Nitrile Assisted, Bronsted Acid Catalyzed Regio and Stereoselective Diarylphosphonylation of Allyl Silyl Ethers

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Experimental Procedures, Analytical and Spectroscopic Data for Compounds

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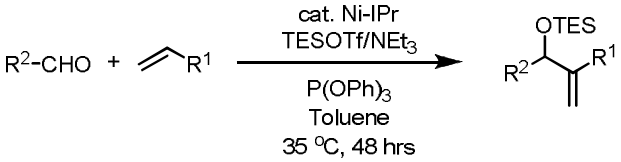
General Aspect.

p-Toluenesulfonic acid monohydrate and phosphorous trichloride were purchased from Acros Organics and used without further purification. Triphenyl phosphite was purchased from International Laboratory and used as received. Sodium hydride (60% in mineral oil) was purchased from Panreac Sintesis. Triethylamine and phosphorous trichloride were distilled over calcium hydride. All solvents were purchased from LAB-SCAN and used as received or dried according to the following procedures. THF: distilled over sodium/benzophenone. Toluene: distilled over calcium hydride. Analytical thin layer chromatography (TLC) was performed using EM Science silica gel 60 F254 plates. The developed chromatogram was analyzed by UV lamp (254 nm), ethanolic phosphomolybdic acid (PMA) or potassium permanganate ($KMnO_4$). Liquid chromatography was performed using a forced flow (flash chromatography) of the indicated solvent system on Silicycle Silica Gel (230–400 mesh). ^{1}H, ^{13}C and ^{31}P NMR spectra were recorded on Bruker 300 MHz or 400 MHz spectrometers in $CDCl_3$. Chemical shifts in ^{1}H NMR spectra are reported in ppm on the δ scale from an internal standard of residual tetramethylsilane (0 ppm). Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad), coupling constant in hertz (Hz), and integration. Chemical shifts of ^{13}C NMR spectra are reported in ppm from the central peak of $CDCl_3$ (77.16 ppm) on the δ scale. Chemical shifts of ^{31}P NMR spectra are reported in ppm referenced to internal $P(OPh)_3$ as standard on the δ scale (127.8 ppm with respect to 85% aq H_3PO_4 at 0 ppm, R. C. Seiceira, C. M. Higa, A. G. Barreto, J. F. Cajaiba da Silva, *Thermochimica Acta* 2005, **428**, 101. The use of internal standard help us to eliminate minor deviations observed when using external 85% aq H_3PO_4 alone. The δ difference between the two phosphonylation regioisomers can be quite small without the internal standard.). Infrared (IR) spectra were recorded on a Perkin−Elmer Spectrum One FT−IR. High resolution mass spectra (HRMS) were obtained on a Finnigan MAT 95XL GC Mass Spectrometer by Miss. Ng, Hau Yan of the Chinese University of Hong Kong, Department of Chemistry.

2/ Preparation of Allyl Silyl Ethers 1a-k.

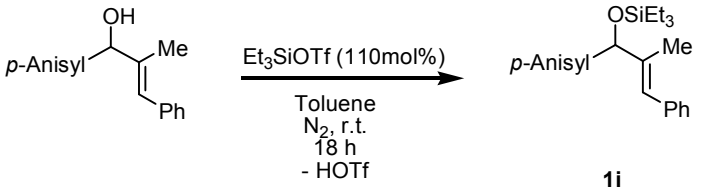
The synthesis of allyl silyl ethers **1a-1h**, **1j** and **1k** followed the literature procedure reported by Ho and Jamison.

Ho, C.-Y.; Jamison, T. F. *Angew. Chem. Int. Ed.* **2007**, *46*, 782-785.



The allylic alcohol below was synthesized by following the procedure reported by Takai. The allylic alcohol was treated with triethylsilyl trifluoromethanesulfonate in toluene to afford allyl silyl ether **1i**. Isolated yield: 82%.

Takai, K.; Sakamoto, S.; Isshiki, T. *Org. Lett.* **2003**, *5,* 653-655.



1i

3/ General Procedure for the Preparation of Triarylphosphites.

The literature procedure reported by Hernández was followed with some modifications.

Hernández, J.; Goycoolea, F. M.; Zepeda-Rivera, D.; Juárez-Onofre, J.; Martínez, K.; Lizardi, J.; Salas-Reyes, M.; Gordillo, B.; Velázquez-Contreras, C.; García-Barradas, O.; Cruz-Sánchezd , S.; Domínguez, Z. *Tetrahedron* **2006**, *62*, 2520-2528.

A 50 mL round bottom flask was equipped with a magnetic stirrer. Under an atmosphere of nitrogen, para-substituted phenols (1.72 mmol, 300 mol%) and sodium hydride (2.16 mmol, 360 mol%) were added to 20 mL dry THF at 0 ºC. After 10 min stirring, the solution was warmed to room temperature and continued to stir for 1 h. Then dry triethylamine (0.11 mmol, 20 mol%) and phosphorous trichloride (0.6 mmol, 100 mol%) were added. The mixture was stirred at room temperature for 20 h. After the removal of solvent under reduced pressure, 10 mL dry toluene was added to the solid residue. The milky white mixture was filtered through dry celite under nitrogen. The filtrate was concentrated and purified via flash chromatography on neutral aluminum oxide with dichloromethane (100 mL). It was then dried under reduced pressure at 90 ºC for 1 h to afford the desired products as the colourless liquid.

4/ General Procedure for the Allyl Diarylphosphonylation and Arylation.

A mixture of 0.1 mmol substrate* with 500 mol% of $P(OPh)_3$ and 5 mol% *p*-TsOH•H_2O in 2 mL CH_3CN (for phosphonylation) or 20 mol% *p*-TsOH•H_2O in 2 mL toluene (for arylation) was stirred at 0 ºC for 2 h in open air and then stirred at r.t. for 9 h. Solvent was removed under reduced pressure. The yield and the selectivity (average of at least two runs) were determined by ^1H NMR analysis using benzaldehyde as standard. Purification via flash chromatography on silica gel (using chloroform as eluent for phosphonylation or 20 % ethyl acetate in hexane as eluent for arylation, unless otherwise indicated), afforded the desired product as oil. The stereochemistry of the olefin was determined by NOESY using isolated product.

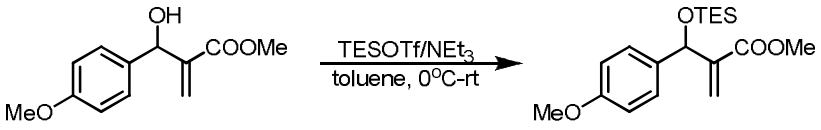
*Vigorous exclusion of other solvents (e.g. chloroform) is necessary, see S5.

Procedure: 1 mL CH_3CN was added to the substrate. Then solvent was removed under reduced pressure.

Procedure for Allyl Diarylphosphonylation of TES protected Baylis-Hillman adduct of methylacrylate and anisaldehyde:

General procedure for the allyl diarylphosphonylation of allyl silyl ethers was followed, except the reaction was conducted at 35 ºC.

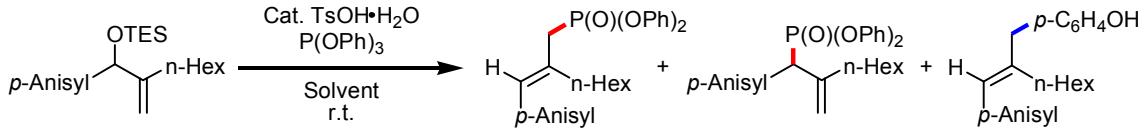
The TES protected Baylis-Hillman adduct of methyl acrylate and anisaldehyde was prepared according to literature procedure with modifications.



To a solution of 1 mmol Baylis-Hillman adduct in 2 mL toluene, 3 equiv. NEt_3 and 1.2 equiv. TESOTf was added at 0 ºC and stir at rt for overnight. Quantative yield.

F. Coelho, W. P. Almeida, C. R. Mateus, L. D. Furtado, J. C. F. Gouveia, *ARKIVOC*, 443.

5/ Solvent Effect on Allyl Diarylphosphonylation.



1a **2a** **2a'** **3a**

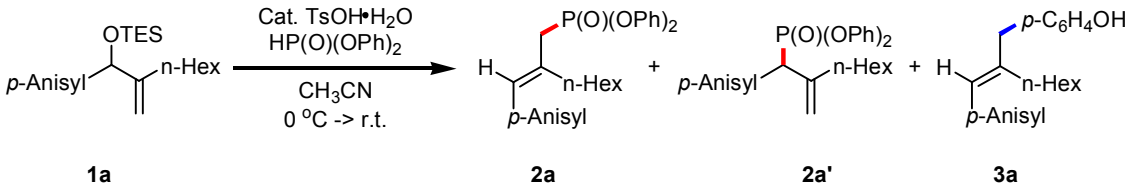
Entry[a]	Solvent	ε_r[b]	Yield % (**2a**+**2a'**:**3**)[c]	**2a**:**2a'** [c]
1[d]	$P(OPh)_3$	n.a.	**1a** recovered	-
2	$P(OPh)_3$	n.a.	90 (38:62)	75:25
3	Toluene	2.38	85 (5:95)	n.d.
4	NEt_3	2.4	**1a** recovered	-
5	$CHCl_3$	4.8	97 (5:95)	n.d.
6	THF	7.6	83 (5:95)	n.d.
7	Acetone	20.7	83 (5:95)	n.d.
8	CH_3NO_2	35.9	90 (24:76)	75:25
9	DMF	36.7	89 (14:86)	78:22
10	CH_3CN	37.5	95 (87:13)	75:25
11	PhCN	26.0	79 (81:19)	78:22

[a] The standard procedure was followed except that the reactions were carried out at r.t. in parallel, using 20 mol% of *p*-TsOH•H_2O and indicated solvent; [b] Dielectric constant; [c] Yield and selectivity were determined by ^1H NMR analysis using benzaldehyde as standard, the olefin stereochemistry was determined as *E*-isomer by NOESY using isolated product; [d] Control experiment without adding *p*-TsOH·H_2O and run in 500 mol% of $P(OPh)_3$.

Dielectric constant:

Landolt-Börnstein: Numerical Data and Functional Relationships in Science and Technology - New Series; Wohlfarth, C., Lechner, M. D., Eds; Group 4: Physical Chemistry; Springer Berlin Heidelberg, 2008; Vol.17.

6/ Phosphonylation Experiment using $HP(O)(OPh)_2$ in place of $P(OPh)_3$.

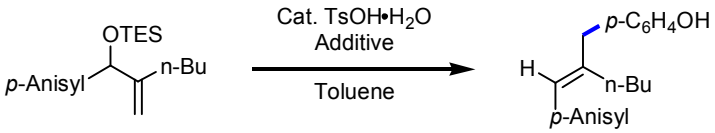


1a **2a** **2a'** **3a**

Entry[a]	Yield % (**2a**+**2a'**)[b]	Yield % (**3a**)[b]	*E*:*Z*[c]
1	No phosphonylation	53	>19:1

[a] The standard procedure was followed, except that $HP(O)(OPh)_2$ was used in place of $P(OPh)_3$; [b] Yield was determined by ^1H NMR analysis using benzaldehyde as standard; [c] Olefin stereochemistry of **3a** was determined by NOESY using isolated product.

7/ Competition Experiments using $P(OPh)_3$, $HP(O)(OPh)_2$ and PhOH in Catalytic Allyl Arylation.



1a **3a**

Entry[a]	Additive	Condition	Conversion %	Yield % (**3**)[b]	*E*:*Z*[c]
1	$P(OPh)_3$	0 ºC, 2h	0	0	n.a.
2		0 ºC, 2h and then r.t., 2h	92	78	>95:5
3	$HP(O)(OPh)_2$	0 ºC, 2h	81	33	>95:5
4		0 ºC, 2h and then r.t., 2h	87	38	>95:5
5	PhOH	0 ºC, 2h	100	92	>95:5

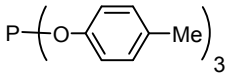
[a] The standard procedure was followed, except that $HP(O)(OPh)_2$ or PhOH was used as indicated above; [b] Yield was determined by ^1H NMR analysis using benzaldehyde as standard; [c] Olefin stereochemistry was determined by NOESY using isolated product.

Compound Characterization Data.

8/ Triarylphosphites.

The spectroscopic data was comparable with the literature:

Nikoletta, S.; Stratakis, M. *ARKIVOC* **2003**, 30-35.

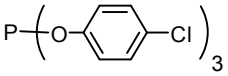
Tris(4-methylphenyl) phosphite **P1** 

The standard procedure was followed, except that 4-methylphenol was used. Yield: 92%.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.10 (d, J = 8.2 Hz, 6H), 7.02 (d, J = 8.2 Hz, 6H), 2.31 (s, 9H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 149.47, 133.78, 130.26, 120.65 (d, J = 7 Hz, ortho), 20.87.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 128.11.

Tris(4-chlorophenyl) phosphite **P2** 

The standard procedure was followed, except that 4-chlorophenol was used. Yield: 88%.

^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.29 (d, J = 9.0 Hz, 6H), 7.04 (d, J = 9.0 Hz, 6H).

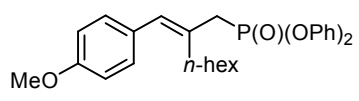
^{13}C NMR (100 MHz, $CDCl_3$) δ: 130.29, 129.96, 129.56, 122.00 (d, J = 5 Hz, ortho), 116.80.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 126.43.

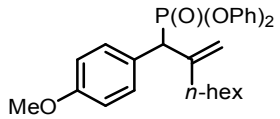
HRMS-EI (m/z): $[M]^+$ calcd for $C_{18}H_{12}Cl_3O_3P$, 412.9662; found, 412.9692.

9/ Phosphonylation products (Table 1).

Table 1, entry 1:



2a



2′a

(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{28}H_{33}O_4PNa$, 487.2009; found, 487.2013.

2a: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.33-7.29 (m, 5H), 7.19-7.10 (m, 7H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.48 (d, *J* = 6.1 Hz, 1H), 3.82 (s, 3H), 3.05 (d, *J* = 22.3 Hz, 2H), 2.47-2.42 (m, 2H), 1.54-1.42 (m, 2H), 1.35-1.25 (m, 6H), 0.88-0.83 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.21, 150.54, 150.45, 131.07, 130.95, 130.65, 130.52, 129.68, 124.98, 120.50, 120.46, 113.53, 55.19, 34.46 (d, *J* = 137 Hz), 31.54, 31.27, 29.13, 27.88, 22.54, 14.00.

IR (neat) cm^{-1}: 2920, 2850, 1725, 1592, 1509, 1488, 1273, 1249, 1214, 1189, 1025, 927, 759.

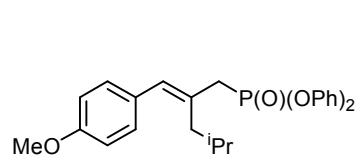
^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.36.

2′a: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 (d, *J* = 8.8 Hz, 2H), 7.31-7.02 (m, 8H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 5.69 (d, *J* = 2.8 Hz, 1H), 5.19 (d, *J* = 2.8 Hz, 1H), 4.06 (d, *J* = 24.8 Hz, 1H), 3.80 (s, 3H), 2.07-1.90 (m, 2H), 1.41-1.37 (m, 2H), 1.36-1.20 (m, 6H), 0.89-0.83 (m, 3H).

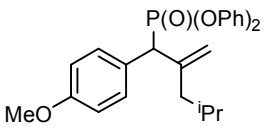
IR (neat) cm^{-1}: 2926, 2855, 1591, 1509, 1489, 1271, 1214, 1188, 1162, 1025, 927, 759, 688, 617, 589, 500.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 18.42.

Table 1, entry 5:



2b



2′b

(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{26}H_{29}O_4PNa$, 459.1696; found, 459.1691.

2b: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.33-7.29 (m, 4H), 7.20-7.11 (m, 8H), 6.85 (d, *J* = 8.7 Hz, 2H), 6.57 (d, *J* = 6.3 Hz, 1H), 3.81 (s, 3H), 3.06 (d, *J* = 22.4 Hz, 2H), 2.38 (dd, *J* = 7.4, 2.4 Hz, 2H), 1.75-1.97 (m, 1H), 0.84 (d, *J* = 6.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.33, 150.74, 150.65, 132.04, 131.91, 130.43, 130.21, 130.18, 129.89, 125.18, 120.69, 120.64, 113.66, 55.37, 39.76, 34.84 (d, *J* = 137 Hz), 26.69, 22.48.

IR (neat) cm^{-1}: 2921, 2851, 1593, 1510, 1490, 1464, 1270, 1250, 1215, 1190, 1026, 930, 765, 689.

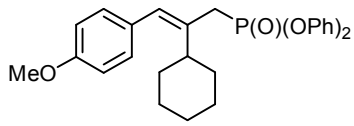
^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.41.

2′b: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 (d, *J* = 8.8 Hz, 2H), 7.31-7.02 (m, 6H), 7.05 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 8.0 Hz, 2H), 6.72 (d, *J* = 8.8 Hz, 2H), 5.76 (d, *J* = 2.8 Hz, 1H), 5.18 (d, *J* = 2.8 Hz, 1H), 4.04 (d, *J* = 25.2 Hz, 1H), 3.80 (s, 3H), 2.35 (d, *J* = 7.4 Hz, 2H), 1.70-1.81 (m, 1H), 0.84 (d, *J* = 6.6 Hz, 6H).

IR (neat) cm^{-1}: 2954, 2923, 2852, 1727, 1593, 1510, 1490, 1465, 1384, 1274, 1250, 1214, 1190, 1163, 1072, 1026, 930, 760, 689, 617.

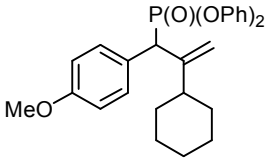
^{31}P NMR (121 MHz, $CDCl_3$) δ: 19.02.

Table 1, entry 6:



2c

(minor, more polar)



2′c

(major, less polar)

The standard procedure was followed.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{28}H_{31}O_4PNa$, 485.1852; found, 485.1845.

2c: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32-7.28 (m, 5H), 7.19-7.10 (m, 7H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.72 (d, *J* = 5.2 Hz, 1H), 3.82 (s, 3H), 3.01 (d, *J* = 22.8 Hz, 2H), 2.84-2.78 (m, 1H), 1.76-1.08 (m, 10H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.34, 150.79, 150.69, 135.09, 134.99, 130.14, 130.01, 129.99, 129.87, 129.75, 129.65, 125.14, 120.76, 120.72, 113.73, 55.39, 40.37, 40.31, 31.11, 30.01, 29.23 (d, *J* = 123 Hz), 26.21, 26.08.

IR (neat) cm^{-1}: 2918, 2849, 1724, 1589, 1506, 1488, 1270, 1248, 1214, 1189, 1025, 926, 760, 688.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 21.55.

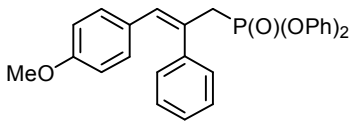
2′c: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 8.8 Hz, 2H), 7.30-7.03 (m, 8H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.6 Hz, 2H), 5.77 (d, *J* = 3.2 Hz, 1H), 5.23 (d, *J* = 3.2 Hz, 1H), 4.12 (d, *J* = 25.3 Hz, 1H), 3.80 (s, 3H), 1.88-1.86 (m, 1H), 1.74-1.05 (m, 10H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.20, 150.93, 150.73, 148.88, 131.21, 131.13, 129.99, 129.87, 129.70, 129.55, 125.00, 124.85, 120.74, 120.64, 114.10, 113.88, 113.81, 55.42, 48.85 (d, *J* = 138 Hz), 45.74, 45.63, 32.80, 32.35, 26.83, 26.68, 26.33.

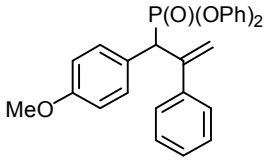
IR (neat) cm^{-1}: 2919, 2850, 1725, 1591, 1509, 1489, 1454, 1251, 1213, 1187, 1161, 1070, 1025, 927, 759, 678.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 18.67.

Table 1, entry 7:



2d



2′d

(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{28}H_{25}O_4PNa$, 479.1383; found, 479.1412.

2d: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32-7.24 (m, 9H), 7.13 (dd, *J* = 8.0, 7.6 Hz, 2H), 7.03 (d, *J* = 8.4 Hz, 4H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.68 (d, *J* = 6.0 Hz, 1H), 6.64 (d, *J* = 8.8 Hz, 2H), 3.73 (s, 3H), 3.38 (d, *J* = 22.0 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ: 159.05, 151.03, 150.91, 140.97, 132.33, 132.16, 130.97, 130.26, 129.64, 129.32, 128.05, 125.59, 121.09, 121.03, 113.95, 55.72, 38.12 (d, *J* = 137 Hz).

IR (neat) cm^{-1}: 2918, 2850, 1722, 1589, 1509, 1488, 1274, 1250, 1213, 1188, 1024, 927, 758, 689.

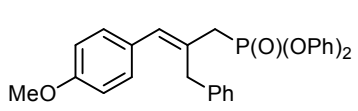
^{31}P NMR (121 MHz, $CDCl_3$) δ: 19.46.

2′d: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.51-7.49 (m, 2H), 7.45-7.29 (m, 4H), 7.23-7.14 (m, 4H), 7.09-7.07 (m, 3H), 6.90-6.86 (m, 4H), 6.80 (d, *J* = 8.5 Hz, 2H), 6.15 (d, *J* = 3.1 Hz, 1H) 5.71 (d, *J* = 3.1 Hz, 1H) 4.63 (d, *J* = 25.6 Hz, 1H), 3.79 (s, 3H).

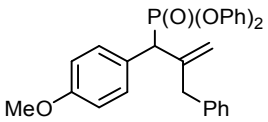
IR (neat) cm^{-1}: 2918, 2850, 1725, 1590, 1509, 1488, 1465, 1384, 1252, 1212, 1182, 1025, 927, 761, 688, 500.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 17.88.

Table 1, entry 8:



2e (major, more polar)



2′e (minor, less polar)

The standard procedure was followed.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{29}H_{27}O_4PNa$, 493.1539; found, 493.1529.

2e: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.37-7.14 (m, 17H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.73 (d, *J* = 6.1 Hz, 1H), 3.88 (d, *J* = 2.4 Hz, 2H), 3.79 (s, 3H), 2.92 (d, *J* = 22.1 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ: 158.76, 150.71, 150.59, 138.84, 132.66, 132.49, 130.00, 129.89, 128.93, 129.77, 126.53, 125.74, 125.20, 120.67, 120.61, 113.91, 55.38, 37.13, 34.20 (d, *J* = 137 Hz).

IR (neat) cm^{-1}: 3061, 3027, 2924, 2836, 1591, 1510, 1490, 1455, 1270, 1251, 1214, 1189, 1162, 1026, 930, 761, 689.

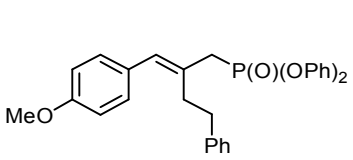
^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.08.

2′e: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (d, *J* = 8.8 Hz, 2H), 7.37-7.01 (m, 13H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.71 (d, *J* = 7.2 Hz, 2H), 5.81 (d, *J* = 2.8 Hz, 1H), 5.21 (d, *J* = 2.8 Hz, 1H), 4.00 (d, *J* = 25.2 Hz, 1H), 3.80 (s, 3H), 3.49 (d, *J* = 15.2 Hz, 1H), 3.27 (d, *J* = 15.2 Hz, 1H).

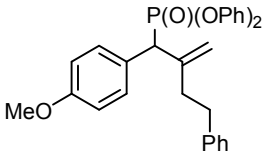
IR (neat) cm^{-1}: 2954, 2922, 2851, 1723, 1590, 1509, 1489, 1463, 1384, 1250, 1213, 1188, 1073, 1025, 928, 823, 758, 689.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 17.95.

Table 1, entry 9:



2f



2′f

(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{30}H_{29}O_4PNa$, 507.1696; found, 507.1688.

2f: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32-7.11 (m, 15H), 7.06 (d, *J* = 8.8 Hz, 2H), 6.84 (d, *J* = 8.8 Hz, 2H), 6.52 (d, *J* = 6.2 Hz, 1H), 3.81 (s, 3H), 3.05 (d, *J* = 22.2 Hz, 2H), 2.84-2.77 (m, 4H).

^{13}C NMR (75 MHz, $CDCl_3$) δ: 158.50, 150.67, 150.55, 141.35, 131.90, 131.72, 129.91, 128.52, 126.16, 125.25, 120.70, 120.64, 113.77, 55.40, 35.02 (d, *J* = 137 Hz), 33.25.

IR (neat) cm^{-1}: 2922, 2852, 1592, 1509, 1490, 1273, 1250, 1204, 1190, 1026, 929, 760, 689.

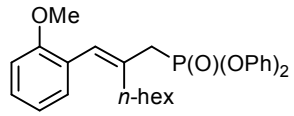
^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.09.

2′f: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.42 (d, *J* = 8.8 Hz, 2H), 7.20-7.02 (m, 13H), 6.86 (d, *J* = 8.4 Hz, 2H), 6.74 (d, *J* = 8.4 Hz, 2H), 5.74 (d, *J* = 2.4 Hz, 1H), 5.23 (d, *J* = 2.4 Hz, 1H), 4.01 (d, *J* = 25.2 Hz, 1H), 3.82 (s, 3H), 2.52-2.41 (m, 2H), 2.38-2.28 (m, 2H).

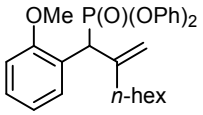
IR (neat) cm^{-1}: 2919, 2851, 1724, 1591, 1509, 1489, 1465, 1384, 1274, 1252, 1213, 1189, 1073, 1025, 929, 823, 759, 689.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 17.98.

Table 1, entry 10:



2g



2′g

(minor) (major)

The standard procedure was followed, except that CH_3CN was distilled over calcium hydride before use, 100 mol% *p*-TsOH•H_2O was used, the reaction mixture was stirred at 35 °C for 18 hours and the product was purified with 5% diethyl ether in toluene. Isolated as a mixture of **2g** and **2′g**.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{28}H_{33}O_4PNa$, 487.2009; found, 487.2039.

2g: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.37-6.82 (m, 14H), 6.61 (d, *J* = 6.2 Hz, 1H), 3.78 (s, 3H), 3.11 (d, *J* = 22.3 Hz, 2H), 2.43-2.38 (m, 2H), 1.49-1.44 (m, 2H), 1.36-1.21 (m, 6H), 0.86-0.81 (m, 3H).

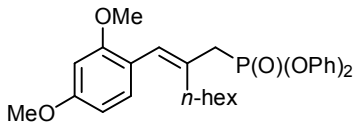
2′g: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75 (d, *J* = 7.8 Hz, 2H), 7.37-6.82 (m, 10H), 6.77 (d, *J* = 8.6 Hz, 2H), 5.67 (d, *J* = 2.9 Hz, 1H), 5.16 (d, *J* = 2.9 Hz, 1H), 4.88 (d, *J* = 25.4 Hz, 1H), 3.77 (s, 3H), 2.17-1.99 (m, 2H), 1.42-1.38 (m, 2H), 1.36-1.21 (m, 6H), 0.86-0.81 (m, 3H).

2g+2′g: ^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.26, 157.10, 150.99, 150.90, 150.74, 150.64, 144.29, 132.36, 132.25, 130.85, 130.79, 130.01, 129.82, 129.67, 129.42, 128.85, 128.34, 127.28, 127.15, 126.52, 125.76, 125.14, 124.94, 124.69, 123.12, 120.88, 120.83, 120.78, 120.74, 120.47, 120.43, 120.29, 120.24, 120.19, 115.22, 115.14, 110.69, 110.48, 55.74, 55.41, 41.07 (d, *J* = 140 Hz), 37.16, 37.06, 34.34 (d, *J* = 137 Hz), 31.82, 31.71, 31.48, 29.19, 28.90, 28.03, 27.49, 22.73, 22.69.

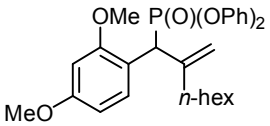
IR (neat) cm^{-1}: 2927, 2856, 1593, 1490, 1454, 1271, 1247, 1215, 1190, 1026, 929, 755, 689.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.53, 19.13.

Table 1, entry11:



2h



2′h

(minor, more polar) (major, less polar)

The standard procedure was followed.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{29}H_{35}O_5P$, 494.22; found, 494.2209.

2h: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32-7.28 (m, 3H), 7.21-7.18 (m, 4H), 7.17-7.13 (m, 3H), 7.05 (d, *J* = 8.4 Hz, 1H), 6.53 (d, *J* = 6.4 Hz, 1H), 6.48-6.43 (m, 2H), 3.82 (s, 3H), 3.76 (s, 3H), 3.09 (d, *J* = 22.4 Hz, 2H), 2.44-2.35 (m, 2H), 1.51-1.43 (m, 2H), 1.29-1.17 (m, 6H), 0.87-0.80 (m, 3H).

IR (neat) cm^{-1}: 2955, 2922, 2852, 1732, 1608, 1591, 1491, 1462, 1378, 1262, 1210, 1190, 1160, 1117, 933, 763, 501.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.68.

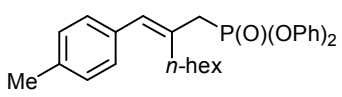
2′h: ^{1}H NMR (300 MHz, $CDCl_3$) δ: 7.42 (dd, *J* = 8.4, 2.1 Hz, 1H), 7.32-7.26 (m, 3H), 7.16-7.14 (m, 4H), 7.05 (m, 1H), 6.80 (d, *J* = 7.8 Hz, 2H), 6.50 (d, *J* = 8.4 Hz, 1H), 6.40 (s, 1H), 5.64 (d, *J* = 2.1 Hz, 1H), 5.13 (s, 1H), 4.75 (d, *J* = 25.5 Hz, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 2.11-2.02 (m, 2H), 1.42-1.35 (m, 2H), 1.26-1.21 (m, 6H), 0.90-0.82 (m, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ: 160.35, 158.15, 158.03, 151.03, 150.89, 150.82, 144.55, 144.50, 131.39, 131.32, 129.66, 129.42, 124.91, 124.66, 120.79, 120.73, 120.49, 120.43, 115.39, 115.33, 114.89, 114.79, 104.58, 98.61, 55.71, 55.51, 40.54 (d, *J* = 140 Hz), 37.05, 36.92, 31.83, 28.91, 27.49, 22.70, 14.23.

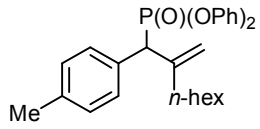
IR (neat) cm^{-1}: 2919, 2851, 1587, 1489, 1455, 1265, 1209, 1189, 1159, 1107, 1026, 926, 761, 688.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 19.39.

Table 1, entry 12:

2i 

(major, more polar)

2′i 

(minor, less polar)

The standard procedure was followed.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{28}H_{33}O_3P$, 448.22; found, 448.2171.

2i: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.34-7.27 (m, 3H), 7.21-7.12 (m, 5H), 7.12-7.09 (m, 4H), 7.07 (d, *J* = 8.4 Hz, 2H), 6.51 (d, *J* = 6.0 Hz, 1H), 3.06 (d, *J* = 22.0 Hz, 2H), 2.45 (t, *J* = 8.4, 2H), 2.35 (s, 3H), 2.14-1.98 (m, 2H), 1.52-1.47 (m, 2H), 1.30-1.19 (m, 4H), 0.89-0.82 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.74, 150.64, 136.45, 132.06, 131.94, 131.25, 131.11, 130.00, 129.88, 129.74, 129.02, 128.94, 128.62, 125.18, 120.76, 120.70, 120.66, 120.60, 34.64 (d, *J* = 137 Hz), 31.73, 31.53, 29.31, 22.73, 22.58, 21.31, 14.19.

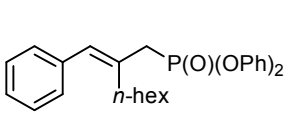
IR (neat) cm^{-1}: 2920, 2851, 1591, 1489, 1272, 1214, 1189, 1162, 1025, 928, 761, 688.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.28.

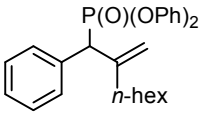
2′i: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38 (d, *J* = 8.8 Hz, 2H), 7.32-7.10 (m, 8H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.79 (d, *J* = 8.4 Hz, 2H), 5.70 (d, *J* = 2.8 Hz, 1H), 5.18 (d, *J* = 2.8 Hz, 1H), 4.06 (d, *J* = 25.2 Hz, 1H), 2.35 (s, 3H), 2.06-1.97 (m, 2H), 1.41-1.37 (m, 2H), 1.36-1.20 (m, 4H), 0.89-0.83 (m, 5H).

^{31}P NMR (121 MHz, $CDCl_3$) δ: 18.38.

Table 1, entry 13:



2j



2′j

(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{27}H_{27}O_3P$, 406.1692; found, 406.1700.

2j: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.34-7.29 (m, 6H), 7.22-7.11 (m, 9H), 6.56 (d, *J* = 6.4 Hz, 1H), 3.08 (d, *J* = 22.4 Hz, 2H), 2.50-2.42 (m, 2H), 2.14-1.98 (m, 2H), 1.54-1.45 (m, 2H), 1.44-1.24 (m, 4H), 0.87 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.73, 150.63, 132.05, 131.93, 131.25, 131.12, 130.01, 129.88, 129.82, 129.74, 129.02, 128.61, 125.19, 120.77, 120.66, 34.63 (d, *J* = 137 Hz), 31.73, 31.52, 29.31, 22.73, 22.58, 21.31, 14.19.

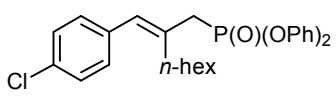
IR (neat) cm^{-1}: 2956, 2925, 2856, 1591, 1490, 1271, 1214, 1189, 1162, 1071, 1025, 1007, 928, 760, 689.

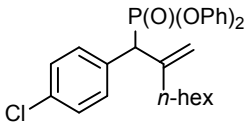
^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.16.

2′j: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52 (d, *J* = 8.4 Hz, 2H), 7.32-7.10 (m, 10H), 7.05 (t, *J* = 7.2 Hz, 1H), 6.76 (d, *J* = 8.4 Hz, 2H), 5.72 (d, *J* = 3.0 Hz, 1H), 5.21 (d, *J* = 3.0 Hz, 1H), 4.11 (d, *J* = 24.8 Hz, 1H), 2.06-1.97 (m, 2H), 1.44-1.35 (m, 2H), 1.36-1.20 (m, 4H), 0.90-0.83 (m, 5H).

^{31}P NMR (121 MHz, $CDCl_3$) δ: 18.19.

Table 1, entry 14:

2k 

2′k 

(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-ES (m/z): $[M]^+$ calcd for $C_{27}H_{30}ClO_3P$, 468.1616; found, 468.1639.

2k: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.35-7.21 (m, 4H), 7.22-7.09 (m, 10H), 6.57 (d, *J* = 6.4 Hz, 1H), 3.09 (d, *J* = 22.4 Hz, 2H), 2.50-2.43 (m, 2H), 2.11-1.99 (m, 2H), 1.55-1.45 (m, 2H), 1.44-1.22 (m, 4H), 0.87 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.55, 150.46, 137.77, 131.28, 131.16, 130.86, 130.72, 130.08, 129.89, 124.98, 120.69, 120.60, 113.73, 34.42 (d, *J* = 137 Hz), 31.70, 31.28, 29.14, 27.87, 22.55, 14.01.

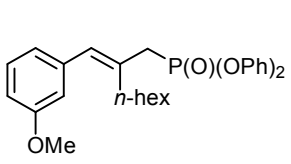
IR (neat) cm^{-1}: 2919, 2851, 1726, 1592, 1489, 1466, 1270, 1212, 1187, 1161, 1091, 1025, 929, 760, 688.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 19.86.

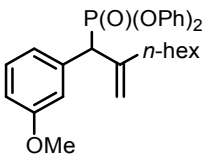
2′k: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.45 (d, *J* = 8.8 Hz, 2H), 7.33-7.29 (m, 4H), 7.22-7.06 (m, 6H), 6.82 (d, *J* = 8.4 Hz, 2H), 5.69 (d, *J* = 3.2 Hz, 1H), 5.21 (d, *J* = 3.2 Hz, 1H), 4.07 (d, *J* = 25.2 Hz, 1H), 2.16-1.94 (m, 2H), 1.42-1.33 (m, 2H), 1.28-1.16 (m, 4H), 0.90-0.81 (m, 5H).

^{31}P NMR (121 MHz, $CDCl_3$) δ: 17.92.

Table 1, entry 15:



2l (major, more polar)



2′l (minor, less polar)

The standard procedure was followed, except that CH_3CN was distilled over calcium hydride before use, 100 mol% *p*-TsOH•H_2O was used, the reaction mixture was stirred at 35 °C for 18 hours and the product was purified with 5% diethyl ether in toluene.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{28}H_{33}O_4PNa$, 487.2009; found, 487.2017.

2l: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.33-7.29 (m, 5H), 7.22-7.14 (m, 6H), 6.80-6.77 (m, 2H), 6.71 (s, 1H), 6.53 (d, *J* = 6.1 Hz, 1H), 3.79 (s, 3H), 3.07 (d, *J* = 22.4 Hz, 2H), 2.48-2.41 (m, 2H), 1.48-1.52 (m, 2H), 1.35-1.20 (m, 6H), 0.88-0.82 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.40, 150.74, 131.27, 131.15, 130.85, 130.72, 130.03, 129.88, 125.18, 124.59, 120.99, 120.70, 120.66, 113.73, 55.39, 34.66 (d, *J* = 137 Hz), 31.75, 31.49, 29.85, 29.34, 28.09, 22.74, 14.21.

IR (neat) cm^{-1}: 2920, 2851, 1725, 1591, 1509, 1489, 1249, 1214, 1189, 1162, 1026, 927, 759, 689.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.11.

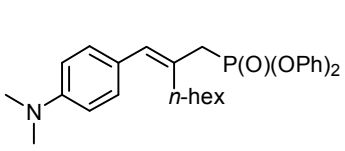
2′l: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.22-7.05 (m, 11H), 6.85-6.79 (m, 3H), 5.70 (d, *J* = 3.1 Hz, 1H), 5.20 (d, *J* = 3.1 Hz, 1H), 4.08 (d, *J* = 25.0 Hz, 1H), 3.77 (s, 3H), 2.18-2.00 (m, 2H), 1.42-1.38 (m, 2H), 1.31-1.20 (m, 6H), 0.88-0.81 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.78, 150.76, 143.65, 143.60, 135.96, 135.90, 129.74, 129.64, 129.55, 125.13, 124.93, 122.58, 122.50, 120.80, 120.76, 120.62, 120.57, 115.57, 115.50, 115.36, 113.48, 55.38, 50.84 (d, *J* = 138 Hz), 37.04, 36.94, 31.81, 28.92, 27.57, 22.71, 14.20.

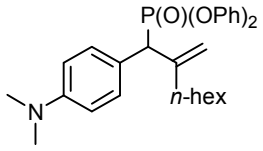
IR (neat) cm^{-1}: 2920, 2851, 1726, 1592, 1489, 1456, 1270, 1213, 1188, 1161, 1046, 928, 760, 688.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 18.00.

Table 1, entry 16:



2m



2′m

(more polar) (less polar)

The standard procedure was followed.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{29}H_{36}NO_3P$, 477.24; found, 477.2438.

2m: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.30-7.26 (m, 4H), 7.19-7.13 (m, 6H), 7.11 (d, *J* = 8.8 Hz, 2H), 6.70 (d, *J* = 8.8 Hz, 2H), 6.43 (d, *J* = 6.4 Hz, 1H), 3.05 (d, *J* = 22.4 Hz, 2H), 2.96 (s, 6H), 2.51-2.46 (m, 2H), 1.58-1.50 (m, 2H), 1.35-1.25 (m, 6H), 0.89-0.82 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 150.76, 150.66, 131.14, 129.85, 129.71, 129.68, 129.49, 125.13, 120.76, 120.72, 112.26, 40.67, 34.90 (d, *J* = 137 Hz), 31.82, 31.68, 29.45, 28.18, 22.77, 14.23.

IR (neat) cm^{-1}: 2925, 2854, 1732, 1610, 1593, 1520, 1490, 1353, 1272, 1215, 1190, 1163, 1071, 1025, 928, 762, 689.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.41.

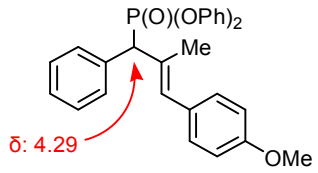
2′m: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32 (d, *J* = 8.8 Hz, 2H), 7.30-7.28 (m, 1H), 7.19-7.11 (m, 6H), 7.05 (t, *J* = 7.4 Hz, 1H), 6.80 (d, *J* = 8.4 Hz, 2H), 6.69 (d, *J* = 8.8 Hz, 2H), 5.66 (d, *J* = 3.2 Hz, 1H), 5.15 (d, *J* = 3.2 Hz, 1H), 4.00 (d, *J* = 24.8 Hz, 1H), 2.94 (s, 6H), 2.16-1.99 (m, 2H), 1.40-1.36 (m, 2H), 1.25-1.20 (m, 6H), 0.89-0.83 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 151.02, 150.92, 150.87, 150.77, 150.16, 144.28, 144.24, 130.73, 130.65, 129.84, 129.68, 129.49, 124.96, 124.78, 120.86, 120.82, 120.76, 120.72, 114.64, 114.56, 112.82, 49.84 (d, *J* = 139 Hz), 40.77, 36.93, 36.83, 31.84, 28.96, 27.58, 22.72, 14.21.

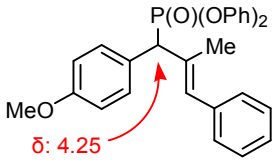
IR (neat) cm^{-1}: 2925, 2854, 1611, 1593, 1520, 1490, 1353, 1270, 1214, 1190, 1162, 1070, 1025, 928, 764, 689.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 19.00.

Table 1, entry 17:



2n
(minor)



2′n
(major)

The standard procedure was followed, except that the product was purified with 20% ethyl acetate in hexane. Isolated as a mixture of **2n** and **2′n**.

HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{29}H_{27}O_4PNa$, 493.1539; found, 493.1523.

2n: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.60 (d, J = 6.4 Hz, 2H), 7.33-7.07 (m, 12H), 6.90-6.74 (m, 6H), 4.29 (d, J = 25.4 Hz, 1H), 3.80 (s, 3H), 1.94 (s, 3H).

2′n: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.52 (d, J = 6.9 Hz, 2H), 7.33-7.07 (m, 11H), 6.97 (d, J = 3.1 Hz, 1H), 6.90-6.74 (m, 6H), 4.25 (d, J = 25.5 Hz, 1H), 3.80 (s, 3H), 1.94 (s, 3H).

2n+2′n: ^{13}C NMR (75 MHz, $CDCl_3$) δ: 159.29, 156.50, 150.87, 150.74, 150.57, 137.54, 133.24, 131.58, 131.04, 130.80, 130.66, 130.40, 129.83, 129.68, 129.51, 129.16, 128.84, 128.21, 126.80, 125.24, 125.17, 120.80, 120.75, 119.97, 115.23, 114.26, 55.43, 53.51 (d, J = 138 Hz), 17.99, 17.90.

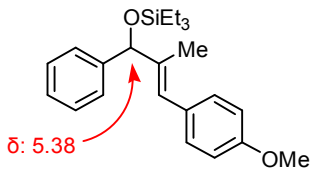
IR (neat) cm^{-1}: 2918, 2851, 1726, 1591, 1510, 1489, 1252, 1213, 1185, 1158, 1025, 929, 757, 689.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 18.00, 18.00.

<u>Comparison with correlated compounds (silyl ethers)</u>

Mahandru, G. M.; Liu, G.; Montgomery, J. *J. Am. Chem. Soc.* **2004**, *126*, 3698-3699.

Silyl ether with a structure similar to **2n** instead of **2′n** gives a slightly more deshielded carbinol proton.



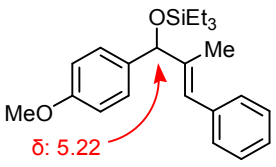
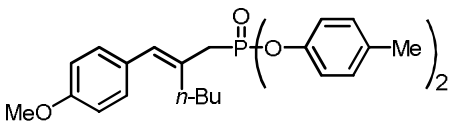
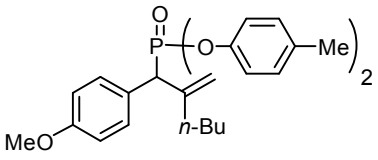


Table 1, entry 18:





(major) (minor)

The standard procedure was followed, except that the tris(4-methylphenyl) phosphite was used instead of triphenyl phosphite and the product was purified with 20% ethyl acetate in hexane. Isolated as a mixture of two isomers.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{28}H_{33}O_4P$, 464.2111; found, 464.2117.

major: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.13-6.95 (m, 10H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.45 (d, *J* = 6.1 Hz, 1H), 3.81 (s, 3H), 3.02 (d, *J* = 22.2 Hz, 2H), 2.48-2.42 (m, 2H), 2.29 (s, 6H), 1.52-1.44 (m, 2H), 1.44-1.21 (m, 2H), 0.89-0.82 (m, 3H).

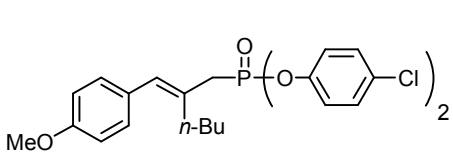
minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.41 (d, *J* = 8.5 Hz, 2H), 7.13-6.95 (m, 6H), 6.86 (d, *J* = 8.7 Hz, 2H), 6.67 (d, *J* = 8.5 Hz, 2H), 5.67 (d, *J* = 2.5 Hz, 1H), 5.17 (d, *J* = 2.5 Hz, 1H), 4.01 (d, *J* = 24.9 Hz, 1H), 3.80 (s, 3H), 2.30 (s, 6H), 2.14-1.97 (m, 2H), 1.44-1.21 (m, 4H), 0.89-0.82 (m, 3H).

major+minor: ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.34, 148.50, 148.41, 134.68, 131.37, 131.25, 131.13, 131.06, 130.68, 130.55, 130.35, 130.27, 130.14, 130.10, 129.97, 129.91, 129.88, 120.47, 120.43, 120.39, 120.35, 120.31, 120.27, 114.07, 113.68, 55.39, 55.35, 49.75 (d, *J* = 138 Hz), 36.62, 34.49 (d, *J* = 137 Hz), 31.22, 31.18, 30.29, 30.27, 29.74, 22.77, 22.33, 20.84, 20.79, 14.05.

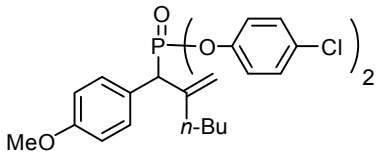
IR (neat) cm^{-1}: 2924, 2855, 1608, 1506, 1465, 1272, 1250, 1221, 1192, 1164, 1105, 1035, 940, 925, 821.

^{31}P NMR (121 MHz, $CDCl_3$) δ: 20.50, 18.59.

Table 1, entry 19:



(major)



(minor)

The standard procedure was followed, except that the tris(4-chlorophenyl) phosphite was used instead of triphenyl phosphite and the product was purified with 20% ethyl acetate in hexane. Isolated as a mixture of two isomers.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{26}H_{27}Cl_2O_4P$, 504.1019; found, 504.1000.

major: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.28-7.08 (m, 8H), 6.86 (d, J = 8.8 Hz, 2H), 6.68-6.65 (m, 2H), 6.48 (d, J = 6.3 Hz, 1H), 3.82 (s, 3H), 3.06 (d, J = 22.3 Hz, 2H), 2.45-2.41 (m, 2H), 1.53-1.45 (m, 2H), 1.45-1.22 (m, 2H), 0.89-0.82 (m, 3H).

minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.38 (d, J = 8.8 Hz, 2H), 7.28-7.08 (m, 8H), 6.86 (d, J = 8.8 Hz, 2H), 5.65 (d, J = 3.0 Hz, 1H), 5.19 (d, J = 2.9 Hz, 1H), 4.04 (d, J = 25.1 Hz, 1H), 3.80 (s, 3H), 2.15-1.97 (m, 2H), 1.45-1.22 (m, 4H), 0.89-0.83 (m, 3H).

major+minor: ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.52, 148.97, 148.87, 131.35, 131.22, 131.10, 131.02, 130.81, 130.37, 130.25, 129.95, 129.86, 129.83, 129.67, 129.60, 129.28, 122.07, 122.03, 121.97, 121.93, 121.84, 121.80, 116.77, 114.26, 113.80, 55.42, 55.37, 49.81 (d, J = 139 Hz), 36.67, 34.57 (d, J = 136 Hz), 31.24, 31.21, 30.24, 31.21, 29.72, 22.73, 22.28, 14.02.

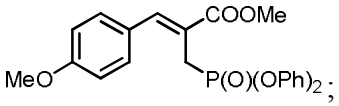
IR (neat) cm^{-1}: 2924, 2850, 1725, 1485, 1460, 1270, 1250, 1218, 1193, 1164, 1090, 924, 830.

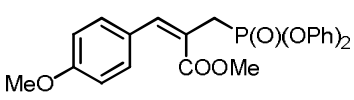
^{31}P NMR (121 MHz, $CDCl_3$) δ: 21.10, 19.19.

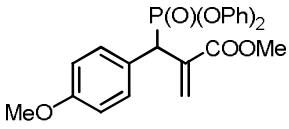
Scheme 5, Allyl diarylphosphonylation of TES protected Baylis-Hillman adduct of methylacrylate and anisaldehyde:

General procedure for the allyl diarylphosphonylation of allyl silyl ethers was followed, except the reaction was conducted at 35 ºC and was purified with 25% ethyl acetate in hexane.

81% yield (**2**+**2′**); **2**:**2′** = 3.2:1; *E:Z* = 18:82. Olefin stereochemistry were determined by NOESY.

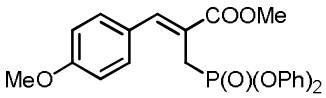


;


;


(Major, **2** *Z*-isomer) (Minor, **2** *E*-isomer) (Minor **2′** product)

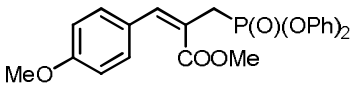
HRMS-EI (m/z): $[M]^+$ calcd for $C_{24}H_{23}O_6P$, 438.1232; found, 438.1232.



(Major, **2** *Z*-isomer): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.89 (d, *J* = 5.2 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 2H), 7.31-7.26 (m, 4H), 7.16-7.12 (m, 6H), 6.91 (d, *J* = 8.4 Hz, 2H), 3.82 (s, 3H), 3.77 (s, 3H), 3.62 (d, *J* = 22.4 Hz, 2H).

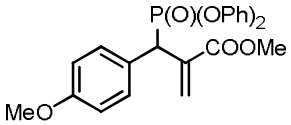
^{13}C NMR (100 MHz, $CDCl_3$) δ: 168.07, 160.62, 150.59, 150.50, 142.86, 142.74, 131.57, 129.84, 125.21, 120.58, 120.53, 114.33, 55.47, 52.52, 27.00 (d, *J* = 142 Hz).

^{31}P NMR (121 MHz, $CDCl_3$) δ: 19.14.



(Minor, **2** *E*-isomer): ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62-7.54 (m, 2H), 7.42-7.34 (m, 2H), 7.31-7.11 (m, 6H), 7.06 (d, *J* = 7.6 Hz, 1H), 7.01 (d, *J* = 8.0 Hz, 1H), 6.95 (d, *J* = 5.6 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 2H), 3.82 (s, 3H), 3.65 (s, 3H), 3.37 (d, *J* = 21.6 Hz, 2H).

^{31}P NMR (121 MHz, $CDCl_3$) δ: 18.53.



(minor **2′** type product) ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.43 (dd, *J* = 8.8, 2.0 Hz, 2H), 7.36-7.27 (m, 2H), 7.20-7.03 (m, 6H), 6.87 (dd, *J* = 8.8, 2.0 Hz, 2H), 6.75-6.71 (m, 2H), 6.65 (d, *J* = 2.8 Hz, 1H), 6.60 (d, *J* = 2.8 Hz, 1H), 4.94 (d, *J* = 24.8 Hz, 1H), 3.79 (s, 3H), 3.75 (s, 3H).

^{31}P NMR (121 MHz, $CDCl_3$) δ: 17.78.

11/ Arylation products (Table 2).

The products relative substitution patterns were determined by ^{1}H NMR coupling patterns and by ^{13}C NMR chemical shift comparison of related compounds if the aromatic region of ^{1}H NMR was not clear. The olefin stereochemistry was determined by NOESY.

For ^{13}C NMR of 2-methylphenol, 3-methylphenol and 4-methylphenol, see Ilczszyn, M.; Latałka, Z.; Ratajczak, H. *Org. Magn. Reson.* **1980**, *13*, 132-136.

^{13}C NMR (25.2 MHz, $CDCl_3$) δ:

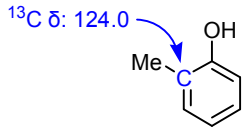
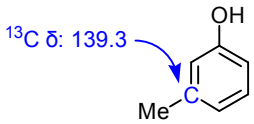
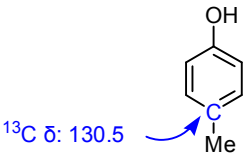
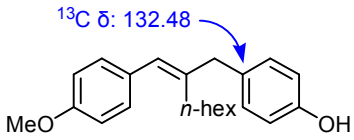
Ortho-	Meta-	Para-
		
153.5, 131.1, 127.7, 124.0, 121.4, 115.9.	154.9, 139.3, 130.3, 122.2, 116.1, 112.7.	152.6, 130.5, 130.2, 115.3.

Table 2, entry 1:



3a

The standard procedure was followed.

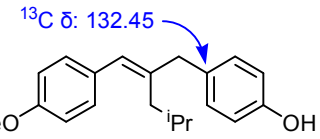
HRMS-EI (m/z): $[M]^+$ calcd for $C_{22}H_{28}O_2$, 324.2084; found, 324.2077.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15 (d, J = 8.6 Hz , 2H), 7.10 (d, J = 8.4 Hz, 2H), 6.85 (d, J = 8.6 Hz, 2H), 6.76 (d, J = 8.4 Hz, 2H), 6.21 (s, 1H), 5.05 (brs, 1H), 3.80 (s, 3H), 3.39 (s, 2H), 2.13 (t, J = 8.2 Hz, 2H), 1.47-1.41 (m, 2H), 1.28-1.20 (m, 6H), 0.86 (t, J = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.97, 154.06, 142.09, 132.48, 131.25, 130.39, 129.97, 126.36, 115.36, 113.75, 55.49, 43.30, 31.85, 30.45, 29.62, 28.35, 22.83, 14.31.

IR (neat) cm^{-1}: 3396, 2925, 1607, 1509, 1464, 1247, 1174, 1035, 822.

Table 2, entry 2:



3b

The standard procedure was followed.

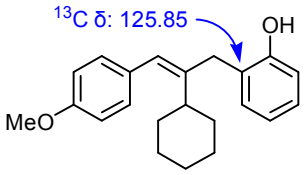
HRMS-EI (m/z): $[M]^+$ calcd for $C_{20}H_{24}O_2$, 296.1771; found, 296.1774.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15 (d, J = 8.6 Hz, 2H), 7.11 (d, J = 8.4 Hz, 2H), 6.84 (d, J = 8.6 Hz, 2H), 6.77 (d, J = 8.4 Hz, 2H), 6.29 (s, 1H), 4.92 (brs, 1H), 3.80 (s, 3H), 3.38 (s, 2H), 2.05 (d, J = 7.4 Hz, 2H), 1.88-1.98 (m, 1H), 0.82 (d, J = 6.6 Hz, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.86, 153.98, 140.98, 132.45, 131.24, 130.29, 130.13, 127.54, 115.27, 113.56, 55.36, 43.23, 38.76, 26.53, 22.65.

IR (neat) cm^{-1}: 3400, 2954, 1608, 1509, 1463, 1246, 1173, 1035, 822.

Table 2, entry 3:



3c *ortho*-product

The standard procedure was followed, except that the product was purified with chloroform.

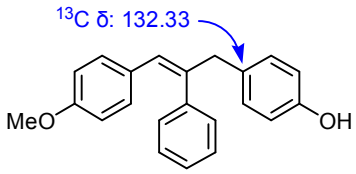
HRMS-EI (m/z): $[M]^+$ calcd for $C_{22}H_{26}O_2$, 322.1927; found, 322.1905.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.18-7.15 (m, 2H), 7.08 (d, J = 8.5 Hz, 2H), 6.91 (t, J = 7.4 Hz, 1H), 6.86-6.84 (m, 3H), 6.05 (s, 1H), 5.10 (brs, 1H), 3.80 (s, 3H), 3.49 (s, 2H), 2.86-2.79 (m, 1H), 1.80-1.48 (m, 5H), 1.45-1.10 (m, 5H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.17, 154.64, 144.64, 131.55, 130.33, 129.94, 128.10, 125.85, 125.72, 120.98, 116.23, 113.69, 55.38, 40.60, 34.79, 31.64, 26.51, 26.21.

IR (neat) cm^{-1}: 3436, 2926, 1602, 1512, 1458, 1384, 1256, 1176, 1032, 836, 754.

Table 2, entry 4:



3d

The standard procedure was followed, except that the product was purified with chloroform.

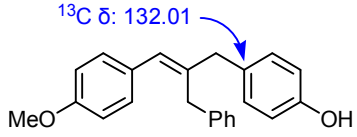
HRMS-EI (m/z): $[M]^+$ calcd for $C_{22}H_{20}O_2$, 316.1458; found, 316.1459.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.24-7.20 (m, 3H), 7.06-7.04 (m, 4H), 6.84 (d, *J* = 8.6 Hz, 2H), 6.72 (d, *J* = 8.6 Hz, 2H), 6.62 (d, *J* = 8.8 Hz, 2H), 6.35 (s, 1H), 4.70 (brs, 1H), 3.72 (s, 3H), 3.68 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.64, 154.36, 140.07, 139.62, 132.33, 130.89, 130.76, 130.05, 129.14, 128.94, 128.62, 126.49, 115.59, 115.49, 114.13, 55.71, 42.96.

IR (neat) cm^{-1}: 3397, 2918, 1724, 1606, 1510, 1465, 1368, 1286, 1251, 1216, 1176, 1124, 1074, 1040, 825, 759, 701.

Table 2, entry 5:



3e

The standard procedure was followed.

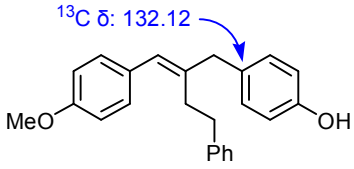
HRMS-EI (m/z): $[M]^+$ calcd for $C_{23}H_{22}O_2$, 330.1614; found, 330.1617.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.32-7.29 (m, 2H), 7.26-7.16 (m, 5H), 7.03 (d, *J* = 8.5 Hz, 2H), 6.83 (d, *J* = 8.8 Hz, 2H), 6.76 (d, *J* = 8.5 Hz, 2H), 6.49 (s, 1H), 4.81 (brs, 1H), 3.79 (s, 3H), 3.53 (s, 2H), 3.29 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.32, 154.05, 139.76, 139.30, 132.01, 130.57, 130.45, 129.74, 128.83, 128.63, 138.30, 126.17, 115.28, 113.81, 55.39, 42.65, 36.00.

IR (neat) cm^{-1}: 3401, 3024, 2922, 1607, 1509, 1451, 1248, 1177, 1031, 822, 733.

Table 2, entry 6:



3f

The standard procedure was followed.

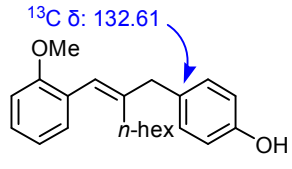
HRMS-ESI (m/z): $[M+Na]^+$ calcd for $C_{24}H_{24}O_2Na$, 367.1669; found, 367.1658.

^1H NMR (400 MHz, $CDCl_3$) δ: 7.25-7.23 (m, 2H), 7.18-7.09 (m, 7H), 6.84 (d, *J* = 8.4 Hz, 2H), 6.77 (d, *J* = 8.2 Hz, 2H), 6.28 (s, 1H), 4.88 (brs, 1H), 3.80 (s, 3H), 3.43 (s, 2H), 2.75-2.71 (m, 2H), 2.50-2.43 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ: 158.09, 154.11, 142.07, 140.70, 132.12, 130.78, 130.33, 129.81, 128.45, 128.42, 127.22, 126.00, 115.34, 113.70, 55.40, 43.52, 34.48, 32.33.

IR (neat) cm^{-1}: 3401, 3025, 2925, 2852, 1606, 1509, 1453, 1247, 1177, 1033, 939, 826, 699.

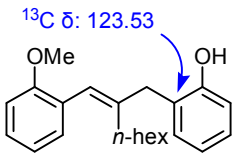
Table 2, entry 7:



3g

(major, more polar)

3g *ortho*-product



(minor, less polar)

The standard procedure was followed, except that 40 mol% of *p*-TsOH•H_2O was used.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{22}H_{28}O_2$, 324.2084; found, 324.2059.

3g: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.23-7.17 (m, 2H), 7.15 (d, *J* = 8.4 Hz, 2H), 6.90 (dd, *J* = 8.2, 7.4 Hz, 1H), 6.86 (d, *J* = 8.2 Hz, 1H), 6.77 (d, *J* = 8.4 Hz, 2H), 6.35 (s, 1H), 4.73 (brs, 1H), 3.82 (s, 3H), 3.44 (s, 2H), 2.06 (t, *J* = 8.1 Hz, 2H), 1.49-1.39 (m, 2H), 1.30-1.17 (m, 6H), 0.85 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.30, 153.92, 142.75, 132.61, 130.25, 130.19, 127.73, 127.50, 122.72, 120.14, 115.21, 110.53, 55.59, 42.86, 31.74, 30.30, 29.42, 28.20, 22.73, 14.20.

IR (neat) cm^{-1}: 3398, 2924, 1706, 1612, 1597, 1512, 1487, 1463, 1376, 1288, 1243, 1171, 1109, 1029, 823, 752.

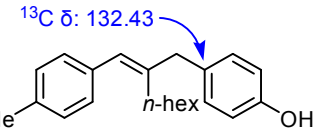
Purified with 10% ethyl acetate in hexane.

3g *ortho*-product: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.23-7.21 (m, 1H), 7.18-7.12 (m, 3H), 6.94-6.85 (m, 4H), 6.54 (s, 1H), 5.86 (brs, 1H), 3.83 (s, 3H), 3.60 (s, 2H), 2.02 (t, *J* = 8.2 Hz, 2H), 1.50-1.39 (m, 2H), 1.34-1.15 (m, 6H), 0.83 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 156.99, 142.41, 131.12, 129.65, 128.13, 128.07, 126.46, 123.53, 120.72, 120.11, 116.85, 110.16, 55.37, 40.16, 31.53, 29.93, 29.12, 27.86, 22.54, 14.05.

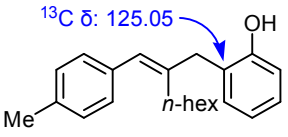
IR (neat) cm^{-1}: 3385, 2953, 2850, 1720, 1594, 1487, 1454, 1369, 1286, 1243, 750.

Table 2, entry 8:



3i

(major, more polar)



3i *ortho*-product

(minor, less polar)

The standard procedure was followed, except that 40 mol% of *p*-TsOH•H_2O was used.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{22}H_{28}O$, 308.2135; found, 308.2112.

3i: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.12-7.09 (m, 6H), 6.77 (d, *J* = 8.4 Hz, 2H), 6.24 (s, 1H), 4.74 (brs, 1H), 3.40 (s, 2H), 2.33 (s, 3H), 2.13 (t, *J* = 8.0 Hz, 2H), 1.50-1.40 (m, 2H), 1.31-1.20 (m, 6H), 0.86 (t, *J* = 6.8 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 153.96, 142.64, 135.76, 135.56, 132.43, 130.31, 128.91, 128.65, 126.76, 115.25, 43.21, 31.76, 30.41, 29.52, 28.28, 22.75, 21.27, 14.22.

IR (neat) cm^{-1}: 3374, 3020, 2854, 1703, 1612, 1510, 1444, 1376, 1222, 1170, 1099, 824.

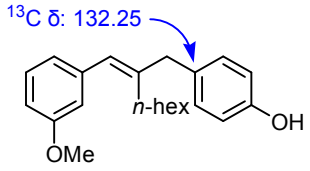
Purified with 10% ethyl acetate in hexane.

3i *ortho*-product: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15 (d, *J* = 7.6 Hz, 2H),7.13-7.09 (m, 4H), 6.93-6.87 (m, 1H), 6.84 (d, *J* = 7.6 Hz, 1H), 6.39 (s, 1H), 5.27 (brs, 1H), 3.54 (s, 2H), 2.33 (s, 3H), 2.20 (t, *J* = 8.0 Hz, 2H), 1.53-1.44 (m, 2H), 1.30-1.19 (m, 6H), 0.85 (t, *J* = 7.0 Hz, 3H).

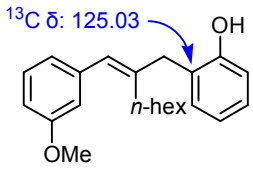
^{13}C NMR (100 MHz, $CDCl_3$) δ: 155.09, 141.60, 136.26, 131.23, 129.01, 128.64, 128.18, 127.19, 125.05, 120.92, 116.17, 39.62, 31.68, 30.62, 29.44, 28.22, 22.70, 21.28, 14.20.

IR (neat) cm^{-1}: 3398, 3021, 2926, 1706, 1604, 1488, 1456, 1377, 1243, 1182, 1096, 1020, 807, 752.

Table 2, entry 9:



3l



3l *ortho*-product

(major, more polar)

(minor, less polar)

The standard procedure was followed, except that 40 mol% of *p*-TsOH•H_2O was used.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{22}H_{28}O_2$, 324.2084; found, 324.2080.

3l: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.24-7.20 (m, 1H), 7.15 (d, *J* = 8.5 Hz, 2H), 6.82-6.74 (m, 5H), 6.25 (s, 1H), 4.78 (brs, 1H), 3.80 (s, 3H), 3.40 (s, 2H), 2.17 (t, *J* = 8.2 Hz, 2H), 1.55-1.40 (m, 2H), 1.30-1.23 (m, 6H), 0.86 (t, *J* = 7.1 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.49, 154.01, 143.61, 139.92, 132.25, 130.34, 129.15, 126.79, 121.35, 115.37, 115.29, 114.18, 114.03, 112.03, 111.89, 55.30, 43.14, 31.79, 30.52, 29.54, 28.34, 22.74, 14.21.

IR (neat) cm^{-1}: 3357, 2916, 2850, 1703, 1575, 1510, 1453, 1367, 1257, 1152, 1072, 1040, 820, 781, 693.

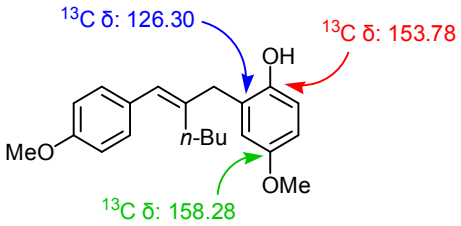
Purified with 10% ethyl acetate in hexane.

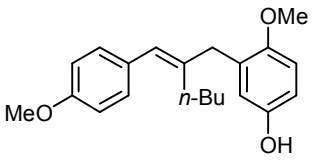
3l *ortho*-product: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.21-7.15 (m, 2H), 6.92-6.75 (m, 6H), 6.37 (s, 1H), 5.22 (brs, 1H), 3.80 (s, 3H), 3.54 (s, 2H), 2.22 (t, *J* = 8.1 Hz, 2H), 1.54-1.45 (m, 2H), 1.38-1.15 (m, 6H), 0.89-0.79 (m, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 159.54, 154.94, 142.54, 139.18, 131.25, 129.26, 128.20, 127.09, 125.03, 121.29, 120.96, 116.13, 114.14, 112.28, 55.32, 39.31, 31.72, 30.79, 29.48, 28.31, 22.71, 14.24, 14.20.

IR (neat) cm^{-1}: 3422, 2926, 2854, 1706, 1597, 1487, 1455, 1261, 1155, 1092, 1042, 873, 753, 696.

Table 2, entry 10:





(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{21}H_{26}O_3$, 326.1876; found, 326.1864.

major: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15 (d, J = 8.4 Hz, 2H), 6.86 (d, J = 8.4 Hz, 2H), 6.78 (d, J = 8.8 Hz, 1H), 6.72-6.69 (m, 2H), 6.34 (s, 1H), 4.92 (brs, 1H), 3.81 (s, 3H), 3.77 (s, 3H), 3.49 (s, 2H), 2.22 (t, J = 8.0 Hz, 2H), 1.53-1.46 (m, 2H), 1.33-1.25 (m, 2H), 0.87 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.28, 153.78, 148.94, 140.59, 130.22, 129.91, 126.78, 126.30, 116.80, 116.76, 114.01, 113.72, 112.81, 55.86, 55.39, 39.80, 30.52, 30.39, 22.94, 14.07.

IR (neat) cm^{-1}: 3401, 2956, 2926, 2871, 1606, 1509, 1464, 1383, 1248, 1203, 1176, 1105, 1039, 808.

Purified with 10% ethyl acetate in hexane.

minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.20 (d, J = 8.4 Hz, 2H), 6.86 (d, J = 8.4 Hz, 2H), 6.74-6.62 (m, 3H), 6.53 (s, 1H), 4.65 (brs, 1H), 3.80 (s, 3H), 3.74 (s, 3H), 3.58 (s, 2H), 2.09 (t, J = 8.0 Hz, 2H), 1.53-1.46 (m, 2H), 1.30-1.23 (m, 2H), 0.90 (t, J = 7.2 Hz, 3H).

Comparison with correlated compounds (^{13}C NMR, $CDCl_3$)

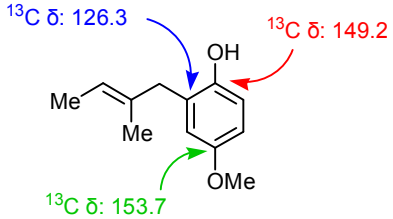
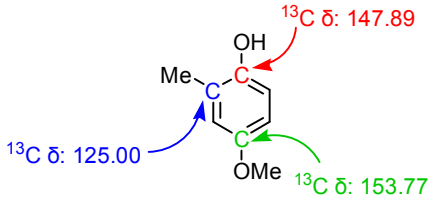
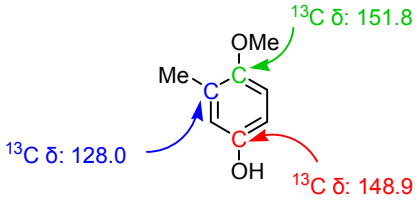
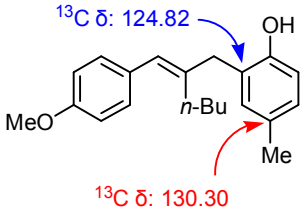
		
(75 MHz) δ: 153.7, 149.2, 134.9, 126.3, 121.6, 116.7, 112.7, 55.9, 42.2, 15.8, 13.7.	(50 MHz) δ: 153.8, 147. 9, 125.0, 116.7, 115.7, 112.0, 55.9, 16.2.	(75 MHz) δ: 151.8, 148.9, 128.0, 118.0, 112.6, 111.6, 56.1, 16.1.
Trend, R. M.; Ramtohul, Y. K.; Stoltz, B. M. *J. Am. Chem. Soc.* **2005**, *127*, 17778.	Mori T.; Grimme S.; Inoue Y. *J. Org. Chem.* **2007**, *72*, 6998.	Vyvyan, J. R.; Loitz, C.; Looper, R. E.; Mattingly, C. S.; Peterson, E. A.; Staben, S. T. *J. Org. Chem.* **2004**, *69*, 2461.

Table 2, entry 11:



The standard procedure was followed

HRMS-EI (m/z): $[M]^+$ calcd for $C_{21}H_{26}O_2$, 310.1927; found, 310.1934.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15 (d, J = 8.4 Hz, 2H), 6.95-6.93 (m, 2H), 6.86 (d, J = 8.4 Hz, 2H), 6.74 (d, J = 8.4 Hz, 1H), 6.34 (s, 1H), 5.11 (brs, 1H), 3.81 (s, 3H), 3.49 (s, 2H), 2.28 (s, 3H), 2.22 (t, J = 8.0 Hz, 2H), 1.54-1.47 (m, 2H), 1.36-1.26 (m, 2H), 0.87 (t, J = 7.3 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.25, 152.79, 140.92, 131.76, 130.30, 130.03, 129.91, 128.56, 126.57, 124.82, 115.97, 113.71, 55.38, 39.62, 30.49, 30.37, 22.93, 20.67, 14.06.

IR (neat) cm^{-1}: 3418, 2956, 2930, 1673, 1606, 1464, 1300, 1250, 1177, 1107, 1035, 937, 814.

Comparison with correlated compounds (^{13}C NMR, 63 MHz, $CDCl_3$)

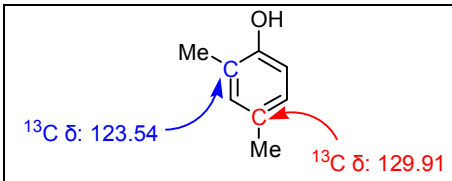
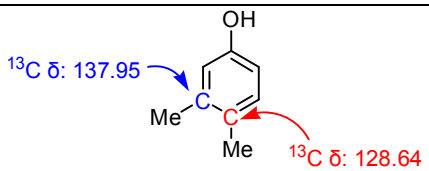
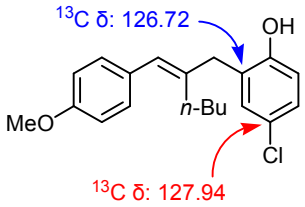
	
δ: 151.36, 131.61, 129.91, 127.34, 123.54, 114.79, 20.36, 15.64.	δ: 153.42, 137.95, 130.47, 128.64, 116.58, 112.34, 19.82, 18.72.
Wigal, C. T.; McKinley, J. D.; Coyle, J.; Porter, D. J.; Lehman, D. E. *J. Org. Chem.***1995**, *60*, 8421-8423.	Livant, P.; Xu, W. *J. Org. Chem.* **1998**, *63*, 636-641.

Table 2, entry 12:



The standard procedure was followed.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{20}H_{23}ClO_2$, 330.1381; found, 330.1389.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15 (d, J = 8.4 Hz, 2H), 7.13-7.10 (m, 2H), 6.87 (d, J = 8.4 Hz, 2H), 6.77 (d, J = 8.0 Hz, 1H), 6.35 (s, 1H), 5.31 (brs, 1H), 3.81 (s, 3H), 3.48 (s, 2H), 2.21 (t, J = 8.0 Hz, 2H), 1.52-1.44 (m, 2H), 1.35-1.25 (m, 2H), 0.87 (t, J = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 153.59, 148.84, 141.50, 130.76, 130.18, 128.73, 128.36, 127.94, 127.75, 126.72, 121.57, 121.52, 117.40, 55.38, 39.11, 29.38, 28.22, 22.69, 14.19.

IR (neat) cm^{-1}: 3345, 2919, 2850, 1484, 1267, 1189, 1092, 971, 832, 698.

Comparison with correlated compounds ^{13}C NMR ($CDCl_3$):

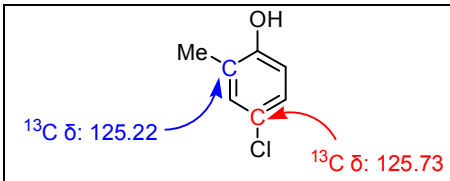
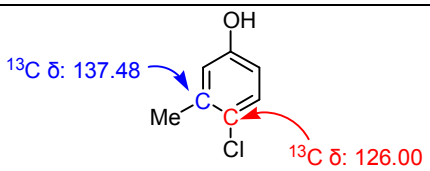
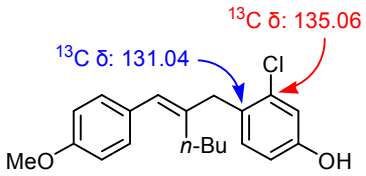
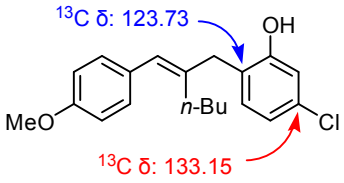
	
125 MHz δ: 152.43, 130.65, 126.76, 125.73, 125.22, 116.04, 15.69.	100 MHz δ: 153.91, 137.48, 129.94, 126.00, 118.05, 114.35, 20.17.
Wang, J.-R.; Manabe, K. *Org. Lett.* **2009**, *11*, 741-744.	Menini, L.; Gusevskaya, E. V. *Appl. Catal., A* **2006**, *309*, 122-128.

Table 2, entry13:





(major, more polar) (minor, less polar)

The standard procedure was followed.

HRMS-EI (m/z): $[M]^+$ calcd for $C_{20}H_{23}ClO_2$, 330.1381; found, 330.1367.

major: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.14-7.12 (m, 3H), 6.89 (d, *J* = 2.8 Hz, 1H), 6.85 (d, *J* = 8.4 Hz, 2H), 6.70 (dd, *J* = 8.4, 2.8 Hz, 1H), 6.06 (s, 1H), 4.99 (brs, 1H), 3.80 (s, 3H), 3.49 (s, 2H), 2.18 (t, *J* = 8.0 Hz, 2H), 1.54-1.46 (m, 2H), 1.37-1.25 (m, 2H), 0.88 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 157.96, 154.62, 140.40, 135.06, 131.88, 131.04, 129.96, 129.82, 126.40, 116.51, 114.18, 113.62, 55.39, 40.03, 30.79, 30.70, 23.02, 14.11.

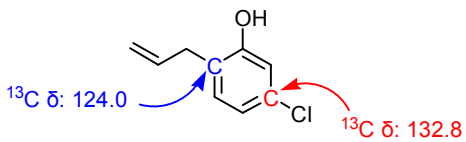
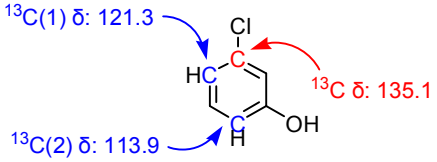
IR (neat) cm^{-1}: 3362, 2957, 2926, 2855, 1703, 1601, 1511, 1466, 1384, 1252, 1175, 1033, 836.

Purified with 10% ethyl acetate in hexane.

minor: ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.15 (d, *J* = 8.4 Hz, 2H), 7.06 (d, *J* = 8.0 Hz, 1H), 6.91-6.77 (m, 4H), 6.36 (s, 1H), 5.40 (brs, 1H), 3.81 (s, 3H), 3.49 (s, 2H), 2.17 (t, *J* = 8.0 Hz, 2H), 1.53-1.44 (m, 2H), 1.37-1.24 (m, 2H), 0.88 (t, *J* = 7.2 Hz, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.39, 155.75, 140.27, 133.15, 131.96, 130.58, 129.98, 129.90, 127.12, 123.73, 121.12, 121.02, 116.55, 116.04, 113.89. 113.78, 55.40, 39.19, 33.35, 31.74, 30.42, 30.28, 22.91, 22.81, 14.28, 14.04. IR (neat) cm^{-1}: 3400, 2956, 2928, 2858, 1702, 1607, 1577, 1509, 1497, 1465, 1441, 1294, 1249, 1177, 1039, 906, 856, 818.

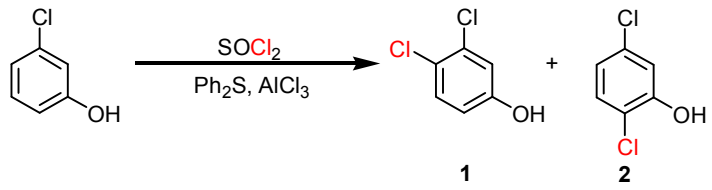
Comparison with correlated compounds ^{13}C NMR ($CDCl_3$)

	
(100 MHz) δ: 154.6, 135.8, 132.8, 131.2, 124.0, 121.0, 116.8, 116.1, 34.5.	(25.2 MHz) δ: 156.3, 135.1, 130.6, 121.3, 116.1, 113.9.
Lin, Y.-L.; Cheng, J.-Y.; Chu, Y.-H. *Tetrahedron* **2007**, *63*, 10949-10957.	Ilczszyn, M.; Latałka, Z.; Ratajczak, H. *Org. Magn. Reson.* **1980**, *13*, 132-136.

The carbon at *ortho*-position to OH, C(2), was more shielded than that to Cl, C(1). Other examples were shown below:

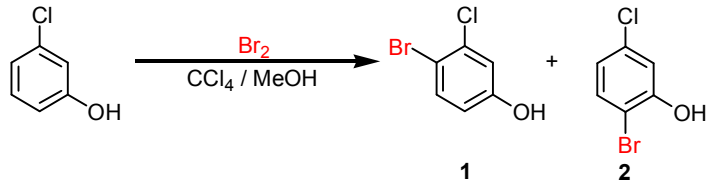
For the regioselective electrophilic aromatic substitution of 3-chlorophenol, see

1) *para*-chlorination with Freidel-Crafts catalyst: Watson, W. D. *J. Org. Chem.* **1985**, *50*, 2145-2148.



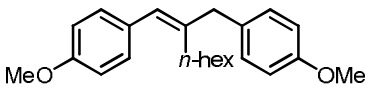
Yield of **1** = 82%
1 : **2** = 86 : 14

2) Bromination: Suresh, P.; Annalakshmi, S.; Pitchumani, K. *Tetrahedron* **2007**, *63*, 4959-4967.



Yield of **1** = 56%
1 : **2** = 64 : 36

Table 2, entry 14:



The standard procedure was followed, except that anisole was used instead of triphenylphosphite.

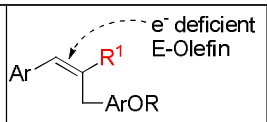
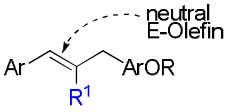
HRMS-EI (m/z): $[M]^+$ calcd for $C_{23}H_{30}O_2$, 338.2240; found, 338.2224.

^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.16 (d, J = 8.4 Hz, 2H), 7.15 (d, J = 8.4 Hz, 2H), 6.85 (d, J = 8.4 Hz, 2H), 6.84 (d, J = 8.4 Hz, 2H), 6.22 (s, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 3.40 (s, 2H), 2.15 (t, J = 8.3 Hz, 2H), 1.50-1.42 (m, 2H), 1.29-1.19 (m, 6H), 0.86 (t, J = 7.1 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ: 157.88, 141.80, 132.11, 130.90, 129.90, 129.66, 126.06, 113.61, 113.41, 55.18, 43.00, 31.57, 30.17, 29.34, 28.07, 22.55, 14.03.

IR (neat) cm^{-1}: 2953, 2926, 2855, 1712, 1608, 1509, 1464, 1441, 1300, 1247, 1175, 1105, 1037, 824.

Other related informations in the preparation of allyl phenolic compounds, with the concomitant formation of a new C-aryl bond and a new trisubstituted olefin:

Allyl Phenolic Targets	 R^1= Ester	 R^1= Alkyl, Aryl
The Selected Phenolic Substrates Acceptors	Acetylated Baylis-Hillman Adducts	e.g. Silyl Allyl Ethers
Phenolic Substrates	$ArylKBF_3$ Arylboronic Acids	Both protected & unprotected phenols

It should be noted that treating unprotected phenols with Baylis-Hillman adducts may result mainly O-allylations, for examples, please see: (a) D. Y. Park, S. Gowrisankar, J. N. Kim, *Bull. Korean Chem. Soc.* 2005, *26*, 1440; (b) E. Ramesh, R. Raghunathan, *Tetrahedron Lett.* 2008, 49, 1125.

For detailed potassium organotrifluoroborates or aryl boronic acid coupling with Baylis-Hillman adducts procedures, please see: (a) Kabalka, G. W.; Venkataiah, B.; Dong, G. *Org. Lett.* 2003, *5*, 3803; (b) Kantam, M. L.; Kumar, K. B. S.; Sreedhar, B. *J. Org. Chem.* 2008, *73*, 320; (c) Gendrineau, T.; Demoulin, N.; Navarre, L.; Genet, J.-P.; Darses, S. *Chem. Eur. J.* 2009, *15*, 4710.

12/ **Summary on the inhibitory effect of the newly synthesized allyl diarylphosphonates on the metabolism of oseltamivir (O) to oseltamivir carboxylate (OC) in rat plasma**

Experimental procedure

The inhibition experiment was modified from our previous study [Chang et al., Biomedical Chromatography, 2009, 23, 852.] and conducted as follow. To 200 μL rat plasma 2 μL of inhibitor working solution in DMSO was added (final 0.18, 0.4, 1, 4 or 10 μM in plasma) and well mixed. 10 μL of 100 μg/mL oseltamivir in H_2O was added, and the mixture was incubated at room temperature for 1 hour. After incubation, 50 μL of the sample mixture was acidified with 1 mL of 0.1% hydrochloric acid prior to solid phase extraction (Waters Oasis MCX cartridge, 30 mg). The detailed solid phase extraction procedure was reference to the previous study and was not produced here. Analytes were eluted with 1 mL of 1% ammonia in methanol and the solvent was evaporated to dryness by a vacuum concentrator. The residue was reconstituted in 150 μL mobile phase (0.1% formic acid : methanol, 1:1 v/v) and 20 μL supernatant were injected for LC-MS/MS analysis (data was acquired by multiple reaction monitoring of O and OC [Chang et al., Biomedical Chromatography, 2009]). Control samples were prepared by adding 2 μL DMSO (instead of inhibitor) to rat plasma.

Data treatment

The percentage inhibition was calculated as $[(ratio_{control} - ratio_{inhibitor})/ratio_{control}] \times 100\%$, where ratio = Area ratio of OC to O. The % inhibition was then plotted against the logarithm of the concentration of inhibitor in μM. The logarithm of IC_{50}, the half maximal inhibitory concentration, was evaluated from the Sigmoidal dose-response equation using Prism program (version 3.03, GraphPad Software, Inc.).

Summary

Our newly synthesized allyl diarylphosphonates were first identified as potent carboxylesterase inhibitors with broad range of inhibitory efficiency (reflected by log IC_{50} from -0.29 to 0.70, or IC_{50} from 0.52 to 4.99 μM) and provided room for fine tuning when necessary. Those exhibit much stronger inhibitory effect than those of $P(OPh)_3$ and $H\text{-}P(O)(OPh)_2$ were quickly identified by using the new phosphonylation methodology developed here. The results obtained here provided us new directions for further optimization, and may lead to the development of a new generation of carboxylesterase inhibitors.

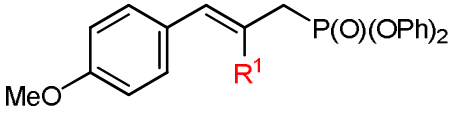
Results and discussion

a. Reference compounds

Reference compound	LOG IC_{50}		IC_{50} (μM)
	Calculated value	Standard error	Calculated value
$P(OPh)_3$	0.55	0.08	3.53
$HP(O)(OPh)_2$	0.97	0.19	9.28

Note: The inhibitory effect of inhibitor on the metabolism of oseltamivir (O) to oseltamivir carboxylate (OC) was presented as Log IC_{50} or IC_{50}. The lower the Log IC_{50} value indicates the lower concentration of inhibitor can be used to exhibit the inhibitory effect and thus the stronger inhibitor.

b. Allyl diarylphosphonates **2** and **2′**

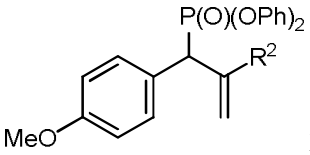


2

R^1	LOG IC_{50}		IC_{50} (μM)
	Calculated value	Standard error	Calculated value
H	-0.21	0.09	0.62
n-Hex	-0.26	0.05	0.55
CH_2-*i*-Pr	0.15	0.03	1.40
Ph	0.22	0.07	1.67
CH_2Ph	-0.13	0.04	0.73
CH_2Bn	0.23	0.09	1.72

Remark: Bn = CH_2Ph

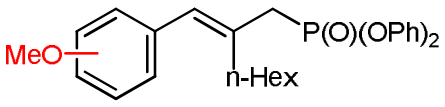
All the above six allyl diarylphosphonates **2** studied exhibits stronger inhibitory effect (log IC_{50} ranges from -0.26 to 0.23) than those of reference compounds (log IC_{50} between 0.55 to 0.97, section a). Among the allyl diarylphosphonates, that with R^1 = *n*-Hex was found to be most potent inhibitor.



2′

Several allyl diarylphosphonates with general structure of **2′** were also examined, however, their inhibitory effects are much lower than those of **2** in general: e.g. for R^2 = Cy and Ph, log IC_{50} are 0.70 and 0.57, respectively (with standard error: 0.02 and 0.07).

c. Effect of OMe in *o*-, *m*-, *p*-position of allyl diarylphosphonates **2** and amine substituent

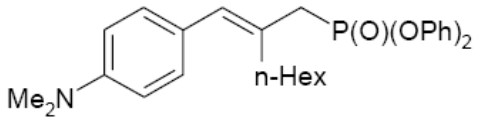


2

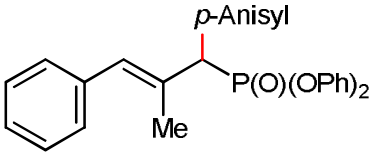
Substituent	LOG IC_{50}		IC_{50} (μM)
	Calculated value	Standard error	Calculated value
p-OMe	-0.26	0.05	0.55
m-OMe	0.29	0.07	1.93
o-OMe	-0.29	0.10	0.52
p-,*o*-*di* OMe	-0.19	0.09	0.64

Among the monosubstituted OMe compounds, *o*-OMe and *p*-OMe (log IC_{50} of -0.29 and -0.26, respectively) exhibit stronger inhibition than *m*-OMe (log IC_{50} of 0.29), but not much enhancement was observed for *o*-, *p*-disubstituted OMe.

In addition, allyl diarylphosphonate with amine substituent (log IC_{50} of 0.46, standard error of 0.14, structure is shown below) lowers the inhibitory effect than those methoxy substituents.

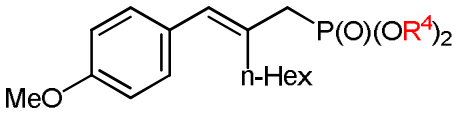


d. α-branch



Inhibitor	LOG IC_{50}		IC_{50} (μM)
	Calculated value	Standard error	Calculated value
2′n	-0.13	0.11	0.74

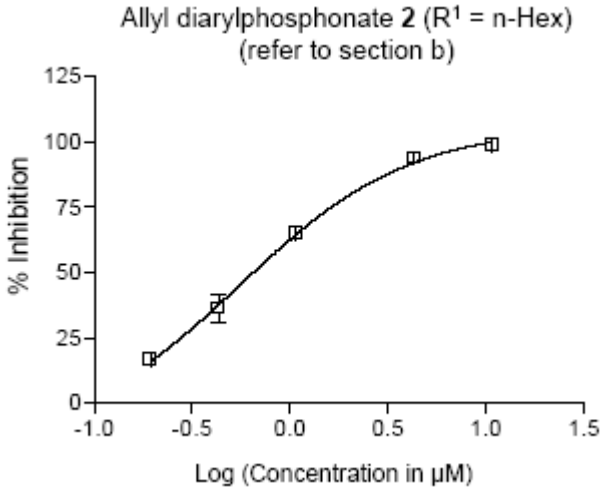
e. Effect of R^4



R^4	LOG IC_{50}		IC_{50} (μM)
	Calculated value	Standard error	Calculated value
p-C_6H_4Me	0.30	0.12	1.98
p-C_6H_4Cl	0.47	0.09	2.93

In both compounds, their inhibitory effect is weaker than those allyl diarylphosphonates **2** in section b (log IC_{50} between -0.26 to 0.23).

f. Representative Sigmoidal dose-response curve of allyl diarylphosphonate inhibitor.



Reference:

Chang Q, Chow MSS and Zuo Z. Studies on the influence of esterase inhibitor to the pharmacokinetic profiles of oseltamivir and oseltamivir carboxylate in rats using an improved LC/MS/MS method. *Biomedical Chromatography* **2009**; *23*: 852-857.

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DE                      6.50 usec
TE                      294.3 K
D1                 1.00000000 sec
TD0                           1

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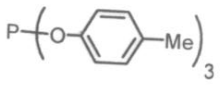

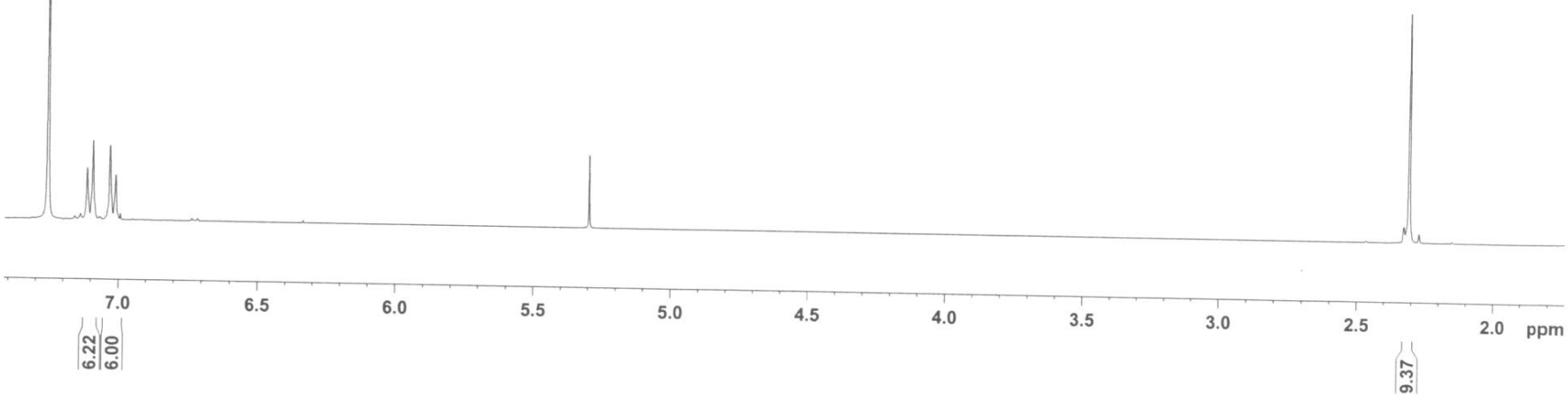

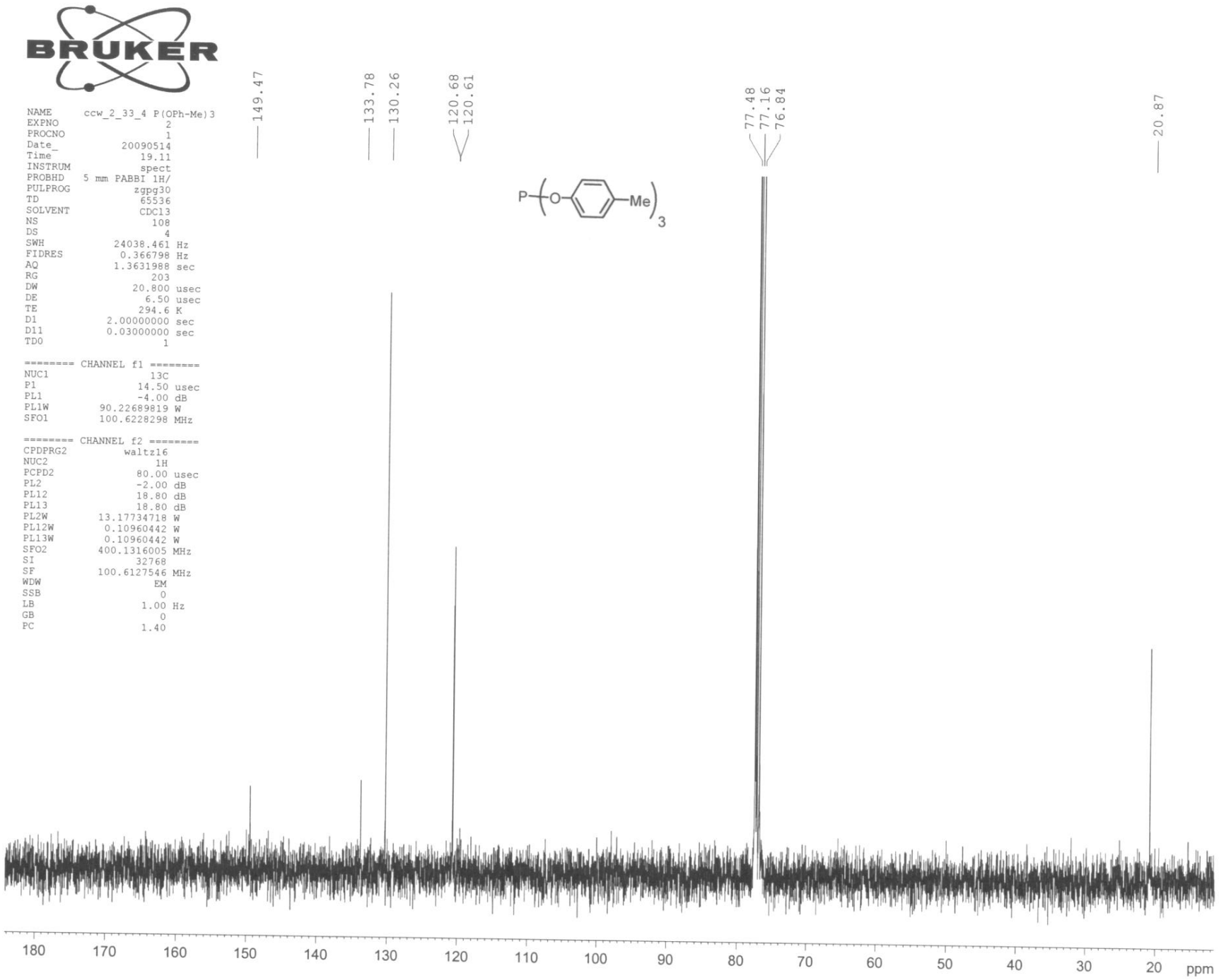
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SFO1 100.6228298 MHz
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PL12 18.80 dB
PL13 18.80 dB
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SSB 0
LB 1.00 Hz
GB 0
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Me
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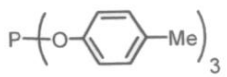

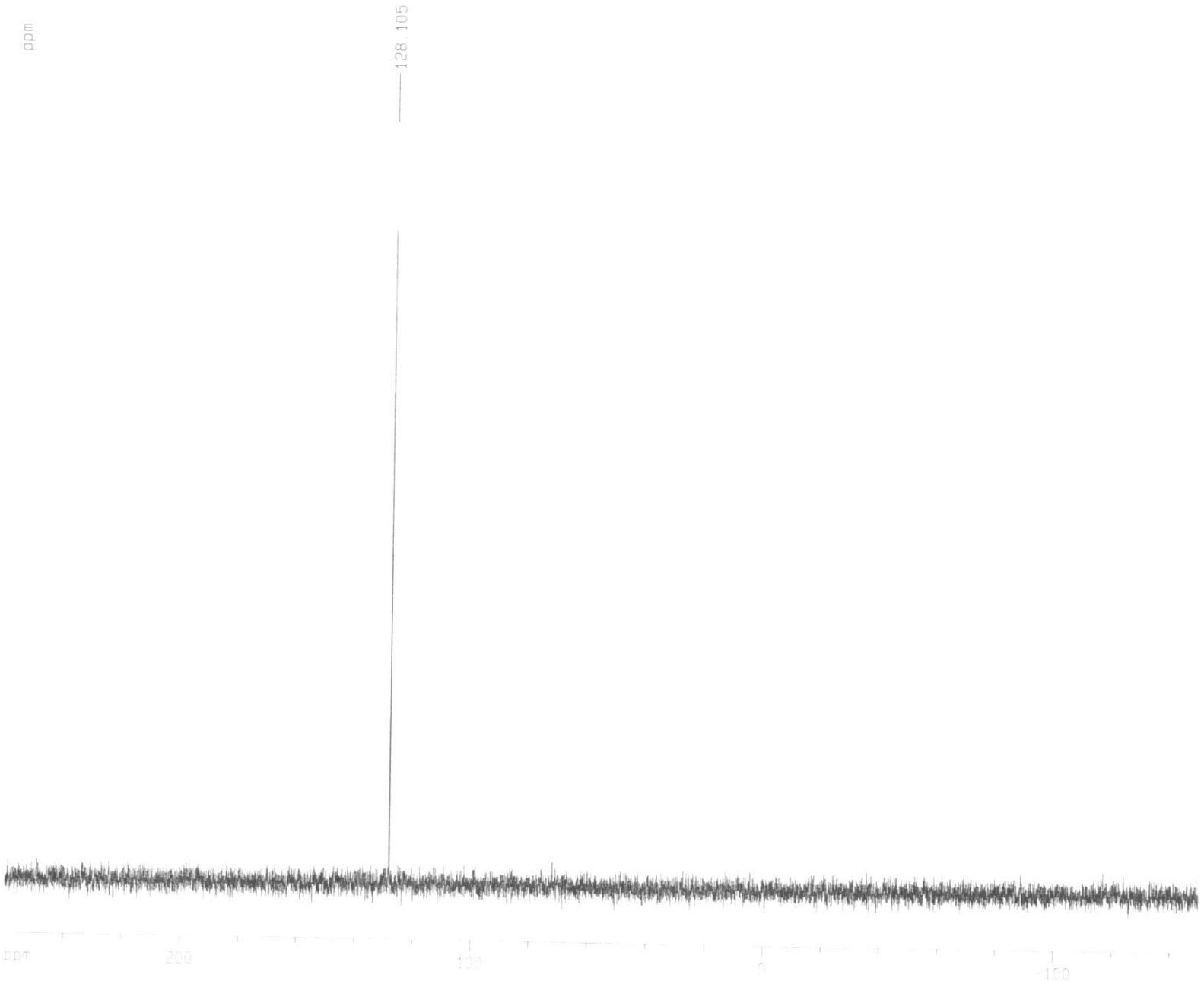


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PC                1.40

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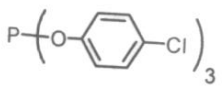

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Integral

6.2678
6.0000

ppm 8.5 8.0 7.5 7.0 6.5

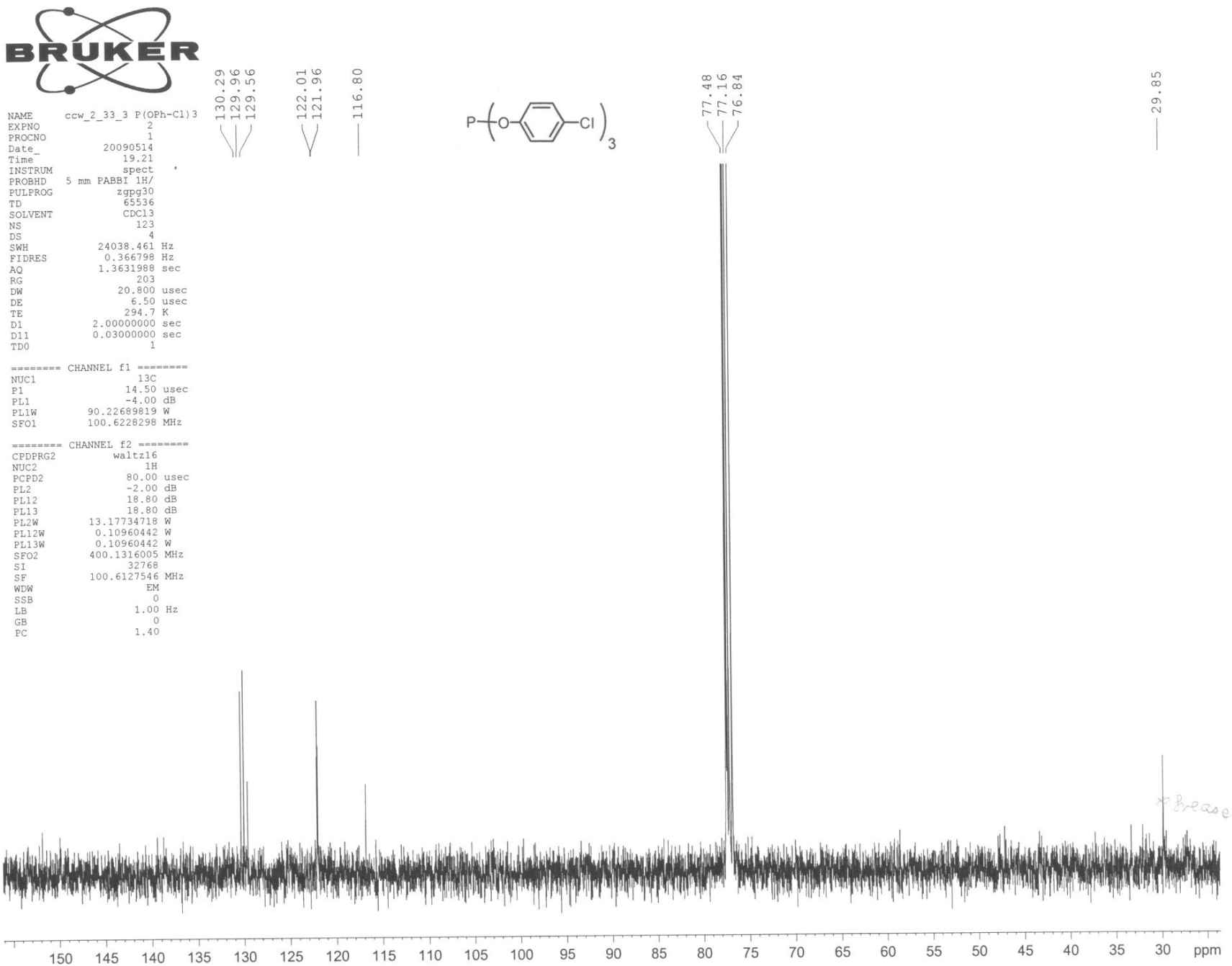
Current Data Parameters
NAME ccw_2_33_3 P(OPh-Cl)3 column
EXPNO 1
PROCNO 1

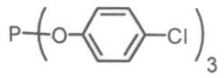
F2 - Acquisition Parameters
Date_ 20090401
Time 13.18
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 42
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 362
DW 55.600 usec
DE 79.43 usec
TE 296.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

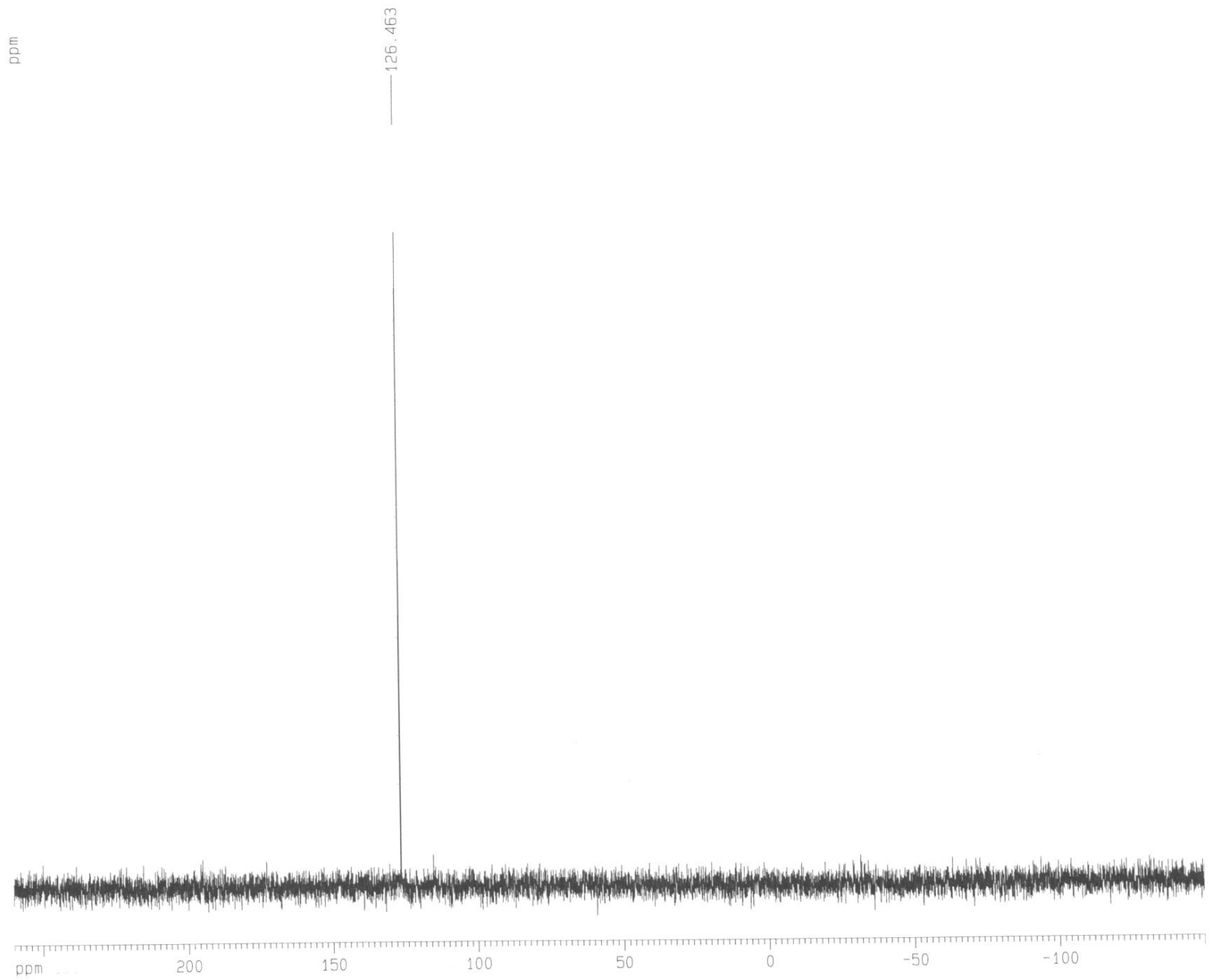
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SF01 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 300.1300056 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 50.00 cm
F1P 9.000 ppm
F1 2701.17 Hz
F2P 6.000 ppm
F2 1800.78 Hz
PPMCM 0.13636 ppm/cm
HZCM 40.92682 Hz/cm


BRUKER
NAME ccw_2_33_3 P(OPh-Cl)3
EXPNO 2
PROCNO 1
Date_ 20090514
Time 19.21
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 123
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 294.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
130.29
129.96
129.56
122.01
121.96
116.80
77.48
77.16
76.84
29.85
P
O
Cl
3
grease
150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 ppm



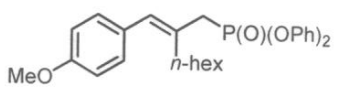


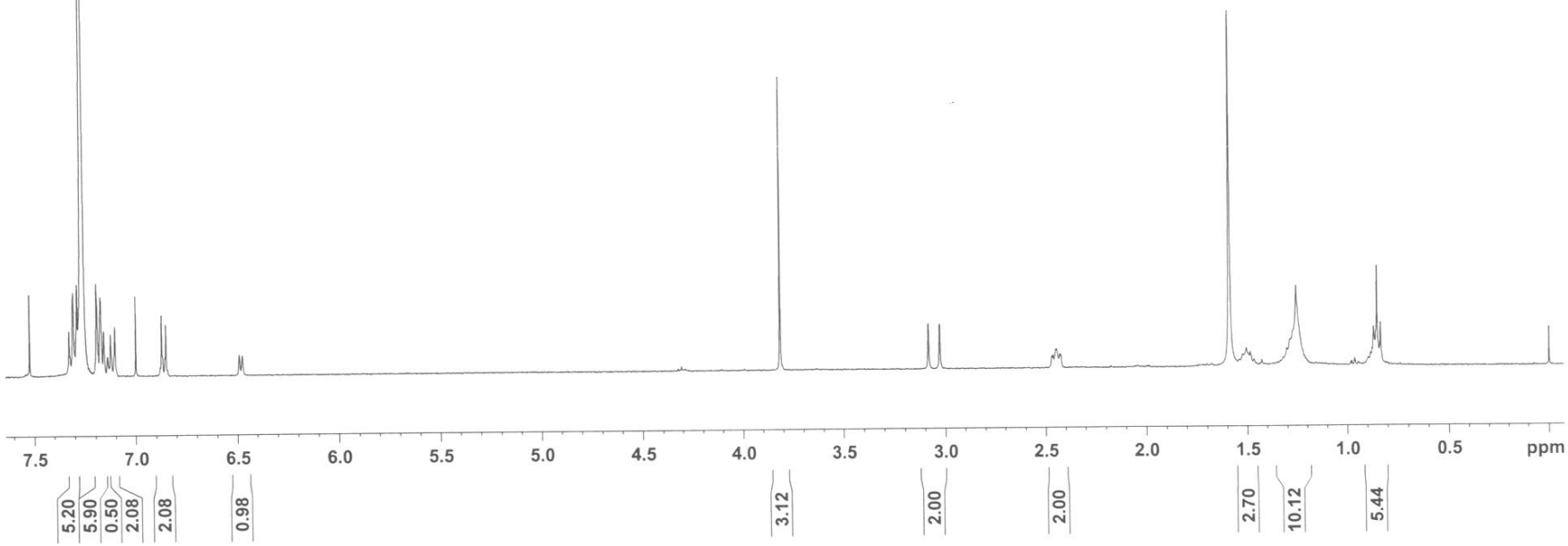

```
Current Data Parameters
NAME      ccw_2_33_3 P(OPh-Cl)3 column
EXPNO                 11
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090401
Time              13.23
INSTRUM         dpx300
PROBHD   5 mm BBO BB-1H
PULPROG           zgdc
TD               65536
SOLVENT          CDCl3
NS                 189
DS                   0
SWH          72992.703 Hz
FIDRES        1.113780 Hz
AQ            0.4489716 sec
RG             9195.2
DW               6.850 usec
DE               6.00 usec
TE              295.2 K
D1            0.10000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1               31P
P1               6.00 usec
PL1             -6.00 dB
SF01       121.4932237 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2          100.00 usec
PL2             120.00 dB
PL12             19.00 dB
SF02       300.1316097 MHz

F2 - Processing parameters
SI                65536
SF          121.4948889 MHz
WDW                EM
SSB                 0
LB               3.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX               22.00 cm
CY               11.84 cm
F1P            260.000 ppm
F1            31588.67 Hz
F2P           -150.000 ppm
F2          -18224.23 Hz
PPMCM         18.63636 ppm/cm
HZCM        2264.22290 Hz/cm
```

P2

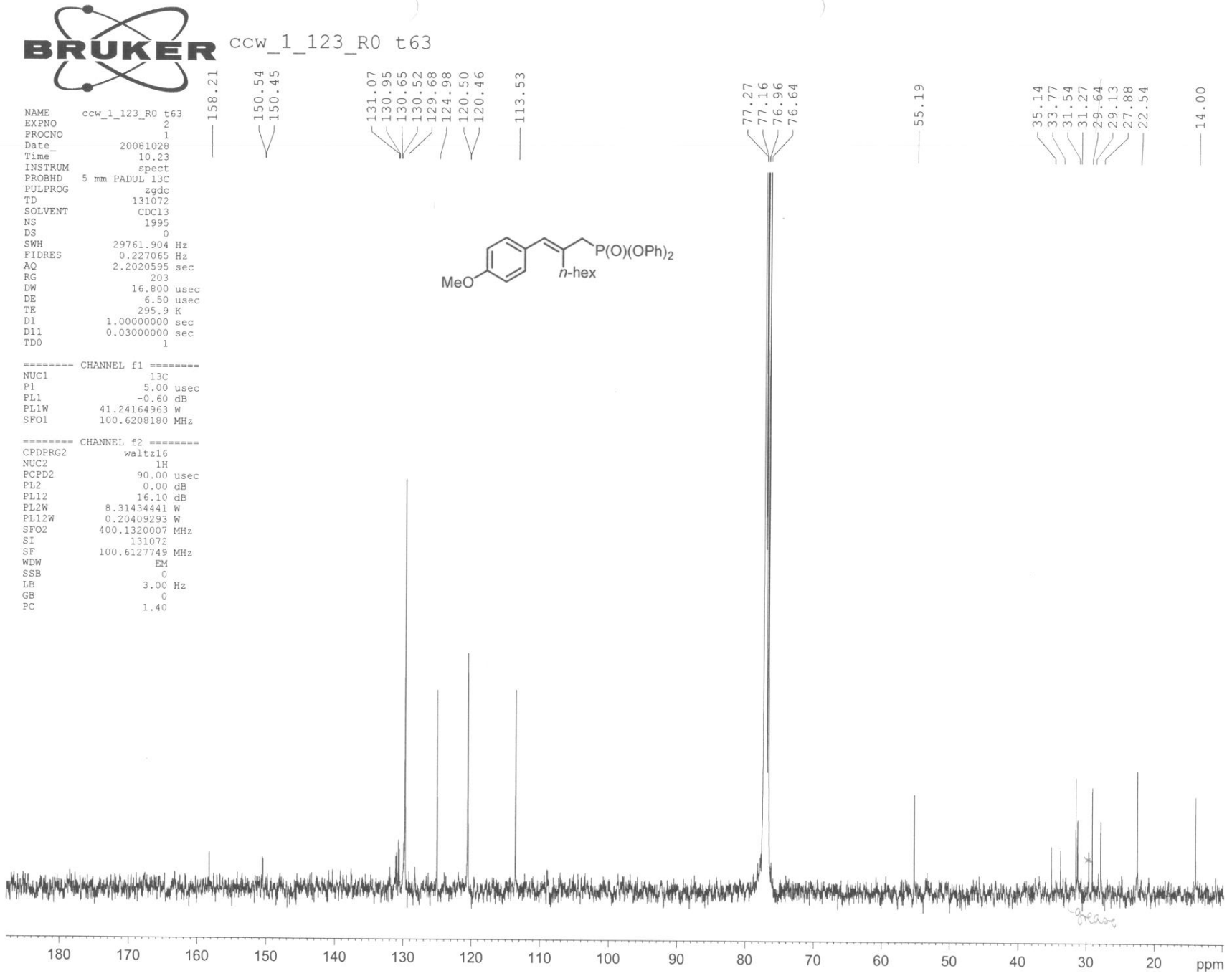
NAME	ccw_1_123_R0 t63
EXPNO	1
PROCNO	1
Date_	20081028
Time	10.15
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	20
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300038 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

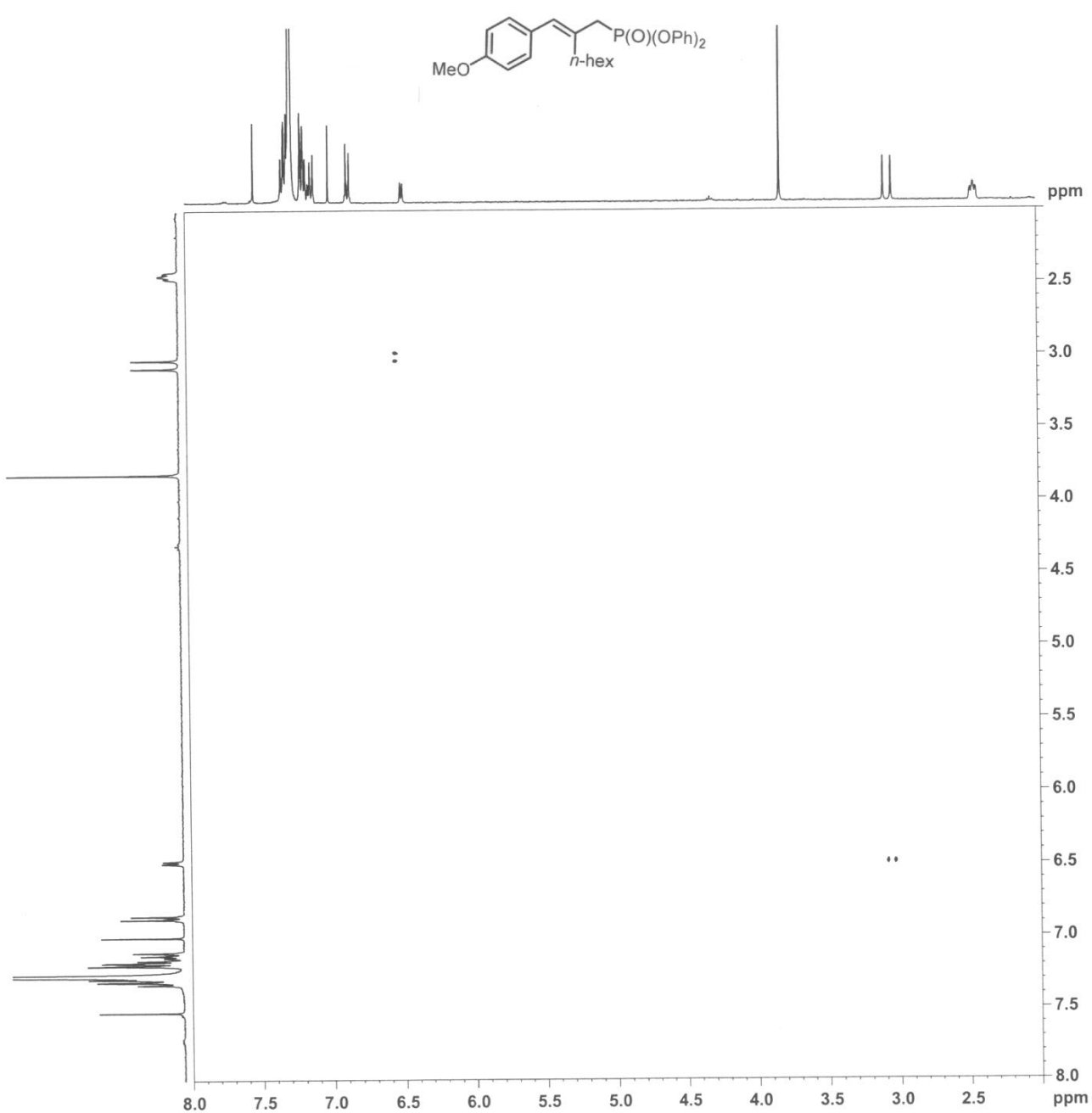
ccw_1_123_R0 t63





A1a1

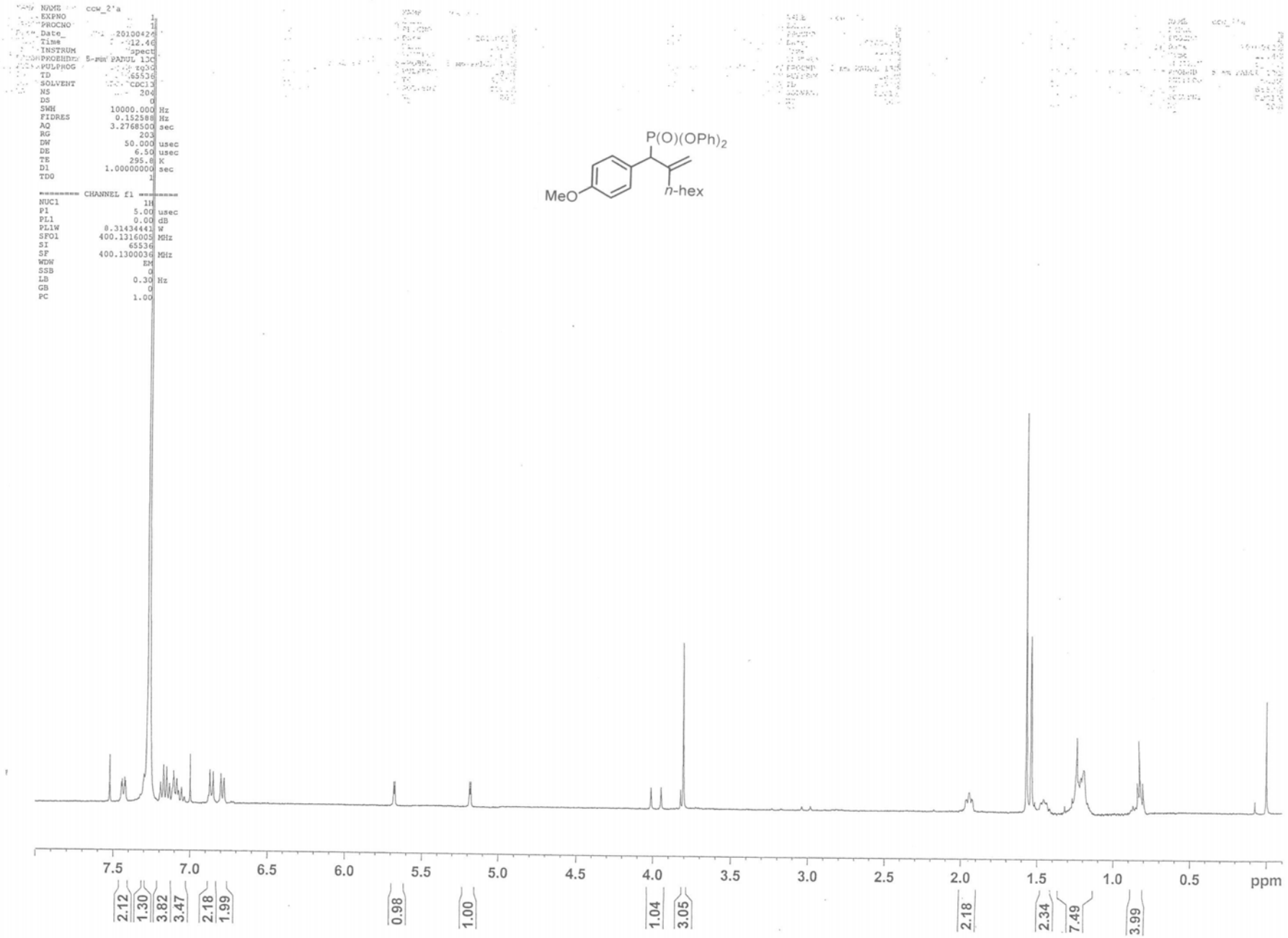

BRUKER
ccw_1_123_R0 t63
158.21
150.54
150.45
131.07
130.95
130.65
130.52
129.68
124.98
120.50
120.46
113.53
77.27
77.16
76.96
76.64
55.19
35.14
33.77
31.54
31.27
29.64
29.13
27.88
22.54
14.00
NAME ccw_1_123_R0 t63
EXPNO 2
PROCNO 1
Date_ 20081028
Time 10.23
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 1995
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL2W 8.31434441 W
PL12W 0.20409293 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127749 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
MeO
P(O)(OPh)2
n-hex
grease
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



BRUKER

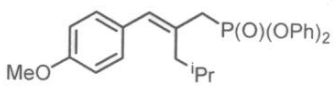
NAME	ccw_1_123_R0 t63
EXPNO	6
PROCNO	1
Date_	20090504
Time	14.13
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	16
SFO1	400.1318 MHz
FIDRES	255.310455 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300127 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300327 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

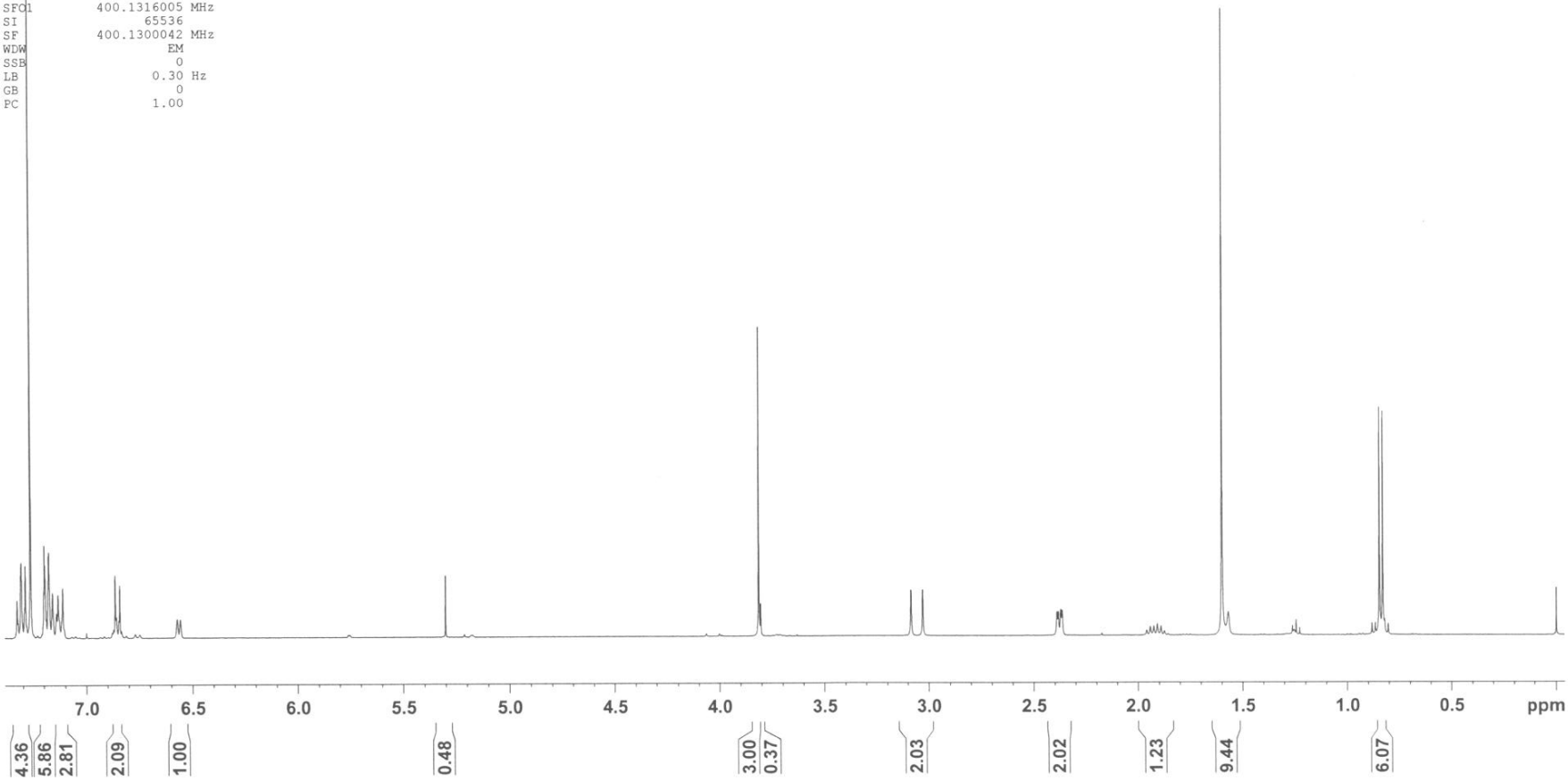
A1q1


NAME ccw_2'a
EXPNO 1
PROCNO 1
Date_ 20100426
Time 12.46
INSTRUM spect
PROBHD 5-mm PABUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 200
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
MeO
n-hex
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.12
1.30
3.82
3.47
2.18
1.99
0.98
1.00
1.04
3.05
2.18
2.34
7.49
3.99

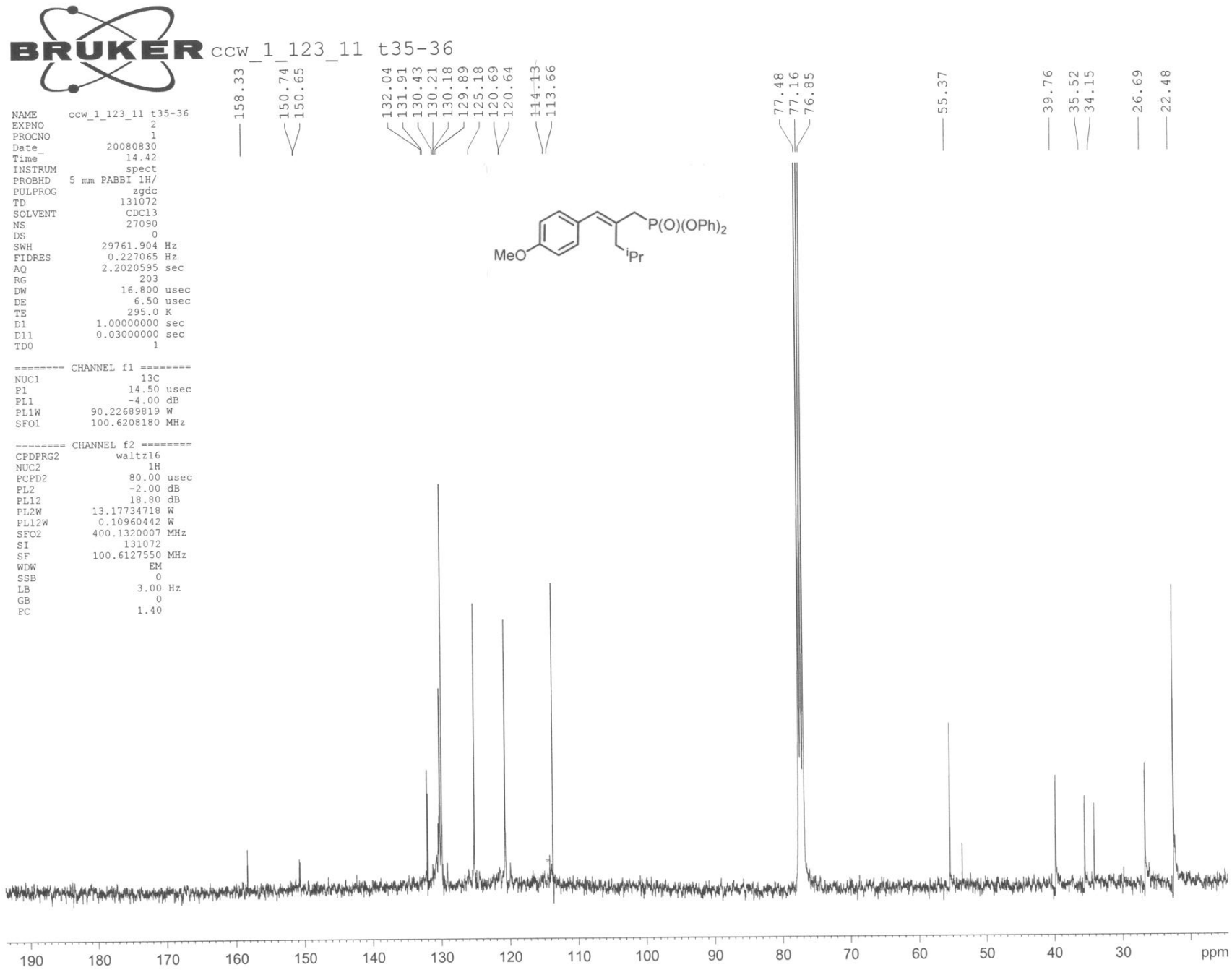
```
NAME      ccw_1_123_11 t35-36
EXPNO                1
PROCNO               1
Date_         20080815
Time             21.19
INSTRUM          spect
PROBHD   5 mm PABBI 1H/
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   0
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ           3.2768500 sec
RG                 203
DW              50.000 usec
DE               6.50 usec
TE              294.7 K
D1           1.00000000 sec
TDO                  1

======== CHANNEL f1 ========
NUC1                1H
P1                5.00 usec
PL1               0.00 dB
PL1W        8.31434441 W
SFO1       400.1316005 MHz
SI              65536
SF         400.1300042 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC               1.00
```

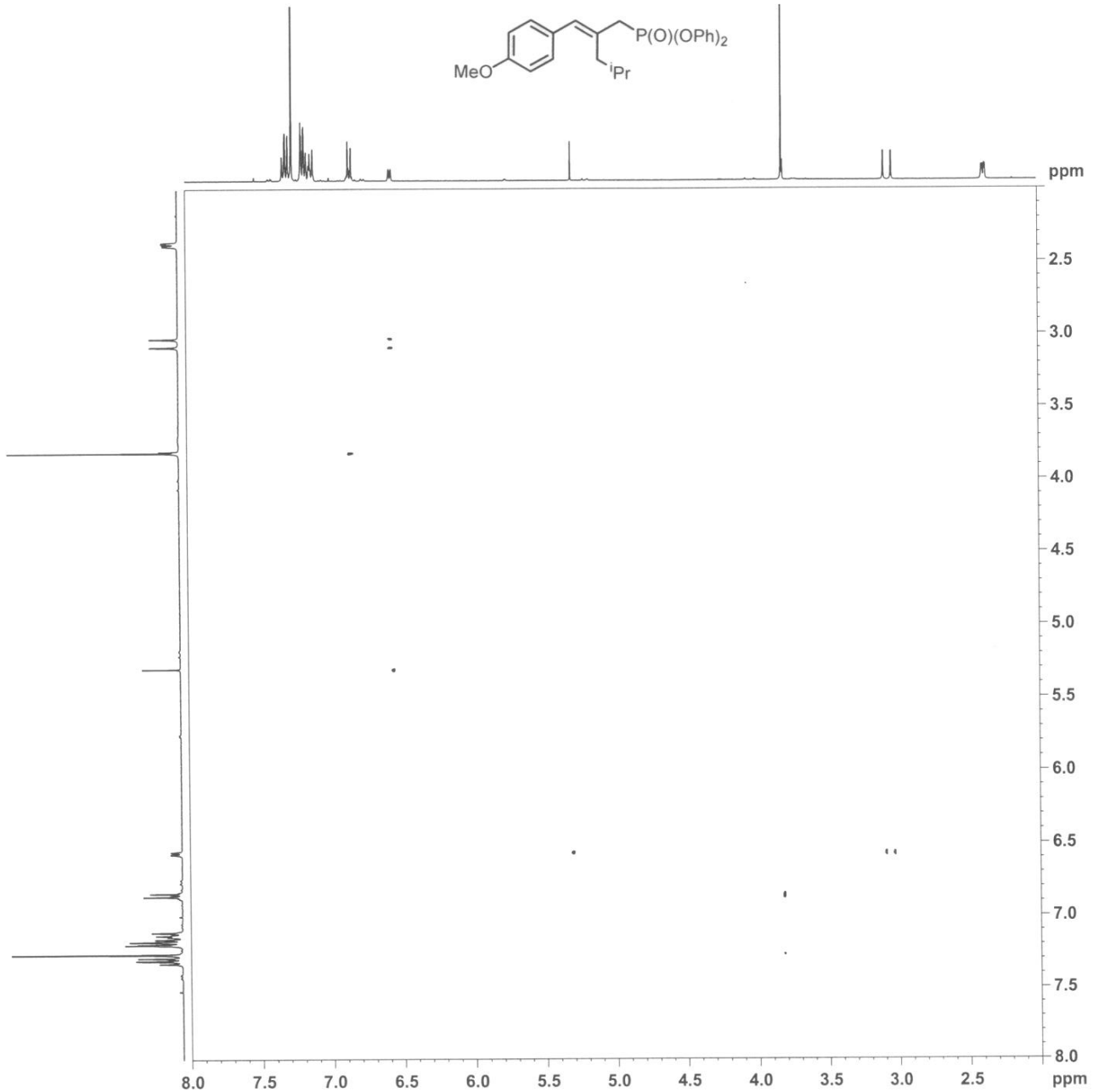
ccw_1_123_11 t35-36





A1b1

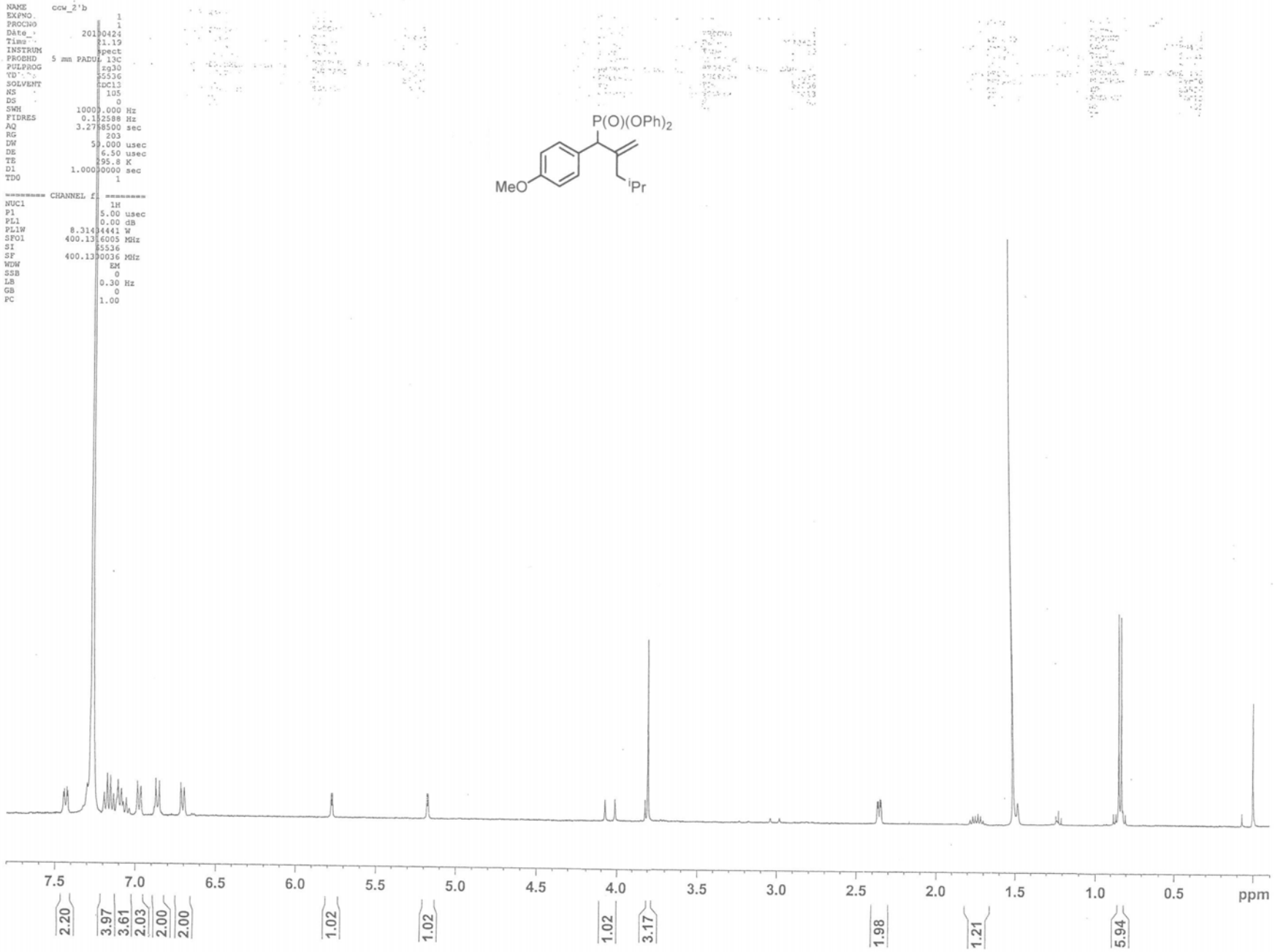

BRUKER
ccw_1_123_11 t35-36
NAME ccw_1_123_11 t35-36
EXPNO 2
PROCNO 1
Date_ 20080830
Time 14.42
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 27090
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127550 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
158.33
150.74
150.65
132.04
131.91
130.43
130.21
130.18
129.89
125.18
120.69
120.64
114.13
113.66
77.48
77.16
76.85
55.37
39.76
35.52
34.15
26.69
22.48
MeO
P(O)(OPh)2
iPr
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm



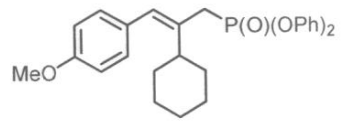
BRUKER

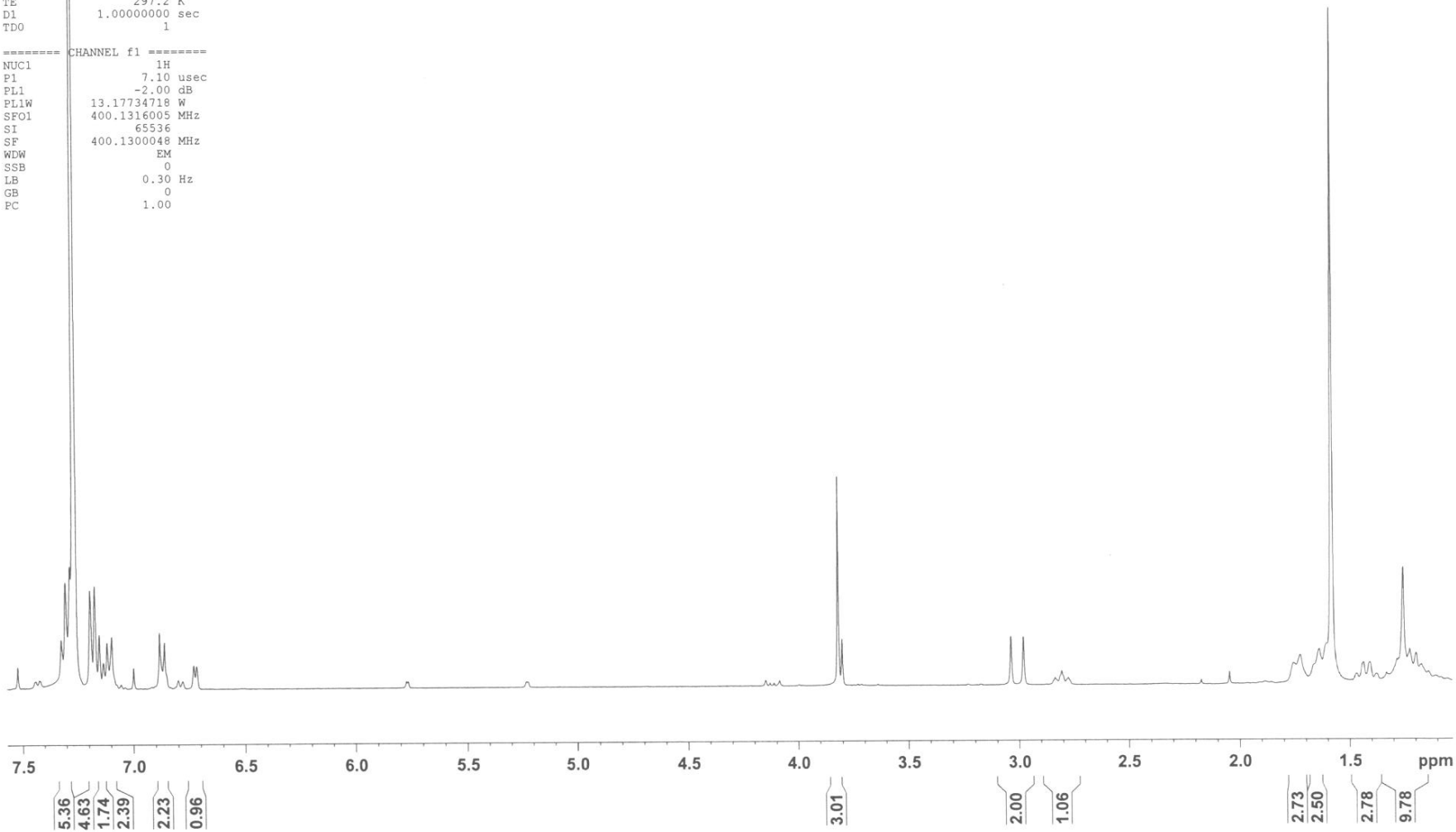
NAME	ccw_1_123_11 t35-36
EXPNO	6
PROCNO	1
Date_	20080829
Time	21.10
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	20
SFO1	400.1318 MHz
FIDRES	204.248367 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

A1b1

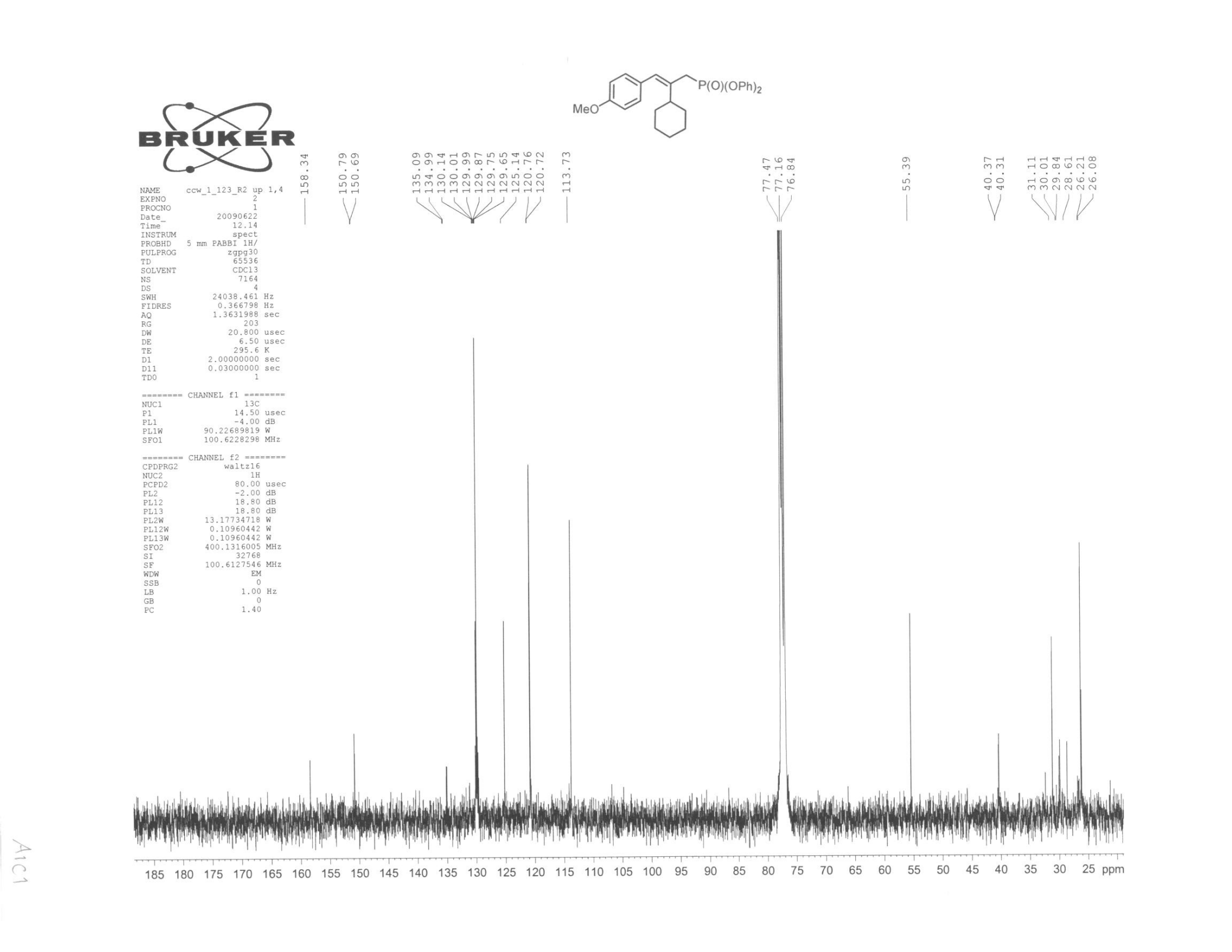

NAME cgw_2'b
EXPNO 1
PROCNO 1
Date_ 20130424
Time 21.19
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 105
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
MeO
iPr
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.20
3.97
3.61
2.03
2.00
2.00
1.02
1.02
1.02
3.17
1.98
1.21
5.94

NAME	ccw_1_123_R2 up 1,4
EXPNO	1
PROCNO	1
Date_	20090622
Time	11.38
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	24
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	297.2 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300048 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

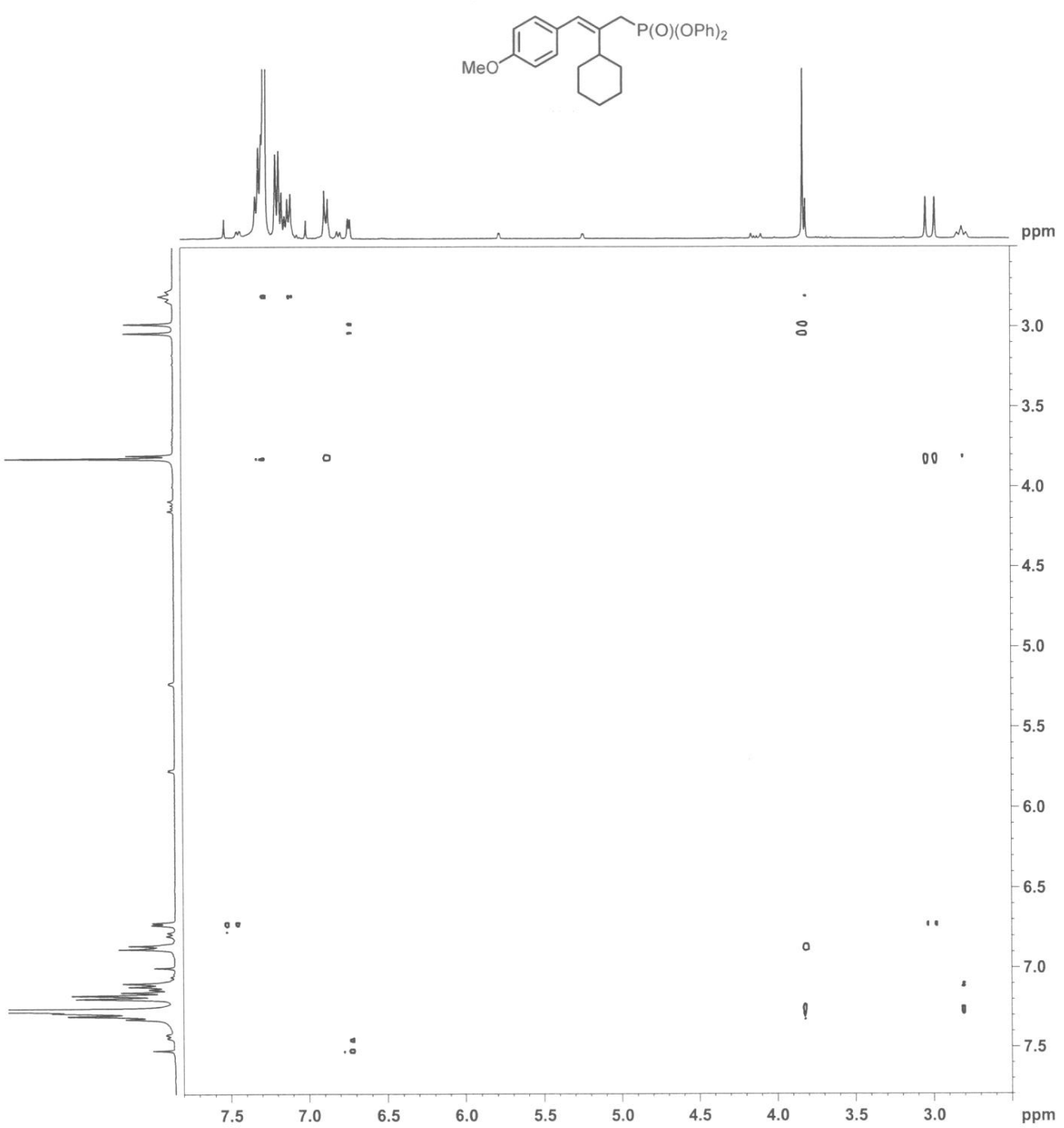




A1c1


MeO
P(O)(OPh)2
158.34
150.79
150.69
135.09
134.99
130.14
130.01
129.99
129.87
129.75
129.65
125.14
120.76
120.72
113.73
77.47
77.16
76.84
55.39
40.37
40.31
31.11
30.01
29.84
28.61
26.21
26.08
NAME ccw_1_123_R2 up 1,4
EXPNO 2
PROCNO 1
Date_ 20090622
Time 12.14
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 7164
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 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 ppm

A1C1



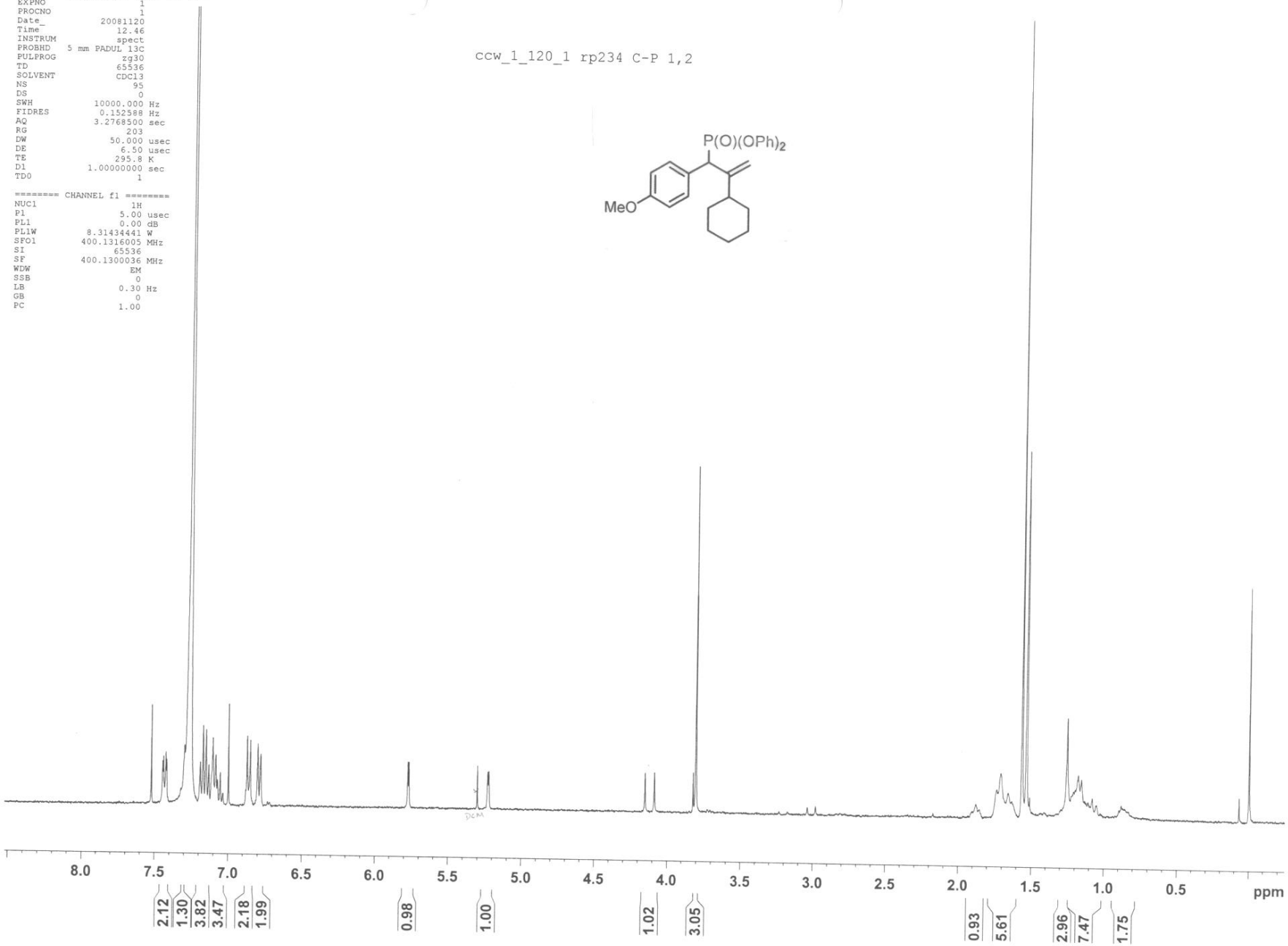
Bruker Advance III 400

NAME	ccw_1_123_R2 up 1,4
EXPNO	6
PROCNO	1
Date_	20090622
Time	11.49
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	296.3 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	27
SFO1	400.1318 MHz
FIDRES	151.295090 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300029 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	States-TPPI
SF	400.1300028 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

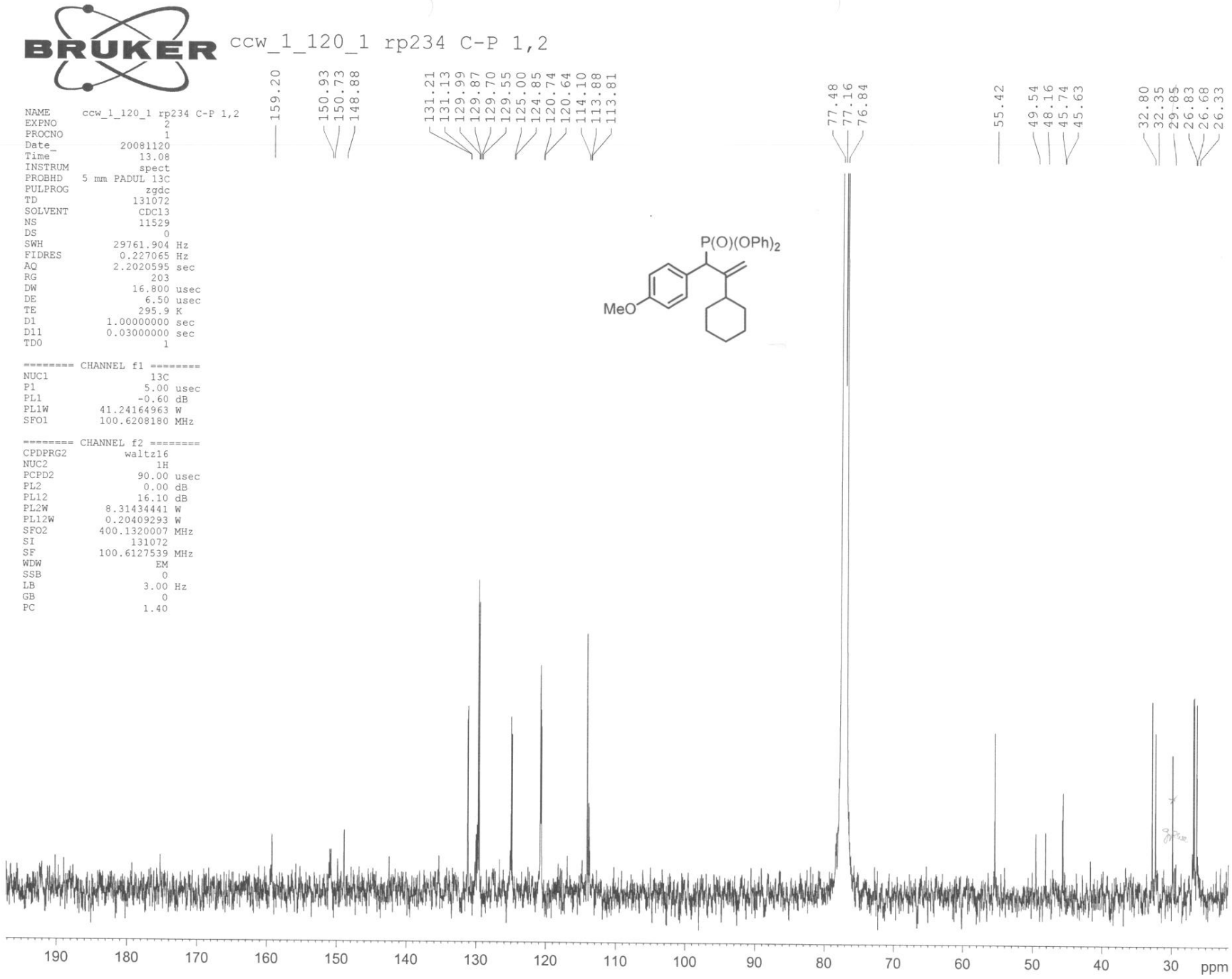
A1c1

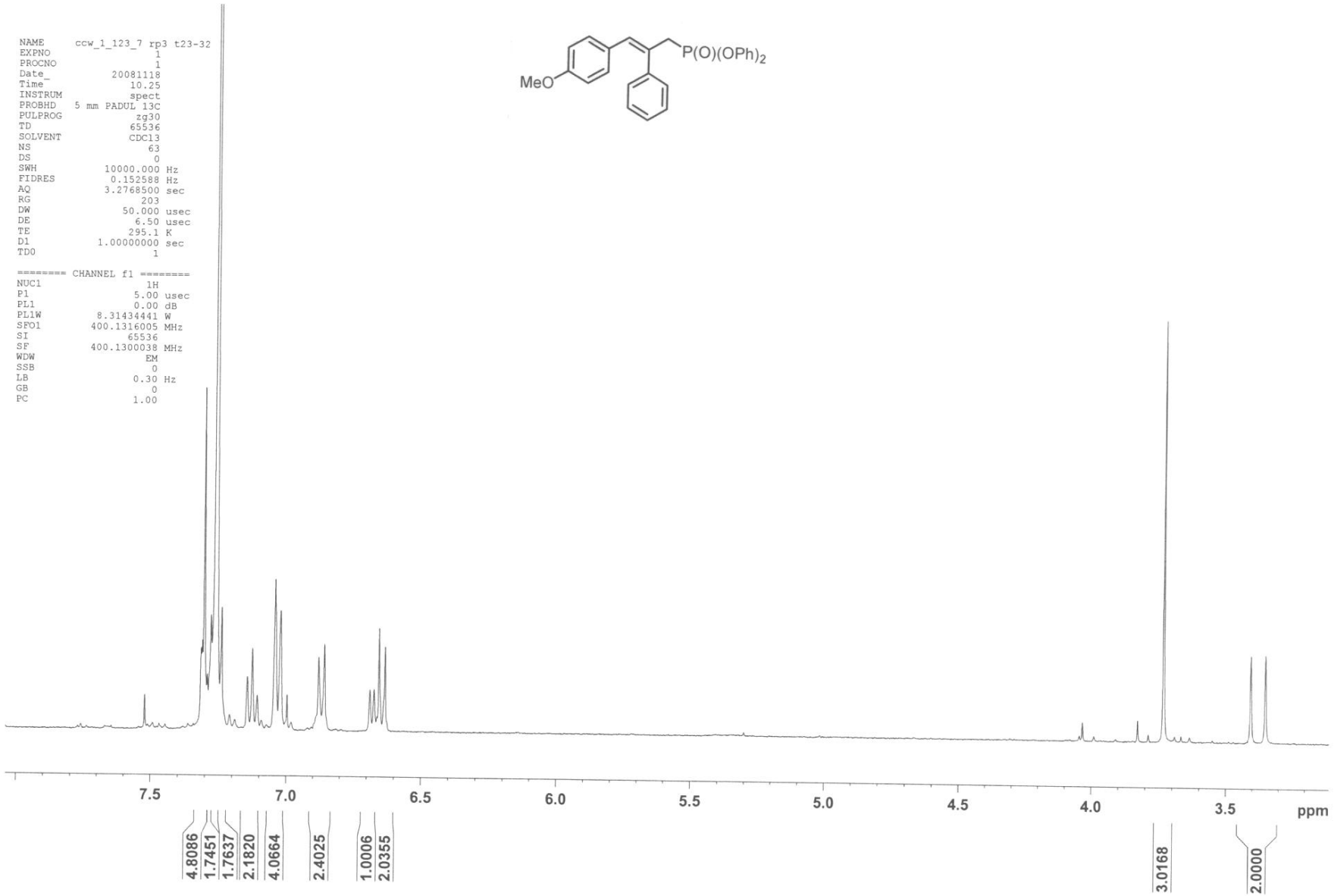
NAME	ccw_1_120_1 rp234 C-P 1,2
EXPNO	1
PROCNO	1
Date_	20081120
Time	12.46
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	95
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	295.8 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300036 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

ccw_1_120_1 rp234 C-P 1,2

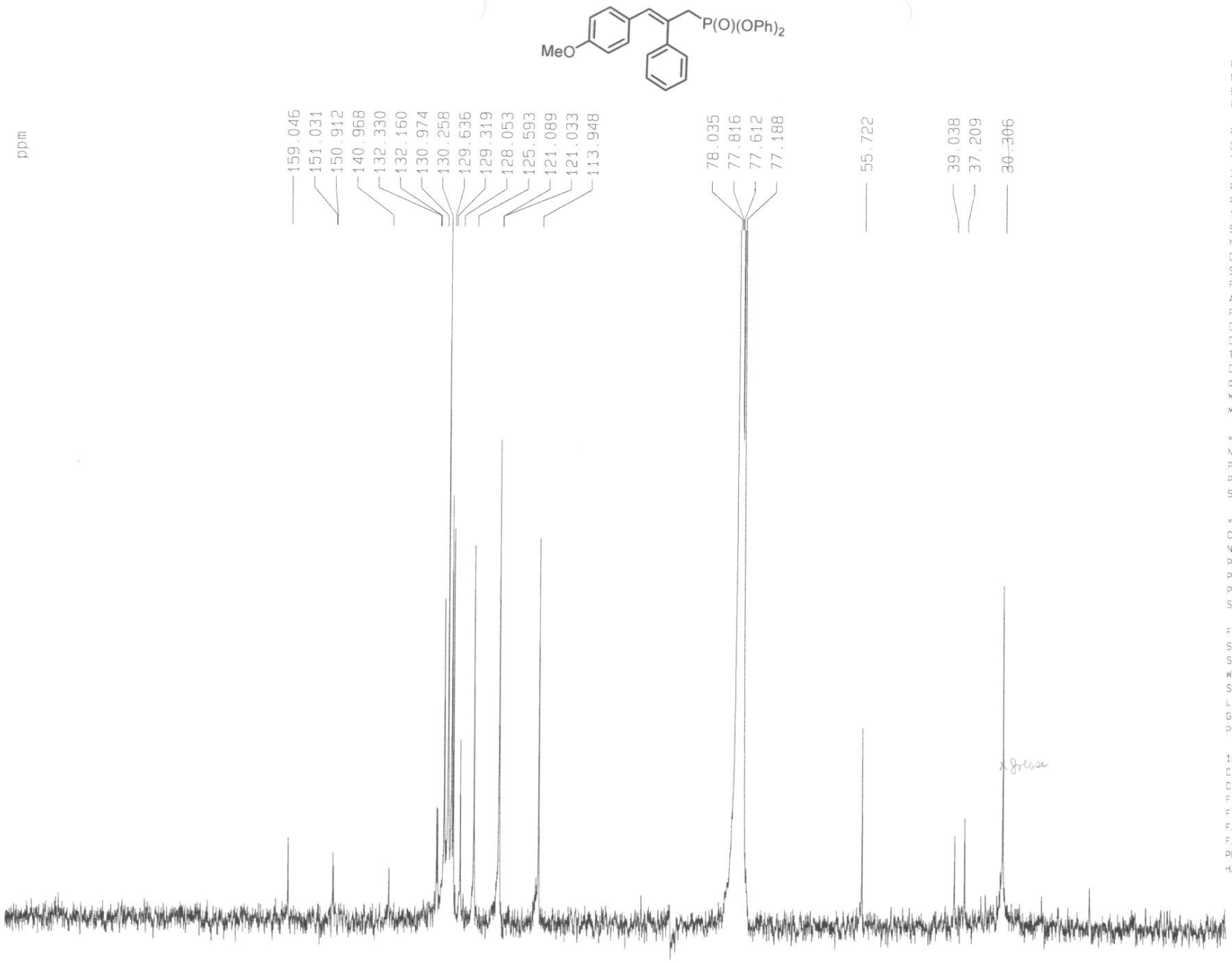


AIC2


ccw_1_120_1 rp234 C-P 1,2
159.20
150.93
150.73
148.88
131.21
131.13
129.99
129.87
129.70
129.55
125.00
124.85
120.74
120.64
114.10
113.88
113.81
77.48
77.16
76.84
55.42
49.54
48.16
45.74
45.63
32.80
32.35
29.85
26.83
26.68
26.33
P(O)(OPh)2
MeO
NAME ccw_1_120_1 rp234 C-P 1,2
EXPNO 2
PROCNO 1
Date_ 20081120
Time 13.08
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 11529
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL2W 8.31434441 W
PL12W 0.20409293 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127539 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm


NAME ccw_1_123_7 rp3 t23-32
EXPNO 1
PROCNO 1
Date_ 20081118
Time 10.25
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 63
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300038 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
MeO
P(O)(OPh)2
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
ppm
4.8086
1.7451
1.7637
2.1820
4.0664
2.4025
1.0006
2.0355
3.0168
2.0000

A1d1



Current Data Parameters

NAME	ccw_1_123_7 rp3 t23-32 13C
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20081127
Time	20.13
INSTRUM	dpx300
PROBHD	5 mm BBO BB-1H
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	20321
DS	0
SWH	22675.736 Hz
FIDRES	0.346004 Hz
AQ	1.4451188 sec
RG	9195.2
DW	22.050 usec
DE	6.00 usec
TE	296.2 K
D1	1.00000000 sec
d11	0.03000000 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

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

NUC1	13C
P1	3.00 usec
PL1	-6.00 dB
SFO1	75.4745111 MHz

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

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	120.00 dB
PL12	19.00 dB
SFO2	300.1315007 MHz

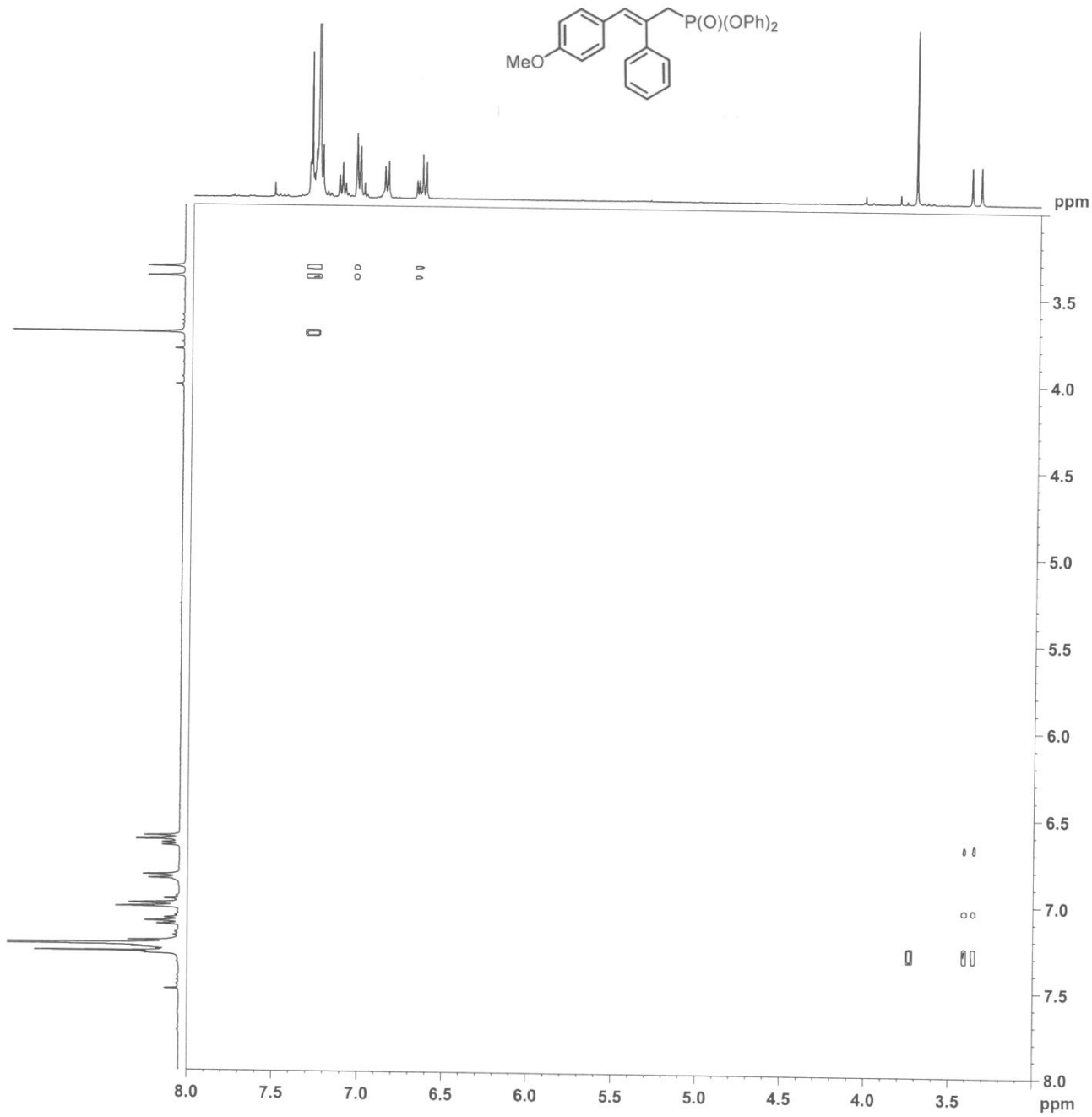
F2 - Processing parameters

SI	65536
SF	75.4677053 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	23.00 cm
CY	75.00 cm
F1P	210.000 ppm
F1	15848.22 Hz
F2P	-10.000 ppm
F2	-754.68 Hz
PPMCM	9.56522 ppm/cm
HZCM	721.86505 Hz/cm

A151



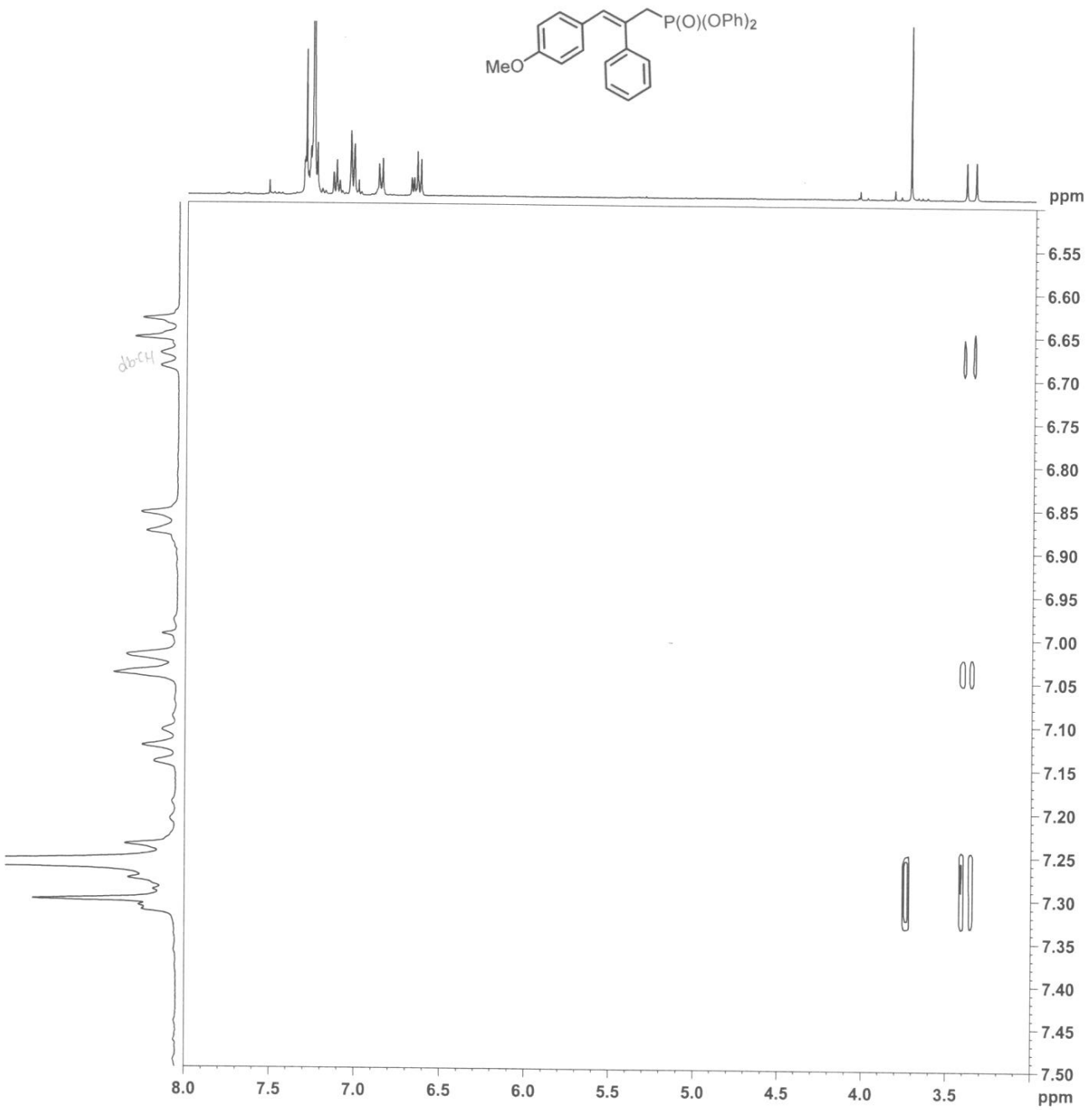
BRUKER

NAME	ccw_1_123_7 rp3 t23-32
EXPNO	6
PROCNO	1
Date_	20081121
Time	17.12
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	295.7 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

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

NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	10
SFO1	400.1318 MHz
FIDRES	408.496735 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

Ald1



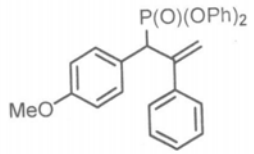
BRUKER

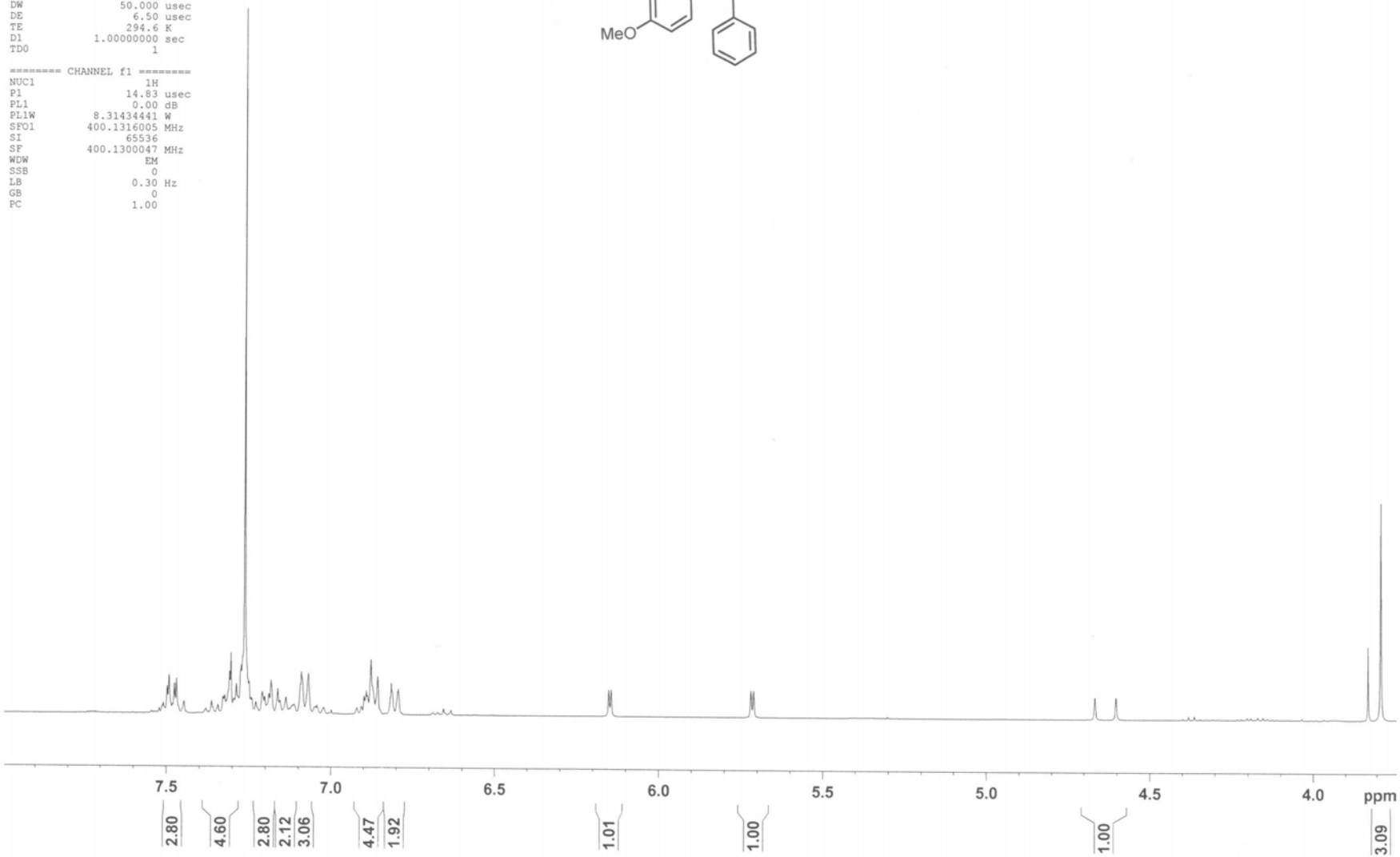
NAME	ccw_1_123_7 rp3 t23-32
EXPNO	6
PROCNO	1
Date_	20081121
Time	17.12
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	295.7 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

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

NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	10
SFO1	400.1318 MHz
FIDRES	408.496735 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

```
NAME        ccw_1_123_7 rp8 t17-22
EXPNO                    1
PROCNO                   1
Date_             20100424
Time                 10.28
INSTRUM              spect
PROBHD    5 mm PABBI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     14
DS                      0
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                     203
DW                50.000 usec
DE                 6.50 usec
TE                 294.6 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 14.83 usec
PL1                 0.00 dB
PL1W          8.31434441 W
SFO1        400.1316005 MHz
SI                  65536
SF          400.1300047 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                   1.00
```

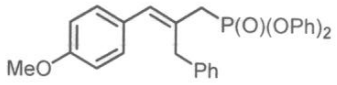


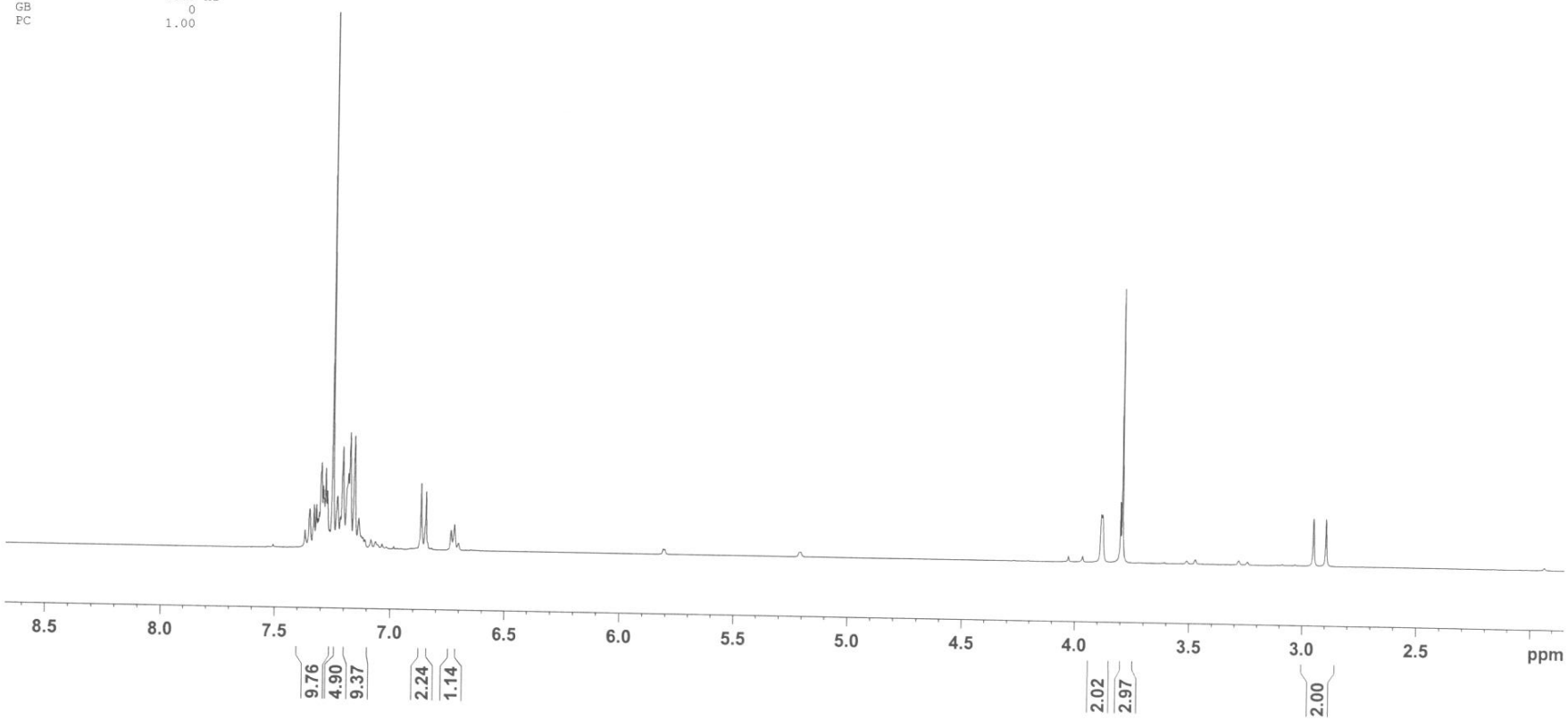


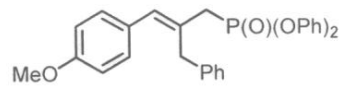
NAME	ccw_1_123_5_3 t5-7
EXPNO	1
PROCNO	1
Date_	20081031
Time	14.41
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	144
DW	50.000 usec
DE	6.50 usec
TE	295.1 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300088 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

ccw_1_123_5_3 t5-7







ppm

158.755
150.707
150.585
138.838
132.655
132.485
129.996
129.885
129.626
129.515
128.929
128.772
128.535
126.532
125.743
125.202
125.093
120.666
120.609
120.484
120.292
120.228
114.216
113.911
77.585
77.365
77.162
76.738
55.384
37.130
35.114
33.289

Current Data Parameters
NAME ccw_1_123_5_3 t5-7 13C
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081030
Time 20.14
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 21305
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 298.2 K
D1 1.00000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz

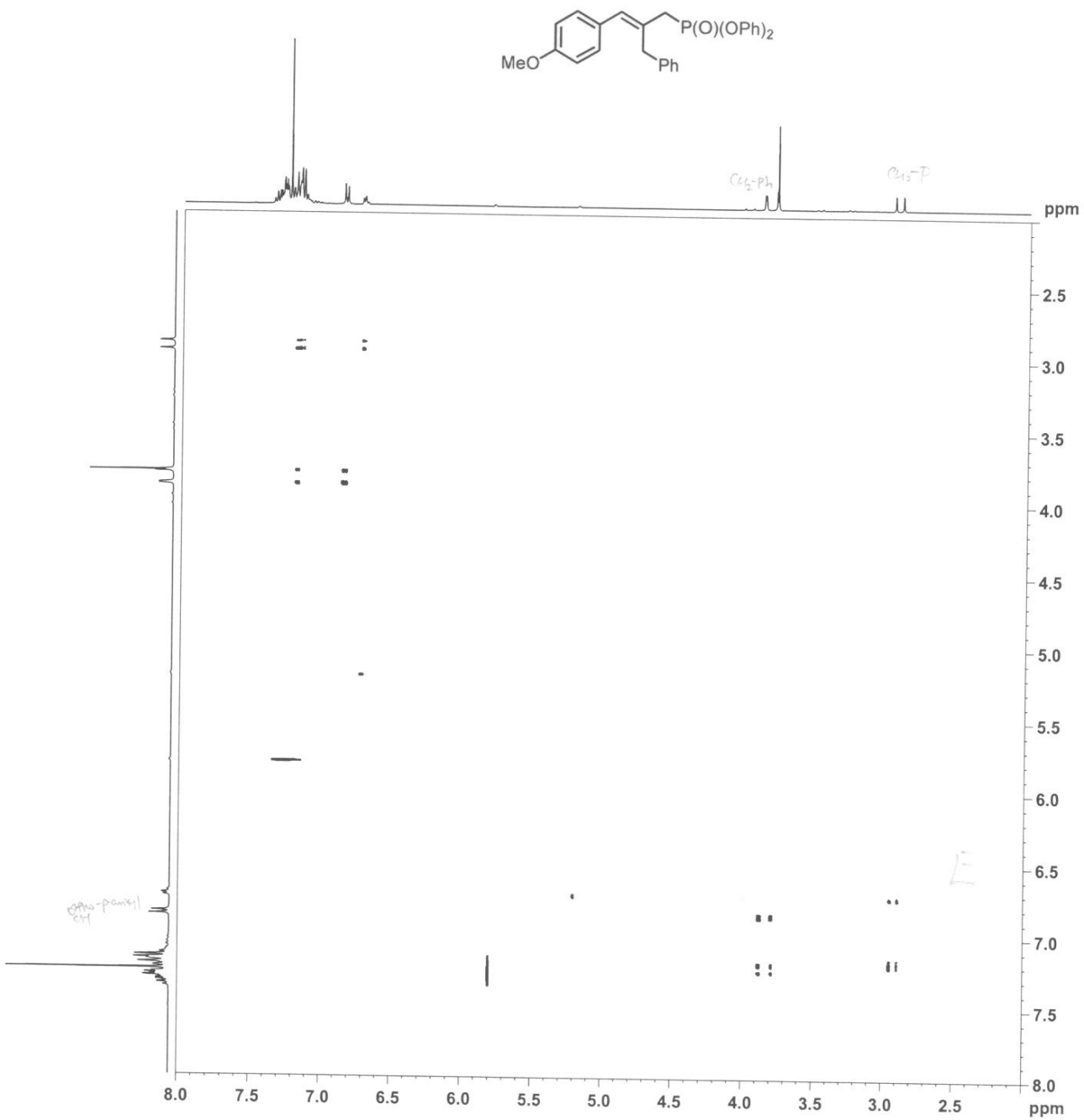
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677403 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 12.00 cm
F1P 204.375 ppm
F1 15423.75 Hz
F2P -7.698 ppm
F2 -580.97 Hz
PPMCM 9.22060 ppm/cm
HZCM 695.85767 Hz/cm

ppm 180 160 140 120 100 80 60 40 20 0

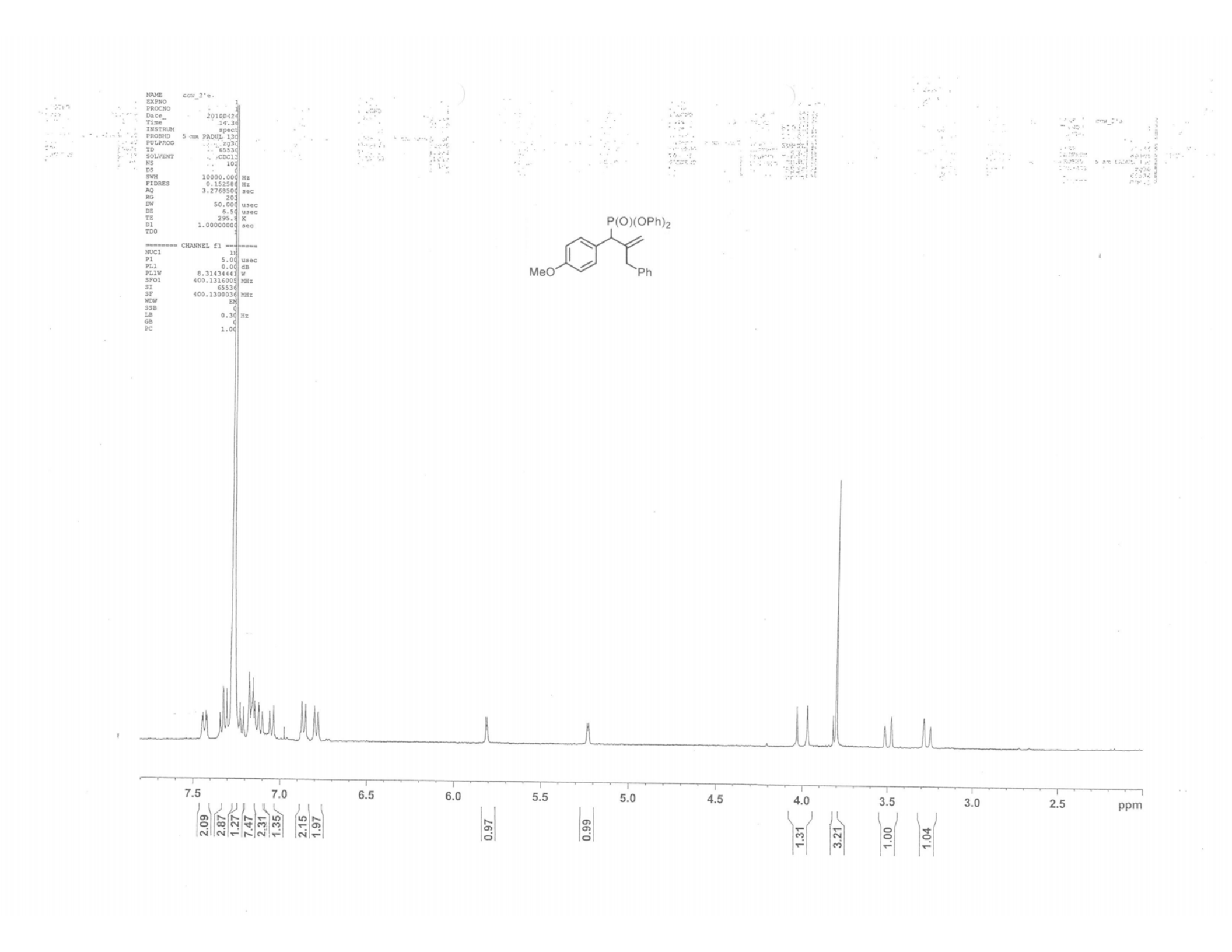
Ale1



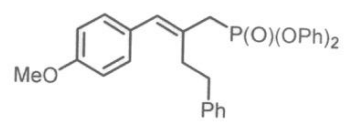
BRUKER

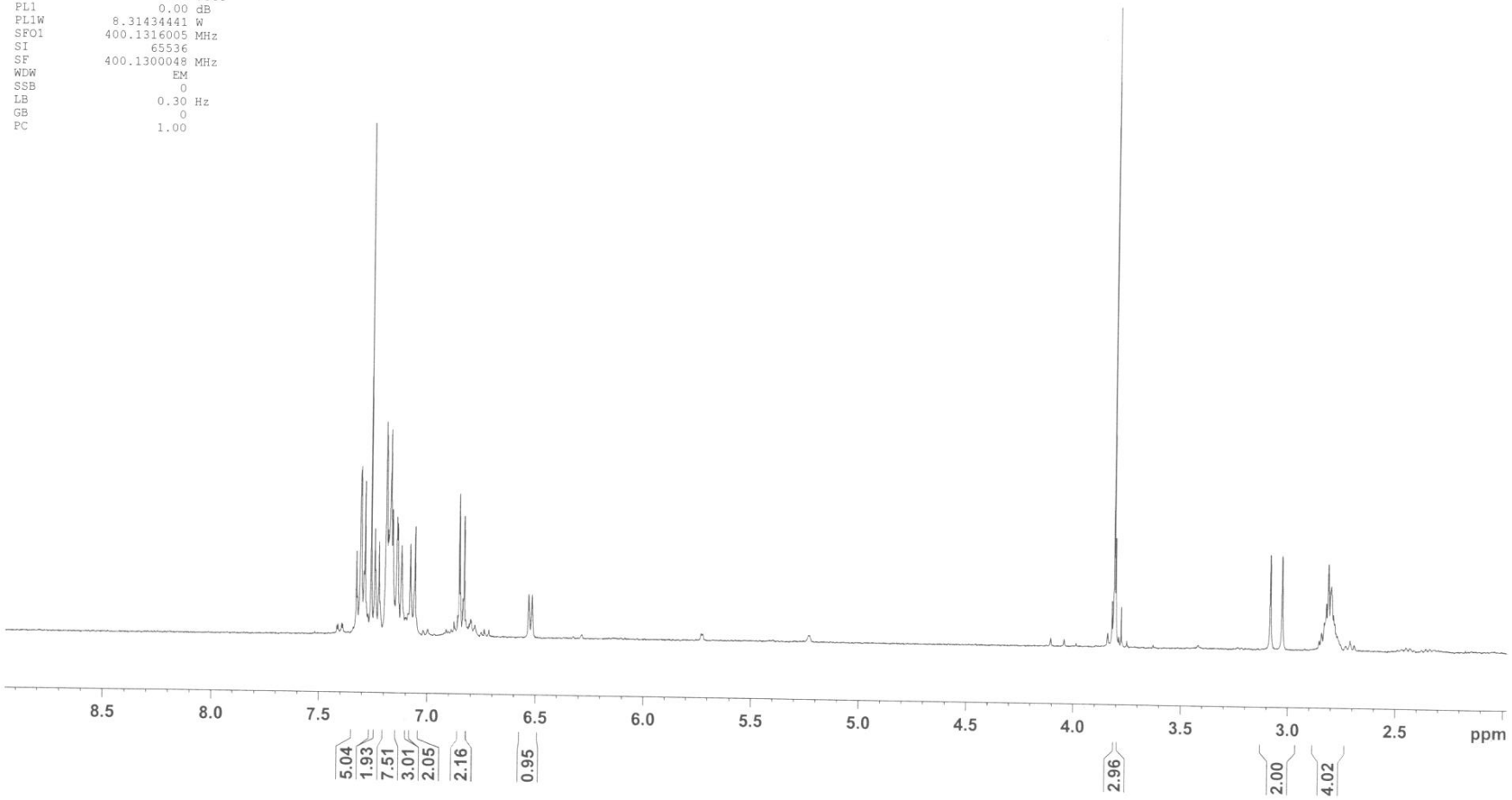
NAME	ccw_1_123_5_3 t5-7
EXPNO	6
PROCNO	1
Date_	20081031
Time	14.43
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	295.1 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	16
SFO1	400.1318 MHz
FIDRES	255.310455 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300088 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300088 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

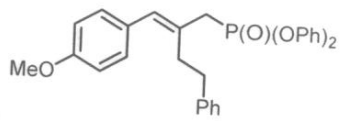

NAME ccw_2'e
EXPNO 1
PROCNO 1
Date_ 20100424
Time 14.36
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 102
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
PL1W 8.31434443 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
MeO
Ph
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
2.09
2.87
1.27
7.47
2.31
1.35
2.15
1.97
0.97
0.99
1.31
3.21
1.00
1.04

NAME	ccw_1_123_15 rp t15
EXPNO	1
PROCNO	1
Date_	20080910
Time	9.50
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300048 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





A181



ppm

158.503
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150.546
141.354
131.897
131.718
129.914
128.520
126.156
125.253
120.701
120.643
113.765
77.582
77.361
77.159
76.736
55.395
35.848
34.198
33.249

Current Data Parameters
NAME ccw_1_123_15 rp t15
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080911
Time 18.04
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 1415
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 9195.2
DW 22.050 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SF01 75.4745111 MHz

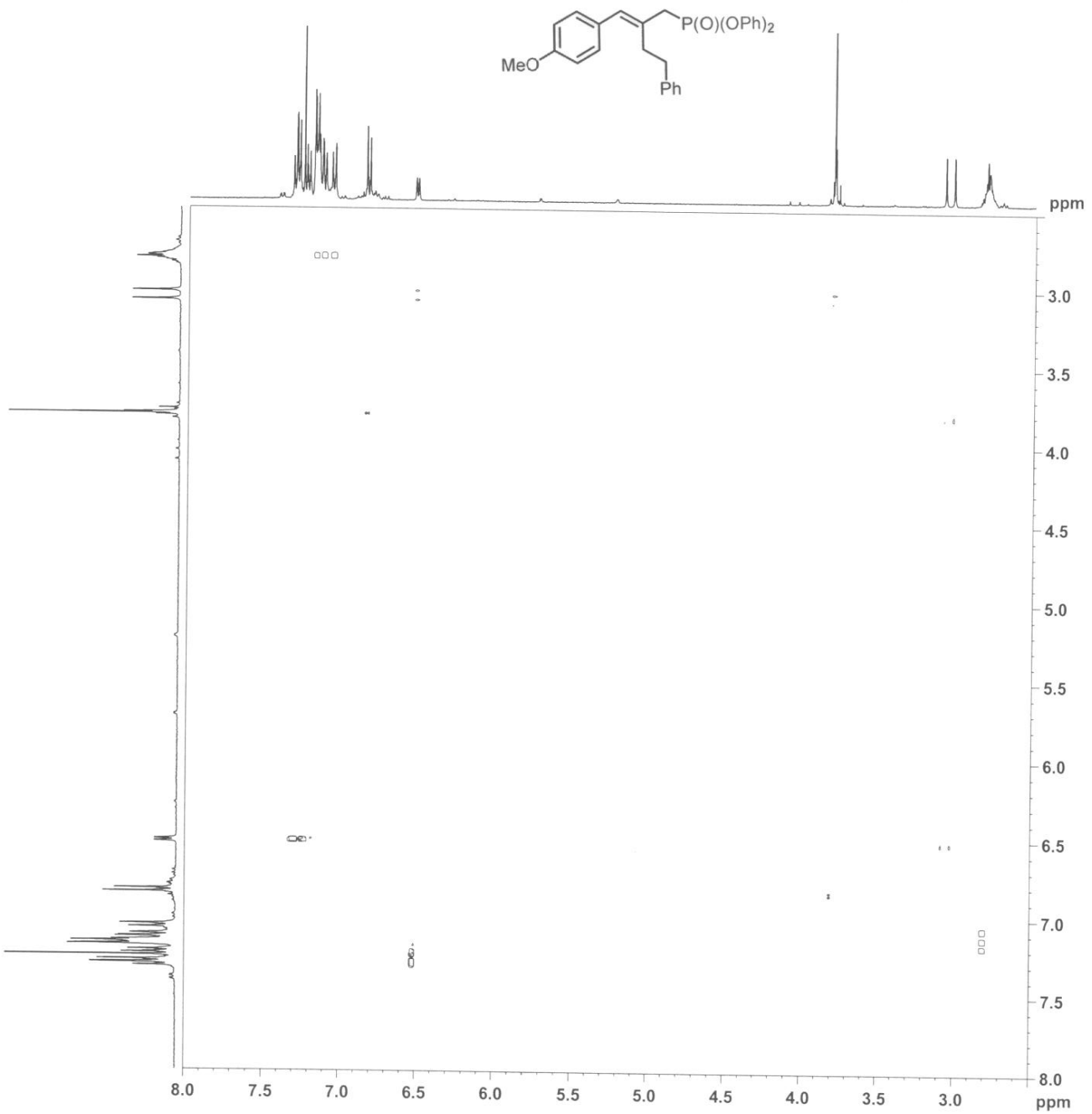
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677400 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 50.00 cm
F1P 210.000 ppm
F1 15848.23 Hz
F2P -10.000 ppm
F2 -754.68 Hz
PPMCM 9.56522 ppm/cm
HZCM 721.86536 Hz/cm

ppm 180 160 140 120 100 80 60 40 20 0

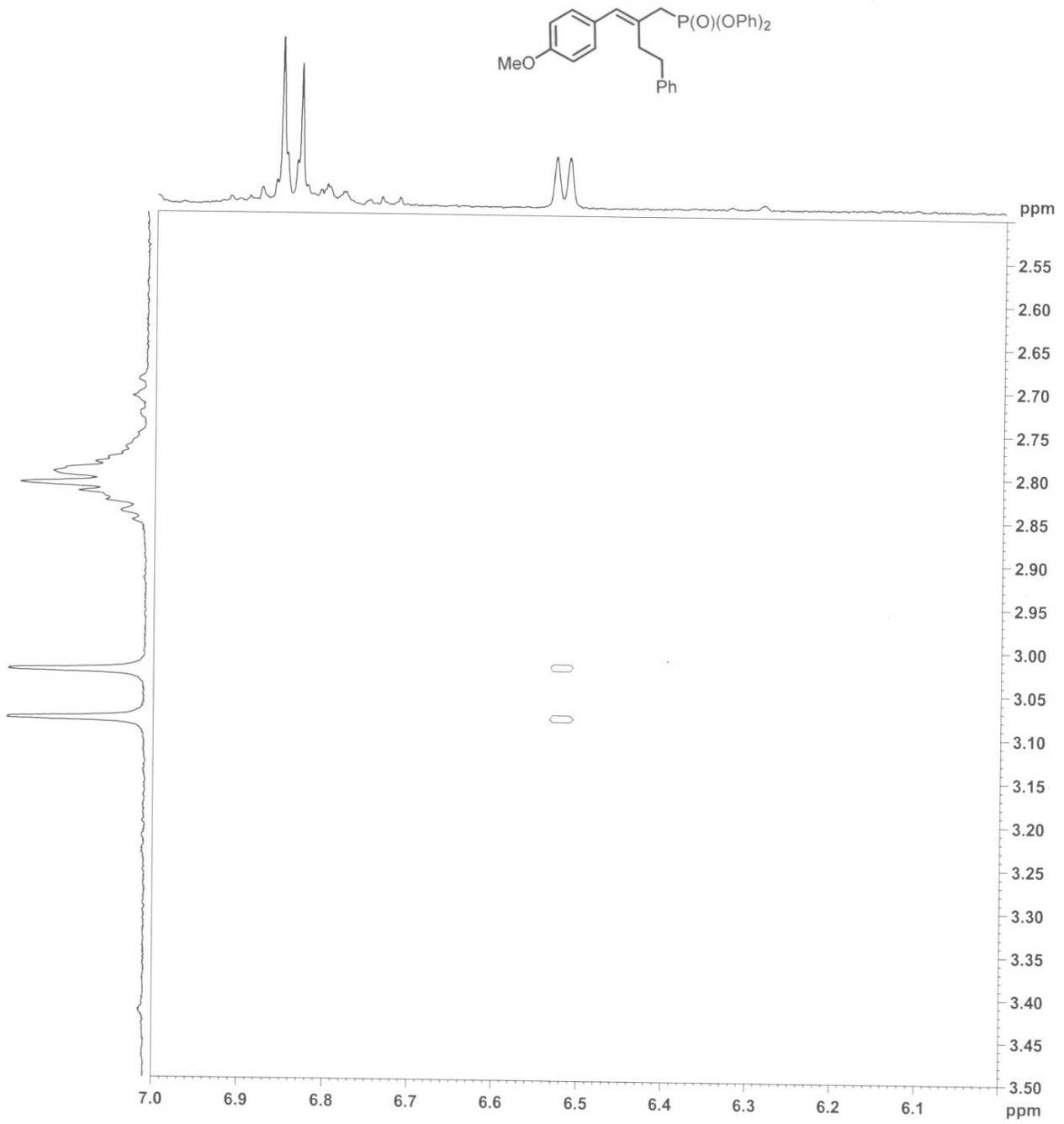
A1f



BRUKER

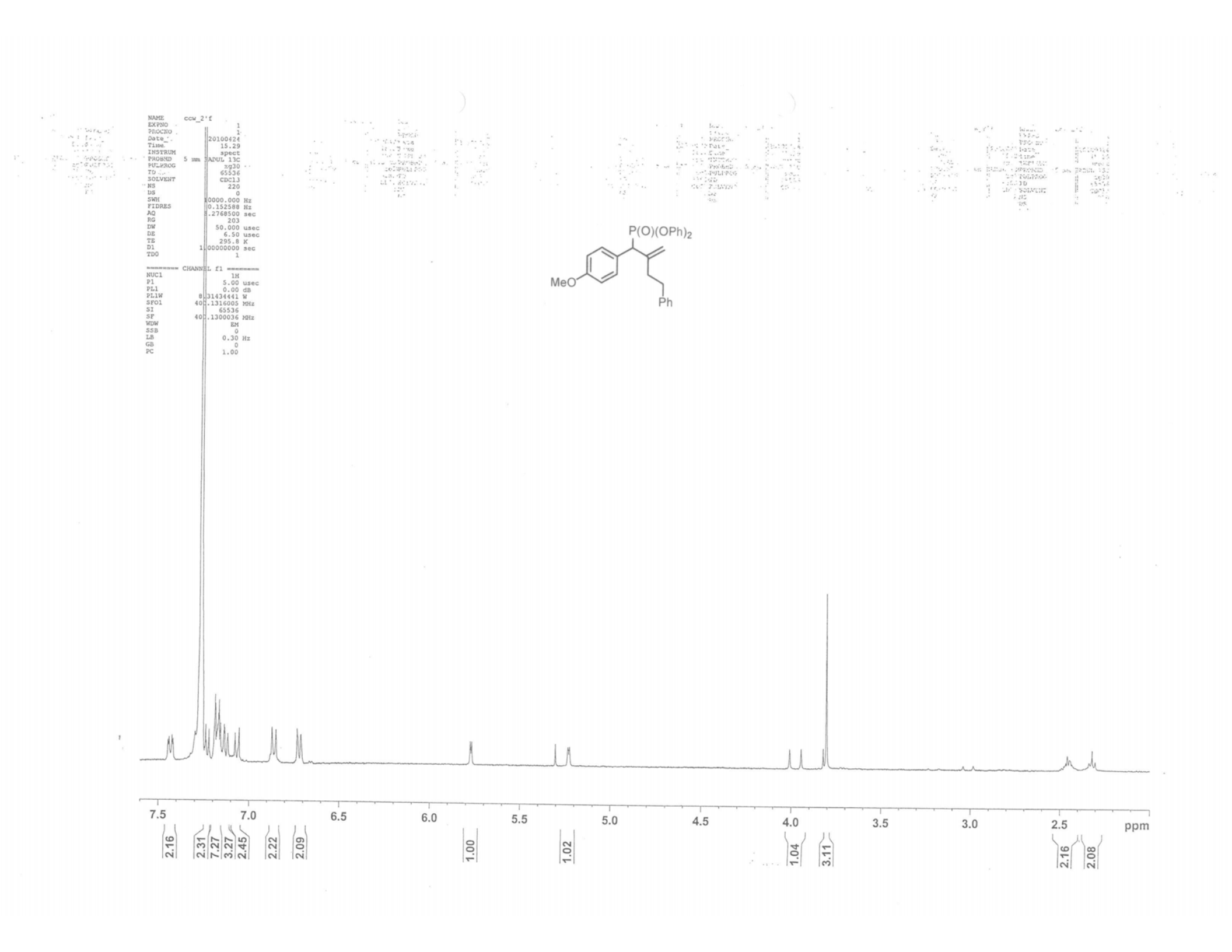
NAME	ccw_1_123_15 rp t15
EXPNO	6
PROCNO	1
Date_	20080910
Time	9.53
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	28
SFO1	400.1318 MHz
FIDRES	145.891693 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

A1f1

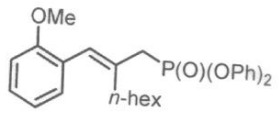


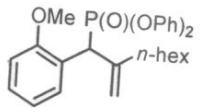
BRUKER

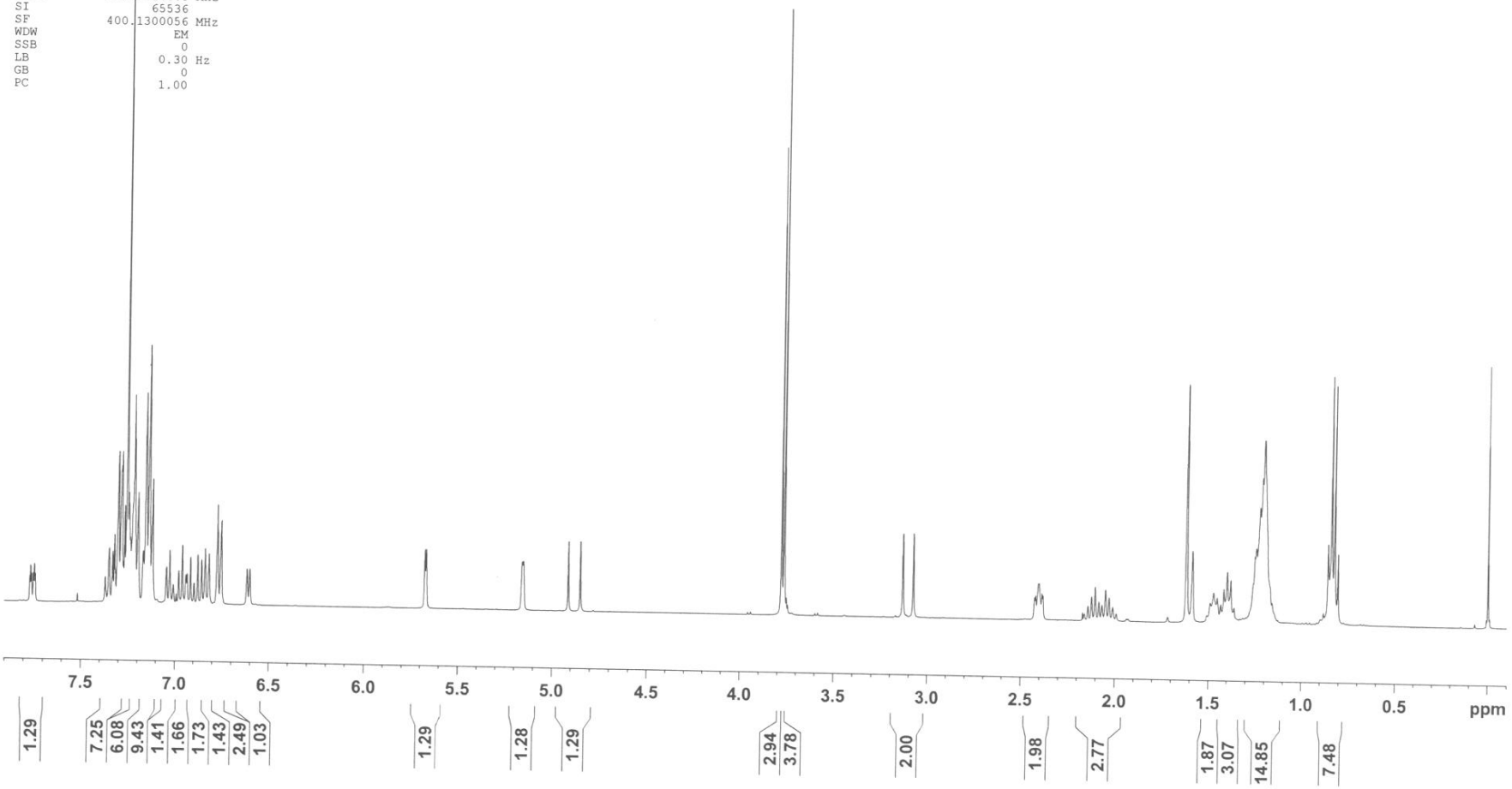
NAME	ccw_1_123_15 rp t15
EXPNO	6
PROCNO	1
Date_	20080910
Time	9.53
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	28
SFO1	400.1318 MHz
FIDRES	145.891693 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0


NAME ccw_2'f
EXPNO 1
PROCNO 1
Date_ 20100424
Time 15.29
INSTRUM spect
PROBND 5 mm BBUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 220
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 295.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300036 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
MeO
Ph
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
ppm
2.16
2.31
7.27
3.27
2.45
2.22
2.09
1.00
1.02
1.04
3.11
2.16
2.08

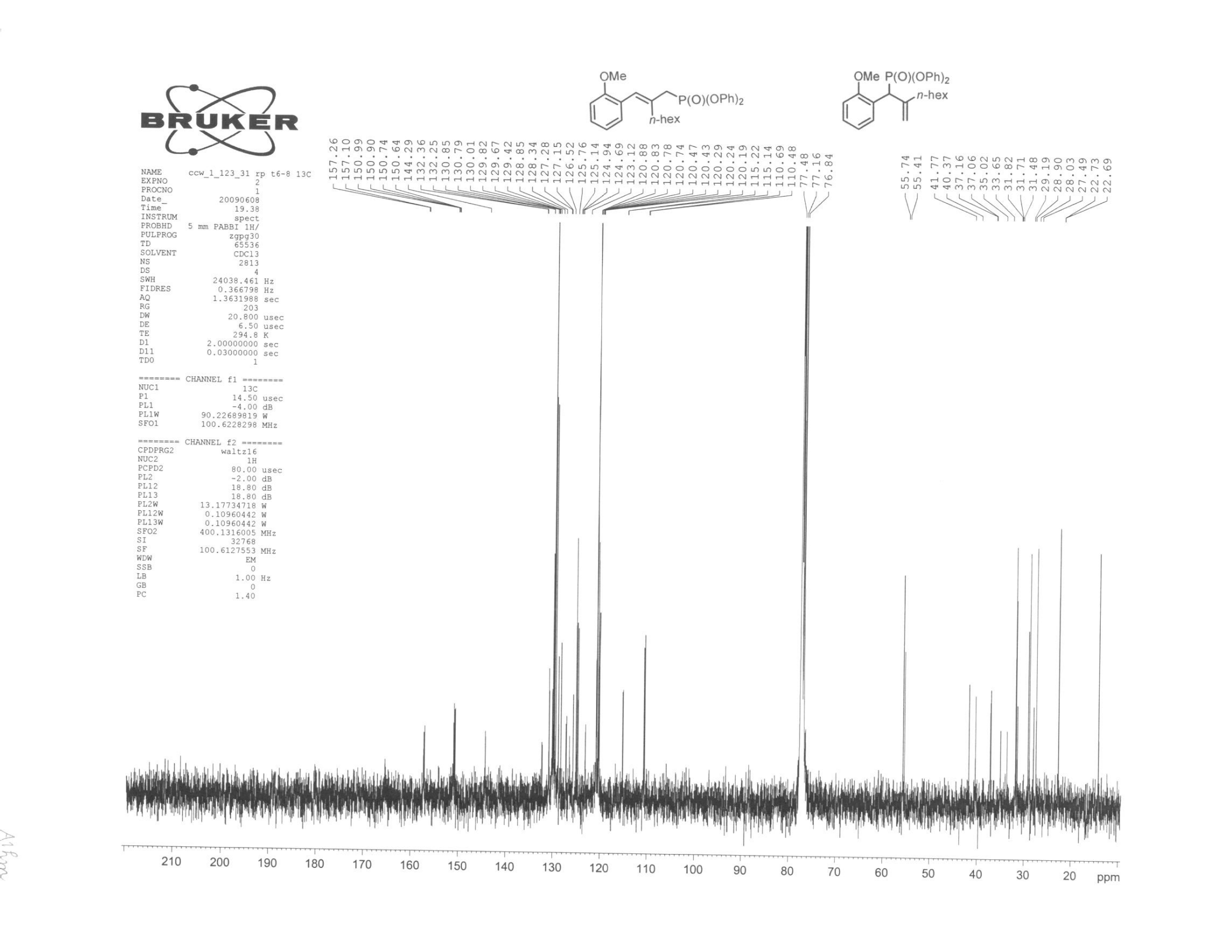
```
NAME        ccw_1_123_31 rp t6-8
EXPNO                  1
PROCNO                 1
Date_           20090423
Time              19.37
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    48
DS                     0
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ            3.27768500 sec
RG                   203
DW                50.000 usec
DE                 6.50 usec
TE                294.7 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.10 usec
PL1               -2.00 dB
PL1W        13.17734718 W
SFO1        400.1316005 MHz
SI               65536
SF          400.1300056 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

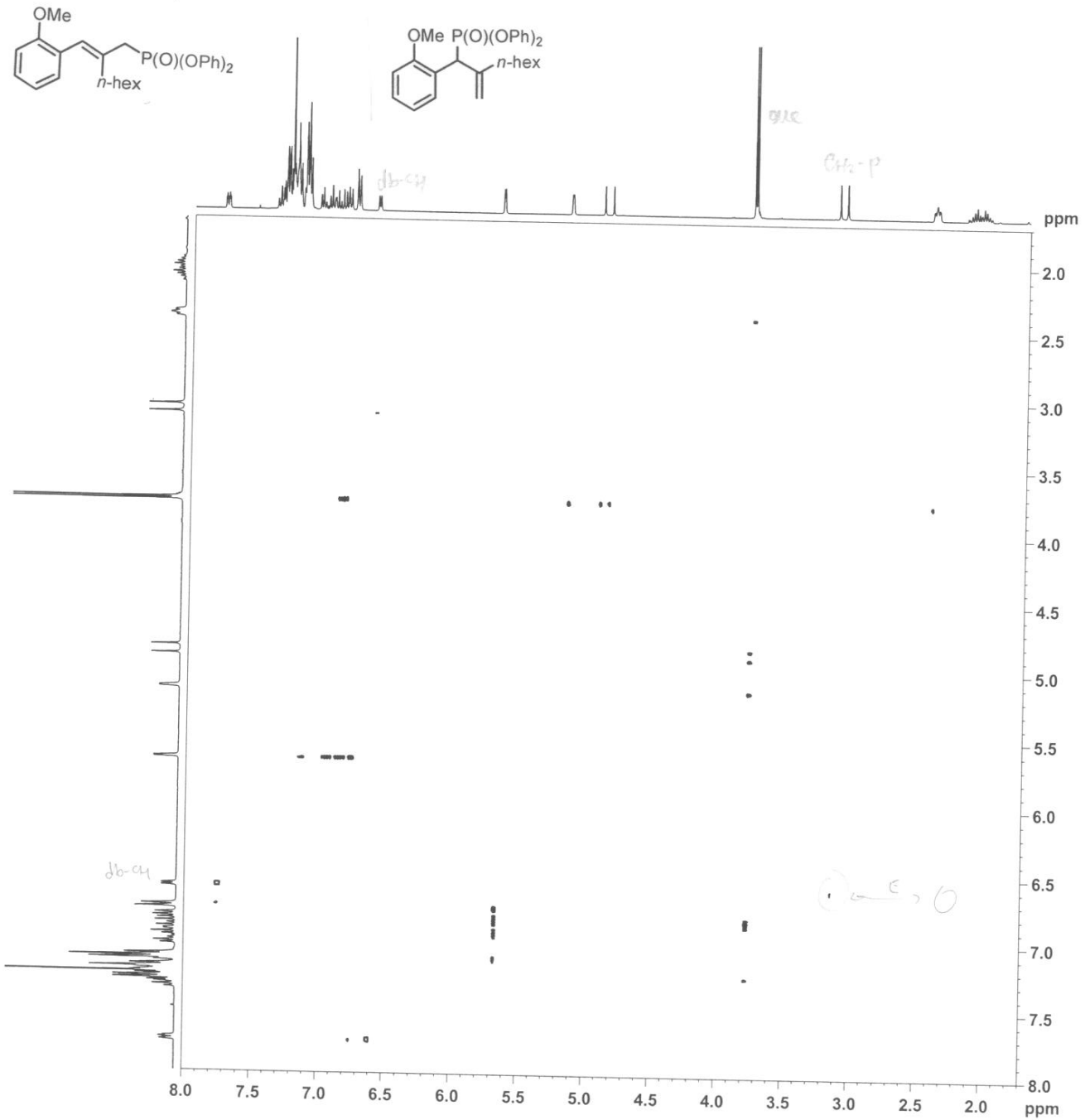






A1f1+A1f2

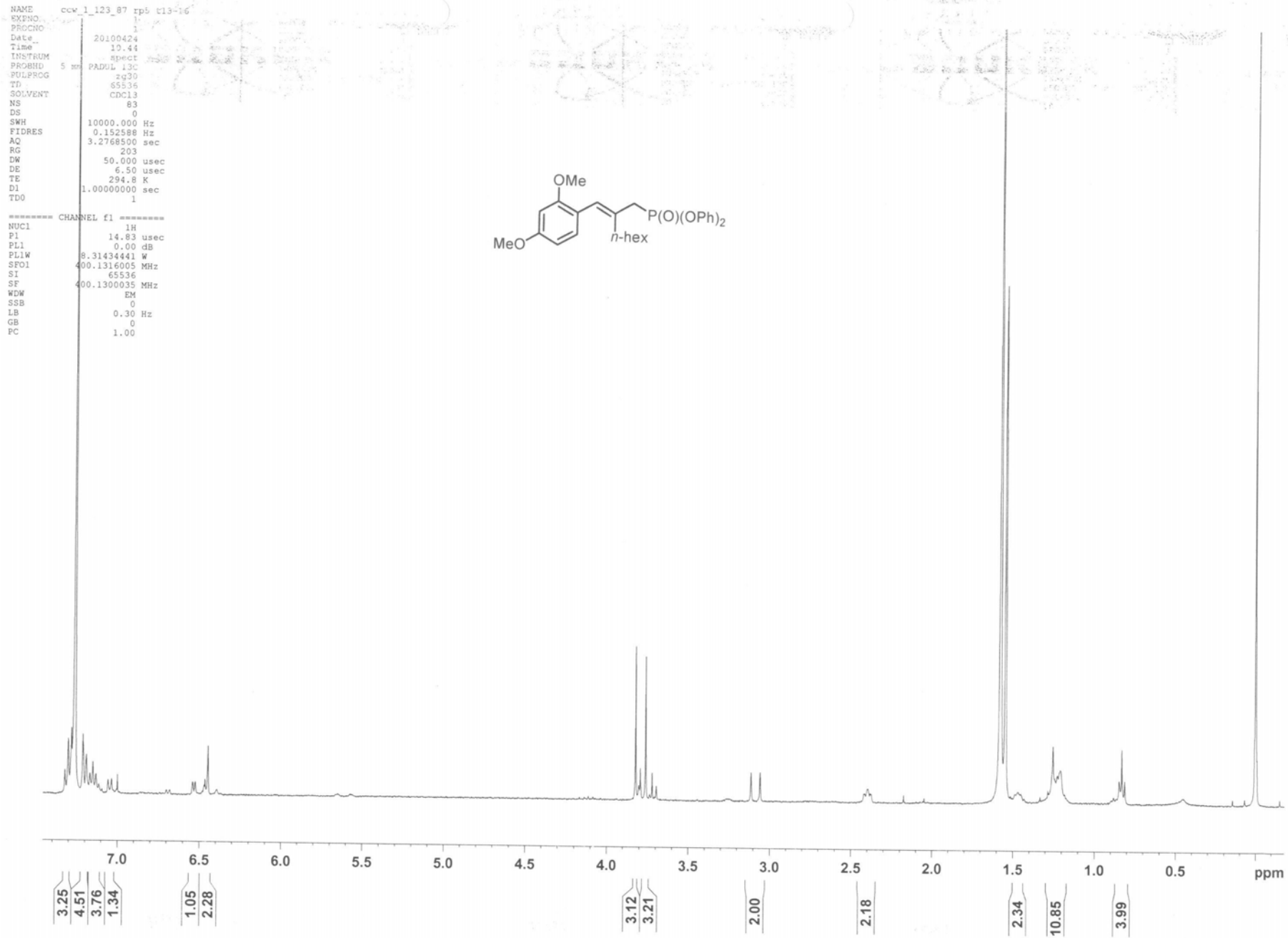

OMe
P(O)(OPh)2
n-hex
OMe P(O)(OPh)2
n-hex
157.26
157.10
150.99
150.90
150.74
150.64
144.29
132.36
132.25
130.85
130.79
130.01
129.82
129.67
129.42
128.85
128.34
127.28
127.15
126.52
125.76
125.14
124.94
124.69
123.12
120.88
120.83
120.78
120.74
120.47
120.43
120.29
120.24
120.19
115.22
115.14
110.69
110.48
77.48
77.16
76.84
55.74
55.41
41.77
40.37
37.16
37.06
35.02
33.65
31.82
31.71
31.48
29.19
28.90
28.03
27.49
22.73
22.69
NAME ccw_1_123_31 rp t6-8 13C
EXPNO 2
PROCNO 1
Date_ 20090608
Time 19.38
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 2813
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 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

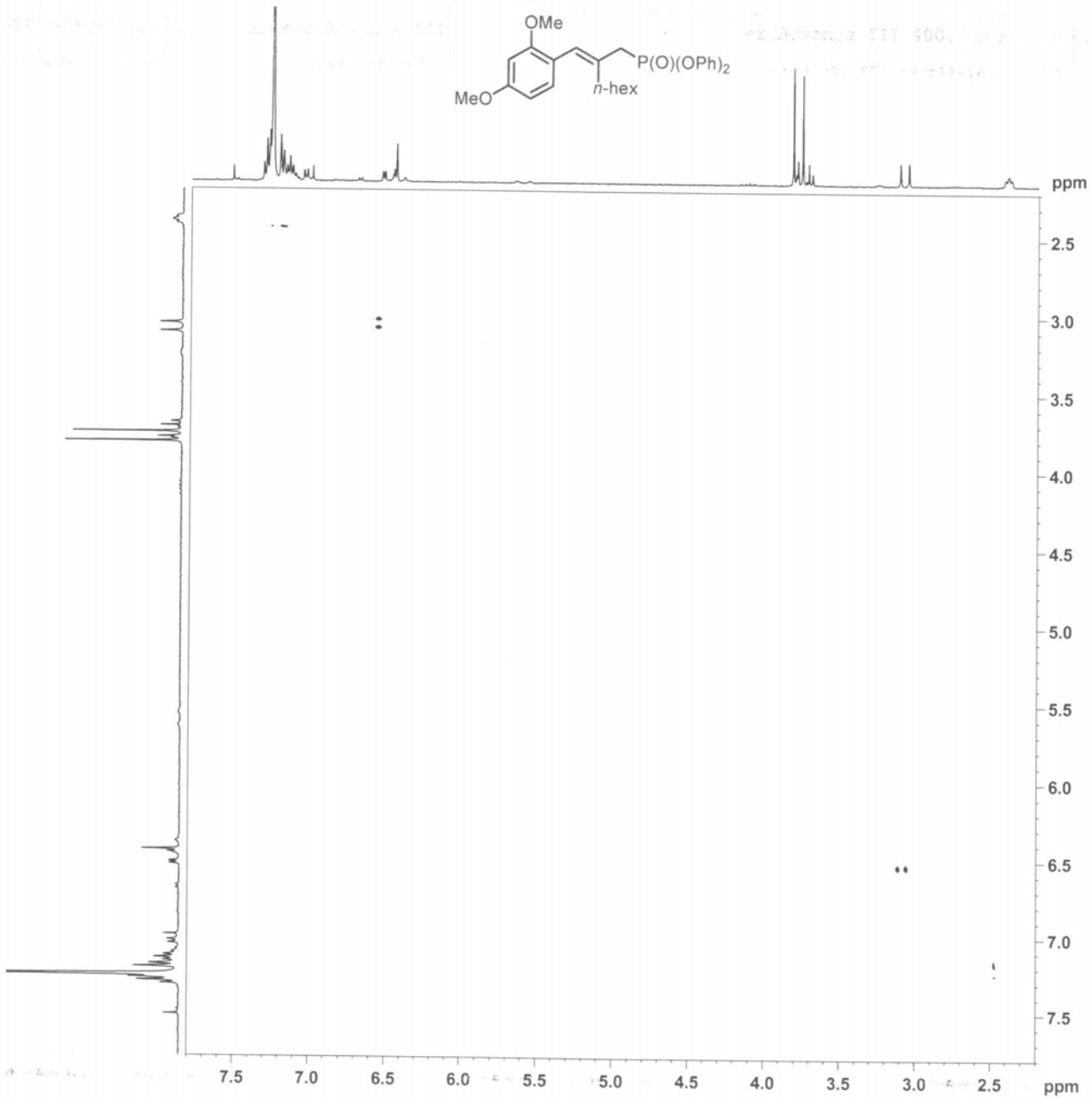


BRUKER

NAME	ccw_1_123_31 rp t6-8
EXPNO	6
PROCNO	1
Date_	20090423
Time	19.39
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

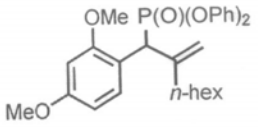
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	18
SFO1	400.1318 MHz
FIDRES	226.942627 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0


NAME ccw_1_123_87 rpb t13-16
EXPNO 1
PROCNO 1
Date_ 20100424
Time 10.44
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 83
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300035 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OMe
MeO
P(O)(OPh)2
n-hex
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
3.25
4.51
3.76
1.34
1.05
2.28
3.12
3.21
2.00
2.18
2.34
10.85
3.99



Bruker Advance III 400

NAME	ccw_1_123_87 rp9 t13-16
EXPNO	6
PROCNO	1
Date_	20100424
Time	16.05
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	52
SFO1	400.1318 MHz
FIDRES	255.310455 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300127 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300327 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0



ppm

7.664 7.657 7.636 7.629 7.319 7.292 7.262 7.162 7.136 7.061 7.037 6.813 6.787 6.517 6.511 6.483 6.400 5.645 5.638 5.130 4.796 4.711 3.799 3.736 2.111 2.086 2.061 2.047 2.017 1.636 1.607 1.419 1.396 1.373 1.349 1.259 1.212 0.899 0.882 0.847 0.824 0.000

water

Integral

1.0558 2.8896 4.7800 0.9400 1.8649 1.0445 1.0182 1.0562 1.0664 1.0000 3.2823 3.3295 2.5812 1.8794 2.4635 9.5038 5.4484

ppm 9 8 7 6 5 4 3 2 1 0

Current Data Parameters
NAME ccw_1_123_86 rp3 t14-15
EXPNO 1
PROCNO 1

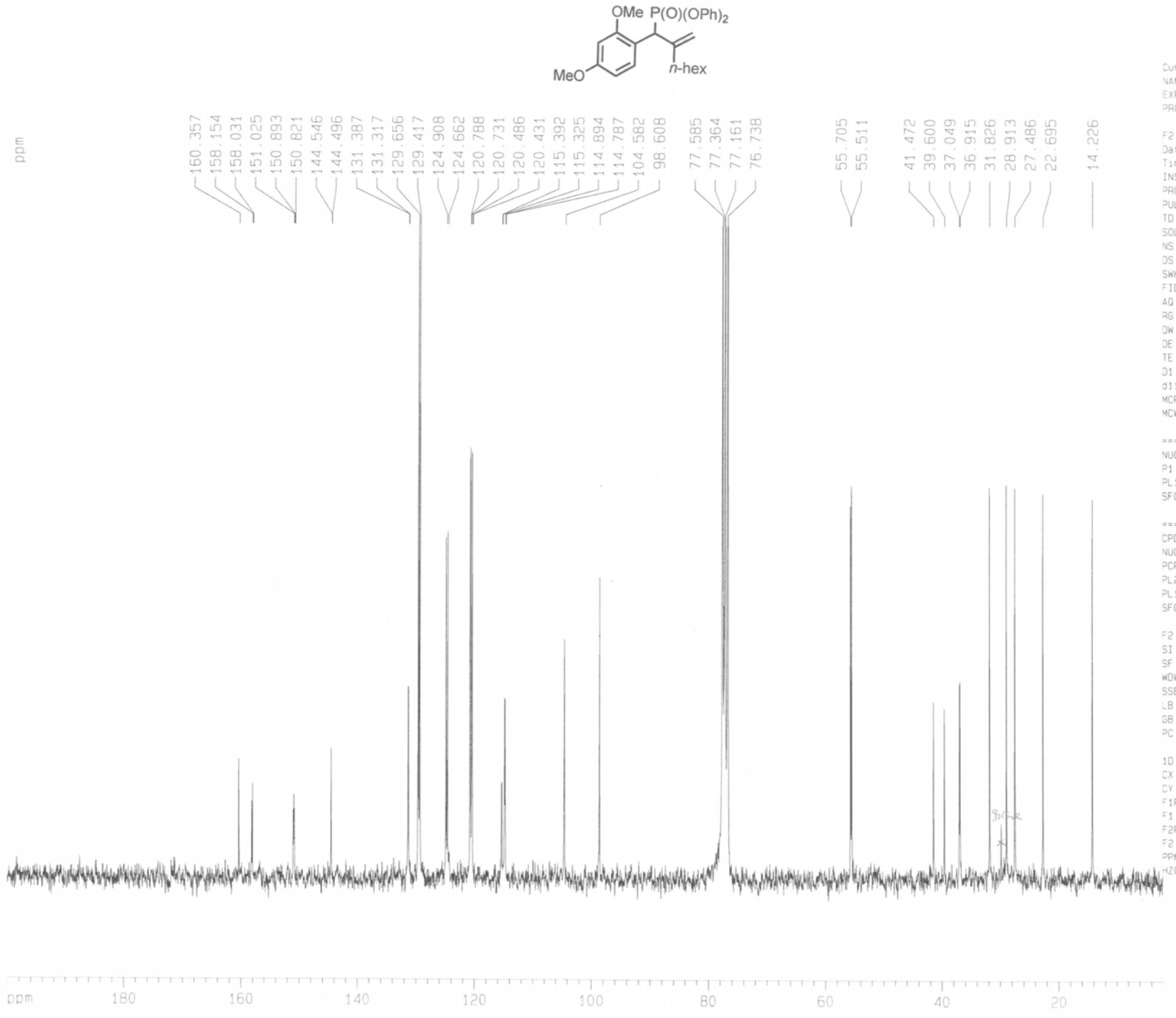
F2 - Acquisition Parameters
Date_ 20090824
Time 19.36
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 29
DS 0
SWH 8992.806 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 203.2
DW 55.600 usec
DE 79.43 usec
TE 295.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 -2.00 dB
SF01 300.1312000 MHz

F2 - Processing parameters
SI 32768
SF 300.1300059 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 22.00 cm
CY 10.00 cm
F1P 10.500 ppm
F1 3151.36 Hz
F2P -0.500 ppm
F2 -150.07 Hz
PPMCM 0.50000 ppm/cm
HZCM 150.06500 Hz/cm

A102



Current Data Parameters
NAME ccw_1_123_87 rp3 t14-15
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090829
Time 18.04
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 5171
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 8192
DW 22.050 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SF01 75.4745111 MHz

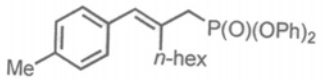
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz

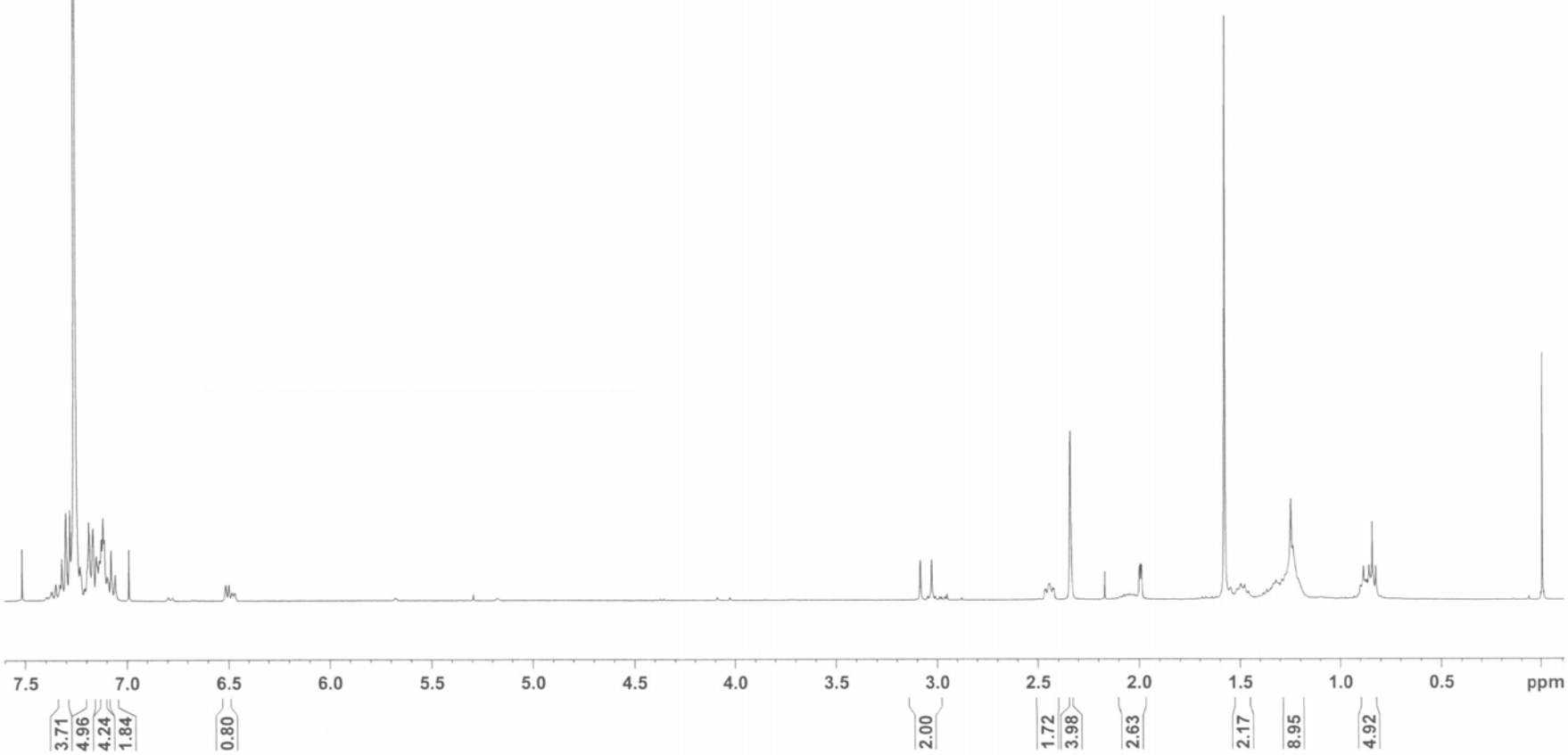
F2 - Processing parameters
SI 65536
SF 75.4677400 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 50.00 cm
F1P 200.000 ppm
F1 15093.55 Hz
F2P 2.000 ppm
F2 150.94 Hz
PPMCM 8.60870 ppm/cm
HZCM 649.67889 Hz/cm

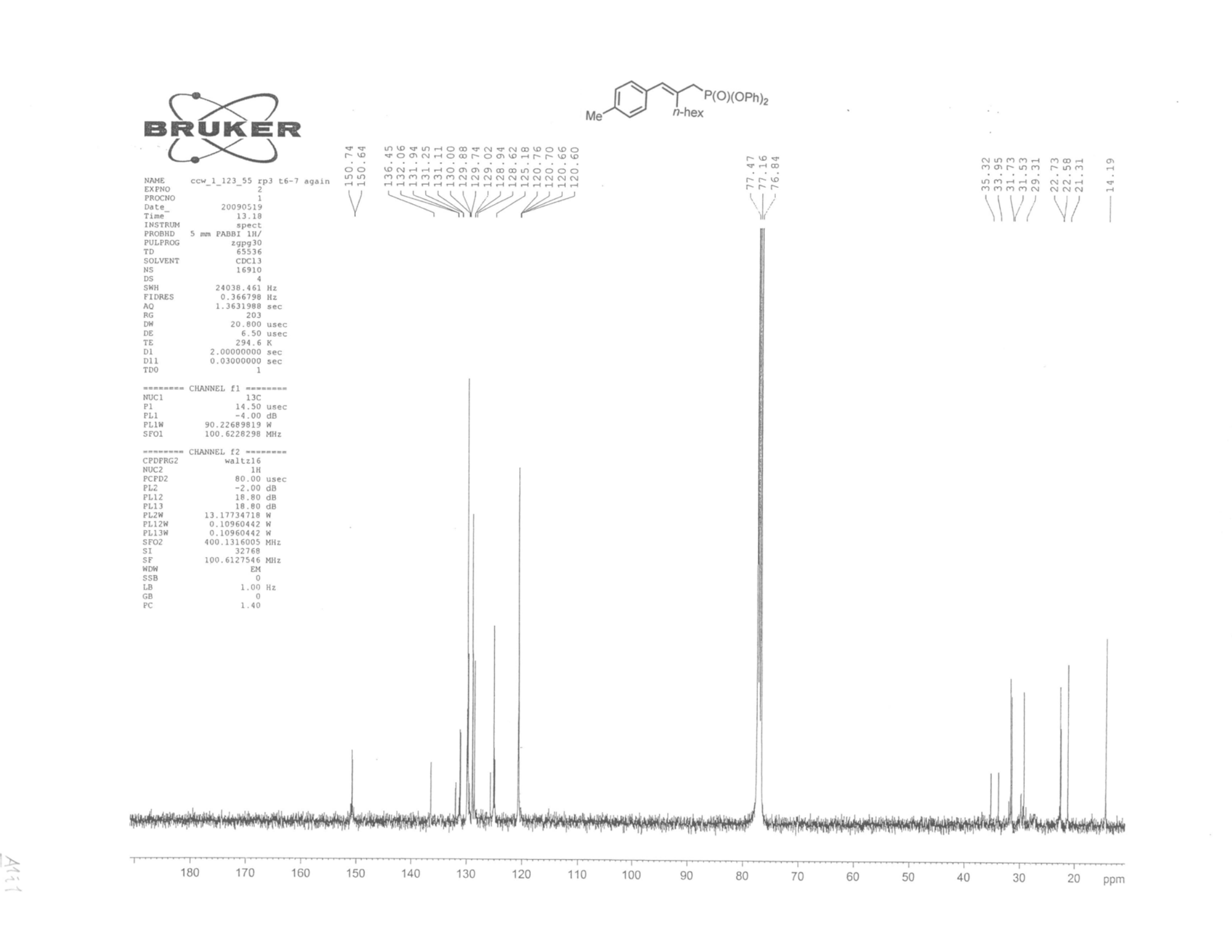
A102

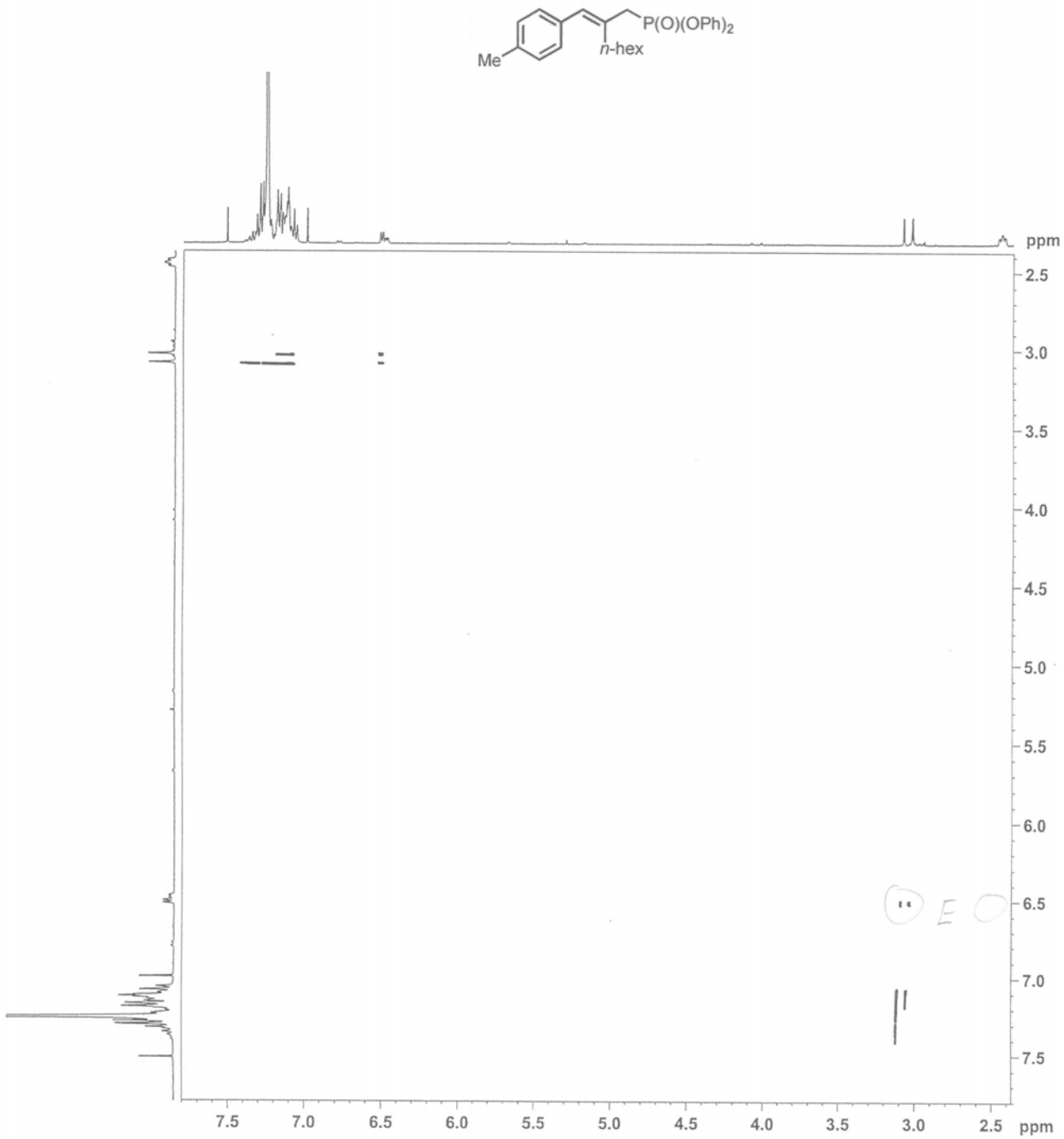
```
NAME      ccw_1_123_55 rp3 t6-7 again
EXPNO                 1
PROCNO                1
Date_          20090514
Time              19.29
INSTRUM           spect
PROBHD    5 mm PABBI 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  23
DS                   0
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2768500 sec
RG                 144
DW                50.000 usec
DE                 6.50 usec
TE                294.4 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.10 usec
PL1               -2.00 dB
PL1W          13.17734718 W
SFO1          400.1316005 MHz
SI                 65536
SF            400.1300070 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```





Ar21

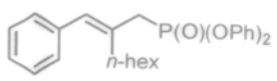

BRUKER
Me
n-hex
P(O)(OPh)2
NAME ccw_1_123_55 rp3 t6-7 again
EXPNO 2
PROCNO 1
Date_ 20090519
Time 13.18
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16910
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150.74
150.64
136.45
132.06
131.94
131.25
131.11
130.00
129.88
129.74
129.02
128.94
128.62
125.18
120.76
120.70
120.66
120.60
77.47
77.16
76.84
35.32
33.95
31.73
31.53
29.31
22.73
22.58
21.31
14.19
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

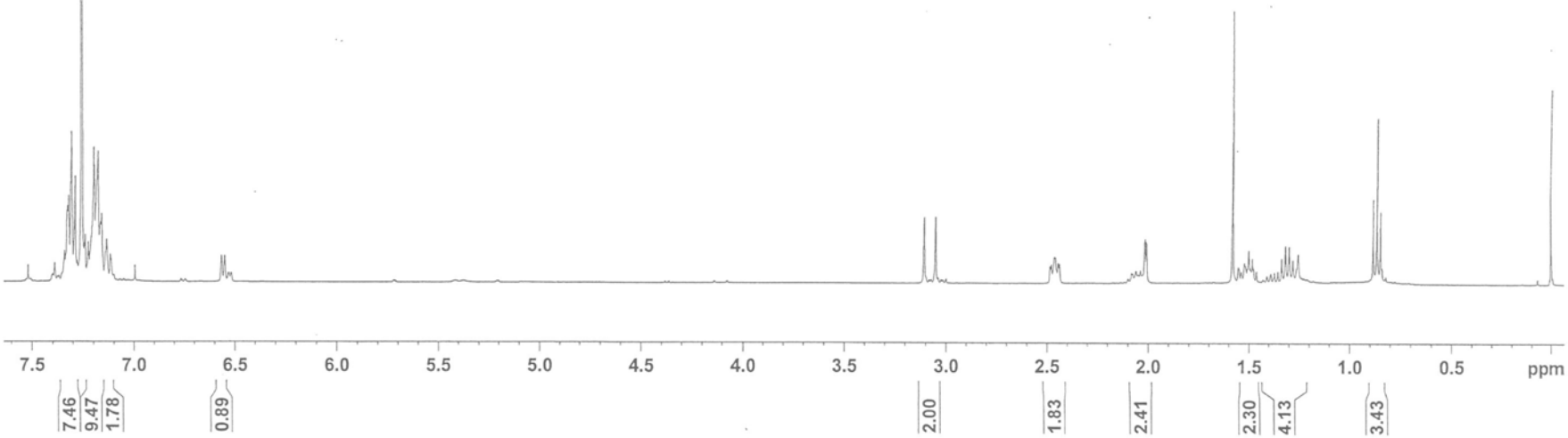


Bruker Advance III 400

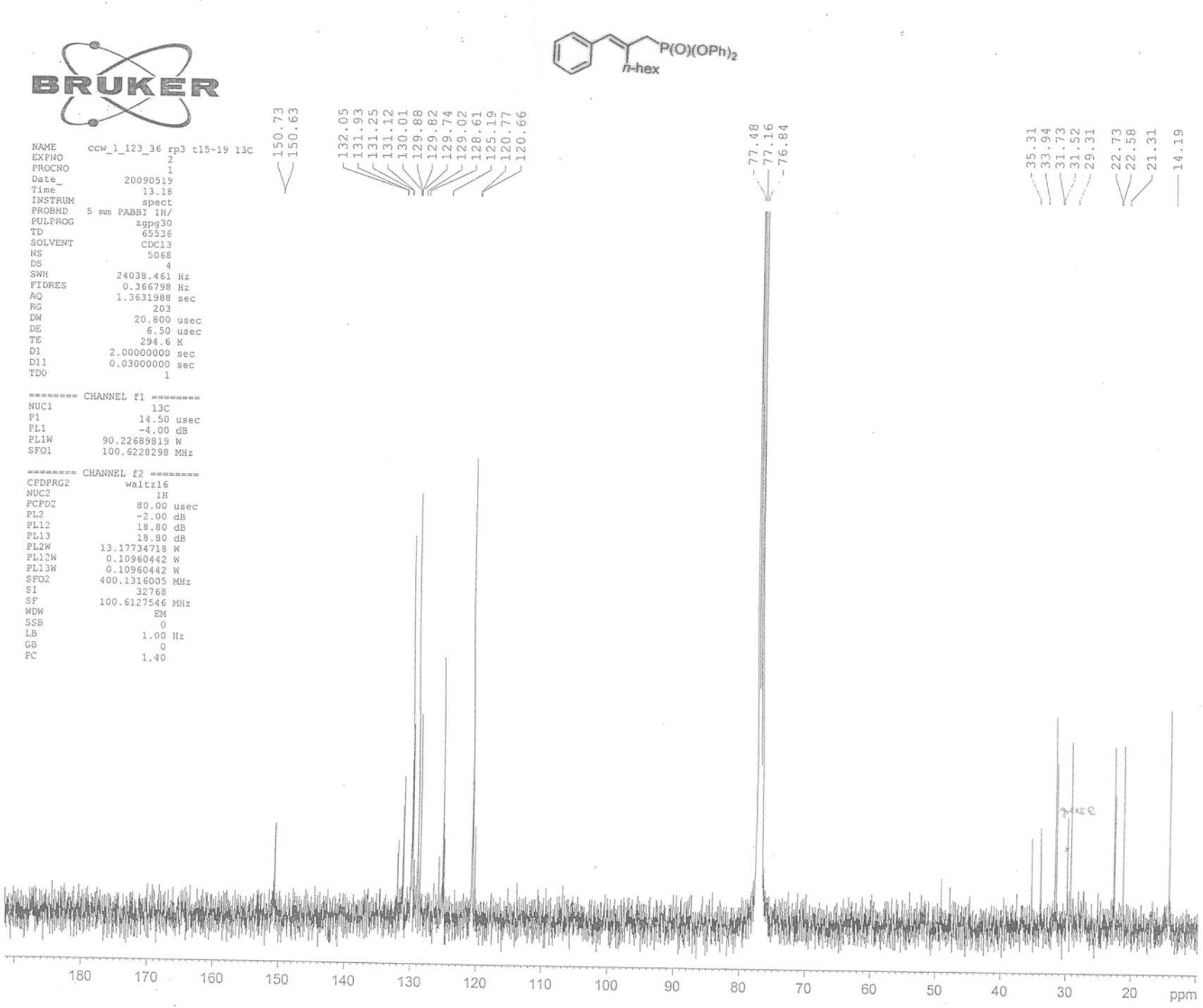
NAME	ccw_1_123_55 rp t9-14
EXPNO	6
PROCNO	1
Date_	20090424
Time	16.53
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	6
SFO1	400.1318 MHz
FIDRES	680.827881 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

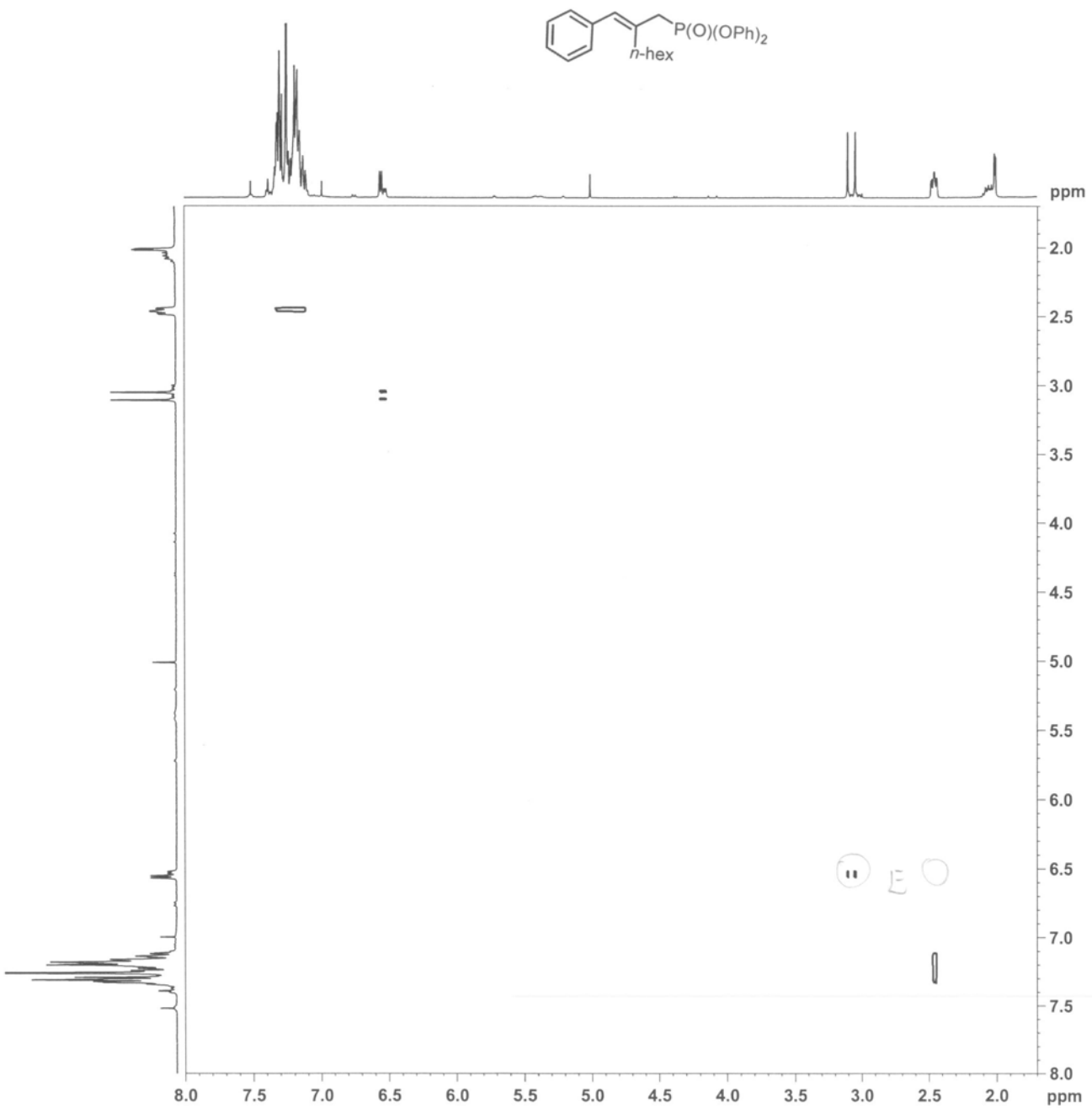
```
NAME       ccw_1_123_36 rp3 t15-19
EXPNO                 1
PROCNO                1
Date_          20090507
Time              14.18
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  15
DS                   0
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ           3.2768500 sec
RG                 144
DW              50.000 usec
DE               6.50 usec
TE              294.6 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                7.10 usec
PL1              -2.00 dB
PL1W       13.17734718 W
SFO1       400.1316005 MHz
SI               65536
SF         400.1300056 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```





A1R1

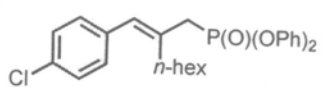

BRUKER
P(O)(OPh)2
n-hex
NAME ccw_1_123_36 rp3 t15-19 13C
EXPNO 2
PROCNO 1
Date_ 20090519
Time 13.18
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5068
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
150.73
150.63
132.05
131.93
131.25
131.12
130.01
129.88
129.82
129.74
129.02
128.61
125.19
120.77
120.66
77.48
77.16
76.84
35.31
33.94
31.73
31.52
29.31
22.73
22.58
21.31
14.19
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

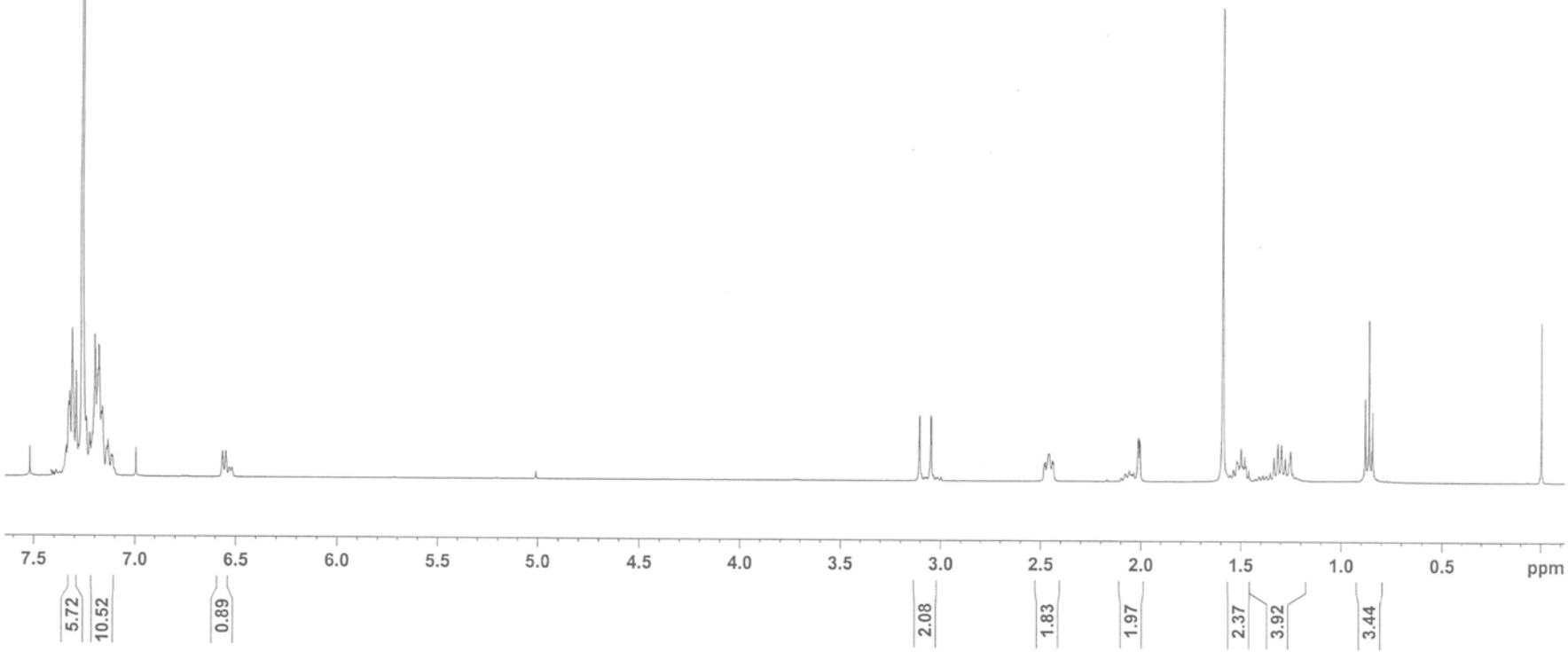


BRUKER

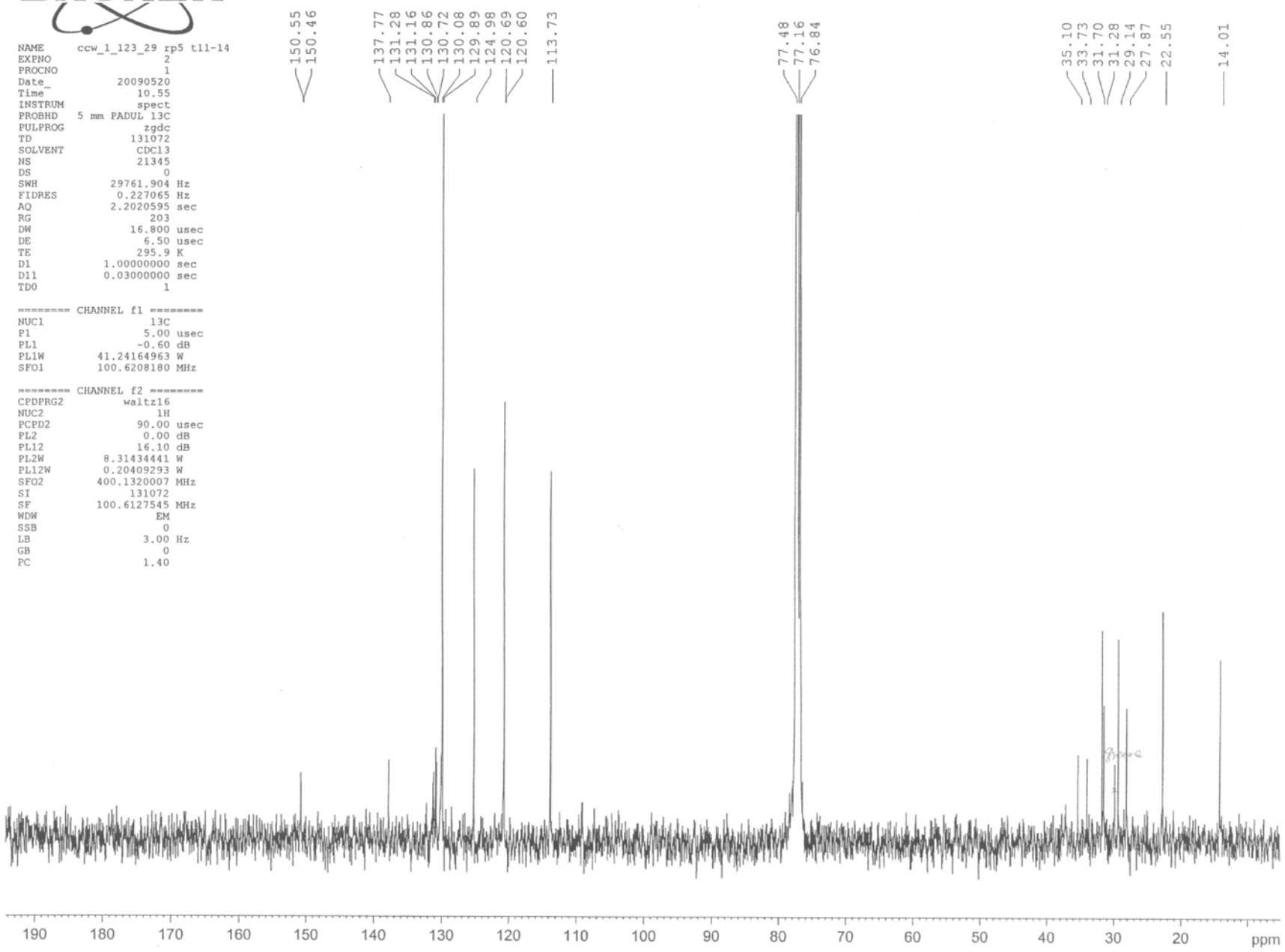
NAME	ccw_1_123_36 rp3 t15-19
EXPNO	6
PROCNO	1
Date_	20090511
Time	14.36
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.8 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	12
SFO1	400.1318 MHz
FIDRES	340.413940 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

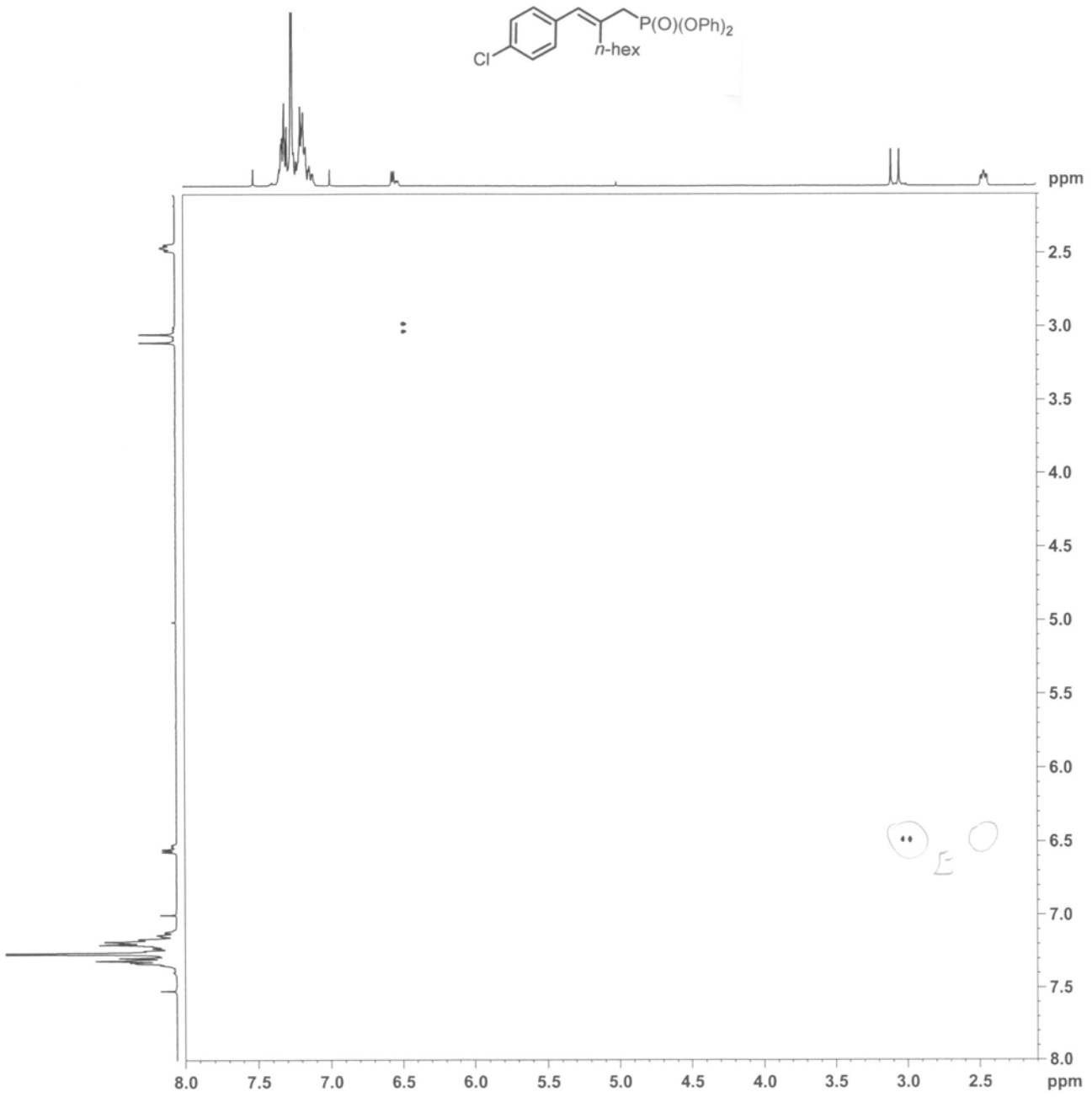
```
NAME      ccw_1_123_29 rp5 t11-14
EXPNO                1
PROCNO               1
Date_         20090511
Time             14.19
INSTRUM          spect
PROBHD    5 mm PABBI 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  27
DS                   0
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ           3.2768500 sec
RG                 203
DW              50.000 usec
DE               6.50 usec
TE              294.7 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                7.10 usec
PL1              -2.00 dB
PL1W       13.17734718 W
SFO1       400.1316005 MHz
SI               65536
SF         400.1300051 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```






```
NAME      ccw_1_123_29 rp5 t11-14
EXPNO                2
PROCNO               1
Date_         20090520
Time             10.55
INSTRUM          spect
PROBHD   5 mm PADUL 13C
PULPROG           zgdc
TD               131072
SOLVENT           CDCl3
NS                21345
DS                   0
SWH           29761.904 Hz
FIDRES         0.227065 Hz
AQ             2.2020595 sec
RG                 203
DW              16.800 usec
DE               6.50 usec
TE              295.9 K
D1           1.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                5.00 usec
PL1              -0.60 dB
PL1W       41.24164963 W
SFO1       100.6208180 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2               1H
PCPD2            90.00 usec
PL2               0.00 dB
PL12             16.10 dB
PL2W        8.31434441 W
PL12W       0.20409293 W
SFO2       400.1320007 MHz
SI              131072
SF         100.6127545 MHz
WDW                EM
SSB                 0
LB               3.00 Hz
GB                  0
PC               1.40
```

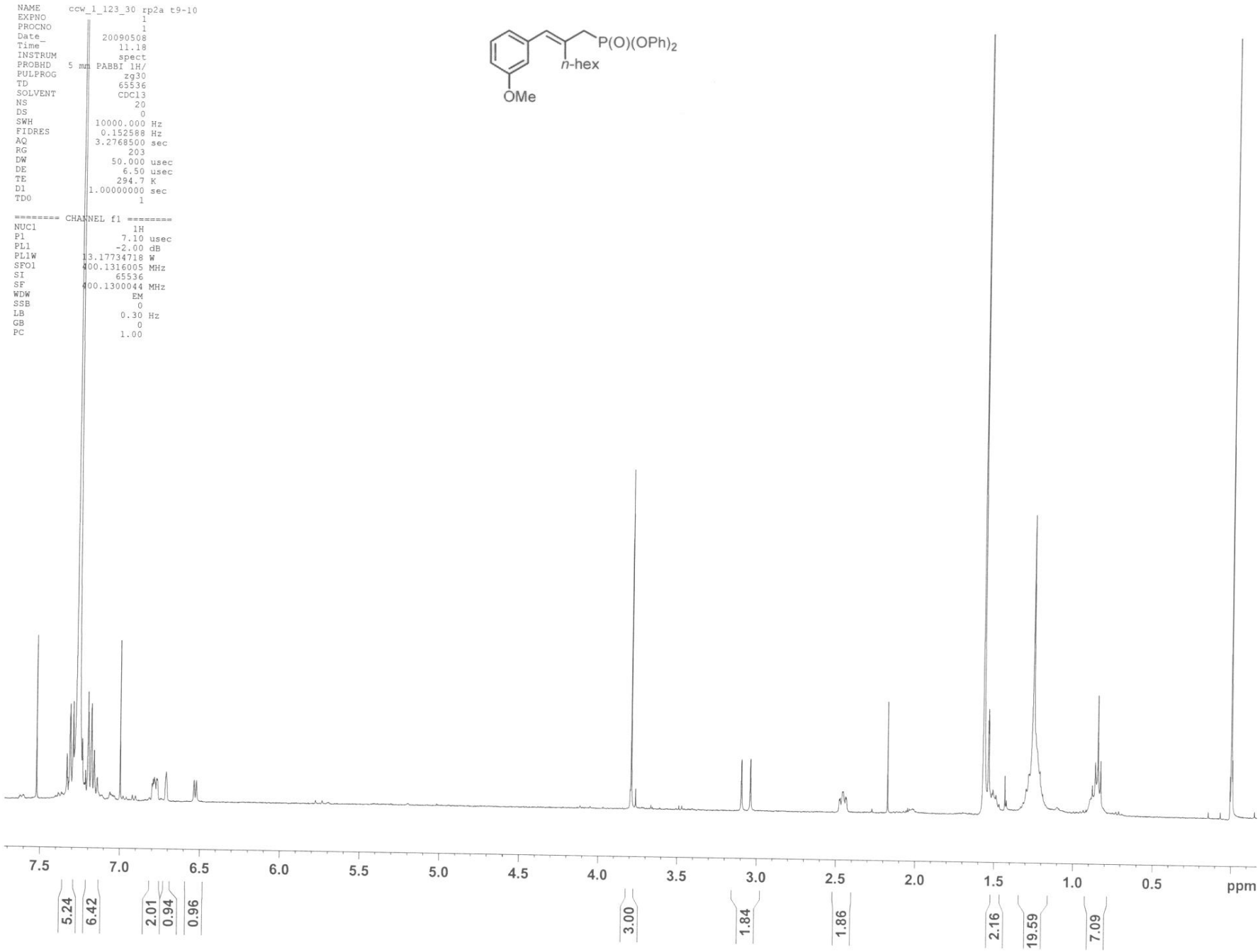


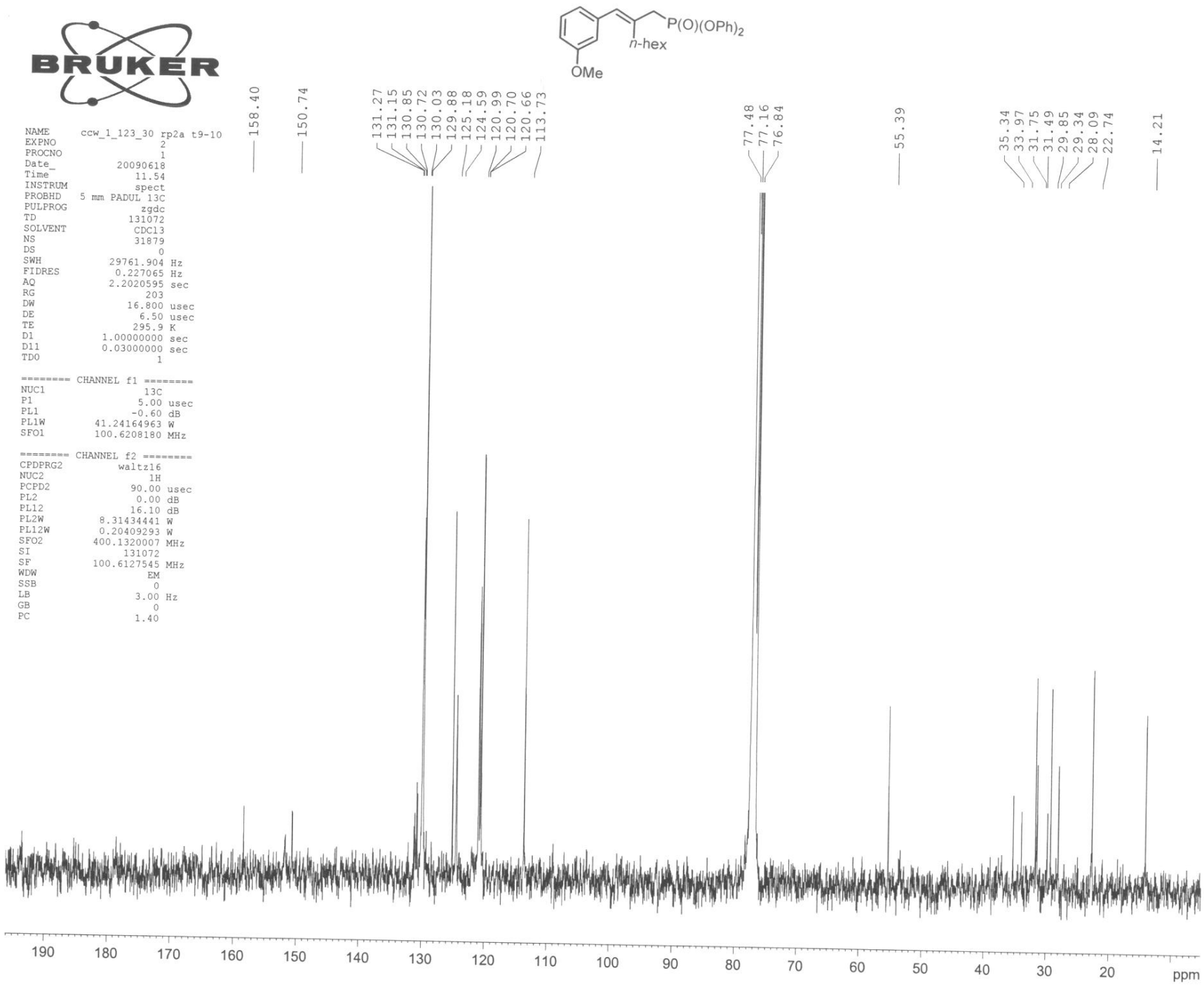


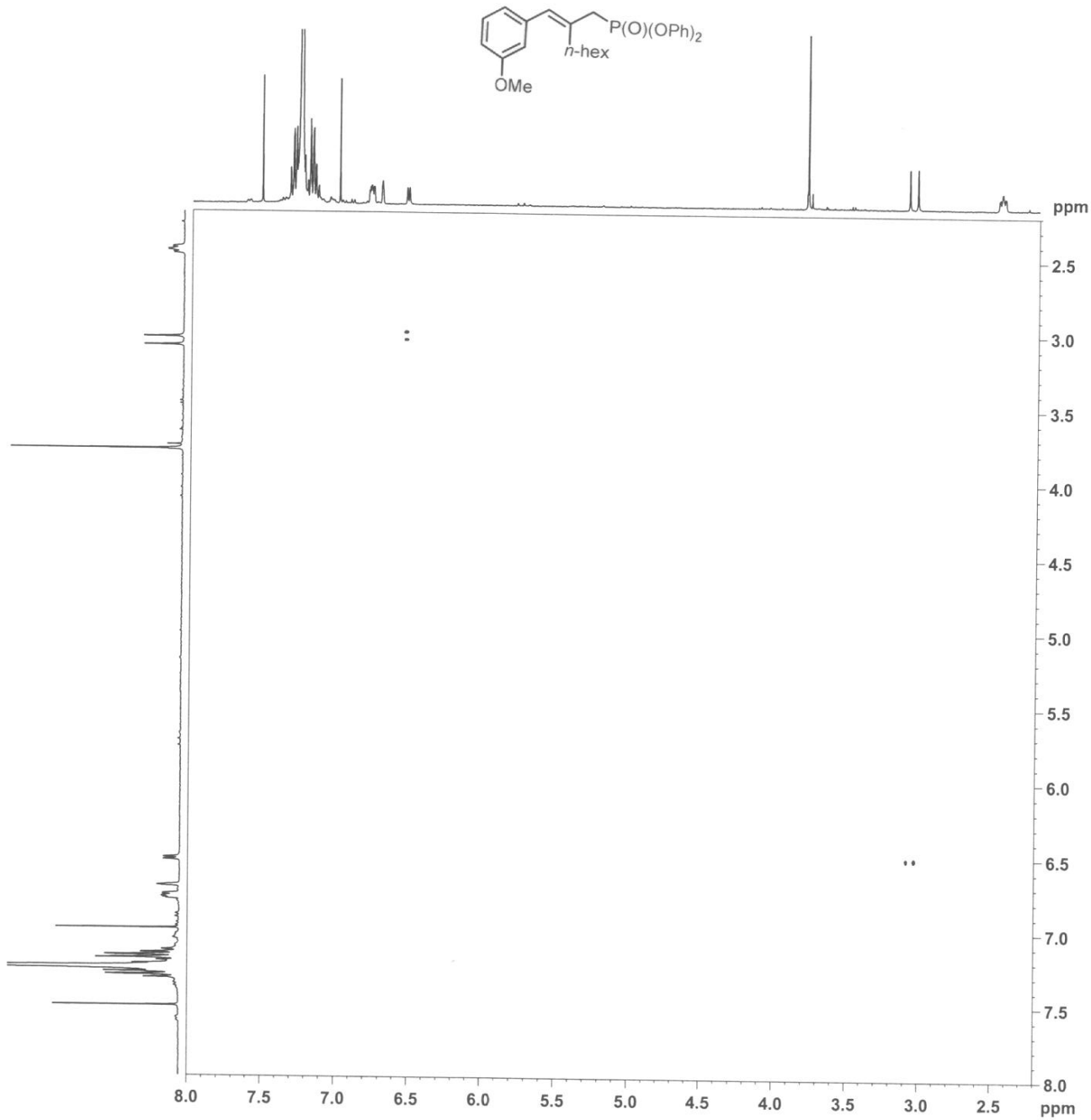
BRUKER

NAME	ccw_1_123_29 rp5 t11-14
EXPNO	6
PROCNO	1
Date_	20090504
Time	14.13
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	16
SFO1	400.1318 MHz
FIDRES	255.310455 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300327 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300327 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

A1j1


NAME ccw_1_123_30 rp2a t9-10
EXPNO 1
PROCNO 1
Date_ 20090508
Time 11.18
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 20
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.10 usec
PL1 -2.00 dB
PL1W 13.17734718 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300044 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
n-hex
OMe
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
5.24
6.42
2.01
0.94
0.96
3.00
1.84
1.86
2.16
19.59
7.09


BRUKER
P(O)(OPh)2
n-hex
OMe
158.40
150.74
131.27
131.15
130.85
130.72
130.03
129.88
125.18
124.59
120.99
120.70
120.66
113.73
77.48
77.16
76.84
55.39
35.34
33.97
31.75
31.49
29.85
29.34
28.09
22.74
14.21
NAME ccw_1_123_30 rp2a t9-10
EXPNO 2
PROCNO 1
Date_ 20090618
Time 11.54
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 31879
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 5.00 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 16.10 dB
PL2W 8.31434441 W
PL12W 0.20409293 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127545 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

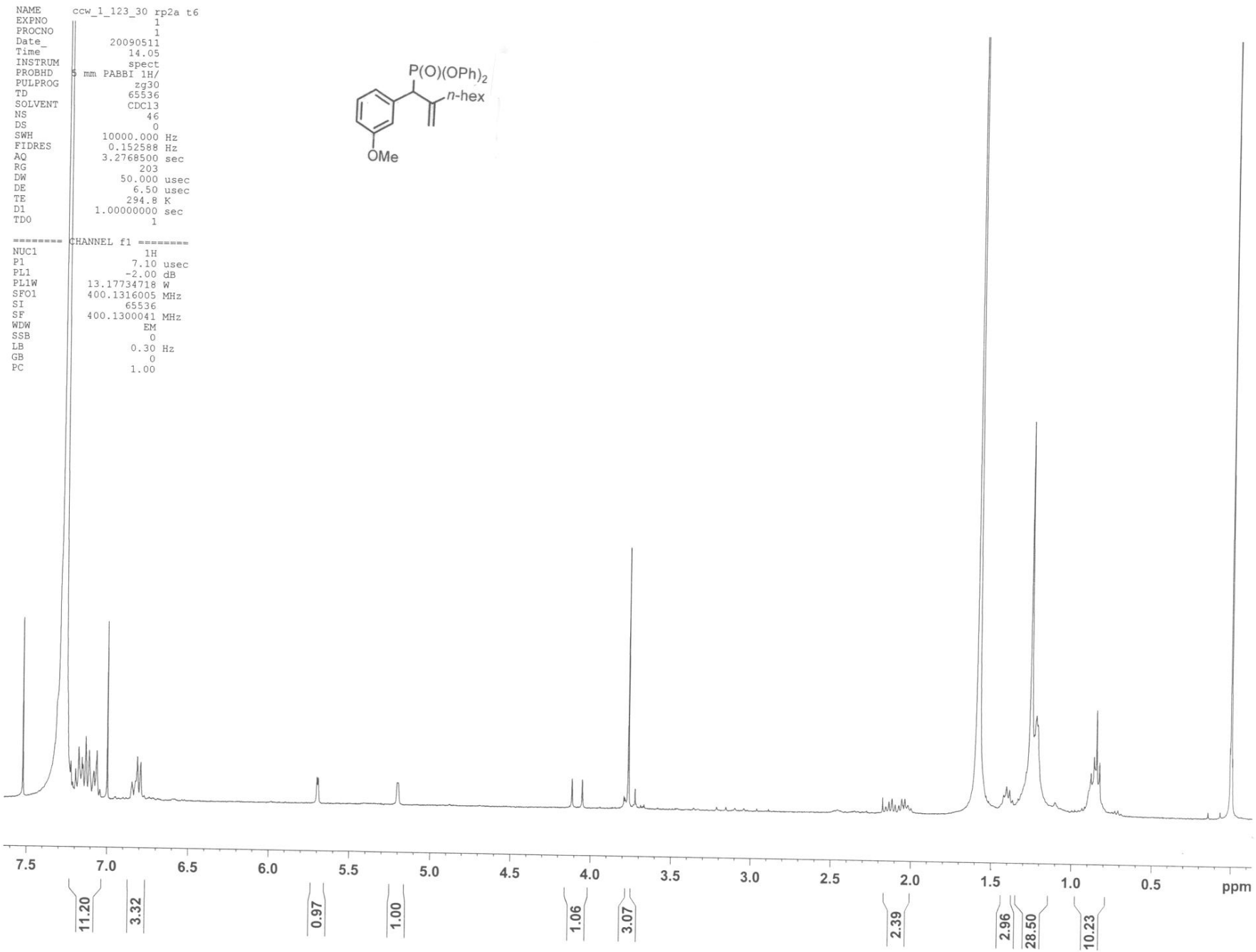


BRUKER

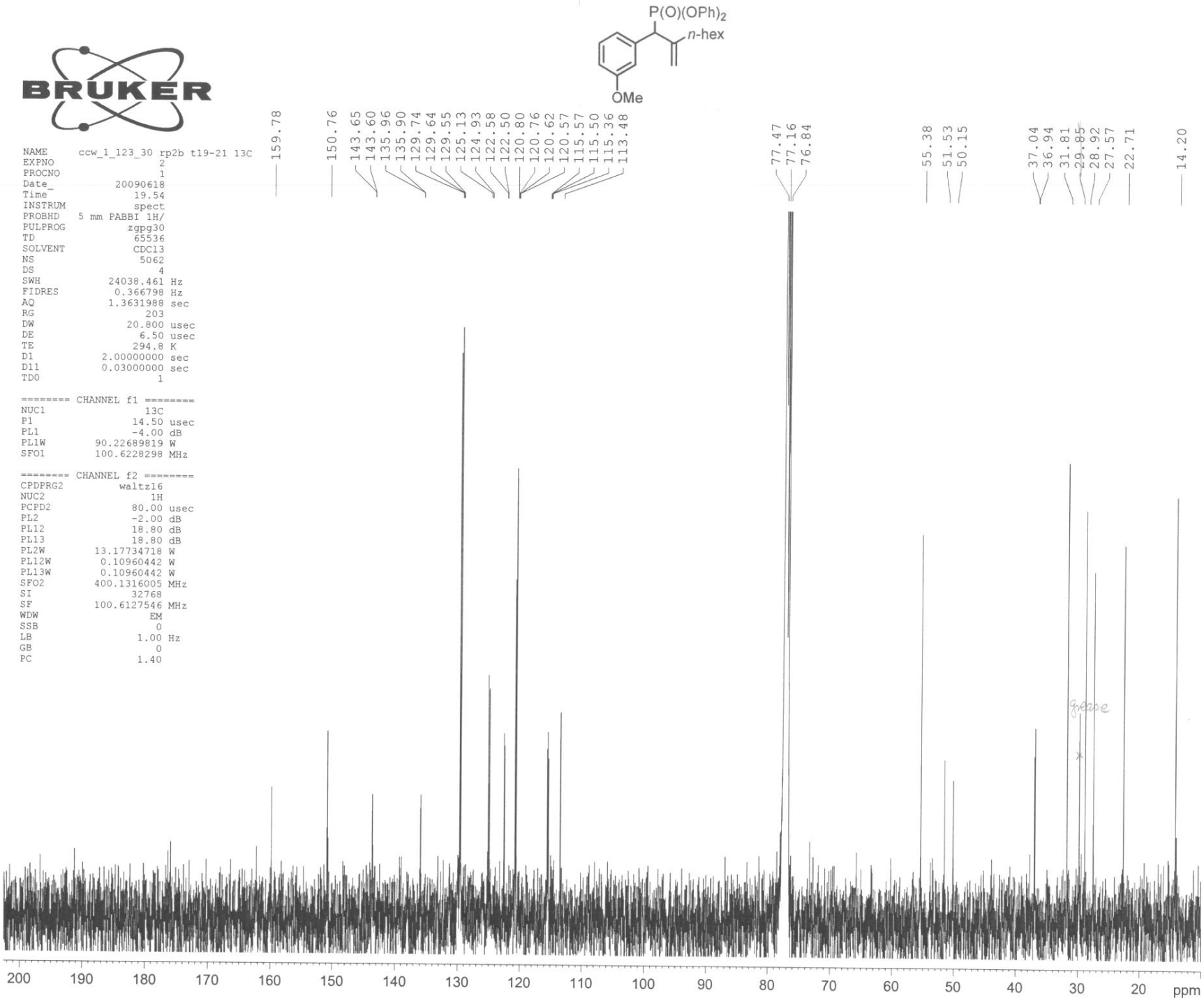
NAME	ccw_1_123_30 rp2a t9-10
EXPNO	6
PROCNO	1
Date_	20090508
Time	12.33
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	36
SFO1	400.1318 MHz
FIDRES	255.310455 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300127 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300247 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

A191


NAME ccw_1_123_30 rp2a t6
EXPNO 1
PROCNO 1
Date_ 20090511
Time 14.05
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 46
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 7.10 usec
PL1 -2.00 dB
PL1W 13.17734718 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
n-hex
OMe
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
11.20
3.32
0.97
1.00
1.06
3.07
2.39
2.96
28.50
10.23

A192



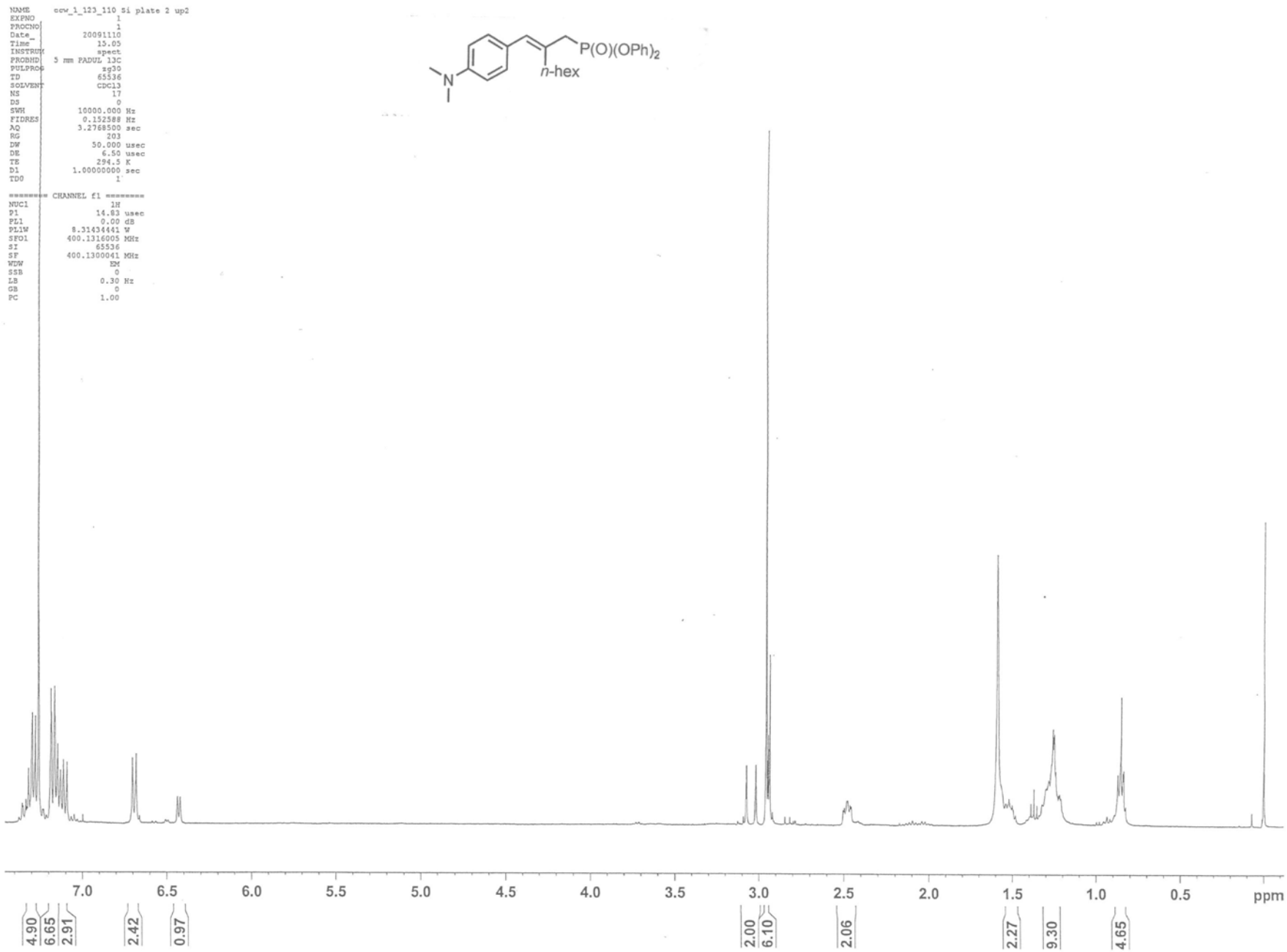
NAME ccw_1_123_30 rp2b t19-21 13C
EXPNO 2
PROCNO 1
Date_ 20090618
Time 19.54
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 5062
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz

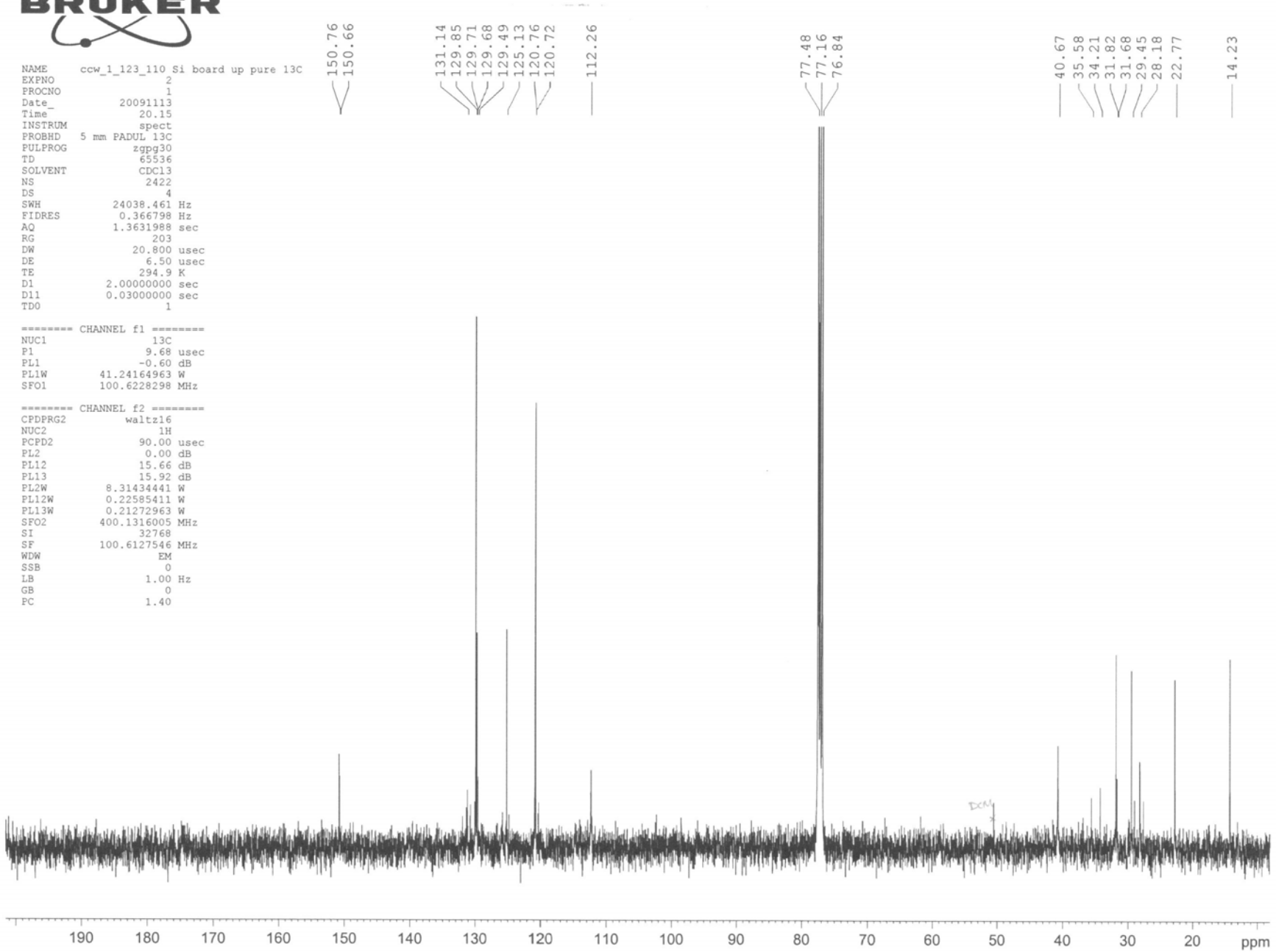
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

gezoe

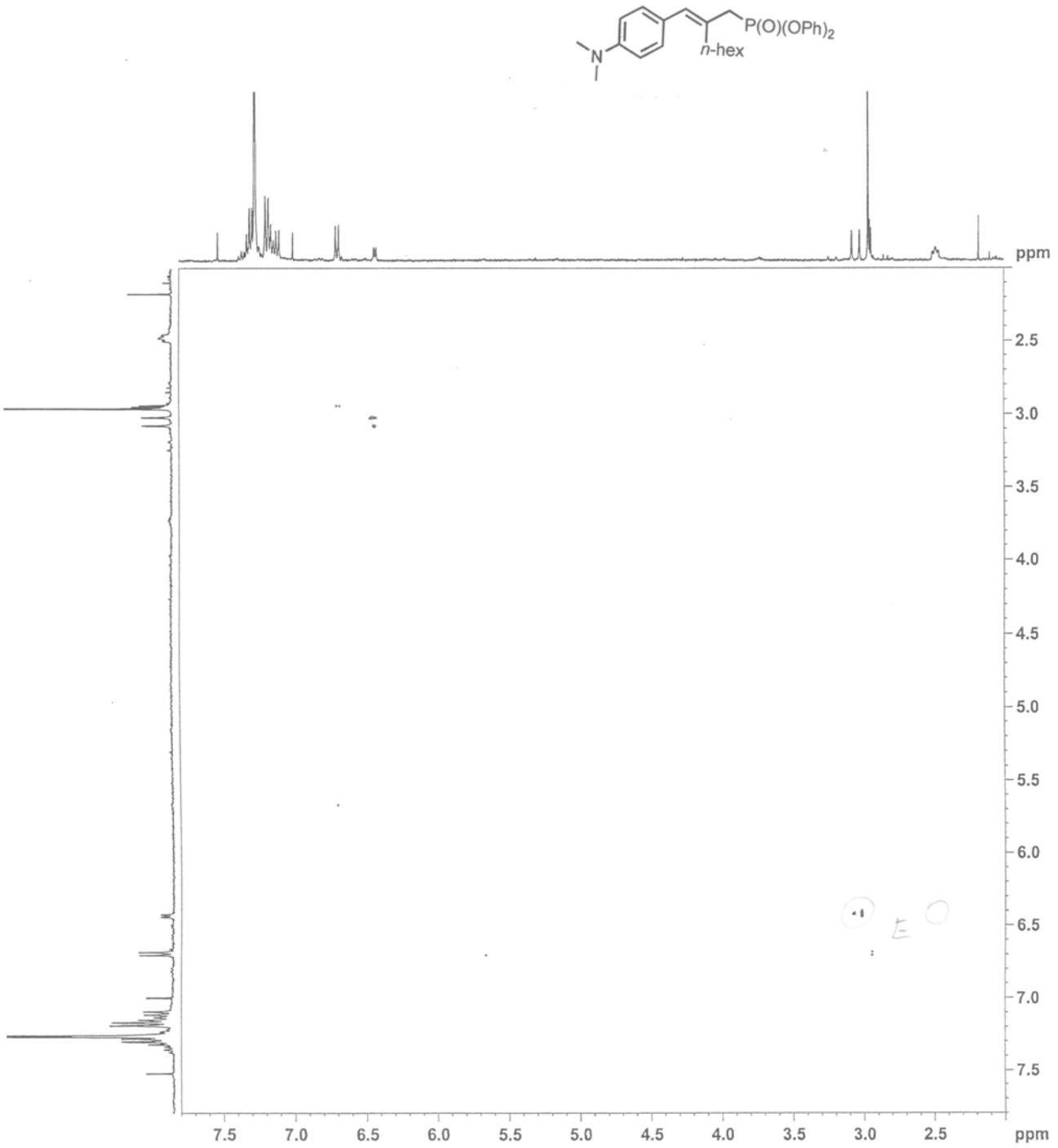
A192


NAME ccw_1_123_110 Si plate 2 up2
EXPNO 1
PROCNO 1
Date_ 20091110
Time 15.05
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 17
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300041 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
n-hex
N
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
4.90
6.65
2.91
2.42
0.97
2.00
6.10
2.06
2.27
9.30
4.65

A1p1



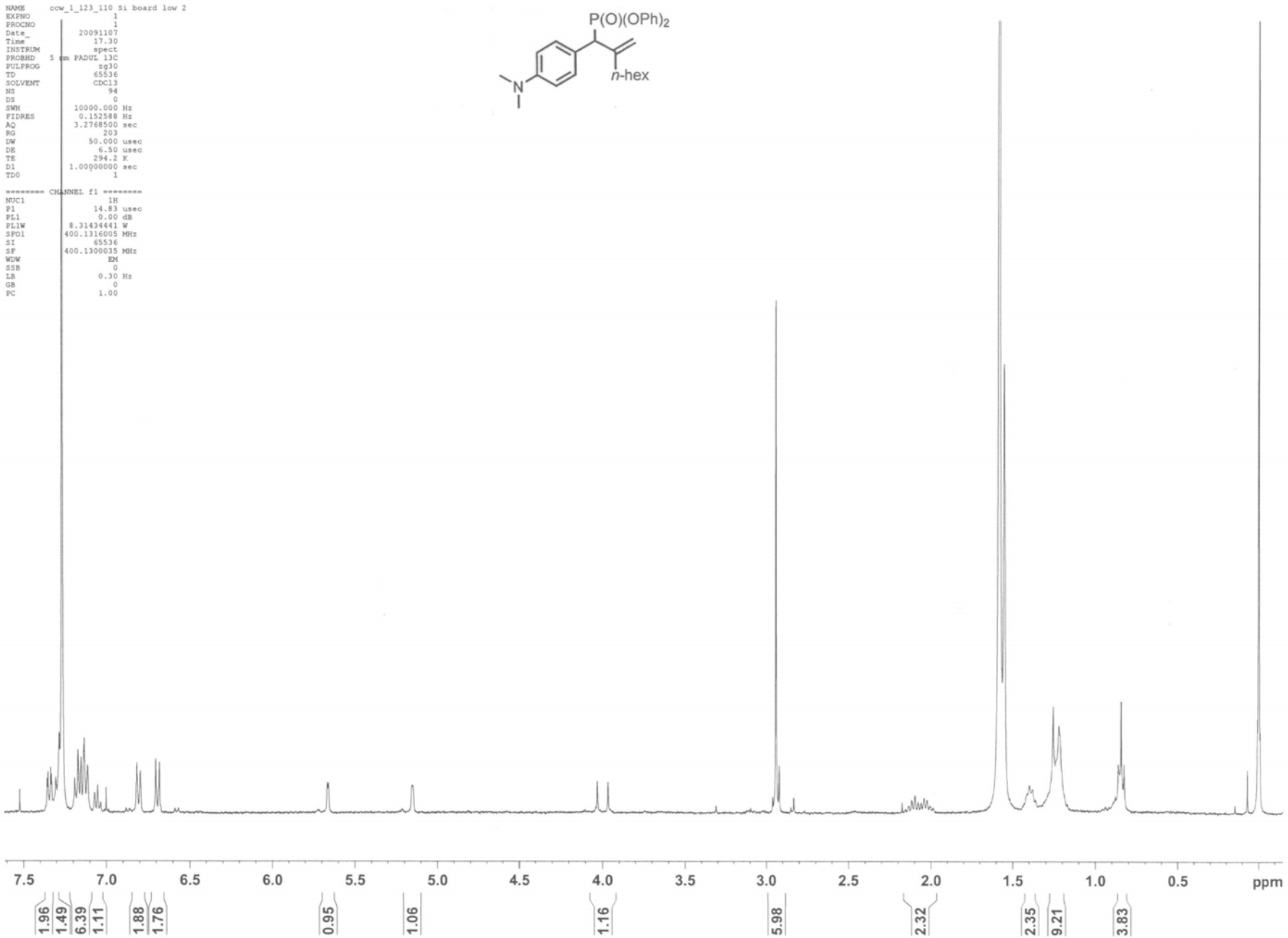

```
NAME      ccw_1_123_110 Si board up pure 13C
EXPNO                 2
PROCNO                1
Date_          20091113
Time              20.15
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                2422
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ           1.3631988 sec
RG                 203
DW              20.800 usec
DE               6.50 usec
TE              294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                9.68 usec
PL1              -0.60 dB
PL1W       41.24164963 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2               0.00 dB
PL12             15.66 dB
PL13             15.92 dB
PL2W        8.31434441 W
PL12W       0.22585411 W
PL13W       0.21272963 W
SFO2       400.1316005 MHz
SI               32768
SF         100.6127546 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



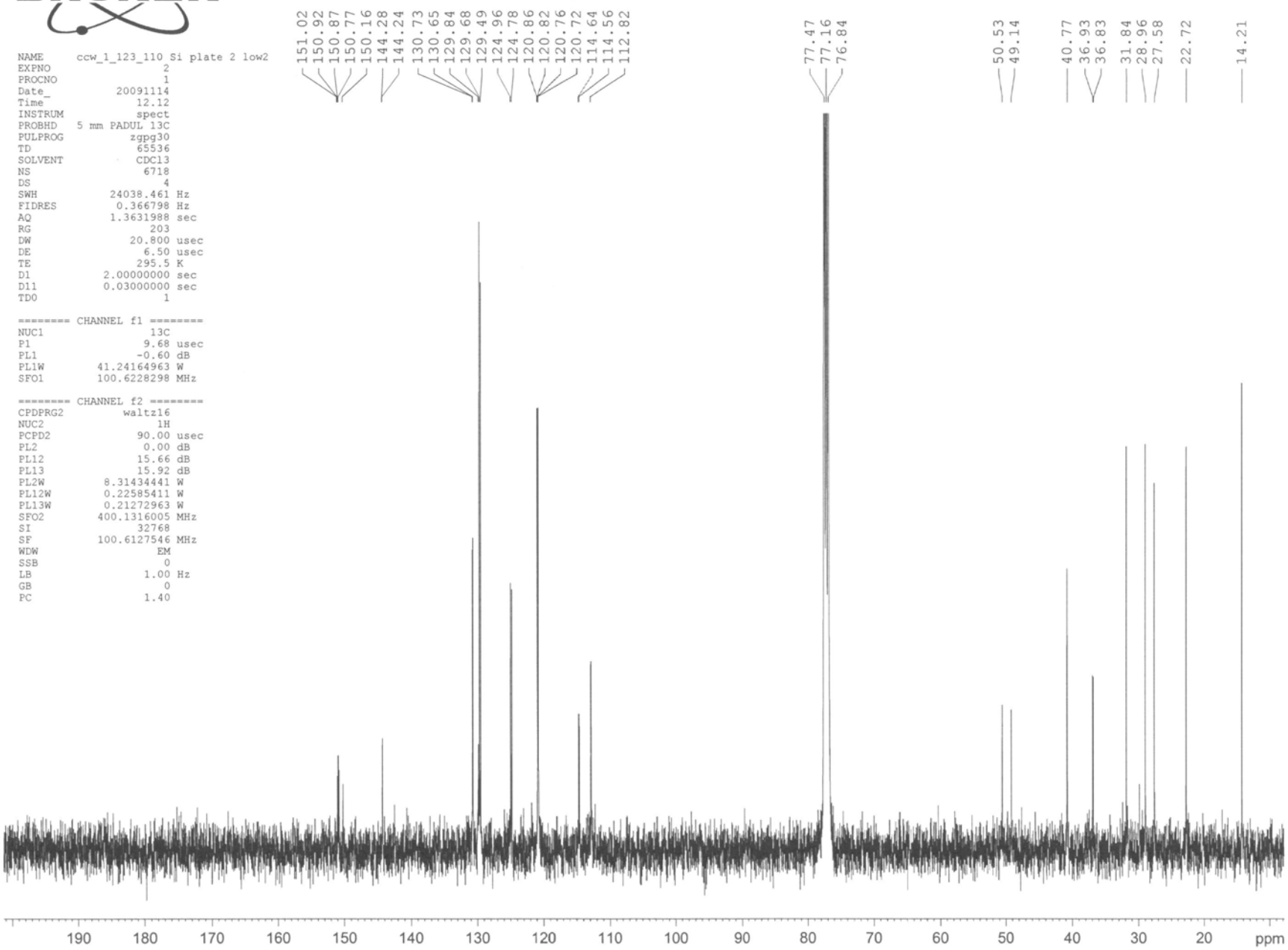
Bruker Advance III 400

NAME	ccw_1_123_110 Si board up 2	
EXPNO	6	
PROCNO	1	
Date_	20091110	
Time	15.19	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	noesyph	
TD	2048	
SOLVENT	CDCl3	
NS	16	
DS	4	
SWH	4084.967	Hz
FIDRES	1.994613	Hz
AQ	0.2507252	sec
RG	203	
DW	122.400	usec
DE	6.50	usec
TE	294.5	K
D0	0.00010352	sec
D1	2.00000000	sec
D8	0.30000001	sec
IN0	0.00024480	sec
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.83	usec
PL1	0.00	dB
PL1W	8.31434441	W
SFO1	400.1318419	MHz
ND0	1	
TD	20	
SFO1	400.1318	MHz
FIDRES	204.248367	Hz
SW	10.209	ppm
FnMODE	States-TPPI	
SI	1024	
SF	400.1300009	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	
PC	1.00	
SI	1024	
MC2	States-TPPI	
SF	400.1300008	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	

A191

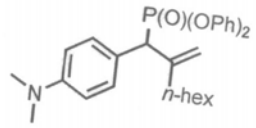

NAME ccw_1_123_110 Si board low 2
EXPNO 1
PROCNO 1
Date_ 20091107
Time 17.30
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 94
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.83 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300035 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
P(O)(OPh)2
N
n-hex
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.96
1.49
6.39
1.11
1.88
1.76
0.95
1.06
1.16
5.98
2.32
2.35
9.21
3.83

A1p2

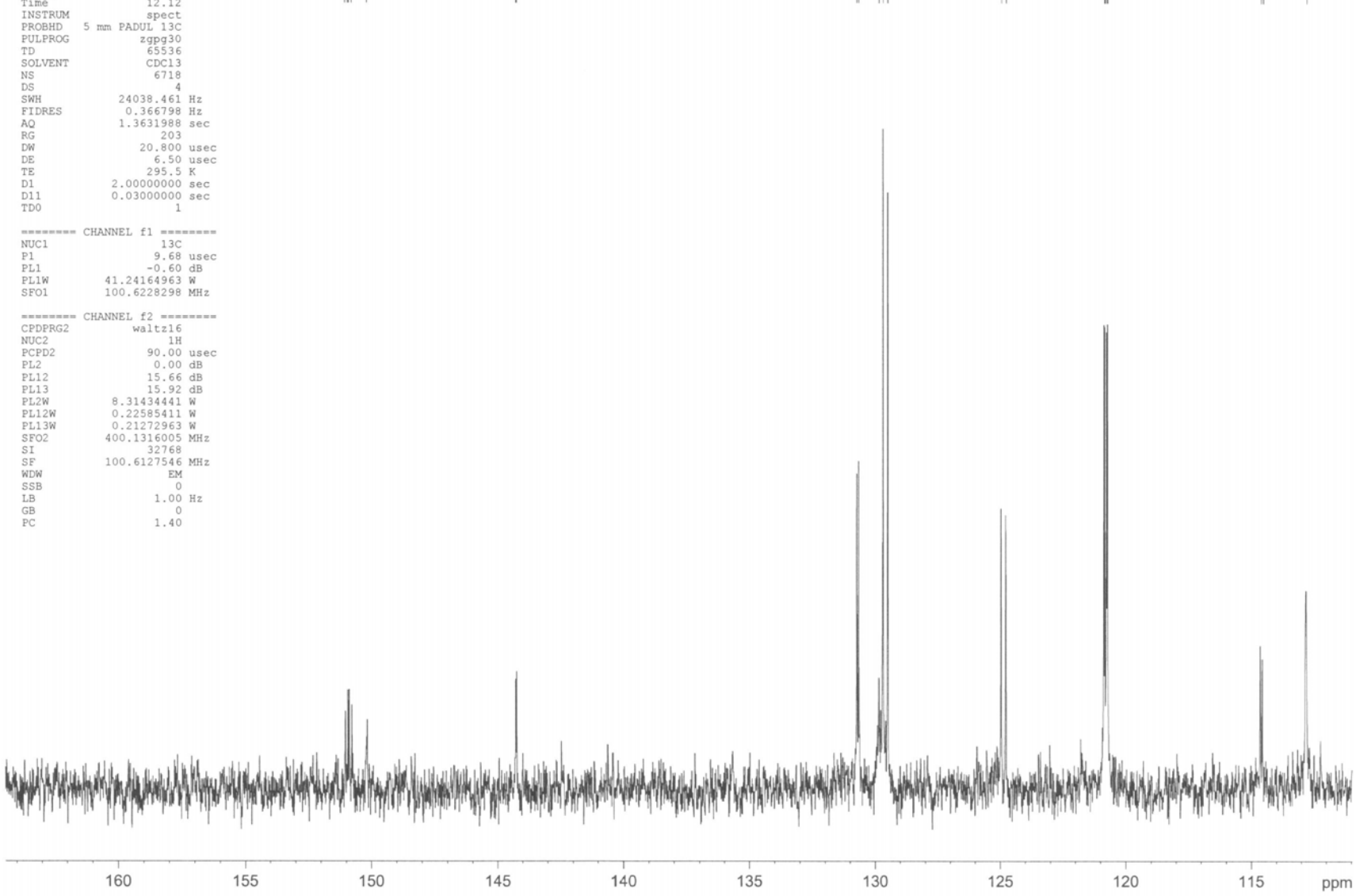



```
NAME        ccw_1_123_110 Si plate 2 low2
EXPNO                    2
PROCNO                   1
Date_             20091114
Time                 12.12
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    6718
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                  295.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.68 usec
PL1                  -0.60 dB
PL1W          41.24164963 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                   0.00 dB
PL12                 15.66 dB
PL13                 15.92 dB
PL2W           8.31434441 W
PL12W          0.22585411 W
PL13W          0.21272963 W
SFO2         400.1316005 MHz
SI                   32768
SF           100.6127546 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

A122




```
NAME      ccw_1_123_110 Si plate 2 low2
EXPNO                 2
PROCNO                1
Date_          20091114
Time              12.12
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                 6718
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                295.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.68 usec
PL1               -0.60 dB
PL1W        41.24164963 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                 0.00 dB
PL12               15.66 dB
PL13               15.92 dB
PL2W         8.31434441 W
PL12W        0.22585411 W
PL13W        0.21272963 W
SFO2       400.1316005 MHz
SI                 32768
SF         100.6127546 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

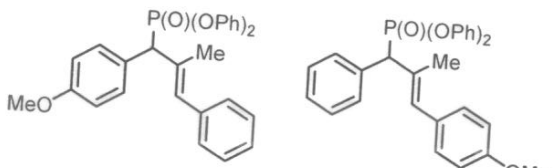


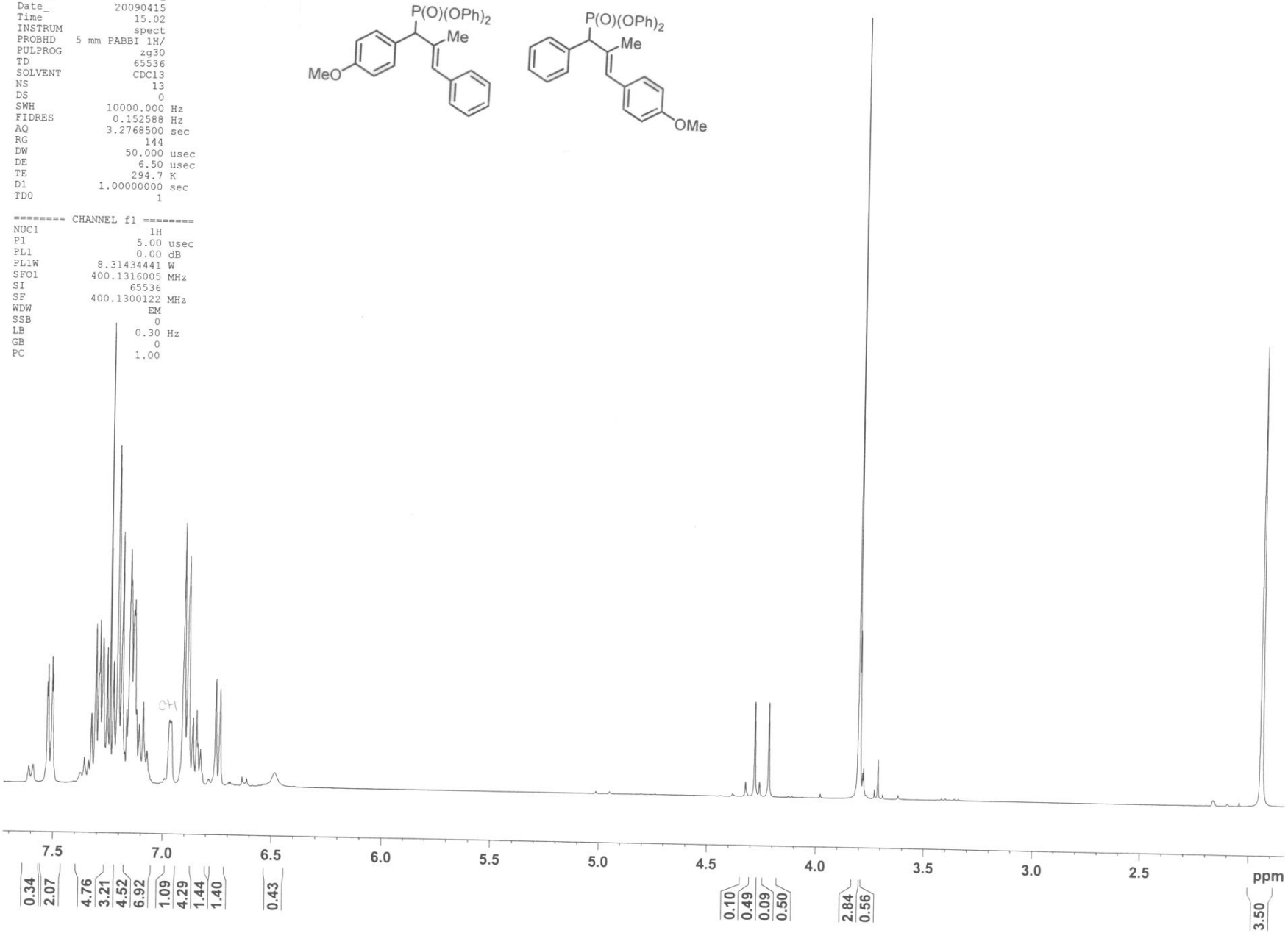
A1p2

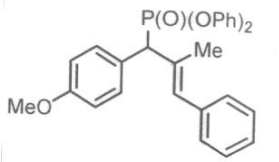
NAME	ccw_1_123_67 t14-16
EXPNO	1
PROCNO	1
Date_	20090415
Time	15.02
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	13
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	144
DW	50.000 usec
DE	6.50 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1

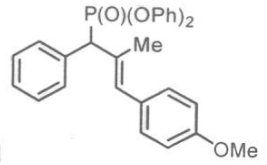
======== CHANNEL f1 ========

NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300122 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00









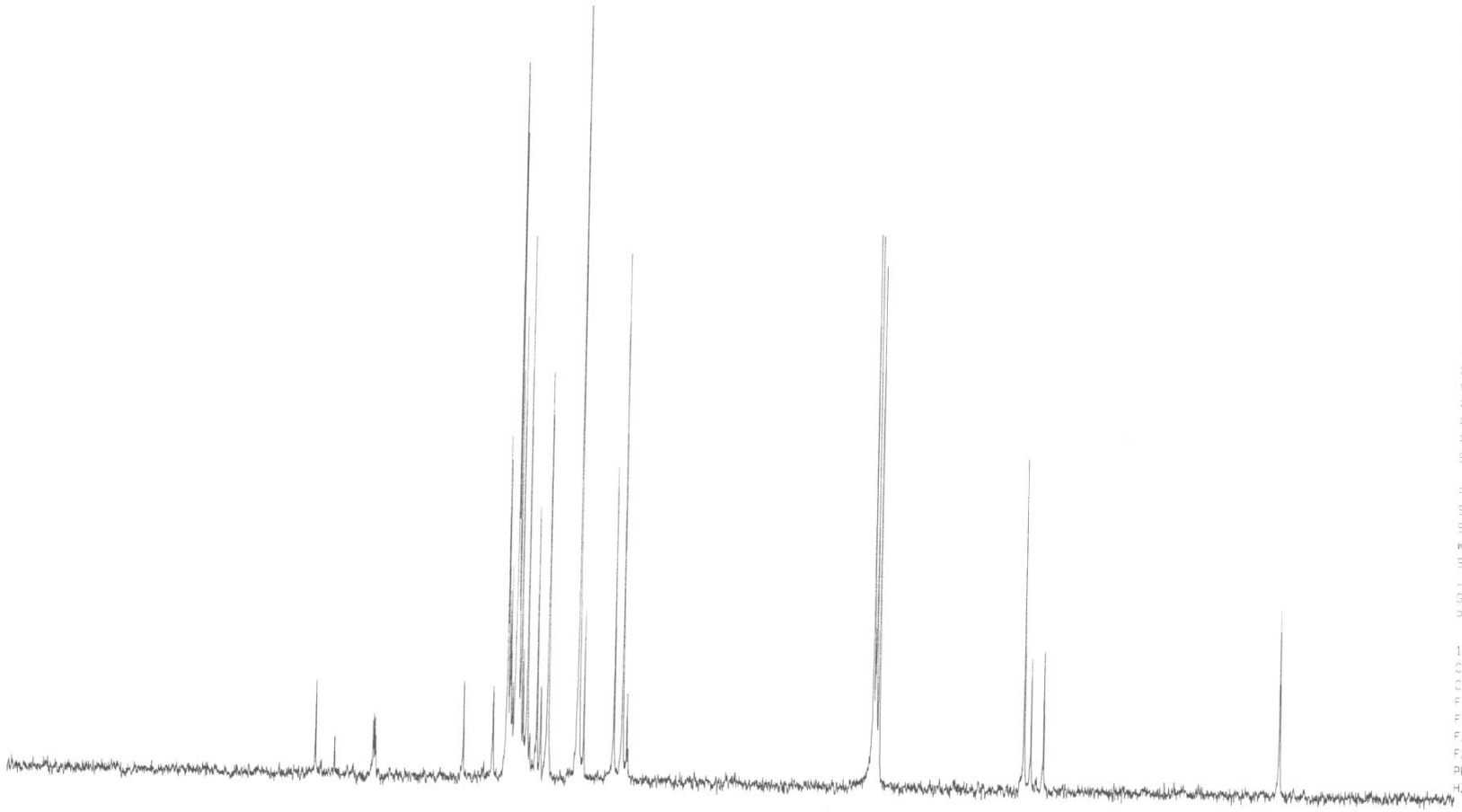
ppm

159.287
156.500
150.868
150.737
150.566
137.540
133.237
131.158
131.042
130.801
130.655
130.402
129.828
129.677
129.507
129.164
128.842
128.212
126.804
125.236
125.167
120.801
120.751
119.967
115.527
114.261

77.601
77.379
77.178
76.754

55.427
54.432
52.591

17.993
17.896



ppm 180 160 140 120 100 80 60 40 20 0

AM1+ Ann 2

Current Data Parameters
NAME ccw_1_123_67 t14-16 13C
EXPNO 2
PROCNO 1

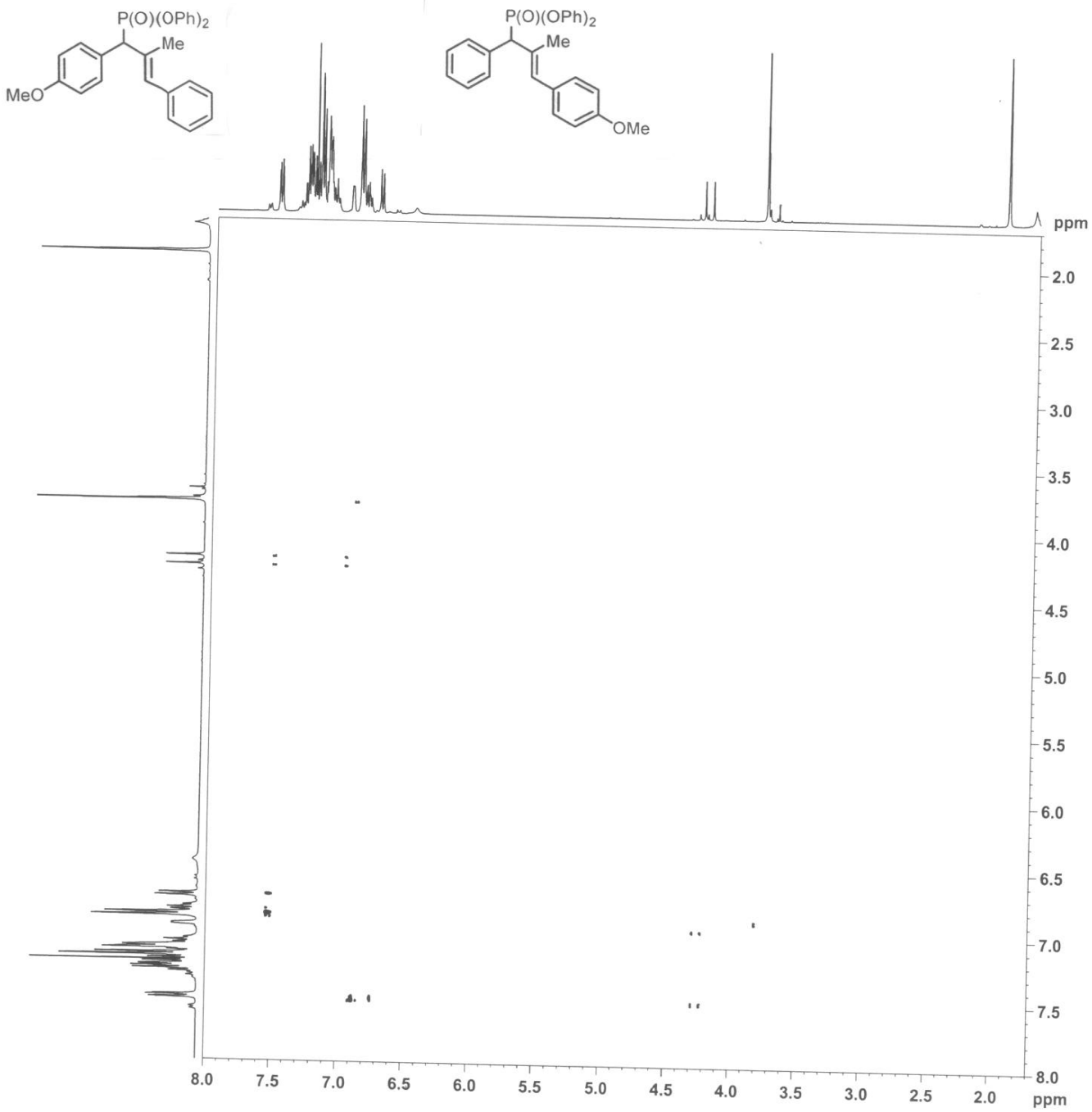
F2 - Acquisition Parameters
Date_ 20090421
Time 19.59
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 1625
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 9195.2
DW 22.050 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SFO1 75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SFO2 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677427 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

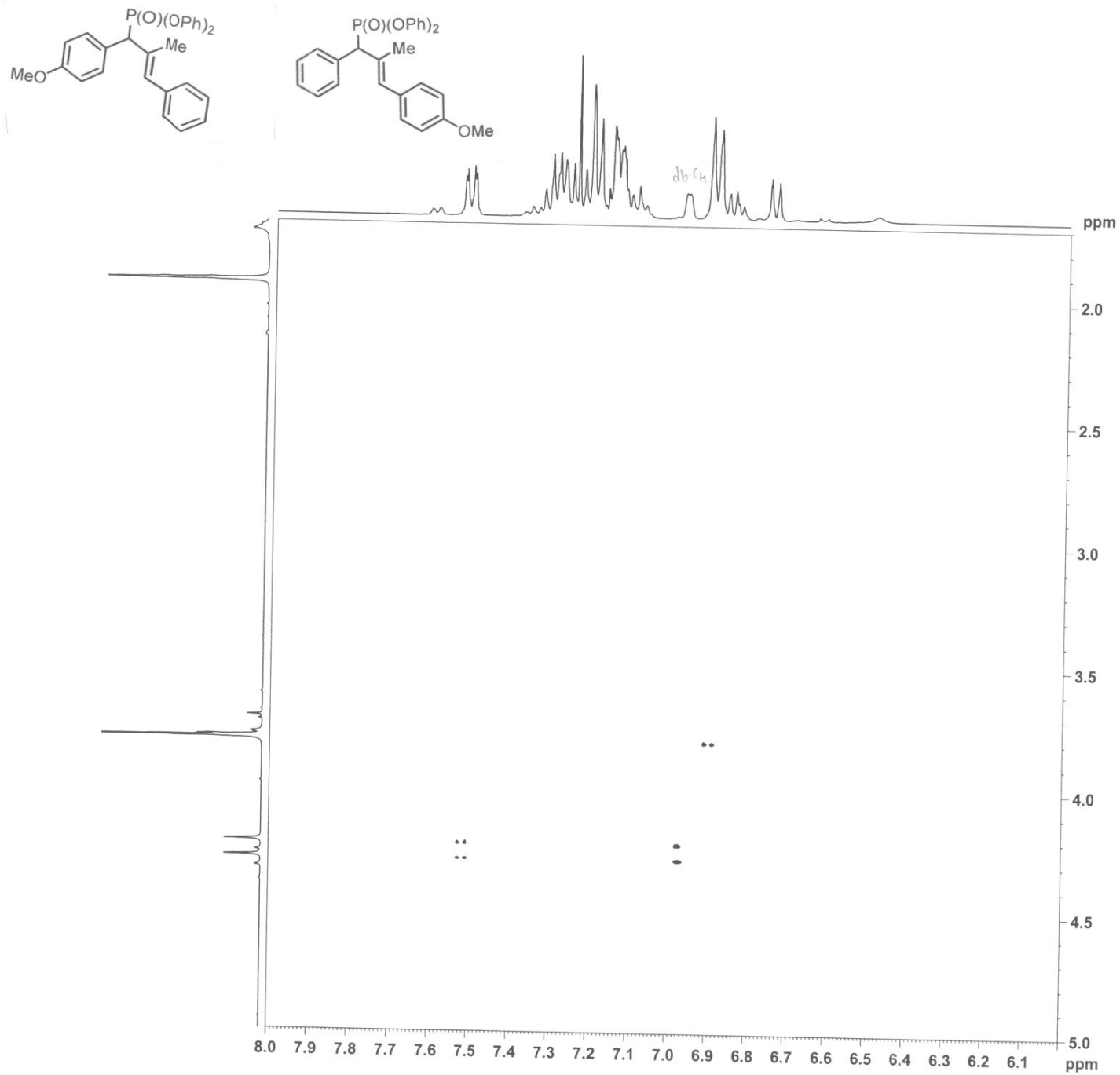
1D NMR plot parameters
CX 23.00 cm
CY 12.00 cm
F1P 204.375 ppm
F1 15423.75 Hz
F2P -7.698 ppm
F2 -580.97 Hz
PPMCM 9.22060 ppm/cm
HZCM 695.85767 Hz/cm



BRUKER

NAME	ccw_1_123_67 t14-16
EXPNO	6
PROCNO	1
Date_	20090415
Time	15.03
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	31
SFO1	400.1318 MHz
FIDRES	131.773132 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300088 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300088 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

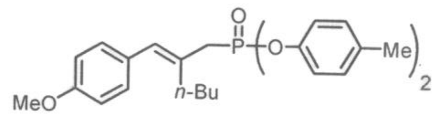


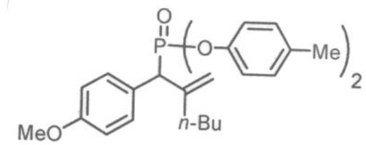
BRUKER

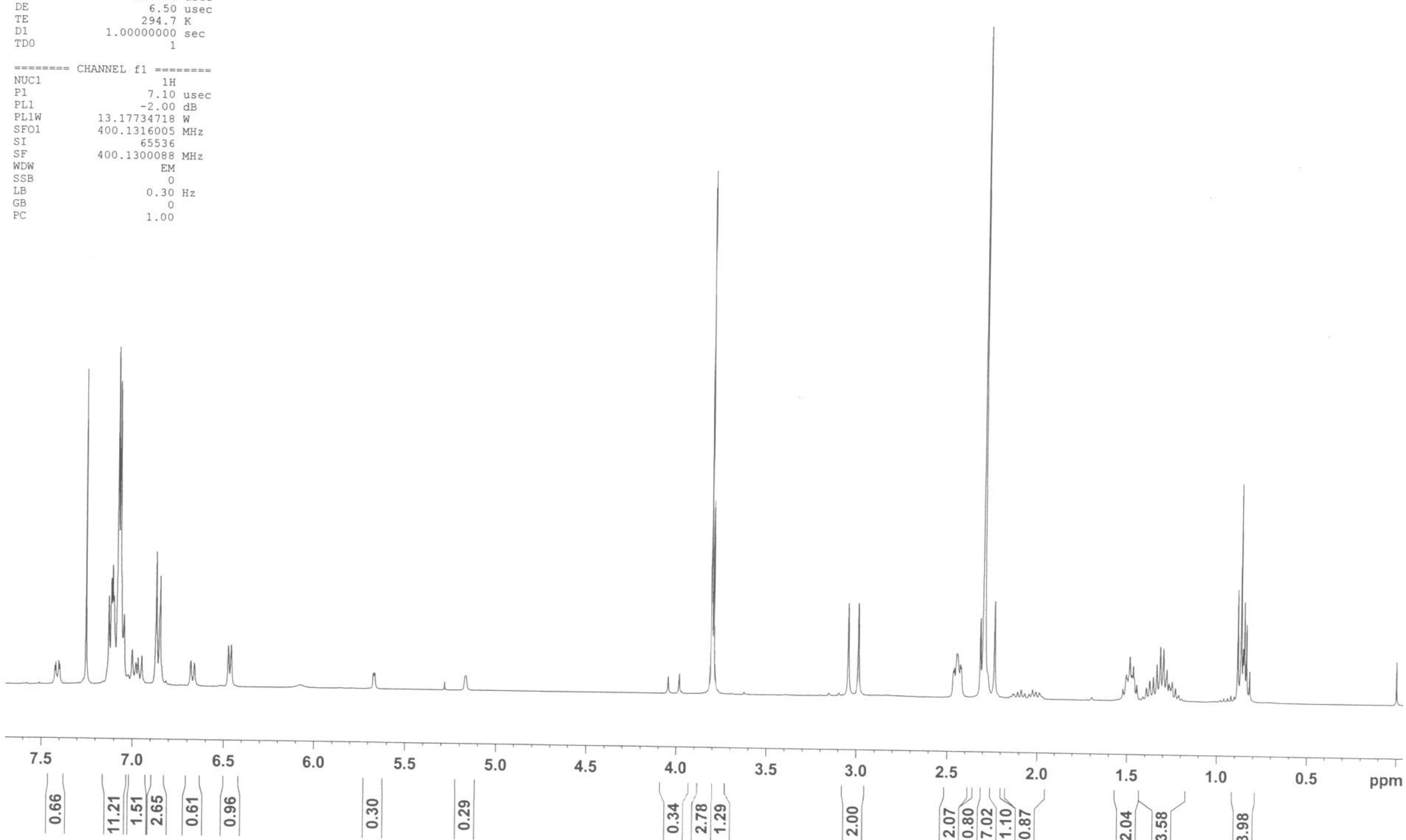
NAME	ccw_1_123_67 t14-16
EXPNO	6
PROCNO	1
Date_	20090415
Time	15.03
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	31
SFO1	400.1318 MHz
FIDRES	131.773132 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300088 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300088 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

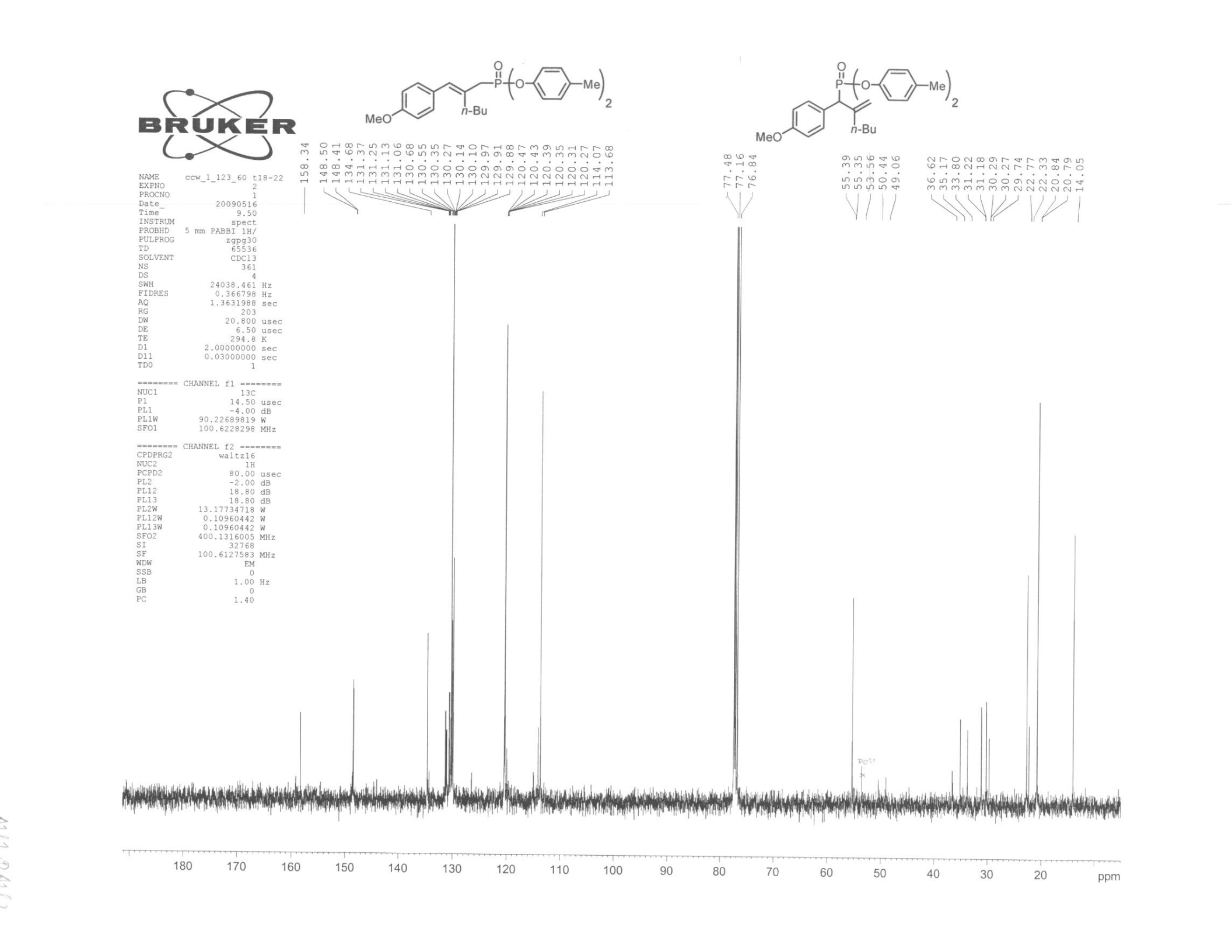
```
NAME       ccw_1_123_60 t18-22
EXPNO                  11
PROCNO                  1
Date_            20090423
Time                19.15
INSTRUM             spect
PROBHD    5 mm PABBI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     18
DS                      0
SWH             10000.000 Hz
FIDRES           0.152588 Hz
AQ              3.2768500 sec
RG                     57
DW                 50.000 usec
DE                   6.50 usec
TE                  294.7 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.10 usec
PL1                 -2.00 dB
PL1W          13.17734718 W
SFO1          400.1316005 MHz
SI                  65536
SF            400.1300088 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```







A121
+ A122


BRUKER
MeO
n-Bu
Me
2
NAME ccw_1_123_60 t18-22
EXPNO 2
PROCNO 1
Date_ 20090516
Time 9.50
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 361
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 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127583 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
158.34
148.50
148.41
134.68
131.37
131.25
131.13
131.06
130.68
130.55
130.35
130.27
130.14
130.10
129.97
129.91
129.88
120.47
120.43
120.39
120.35
120.31
120.27
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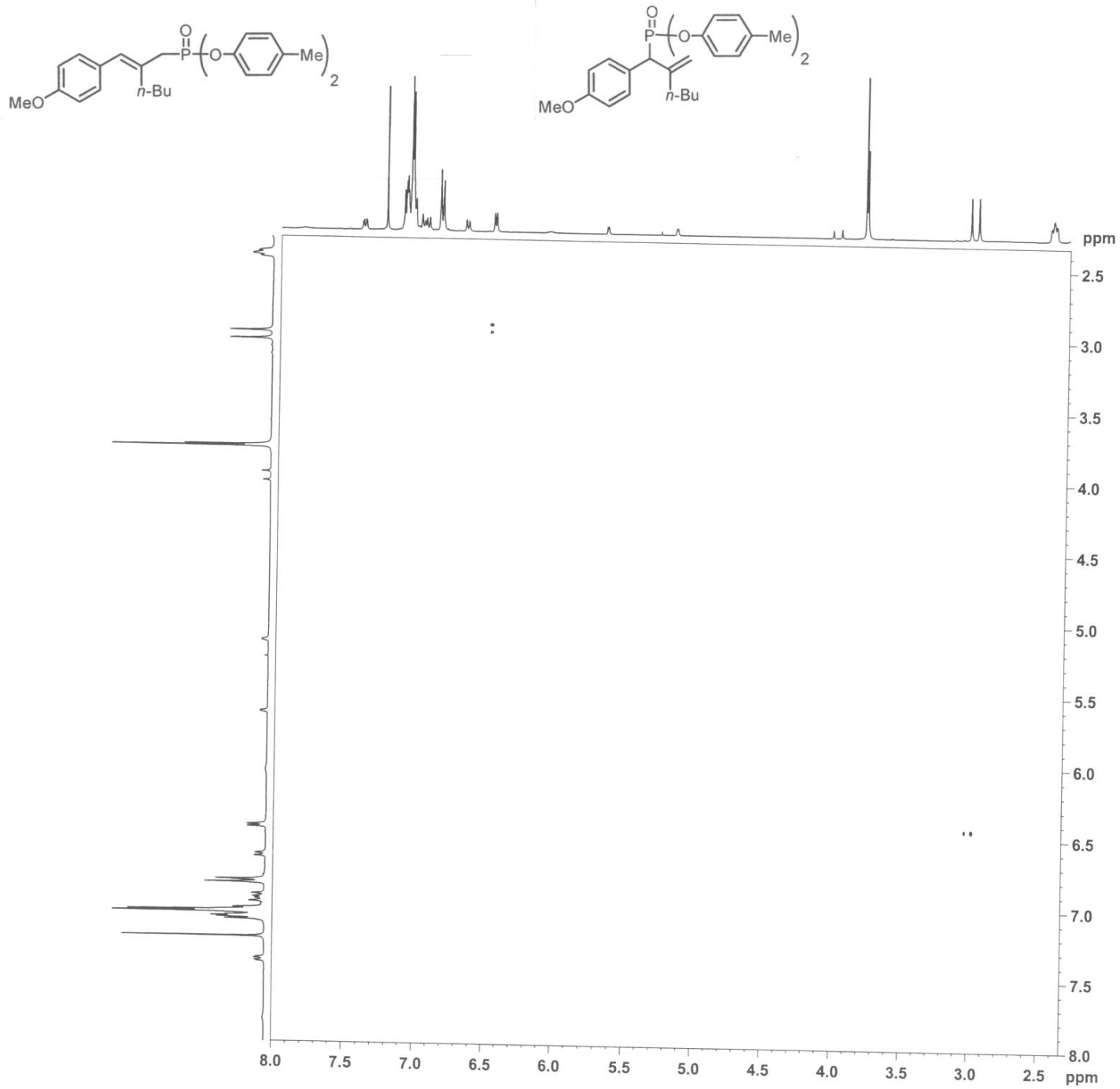
113.68
77.48
77.16
76.84
55.39
55.35
53.56
50.44
49.06
36.62
35.17
33.80
31.22
31.18
30.29
30.27
29.74
22.77
22.33
20.84
20.79
14.05
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

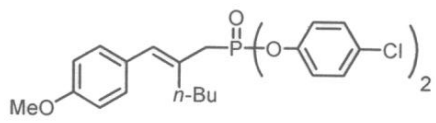


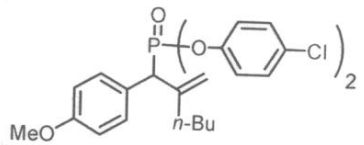
BRUKER

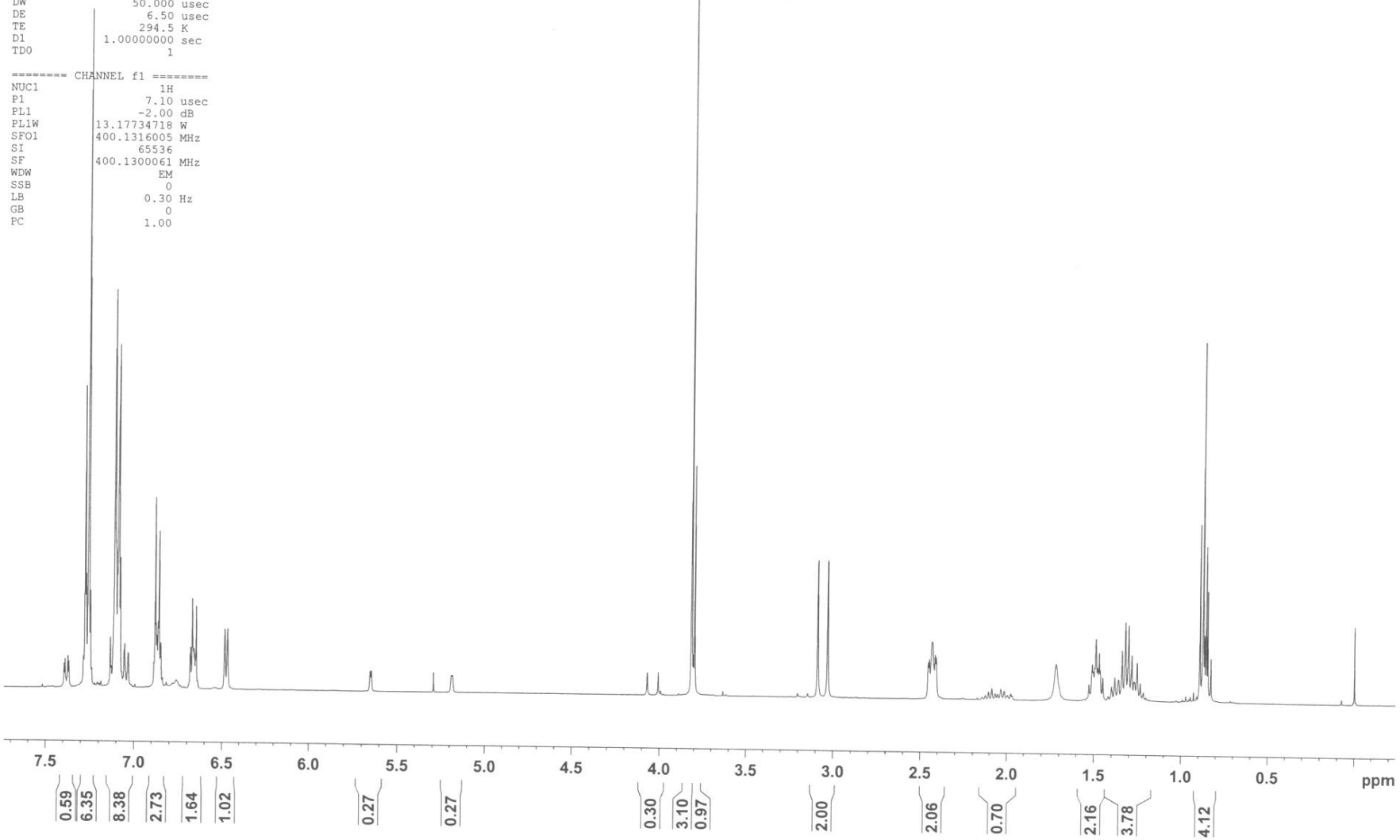
NAME	ccw_1_123_60 t18-22
EXPNO	6
PROCNO	1
Date_	20090506
Time	17.56
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

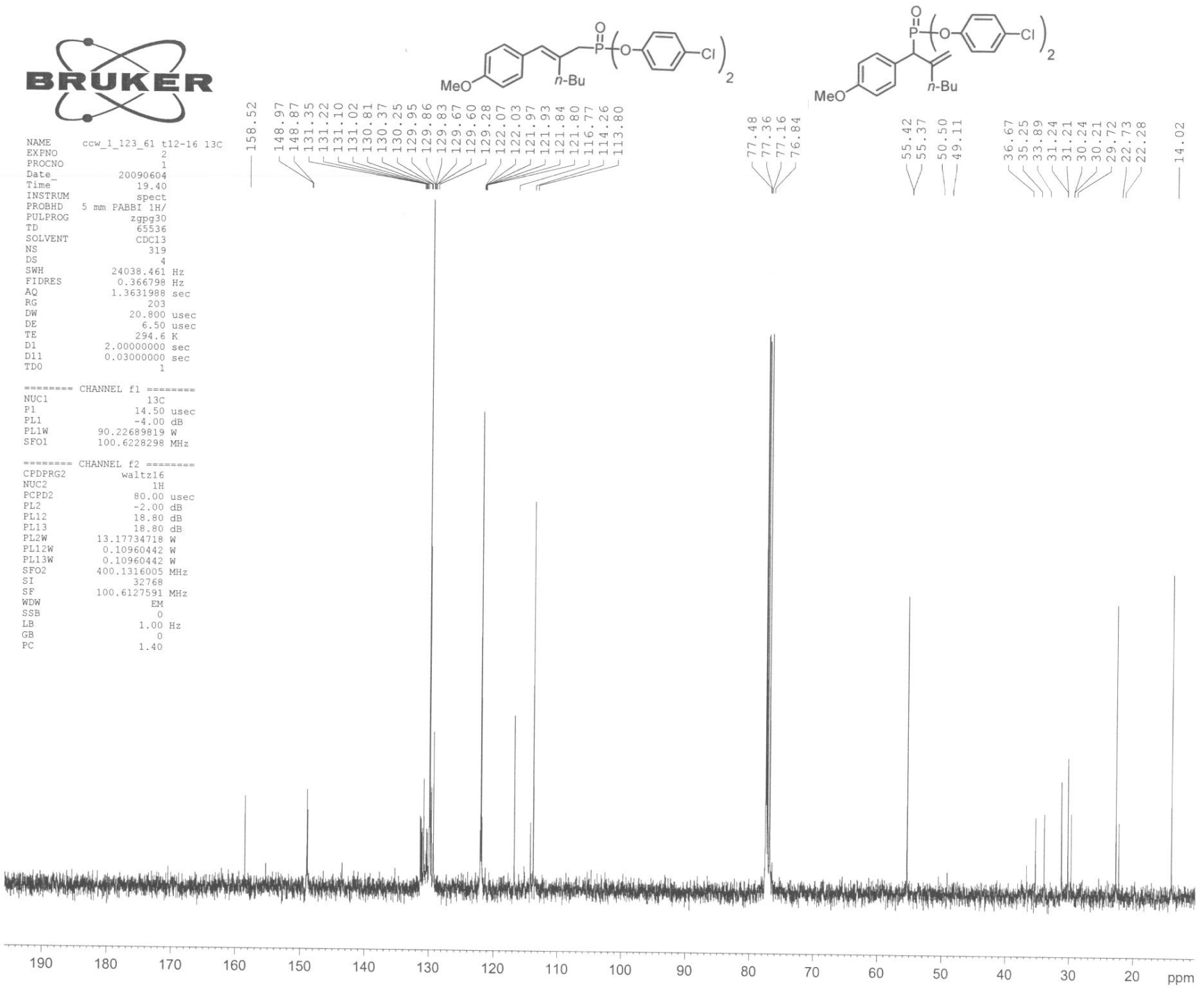
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	36
SFO1	400.1318 MHz
FIDRES	255.310455 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300328 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300527 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

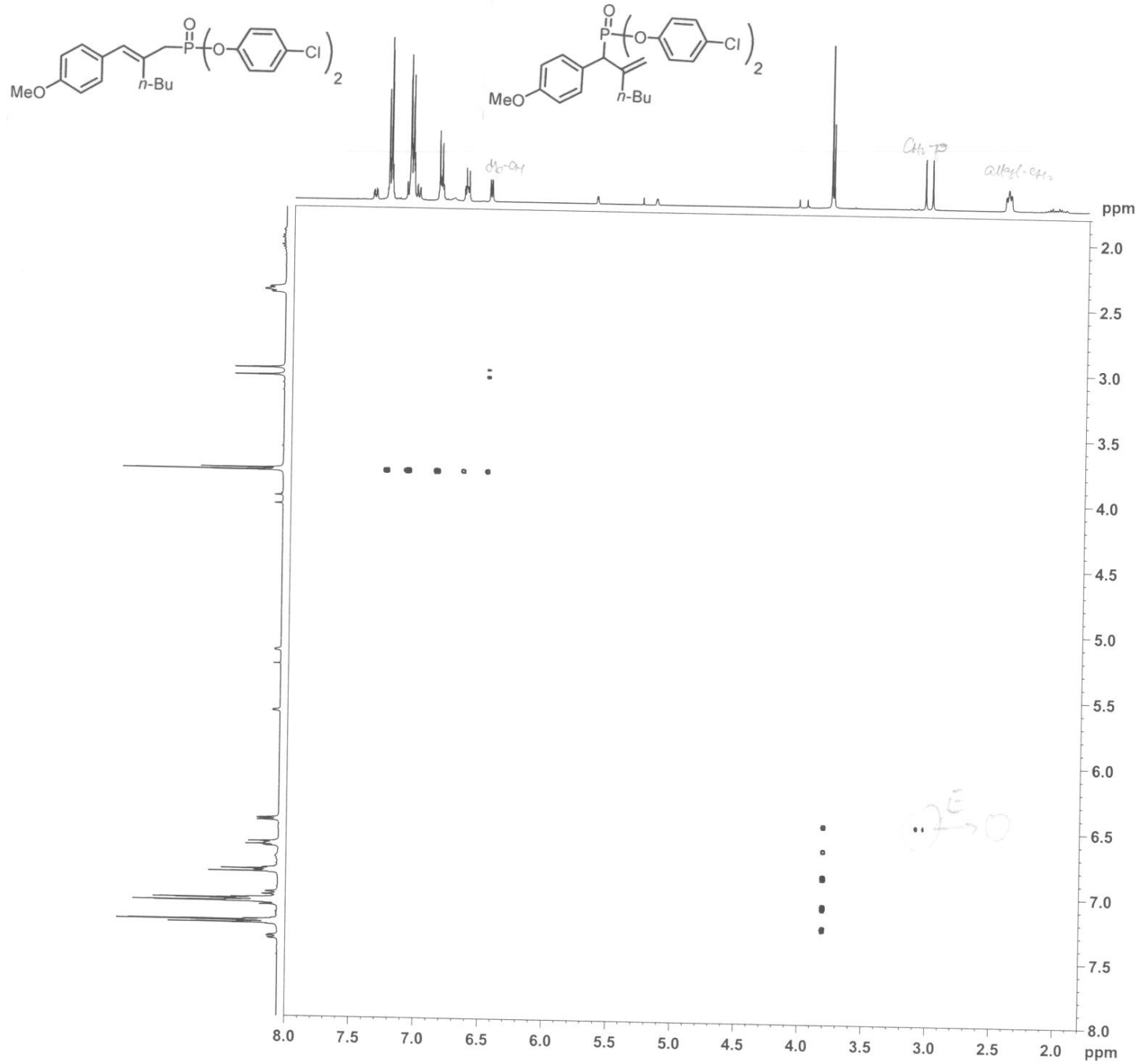
```
NAME      ccw_1_123_61 t12-16
EXPNO                 11
PROCNO                 1
Date_           20090423
Time_             20.00
INSTRUM           spect
PROBHD    5 mm PABBI 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  25
DS                   0
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ           3.2768500 sec
RG                90.5
DW              50.000 usec
DE               6.50 usec
TE              294.5 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                 1H
P1                7.10 usec
PL1              -2.00 dB
PL1W        13.17734718 W
SFO1        400.1316005 MHz
SI               65536
SF          400.1300061 MHz
WDW                 EM
SSB                  0
LB               0.30 Hz
GB                   0
PC                1.00
```








BRUKER
MeO
n-Bu
Cl
2
158.52
148.97
148.87
131.35
131.22
131.10
131.02
130.81
130.37
130.25
129.95
129.86
129.83
129.67
129.60
129.28
122.07
122.03
121.97
121.93
121.84
121.80
116.77
114.26
113.80
77.48
77.36
77.16
76.84
55.42
55.37
50.50
49.11
36.67
35.25
33.89
31.24
31.21
30.24
30.21
29.72
22.73
22.28
14.02
NAME ccw_1_123_61 t12-16 13C
EXPNO 2
PROCNO 1
Date_ 20090604
Time 19.40
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 319
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 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127591 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm



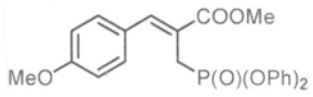
BRUKER

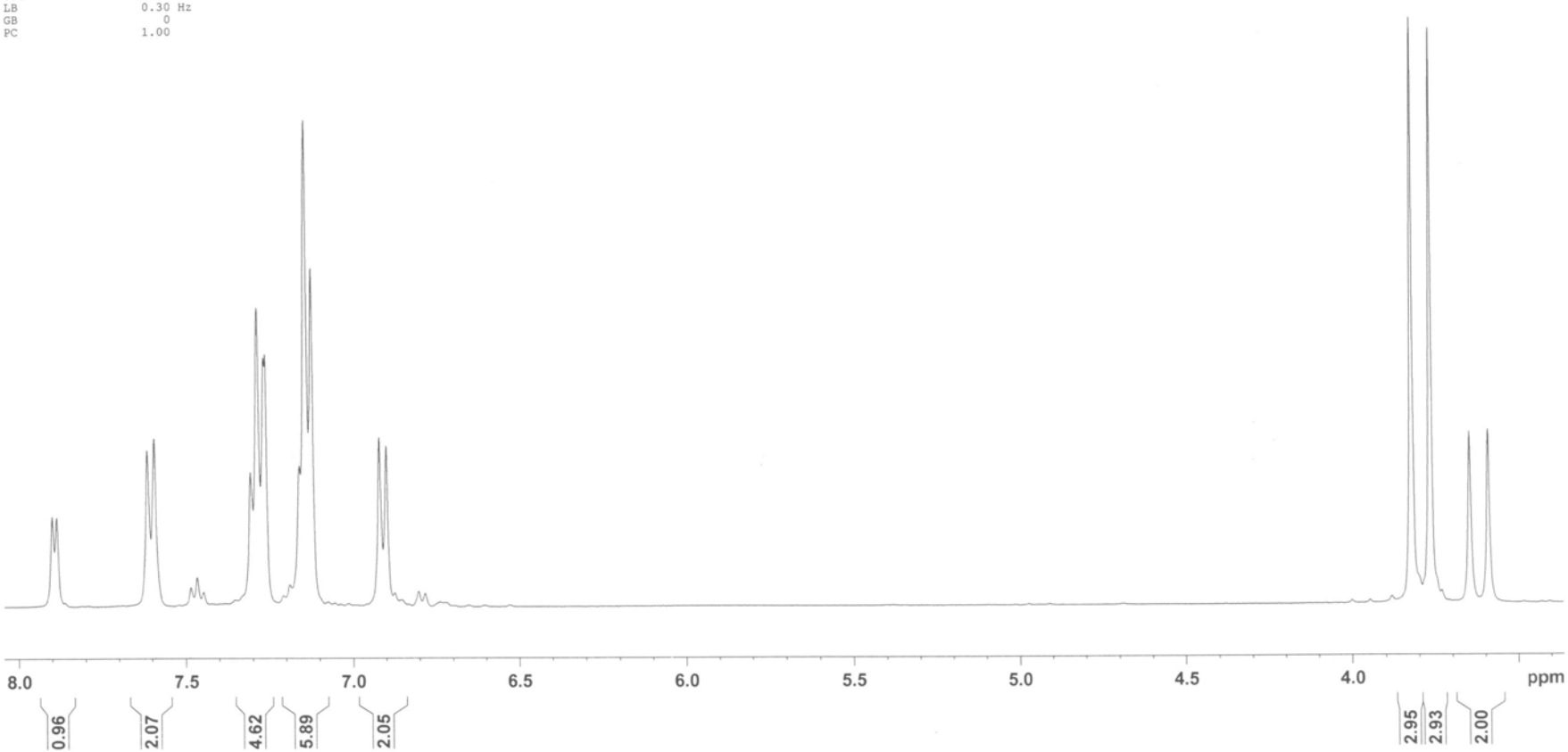
NAME	ccw_1_123_61 t12-16
EXPNO	6
PROCNO	1
Date_	20090423
Time	19.54
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

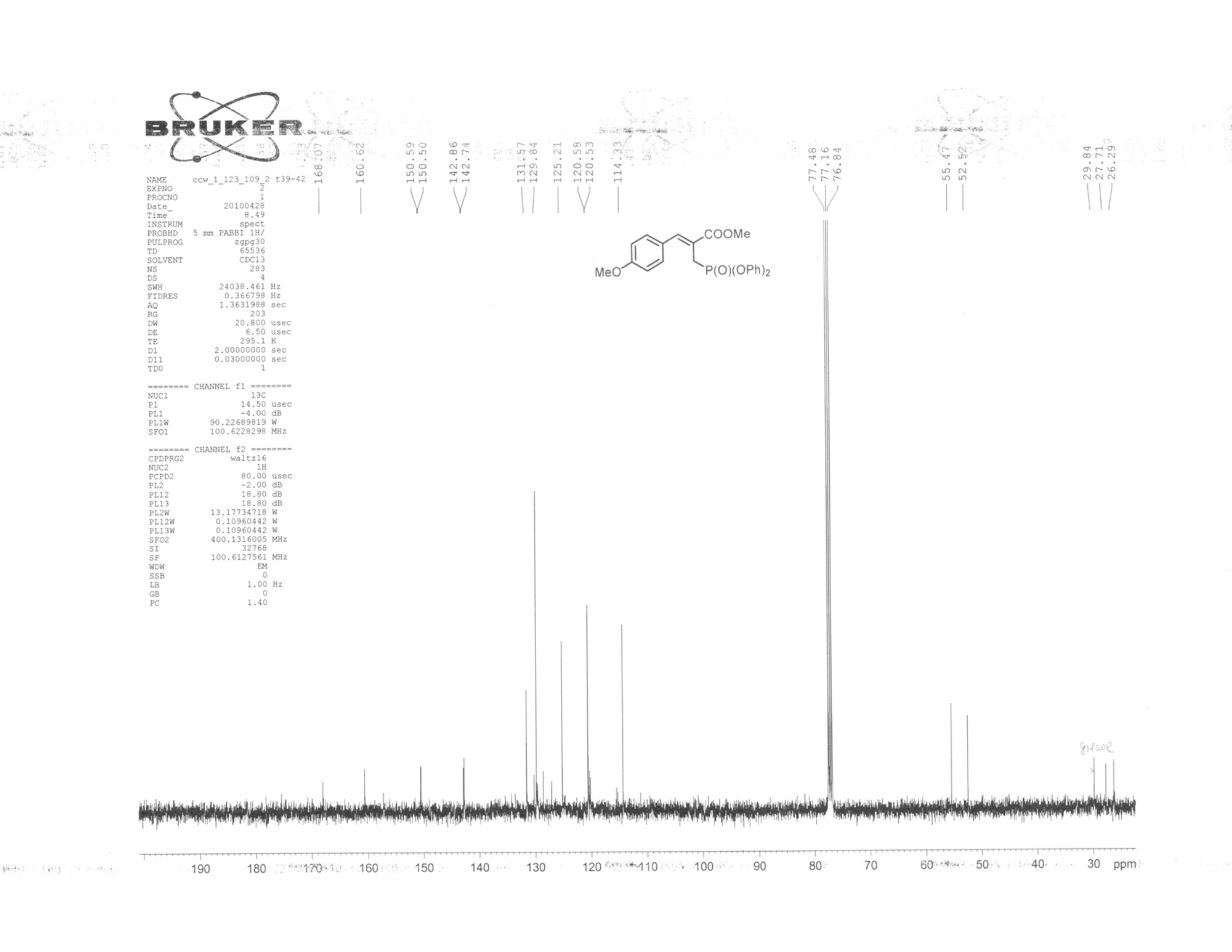
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	6
SFO1	400.1318 MHz
FIDRES	680.827881 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

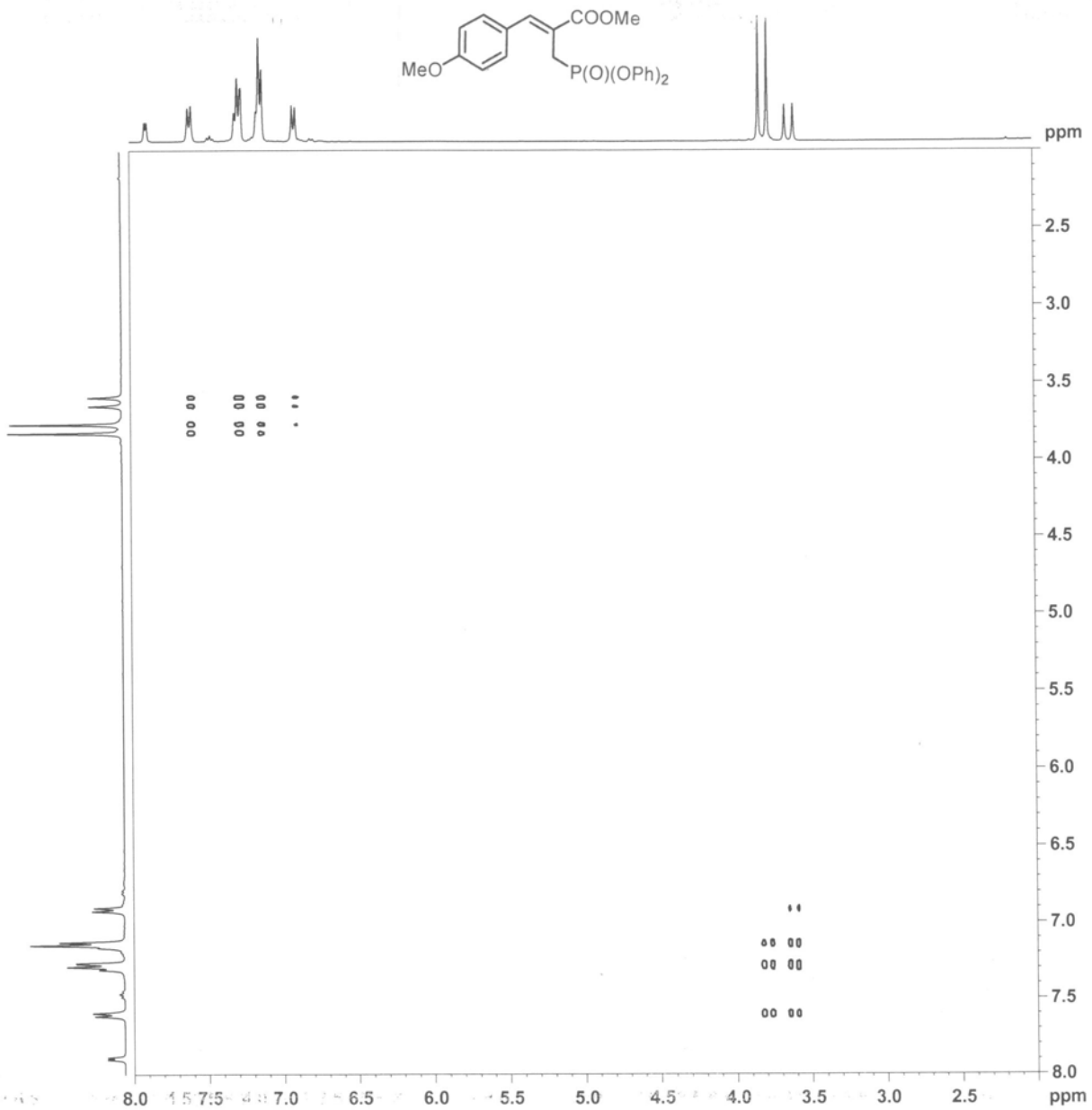
A1m1 + A1m2

```
NAME      ccw_1_123_109_2 t39-42
EXPNO                      1
PROCNO                     1
Date_               20100428
Time                    8.35
INSTRUM                 spect
PROBHD   5 mm PABBI 1H/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        10
DS                         0
SWH                10000.000 Hz
FIDRES                 0.152588 Hz
AQ                 3.2768500 sec
RG                       203
DW                    50.000 usec
DE                     6.50 usec
TE                    295.3 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     5.00 usec
PL1                    0.00 dB
PL1W               8.31434441 W
SFO1             400.1316005 MHz
SI                     65536
SF               400.1300061 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                     1.00
```





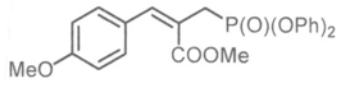

BRUKER
NAME ccw_1_123_109_2 t39-42
EXPNO 2
PROCNO 1
Date_ 20100428
Time 8.49
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 283
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127561 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
168.07
160.62
150.59
150.50
142.86
142.74
131.57
129.84
125.21
120.58
120.53
114.33
77.48
77.16
76.84
55.47
52.52
29.84
27.71
26.29
COOMe
MeO
P(O)(OPh)2
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

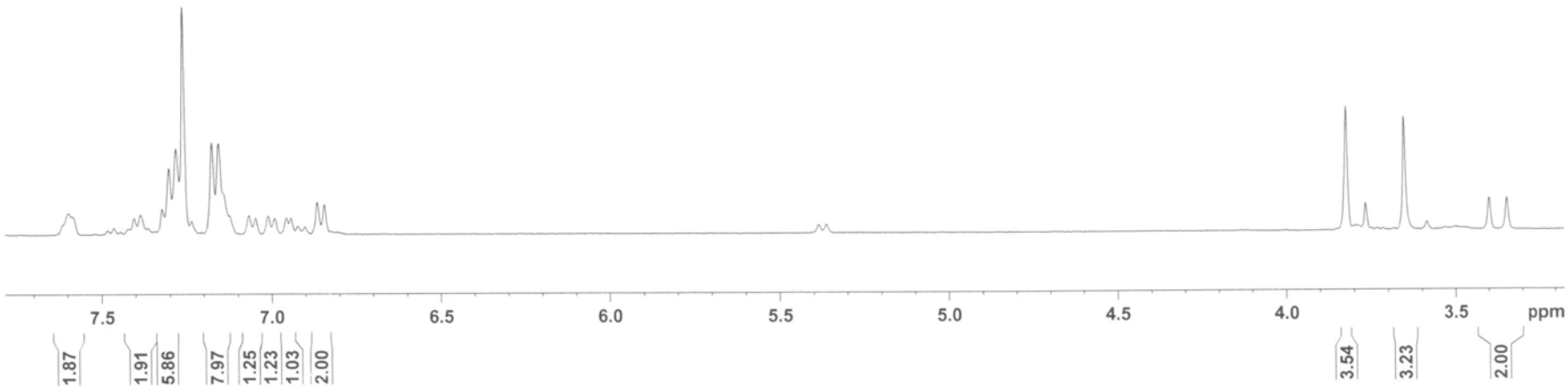


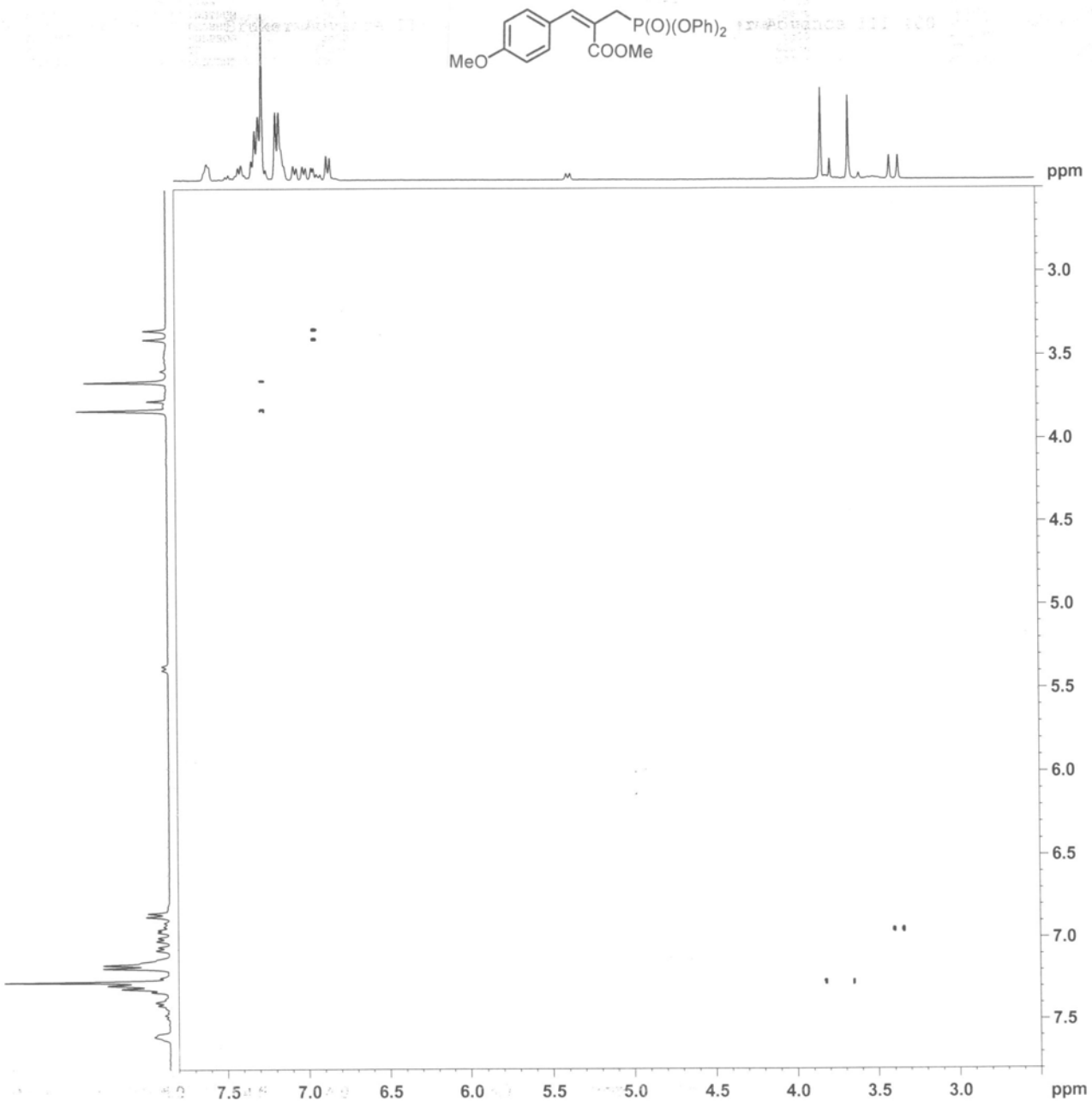
Bruker Advance III 400

NAME	ccw_1_123_109_2 t39-42
EXPNO	6
PROCNO	1
Date_	20100428
Time	8.37
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	295.3 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	12
SFO1	400.1318 MHz
FIDRES	340.413940 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

```
NAME        ccw_1_123_109_2 t43-45
EXPNO                     1
PROCNO                    1
Date_              20100428
Time                  9.13
INSTRUM              spect
PROBHD    5 mm PABBI 1H/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      14
DS                       0
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                     203
DW                  50.000 usec
DE                   6.50 usec
TE                  294.7 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   5.00 usec
PL1                  0.00 dB
PL1W            8.31434441 W
SFO1          400.1316005 MHz
SI                   65536
SF            400.1300056 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                   1.00
```

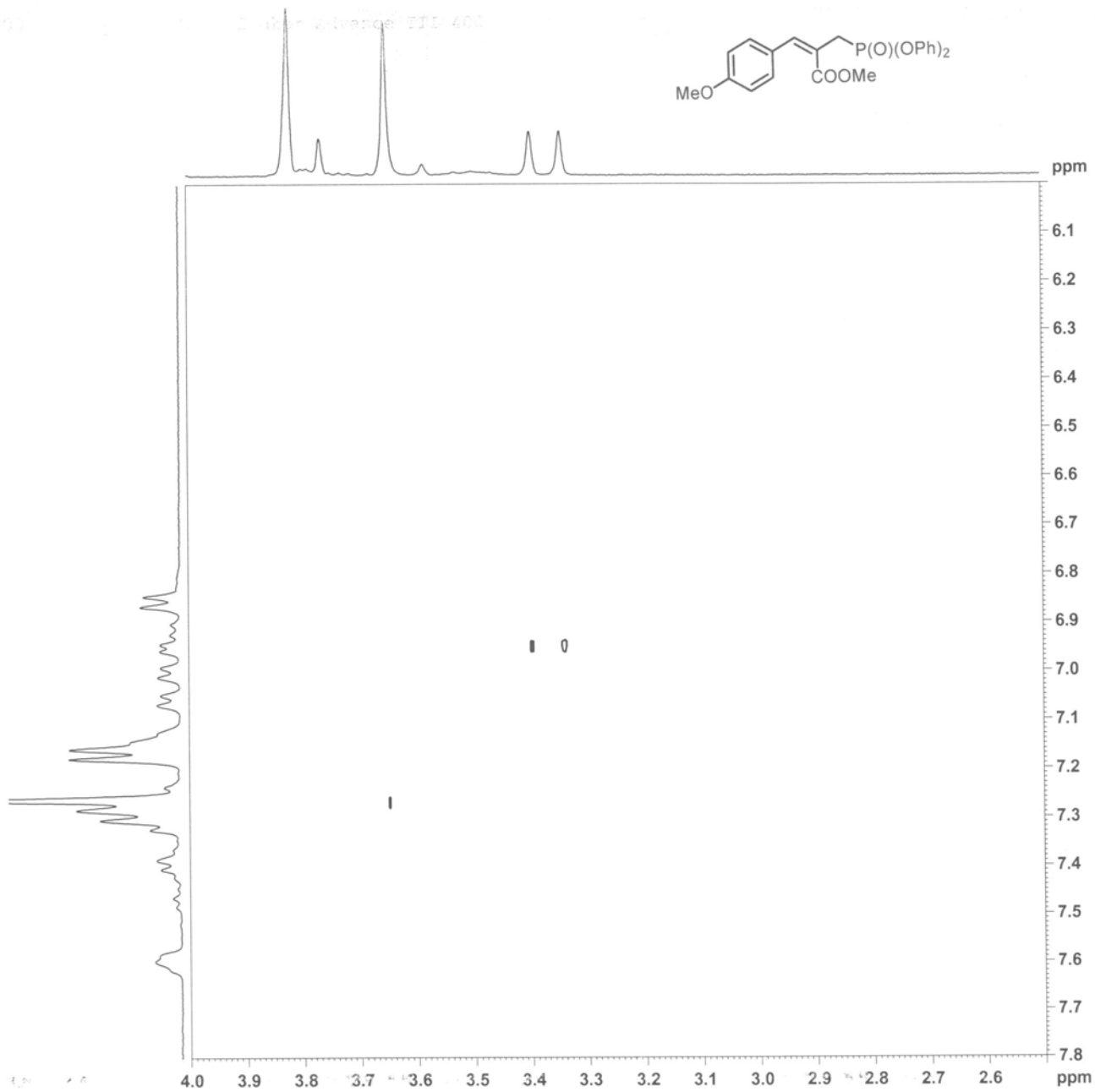






Bruker Advance III 400

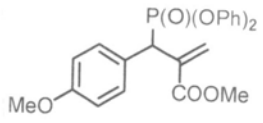
NAME	ccw_1_123_109_2 t43-45
EXPNO	6
PROCNO	1
Date_	20100428
Time	9.19
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	17
SFO1	400.1318 MHz
FIDRES	240.292191 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

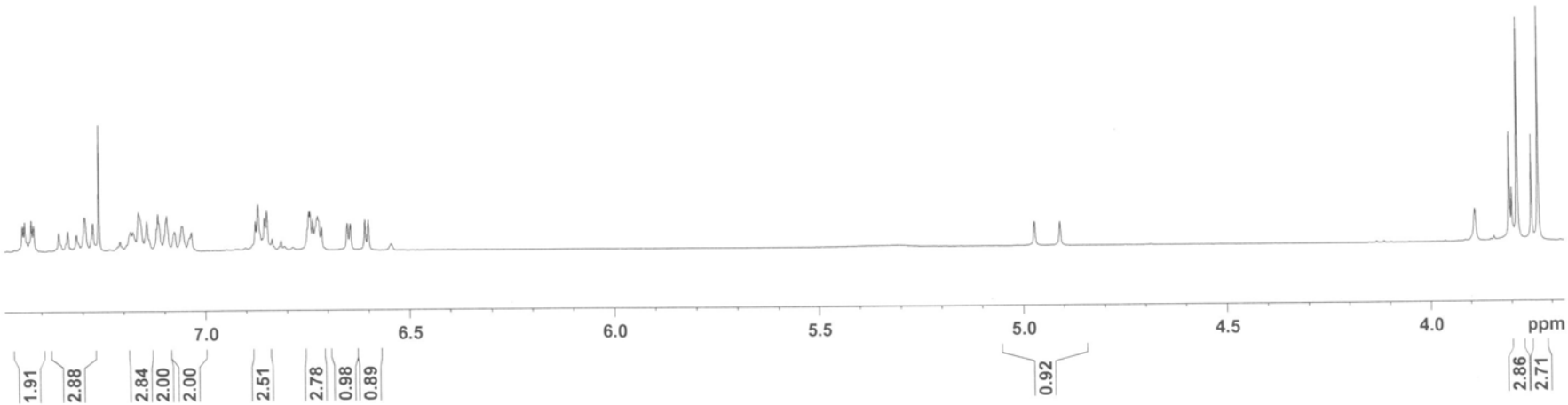


Bruker Advance III 400

NAME	ccw_1_123_109_2 t43-45
EXPNO	6
PROCNO	1
Date_	20100428
Time	9.19
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
========	CHANNEL f1 ========
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	17
SFO1	400.1318 MHz
FIDRES	240.292191 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

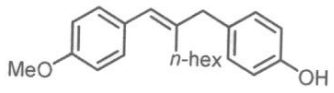
```
NAME      ccw_1_123_109_2 t28-36
EXPNO                    1
PROCNO                   1
Date_             20100427
Time                 18.53
INSTRUM              spect
PROBHD   5 mm PABBI 1H/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2768500 sec
RG                    203
DW               50.000 usec
DE                6.50 usec
TE                294.8 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  5.00 usec
PL1                 0.00 dB
PL1W          8.31434441 W
SFO1        400.1316005 MHz
SI                  65536
SF          400.1300053 MHz
WDW                    EM
SSB                     0
LB                  0.30 Hz
GB                      0
PC                  1.00
```

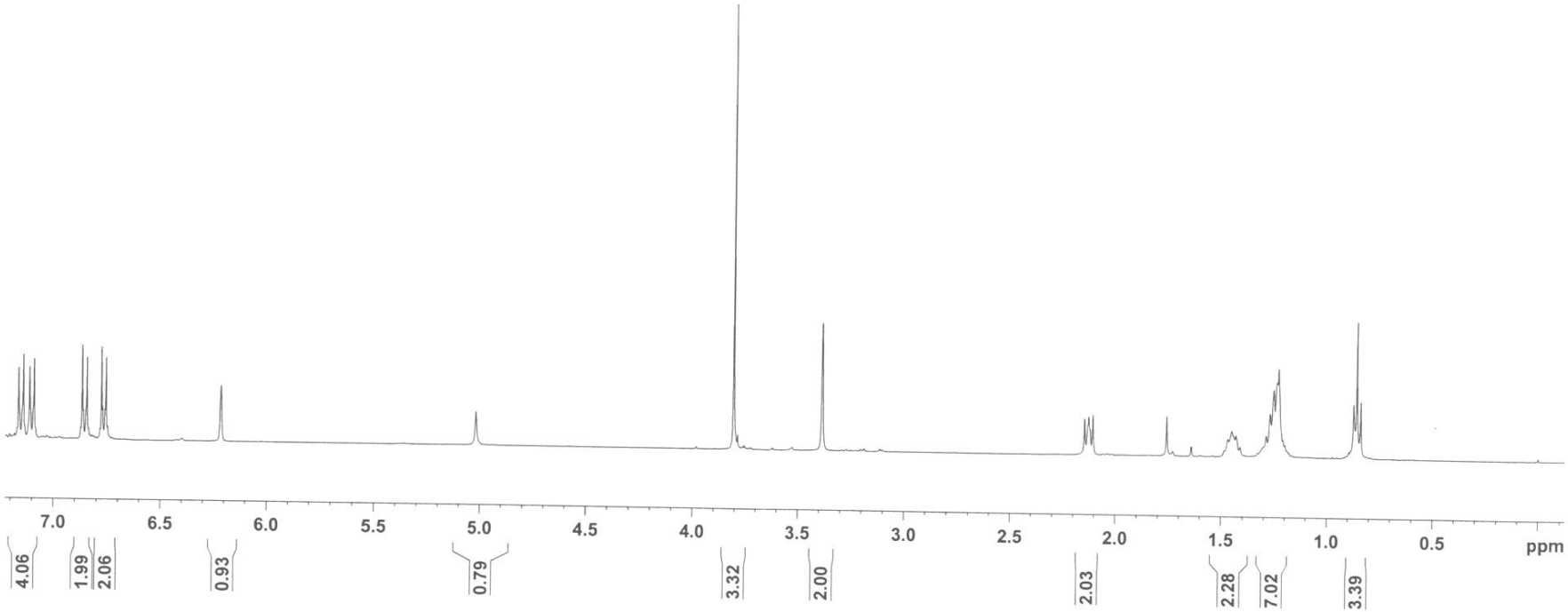




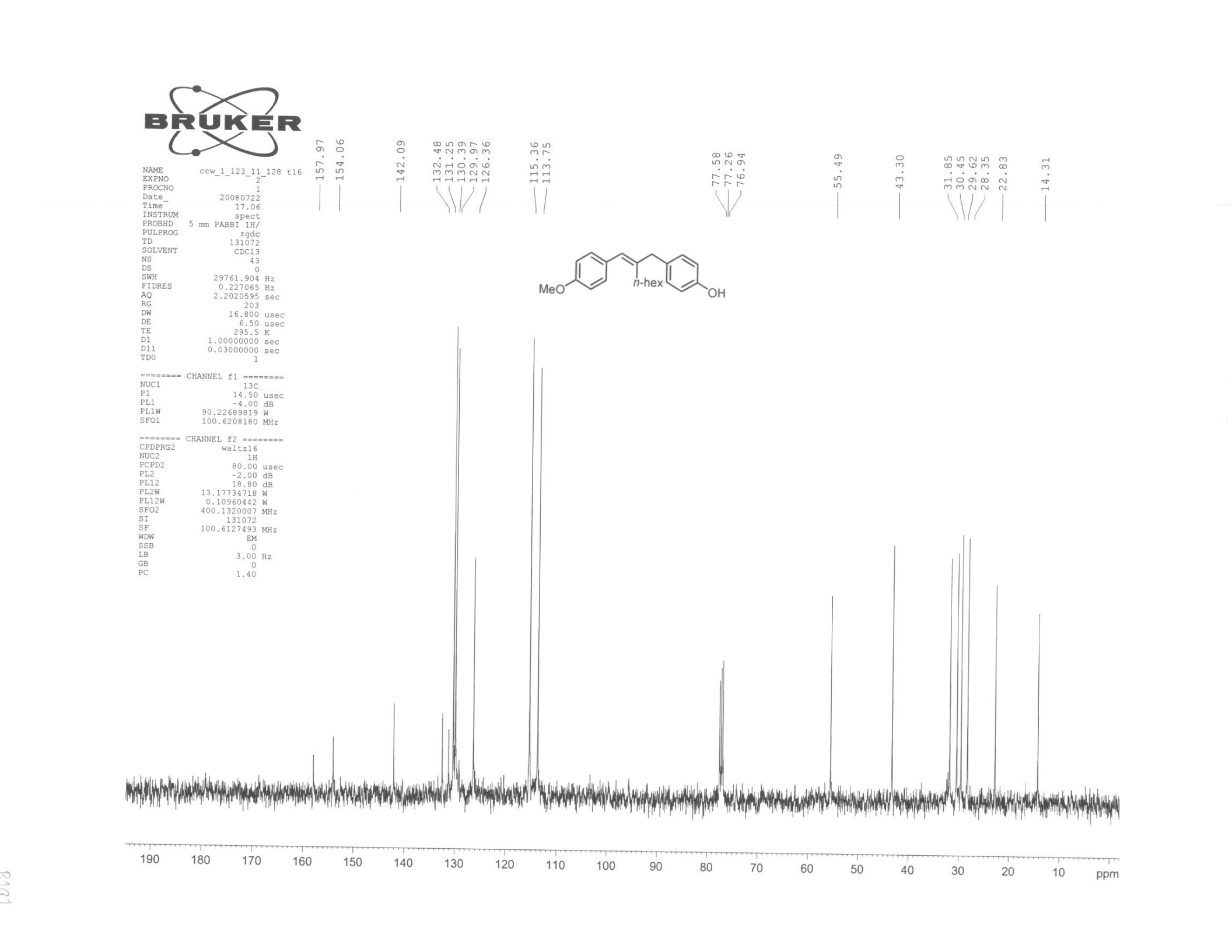
NAME	ccw_1_123_11_128 t16
EXPNO	1
PROCNO	1
Date_	20080728
Time	14.28
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg
TD	65536
SOLVENT	CDCl3
NS	3
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	32
DW	50.000 usec
DE	6.50 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1

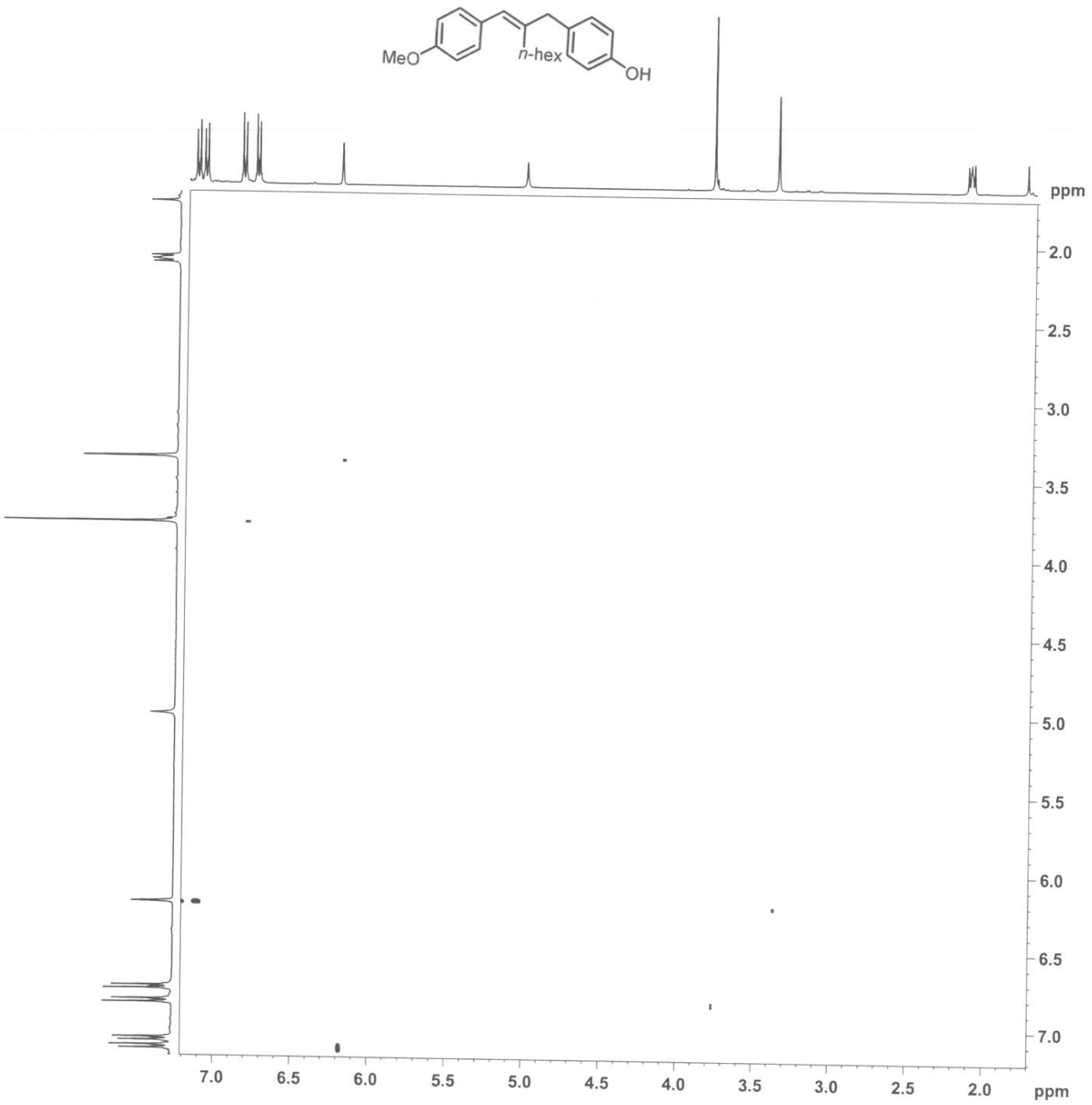
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300104 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00





B101


BRUKER
157.97
154.06
142.09
132.48
131.25
130.39
129.97
126.36
115.36
113.75
77.58
77.26
76.94
55.49
43.30
31.85
30.45
29.62
28.35
22.83
14.31
MeO
n-hex
OH
NAME ccw_1_123_11_128 t16
EXPNO 2
PROCNO 1
Date_ 20080722
Time 17.06
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 43
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127493 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



BRUKER

NAME	ccw_1_123_11_128 t16
EXPNO	6
PROCNO	1
Date_	20081011
Time	15.10
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	295.2 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	23
SFO1	400.1318 MHz
FIDRES	177.607269 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300192 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300028 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

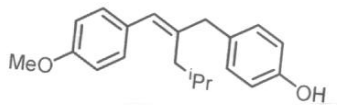
B1a1

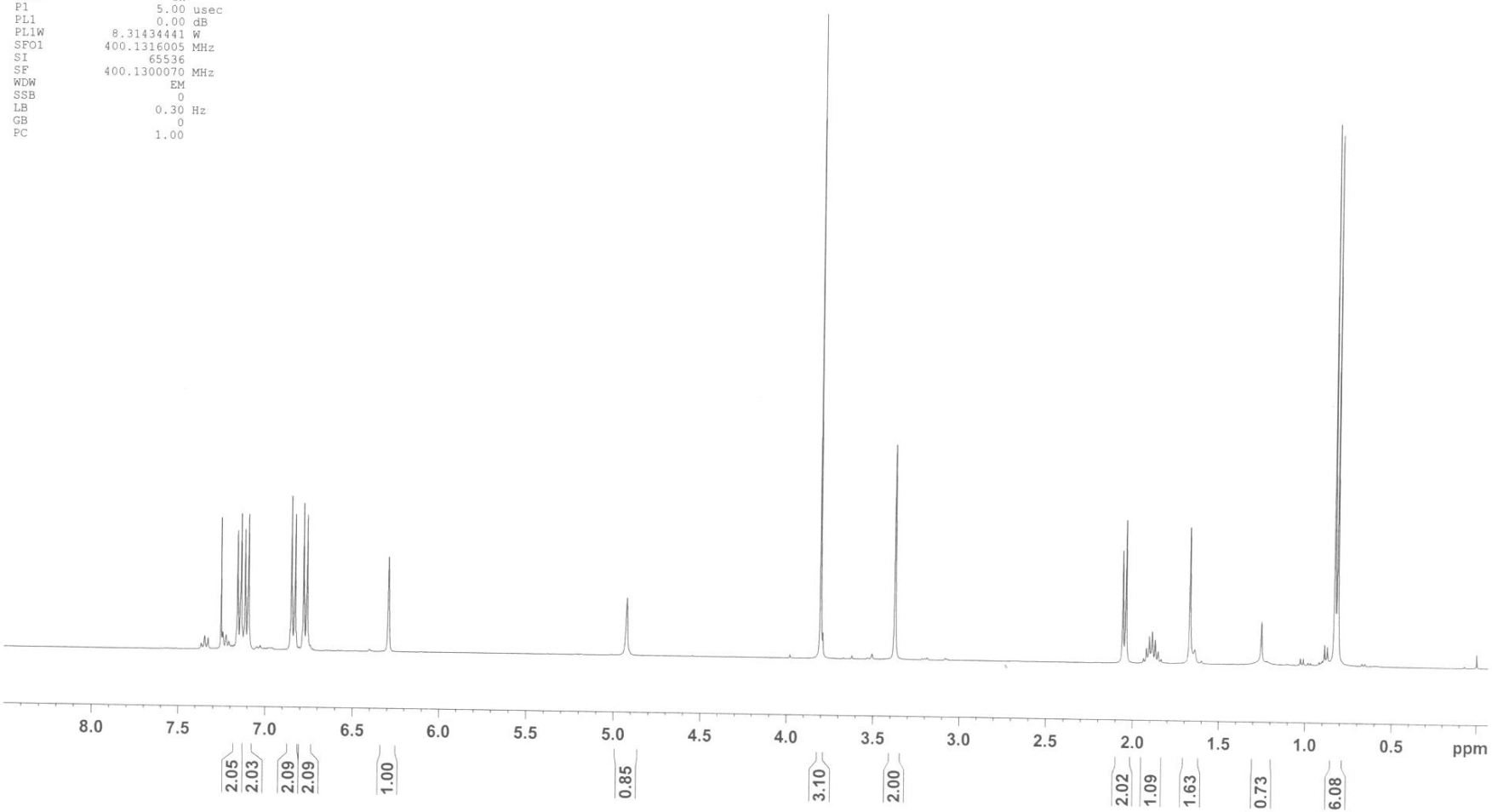
NAME	ccw_1_123_12 rp t7-8
EXPNO	1
PROCNO	1
Date_	20080818
Time	19.13
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1

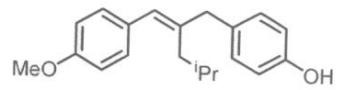
======== CHANNEL f1 ========

NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300070 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

ccw_1_123_12 rp t7-8



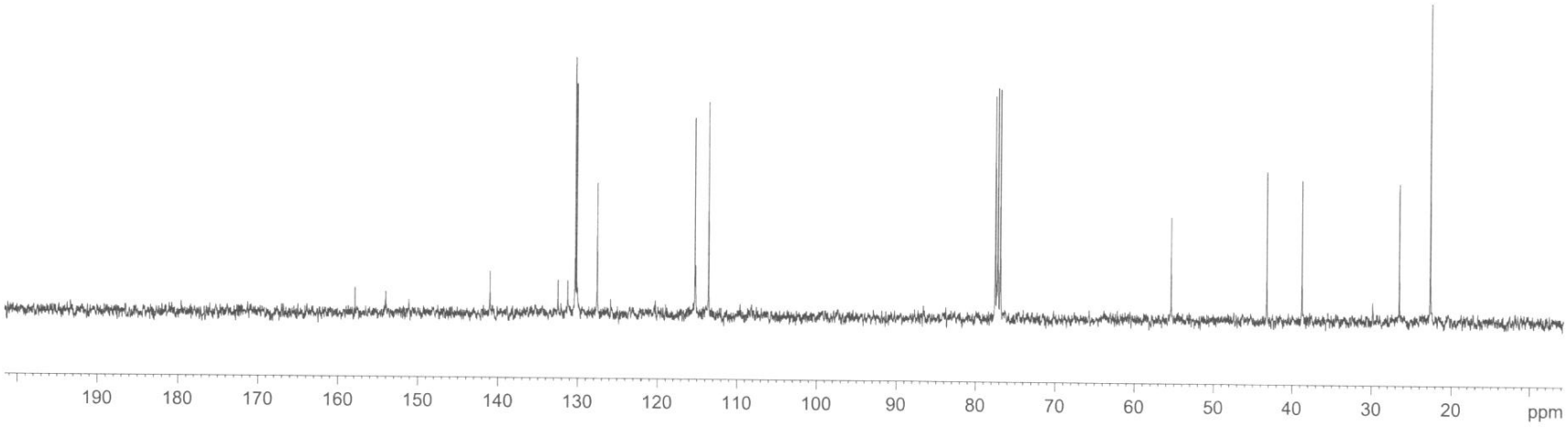




ccw_1_123_12_rp t7-8

NAME	ccw_1_123_12 rp t7-8
EXPNO	2
PROCNO	1
Date_	20080818
Time	19.17
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zgdc
TD	131072
SOLVENT	CDCl3
NS	290
DS	0
SWH	29761.904 Hz
FIDRES	0.227065 Hz
AQ	2.2020595 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	294.8 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	14.50 usec
PL1	-4.00 dB
PL1W	90.22689819 W
SFO1	100.6208180 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	18.80 dB
PL2W	13.17734718 W
PL12W	0.10960442 W
SFO2	400.1320007 MHz
SI	131072
SF	100.6127562 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40

157.86 153.98 140.98 132.45 131.24 130.29 130.13 127.54 115.27 113.56 77.48 77.16 76.84 55.36 43.23 38.76 26.53 22.65



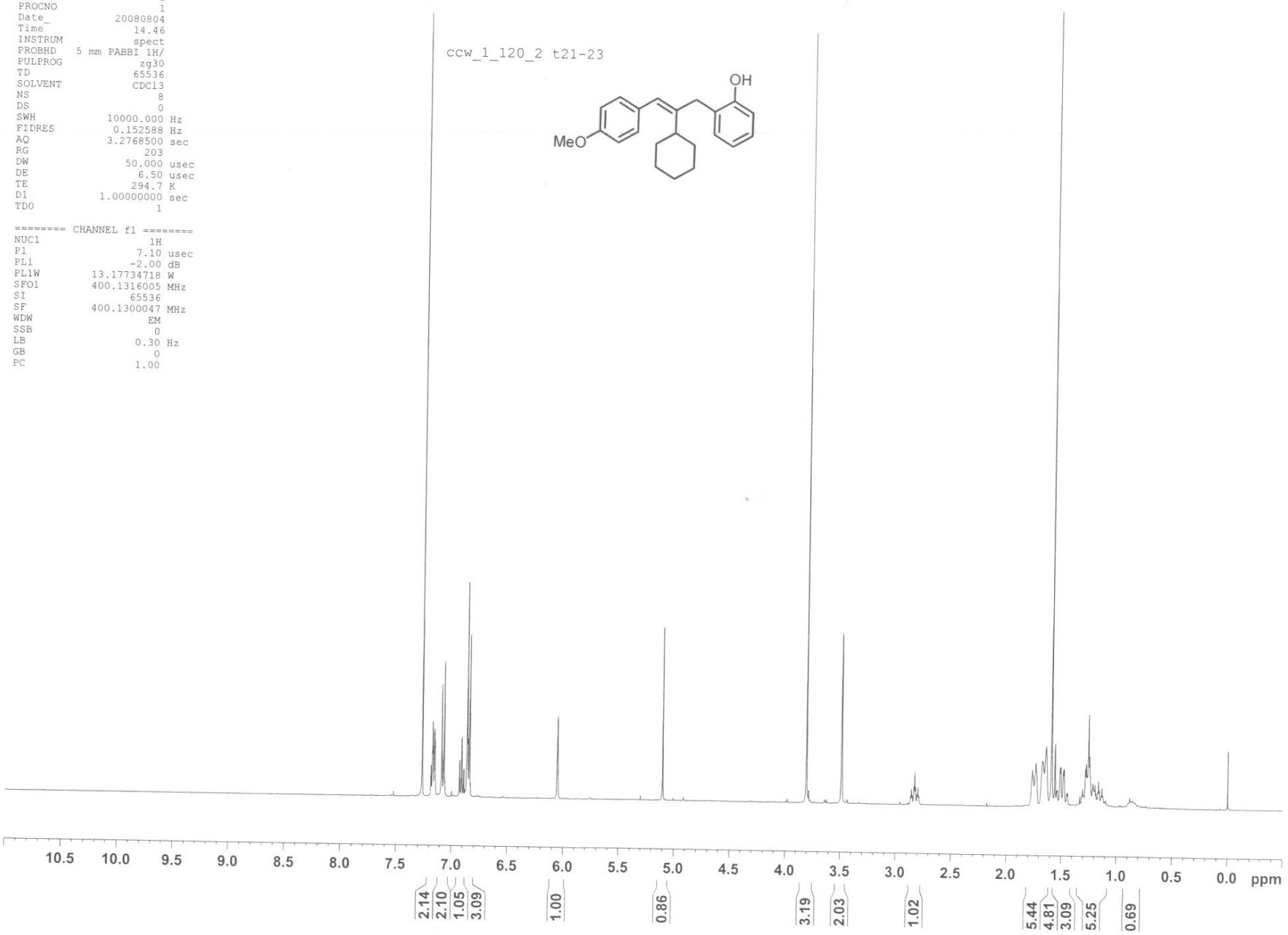
BRUKER

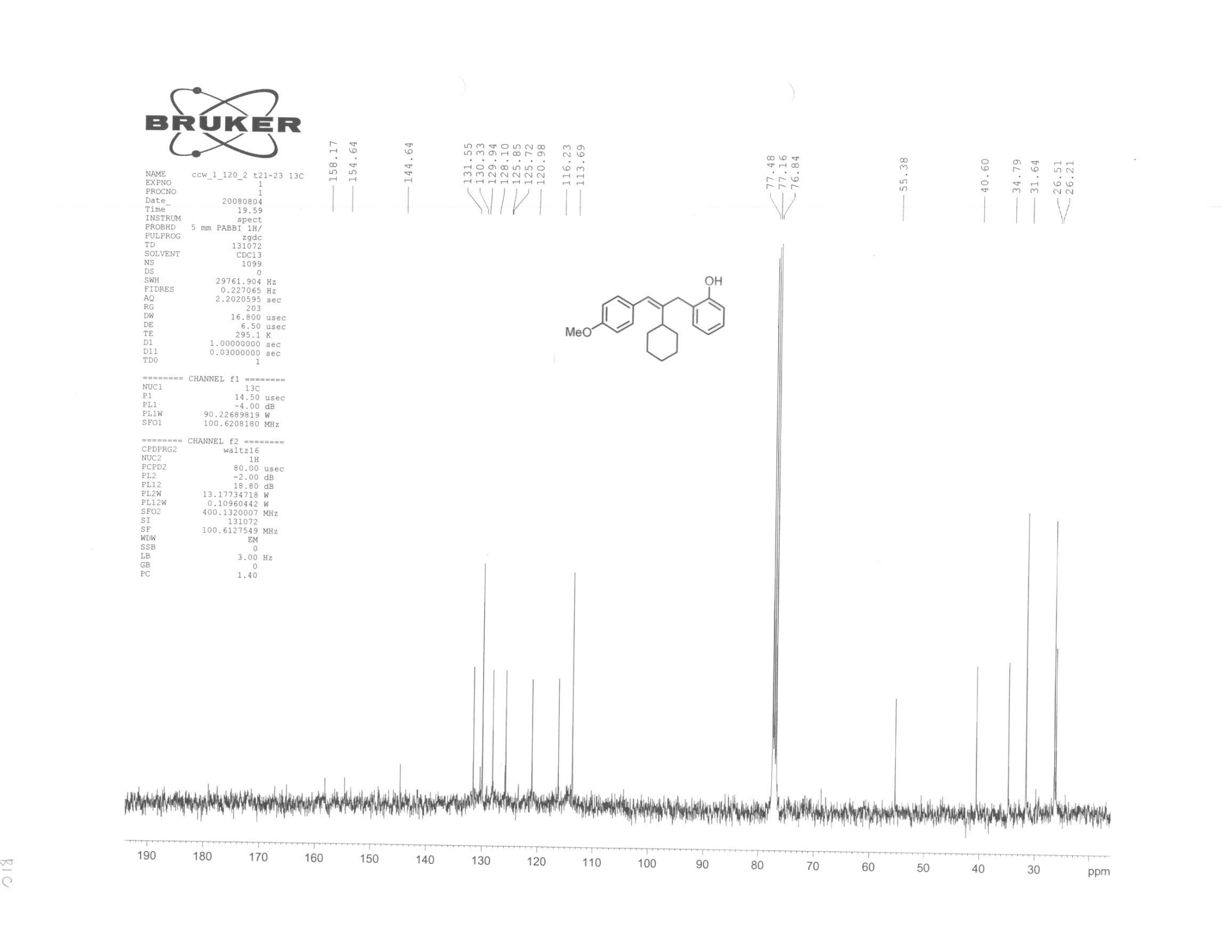
NAME	ccw_1_123_12 rp t7-8
EXPNO	6
PROCNO	1
Date_	20080818
Time	19.34
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

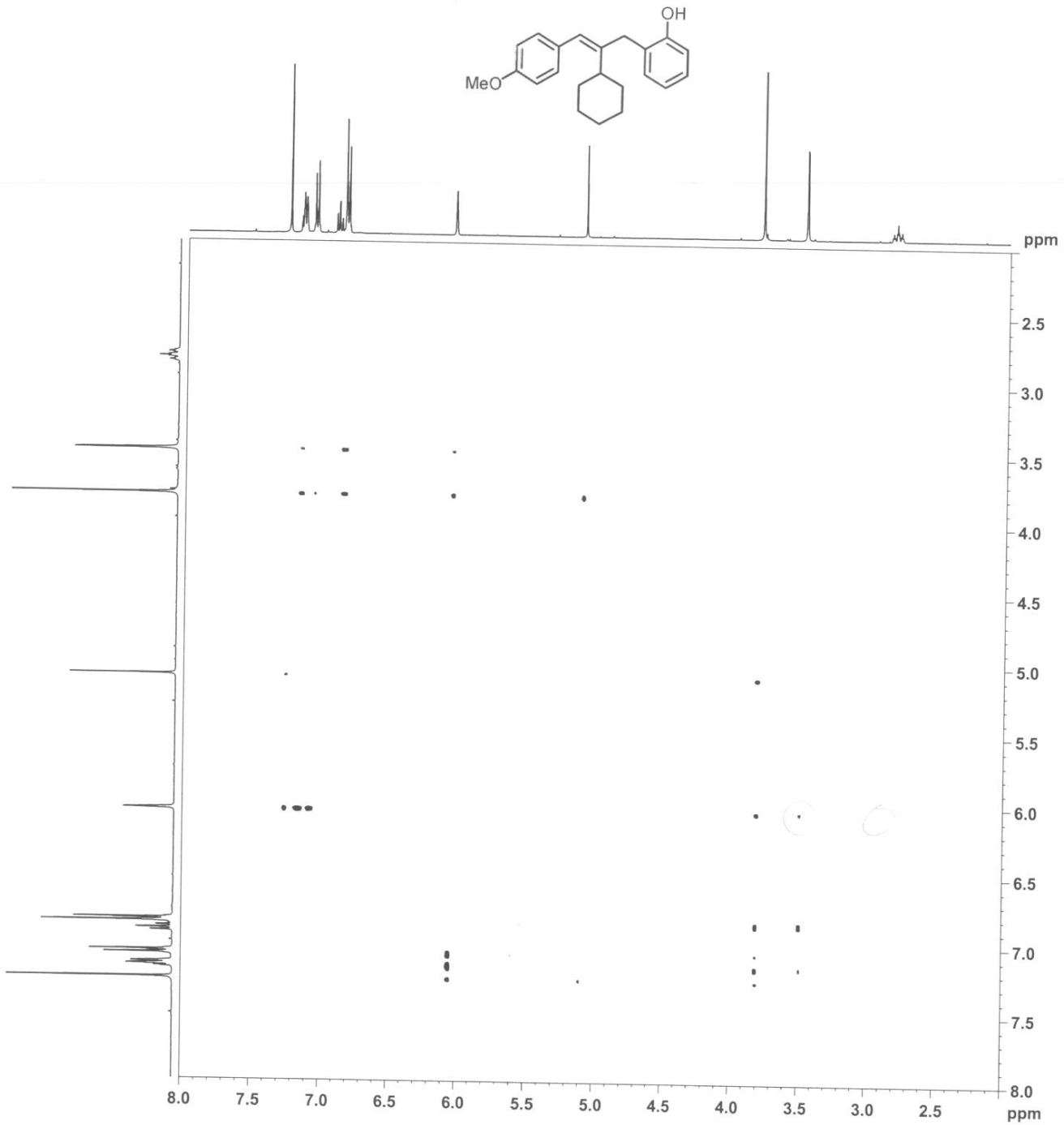
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	25
SFO1	400.1318 MHz
FIDRES	163.398697 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

NAME	ccw_1_120_2 t21-23
EXPNO	1
PROCNO	1
Date_	20080804
Time	14.46
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.7 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300047 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00




BRUKER
158.17
154.64
144.64
131.55
130.33
129.94
128.10
125.85
125.72
120.98
116.23
113.69
77.48
77.16
76.84
55.38
40.60
34.79
31.64
26.51
26.21
OH
MeO
NAME ccw_1_120_2 t21-23 13C
EXPNO 1
PROCNO 1
Date_ 20080804
Time 19.59
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 1099
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127549 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 ppm



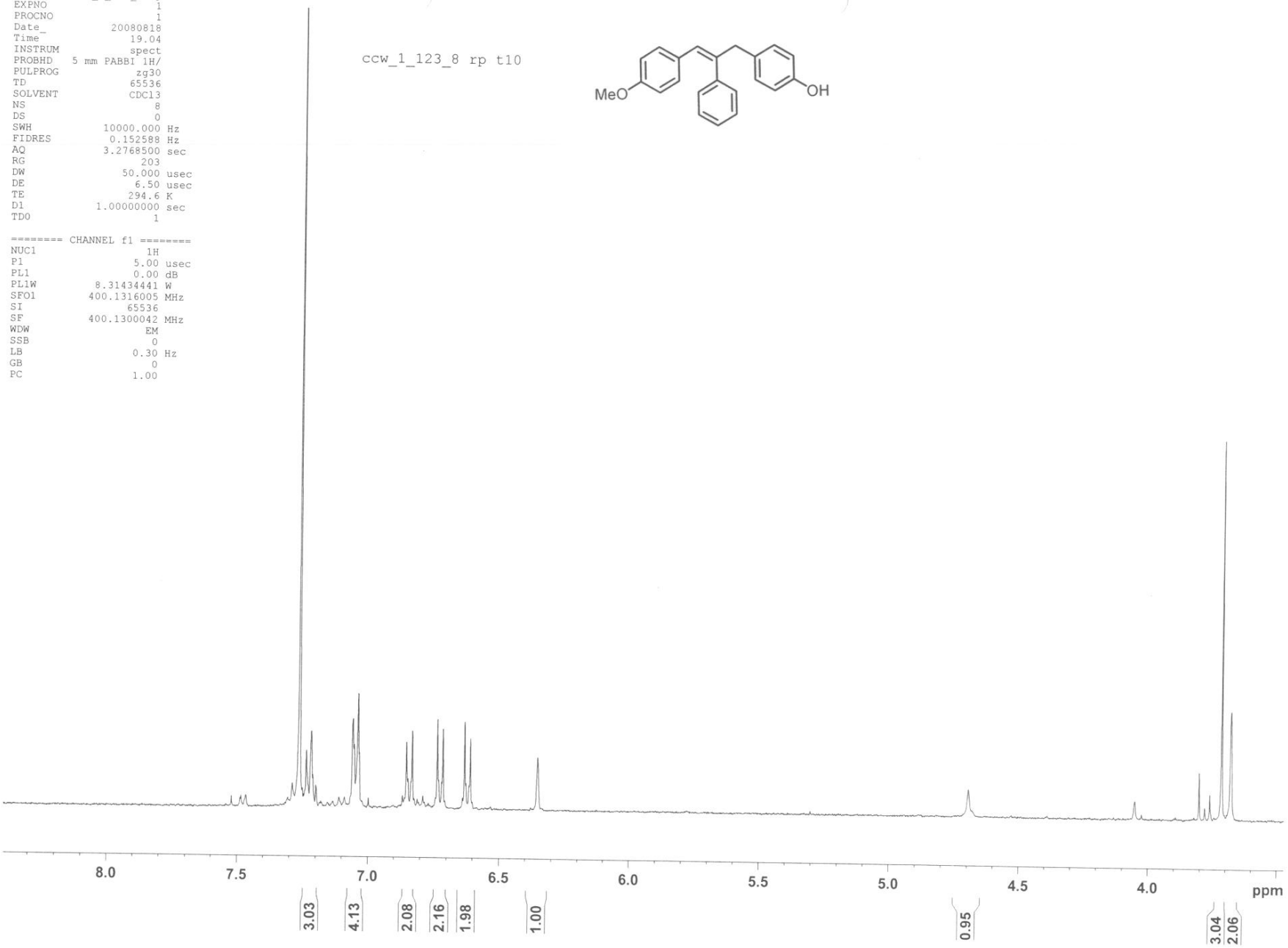
BRUKER

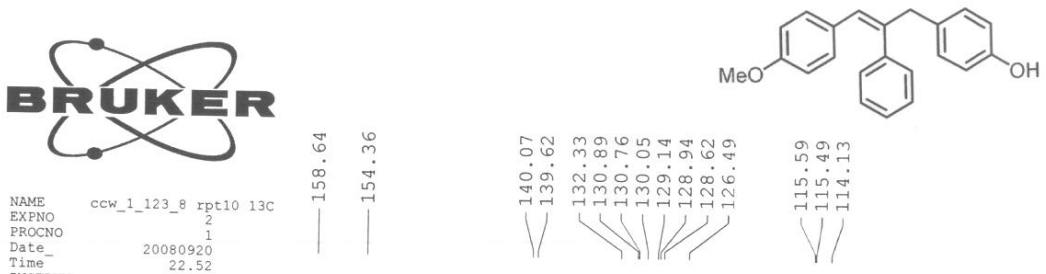
NAME	ccw_1_120_2 t21-23
EXPNO	6
PROCNO	1
Date_	20080804
Time	14.48
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	19
SFO1	400.1318 MHz
FIDRES	214.998276 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300029 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300028 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

NAME	ccw_1_123_8 rp t10
EXPNO	1
PROCNO	1
Date_	20080818
Time	19.04
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300042 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

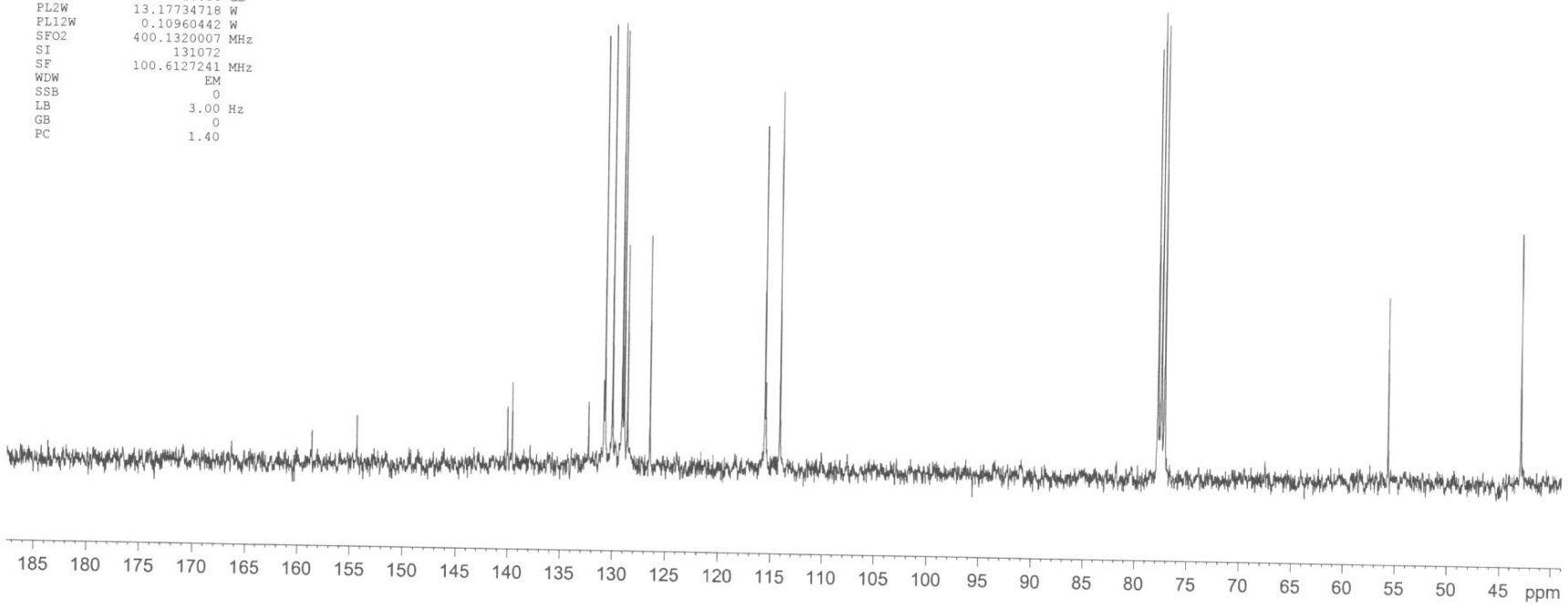
ccw_1_123_8 rp t10

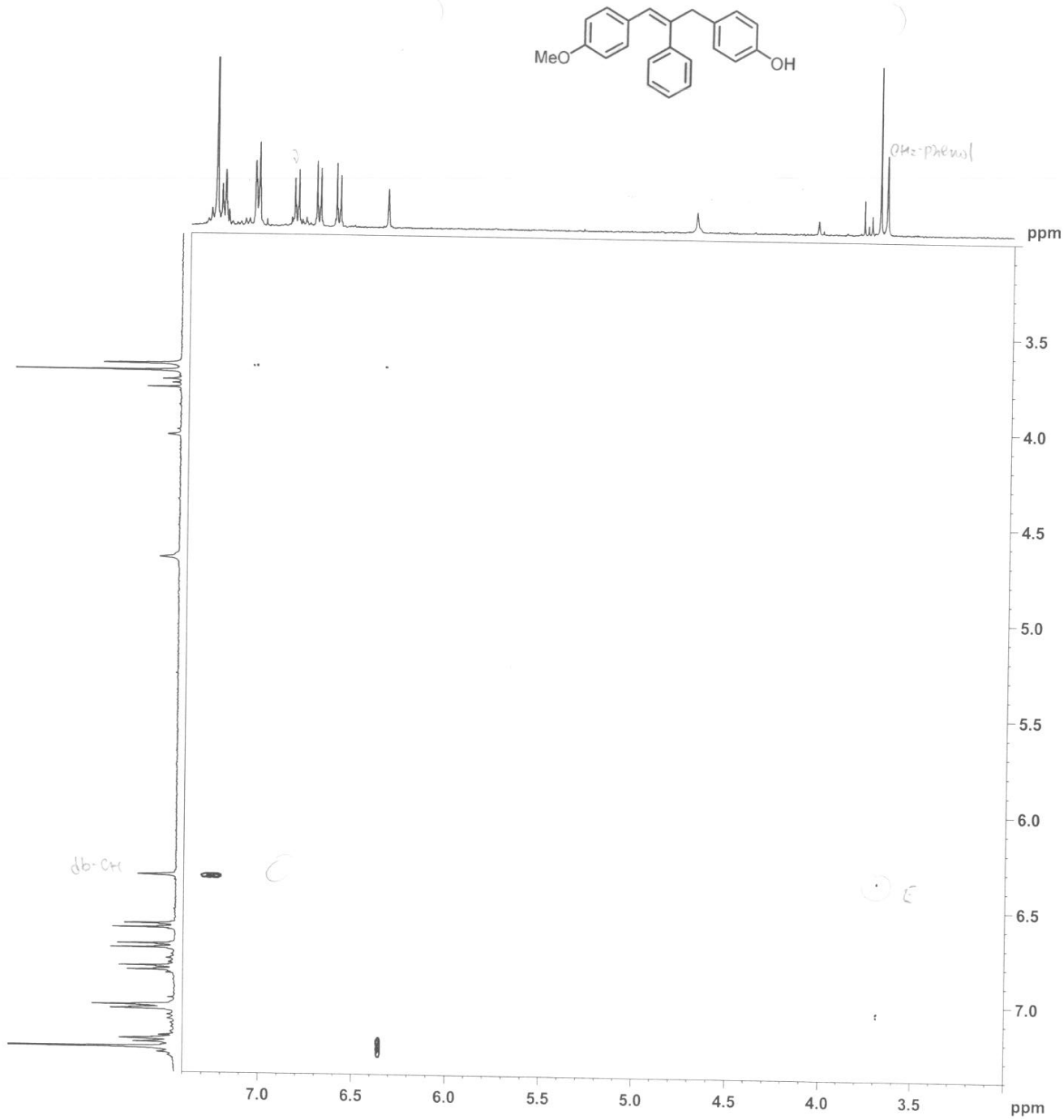




158.64
154.36
140.07
139.62
132.33
130.89
130.76
130.05
129.14
128.94
128.62
126.49
115.59
115.49
114.13
77.48
77.16
76.84
55.71
42.96

NAME	ccw_1_123_8 rpt10 13C
EXPNO	2
PROCNO	1
Date_	20080920
Time	22.52
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zgdc
TD	131072
SOLVENT	CDCl3
NS	32549
DS	0
SWH	29761.904 Hz
FIDRES	0.227065 Hz
AQ	2.2020595 sec
RG	203
DW	16.800 usec
DE	6.50 usec
TE	294.8 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	14.50 usec
PL1	-4.00 dB
PL1W	90.22689819 W
SFO1	100.6208180 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-2.00 dB
PL12	18.80 dB
PL2W	13.17734718 W
PL12W	0.10960442 W
SFO2	400.1320007 MHz
SI	131072
SF	100.6127241 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.40





BRUKER

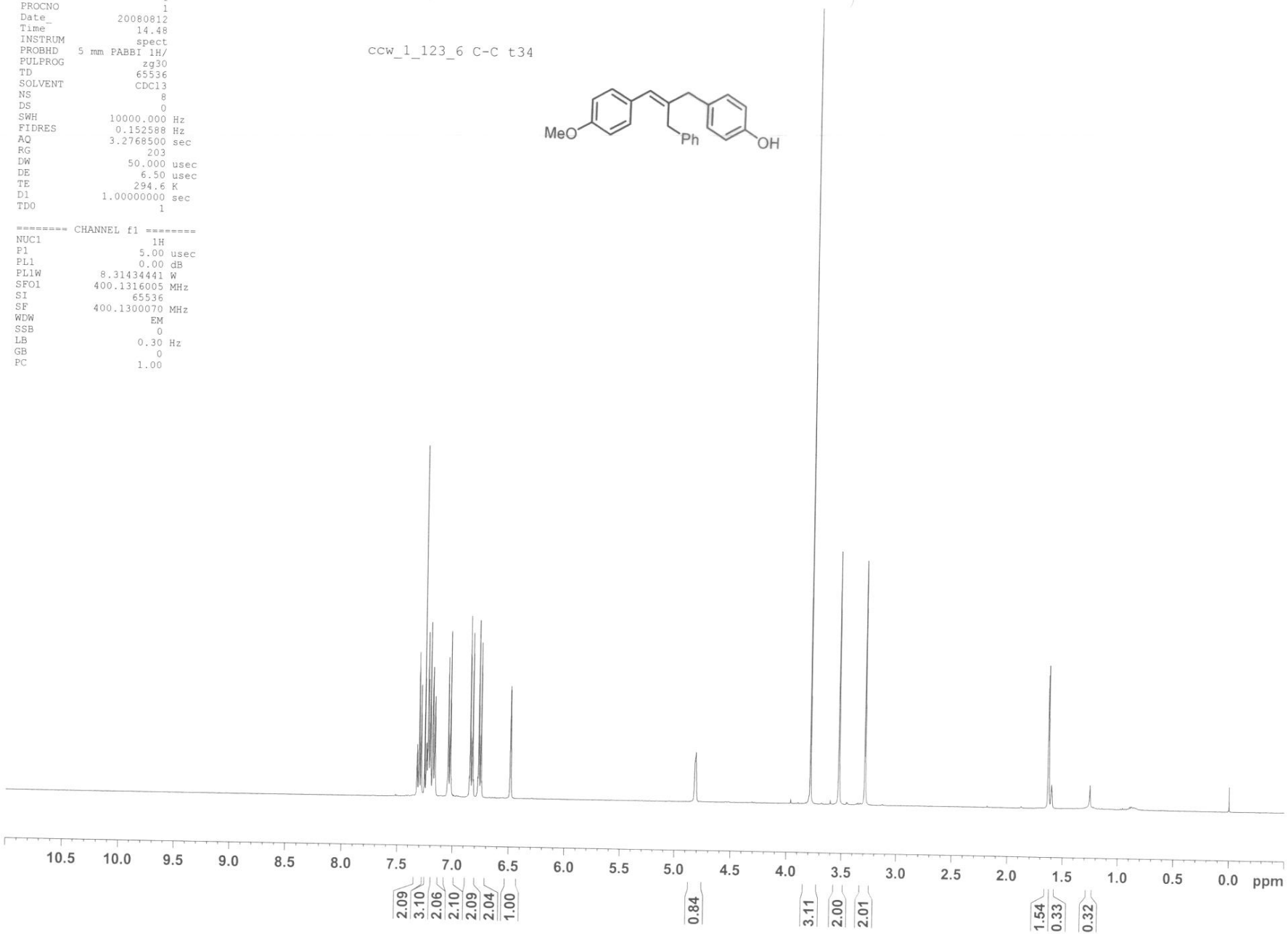
NAME	ccw_1_123_8 rp t10
EXPNO	6
PROCNO	1
Date_	20080820
Time	10.00
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

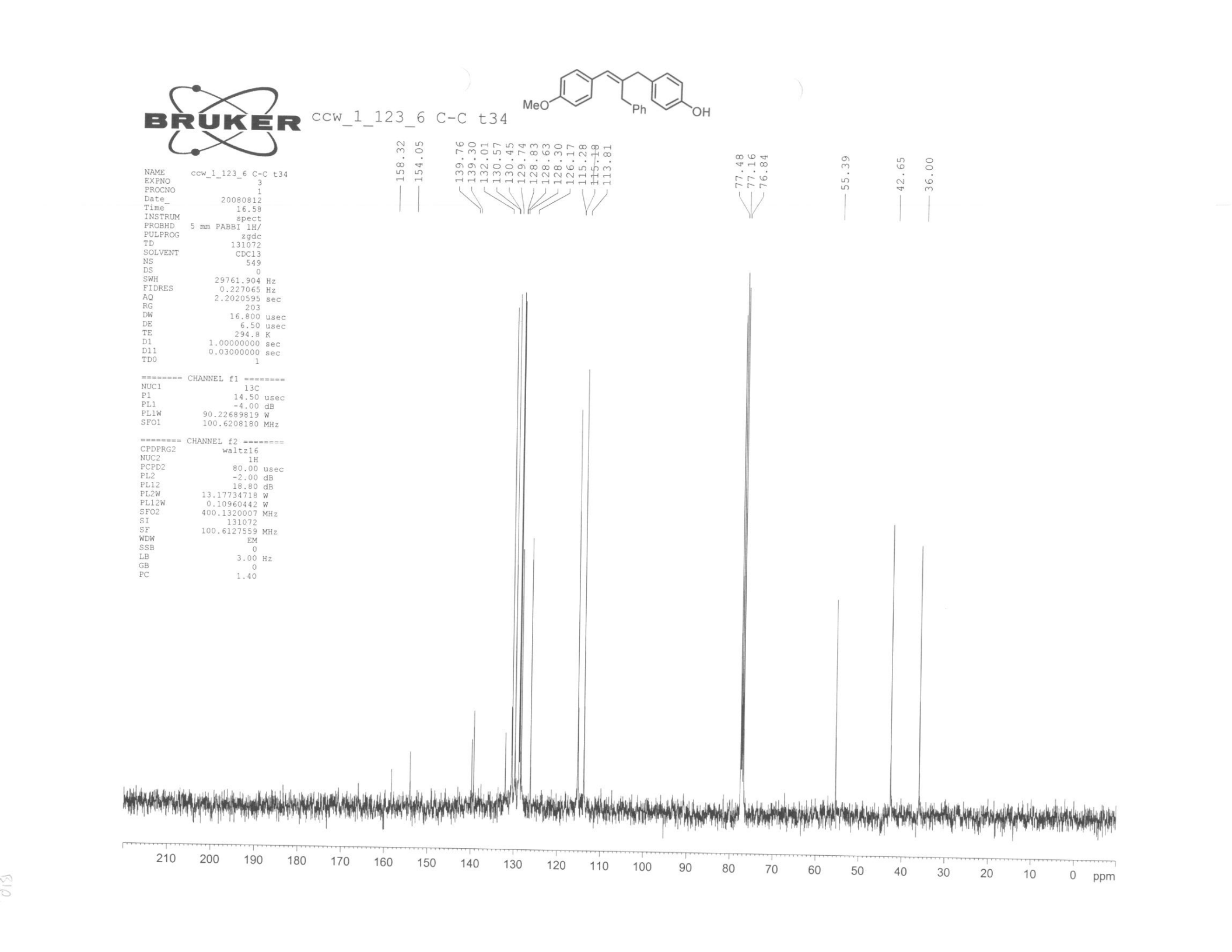
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	26
SFO1	400.1318 MHz
FIDRES	157.114120 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

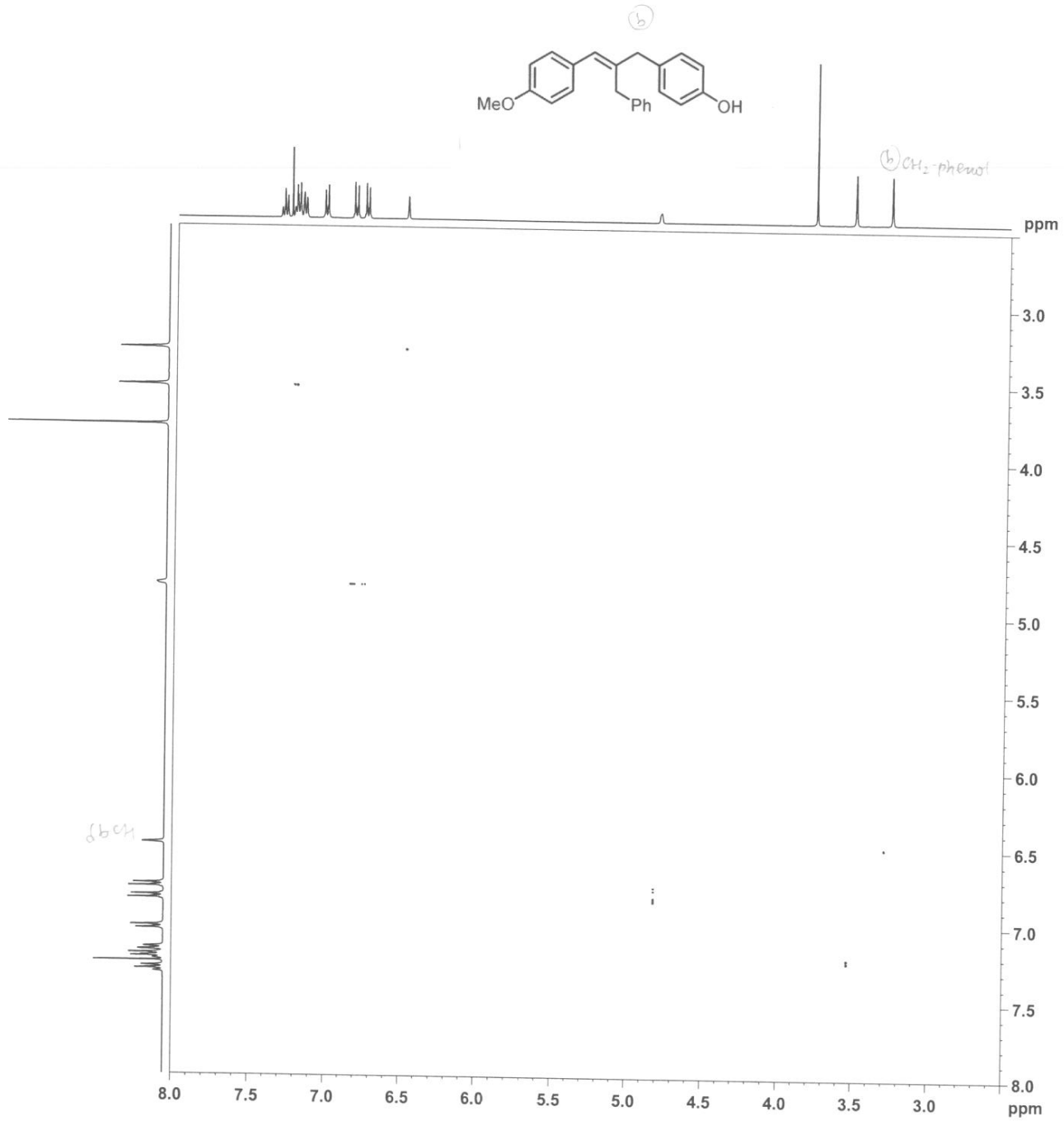
NAME	ccw_1_123_6 C-C t34
EXPNO	1
PROCNO	1
Date_	20080812
Time	14.48
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300070 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

ccw_1_123_6 C-C t34



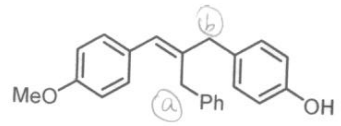

BRUKER
ccw_1_123_6 C-C t34
MeO
Ph
OH
158.32
154.05
139.76
139.30
132.01
130.57
130.45
129.74
128.83
128.63
128.30
126.17
115.28
115.18
113.81
77.48
77.16
76.84
55.39
42.65
36.00
NAME ccw_1_123_6 C-C t34
EXPNO 3
PROCNO 1
Date_ 20080812
Time 16.58
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 549
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127559 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

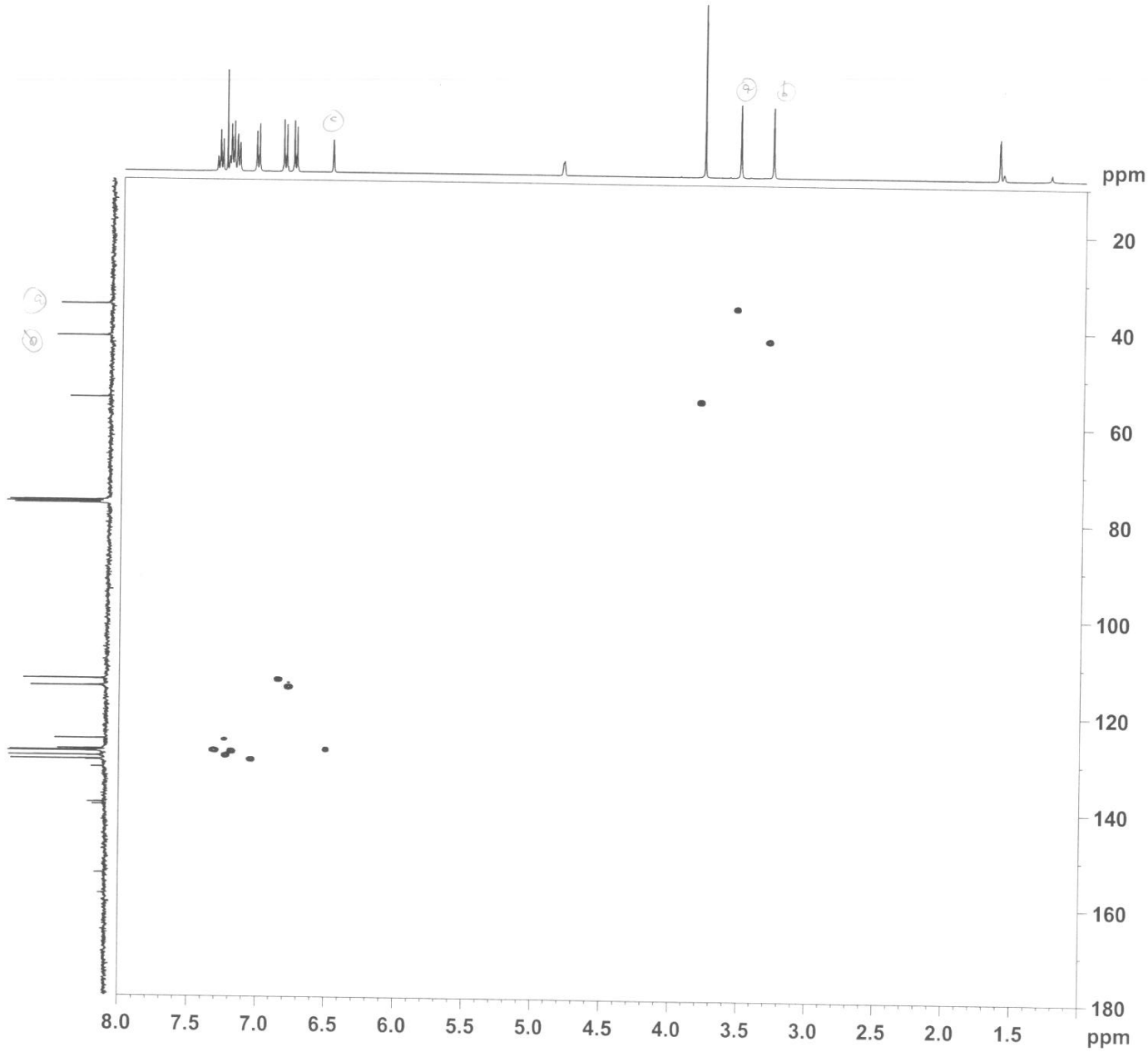


BRUKER

NAME	ccw_1_123_6 C-C t34
EXPNO	6
PROCNO	1
Date_	20080812
Time	15.01
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	10
SFO1	400.1318 MHz
FIDRES	408.496735 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300049 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

ccw_1_123_6 C-C t34





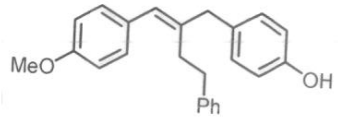
BRUKER

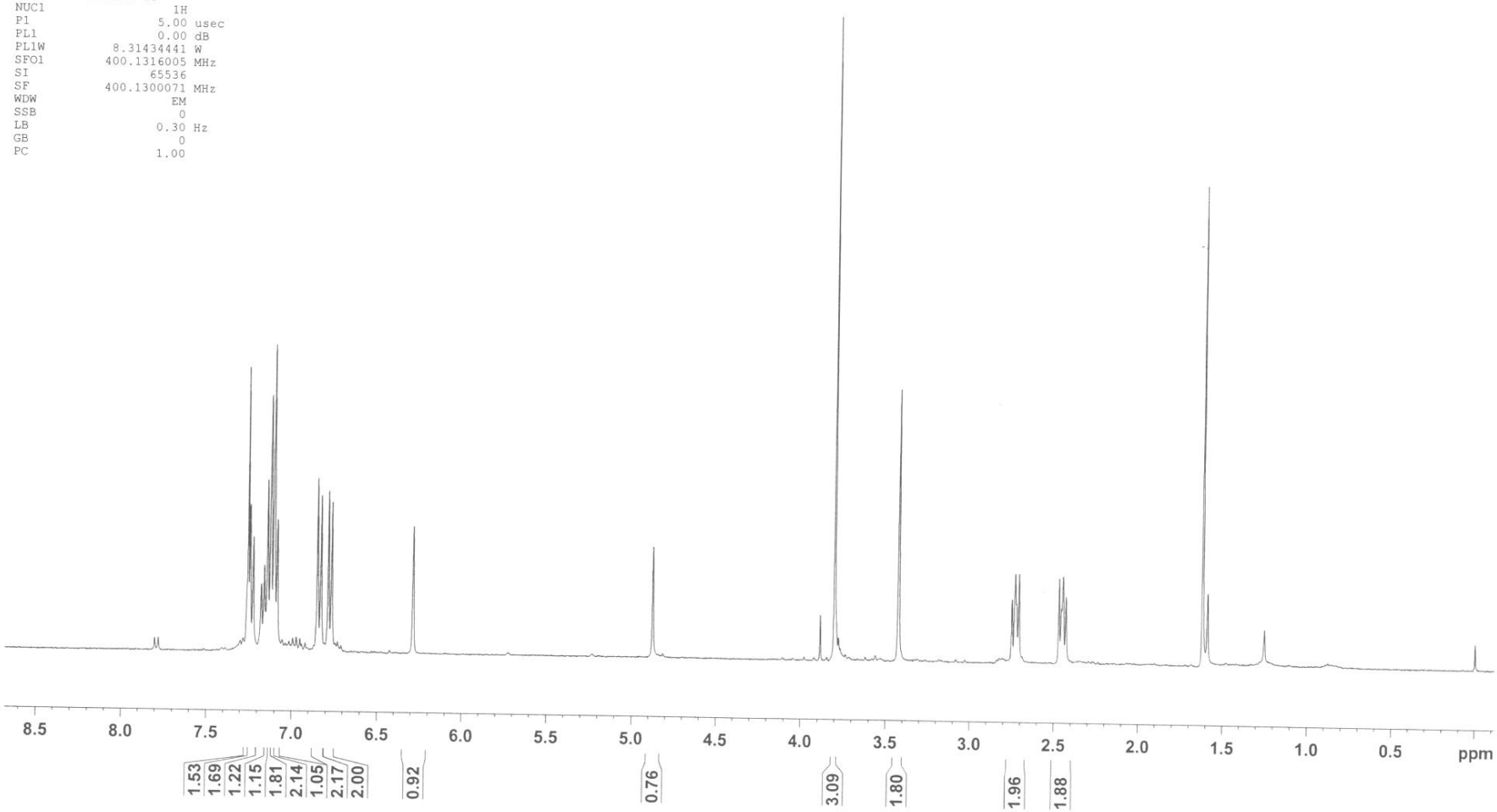
NAME	ccw_1_123_6 C-C t34
EXPNO	4
PROCNO	1
Date_	20080823
Time	17.02
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	hsqcetgp
TD	1024
SOLVENT	CDCl3
NS	2
DS	16
SWH	5341.880 Hz
FIDRES	5.216680 Hz
AQ	0.0958964 sec
RG	203
DW	93.600 usec
DE	6.50 usec
TE	294.8 K
CNST2	145.0000000
D0	0.00000300 sec
D1	1.50000000 sec
D4	0.00172414 sec
D11	0.03000000 sec
D13	0.00000400 sec
D16	0.00020000 sec
IN0	0.00003000 sec
ZGOPTNS	
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
P2	14.20 usec
P28	1000.00 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1324057 MHz
======== CHANNEL f2 ========	
CPDPRG2	garp
NUC2	13C
P3	13.20 usec
P4	26.40 usec
PCPD2	70.00 usec
PL2	-4.00 dB
PL12	10.00 dB
PL2W	90.22689819 W
PL12W	3.59199762 W
SFO2	100.6202727 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPZ1	80.00 %
GPZ2	20.10 %
P16	1000.00 usec
NDO	2
TD	256
SFO1	100.6203 MHz
FIDRES	65.104164 Hz
SW	165.639 ppm
FnMODE	Echo-Antiecho
SI	1024
SF	400.1300000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	echo-antiecho
SF	100.6127690 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

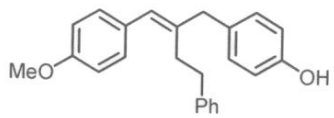
NAME	ccw_1_123_16 rp t10
EXPNO	1
PROCNO	1
Date_	20080819
Time	21.06
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.8 K
D1	1.00000000 sec
TD0	1

======== CHANNEL f1 ========	
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300071 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

ccw_1_123_16 t10







ppm

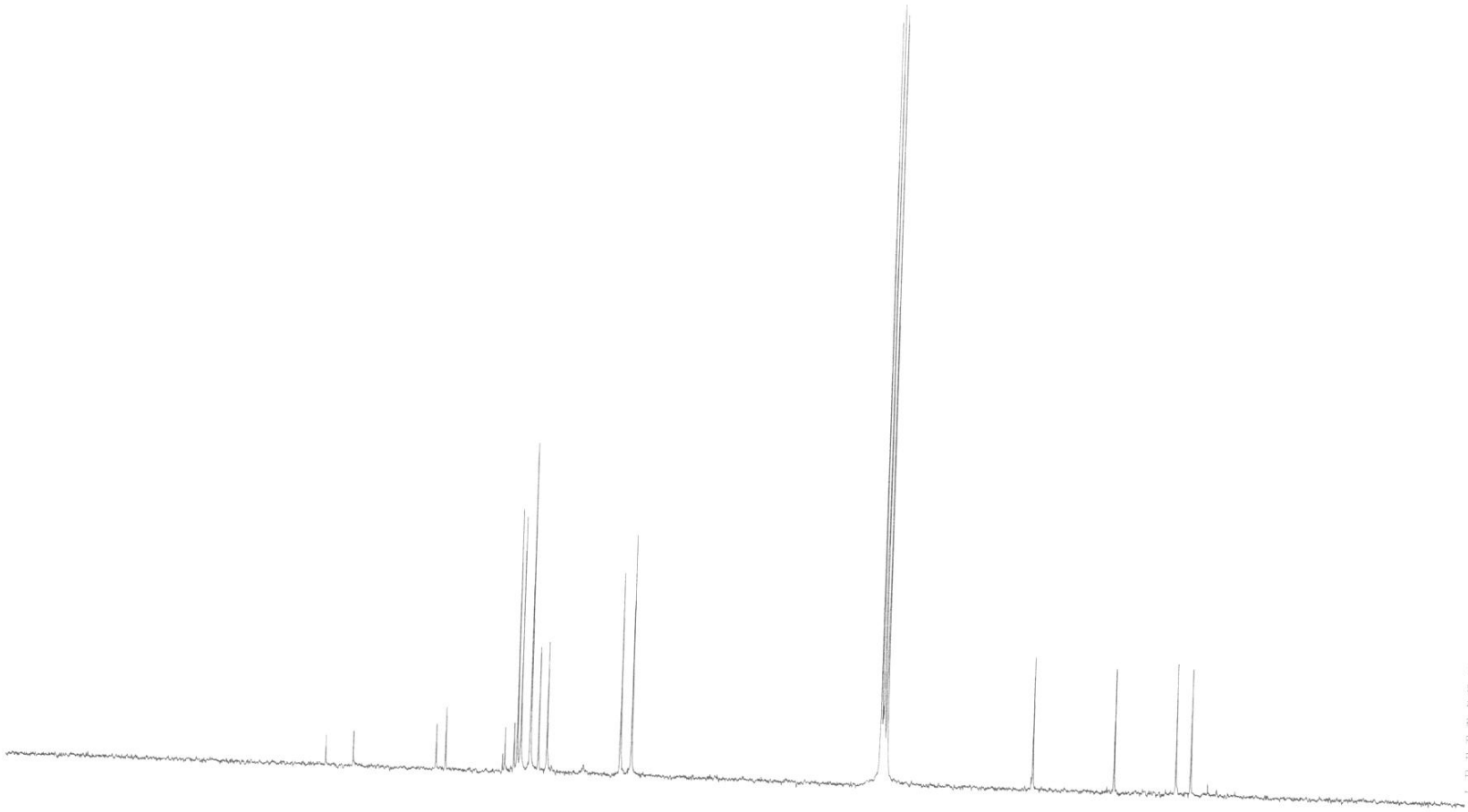
158.088
154.106
142.068
140.704
132.430
132.115
130.783
130.327
129.809
128.452
128.415
127.218
125.996
115.341
113.698

77.582
77.359
77.159
76.735

55.397

43.518

34.475
32.330



ppm 180 160 140 120 100 80 60 40 20 0

Current Data Parameters
NAME ccw_1_123_16 rp t10 13C
EXPNO 2
PROCNO 1

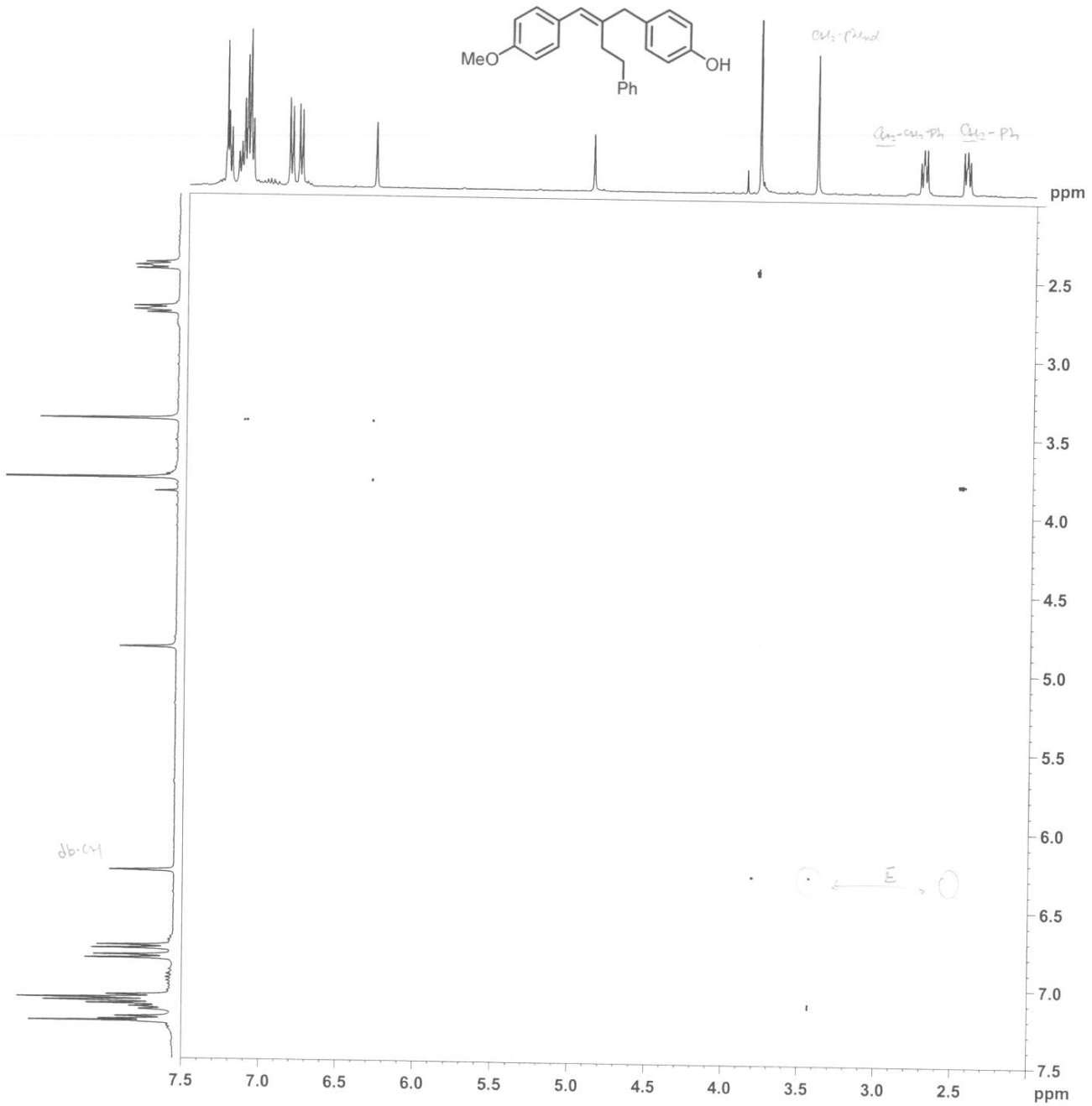
F2 - Acquisition Parameters
Date_ 20080819
Time 22.49
INSTRUM dpx300
PROBHD 5 mm BBO BB-1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 14265
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 9195.2
DW 22.050 usec
DE 6.00 usec
TE 295.2 K
D1 1.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 3.00 usec
PL1 -6.00 dB
SF01 75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 19.00 dB
SF02 300.1315007 MHz

F2 - Processing parameters
SI 65536
SF 75.4677400 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 23.00 cm
CY 12.00 cm
F1P 204.375 ppm
F1 15423.75 Hz
F2P -7.698 ppm
F2 -580.97 Hz
PPMCM 9.22060 ppm/cm
HZCM 695.85767 Hz/cm

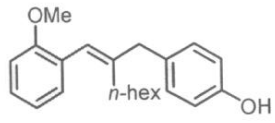


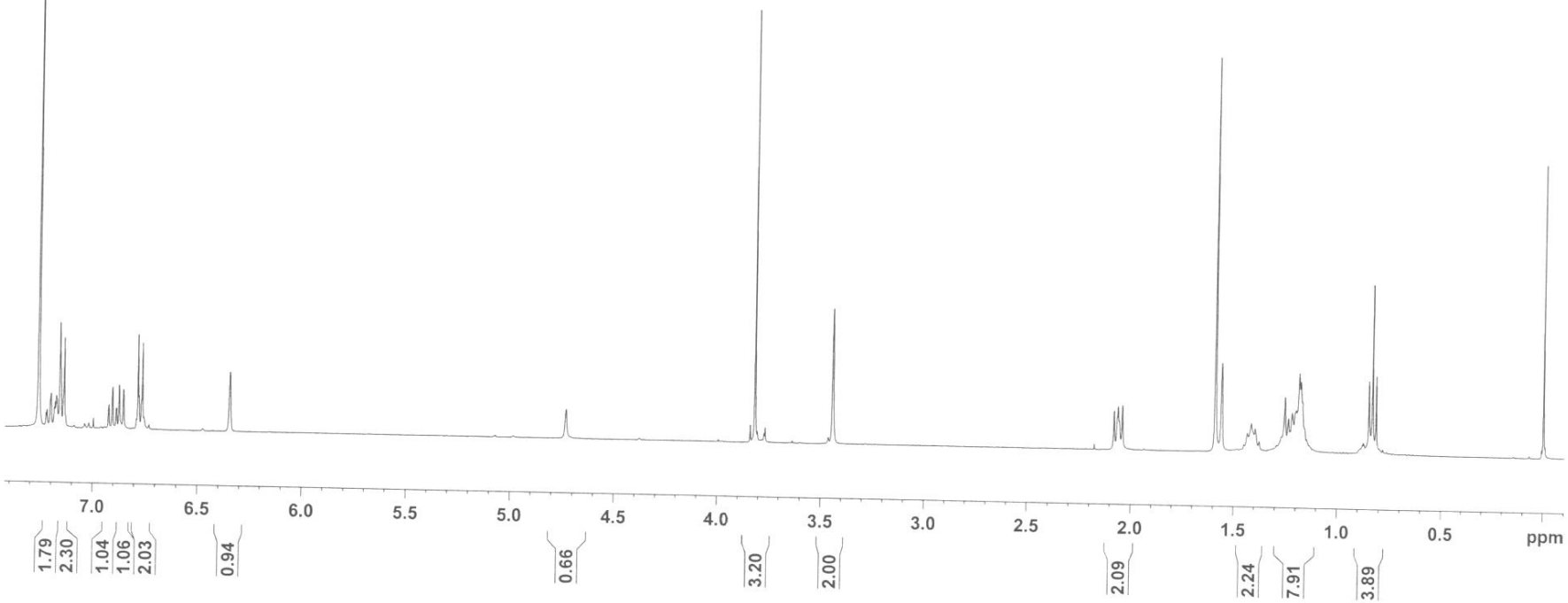
BRUKER

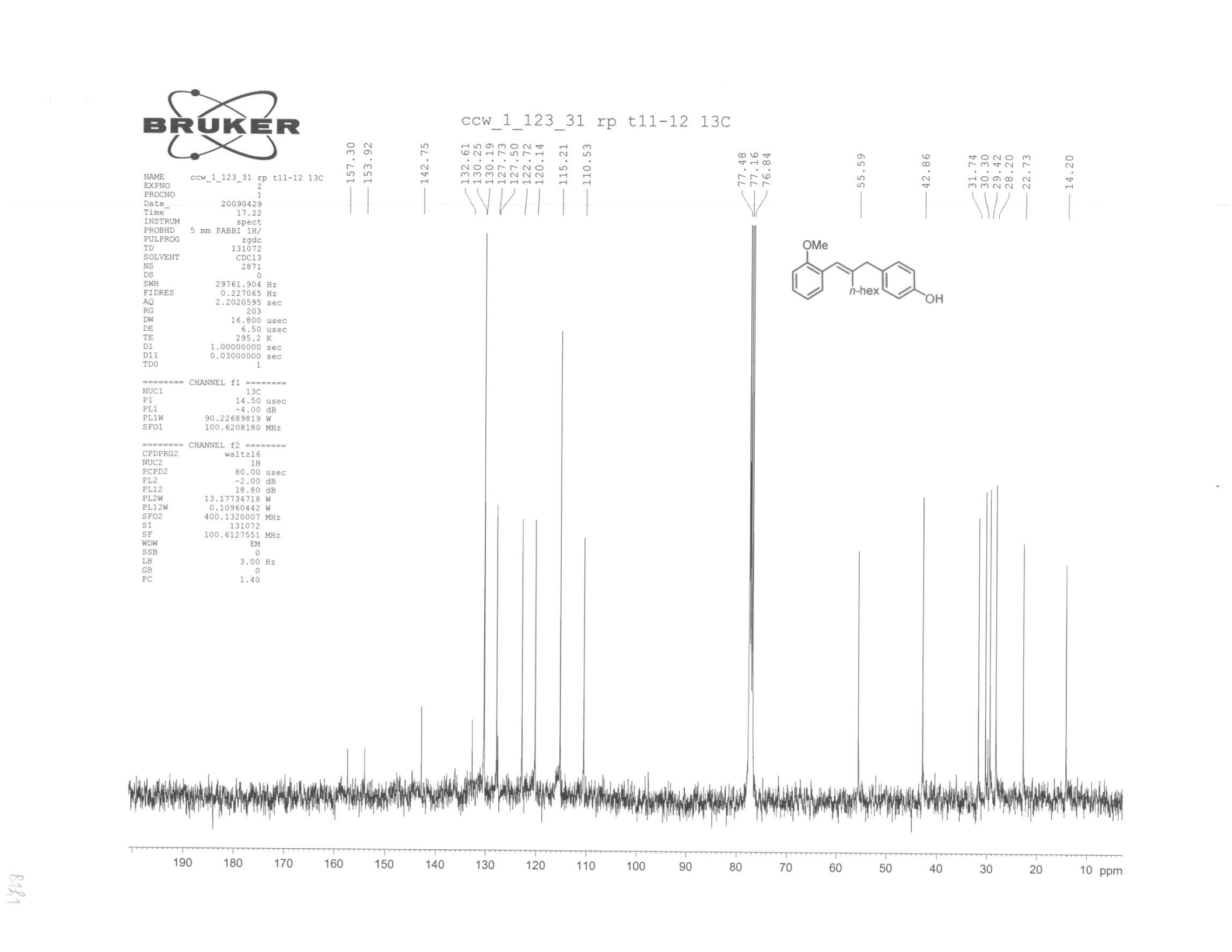
NAME	ccw_1_123_16 rp t10
EXPNO	6
PROCNO	1
Date_	20080820
Time	10.25
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

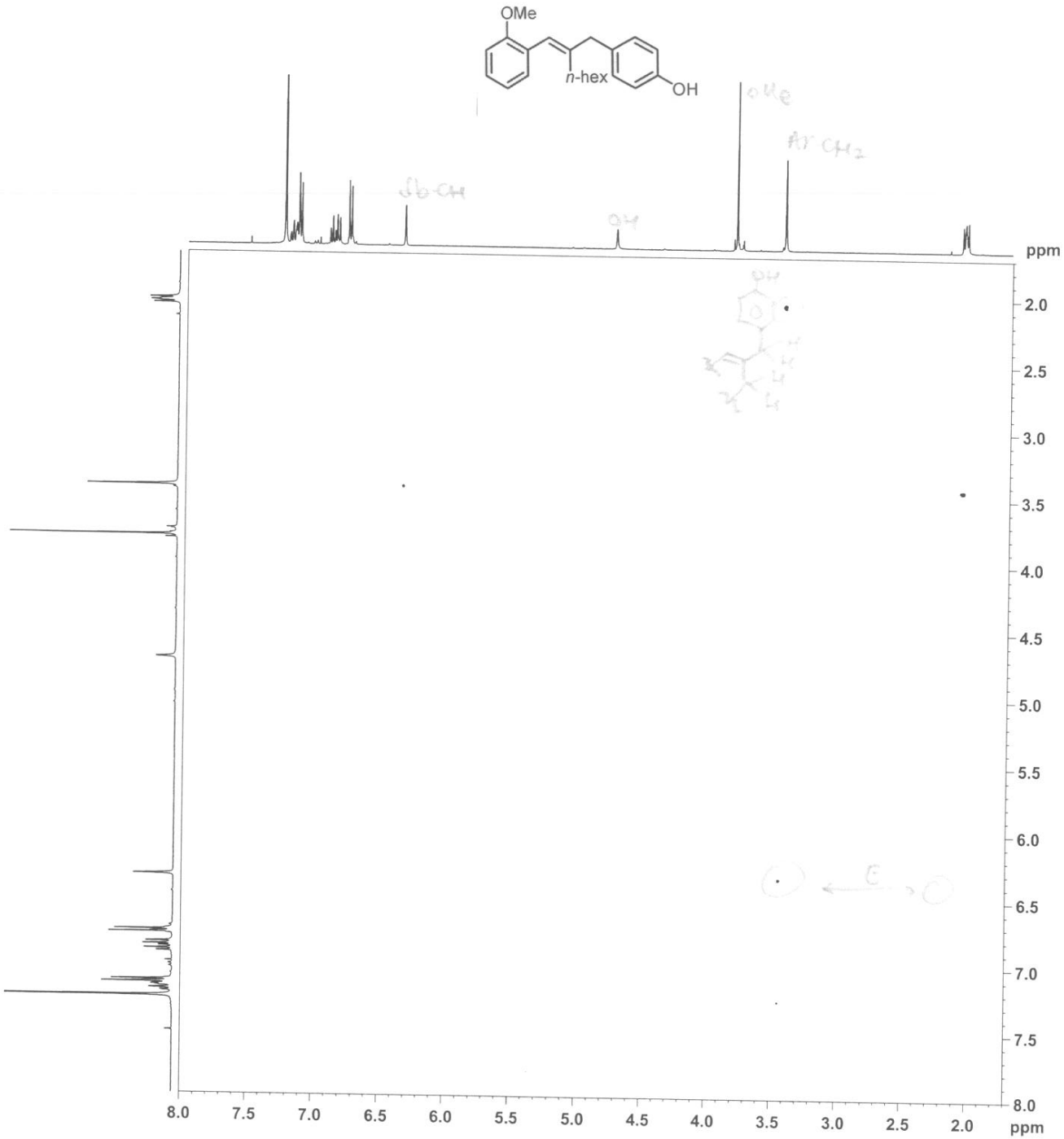
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	17
SFO1	400.1318 MHz
FIDRES	240.292191 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

```
NAME        ccw_1_123_31 rp t11-12
EXPNO                  1
PROCNO                 1
Date_           20090423
Time                19.19
INSTRUM             spect
PROBHD   5 mm PABBI 1H/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    25
DS                     0
SWH             10000.000 Hz
FIDRES              0.152588 Hz
AQ              3.2768500 sec
RG                   203
DW                50.000 usec
DE                 6.50 usec
TE                294.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 7.10 usec
PL1                -2.00 dB
PL1W        13.17734718 W
SFO1        400.1316005 MHz
SI                 65536
SF          400.1300048 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```






BRUKER
ccw_1_123_31 rp t11-12 13C
NAME ccw_1_123_31 rp t11-12 13C
EXPNO 2
PROCNO 1
Date_ 20090429
Time 17.22
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgdc
TD 131072
SOLVENT CDCl3
NS 2871
DS 0
SWH 29761.904 Hz
FIDRES 0.227065 Hz
AQ 2.2020595 sec
RG 203
DW 16.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6208180 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
SFO2 400.1320007 MHz
SI 131072
SF 100.6127551 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
157.30
153.92
142.75
132.61
130.25
130.19
127.73
127.50
122.72
120.14
115.21
110.53
77.48
77.16
76.84
55.59
42.86
31.74
30.30
29.42
28.20
22.73
14.20
OMe
n-hex
OH
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

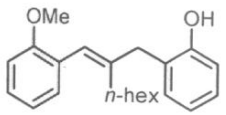


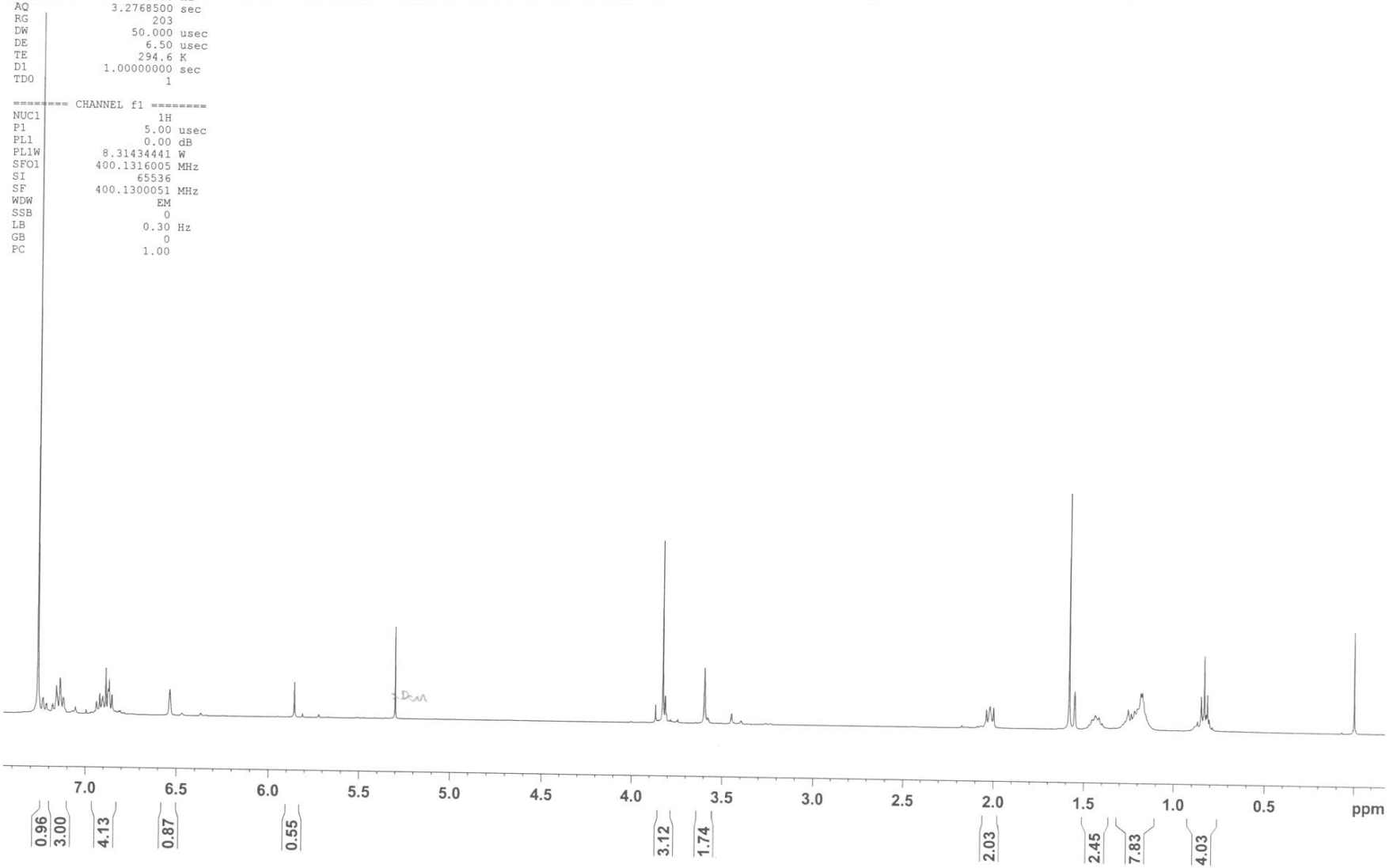
BRUKER

NAME	ccw_1_123_31 rp t11-12
EXPNO	6
PROCNO	1
Date_	20090423
Time	19.22
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.8 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	14
SFO1	400.1318 MHz
FIDRES	291.783386 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

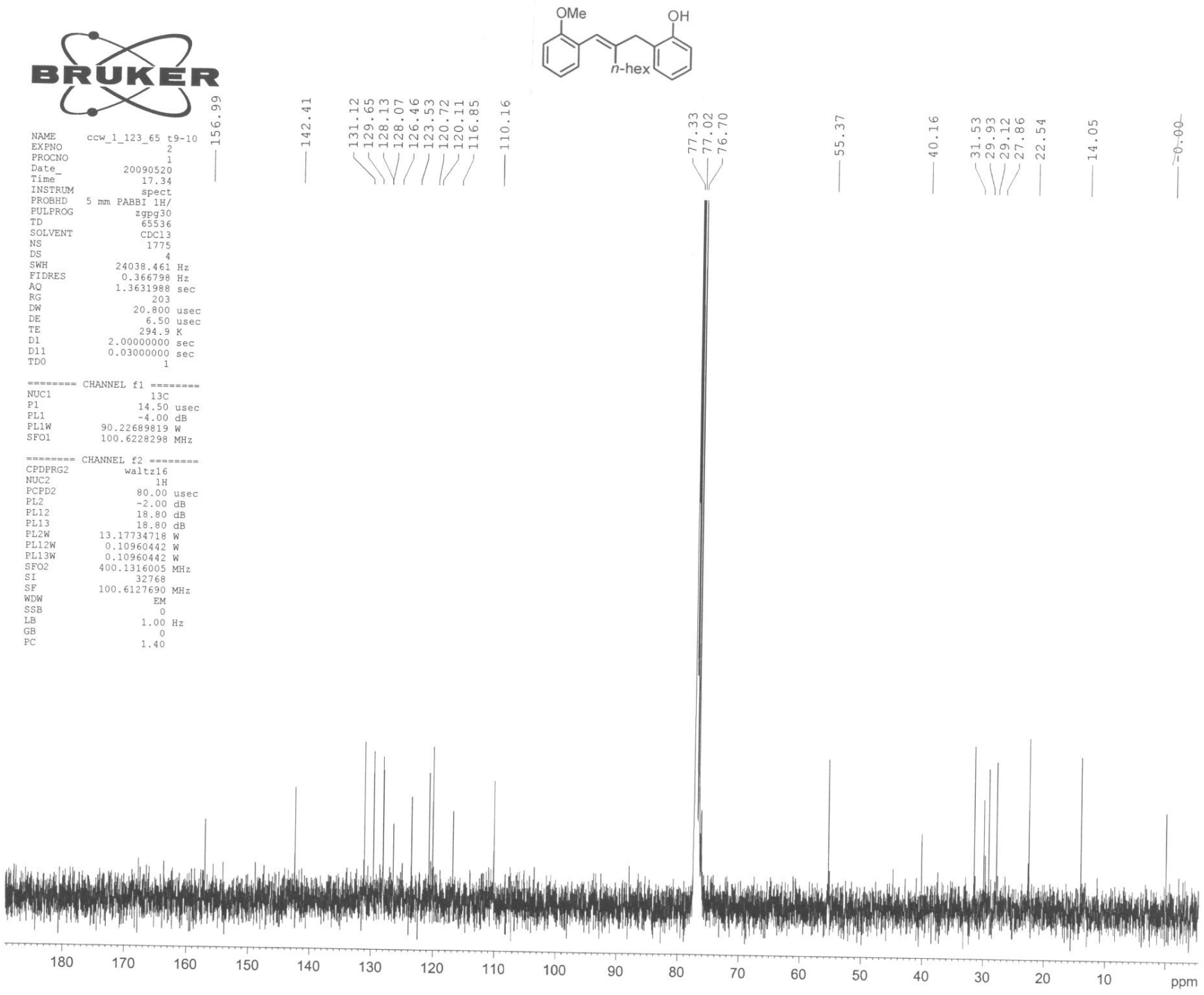
NAME	ccw_1_123_65 t9-10
EXPNO	1
PROCNO	1
Date_	20090423
Time	10.33
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	15
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.6 K
D1	1.00000000 sec
TD0	1
========	CHANNEL f1 ========
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300051 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

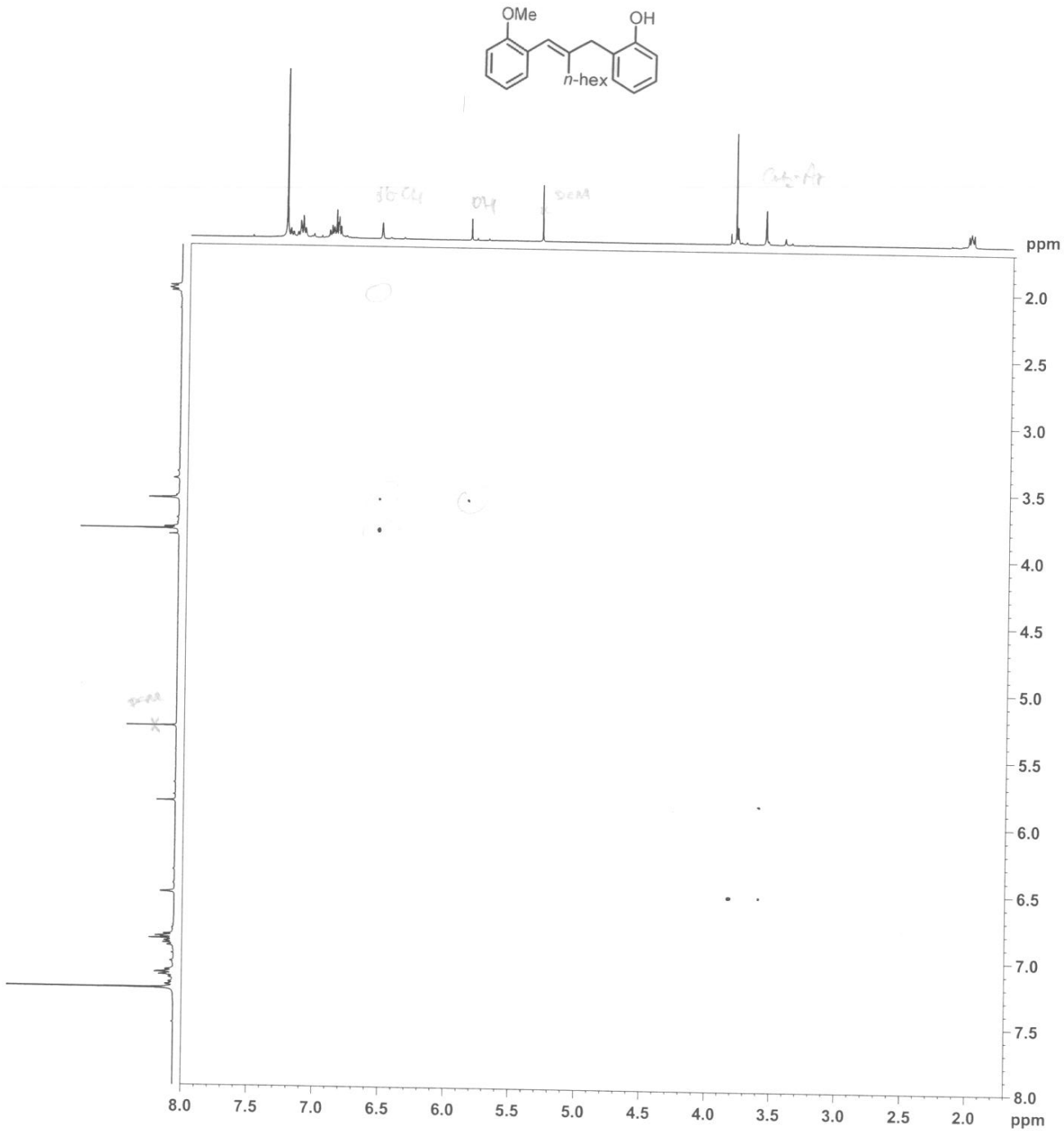
ccw_1_123_65 t9-10





B1h2


OMe
OH
n-hex
156.99
142.41
131.12
129.65
128.13
128.07
126.46
123.53
120.72
120.11
116.85
110.16
77.33
77.02
76.70
55.37
40.16
31.53
29.93
29.12
27.86
22.54
14.05
0.00
NAME ccw_1_123_65 t9-10
EXPNO 2
PROCNO 1
Date_ 20090520
Time 17.34
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1775
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 294.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



BRUKER

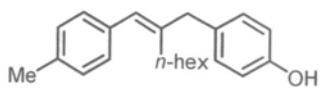
NAME	ccw_1_123_65 t9-10
EXPNO	6
PROCNO	1
Date_	20090423
Time	14.55
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	294.8 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

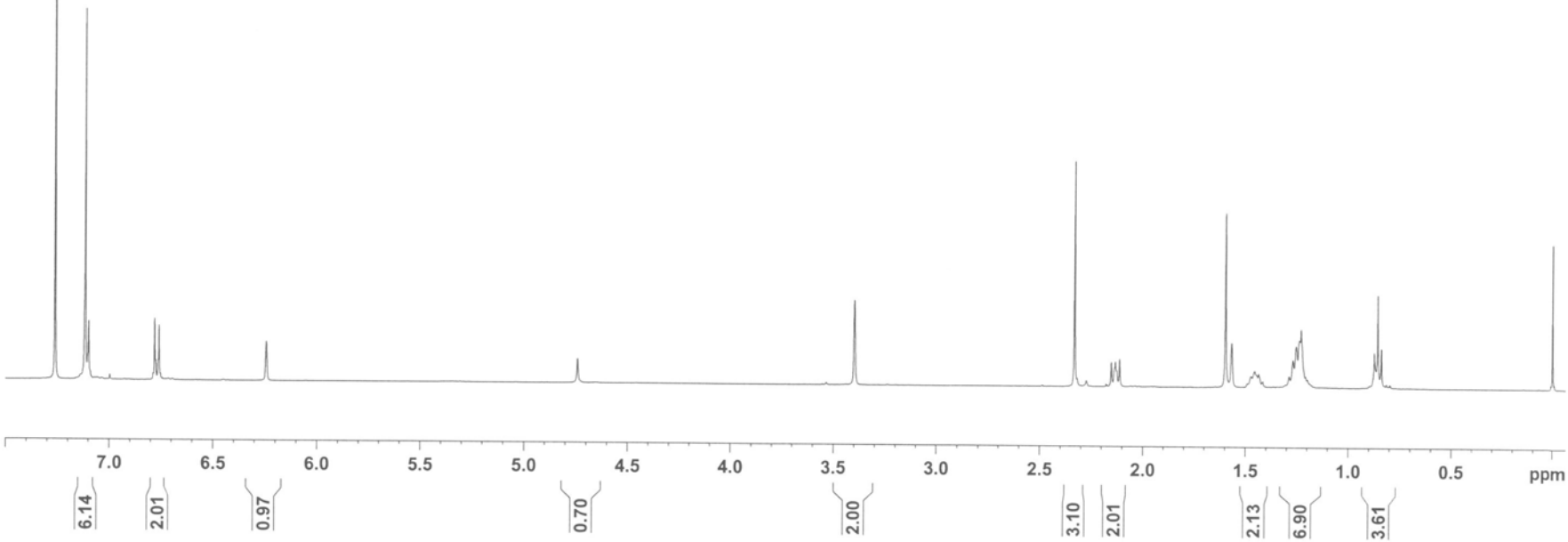
======== CHANNEL f1 ========

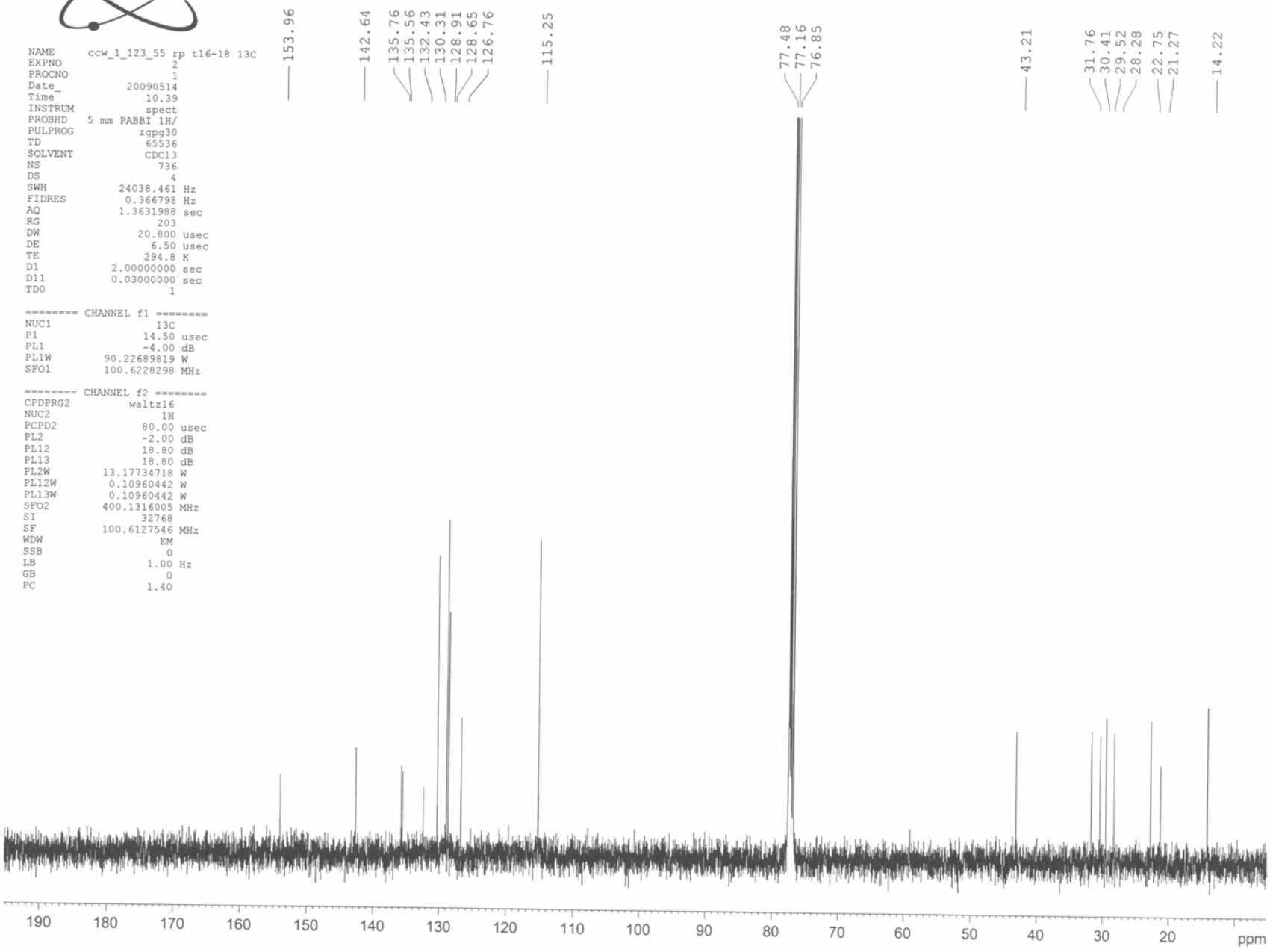
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	8
SFO1	400.1318 MHz
FIDRES	510.620911 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

```
NAME      ccw_1_123_55 rp t16-18
EXPNO                1
PROCNO               1
Date_         20090421
Time_            20.43
INSTRUM          spect
PROBHD   5 mm PABBI 1H/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  38
DS                   0
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ           3.2768500 sec
RG                 203
DW              50.000 usec
DE               6.50 usec
TE              294.9 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               5.00 usec
PL1              0.00 dB
PL1W       8.31434441 W
SFO1     400.1316005 MHz
SI             65536
SF       400.1300051 MHz
WDW               EM
SSB                  0
LB               0.30 Hz
GB                   0
PC                1.00
```

ccw_1_123_55 rp t16-18





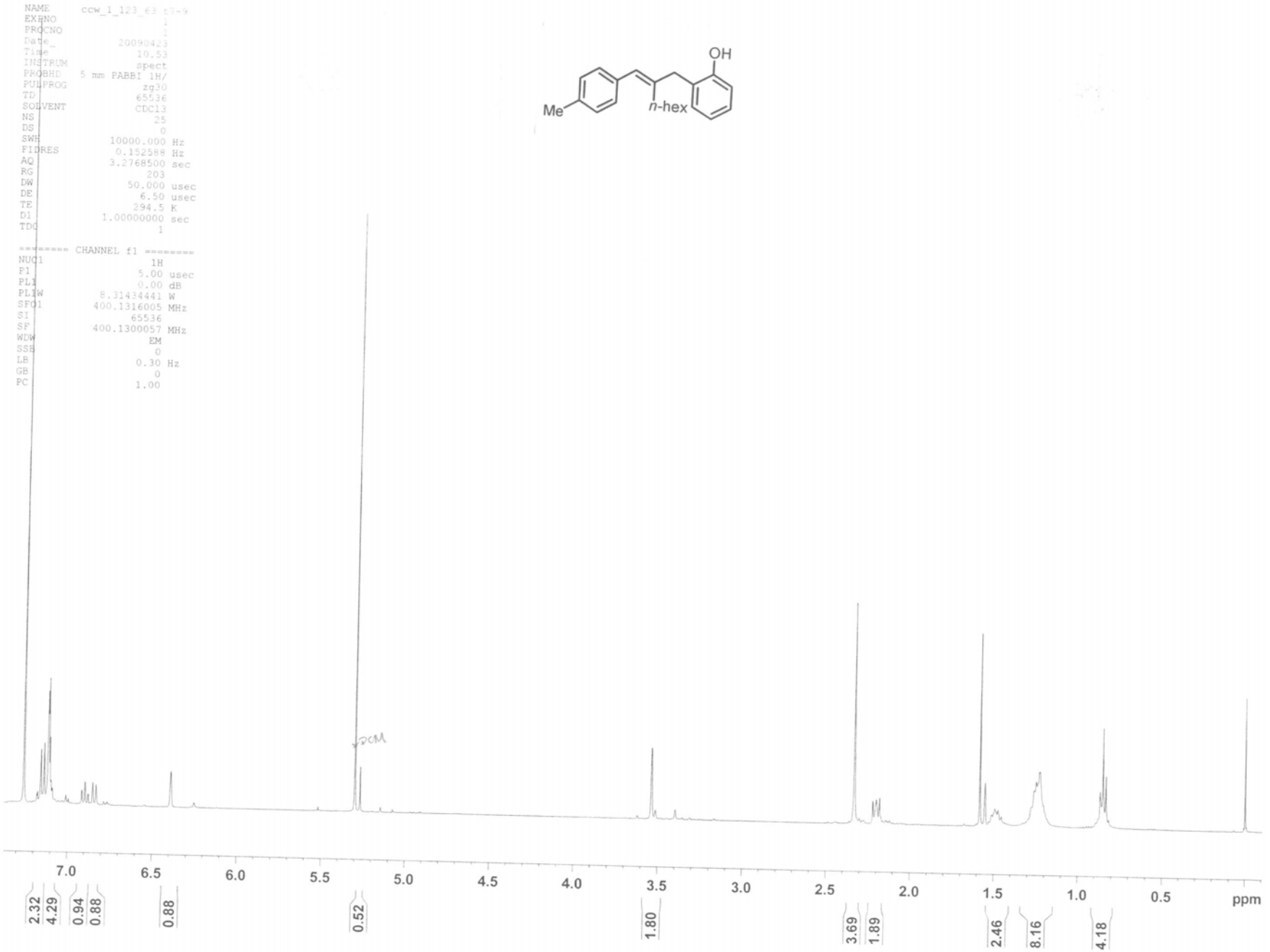


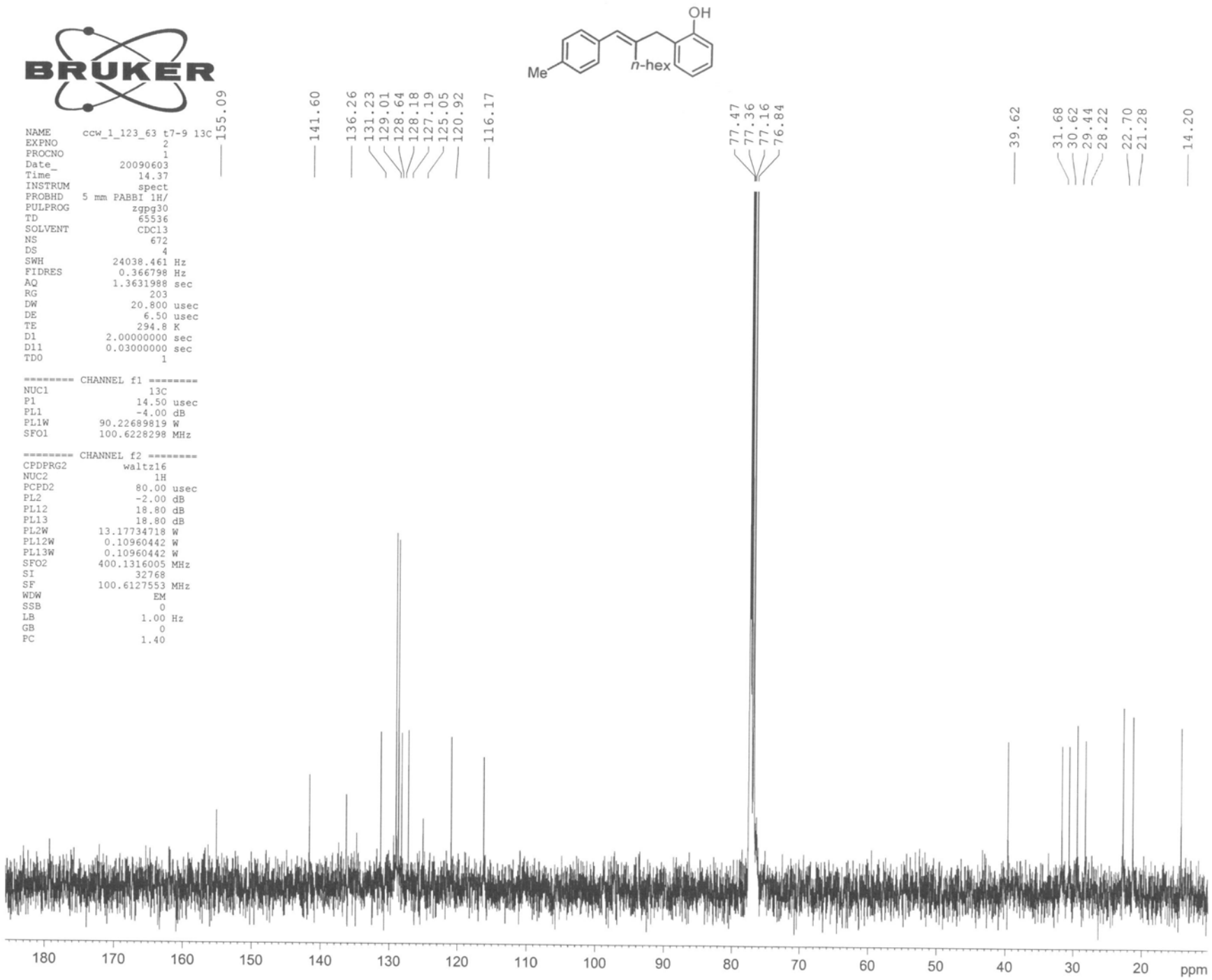

```
NAME      ccw_1_123_55 rp t16-18 13C
EXPNO                 2
PROCNO                1
Date_          20090514
Time              10.39
INSTRUM           spect
PROBHD   5 mm PABBI 1H/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  736
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631988 sec
RG                  203
DW               20.800 usec
DE                6.50 usec
TE               294.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                14.50 usec
PL1               -4.00 dB
PL1W        90.22689819 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2             80.00 usec
PL2               -2.00 dB
PL12              18.80 dB
PL13              18.80 dB
PL2W        13.17734718 W
PL12W        0.10960442 W
PL13W        0.10960442 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127546 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

Bruker Advance III 400

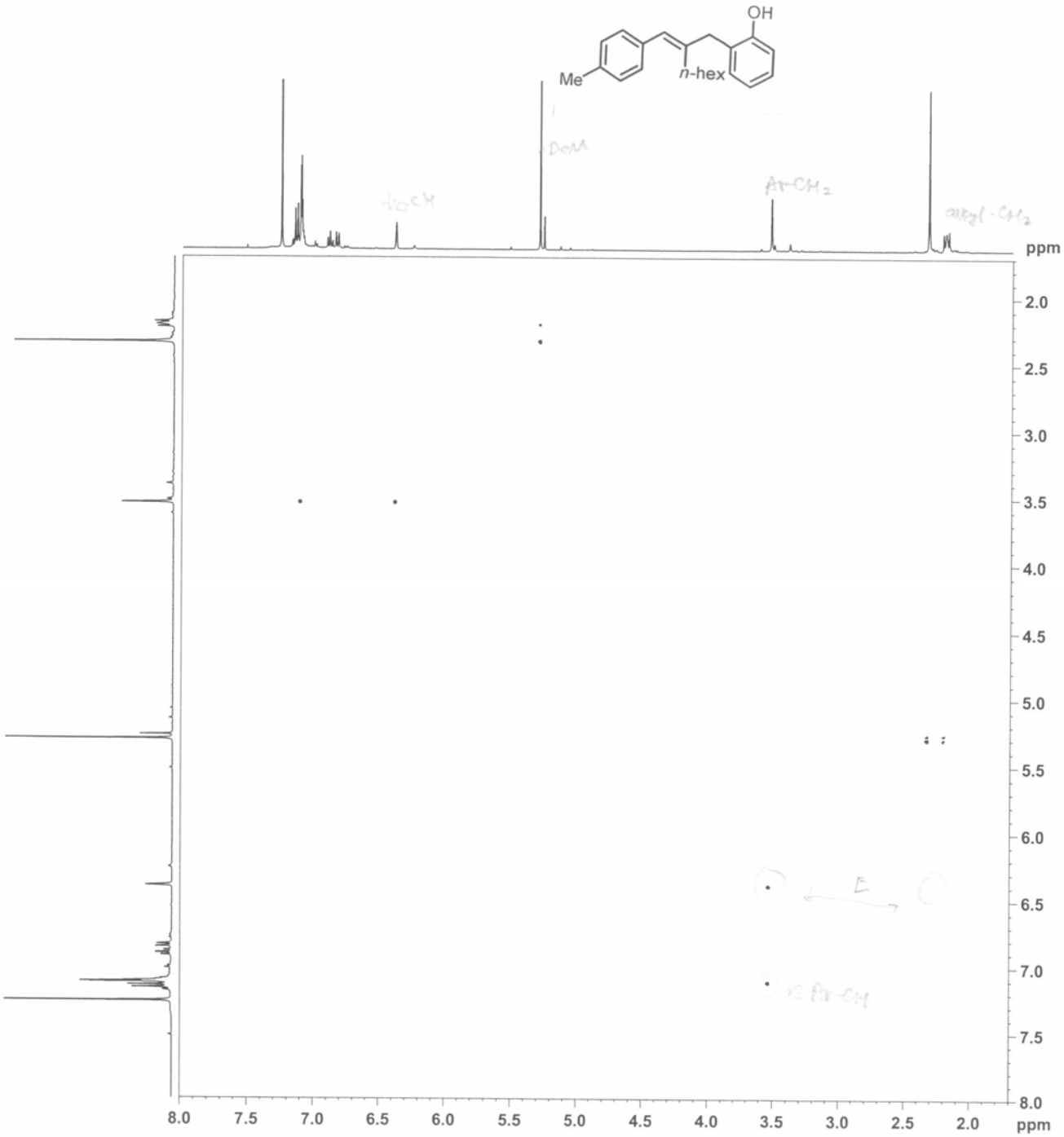
NAME	ccw_1_123_55 rp t16-18
EXPNO	6
PROCNO	1
Date_	20090422
Time	17.10
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.5 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
========	CHANNEL f1 ========
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	23
SFO1	400.1318 MHz
FIDRES	177.607269 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

B111


NAME ccw_1_123_63 t7-9
EXPNO 1
PROCNO 1
Date_ 20090423
Time 10.53
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 25
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 294.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300057 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
Me
n-hex
PCM
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.32
4.29
0.94
0.88
0.88
0.52
1.80
3.69
1.89
2.46
8.16
4.18


BRUKER
OH
Me
n-hex
155.09
141.60
136.26
131.23
129.01
128.64
128.18
127.19
125.05
120.92
116.17
77.47
77.36
77.16
76.84
39.62
31.68
30.62
29.44
28.22
22.70
21.28
14.20
NAME ccw_1_123_63 t7-9 13C
EXPNO 2
PROCNO 1
Date_ 20090603
Time 14.37
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 672
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 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

B112



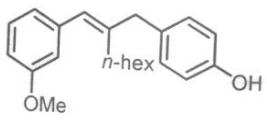
BRUKER

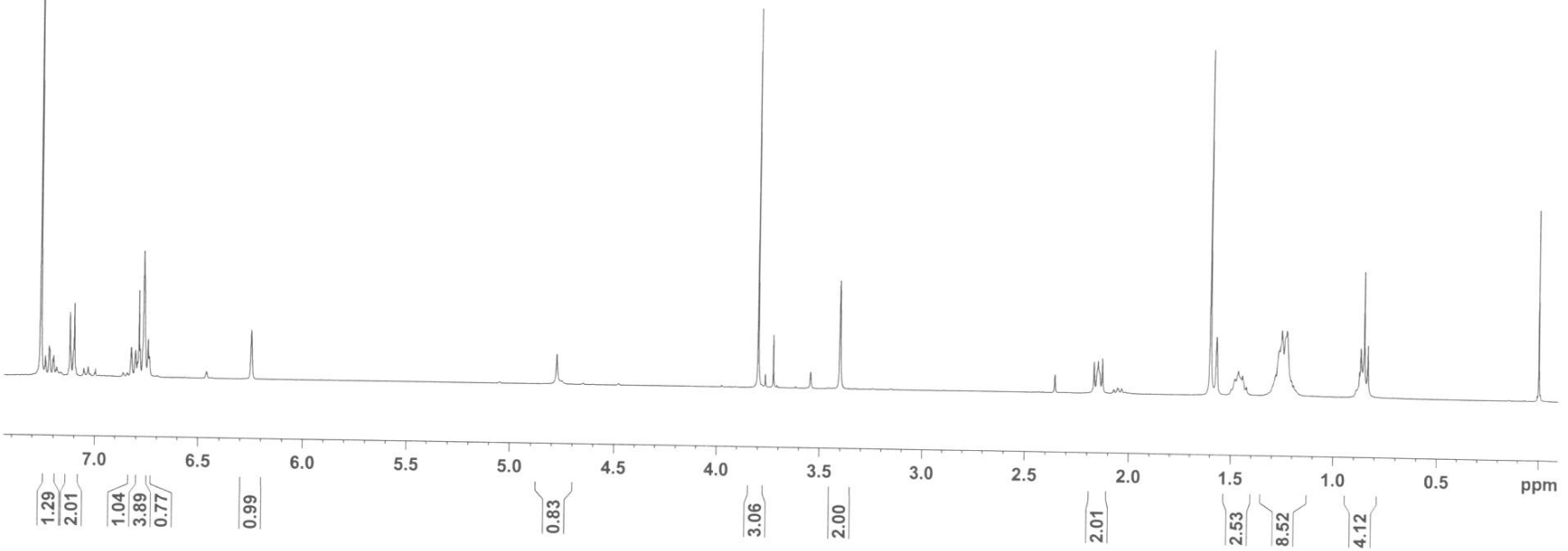
NAME	ccw_1_123_63 t7-9
EXPNO	6
PROCNO	1
Date_	20090423
Time	15.15
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.6 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

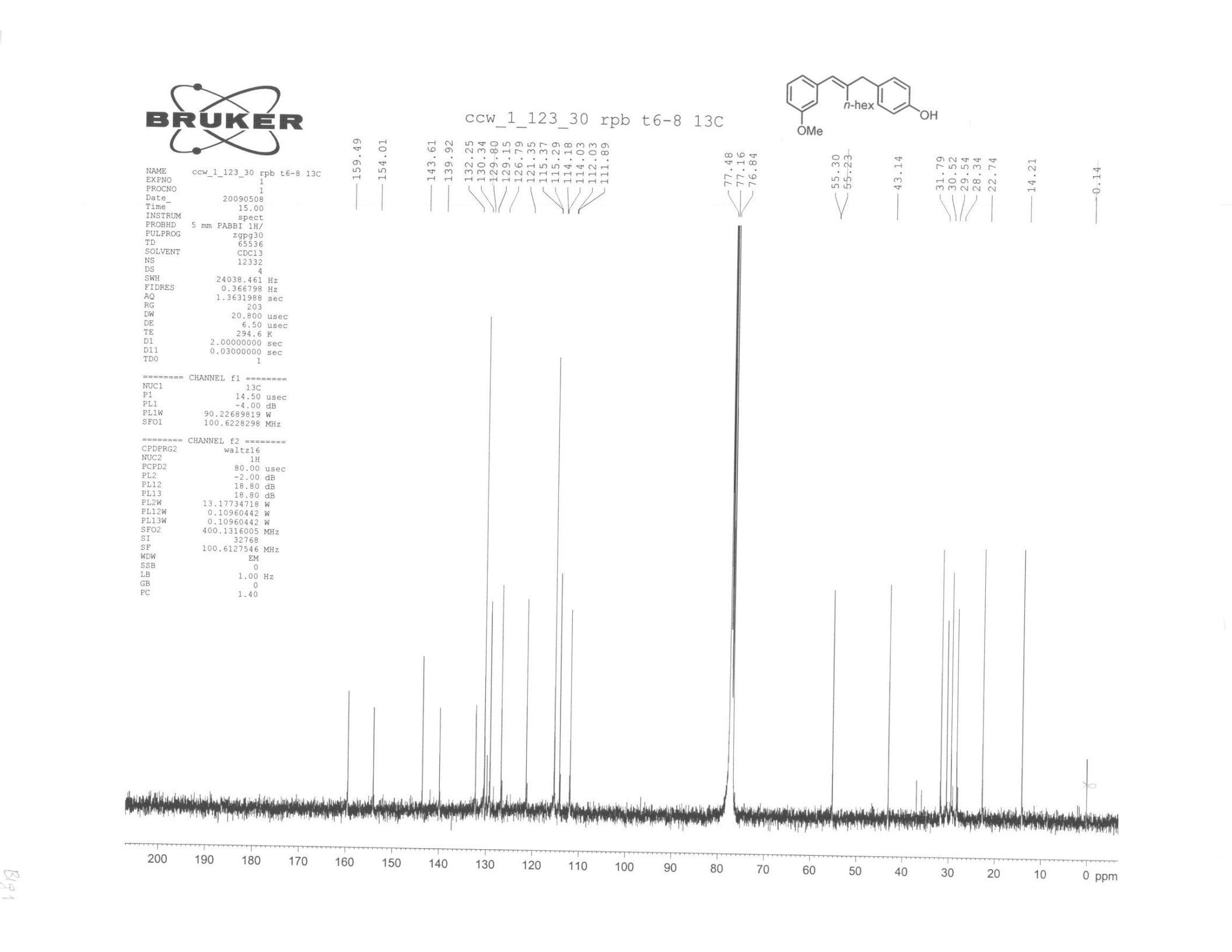
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	6
SFO1	400.1318 MHz
FIDRES	680.827881 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

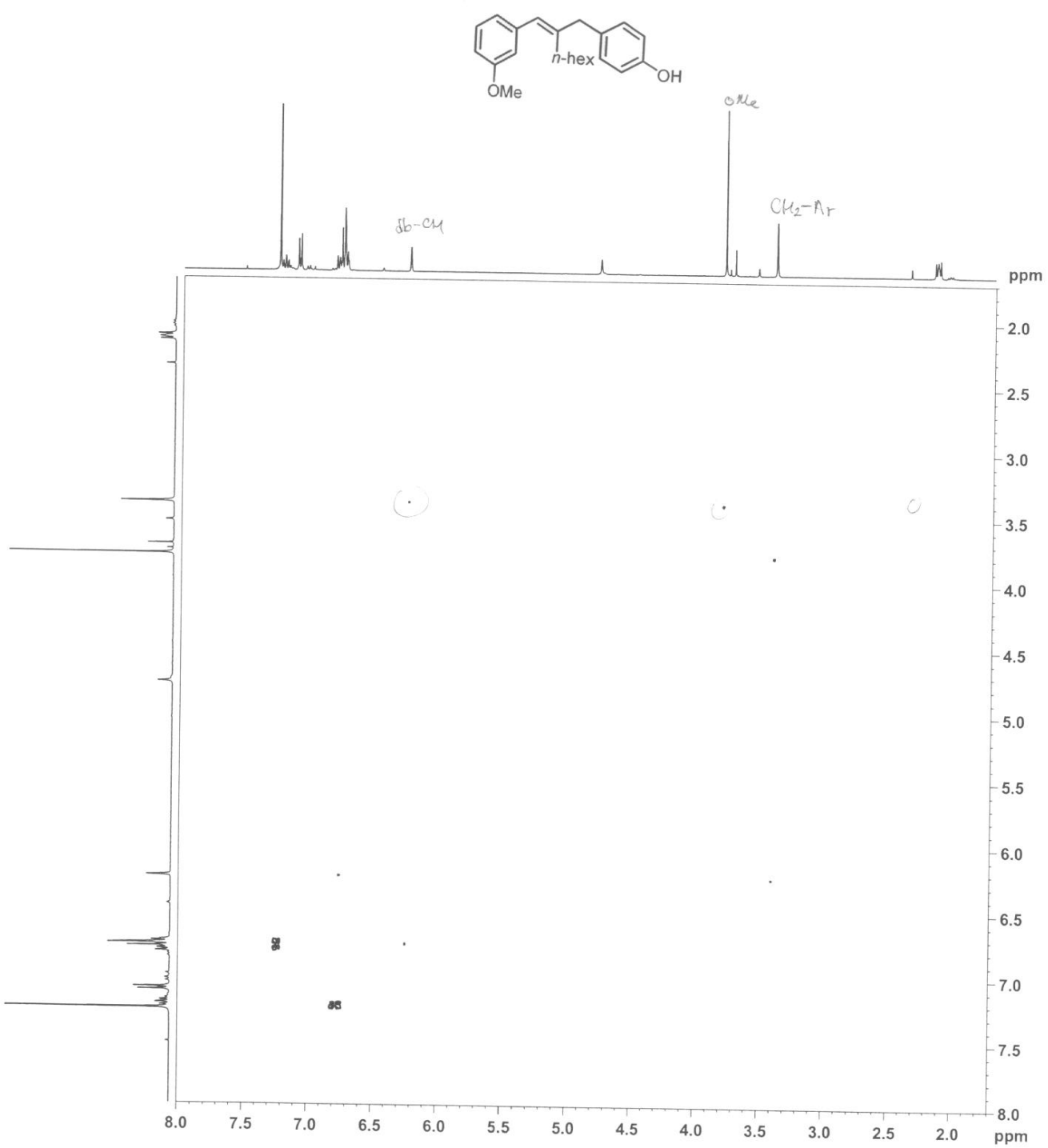
NAME	ccw_1_123_30 rpb t6-8
EXPNO	1
PROCNO	1
Date_	20090421
Time_	20.25
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	41
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	294.9 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	5.00 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300050 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

ccw_1_123_30 rpb t6-8






BRUKER
ccw_1_123_30 rpb t6-8 13C
n-hex
OH
OMe
159.49
154.01
143.61
139.92
132.25
130.34
129.80
129.15
126.79
121.35
115.37
115.29
114.18
114.03
112.03
111.89
77.48
77.16
76.84
55.30
55.23
43.14
31.79
30.52
29.54
28.34
22.74
14.21
0.14
NAME ccw_1_123_30 rpb t6-8 13C
EXPNO 1
PROCNO 1
Date_ 20090508
Time 15.00
INSTRUM spect
PROBHD 5 mm PABBI 1H/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 12332
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 294.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 14.50 usec
PL1 -4.00 dB
PL1W 90.22689819 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 18.80 dB
PL13 18.80 dB
PL2W 13.17734718 W
PL12W 0.10960442 W
PL13W 0.10960442 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



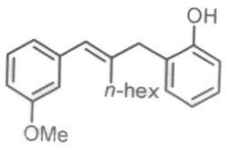
BRUKER

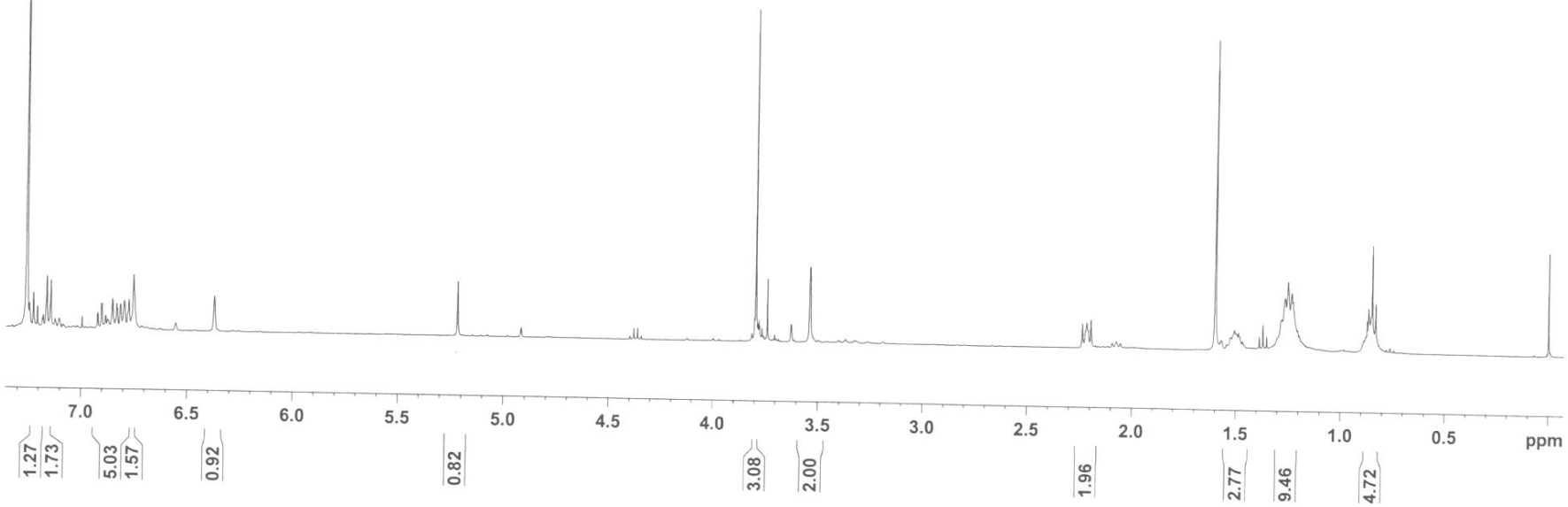
NAME	ccw_1_123_30 rpb t6-8
EXPNO	6
PROCNO	1
Date_	20090422
Time	14.58
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.5 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

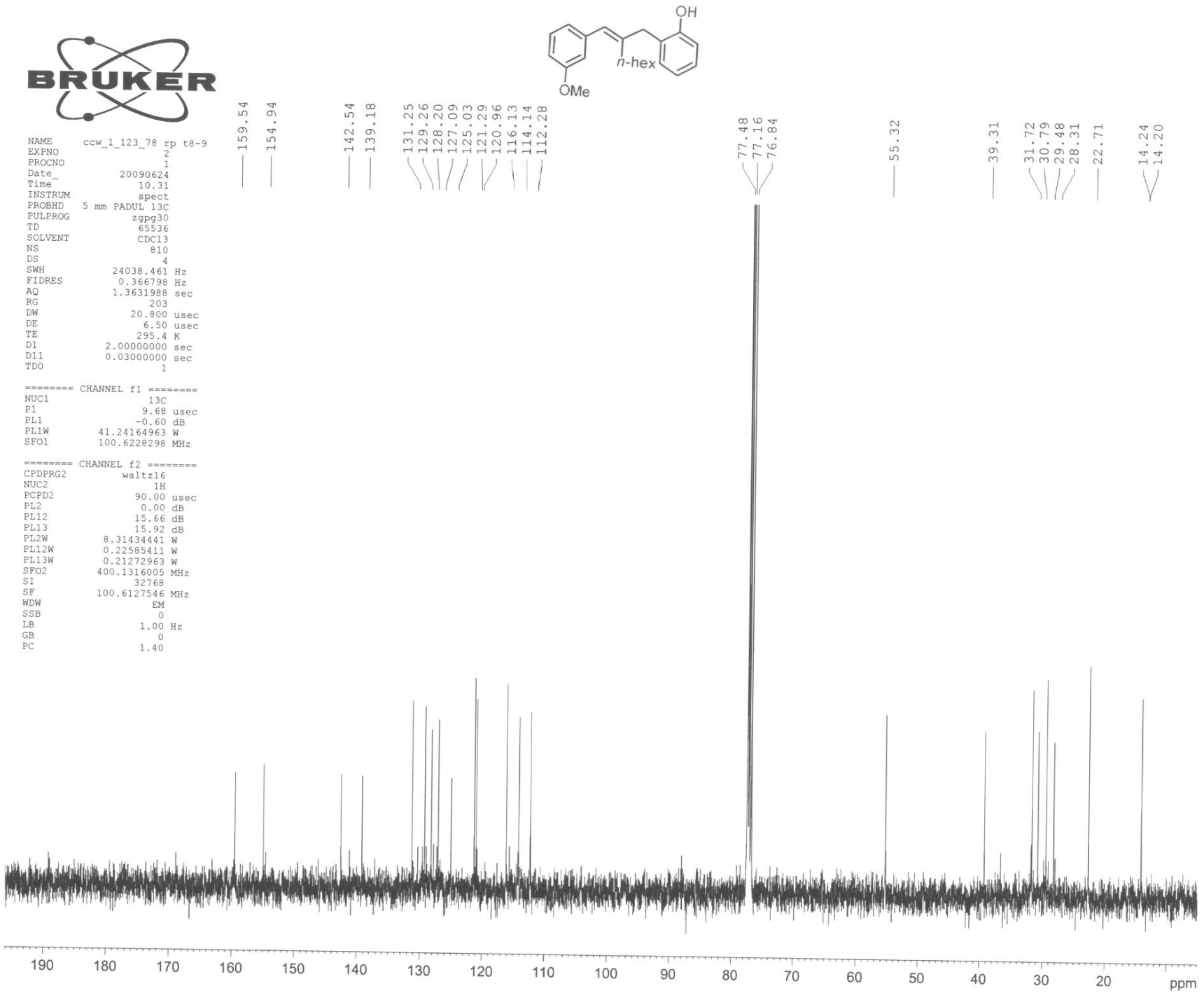
======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	53
SFO1	400.1318 MHz
FIDRES	77.074852 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

B191

```
NAME        ccw_1_123_78 rp t8-9
EXPNO                    1
PROCNO                   1
Date_             20090624
Time                 9.43
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                       9
DS                       0
SWH              10000.000 Hz
FIDRES           0.152588 Hz
AQ               3.2768500 sec
RG                     203
DW                50.000 usec
DE                 6.50 usec
TE                294.6 K
D1               1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  14.83 usec
PL1                  0.00 dB
PL1W          8.31434441 W
SFO1         400.1316005 MHz
SI                   65536
SF           400.1300051 MHz
WDW                    EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                  1.40
```

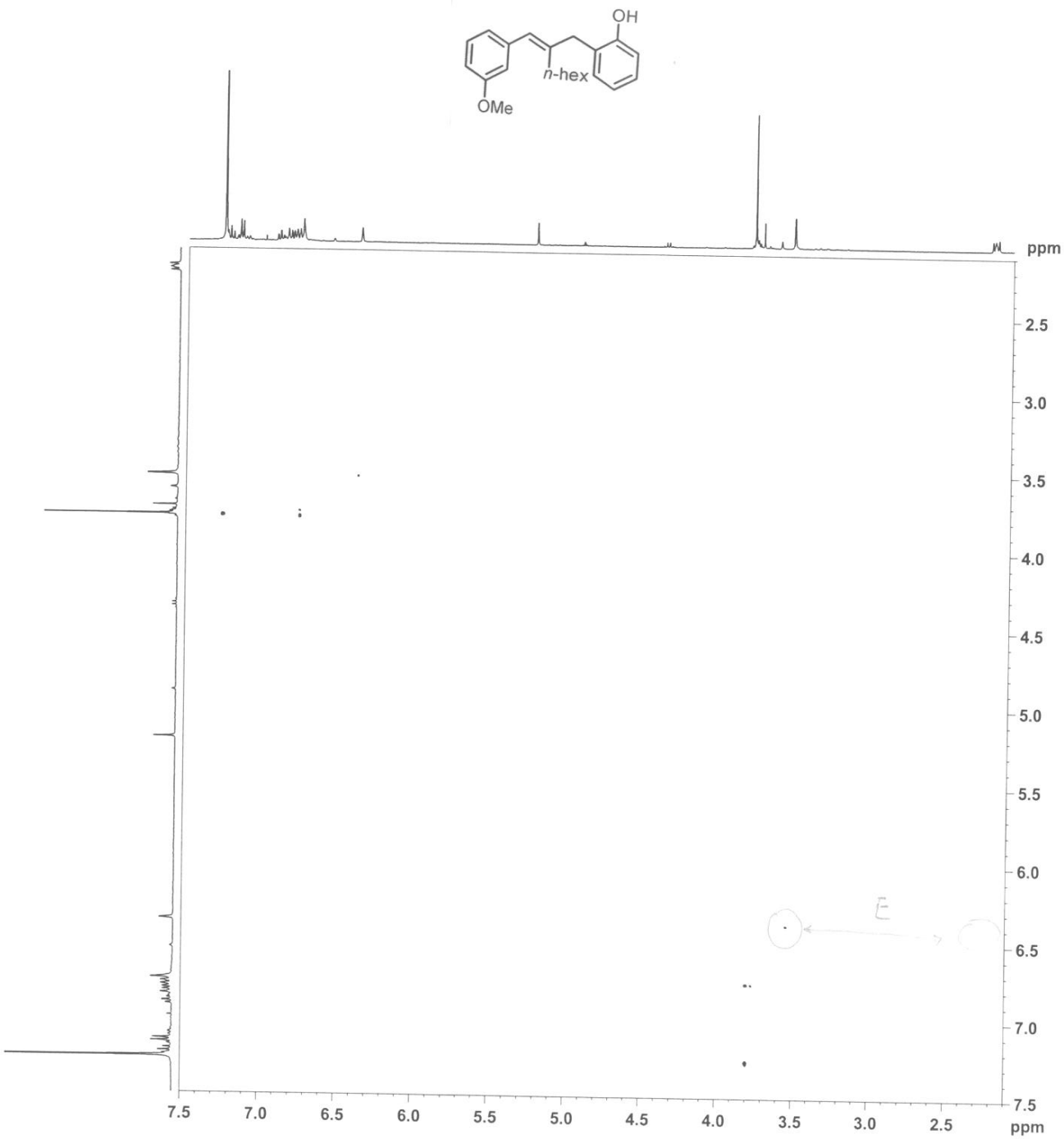






NAME	ccw_1_123_78 rp t8-9
EXPNO	2
PROCNO	1
Date_	20090624
Time	10.31
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	810
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.66 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.22585411 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127546 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

B192

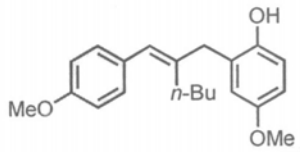


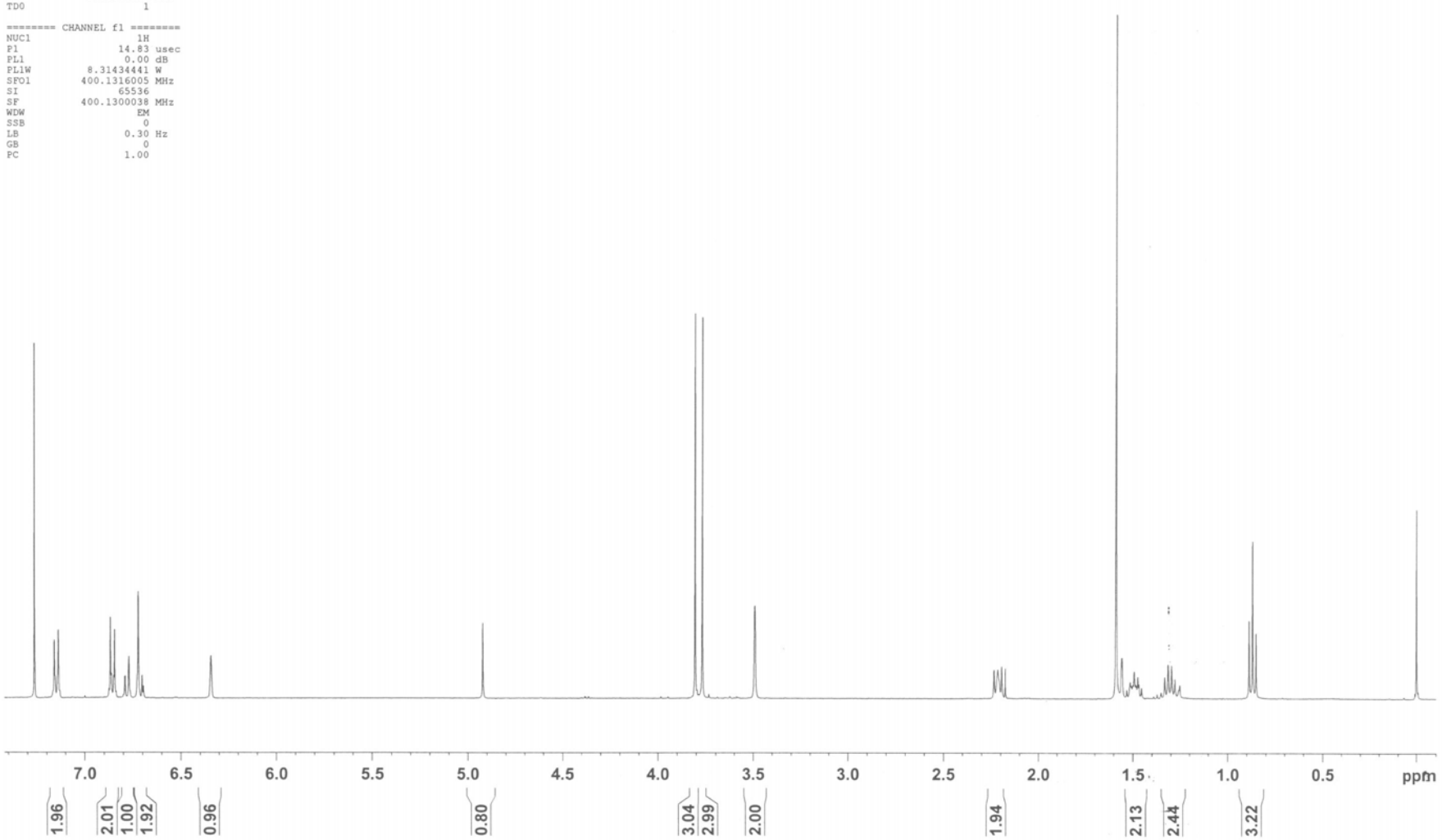
Bruker Advance III 400

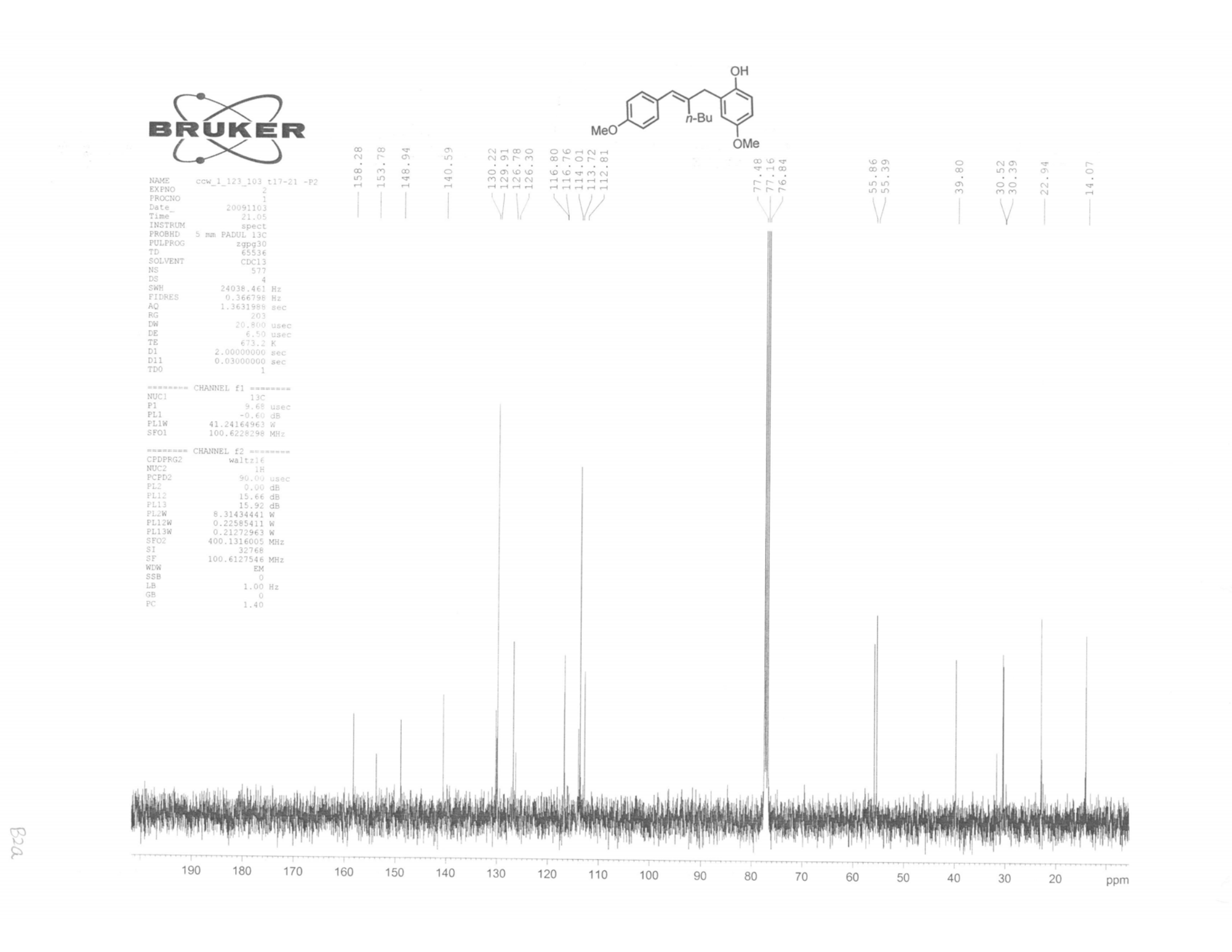
NAME	ccw_1_123_78 rp t8-9
EXPNO	6
PROCNO	1
Date_	20090424
Time	16.15
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	294.7 K
D0	0.00011336 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec

======== CHANNEL f1 ========	
NUC1	1H
P1	7.10 usec
PL1	-2.00 dB
PL1W	13.17734718 W
SFO1	400.1318419 MHz
ND0	1
TD	6
SFO1	400.1318 MHz
FIDRES	680.827881 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300009 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

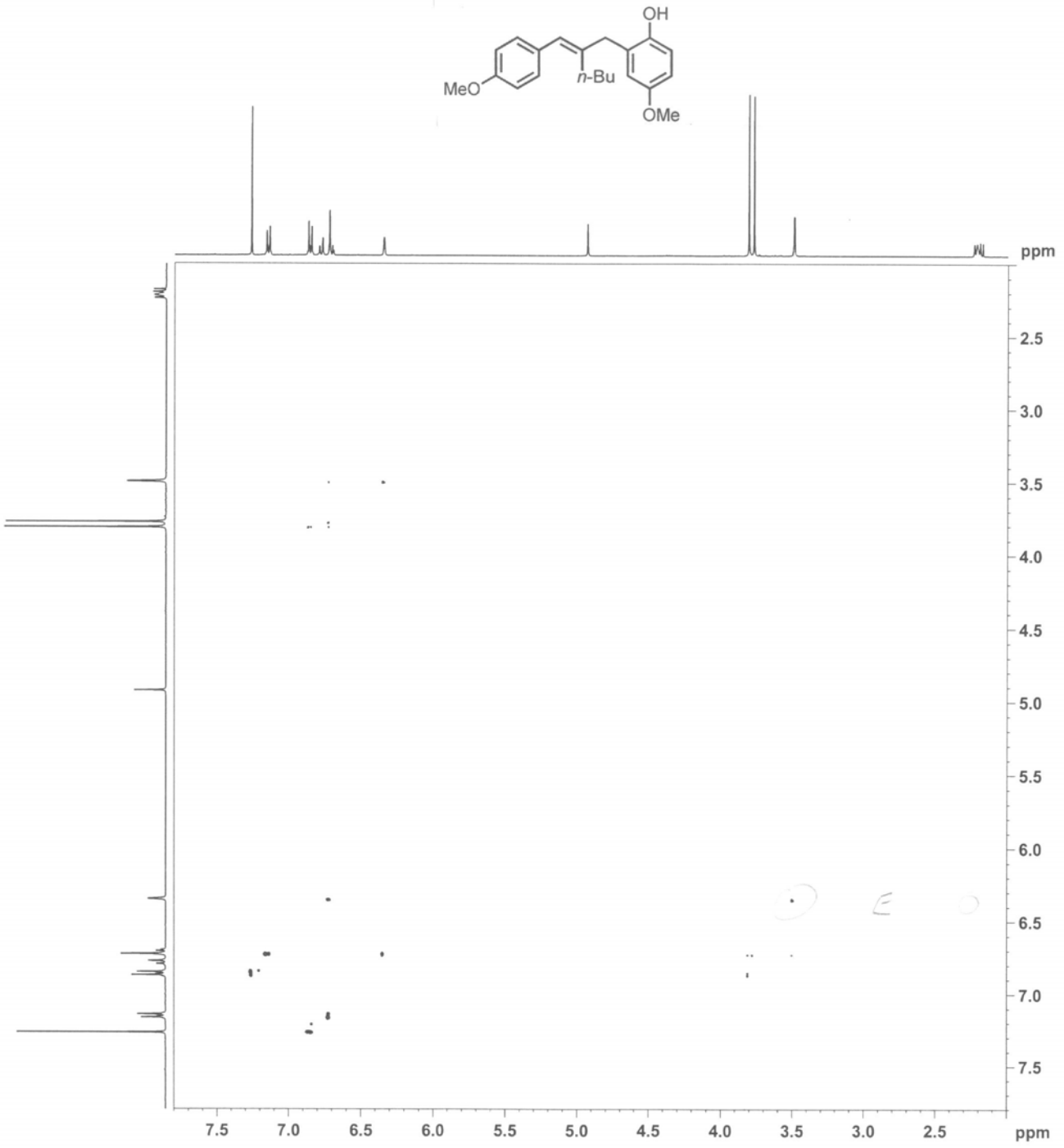
```
NAME      ccw_1_123_103 t17-21 -P2
EXPNO                        1
PROCNO                       1
Date_                 20091110
Time                     21.11
INSTRUM                  spect
PROBHD   5 mm PADUL 13C
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                 10000.000 Hz
FIDRES                0.152588 Hz
AQ                    3.2768500 sec
RG                        203
DW                     50.000 usec
DE                      6.50 usec
TE                     294.5 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       14.83 usec
PL1                       0.00 dB
PL1W                 8.31434441 W
SFO1               400.1316005 MHz
SI                      65536
SF                 400.1300038 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```






BRUKER
OH
MeO
n-Bu
OMe
158.28
153.78
148.94
140.59
130.22
129.91
126.78
126.30
116.80
116.76
114.01
113.72
112.81
77.48
77.16
76.84
55.86
55.39
39.80
30.52
30.39
22.94
14.07
NAME ccw_1_123_103 t17-21 -P2
EXPNO 2
PROCNO 1
Date_ 20091103
Time 21.05
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 577
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 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.68 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 15.66 dB
PL13 15.92 dB
PL2W 8.31434441 W
PL12W 0.22585411 W
PL13W 0.21272963 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127546 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

B2a

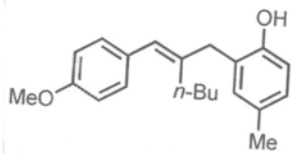


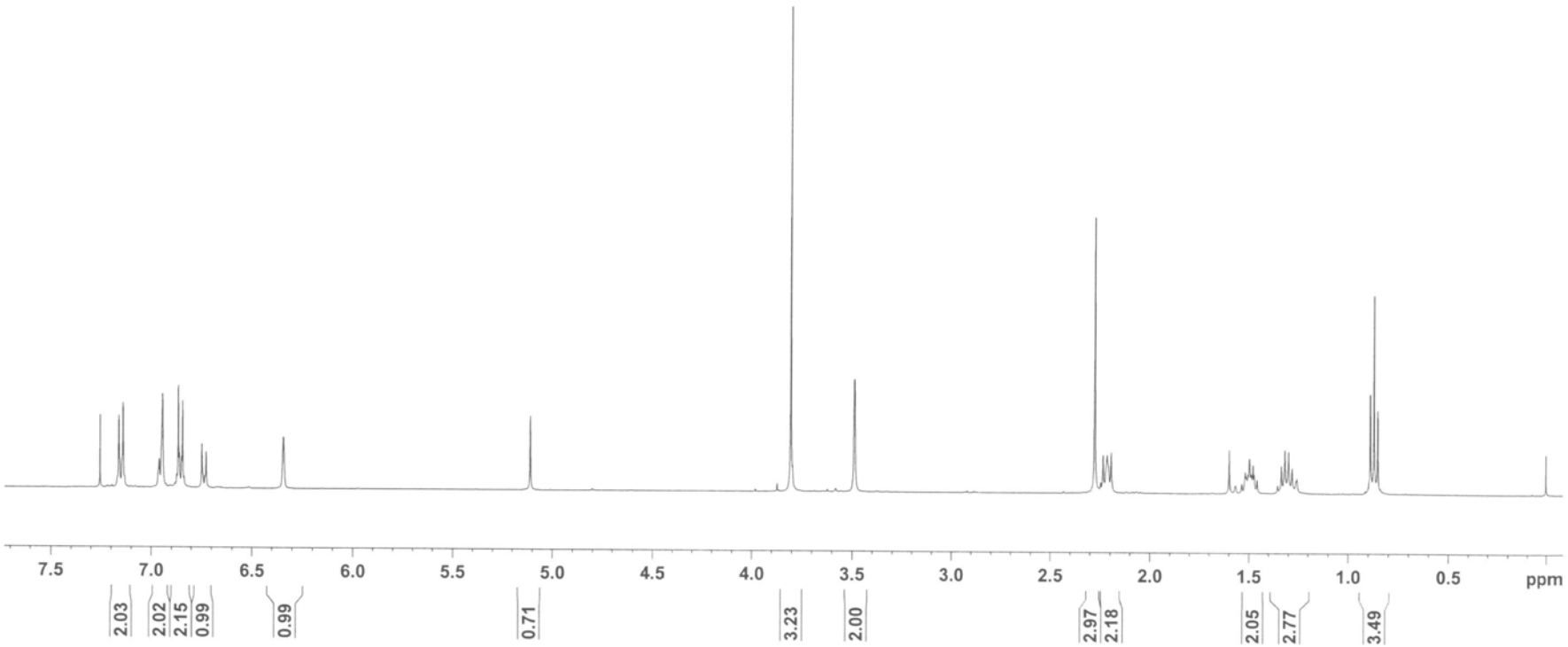
Bruker Advance III 400

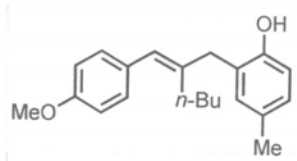
NAME	ccw_1_123_103 t17-21 -P2
EXPNO	6
PROCNO	1
Date_	20091103
Time	21.09
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	673.2 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
========	CHANNEL f1 ========
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	59
SFO1	400.1318 MHz
FIDRES	69.236732 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300009 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300009 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

B2a

```
NAME      ccw_1_123_105 t9-17 -P
EXPNO                 1
PROCNO                1
Date_          20091103
Time              15.02
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   21
DS                    0
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2768500 sec
RG                  203
DW                50.000 usec
DE                 6.50 usec
TE                673.2 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                14.83 usec
PL1                0.00 dB
PL1W         8.31434441 W
SFO1        400.1316005 MHz
SI                65536
SF          400.1300067 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

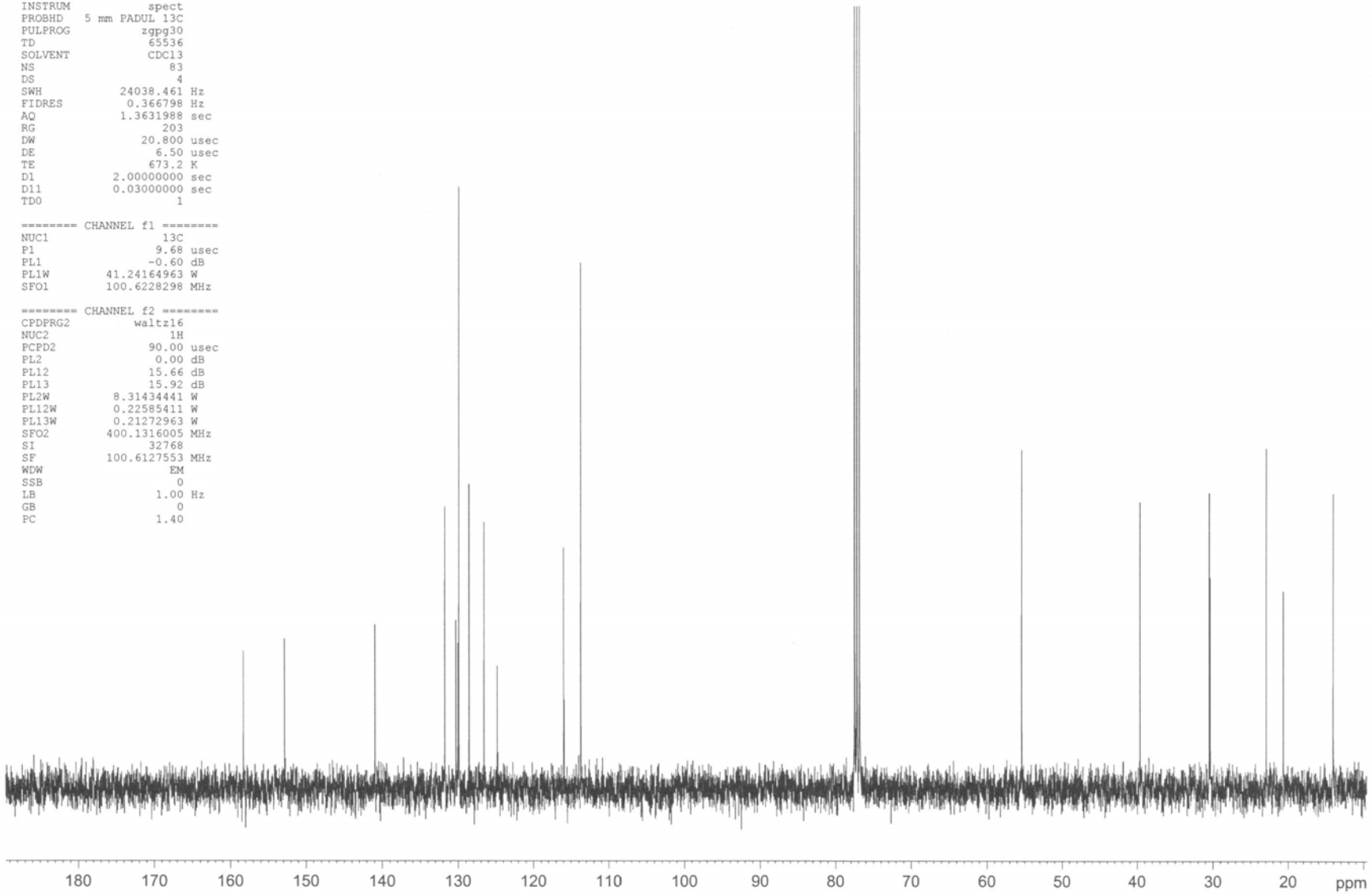




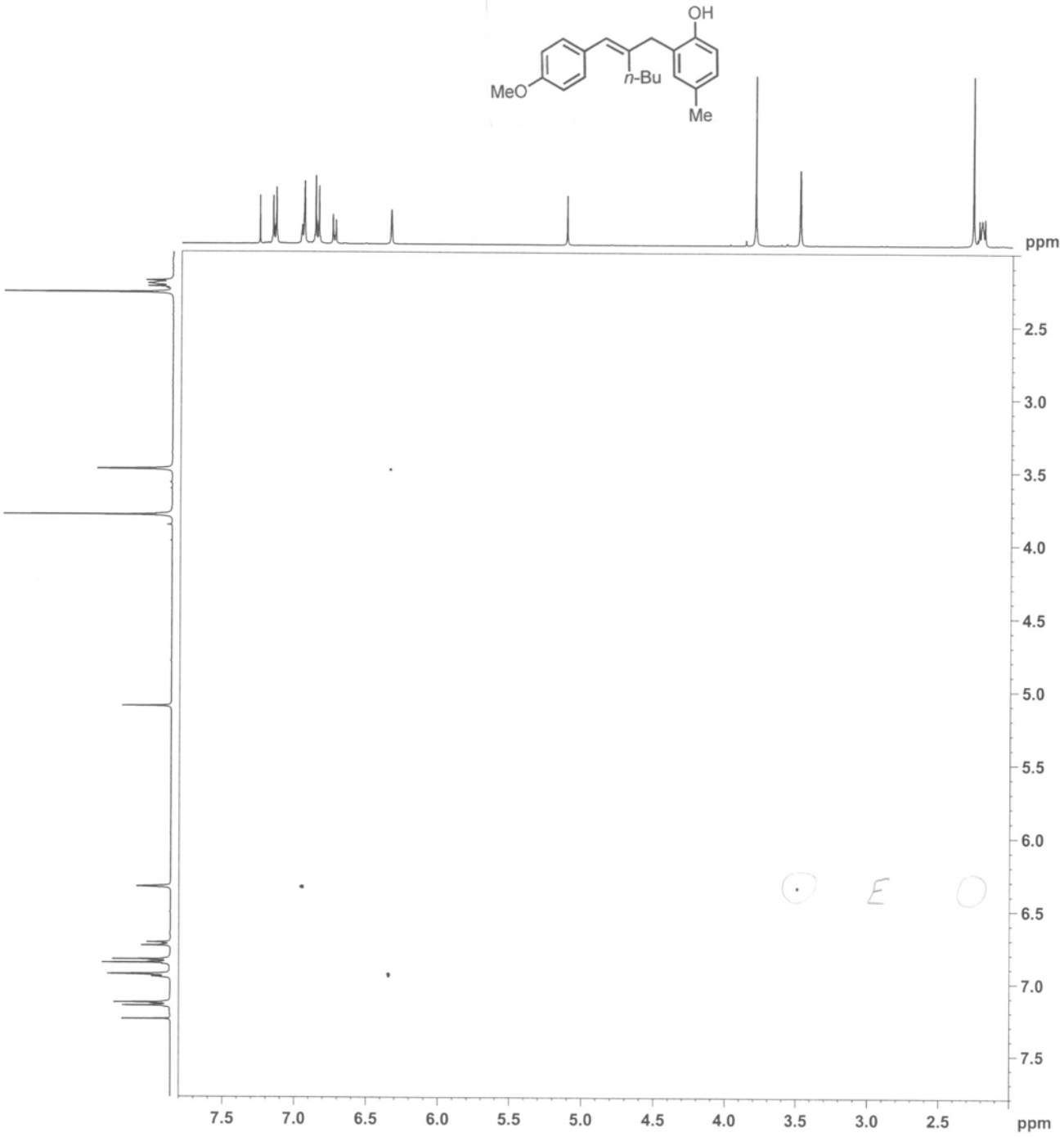



```
NAME        ccw-2-105 t9-17p
EXPNO                     2
PROCNO                    1
Date_              20091104
Time                   8.08
INSTRUM               spect
PROBHD    5 mm PADUL 13C
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      83
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                  673.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                    9.68 usec
PL1                  -0.60 dB
PL1W          41.24164963 W
SFO1          100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                   0.00 dB
PL12                 15.66 dB
PL13                 15.92 dB
PL2W           8.31434441 W
PL12W          0.22585411 W
PL13W          0.21272963 W
SFO2          400.1316005 MHz
SI                   32768
SF            100.6127553 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

158.25 152.79 140.92 131.76 130.30 130.03 129.91 128.56 126.57 124.82 115.97 113.71 77.48 77.16 76.85 55.38 39.62 30.49 30.37 22.93 20.67 14.06



B26

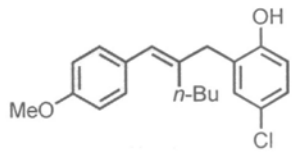


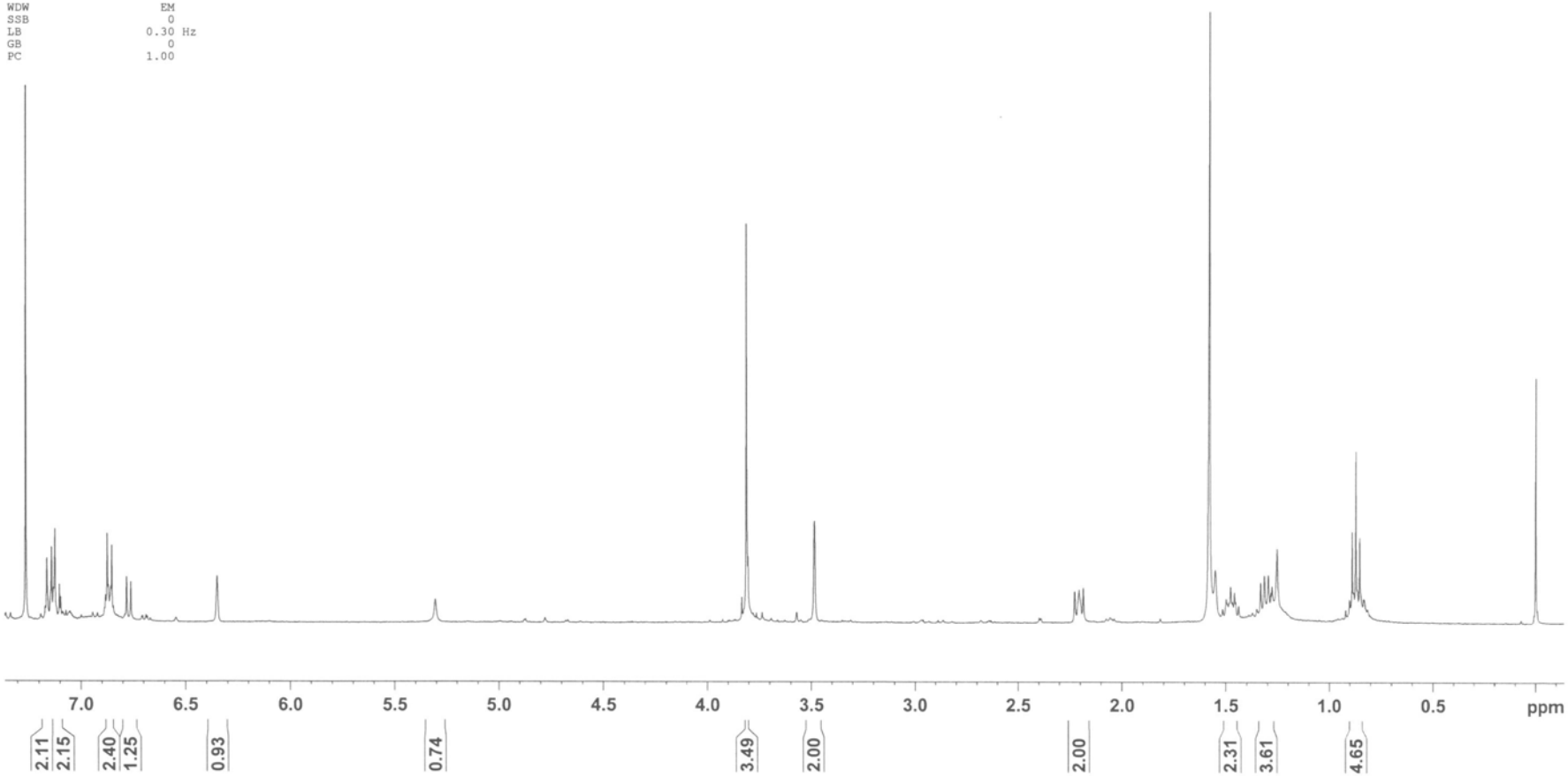
Bruker Advance III 400

NAME	ccw_1_123_105 t9-17 -P
EXPNO	6
PROCNO	1
Date_	20091103
Time	15.10
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	673.2 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	36
SFO1	400.1318 MHz
FIDRES	113.471313 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300048 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

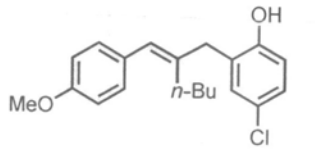
Bz b

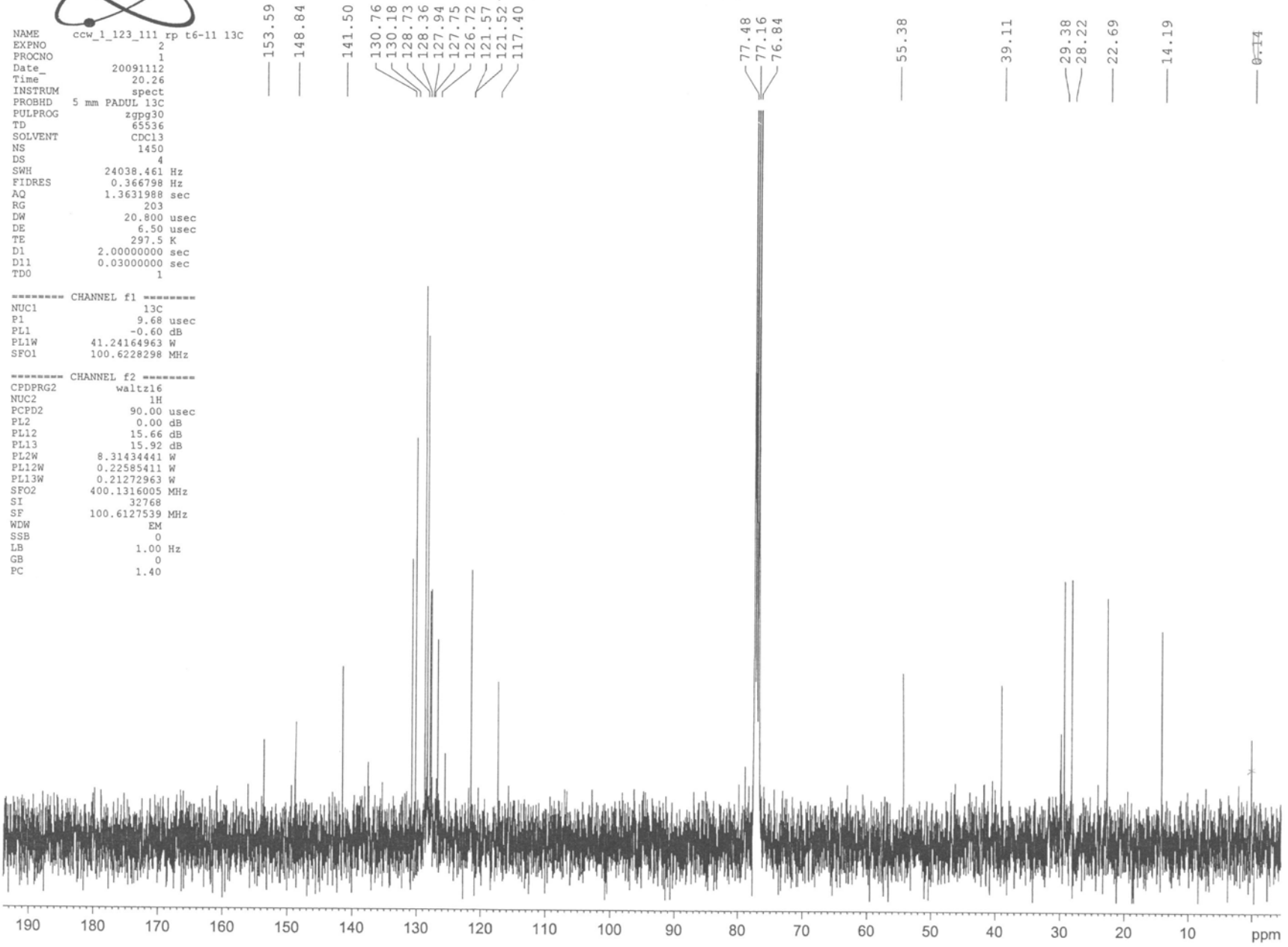
```
NAME      ccw_1_123_111 rp t6-11
EXPNO                  1
PROCNO                 1
Date_           20091112
Time              17.26
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    13
DS                     0
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG                   203
DW              50.000 usec
DE               6.50 usec
TE              295.8 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                14.83 usec
PL1                0.00 dB
PL1W         8.31434441 W
SFO1        400.1316005 MHz
SI                 65536
SF          400.1300041 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```





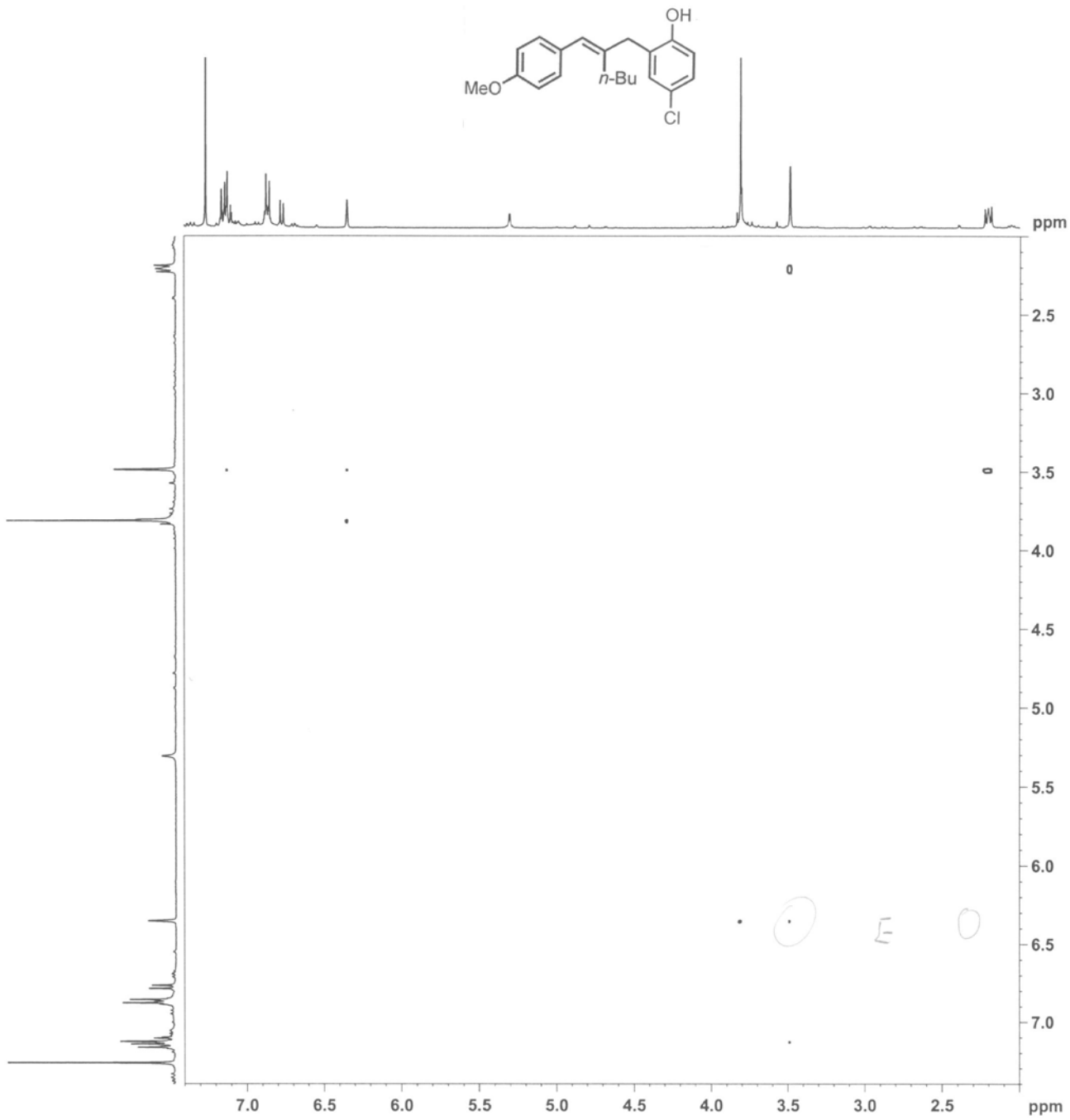
B2C






```
NAME        ccw_1_123_111 rp t6-11 13C
EXPNO                 2
PROCNO                1
Date_          20091112
Time              20.26
INSTRUM           spect
PROBHD    5 mm PADUL 13C
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1450
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                297.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                 9.68 usec
PL1                -0.60 dB
PL1W        41.24164963 W
SFO1       100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              90.00 usec
PL2                 0.00 dB
PL12               15.66 dB
PL13               15.92 dB
PL2W         8.31434441 W
PL12W        0.22585411 W
PL13W        0.21272963 W
SFO2       400.1316005 MHz
SI                32768
SF         100.6127539 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

820

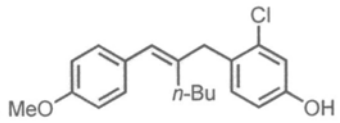


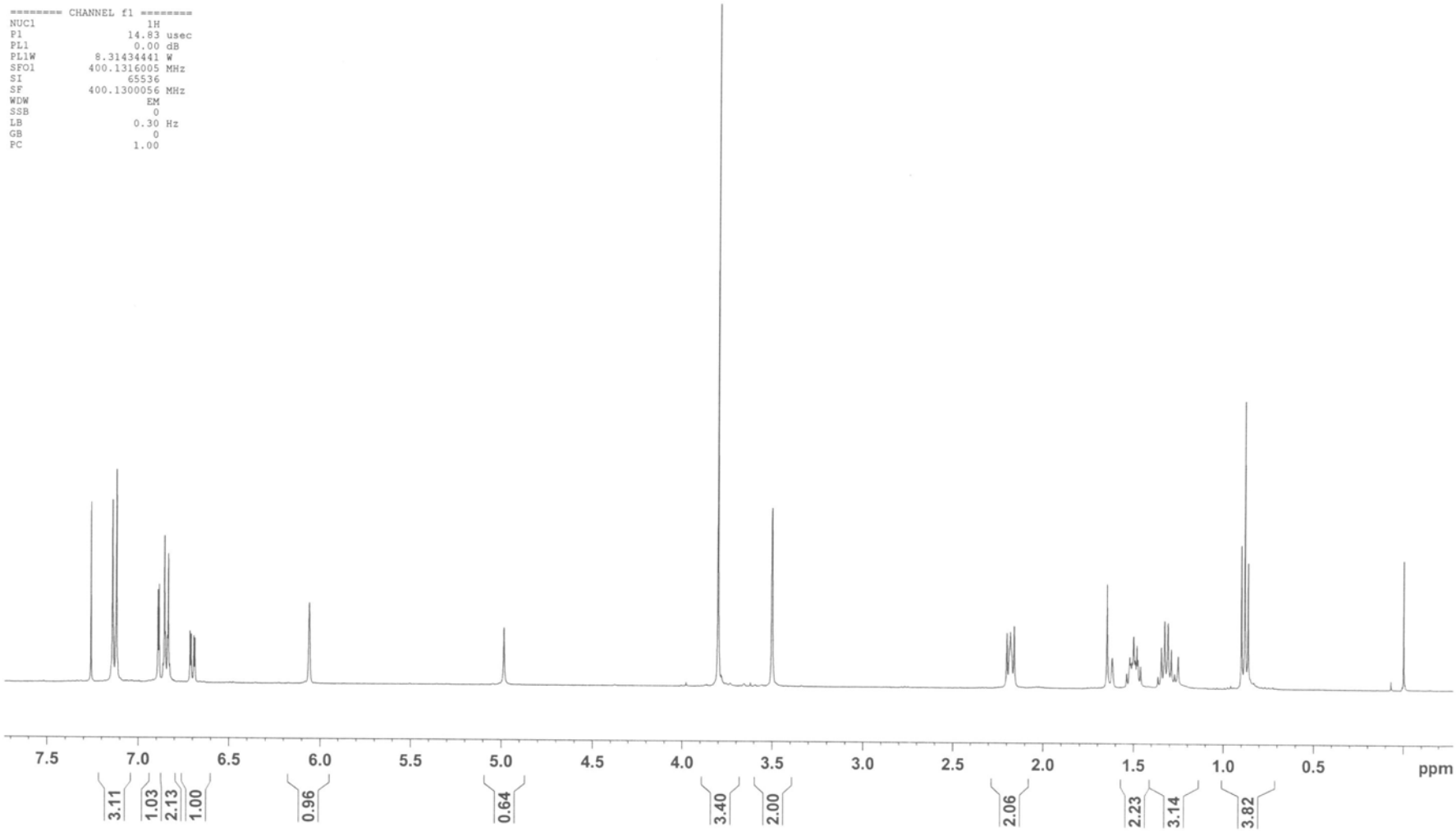
Bruker Advance III 400

NAME	ccw_1_123_111 rp t6-11
EXPNO	6
PROCNO	1
Date_	20091112
Time	17.43
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	296.3 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	17
SFO1	400.1318 MHz
FIDRES	240.292191 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300009 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

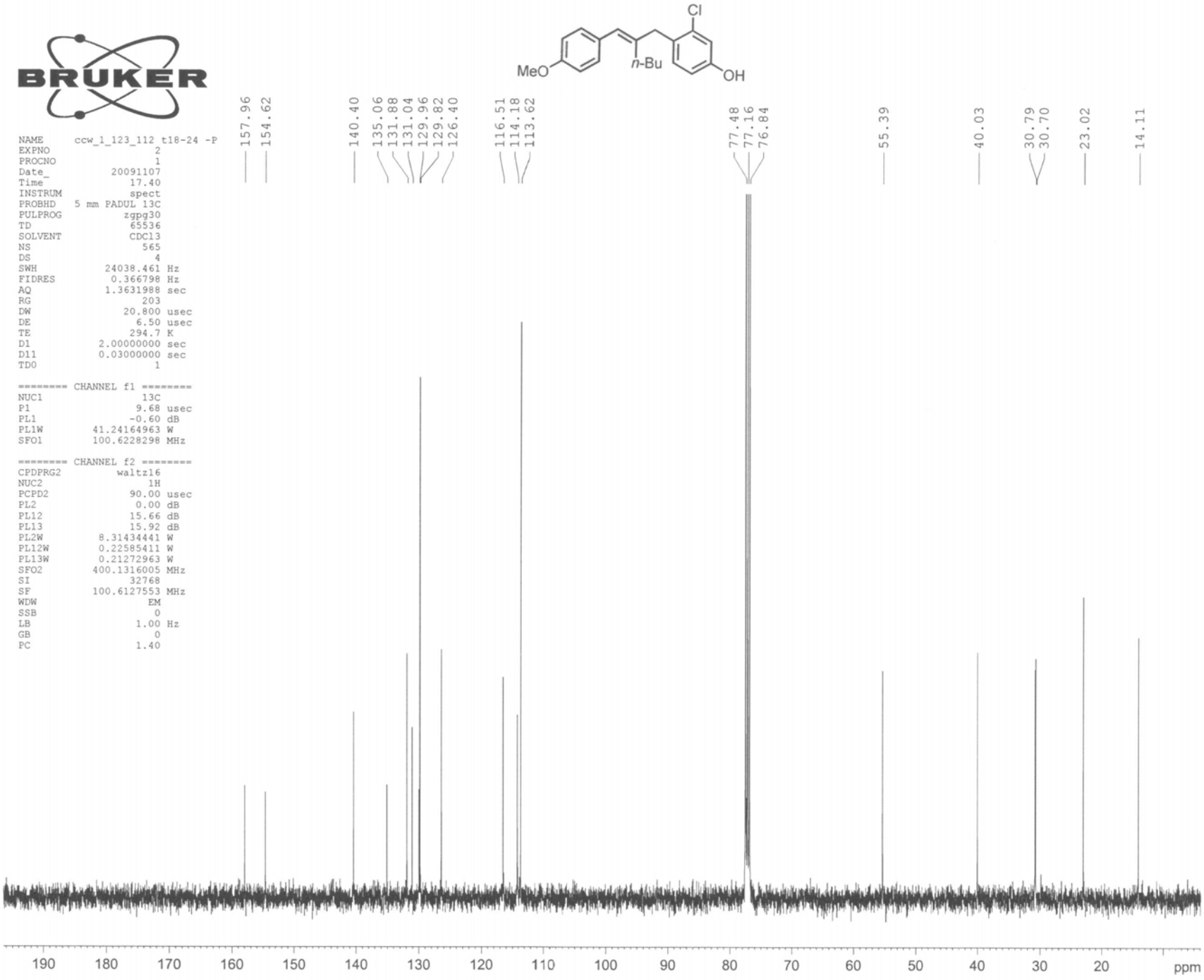
B2C

```
NAME       ccw_1_123_112 t18-24 -P
EXPNO                   1
PROCNO                  1
Date_            20091104
Time_               13.29
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     17
DS                      0
SWH             10000.000 Hz
FIDRES          0.152588 Hz
AQ              3.2768500 sec
RG                    203
DW                 50.000 usec
DE                  6.50 usec
TE                 673.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  14.83 usec
PL1                  0.00 dB
PL1W          8.31434441 W
SFO1         400.1316005 MHz
SI                  65536
SF           400.1300056 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```





B2d


BRUKER
Cl
MeO
n-Bu
OH
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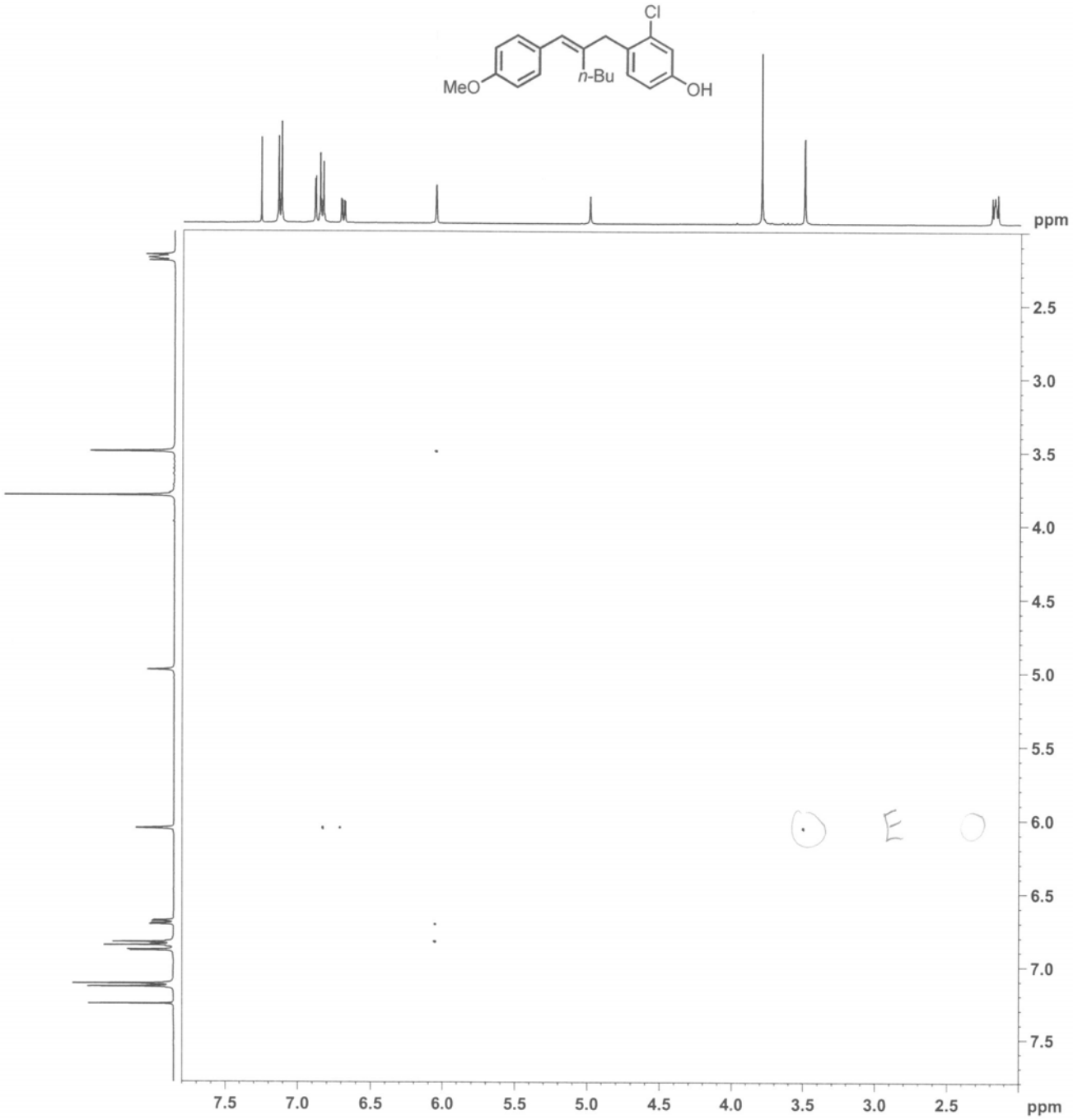
154.62
140.40
135.06
131.88
131.04
129.96
129.82
126.40
116.51
114.18
113.62
77.48
77.16
76.84
55.39
40.03
30.79
30.70
23.02
14.11
NAME ccw_1_123_112 t18-24 -P
EXPNO 2
PROCNO 1
Date_ 20091107
Time 17.40
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 565
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 294.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.68 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 15.66 dB
PL13 15.92 dB
PL2W 8.31434441 W
PL12W 0.22585411 W
PL13W 0.21272963 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127553 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

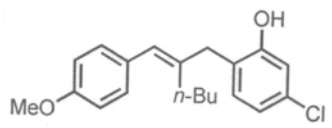


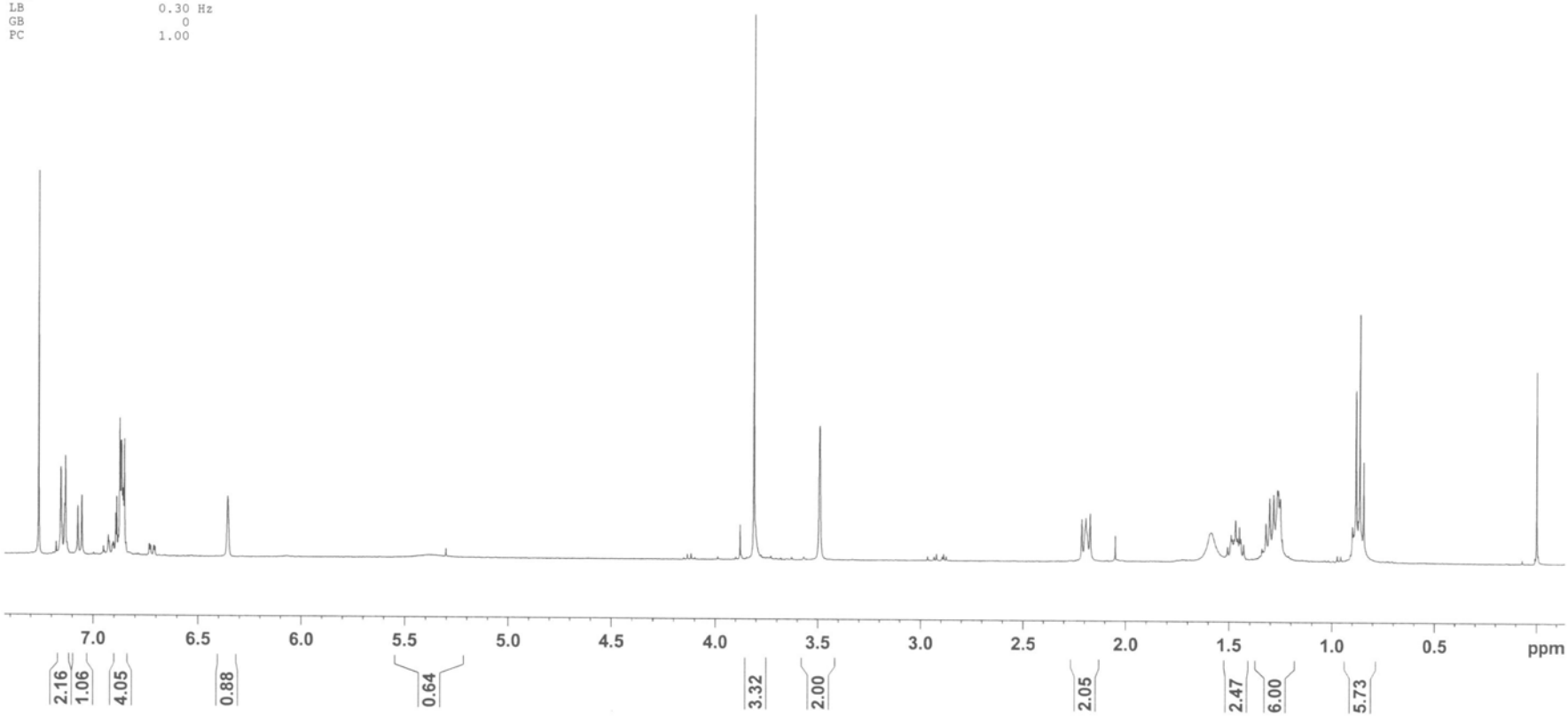
Bruker Advance III 400

NAME	ccw_1_123_112 t18-24 -P	
EXPNO	6	
PROCNO	1	
Date_	20091106	
Time	20.35	
INSTRUM	spect	
PROBHD	5 mm PADUL 13C	
PULPROG	noesyph	
TD	2048	
SOLVENT	CDCl3	
NS	16	
DS	4	
SWH	4084.967	Hz
FIDRES	1.994613	Hz
AQ	0.2507252	sec
RG	203	
DW	122.400	usec
DE	6.50	usec
TE	294.4	K
D0	0.00010352	sec
D1	2.00000000	sec
D8	0.30000001	sec
IN0	0.00024480	sec
======== CHANNEL f1 ========		
NUC1	1H	
P1	14.83	usec
PL1	0.00	dB
PL1W	8.31434441	W
SFO1	400.1318419	MHz
ND0	1	
TD	35	
SFO1	400.1318	MHz
FIDRES	116.713348	Hz
SW	10.209	ppm
FnMODE	States-TPPI	
SI	1024	
SF	400.1300048	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	
PC	1.00	
SI	1024	
MC2	States-TPPI	
SF	400.1300048	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	

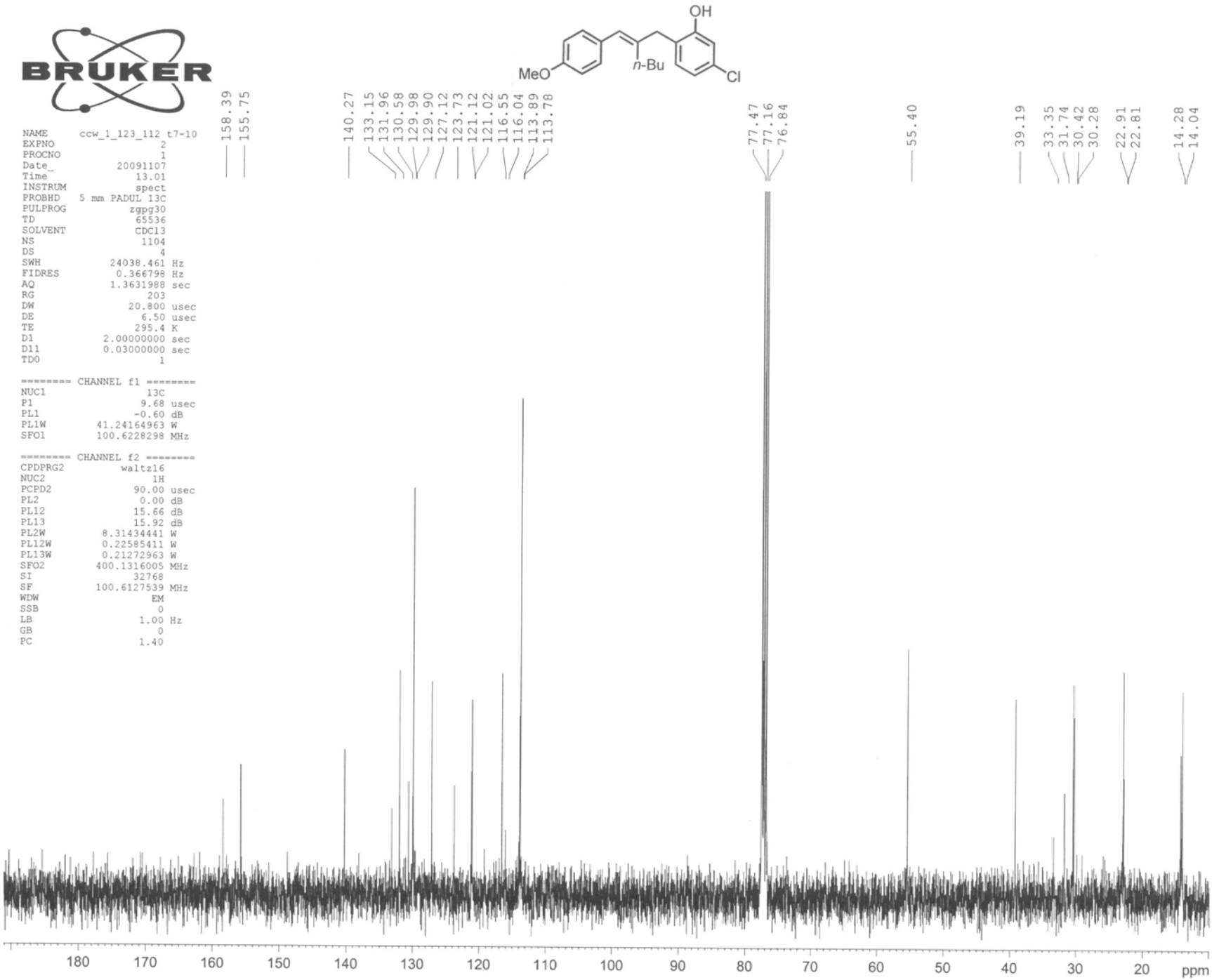
B2d

NAME	ccw_1_123_112 t7-10
EXPNO	1
PROCNO	1
Date_	20091103
Time	15.05
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	12
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	203
DW	50.000 usec
DE	6.50 usec
TE	673.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1316005 MHz
SI	65536
SF	400.1300045 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

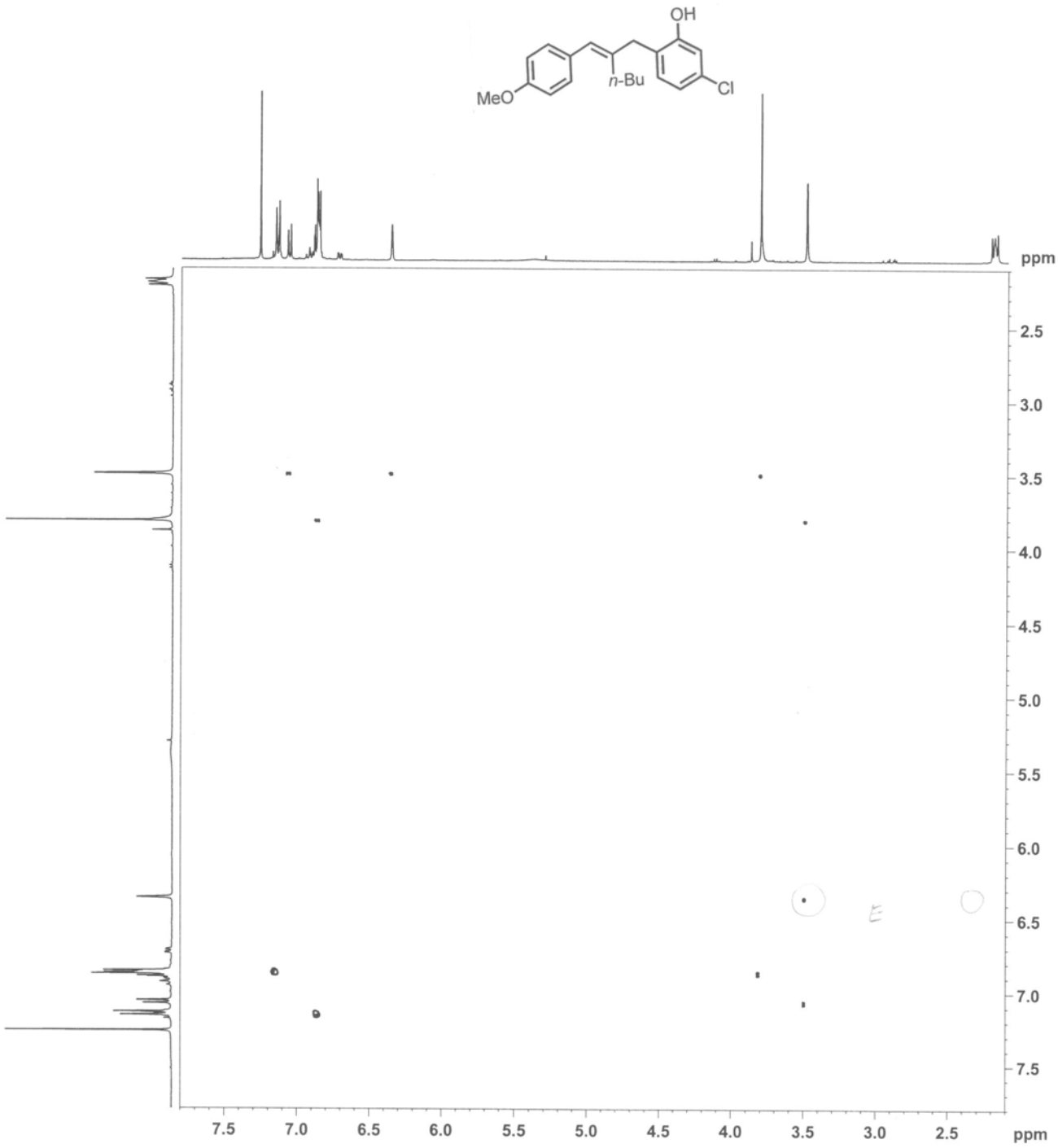




B2e


BRUKER
OH
MeO
n-Bu
Cl
158.39
155.75
140.27
133.15
131.96
130.58
129.98
129.90
127.12
123.73
121.12
121.02
116.55
116.04
113.89
113.78
77.47
77.16
76.84
55.40
39.19
33.35
31.74
30.42
30.28
22.91
22.81
14.28
14.04
NAME ccw_1_123_112 t7-10
EXPNO 2
PROCNO 1
Date_ 20091107
Time 13.01
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1104
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 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.68 usec
PL1 -0.60 dB
PL1W 41.24164963 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 0.00 dB
PL12 15.66 dB
PL13 15.92 dB
PL2W 8.31434441 W
PL12W 0.22585411 W
PL13W 0.21272963 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127539 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

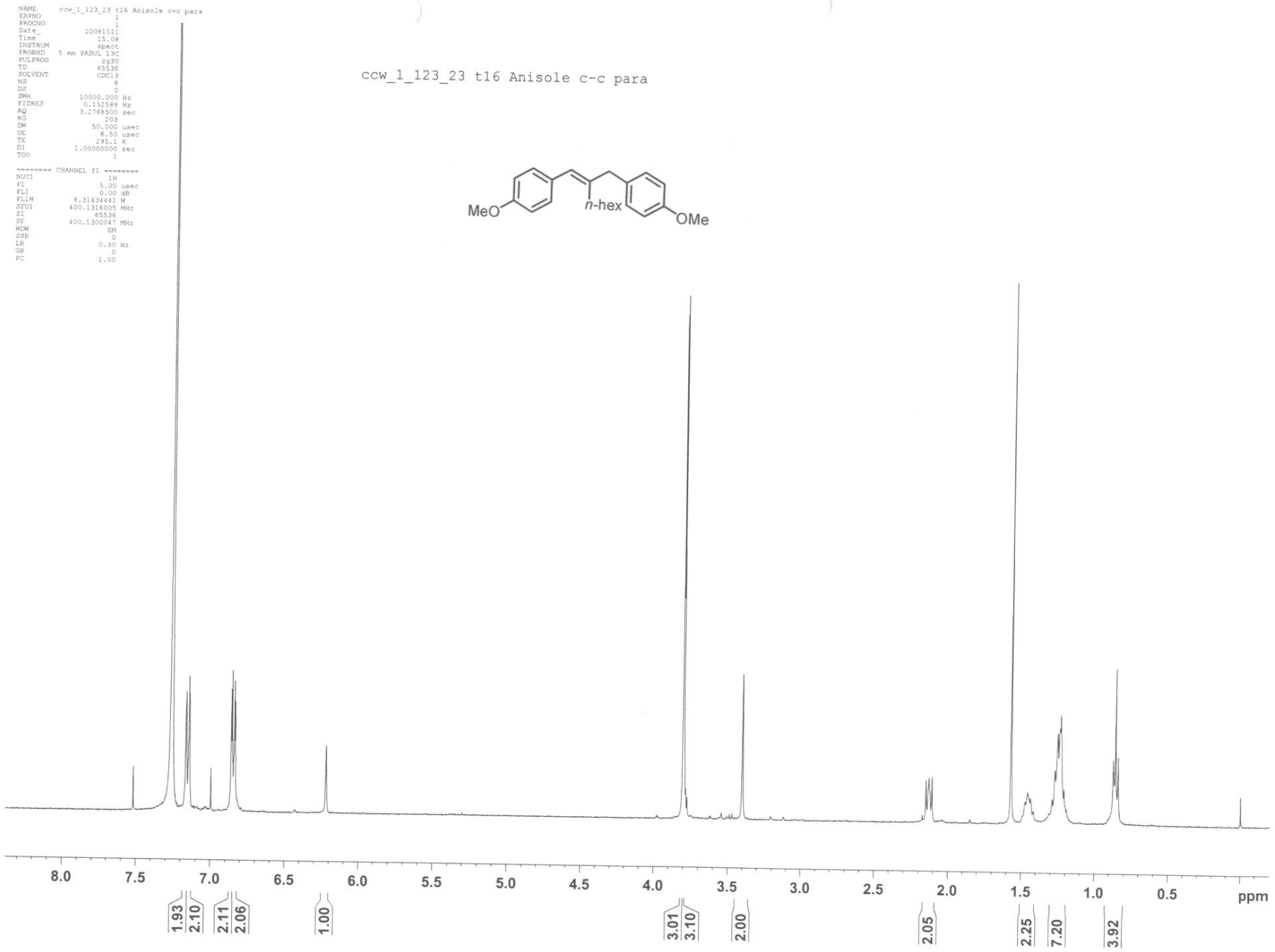
B2e



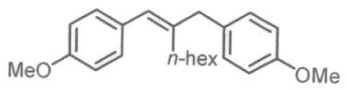
Bruker Advance III 400

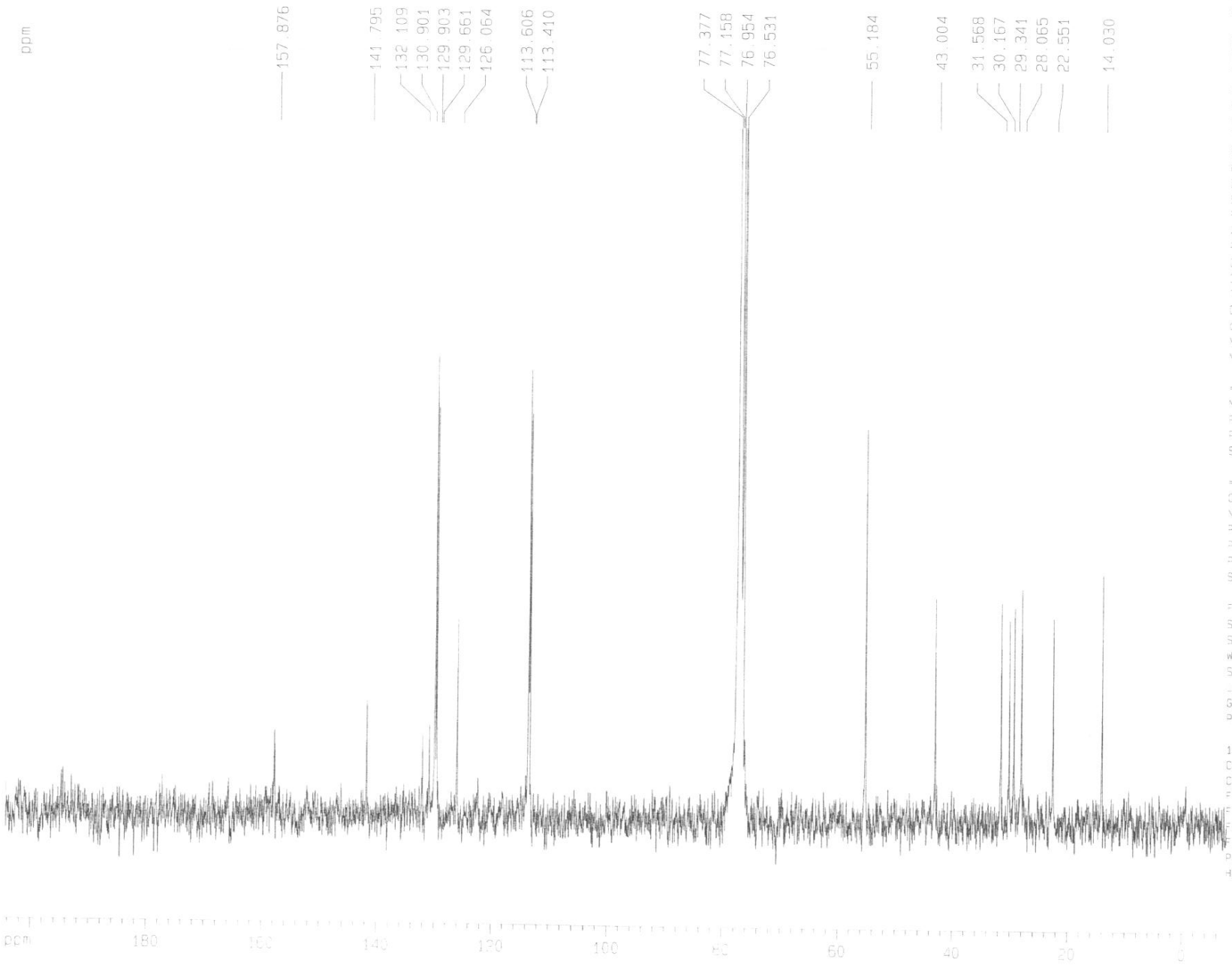
NAME	ccw_1_123_112 t7-10
EXPNO	6
PROCNO	1
Date_	20091103
Time	15.58
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	203
DW	122.400 usec
DE	6.50 usec
TE	673.2 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	90
SFO1	400.1318 MHz
FIDRES	45.388527 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300009 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300008 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

B2e


NAME ccw_1_123_23 t16 Anisole c-c para
EXPNO 1
PROCNO 1
Date_ 20081101
Time 15.08
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 203
DW 50.000 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 5.00 usec
PL1 0.00 dB
PL1W 8.31434441 W
SFO1 400.1316005 MHz
SI 65536
SF 400.1300047 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ccw_1_123_23 t16 Anisole c-c para
MeO
n-hex
OMe
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.93
2.10
2.11
2.06
1.00
3.01
3.10
2.00
2.05
2.25
7.20
3.92

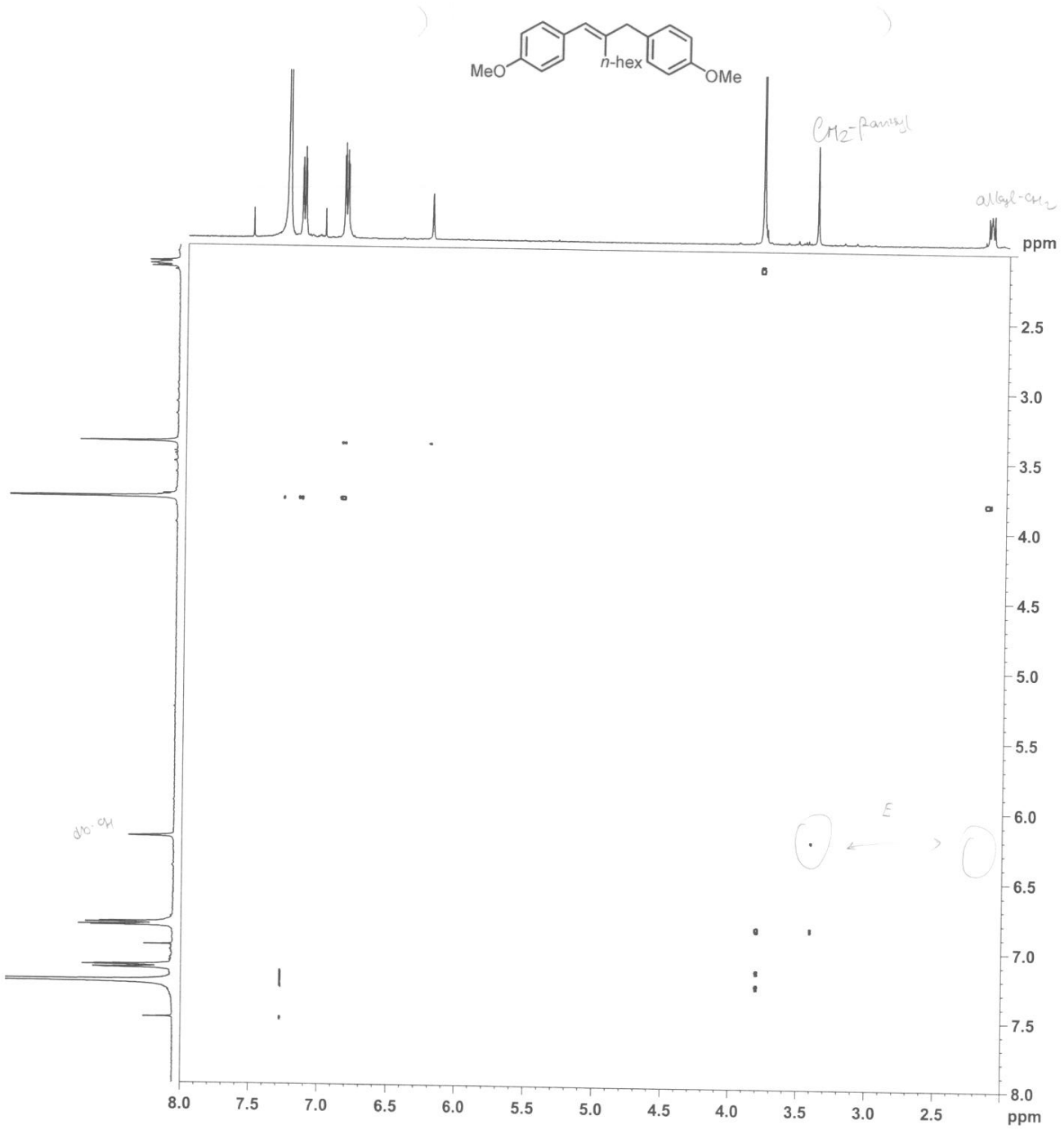
B102






```
Current Data Parameters
NAME        ccw_1_123_23 t16 13c
EXPNO                          2
PROCNO                         1

F2 - Acquisition Parameters
Date_               20081011
Time                   10.35
INSTRUM              dpx300
PROBHD     5 mm BBO BB-1H
PULPROG              zgdc
TD                    65536
SOLVENT              CDCl3
NS                     2274
DS                        0
SWH              22675.736 Hz
FIDRES           0.346004 Hz
AQ               1.4451888 sec
RG                     8192
DW                 22.050 usec
DE                  6.00 usec
TE                 296.2 K
D1              1.00000000 sec
D11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    3.00 usec
PL1                  -6.00 dB
SFO1           75.4745111 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                 120.00 dB
PL12                 19.00 dB
SFO2           300.1315007 MHz

F2 - Processing parameters
SI                    65536
SF             75.4677546 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                   23.00 cm
CY                   50.00 cm
F1P                204.375 ppm
F1                15423.75 Hz
F2P                 -7.698 ppm
F2                 -580.97 Hz
PPMCM              9.22060 ppm/cm
HZCM             695.85785 Hz/cm
```

B102



BRUKER

NAME	ccw_1_123_23 t16 Anisole c-c para
EXPNO	6
PROCNO	1
Date_	20081011
Time	15.10
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	noesyph
TD	2048
SOLVENT	CDCl3
NS	16
DS	4
SWH	4084.967 Hz
FIDRES	1.994613 Hz
AQ	0.2507252 sec
RG	64
DW	122.400 usec
DE	6.50 usec
TE	295.2 K
D0	0.00010352 sec
D1	2.00000000 sec
D8	0.30000001 sec
IN0	0.00024480 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.83 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1318419 MHz
ND0	1
TD	13
SFO1	400.1318 MHz
FIDRES	314.228241 Hz
SW	10.209 ppm
FnMODE	States-TPPI
SI	1024
SF	400.1300029 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.00
SI	1024
MC2	States-TPPI
SF	400.1300028 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

Bla2