Supporting Information for
Nickel-Catalyzed Formation of Cyclopentenone Derivatives via the Unique Cycloaddition of α,β-Unsaturated Phenylesters with Alkynes

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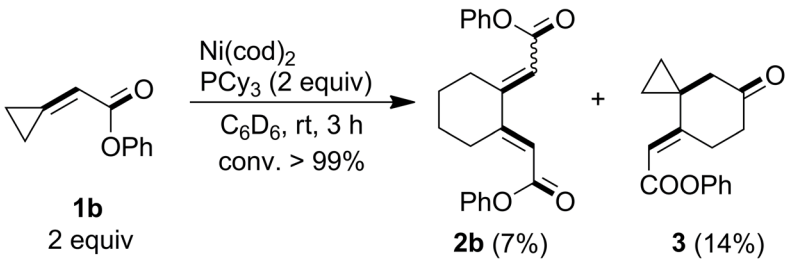
Experimental Section

General: All manipulations were conducted under a nitrogen atmosphere using standard Schlenk or dry box techniques. ^{1}H, ^{31}P, and ^{13}C nuclear magnetic resonance spectra were recorded on Brucker Avance III 400 and Varian Unity Inova 600 spectrometers. The chemical shifts in ^{1}H NMR spectra were recorded relative to either Me_4Si or residual protiated solvent (C_6D_5H (δ 7.16), $CHCl_3$ (δ 7.27), or toluene-d_7 (δ 2.09)). The chemical shifts in the ^{13}C NMR spectra were recorded relative to Me_4Si. The chemical shifts in the ^{31}P NMR spectra were recorded using 85% H_3PO_4 as external standard. Elemental analyses were performed at Instrumental Analysis Center, Faculty of Engineering, Osaka University. X-ray crystal data were collected by a Rigaku RAXIS-RAPID Imaging Plate diffractometer.

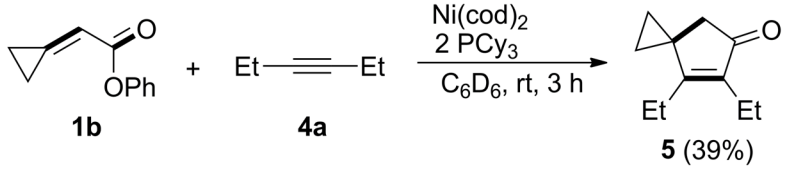
Materials: The degassed and distilled solvents (toluene and hexane) used in this work were commercially available. THF, C_6D_6, and toluene-d_8 were distilled from sodium benzophenone ketyl. ^{i}PrOH was distilled from sodium. ^{i}PrOD (>98% D) was purchased from Merck and used as received. All commercially available reagents were distilled and degassed prior to use.



Stoichiometric reaction of 1b with Ni(cod)$_2$ in the presence of PCy$_3$: To a C_6D_6 solution of Ni(cod)$_2$ (11.1 mg, 0.04 mmol) and PCy$_3$ (22.7 mg, 0.08 mmol) was added a C_6D_6 solution of **1b** (17.6 mg, 0.10 mmol), resulting in change of the color from orange to dark brown. The reaction mixture was stirred for 3 h at ambient temperature, and then insoluble was filtered to remove by passing through a pad of celite. The filtrate was concentrated *in vacuo* to give brown residue. NMR analysis using 2-methoxynaphthalene as internal standard revealed the formation of **2b** and **3** in 7% and 14% yield, respectively. Isolation of the products was conducted with 0.20 mmol of **1b**, and purification of the crude product by HPLC gave **2b** and **3** in 4 mg and 13 mg yield, respectively.

Spectral data of **3**: ^{1}H NMR (400 MHz, C_6D_6, rt): δ 0.14 (dd, *J* = 4.4, 6.4 Hz, 2H, C*H*$_2$ of C^3-ring), 0.42 (dd, *J* = 4.4, 6.4 Hz, 2H, C*H*$_2$ of C^3-ring), 1.81 (s, 2H, -COC*H*$_2$C), 2.11 (t, *J* = 6.8 Hz, 2H, -C*H*$_2$CH$_2$CO), 3.09 (m, 2H, -CH$_2$C*H*$_2$CO), 5.55 (s, 1H, =C*H*CO$_2$Ph), 6.94 (t, *J* = 7.2 Hz, 1H, -*Ph*), 7.10 (t, *J* = 8.0 Hz, 2H, -*Ph*), 7.18 (m, 2H, -*Ph*). ^{13}C NMR (100 MHz, C_6D_6, rt): δ 15.8 (C^3), 22.8 (C^3), 27.0 (-*C*H$_2$CH$_2$CO) 37.9 (-CH$_2$*C*H$_2$CO), 47.4 (-CO*C*H$_2$C-), 110.0 (-C=*C*HCO$_2$Ph), 122.1 (-*Ph*), 125.7 (-*Ph*), 129.6 (-*Ph*), 151.4 (*ipso*-*Ph*), 164.5 (-*C*O$_2$Ph), 165.8 (-*C*=CHCO$_2$Ph), 207.1 (-*C*O). HRMS (CI) Calcd for $C_{16}H_{17}O_3$ 257.1178 (M + H), Found m/z 257.1180.

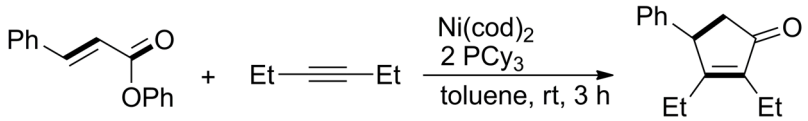
Spectral data for **2b**: ^{1}H NMR (400 MHz, C_6D_6, rt): δ 1.21 (br s, 4H, -C*H*$_2$CH$_2$C-), 2.96 (br s, 4H, -CH$_2$C*H*$_2$C-), 6.01 (s, 2H, C=C*H*-), 6.94 (t, J = 7.2 Hz, 2H, -O*Ph*), 7.00-7.16 (m, 8H, -O*Ph*). ^{13}C NMR (100 MHz, C_6D_6, rtm): δ 25.6 (*C*H$_2$CH$_2$C), 30.4 (CH$_2$*C*H$_2$C), 114.9 (-C=*C*H-), 122.0 (-O*Ph*), 125.8 (-O*Ph*), 129.6 (-O*Ph*), 151.3 (ipso-*Ph*), 162.5 (-*C*=CH-), 164.0 (-*C*O). HRMS (CI) Calcd for $C_{22}H_{21}O_4$ 349.1440 (M + H), Found m/z .349.1443.



Stoichiometric reaction of 1b with 4a in the presence of Ni(cod)$_2$ and PCy$_3$: To a C_6D_6 solution of Ni(cod)$_2$ (11.1 mg, 0.04 mmol) and PCy$_3$ (21.8 mg, 0.08 mmol) was added a C_6D_6 solution of **1b** (25.0

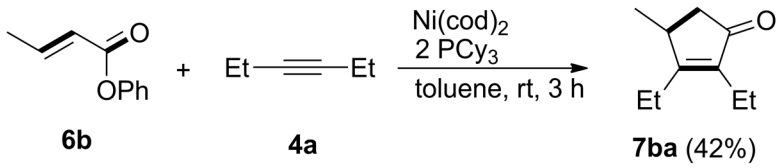
mg, 0.12 mmol), resulting in change of the color from orange to dark brown. Then, 3-hexyne (**4a**, 3.3 mg, 0.04 mmol) was added to the solution, and the reaction mixture was transferred into a J-Young NMR tube. Monitoring of the reaction by NMR spectroscopy demonstrated that all of **1b** were consumed after 3 hours. The yield of **5** (3.4 mg, 52%) was determined by GC analysis using tetradecane as an internal standard. Following the aforementioned procedure, isolation of **5** was conducted with 0.20 mmol of $Ni(cod)_2$. After the reaction mixture was stirred for 3 h at room temperature, insoluble was filtered to remove by passing through a pad of silica, and then the filtrate was concentrated *in vacuo*. To the residue was added 1.0 M aqueous sodium hydroxide (30 mL). The organic layer was extracted with diethyl ether (20 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (hexane/ethyl acetate = 98/2) afforded **5** as colorless oil (13.8 mg, 39%).

^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.94 (m, 2H, -C*H*$_2$ of C^3-ring), 1.02-1.08 (m, 8H, -C*H*$_2$ of C^3-ring and two -C*H*$_3$ groups), 1.98 (q, *J* = 7.6 Hz, 2H, -C*H*$_2$CH$_3$), 2.21 (q, *J* = 7.6 Hz, 2H, -C*H*$_2$CH$_3$), 2.43 (s, 2H, -C*H*$_2$-). ^{13}C NMR (100 MHz, $CDCl_3$, rt): δ 12.1 (-*C*H$_2$ of C^3-ring), 13.2 (-CH$_2$*C*H$_3$), 13.8 (-CH$_2$*C*H$_3$), 17.1 (-*C*H$_2$CH$_3$), 18.1 (-*C*H$_2$CH$_3$), 24.7 (-*C*H$_2$C-), 45.1(C^3-ring), 141.5 (-C=*C*-C=O), 176.9 (-*C*=C-C=O), 208.1 (-C=C-*C*=O). HRMS (EI) Calcd for $C_{11}H_{16}O$ 164.1201, Found m/z 164.1198.



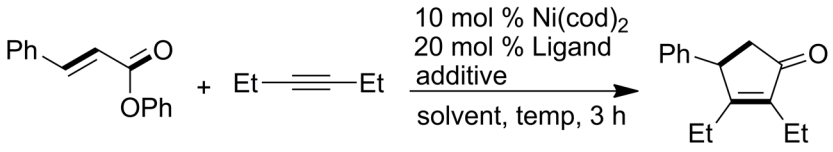
6a **4a** **7aa** (44%)

Stoichiometric reaction of 6a with 4a in the presence of Ni(cod)$_2$ and PCy$_3$: To a C_6D_6 solution of $Ni(cod)_2$ (11.6 mg, 0.04 mmol) and PCy_3 (21.8 mg, 0.08 mmol) was added a C_6D_6 solution of **6a** (9.0 mg, 0.04 mmol), resulting in change of the color from orange to deep red. Then, **4a** (3.6 mg, 0.04 mmol) was added to the solution, and the reaction mixture was transferred into a J-Young NMR tube. Monitoring of the reaction by NMR spectroscopy demonstrated that all of **6a** were consumed after 3 hours. The yield of **7aa** (3.7 mg, 44%) was determined by GC analysis using tetradecane as an internal standard.



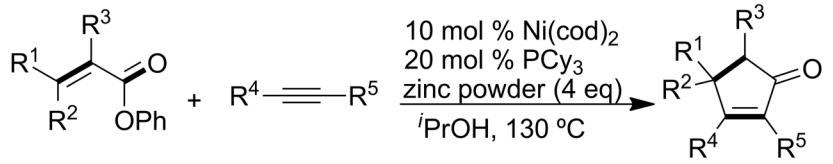
6b **4a** **7ba** (42%)

Stoichiometric reaction of 6b with 4a in the presence of Ni(cod)$_2$ and PCy$_3$: To a C_6D_6 solution of Ni(cod)$_2$ (11.6 mg, 0.04 mmol) and PCy$_3$ (21.8 mg, 0.08 mmol) was added a C_6D_6 solution of **6b** (6.5 mg, 0.04 mmol), resulting in change of the color from orange to deep red. Then, **4a** (3.6 mg, 0.04 mmol) was added to the solution, and the reaction mixture was transferred into a J-Young NMR tube. Monitoring of the reaction by NMR spectroscopy demonstrated that all of **6b** were consumed after 3 hours. The yield of **7ba** (3.0 mg, 42%) was determined by GC analysis using tetradecane as an internal standard.



6a **4a** (4 eq) **7aa**

Optimization for Ni(0)-catalyzed dephenoxylative [3 + 2] cycloaddition of 6a with 4a: All catalytic reactions listed in Table 1, in which 0.10 mmol of **6a** (22.4 mg) was served in 5.0 mL of solvent, were conducted by using a pressure-tight test-tube. After the reaction mixture was thermostated at a given temperature for 3 hours, the yield of **7aa** was determined by GC analysis using tetradecane as an internal standard.

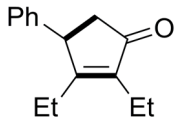


6 **4** (4 eq) **7**

Ni(0)-Catalyzed Dephenoxylative Cycloaddition Reaction of 6 with 4:

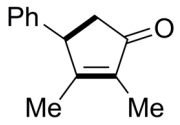
All catalytic reactions listed in Table 2 were conducted by using a pressure-tight test-tube. Ni(cod)$_2$ (0.10 mmol), PCy$_3$ (0.20 mmol), Zn powder (4.0 mmol), and α,β-Unsaturated Phenyl Ester **6** (1.0 mmol) were charged into the test-tube, and then 15.0 mL of ^{i}PrOH and alkyne **4** (4.0 mmol) were added to the mixture in this order. The test-tube was tightly sealed up and thermostated at 130 ºC for 3 hours. After the reaction mixture was cooled down to room temperature, insoluble was filtered to remove by passing

through a pad of celite, and then the filtrate was concentrated *in vacuo*. The resulting crude product was further purified by the method hereinafter described separately.



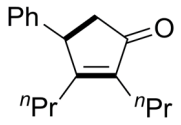
7aa

Table 1, run 3, 2,3-diethyl-4-phenylcyclopenten-2-one (7aa): The resulting crude product was further purified by passing through a pad of SiO_2 (eluent: hexane) followed by HPLC (eluent: $CHCl_3$), yielding **7aa** as colorless oil (194.5 mg, 91%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.99 (t, *J* = 7.6 Hz, 3H, 2-CH_2CH_3), 1.07 (t, *J* = 7.6 Hz, 3H, 3-CH_2CH_3), 1.99 (dq, *J* = 7.6, 7.6 Hz, 1H, 3-$CHHCH_3$), 2.30 (q, *J* = 7.6 Hz, 2H, 2-CH_2CH_3), 2.33 (dd, *J* = 2.4, 18.8 Hz, 1H, -C*H*H-), 2.46 (dq, *J* = 7.6, 7.6 Hz, 1H, 3-$CHHCH_3$), 2.88 (dd, *J* = 6.8, 18.8 Hz, 1H, -CH*H*-), 3.96 (d, *J* = 6.8 Hz, 1H, -CH_2C(Ph)*H*-), 7.07 (d, *J* = 6.8 Hz, 2H, *o-Ph*), 7.20 (t, *J* = 6.8 Hz, 1H, *p-Ph*), 7.28 (t, *J* = 6.8 Hz, 2H, *m-Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.1 (-CH_2CH_3), 13.5 (-CH_2CH_3), 16.5 (-CH_2CH_3), 22.1 (-CH_2CH_3), 44.9 (5-*C*), 46.2 (4-*C*), 127.0 (-*Ph*), 127.4 (-*Ph*), 128.9 (-*Ph*), 142.2 (-*Ph*), 142.2 (2-*C*), 176.1 (3-*C*), 209.1 (1-*C*O). HRMS (EI) Calcd for $C_{15}H_{18}O$ 214.1358, Found m/z 214.1365.



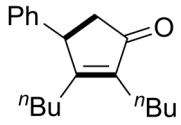
7ab

Table 2, run 1, 2,3-dimethyl-4-phenylcyclopenten-2-one (7ab): The resulting crude product was further purified by passing through a pad of SiO_2 (eluent: hexane) followed by HPLC (eluent: $CHCl_3$), yielding **7ab** as colorless oil (156.5 mg, 84%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 1.78 (dd, *J* = 0.8, 2.0 Hz, 3H, 2-CH_3), 1.81 (s, 3H, 3-CH_3), 2.35 (dd, *J* = 2.0, 19.2 Hz, 1H, -C*H*H-), 2.88 (dd, *J* = 6.8, 19.2 Hz, 1H, -CH*H*-), 3.81 (m, *J* = 6.8 Hz, 1H, -CH_2C(Ph)*H*-), 7.08 (d, *J* = 7.2 Hz, 2H, *o-Ph*), 7.24 (t, *J* = 7.2 Hz, 1H, *p-Ph*), 7.33 (t, *J* = 7.2 Hz, 2H, *m-Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 8.3 (-CH_3), 15.6 (-CH_3), 44.6 (5-*C*), 49.2 (4-*C*), 127.1 (-*Ph*), 127.4 (-*Ph*), 128.9 (-*Ph*), 137.1 (2-C), 142.1 (-*Ph*), 171.6 (3-*C*), 209.1 (1-*C*O). HRMS (EI) Calcd for $C_{13}H_{14}O$ 186.1045, Found m/z 186.1037.



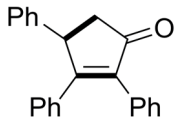
7ac

Table 2, run 2, 4-phenyl-2,3-dipropylcyclopenten-2-one (7ac): The resulting crude product was further purified by passing through a pad of SiO_2 (eluent: hexane) followed by HPLC (eluent: $CHCl_3$), yielding **7ac** as colorless oil (160.0 mg, 66%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.84 (t, *J* = 7.2 Hz, 3H, 2-$CH_2CH_2CH_3$), 0.92 (t, *J* = 7.2 Hz, 3H, 3-$CH_2CH_2CH_3$), 1.35 (m, 1H, 3-CH_2CHHCH_3), 1.50 (dt, *J* = 7.2, 7.2 Hz, 2H, 2-$CH_2CH_2CH_3$), 1.48-1.56 (m, 1H, 3-CH_2CHHCH_3), 1.94 (ddd, *J* = 5.2, 9.2, 14.0 Hz, 1H, 3-$CHHCH_2CH_3$), 2.18-2.31, (m, 2H, 2-$CH_2CH_2CH_3$), 2.32 (dd, *J* = 2.0, 18.8 Hz, 1H, -CHH-), 2.39 (ddd, *J* = 7.2, 9.2, 14.9 Hz, 1H, 3-$CHHCH_2CH_3$), 2.88 (dd, *J* = 7.2, 18.8 Hz, 1H, -CHH-), 3.91 (m, *J* = 7.2 Hz, 1H, -CH_2CH-), 7.06 (d, *J* = 6.8 Hz, 2H, *o*-*Ph*), 7.20 (t, *J* = 6.8 Hz, 1H, *p*-*Ph*), 7.28 (t, *J* = 6.8 Hz, 2H, *m*-*Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 14.2 (-$CH_2CH_2CH_3$), 14.2 (-$CH_2CH_2CH_3$), 20.8 (-$CH_2CH_2CH_3$), 22.0 (-$CH_2CH_2CH_3$), 25.3 (-$CH_2CH_2CH_3$), 31.0 (-$CH_2CH_2CH_3$), 44.9 (5-*C*), 46.5 (4-*C*), 127.0 (-*Ph*), 127.3 (-*Ph*), 128.9 (-*Ph*), 141.4 (-*Ph*), 142.4 (2-*C*), 175.3 (3-*C*), 209.2 (1-*C*O). HRMS (EI) Calcd for $C_{17}H_{22}O$ 242.1671, Found m/z .242.1672.



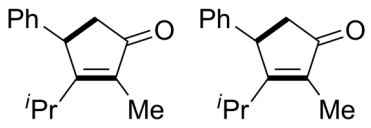
7ad

Table 2, run 3, 2,3-dibutyl-4-phenylcyclopenten-2-one (7ad): An authentic sample was prepared by using a stoichiometric amount of $Ni(cod)_2$ (0.20 mmol). To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7ad** as colorless oil (55.4 mg, 98%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.77 (t, *J* = 6.8 Hz, 3H, 2-$(CH_2)_3CH_3$), 0.87 (t, *J* = 6.8 Hz, 3H, 3-$(CH_2)_3CH_3$), 1.12-1.38 (m, 8H, -CH_2- x 4), 1.88 (m, 1H, 3-$CHH(CH_2)_2CH_3$), 2.17 (m, 2H, 2-$CH_2(CH_2)_2CH_3$), 2.25 (dd, *J* = 1.6, 18.8 Hz, 1H, -CHH-), 2.33 (m, 1H, 3-$CHH(CH_2)_2CH_3$), 2.79 (dd, *J* = 6.8, 18.8 Hz, 1H, -CHH-), 3.85 (d, *J* = 6.8 Hz, 1H, -$CH_2C(Ph)H$-), 7.01 (d, *J* = 7.2 Hz, 2H, *o*-*Ph*), 7.19 (m, 1H, *p*-*Ph*), 7.24 (t, *J* = 7.2 Hz, 2H, *m*-*Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 13.9 (-CH_3), 14.1 (-CH_3), 22.8 (-CH_2-), 22.9 (-CH_2-), 23.2 (-CH_2-), 28.7 (-CH_2-), 29.6 (-CH_2-), 31.0 (-CH_2-), 44.9 (5-*C*), 46.5 (4-*C*), 127.0 (-*Ph*), 127.4 (-*Ph*), 128.5 (-*Ph*), 141.4 (-*Ph*), 142.4 (2-*C*), 175.4 (3-*C*), 209.3 (1-*C*O). HRMS (EI) Calcd for $C_{19}H_{26}O$ 270.1984, Found m/z 270.1982.



7ae

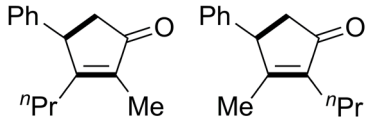
Table 2, run 4, 2,3,4-triphenylcyclopenten-2-one (7ae): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Recrystallization of the residue from hexane at -15 °C gave **7ae** as white microcrystalline (198.2 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 2.62 (dd, *J* = 2.0, 18.8 Hz, 1H, -C*H*H-), 3.23 (dd, *J* = 7.2, 18.8 Hz, 1H, -CH*H*-), 4.57 (dd, *J* = 2.0, 7.2 Hz, 1H, -CH_2C(Ph)*H*-), 7.03-7.29 (m, 15H, *Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 46.0 (5-*C*), 47.2 (4-*C*), 126.9 (-*Ph*), 127.4 (-*Ph*), 128.0 (-*Ph*), 128.2 (-*Ph*), 128.4 (-*Ph*), 128.7 (-*Ph*), 128.9 (-*Ph*), 129.2 (-*Ph*), 129.6 (-*Ph*), 131.8 (*ipso*-*Ph*), 134.8 (*ipso*-*Ph*), 140.8 (*ipso*-*Ph*), 142.2 (2-*C*), 177.0 (3-*C*), 207.5 (1-*C*O). HRMS (EI) Calcd for $C_{23}H_{18}O$ 310.1358, Found m/z 310.1352.



7af ***minor*-7af**

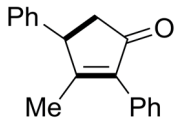
Table 2, run 5, 3-isopropyl-2-methyl-4-phenylcyclopenten-2-one (7af): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7af** as colorless oil (97.9 mg, 46%,) and its regioisomer as colorless oil (52.3 mg, 24%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.79 (d, *J* = 6.0 Hz, 3H, -CH(CH_3)$_2$), 1.06 (d, *J* = 6.0 Hz, 3H, -CH(CH_3)$_2$), 1.82 (s, 3H, -CH_3), 2.29 (d, *J* = 18.8 Hz, 1H, -C*H*H-), 2.77 (m, 1H, -C*H*(CH_3)$_2$), 2.85 (dd, *J* = 6.8, 18.8 Hz, 1H, -CH*H*-), 3.96 (m, 1H, -CH_2C(Ph)*H*-), 7.10 (d, *J* = 6.8 Hz, 2H, *o*-*Ph*), 7.20-7.28 (m, 3H, *Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 8.4 (-CH(*C*H_3)$_2$), 19.8 (-*C*H_3), 20.9 (-CH(*C*H_3)$_2$), 30.4 (-*C*H(CH_3)$_2$), 45.3 (5-*C*), 46.5 (4-*C*), 126.9 (-*Ph*), 127.6 (-*Ph*), 128.6 (-*Ph*), 136.0 (-*Ph*), 142.8 (2-*C*), 179.4 (3-*C*), 209.8 (1-*C*O). HRMS (EI) Calcd for $C_{15}H_{18}O$ 214.1358, Found m/z 214.1360. Spectral data of the regioisomer of **7af**: ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 1.15 (d, *J* = 7.2 Hz, 3H, -CH(CH_3)$_2$), 1.17 (d, *J* = 7.2 Hz, 3H, -CH(CH_3)$_2$), 1.75 (s, 3H, -CH_3), 2.20 (dd, *J* = 2.0, 18.8 Hz, 1H, -C*H*H-), 2.76 (dd, *J* = 6.8, 18.8 Hz, 1H, -CH*H*-), 2.75 (m, 1H, -C*H*(CH_3)$_2$), 3.67 (d, *J* = 6.8 Hz, 1H, -CH_2C*H*-), 7.00 (d, *J* = 7.2 Hz,

2H, *o*-*Ph*), 7.17 (m, 1H, *p*-*Ph*), 7.23 (t, *J* = 7.2 Hz, 2H, *m*-*Ph*). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$, rt): δ 15.6 (-*C*H_3), 20.1 (-CH(*C*H_3)$_2$), 20.4 (-CH(*C*H_3)$_2$), 25.0 (-*C*H(CH$_3$)$_2$), 44.9 (5-*C*), 48.9 (4-*C*), 126.9 (-*Ph*), 127.2 (-*Ph*), 128.9 (-*Ph*), 142.3 (2-*C*), 145.4 (-*Ph*), 170.3 (3-*C*), 208.5 (1-*C*O). HRMS (EI) Calcd for $C_{15}H_{18}O$ 214.1358, Found m/z 214.1355.



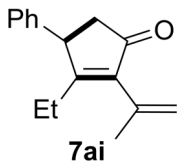
7ag ***minor*-7ag**

Table 2, run 6, 2-methyl-4-phenyl-3-propylcyclopenten-2-one (7ag): The resulting crude product was further purified by passing through a pad of SiO_2 (eluent: hexane) followed by HPLC (eluent: $CHCl_3$), yielding a mixture of the isomers of **7ag** as yellow oil (111.3 mg, 52%, major/minor = 72/28) The ratio of products was determined by NMR analysis. ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.77 (t, *J* = 7.2 Hz, 3H, -$CH_2CH_2CH_3$), 1.32-1.38 (m, 1H, -CH_2CHHCH_3), 1,45-1.52 (m, 1H, -CH_2CHHCH_3), 1.73 (d, *J* = 1.2 Hz, 3H, -CH_3), 1.90 (m, 1H, -C*H*HCH_2CH_3), 2.22 (m, 1H, -CH*H*CH_2CH_3), 2.23-2.37 (m, 1H, -C*H*H-), 2.85 (dd, *J* = 2.0, 18.8 Hz, 1H, -CH*H*-), 3.84 (m, *J* = 7.2 Hz, 1H, -CH_2C(Ph)*H*-), 6.80 (d, *J* = 6.8 Hz, 2H, *o*-*Ph*), 7.14-7.23 (m, 3H, *Ph*). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$, rt): δ 8.3 (-$CH_2CH_2CH_3$), 14.1 (-CH_3), 20.5 (-$CH_2CH_2CH_3$), 31.1 (-$CH_2CH_2CH_3$), 44.7 (5-*C*), 46.9 (4-*C*), 127.0 (-*Ph*), 127.3 (-*Ph*), 127.4 (-*Ph*), 128.9 (-*Ph*), 142.2 (2-*C*), 175.2 (3-*C*), 209.4 (1-*C*O). HRMS (EI) Calcd for $C_{15}H_{18}O$ 214.1358, Found m/z 214.1358. Spectral data of minor product: ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.83 (t, *J* = 7.2 Hz, 3H, -$CH_2CH_2CH_3$), 1.38 (m, 2H, -$CH_2CH_2CH_3$), 1.74 (s, 3H, -CH_3), 2.18 (m, 2H, -C$H_2CH_2CH_3$), 2.23-2.37 (m, 1H, -C*H*H-), 2.85-2.93 (m, 1H, -CH*H*-), 3.60 (m, *J* = 7.2 Hz, 1H, -CH_2C(Ph)*H*-), 7.04 (d, *J* = 6.8 Hz, 2H, *o*-*Ph*), 7.18-7.29 (m, 3H, *Ph*). $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$, rt): δ 14.0 (-$CH_2CH_2CH_3$), 15.5 (-CH_3), 21.7 (-$CH_2CH_2CH_3$), 25.2 (-$CH_2CH_2CH_3$), 44.7 (5-*C*), 49.1 (4-*C*), 127.0 (-*Ph*), 127.0 (-*Ph*), 128.9 (-*Ph*), 137.2 (-*Ph*), 141.3 (2-*C*), 171.9 (3-*C*), 208.9 (1-*C*O).



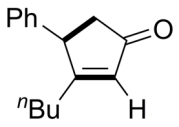
7ah

Table 2, run 7, 3-methyl-2,4-diphenylcyclopenten-2-one (7ah): Monitoring of the crude product by GC revealed the formation of the cycloaddition product as a mixture of regioisomers mixture (major/minor = 97/3). The resulting crude product was further purified by passing through a pad of SiO_2 (eluent: hexane) followed by HPLC (eluent: $CHCl_3$), yielding the major regioisomer **7ah** as colorless oil (159.7 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 1.93 (s, 3H, -C*H*$_3$), 2.52 (dd, *J* = 2.0, 19.2 Hz, 1H, -C*H*H-), 3.03 (dd, *J* = 7.2, 19.2 Hz, 1H, -CH*H*-), 3.92 (br d, *J* = 7.2 Hz, 1H, -CH$_2$C(Ph)*H*), 7.13 (d, *J* = 7.2 Hz, 2H, *Ph*), 7.14-7.40 (m, 8H, *Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 16.8 (-*C*H$_3$), 45.1 (5-*C*), 49.2 (4-*C*), 127.3 (-*Ph*), 127.5 (-*Ph*), 127.9 (-*Ph*), 128.4 (-*Ph*), 129.2 (-*Ph*), 129.3 (-*Ph*), 131.7 (-*Ph*), 140.8 (-*Ph*), 141.9 (2-*C*), 173.3 (3-*C*), 206.8 (1-*C*O). HRMS (EI) Calcd for $C_{18}H_{16}O$ 248.1201, Found m/z 248.1197.



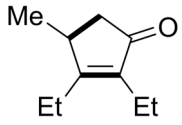
7ai

Table 2, Entry 8, 3-ethyl-4-phenyl-2-(1-propen-2-yl)cyclopenten-2-one (7ai): Monitoring of the crude product by GC revealed the formation of the cycloaddition product as a mixture of regioisomers mixture (major/minor = >99/1). To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave the major regioisomer **7ai** as colorless oil (43.0 mg, 19%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 1.18 (t, *J* = 7.6 Hz, 3H, -C*H*$_3$), 2.17 (dq, 1H, 3-C*H*HCH$_3$), 2.17 (s, 3H, -C*H*$_3$), 2.57 (dd, *J* = 2.0, 18.8 Hz, 1H, -CH*H*C(Ph)H-), 2.75 (dq, *J* = 7.6, 14.0 Hz, 1H, 3-CH*H*CH$_3$), 3.11 (dd, *J* = 6.8, 18.8 Hz, 1H, -C*H*HC(Ph)H-), 4.20 (br d, *J* = 6.8 Hz, 1H, -CH$_2$C(Ph)*H*-), 5.05 (s, 1H, =C*H*H), 5.43 (s, 1H, =CH*H*), 7.30 (d, *J* = 6.8 Hz, 2H, -*Ph*), 7.45 (m, 1H, -*Ph*), 7.51 (m, 2H, -*Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.3 (-*C*H$_3$), 22.5 (-*C*H$_3$), 22.6 (-*C*H$_2$CH$_3$), 45.0 (5-*C*), 46.2 (4-*C*), 116.7 (-C=*C*H$_2$), 127.1 (-*Ph*), 127.4 (-*Ph*), 129.0 (-*Ph*), 137.4 (2-*C*), 141.9 (-*Ph*), 143.3 (-*C*=CH$_2$), 177.0 (3-*C*), 207.5 (1-*C*O). HRMS (EI) Calcd for $C_{16}H_{18}O$ 226.1358, Found m/z 226.1361.



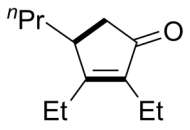
7aj

Table 2, Entry 9, 3-butyl-4-phenylcyclopenten-2-one (7aj): Monitoring of the crude product by GC revealed the formation of the cycloaddition product as a mixture of regioisomers mixture (42% yield, major/minor = 76/24). The resulting crude product was further purified by passing through a pad of SiO_2 (eluent: hexane) followed by HPLC (eluent: $CHCl_3$), yielding the major regioisomer **7aj** as colorless oil (64.2 mg, 30%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.79 (t, *J* = 7.2 Hz, 3H, -CH_2 $CH_2CH_2CH_3$), 1.22 (m, 2H, -CH_2 $CH_2CH_2CH_3$), 1.41 (m, 2H, -$CH_2CH_2CH_2CH_3$), 2.05 (m, 2H, -$CH_2CH_2CH_2CH_3$), 2.32 (d, *J* = 18.8 Hz, 1H, -C*H*HCH-), 2.84 (dd, *J* = 6.4, 18.8 Hz, 1H, -CH*H*CH-), 3.88 (m, *J* = 6.4Hz., 1H, -CH_2C*H*-), 6.02 (s, 1H, -C*H*=C-), 7.04 (m, *J* = 6.8 Hz, 2H, *o-Ph*), 7.17-7.24 (m, 3H, *Ph*). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 13.8 (-$CH_2CH_2CH_2CH_3$), 22.3 (-$CH_2CH_2CH_2CH_3$), 29.1 (-$CH_2CH_2CH_2CH_3$), 31.2 (-$CH_2CH_2CH_2CH_3$), 45.7 (-CH_2CHPh), 49.6 (-CH_2CHPh), 126.9 (-*Ph*), 127.2 (-*Ph*), 128.9 (-*Ph*), 129.8 (-C=*C*-CO), 141.5 (-*Ph*), 184.6 (-*C*=C-CO), 209.1 (-*C*O). $C_{15}H_{18}O$ 214.1358, Found m/z 214.1355.



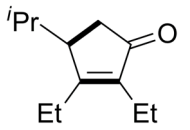
7ba

Table 2, Entry 14, 2,3-diethyl-4-methylcyclopenten-2-one (7ba): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7ba** as colorless oil (260.0 mg, 86%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.93 (t, *J* = 7.2 Hz, 3H, 2-CH_2CH_3), 1.05 (t, *J* = 7.2 Hz, 3H, 3-CH_2CH_3), 1.08 (d, *J* = 7.2 Hz, 3H, -CH_3), 1.90 (dd, *J* = 2.0, 18.4 Hz, 1H, 3-C*H*HCH-), 2.09 (q, *J* = 7.2 Hz, 2H, 2-CH_2CH_3), 2.25 (m, 1H, 3-C*H*HCH_3), 2.45 (m, 1H, -C*H*HCH_3), 2.60 (dd, *J* = 7.2 ,18.4 Hz, 1H, -C*H*H-), 2.81 (m, 1H, -CH_2C(Me)*H*-). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.2 (-CH_2CH_3), 13.4 (-CH_2CH_3), 16.3 (-CH_2CH_3), 19.1 (-CH_3), 21.4 (-CH_2CH_3), 34.4 (5-*C*), 43.2 (4-*C*), 140.1 (2-*C*), 178.5 (3-*C*), 209.0 (1-*C*O). HRMS (EI) Calcd for $C_{10}H_{16}O$ 152.1201, Found m/z .152.1194.



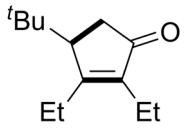
7ca

Table 2, Entry 15, 2,3-diethyl-4-propylcyclopenten-2-one (7ca): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7ca** as colorless oil (141.2 mg, 79%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.86 (t, *J* = 7.2 Hz, 3H, 2-CH_2CH_3), 0.86 (t, *J* = 7.6 Hz, 3H, 3-CH_2CH_3), 1.00 (t, *J* = 7.6 Hz, 3H, -$CH_2CH_2CH_3$), 1.00-1.09 (m, 1H, -$CHHCH_2CH_3$), 1.09-1.27 (m, 2H, -$CH_2CH_2CH_3$), 1.58-1.67 (m, 1H, -$CHHCH_2CH_3$), 1.93 (dd, *J* = 2.0 ,18.4 Hz, 1H, -C*H*H-), 2.04 (q, *J* = 7.6 Hz, 2H, 2-CH_2CH_3), 2.21 (m, 1H, 3-$CHHCH_3$), 2.40 (dd, *J* = 6.8, 18.8 Hz, 1H, -CH*H*-), 2.48 (m, 1H, -$CHHCH_3$), 2.67 (m, 1H, -$CH_2C(^{n}Pr)H$-). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.3 (-CH_3), 13.4 (-CH_3), 14.1 (-CH_3), 16.3 (-CH_2-), 20.4 (-CH_2CH_3), 21.6 (-CH_2CH_3), 35.1 (-$CH_2C_2H_5$), 39.6 (5-*C*), 40.7 (4-*C*), 141.3 (2-*C*), 177.6 (3-*C*), 209.1 (1-*C*O). HRMS (EI) Calcd for $C_{12}H_{20}O$,180.1514, Found m/z 180.1514.



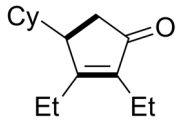
7da

Table 2, Entry 16, 2,3-diethyl-4-isopropylcyclopenten-2-one (7da): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7da** as colorless oil (151.3 mg, 84%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.53 (d, *J* = 6.8 Hz, 3H, -$CH(CH_3)_2$), 0.91 (t, *J* = 7.6 Hz, 3H, 2-CH_2CH_3), 0.94 (d, *J* = 6.8 Hz, 3H, -$CH(CH_3)_2$), 1.06 (t, *J* = 7.6 Hz, 3H, 3-CH_2CH_3), 2.01-2.23, (m, 6H, 2-CH_2CH_3, 3-$CHHCH_3$, -CH_2CH-, -$CH(CH_3)_2$), 2.49 (dq, *J* = 7.6, 15.2 Hz 1H, 3-$CHHCH_3$), 2.81 (m, 1H, -$CH_2C(^{i}Pr)H$-). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.1 (-CH_3), 13.4 (-CH_3), 14.4 (-$CH(CH_3)_2$), 16.1 (-$CH(CH_3)_2$), 21.4 (-CH_2CH_3), 21.7 (-CH_2CH_3), 27.3 (-$CH(CH_3)_2$), 34.8 (5-*C*), 44.9 (4-*C*), 142.1 (2-*C*), 176.7 (3-*C*), 209.2 (1-*C*O). HRMS (EI) Calcd for $C_{12}H_{20}O$ 180.1514, Found m/z 180.1514.



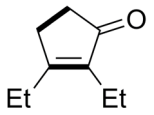
7ea

Table 2, Entry 17, 2,3-diethyl-4-isopropylcyclopenten-2-one (7ea): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7ea** as colorless oil (110.6 mg, 57%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.88 (s, 9H, -C(CH_3)$_3$), 0.90 (t, *J* = 7.6 Hz, 3H, 2-CH_2CH_3), 1.06 (t, *J* = 7.6 Hz, 3H, 3-CH_2CH_3), 2.13 (q, 2H, 2-CH_2CH_3), 2.17 (dd, *J* = 2.0 ,18.8 Hz, 1H, -C*H*HCH-), 2.27 (dd, *J* = 6.4 ,18.8 Hz, 1H, -C*H*HCH-), 2.38 (dq, *J* = 7.6, 11.6 Hz, 1H, 3-CH*H*CH_3), 2.60 (dq, *J* = 7.6, 11.6 Hz, 1H, 3-CH*H*CH_3), 2.66 (br d, *J* = 6.4 Hz, 1H, -CH_2CH-). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.7 (-CH_2CH_3), 13.5 (-CH_2CH_3), 16.4 (-CH_2CH_3), 24.2 (-CH_2CH_3), 28.3 (-C(CH_3)$_3$), 34.4 (-C(CH_3)$_3$), 39.6 (5-*C*), 49.8 (4-*C*), 143.7 (2-*C*), 176.7 (3-*C*), 209.1 (1-*C*O). HRMS (EI) Calcd for $C_{13}H_{22}O$ 1941671, Found m/z .194.1669.



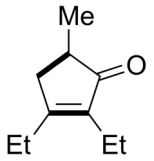
7fa

Table 2, Entry 18, 4-cyclohexyl-2,3-diethylcyclopenten-2-one (7fa): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7fa** as colorless oil (113.9 mg, 49%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.74 (m, 1H, -*Cy*), 0.92 (t, *J* = 7.6 Hz, 3H, 2-CH_2CH_3), 1.07 (t, *J* = 7.6 Hz, 3H, 3-CH_2CH_3), 1.00-1.28 (m, 5H, -*Cy*), 1.55-1.75 (m, 5H, -*Cy*), 2.09-2.25 (m, 5H, -CH_2C(Cy)H-, 2-CH_2CH_3, 3-C*H*HCH_3), 2.51 (dq, *J* = 7.6, 14.0 Hz 1H, 3-CH*H*CH_3), 2.78 (m, 1H, -CH_2C(Cy)*H*-). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.3 (-CH_3), 13.5 (-CH_3), 16.2 (-Cy), 21.7 (-CH_2CH_3), 25.2 (-CH_2CH_3), 26.1 (-Cy), 26.4 (-Cy), 26.8 (-Cy), 32.5 (-Cy), 36.2 (5-*C*), 38.2 (-Cy), 44.8 (4-*C*), 142.2 (2-*C*), 176.4 (3-*C*), 209.4 (1-*C*O). HRMS (EI) Calcd for $C_{15}H_{24}O$ 220.1827, Found m/z 220.1819.



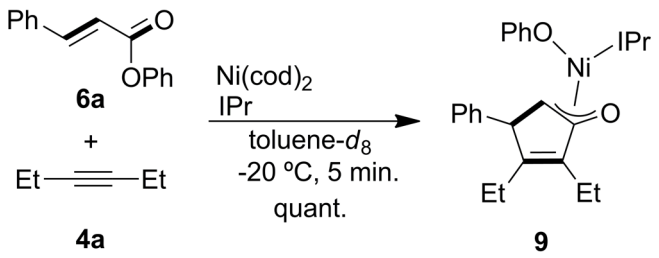
7ga

Table 2, Entry 19, 2,3-diethylcyclopenten-2-one (7ga): An authentic sample was prepared by using a stoichiometric amount of $Ni(cod)_2$ (0.50 mmol). To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) and HPLC (eluent: $CHCl_3$) gave **7ga** as colorless oil (15.6 mg, 23%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.97 (t, *J* = 7.6 Hz, 3H, 2-CH_2CH_3), 1.14 (t, *J* = 7.6 Hz, 3H, 3-CH_2CH_3), 2.17 (q, *J* = 7.6 Hz, 2H, 2-CH_2CH_3), 2.34 (m, 2H, 4-CH_2-), 2.43 (q, *J* = 7.6 Hz, 2H, 3-CH_2CH_3), 2.48 (m, 2H, 5-CH_2-). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.1 (-CH_3), 13.3 (-CH_3), 16.2 (-CH_2CH_3), 24.1 (-CH_2CH_3), 28.5 (5-*C*) 34.3 (4-*C*), 141.3 (2-*C*), 174.8 (3-*C*), 210.1 (*C*O). HRMS (EI) Calcd for $C_9H_{14}O$ 138.1045, Found m/z 138.1048..

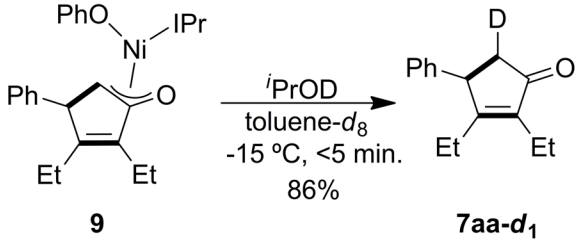


7ha

Table 2, Entry 20, 2,3-diethyl-5-methylcyclopenten-2-one (7ha): To the resulting crude product was added NaOH*aq* (1.0 M, 50 mL), and then the organic layer was extracted with diethyl ether (50 mL x 3). The combined organic layer was dried over magnesium sulfate, filtered and concentrated *in vacuo*. Further purification by silica gel chromatography (eluent: hexane/AcOEt = 95/5) gave **7ha** as colorless oil (94.2 mg, 62%). ^{1}H NMR (400 MHz, $CDCl_3$, rt): δ 0.96 (t, *J* = 7.2 Hz, 3H, 2-CH_2CH_3), 1.12 (t, *J* = 7.2 Hz, 3H, 3-CH_2CH_3), 1.14 (d, *J* = 7.2 Hz, 3H, -CH_3), 2.07 (br d, *J* = 18.0 Hz, 1H, -C*H*H-), 2.19 (q, *J* = 7.2 Hz, 2H, 3-CH_2CH_3), 2.33 (ddq, *J* = 2.4, 7.2, 7.2 Hz, 1H, -CH_2C(Me)*H*), 2.42 (q, *J* = 7.2 Hz, 2H, 2-CH_2CH_3), 2.75 (dd, *J* = 7.2 ,18.0 Hz, 1H, -C*H*H-). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$, rt): δ 12.1 (-CH_2CH_3), 13.4 (-CH_2CH_3), 16.3 (-CH_2CH_3), 16.6 (-CH_3), 23.9 (-CH_2CH_3), 37.7 (4-*C*), 39.5 (5-*C*), 139.9 (2-*C*), 172.9 (3-*C*), 212.4 (1-*C*O). HRMS (EI) Calcd for $C_{10}H_{16}O$ 152.1201, Found m/z .152.1193.

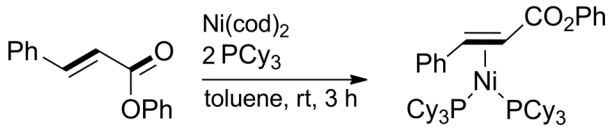


Observation of a reaction intermediate of 6a and 4a in the presence of Ni(cod)$_2$ and IPr: To a toluene-d_8 solution of Ni(cod)$_2$ (22.0 mg, 0.08 mmol) and IPr (31.3 mg, 0.08 mmol) was added a toluene-d_8 solution of **6a** (18.0 mg, 0.08 mmol) and **4a** (6.6 mg, 0.08 mmol) at -35 °C. The color of the solution turned into deep red. NMR observation at -20 ºC revealed the quantitative formation of **9**. ^{1}H NMR (600 MHz, toluene-d_8, -20 °C): δ 0.89−0.98 (m, 6H, -CH_3), 1.07 (d, J = 6.0 Hz, 6H, -CH_3 groups of IPr), 1.13 (d, J = 6.0 Hz, 6H, -CH_3 groups of IPr), 1.14 , (m, 1H, -CHHCH$_3$), 1.20 (d, J = 6.0 Hz, 6H, -CH_3 groups of IPr), 1.74 (d, J = 6.0 Hz, 6H, -CH_3 groups of IPr), 1.75 (m, 1H, -CHHCH$_3$), 1.86 (m, 1H, -CHHCH$_3$), 2.20 (m, 1H, -CHHCH$_3$), 2.54 (m, 2H, -CH(CH$_3$)$_2$), 2.58 (s, 1H, -CHPh), 3.54 (m, 2H, -CH(CH$_3$)$_2$), 5.03 (s, 1H, -CHCO), 6.34 (d, J = 6.6 Hz, 2H, *o*-Ph), 6.52 (s, 2H, =CH-), 6.58 (d, J = 7.2 Hz, 2H, o-Ph), 6.66 (t, J = 6.6 Hz, 1H, *p*-Ph), 6.94 (m, 1H, *p*-Ph), 7.00 (m, 4H, *m*-Ph), 7.12 (m, 4H, *m*-Ph (IPr)), 7.38 (m, 2H, *p*-Ph (IPr)). ^{13}C{^{1}H} NMR (150 MHz, toluene-d_8, -20 °C): δ 13.9 (-CH$_2$*C*H$_3$), 14.1 (-CH$_2$*C*H$_3$), 16.1 (-*C*H$_2$-), 20.0 (-*C*H$_2$-), 22.6 (-*C*H$_3$), 23.9 (-*C*H$_3$), 25.4 (-*C*H$_3$), 26.0 (-*C*H$_3$), 28.7 (-*C*H(CH$_3$)$_2$), 28.9 (-*C*H(CH$_3$)$_2$), 52.6 (-*C*(Ph)H-), 81.8 (-*C*HC(O)-), 124.1 (=*C*H-), 124.3 (*m*-Ph), 124.4 (*m*-Ph), 129.8 (*p*-Ph), 136.5 (*ipso*-Ph), 137.6 (C(CO)=C), 139.7 (*ipso*-Ph), 146.4 (*o*-Ph (IPr)), 146.5 (*o*-Ph (IPr)), 157.0 (C(CO)=C), 162.9 (CHCO), 170.7 (*ipso*-OPh), 176.9 (N*C*N).



Reaction of 9 with ^{i}PrOD-d_1: Treatment of **9** with ^{i}PrOD (>98% D) at -15 ºC gave **7aa-d_1** (98% D) in 86% yield. ^{1}H (and ^{2}D) NMR observation of the reaction mixture revealed that deuterium was selectively incorporated at the 5-position.

Isolation of (η^2-(*E*)-PhCH=CHCO$_2$Ph)Ni(PCy$_3$)$_2$



6a 92%

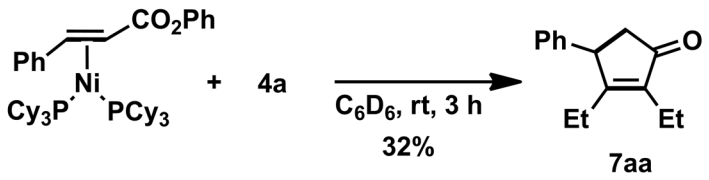
To toluene solution (5 mL) of Ni(cod)$_2$ (128.3 mg, 0.50 mmol) and PCy$_3$ (281.0 mg, 1.00 mmol) was added **6a** (128.3 mg, 0.53 mmol) at room temperature. The solution changed from yellow to red. The reaction mixture was stirred for 3 h and then concentration in *vacuo*. The residue was dissolved in toluene followed by the concentration in *vacuo* again. All volatiles were removed under reduced pressure, and then the deep red residue was purified by recrystallization from toluene/hexane at -35 ºC, affording (η^2-(*E*)-PhCH=CHCO$_2$Ph)Ni(PCy$_3$)$_2$ (248.9 mg, 60%) as a red solid. The resulting supernatant was concentrated again, and then the recrystallization procedure was repeated twice, yielding another title complex (130.5 mg, 31%). The title compound was found to be intact in solution at room temperature. ^{1}H NMR (400 MHz, C$_6$D$_6$, rt): δ 1.00 (m, 2H, -*Cy*), 1.16−1.41 (m, 20H, -*Cy*), 1.56−1.92 (m, 32H, -*Cy*), 1.96−2.10 (m, 4H, -*Cy*), 2.16−2.33 (m, 8H, -*Cy*), 3.40 (m, *J* = 9.2 Hz, 1H, C(Ph)*H*=C(CO$_2$Ph)H), 4.17 (m, 1H, C(Ph)H=C(CO$_2$Ph)*H*), 6.94 (m, 1H, *p*-*Ph*), 7.06 (m, 1H, *p*-*Ph*), 7.18 (m, 4H, *m*-*Ph* x 2), 7.40 (d, *J* = 7.2 Hz, 2H, *o*-*Ph*), 7.64 (d, *J* = 6.8 Hz, 2H, *o*-*Ph*). ^{13}C{^{1}H} NMR (100 MHz, C$_6$D$_6$, rt): δ 30.3 (-*Cy*), 30.5 (-*Cy*), 31.2 (d, *J* = 9.9 Hz, -*Cy*), 31.4 (d, *J* = 7.6 Hz, -*Cy*), 31.6 (d, *J* = 8.6 Hz, -*Cy*), 31.9 (d, *J* = 6.9 Hz, -*Cy*), 33.3 (-*Cy*), 34.5 (-*Cy*), 39.3 (-*Cy*), 40.3 (-*Cy*), 48.5 (d, *J* = 12.2 Hz, *C*(Ph)H=C(CO$_2$Ph)H), 51.9 (d, *J* = 19.2 Hz, C(Ph)H=*C*(CO$_2$Ph)H), 122.5 (-*Ph*), 123.5 (-*Ph*), 124.2 (-*Ph*), 128.7 (-*Ph*), 129.0 (-*Ph*), 129.3 (-*Ph*), 148.2 (ipso-*Ph*), 152.3 (*ipso*-*OPh*), 169.8 (*C*O). ^{31}P{^{1}H} NMR (109 MHz, C$_6$D$_6$, rt): δ 31.9 (m). Anal. Calcd for C$_{51}$H$_{78}$NiO$_2$P$_2$: C, 72.59; H, 9.32. Found: C, 72.94; H, 9.49. X-ray data for (η^2-(*E*)-PhCH=CHCO$_2$Ph)Ni(PCy$_3$)$_2$. *M* = 843.78**,** yellow, Monoclinic, *P*2$_1$/*n* (No. 14), a = 13.9259(12) Å, b = 18.0957(14) Å, c = 18.4593(17) Å, β = 90.150(3)°, V = 4651.7(7) Å^3, Z = 4, D$_{calcd}$ = 1.205 g/cm^3, T = −150 (2)°C, R_1(*wR*$_2$) = 0.061 (0.131). Figure S1 shows an ORTEP drawing of the title complex.

Isolation of (η^2-(*E*)-PhCH=CHCO$_2$Ph)$_2$Ni(PCy$_3$)

6a 86%

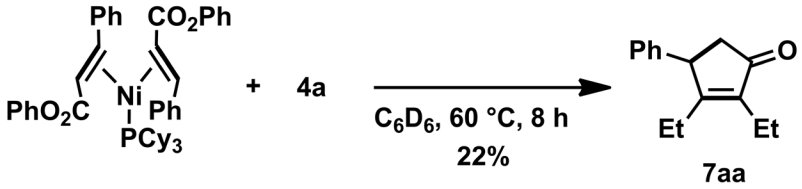
To toluene solution (5 mL) of Ni(cod)$_2$ (276.3 mg, 1.00 mmol) and PCy$_3$ (286.4 mg, 1.02 mmol) was added **6a** (452.3 mg, 2.02 mmol) at room temperature. The solution changed from yellow to bright red. The reaction mixture was stirred for 1 h and then concentration in *vacuo*. The residue was washed by hexane followed by the concentration in *vacuo* again. All volatiles were removed under reduced pressure, and then the orange residue was purified by recrystallization from toluene/hexane at -35 ºC, affording (η^2-(*E*)-PhCH=CHCO$_2$Ph)$_2$Ni(PCy$_3$) (675.5 mg, 86%) as an orange microcrystalline. The title compound was found to be intact in solution at room temperature. ^{1}H NMR (400 MHz, C_6D_6, rt): δ 0.78–1.04 (m, 2H, -*Cy*), 1.04-1.50 (m, 14H, -*Cy*), 1.50-1.84 (m, 11H, -*Cy*), 1.90-2.14 (m, 4H, -*Cy*), 2.27-2.46 (m, 2H, -*Cy*), 3.93 (d, *J* = 9.6 Hz, 1H, C(Ph)*H*=C(CO$_2$Ph)H), 4.45 (dd, *J* = 10.0, 10.0 Hz, 1H, C(Ph)H=C(CO$_2$Ph)*H*), 5.33 (dd, *J* = 10.4, 10.4 Hz, 1H, C(Ph)H=C(CO$_2$Ph)*H*), 6.21 (d, *J* = 10.8 Hz, 1H, C(Ph)*H*=C(CO$_2$Ph)H), 6.21 (m, 1H, -*Ph*), 6.74-7.05 (m, 10H, -*Ph*), 7.21 (m, 5H, -*Ph*), 7.29 (d, *J* = 8.0 Hz, 2H, *o*-*Ph*), 7.60 (d, *J* = 7.2 Hz, 2H, *o*-*Ph*). ^{13}C{^{1}H} NMR (100 MHz, C_6D_6, rt): δ 26.9 (-*Cy*), 27.5 (d, *J* = 9.9 Hz, -*Cy*), 28.0 (d, *J* = 9.1 Hz, -*Cy*), 30.3 (-*Cy*), 30.7 (-*Cy*), 34.4 (d, *J* = 15.3 Hz, -*Cy*), 55.1 (C(Ph)H=*C*(CO$_2$Ph)H), 69.1 (*C*(Ph)H=C(CO$_2$Ph)H), 70.1 (d, *J* = 6.9 Hz, C(Ph)H=*C*(CO$_2$Ph)H), 75.1 (d, *J* = 7.2 Hz, *C*(Ph)H=*C*(CO$_2$Ph)H), 122.6 (-*Ph*), 123.0 (-*Ph*), 124.8 (-*Ph*), 125.0 (-*Ph*), 125.4 (-*Ph*), 126.6 (-*Ph*), 126.7 (-*Ph*), 128.5 (-*Ph*), 128.8 (-*Ph*), 129.0 (-*Ph*), 129.4 (-*Ph*), 140.2 (*ipso*-*Ph*), 142.6 (*ipso*-*Ph*), 151.7 (*ipso*-*OPh*), 151.9 (*ipso*-*OPh*), 167.4 (*C*O), 169.8 (*C*O), one peak attributed Phenyl unit might be obscured by the solvent signal. ^{31}P{^{1}H} NMR (109 MHz, C_6D_6, rt): δ 27.8 (s). Anal. Calcd for $C_{48}H_{57}NiO_4P$: C, 73.20; H, 7.29. Found: C, 72.92; H, 7.58. X-ray data for (η^2-(*E*)-PhCH=CHCO$_2$Ph)$_2$Ni(PCy$_3$). *M* = 787.62**,** yellow, Triclinic, *P*-1 (No. 2), a = 11.9158(9) Å, b = 12.1881(9) Å, c = 14.9957(10) Å, α = 87.464(3)°, β = 83.542(3)°, γ = 69.604(2)°, V = 2028.3(2) Å^3, Z = 2, D_{calcd} = 1.290 g/cm^3, T = −150 (2)°C, $R_1(wR_2)$ = 0.072 (0.152). Figure S2 shows an ORTEP drawing of the title complex.

Reaction of (η^2-(*E*)-PhCH=CHCO$_2$Ph)Ni(PCy$_3$)$_2$ with 3-hexyne



To a C_6D_6 solution of (η^2-(*E*)-PhCH=CHCO$_2$Ph)Ni(PCy$_3$)$_2$ (34.0 mg, 0.04 mmol) was added 3-hexyne at room temperature. The reaction mixture was transferred into a J-Young NMR tube. Monitoring of the reaction by NMR spectroscopy demonstrated that all of (η^2-(*E*)-PhCH=CHCO$_2$Ph)Ni(PCy$_3$)$_2$ were consumed within 3 hours, whereas no significant intermediates were observed. The yield of **7aa** (2.7 mg, 32%) was determined by GC analysis using tetradecane as an internal standard.

Reaction of (η^2-(*E*)-PhCH=CHCO$_2$Ph)$_2$Ni(PCy$_3$) with 3-hexyne



To a C_6D_6 solution of (η^2-(*E*)-PhCH=CHCO$_2$Ph)$_2$Ni(PCy$_3$) (31.0 mg, 0.04 mmol) was added 3-hexyne at room temperature. The reaction mixture was transferred into a J-Young NMR tube and kept at 60 ºC. Monitoring of the reaction by NMR spectroscopy demonstrated that all of (η^2-(*E*)-PhCH=CH-CO$_2$Ph)$_2$Ni(PCy$_3$) was consumed after 8 hours, whereas no significant intermediates were observed. The reaction did not proceed at room temperature. The yield of **7aa** (2.0 mg, 22%) was determined by GC analysis using tetradecane as an internal standard.

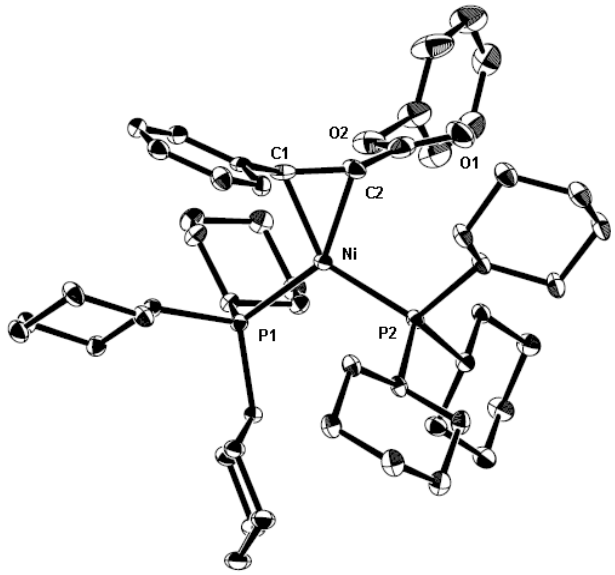


Figure S1. ORTEP drawing of (η^2-(*E*)-PhCH=CHCO$_2$Ph)Ni-(PCy$_3$)$_2$ with thermal ellipsoids at the 30% probability level. H atoms are omitted for clarity.

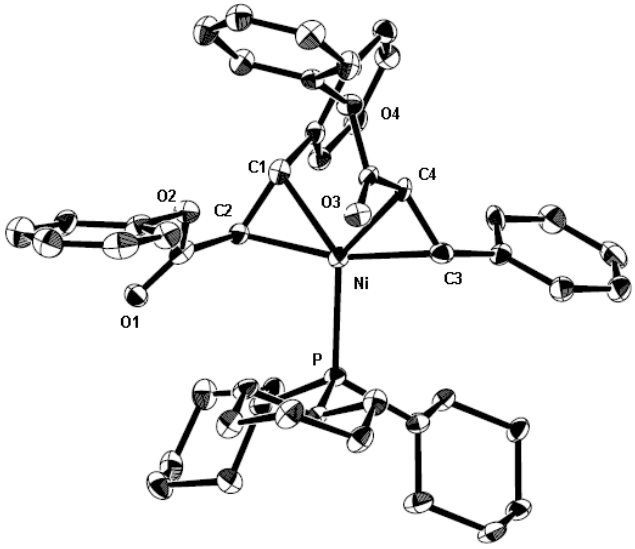
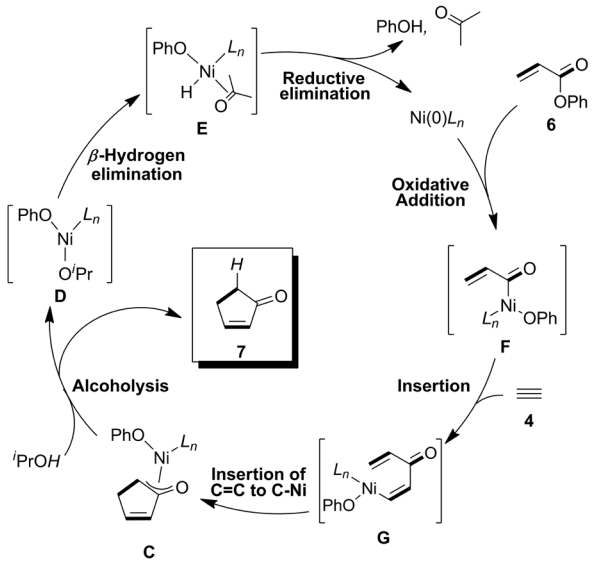
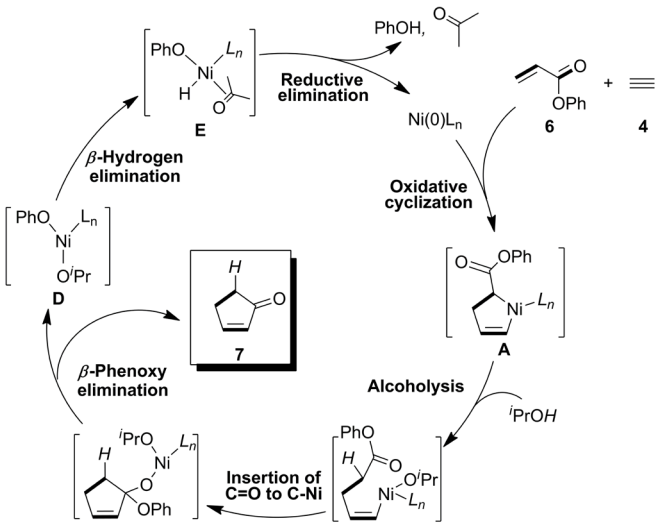


Figure S2. ORTEP drawing of (η^2-(*E*)-PhCH=CHCO$_2$Ph)$_2$Ni-(PCy$_3$) with thermal ellipsoids at the 30% probability level. H atoms are omitted for clarity.



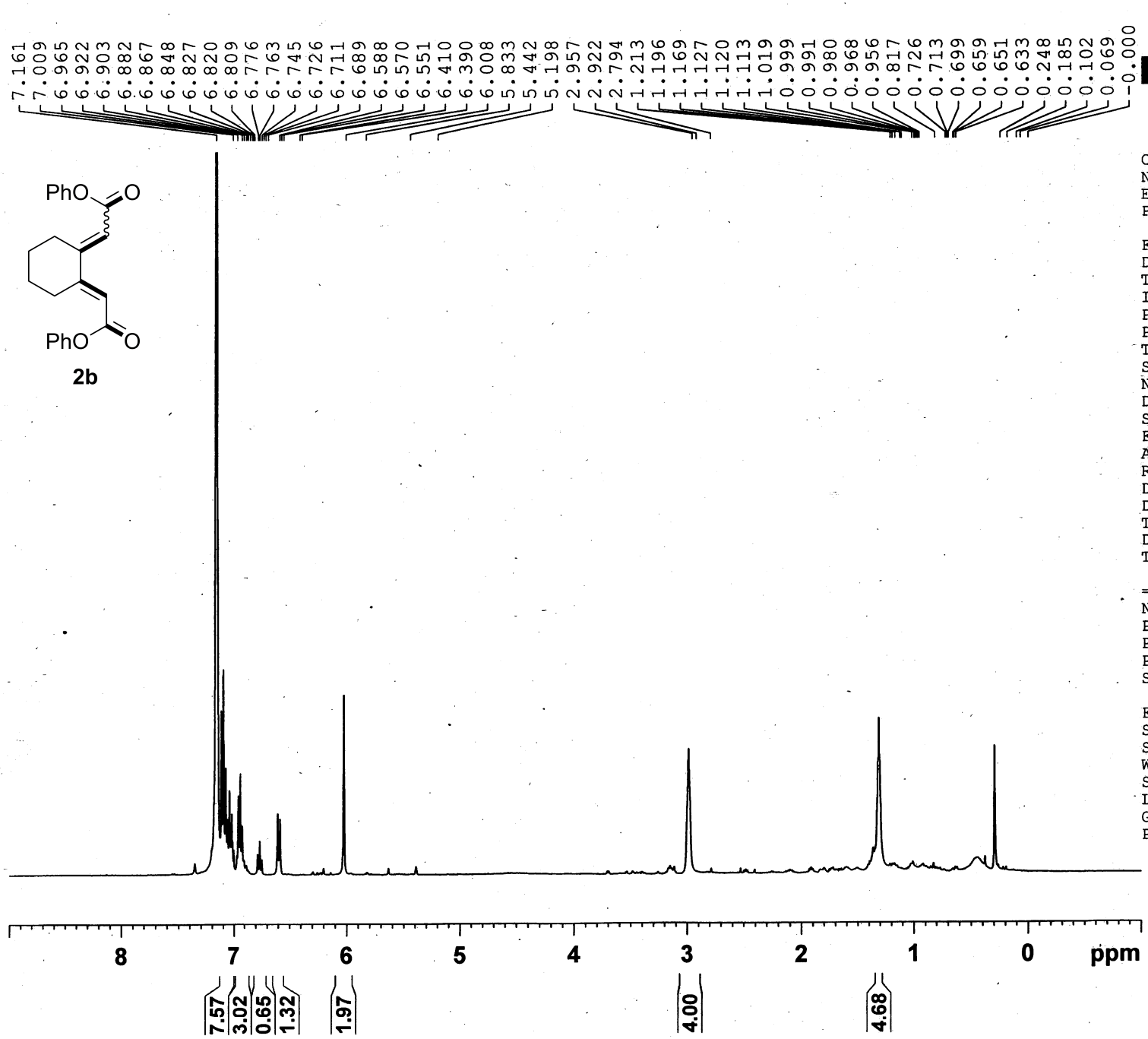
Scheme S1. An alternative reaction mechanism, involving a C−O bond activation of phenylester, for the Ni-catalyzed dephenoxylative cycloaddition reaction



Scheme S2. An alternative reaction mechanism, involving alcoholysis of **A** followed by ester carbonyl insertion into the Ni−C bond and β-phenoxy elimination, for the Ni-catalyzed dephenoxylative cycloaddition reaction

References

[1] Takahashi, T.; Xi, Z.; Nishihara, Y.; Huo, S.; Kasai, K; Aoyagi, K.; Denisov, V.; Negishi, E. *Tetrahedron*, **1997**, *53*, 9123.



BRUKER

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PROCNO 1

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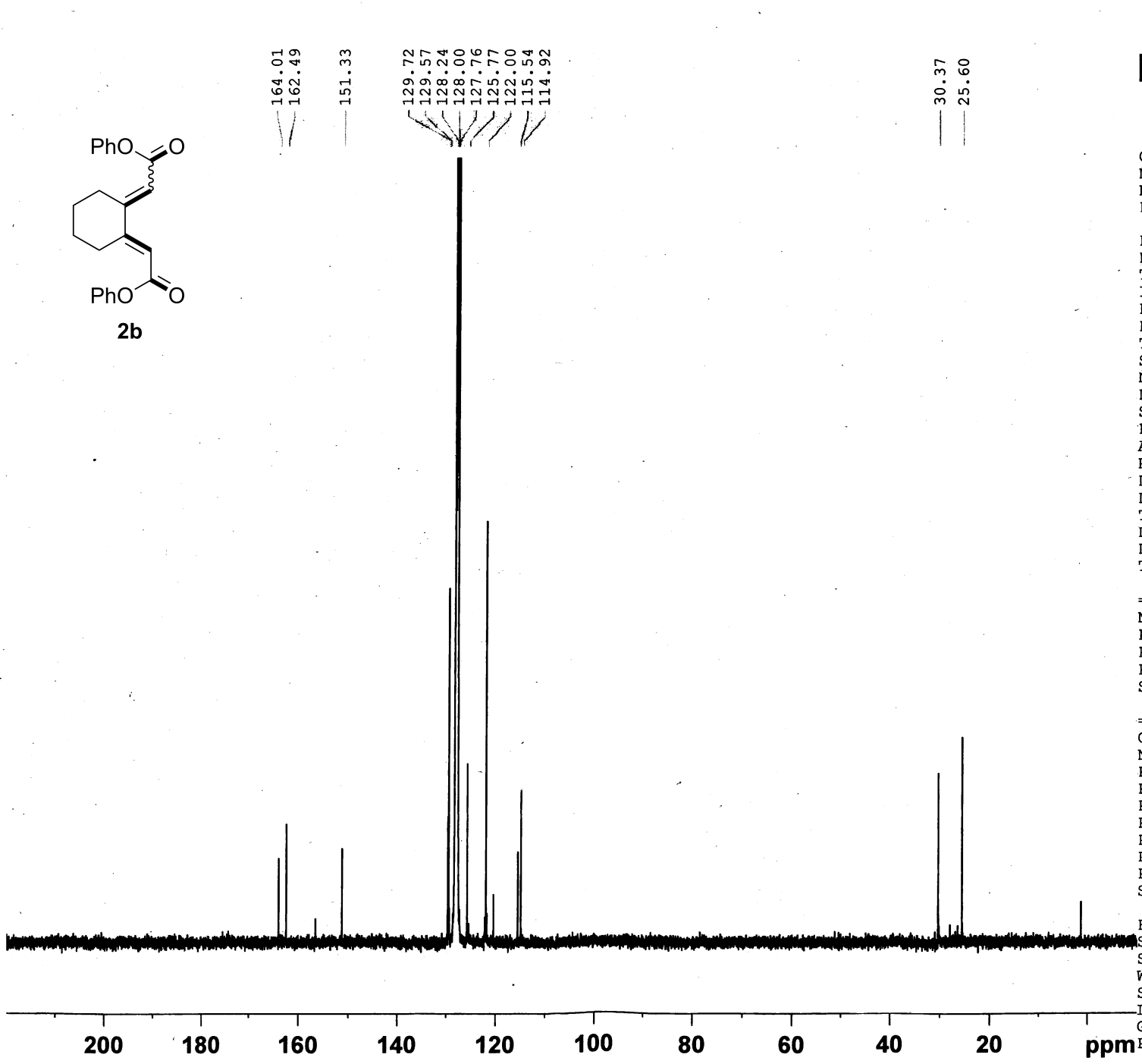

BRUKER

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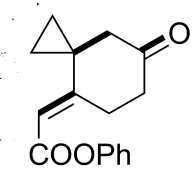

3

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PROCNO 1

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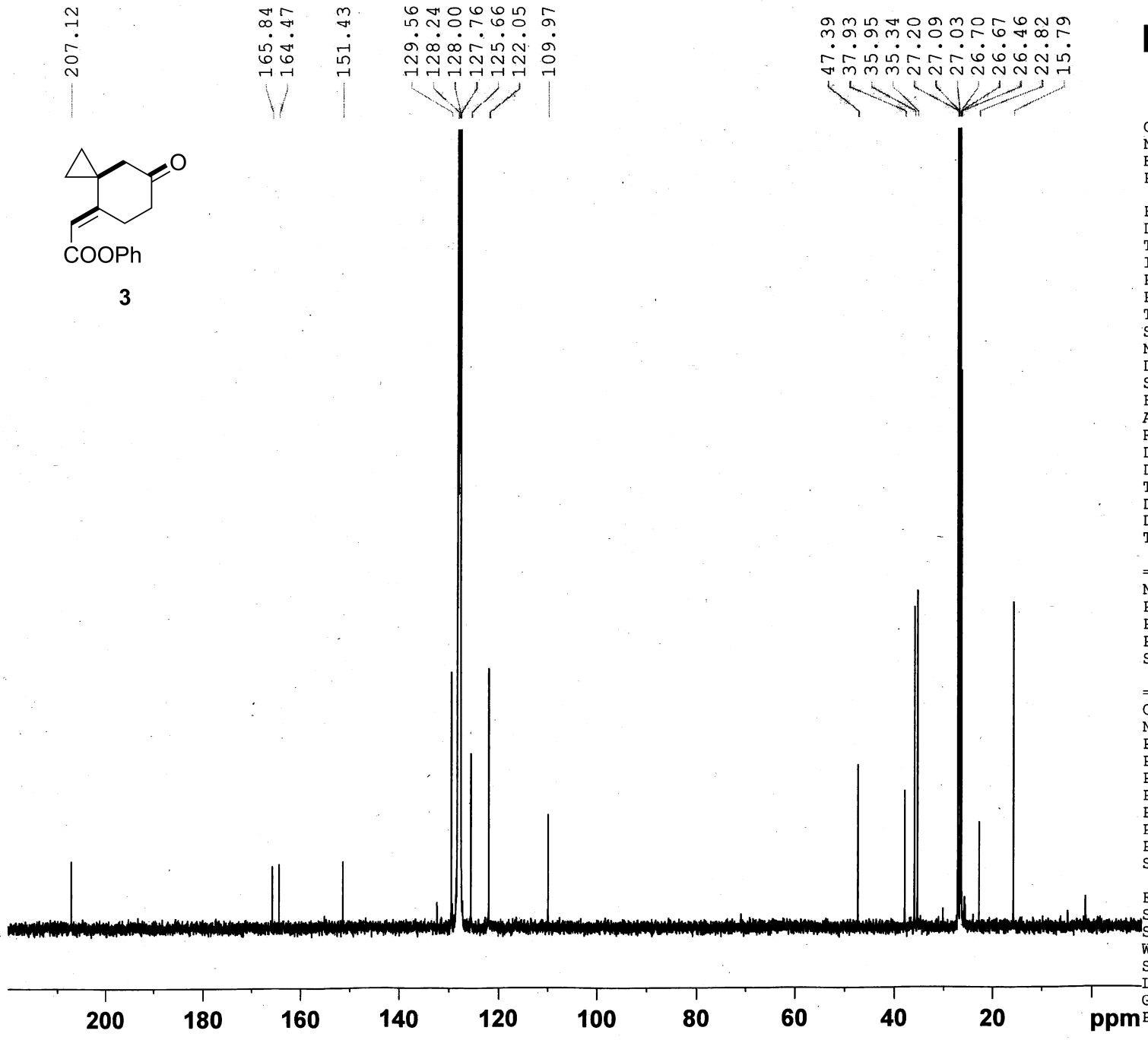

BRUKER

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D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
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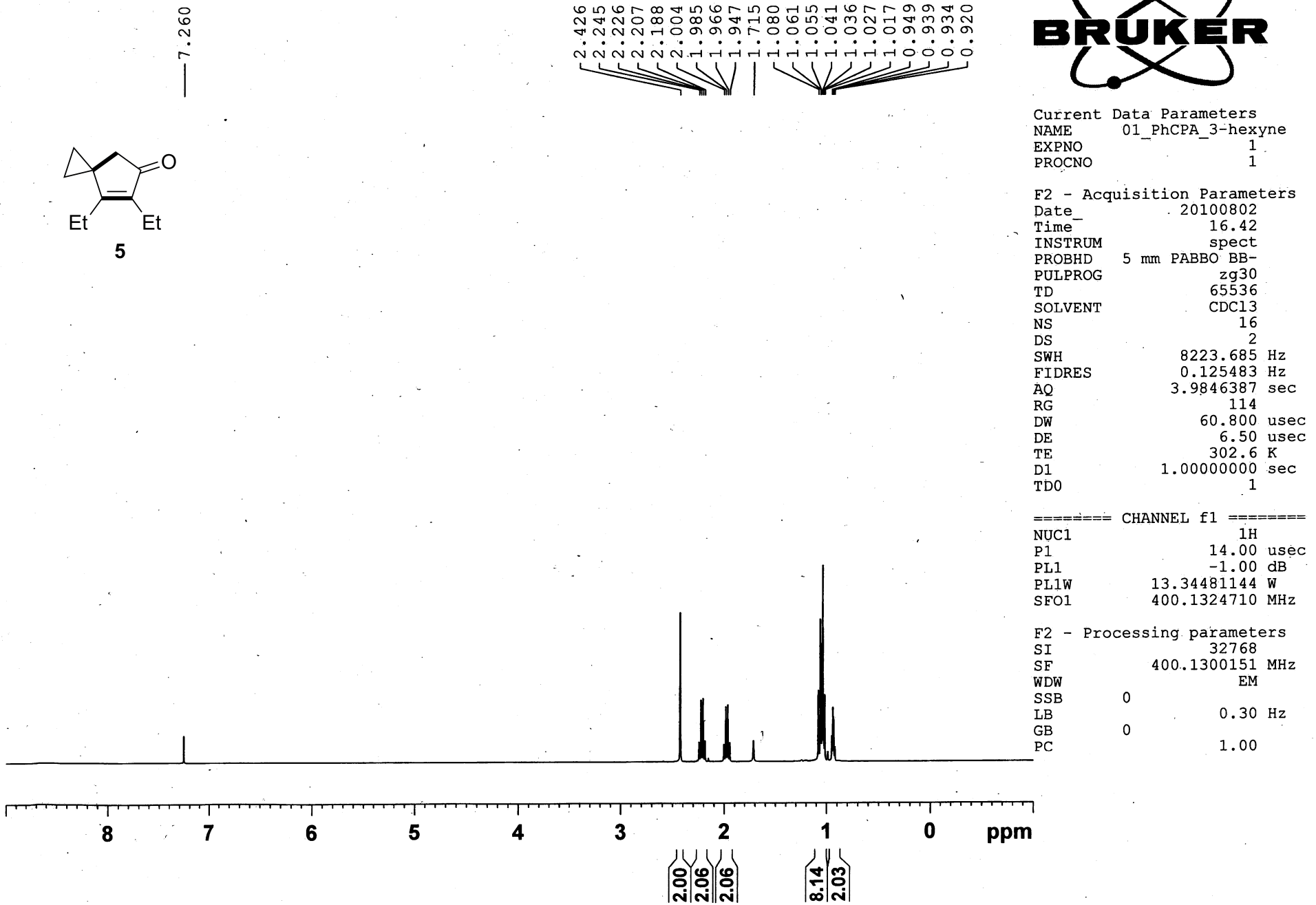
F2 - Processing parameters
SI 32768
SF 100.6127369 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



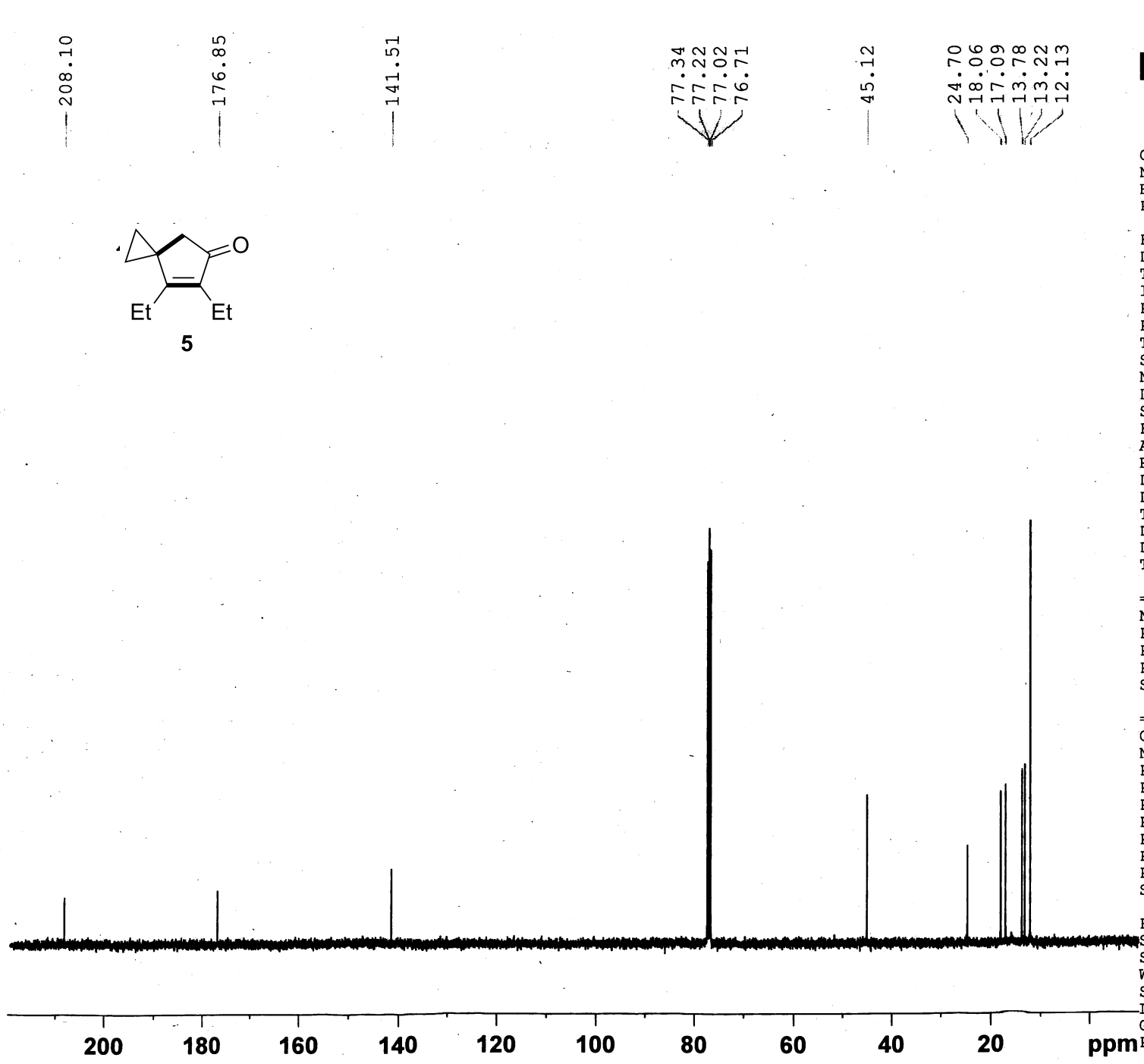
BRUKER

Current Data Parameters
NAME 01_PhCPA_3-hexyne
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100802
Time 16.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 114
DW 60.800 usec
DE 6.50 usec
TE 302.6 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.34481144 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300151 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

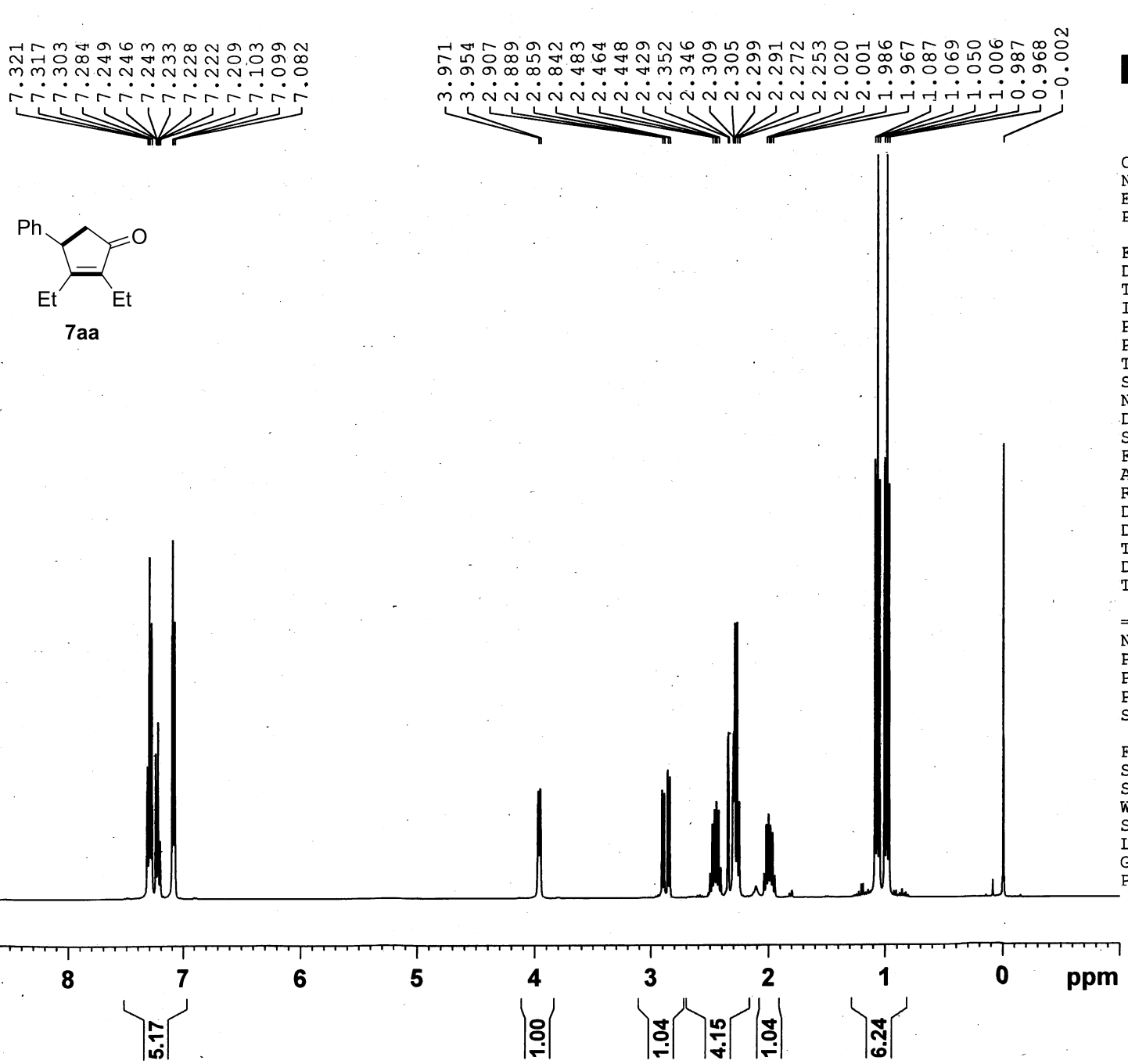
Current Data Parameters
NAME 01_PhCPA_3-hexyne
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100802
Time 16.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 165
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 303.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



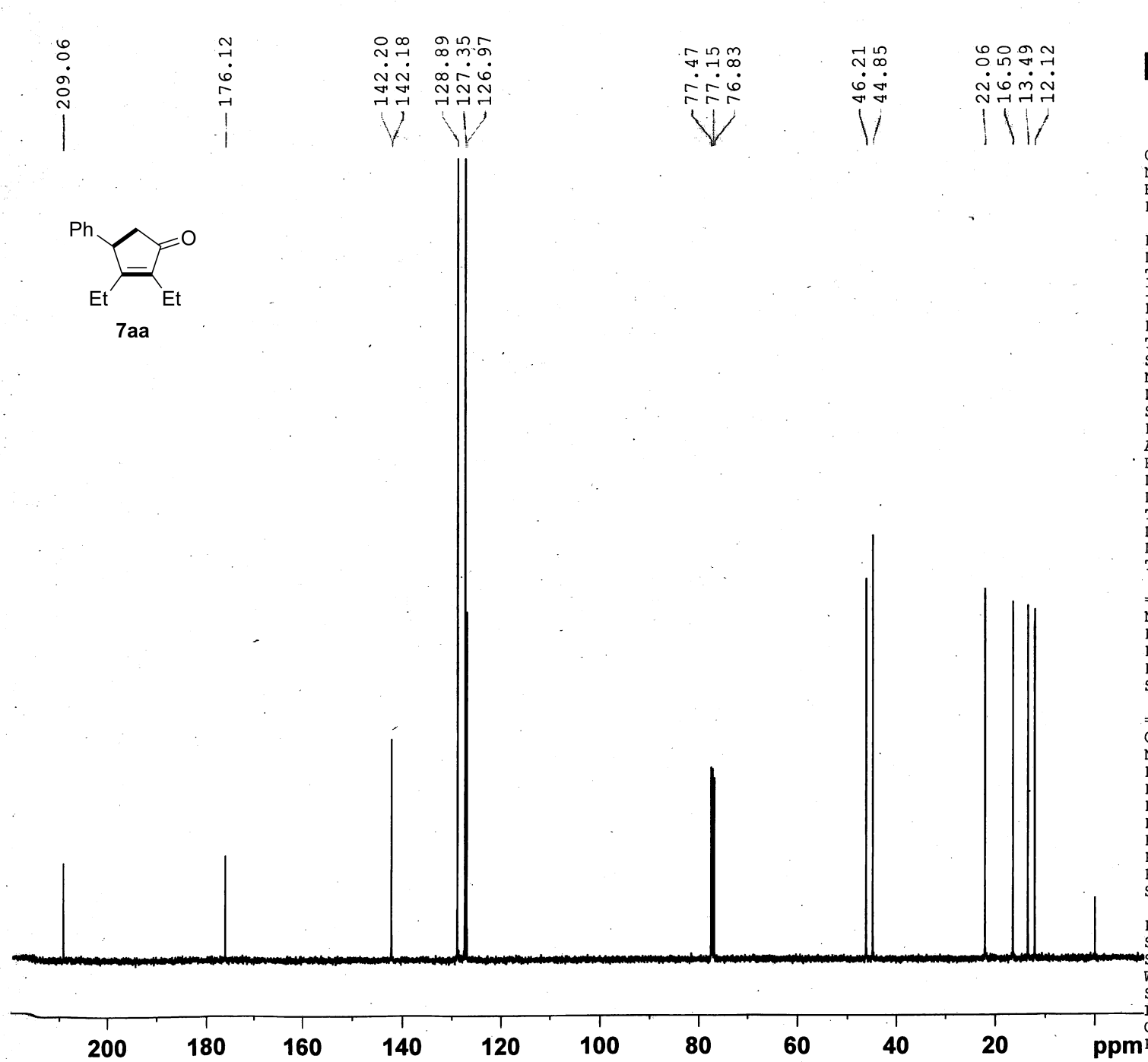
BRUKER

Current Data Parameters
NAME 02_PhCi_3-hexyne
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100226
Time 18.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 28.5
DW 60.800 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.34481144 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300083 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



BRUKER

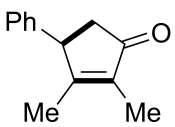
Current Data Parameters
NAME 02_PhCi_3-hexyne
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100226
Time 18.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 63
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



7ab

Current Data Parameters

NAME	03_PhCi_2-butyne
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

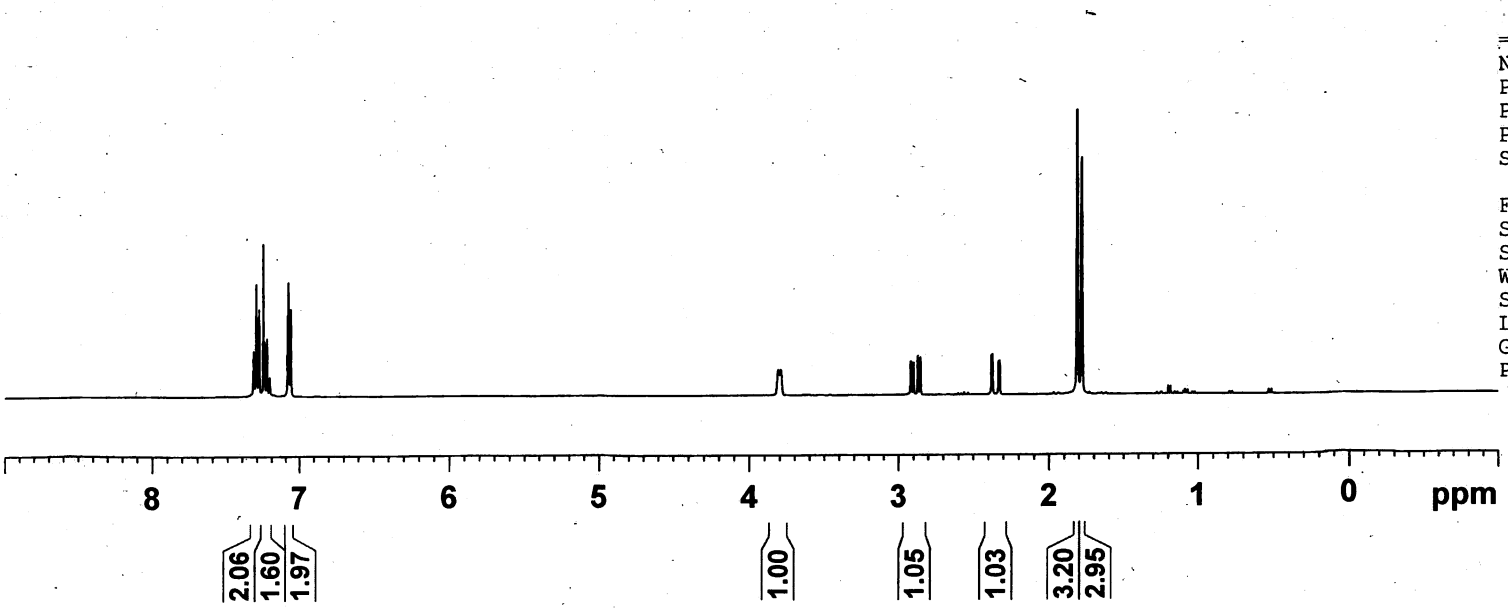
Date_	20100302
Time	17.58
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	71.8
DW	60.800 usec
DE	6.50 usec
TE	294.9 K
D1	1.00000000 sec
TD0	1

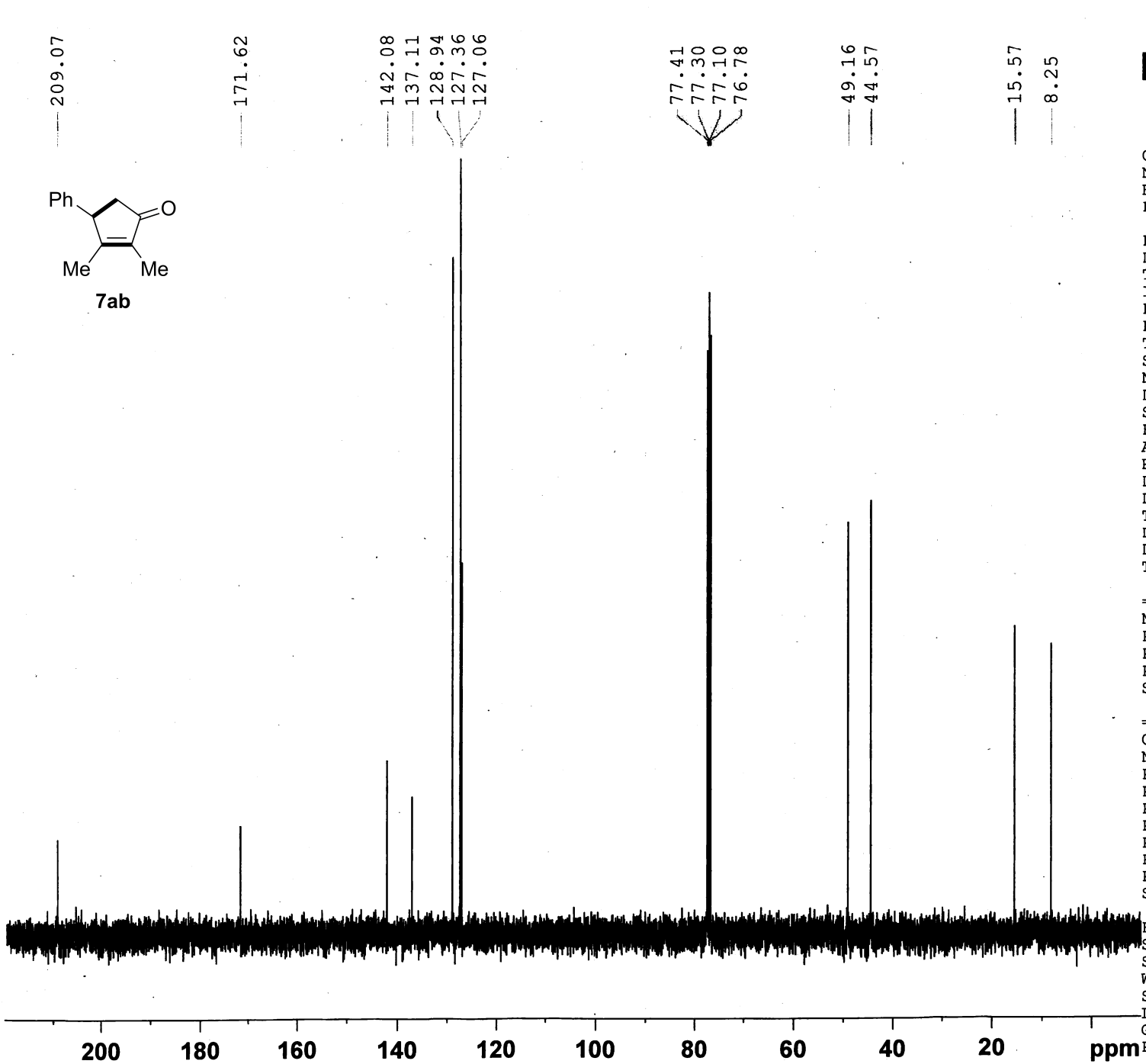
======== CHANNEL f1 ========

NUC1	1H
P1	14.00 usec
PL1	-1.00 dB
PL1W	13.34481144 W
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300172 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





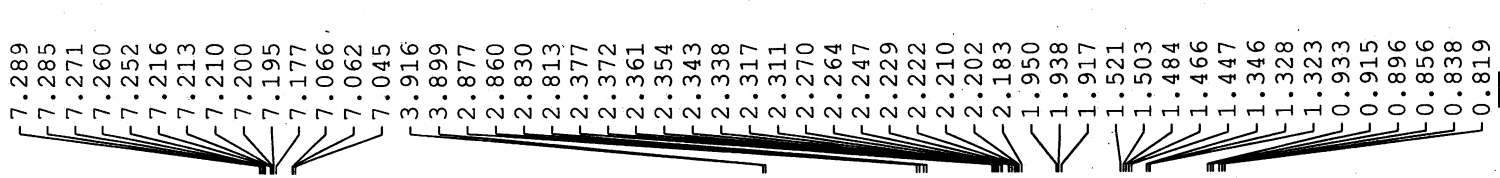
Current Data Parameters
NAME 03_PhCi_2-butyne
EXPNO 2
PROCNO 1

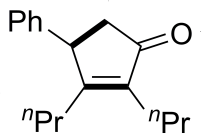
F2 - Acquisition Parameters
Date_ 20100302
Time 18.01
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

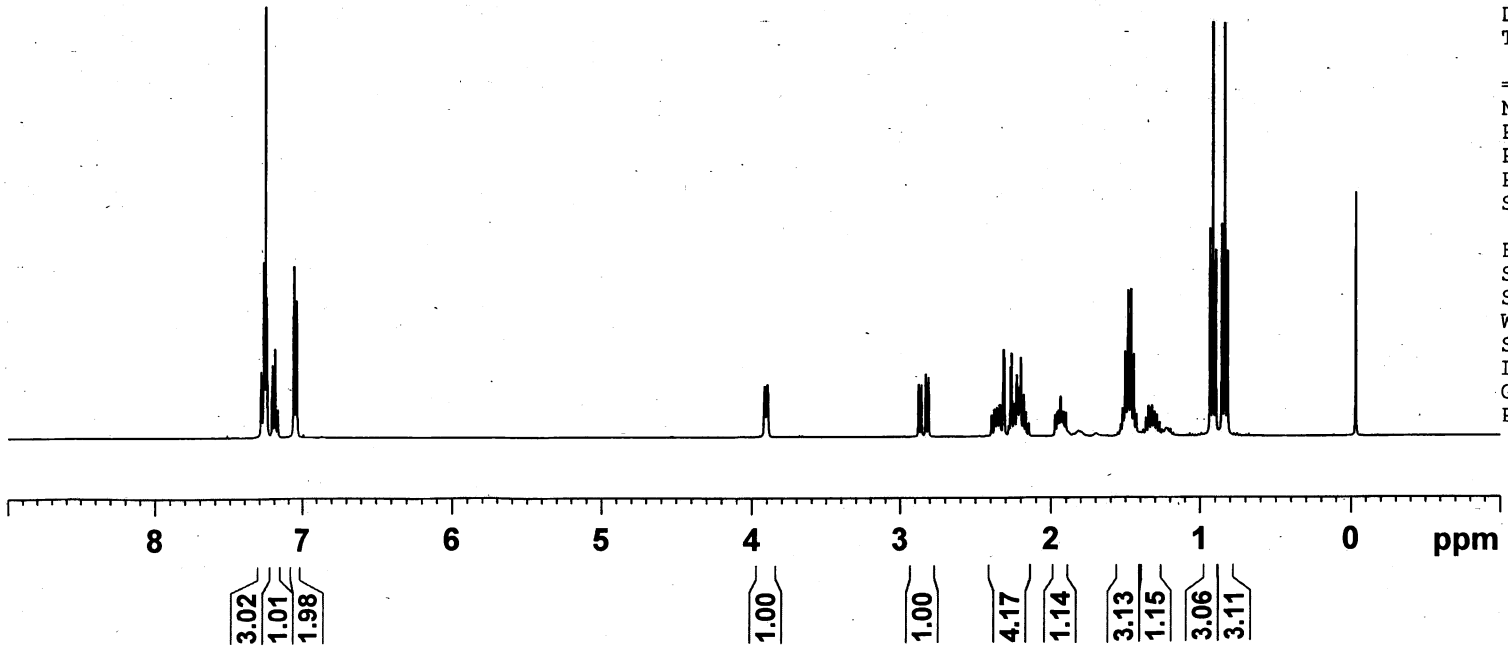
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





7ac




```
Current Data Parameters
NAME      04_PhCi_4-octyne
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100304
Time                 21.55
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                   22.6
DW                 60.800 usec
DE                  6.50 usec
TE                 294.9 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W          13.34481144 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300167 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
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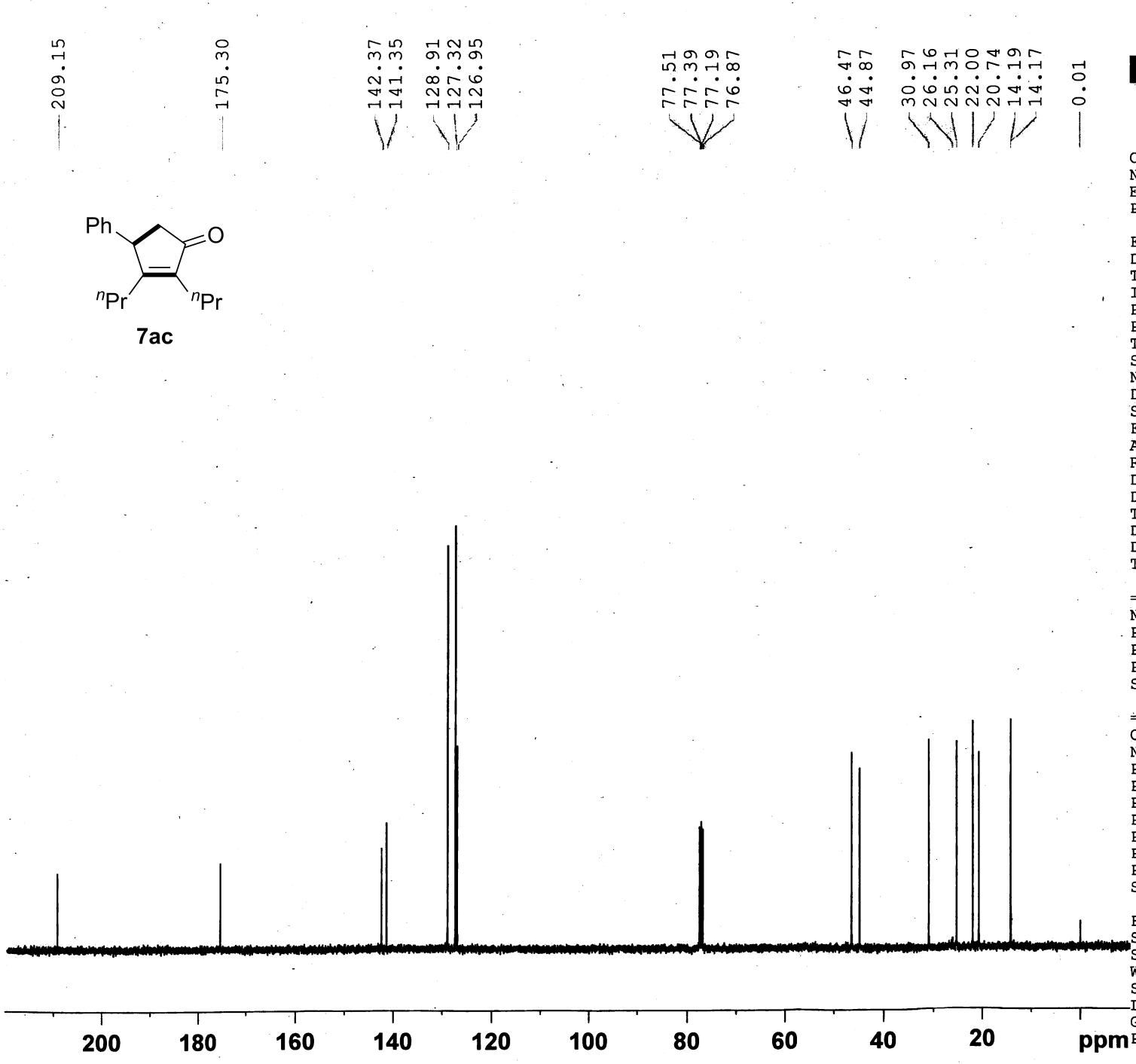

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Current Data Parameters

NAME	04_PhCi_4-octyne
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20100304
Time	21.56
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

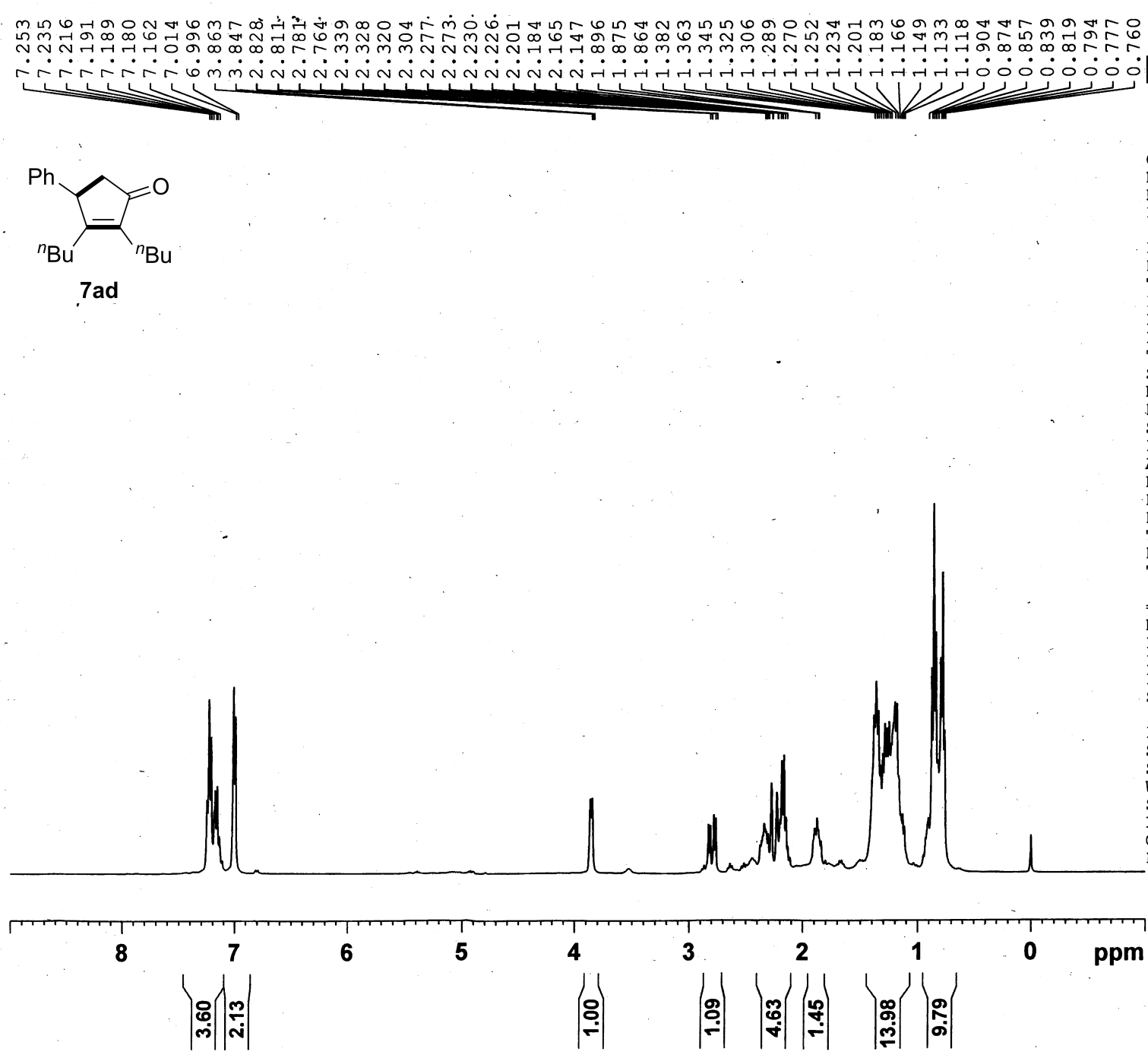
NUC1	13C
P1	10.00 usec
PL1	2.20 dB
PL1W	21.94663811 W
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	14.20 dB
PL13	15.00 dB
PL2W	13.34481144 W
PL12W	0.40300688 W
PL13W	0.33520651 W
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



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Current Data Parameters
NAME 17_PhCi_5-decyne
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110216
Time 8.29
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 50.8
DW 60.800 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.20 dB
PL1W 13.97373390 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300447 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

209.26
175.43
142.38
141.41
128.92
128.46
128.34
127.37
126.97
77.39
77.07
76.75
46.54
44.87
30.98
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28.70
23.61
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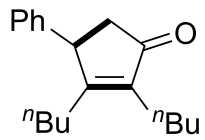
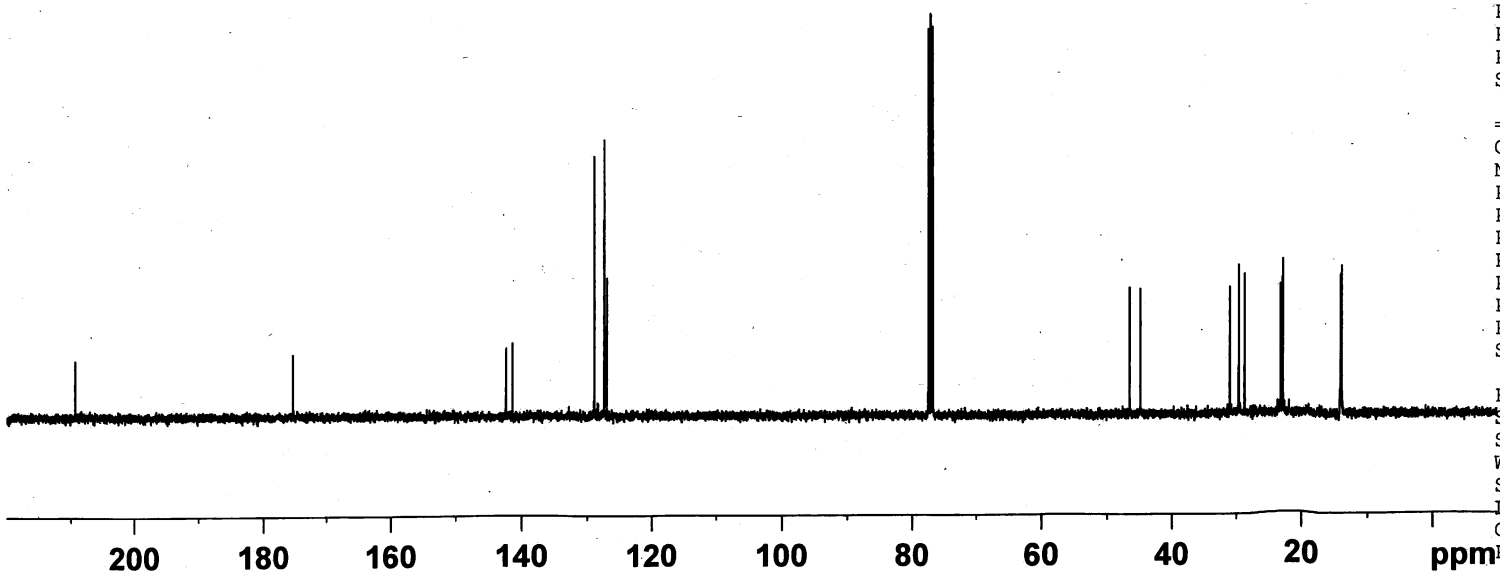
22.88
22.77
21.83
14.12
13.99
13.90
13.83



7ad



BRUKER

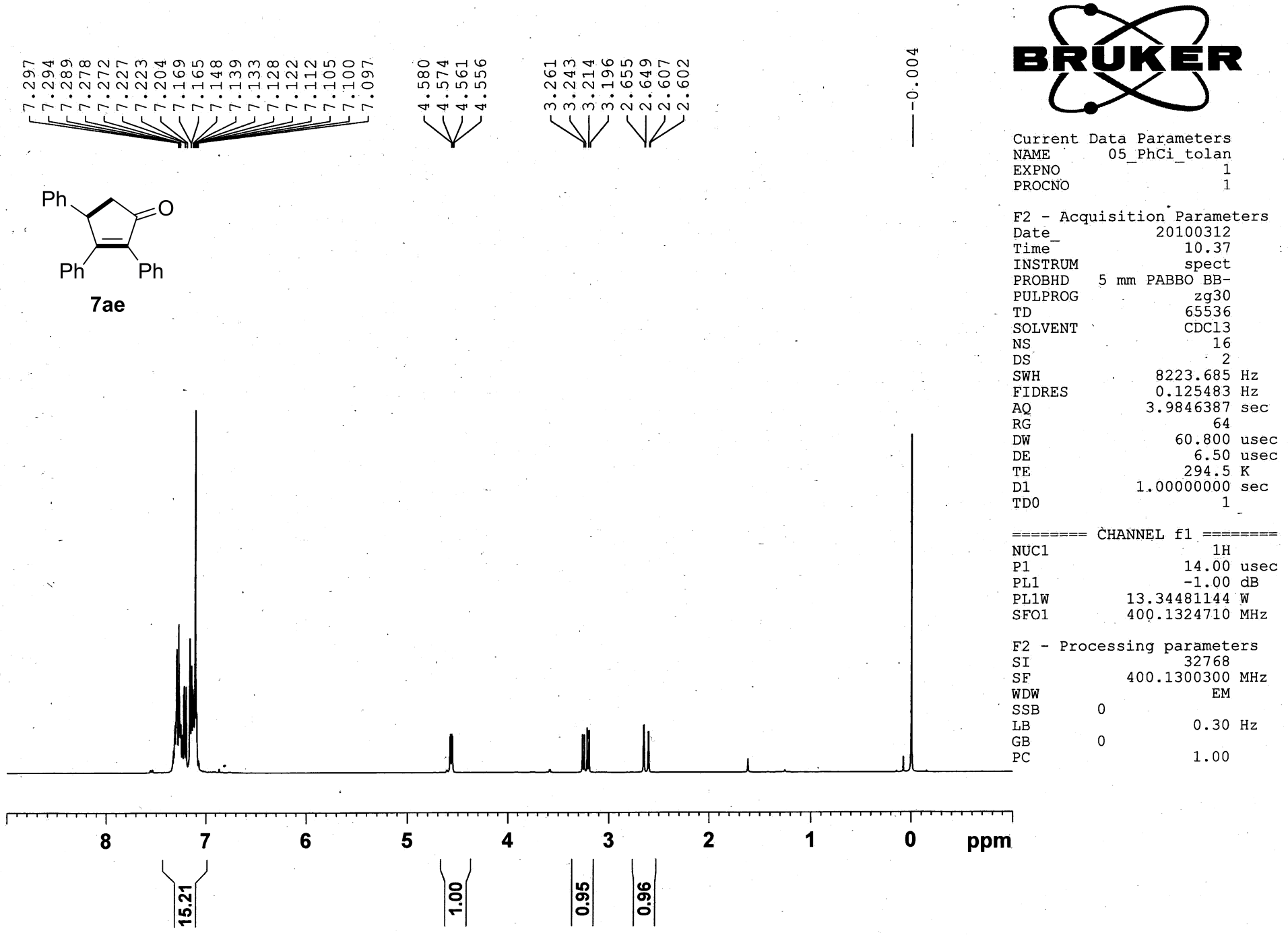
Current Data Parameters
NAME 17_PhCi_5-decyne
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110214
Time 20.54
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 114
DW 19.800 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -1.20 dB
PL12 15.88 dB
PL13 16.00 dB
PL2W 13.97373390 W
PL12W 0.27372372 W
PL13W 0.26626399 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



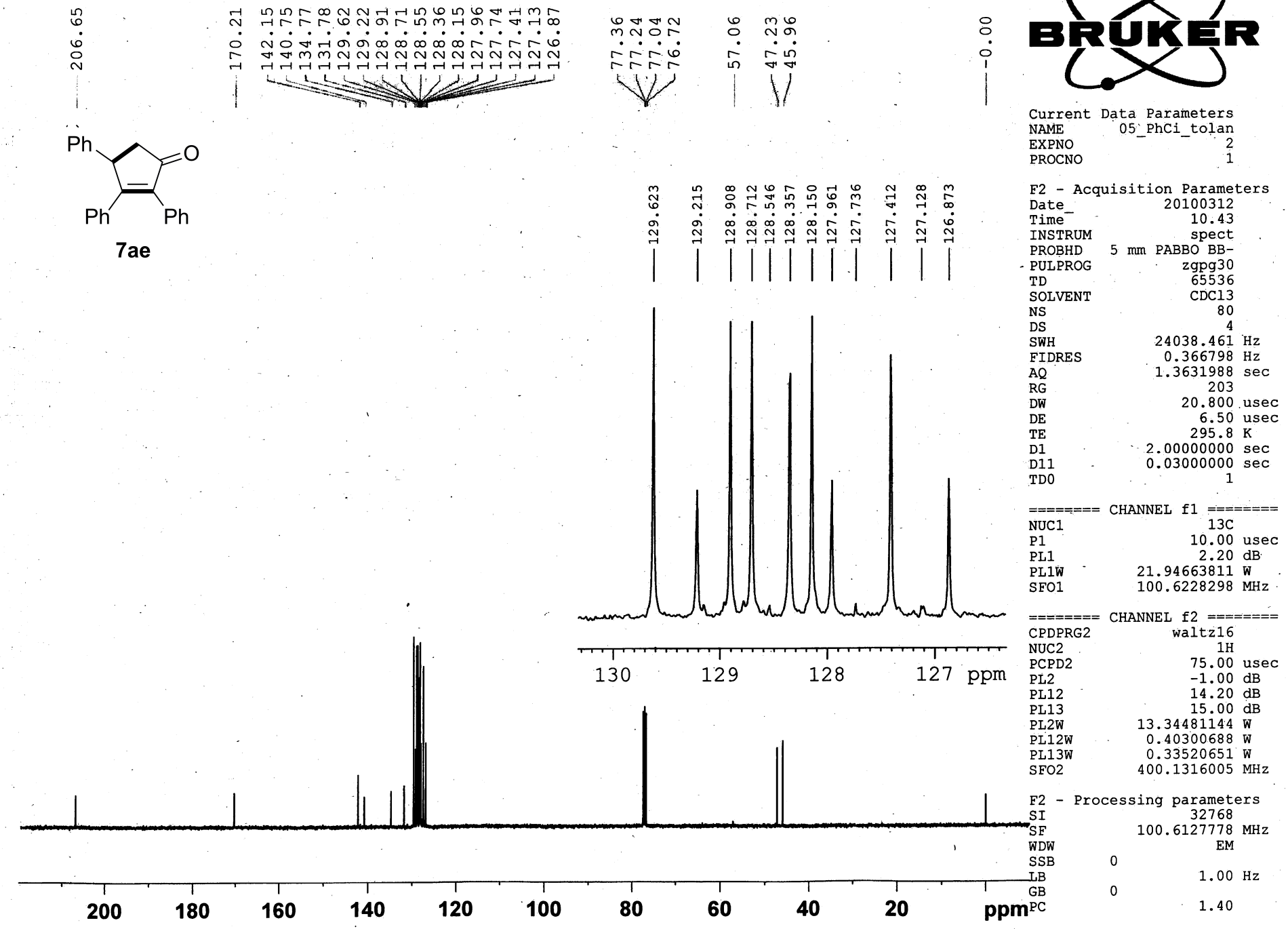
BRUKER

Current Data Parameters
NAME 05_PhCi_tolan
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100312
Time 10.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 64
DW 60.800 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.34481144 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300300 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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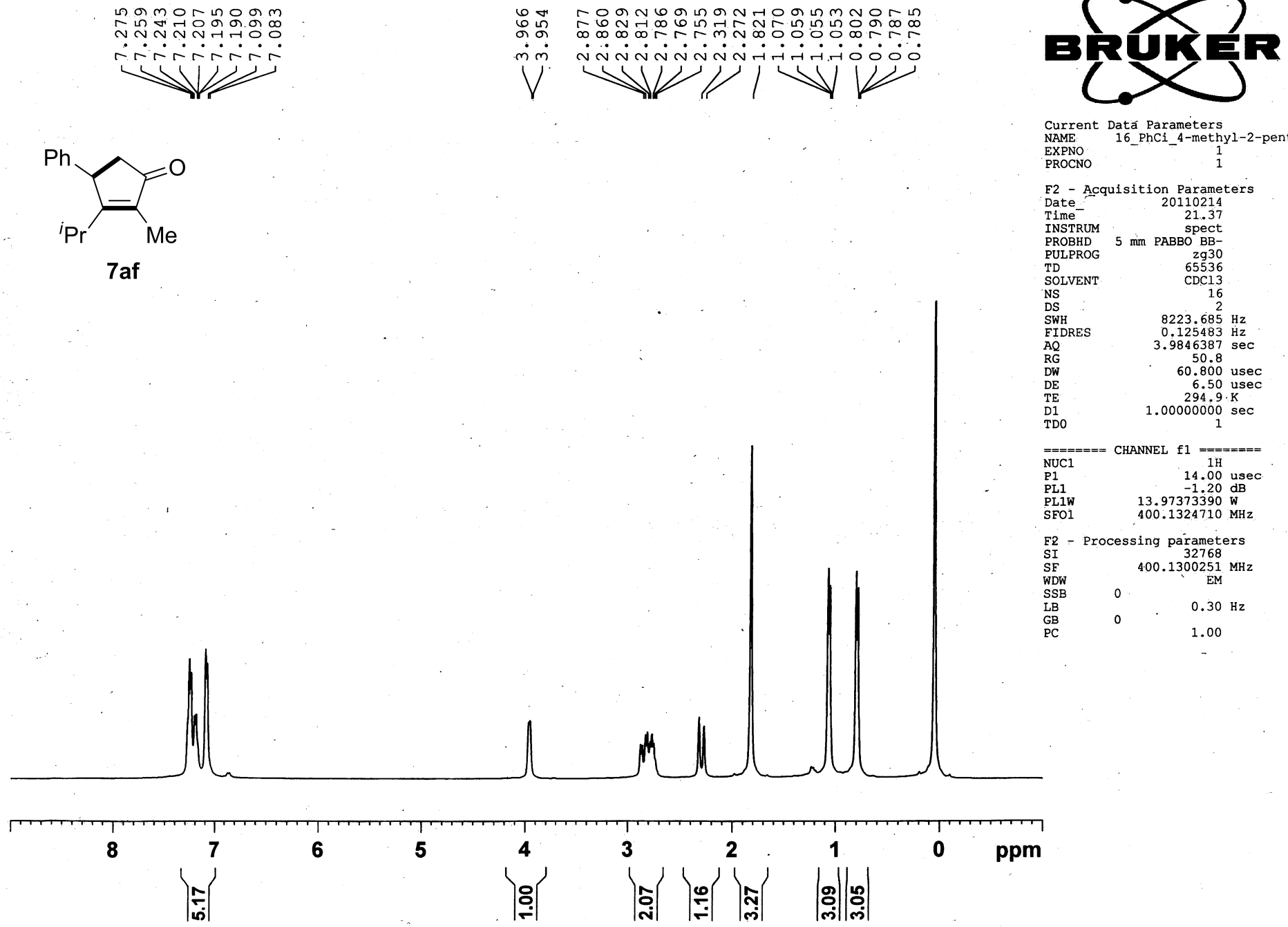
Current Data Parameters
NAME 05_PhCi_tolan
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100312
Time 10.43
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127778 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

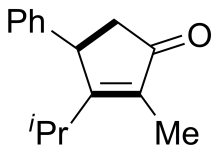


Current Data Parameters
NAME 16_PhCi_4-methyl-2-pentyne
EXPNO 1
PROCNO 1

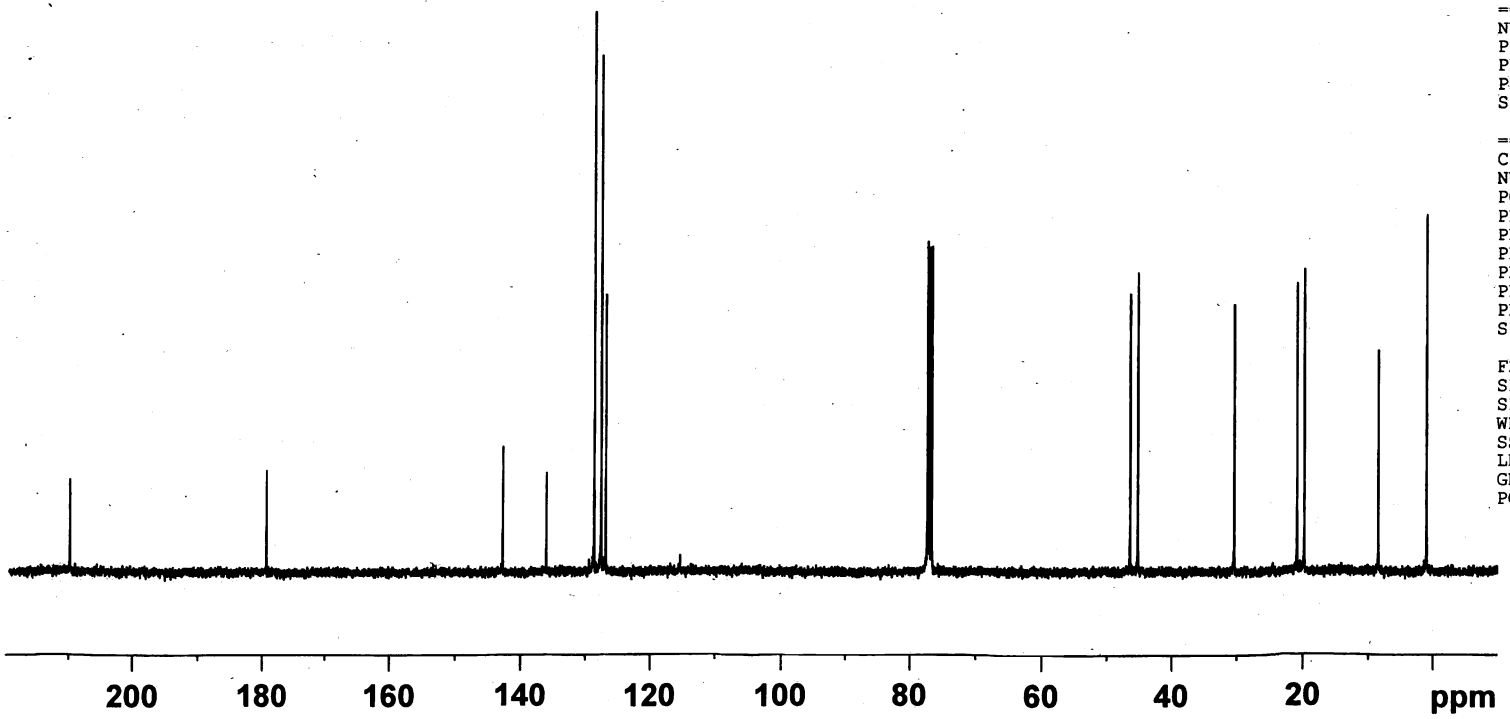
F2 - Acquisition Parameters
Date_ 20110214
Time 21.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 50.8
DW 60.800 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.20 dB
PL1W 13.97373390 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 32768
SF 400.1300251 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



7af



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```
Current Data Parameters
NAME      16_PhCi_4-methyl-2-pentyne
EXPNO                 13
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110214
Time              21.51
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                  114
DW                20.800 usec
DE                 6.50 usec
TE                296.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                10.00 usec
PL1                2.20 dB
PL1W         21.94663811 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                -1.20 dB
PL12               15.88 dB
PL13               16.00 dB
PL2W         13.97373390 W
PL12W         0.27372372 W
PL13W         0.26626399 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF           100.6127792 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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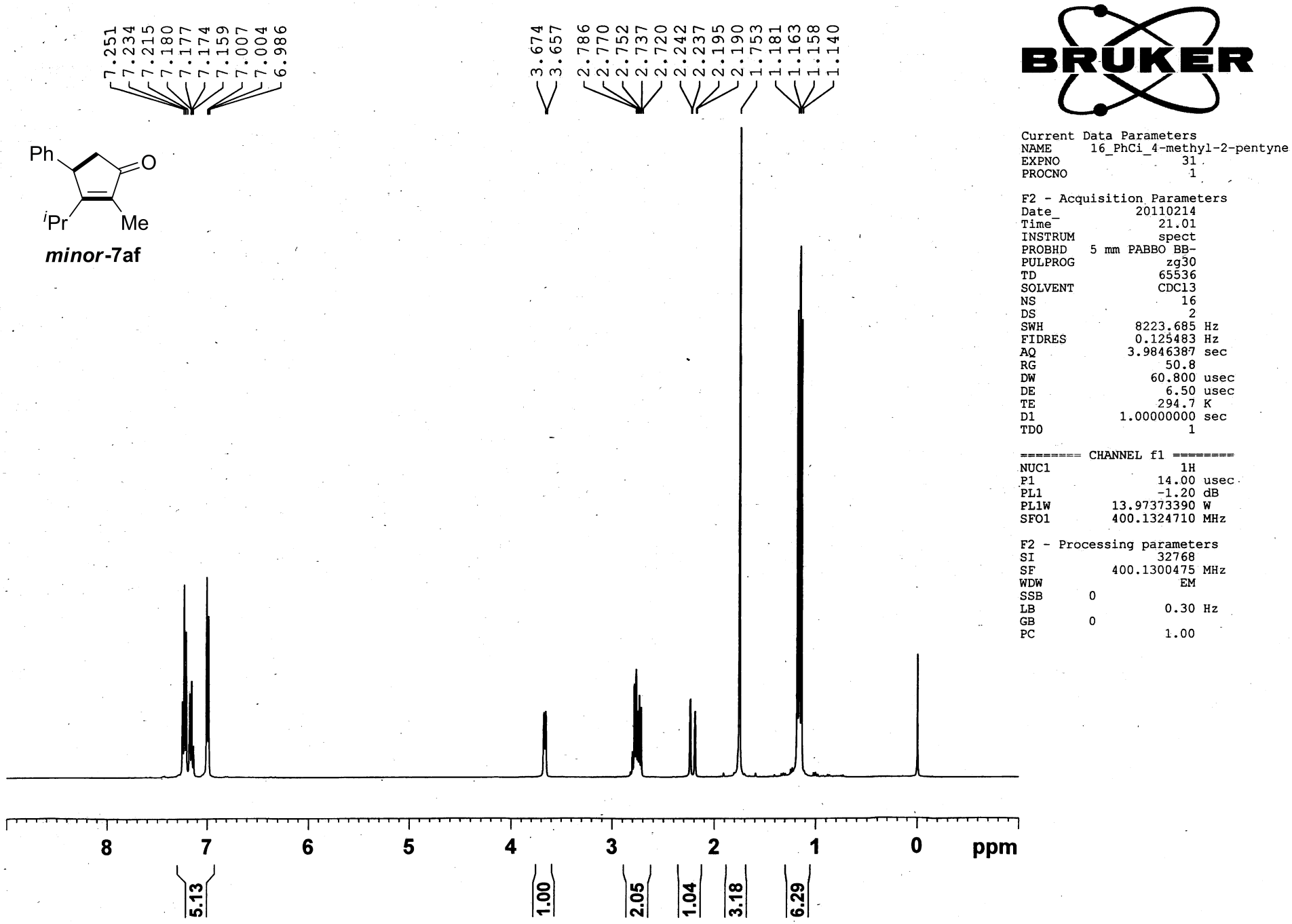

BRUKER

```
Current Data Parameters
NAME      16_PhCi_4-methyl-2-pentyne
EXPNO                 31
PROCNO                 1

F2 - Acquisition Parameters
Date_           20110214
Time              21.01
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                50.8
DW              60.800 usec
DE               6.50 usec
TE              294.7 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              14.00 usec
PL1             -1.20 dB
PL1W       13.97373390 W
SFO1      400.1324710 MHz

F2 - Processing parameters
SI                32768
SF         400.1300475 MHz
WDW                EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
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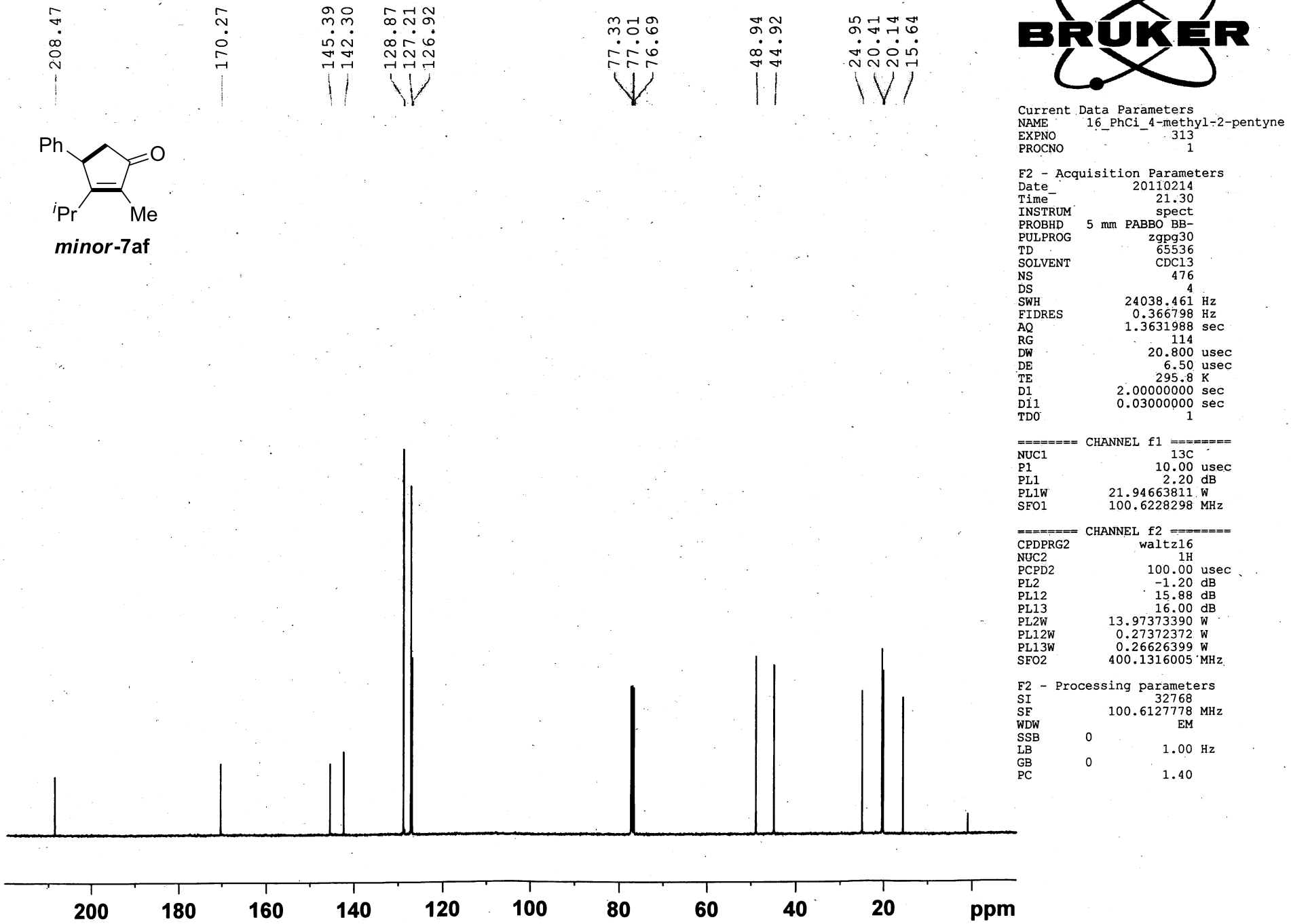

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Current Data Parameters
NAME 16_PhCi_4-methyl-2-pentyne
EXPNO 313
PROCNO 1

F2 - Acquisition Parameters

Date_	20110214
Time	21.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	476
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	114
DW	20.800 usec
DE	6.50 usec
TE	295.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

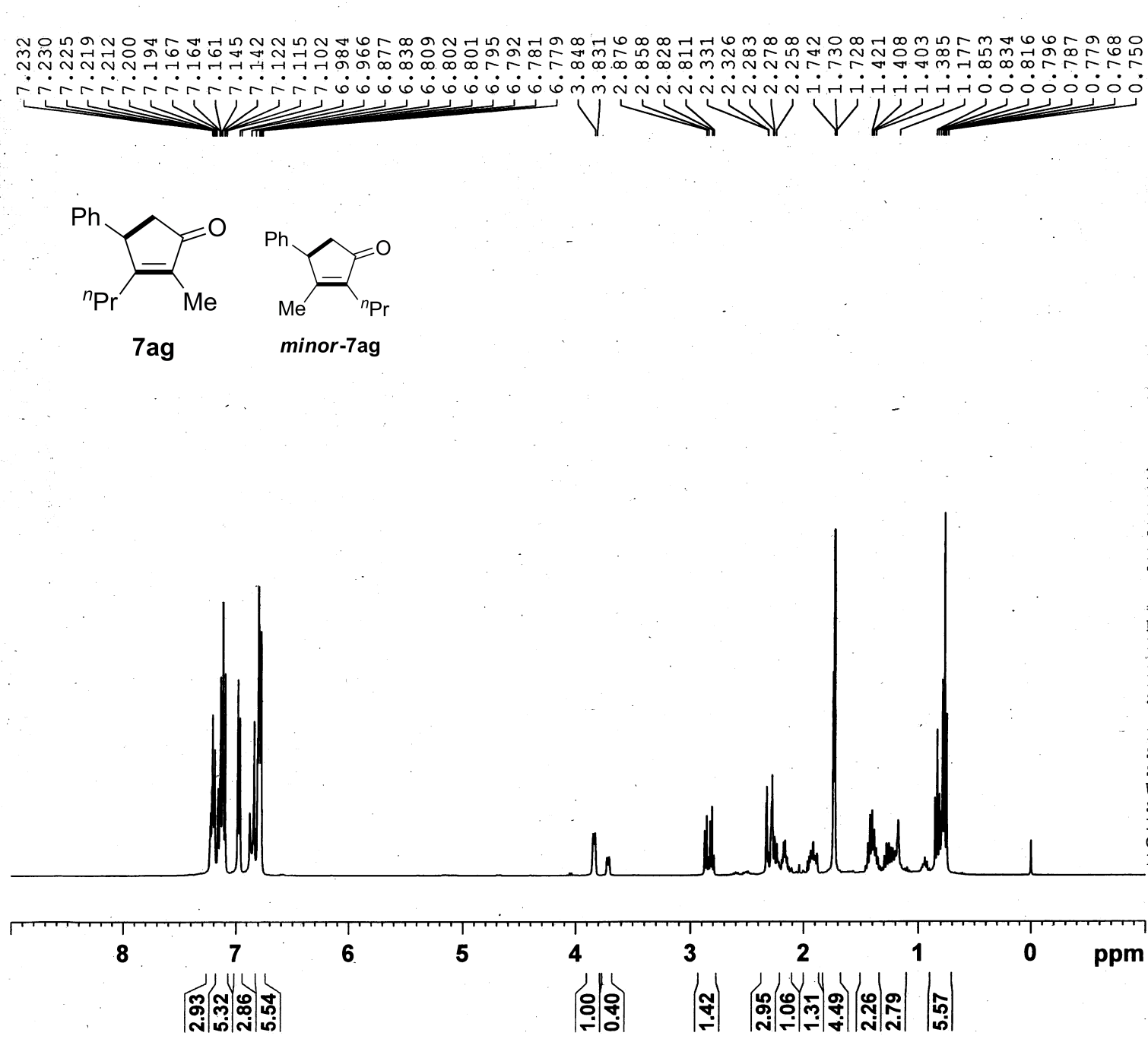
NUC1	13C
P1	10.00 usec
PL1	2.20 dB
PL1W	21.94663811 W
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.20 dB
PL12	15.88 dB
PL13	16.00 dB
PL2W	13.97373390 W
PL12W	0.27372372 W
PL13W	0.26626399 W
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127778 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

Current Data Parameters
NAME 06_PhCi_2-hexyne
EXPNO 1
PROCNO 1

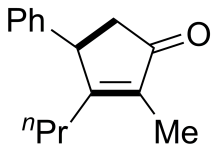
F2 - Acquisition Parameters
Date_ 20100324
Time 14.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 40.3
DW 60.800 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.34481144 W
SFO1 400.1324710 MHz

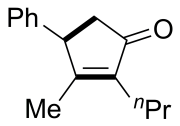
F2 - Processing parameters
SI 32768
SF 400.1300638 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

PhCi + 2-hexyne

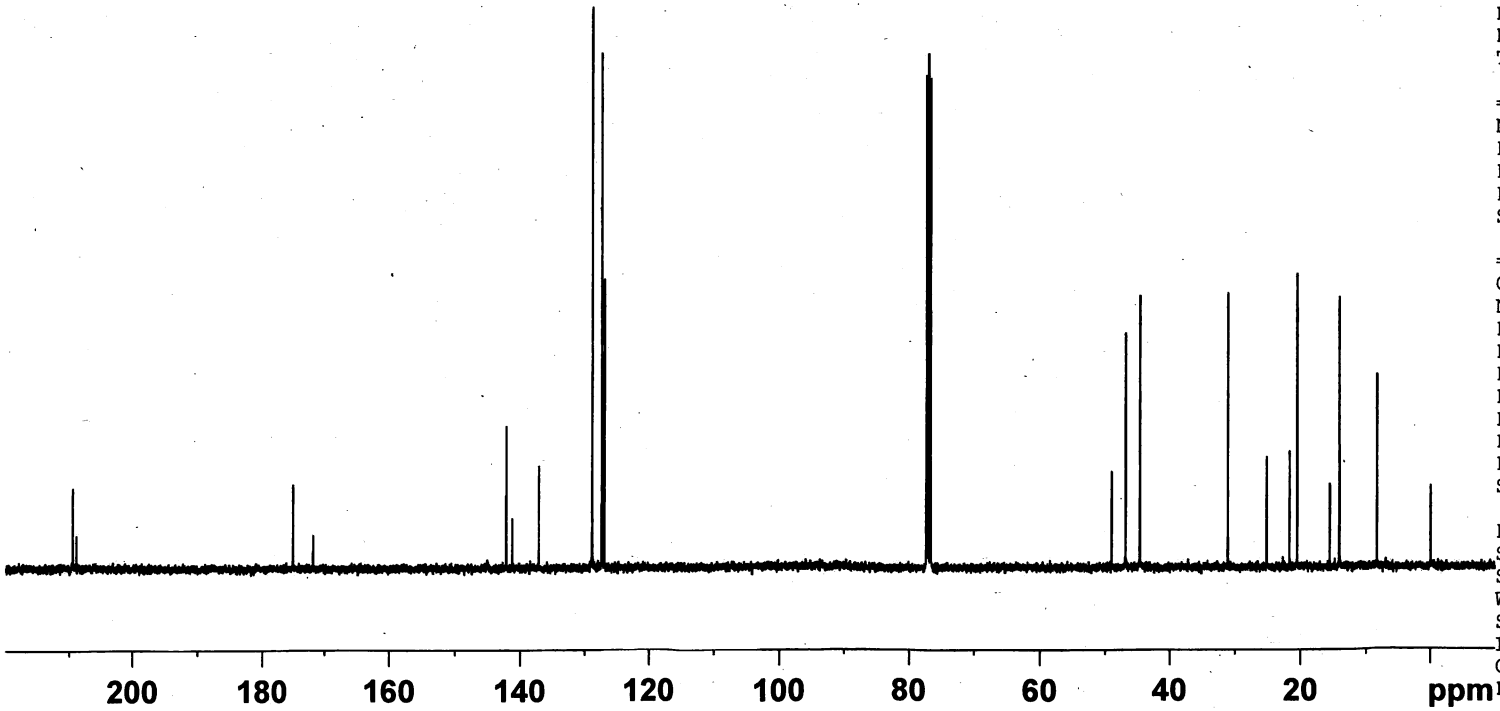
209.39
208.81
175.17
171.85
142.18
141.27
137.18
128.93
128.87
127.40
127.29
127.00
126.98
77.38
77.27
77.06
76.75
49.08
46.90
44.67
31.11
25.16
22.72
21.68
20.53
15.54
14.08
14.04
8.32
-0.00



7ag



***minor*-7ag**



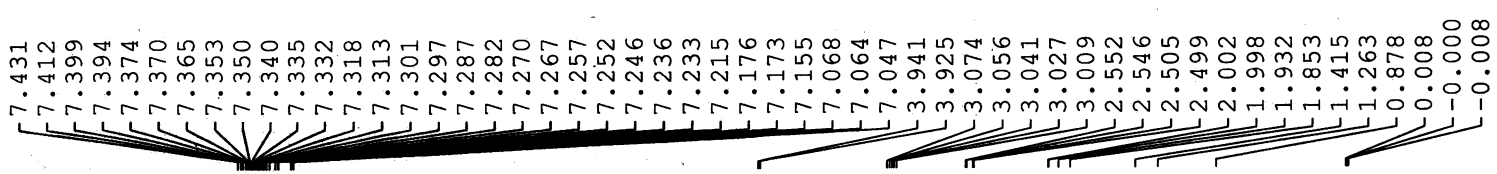

```
Current Data Parameters
NAME      06_PhCi_2-hexyne
EXPNO                    2
PROCNO                   1

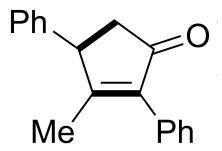
F2 - Acquisition Parameters
Date_             20100325
Time                 14.17
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     196
DS                       4
SWH              25252.525 Hz
FIDRES           0.385323 Hz
AQ               1.2976629 sec
RG                     203
DW                19.800 usec
DE                 6.50 usec
TE                 295.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                10.00 usec
PL1                2.20 dB
PL1W         21.94663811 W
SFO1         100.6228298 MHz

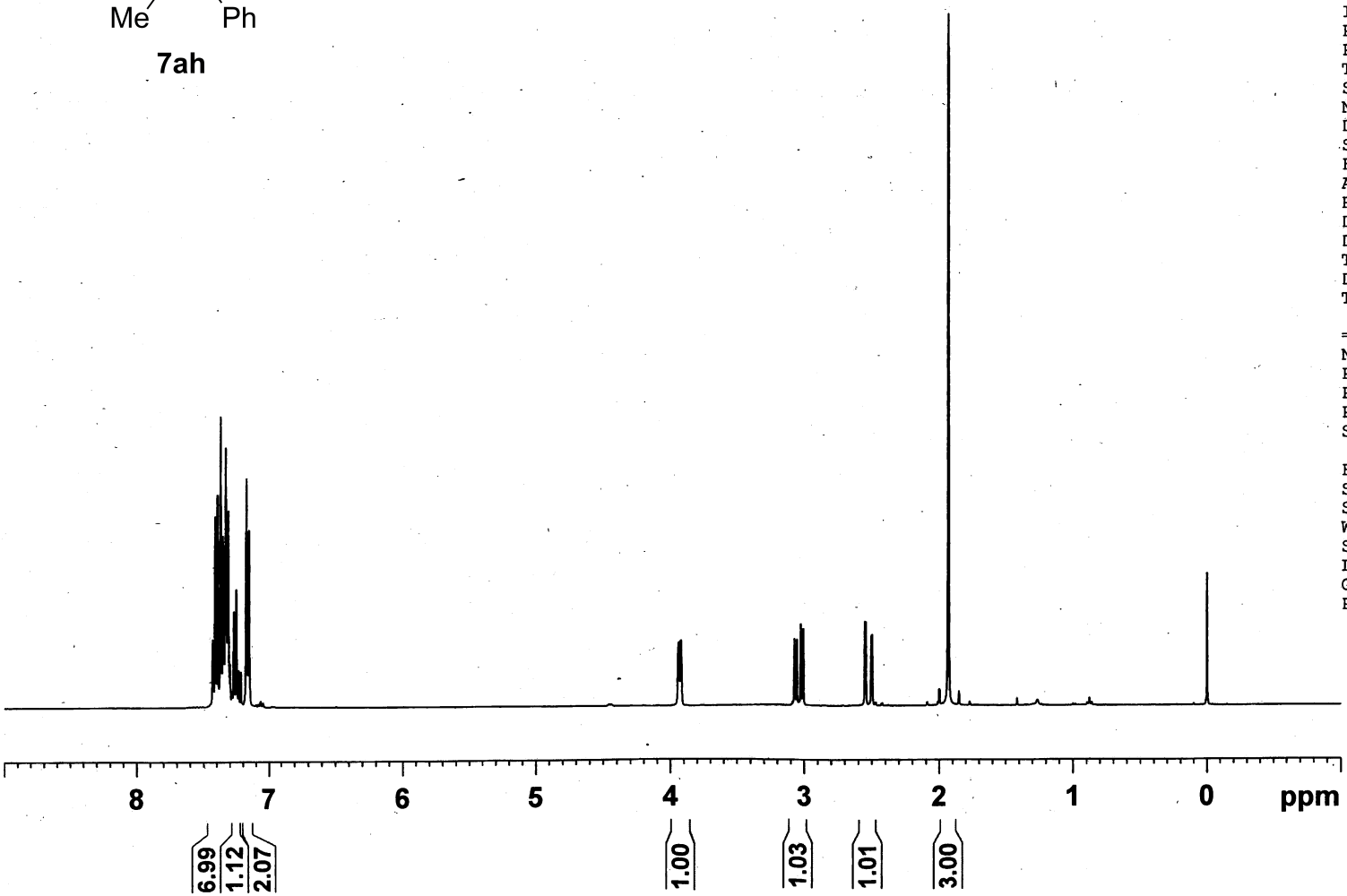
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             75.00 usec
PL2               -1.00 dB
PL12              14.20 dB
PL13              15.00 dB
PL2W         13.34481144 W
PL12W         0.40300688 W
PL13W         0.33520651 W
SFO2         400.1316005 MHz

F2 - Processing parameters
SI                   32768
SF           100.6127714 MHz
WDW                     EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```





7ah



Current Data Parameters
NAME 07_PhCi_1-Ph-3-propyne
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

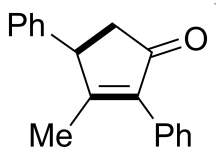
Date_	20100310
Time	15.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	32
DW	60.800 usec
DE	6.50 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========

NUC1	1H
P1	14.00 usec
PL1	-1.00 dB
PL1W	13.34481144 W
SFO1	400.1324710 MHz

F2 - Processing parameters

SI	32768
SF	400.1300346 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



7ah

206.78
173.33
141.90
140.83
131.67
129.31
129.17
128.85
128.58
128.40
128.19
127.93
127.48
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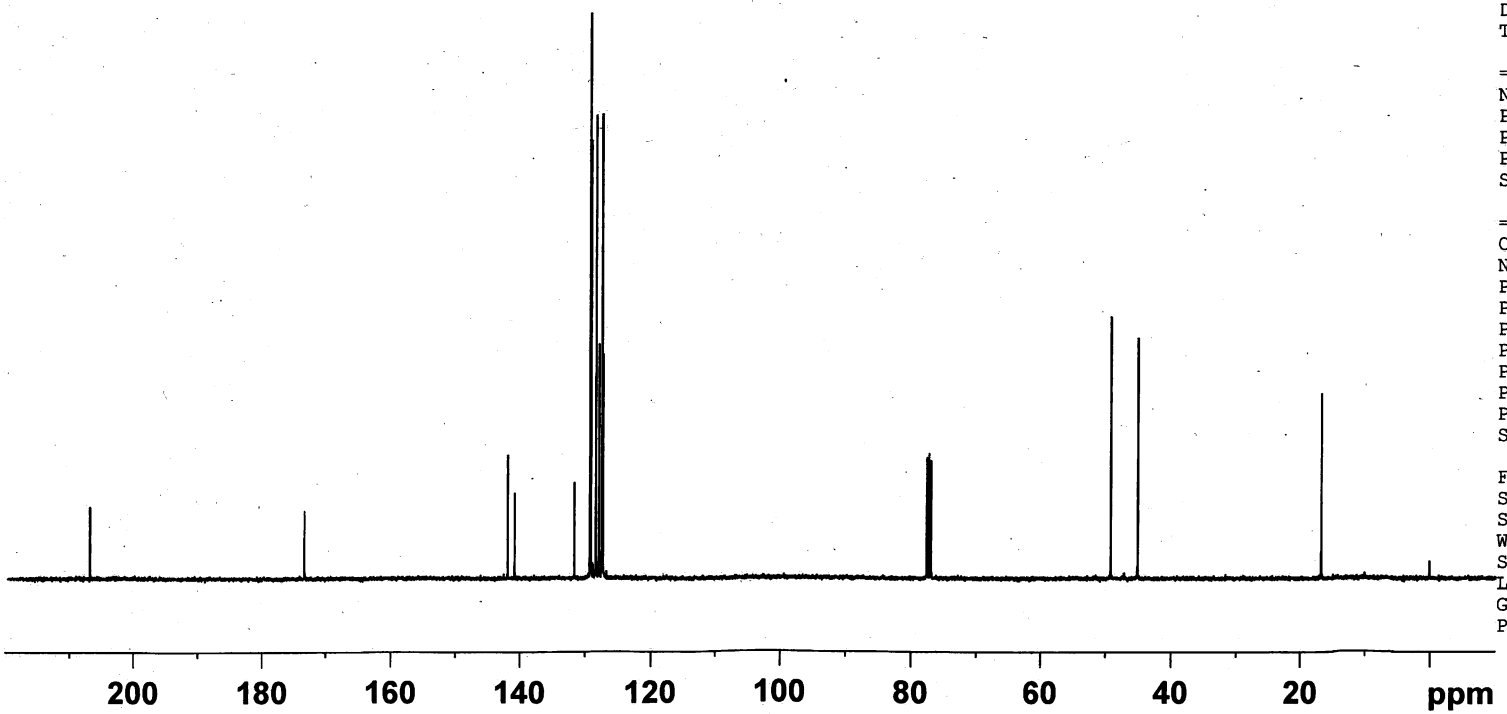
76.95
49.21
47.18
45.11
16.79
0.14



Current Data Parameters
NAME 07_PhCi_1-Ph-3-propyne
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters

Date_	20100310
Time	15.31
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	54
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

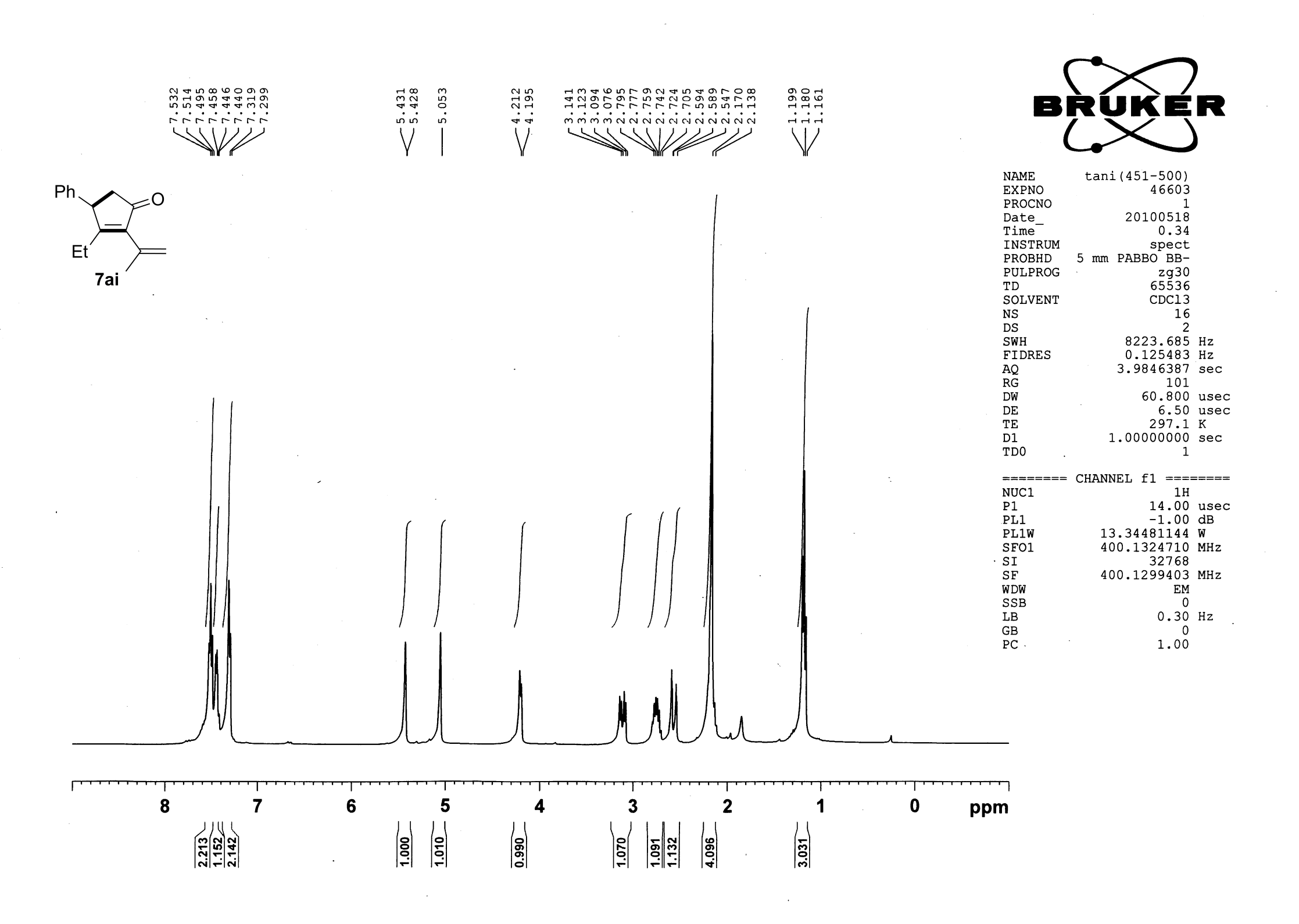
NUC1	13C
P1	10.00 usec
PL1	2.20 dB
PL1W	21.94663811 W
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	14.20 dB
PL13	15.00 dB
PL2W	13.34481144 W
PL12W	0.40300688 W
PL13W	0.33520651 W
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



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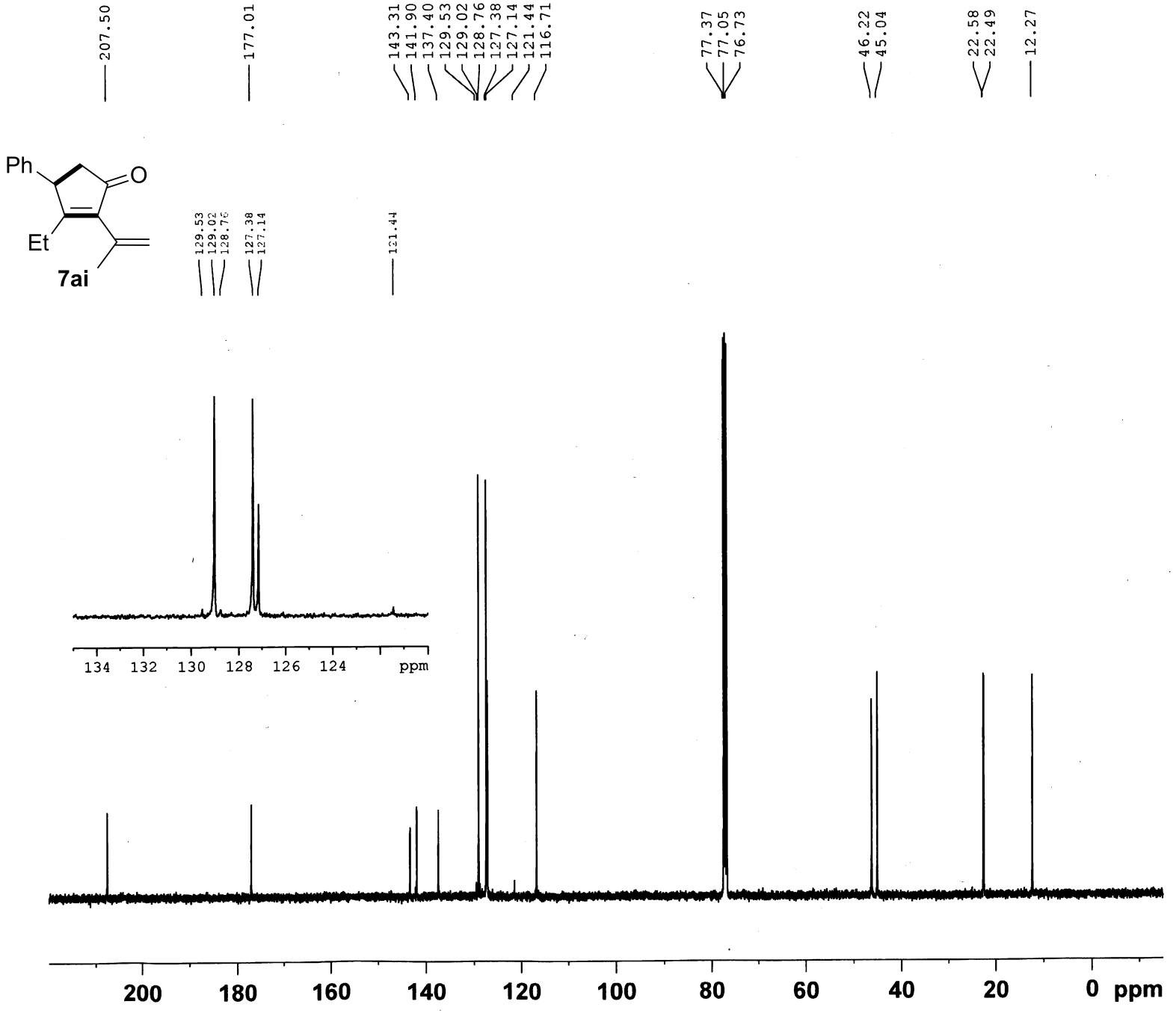
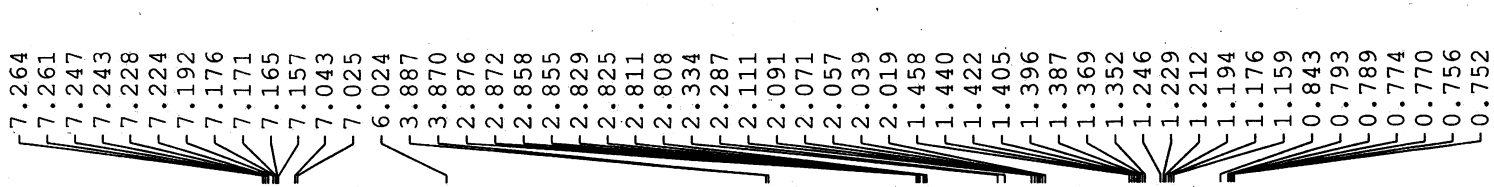
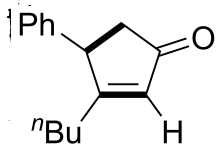
NAME	tani(451-500)	
EXPNO	46603	
PROCNO	1	
Date_	20100518	
Time	0.34	
INSTRUM	spect	
PROBHD	5 mm PABBO BB-	
PULPROG	zg30	
TD	65536	
SOLVENT	CDCl3	
NS	16	
DS	2	
SWH	8223.685	Hz
FIDRES	0.125483	Hz
AQ	3.9846387	sec
RG	101	
DW	60.800	usec
DE	6.50	usec
TE	297.1	K
D1	1.00000000	sec
TD0	1	
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.00	usec
PL1	-1.00	dB
PL1W	13.34481144	W
SFO1	400.1324710	MHz
SI	32768	
SF	400.1299403	MHz
WDW	EM	
SSB	0	
LB	0.30	Hz
GB	0	
PC	1.00	

PhCi + eneyne

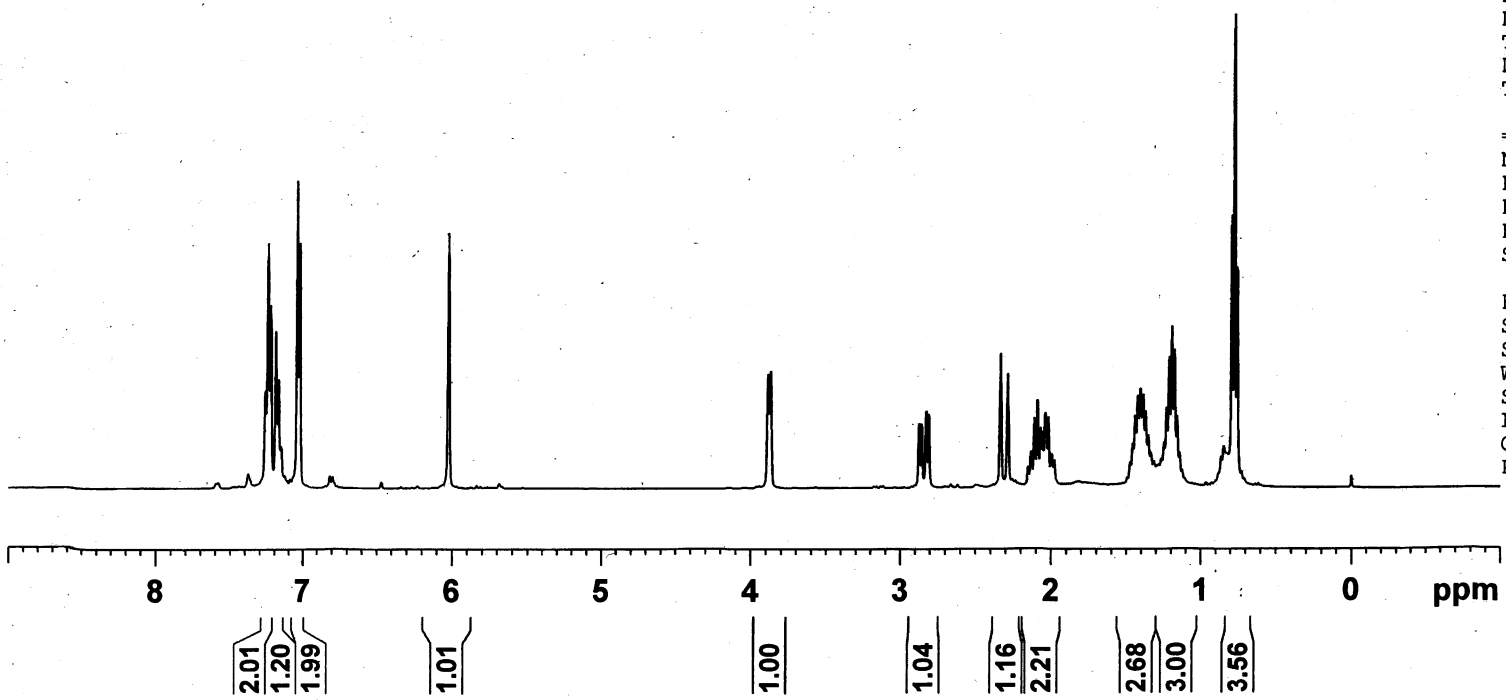


NAME	tani(451-500)
EXPNO	46604
PROCNO	1
Date_	20100518
Time	1.30
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	979
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	203
DW	19.800 usec
DE	6.50 usec
TE	298.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	2.20 dB
PL1W	21.94663811 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	14.20 dB
PL13	15.00 dB
PL2W	13.34481144 W
PL12W	0.40300688 W
PL13W	0.33520651 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





7aj

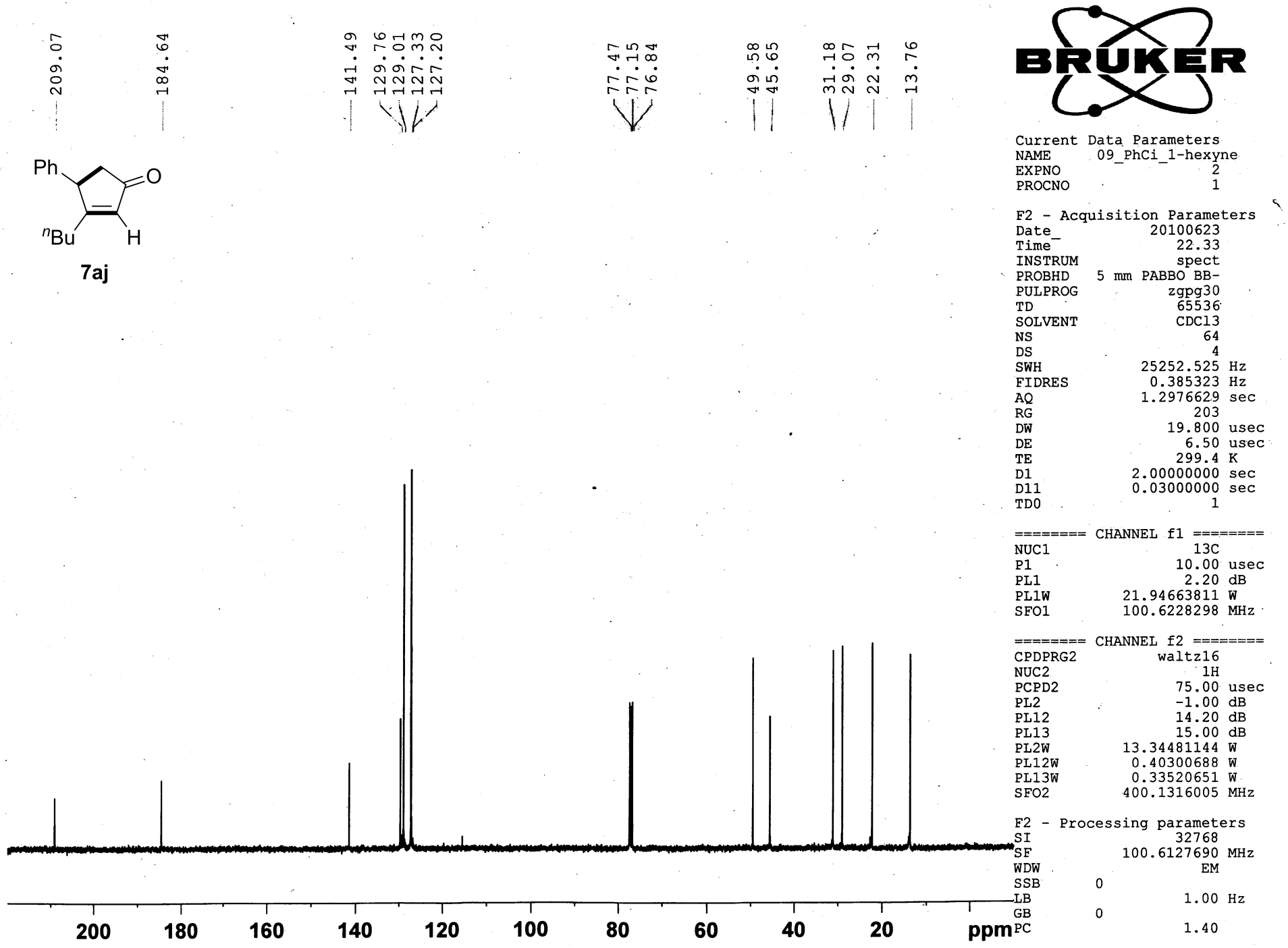



```
Current Data Parameters
NAME      09_PhCi_1-hexyne
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100623
Time                23.22
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                   50.8
DW                 60.800 usec
DE                  6.50 usec
TE                 298.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W         13.34481144 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300452 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



BRUKER

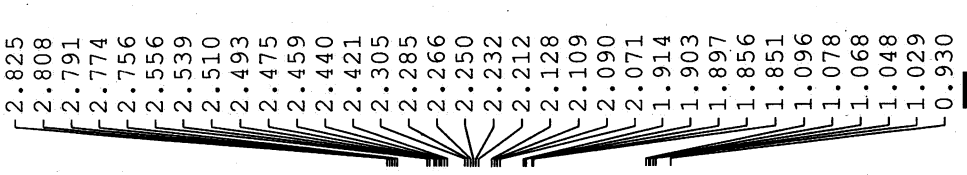
Current Data Parameters
NAME 09_PhCi_1-hexyne
EXPNO 2
PROCNO 1

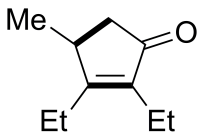
F2 - Acquisition Parameters
Date_ 20100623
Time 22.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 203
DW 19.800 usec
DE 6.50 usec
TE 299.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





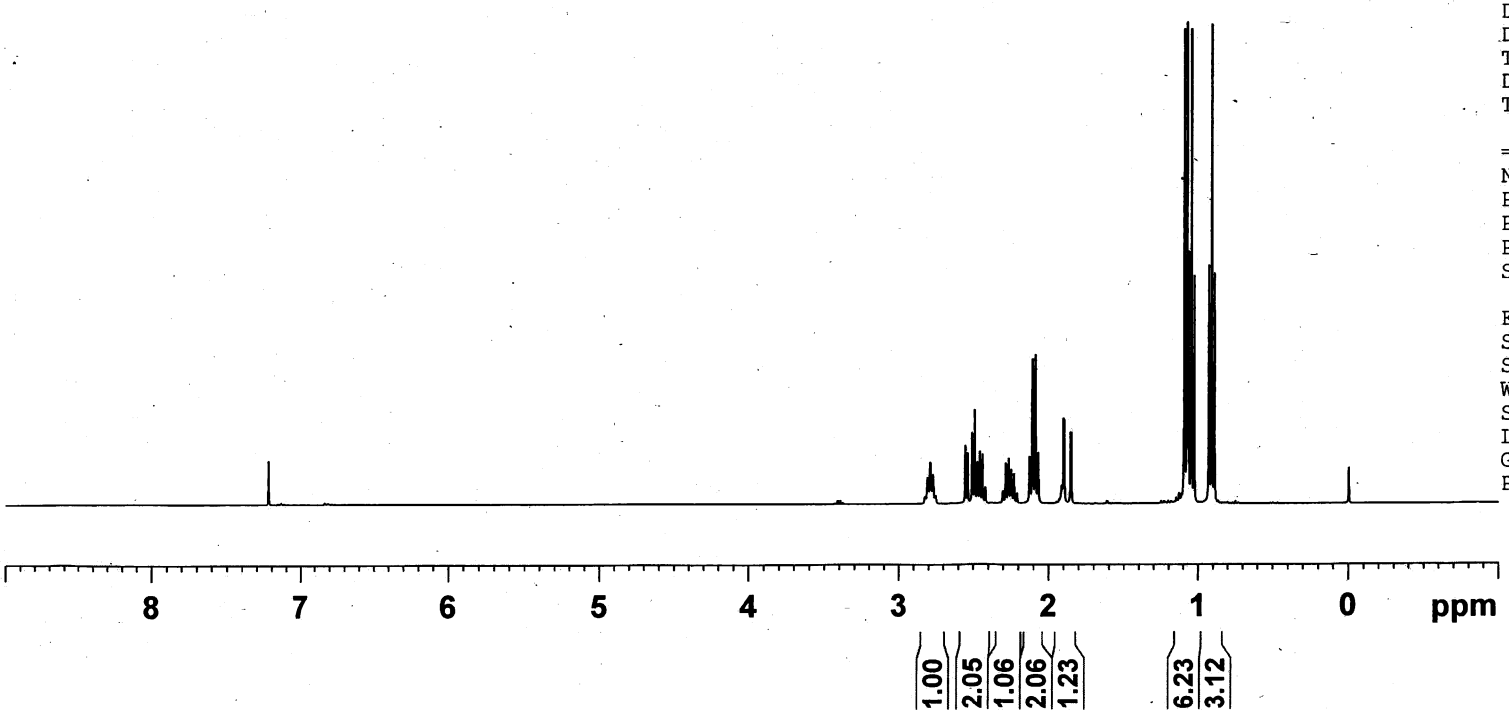
7ba

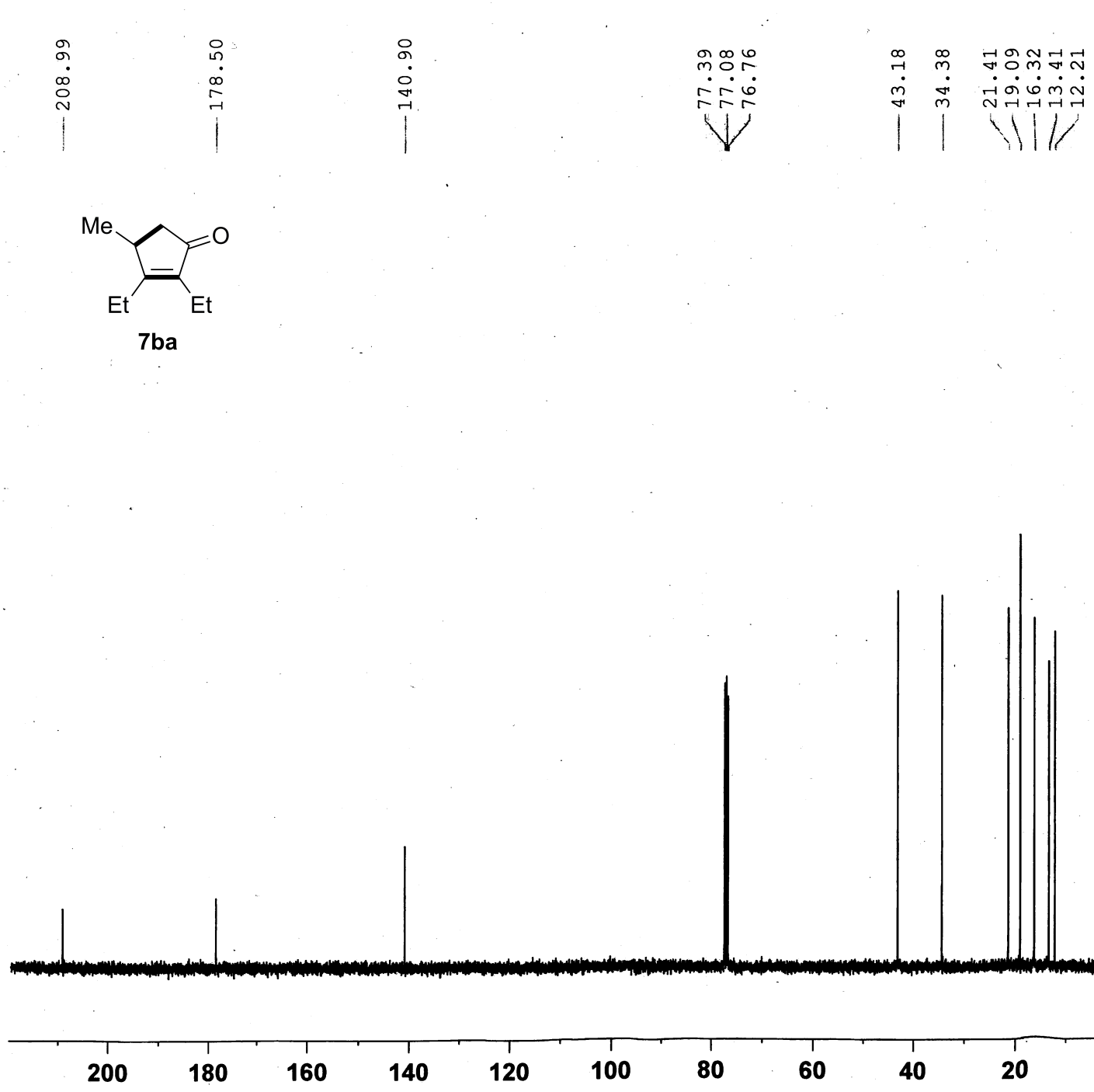
```
Current Data Parameters
NAME      10_PhCr_3-hexyne
EXPNO              45511
PROCNO                 1

F2 - Acquisition Parameters
Date_           20100506
Time              22.17
INSTRUM           spect
PROBHD   5 mm PABBO BB-
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8223.685 Hz
FIDRES         0.125483 Hz
AQ             3.9846387 sec
RG                   57
DW               60.800 usec
DE                6.50 usec
TE                296.8 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.00 usec
PL1               -1.00 dB
PL1W        13.34481144 W
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                32768
SF           400.1300306 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```





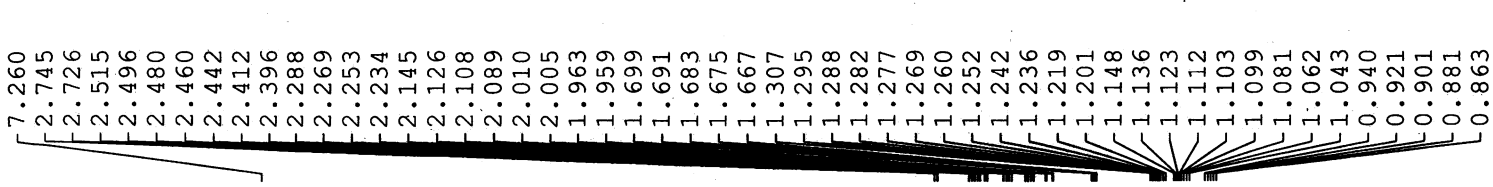
Current Data Parameters
NAME 10_PhCr_3-hexyne
EXPNO 45501
PROCNO 1

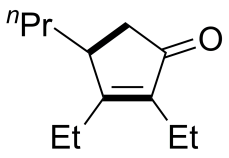
F2 - Acquisition Parameters
Date_ 20100506
Time 22.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 48
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

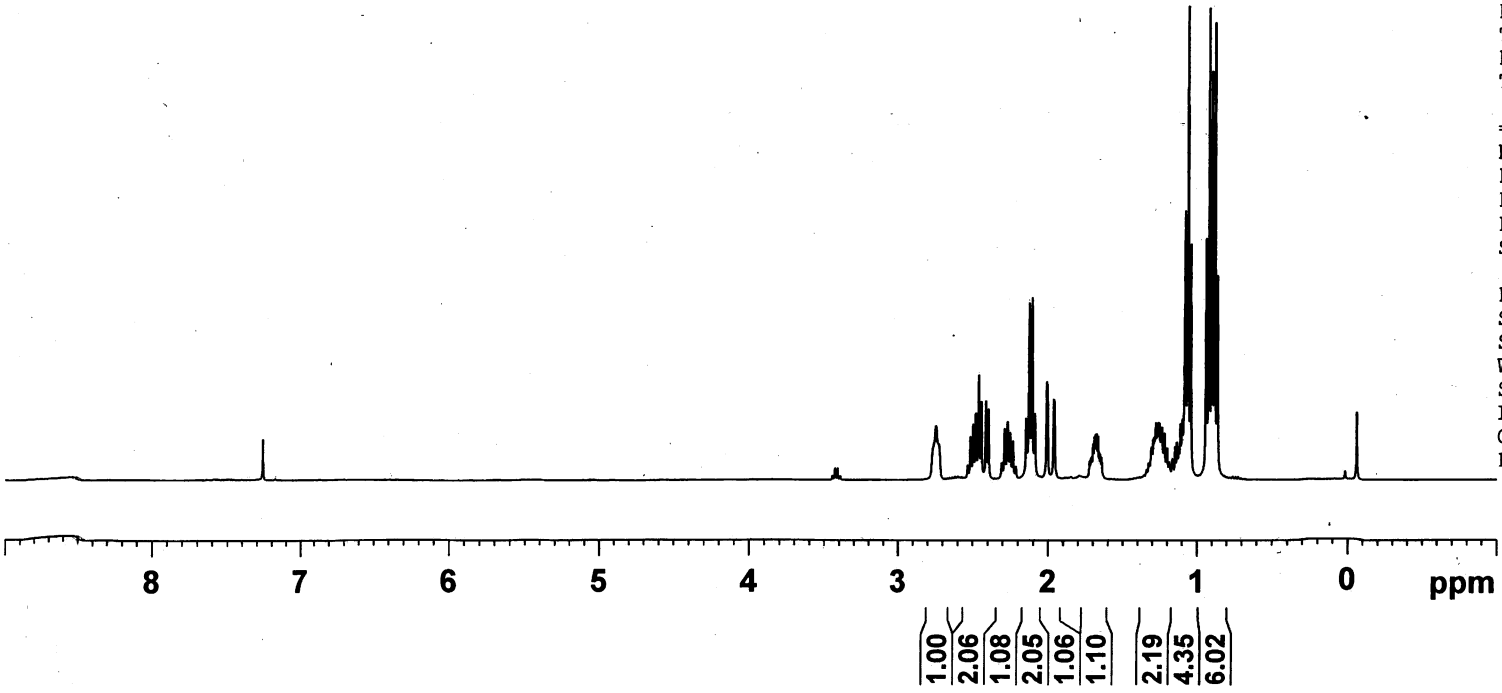
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





7ca

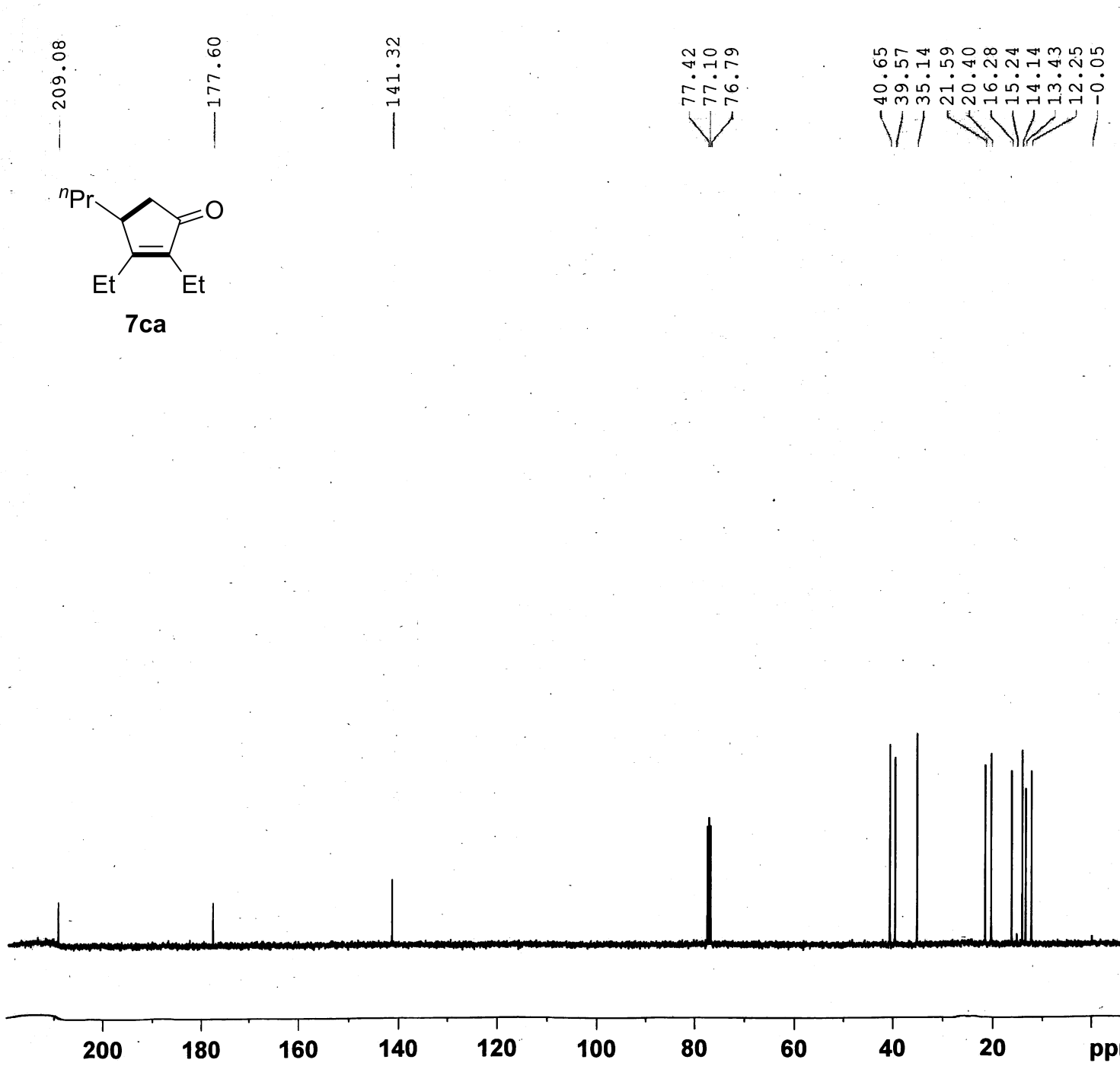



```
Current Data Parameters
NAME      11_PhHex_3-hexyne
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100423
Time                 17.39
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                      36
DW                 60.800 usec
DE                  6.50 usec
TE                  294.8 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.00 usec
PL1                 -1.00 dB
PL1W          13.34481144 W
SFO1          400.1324710 MHz

F2 - Processing parameters
SI                   32768
SF            400.1300170 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
```



BRUKER

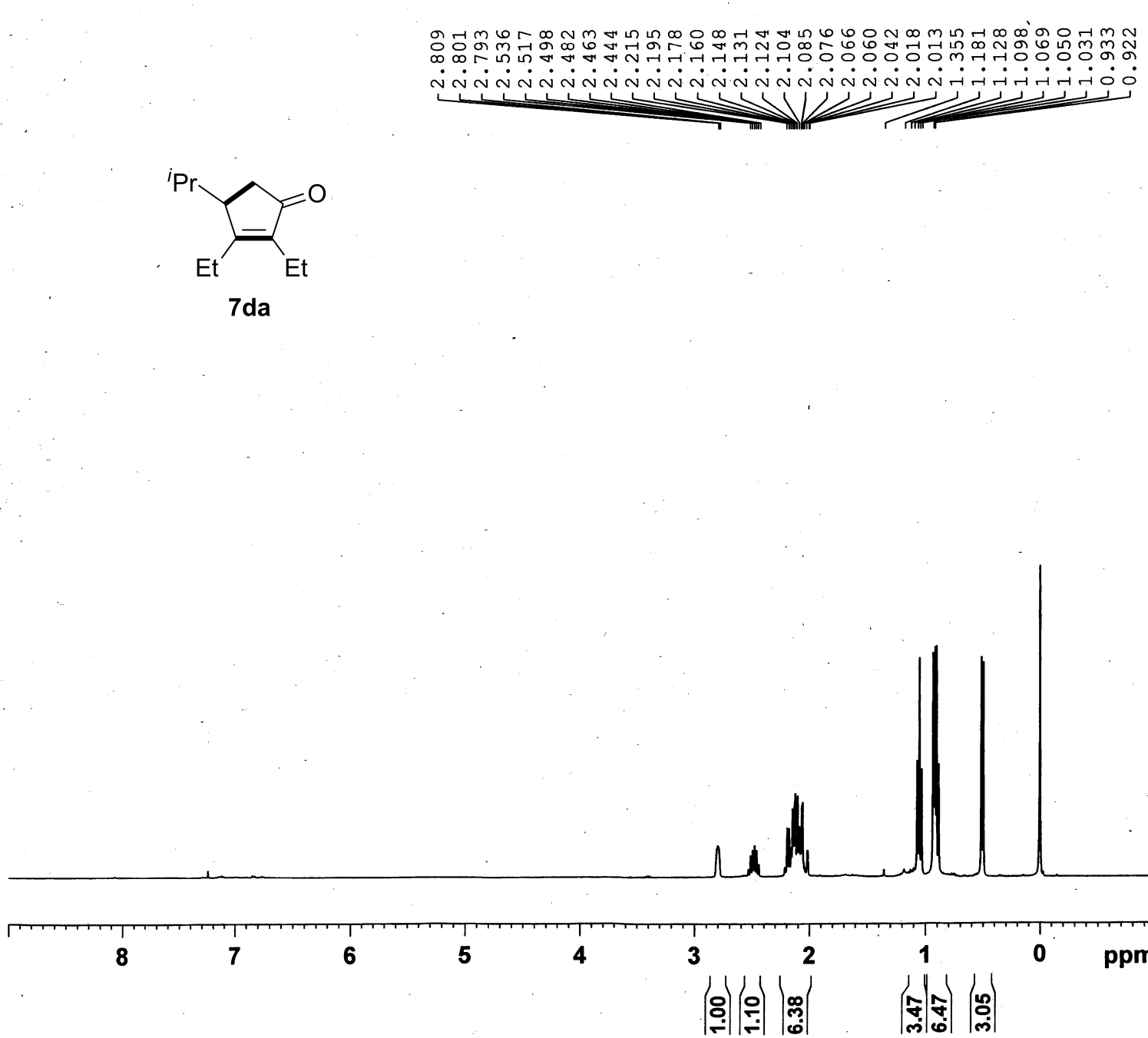
Current Data Parameters
NAME 11_PhHex_3-hexyne
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100423
Time 17.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 31
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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2.498
2.482
2.463
2.444
2.215
2.195
2.178
2.160
2.148
2.131
2.124
2.104
2.085
2.076
2.066
2.060
2.042
2.018
2.013
1.355
1.181
1.128
1.098
1.069
1.050
1.031
0.933
0.922
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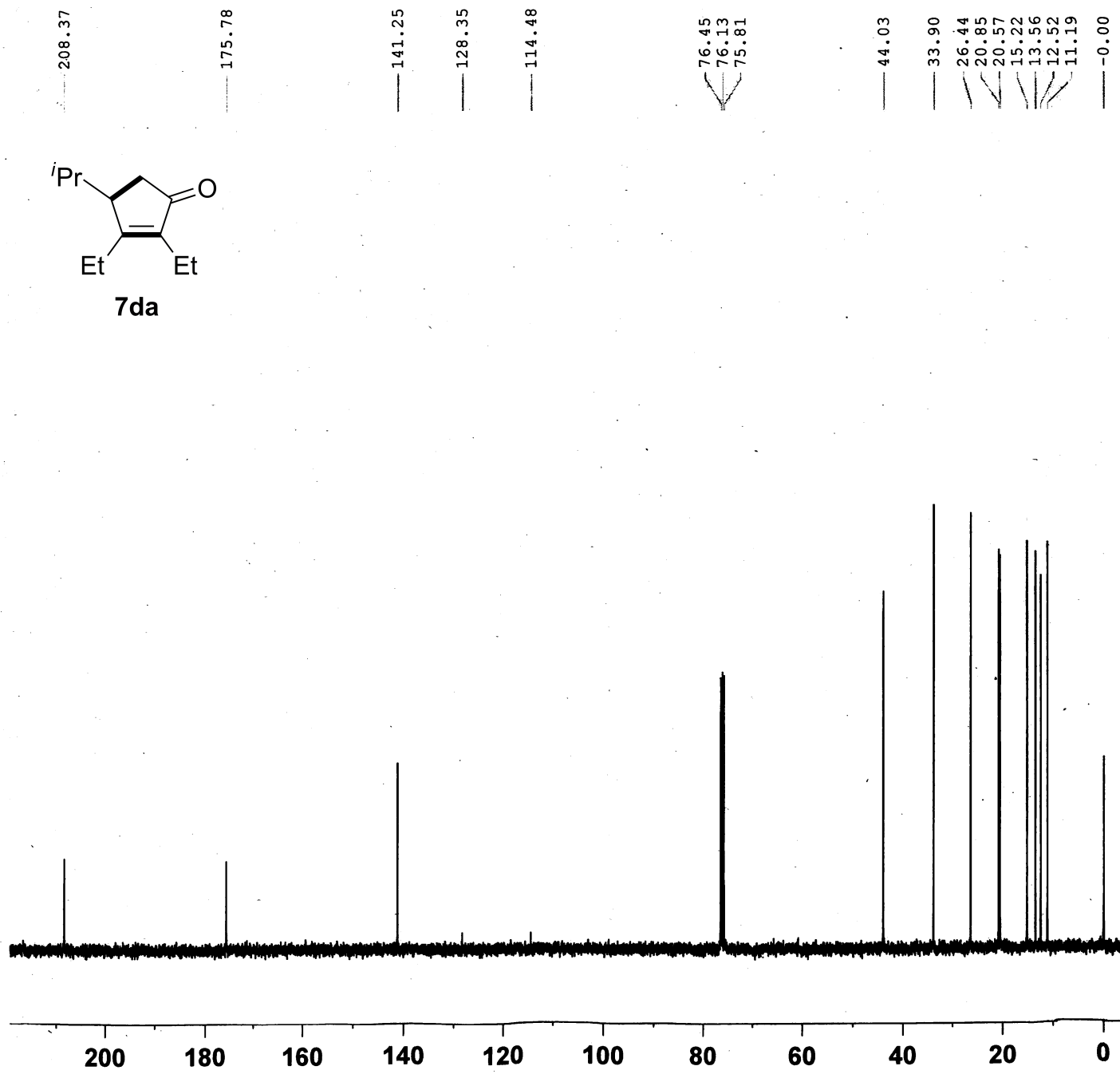
Current Data Parameters
NAME 12_PhMPen_3-hexyne
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100512
Time 18.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 25.4
DW 60.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.34481144 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300224 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0
ppm
1.00
1.10
6.38
3.47
6.47
3.05



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Current Data Parameters
NAME 12_PhMPen_3-hexyne
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_	20100512
Time	18.33
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	25
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	203
DW	19.800 usec
DE	6.50 usec
TE	296.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

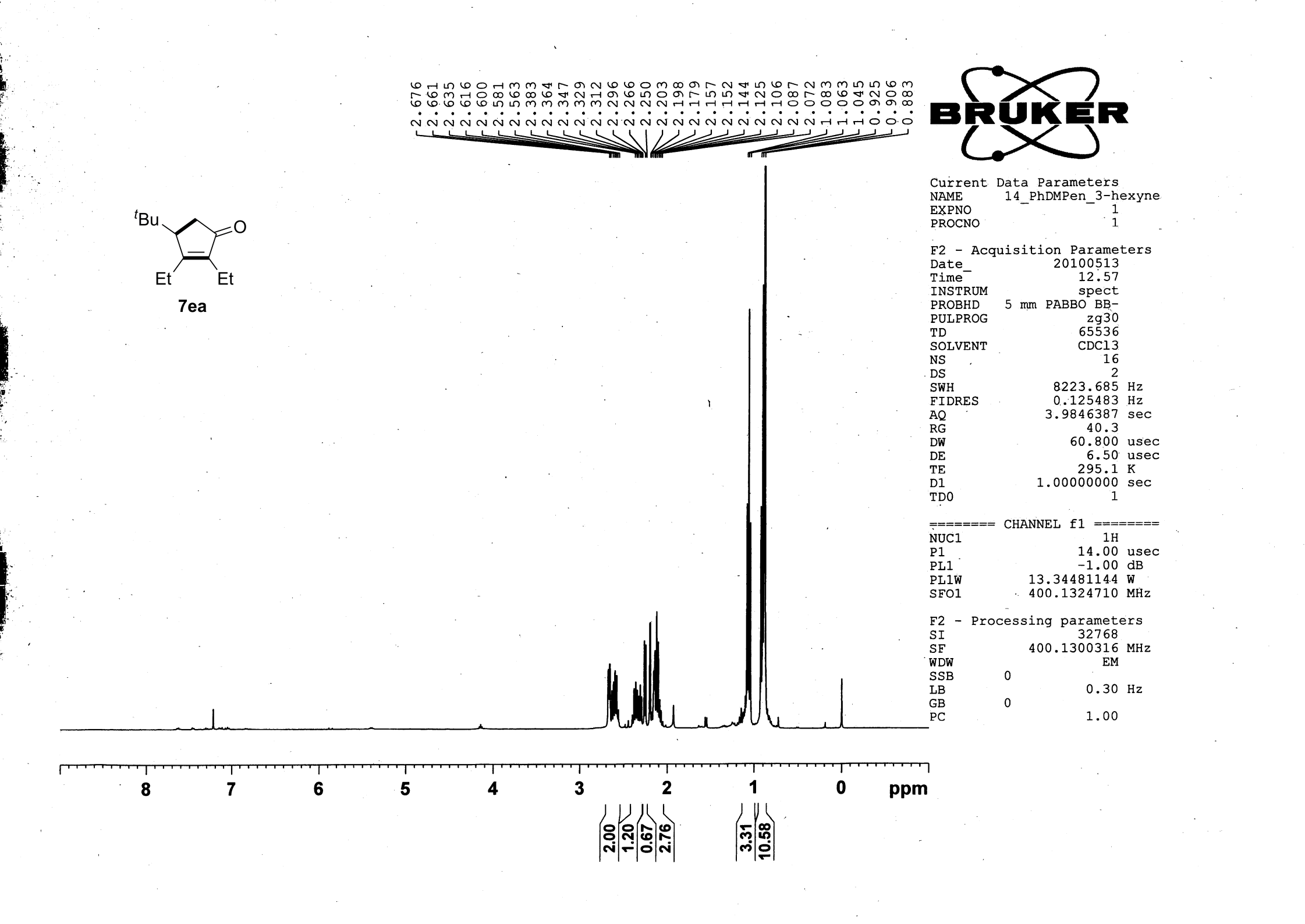
NUC1	13C
P1	10.00 usec
PL1	2.20 dB
PL1W	21.94663811 W
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	14.20 dB
PL13	15.00 dB
PL2W	13.34481144 W
PL12W	0.40300688 W
PL13W	0.33520651 W
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6128677 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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2.616
2.600
2.581
2.563
2.383
2.364
2.347
2.329
2.312
2.296
2.266
2.250
2.203
2.198
2.179
2.157
2.152
2.144
2.125
2.106
2.087
2.072
1.083
1.063
1.045
0.925
0.906
0.883
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Current Data Parameters
NAME 14_PhDMPen_3-hexyne
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100513
Time 12.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 40.3
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
PL1 -1.00 dB
PL1W 13.34481144 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 32768
SF 400.1300316 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
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ppm
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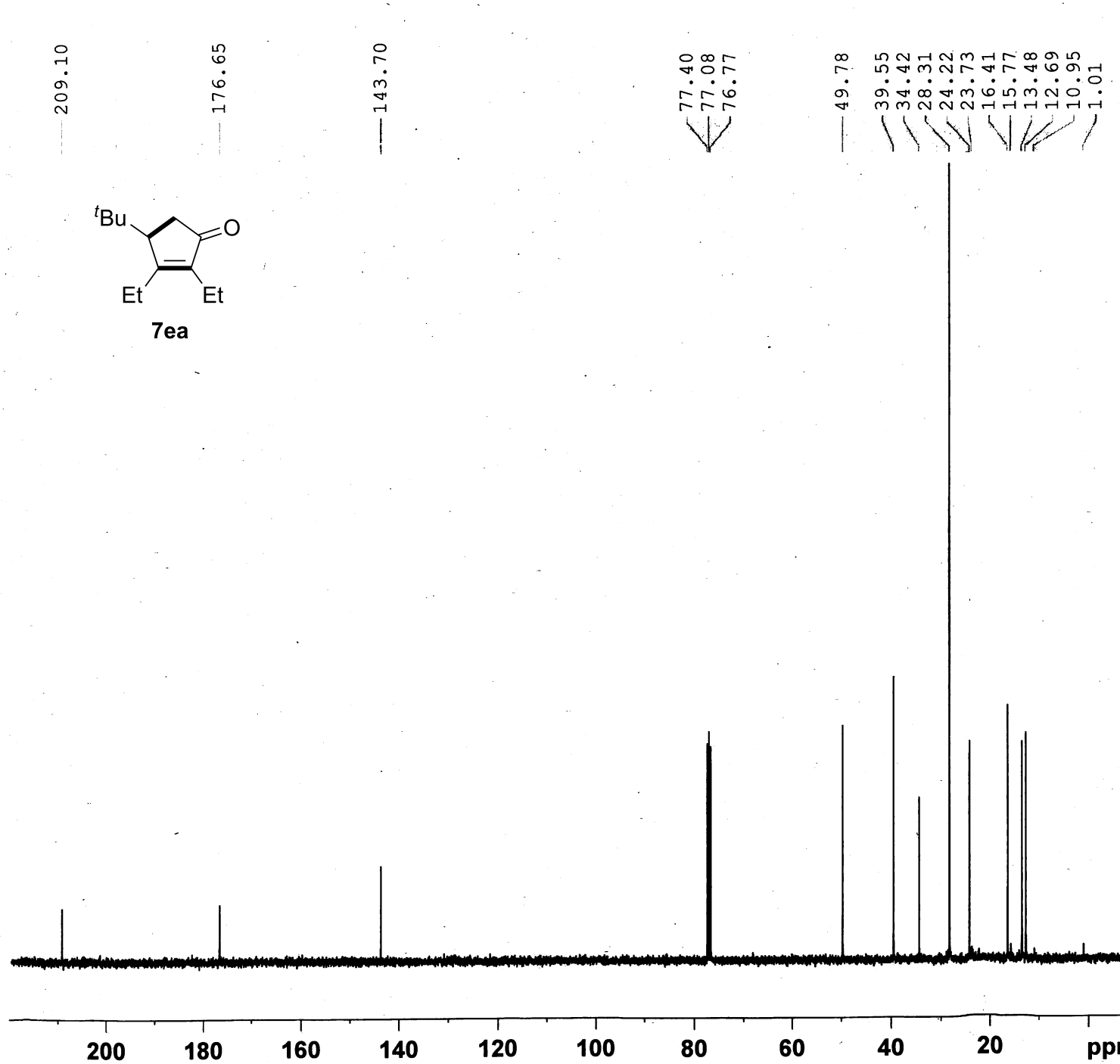
2.76
3.31
10.58



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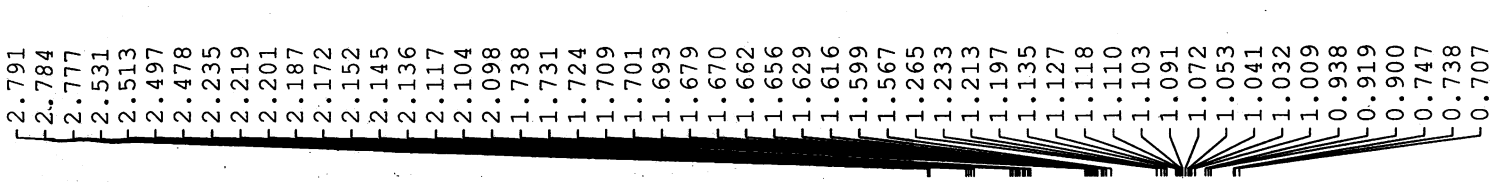
Current Data Parameters
NAME 14_PhDMPen_3-hexyne
EXPNO 13
PROCNO 1

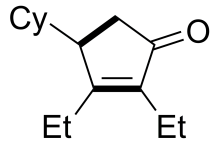
F2 - Acquisition Parameters
Date_ 20100513
Time 13.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 53
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PL1 2.20 dB
PL1W 21.94663811 W
SFO1 100.6228298 MHz

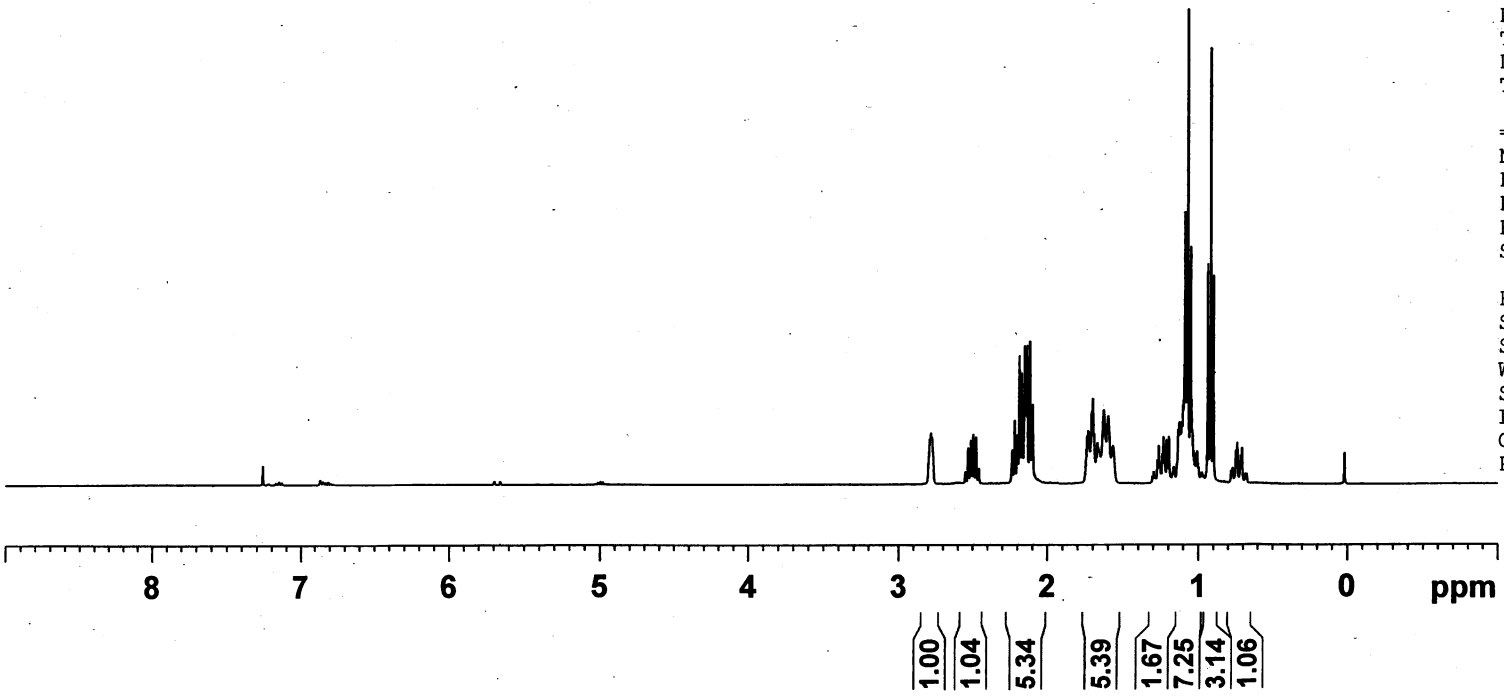
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 75.00 usec
PL2 -1.00 dB
PL12 14.20 dB
PL13 15.00 dB
PL2W 13.34481144 W
PL12W 0.40300688 W
PL13W 0.33520651 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





7fa

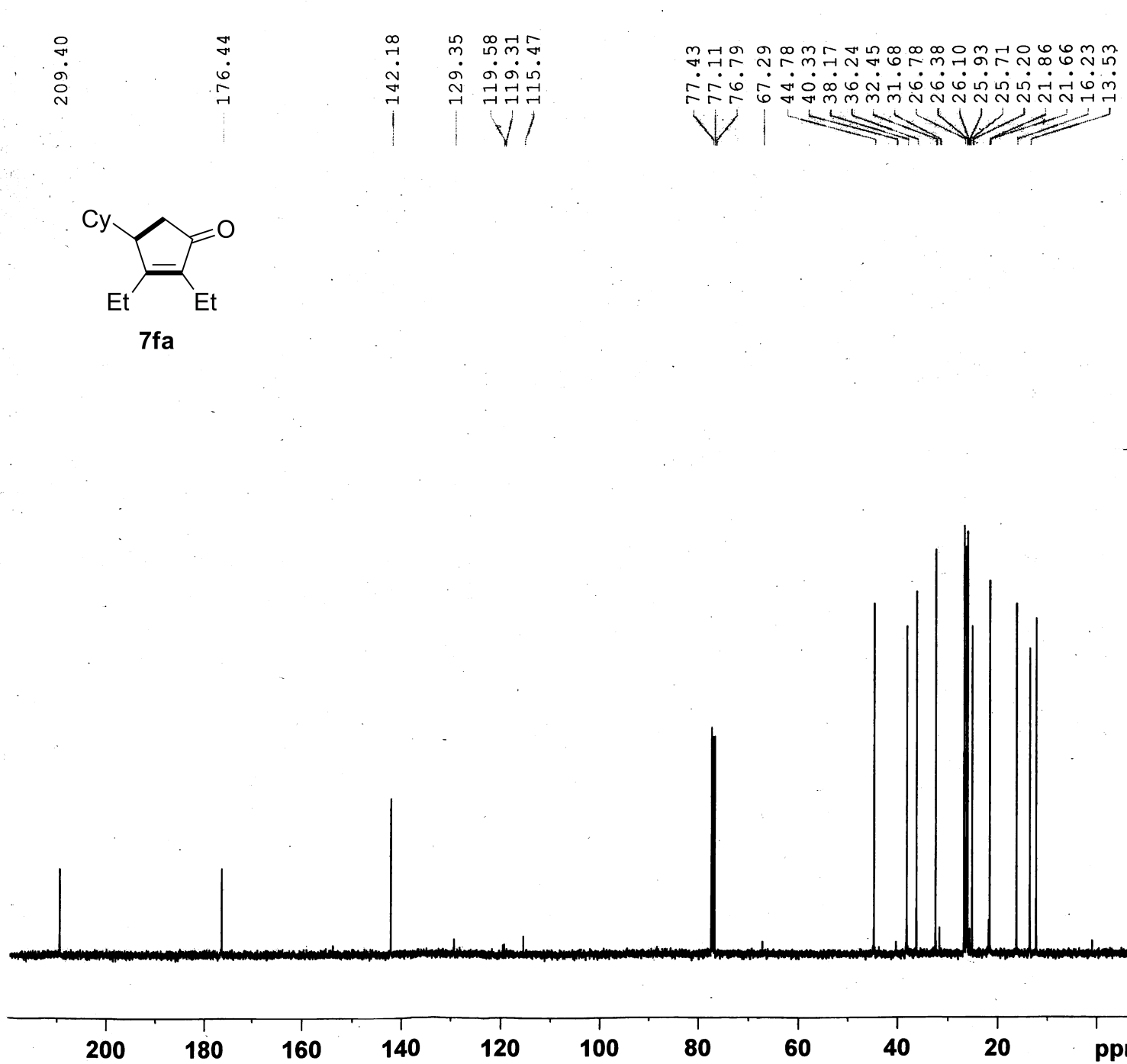



```
Current Data Parameters
NAME      13_PhCy_3-hexyne
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100518
Time                 23.07
INSTRUM              spect
PROBHD   5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                   28.5
DW                 60.800 usec
DE                  6.50 usec
TE                 297.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W         13.34481144 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300170 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```



Current Data Parameters

NAME	13_PhCy_3-hexyne
EXPNO	13
PROCNO	1

F2 - Acquisition Parameters

Date_	20100518
Time	23.10
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	49
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	297.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

======== CHANNEL f1 ========

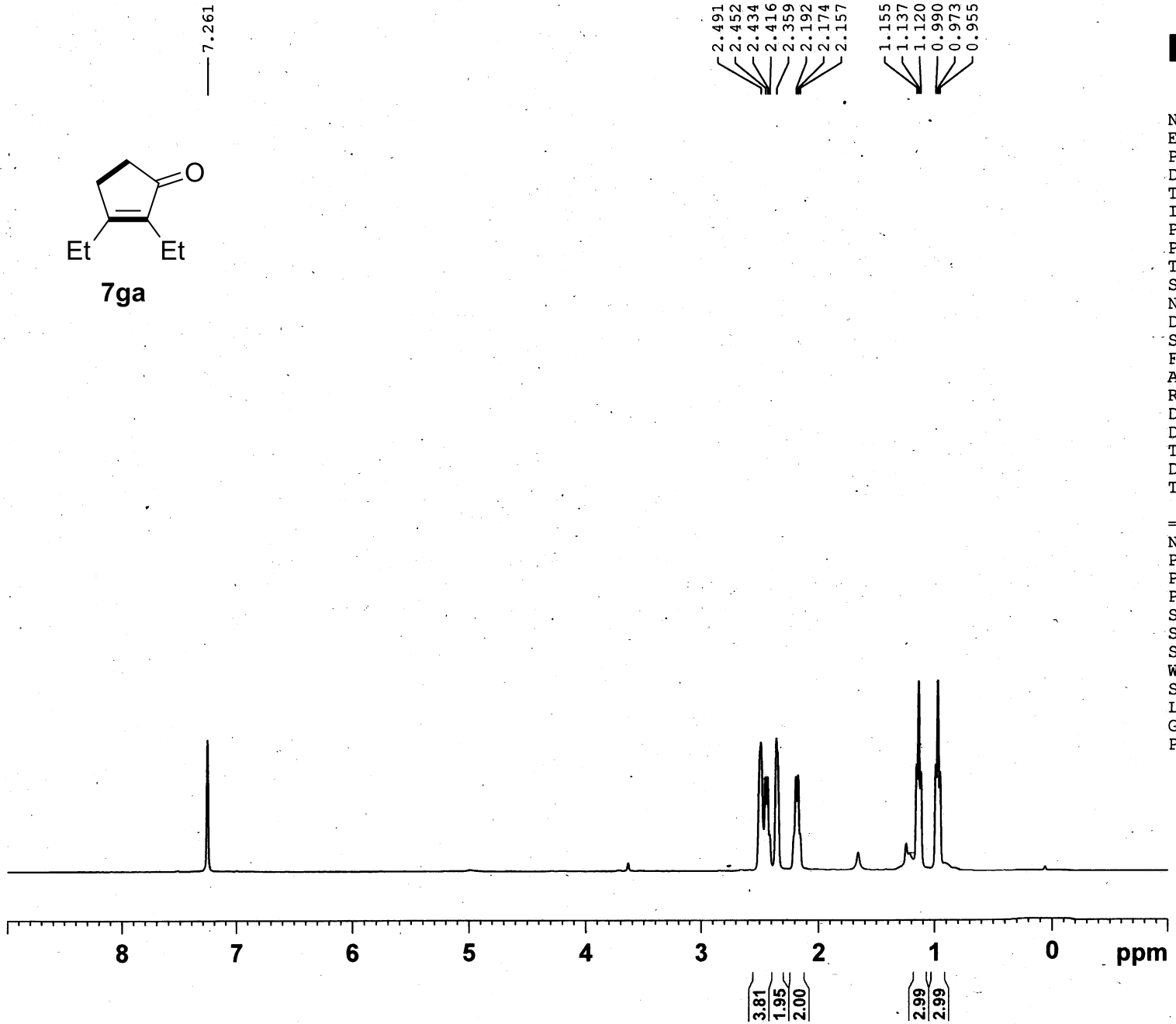
NUC1	13C
P1	10.00 usec
PL1	2.20 dB
PL1W	21.94663811 W
SFO1	100.6228298 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	75.00 usec
PL2	-1.00 dB
PL12	14.20 dB
PL13	15.00 dB
PL2W	13.34481144 W
PL12W	0.40300688 W
PL13W	0.33520651 W
SFO2	400.1316005 MHz

F2 - Processing parameters

SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



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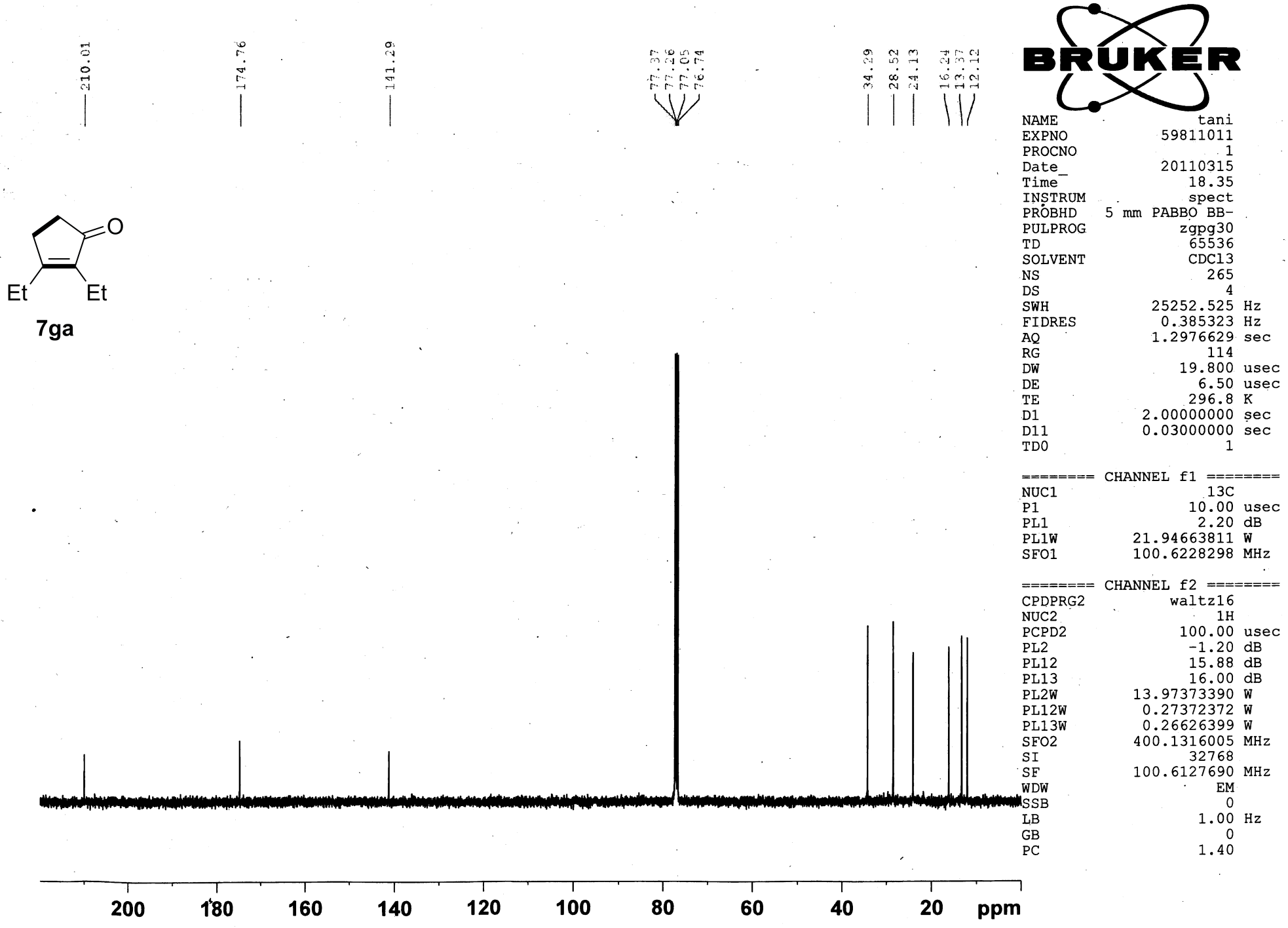
NAME	tani
EXPNO	59812010
PROCNO	1
Date_	20110315
Time	18.54
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	296.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.20 dB
PL1W	13.97373390 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300177 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



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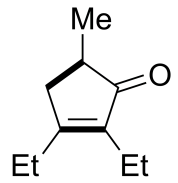
NAME	tani
EXPNO	59811011
PROCNO	1
Date_	20110315
Time	18.35
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	265
DS	4
SWH	25252.525 Hz
FIDRES	0.385323 Hz
AQ	1.2976629 sec
RG	114
DW	19.800 usec
DE	6.50 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	10.00 usec
PL1	2.20 dB
PL1W	21.94663811 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-1.20 dB
PL12	15.88 dB
PL13	16.00 dB
PL2W	13.97373390 W
PL12W	0.27372372 W
PL13W	0.26626399 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

PhMMA + 3-Hex



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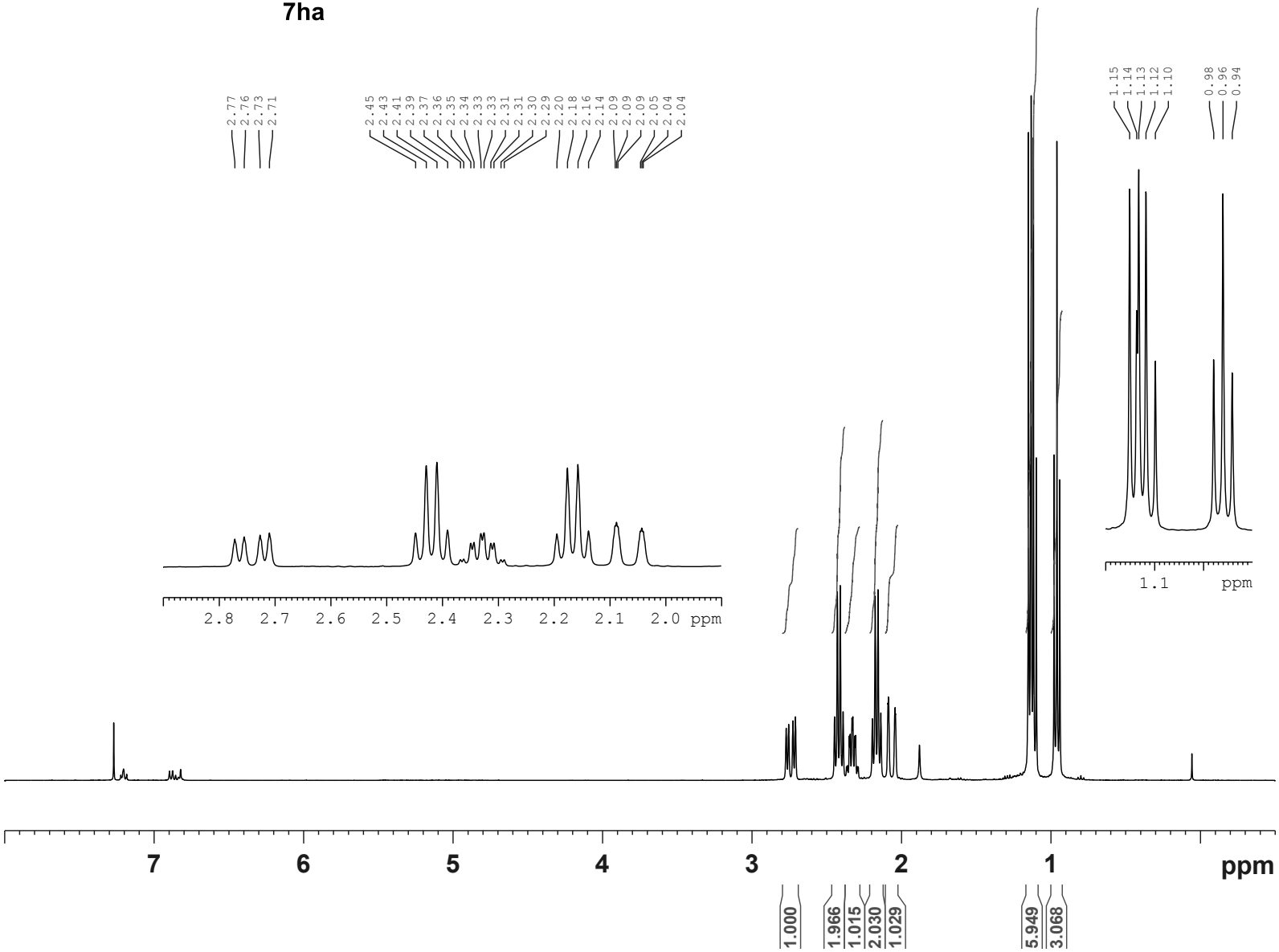
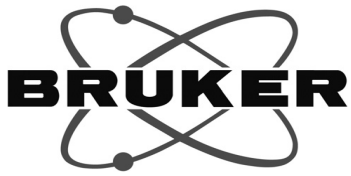




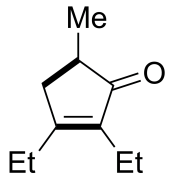

```
Current Data Parameters
NAME      15_PhMA_3-hexyne
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20100518
Time                 23.32
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     16
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ              3.9846387 sec
RG                     64
DW                 60.800 usec
DE                  6.50 usec
TE                 297.1 K
D1            1.00000000 sec
TD0                     1

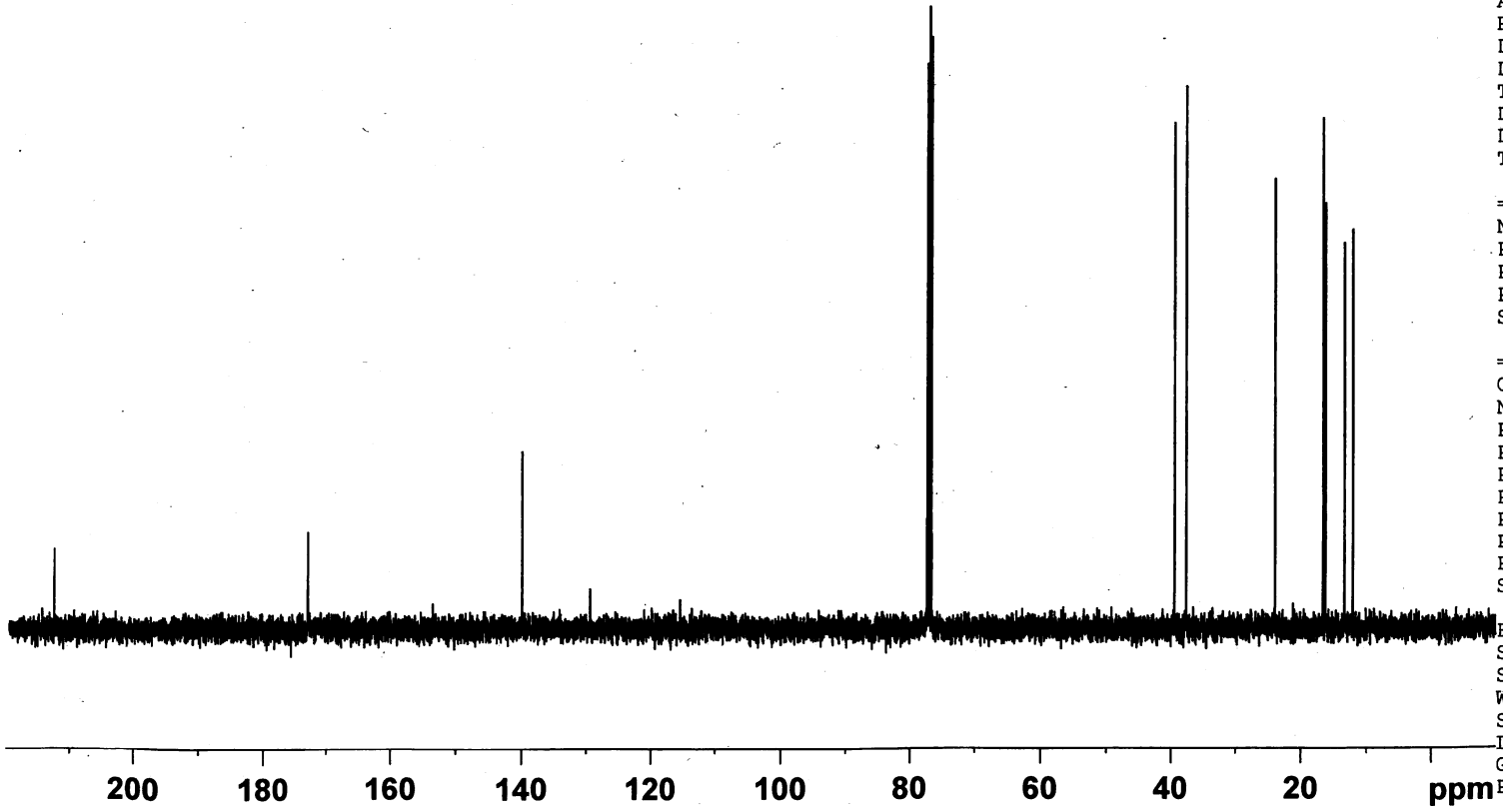
======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W         13.34481144 W
SFO1         400.1324710 MHz

F2 - Processing parameters
SI                  32768
SF           400.1300132 MHz
WDW                    EM
SSB       0
LB                  0.30 Hz
GB        0
PC                  1.00
```

PhMMA + 3-Hex



7ha



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```
Current Data Parameters
NAME      15_PhMA_3-hexyne
EXPNO                   13
PROCNO                   1

F2 - Acquisition Parameters
Date_              20100518
Time                 23.35
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      33
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     203
DW                  20.800 usec
DE                   6.50 usec
TE                  298.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   10.00 usec
PL1                   2.20 dB
PL1W         21.94663811 W
SFO1        100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2                75.00 usec
PL2                  -1.00 dB
PL12                 14.20 dB
PL13                 15.00 dB
PL2W         13.34481144 W
PL12W         0.40300688 W
PL13W         0.33520651 W
SFO2        400.1316005 MHz

F2 - Processing parameters
SI                    32768
SF           100.6127690 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```