Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

$CoBr_2$/TMTU/Zinc Catalysed-Pauson—Khand Reaction

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General Information

The boiling point of petroleum ether is between 60-90 ºC. Silica gel (200-300 mesh) for purification was purchased from Tsingtao Haiyang Chemicals (China). Solvent purification was conducted according to *Purification of Laboratory Chemicals* (Peerrin, D. D.; Armarego, W. L. and Perrins, D. R., Pergamon Press: Oxford, 1980). Yields refer to chromatographically and spectroscopically (1H NMR) homogeneous materials. Reactions were monitored by Thin Layer Chromatography on plates (GF254) supplied by Yantai Chemicals (China) using UV light as visualizing agent and basic aqueous solution of potassium permanganate, and heat as developing agents..

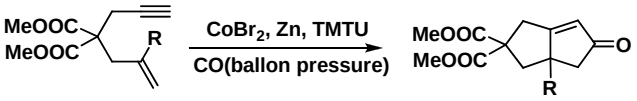
^{1}H-NMR and ^{13}C-NMR were recorded on 400.1 MHz and 100.6 MHz with Brucker AVANCE III spectrometer. TMS was used as internal standard for ^{1}H NMR (0 ppm), and solvent signal was used as reference for ^{13}C NMR ($CDCl_3$, 77.0 ppm). Infrared (IR) spectra were recorded on a Thermo Nicolet Avatar 330 FT-IR spectrometer. High-resolution mass spectra (HRMS) were recorded on a Bruker Apex IV FTMS mass spectrometer using ESI (electrospray ionization). Cobalt bromide was purchased from Alfa Aesar. Tetramethyl thiourea was purchased from Acros. Zinc dust was purchased from Beijing Yili Chemicals (China).

Experimental Data

Optimization of the reaction conditions:

To a mixture of anhydrous $CoBr_2$ (0.100 mmol, 21.9 mg, 10 mol%), TMTU (0.6 mmol, 79.3mg, 0.60 equiv) and zinc dust (2.0 mmol, 130 mg, 2.0 equiv) in flame-dried 10 mL-schlenk tube, dry solvent (1.5 mL) was added under N_2 balloon protection, then degased with CO balloon, and the mixture was stirred under a balloon pressure of CO at 70 ^oC for 3h after the color of the reaction mixture was changed from original deep green to colorless or yellowish (except DMF and CH_3CN). To this solution enyne (0.5 mmol, 1.0 equiv) was added, and the formed mixture was stirred at 70 ^oC for additional 3-24 hours. After cooling to room temperature, the reaction mixture was purified by column chromatography on silica gel with gradient solvent (petroleum ether and ethyl acetate) to give the corresponding product. For the reactions with molecular sieves (entries 13 and 17), their procedures are given in Page 6.

Table 1. **CoBr2/TMTU/Zinc Catalyzed PK Reaction: Survey of Best Condition**



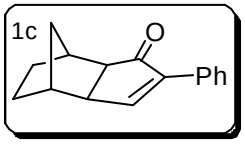
entry	solvent	$CoBr_2$/ligand (loading)	condition	time / h	yield (%)
1	CH_3CN	1/6 (20%)	70 ^oC, R = H	12h	N. R.
2	DMF	1/6 (20%)	70 ^oC, R = H	12h	N. R.
3	THF	1/6 (20%)	65 ^oC, R = H	24h	18
4	PhH	1/6 (20%)	70 ^oC, R = H	3h	86
5	PhMe	1/6 (20%)	70 ^oC, R = H	3h	87
6	DCE	1/6 (20%)	70 ^oC, R = Me	3h	70 (85)
7	PhMe	1/6 (20%)	70 ^oC, R = Me	3h	88
8	PhH	1/6 (20%)	70 ^oC, R = Me	3h	70 (73)
9	PhMe	1/1 (20%)	70 ^oC, R = Me	3h	N. R.
10	PhMe	1/2 (20%)	70 ^oC, R = Me	3h	N. R.
11	PhMe	1/4 (20%)	70 ^oC, R = Me	3h	71 (77)
12	PhMe	1/10 (20%)	70 ^oC, R = Me	3h	75 (79)
13	PhMe	1/6 (10%)	70 ^oC, R = Me	3h	81
14	PhMe	1/6 (10%)	70 ^oC, R = Me molecular seives	3h	92
15	PhMe	1/6 (5%)	70 ^oC, R = Me	3h	78 (84)
16	PhMe	1/6 (5%)	110 ^oC, R = Me	1h	68 (84)
17	PhMe	1/6 (5%)	70 ^oC, R = H molecular seives	3h	86
18	PhH	1/6 (5%)	70 ^oC, R = Me molecular seives	3h	35 (71)

[a] Reaction conditions: enyne (0.50 mmol), $CoBr_2$, TMTU, Zn under CO (balloon pressure).
[b] Isolated yield. [c] The yields in parentheses based on starting material recover.

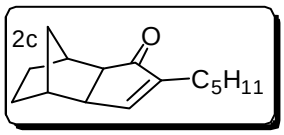
Triphenylphosphine (PPh_3), Triphenyl phosphite ($P(OPh)_3$), simple thiourea (TU) and dimethyl thiourea (DMTU) ligands were all tested in the same condition. But no reaction occurred when these ligands were used.

General procedure for the intermolecular reaction

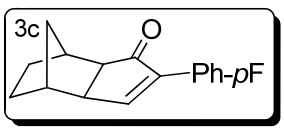
To a mixture of dry molecular sieves (30 mg), anhydrous $CoBr_2$ (0.100 mmol, 21.9 mg, 10 mol%), TMTU (0.6 mmol, 79.3mg, 0.60 equiv) and zinc dust (2.0 mmol, 130 mg, 2.0 equiv) in flame-dried 10 mL-schlenk tube, dry toluene (1.5 mL) was added under N_2 balloon protection, then degassed with CO balloon, and the mixture was stirred under a balloon pressure of CO at 70 ^oC for 3h after the color of the reaction mixture was changed from original deep green to colorless or yellowish. To this solution was added alkyne (1.00 mmol, 1.0 equiv) and olefin (2.00 mmol, 2.0 equiv), and the formed mixture was stirred at 70 ^oC for additional 3 hours. After cooling to room temperature, the reaction mixture was purified by column chromatography on silica gel with gradient solvent (petroleum ether and ethyl acetate) to give the corresponding product.



1c: Alkyne (93.6 mg, 0.916 mmol); eluent: petroleum ether/EtOAc = 30/1 to 25/1, product obtained 174.1 mg (0.776 mmol), yield 85%;mp 74-76 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.71-7.68 (m, 2H), 7.62 (d, *J* = 3.2 Hz, 1H), 7.38-7.29 (m, 3H), 2.69-2.67 (m, 1H), 2.49 (d, *J* = 3.6 Hz, 1H), 2.35 (d, *J* = 5.2 Hz, 1H), 2.26 (d, *J* = 4.0 Hz, 1H), 1.74-1.56 (m, 2H), 1.39-1.26 (m, 2H) 1.12 (dt, *J* = 10.4, 1.6 Hz, 1H), 0.99 (dt, *J* = 10.4, 1.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 208.9, 160.1, 146.1, 131.5, 128.3, 127.0, 54.9, 47.7, 39.4, 38.3, 31.2, 29.1, 28.4 ppm; IR (neat): 2955, 1694, 1296 (w), 1134 (w), 765, 690 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{16}H_{16}NaO$ $[M+Na]^+$: 247.1093, found 247.1091.[1]



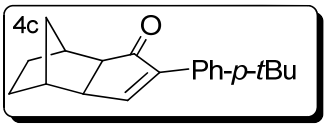
2c: **A**lkyne (103.7 mg, 1.08 mmol); eluent: petroleum ether/EtOAc = 40/1 to 30/1, product obtained 218.8 mg (1.00 mmol), yield 93%; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.02 (s, 1H), 2.49 (br, 1H), 2.30 (br, 1H), 2.14-2.01 (m, 4H), 1.63-1.46 (m, 2H), 1.43-1.36 (m, 2H), 1.25-1.18 (m, 6H), 0.93-0.79 (m, 5H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 211.0, 158.5, 149.4, 53.8, 48.0, 38.9, 38.0, 31.5, 30.9, 29.0, 28.4, 27.4, 24.6, 22.3, 13.9 ppm; IR (neat): 2951, 2926, 2868, 1694 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{15}H_{23}O$ $[M+H]^+$: 219.1743, found 219.1740.[2]



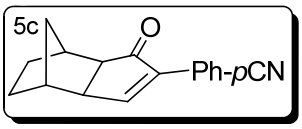
3c: Alkyne (116 mg, 0.97 mmol); eluent: petroleum ether/EtOAc = 35/1, product obtained 203 mg (0.84 mmol), yield 86%; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.72- 7.69 (m, 2H), 7.61 (d, *J* = 2.8 Hz, 1H), 7.08-7.04 (m, 2H), 2.71-2.69 (m, 1H), 2.50 (d, *J* = 3.6 Hz, 1H), 2.37 (d, *J* = 5.2 Hz, 1H), 2.28 (d, *J* = 4.0 Hz, 1H), 1.76-1.58 (m, 2H), 1.37-1.32 (m, 2H), 1,11 (d, *J* = 10.6 Hz, 1H), 1.02 (d, *J* = 10.6 Hz, 1H) ppm; ^{13}C

[1] Kobayashi, T.; Koga, Y.; Narasaka, K. *J. Organomet.Chem.* **2001**, *624*, 73.
[2] Periasamy, M.; Reddy, M. R.; Devasagayaraj, A. *Tetrahedron* **1994**, *50*, 6955.

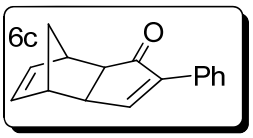
NMR (100 MHz, $CDCl_3$) δ = 209.0, 164.1 (d, *J* = 246 Hz), 159.8, 145.0, 128.9 (d, *J* = 8 Hz), 127.7, 115.4 (d, *J* = 22 Hz), 54.9, 47.7, 39.5, 38.4, 31.3, 29.2, 28.4 ppm; IR (neat): 2955, 1693, 1503, 1220, 870, 854, 844, 824, 809 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{16}H_{16}FO$ $[M+H]^+$: 243.1180, found 243.1178.



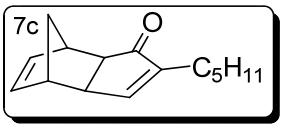
4c: Alkyne (186 mg, 1.17 mmol); eluent: petroleum ether/EtOAc = 40/1, product obtained 248 mg (0.88 mmol), yield 75%; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.65 (d, *J* = 8.4 Hz, 2H), 7.60 (d, *J* = 2.8 Hz, 1H), 7.41 (d, *J* = 8.4 Hz, 2H), 2.69-2.68 (m, 1H), 2.49 (d, *J* = 3.6 Hz, 1H), 2.36 (d, *J* = 5.2 Hz, 1H), 2.26 (d, *J* = 4.0 Hz), 1.74-1.57 (m, 2H), 1.32 (s, 9H) , 1.12 (d, *J* = 10.6 Hz, 1H), 0.99 (d, *J* = 10.6 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 209.2, 159.6, 151.5, 146.0, 128.7, 126.8, 125.4, 54.9, 47.8, 39.5, 38.4, 34.6, 31.3, 29.2, 28.4 ppm; IR (neat): 2959, 1694, 844, 830, 816 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{20}H_{25}O$ $[M+H]^+$: 281.1900, found 281.1900.



5c: Alkyne (119.9 mg, 0.94 mmol); eluent: petroleum ether/EtOAc = 15/1 to 12/1, product obtained 207.5 mg (0.83 mmol), yield 88%; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.85 (d, *J* = 8.4 Hz, 2H), 7.79 (d, *J* = 3.2 Hz, 1H), 7.67 (d, *J* = 8.4 Hz, 2H), 2.78 (dd, *J* = 4.8, 3.2 Hz, 1H), 2.52 (d, *J* = 3.6 Hz, 1H), 2.41 (d, 5.2 Hz, 1H), 2.33 (d, 4.0 Hz, 1H), 1.78-1.60 (m, 2H), 1.43-1.31 (m, 2H), 1.10 (d, *J* = 10.8 Hz, 1H), 1.05 (d, *J* = 10.8 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 208.2, 162.5, 144.5, 135.9, 132.2, 127.6, 118.8, 111.9, 55.0, 48.0, 39.6, 38.4, 31.4, 29.2, 28.3 ppm; IR (neat): 2954, 2225, 1691, 871, 856, 849, 841, 822 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{17}H_{16}NO$ $[M+H]^+$: 250.1226, found 250.1227.



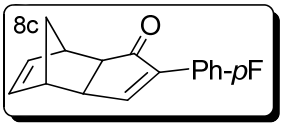
6c: **A**lkyne (156.4 mg, 1.53 mmol); eluent: petroleum ether/EtOAc = 50/1, product obtained 257 mg (1.16 mmol), yield 76%; mp 77-79 ºC; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.70-7.68 (m, 3H), 7.39-7.32 (m, 3H), 6.32 (s, 1H), 6.25 (s, 1H), 3.02 (s, 1H), 2.83 (s, 1H), 2.78 (s, 1H), 2.46 (d, J = 4.0 Hz, 1H), 1.43 (d, *J* = 9.2 Hz, 1H), 1.34 (d, *J* = 9.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 207.6, 159.8, 147.2, 138.5, 137.1, 131.6, 128.4(2), 128.3(5), 127.0, 53.5, 47.1, 44.1, 43.3, 41.3 ppm; IR (neat): 2967, 1702, 757 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{16}H_{14}NaO$ $[M+Na]^+$: 245.0937, found 245.0935.[3]



7c: Alkyne (128.2 mg, 1.33 mmol); eluent: petroleum ether/EtOAc = 60/1 to 40/1, product obtained 242 mg (1.12 mmol), yield 84%; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.16 (d, *J* = 1.2 Hz, 1H), 6.29-6.27 (m, 1H), 6.21-6.18 (m, 1H), 2.90 (s, 1H), 2.70 (br, 1H), 2.66 (br, 1H), 2.28-2.27 (m, 1H), 2.17-2.13 (m, 1H), 1.51-1.44 (m, 2H), 1.38-1.26 (m, 6H), 1.22-1.20 (m, 1H), 0.90 (t, *J* = 6.8 Hz, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 209.9, 158.6, 150.8, 138.3, 137.0, 52.5, 47.6, 43.6, 42.9, 41.1, 31.6,

[3] Park, K. H.; Chung, Y. K. *Adv. Syn. Catal.* **2005**, *347*, 854.

27.4, 24.9, 22.4, 13.9 ppm; IR (neat): 2951, 2913, 2851, 1698, 1453, 1366 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{15}H_{20}NaO$ $[M+Na]^+$: 239.1406, found 239.1404.[4]

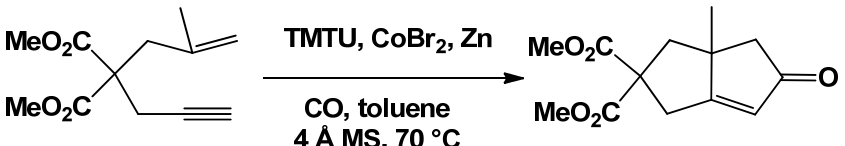


8c: Alkyne (116.4 mg, 0.97 mmol); eluent: petroleum ether/EtOAc = 40/1, product obtained 156.2 mg (0.650 mmol), yield 67%; ^1H NMR (400 MHz, $CDCl_3$) δ = 7.72- 7.69 (m, 2H), 7.66 (d, *J* = 2.8 Hz, 1H), 7.08-7.03 (m, 2H), 6.34-6.32 (m, 1H), 6.26-6.24 (m, 1H), 3.01 (s, 1H), 2.84-2.82 (m, 1H), 2.78 (s, 1H), 2.47 (d, *J* = 5.2 Hz, 1H), 1.44 (d, *J* = 9.4 Hz, 1H), 1.32 (d, *J* = 9.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 207.6, 164.1 (d, *J* = 247 Hz), 159.5, 146.1, 138.6, 137.2, 128.9 (d, *J* = 8 Hz), 127.8 (d, *J* = 4 Hz), 115.5 (d, *J* = 22 Hz), 53.6, 47.1, 44.1, 43.4, 41.4 ppm; IR (neat): 2980, 2967, 1688, 1507, 1230, 1165, 841, 715 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{16}H_{14}FO$ $[M+H]^+$: 241.1023, found 241.1021.

General procedure for the intramolecular reaction

To a solution of dry molecular sieves (30 mg), anhydrous $CoBr_2$ (0.05 mmol, 10.9 mg, 10 mol%), TMTU (0.30 mmol, 39.7 mg, 0.6 equiv.) and zinc dust (1.0 mmol, 65 mg, 2.0 equiv.) in a flame-dried 10 mL-schlenk tube, dry toluene (1.0 mL) was added under N_2 balloon protection, then degassed with CO balloon, and the mixture was stirred under a balloon pressure of CO at 70 °C for 3h after the color of the reaction mixture was changed from original deep green to colorless or yellowish. To this solution was added enyne (0.5 mmol, 1.0 equiv.), and the formed mixture was stirred at 70 °C for additional 3-5h hours. After cooling to room temperature, the reaction mixture was purified by column chromatography on silica gel with gradient solvent (petroleum ether and ethyl acetate) to give the corresponding product.

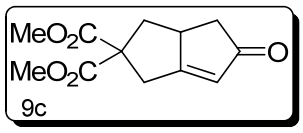
It is worthwhile to mention that when the amount of zinc was reduced from 1.0 mmol to 0.2 mmol, the annulation of substrate **10a** could also proceed smoothly under the identical conditions listed above. However, further reduction of the zinc amount to less than 0.1 mmol, no desired reaction occurred.



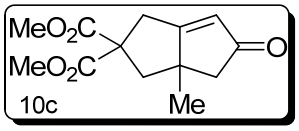
10a 10c

entry	$CoBr_2$ loading	Co:Zn	yield
1	10 mol %	1/20	91%
2	10 mol %	1/2	91%
3	10 mol %	1/1	trace

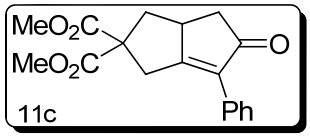
[4] Billington, D. C.; Heps, I. M.; Pauson, P. L.; Thomson, W.; Willison, D. *J. Organomet.Chem.* **1988**, *354*, 233.



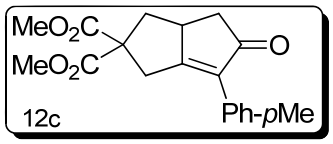
9c: Substrate (89.5 mg, 0.426 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 3/1, product obtained 87.3 mg (0.366 mmol), yield 86%; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.94 (d, *J* = 1.2 Hz, 1H), 3.80 (s, 3H), 3.76 (s, 3H), 3.39 (d, *J* = 19.0 Hz, 1H), 3.30 (d, *J* = 19.0 Hz, 1H), 3.13-3.08 (m, 1H), 2.85 (dd, *J* = 12.8, 7.6 Hz, 1H), 2.66 (dd, *J* = 17.6, 6.4 Hz, 1H), 2.16 (dd, *J* = 17.6, 3.2 Hz, 1H), 1.78 (t, *J* = 12.8 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 209.3, 185.1, 171.8, 171.1, 125.6, 60.6, 53.2, 53.1, 44.9, 42.0, 38.9, 35.2 ppm; IR (neat): 2956, 1731, 1707, 1636, 1254 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{12}H_{14}NaO_5$ $[M+Na]^+$: 261.0733, found 261.0731.[5]



10c:[5] Substrate (289 mg, 1.29 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 4/1, product obtained 298 mg (1.18 mmol), yield 91%; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.84 (d, *J* = 0.8 Hz, 1H), 3.81 (s, 3H), 3,74 (s, 3H), 3.53 (d, *J* = 17.6 Hz, 1H), 3.25 (d, *J* = 17.6 Hz, 1H), 2.62 (d, *J* = 13.6 Hz, 1H), 2.39 (s, 2H), 2.26 (d, *J* = 13.6 Hz, 1H), 1.16 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 208.7, 188.0, 171.8, 171.5, 124.1, 59.7, 53.1, 53.0, 51.6, 49.6, 44.3, 34.1, 26.3 ppm; IR (neat): 2956, 1732, 1710, 1636, 1252 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{13}H_{16}NaO_5$ $[M+Na]^+$: 275.0890, found 275.0888.[5]

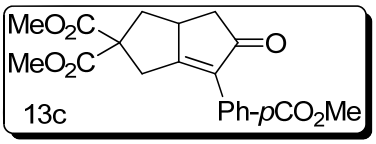


11c: Substrate (222.5 mg, 0.777 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 4/1, product obtained 220 mg (0.700 mmol), yield 90%; mp 125-126 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.57 (d, *J* = 7.2 Hz, 2H), 7.43 (dd, *J* = 7.2, 7.2 Hz, 2H), 7.35 (dd, *J* = 7.2, 7.2 Hz, 1H), 3.83 (s, 3H), 3.72 (s, 3H), 3.69 (d, *J* = 19.2 Hz, 1H), 3.33 (d, *J* = 19.2 Hz, 1H), 3.15-3.13 (m, 1H), 2.88-2.80 (m, 2H), 2.34 (dd, *J* = 17.6, 3.2 Hz, 1H), 1.80 (t, *J* = 12.8 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 207.0, 178.6, 172.0, 171.1, 135.6, 130.8, 128.4(3), 128.4(0), 128.2, 61.2, 53.3, 53.1, 42.8, 42.6, 38.9, 36.0 ppm; IR (neat): 1732, 1702, 1653, 1276 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{18}H_{19}O_5$ $[M+H]^+$: 315.1227, found 315.1229.[5]

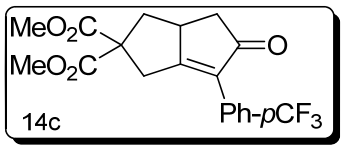


12c: Substrate (130.9 mg, 0.436 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 4/1, product obtained 134.2 mg (0.409 mmol), yield 94%; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.47 (d, *J* = 8.0 Hz, 2H), 7.22 (d, *J* = 8.0 Hz, 2H), 3.82 (s, 3H), 3.71 (s, 3H), 3.68 (d, *J* = 19.2 Hz, 1H), 3.32 (d, *J* = 19.2 Hz, 1H), 3.13-3.11 (m, 1H), 2.86-2.79 (m, 2H), 2.36 (s, 3H), 2.31 (dd, *J* = 18.0, 2.0 Hz, 1H), 1.79 (t, 12.8 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 207.1, 177.7, 172.0, 171.1, 138.1, 135.4, 129.1, 128.3, 128.0, 61.2, 53.3, 53.0, 42.7, 42.6, 38.9, 36.0, 21.3 ppm; IR (neat): 1732, 1701, 1653, 1275, 818 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{19}H_{21}O_5$ $[M+H]^+$: 329.1384, found 329.1382.

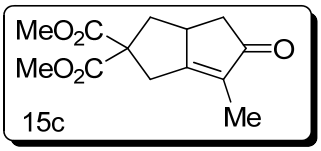
[5] Tang, Y. F.; Deng, L. J.; Zhang, Y. D.; Dong, G. B.; Chen, J. H.; Yang, Z. *Org. Lett*. **2005**, *7*, 593.



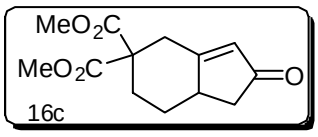
13c: Substrate (267 mg, 0.775 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc/CH_2Cl_2 = 3/1/1, product obtained 255 mg (0.685 mmol), yield 88%; mp 115-117 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 8.08 (d, *J* = 8.8 Hz, 2H), 7.66 (d, *J* = 8.4 Hz, 2H), 3.93 (s, 3H), 3.84 (s, 3H), 3.73 (s, 3H), 3.72 (d, *J* = 19.4 Hz, 1H), 3.34 (d, *J* = 19.4 Hz, 1H), 3.19 (m, 1H), 2.91 (m, 2H), 2.36 (dd, *J* = 18.0, 3.2 Hz, 1H), 1.82 (t, *J* = 12.8, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 206.4, 180.5, 171.8, 170.9, 166.7, 135.2, 134.6, 130.0, 129.5, 128.2, 61.1, 53.3, 53.1, 52.1, 43.1, 42.5, 38.7, 36.1 ppm; IR (neat): 1731, 1707, 1277, 775 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{20}H_{21}O_7$ $[M+H]^+$: 373.1282, found 373.1282.



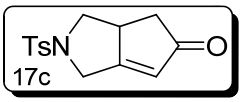
14c: Substrate (200.1 mg, 0.565 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 3/1, product obtained 199.8 mg (0.523 mmol), yield 93%; mp 113-115 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.70 (d, *J* = 12.0 Hz, 2H), 7.68 (d, *J* = 12.0 Hz, 2H), 3.84 (s, 3H), 3.73 (s, 3H), 3.71 (d, *J* = 19.4 Hz, 1H), 3.33 (d, *J* = 19.4 Hz, 1H), 3.20 (m, 1H), 2.91 (m, 2H), 2.36 (dd, *J* = 17.6, 3.2 Hz, 1H), 1.82 (d, *J* = 12.8, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 206.3, 180.6, 171.7, 170.9, 134.3(1), 134.2(6), 130.3 (q, *J* = 32 Hz), 128.6, 128.0 (q, *J* = 271 Hz), 125.3 (q, *J* = 4 Hz), 61.0, 53.3, 53.1, 43.1, 42.4, 38.6, 35.9 ppm; IR (neat): 1734, 1705, 1653, 1617, 1325, 733 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{19}H_{18}F_3O_5$ $[M+H]^+$: 383.1101, found 383.1105.



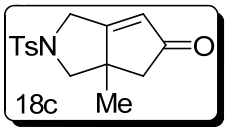
15c: Substrate (128.6 mg, 0.573 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 3/1, product obtained 120.8 mg (0.479 mmol), yield 83%; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 3.80 (s, 3H), 3.76 (s, 3H), 3.28 (d, *J* = 18.6 Hz, 1H), 3.21 (d, *J* = 18.6 Hz, 1H), 2.98 (br, 1H), 2.83 (dd, *J* = 12.6, 7.4 Hz, 1H), 2.68 (dd, *J* = 18.0, 6.2 Hz, 1H), 2.11 (d, *J* = 18.0 Hz, 1H), 1.72 (s, 3H), 1.69 (t, *J* =12.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 209.2, 177.3, 172.0, 171.4, 133.0, 60.8, 53.1, 53.0, 42.6, 41.3, 39.3, 34.1, 8.5 ppm; IR (neat): 1735, 1711, 1676, 1276 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{13}H_{17}O_5$ $[M+H]^+$: 253.1071, found 253.1069. [5]



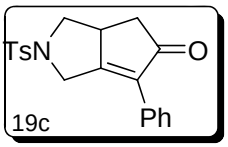
16c: Substrate (153.1 mg, 0.683 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 2.5/1, product obtained 123.9 mg (0.491 mmol), yield 72%; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 5.97 (s, 1H), 3.77 (s, 3H), 3.72 (s, 3H), 3.52 (d, *J* = 14.0 Hz, 1H), 2.69 (d, *J* = 14.0 Hz, 2H), 2.61-2.50 (m, 2H), 2.20-2.14 (m, 1H), 2.00-1.91 (m, 2H), 1.33 (ddd, *J* = 26.0, 13.2, 3.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 208.1, 178.6, 171.3, 170.0, 129.6, 56.6, 53.1, 52.8, 41.8, 40.6, 35.4, 30.7(4), 30.6(8) ppm; IR (neat): 2955, 1733, 1706, 1627, 1250 cm^{-1}; HRMS (ESI): *m/z* calcd for $C_{13}H_{16}NaO_5$ $[M+Na]^+$: 275.0890, found 275.0887. [5]



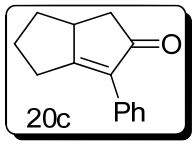
17c: Substrate (140.1 mg, 0.562 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 1/1 (0.3% Et_3N added), product obtained 138.4 mg (0.501 mmol), yield 89%; mp 158-159 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.74 (d, *J* = 8.2 Hz, 2H), 7.36 (d, *J* = 8.2 Hz, 2H), 5.99 (s, 1H), 4.36 (d, *J* = 16.4 Hz, 1H), 4.05-4.01 (m, 2H), 3.16-3.14 (m, 1H), 2.65-2.56 (m, 2H), 2.44 (s, 3H), 2.09 (dd, *J* = 17.6, 3.2 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 207.4, 178.7, 144.2, 133.4, 130.0, 127.4, 126.2, 52.4, 47.6, 43.9, 39.8, 21.5 ppm; IR (neat): 2922, 1711, 1650, 1598, 1343, 1159 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{14}H_{16}NO_3S$ $[M+H]^+$: 278.0845, found 278.0846. [5]



18c: Substrate (126.7 mg, 0.481 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 1/1 (0.3% Et_3N added), product obtained 102.4 mg (0.351 mmol), yield 73%; mp 115-116 ºC; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.74 (d, *J* = 8.0 Hz, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 5.86 (s, 1H), 4.36 (dd, *J* = 16.4, 1.6 Hz, 1H), 4.08 (d, *J* = 16.4 Hz, 1H), 3.70 (d, *J* = 9.2 Hz, 1H), 2.85 (d, *J* = 9.2 Hz, 1H), 2.44 (s, 3H), 2.39 (d, *J* = 17.6 Hz, 1H), 2.30 (d, *J* = 17.6 Hz, 1H), 1.18 (s, 3H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 207.0, 182.0, 144.0, 133.6, 129.9, 127.3, 124.6, 57.8, 49.0, 48.2, 46.3, 24.9, 21.5 ppm; IR (neat): 2972, 1714, 1653, 1598, 1345, 1170, 1154, 1093 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{15}H_{17}NNaO_3S$ $[M+Na]^+$: 314.0821, found 314.0824. [5]



19c: **S**ubstrate (144 mg, 0.443 mmol); eluent: petroleum ether/EtOAc/CH_2Cl_2 = 13/1/1 to petroleum ether/EtOAc = 3/1, product obtained 145.5 mg (0.412 mmol), yield 93%; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.72 (d, *J* = 8.0 Hz, 2H), 7.46-7.34 (m, 5H), 7.31 (d, *J* = 8.4 Hz, 2H), 4.65 (d, *J* = 16.8 Hz, 1H), 4.10-4.04 (m, 2H), 3.21-3.19 (m, 1H), 2.82 (dd, *J* = 17.6, 6.4 Hz, 1H), 2.64 (t, *J* = 10.2 Hz, 1H), 2.40 (s, 3H), 2.28 (dd, *J* = 17.6, 3.6 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 205.4, 171.9, 144.0, 136.0, 133.6, 129.9, 129.8, 128.9, 128.6, 128.1, 127.3, 51.9, 48.3, 41.8, 40.6, 21.4 ppm; IR (neat): 1708, 1662, 1598, 1345, 1158 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{20}H_{20}NO_3S$ $[M+H]^+$: 354.1158, found 354.1160. [5]



20c: Substrate (129.9 mg, 0.763 mmol); eluent: petroleum ether/EtOAc = 40/1 to petroleum ether/EtOAc = 20/1, product obtained 94.5 mg (0.477 mmol), yield 62%; ^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.60-7.58 (m, 2H), 7.41-7.38 (m, 2H), 7.32-7.28 (m, 1H), 2.94-2.86 (m, 2H), 2.85 (dd, *J* = 17.6, 6.8 Hz, 1H), 2.70 (m, 1H), 2.29 (m, 2H), 2.14 (m, 2H), 1.19 (m, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$) δ = 208.6, 185.2, 134.5, 131.7, 128.2(2), 128.1(8), 127.6, 44.6, 42.9, 30.9, 27.2, 25.9 ppm; IR (neat): 1696 cm^{-1}; HRMS (ESI): *m*/*z* calcd for $C_{14}H_{14}NaO$ $[M+Na]^+$: 221.0937, found 221.0934. [5]

Spectrum of the synthesized compounds

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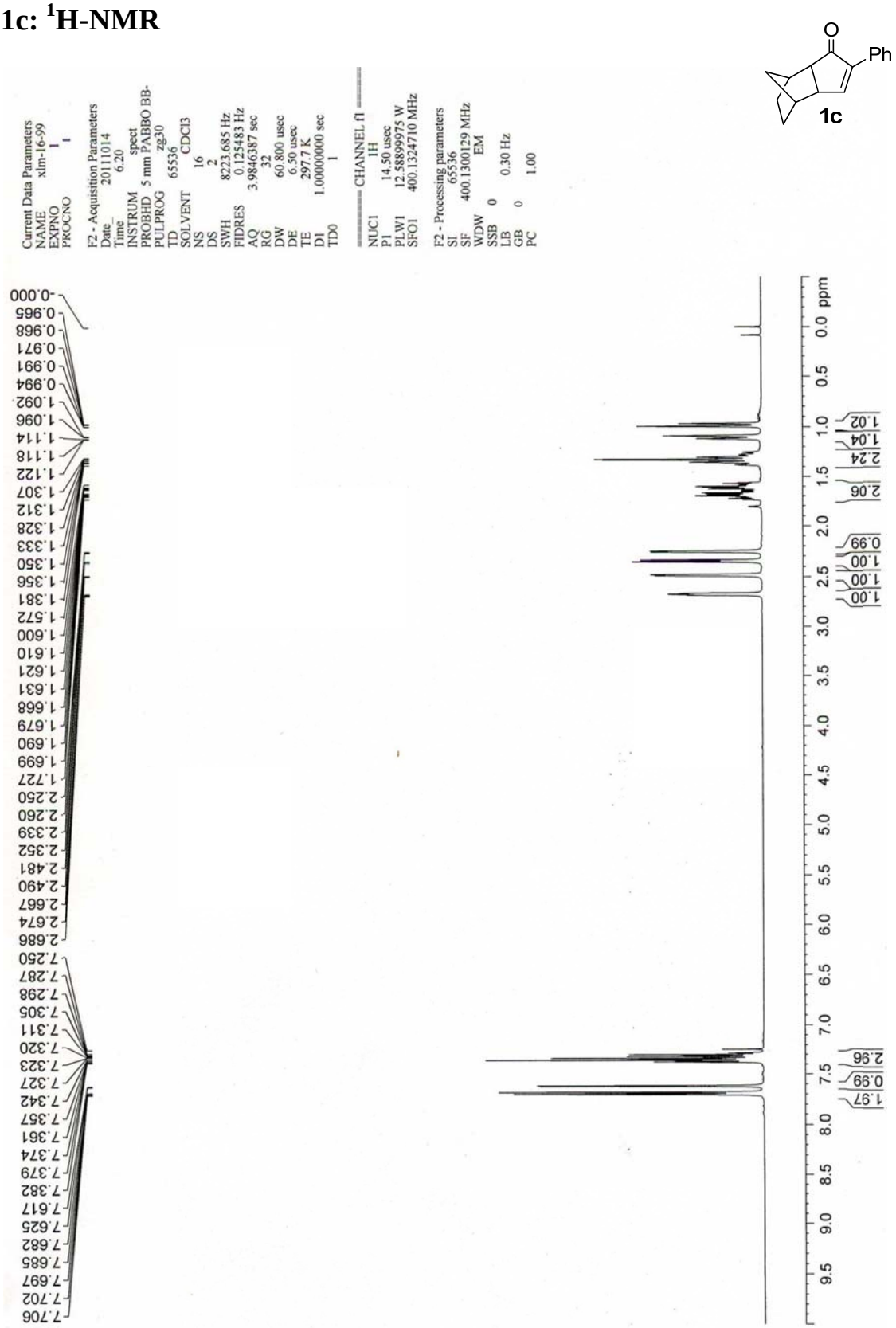

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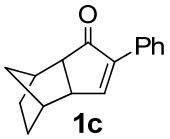
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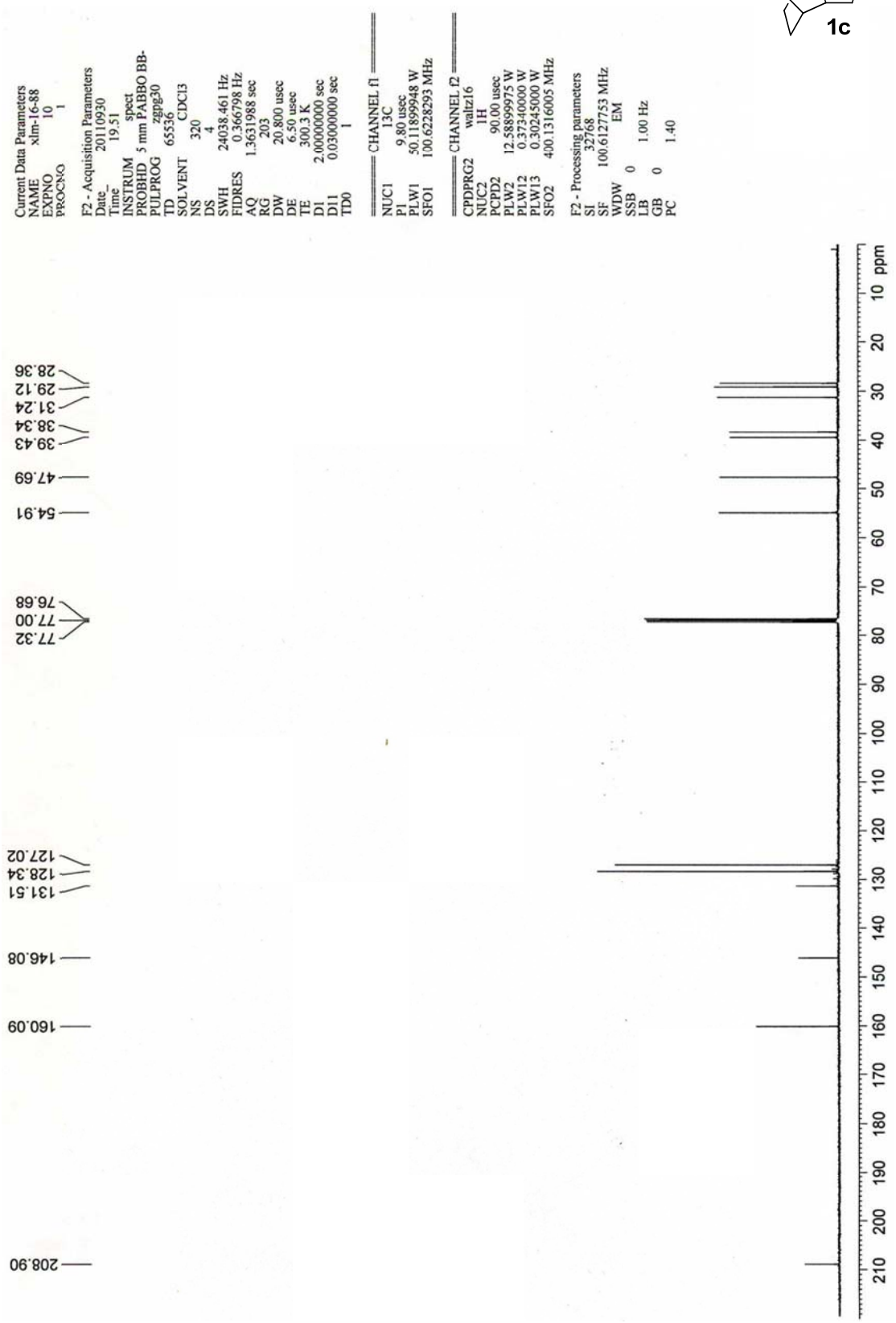
1c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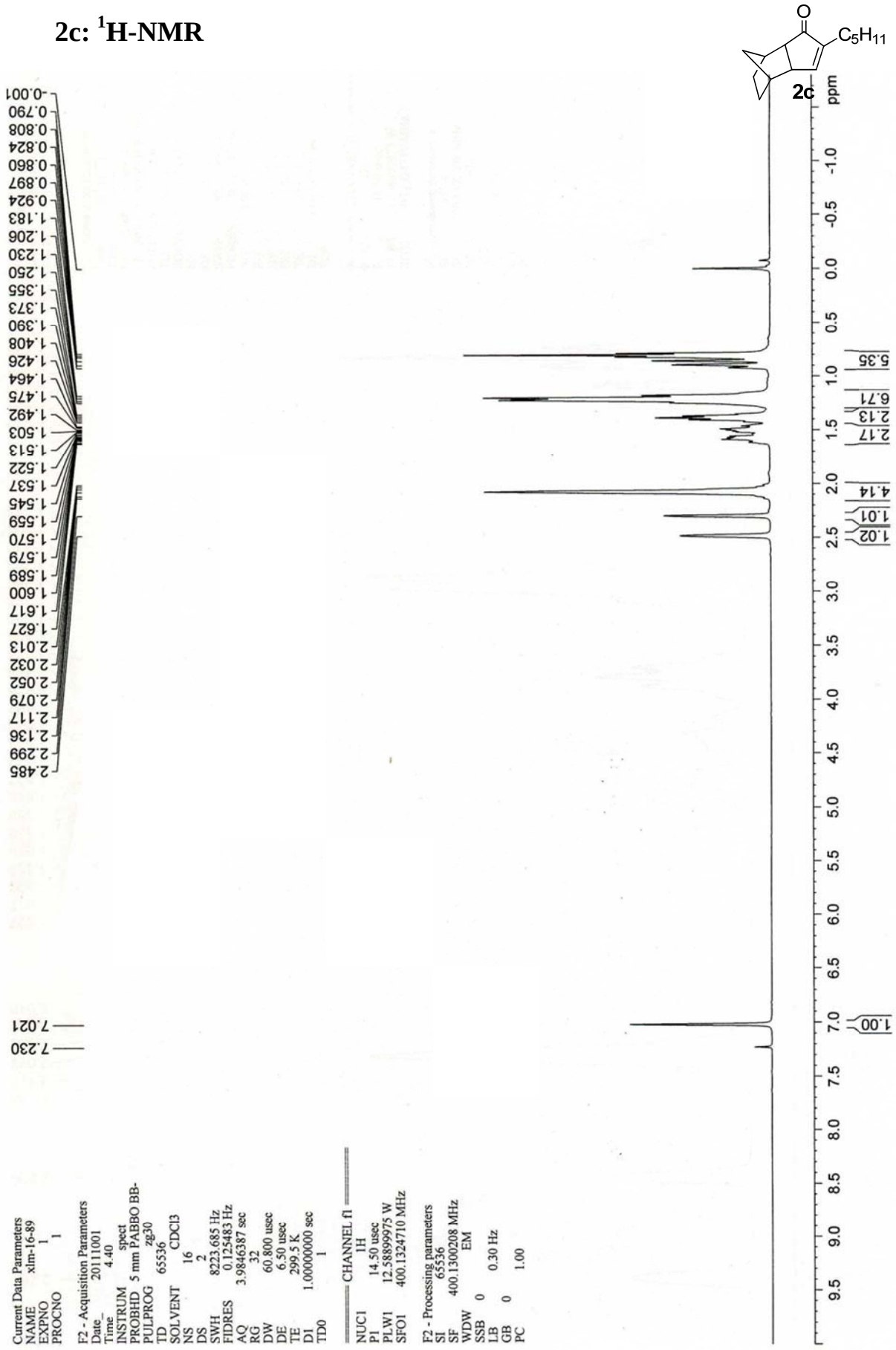
2c: ^{1}H-NMR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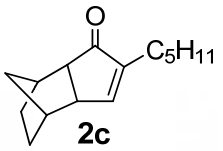
2c: ^{13}C-NMR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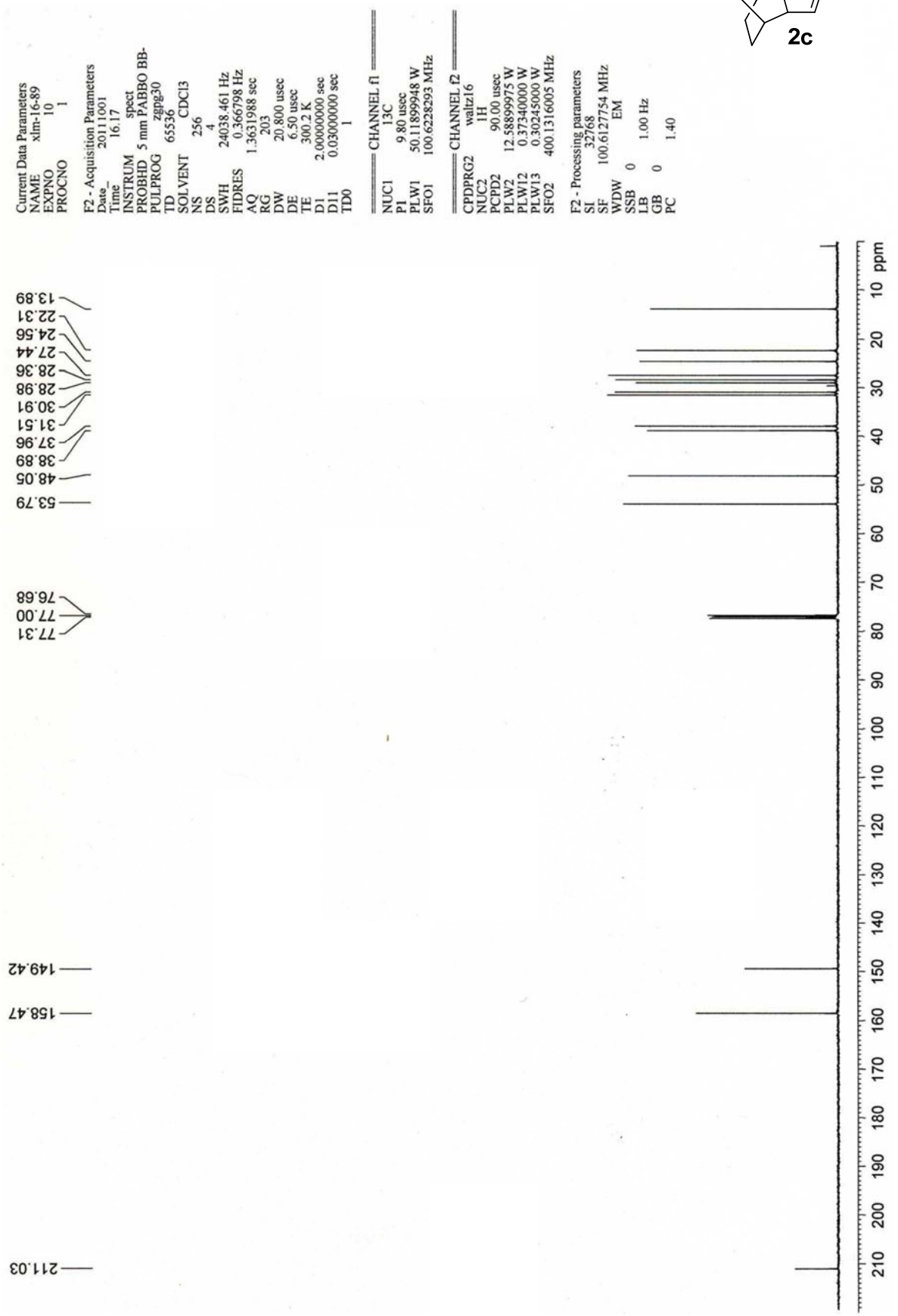
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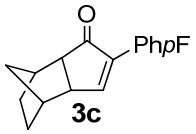
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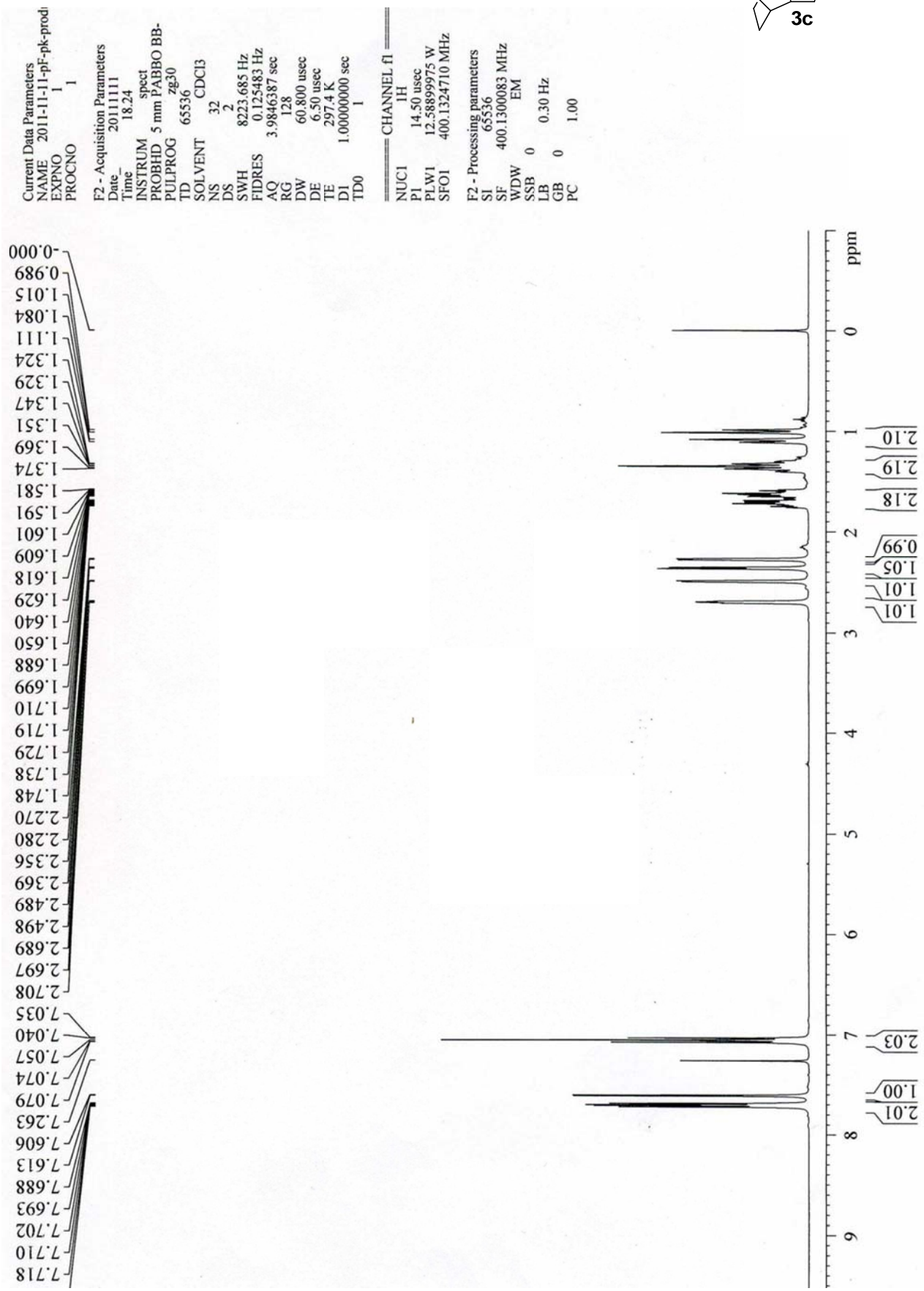
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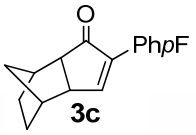
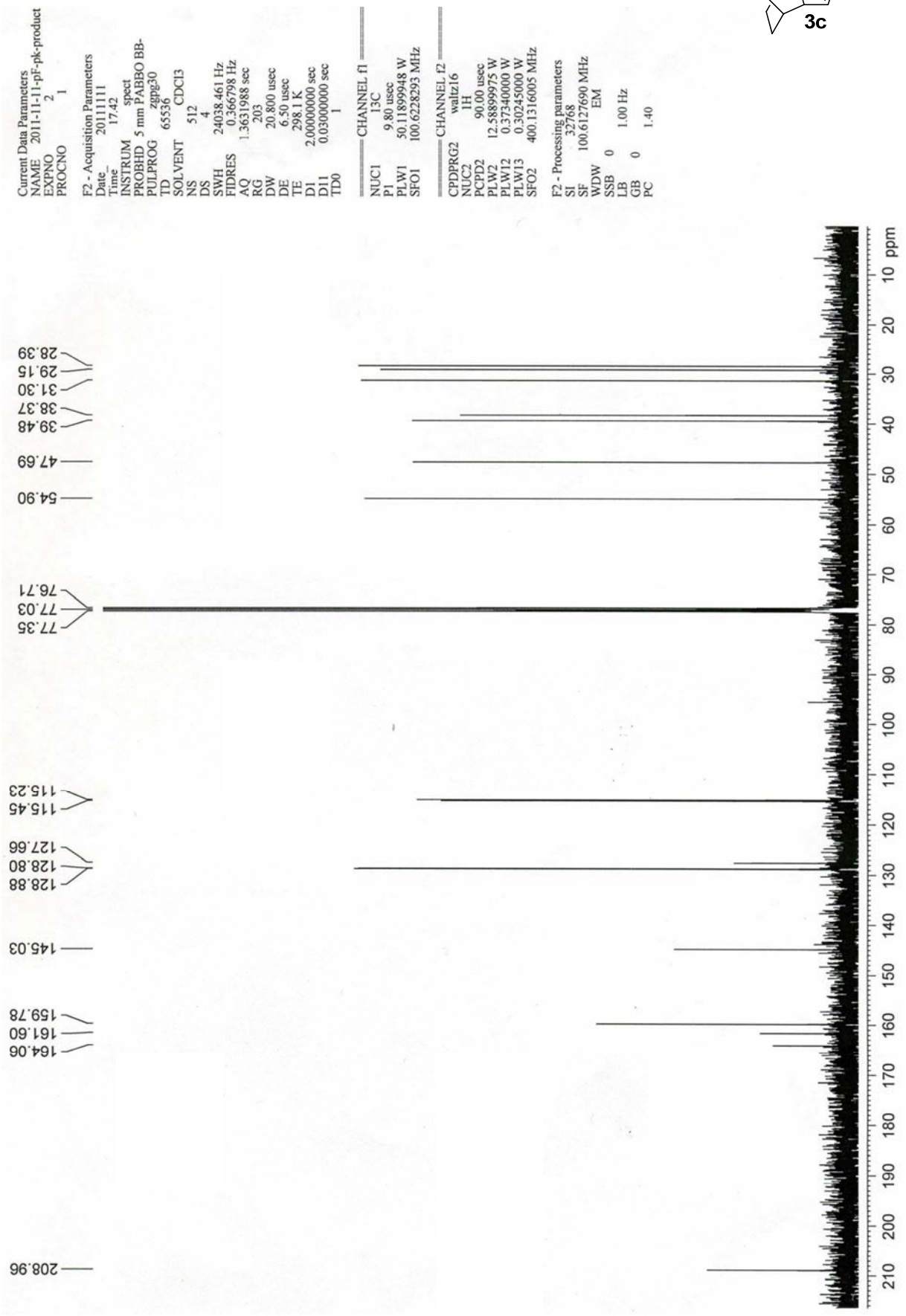
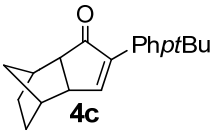
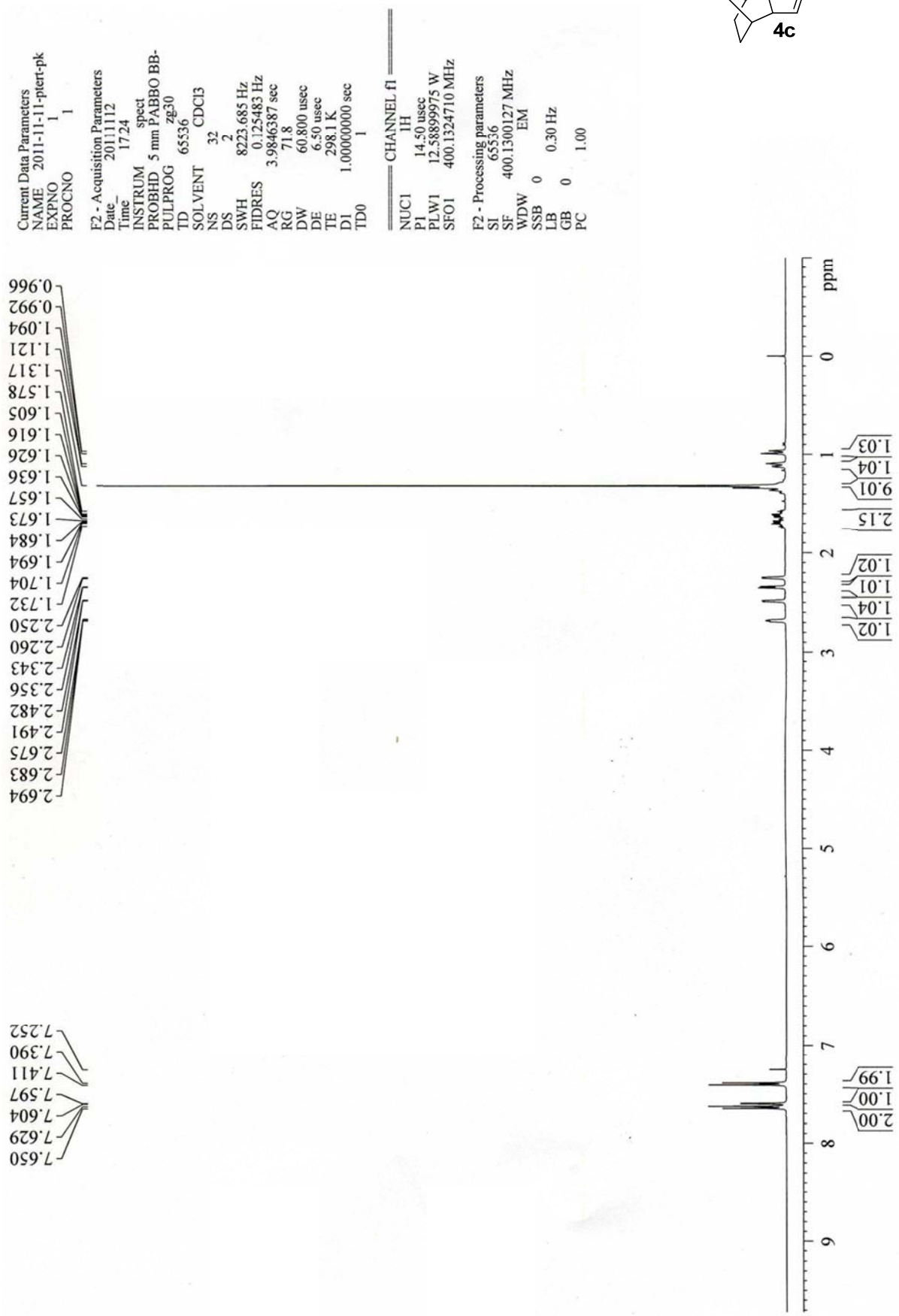

O
PhpF
3c

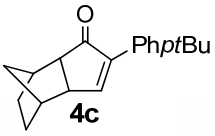

Current Data Parameters
NAME 2011-11-11-pF-pk-product
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111111
Time 17.42
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.39
29.15
31.30
38.37
39.48
47.69
54.90
76.71
77.03
77.35
115.23
115.45
127.66
128.80
128.88
145.03
159.78
161.60
164.06
208.96
ppm

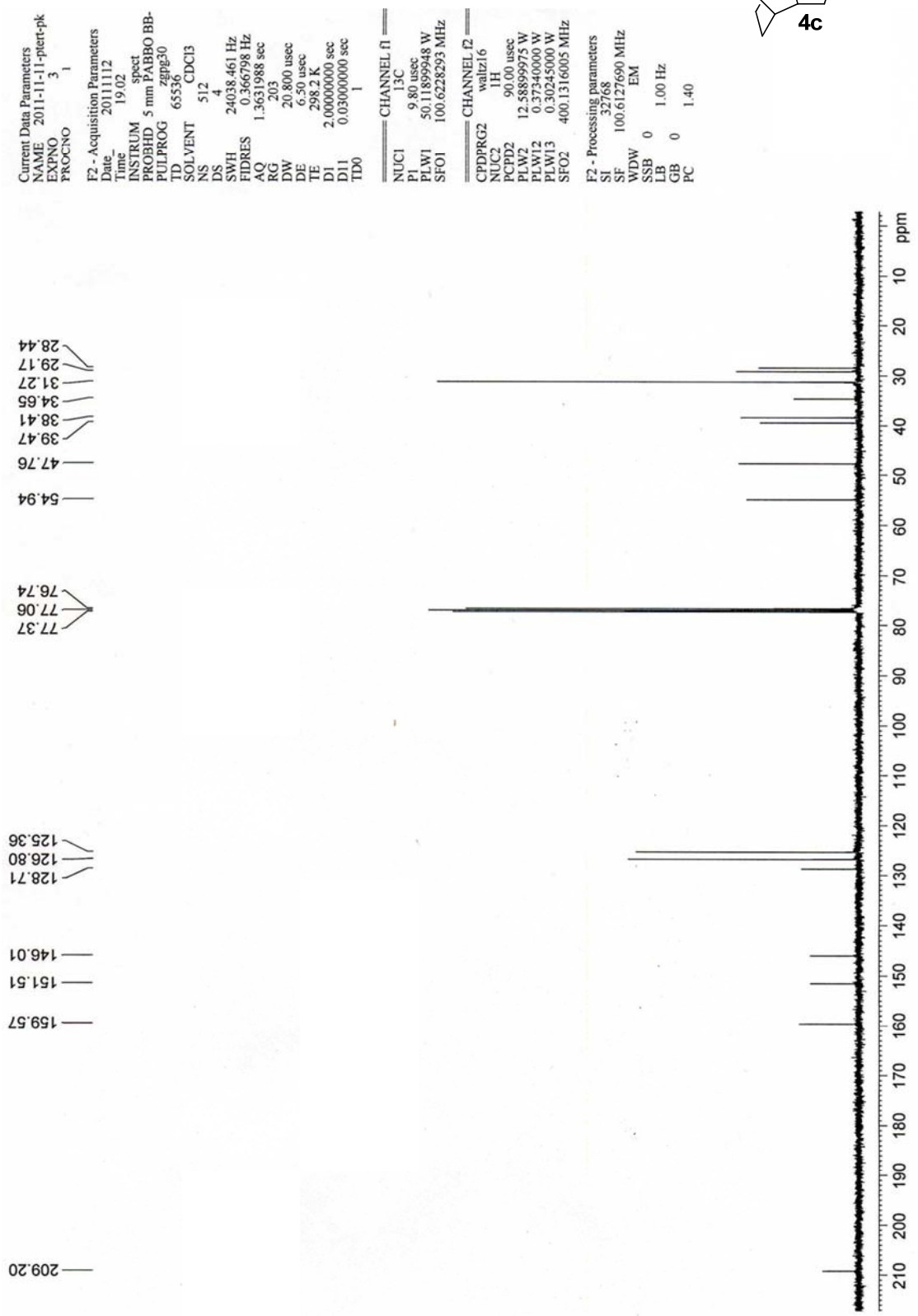
4c: ^{1}H-NMR


O
PhptBu
4c

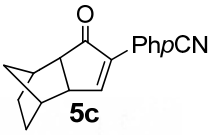

Current Data Parameters
NAME 2011-11-11-ptert-pk
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111112
Time 17:24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.8
DW 60.800 usec
DE 6.50 usec
TE 298.1 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.50 usec
PLW1 12.58899975 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300127 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.966
0.992
1.094
1.121
1.317
1.578
1.605
1.616
1.626
1.636
1.657
1.673
1.684
1.694
1.704
1.732
2.250
2.260
2.343
2.356
2.482
2.491
2.675
2.683
2.694
7.252
7.390
7.411
7.597
7.604
7.629
7.650
ppm
0
1
2
3
4
5
6
7
8
9
1.03
9.01
1.04
2.15
1.02
1.01
1.04
1.02
2.00
1.00
1.99

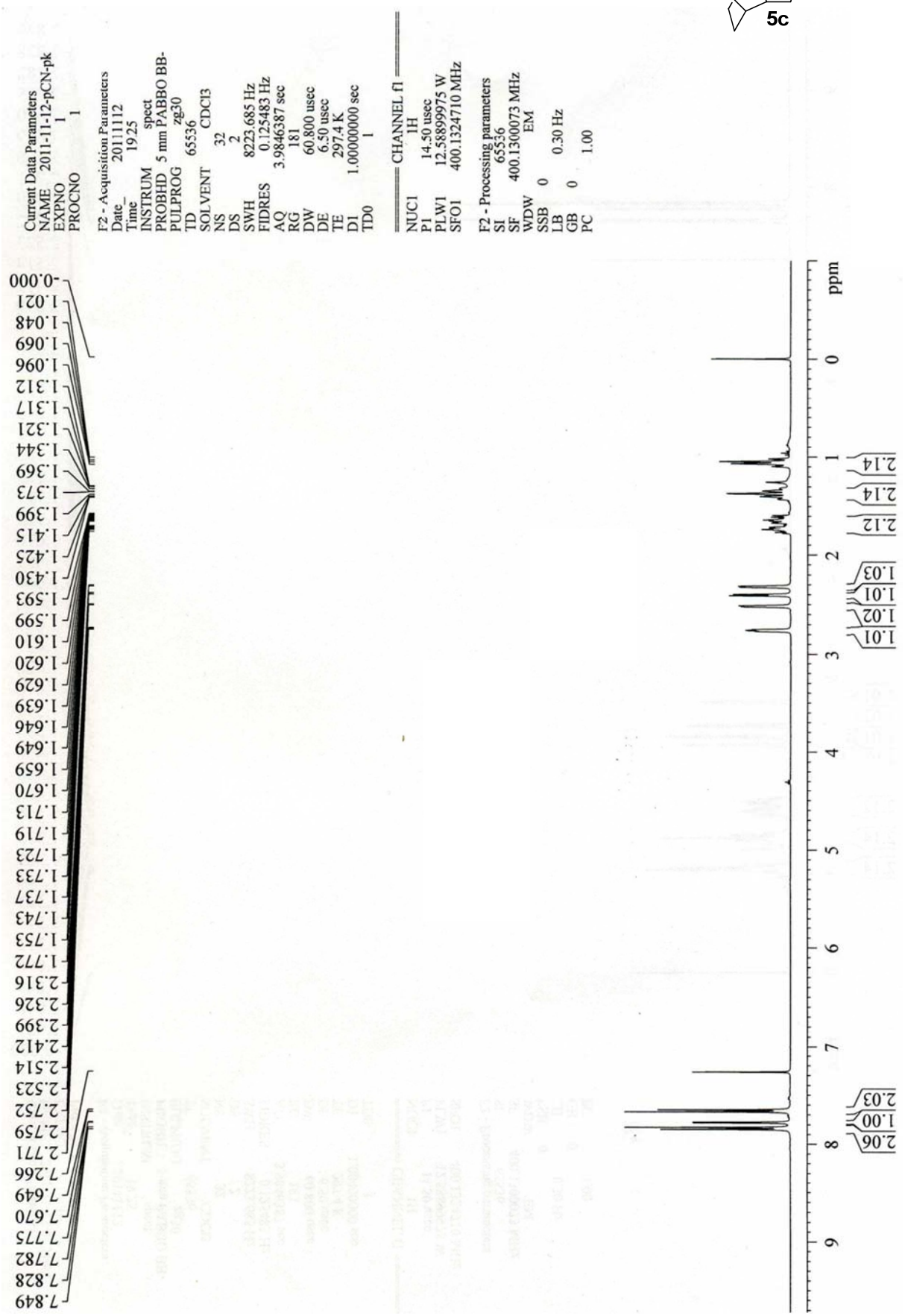
4c: ^{13}C-NMR


O
PhptBu
4c

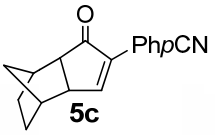

Current Data Parameters
NAME 2011-11-11-ptert-pk
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111112
Time 19.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.44
29.17
31.27
34.65
38.41
39.47
47.76
54.94
76.74
77.06
77.37
125.36
126.80
128.71
146.01
151.51
159.57
209.20
ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

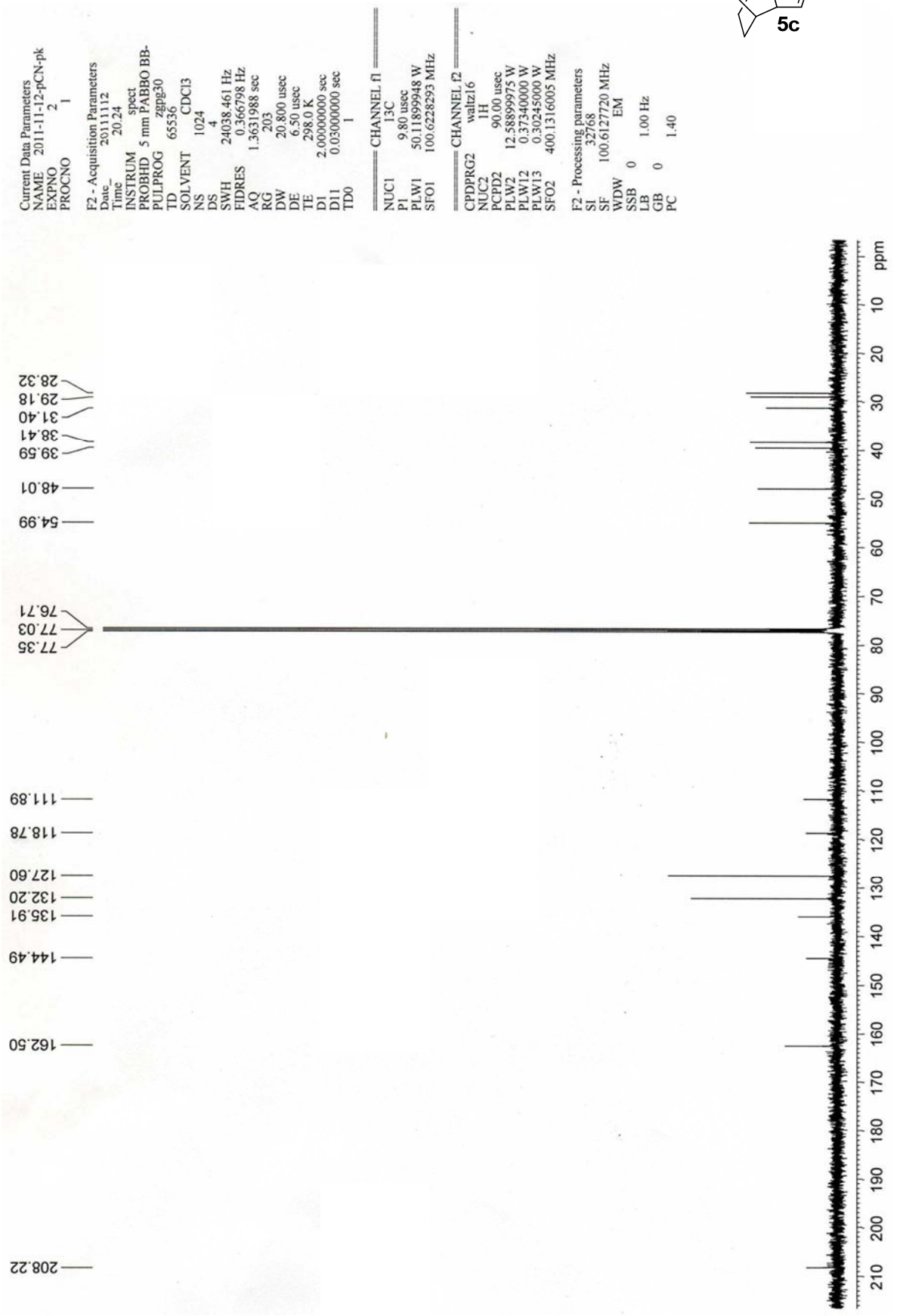
5c: ^{1}H-NMR


O
PhpCN
5c

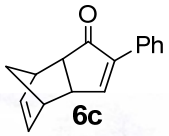

Current Data Parameters
NAME 2011-11-12-pCN-pk
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111112
Time 19.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 181
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.50 usec
PLW1 12.58899975 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300073 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
2.06
1.00
2.03
1.01
1.02
1.01
1.03
2.12
2.14
2.14

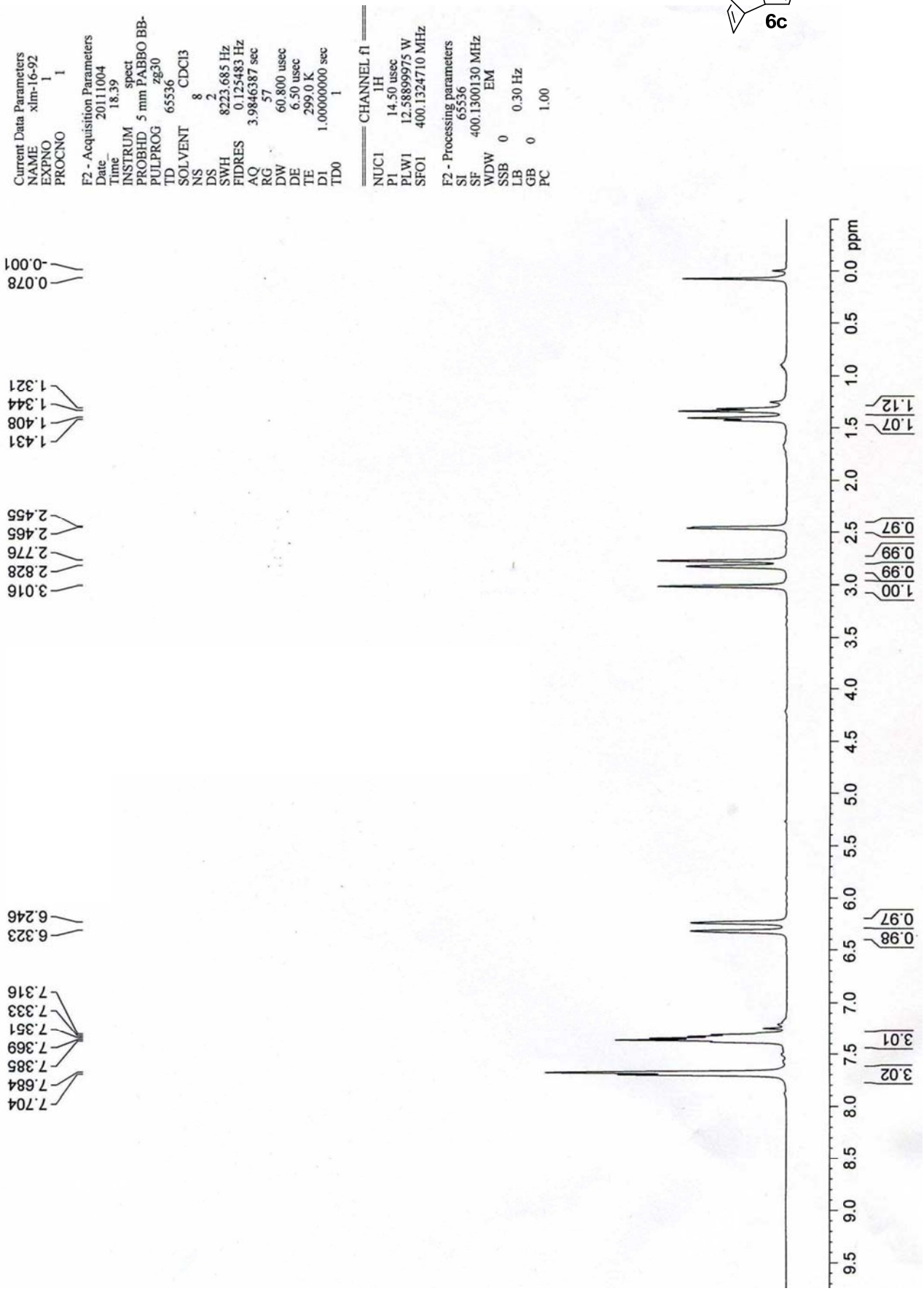
5c: ^{13}C-NMR


O
PhpCN
5c

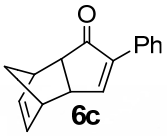

Current Data Parameters
NAME 2011-11-12-pCN-pk
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111112
Time_ 20.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127720 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
28.32
29.18
31.40
38.41
39.59
48.01
54.99
76.71
77.03
77.35
111.89
118.78
127.60
132.20
135.91
144.49
162.50
208.22
ppm

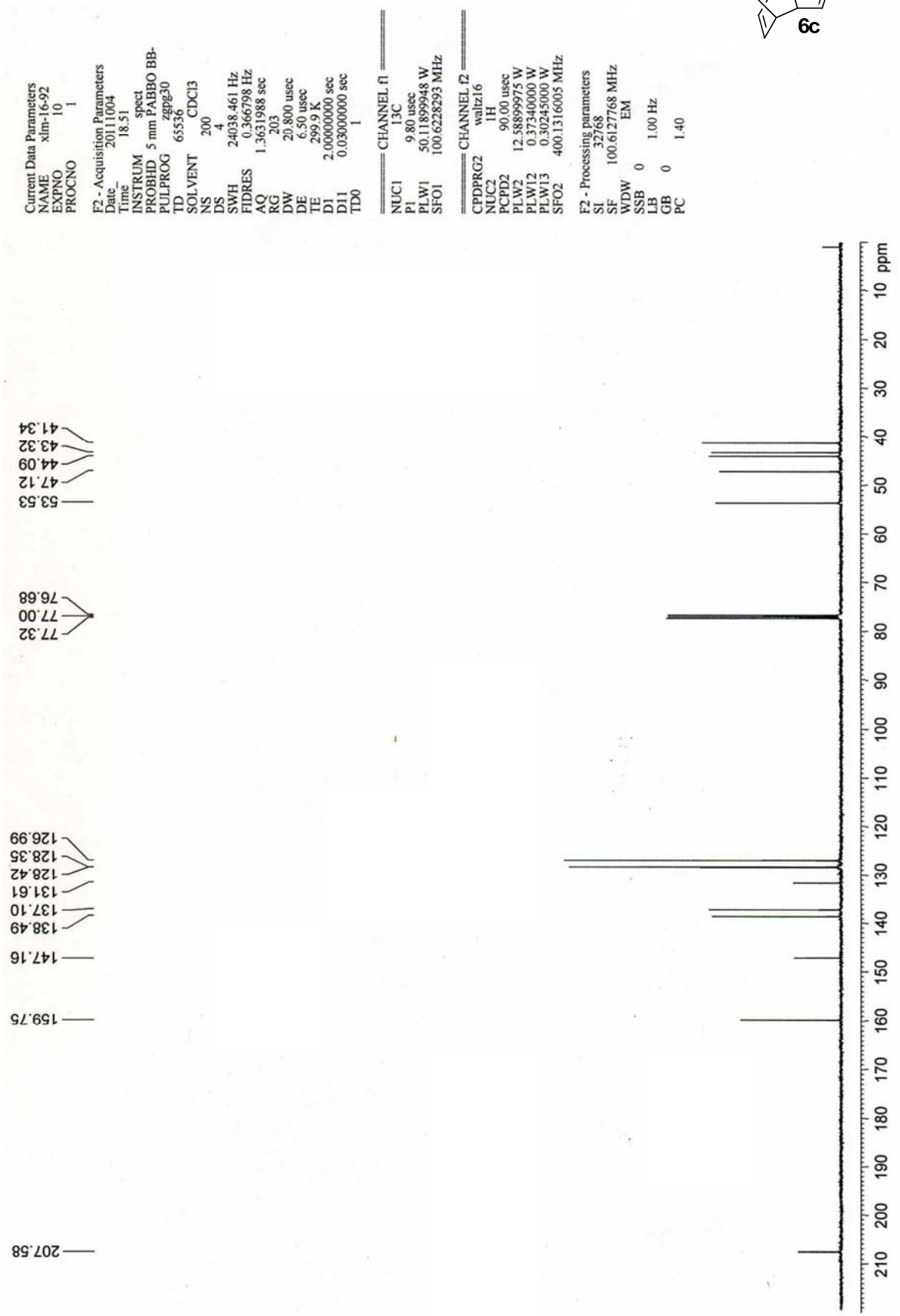
6c: ^{1}H-NMR


O
Ph
6c

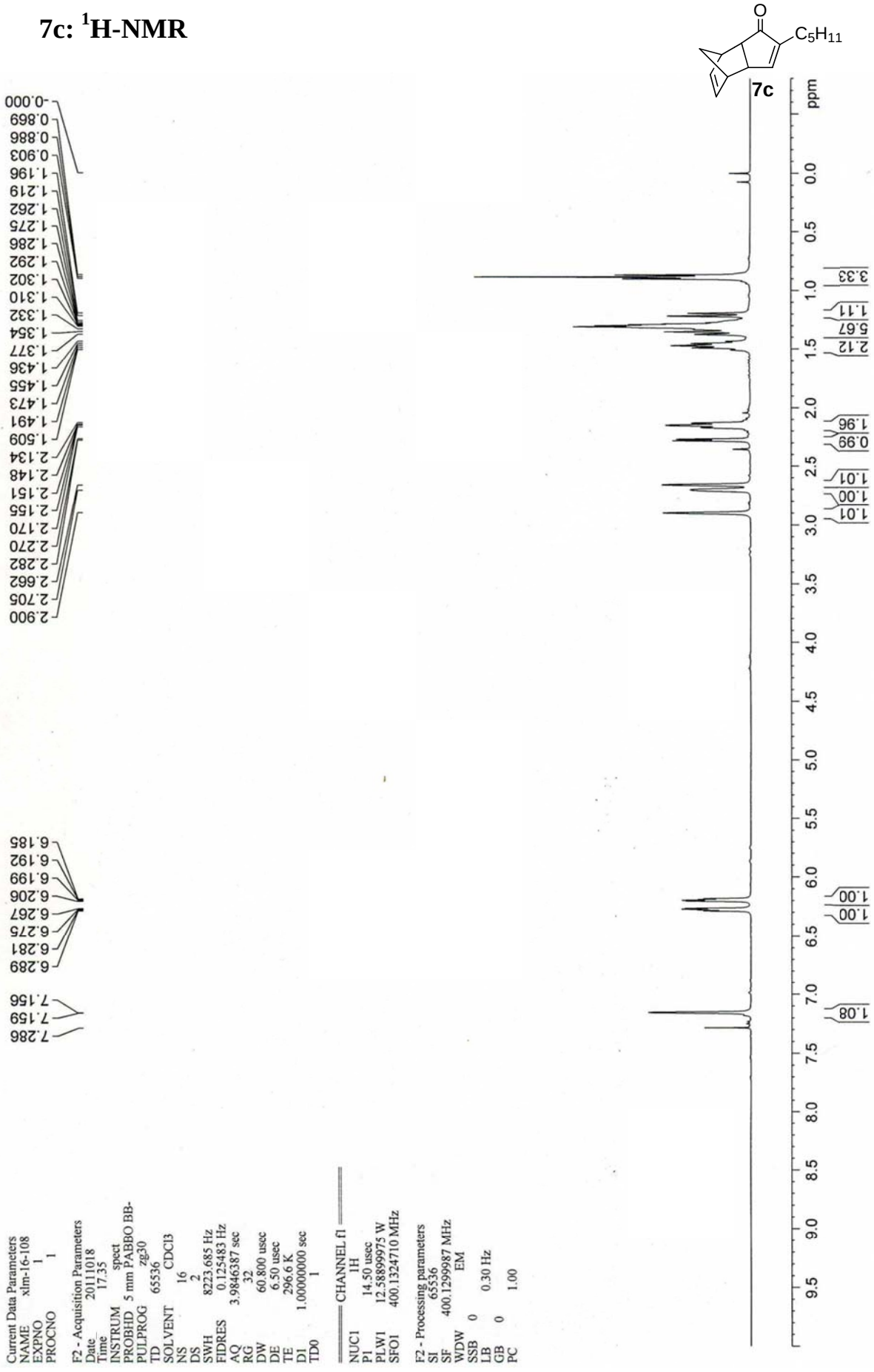

Current Data Parameters
NAME xlm-16-92
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 18.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 57
DW 60.800 usec
DE 6.50 usec
TE 299.0 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.50 usec
PLW1 12.58899975 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300130 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
0.078
-0.001
1.321
1.344
1.408
1.431
2.455
2.465
2.776
2.828
3.016
6.246
6.323
7.316
7.333
7.351
7.369
7.385
7.684
7.704
1.12
1.07
0.97
0.99
0.99
1.00
0.97
0.98
3.01
3.02
ppm

6c: ^{13}C-NMR

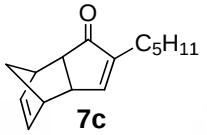

O
Ph
6c

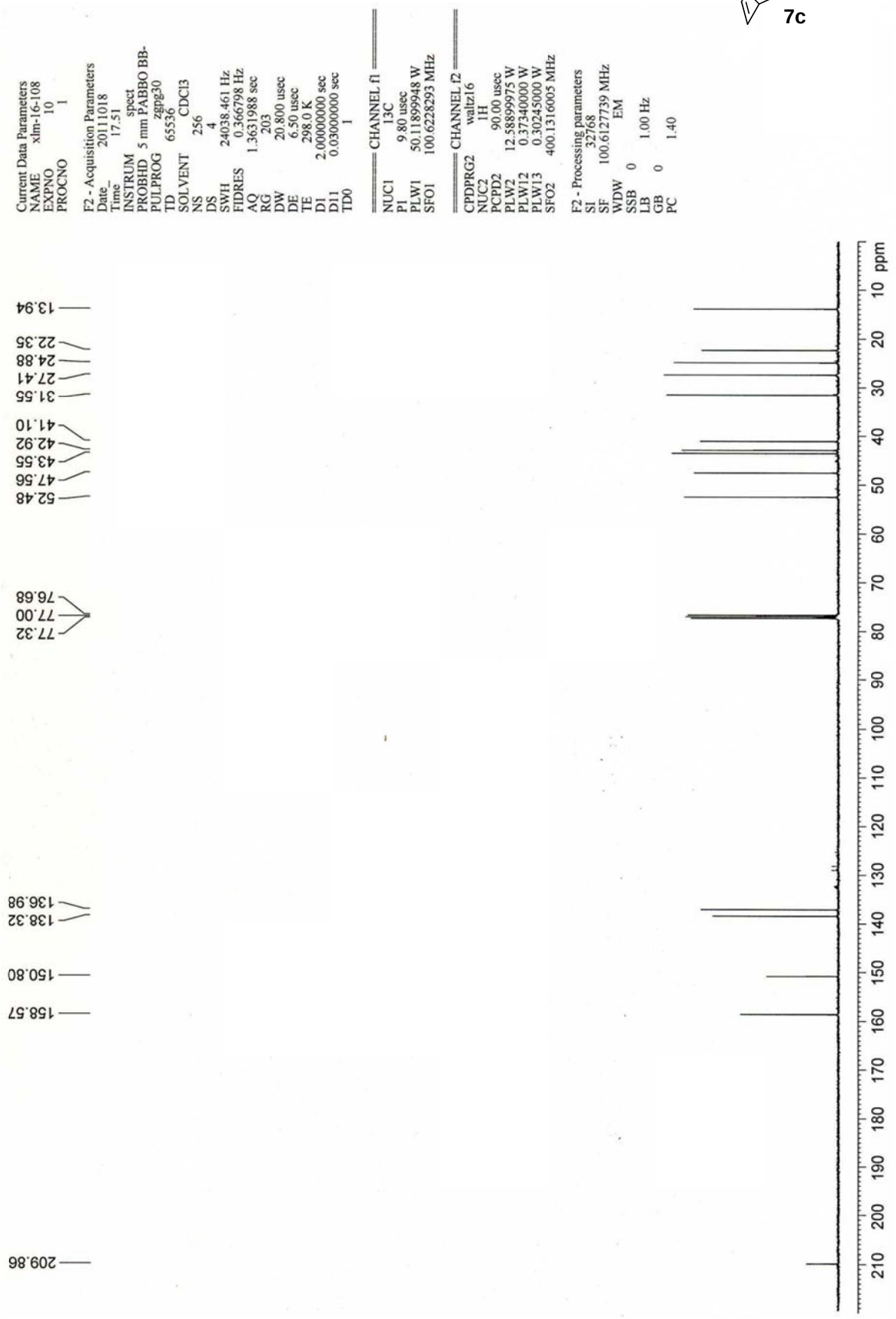

Current Data Parameters
NAME xlm-16-92
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 18.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 299.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127768 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
41.34
43.32
44.09
47.12
53.53
76.68
77.00
77.32
126.99
128.35
128.42
131.61
137.10
138.49
147.16
159.75
207.58
ppm

7c: ^{1}H-NMR

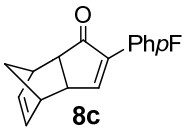

O
C5H11
7c
ppm
0.0
0.5
1.0
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
6.0
6.5
7.0
7.5
8.0
8.5
9.0
9.5
3.33
1.11
5.67
2.12
1.96
0.99
1.01
1.00
1.01
1.00
1.00
1.08
-0.000
0.869
0.886
0.903
1.196
1.219
1.262
1.275
1.286
1.292
1.302
1.310
1.332
1.354
1.377
1.436
1.455
1.473
1.491
1.509
2.134
2.148
2.151
2.155
2.170
2.270
2.282
2.662
2.705
2.900
6.185
6.192
6.199
6.206
6.267
6.275
6.281
6.289
7.156
7.159
7.286
Current Data Parameters
NAME xlm-16-108
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date 20111018
Time 17.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 32
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.50 usec
PLW1 12.58899975 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1299987 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

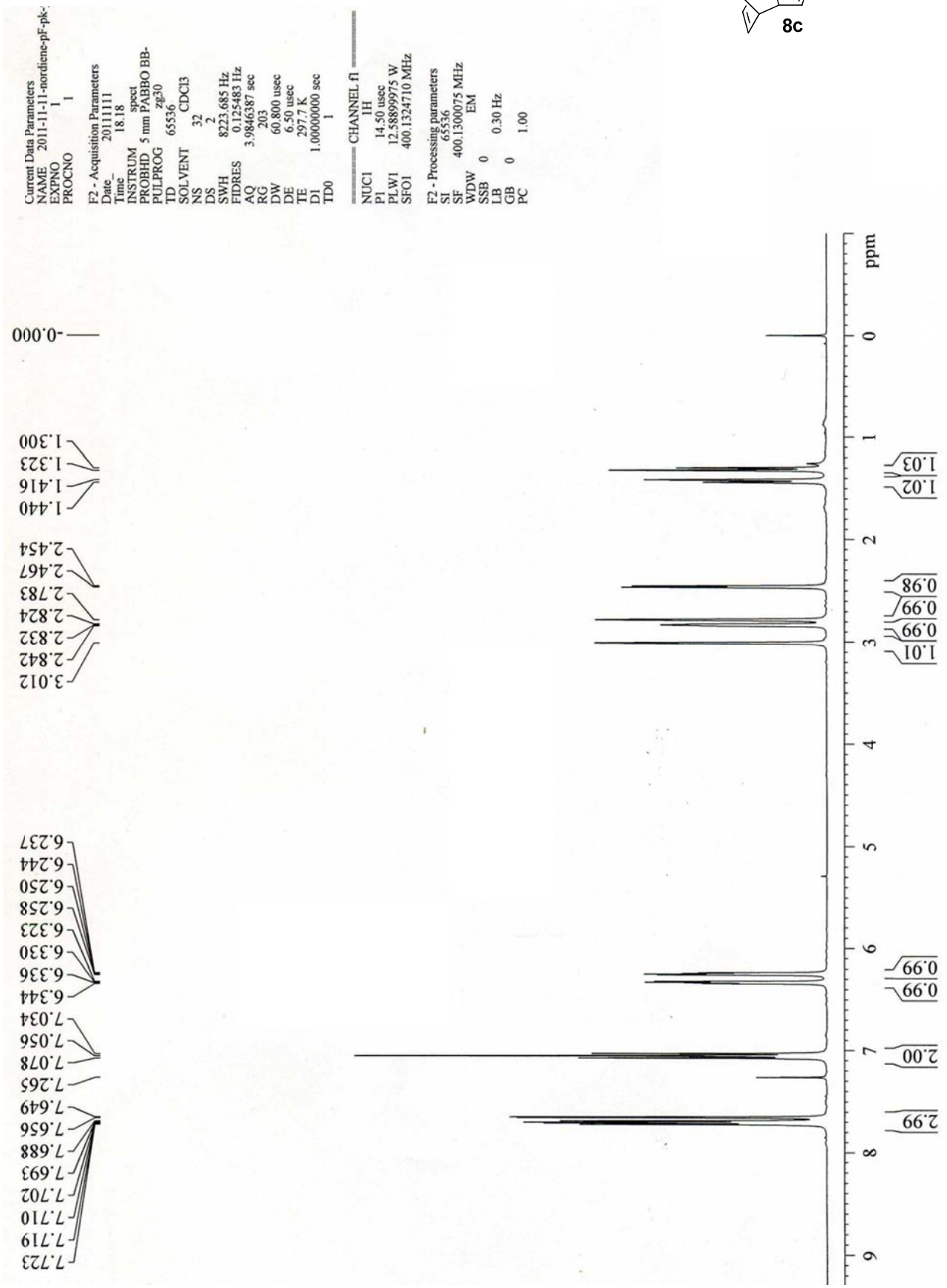
7c: ^{13}C-NMR


O
C5H11
7c

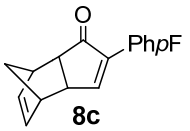

Current Data Parameters
NAME xlm-16-108
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111018
Time 17.51
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127739 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
13.94
22.35
24.88
27.41
31.55
41.10
42.92
43.55
47.56
52.48
76.68
77.00
77.32
136.98
138.32
150.80
158.57
209.86
ppm

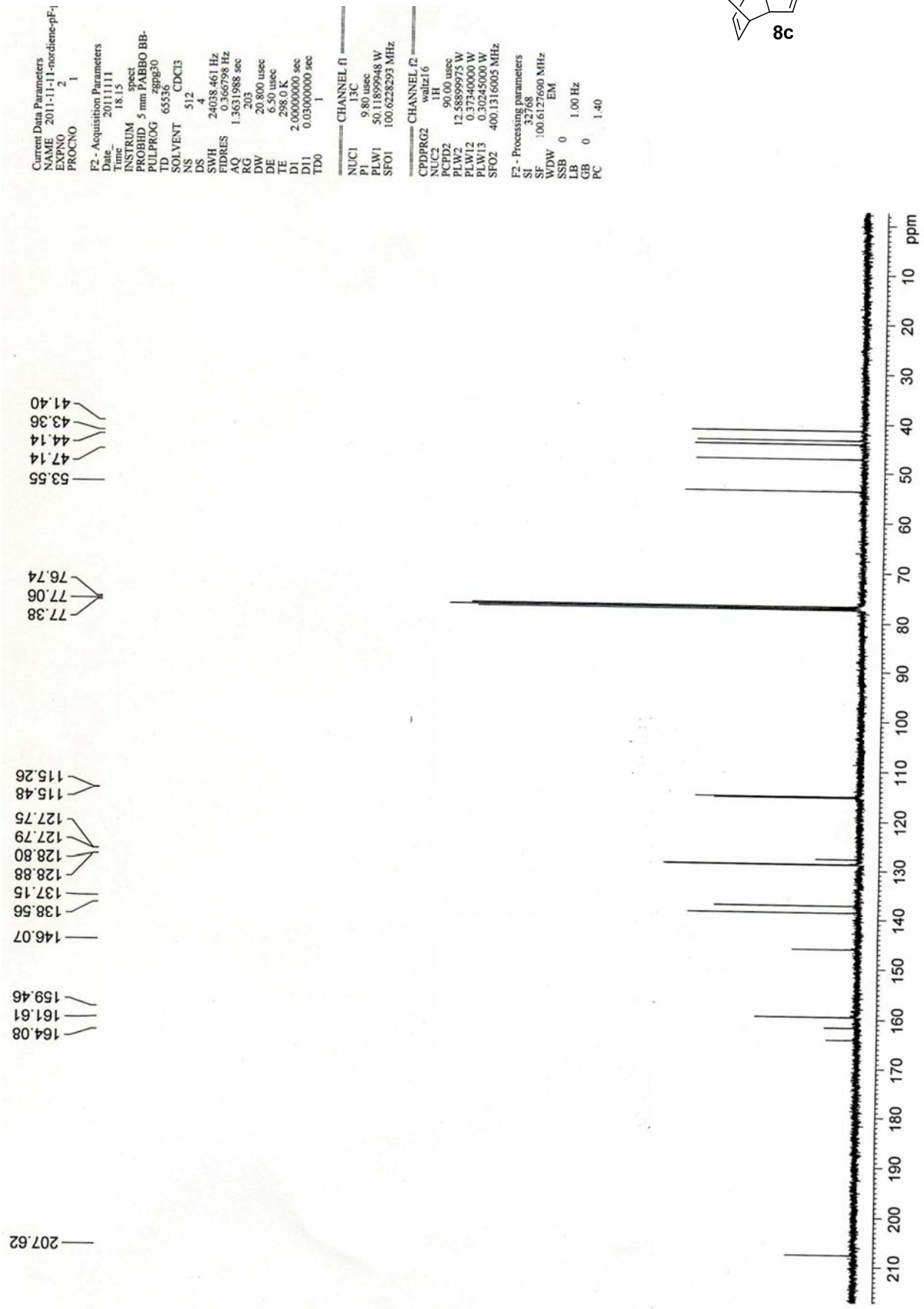
8c: ^{1}H-NMR


O
PhpF
8c

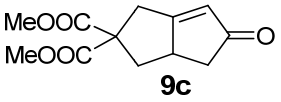

Current Data Parameters
NAME 2011-11-11-nordiene-pF-pk-
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111111
Time 18.18
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.98466387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 297.7 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.50 usec
PLW1 12.58899975 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300075 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
ppm
1.03
1.02
0.98
0.99
0.99
1.01
0.99
0.99
2.00
2.99

8c: ^{13}C-NMR

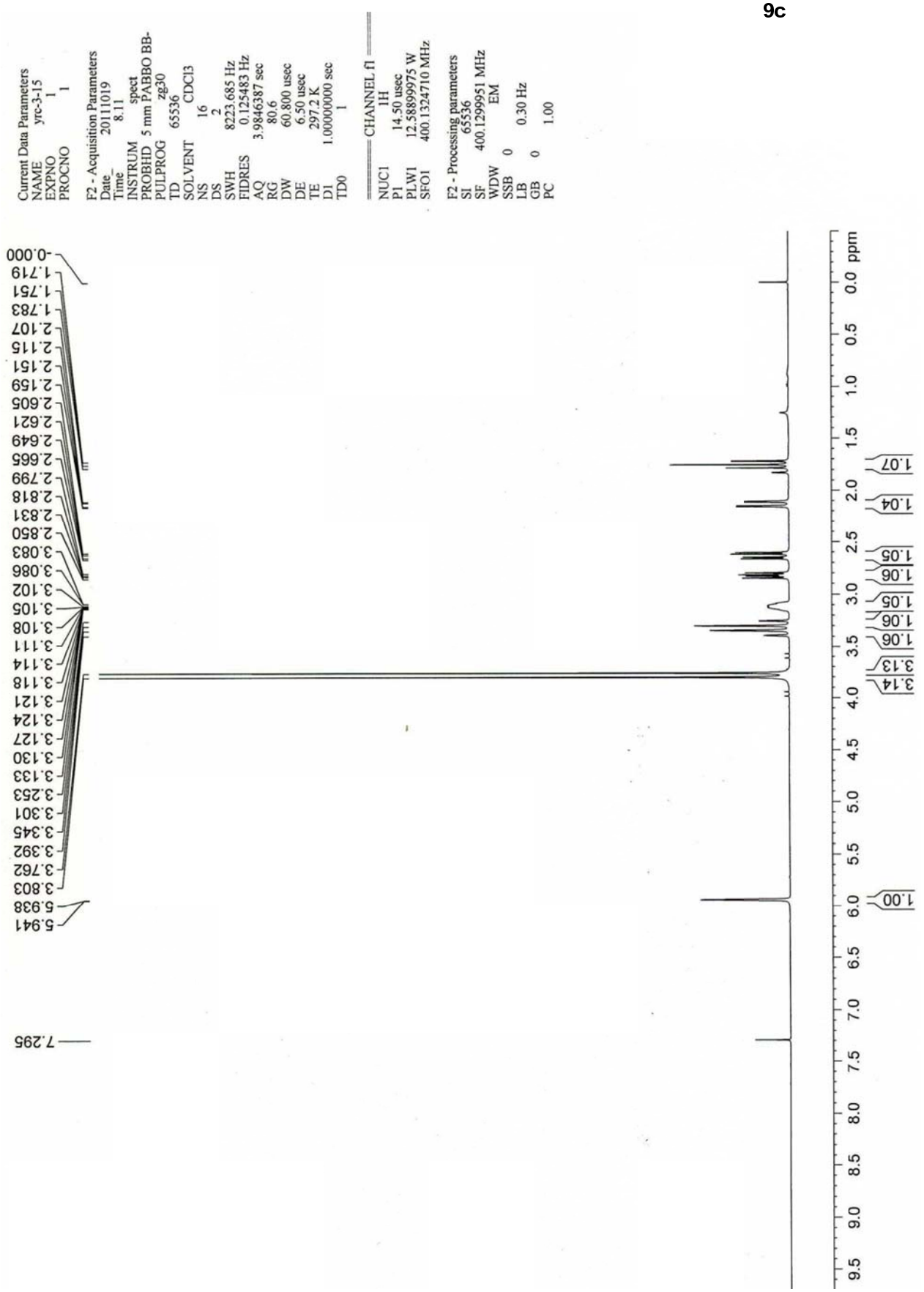

O
PhpF
8c


Current Data Parameters
NAME 2011-11-11-nordiene-pF-
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111111
Time 18.15
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
41.40
43.36
44.14
47.14
53.55
76.74
77.06
77.38
115.26
115.48
127.75
127.79
128.80
128.88
137.15
138.56
146.07
159.46
161.61
164.08
207.62
ppm

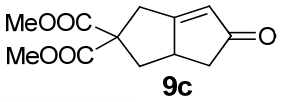
9c: ^{1}H-NMR


MeOOC
MeOOC
O

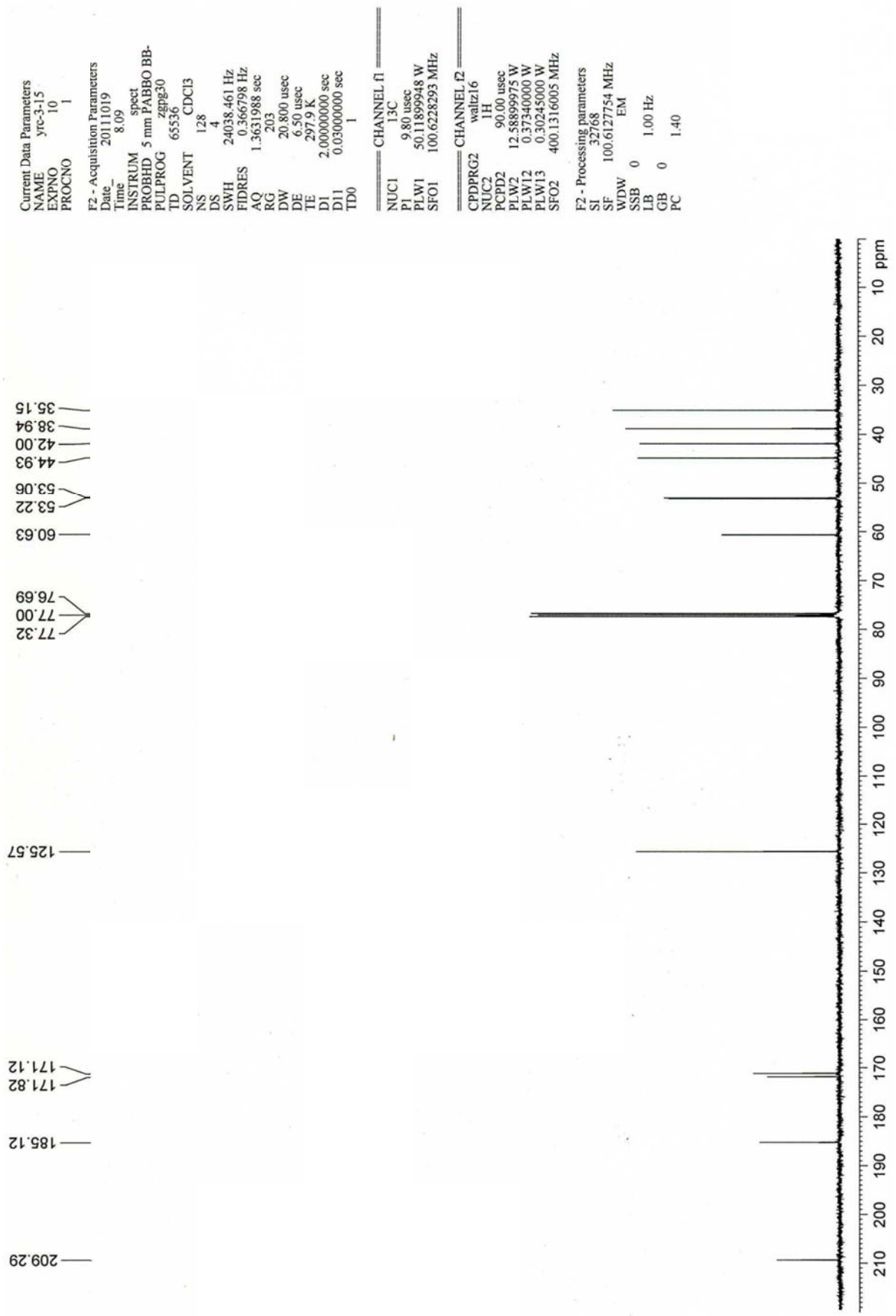
9c


Current Data Parameters
NAME yrc-3-15
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111019
Time_ 8.11
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 80.6
DW 60.800 usec
DE 6.50 usec
TE 297.2 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.50 usec
PLW1 12.58899975 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1299951 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.719
1.751
1.783
2.107
2.115
2.151
2.159
2.605
2.621
2.649
2.665
2.799
2.818
2.831
2.850
3.083
3.086
3.102
3.105
3.108
3.111
3.114
3.118
3.121
3.124
3.127
3.130
3.133
3.253
3.301
3.345
3.392
3.762
3.803
5.938
5.941
7.295
1.07
1.04
1.05
1.06
1.05
1.06
1.06
3.13
3.14
1.00
ppm

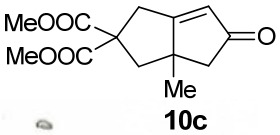
9c: ^{13}C-NMR


MeOOC
MeOOC
O

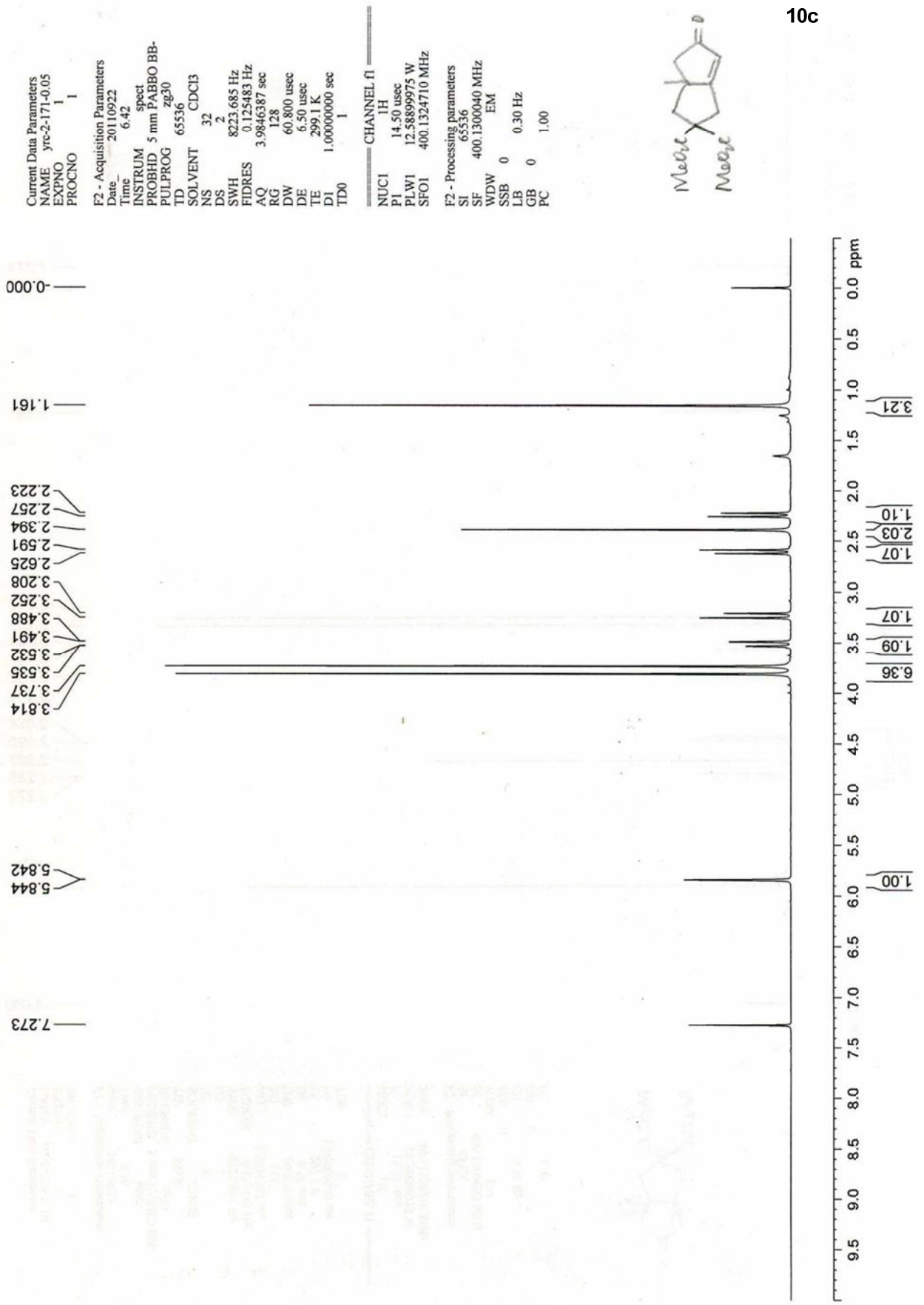
9c


Current Data Parameters
NAME yrc-3-15
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111019
Time 8.09
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127754 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
35.15
38.94
42.00
44.93
53.06
53.22
60.63
76.69
77.00
77.32
125.57
171.12
171.82
185.12
209.29
ppm

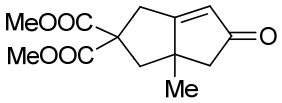
10c: ^{1}H-NMR



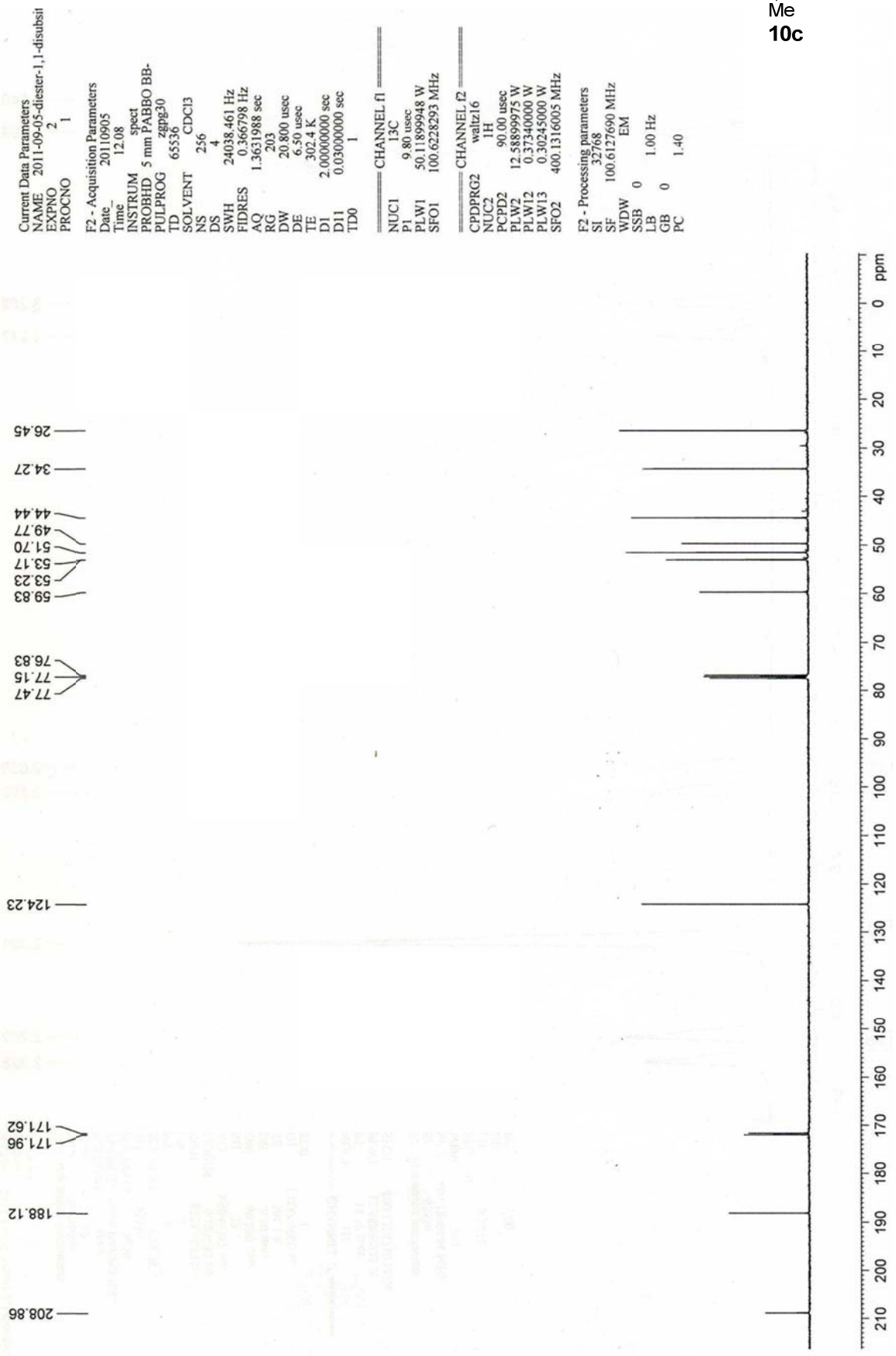
10c



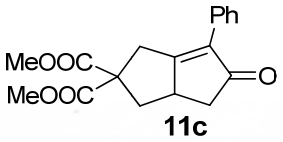
10c: ^{13}C-NMR



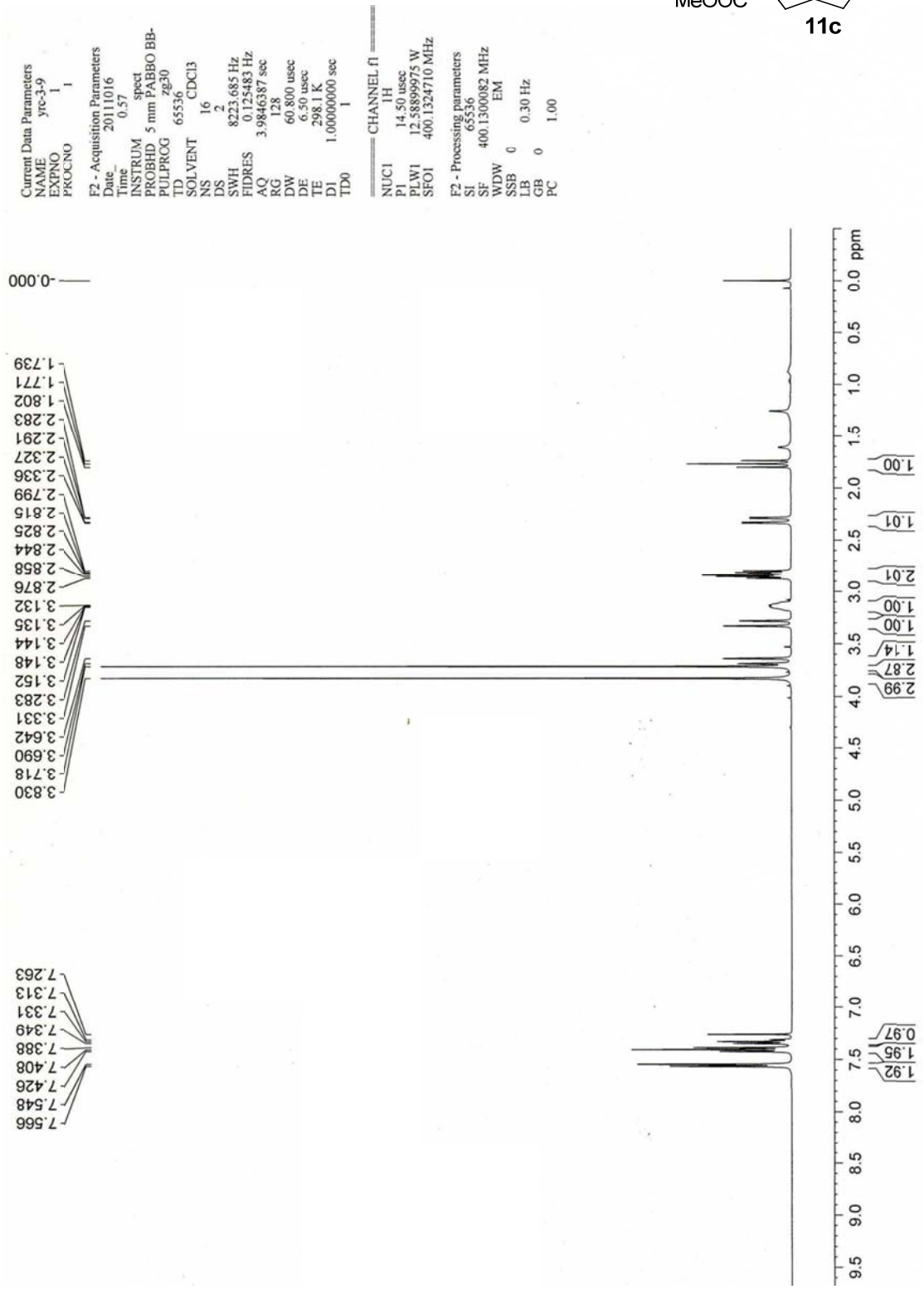
10c



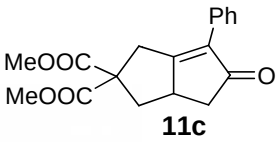
11c: ^{1}H-NMR



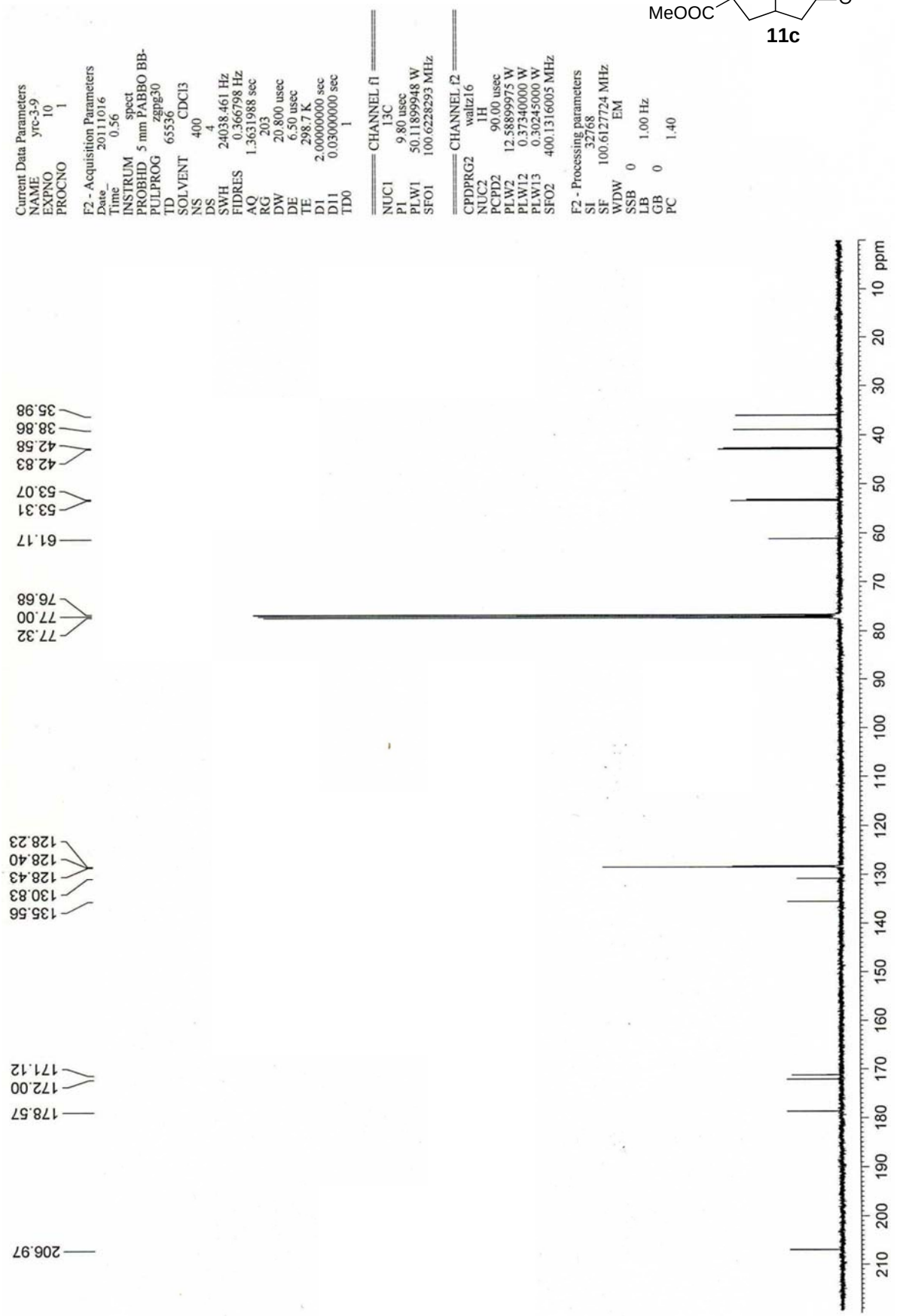
11c



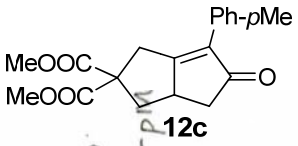
11c: ^{13}C-NMR

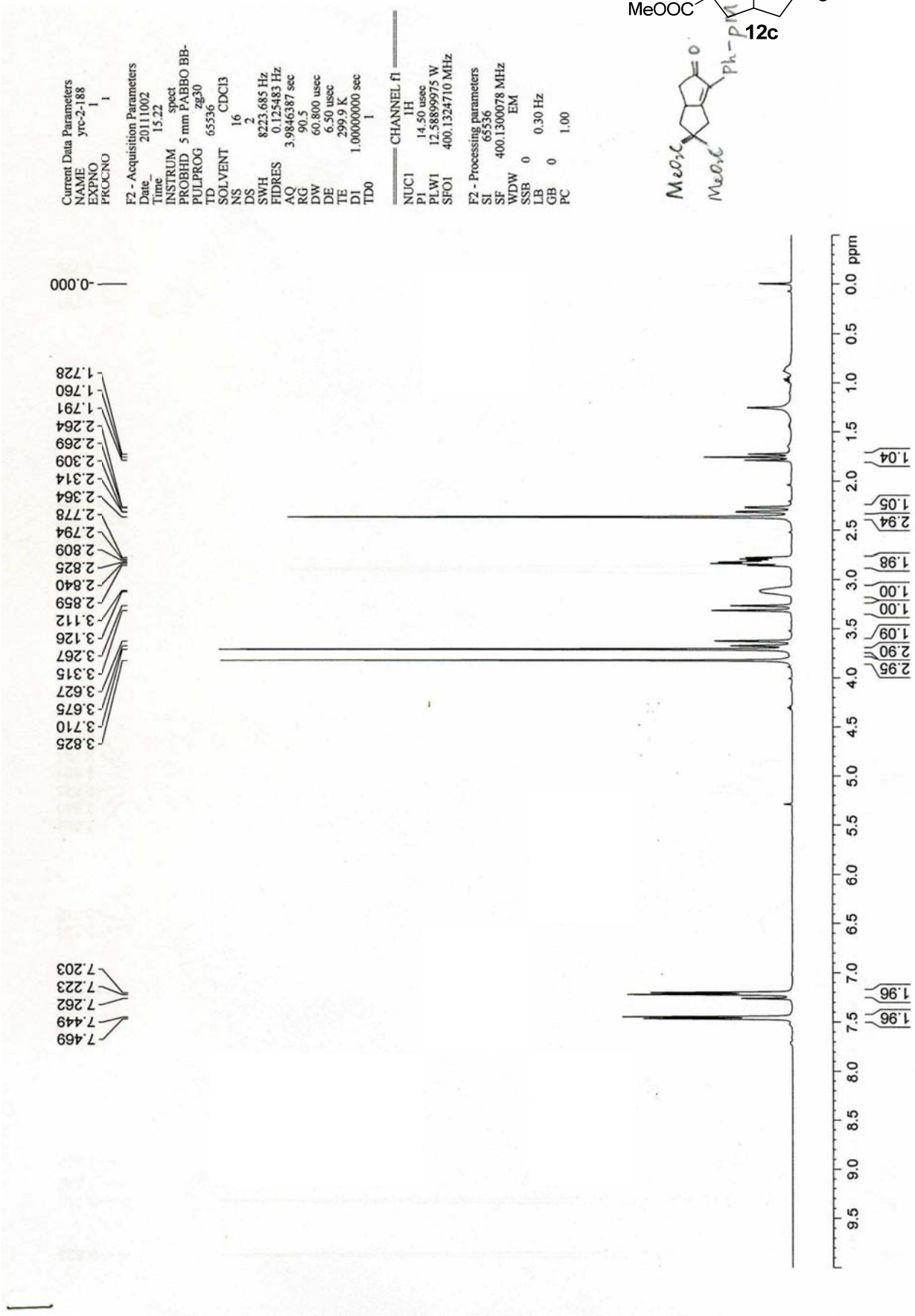


11c

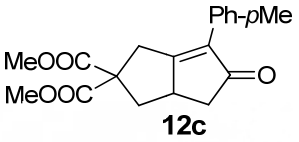


12c: ^{1}H-NMR

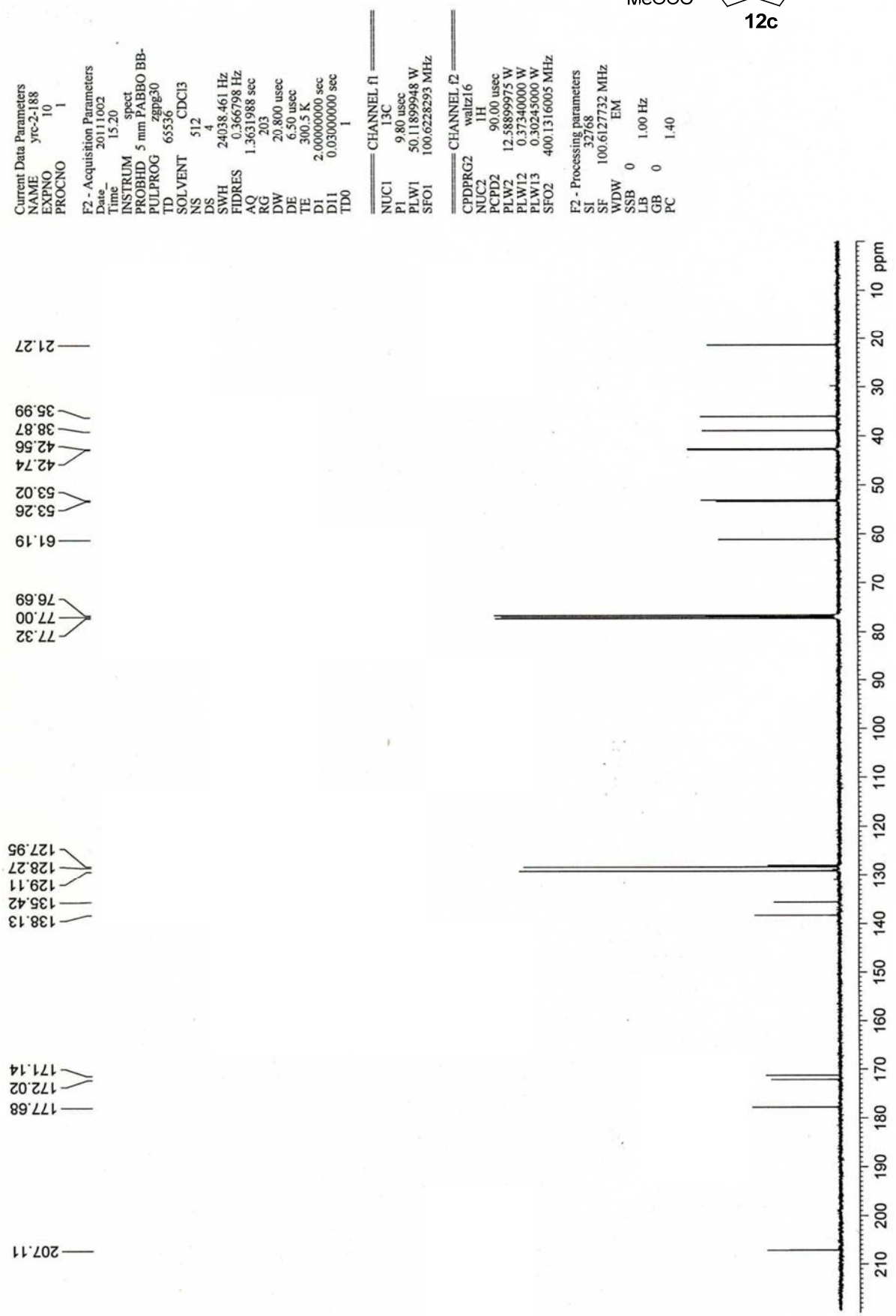

Ph-pMe
MeOOC
MeOOC
O
12c


Current Data Parameters
NAME yrc-2-188
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111002
Time 15.22
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 90.5
DW 60.800 usec
DE 6.50 usec
TE 299.9 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 14.50 usec
PLW1 12.58899975 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300078 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
-0.000
1.728
1.760
1.791
2.264
2.269
2.309
2.314
2.364
2.778
2.794
2.809
2.825
2.840
2.859
3.112
3.126
3.267
3.315
3.627
3.675
3.710
3.825
7.203
7.223
7.262
7.449
7.469
1.04
1.05
2.94
1.98
1.00
1.00
1.09
2.90
2.95
1.96
1.96
ppm

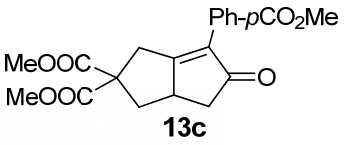
12c: ^{13}C-NMR



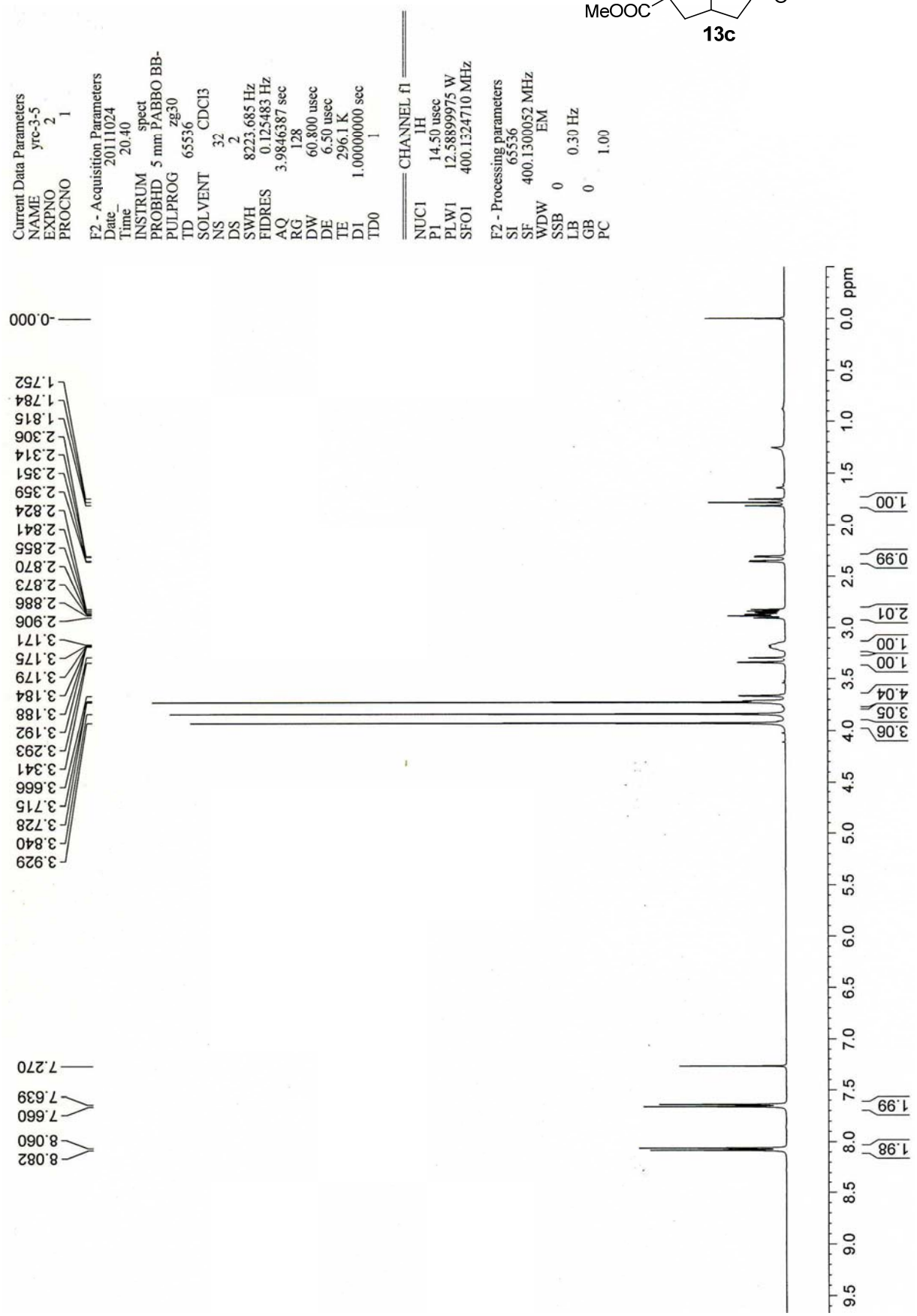
12c



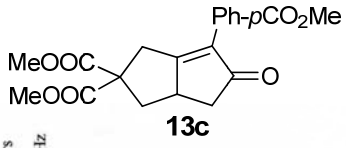
13c: ^{1}H-NMR



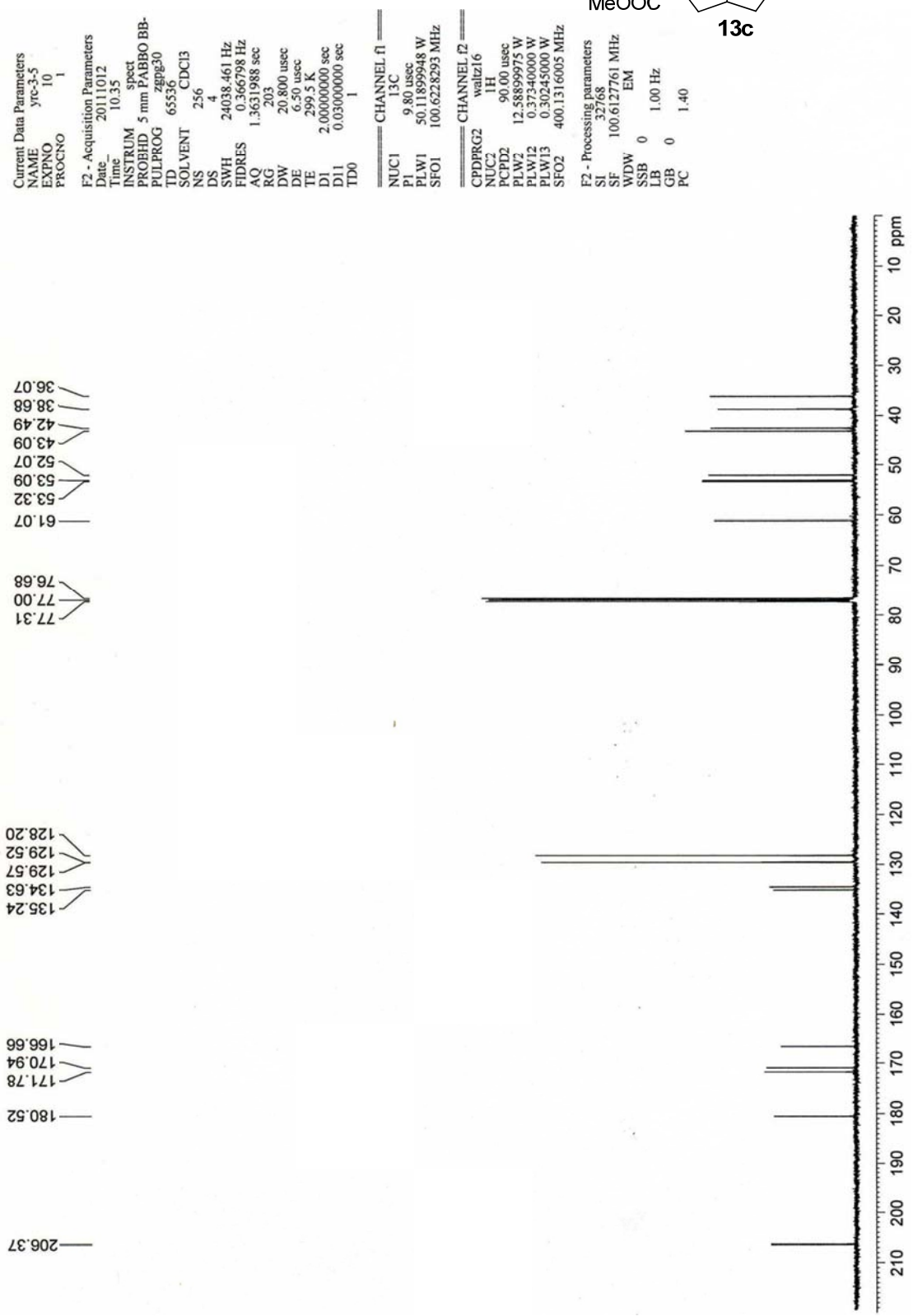
13c



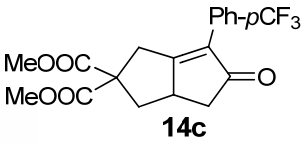
13c: ^{13}C-NMR



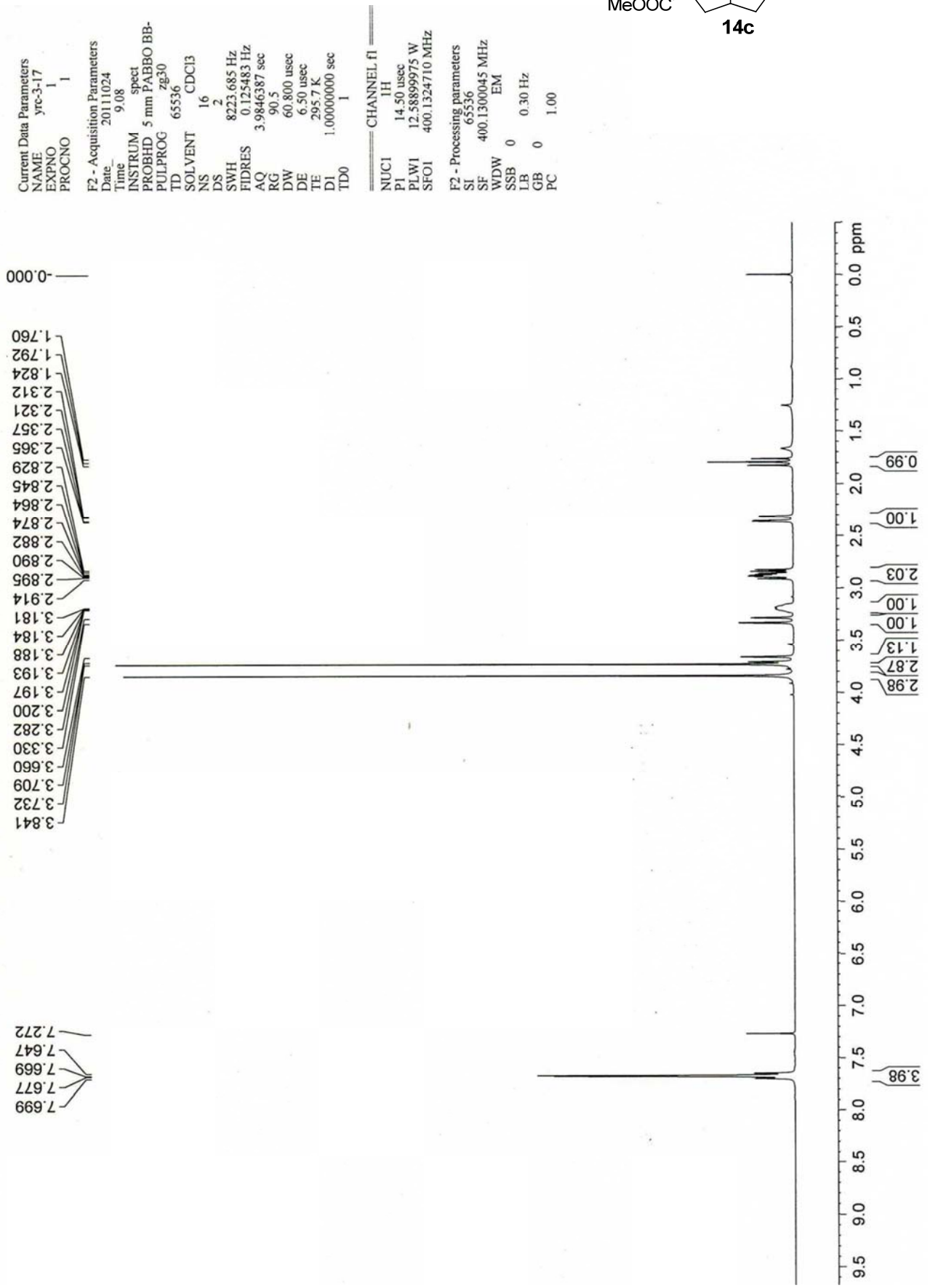
13c



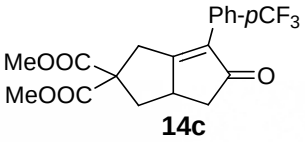
14c: ^{1}H-NMR



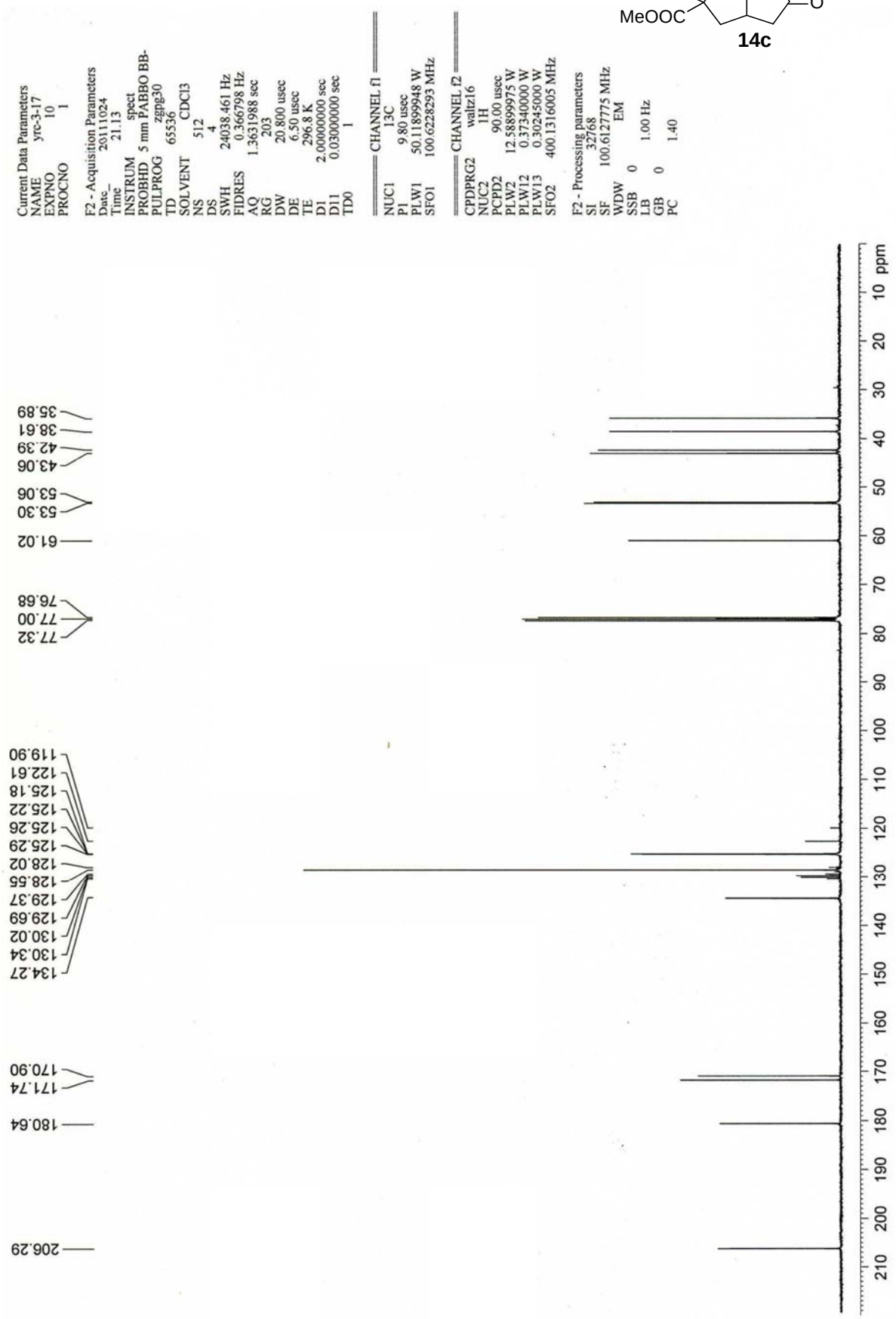
14c



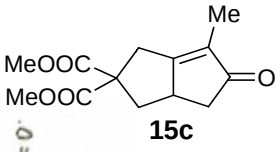
14c: ^{13}C-NMR



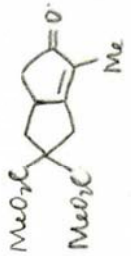
14c



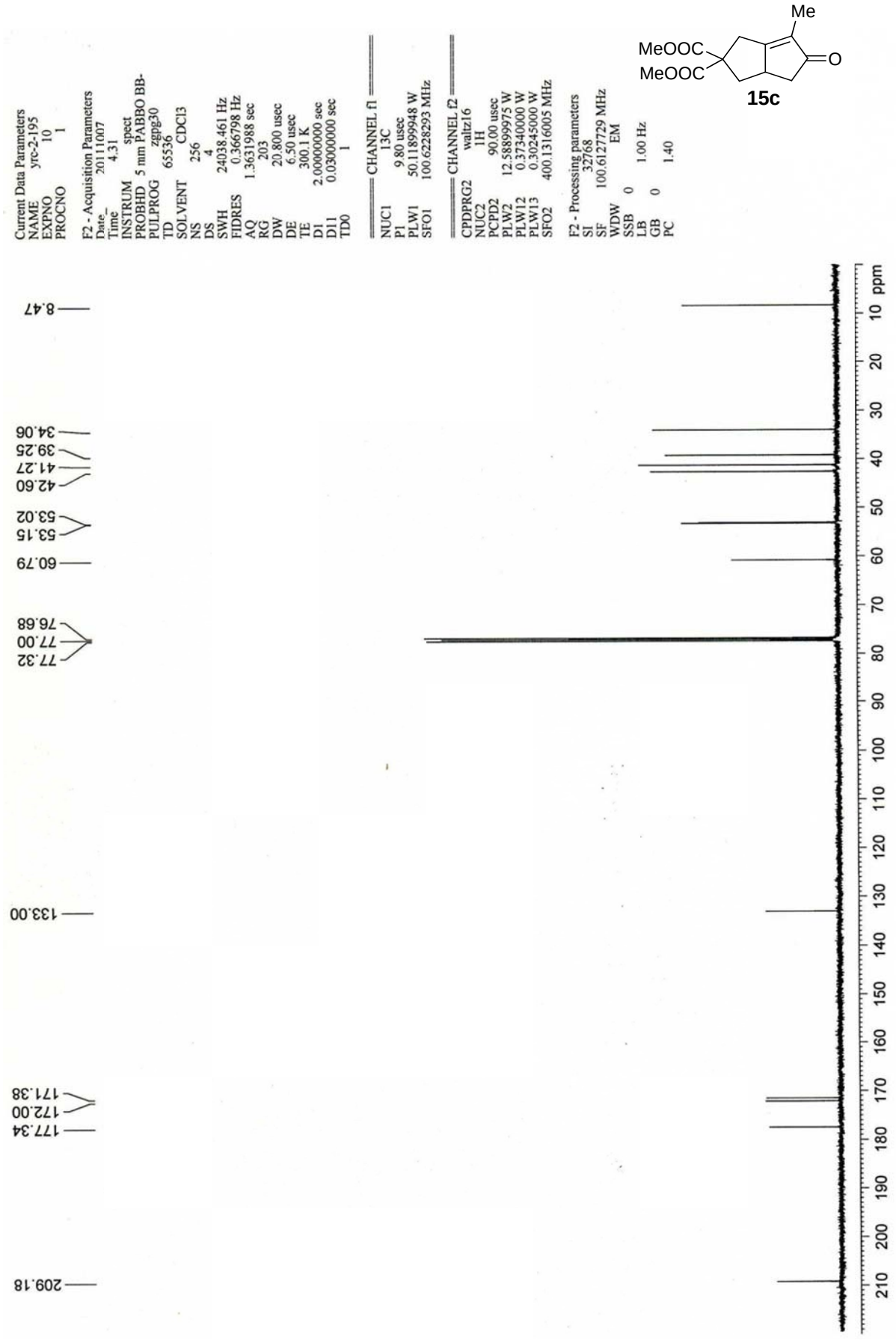
15c: ^{1}H-NMR



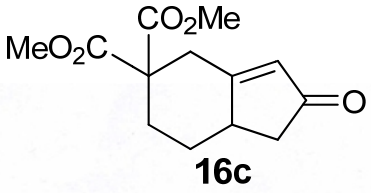
15c



15c: ^{13}C-NMR

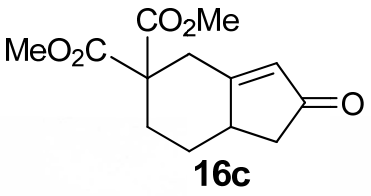

Me
MeOOC
MeOOC
O
15c
Current Data Parameters
NAME yrc-2-195
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111007
Time 4.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 256
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
8.47
34.06
39.25
41.27
42.60
53.02
53.15
60.79
76.68
77.00
77.32
133.00
171.38
172.00
177.34
209.18
ppm

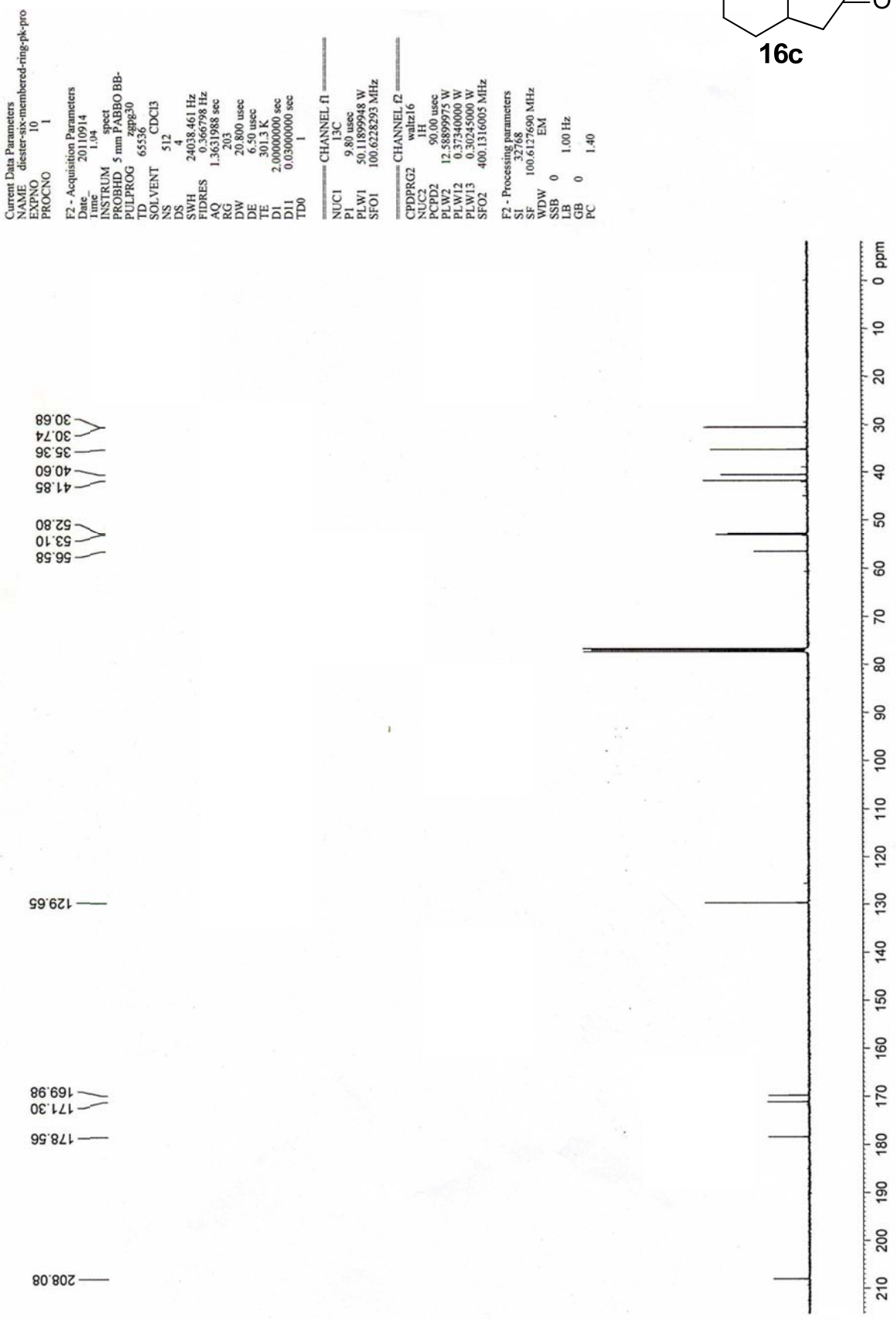
16c: ^{1}H-NMR



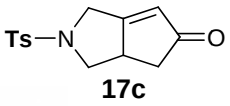
16c

16c: ^{13}C-NMR

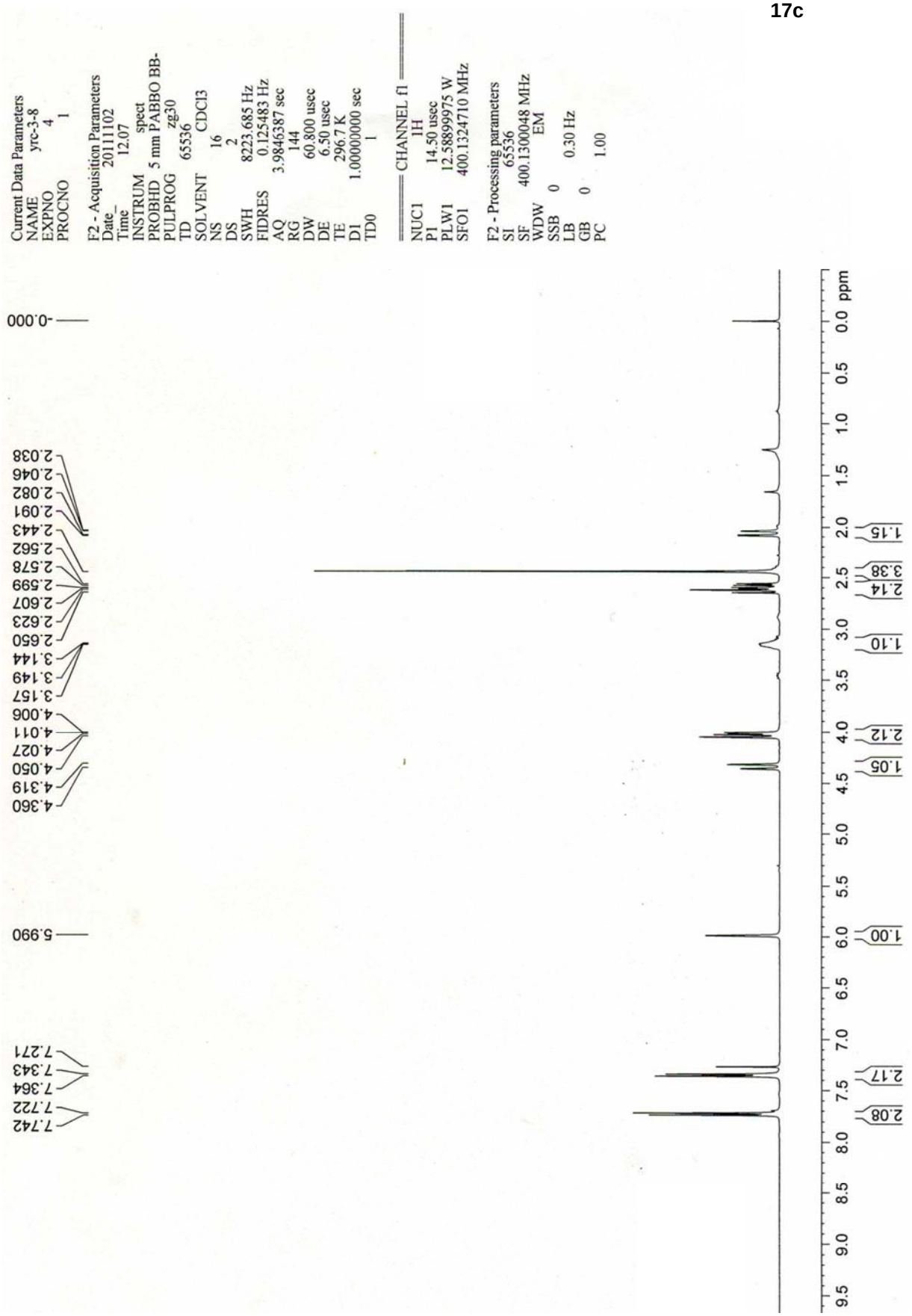

CO2Me
MeO2C
O
16c


Current Data Parameters
NAME diester-six-membered-ring-pk-pro
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110914
Time 1.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
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 301.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
30.68
30.74
35.36
40.60
41.85
52.80
53.10
56.58
129.65
169.98
171.30
178.56
208.08
0 ppm
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210

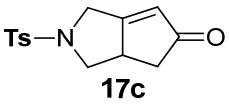
17c: ^{1}H-NMR



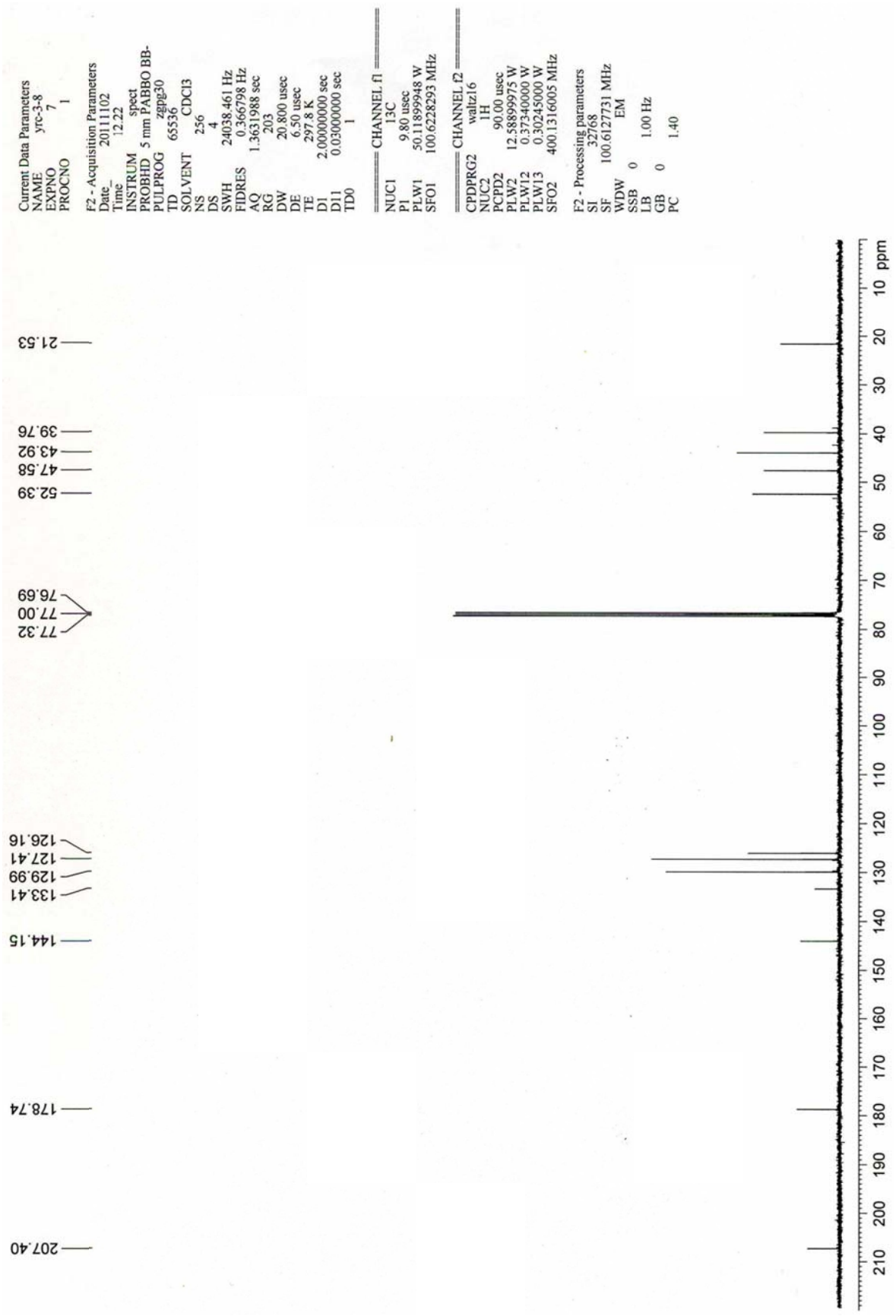
17c



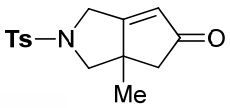
17c: ^{13}C-NMR



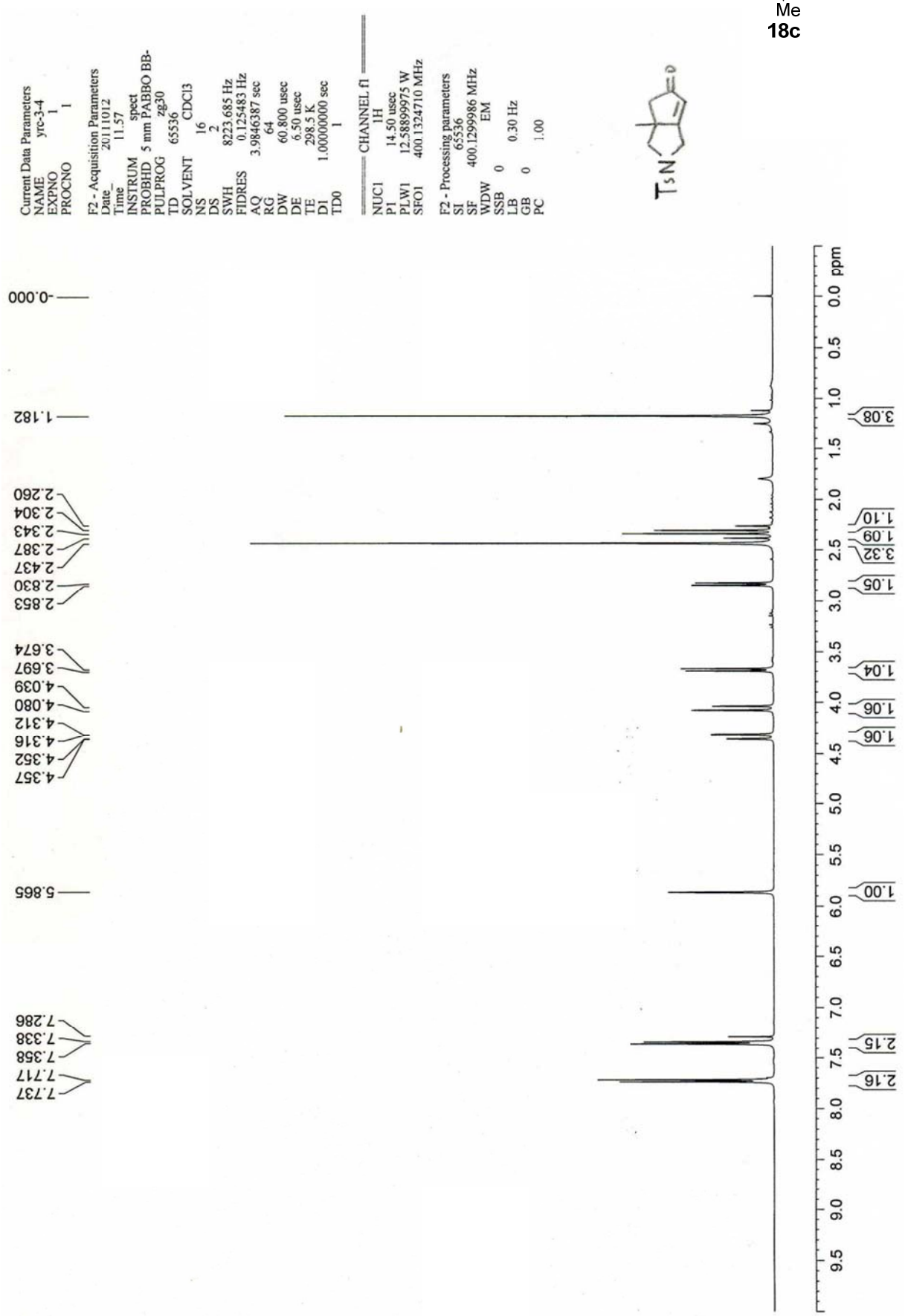
17c



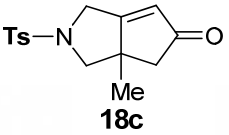
18c: ^{1}H-NMR



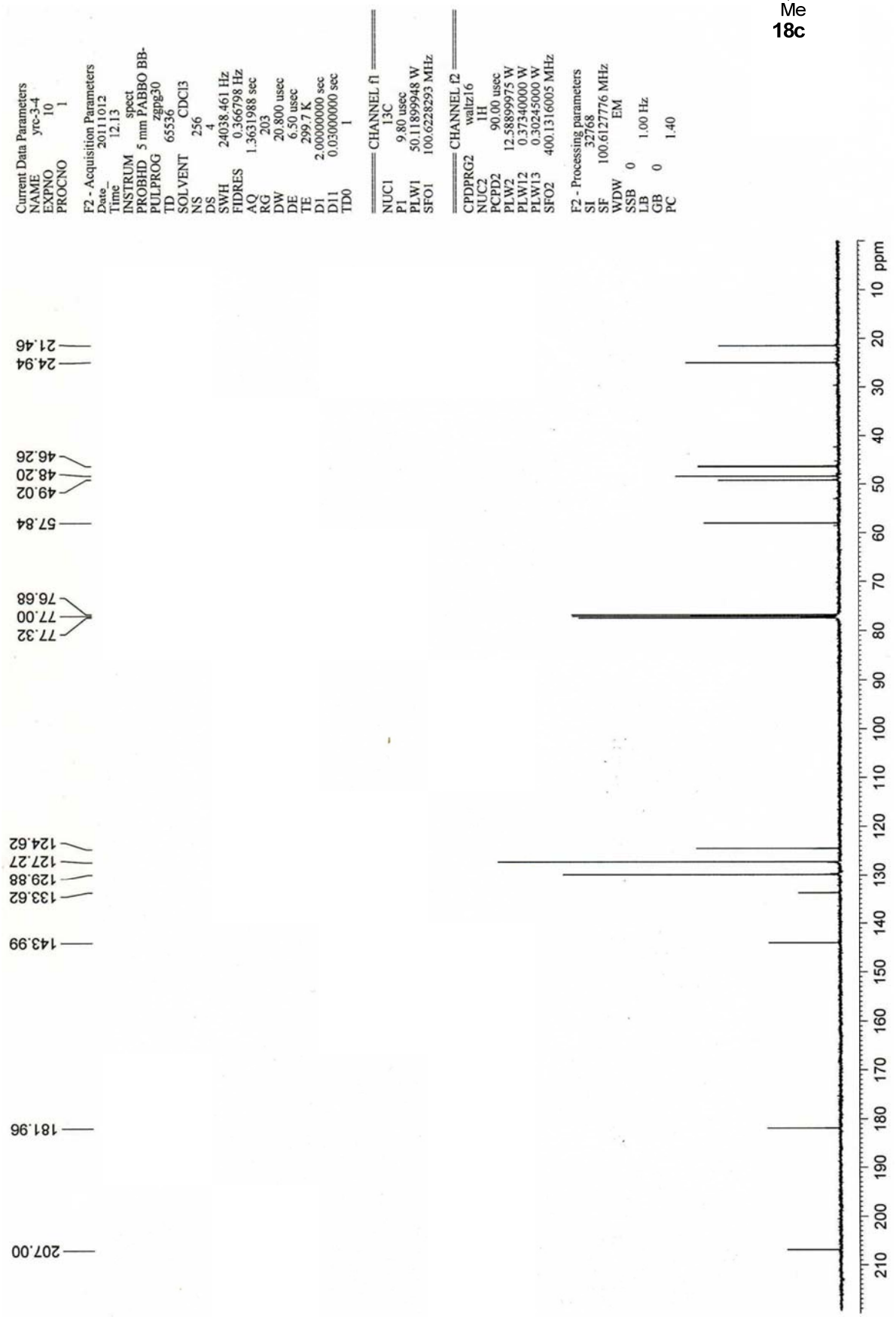
18c



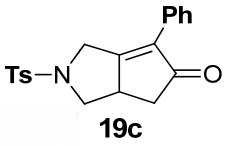
18c: ^{13}C-NMR



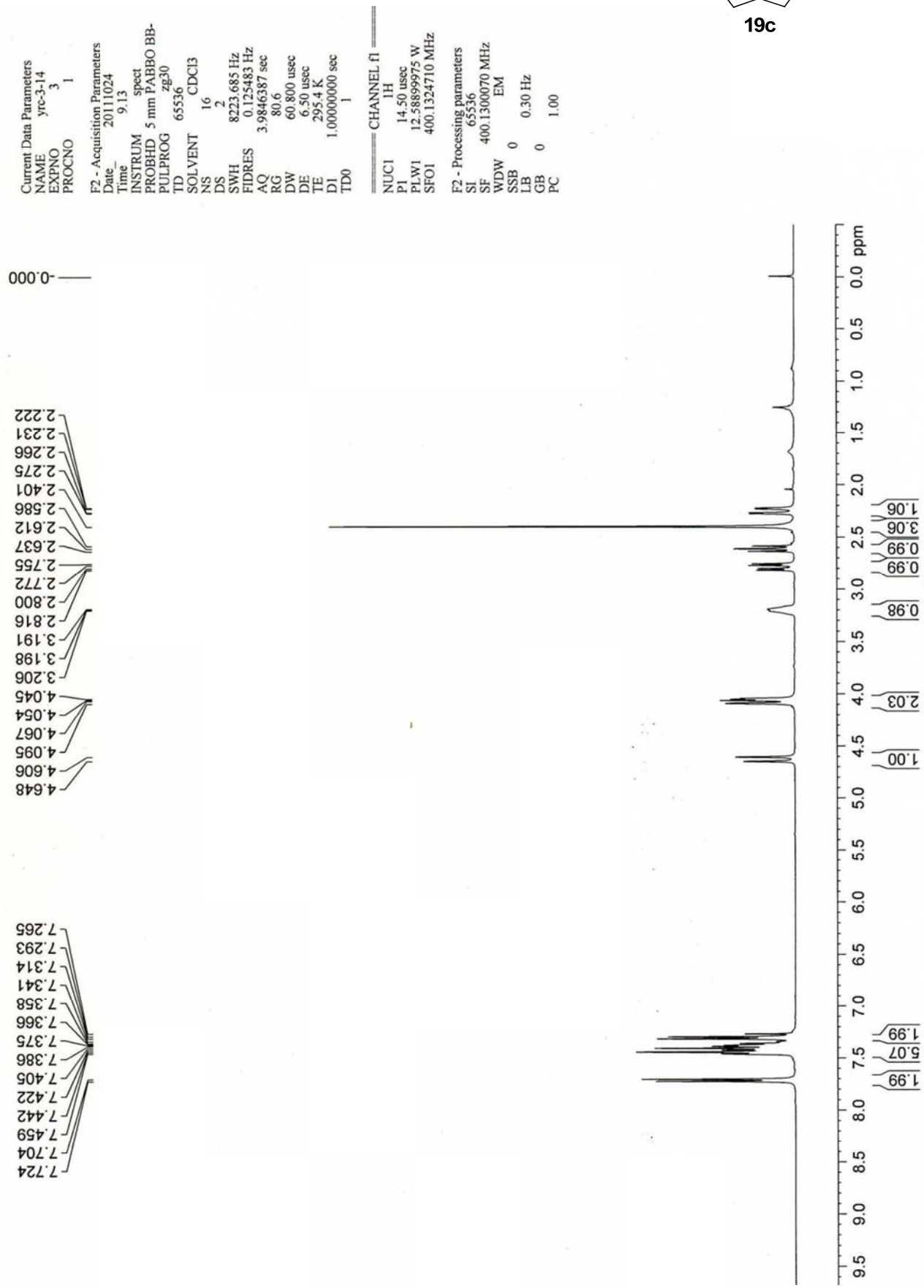
18c



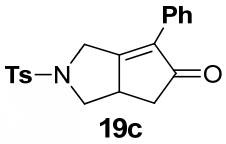
19c: ^{1}H-NMR

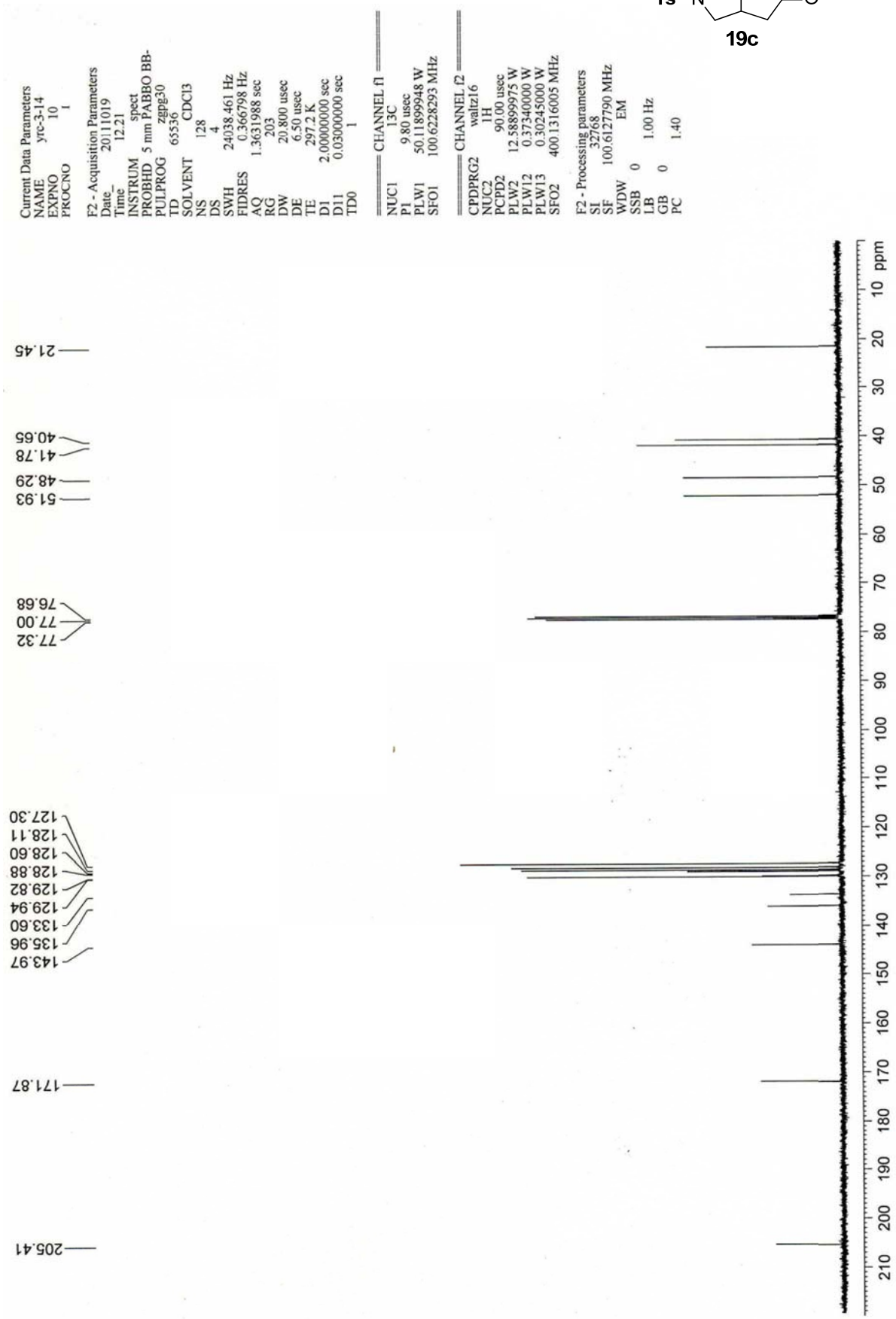


19c

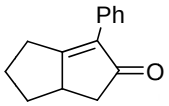


19c: ^{13}C-NMR

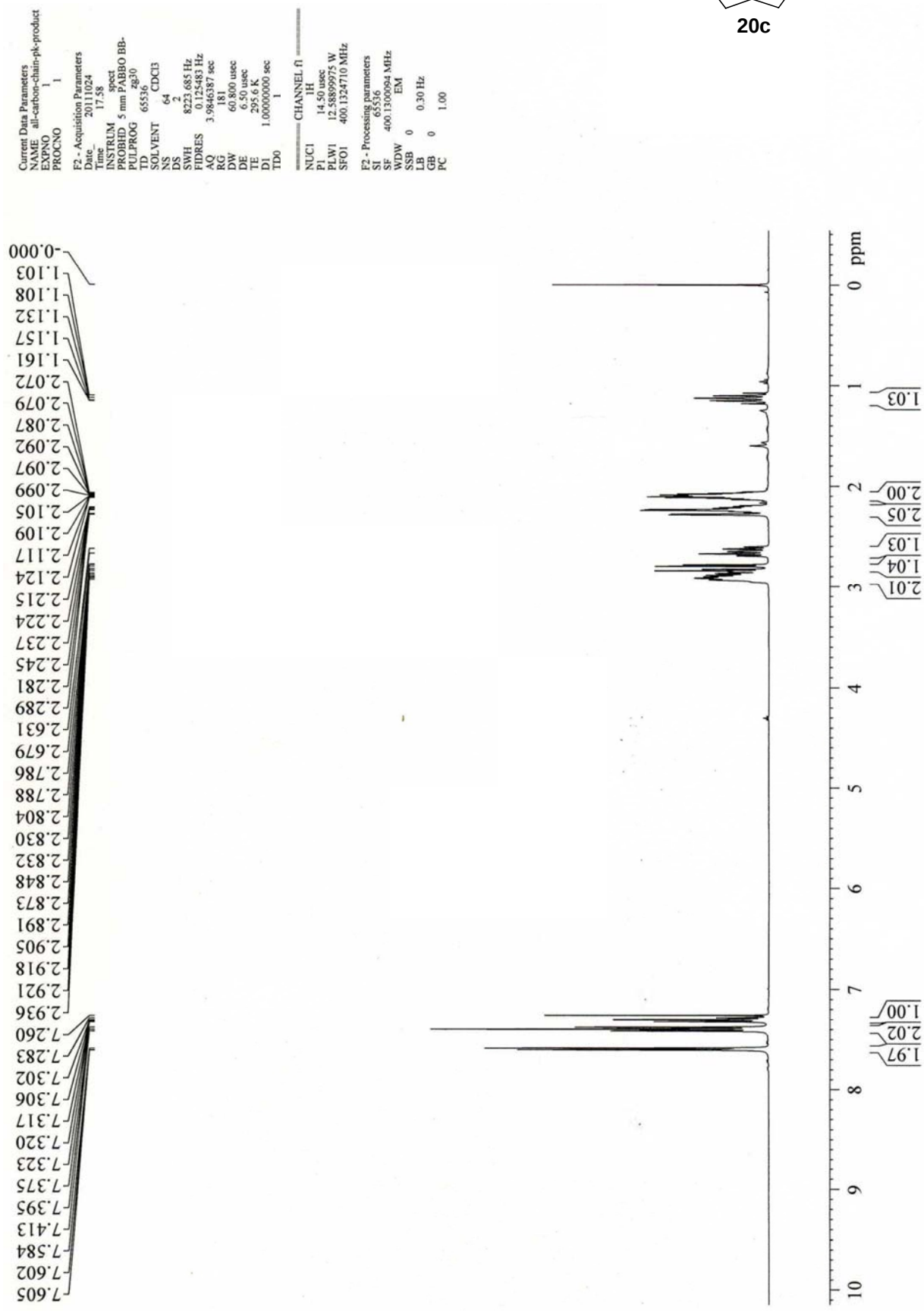

Ph
Ts–N
O
19c


Current Data Parameters
NAME yrc-3-14
EXPNO 10
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111019
Time_ 12.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 128
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.80 usec
PLW1 50.11899948 W
SFO1 100.6228293 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 12.58899975 W
PLW12 0.37340000 W
PLW13 0.30245000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127790 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
21.45
40.65
41.78
48.29
51.93
76.68
77.00
77.32
127.30
128.11
128.60
128.88
129.82
129.94
133.60
135.96
143.97
171.87
205.41
10 ppm

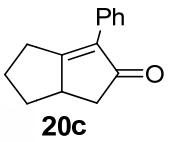
20c: ^{1}H-NMR



20c



20c: ^{13}C-NMR



20c