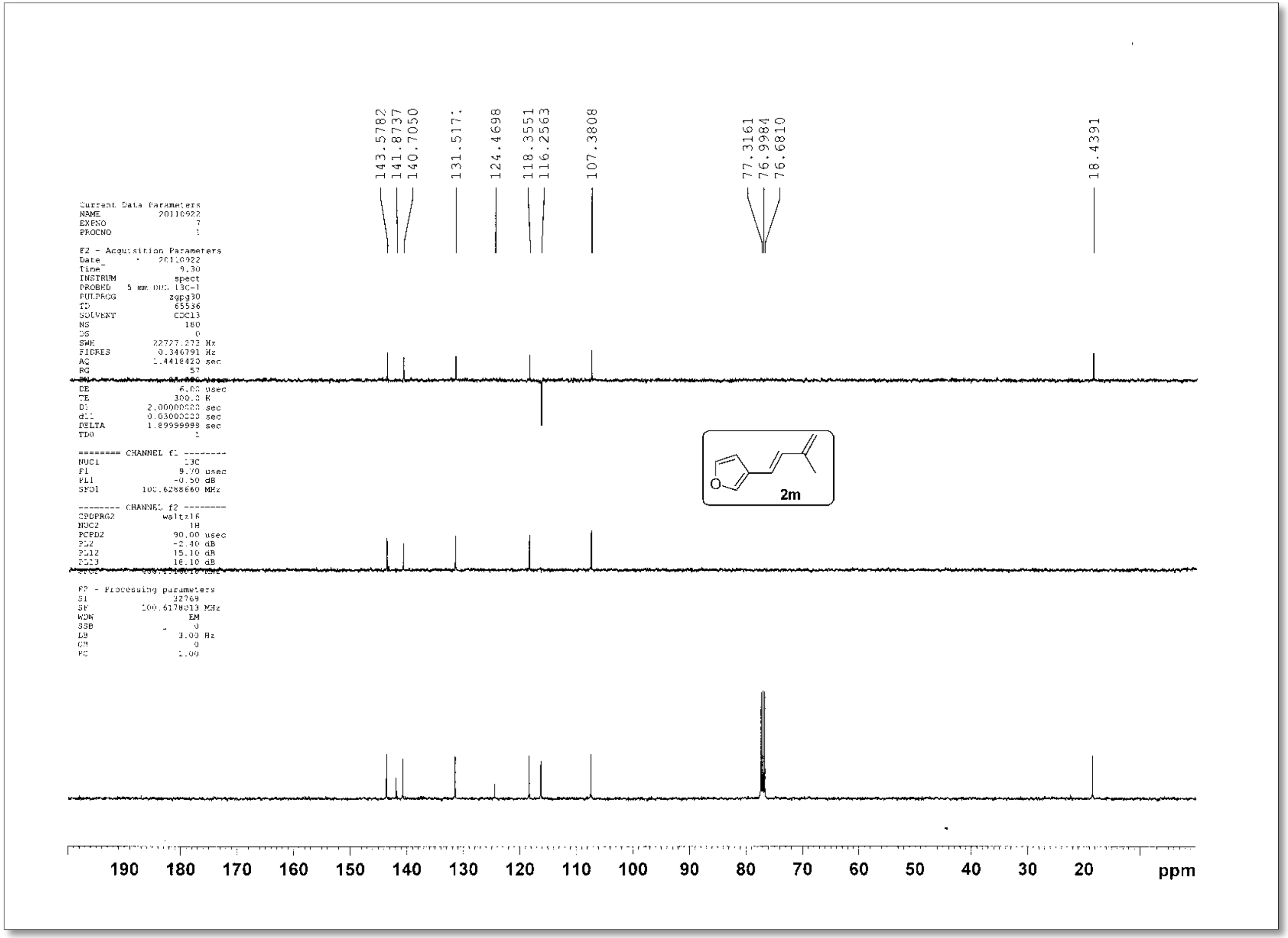
```
Current Data Parameters
NAME             20110813
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110813
Time                13.05
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     34
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    724
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



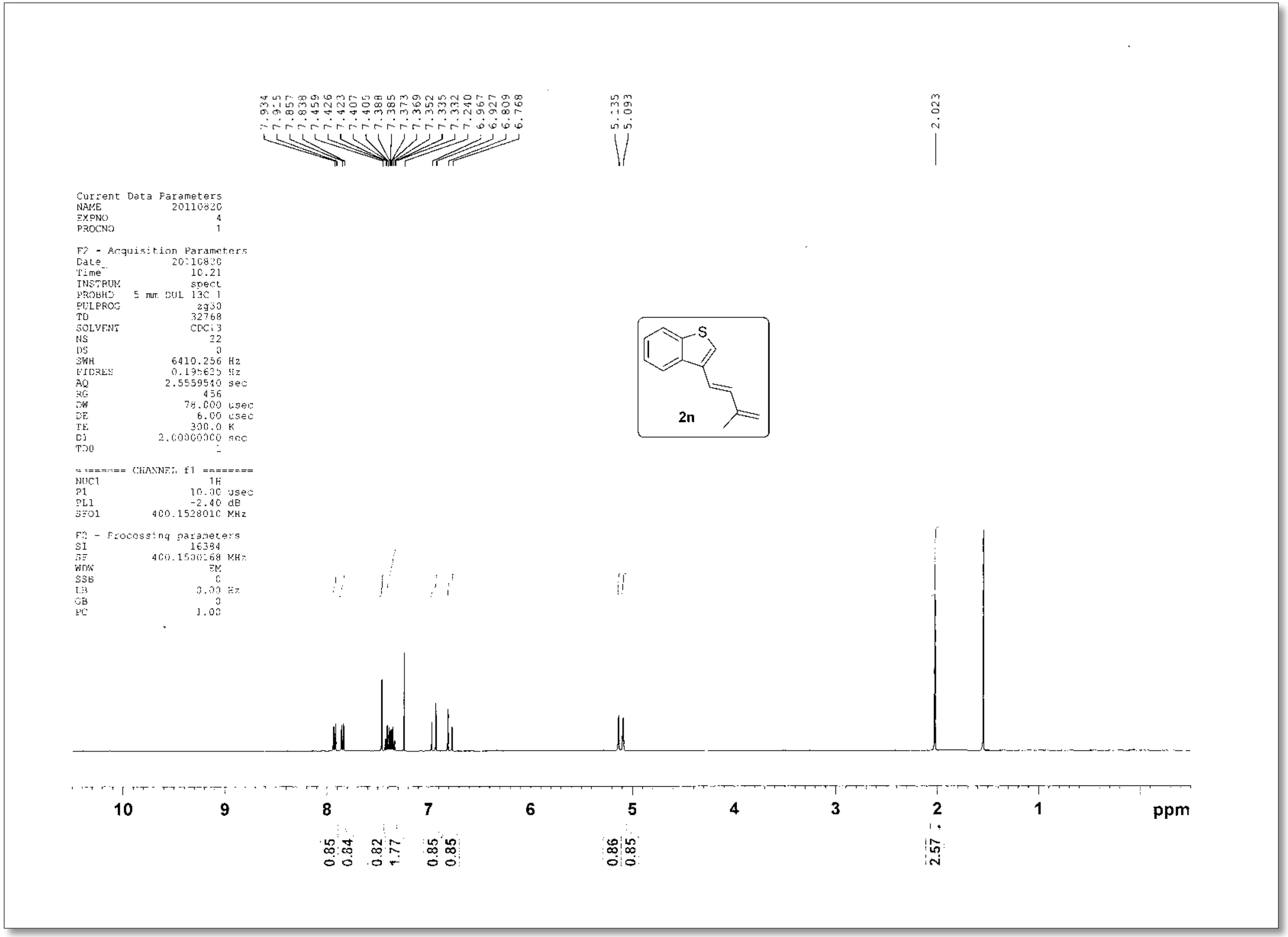
```
Current Data Parameters
NAME             20110813
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date             20110813
Time                12.38
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    156
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                   90.6
DW                 22.000 usec
DE                   6.00 usec
TE
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6268660 MHz

-------- CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178027 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



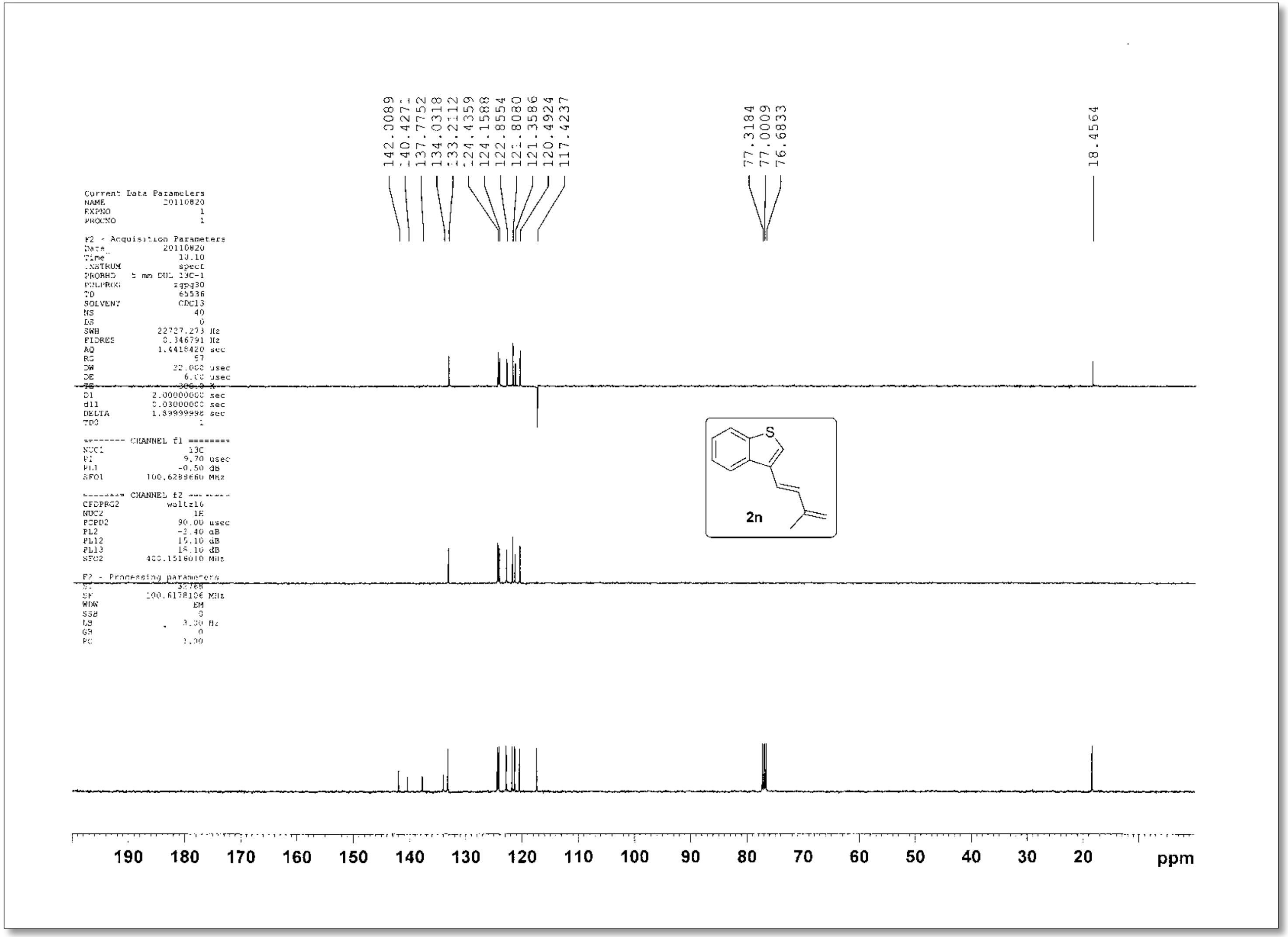
```
Current Data Parameters
NAME              20110922
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20110921
Time                  9.24
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ               2.5559540 sec
RG                     645
DW                  78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1           400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF             400.1500168 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

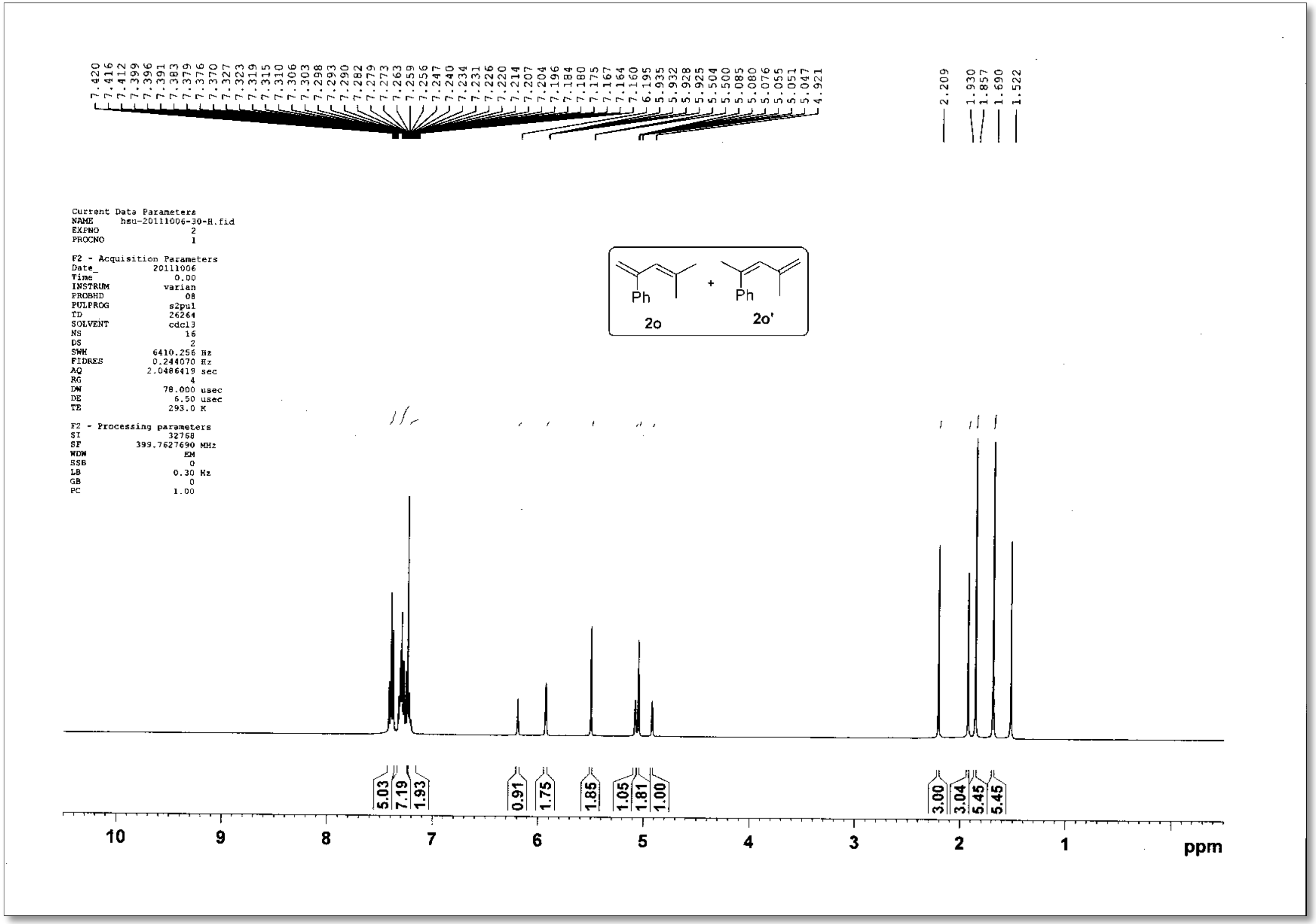


```
Current Data Parameters
NAME             20110922
EXPNO                   7
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110922
Time                 9.30
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   180
DS                     0
SWH         22727.273 Hz
FIDRES       0.346791 Hz
AQ          1.4418420 sec
RG                    57
DE               6.00 usec
TE              300.0 K
D1         2.00000000 sec
d11        0.03000000 sec
DELTA      1.89999998 sec
TD0                    1

======== CHANNEL f1 --------
NUC1                 13C
P1               9.70 usec
PL1             -0.50 dB
SFO1      100.6288660 MHz

-------- CHANNEL f2 --------
CPDPRG2         waltz16
NUC2                  1H
PCPD2           90.00 usec
PL2             -2.40 dB
PL12            15.10 dB
PL13            18.10 dB

F2 - Processing parameters
SI                 32768
SF        100.6178013 MHz
WDW                   EM
SSB                    0
LB               3.00 Hz
GB                     0
PC                  1.00
```

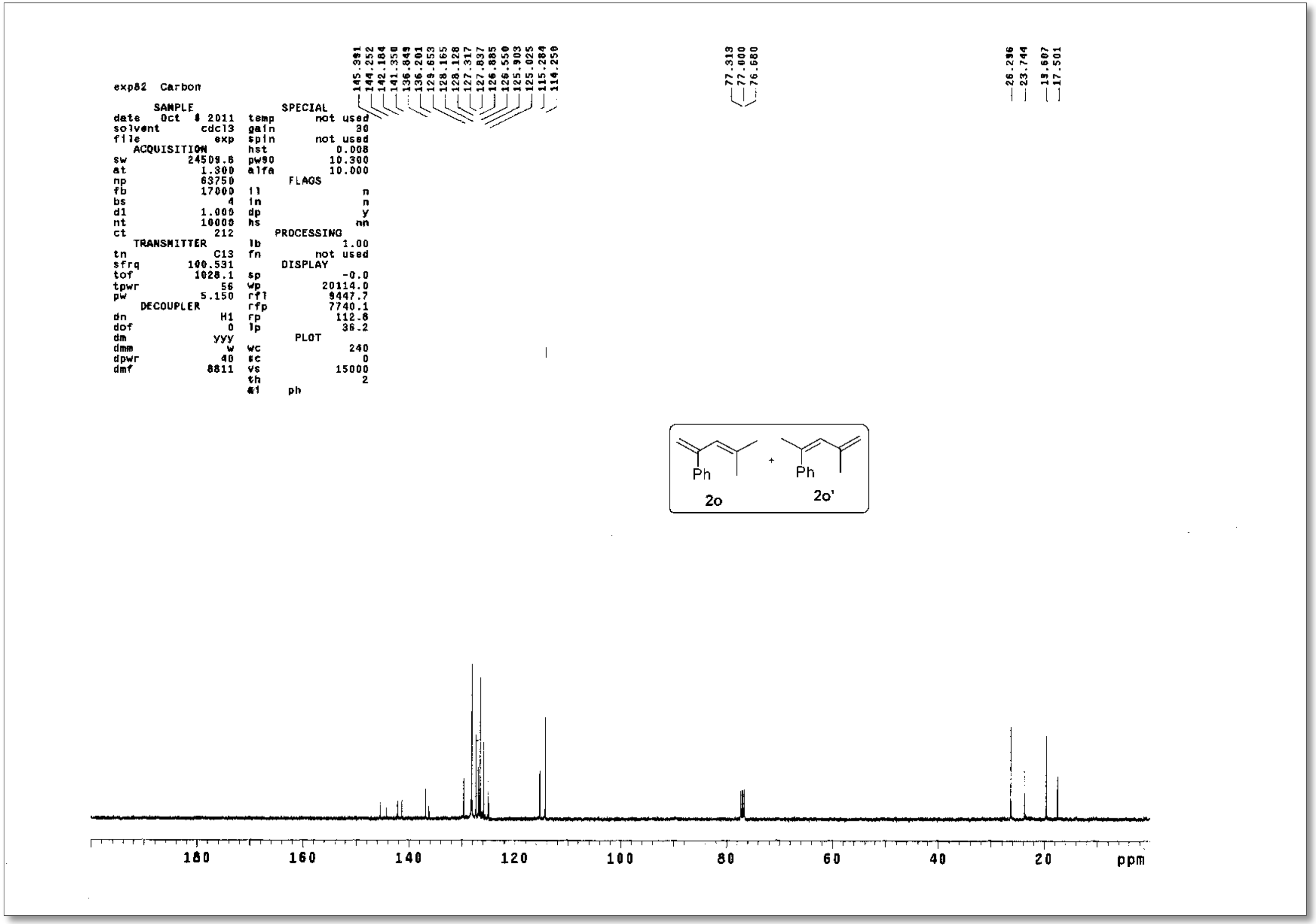


```
Current Data Parameters
NAME            20110820
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110820
Time               10.21
INSTRUM            spect
PROBHD   5 mm DUL 13C 1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    22
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   456
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500168 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

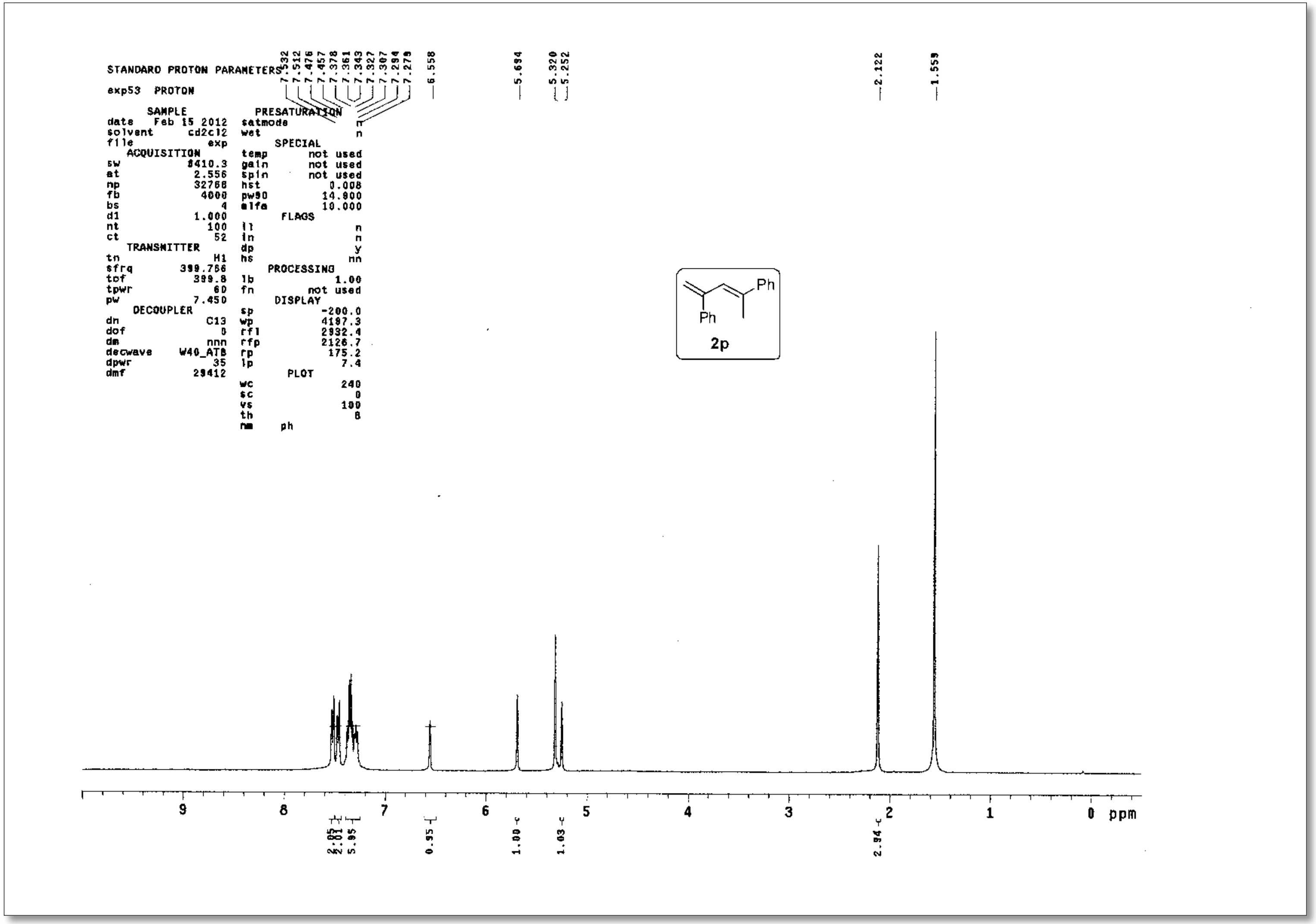

142.3089
140.427
137.7752
134.0318
133.2112
124.4359
124.1588
122.8554
121.8080
121.3586
120.4924
117.4237
77.3184
77.0009
76.6833
18.4564
Current Data Parameters
NAME 20110820
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110820
Time 10.10
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 40
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 15.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178106 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
S
2n
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



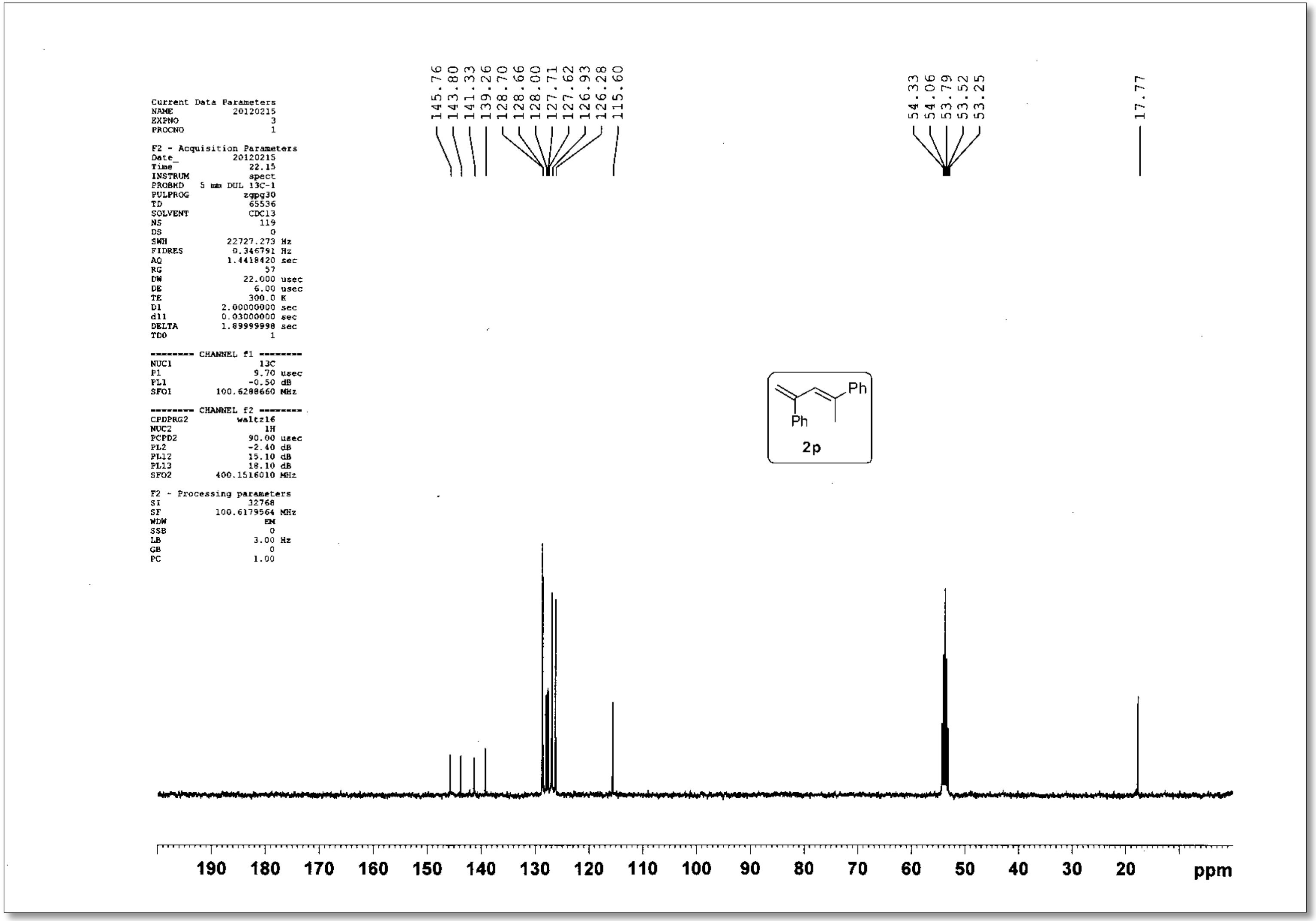
```
Current Data Parameters
NAME      hsu-20111006-30-H.fid
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20111006
Time                   0.00
INSTRUM              varian
PROBHD                   08
PULPROG               s2pul
TD                    26264
SOLVENT               cdcl3
NS                       16
DS                        2
SWH                6410.256 Hz
FIDRES             0.244070 Hz
AQ                2.0486419 sec
RG                        4
DW                   78.000 usec
DE                     6.50 usec
TE                    293.0 K

F2 - Processing parameters
SI                    32768
SF              399.7627690 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



```
exp82  Carbon

      SAMPLE                  SPECIAL
date   Oct  8 2011   temp        not used
solvent       cdcl3  gain              30
file            exp  spin        not used
   ACQUISITION       hst            0.008
sw          24509.8  pw90          10.300
at            1.300  alfa          10.000
np            63750          FLAGS
fb            17000  il                 n
bs                4  in                 n
d1            1.000  dp                 y
nt            10000  hs                nn
ct              212        PROCESSING
   TRANSMITTER       lb              1.00
tn              C13  fn          not used
sfrq        100.531          DISPLAY
tof          1028.1  sp              -0.0
tpwr             56  wp           20114.0
pw            5.150  rfl           9447.7
    DECOUPLER        rfp           7740.1
dn               H1  rp             112.8
dof               0  lp              36.2
dm              yyy            PLOT
dmm               w  wc               240
dpwr             40  sc                 0
dmf            8811  vs             15000
                     th                 2
                     ai     ph
```

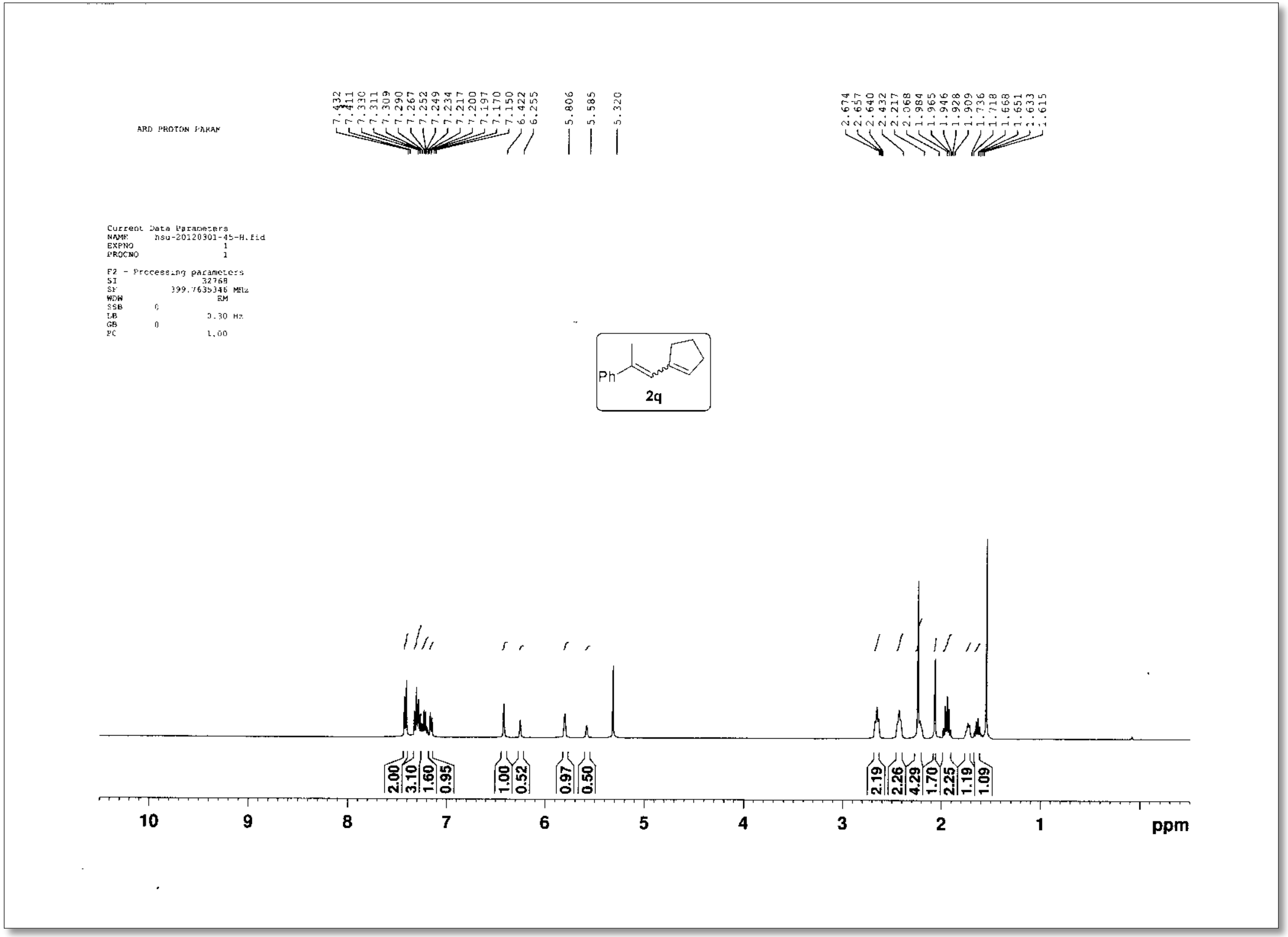


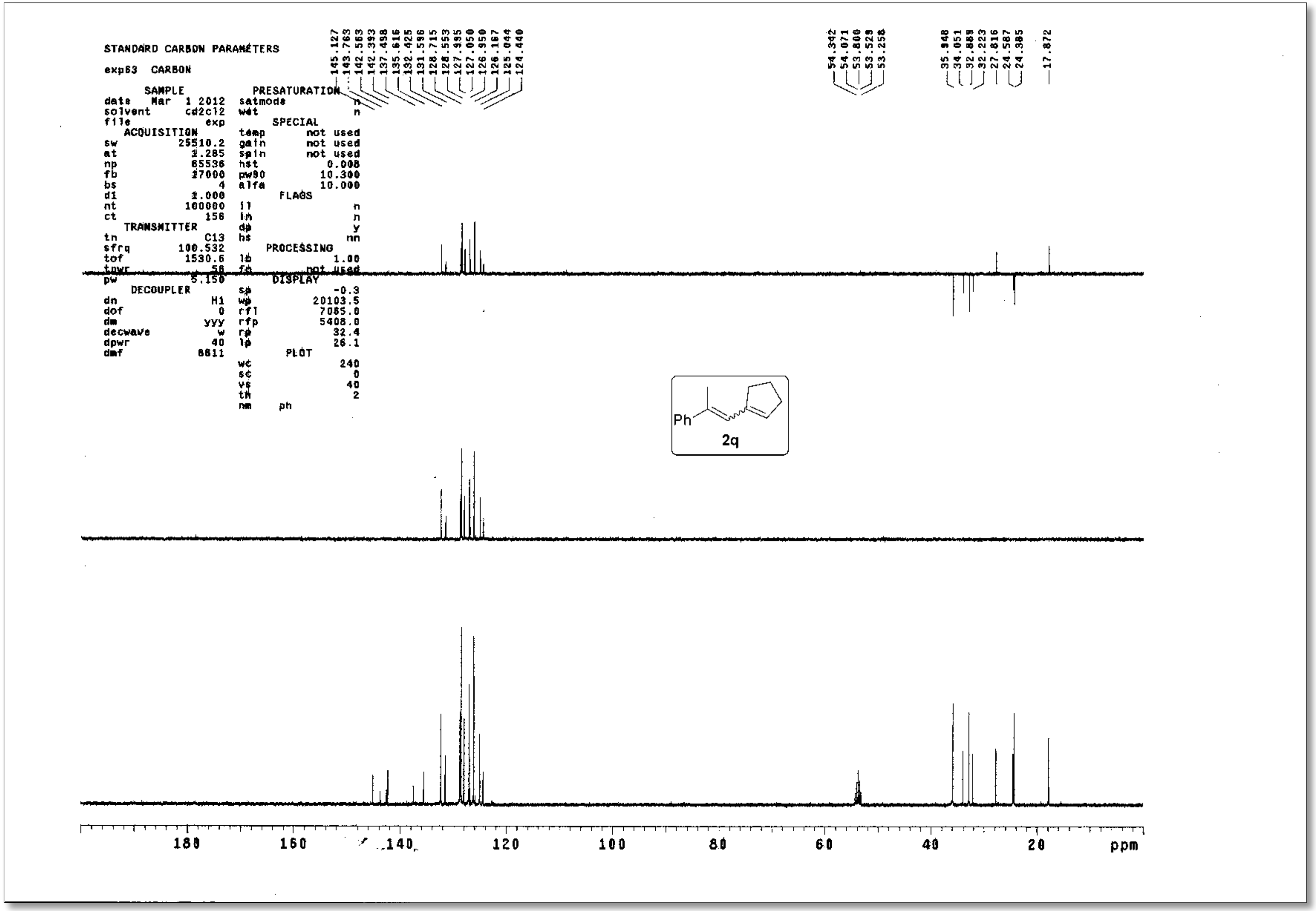
```
STANDARD PROTON PARAMETERS

exp53  PROTON

      SAMPLE               PRESATURATION
date    Feb 15 2012   satmode            n
solvent      cd2cl2   wet                n
file            exp         SPECIAL
   ACQUISITION        temp        not used
sw           6410.3   gain        not used
at            2.556   spin        not used
np            32768   hst            0.008
fb             4000   pw90          14.900
bs                4   alfa          10.000
d1            1.000          FLAGS
nt              100   il                 n
ct               52   in                 n
   TRANSMITTER        dp                 y
tn               H1   hs                nn
sfrq        399.766        PROCESSING
tof           399.8   lb              1.00
tpwr             60   fn          not used
pw            7.450         DISPLAY
    DECOUPLER         sp            -200.0
dn              C13   wp            4197.3
dof               0   rfl           2932.4
dm              nnn   rfp           2126.7
decwave     W40_ATB   rp             175.2
dpwr             35   lp               7.4
dmf           29412           PLOT
                      wc               240
                      sc                 0
                      vs               100
                      th                 8
                      nm      ph
```



```
Current Data Parameters
NAME             20120215
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120215
Time                22.15
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   119
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6179564 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```

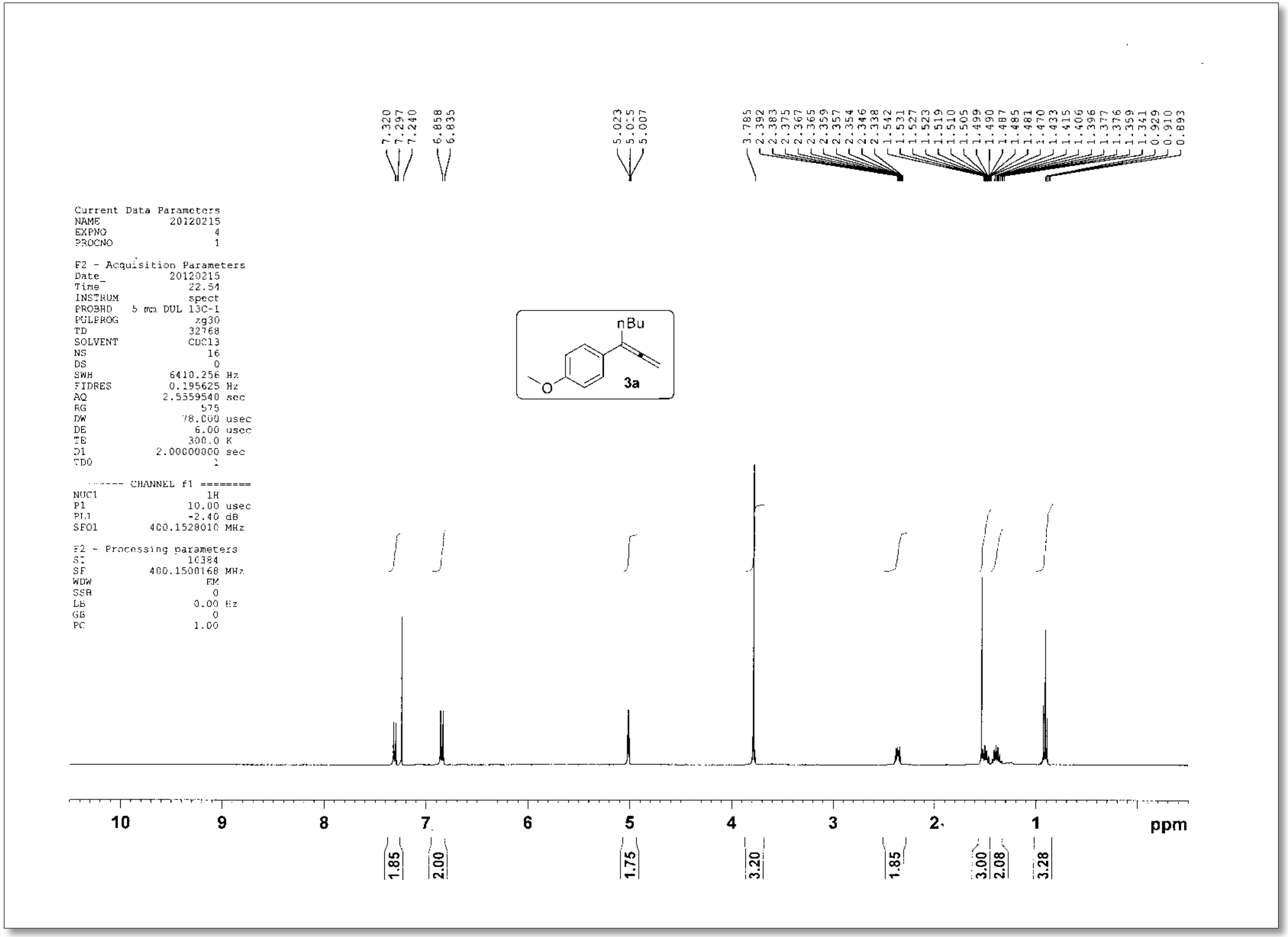
2p


ARD PROTON PARAM
7.432
7.411
7.330
7.311
7.309
7.290
7.267
7.252
7.249
7.234
7.217
7.200
7.197
7.170
7.150
6.422
6.255
5.806
5.585
5.320
2.674
2.657
2.640
2.432
2.217
2.068
1.984
1.965
1.946
1.928
1.909
1.736
1.718
1.668
1.651
1.633
1.615
Current Data Parameters
NAME hsu-20120301-45-H.fid
EXPNO 1
PROCNO 1
F2 - Processing parameters
SI 32768
SF 399.7635346 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Ph
2q
2.00
3.10
1.60
0.95
1.00
0.52
0.97
0.50
2.19
2.26
4.29
1.70
2.25
1.19
1.09
10
9
8
7
6
5
4
3
2
1
ppm

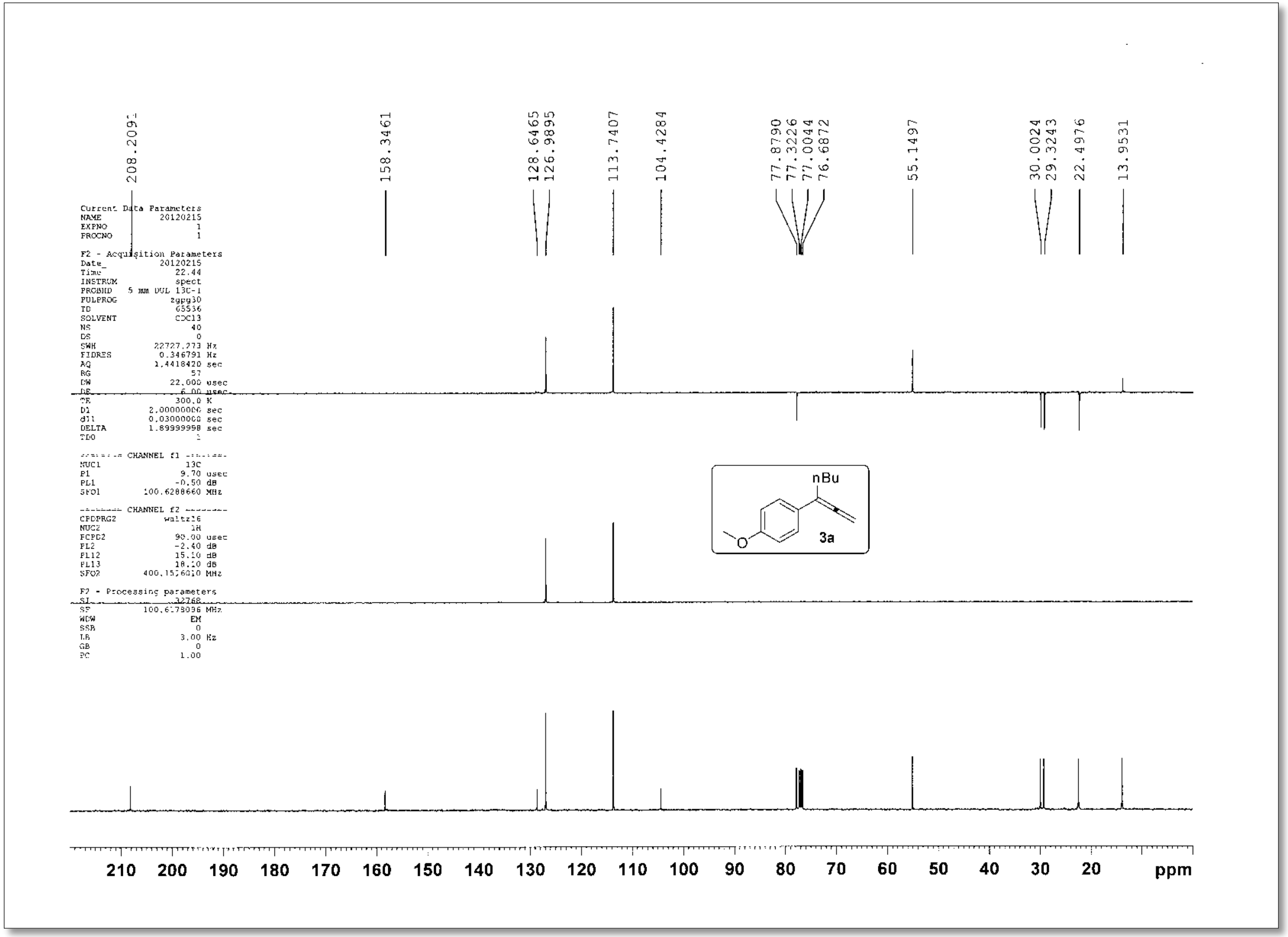


```
STANDARD CARBON PARAMETERS

exp63  CARBON

      SAMPLE              PRESATURATION
date   Mar  1 2012   satmode              n
solvent     cd2cl2   wet                  n
file           exp           SPECIAL
   ACQUISITION       temp          not used
sw         25510.2   gain          not used
at           1.285   spin          not used
np           65536   hst              0.008
fb           17000   pw90            10.300
bs               4   alfa            10.000
d1           1.000           FLAGS
nt          100000   il                   n
ct             156   in                   n
   TRANSMITTER       dp                   y
tn             C13   hs                  nn
sfrq       100.532         PROCESSING
tof         1530.6   lb                1.00
tpwr            56   fn            not used
pw           5.150           DISPLAY
    DECOUPLER        sp                -0.3
dn              H1   wp             20103.5
dof              0   rfl             7085.0
dm             yyy   rfp             5408.0
decwave          w   rp                32.4
dpwr            40   lp                26.1
dmf           6611            PLOT
                     wc                 240
                     sc                   0
                     vs                  40
                     th                   2
                     nm       ph
```

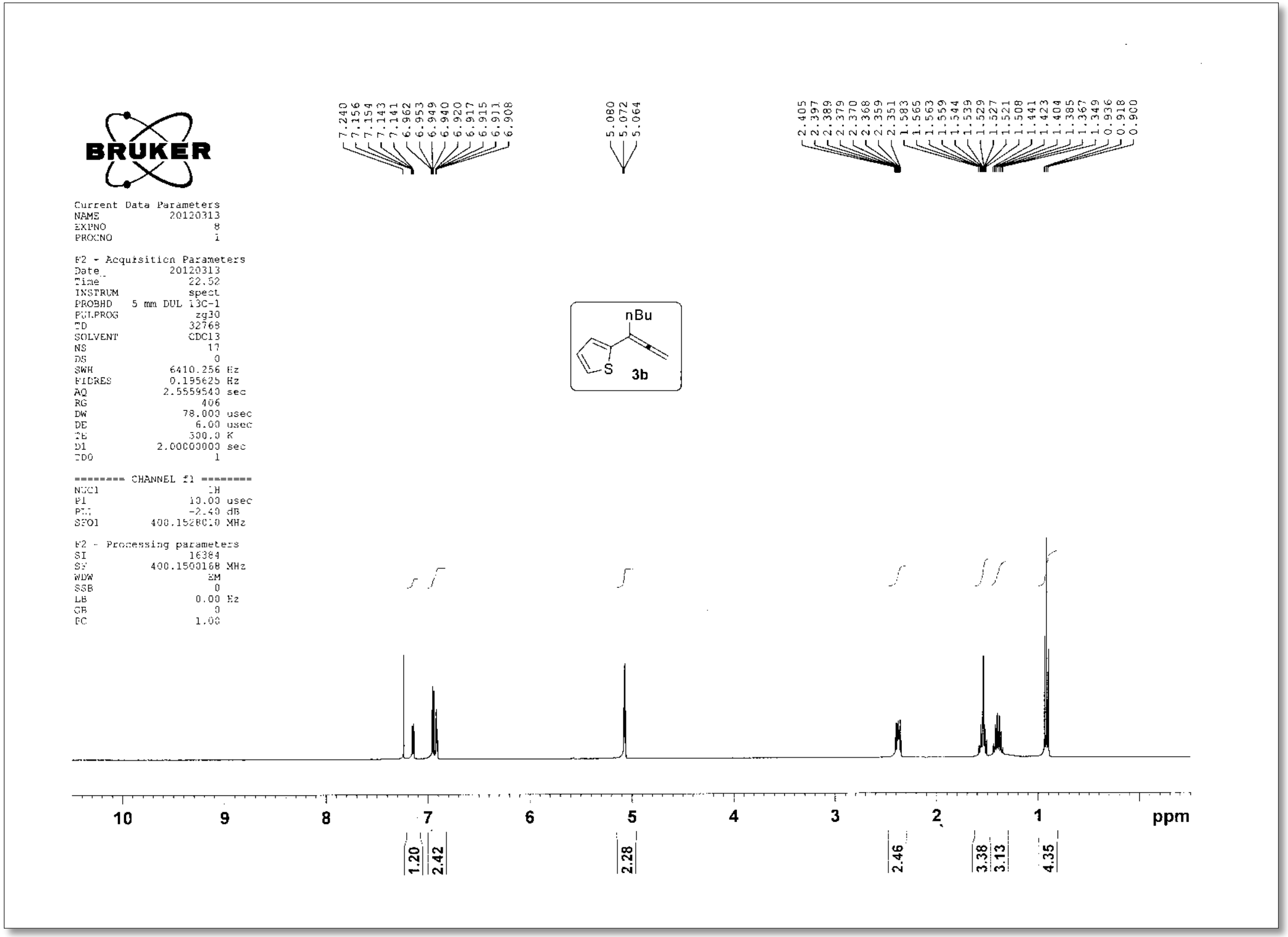
2q



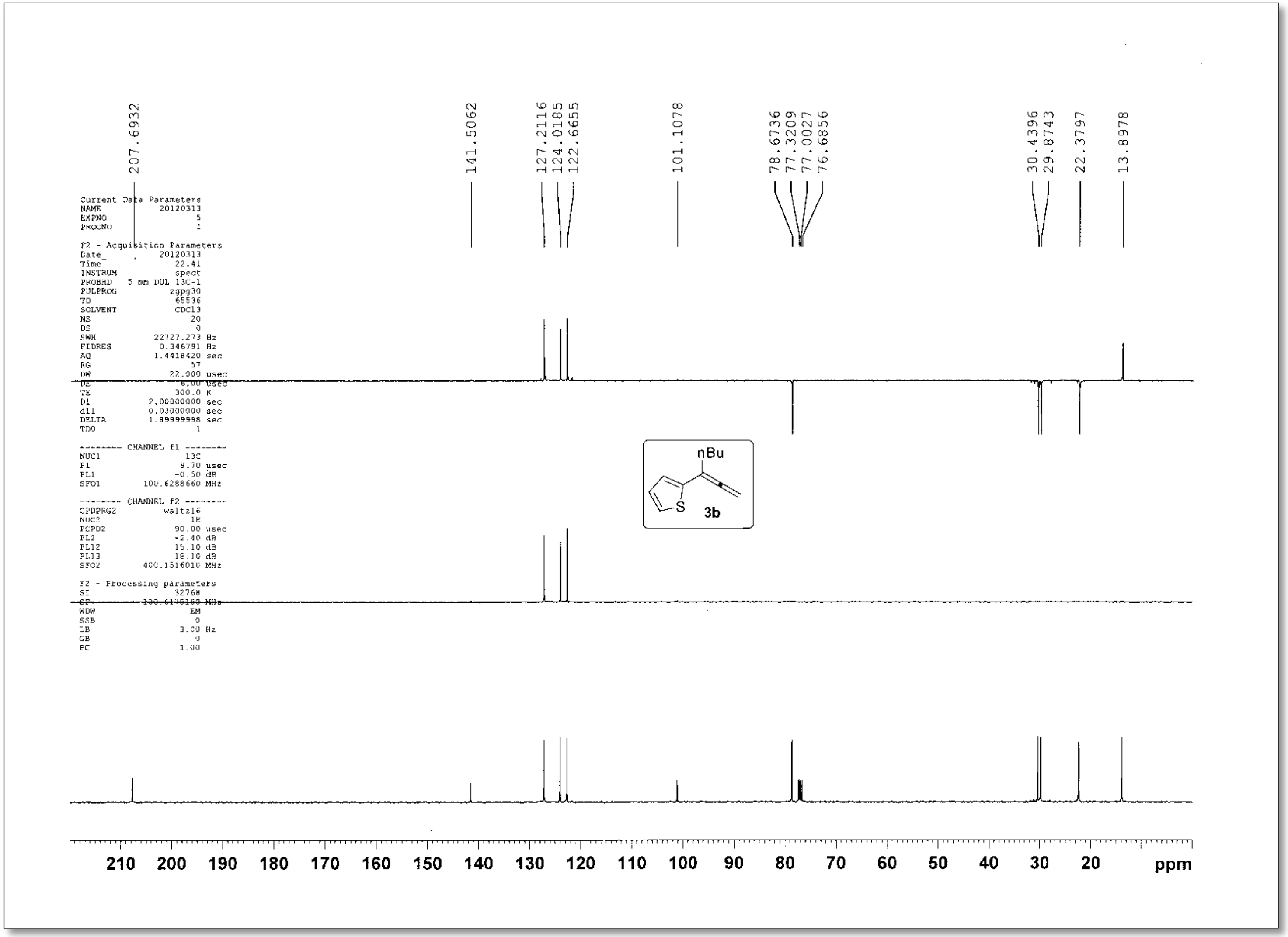
```
Current Data Parameters
NAME             20120215
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120215
Time                22.54
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    575
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



```
Current Data Parameters
NAME              20120215
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20120215
Time                 22.44
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      40
DS                       0
SWH              22727.273 Hz
FIDRES            0.346791 Hz
AQ               1.4418420 sec
RG                      57
DW                  22.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                  -0.50 dB
SFO1           100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -2.40 dB
PL12                 15.10 dB
PL13                 18.10 dB
SFO2           400.1576010 MHz

F2 - Processing parameters
SI                   32768
SF             100.6178036 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```

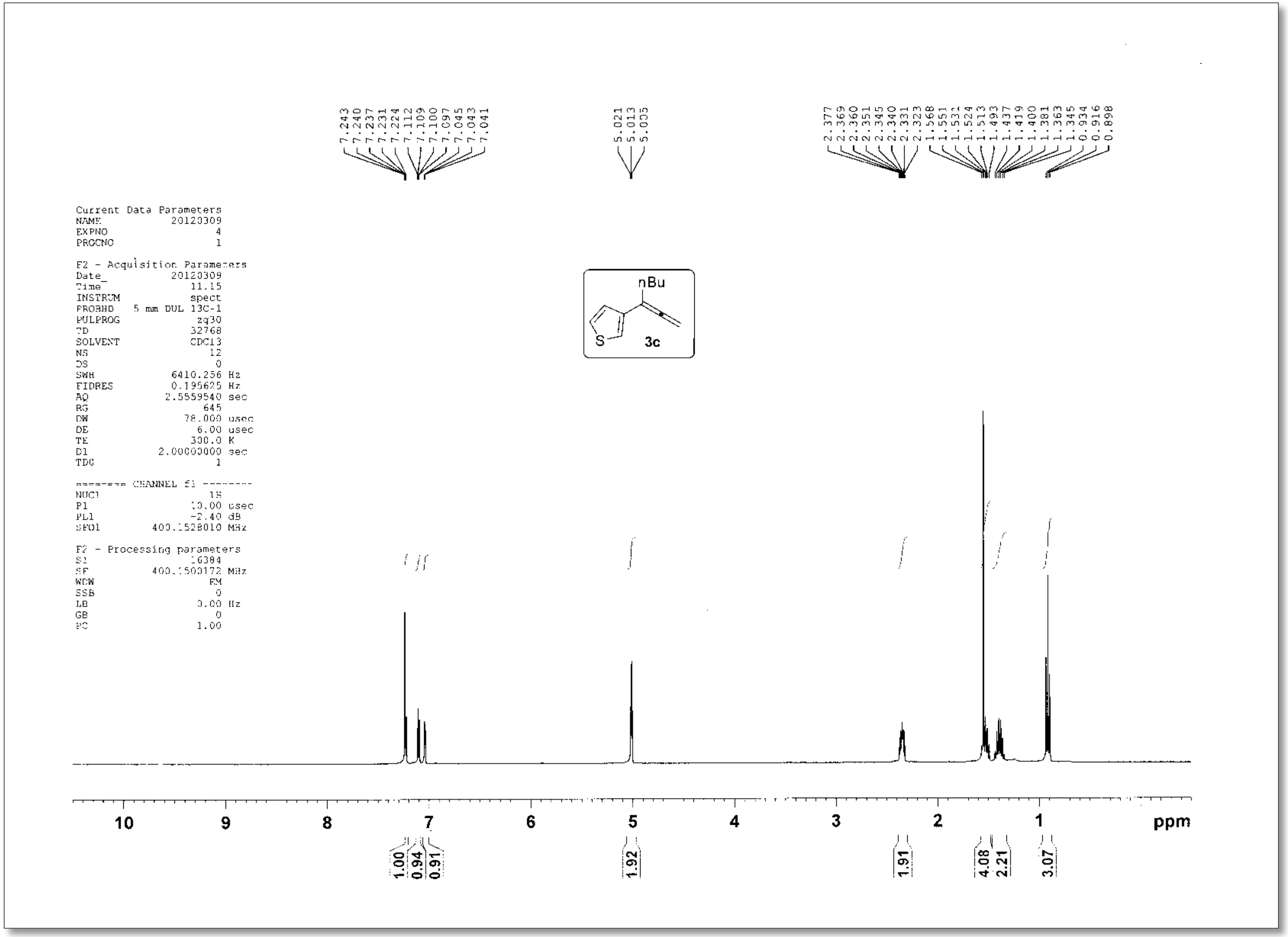


```
Current Data Parameters
NAME              20120313
EXPNO                    8
PROCNO                   1

F2 - Acquisition Parameters
Date_             20120313
Time                 22.52
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      17
DS                       0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ               2.5559540 sec
RG                     406
DW                  78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1           400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF             400.1500168 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```

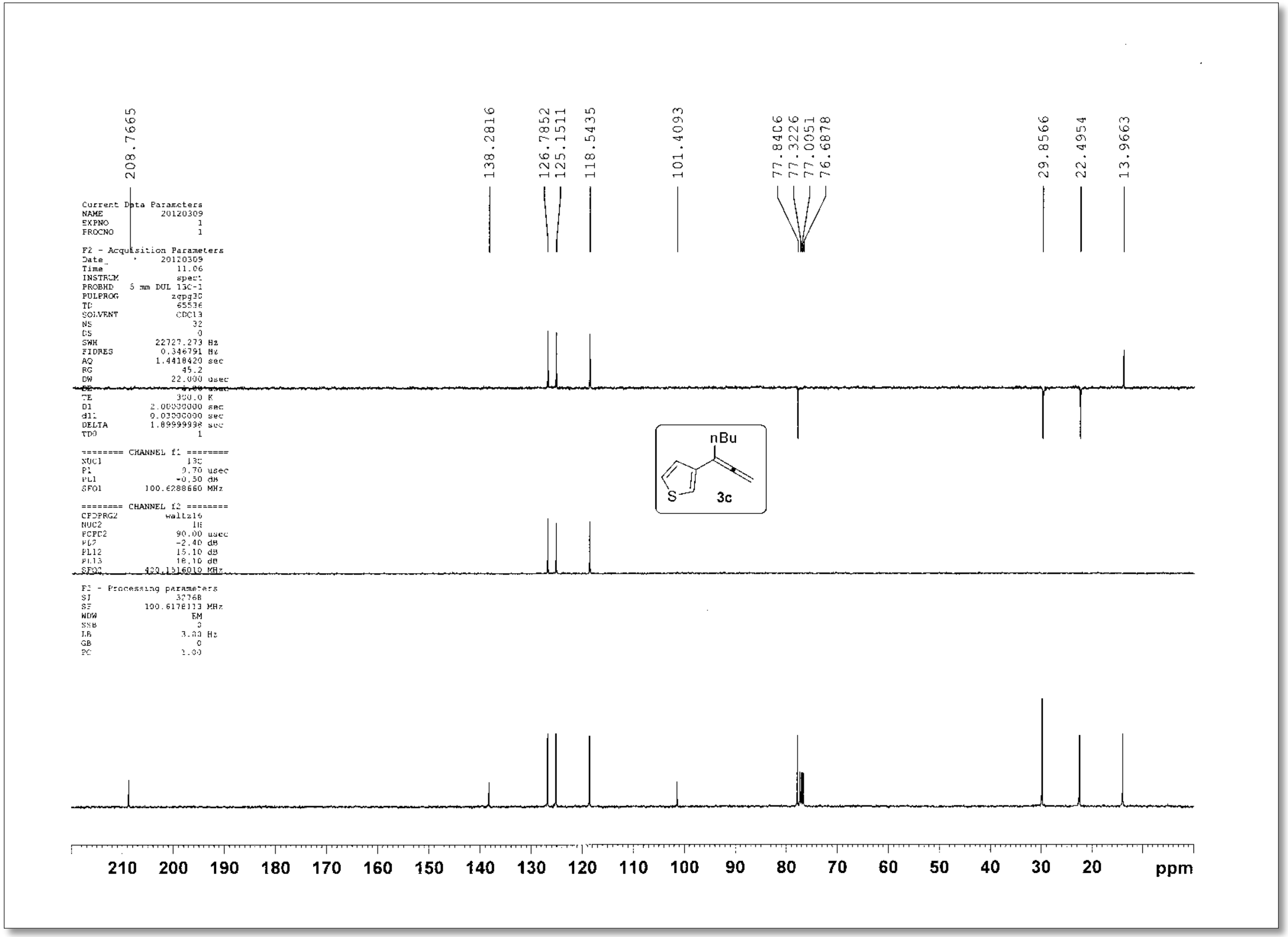


3b

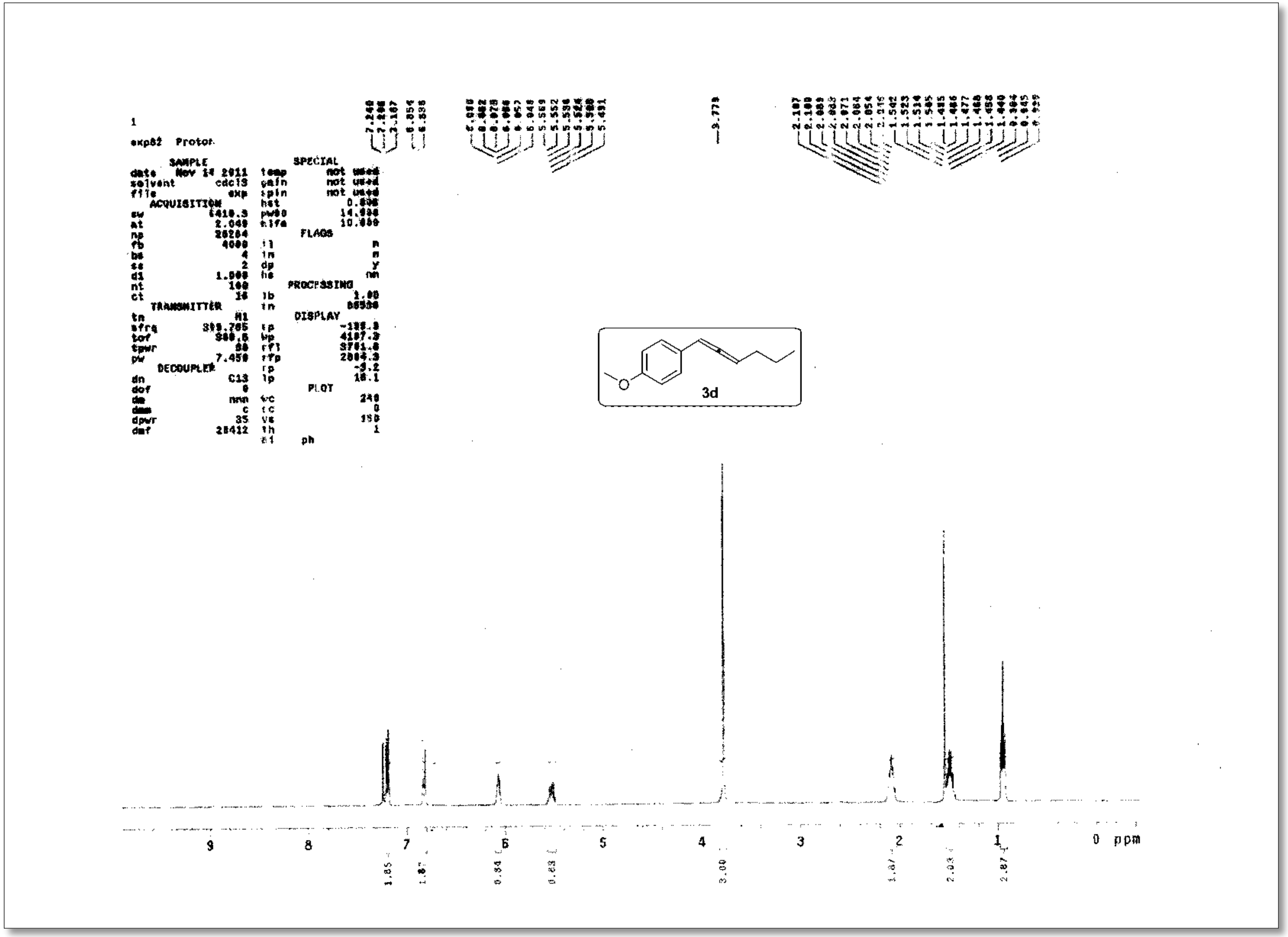
```
Current Data Parameters
NAME             20120313
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120313
Time                22.41
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     20
DS                      0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF           [illegible] MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



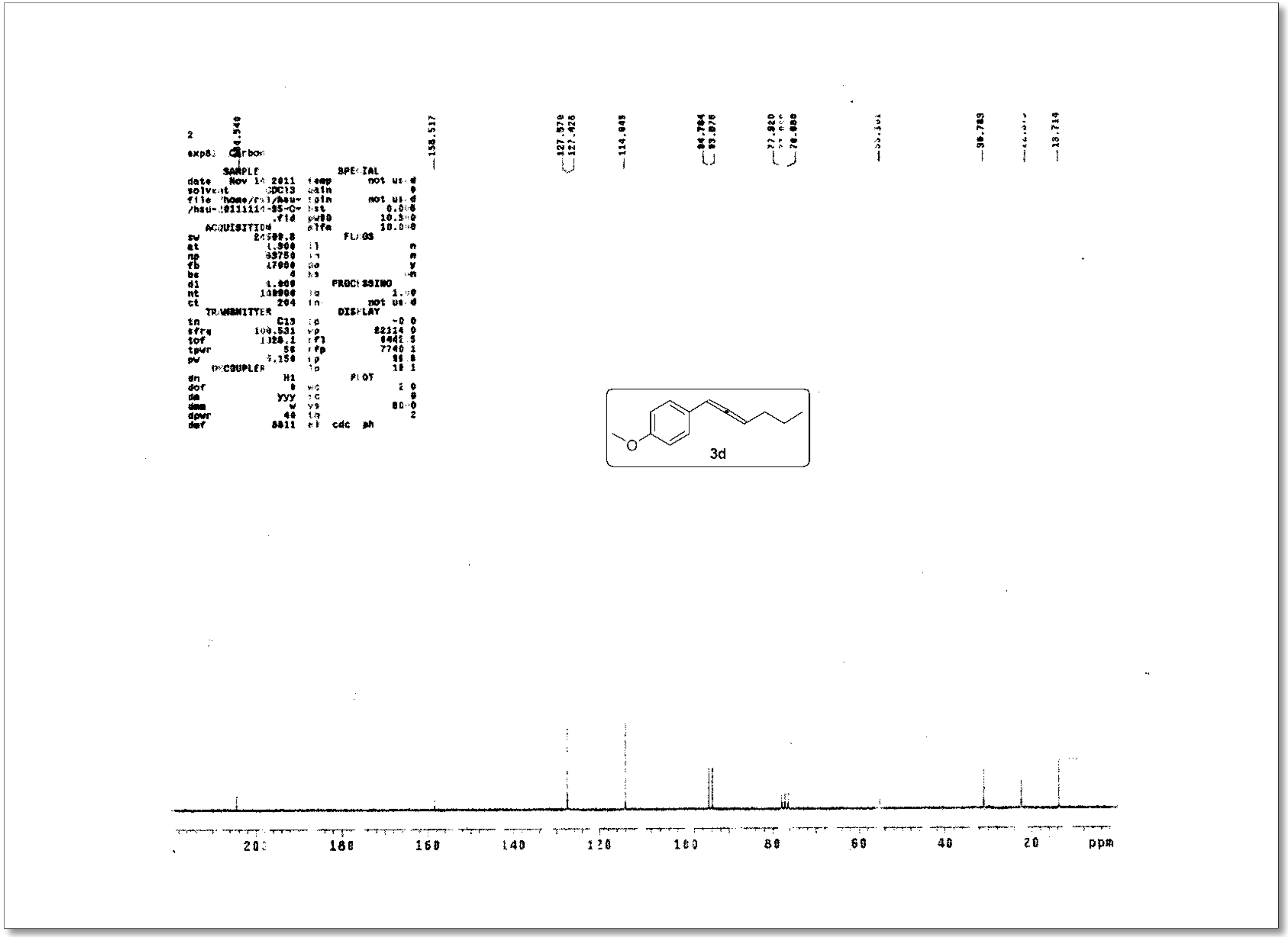
```
Current Data Parameters
NAME          20120309
EXPNO                4
PROCNO               1

F2 - Acquisition Parameters
Date_         20120309
Time             11.15
INSTRUM          spect
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD               32768
SOLVENT          CDCl3
NS                  12
DS                   0
SWH           6410.256 Hz
FIDRES        0.195625 Hz
AQ           2.5559540 sec
RG                 645
DW              78.000 usec
DE                6.00 usec
TE               300.0 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               10.00 usec
PL1              -2.40 dB
SFO1       400.1528010 MHz

F2 - Processing parameters
SI               16384
SF         400.1500172 MHz
WDW                 EM
SSB                  0
LB                0.00 Hz
GB                   0
PC                1.00
```



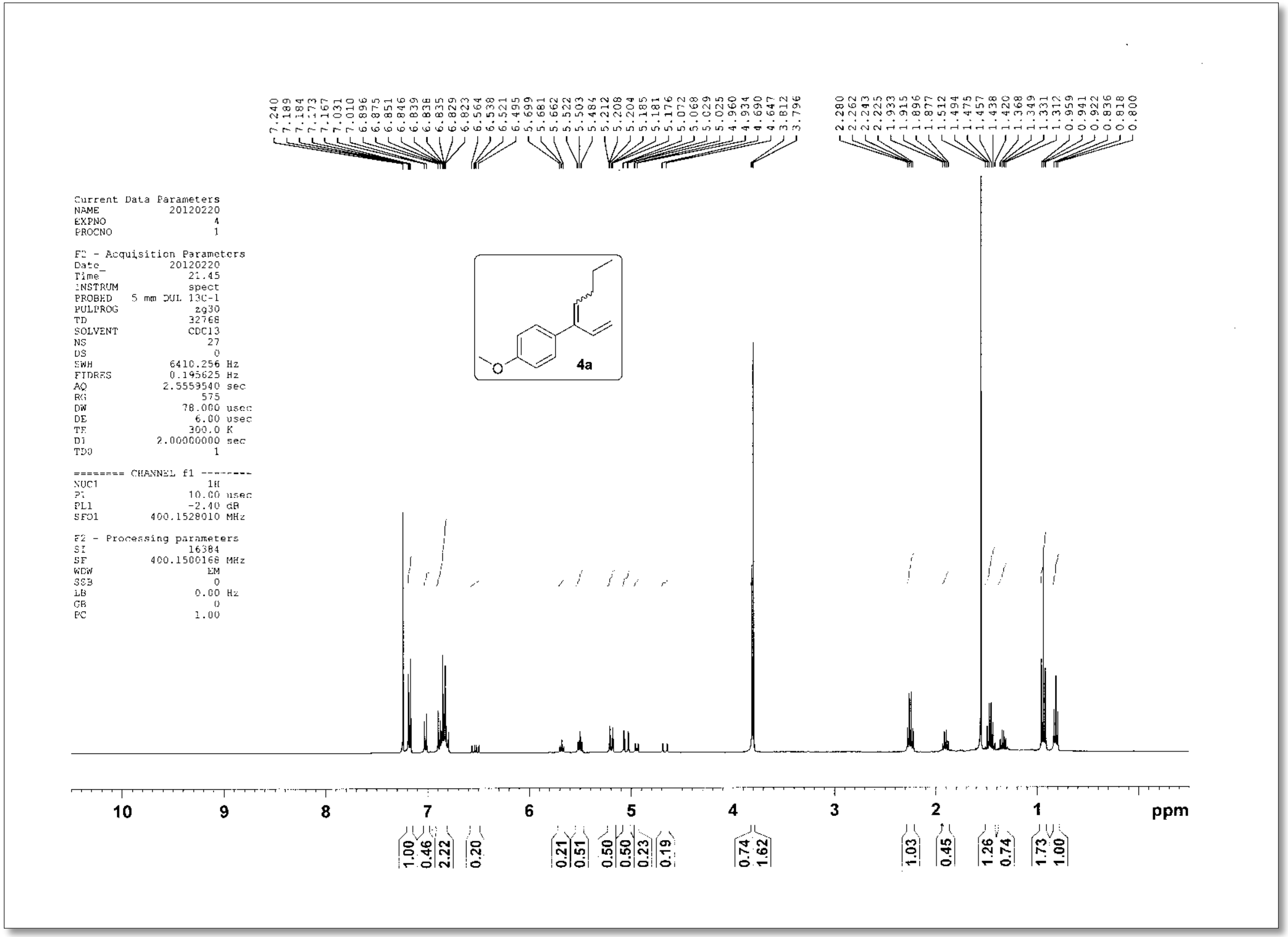
```
Current Data Parameters
NAME            20120309
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120309
Time               11.06
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT            CDCl3
NS                    32
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                  45.2
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178113 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



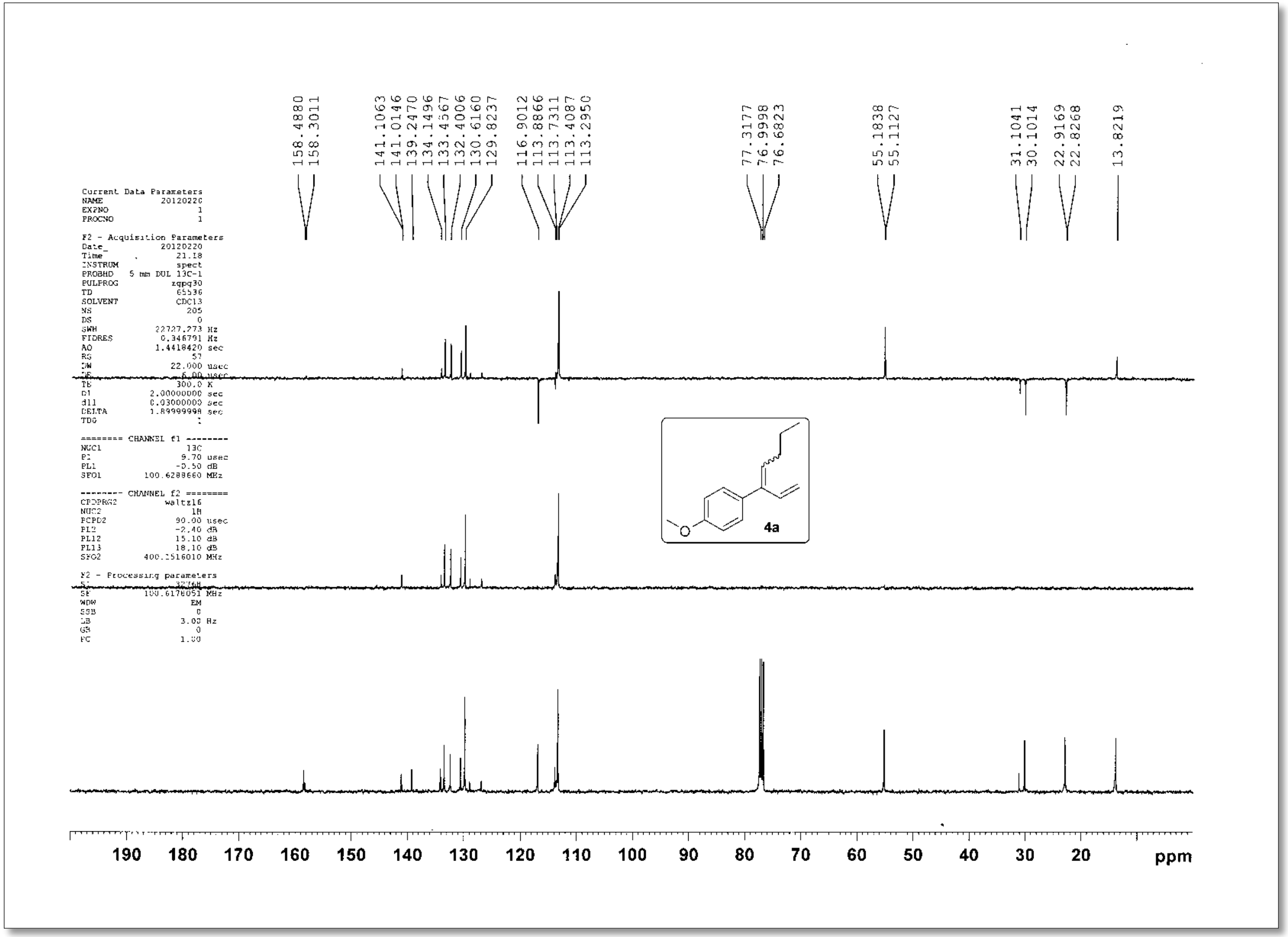
```
1

exp82   Proton

      SAMPLE                  SPECIAL
date    Nov 14 2011    temp        not used
solvent       cdcl3    gain        not used
file            exp    spin        not used
    ACQUISITION        hst            0.008
sw           6410.3    pw90          14.900
at            2.049    alfa          10.000
np            26264           FLAGS
fb             4000    il                 n
bs                4    in                 n
ss                2    dp                 y
d1            1.000    hs                nn
nt              100         PROCESSING
ct               16    lb              1.00
    TRANSMITTER        fn             65536
tn               H1           DISPLAY
sfrq        399.765    sp            -135.8
tof           500.6    wp            4107.3
tpwr             60    rfl           3701.6
pw            7.450    rfp           2904.3
     DECOUPLER         rp              -3.2
dn              C13    lp              16.1
dof               0           PLOT
dm              nnn    wc               240
dmm               c    sc                 0
dpwr             35    vs               150
dmf           28412    th                 1
                       ai     ph
```

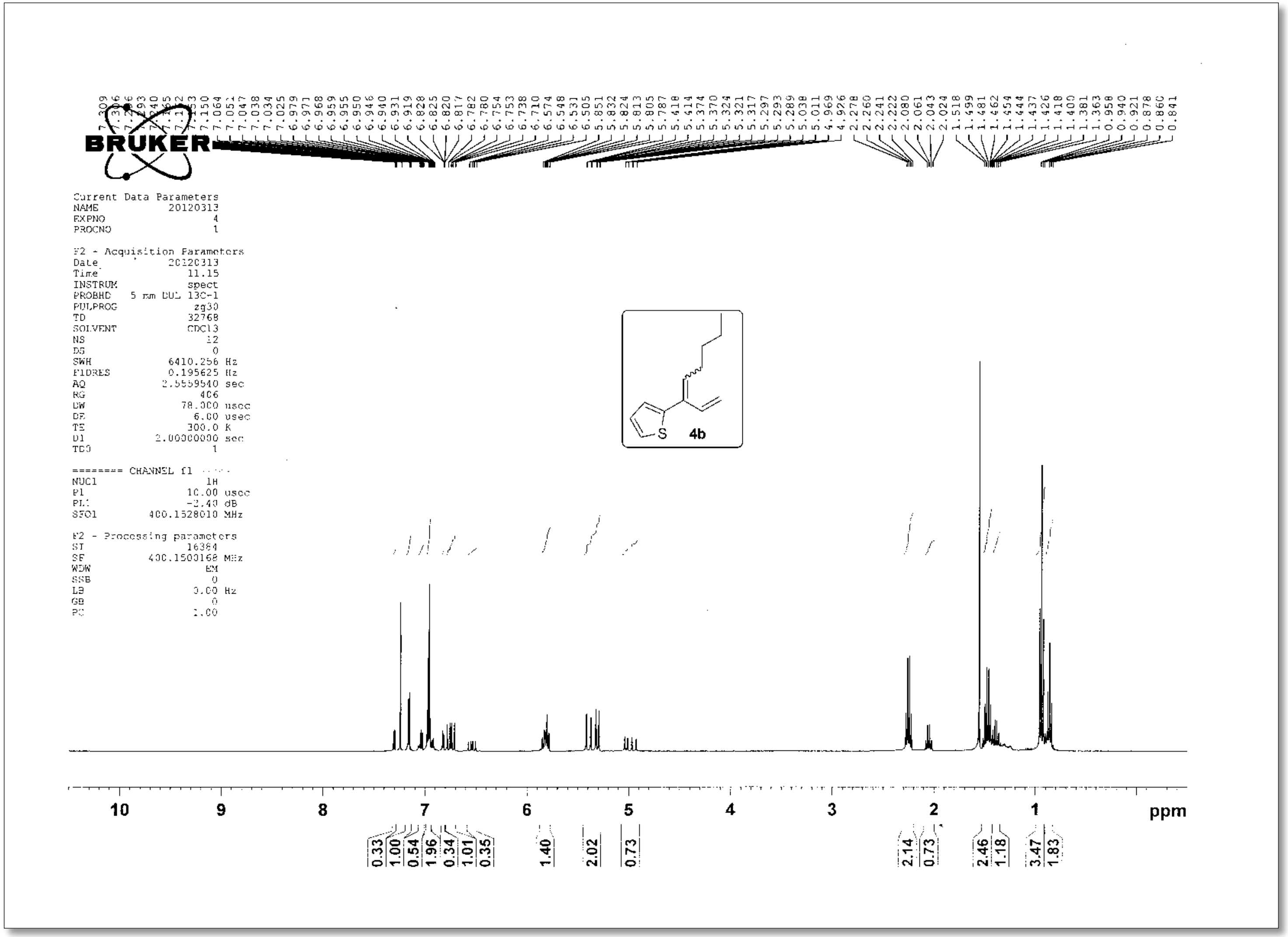

exp8 Carbon
SAMPLE
date Nov 14 2011
solvent CDCl3
ACQUISITION
TRANSMITTER
DECOUPLER
SPECIAL
FLAGS
PROCESSING
DISPLAY
PLOT
158.517
127.570
127.426
114.045
94.704
93.076
77.320
70.880
30.789
13.714
200
180
160
140
120
100
80
60
40
20
ppm

3d

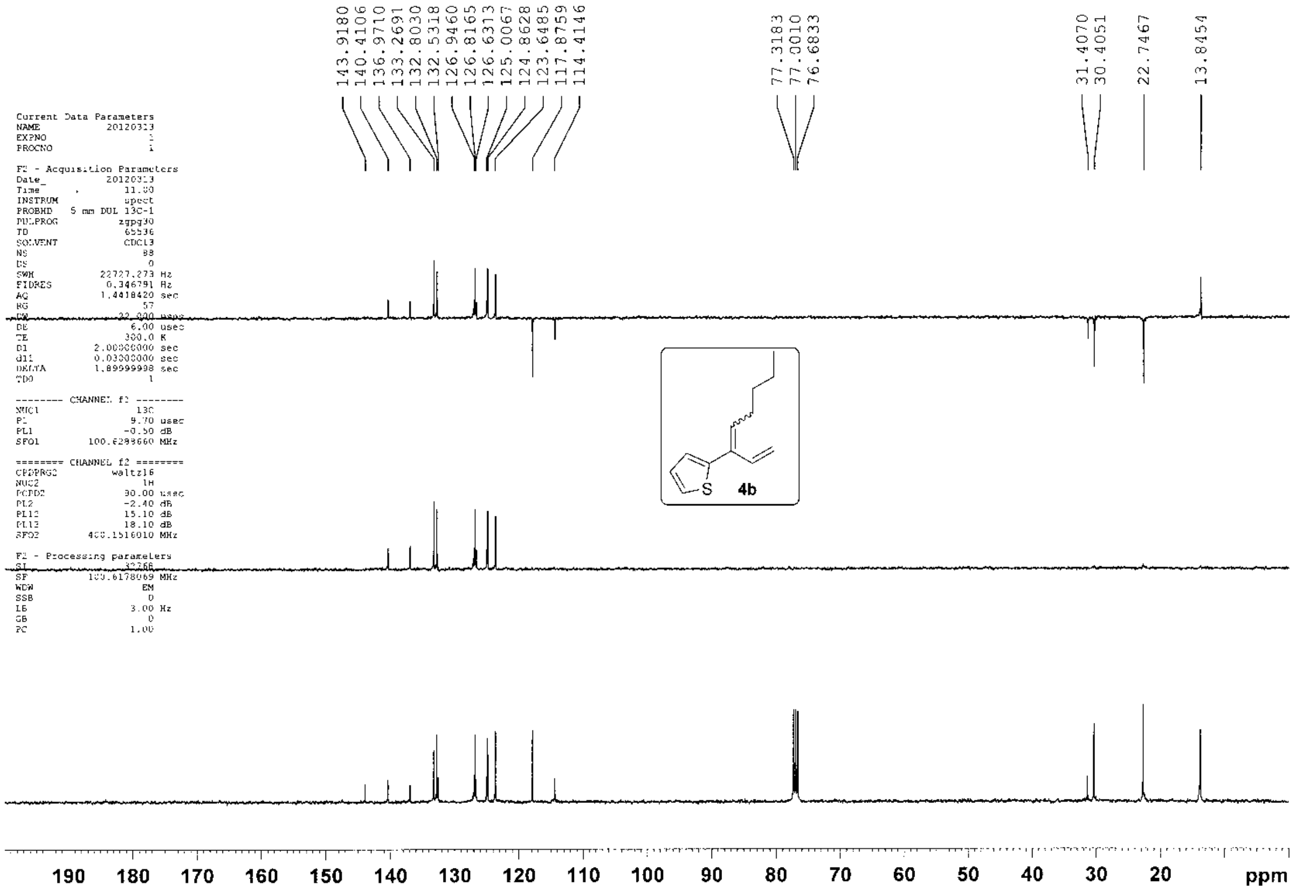


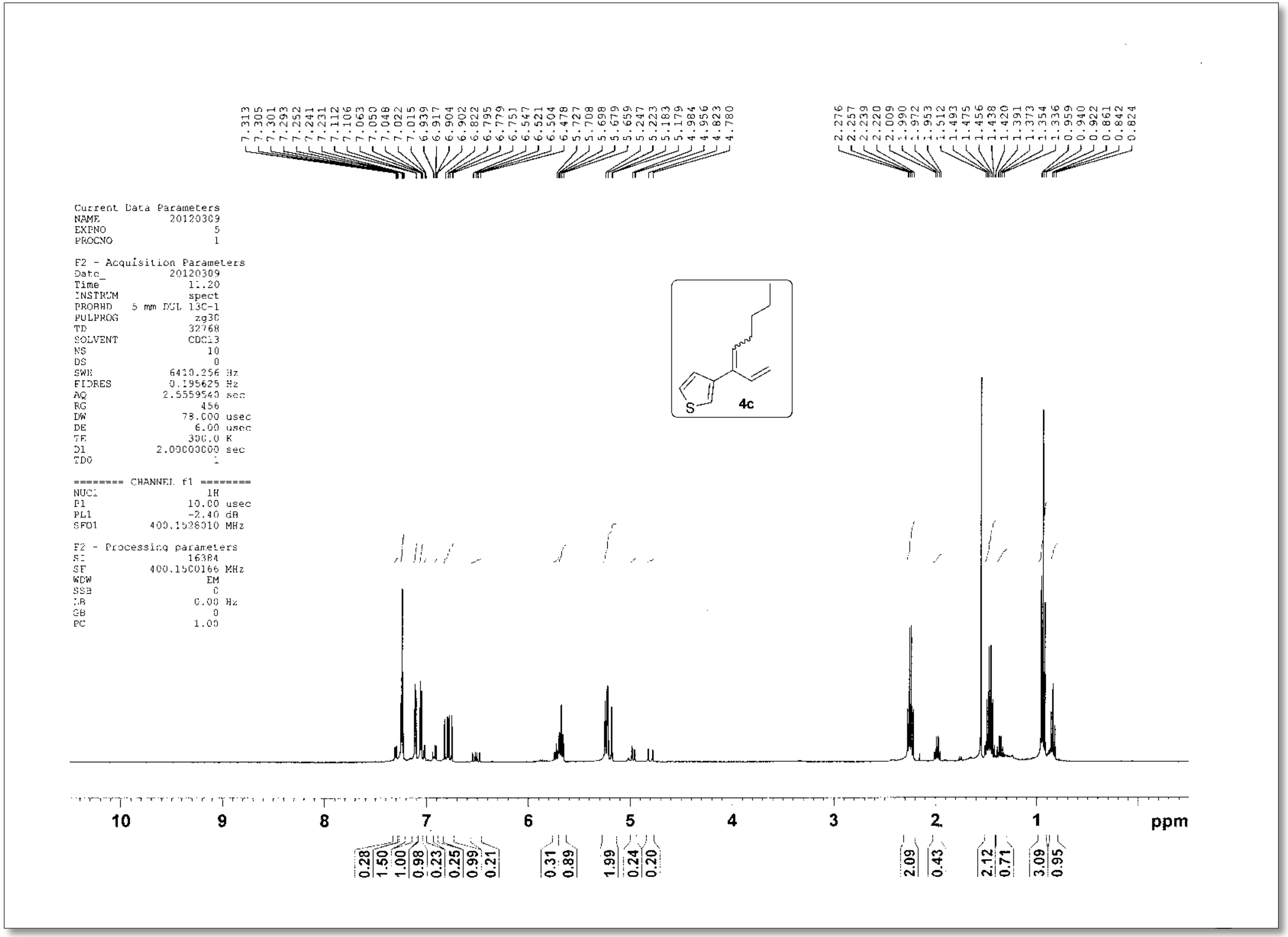
```
Current Data Parameters
NAME              20120220
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20120220
Time                 21.45
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      27
DS                       0
SWH               6410.256 Hz
FIDRES            0.195625 Hz
AQ               2.5559540 sec
RG                     575
DW                  78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
TD0                      1

======== CHANNEL f1 --------
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1           400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF             400.1500168 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```


158.4880
158.3011
141.1063
141.0146
139.2470
134.1496
133.4567
132.4006
130.6160
129.8237
116.9012
113.8866
113.7311
113.4087
113.2950
77.3177
76.9998
76.6823
55.1838
55.1127
31.1041
30.1014
22.9169
22.8268
13.8219
Current Data Parameters
NAME 20120220
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120220
Time 21.18
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 205
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 --------
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6288660 MHz
-------- CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.2516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178051 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

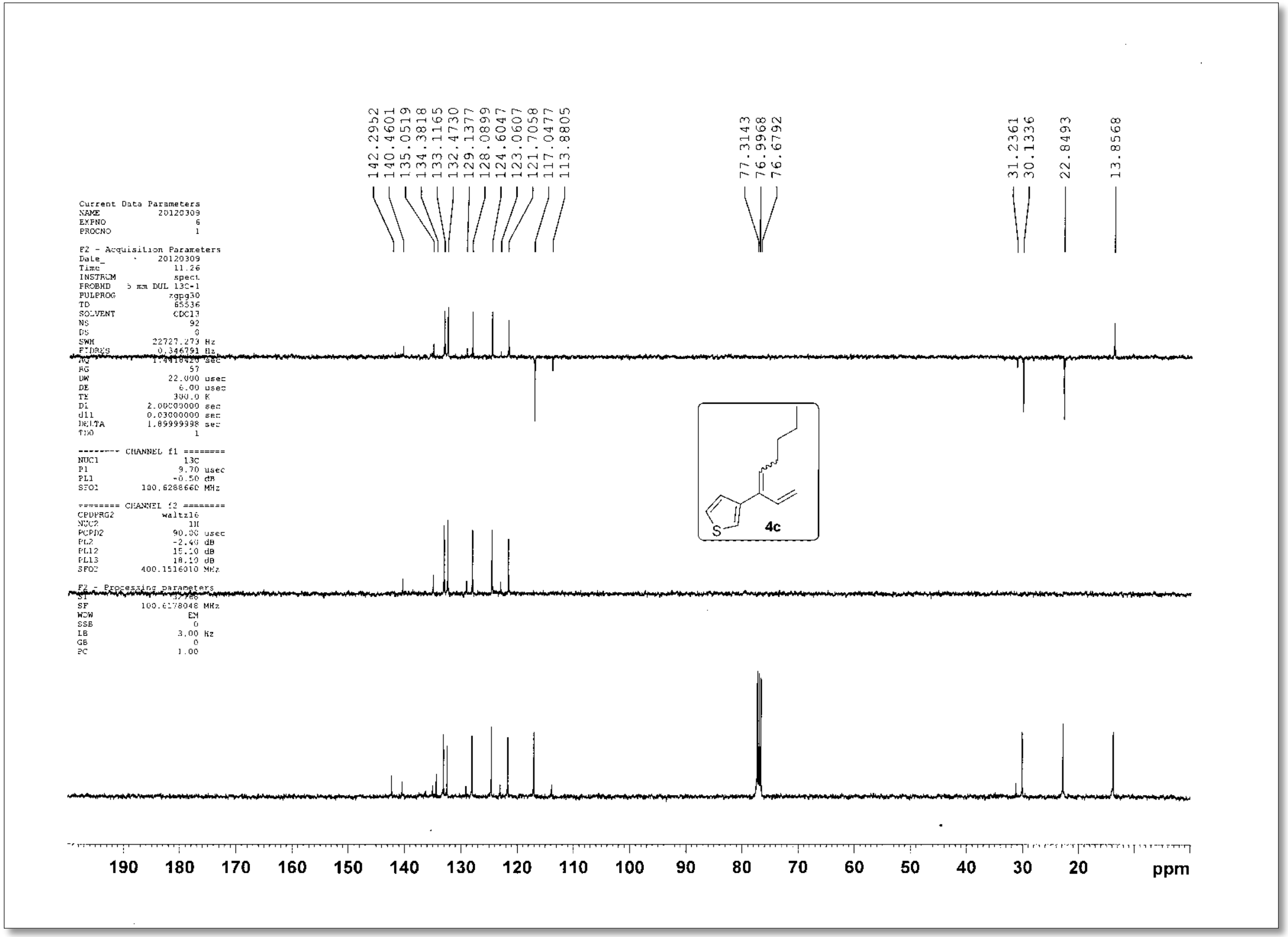


```
Current Data Parameters
NAME             20120313
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date             20120313
Time                11.15
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     12
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    406
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

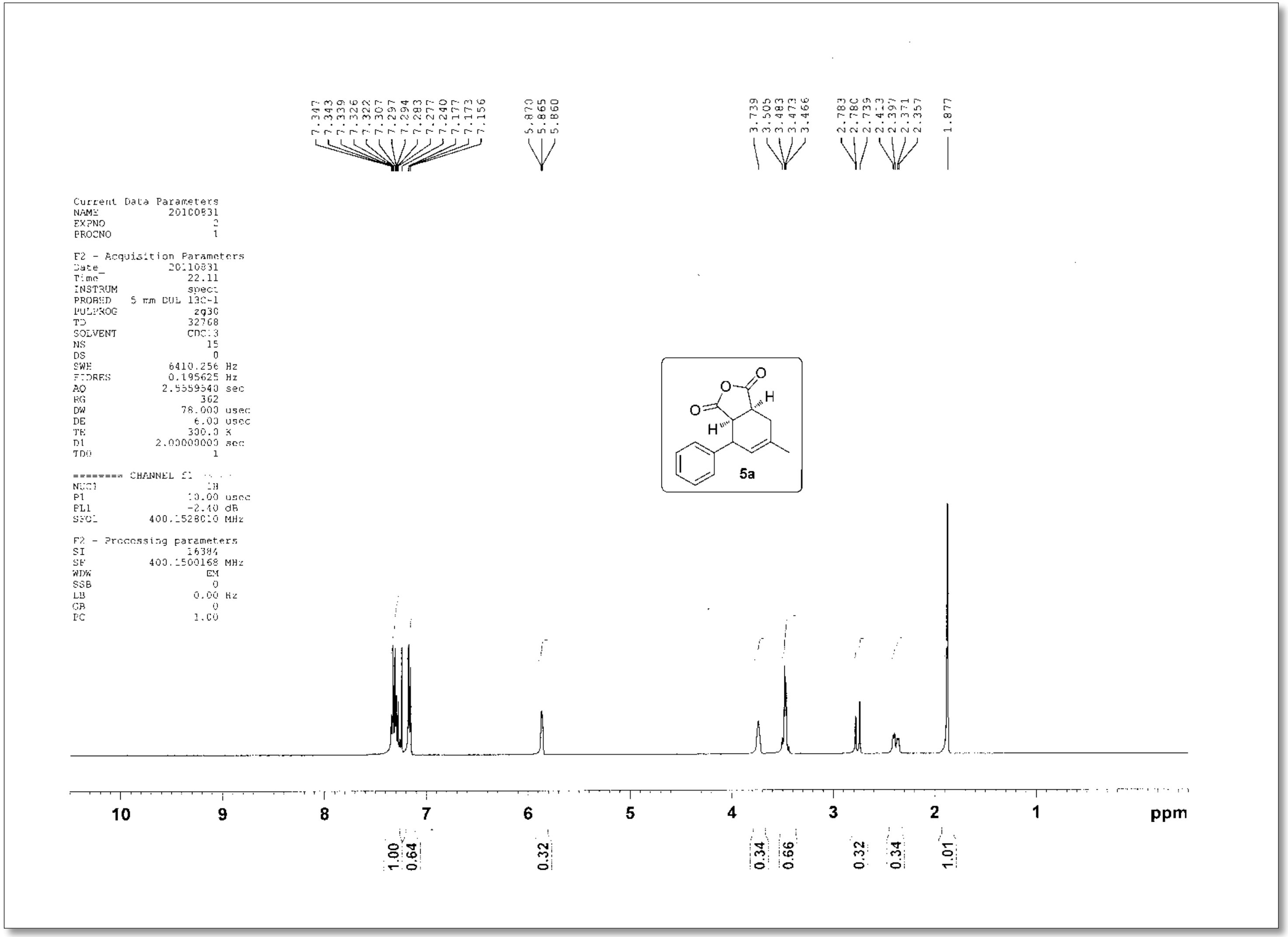

143.9180
140.4106
136.9710
133.2691
132.8030
132.5318
126.9460
126.8165
126.6313
125.0067
124.8628
123.6485
117.8759
114.4146
77.3183
77.0010
76.6833
31.4070
30.4051
22.7467
13.8454
Current Data Parameters
NAME 20120313
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120313
Time 11.30
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 88
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 9.70 usec
PL1 -0.50 dB
SFO1 100.6298660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.10 dB
PL13 18.10 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178069 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
S
4b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



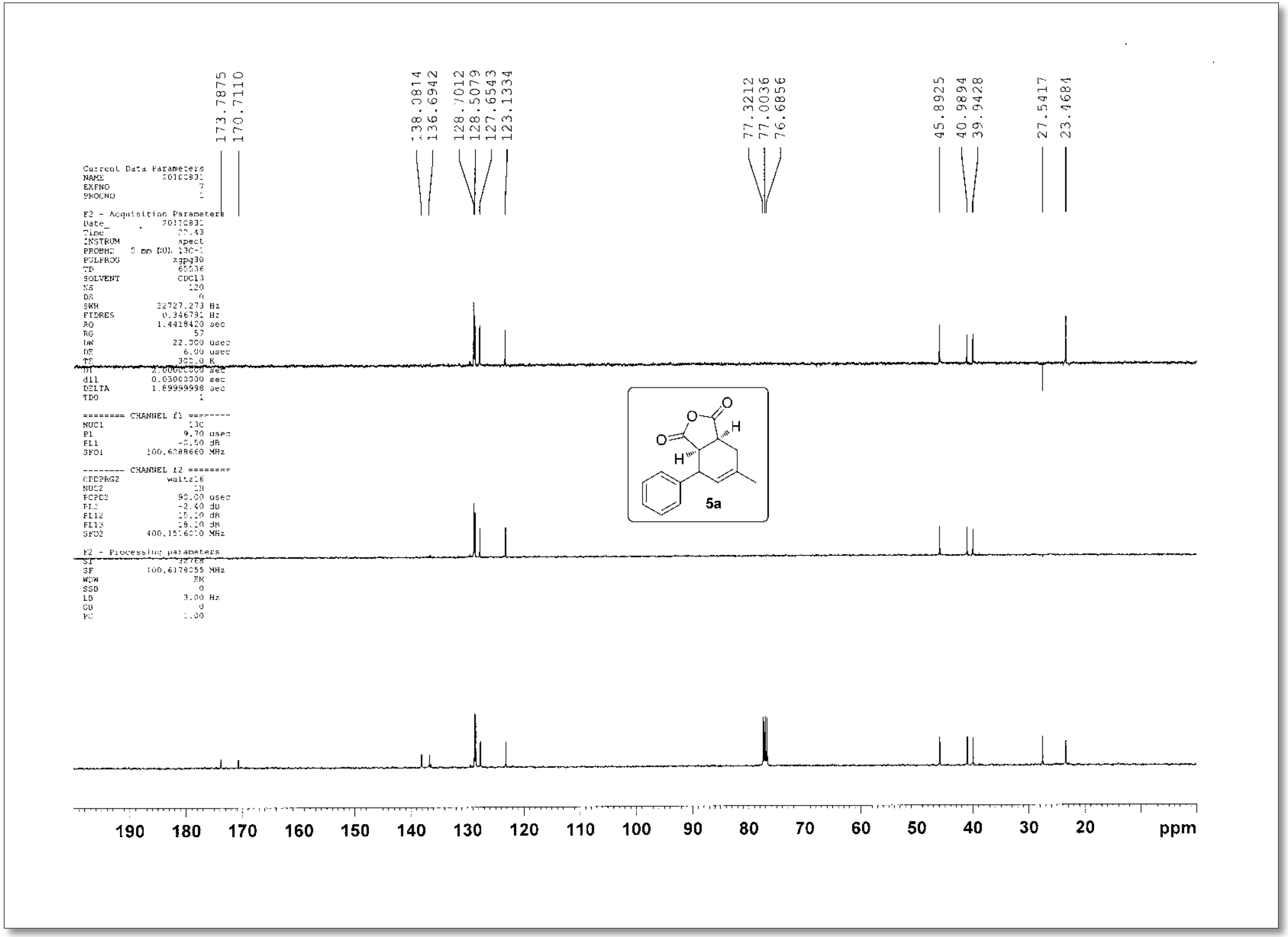
```
Current Data Parameters
NAME            20120309
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120309
Time               11.20
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    10
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   456
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -2.40 dB
SFO1         400.1526010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500166 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



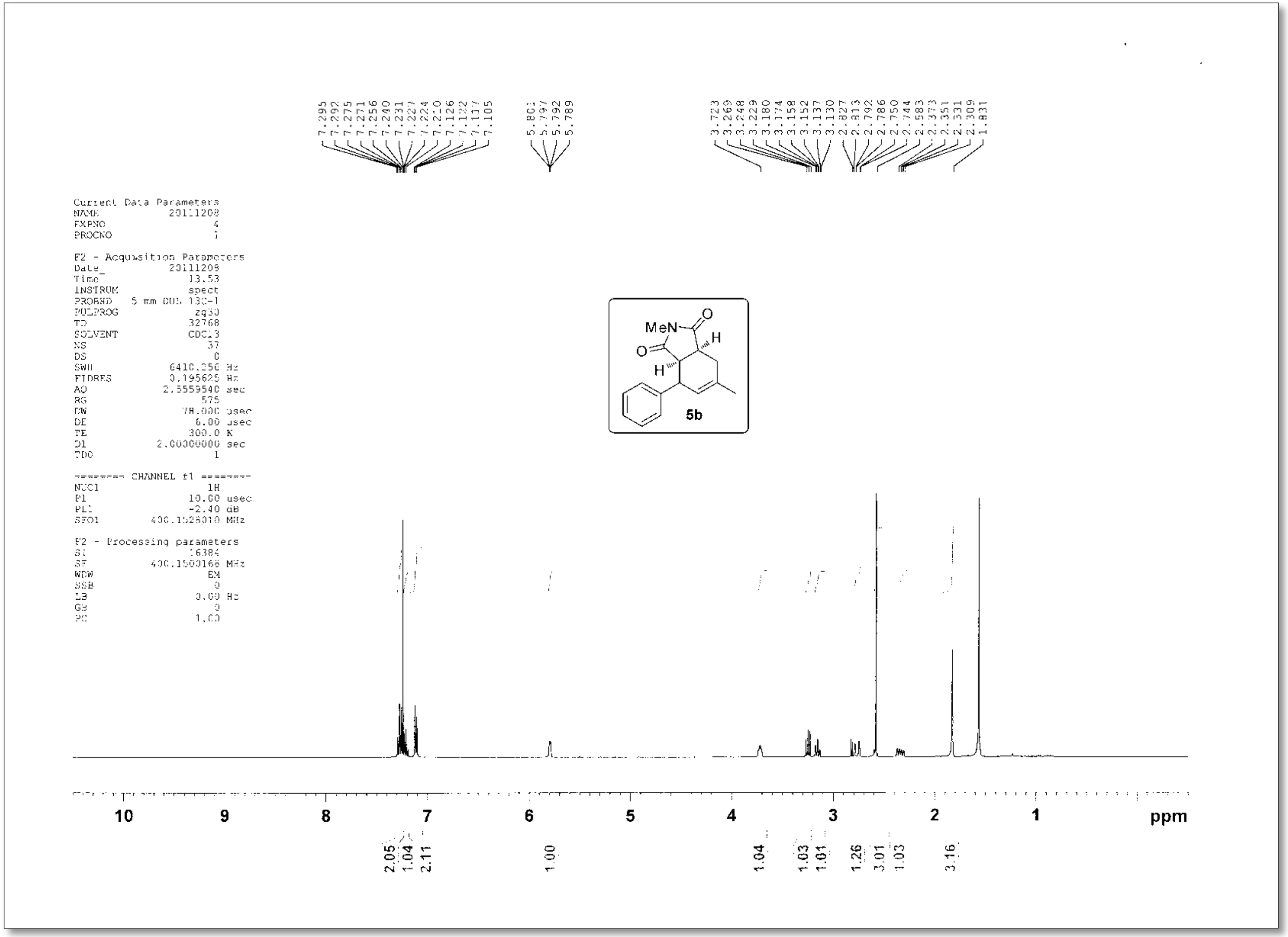
```
Current Data Parameters
NAME             20120309
EXPNO                   6
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120309
Time                11.26
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    92
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178048 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```



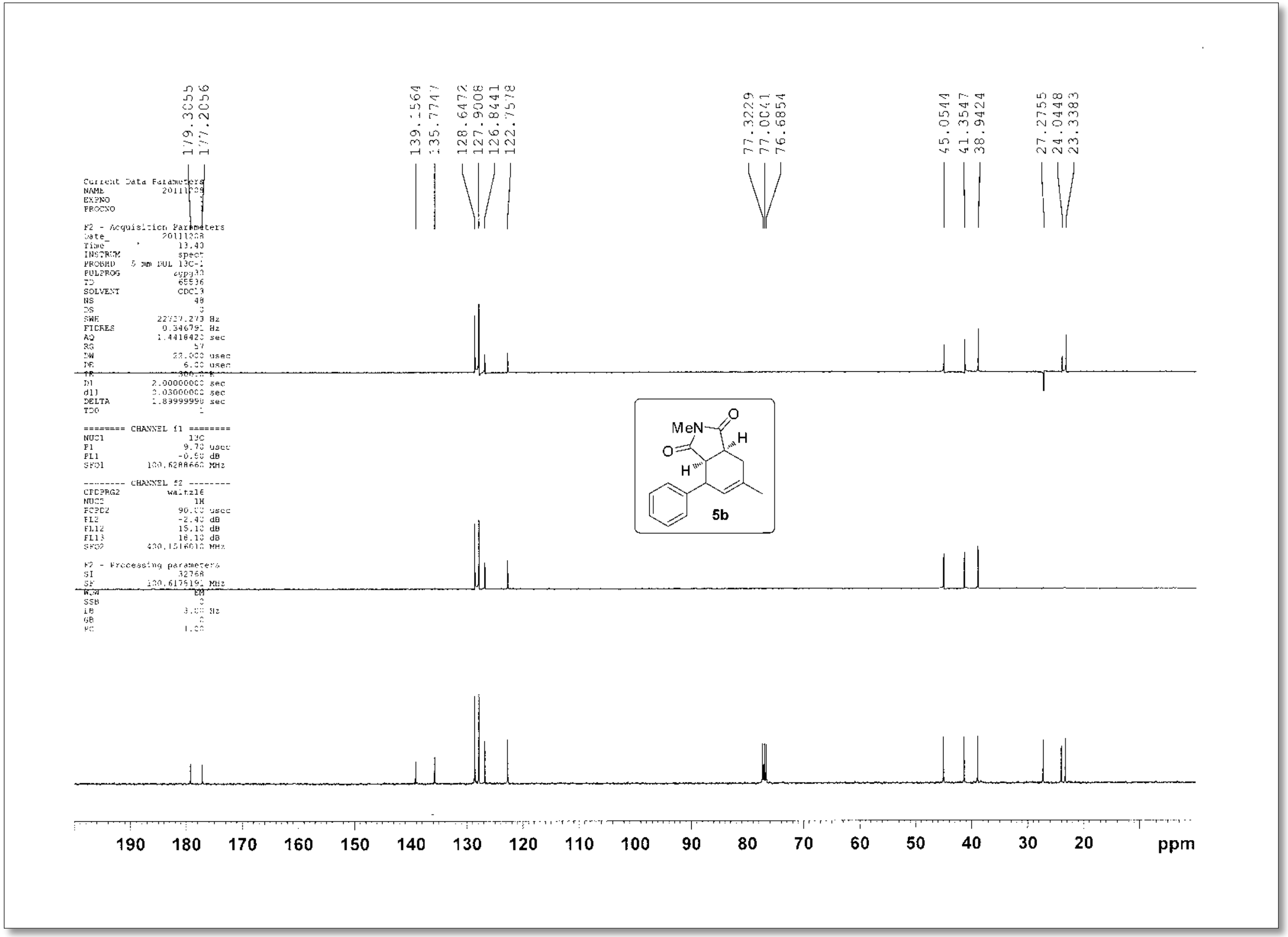
```
Current Data Parameters
NAME             20100831
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_            20110831
Time                22.11
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     15
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    362
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



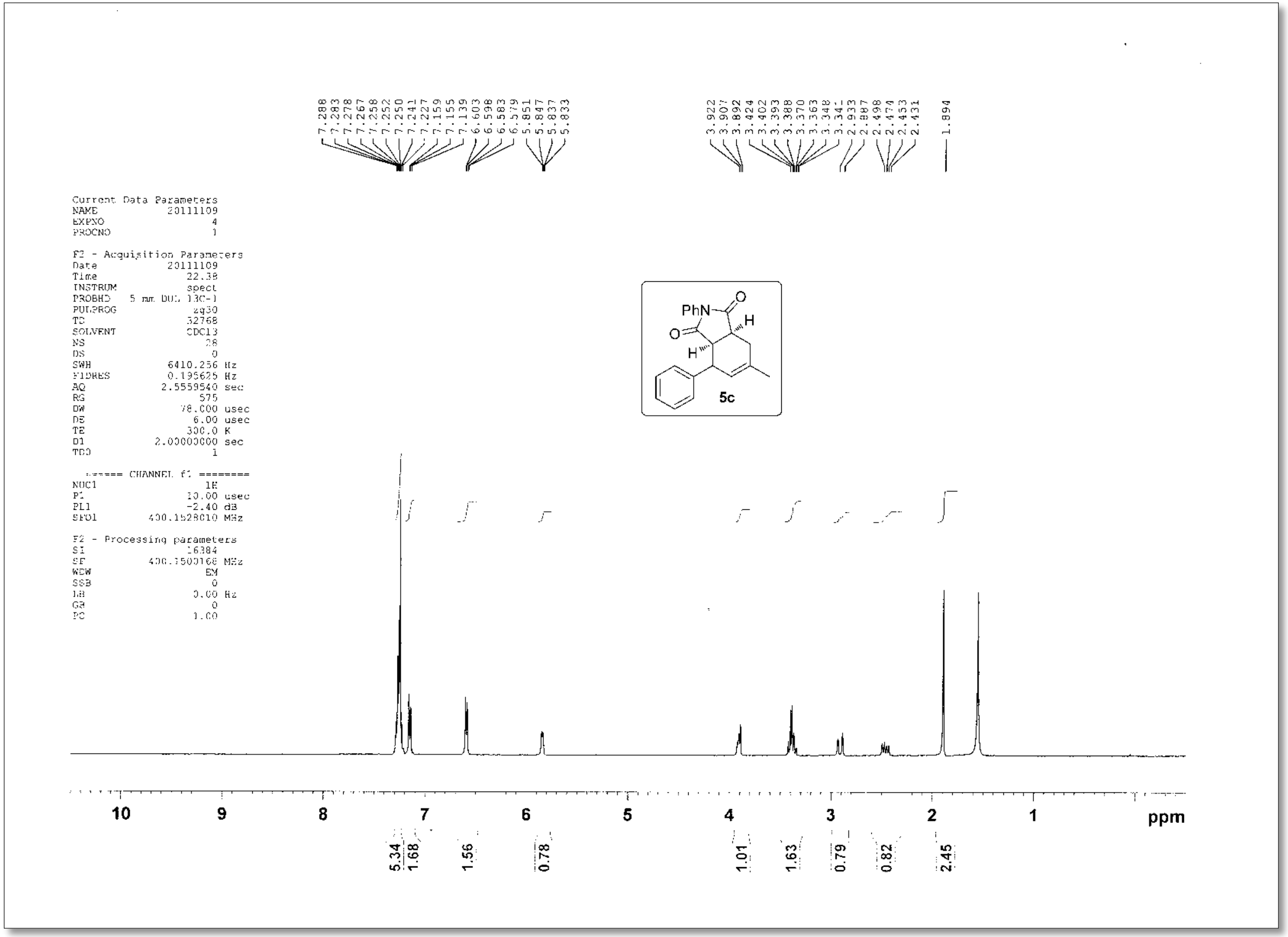
```
Current Data Parameters
NAME              20100831
EXPNO                    7
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100831
Time                 22.43
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     120
DS                       6
SWH              22727.273 Hz
FIDRES            0.346791 Hz
AQ               1.4418420 sec
RG                      57
DW                  22.000 usec
DE                    6.00 usec
TE                   300.0 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.70 usec
PL1                  -0.50 dB
SFO1           100,6288660 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -2.40 dB
PL12                 15.10 dB
PL13                 18.10 dB
SFO2           400,1516010 MHz

F2 - Processing parameters
SI                   32768
SF             100,6178055 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.00
```



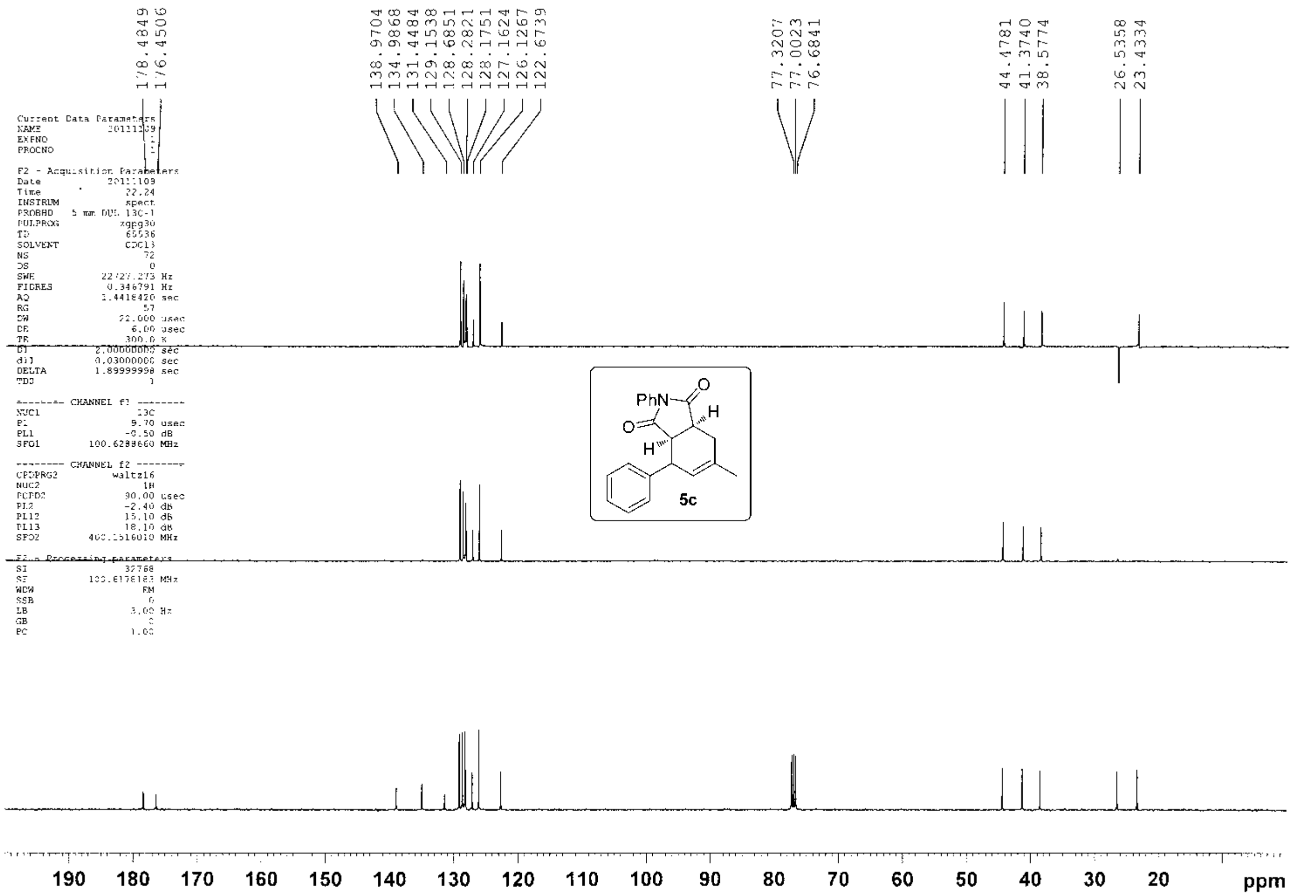
```
Current Data Parameters
NAME            20111208
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111209
Time               13.53
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    37
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   575
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -2.40 dB
SFO1         400.1526010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500166 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



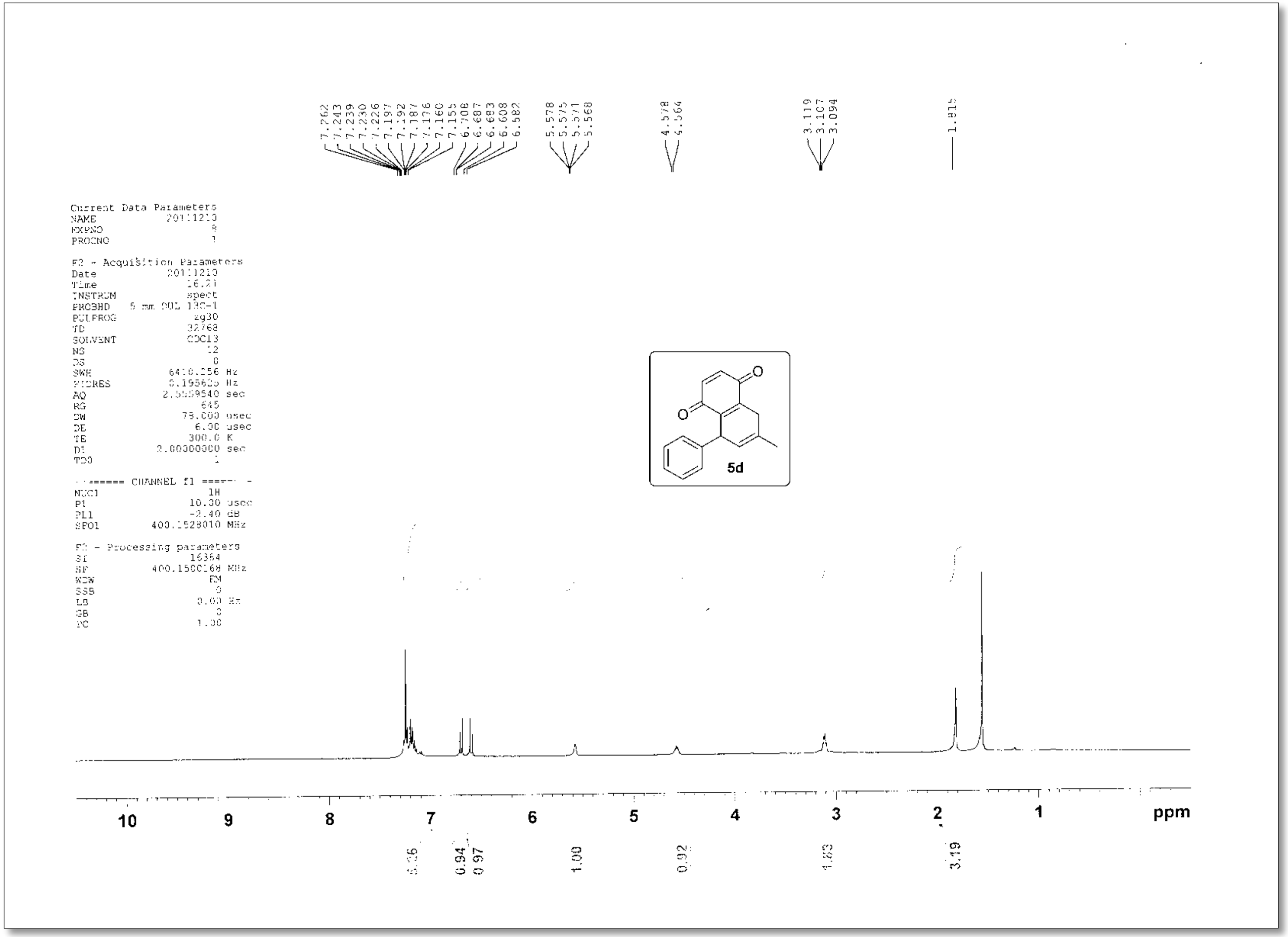
```
Current Data Parameters
NAME             20111209
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111208
Time                13.40
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     48
DS                      0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1        100.6288660 MHz

-------- CHANNEL f2 --------
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                16.10 dB
SFO2        400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF          100.6176191 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```



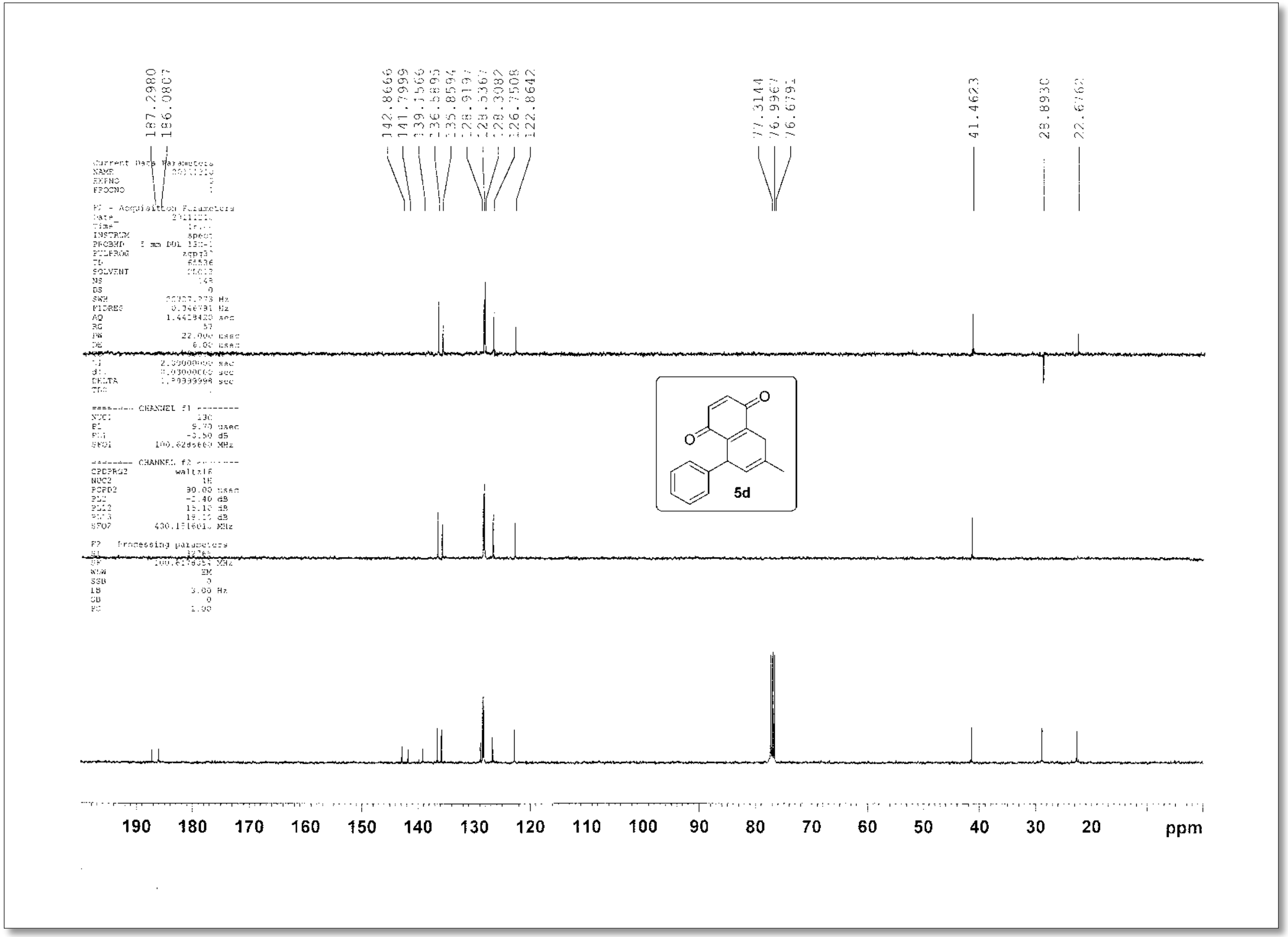
```
Current Data Parameters
NAME            20111109
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date            20111109
Time               22.38
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    28
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   575
DW                78.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PL1                -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500168 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```



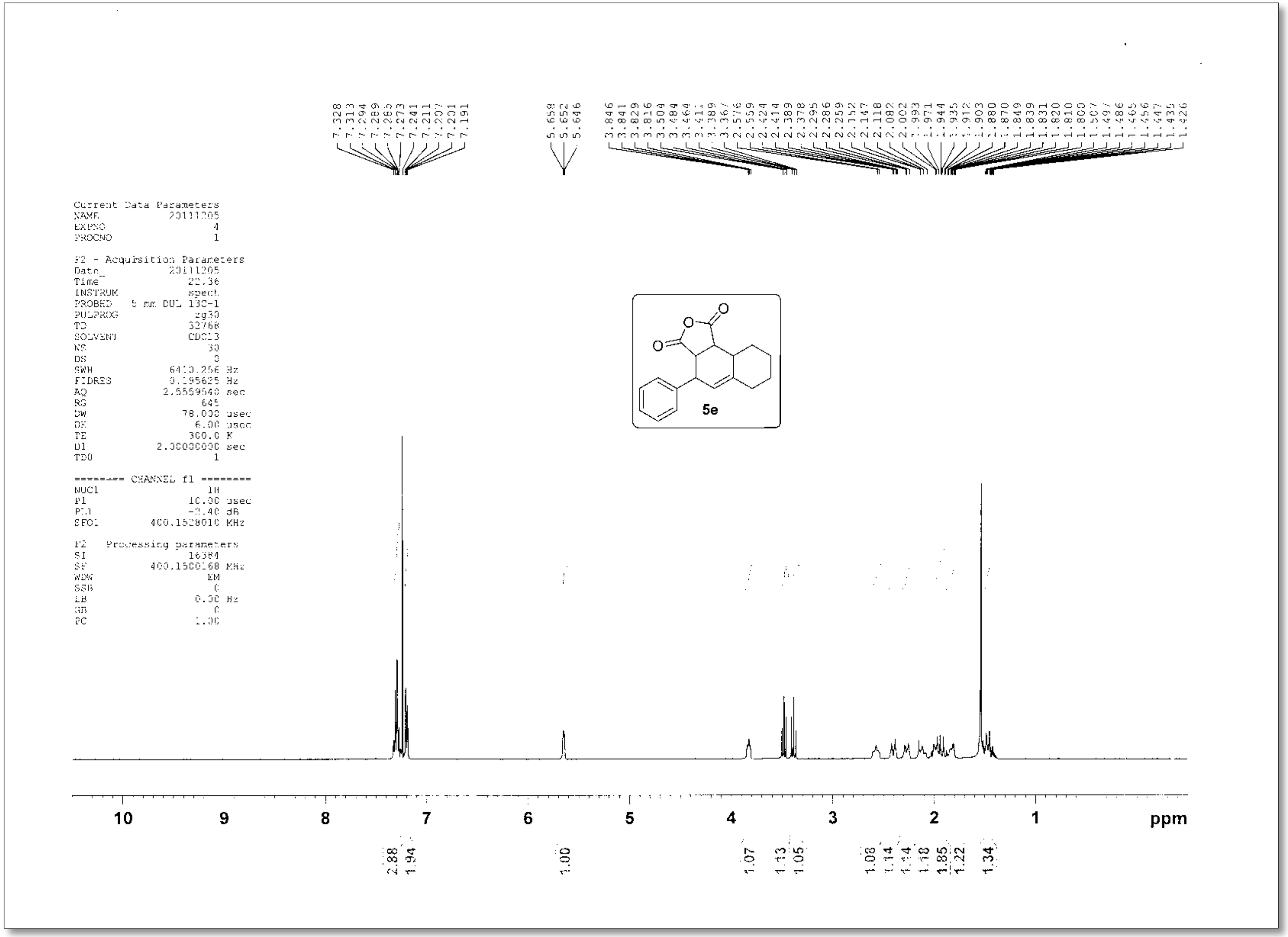
```
Current Data Parameters
NAME          20111109
EXPNO                1
PROCNO               1

F2 - Acquisition Parameters
Date_         20111109
Time             22.24
INSTRUM          spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  72
DS                   0
SWH          22727.273 Hz
FIDRES        0.346791 Hz
AQ           1.4418420 sec
RG                  57
DW              22.000 usec
DE                6.00 usec
TE               300.0 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                9.70 usec
PL1              -0.50 dB
SFO1       100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2                1H
PCPD2            90.00 usec
PL2              -2.40 dB
PL12             15.10 dB
PL13             18.10 dB
SFO2       400.1516010 MHz

F2 - Processing parameters
SI               32768
SF         100.6178183 MHz
WDW                 EM
SSB                  0
LB                3.00 Hz
GB                   0
PC                1.00
```

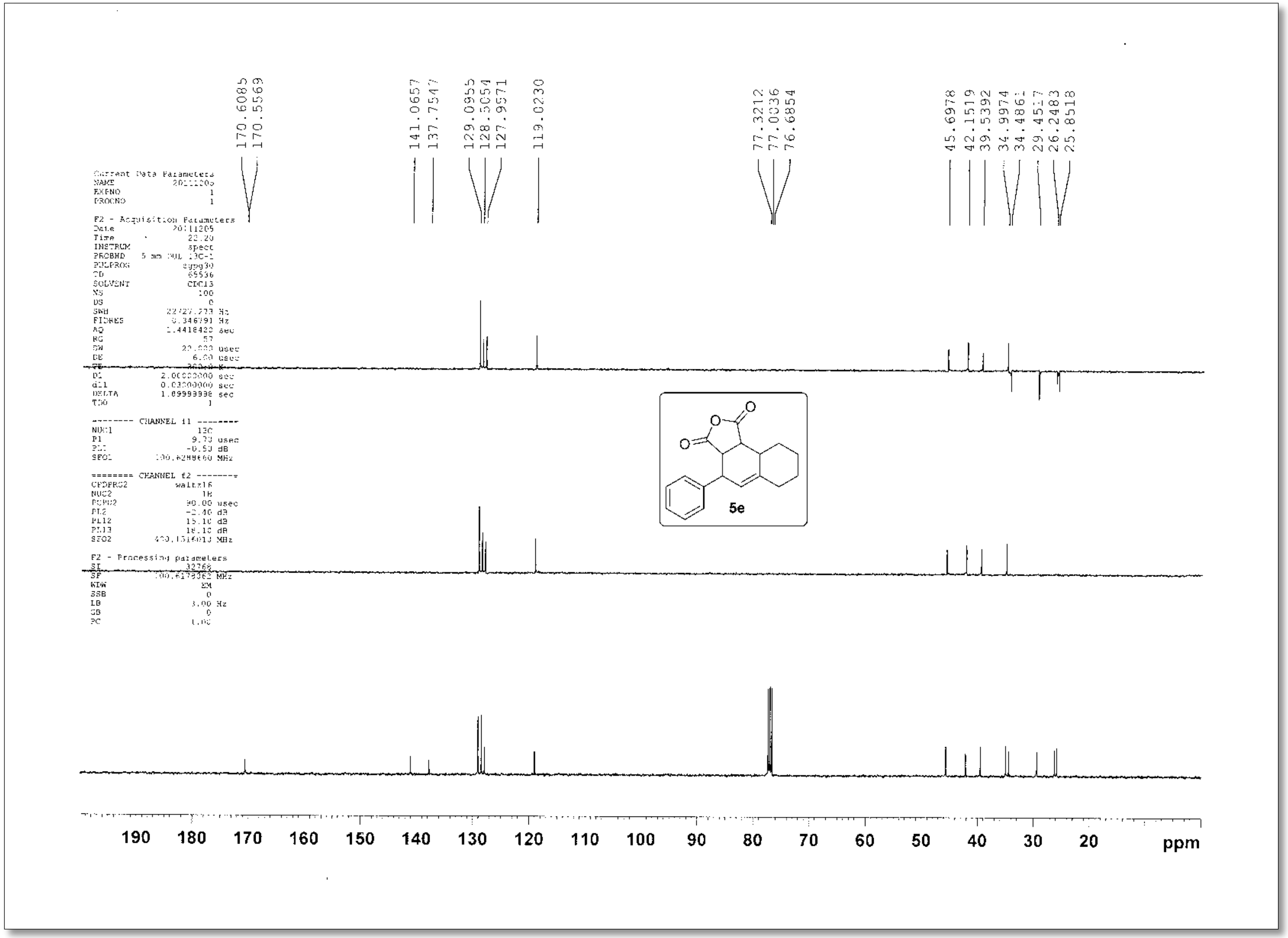


```
Current Data Parameters
NAME             20111210
EXPNO                   8
PROCNO                  1

F2 - Acquisition Parameters
Date_            20111210
Time                16.21
INSTRUM             spect
PROBHD   5 mm QNP 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     12
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    645
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.30 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF          400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```

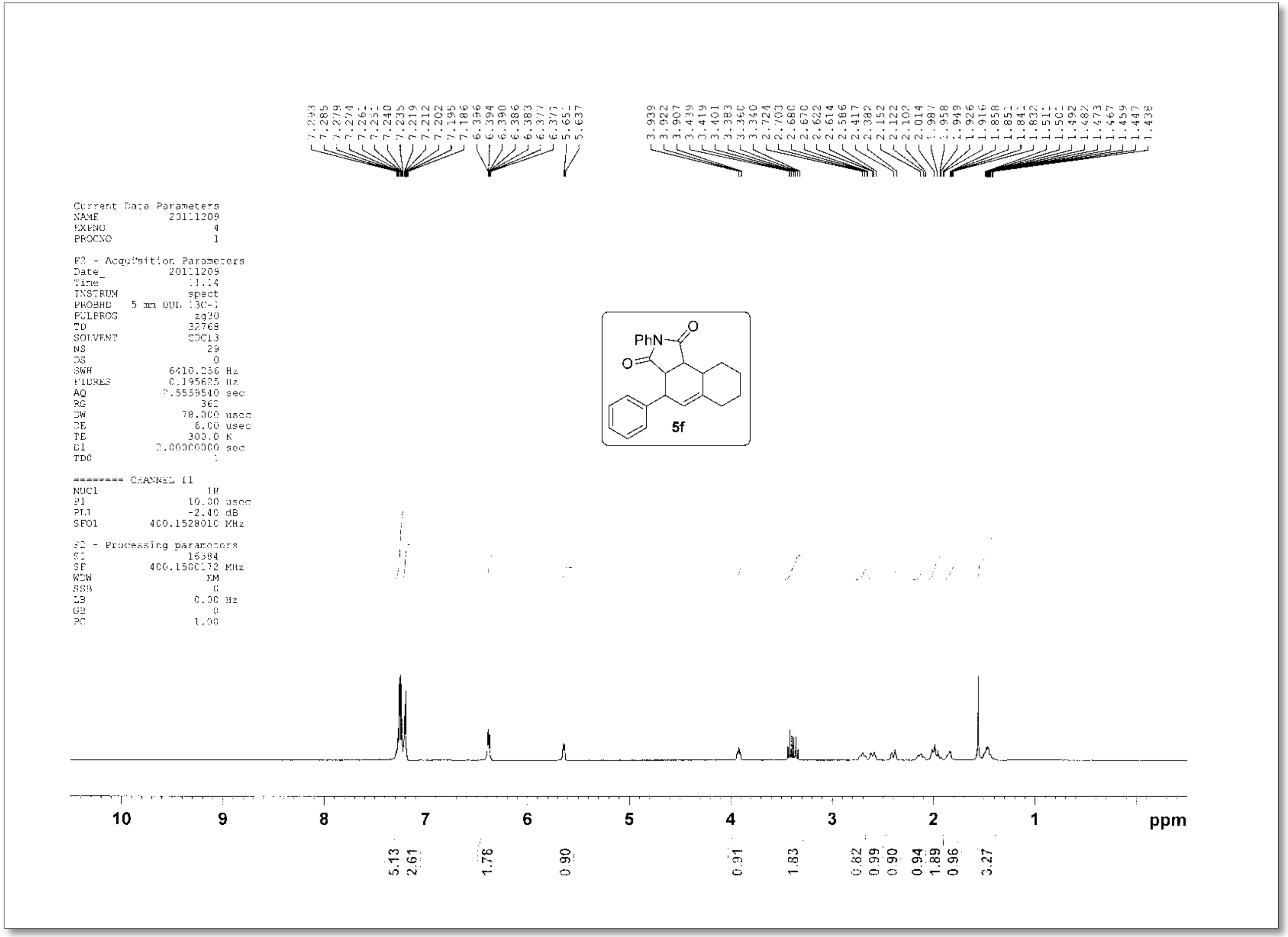

187.2980
186.0867
142.8666
141.7999
139.3566
136.5895
135.8594
128.9197
128.5367
128.3082
126.7508
122.8642
77.3144
76.9967
76.6791
41.4623
28.8930
22.6762
Current Data Parameters
NAME
EXPNO
PROCNO
F2 - Acquisition Parameters
Date_
Time
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS
DS 0
SWH Hz
FIDRES Hz
AQ sec
RG
DW 22.000 usec
DE 6.00 usec
TE
d1 sec
DELTA sec
TD0
======== CHANNEL f1 ========
NUC1 13C
P1 9.70 usec
PL1 dB
SFO1 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 dB
PL12 dB
PL13 dB
SFO2 MHz
F2 - Processing parameters
SI
SF MHz
WDW EM
SSB 0
LB Hz
GB 0
PC 1.00
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

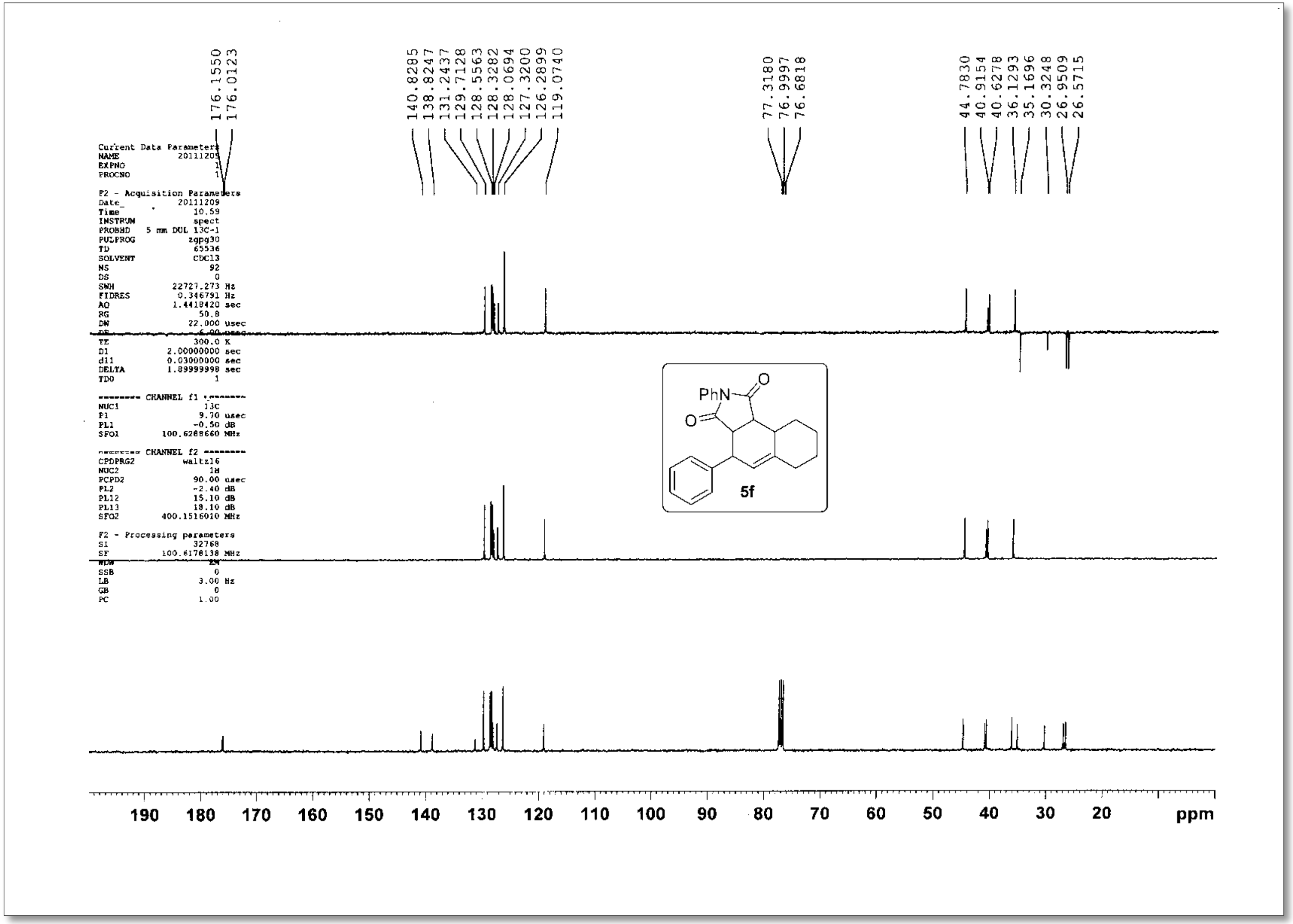
5d


7.328
7.313
7.294
7.289
7.265
7.273
7.241
7.211
7.207
7.201
7.191
5.659
5.652
5.646
3.846
3.841
3.829
3.816
3.504
3.484
3.464
3.411
3.389
3.367
2.576
2.559
2.424
2.414
2.389
2.378
2.295
2.286
2.259
2.152
2.147
2.118
2.082
2.002
1.993
1.971
1.944
1.935
1.912
1.903
1.880
1.870
1.849
1.839
1.831
1.820
1.810
1.800
1.507
1.497
1.486
1.465
1.456
1.447
1.435
1.426
Current Data Parameters
NAME 20111205
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111205
Time 22.36
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 30
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 645
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
O
5e
10
9
8
7
6
5
4
3
2
1
ppm
2.88
1.94
1.00
1.07
1.13
1.05
1.08
1.14
1.14
1.18
1.85
1.22
1.34

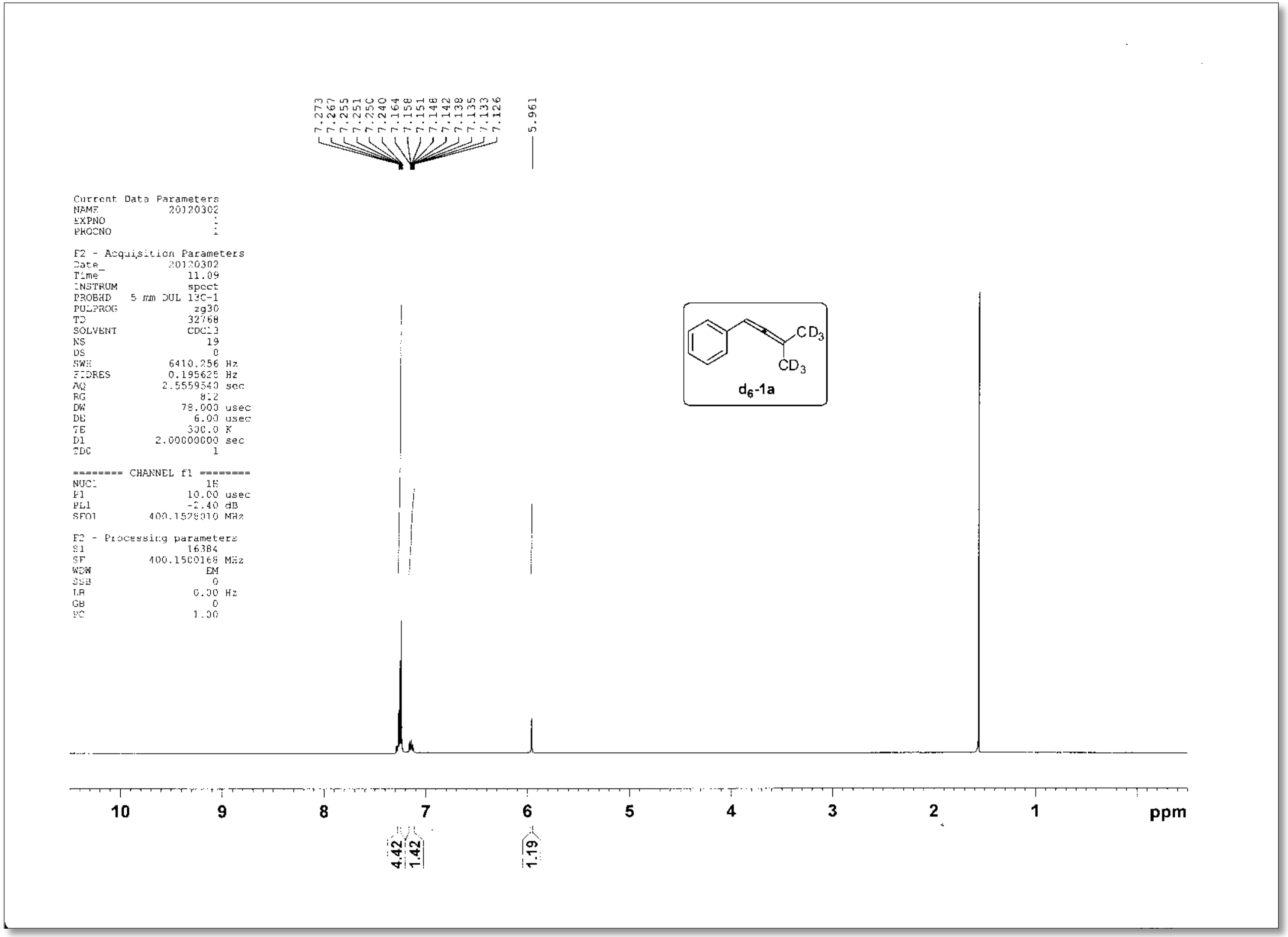


```
Current Data Parameters
NAME             20111205
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date             20111205
Time                22.20
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    100
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                     57
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1316013 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178362 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```


7.293
7.285
7.279
7.274
7.261
7.251
7.240
7.235
7.219
7.212
7.202
7.195
7.186
6.396
6.394
6.390
6.386
6.383
6.377
6.371
5.651
5.637
3.939
3.922
3.907
3.439
3.419
3.401
3.383
3.360
3.340
2.724
2.703
2.680
2.670
2.622
2.614
2.586
2.417
2.382
2.152
2.122
2.102
2.014
1.987
1.958
1.949
1.926
1.916
1.858
1.851
1.841
1.832
1.511
1.501
1.492
1.482
1.473
1.467
1.459
1.447
1.438
Current Data Parameters
NAME 20111209
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111209
Time 11.14
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 29
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 362
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500172 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
PhN
O
O
5f
10
9
8
7
6
5
4
3
2
1
ppm
5.13
2.61
1.78
0.90
0.91
1.83
0.82
0.99
0.90
0.94
1.89
0.96
3.27

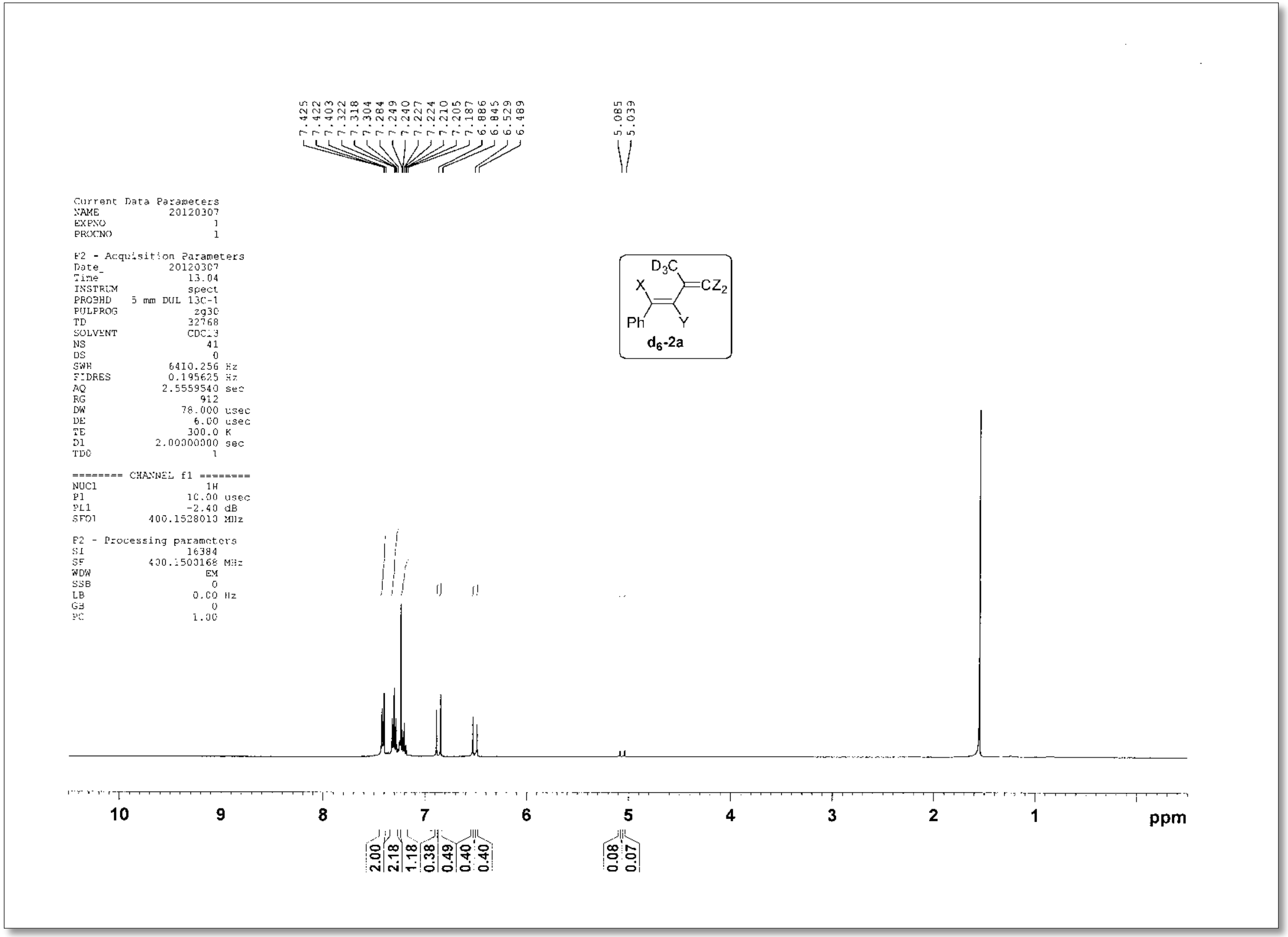


```
Current Data Parameters
NAME              20111209
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20111209
Time                 10.59
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     92
DS                      0
SWH             22727.273 Hz
FIDRES           0.346791 Hz
AQ              1.4418420 sec
RG                   50.8
DW                 22.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                   9.70 usec
PL1                 -0.50 dB
SFO1          100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -2.40 dB
PL12                15.10 dB
PL13                18.10 dB
SFO2          400.1516010 MHz

F2 - Processing parameters
SI                  32768
SF            100.6178138 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.00
```

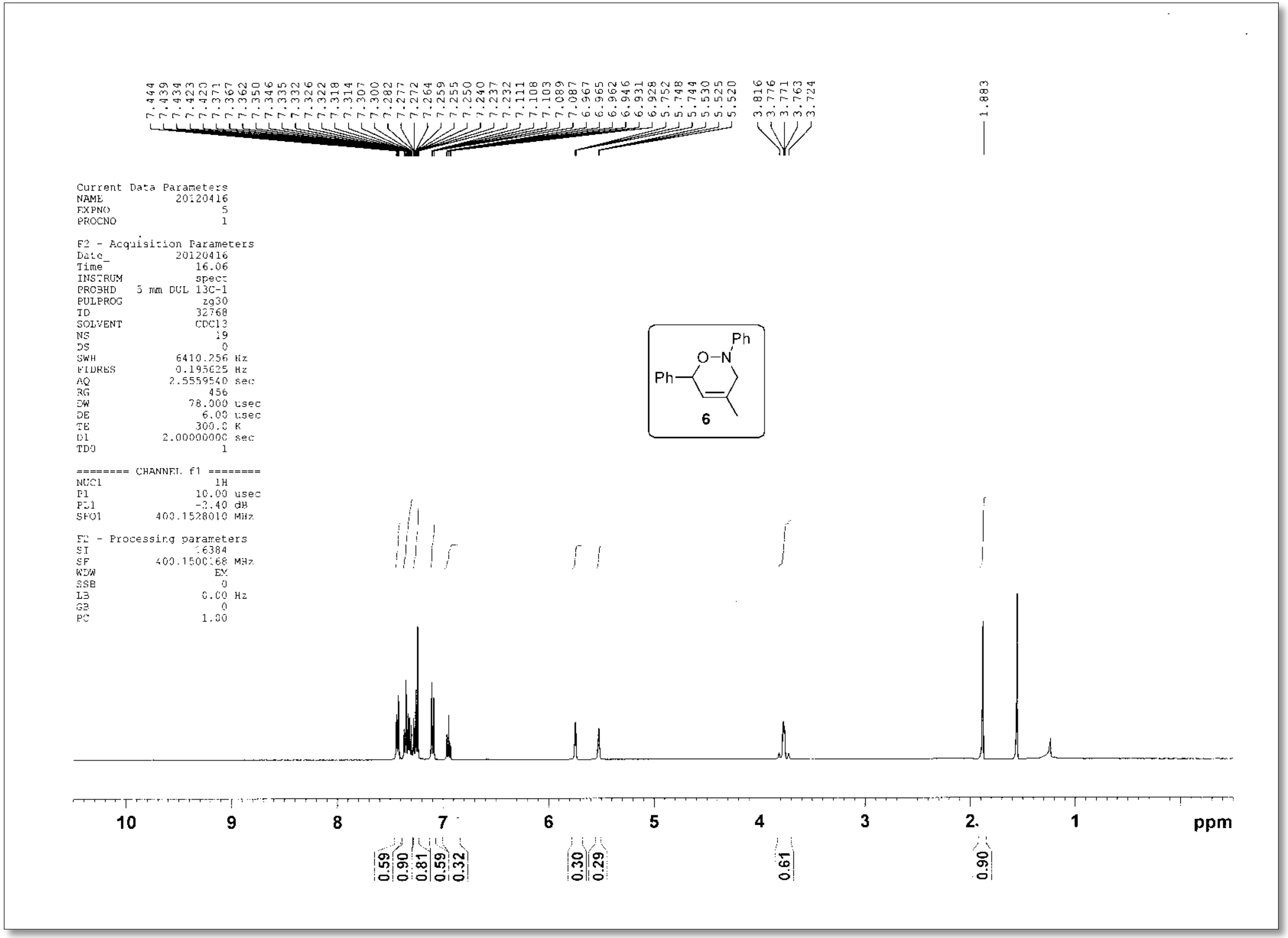


```
Current Data Parameters
NAME             20120302
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120302
Time                11.09
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     19
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    812
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

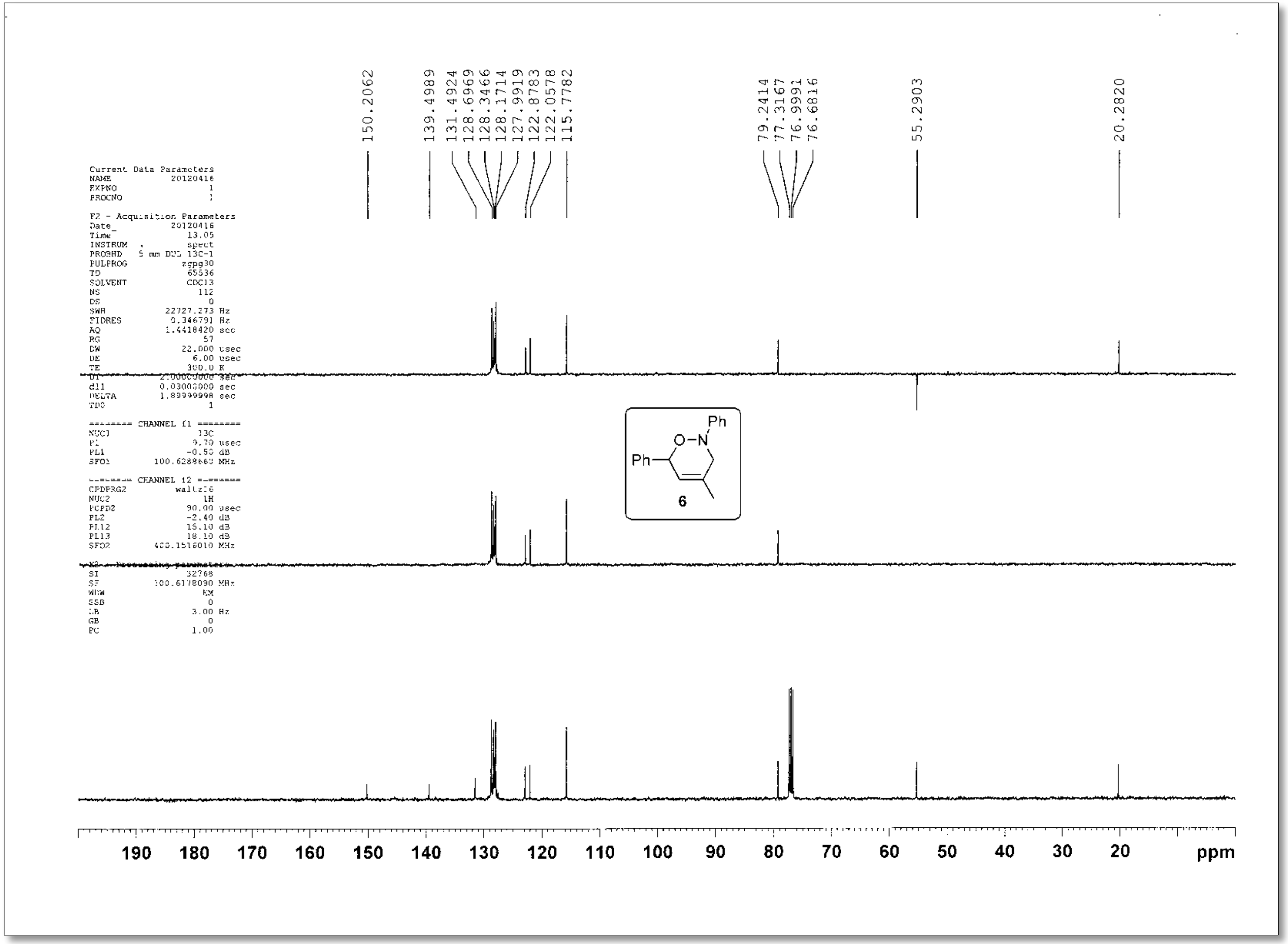
d_6-**1a**



```
Current Data Parameters
NAME               20120307
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20120307
Time                  13.04
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       41
DS                        0
SWH                6410.256 Hz
FIDRES             0.195625 Hz
AQ                2.5559540 sec
RG                      912
DW                   78.000 usec
DE                     6.00 usec
TE                    300.0 K
D1               2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.00 usec
PL1                   -2.40 dB
SFO1            400.1528010 MHz

F2 - Processing parameters
SI                    16384
SF              400.1500168 MHz
WDW                      EM
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.00
```



```
Current Data Parameters
NAME             20120416
EXPNO                   5
PROCNO                  1

F2 - Acquisition Parameters
Date_            20120416
Time                16.06
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     19
DS                      0
SWH              6410.256 Hz
FIDRES           0.195625 Hz
AQ              2.5559540 sec
RG                    456
DW                 78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.00 usec
PL1                 -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                  16384
SF            400.1500168 MHz
WDW                    EM
SSB                     0
LB                   0.00 Hz
GB                      0
PC                   1.00
```



```
Current Data Parameters
NAME            20120416
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120416
Time               13.05
INSTRUM   .        spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   112
DS                     0
SWH            22727.273 Hz
FIDRES          0.346791 Hz
AQ             1.4418420 sec
RG                    57
DW                22.000 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                  9.70 usec
PL1                -0.50 dB
SFO1         100.6288663 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PL2                -2.40 dB
PL12               15.10 dB
PL13               18.10 dB
SFO2         400.1516010 MHz

F2 - Processing parameters
SI                 32768
SF           100.6178090 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00
```