Supplementary Material (ESI) for Chemical Communications

Supporting Information

The Use of Halogen to Promote Regioselective Gold-Catalyzed Rearrangement of Propargylic Carboxylates: Efficient Syntheis of (1Z, 3E)-1-Halo-2-Carboxy-1,3 Dienes from Terminally Halogenated Propargyl Carboxylates

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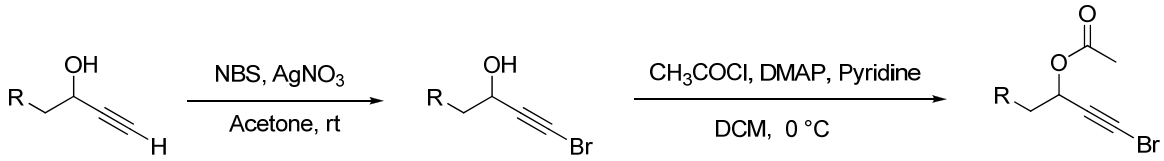
General. Ethyl acetate (ACS grade), hexanes (ACS grade) and diethyl ether (ACS grade) were purchased from Fisher Scientific and used without further purification. Anhydrous 1, 2-dichloroethane (HPLC grade) was purified by distillation over calcium hydride. Anhydrous tetrahydrofuran in Pure-Pac™ from Aldrich was used directly without further purification. Commercially available reagents were used without further purification. Reactions were monitored by thin layer chromatography (TLC) using silicycle pre-coated silica gel plates. Flash column chromatography was performed over silicycle silica gel (230-400 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian 500 MHz Unity plus spectrometer and a Varian 400 MHz spectrometer using residue solvent peaks as internal standards. Residue solvent peaks as internal standards ($CHCl_3$, ^{1}H: 7.26 ppm; ^{13}C: 77.2 ppm). Infrared spectra were recorded with a Perkin Elmer FT-IR spectrum 2000 spectrometer and are reported in reciprocal centimeter (cm^{-1}). Mass spectra were recorded with Micromass QTOF2 Quadrupole/Time-of-Flight Tandem mass spectrometer using electron spray ionization.

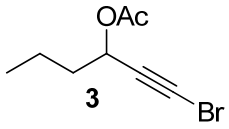
General procedure A: preparation of bromopropargyl alcohols and acetates



A solution of a propargyl alcohol (10 mmol) and $AgNO_3$ (2 mmol) in acetone (1.0 M) was stirred at rt for 25 min, which was followed by the addition of NBS (11 mmol). The reaction mixture was stirred at rt until no starting material was left as indicated by TLC. The reaction mixture was filtered through a small pad of celite. The filtrate was concentrated under *vacuum* and the crude bromopropargyl alcohol was purified by silica gel flash column chromatography.

To a solution of the above bromopropargyl alcohol (2.0 mmol), pyridine (20.0 mmol) and DMAP (0.2 mmol) in anhydrous CH_2Cl_2 (6 mL) at 0 °C was slowly added acetyl chloride (0.29 mL, 4 mmol). The reaction was stired at the same temperature for 30 min before being diluted with hexanes (30 mL). The solid precipitates were filtered off and the filtrate obtained was concentrated. The residue was purified by flash column chromatography to give desired bromopropargyl acetates.

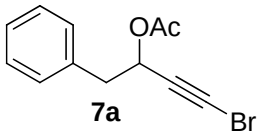
1-Bromohex-1-yn-3-yl acetate



3

Compound **3** was prepared in 81 % yield according to the general procedure **A** (eluents: ethyl acetate: hexanes = 1: 70). ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.35 (t, 1H, *J* = 6.5 Hz), 2.08 (s, 3H), 1.76 – 1.72 (m, 2H), 1.49 – 1.42 (m, 2H), 0.94 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.1, 77.9, 64.7, 45.9, 36.8, 21.1, 18.5, 13.8; IR (neat): 2962, 2875, 2220, 1747, 1651, 1373, 1230, 1020; MS (ES^+) Calculated for $[C_8H_{12}BrO_2]^+$: 219.0; Found: 219.0.

4-Bromo-1-phenylbut-3-yn-2-yl acetate

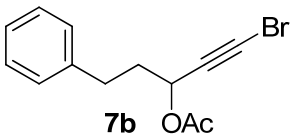


7a

Compound **7a** was prepared in 76 % yield according to the general procedure **A** (eluents: ethyl acetate: hexanes = 1: 100). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 – 7.32 (m, 2H), 7.22 – 7.27 (m, 3H), 5.52 (t, 1H, *J* = 6.8 Hz), 3.06 (d, 2H, *J* = 6.8 Hz), 2.04 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.9, 135.7, 129.8, 128.6, 127.3, 77.3, 65.3, 47.1, 41.2, 21.1;

IR (neat): 3448, 2933, 2220, 1747, 1373, 1228, 1022; MS (ES^+) Calculated for $[C_{12}H_{11}BrNa\ O_2]^+$: 289.0; Found: 289.0.

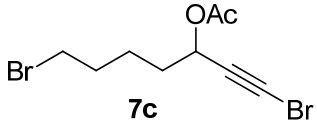
1-Bromo-5-phenylpent-1-yn-3-yl acetate



7b

Compound **7b** was prepared in 86 % yield according to the general procedure **A** (eluents: ethyl acetate: hexanes = 1: 100). ^1H NMR (500 MHz, $CDCl_3$) δ 7.32 – 7.29 (m, 2H), 7.19 – 7.23 (m, 3H), 5.36 (t, 1H, *J* = 6.5 Hz), 2.78 (t, 1H, *J* = 7.5 Hz), 2.09 – 2.14 (m, 2H), 2.08 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.0, 140.7, 128.7, 128.6, 126.4, 77.6, 64.3, 46.6, 36.3, 31.4, 21.1; IR (neat): 3428, 2217, 1739, 1643, 1371, 1228, 1022, 700; MS (ES^+) Calculated for $[C_{13}H_{13}BrNaO_2]^+$: 303.0; Found: 303.0.

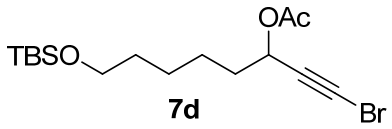
1, 7-Dibromohept-1-yn-3-yl acetate



7c

Compound **7c** was prepared in 89 % yield according to the general procedure **A** (eluents: ethyl acetate: hexanes = 1: 50). ^1H NMR (500 MHz, $CDCl_3$) δ 5.36 (t, 1H, *J* = 6.0 Hz), 3.41(t, 2H, *J* = 7.0Hz), 2.09 (s, 3H), 1.88 – 1.91 (m, 2H), 1.77 – 1.82 (m, 2H), 1.58 – 1.62 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.0, 64.5, 46.5, 33.9, 33.3, 32.3, 23.8, 21.1; IR (neat): 2218, 1747, 1643, 1371, 1228, 1020; MS (ES^+) Calculated for $[C_9H_{12}Br_2NaO_2]^+$: 332.9; Found: 332.9.

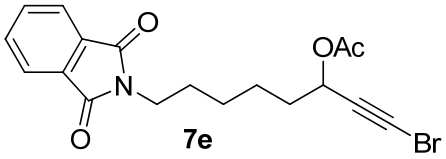
1-Bromo-8-(tert-butyldimethylsilyloxy)oct-1-yn-3-yl acetate



7d

Compound **7d** was prepared in 80 % yield according to the general procedure **A** (eluents: ethyl acetate: hexanes = 1: 100). ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.34 (t, 1H, *J* = 6.8 Hz), 3.60 (t, 2H, *J* = 6.5 Hz), 2.07 (s, 3H), 1.73 – 1.78 (m, 2H), 1.49 – 1.55 (m, 2H), 1.39 – 1.43 (m, 2H), 1.34 – 1.37 (m, 2H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.1, 77.8, 64.8, 63.1, 45.9, 34.8, 32.7, 26.1, 25.5, 24.9, 21.1, 18.5, -5.1; IR (neat): 2931, 2858, 2218, 1747, 1651, 1373, 1230, 1097, 835; MS (ES^+) Calculated for $[C_{16}H_{29}BrNaO_3Si]^+$: 399.1; Found: 399.1.

1-Bromo-8-*N*-phth-oct-1-yn-3-yl acetate

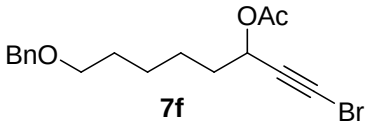


7e

Compound **7e** was prepared in a 72 % yield according the following procesure: To a stirred solution of **7d** (5 mmol) in THF (30mL) is added TBAF (6 mmol, 1M in THF) at rt and leave the reaction overnight. The reaction was concentrated under *vacuum* and the residue was purified through silica gel flash column chromatography (hexane/ethyl acetate = 10:1). To a solution of the alcohol above (1.33 mmol), phthalimide (1.463 mmol), PPh_3 (1.463 mmol) in anhydrous THF (0.3M) were added DEAD (1.463 mmol) dropwise at 0 ^{o}C. The resulting mixture was stirred at rt for 2h. After all staring material was consumed, the mixture was concentrated under *vacuum* and the residue was purified through silica gel flash column chromatography (eluents: ethyl acetate: hexanes = 1:15). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 – 7.85 (m, 2H), 7.70 – 7.72 (m, 2H), 5.33 (t, 1H, *J* = 7.0 Hz), 3.68(t, 2H, *J* = 7.0 Hz), 2.07 (s, 3H), 1.74 – 1.77 (m, 2H), 1.68 – 1.72(m, 2H), 1.46 – 1.49 (m, 2H), 1.36 – 1.40 (m, 2H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.1, 168.6,

134.1, 132.3, 123.4, 77.7, 64.7, 64.6, 46.1, 38.0, 34.6, 28.6, 26.6, 21.1; IR (neat): 3461, 2939, 2218, 1747, 1709, 1396, 1373, 1230, 721; MS (ES^+) Calculated for $[C_{18}H_{18}BrNNaO_4]^+$: 414.0; Found: 414.0.

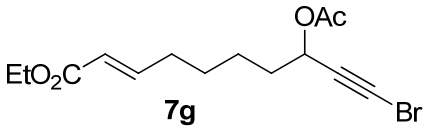
8-(Benzyloxy)-1-bromooct-1-yn-3-yl acetate



7f

Compound **7f** was prepared in 91 % yield according to the general procedure **A** (eluents: ethyl acetate: hexanes = 1: 100). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.35 (m, 4H), 7.27 – 7.29 (m, 1H), 5.34 (t, 1H, *J* = 6.4 Hz), 4.50 (s, 2h), 3.47 (t, 2H, *J* = 6.4 Hz), 2.08 (s, 3H), 1.74 – 1.79 (m, 2H), 1.62 – 1.65 (m, 2H), 1.40 – 1.46 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.1, 138.8, 128.6, 127.8, 127.7, 77.8, 73.1, 70.4, 64.8, 46.0, 34.8, 29.8, 26.0, 25.0, 21.2.; IR (neat): 2860, 2935, 2860, 2216, 1747, 1371, 1230, 1101, 1022; MS (ES^+) Calculated for $[C_{17}H_{21}BrNaO_3]^+$: 375.1; Found: 375.1.

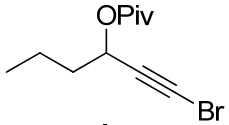
(*E*)-Ethyl 8-acetoxy-10-bromodec-2-en-9-ynoate



7g

Compound **7g** was prepared in overall 72 % yield via routine deprotection, oxidation and subsequent HWE reaction. The residue was purified through silica gel flash column chromatography (eluents: ethyl acetate: hexanes = 1: 10). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.94 (dt, 1H, *J* = 15.5, 7.0 Hz), 5.81 (dt, 1H, *J* = 15.5, 1.5 Hz), 5.34 (t, 1H, *J* = 6.5 Hz), 4.18 (q, 2H, *J* = 7.0 Hz), 2.19 – 2.24 (m, 2H), 2.07 (s, 3H), 1.74 – 1.79 (m, 2H), 1.44 – 1.51 (m, 4H), 1.28 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 170.1, 166.9, 148.8, 121.9, 77.7, 64.6, 60.4, 46.3, 34.5, 32.1, 27.7, 24.6, 21.1, 14.5; IR (neat): 2938, 2218, 1747, 1718, 1651, 1371, 1230, 1022; MS (ES^+) Calculated for $[C_{14}H_{19}BrNaO_4]^+$: 353.0; Found: 353.0.

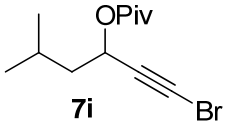
1-Bromohex-1-yn-3-yl pivalate



7h

Compound **7h** was prepared in 81 % yield according to the general procedure **A** using pivaloyl chloride instead as the acylating reagent (eluents: ethyl acetate: hexanes = 1: 50). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.33 (t, 1H, *J* = 6.8 Hz), 1.71 – 1.77 (m, 2H), 1.40 – 1.49 (m, 2H), 1.20 (s, 9H), 0.94 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.4, 78.1, 64.4, 45.4, 39.0, 36.9, 27.3, 18.5, 13.9; IR (neat): 3445, 2956, 2874, 2213, 1729, 1642, 1276, 1143; MS (ES^+) Calculated for $[C_{11}H_{17}BrNaO_2]^+$: 283.03; Found: 283.06.

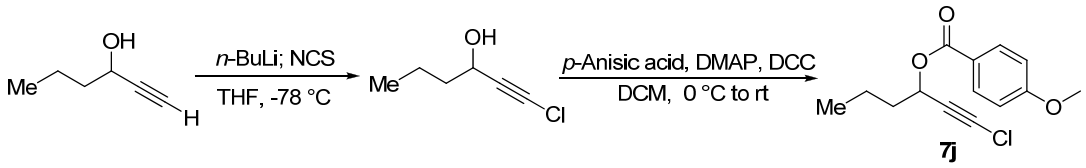
1-Bromo-5-methylhex-1-yn-3-yl pivalate



7i

Compound **7i** was prepared in 77 % yield according to the general procedure **A** using pivaloyl chloride instead as the acylating reagent (eluents: ethyl acetate: hexanes = 1: 50). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.38 (t, 1H, *J* = 7.2 Hz), 1.61 – 1.79 (m, 3H), 1.21 (s, 9H), 0.94 (d, 3H, *J* = 5.2 Hz), 0.93(d, 3H, *J* = 5.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 177.6, 78.2, 63.4, 45.4, 43.5, 38.9, 27.2, 24.9, 22.7, 22.5; IR (neat): 2960, 2220, 1738, 1458, 1281, 1153, 1138, 1034; MS (ES^+) Calculated for $[C_{12}H_{19}BrNaO_2]^+$: 297.1; Found: 297.1.

1-Chlorohex-1-yn-3-yl 4-methoxybenzoate



7j

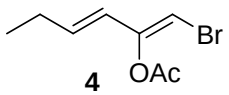
To a solution of hex-1-yn-3-ol in THF (0.2 M) was added *n*-BuLi (1.6 M in hexane) (2.2 equiv) at –78 °C; after stirring for 10 min, NCS (2.2 equiv) was added into the mixture. The reaction mixture was stirred at –78 °C for 1 h, and the dry ice/acetone bath was

removed to allow the reaction to warm to room temperature. The resulting mixture was then diluted with H_2O (20 mL) and extracted with CH_2Cl_2 (150 mL). The organic layer was dried ($MgSO_4$). The solvent was removed under reduced pressure. The residue was purified by flash column chromatography on silica gel to give 1-chlorohex-1-yn-3-ol as the desired product.

To a stirred solution of anisic acid (10 mmol) in 10 mL anhydrous CH_2Cl_2 was added DMAP (1 mmol) and 1-chlorohex-1-yn-3-ol (10 mmol). DCC (11 mmol) was added to the reaction mixture at 0 °C, and the resulting mixture was stirred overnight at room temperature. The precipitated urea was then filtered off and the filtrate evaporated in *vacuum.* The residue was purified by flash column chromatography on silica gel to yield product **7j** in 64% yield (eluents: ethyl acetate: hexane = 1: 100). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 (d, 2H, *J* = 8.5 Hz), 6.92 (d, 2H, *J* = 8.5 Hz), 5.56 (t, 1H, *J* = 6.5 Hz), 3.86 (s, 3H), 1.86 – 1.91 (m, 2H), 1.49 – 1.52 (m, 2H), 1.33 – 1.35 (m, 4H), 0.90 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 165.4, 163.8, 132.1, 122.4, 113.9, 67.5, 64.6, 64.2, 55.7, 35.0, 31.5, 24.9, 22.7, 14.2; IR (neat): 2956, 2931, 2243, 1718, 1606, 1510, 1257, 1169, 1095, 1031, 769; MS (ES^+) Calculated for $[C_{16}H_{19}ClNaO_3]^+$: 317.1; Found: 317.1.

General procedure for gold catalysis: A halopropargyl acetate or pivalate (0.30 mmol) was dissolved in anhydrous DCM (1.5 mL) at room temperature and then PPh_3AuNTf_2 (5 mol %) was added. The reaction mixture was stirred at room temperature and the progress of the reaction was monitored by TLC. The reactions typically took 10 min but for **7j** 3 h was necessary. Upon completion, the reaction was quenched by Et_3N and then the mixture was concentrated under *vacuum*. The residue was purified through silica gel flash column chromatography to afford the desired product.

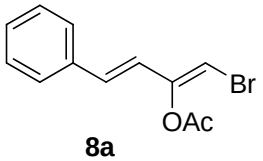
(1*Z*, 3*E*)-1-Bromohexa-1, 3-dien-2-yl acetate



4

Compound **4** was prepared in 85 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 100). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.04 (s, 1H), 5.98 (d, 1H, *J* = 15.0 Hz), 5.86 (dt, 1H, *J* = 15.0, 6.0 Hz), 2.29 (s, 3H), 2.07 – 2.14 (m, 2H), 1.02 (t, 3H, *J* = 7.5 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.2, 150.0, 135.0, 121.4, 97.7, 25.5, 20.7, 13.0; IR (neat): 2972, 2937, 1770, 1699, 1373, 1196; MS (ES^+) Calculated for $[C_8H_{11}BrNaO_2]^+$: 241.0; Found: 241.0.

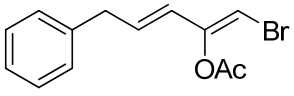
(1*Z*, 3*E*)-1-Bromo-4-phenylbuta-1, 3-dien-2-yl acetate



8a

Compound **8a** was prepared in 85 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 40). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.43 (m, 2H), 7.29 – 7.36 (m, 3H), 6.63 – 6.70 (m, 2H), 6.29 (s, 1H), 2.39 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.1, 150.1, 135.8, 130.4, 129.0, 128.8, 127.1, 120.6, 100.3, 20.7; IR (neat): 3502, 3084, 1770, 1371, 1190, 1016, 945; MS (ES^+) Calculated for $[C_{12}H_{11}BrNaO_2]^+$: 289.0; Found: 289.0.

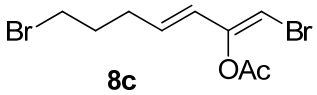
(1*Z*, 3*E*)-1-Bromo-5-phenylpenta-1, 3-dien-2-yl acetate



8b

Compound **8b** was prepared in 91 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 40). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 – 7.32 (m, 2H), 7.23 – 7.25 (m, 1H), 7.16 – 7.17 (m, 1H), 6.09 (s, 1H), 5.97 – 6.03 (m, 2H), 3.43 (d, J = 5.5 Hz), 2.28 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.1, 149.7, 139.0, 131.7, 128.9, 128.8, 126.7, 123.6, 98.8, 38.6, 20.7; IR (neat): 3419, 1768, 1653, 1643, 1633, 1196, 700; MS (ES^+) Calculated for $[C_{13}H_{13}BrNaO_2]^+$: 303.0; Found: 303.0.

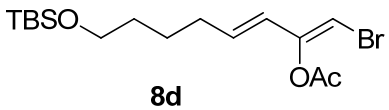
(1*Z*, 3*E*)-1, 7-Dibromohepta-1, 3-dien-2-yl acetate



8c

Compound **8c** was prepared in 96 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 40). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.09 (s, 1H), 6.06 (d, 1H, *J* = 16.0 Hz), 5.76 (dt, 1H, *J* = 16.0, 7.0 Hz), 3.39 (t, 2H, *J* = 7.0 Hz), 2.30 (s, 3H), 2.25 – 2.28 (m, 2H), 1.94 – 1.99 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.1, 149.6, 131.0, 123.7, 98.7, 33.0, 31.6, 30.7, 20.7; IR (neat): 2920, 1770, 1702, 1432, 1371, 1194, 1016, 937; MS (ES^+) Calculated for $[C_9H_{12}Br_2KO_2]^+$: 348.9; Found: 348.9.

(1*Z*, 3*E*)-1-Bromo-8-(tert-butyldimethylsilyloxy)octa-1, 3-dien-2-yl acetate

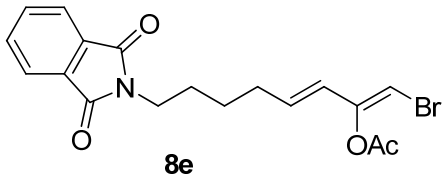


8d

Compound **8d** was prepared in 86 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 50). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.04 (s, 1H), 5.99 (d, 1H, *J* = 15.5Hz), 5.81 (dt, 1H, *J* = 15.5, 6.5 Hz), 3.59 (t, 2H, *J* = 5.8 Hz), 2.29 (s, 3H), 2.11 (q, 2H, *J* = 7.0 Hz), 1.43 – 1.52 (m, 4H), 0.89 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.2, 149.9, 133.5, 122.4, 97.8, 63.1, 32.5, 32.3, 26.2, 25.2, 20.7, 18.6, –5.1;

IR (neat): 2939, 1736, 1651, 1240, 1198, 1045; MS (ES^+) Calculated for $[C_{16}H_{29}BrNaO_3Si]^+$: 399.1; Found: 399.1.

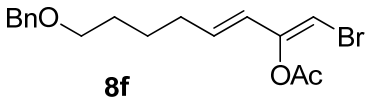
(1*Z*, 3*E*)-1-Bromo-8-(2, 5-dioxo-2, 5-dihydro-1H-pyrrol-1-yl) octa-1, 3-dien-2-yl acetate



8e

Compound **8e** was prepared in 88 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 30). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.81– 7.85 (m, 2H), 7.69 – 7.73 (m, 2H), 6.04 (s, 1H), 5.98 (d, 1H, *J* = 16.0 Hz), 5.78 (dt, 1H, *J* = 16.0, 7.0 Hz), 3.68 (t, 2H, *J* = 7 Hz), 2.28 (s, 3H), 2.11 – 2.16 (m, 2H), 1.71 – 1.65 (m, 2H), 1.48 – 1.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 168.6, 167.1, 149.7, 134.1, 132.7, 132.3, 123.4, 122.8, 98.1, 37.8, 31.9, 28.2, 26.0, 20.7; IR (neat): 3460, 1768, 1709, 1396, 1371, 1188, 721; MS (ES^+) Calculated for $[C_{18}H_{18}BrNNaO_4]^+$: 414.0; Found: 414.0.

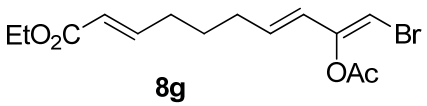
(1*Z*, 3*E*)-8-(Benzyloxy)-1-bromoocta-1, 3-dien-2-yl acetate



8f

Compound **8f** was prepared in 90 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 30). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.37 (m, 4H), 7.28 – 7.32 (m, 1H), 6.04 (s, 1H), 6.00 (d, 1H, *J* = 15.5 Hz), 5.81 (dt, 1H, *J* = 15.5, 7.0 Hz), 4.50 (s, 2H), 3.47 (t, 2H, *J* = 6.0 Hz), 2.29 (s, 3H), 2.09 – 2.14 (m, 2H), 1.59 – 1.63 (m, 2H), 1.49 – 1.54 (m, 2H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.1, 149.8, 138.7, 133.3, 128.6, 127.8, 127.7, 122.5, 73.1, 70.2, 32.2, 29.4, 25.5, 20.7; IR (neat): 2918, 2850, 1770, 1718, 1371, 1194; MS (ES^+) Calculated for $[C_{17}H_{21}BrNaO_3]^+$: 352.1; Found: 352.1.

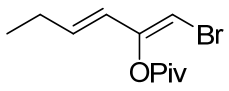
(2*E*, 7*E*, 9*Z*)-Ethyl 9-acetoxy-10-bromodeca-2, 7, 9-trienoate



8g

Compound **8g** was prepared in 88 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 60). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.92 (dt, 1H, *J* = 15.5, 7.0 Hz), 6.06 (s, 1H), 5.99 (d, 1H, *J* = 15.5 Hz), 5.74 – 5.82 (m, 2H), 4.18 (q, 2H, *J* = 7.0 Hz), 2.29 (s, 3H), 2.17 – 2.21 (m, 2H), 2.10 – 2.14 (m, 2H), 1.55 – 1.63 (m, 2H), 1.28 (t, 3H, *J* = 7.0 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 167.1, 166.8, 149.7, 148.5, 132.4, 123.0, 122.0, 60.4, 31.8, 31.6, 27.1, 20.7, 14.5; IR (neat): 2929, 1770, 1716, 1651, 1371, 1190, 1043; MS (ES^+) Calculated for $[C_{14}H_{19}BrNaO_4]^+$: 353.0; Found: 353.0.

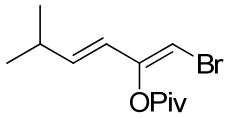
(1*Z*, 3*E*)-1-Bromohexa-1, 3-dien-2-yl pivalate



8h

Compound **8h** was prepared in 88 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 40). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.03 (s, 1H), 5.99 (d, 1H, *J* = 15.6 Hz), 5.74 (dt, 1H, *J* = 15.6, 6.4 Hz), 2.08 – 2.12 (m, 2H), 1.37 (s, 9H), 1.01 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.5, 149.8, 134.5, 121.7, 101.8, 97.5, 39.6, 27.6, 25.5, 13.1; IR (neat): 3424, 2966, 1760, 1642, 1265, 1108; Calculated for $[C_{11}H_{17}BrNaO_2]^+$: 283.03; Found: 283.06.

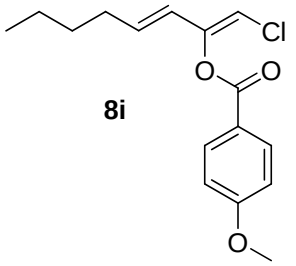
(1*Z*, 3*E*)-1-bromo-5-methylhexa-1, 3-dien-2-yl pivalate



8i

Compound **8i** was prepared in 90 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 40). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.04 (s, 1H), 5.95 (dd, 1H, *J* = 16.0, 1.5 Hz), 5.74 (dd, 1H, *J* = 16.0, 6.5 Hz), 2.32 – 2.35 (m, 1H), 1.37 (s, 9H), 1.00 (d, *J* = 6.5 Hz, 6H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 174.5, 149.9, 139.7, 119.9, 97.6, 39.6, 30.9, 27.5, 22.0; IR (neat): 2962, 2871, 1759, 1479, 1265, 1107, 962, 748; MS (ES^+) Calculated for $[C_{12}H_{19}BrNaO_2]^+$: 297.1; Found: 297.1.

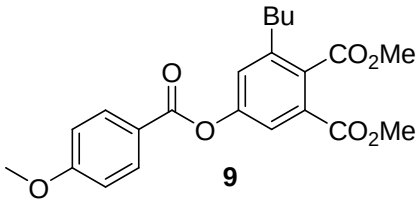
(1*Z*, 3*E*)-1-chloroocta-1, 3-dien-2-yl 4-methoxybenzoate



8i

Compound **8i** was prepared in 85 % yield according to the general procedure (eluents: ethyl acetate: hexanes = 1: 80). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 – 8.16 (m, 2H), 6.97 – 6.99 (m, 2H), 6.04 (d, 1H, *J* = 16.0 Hz), 6.03 (s, 1H), 5.86 (dt, 1H, J = 16.0, 7.2 Hz), 3.89 (s, 3H), 2.08 – 2.12 (m, 2H), 1.19 – 1.39 (m, 4H), 0.87 (t, 3H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 164.2, 162.7, 148,1, 133.4, 132.6, 121.8, 121.1, 114.1, 108.9, 55.7, 332.2, 31.0, 22.4, 14.0; IR (neat): 1738, 1604, 1510, 1246, 1167, 1072, 845.; MS (ES^+) Calculated for $[C_{16}H_{19}ClNaO_3]^+$: 317.1; Found: 317.1.

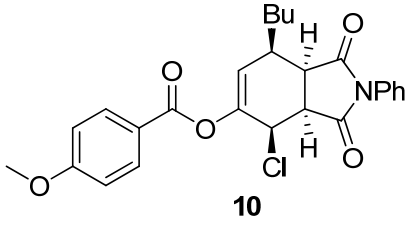
Dimethyl 3-butyl-5-(4-methoxybenzoyloxy) phthalate



9

An oven-dried vial was charged with diene **8j** (0.1mmol) and dimethyl acetylenedicarboxylate (0.2 mmol). Anisole (0.25 mL) was added. The reaction mixture was stirred at 185 ℃ for 10 h and then cooled to room temperature. After removed solvent under *vacuum* and the residue was purified to give **9** in 75% yield through silica gel flash column chromatography (eluents: ethyl acetate: hexanes = 1:15). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 – 8.16 (m, 2H), 7.70 (d, 1H, *J* = 2.4 Hz), 7.30 (d, 1H, *J* = 2.4 Hz), 6.97 – 7.00 (m, 2H), 3.94 (s, 3H), 3.90 (s, 3H), 3.88 (s, 3H), 2.63 (t, 2H, *J* = 6.0 Hz), 1.58 – 1.64 (m, 2H), 1.34 – 1.39 (m, 2H), 0.92 (t, 2H, *J* = 7.2 Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 169.6, 165.7, 164.6, 164.4, 151.3, 142.6, 132.7, 132.6, 129.5,
127.0, 121.4, 121.3, 114.1, 55.7, 52.8, 52.7, 33.3, 33.1, 22.7, 14.0; IR (neat): 3450, 2954, 1738, 1730, 1510, 1252, 1169, 1134, 1057, 847; MS (ES^+) Calculated for $[C_{22}H_{24}NaO_7]^+$: 423.1; Found: 423.1.

7-Butyl-4-chloro-1, 3-dioxo-2-phenyl-2, 3, 3a, 4, 7, 7a-hexahydro-1H-isoindol-5-yl 4-methoxybenzoate

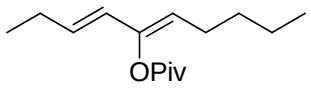


10

An oven-dried vial was charged with diene **8j** (0.1 mmol) and *N*-phenylmaleimide (0.15 mmol). Anisole (0.25 mL) was added. The reaction mixture was stirred at 100 ℃ for 18 h and then cooled to room temperature. After removed solvent under *vacuum*, the residue was purified to give cycloadduct **10** in 77% yield through silica gel flash column flash chromatography (eluents: ethyl acetate: hexanes =1:8). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 8.09 (m, 2H), 7.47 – 7.51(m, 2H), 7.39 – 7.43 (m, 1H), 7.33 – 7.35 (m, 2H), 6.96 – 6.98 (m, 2H), 6.05 (d, 1H, *J* = 6.8 Hz), 5.10 (d, 1H, *J* = 6.4 Hz), 3.94 (dd, 1H, J = 10.4, 6.4Hz), 3.91 (s, 3H), 3.54 – 3.59 (m, 1H), 3.11 – 3.16 (m, 1H), 2.18 – 2.24 (m, 1H), 1.31 – 1.49 (m, 3H), 0.92 (t, 3H, *J* = 6.4 Hz); ^{13}C NMR (125 MHz, $CDCl_3$)

δ 175.8, 173.6, 165.3, 164.5, 146.9, 132.6, 131.9, 129.4, 129.0, 126.8, 123.7, 120.9, 114.2 , 55.8, 51.2, 46.9, 40.5, 33.9, 31.3, 30.3, 22.9, 14.2; IR (neat): 3450, 1716, 1604, 1510, 1384, 1257, 1169; MS (ES^+) Calculated for $[C_{26}H_{26}ClNNaO_5]^+$: 490.1; Found: 490.1.

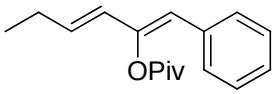
(*3E, 5Z*)-Deca-3, 5-dien-5-yl pivalate



11

To a mixture of $NiCl_2$(dppp) (11mg, 8 mol %) and pivalate **8h** (50 mg) in dry THF (2mL) was added n-BuMgCl (0.2 mL, 2N in THF, 2eq) dropwise at 0 ℃ under N_2. After stirred for 6h at room temperture, the mixture was cooled to 0 ℃. Another n-BuMgCl (0.2mL, 2N in THF, 2eq) was added dropwise. The reaction mixture was stirred overnight at room temperature and then quenched with saturated NH_4Cl. After extracted with ether, dried and concentrated, the residue was purified to give product **11** (30 mg) in 67% yield through flash column chromatography on silica gel (eluents: ethyl acetate: hexanes =1: 20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 5.92 (d, 1H, *J* = 15.6 Hz), 5.54 – 5.61 (m, 1H), 5.21 (t, 1H, *J* = 7.6 Hz), 2.06 – 2.17 (m, 2H), 2.06 – 2.17 (m, 2H), 1.91– 1.97 (m, 2H), 1.27– 1.34 (m, 4H), 1.32 (s, 9H), 1.01 (t, *J* = 7.6 Hz, 3H), 0.87(t, *J* = 7.2 Hz, 3H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.9, 145.7, 130.8, 124.1, 120.0, 39.4, 31.2, 27.6, 25.6, 25.4, 22.7, 14.1, 13.5.; IR (neat): 3423, 3002, 1751, 1128, 1287, 952; Calculated for $[C_{15}H_{26}NaO_2]^+$: 216.2; Found: 216.2.

(1*Z*, 3*E*)-1-Phenylhexa-1,3-dien-2-yl pivalate

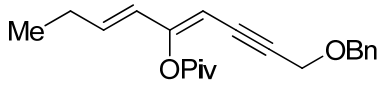


12

An oven-dried Schlenk tube was charged with SPhos (6.7 mg, 8 mol %)), $Pd_2(dba)_3$ (7.5mg, 4mol %)), phenylboronic acid (50 mg, 2eq)) and anhydrous K_3PO_4 (130 mg, 3eq). The mixture was evacuated and flushed with N_2 for 3 times. A solution of pivalate **8h**

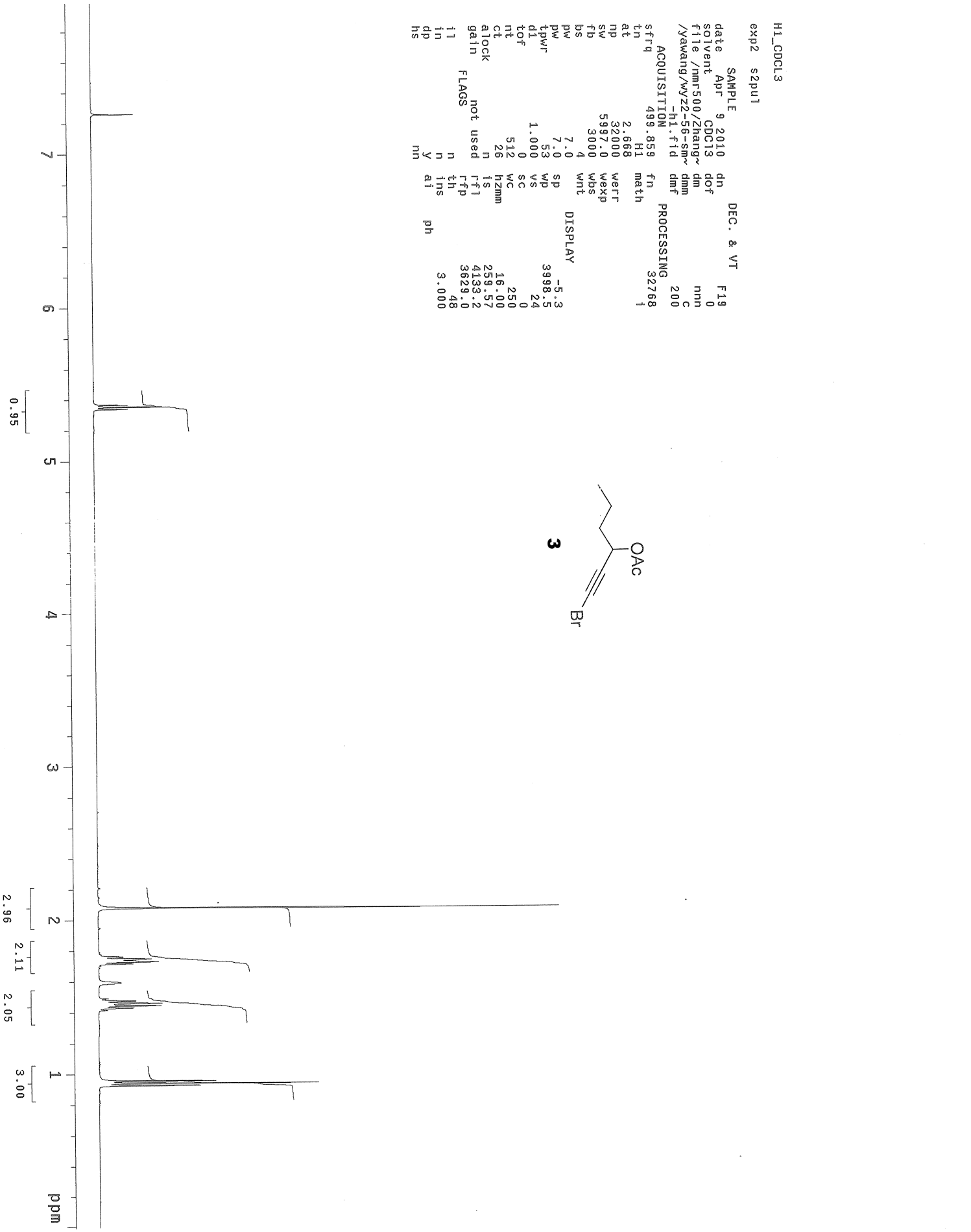
(50 mg) in dry toluene (1.5 mL) was added. The reaction mixture was stirred at 100 ℃ for 8 h and then cooled to room temperature. After filtered through a small pad of celite and removed solvent under *vacuum*, the residue was purified to give product **12** (31mg) in 63% yield through flash column chromatography on silica gel (eluents: ethyl acetate: hexanes =1: 20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.40 (m, 2H), 7.27 – 7.31 (m, 2H), 7.19 – 7.22 (m, 1H), 6.17 (s, 1H), 5.99 (d, 1H, *J* = 15.6 Hz), 5.79 (dt, 1H, *J* = 15.6, 6.4 Hz), 2.14 – 2.20 (m, 2H), 1.33 (s, 9H), 1.05 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.4, 146.2, 134.6, 133.1, 128.8, 128.3,127.4, 124.9, 118.8, 39.4, 27.7, 25.6, 13.4; IR (neat): 3705, 2966, 2874, 1749, 1586, 1107, 956; Calculated for $[C_{17}H_{22}NaO_2]^+$: 281.2; Found: 281.2.

(4*Z*,6*E*)-1-(Benzyloxy)nona-4,6-dien-2-yn-5-yl pivalate



13

To a mixture of $PdCl_2(Ph_3P)_2$ (10 mg, 7.5 mol %), CuI (9.7 mg, 25 mol%) and Et_2NH (0.2mL) was added a solution of pivalate **8h** (50 mg) and (prop-2-ynyloxy) methylbenzene (1.5eq) in dry THF (2mL) under N_2. The reaction mixture was stirred at 60 ℃ for 18 h and then cooled to room temperature. After filtered through a small pad of Celite™ and removed solvent under *vacuum*, the residue was purified to give product **13** (47mg) in 76% yield through flash column chromatography on silica gel (eluents: ethyl acetate: hexanes =1: 20). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 –7.35 (m, 4H), 7.27 – 7.29 (m, 1H), 6.02 (d, 1H, *J* = 15.6 Hz), 5.81 (dt, 1H, *J* = 15.6, 6.4 Hz), 5.35 (s, 1H), 4.57 (s, 2H), 4.29 (d, 2H, *J* = 2.4Hz), 2.13 – 2.20 (m, 2H), 1.35(s, 9H), 1.01(t, *J* = 7.6 Hz, 3H) ; ^{13}C NMR (125 MHz, $CDCl_3$) δ 175.1, 155.8, 137.6, 136.3, 128.5, 128.2, 128.0, 123.1, 98.4, 92.7, 81.3, 71.6, 58.1, 39.5, 27.5, 25.6, 13.1; IR (neat): 3419, 2971, 2213, 1754, 1642, 1352, 1113, 736; Calculated for $[C_{21}H_{26}NaO_3]^+$: 349.2; Found: 349.2.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date Apr 9 2010
solvent CDCl3
file /nmr500/Zhang~
/yawang/wyz2-56-sm~
-h1.fid
ACQUISITION
sfrq 499.859
tn H1
at 2.668
np 32000
sw 5997.0
fb 3000
bs 4
pw 7.0
pw 7.0
tpwr 53
d1 1.000
tof 0
nt 512
ct 26
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DEC. & VT
dn F19
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
fn 32768
math i
DISPLAY
sp -5.3
wp 3998.5
vs 24
sc 0
wc 250
hzmm 16.00
is 259.57
rfl 4133.2
rfp 3629.0
th 48
ins 3.000
ai ph
OAc
Br
3
0.95
2.96
2.11
2.05
3.00
7
6
5
4
3
2
1
ppm

STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
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solvent	cdcl3	gain	54
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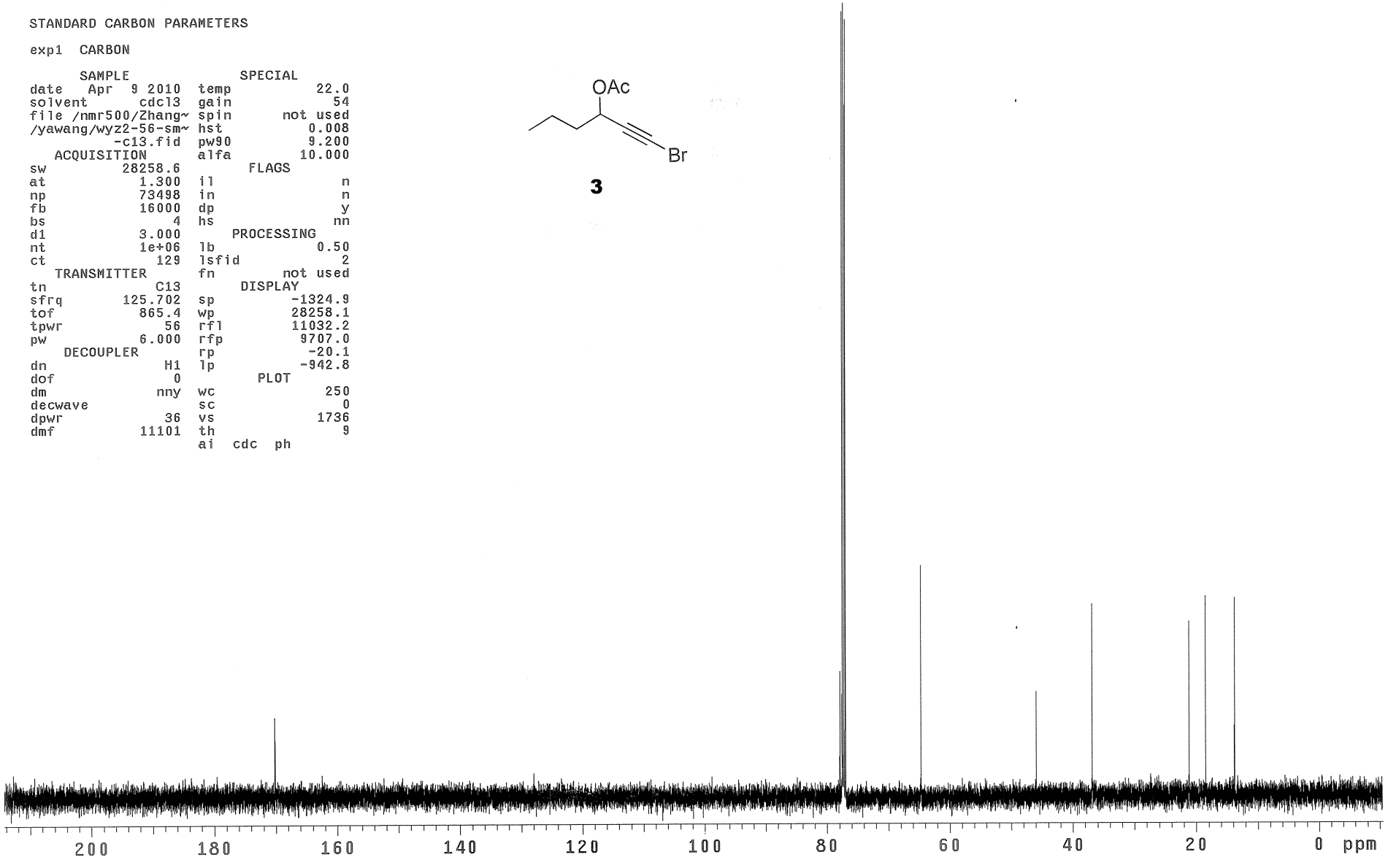
	-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	129	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-1324.9
tof	865.4	wp	28258.1
tpwr	56	rfl	11032.2
pw	6.000	rfp	9707.0
DECOUPLER		rp	-20.1
dn	H1	lp	-942.8
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1736
dmf	11101	th	9
		ai cdc ph	

3



H1_CDCL3

exp2 s2pul

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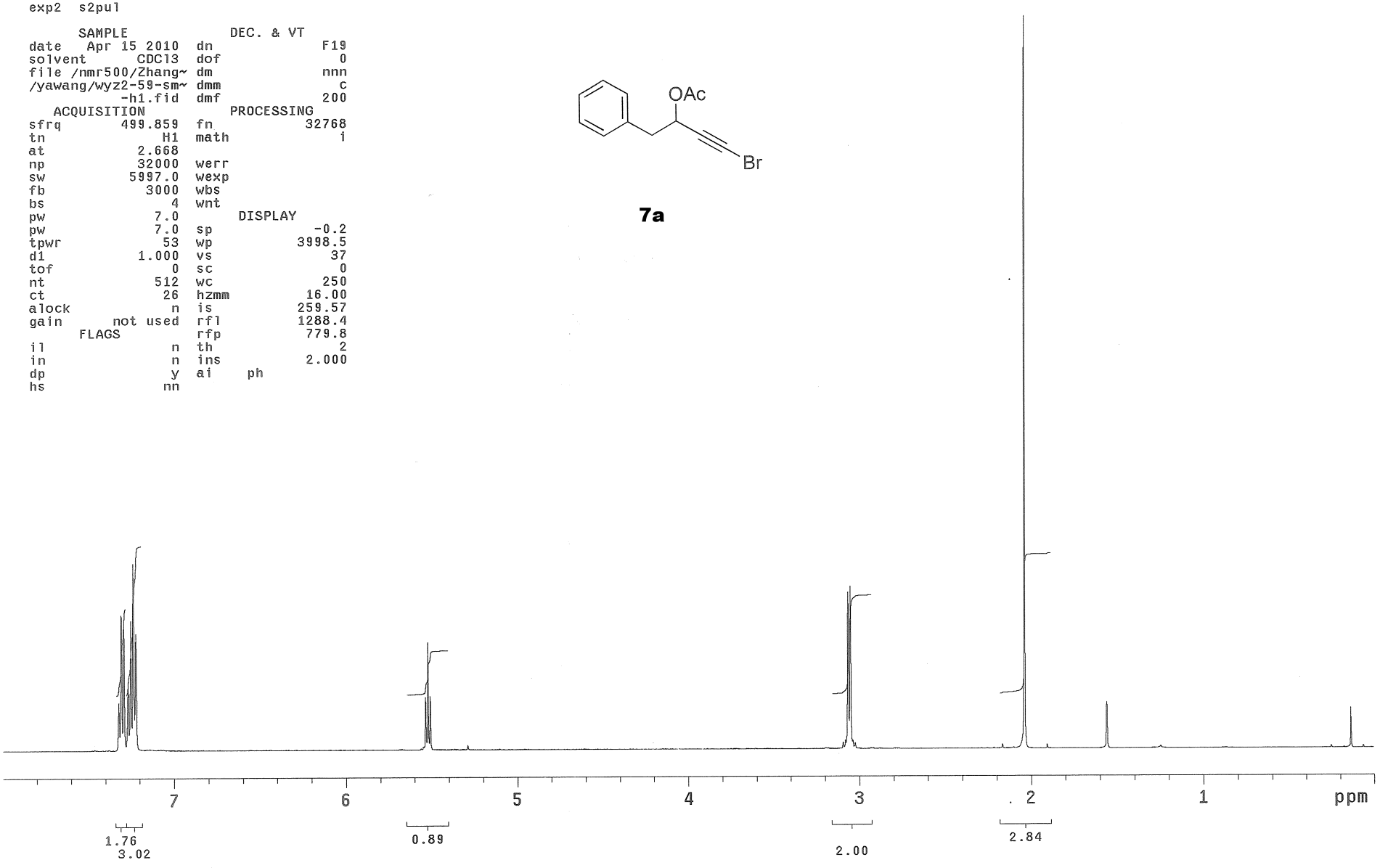
	/yawang/wyz2-59-sm~	dmm	c
	-h1.fid	dmf	200
ACQUISITION		PROCESSING	
sfrq	499.859	fn	32768
tn	H1	math	i
at	2.668		
np	32000	werr	
sw	5997.0	wexp	
fb	3000	wbs	
bs	4	wnt	
pw	7.0	DISPLAY	
pw	7.0	sp	-0.2
tpwr	53	wp	3998.5
d1	1.000	vs	37
tof	0	sc	0
nt	512	wc	250
ct	26	hzmm	16.00
alock	n	is	259.57
gain	not used	rfl	1288.4
FLAGS		rfp	779.8
il	n	th	2
in	n	ins	2.000
dp	y	ai ph	
hs	nn		

7a



STANDARD CARBON PARAMETERS

exp1 CARBON

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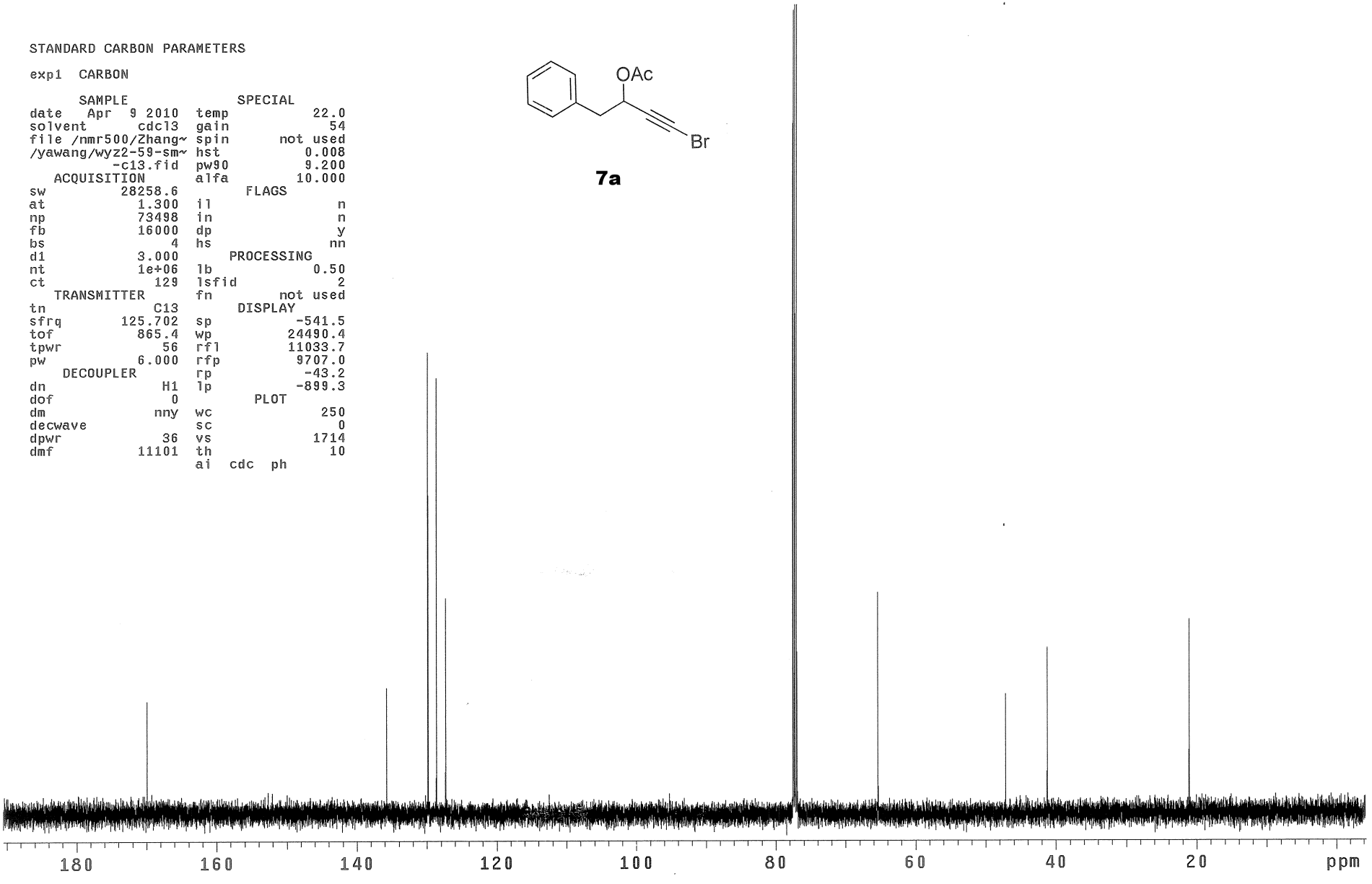
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	129	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-541.5
tof	865.4	wp	24490.4
tpwr	56	rfl	11033.7
pw	6.000	rfp	9707.0
DECOUPLER		rp	-43.2
dn	H1	lp	-899.3
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1714
dmf	11101	th	10
		ai cdc ph	

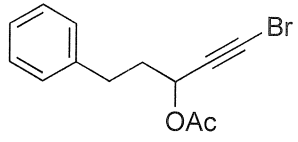
7a



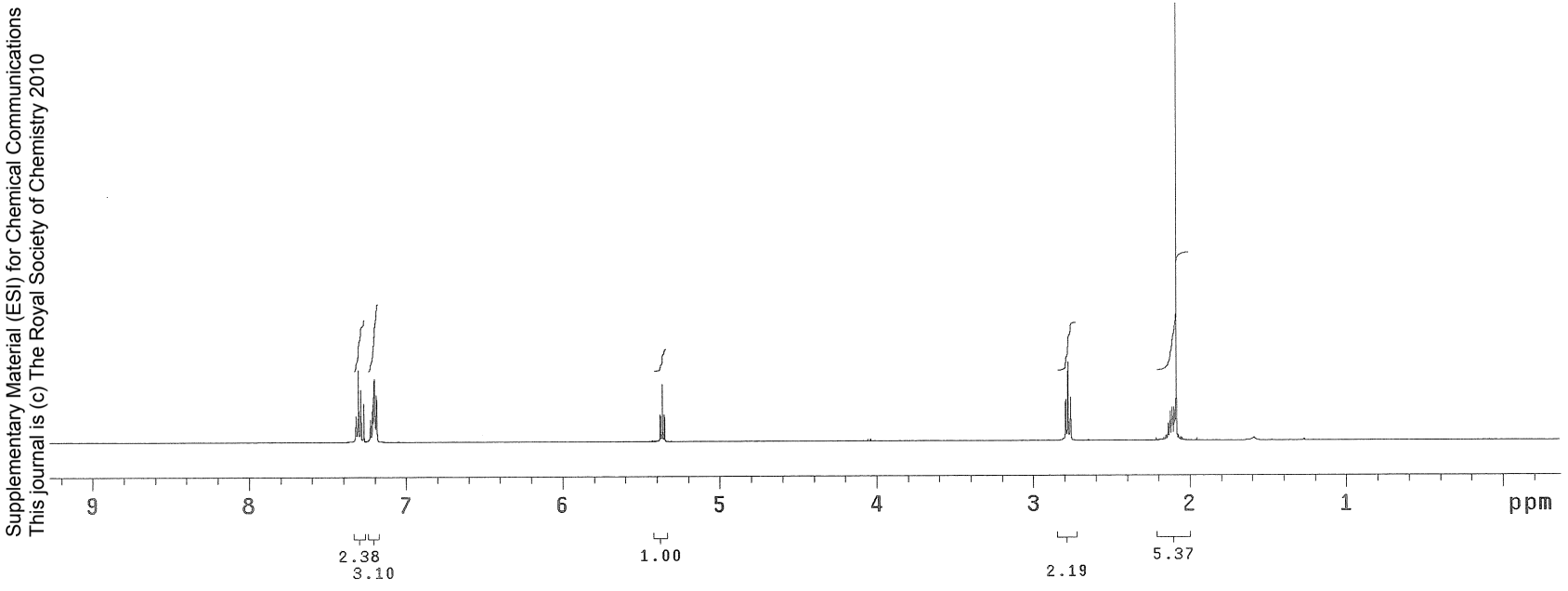
lb-I-136-2

exp1 PROTON

SAMPLE		PRESATURATION	
date	Mar 16 2010	satmode	n
solvent	CDCl3	wet	n
file	exp	SPECIAL	
ACQUISITION		temp	22.0
sw	7997.6	gain	not used
at	2.049	spin	20
np	32768	hst	0.008
fb	4000	pw90	9.100
bs	2	alfa	6.600
d1	1.000	FLAGS	
nt	8	il	n
ct	8	in	n
TRANSMITTER		dp	y
tn	H1	hs	nn
sfrq	499.859	PROCESSING	
tof	499.9	fn	not used
tpwr	53	DISPLAY	
pw	4.550	sp	-184.0
DECOUPLER		wp	4819.8
dn	C13	rfl	999.7
dof	0	rfp	0
dm	nnn	rp	-25.3
decwave	W40_5mm3Gi~dp	lp	3.0
		PLOT	
dpwr	47	wc	215
dmf	32258	sc	35
		vs	62
		th	2
		nm	cdc ph



7b

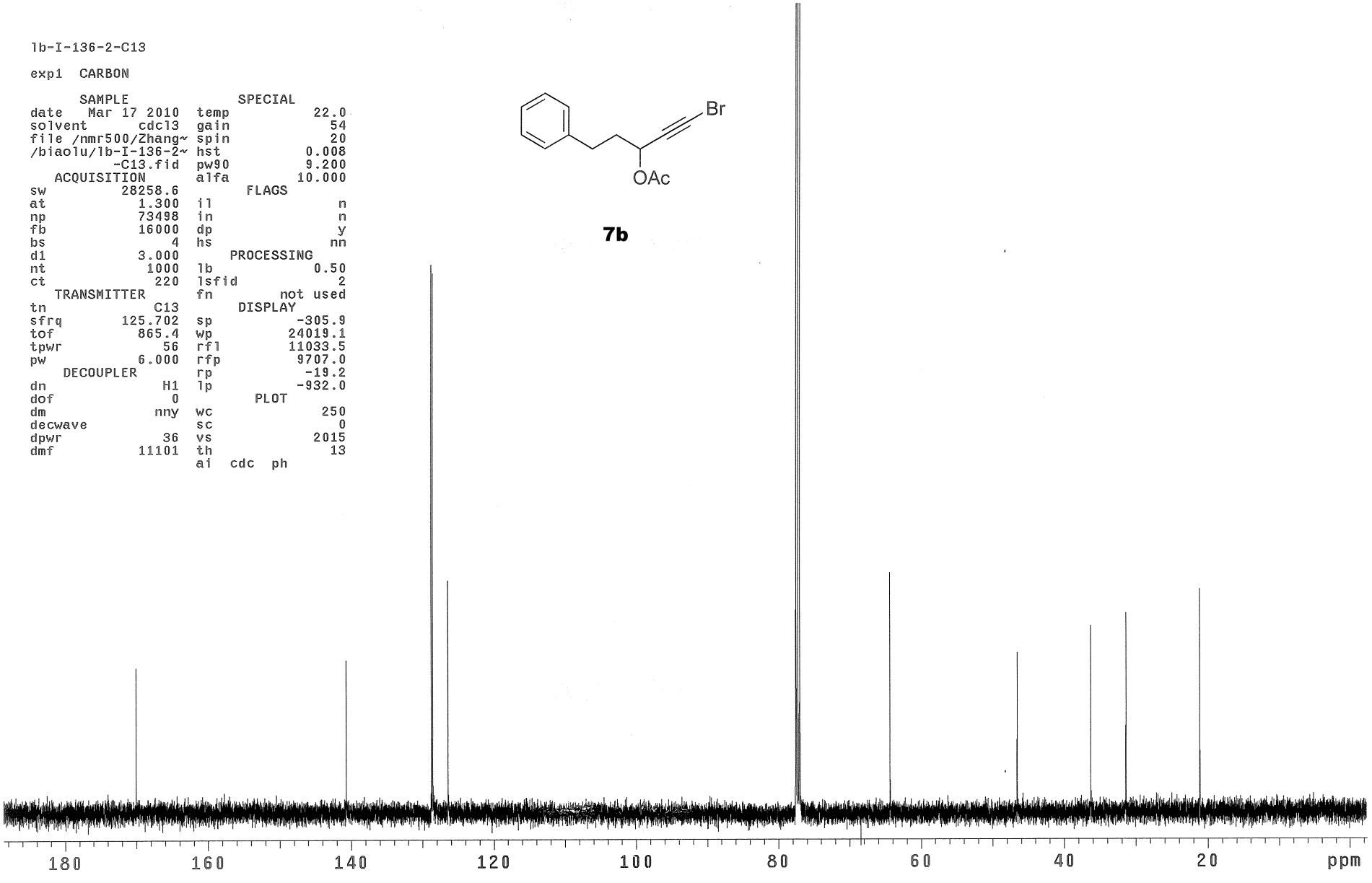


lb-I-136-2-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Mar 17 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	20
	/biaolu/lb-I-136-2~	hst	0.008
	-C13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1000	lb	0.50
ct	220	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-305.9
tof	865.4	wp	24019.1
tpwr	56	rfl	11033.5
pw	6.000	rfp	9707.0
DECOUPLER		rp	-19.2
dn	H1	lp	-932.0
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	2015
dmf	11101	th	13
		ai cdc ph	

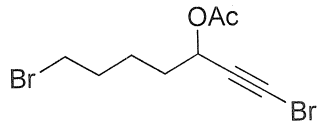
7b



STANDARD PROTON PARAMETERS

exp1 PROTON

SAMPLE		PRESATURATION	
date	Mar 14 2010	satmode	n
solvent	CDCl3	wet	n
file	/nmr500/Zhang~	SPECIAL	
/biaolu/lb-I-130-2~	.fid	temp	22.0
ACQUISITION		gain	not used
sw	7997.6	spin	20
at	2.049	hst	0.008
np	32768	pw90	9.100
fb	4000	alfa	6.600
bs	2	FLAGS	
d1	1.000	il	n
nt	8	in	n
ct	8	dp	y
TRANSMITTER		hs	nn
tn	H1	PROCESSING	
sfrq	499.859	fn	not used
tof	499.9	DISPLAY	
tpwr	53	sp	-254.7
pw	4.550	wp	4760.8
DECOUPLER		rfl	4631.9
dn	C13	rfp	3629.0
dof	0	rp	-38.2
dm	nnn	lp	-2.4
decwave	W40_5mm3Gi~	PLOT	
	dp	wc	250
dpwr	47	sc	0
dmf	32258	vs	13
		th	4
		ai ph	



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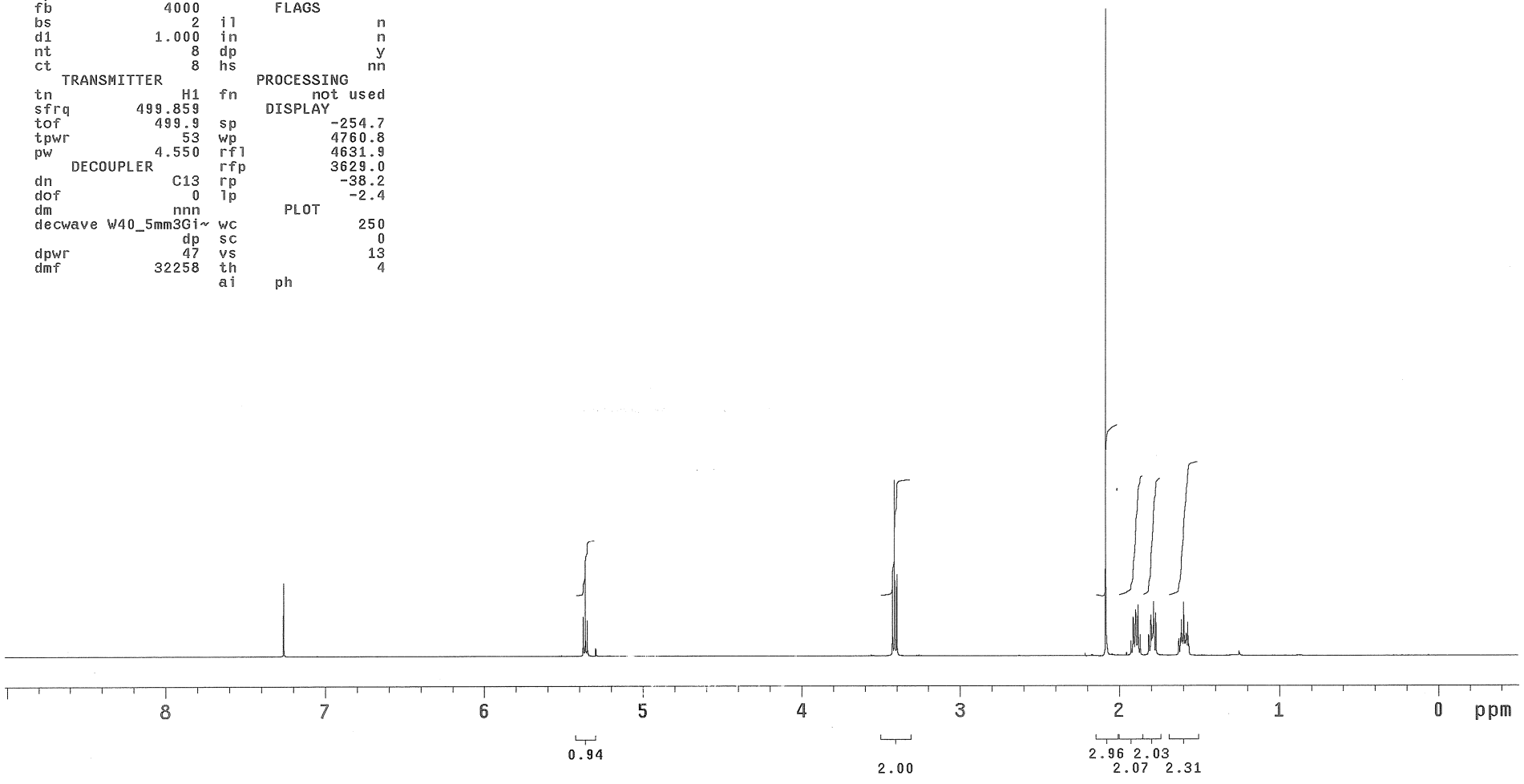
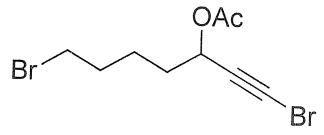


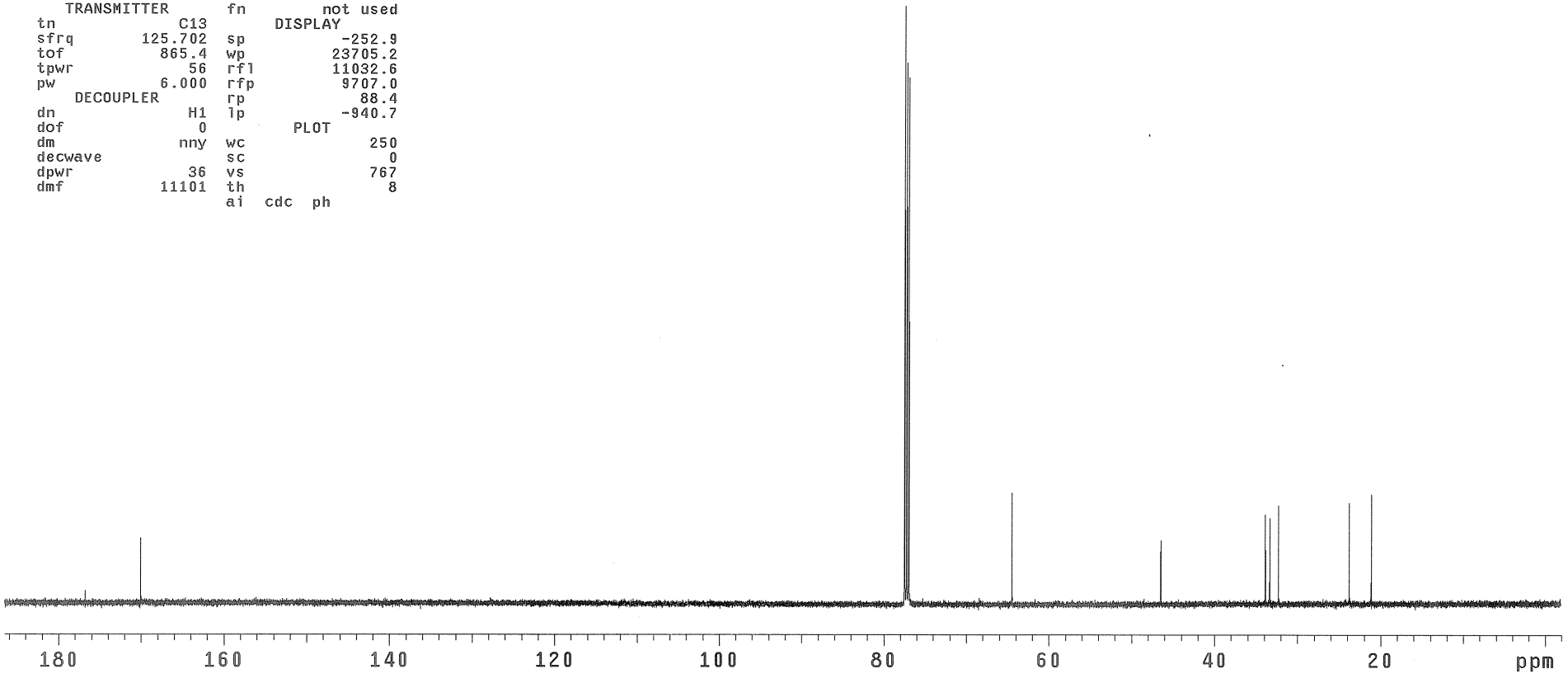
lb-I-130-2-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Mar 15 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	20
/biaolu/lb-I-130-2~		hst	0.008
	-C13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	10	hs	nn
d1	3.000	PROCESSING	
nt	1000	lb	0.50
ct	630	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-252.9
tof	865.4	wp	23705.2
tpwr	56	rfl	11032.6
pw	6.000	rfp	9707.0
DECOUPLER		rp	88.4
dn	H1	lp	-940.7
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	767
dmf	11101	th	8
		ai cdc ph	



7c

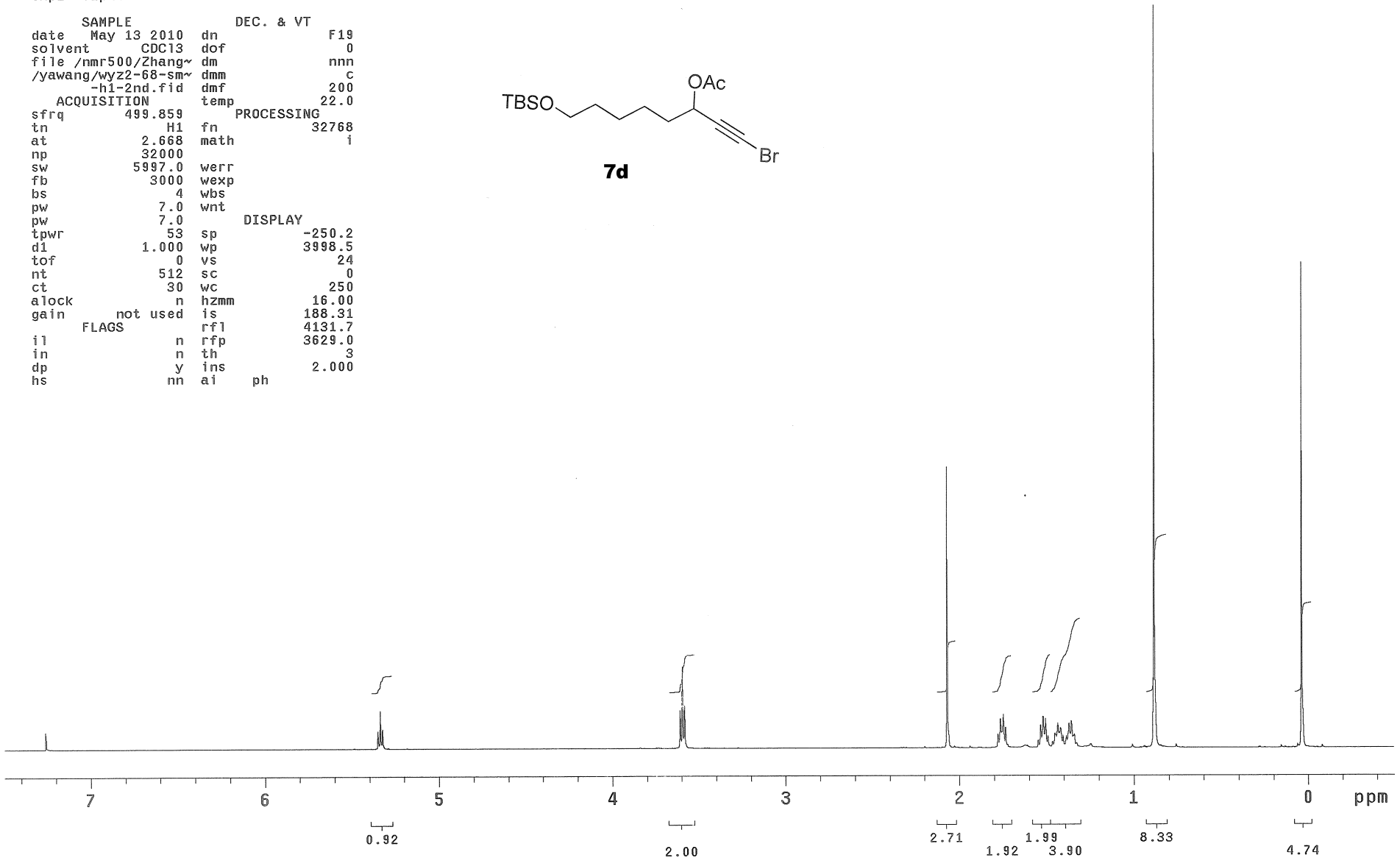


H1_CDCL3

exp2 s2pul

SAMPLE		DEC. & VT	
date	May 13 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-68-sm~	dmm	c
	-h1-2nd.fid	dmf	200
ACQUISITION		temp	22.0
sfrq	499.859	PROCESSING	
tn	H1	fn	32768
at	2.668	math	i
np	32000		
sw	5997.0	werr	
fb	3000	wexp	
bs	4	wbs	
pw	7.0	wnt	
pw	7.0	DISPLAY	
tpwr	53	sp	-250.2
d1	1.000	wp	3998.5
tof	0	vs	24
nt	512	sc	0
ct	30	wc	250
alock	n	hzmm	16.00
gain	not used	is	188.31
FLAGS		rfl	4131.7
il	n	rfp	3629.0
in	n	th	3
dp	y	ins	2.000
hs	nn	ai ph	

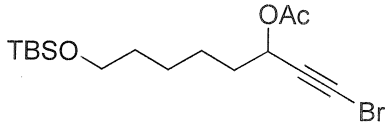
7d



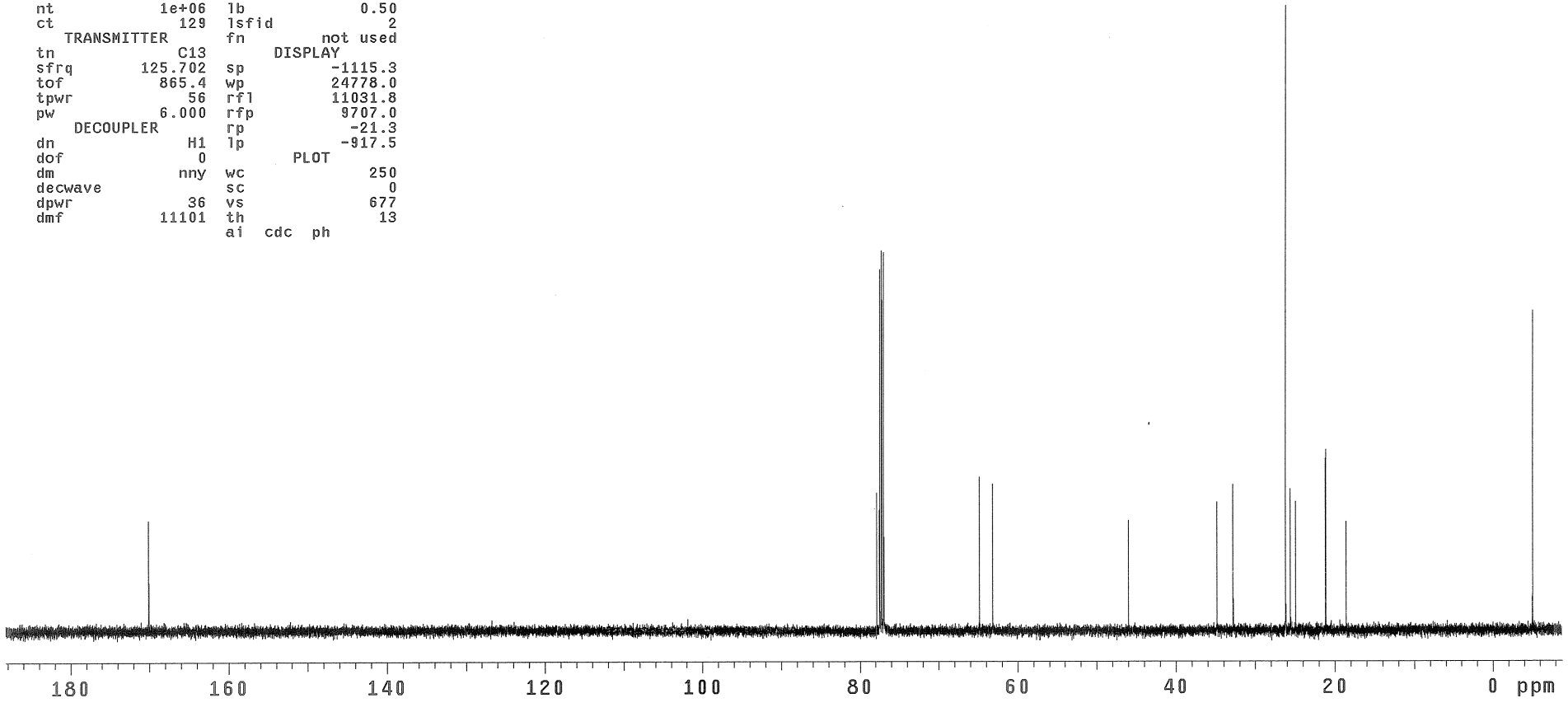
STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	May 13 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
	/yawang/wyz2-68-sm~	hst	0.008
	-c13-2nd.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	129	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-1115.3
tof	865.4	wp	24778.0
tpwr	56	rfl	11031.8
pw	6.000	rfp	9707.0
DECOUPLER		rp	-21.3
dn	H1	lp	-917.5
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	677
dmf	11101	th	13
		ai cdc ph	



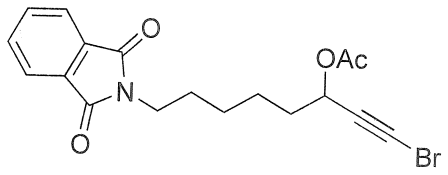
7d



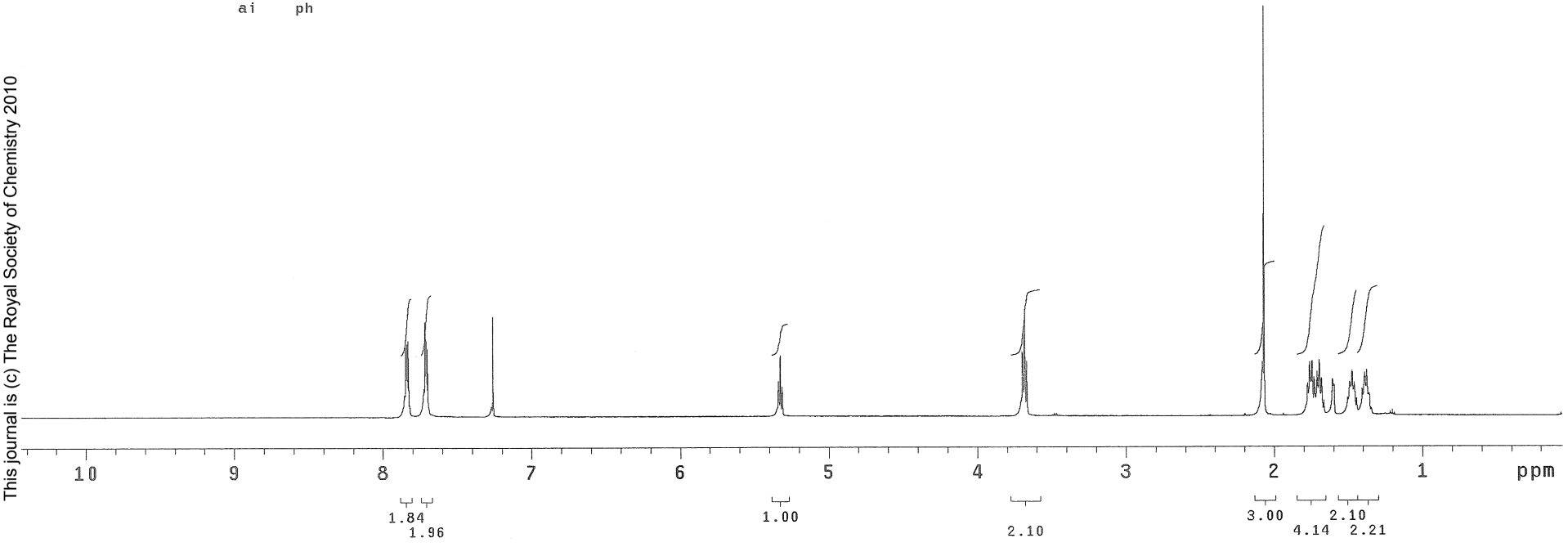
wyz-2-102-sm

exp1 s2pul

SAMPLE		DEC. & VT	
date	Aug 14 2010	dn	F19
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	499.859	dmf	200
tn	H1	PROCESSING	
at	2.699	lb	0.20
np	43200	fn	32768
sw	8003.2	math	i
fb	4000		
bs	2	werr	
pw	9.0	wexp	
pw	9.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	0	sp	28.4
nt	16	wp	5188.6
ct	16	vs	31
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	20.75
il	n	is	259.57
in	n	rfl	5133.8
dp	y	rfp	3629.0
hs	nn	th	3
		ins	3.000
		ai ph	



7e

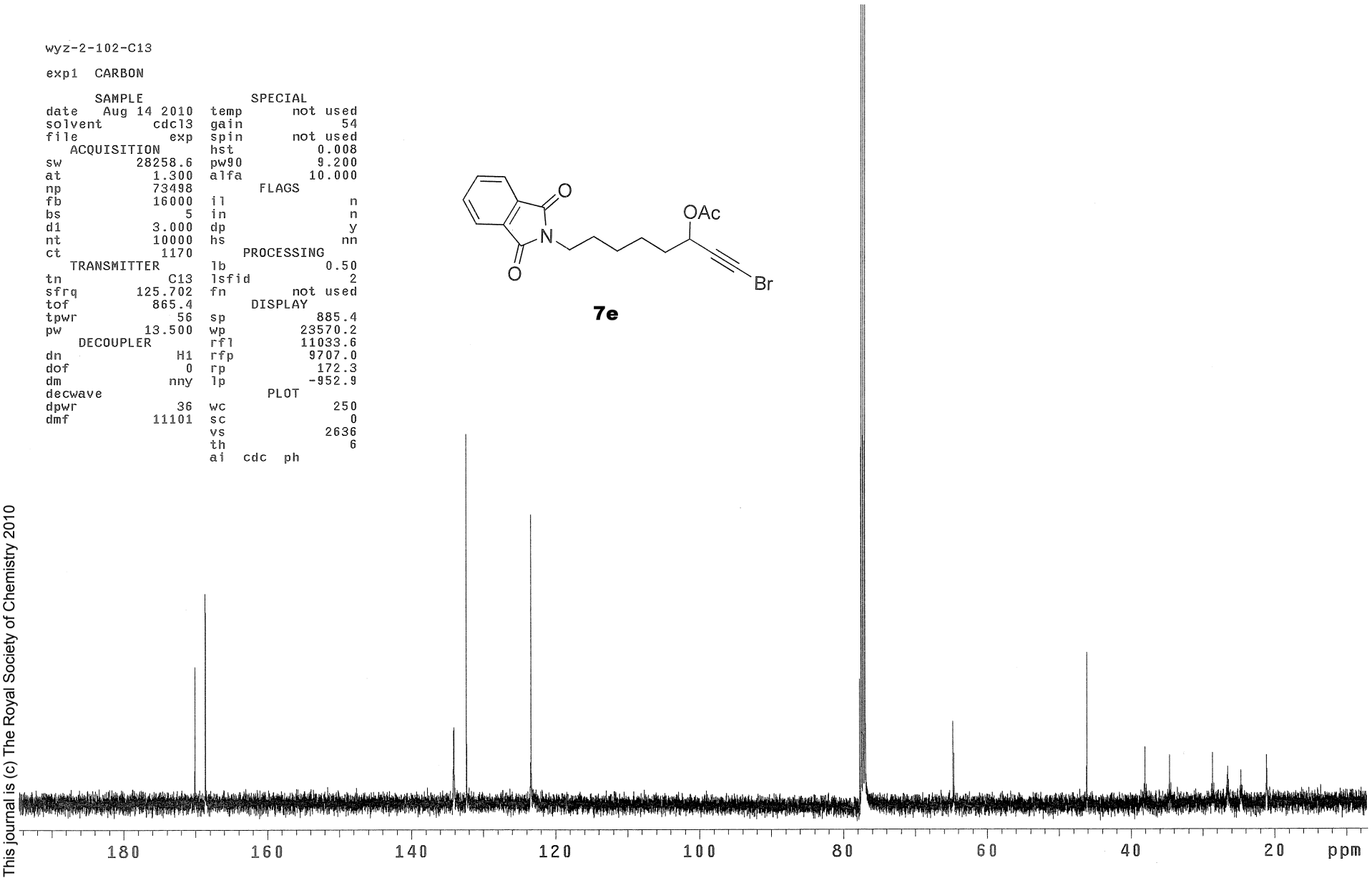


wyz-2-102-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Aug 14 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	5	in	n
d1	3.000	dp	y
nt	10000	hs	nn
ct	1170	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	885.4
pw	13.500	wp	23570.2
DECOUPLER		rfl	11033.6
dn	H1	rfp	9707.0
dof	0	rp	172.3
dm	nny	lp	-952.9
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	2636
		th	6
		ai cdc ph	

7e

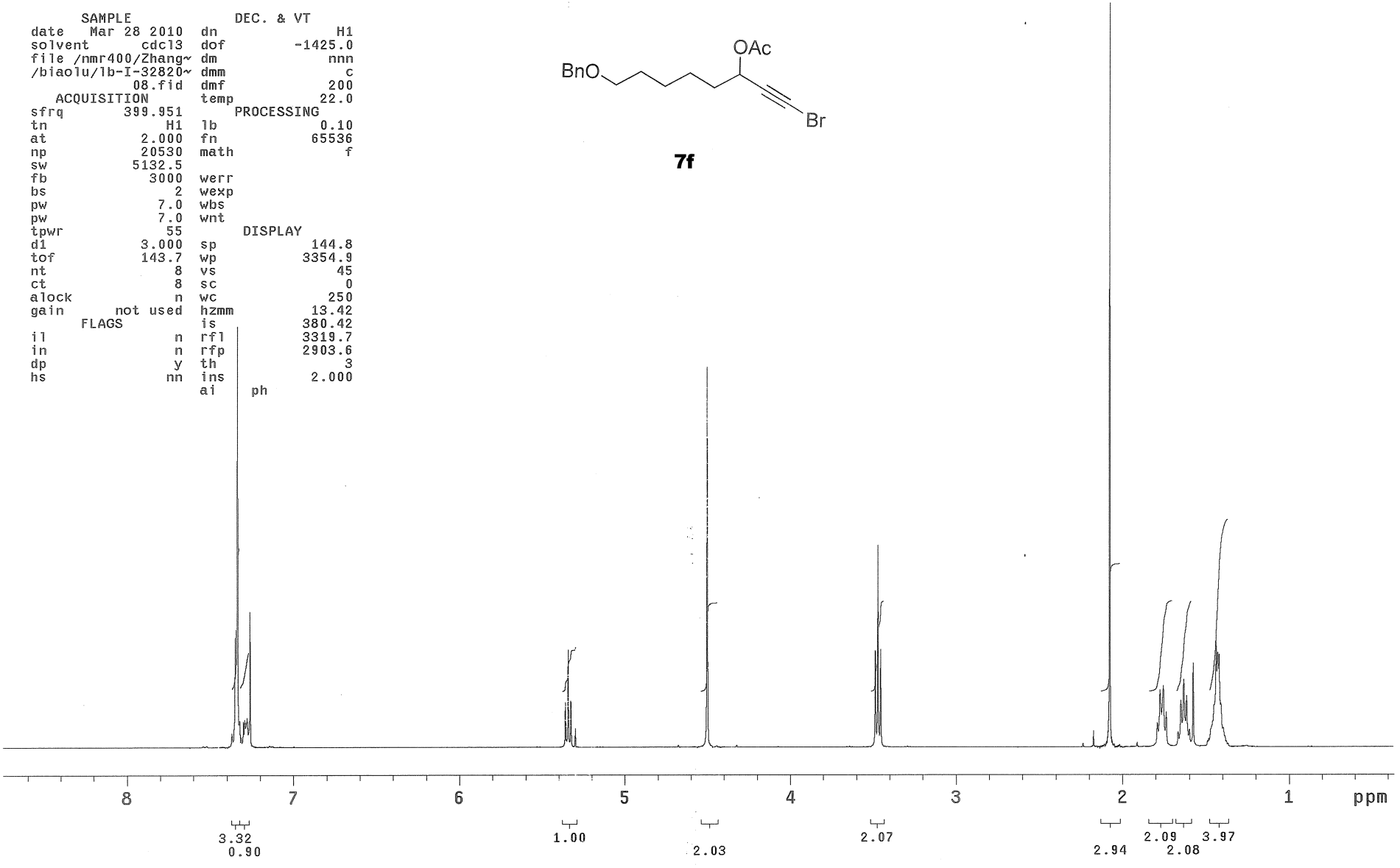


lb-I-3282008

exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 28 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	/nmr400/Zhang~	dm	nnn
	/biaolu/lb-I-32820~	dmm	c
	08.fid	dmf	200
ACQUISITION		temp	22.0
sfrq	399.951	PROCESSING	
tn	H1	lb	0.10
at	2.000	fn	65536
np	20530	math	f
sw	5132.5		
fb	3000	werr	
bs	2	wexp	
pw	7.0	wbs	
pw	7.0	wnt	
tpwr	55	DISPLAY	
d1	3.000	sp	144.8
tof	143.7	wp	3354.9
nt	8	vs	45
ct	8	sc	0
alock	n	wc	250
gain	not used	hzmm	13.42
FLAGS		is	380.42
il	n	rfl	3319.7
in	n	rfp	2903.6
dp	y	th	3
hs	nn	ins	2.000
		ai ph	

7f

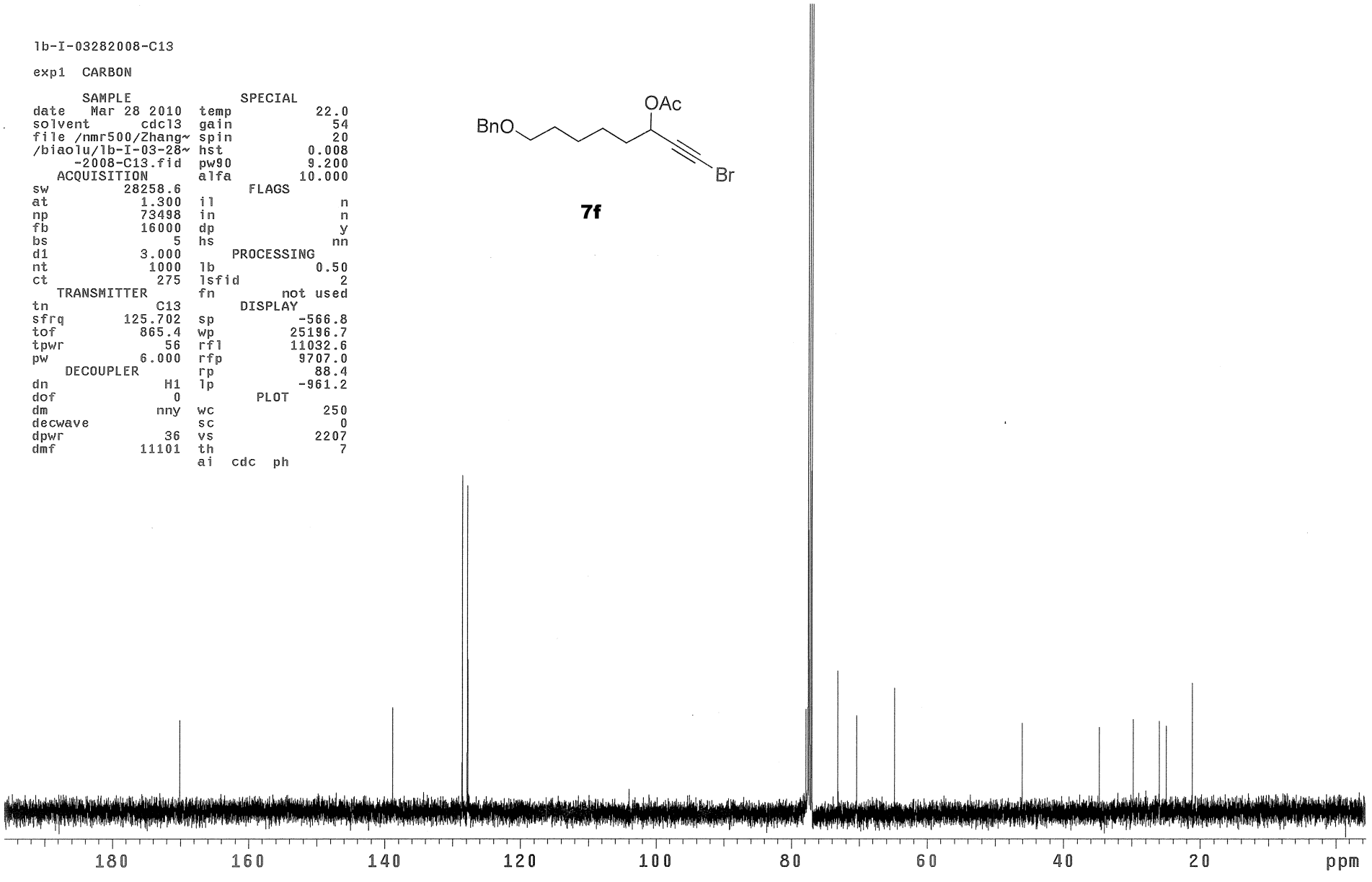


lb-I-03282008-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Mar 28 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	20
/biaolu/lb-I-03-28~		hst	0.008
-2008-C13.fid		pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	5	hs	nn
d1	3.000	PROCESSING	
nt	1000	lb	0.50
ct	275	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-566.8
tof	865.4	wp	25196.7
tpwr	56	rfl	11032.6
pw	6.000	rfp	9707.0
DECOUPLER		rp	88.4
dn	H1	lp	-961.2
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	2207
dmf	11101	th	7
		ai cdc ph	

7f

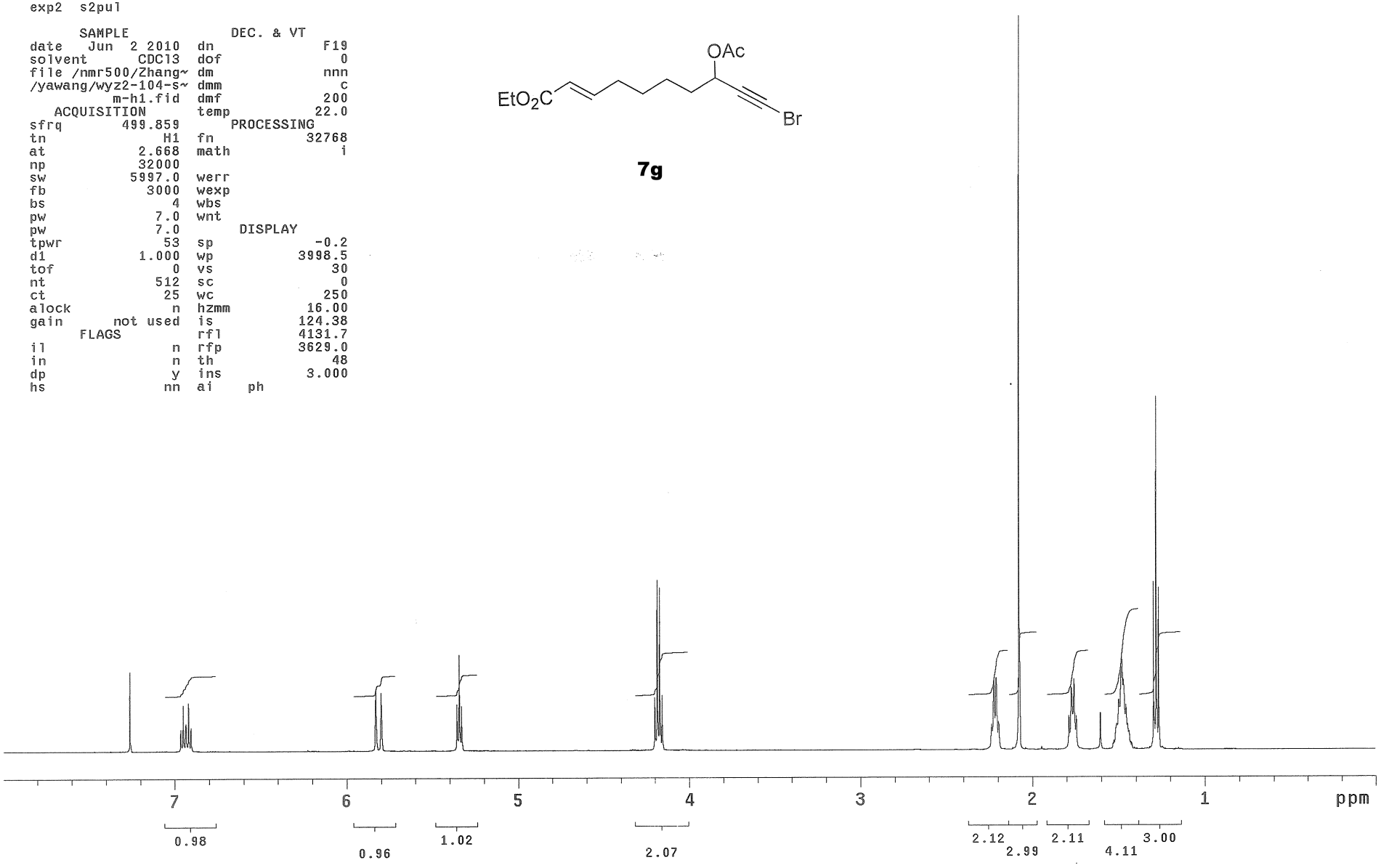


H1_CDCL3

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 2 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-104-s~	dmm	c
	m-h1.fid	dmf	200
ACQUISITION		temp	22.0
sfrq	499.859	PROCESSING	
tn	H1	fn	32768
at	2.668	math	i
np	32000		
sw	5997.0	werr	
fb	3000	wexp	
bs	4	wbs	
pw	7.0	wnt	
pw	7.0	DISPLAY	
tpwr	53	sp	-0.2
d1	1.000	wp	3998.5
tof	0	vs	30
nt	512	sc	0
ct	25	wc	250
alock	n	hzmm	16.00
gain	not used	is	124.38
FLAGS		rfl	4131.7
il	n	rfp	3629.0
in	n	th	48
dp	y	ins	3.000
hs	nn	ai ph	

7g

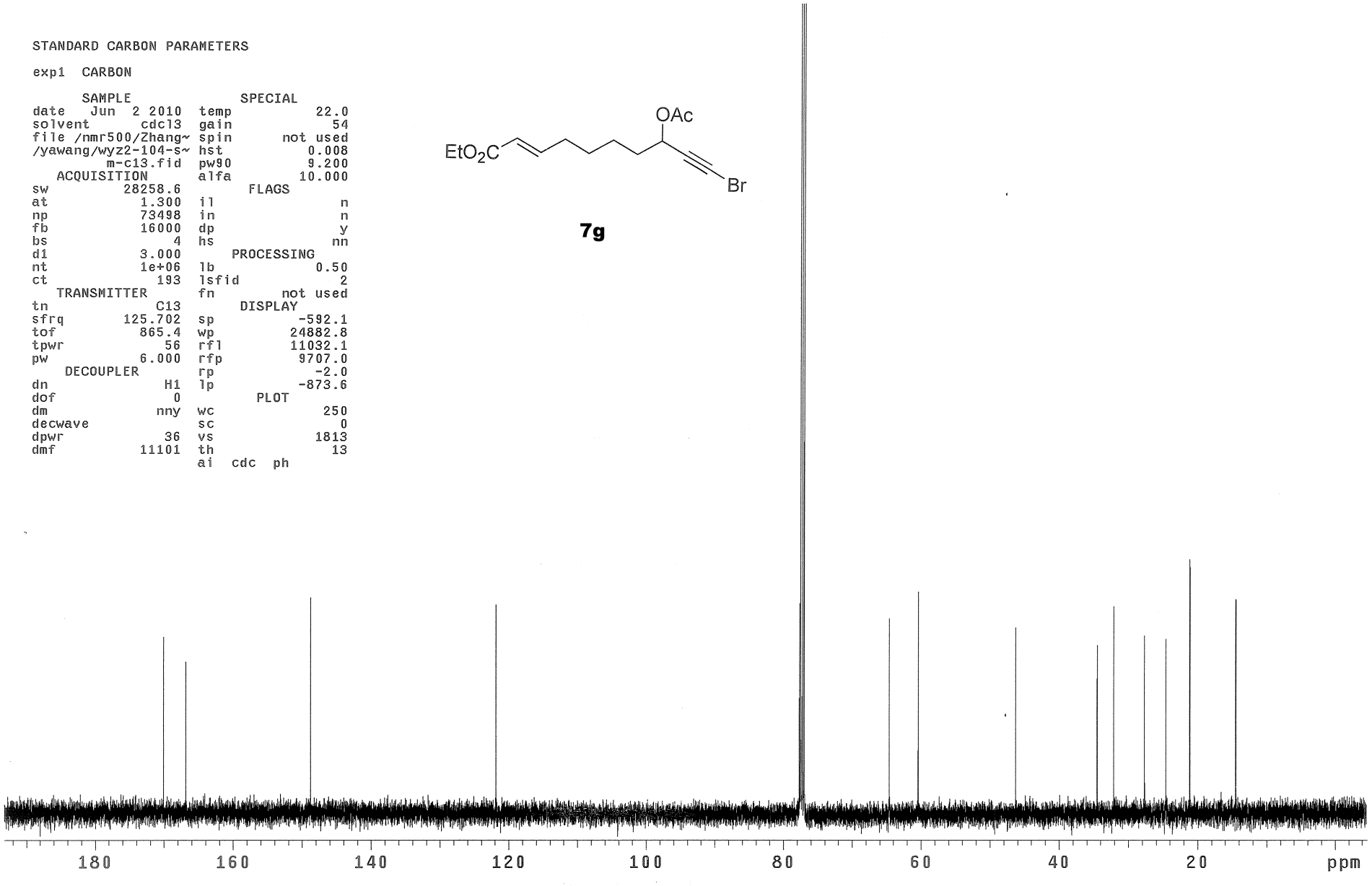


STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Jun 2 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
	/yawang/wyz2-104-s~	hst	0.008
	m-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	193	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-592.1
tof	865.4	wp	24882.8
tpwr	56	rfl	11032.1
pw	6.000	rfp	9707.0
DECOUPLER		rp	-2.0
dn	H1	lp	-873.6
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1813
dmf	11101	th	13
		ai cdc ph	

7g

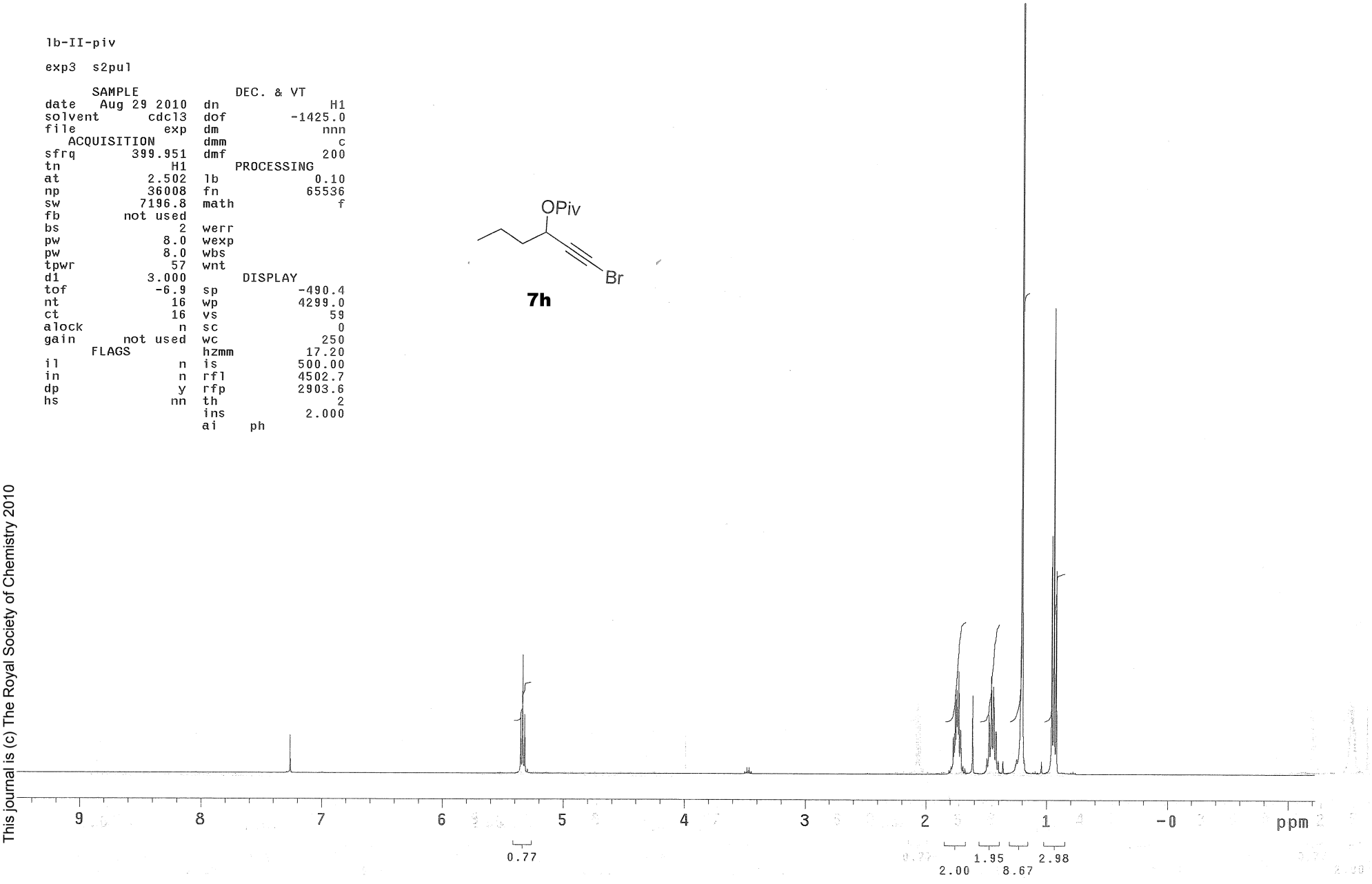


lb-II-piv

exp3 s2pul

SAMPLE		DEC. & VT	
date	Aug 29 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	PROCESSING	
at	2.502	lb	0.10
np	36008	fn	65536
sw	7196.8	math	f
fb	not used		
bs	2	werr	
pw	8.0	wexp	
pw	8.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	-6.9	sp	-490.4
nt	16	wp	4299.0
ct	16	vs	59
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	17.20
il	n	is	500.00
in	n	rfl	4502.7
dp	y	rfp	2903.6
hs	nn	th	2
		ins	2.000
		ai ph	

7h

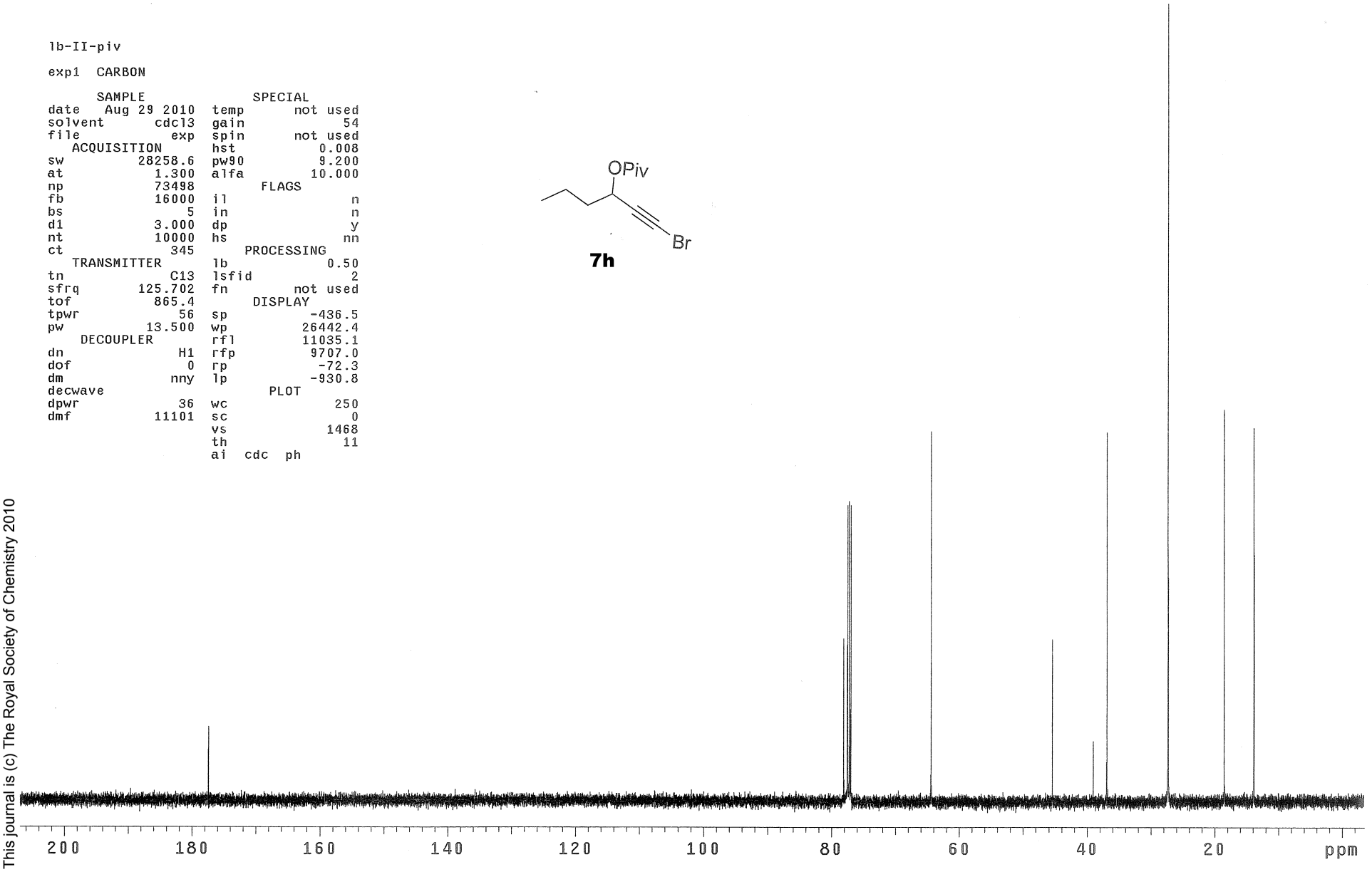


lb-II-piv

exp1 CARBON

SAMPLE		SPECIAL	
date	Aug 29 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	5	in	n
d1	3.000	dp	y
nt	10000	hs	nn
ct	345	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	-436.5
pw	13.500	wp	26442.4
DECOUPLER		rfl	11035.1
dn	H1	rfp	9707.0
dof	0	rp	-72.3
dm	nny	lp	-930.8
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	1468
		th	11
		ai cdc ph	

7h

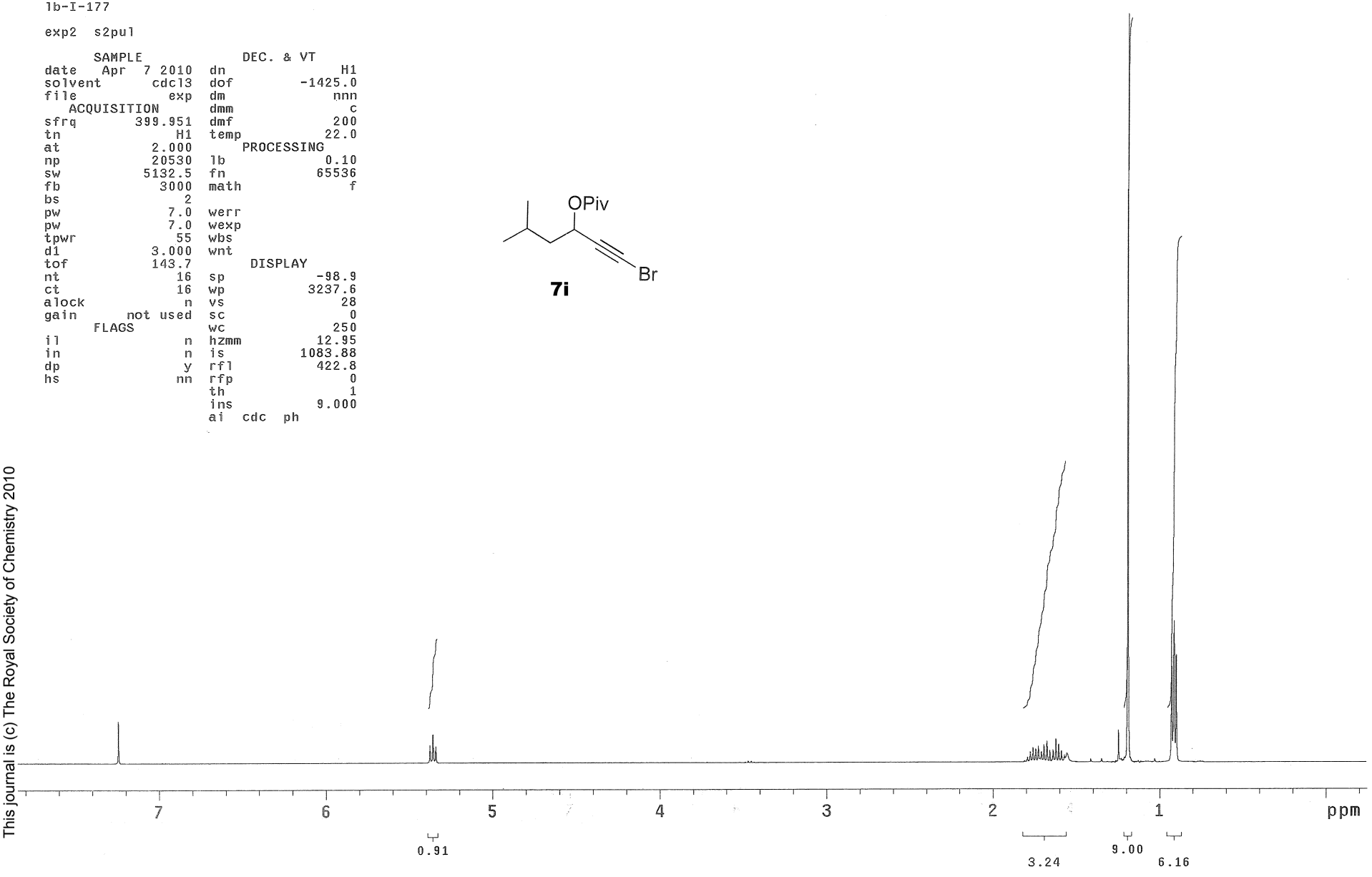


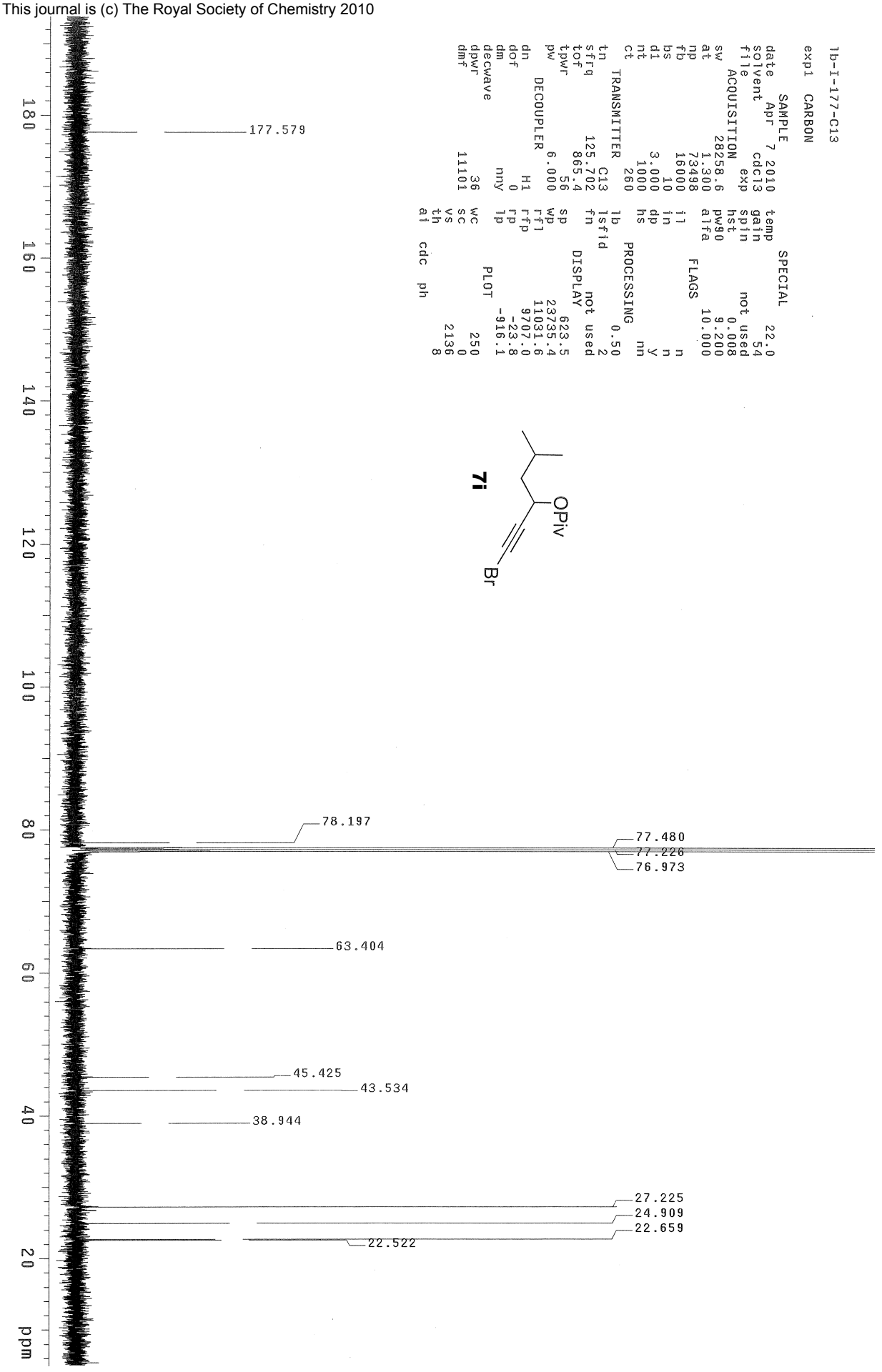
lb-I-177

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 7 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	temp	22.0
at	2.000	PROCESSING	
np	20530	lb	0.10
sw	5132.5	fn	65536
fb	3000	math	f
bs	2		
pw	7.0	werr	
pw	7.0	wexp	
tpwr	55	wbs	
d1	3.000	wnt	
tof	143.7	DISPLAY	
nt	16	sp	-98.9
ct	16	wp	3237.6
alock	n	vs	28
gain	not used	sc	0
FLAGS		wc	250
il	n	hzmm	12.95
in	n	is	1083.88
dp	y	rfl	422.8
hs	nn	rfp	0
		th	1
		ins	9.000
		ai cdc ph	

7i



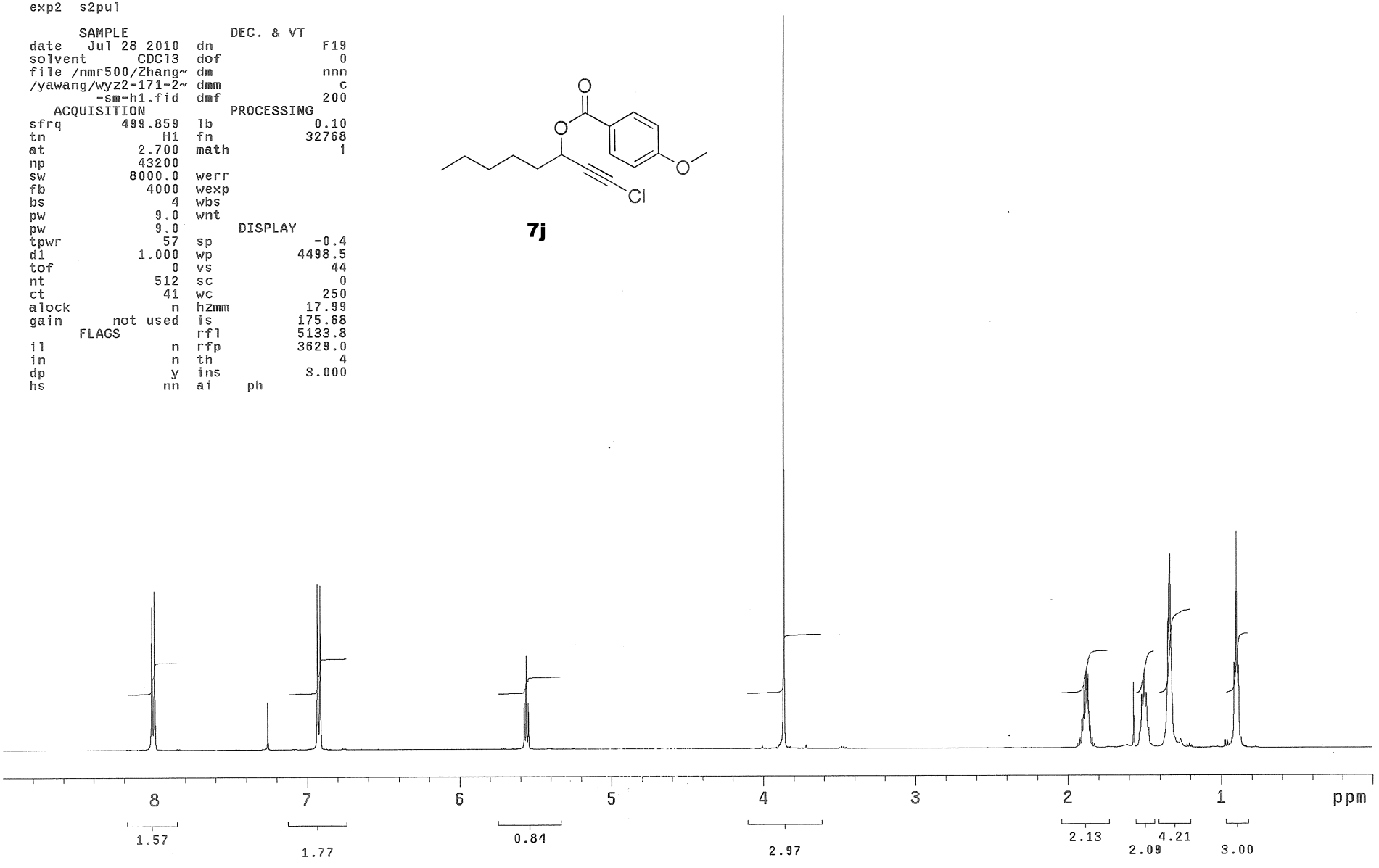

1b-I-177-C13
exp1 CARBON
SAMPLE
date Apr 7 2010
solvent cdcl3
file exp
ACQUISITION
sw 28258.6
at 1.300
np 73498
fb 16000
bs 10
d1 3.000
nt 1000
ct 260
TRANSMITTER
tn C13
sfrq 125.702
tof 865.4
tpwr 56
pw 6.000
DECOUPLER
dn H1
dof 0
dm nny
decwave
dpwr 36
dmf 11101
temp 22.0
gain 54
spin not used
hst 0.008
pw90 9.200
alfa 10.000
FLAGS
n
n
y
nn
PROCESSING
lb 0.50
lsfid 2
fn not used
DISPLAY
sp 623.5
wp 23735.4
rfl 11031.6
rfp 9707.0
rp -23.8
lp -916.1
PLOT
250
0
2136
8
ai cdc ph
7i
OPiv
Br
177.579
78.197
77.480
77.226
76.973
63.404
45.425
43.534
38.944
27.225
24.909
22.659
22.522
180
160
140
120
100
80
60
40
20
ppm

new experiment

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jul 28 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-171-2~	dmm	c
	-sm-h1.fid	dmf	200
ACQUISITION		PROCESSING	
sfrq	499.859	lb	0.10
tn	H1	fn	32768
at	2.700	math	i
np	43200		
sw	8000.0	werr	
fb	4000	wexp	
bs	4	wbs	
pw	9.0	wnt	
pw	9.0	DISPLAY	
tpwr	57	sp	-0.4
d1	1.000	wp	4498.5
tof	0	vs	44
nt	512	sc	0
ct	41	wc	250
alock	n	hzmm	17.99
gain	not used	is	175.68
FLAGS		rfl	5133.8
il	n	rfp	3629.0
in	n	th	4
dp	y	ins	3.000
hs	nn	ai ph	

7j

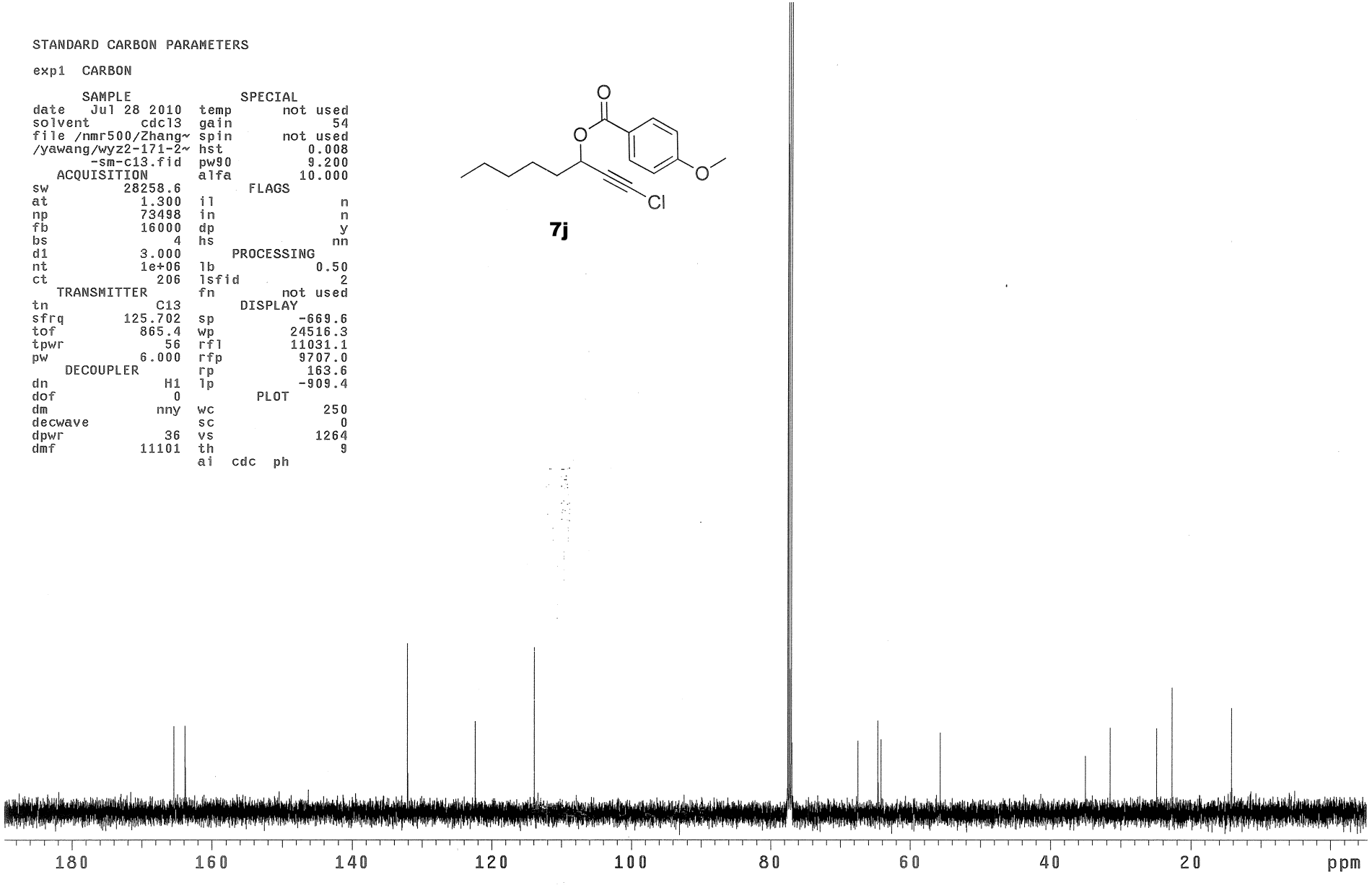


STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Jul 28 2010	temp	not used
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
/yawang/wyz2-171-2~		hst	0.008
	-sm-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	206	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-669.6
tof	865.4	wp	24516.3
tpwr	56	rfl	11031.1
pw	6.000	rfp	9707.0
DECOUPLER		rp	163.6
dn	H1	lp	-909.4
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1264
dmf	11101	th	9
		ai cdc ph	

7j

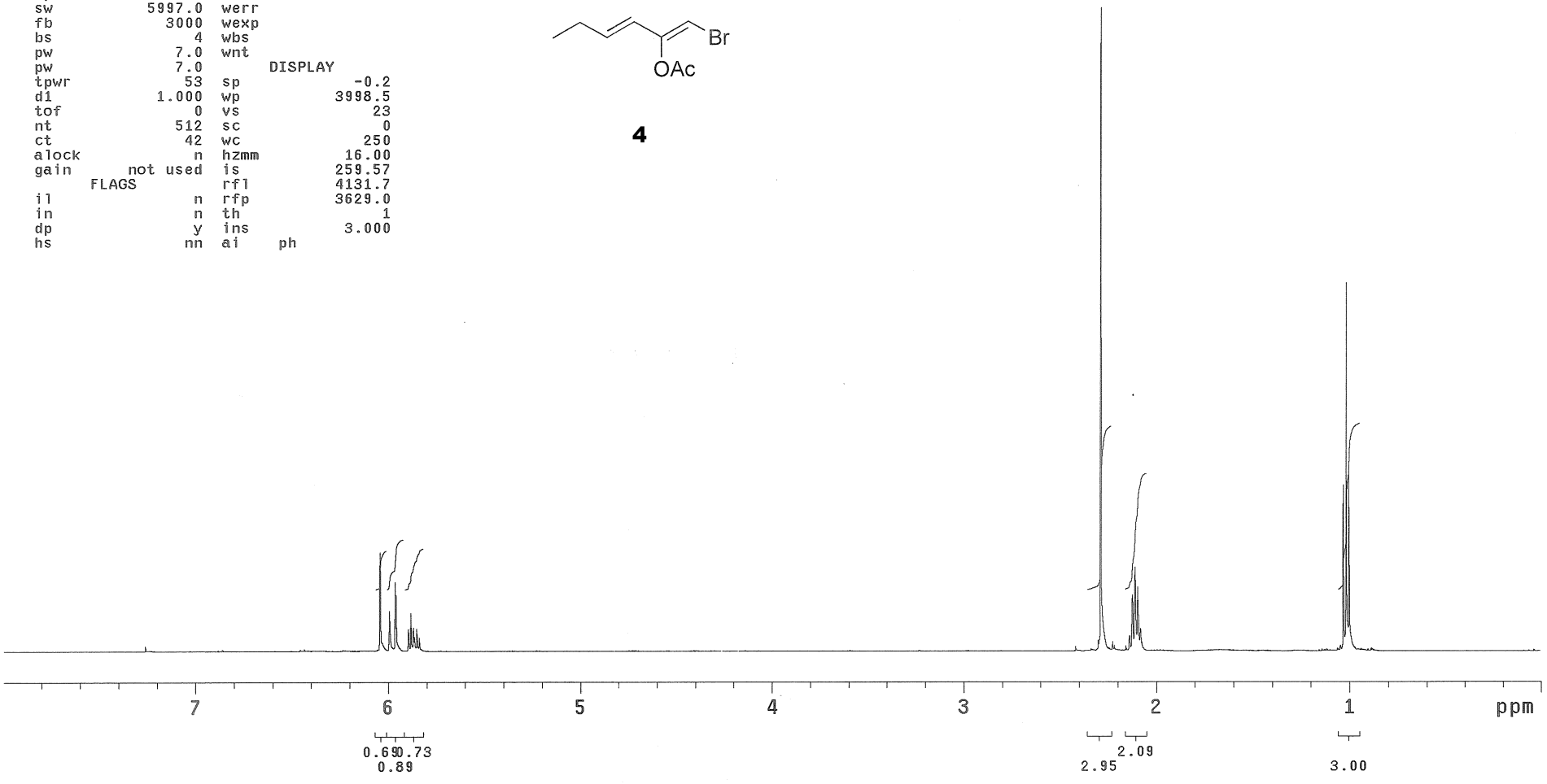


H1_CDCL3

exp2 s2pul

SAMPLE		DEC. & VT	
date	May 13 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-56-pr~	dmm	c
	o-h1.fid	dmf	200
ACQUISITION		temp	22.0
sfrq	499.859	PROCESSING	
tn	H1	fn	32768
at	2.668	math	i
np	32000		
sw	5997.0	werr	
fb	3000	wexp	
bs	4	wbs	
pw	7.0	wnt	
pw	7.0	DISPLAY	
tpwr	53	sp	-0.2
d1	1.000	wp	3998.5
tof	0	vs	23
nt	512	sc	0
ct	42	wc	250
alock	n	hzmm	16.00
gain	not used	is	259.57
FLAGS		rfl	4131.7
il	n	rfp	3629.0
in	n	th	1
dp	y	ins	3.000
hs	nn	ai ph	

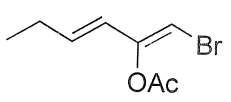
4



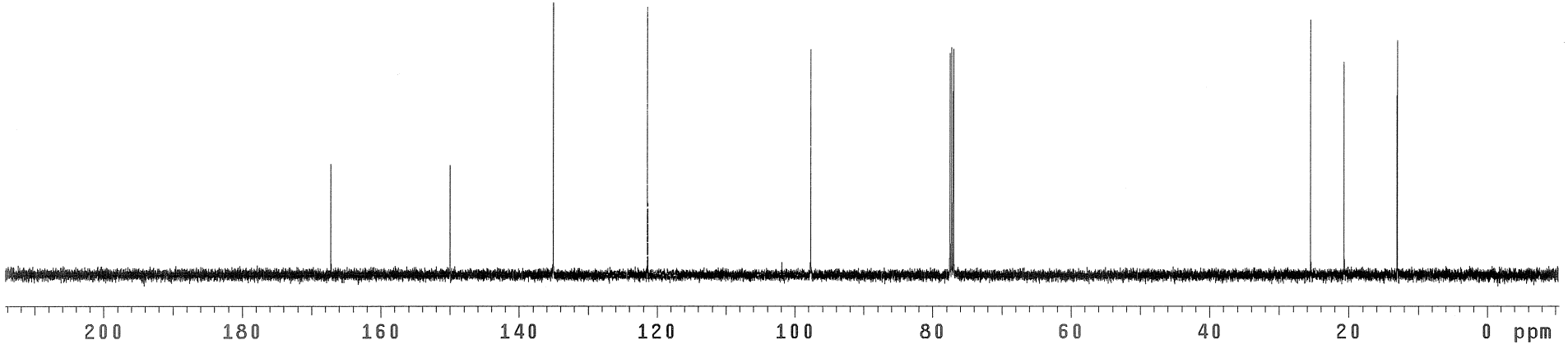
STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	May 13 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
/yawang/wyz2-56-pr~		hst	0.008
	o-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	194	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-1324.2
tof	865.4	wp	28258.1
tpwr	56	rfl	11031.6
pw	6.000	rfp	9707.0
DECOUPLER		rp	-28.2
dn	H1	lp	-885.4
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	791
dmf	11101	th	12
		ai cdc ph	



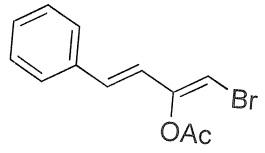
4



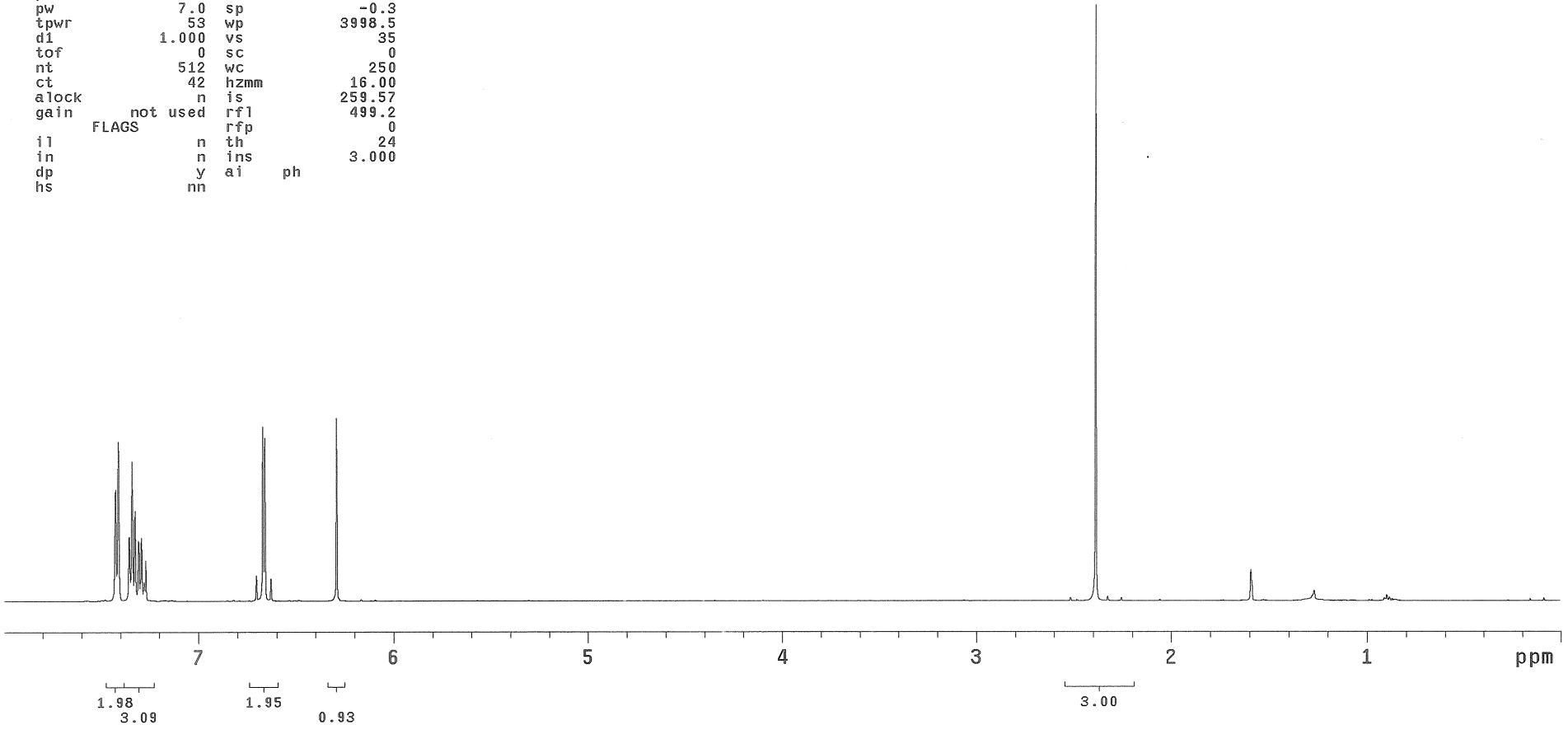
H1_CDCL3

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 15 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-59-pr~	dmm	c
	o-h1.fid	dmf	200
ACQUISITION		PROCESSING	
sfrq	499.859	fn	32768
tn	H1	math	i
at	2.668		
np	32000	werr	
sw	5997.0	wexp	
fb	3000	wbs	
bs	4	wnt	
pw	7.0	DISPLAY	
pw	7.0	sp	-0.3
tpwr	53	wp	3998.5
d1	1.000	vs	35
tof	0	sc	0
nt	512	wc	250
ct	42	hzmm	16.00
alock	n	is	259.57
gain	not used	rfl	499.2
FLAGS		rfp	0
il	n	th	24
in	n	ins	3.000
dp	y	ai ph	
hs	nn		



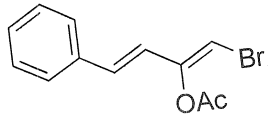
8a



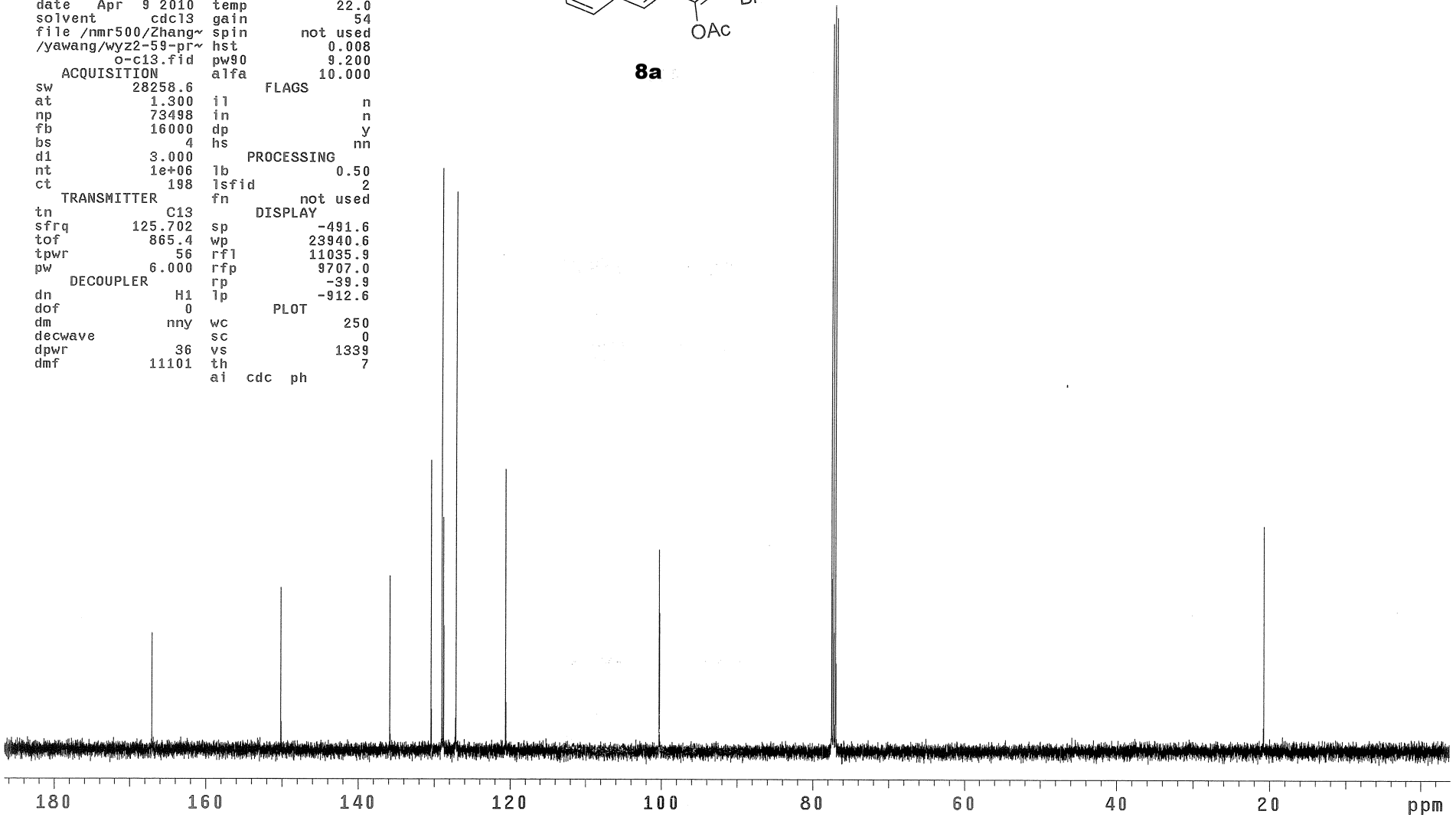
STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Apr 9 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
/yawang/wyz2-59-pr~		hst	0.008
	o-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	198	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-491.6
tof	865.4	wp	23940.6
tpwr	56	rfl	11035.9
pw	6.000	rfp	9707.0
DECOUPLER		rp	-39.9
dn	H1	lp	-912.6
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1339
dmf	11101	th	7
		ai cdc ph	



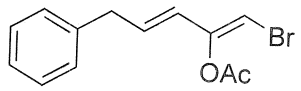
8a



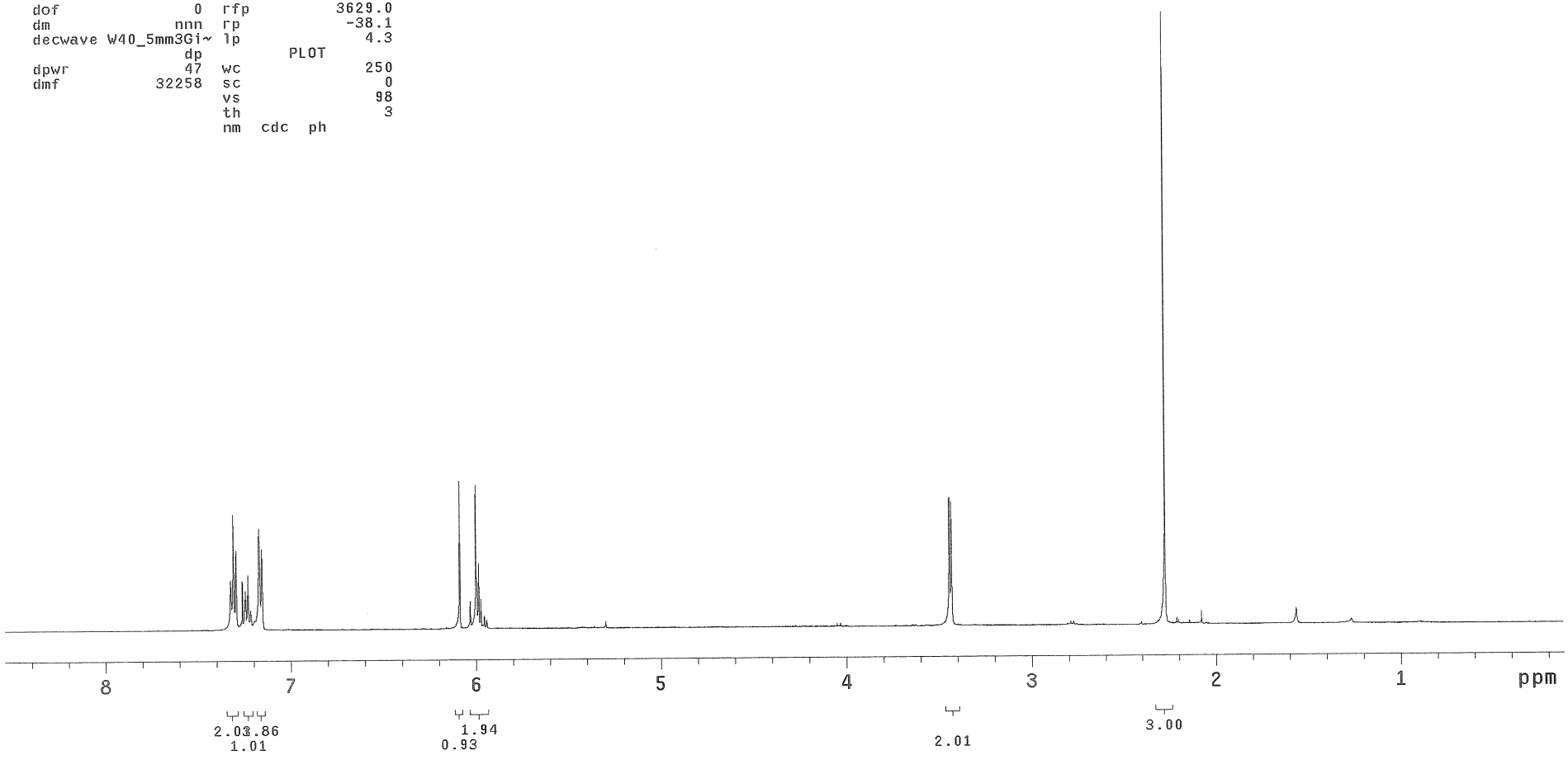
lb-I-140

exp1 PROTON

SAMPLE		PRESATURATION	
date	Mar 17 2010	satmode	n
solvent	CDCl3	wet	n
file	exp	SPECIAL	
ACQUISITION		temp	22.0
sw	7997.6	gain	not used
at	2.049	spin	20
np	32768	hst	0.008
fb	4000	pw90	9.100
bs	2	alfa	6.600
d1	1.000	FLAGS	
nt	8	il	n
ct	8	in	n
TRANSMITTER		dp	y
tn	H1	hs	nn
sfrq	499.859	PROCESSING	
tof	499.9	fn	not used
tpwr	53	DISPLAY	
pw	4.550	sp	58.7
DECOUPLER		wp	4213.6
dn	C13	rfl	4631.9
dof	0	rfp	3629.0
dm	nnn	rp	-38.1
decwave	W40_5mm3Gi~dp	lp	4.3
		PLOT	
dpwr	47	wc	250
dmf	32258	sc	0
		vs	98
		th	3
		nm cdc ph	



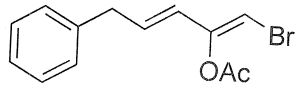
8b



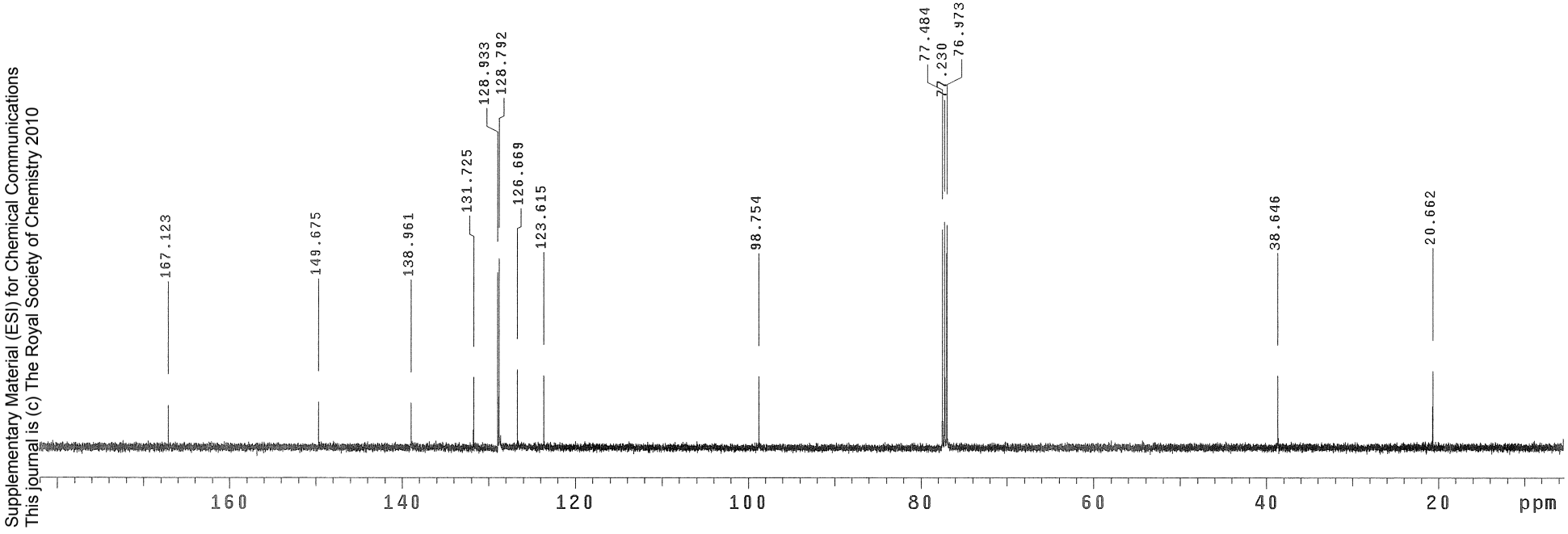
lb-I-140-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Mar 17 2010	temp	22.0
solvent	cdcl3	gain	54
file	exp	spin	20
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	4	in	n
d1	3.000	dp	y
nt	1000	hs	nn
ct	104	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	677.4
pw	6.000	wp	22205.1
DECOUPLER		rfl	11035.1
dn	H1	rfp	9707.0
dof	0	rp	5.8
dm	nny	lp	-952.5
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	412
		th	4
		ai cdc ph	



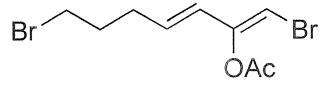
8b



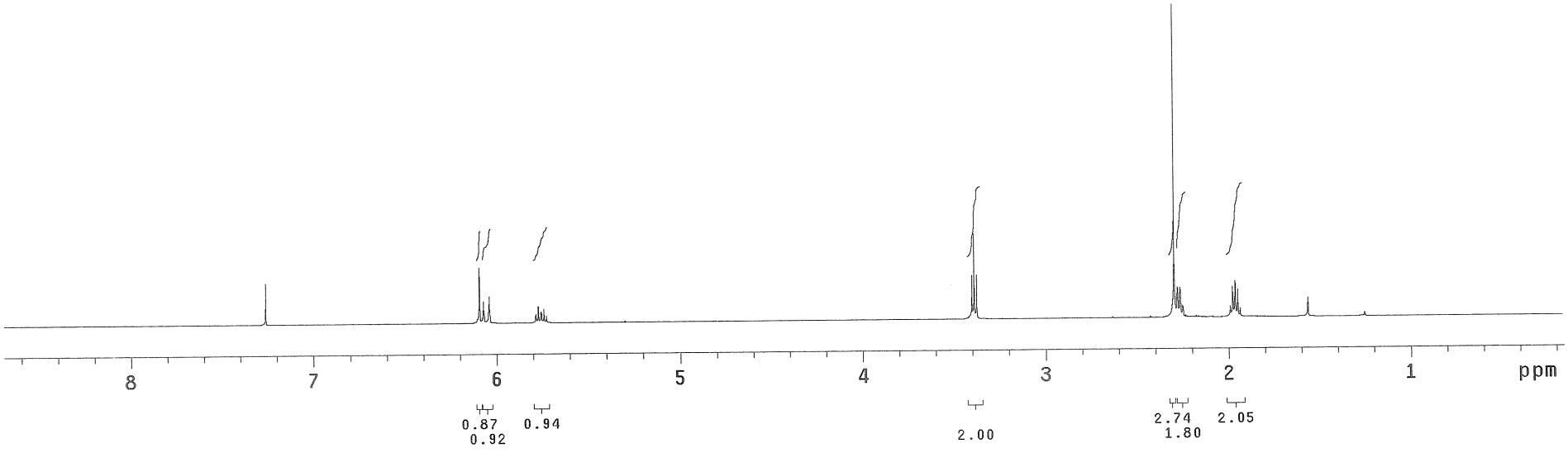
lb-I-141

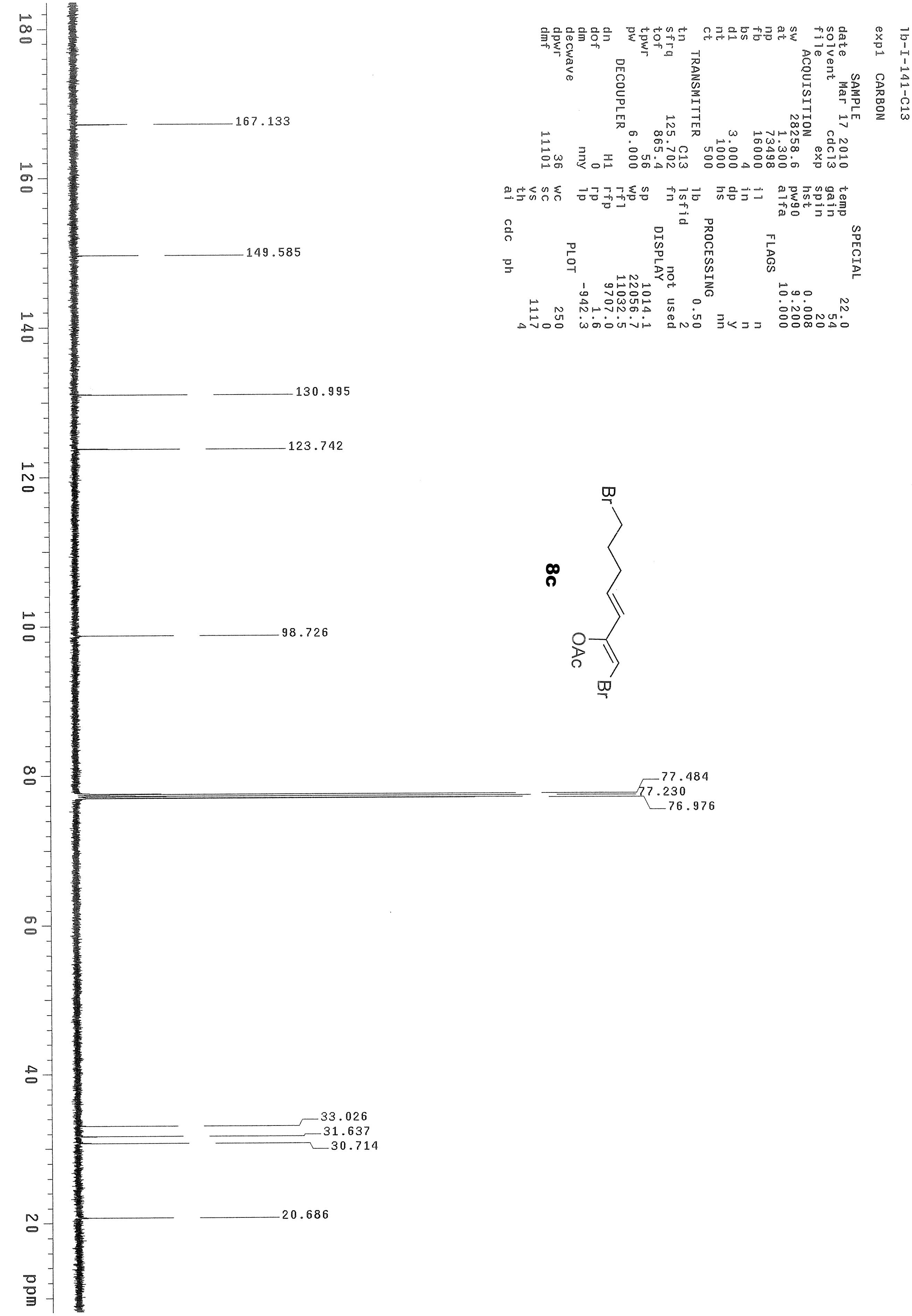
exp1 PROTON

SAMPLE		PRESATURATION	
date	Mar 17 2010	satmode	n
solvent	CDCl3	wet	n
file	exp	SPECIAL	
ACQUISITION		temp	22.0
sw	7997.6	gain	not used
at	2.049	spin	20
np	32768	hst	0.008
fb	4000	pw90	9.100
bs	4	alfa	6.600
d1	1.000	FLAGS	
nt	16	il	n
ct	16	in	n
TRANSMITTER		dp	y
tn	H1	hs	nn
sfrq	499.859	PROCESSING	
tof	499.9	fn	not used
tpwr	53	DISPLAY	
pw	4.550	sp	79.7
DECOUPLER		wp	4266.3
dn	C13	rfl	4631.9
dof	0	rfp	3629.0
dm	nnn	rp	-39.8
decwave	W40_5mm3Gi~dp	lp	-3.4
		PLOT	
dpwr	47	wc	250
dmf	32258	sc	0
		vs	50
		th	1
		nm cdc ph	



8c



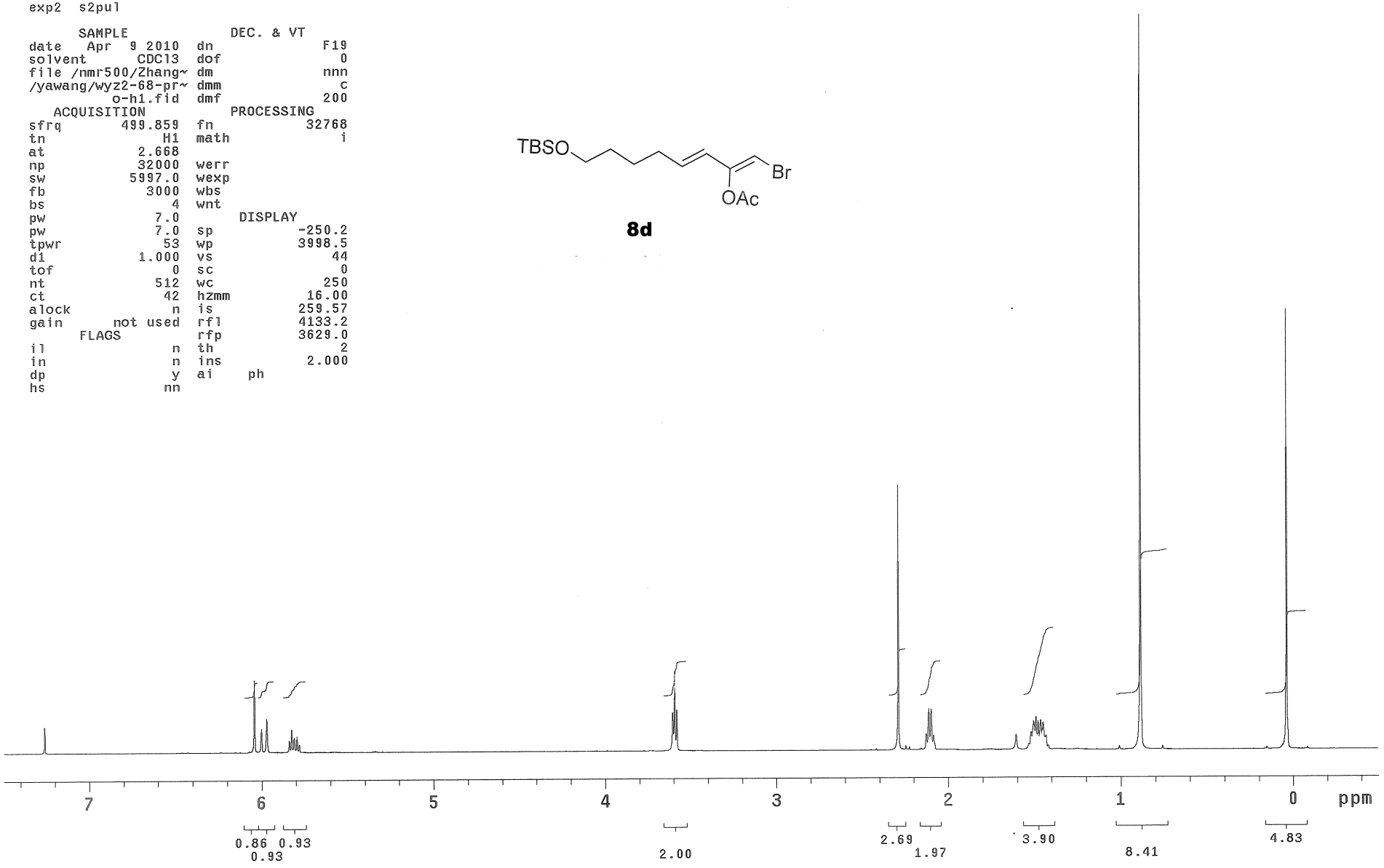

1b-I-141-C13
exp1 CARBON
167.133
149.585
130.995
123.742
98.726
77.484
77.230
76.976
33.026
31.637
30.714
20.686
180
160
140
120
100
80
60
40
20
ppm
Br
OAc
Br
8c

H1_CDCL3

exp2 s2pul

SAMPLE		DEC. & VT	
date	Apr 9 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-68-pr~	dmm	c
	o-h1.fid	dmf	200
ACQUISITION		PROCESSING	
sfrq	499.859	fn	32768
tn	H1	math	i
at	2.668		
np	32000	werr	
sw	5997.0	wexp	
fb	3000	wbs	
bs	4	wnt	
pw	7.0	DISPLAY	
pw	7.0	sp	-250.2
tpwr	53	wp	3998.5
d1	1.000	vs	44
tof	0	sc	0
nt	512	wc	250
ct	42	hzmm	16.00
alock	n	is	259.57
gain	not used	rfl	4133.2
FLAGS		rfp	3629.0
il	n	th	2
in	n	ins	2.000
dp	y	ai ph	
hs	nn		

8d

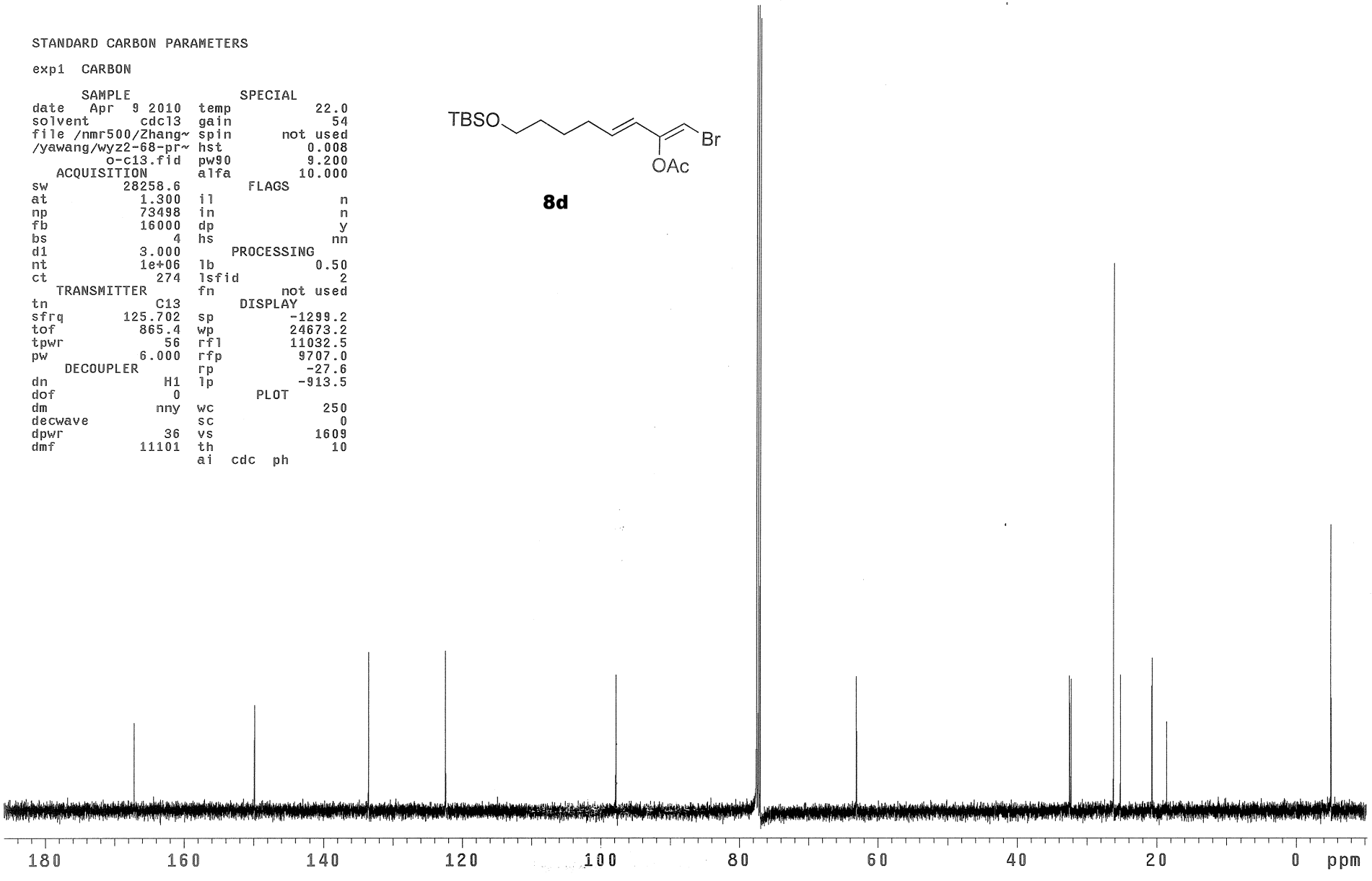


STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Apr 9 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
	/yawang/wyz2-68-pr~	hst	0.008
	o-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	274	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-1299.2
tof	865.4	wp	24673.2
tpwr	56	rfl	11032.5
pw	6.000	rfp	9707.0
DECOUPLER		rp	-27.6
dn	H1	lp	-913.5
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1609
dmf	11101	th	10
		ai cdc ph	

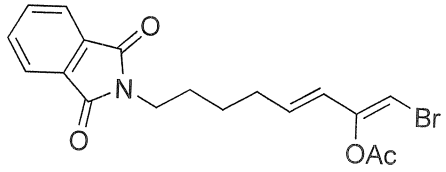
8d



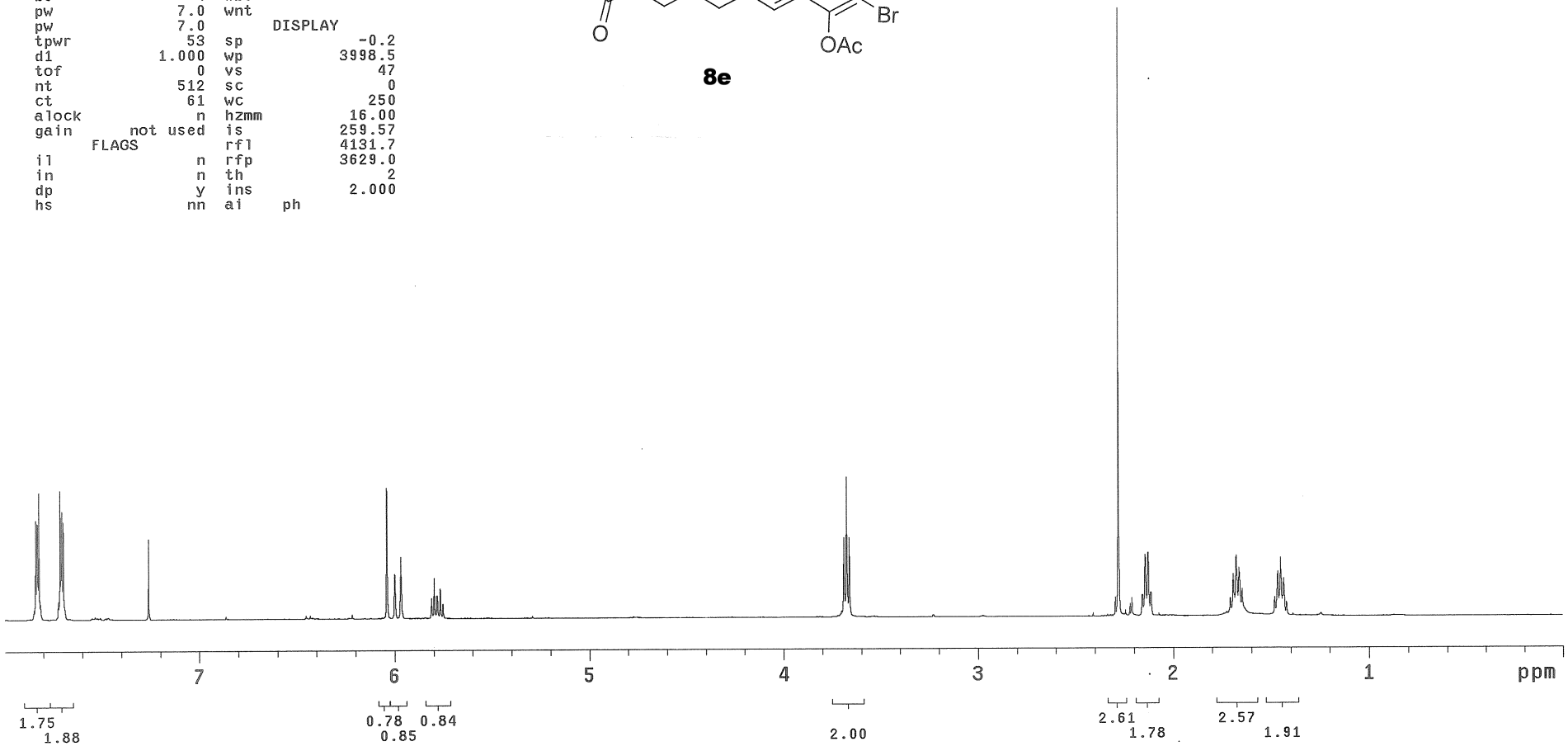
H1_CDCL3

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 1 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-102-p~	dmm	c
	ro-h1.fid	dmf	200
ACQUISITION		temp	22.0
sfrq	499.859	PROCESSING	
tn	H1	fn	32768
at	2.668	math	i
np	32000		
sw	5997.0	werr	
fb	3000	wexp	
bs	4	wbs	
pw	7.0	wnt	
pw	7.0	DISPLAY	
tpwr	53	sp	-0.2
d1	1.000	wp	3998.5
tof	0	vs	47
nt	512	sc	0
ct	61	wc	250
alock	n	hzmm	16.00
gain	not used	is	259.57
FLAGS		rfl	4131.7
il	n	rfp	3629.0
in	n	th	2
dp	y	ins	2.000
hs	nn	ai ph	



8e

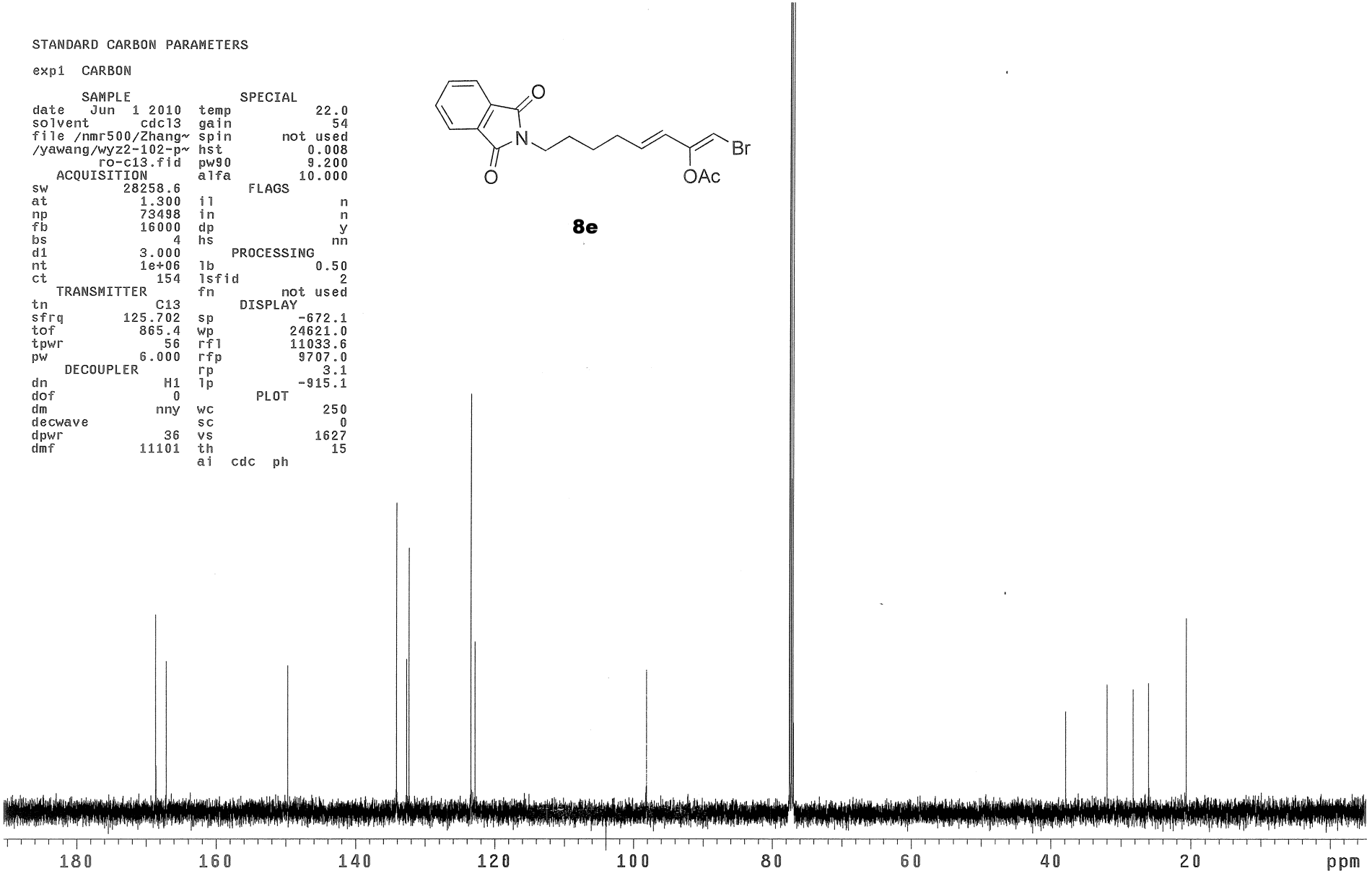


STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Jun 1 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
/yawang/wyz2-102-p~		hst	0.008
	ro-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	154	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-672.1
tof	865.4	wp	24621.0
tpwr	56	rfl	11033.6
pw	6.000	rfp	9707.0
DECOUPLER		rp	3.1
dn	H1	lp	-915.1
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1627
dmf	11101	th	15
		ai cdc ph	

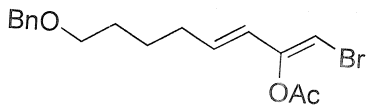
8e



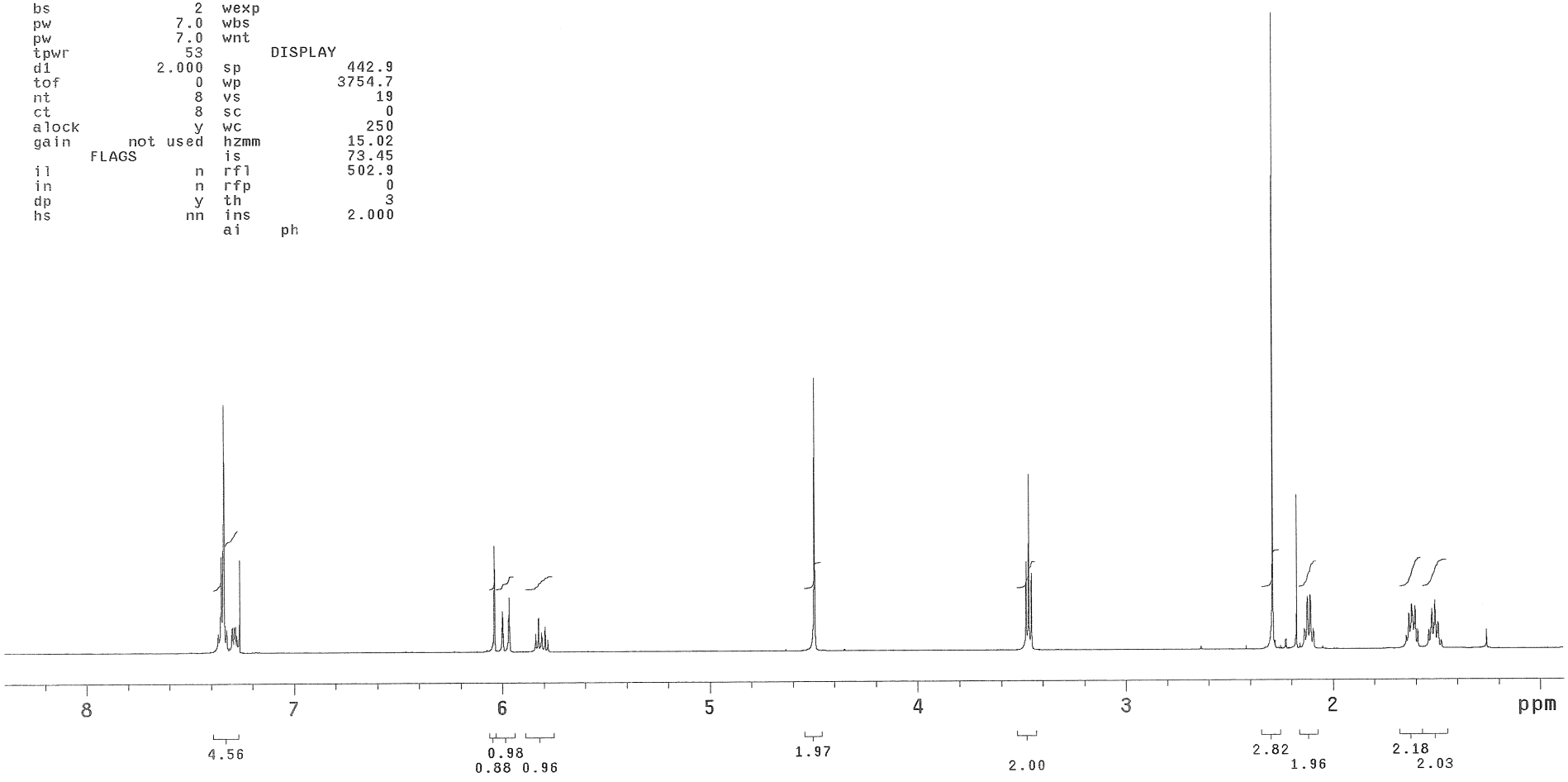
1b-I-165

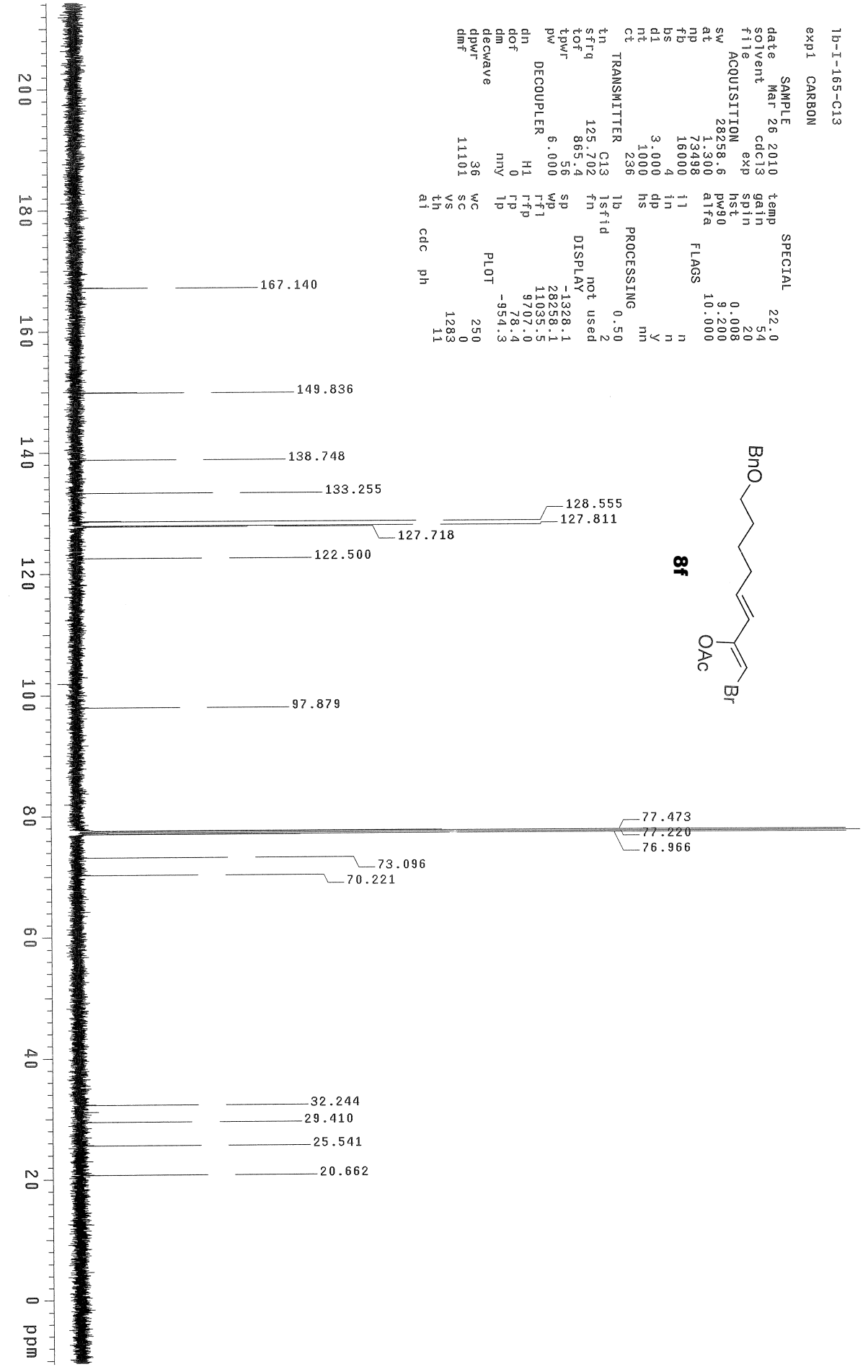
exp1 s2pul

SAMPLE		DEC. & VT	
date	Mar 26 2010	dn	F19
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	499.859	dmf	200
tn	H1	PROCESSING	
at	2.668	fn	32768
np	32000	math	i
sw	5997.0		
fb	3000	werr	
bs	2	wexp	
pw	7.0	wbs	
pw	7.0	wnt	
tpwr	53	DISPLAY	
d1	2.000	sp	442.9
tof	0	wp	3754.7
nt	8	vs	19
ct	8	sc	0
alock	y	wc	250
gain	not used	hzmm	15.02
FLAGS		is	73.45
il	n	rfl	502.9
in	n	rfp	0
dp	y	th	3
hs	nn	ins	2.000
		ai ph	



8f

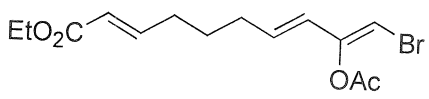



1b-I-165-C13
exp1 CARBON
SAMPLE
date Mar 26 2010
solvent cdcl3
file exp
ACQUISITION
sw 28258.6
at 1.300
np 73498
fb 16000
bs 4
d1 3.000
nt 1000
ct 236
TRANSMITTER
tn C13
sfrq 125.702
tof 865.4
tpwr 56
pw 6.000
DECOUPLER
dn H1
dof 0
dm nny
decwave
dpwr 36
dmf 11101
SPECIAL
temp 22.0
gain 54
spin 20
hst 0.008
pw90 9.200
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
lsfid 2
fn not used
DISPLAY
sp -1328.1
wp 28258.1
rfl 11035.5
rfp 9707.0
rp 78.4
lp -954.3
PLOT
wc 250
sc 0
vs 1283
th 11
ai cdc ph
167.140
149.836
138.748
133.255
128.555
127.811
127.718
122.500
97.879
77.473
77.220
76.966
73.096
70.221
32.244
29.410
25.541
20.662
200
180
160
140
120
100
80
60
40
20
0 ppm
8f
BnO
OAc
Br

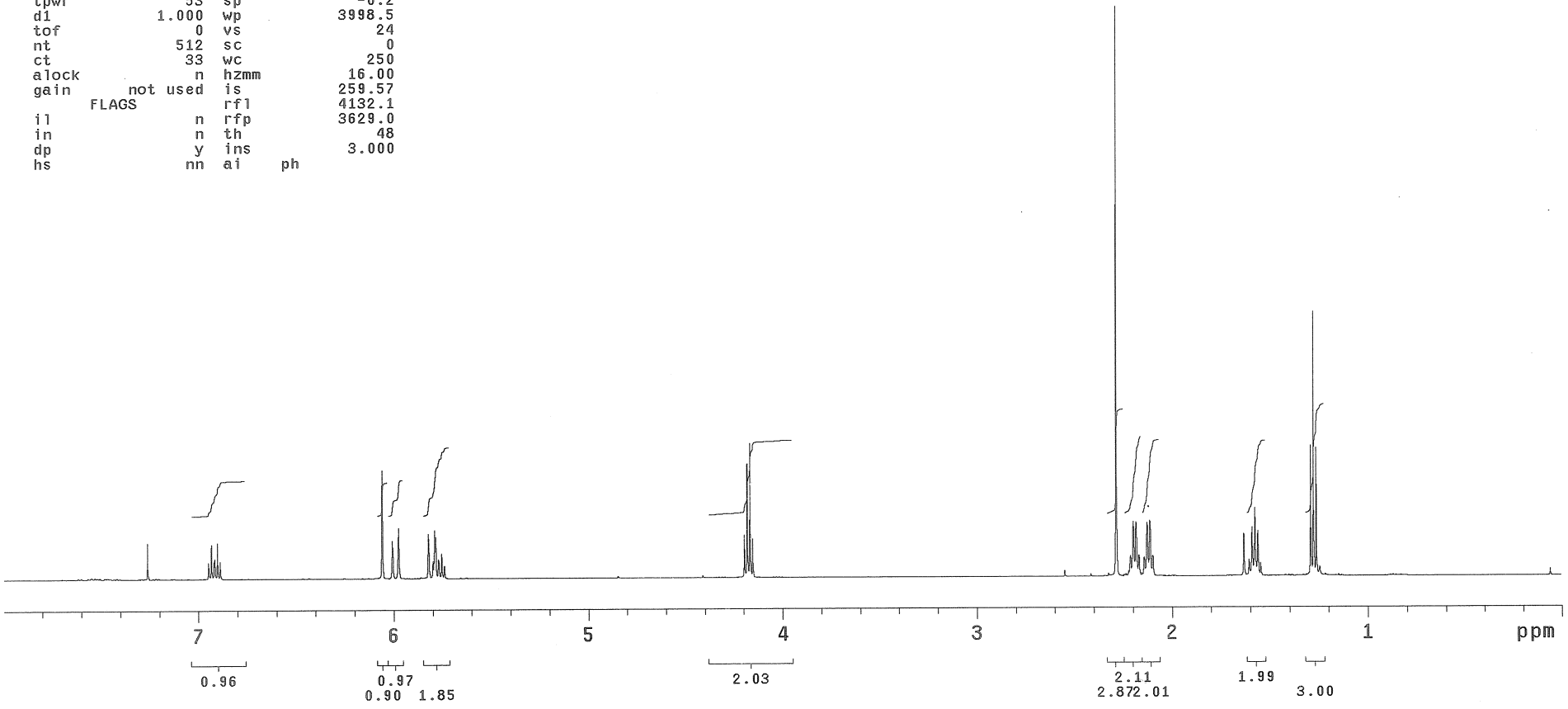
H1_CDCL3

exp2 s2pul

SAMPLE		DEC. & VT	
date	Jun 2 2010	dn	F19
solvent	CDCl3	dof	0
file	/nmr500/Zhang~	dm	nnn
	/yawang/wyz2-104-p~	dmm	c
	ro-h1.fid	dmf	200
ACQUISITION		temp	22.0
sfrq	499.859	PROCESSING	
tn	H1	fn	32768
at	2.668	math	i
np	32000		
sw	5997.0	werr	
fb	3000	wexp	
bs	4	wbs	
pw	7.0	wnt	
pw	7.0	DISPLAY	
tpwr	53	sp	-0.2
d1	1.000	wp	3998.5
tof	0	vs	24
nt	512	sc	0
ct	33	wc	250
alock	n	hzmm	16.00
gain	not used	is	259.57
FLAGS		rfl	4132.1
il	n	rfp	3629.0
in	n	th	48
dp	y	ins	3.000
hs	nn	ai ph	



8g

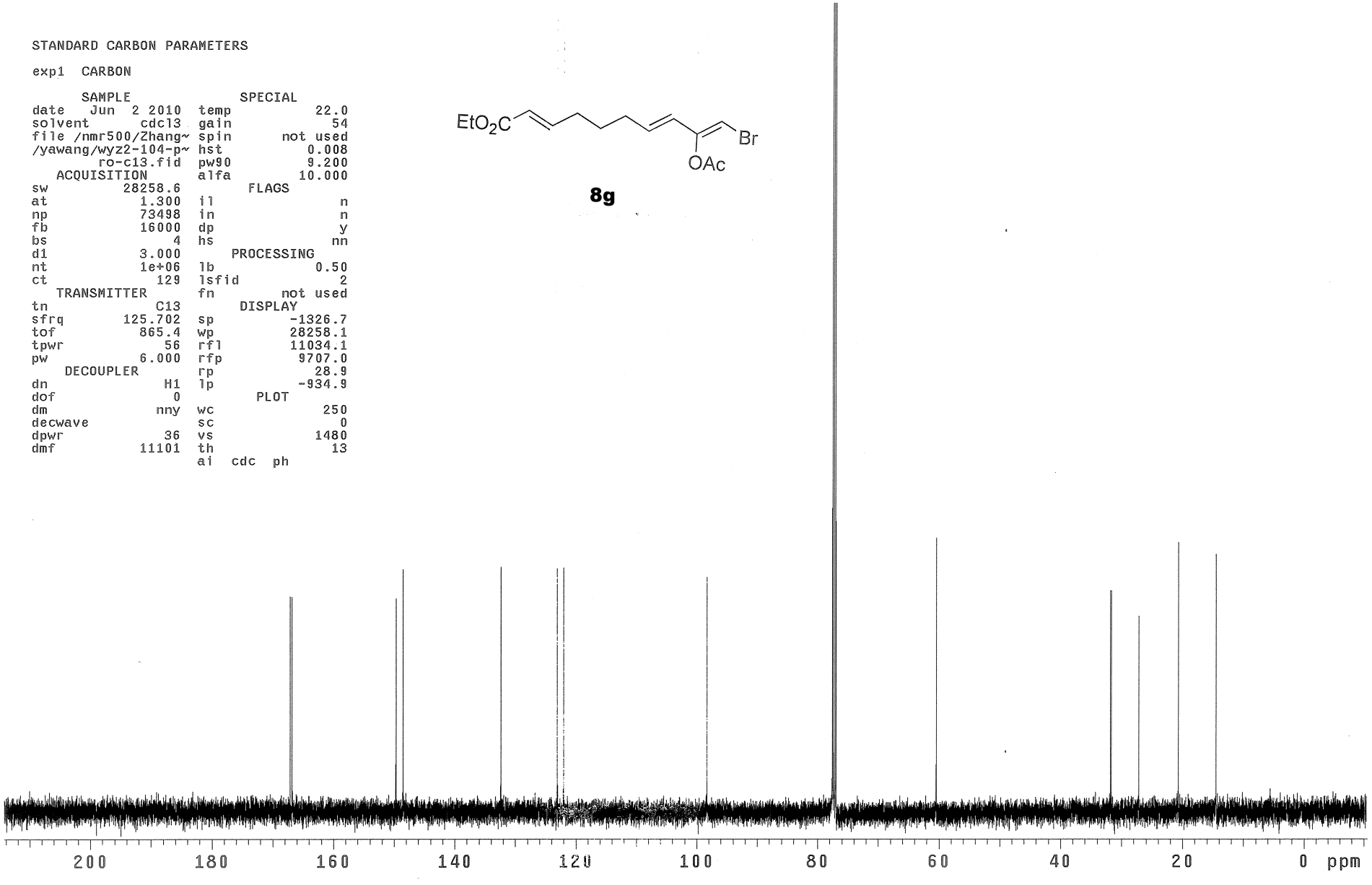


STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Jun 2 2010	temp	22.0
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
/yawang/wyz2-104-p~		hst	0.008
	ro-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	129	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-1326.7
tof	865.4	wp	28258.1
tpwr	56	rfl	11034.1
pw	6.000	rfp	9707.0
DECOUPLER		rp	28.9
dn	H1	lp	-934.9
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1480
dmf	11101	th	13
		ai cdc ph	

8g

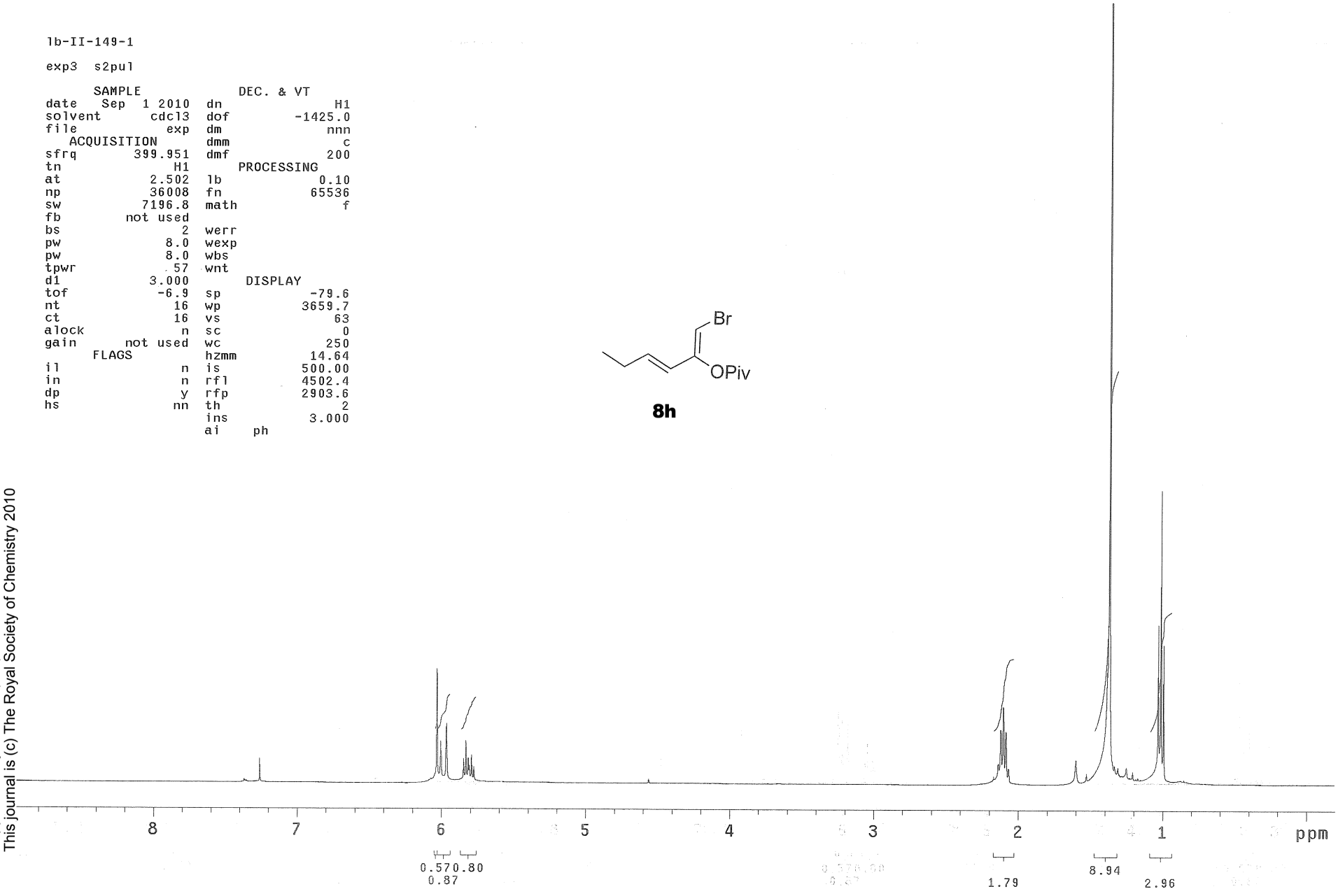


lb-II-149-1

exp3 s2pul

SAMPLE		DEC. & VT	
date	Sep 1 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	PROCESSING	
at	2.502	lb	0.10
np	36008	fn	65536
sw	7196.8	math	f
fb	not used		
bs	2	werr	
pw	8.0	wexp	
pw	8.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	-6.9	sp	-79.6
nt	16	wp	3659.7
ct	16	vs	63
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	14.64
il	n	is	500.00
in	n	rfl	4502.4
dp	y	rfp	2903.6
hs	nn	th	2
		ins	3.000
		ai ph	

8h

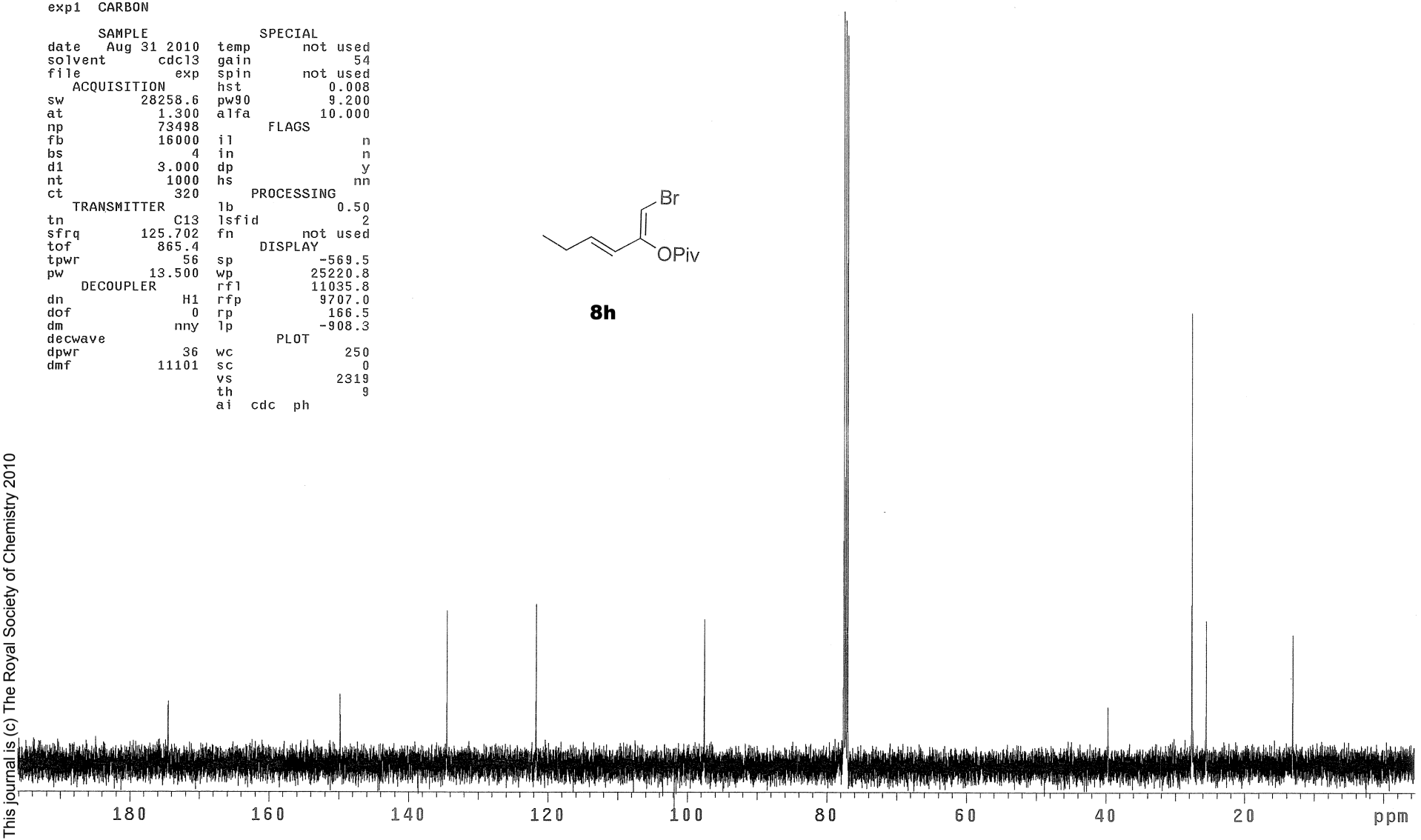


lb-II-149-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Aug 31 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	4	in	n
d1	3.000	dp	y
nt	1000	hs	nn
ct	320	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	-569.5
pw	13.500	wp	25220.8
DECOUPLER		rfl	11035.8
dn	H1	rfp	9707.0
dof	0	rp	166.5
dm	nny	lp	-908.3
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	2319
		th	9
		ai cdc ph	

8h

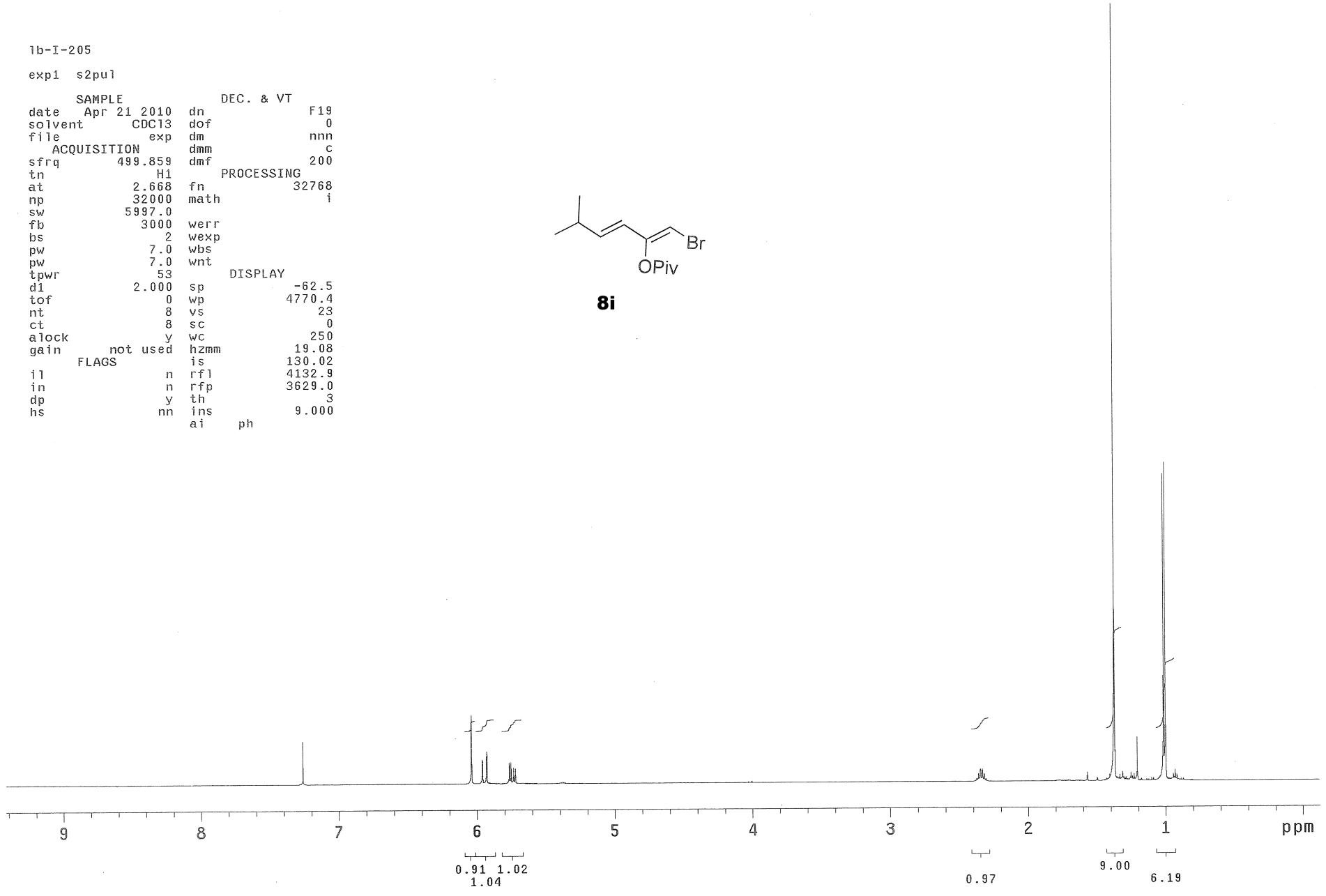


1b-I-205

exp1 s2pul

SAMPLE		DEC. & VT	
date	Apr 21 2010	dn	F19
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	499.859	dmf	200
tn	H1	PROCESSING	
at	2.668	fn	32768
np	32000	math	i
sw	5997.0		
fb	3000	werr	
bs	2	wexp	
pw	7.0	wbs	
pw	7.0	wnt	
tpwr	53	DISPLAY	
d1	2.000	sp	-62.5
tof	0	wp	4770.4
nt	8	vs	23
ct	8	sc	0
alock	y	wc	250
gain	not used	hzmm	19.08
FLAGS		is	130.02
il	n	rfl	4132.9
in	n	rfp	3629.0
dp	y	th	3
hs	nn	ins	9.000
		ai ph	

8i

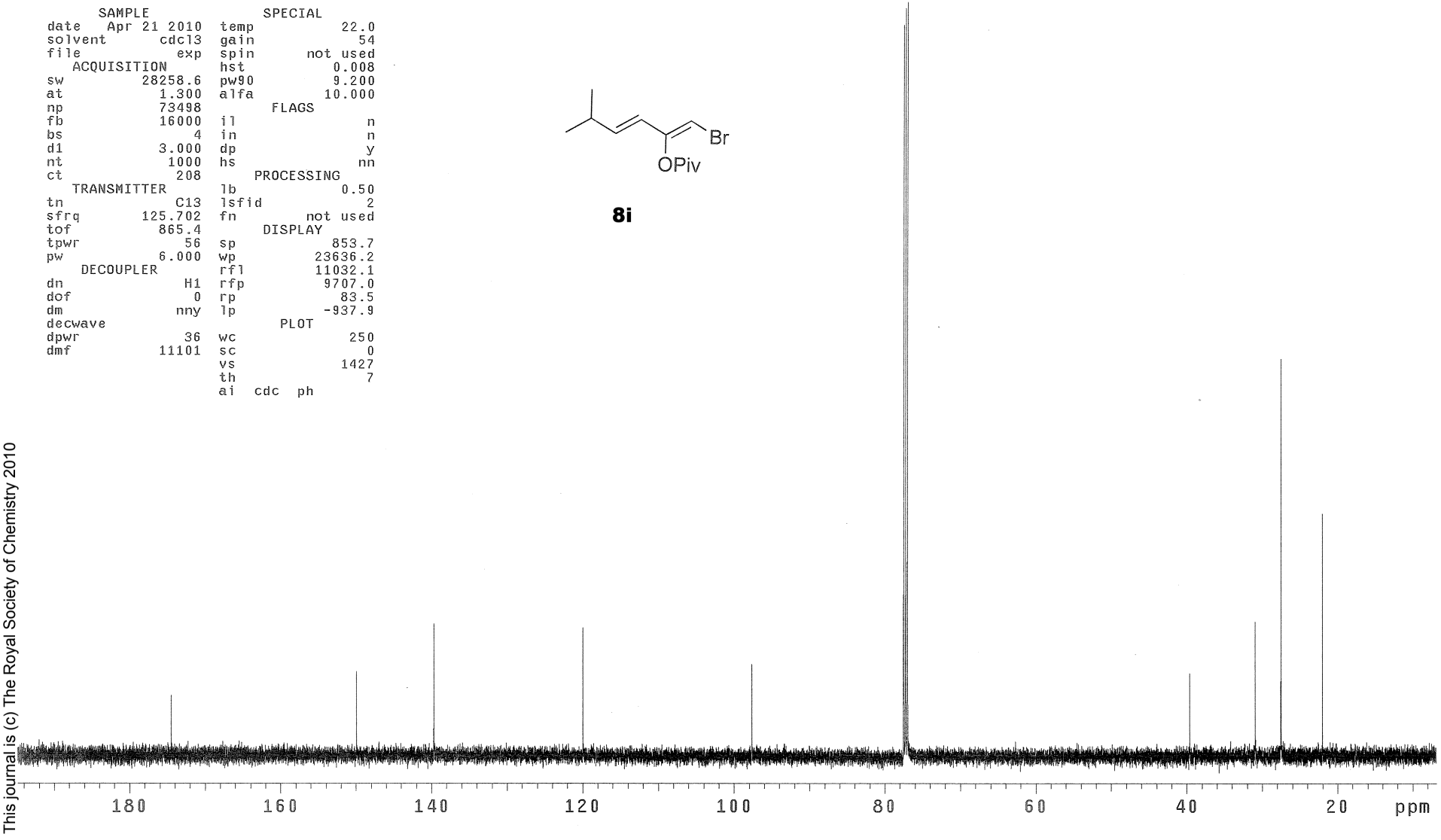


lb-I-205-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Apr 21 2010	temp	22.0
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	4	in	n
d1	3.000	dp	y
nt	1000	hs	nn
ct	208	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	853.7
pw	6.000	wp	23636.2
DECOUPLER		rfl	11032.1
dn	H1	rfp	9707.0
dof	0	rp	83.5
dm	nny	lp	-937.9
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	1427
		th	7
		ai cdc ph	

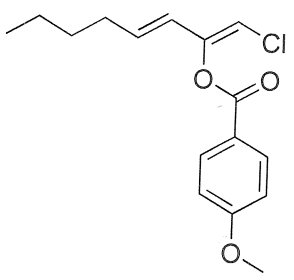
8i



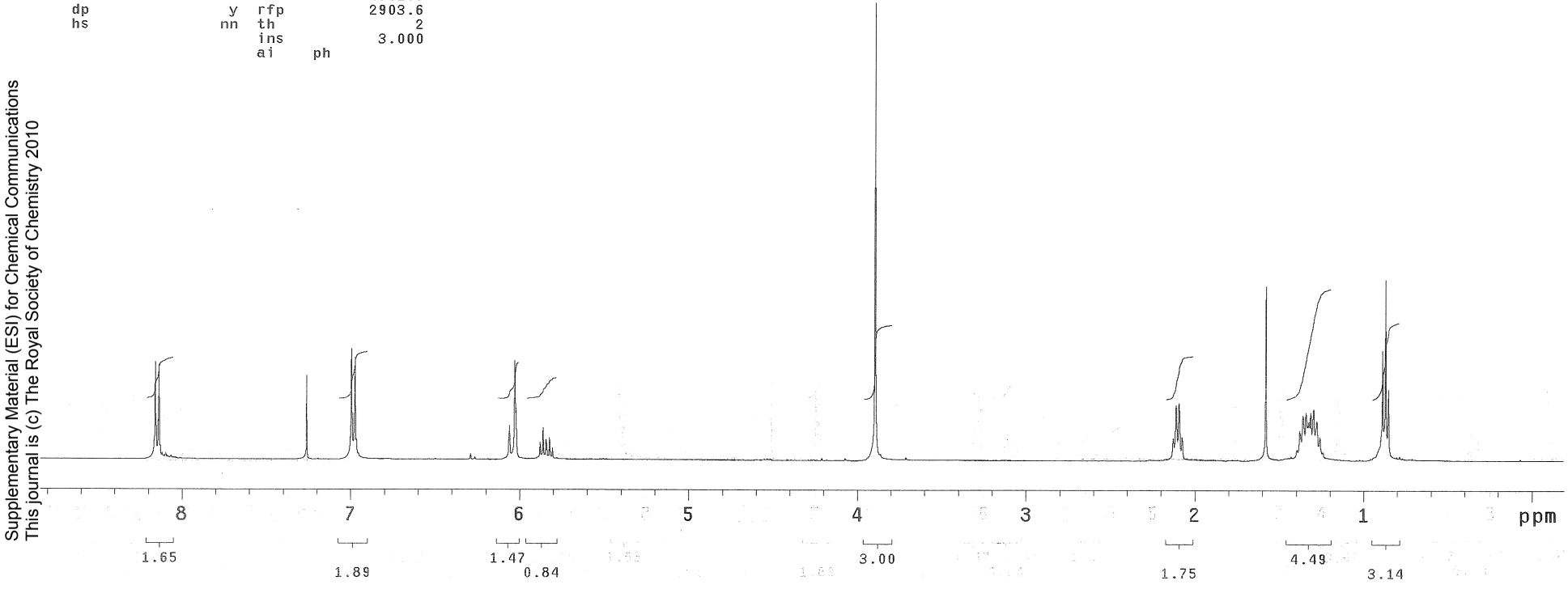
wyz-2-171-product

exp1 s2pul

SAMPLE		DEC. & VT	
date	Aug 26 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	PROCESSING	
at	2.502	lb	0.10
np	36008	fn	65536
sw	7196.8	math	f
fb	not used		
bs	2	werr	
pw	8.0	wexp	
pw	8.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	-6.9	sp	-76.4
nt	16	wp	3612.7
ct	16	vs	45
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	14.45
il	n	is	291.74
in	n	rfl	4502.5
dp	y	rfp	2903.6
hs	nn	th	2
		ins	3.000
		ai ph	



8j

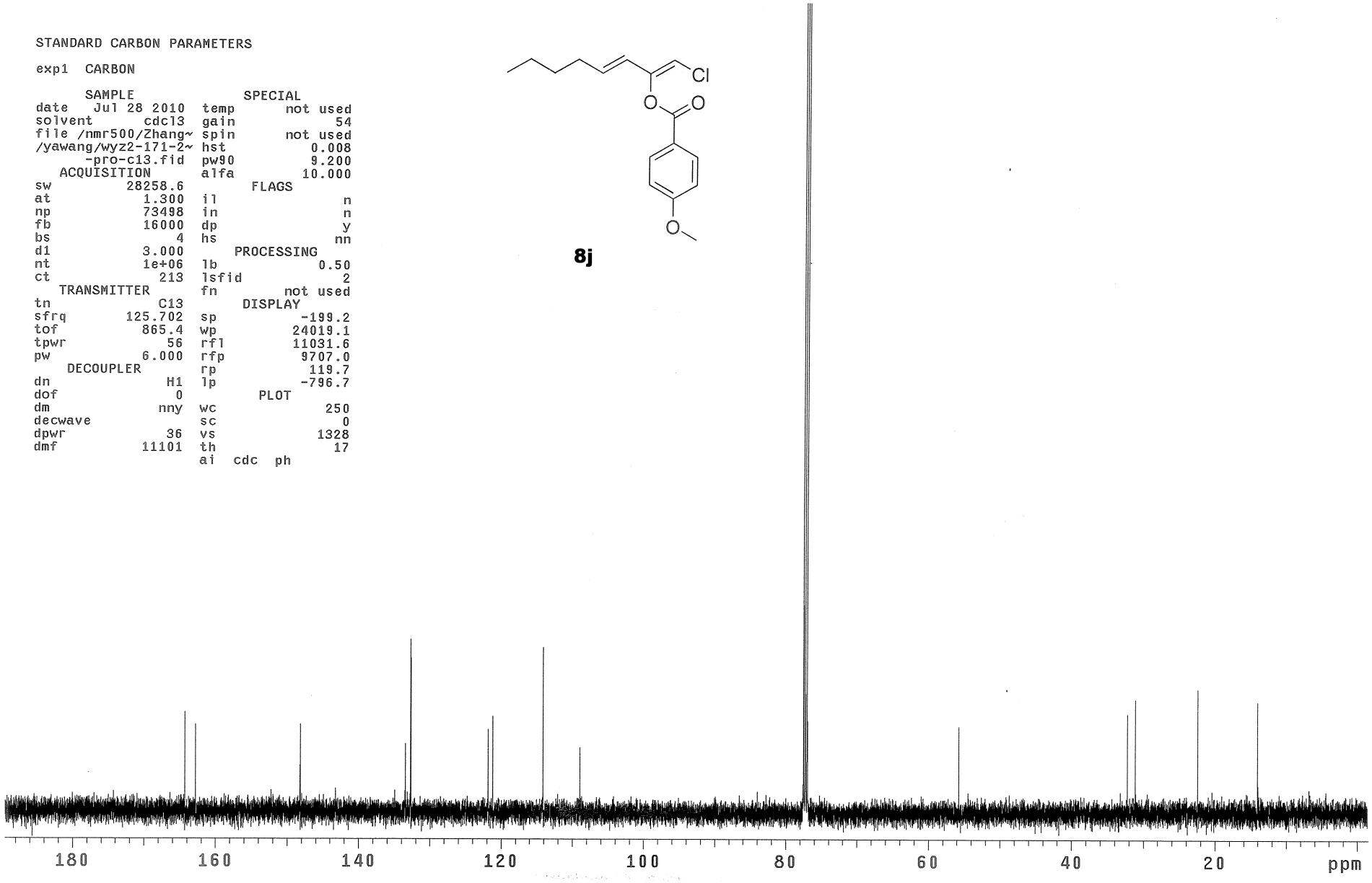


STANDARD CARBON PARAMETERS

exp1 CARBON

SAMPLE		SPECIAL	
date	Jul 28 2010	temp	not used
solvent	cdcl3	gain	54
file	/nmr500/Zhang~	spin	not used
	/yawang/wyz2-171-2~	hst	0.008
	-pro-c13.fid	pw90	9.200
ACQUISITION		alfa	10.000
sw	28258.6	FLAGS	
at	1.300	il	n
np	73498	in	n
fb	16000	dp	y
bs	4	hs	nn
d1	3.000	PROCESSING	
nt	1e+06	lb	0.50
ct	213	lsfid	2
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	125.702	sp	-199.2
tof	865.4	wp	24019.1
tpwr	56	rfl	11031.6
pw	6.000	rfp	9707.0
DECOUPLER		rp	119.7
dn	H1	lp	-796.7
dof	0	PLOT	
dm	nny	wc	250
decwave		sc	0
dpwr	36	vs	1328
dmf	11101	th	17
		ai cdc ph	

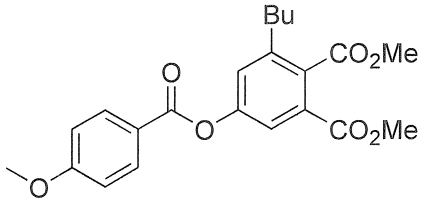
8j



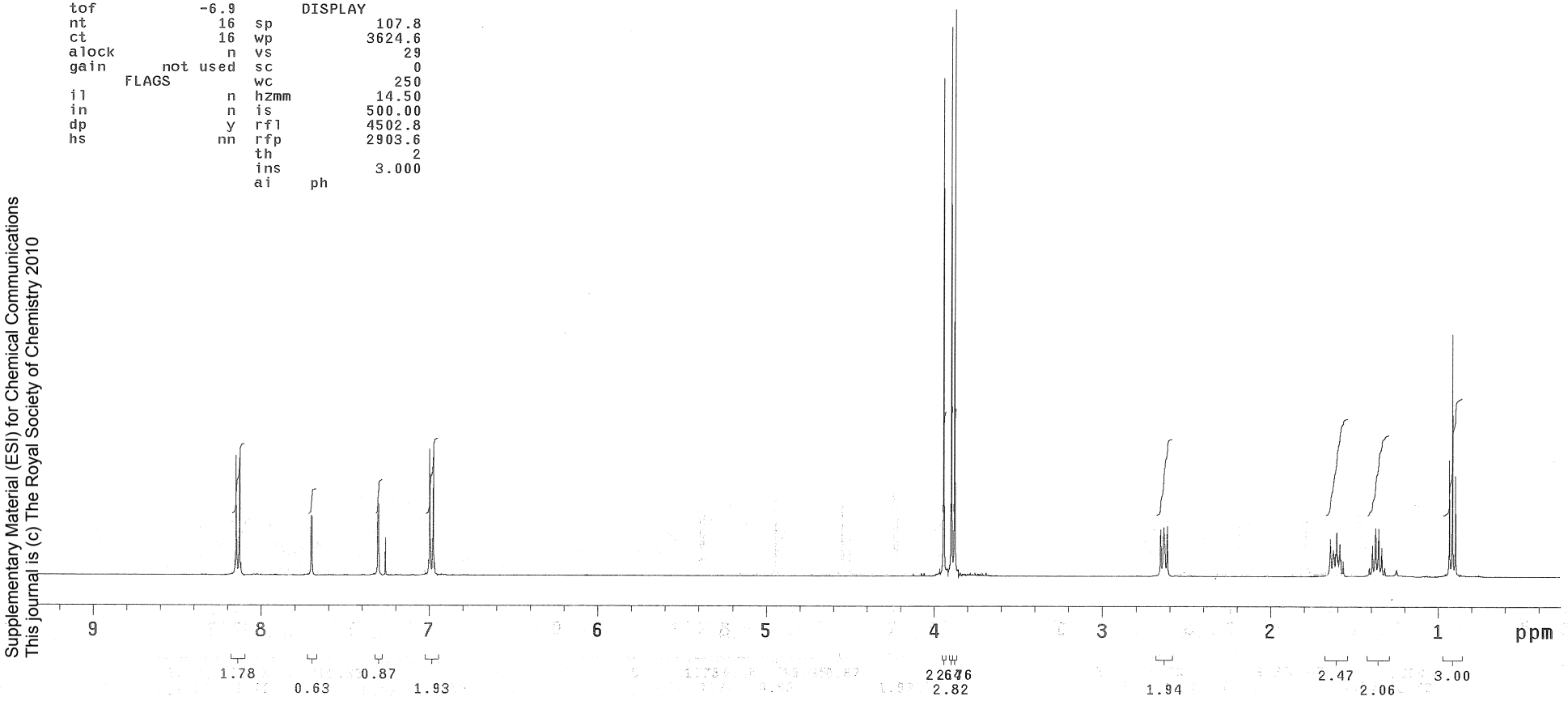
wyz-DA-1

exp3 s2pul

SAMPLE		DEC. & VT	
date	Aug 23 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	temp	22.0
at	2.502	PROCESSING	
np	36008	lb	0.10
sw	7196.8	fn	65536
fb	not used	math	f
bs	2		
pw	8.0	werr	
pw	8.0	wexp	
tpwr	57	wbs	
d1	3.000	wnt	
tof	-6.9	DISPLAY	
nt	16	sp	107.8
ct	16	wp	3624.6
alock	n	vs	29
gain	not used	sc	0
FLAGS		wc	250
il	n	hzmm	14.50
in	n	is	500.00
dp	y	rfl	4502.8
hs	nn	rfp	2903.6
		th	2
		ins	3.000
		ai ph	



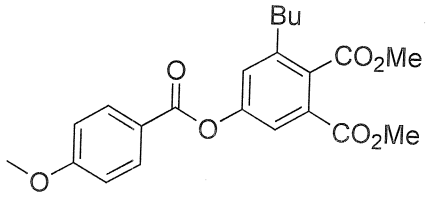
9



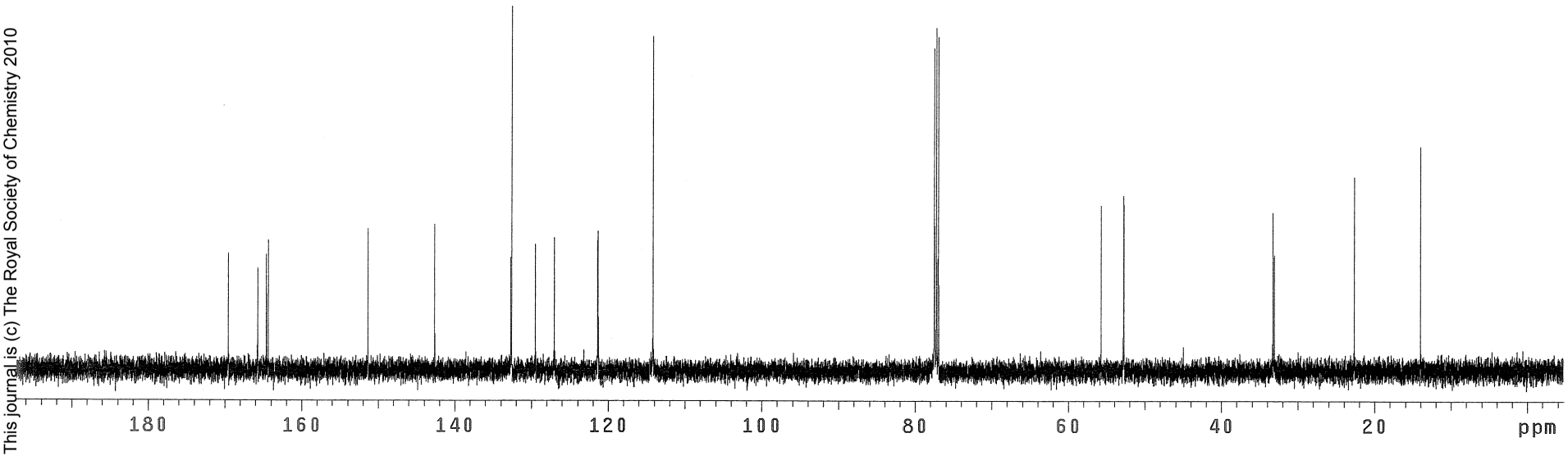
wyz-DA-1

exp1 CARBON

SAMPLE		SPECIAL	
date	Aug 23 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	**FLAGS**	
fb	16000	il	n
bs	4	in	n
d1	3.000	dp	y
nt	1000	hs	nn
ct	108	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	**DISPLAY**	
tpwr	56	sp	-600.9
pw	13.500	wp	25386.0
DECOUPLER		rfl	11034.4
dn	H1	rfp	9707.0
dof	0	rp	-108.7
dm	nny	lp	-930.7
decwave		**PLOT**	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	969
		th	14
		ai cdc ph	



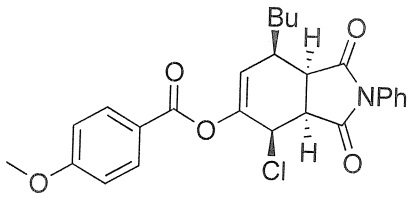
9



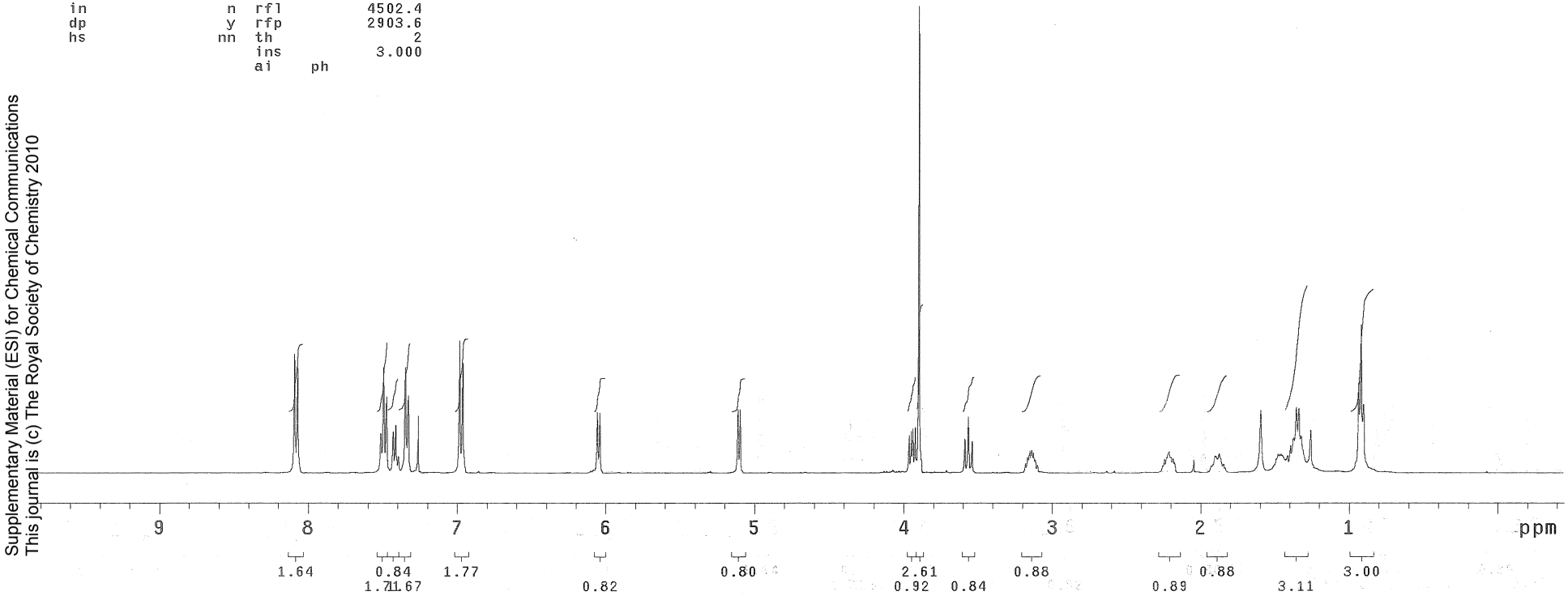
lb-II-142-C-purification

exp3 s2pul

SAMPLE		DEC. & VT	
date	Aug 23 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	PROCESSING	
at	2.502	lb	0.10
np	36008	fn	65536
sw	7196.8	math	f
fb	not used		
bs	2	werr	
pw	8.0	wexp	
pw	8.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	-6.9	sp	-179.3
nt	32	wp	4105.5
ct	32	vs	59
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	16.42
il	n	is	500.00
in	n	rfl	4502.4
dp	y	rfp	2903.6
hs	nn	th	2
		ins	3.000
		ai ph	



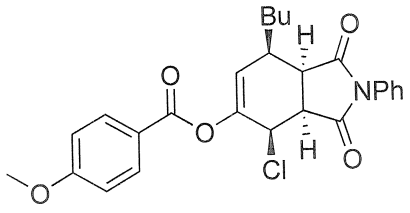
10



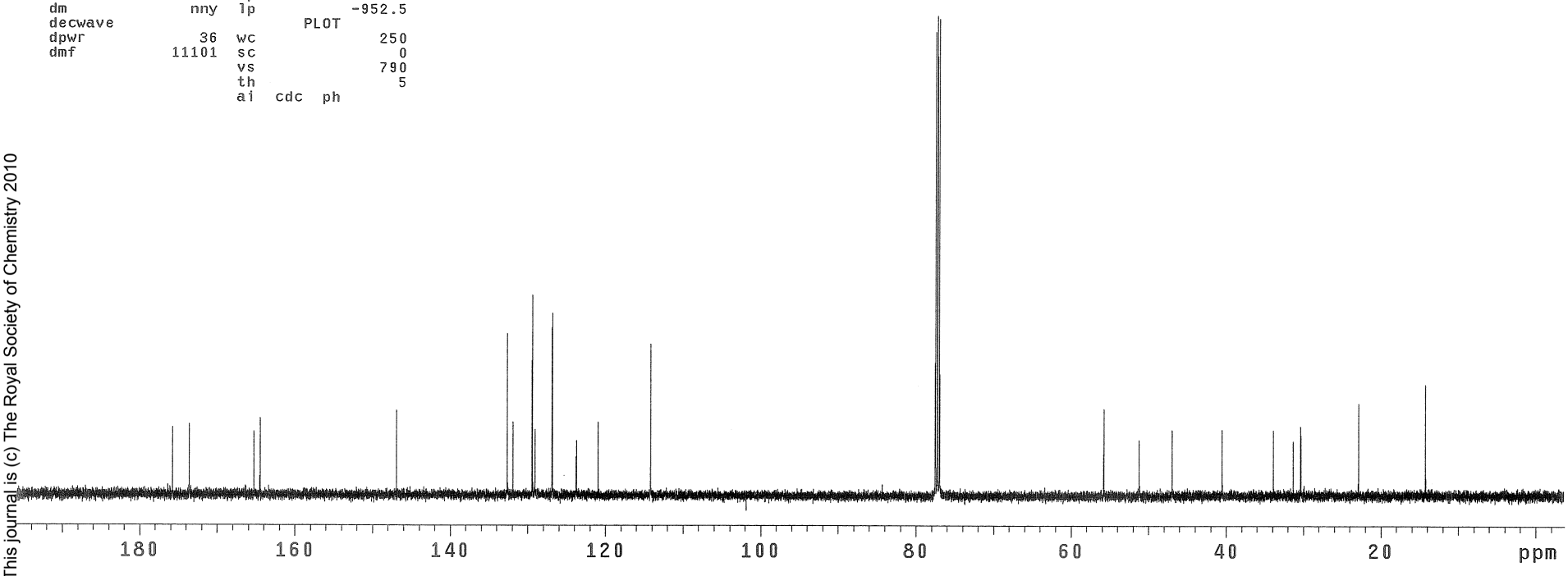
lb-II-142-C-13

exp2 CARBON

SAMPLE		SPECIAL	
date	Aug 23 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	**FLAGS**	
fb	16000	il	n
bs	5	in	n
d1	3.000	dp	y
nt	10000	hs	nn
ct	380	**PROCESSING**	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	**DISPLAY**	
tpwr	56	sp	-467.8
pw	13.500	wp	25088.9
DECOUPLER		rfl	11033.3
dn	H1	rfp	9707.0
dof	0	rp	-118.6
dm	nny	lp	-952.5
decwave		**PLOT**	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	790
		th	5
		ai cdc ph	



10

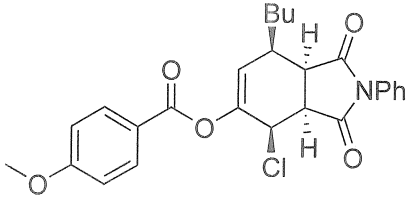


lb-II-142-C

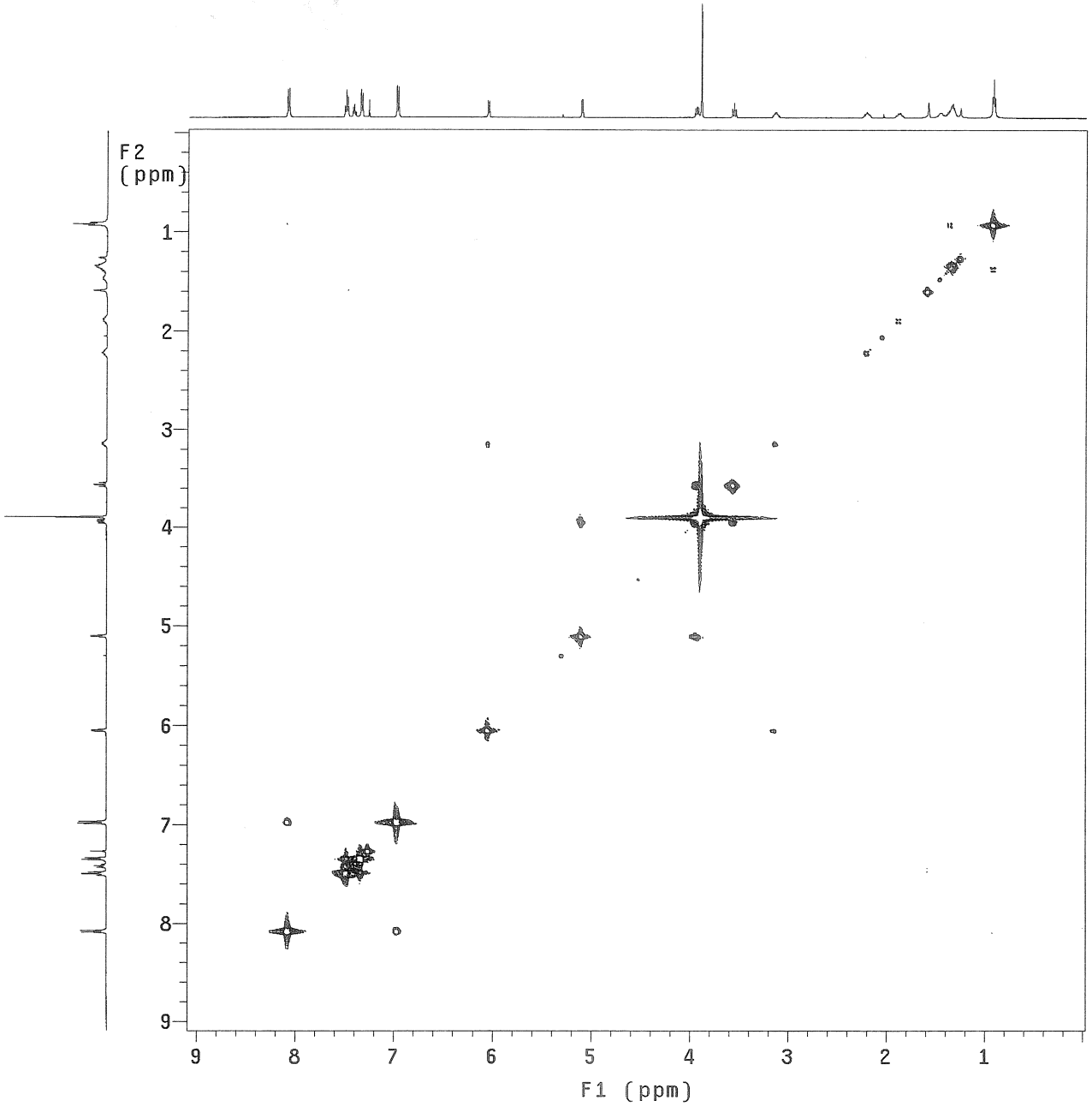
File: xp

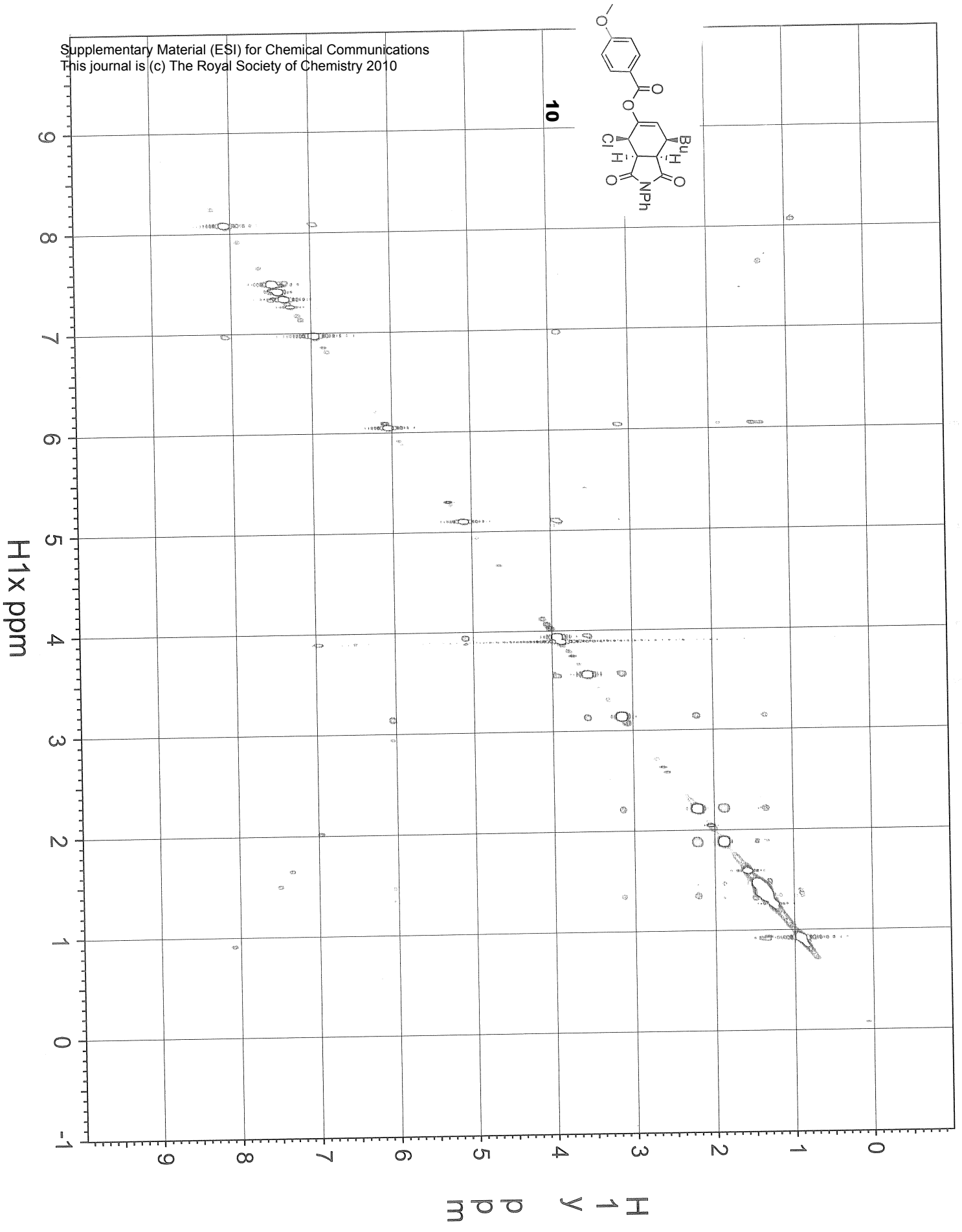
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
Operator: biaolu
INOVA-500 "nmr500"

Relax. delay 3.000 sec
COSY 90-90
Acq. time 0.224 sec
Width 4566.2 Hz
2D Width 4566.2 Hz
3 repetitions
256 increments
OBSERVE H1, 499.8560507 MHz
DATA PROCESSING
Sine bell 0.112 sec
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 1024 x 1024
Total time 42 min, 12 sec



10



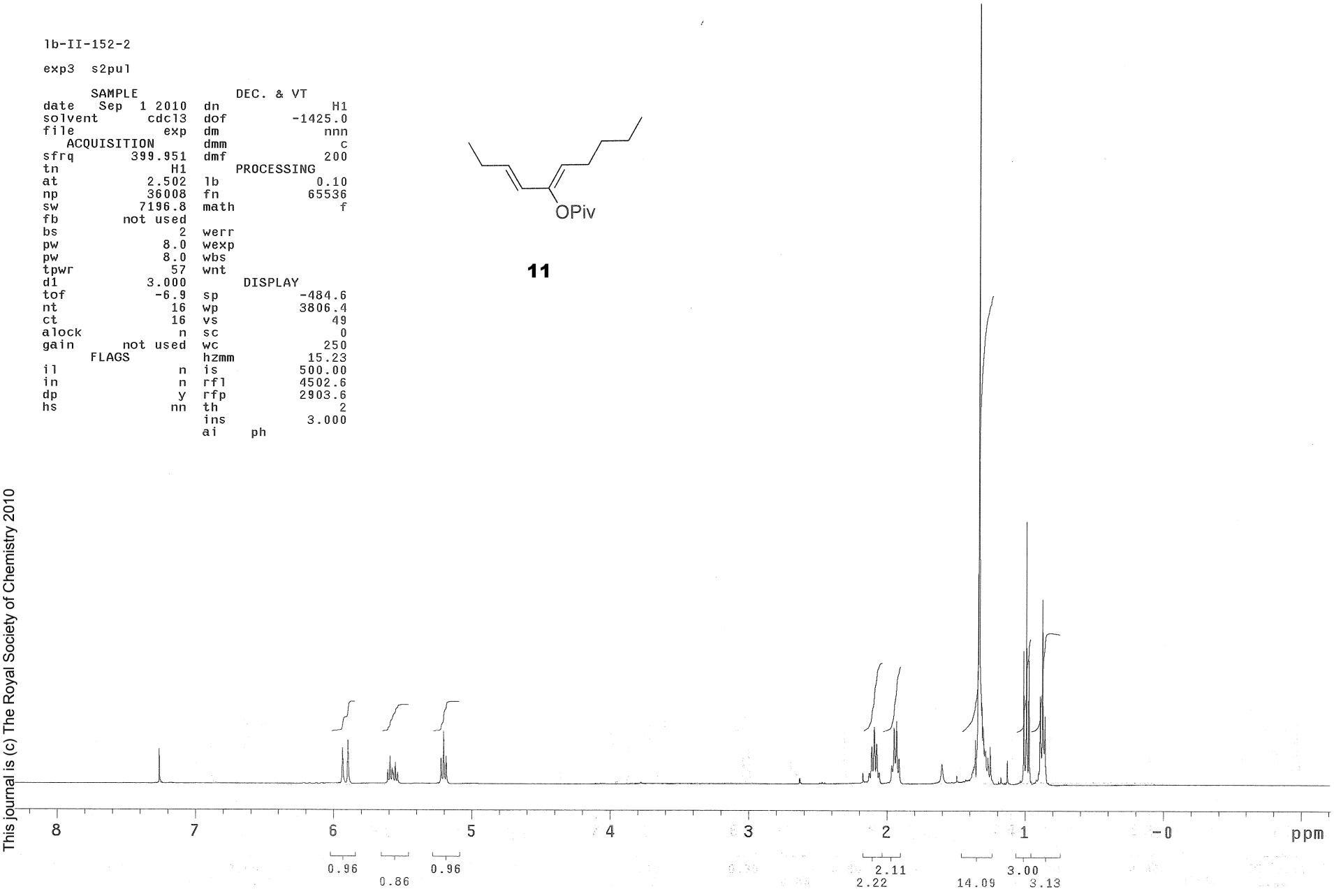

10
H1x ppm
H1y ppm

lb-II-152-2

exp3 s2pul

SAMPLE		DEC. & VT	
date	Sep 1 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	PROCESSING	
at	2.502	lb	0.10
np	36008	fn	65536
sw	7196.8	math	f
fb	not used		
bs	2	werr	
pw	8.0	wexp	
pw	8.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	-6.9	sp	-484.6
nt	16	wp	3806.4
ct	16	vs	49
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	15.23
il	n	is	500.00
in	n	rfl	4502.6
dp	y	rfp	2903.6
hs	nn	th	2
		ins	3.000
		ai ph	

11

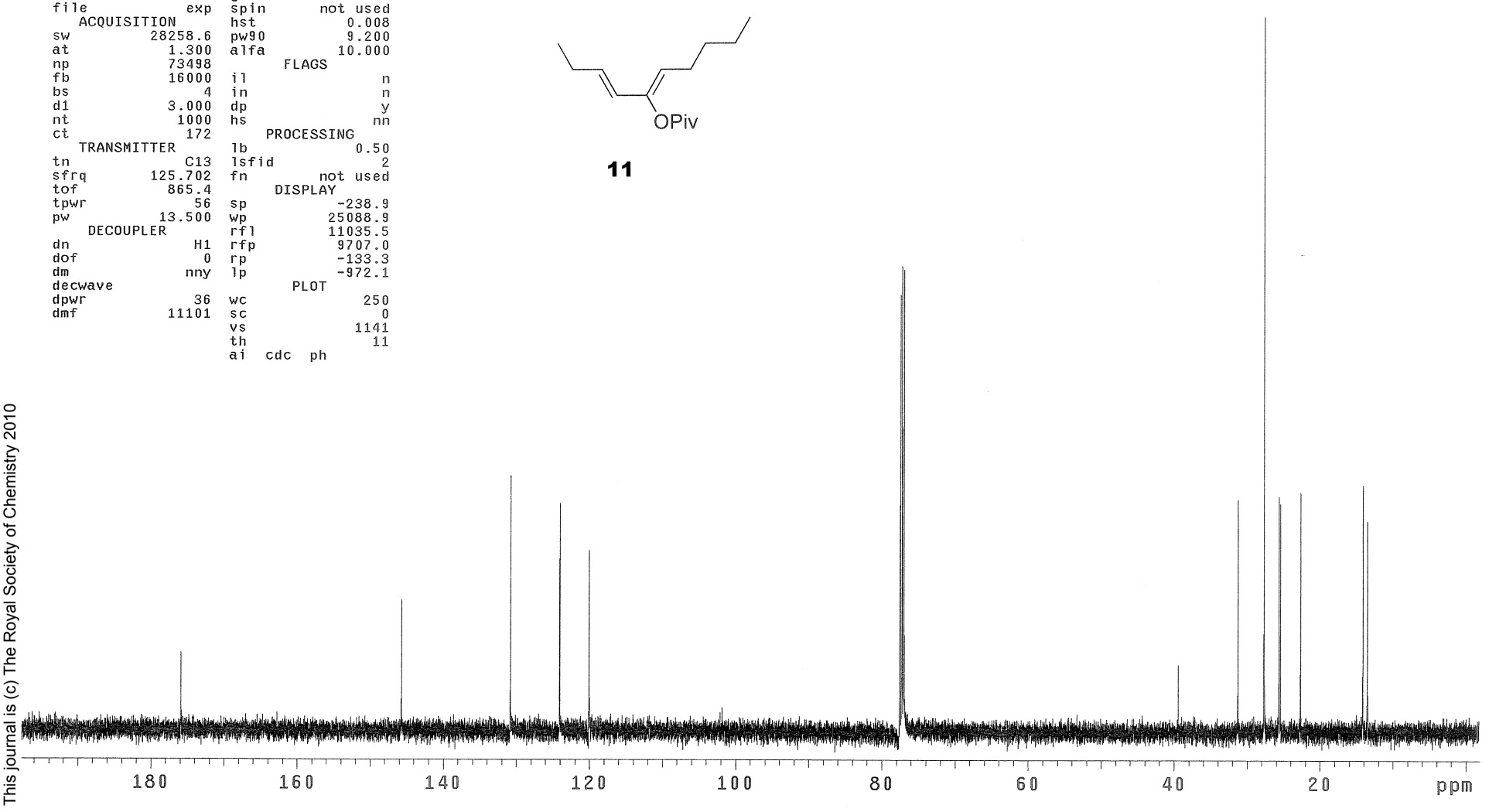


lb-II-152-2-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Sep 1 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	4	in	n
d1	3.000	dp	y
nt	1000	hs	nn
ct	172	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	-238.9
pw	13.500	wp	25088.9
DECOUPLER		rfl	11035.5
dn	H1	rfp	9707.0
dof	0	rp	-133.3
dm	nny	lp	-972.1
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	1141
		th	11
		ai cdc ph	

11

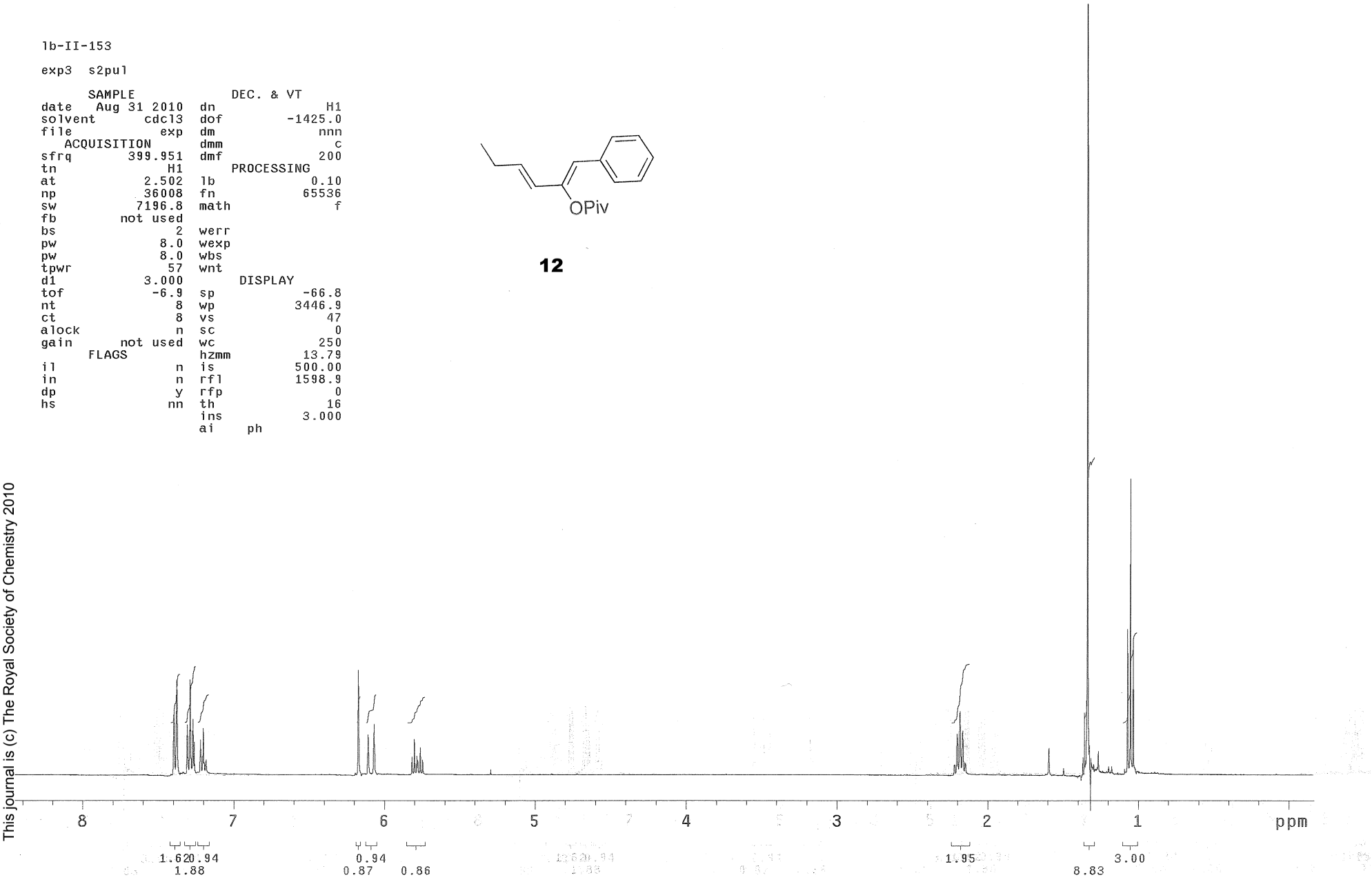


lb-II-153

exp3 s2pul

SAMPLE		DEC. & VT	
date	Aug 31 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	PROCESSING	
at	2.502	lb	0.10
np	36008	fn	65536
sw	7196.8	math	f
fb	not used		
bs	2	werr	
pw	8.0	wexp	
pw	8.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	-6.9	sp	-66.8
nt	8	wp	3446.9
ct	8	vs	47
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	13.79
il	n	is	500.00
in	n	rfl	1598.9
dp	y	rfp	0
hs	nn	th	16
		ins	3.000
		ai ph	

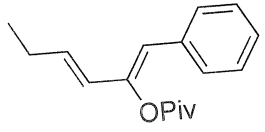
12



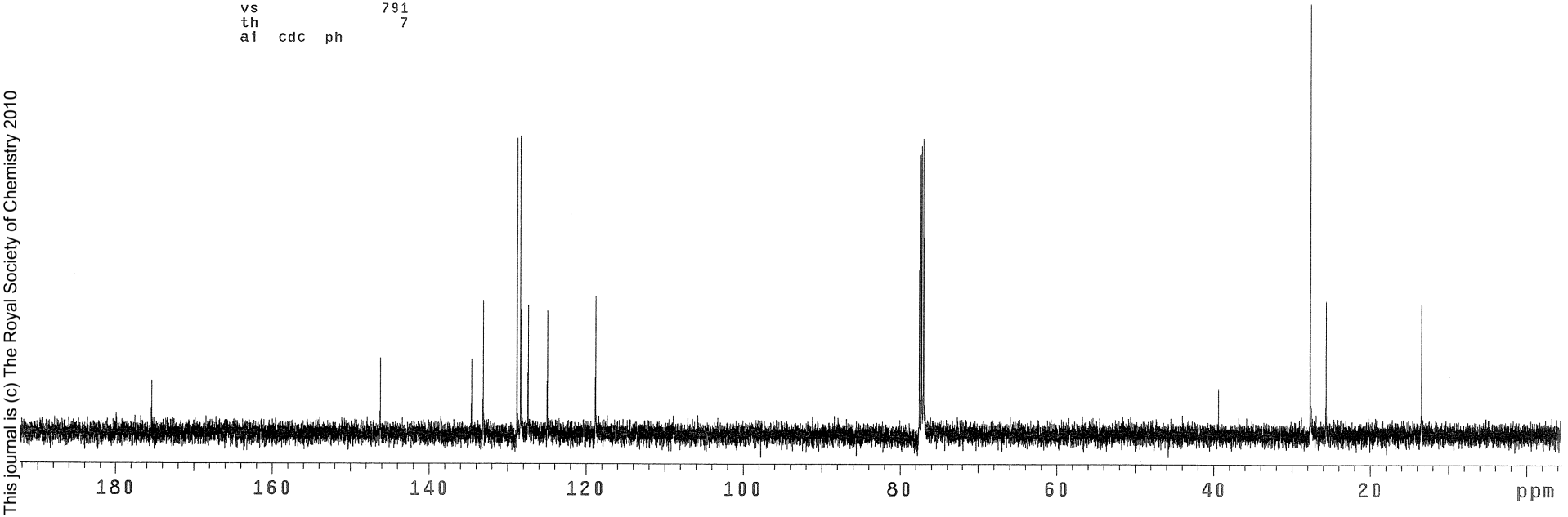
lb-II-153-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Aug 31 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	5	in	n
d1	3.000	dp	y
nt	10000	hs	nn
ct	90	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	-571.7
pw	13.500	wp	24725.8
DECOUPLER		rfl	11038.0
dn	H1	rfp	9707.0
dof	0	rp	145.6
dm	nny	lp	-923.1
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	791
		th	7
		ai cdc ph	



12



lb-II-154-2

exp3 s2pul

SAMPLE		DEC. & VT	
date	Sep 1 2010	dn	H1
solvent	cdcl3	dof	-1425.0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	399.951	dmf	200
tn	H1	PROCESSING	
at	2.502	lb	0.10
np	36008	fn	65536
sw	7196.8	math	f
fb	not used		
bs	2	werr	
pw	8.0	wexp	
pw	8.0	wbs	
tpwr	57	wnt	
d1	3.000	DISPLAY	
tof	-6.9	sp	-214.6
nt	16	wp	3782.9
ct	14	vs	49
alock	n	sc	0
gain	not used	wc	250
FLAGS		hzmm	15.13
il	n	is	500.00
in	n	rfl	4502.6
dp	y	rfp	2903.6
hs	nn	th	2
		ins	3.000
		ai ph	

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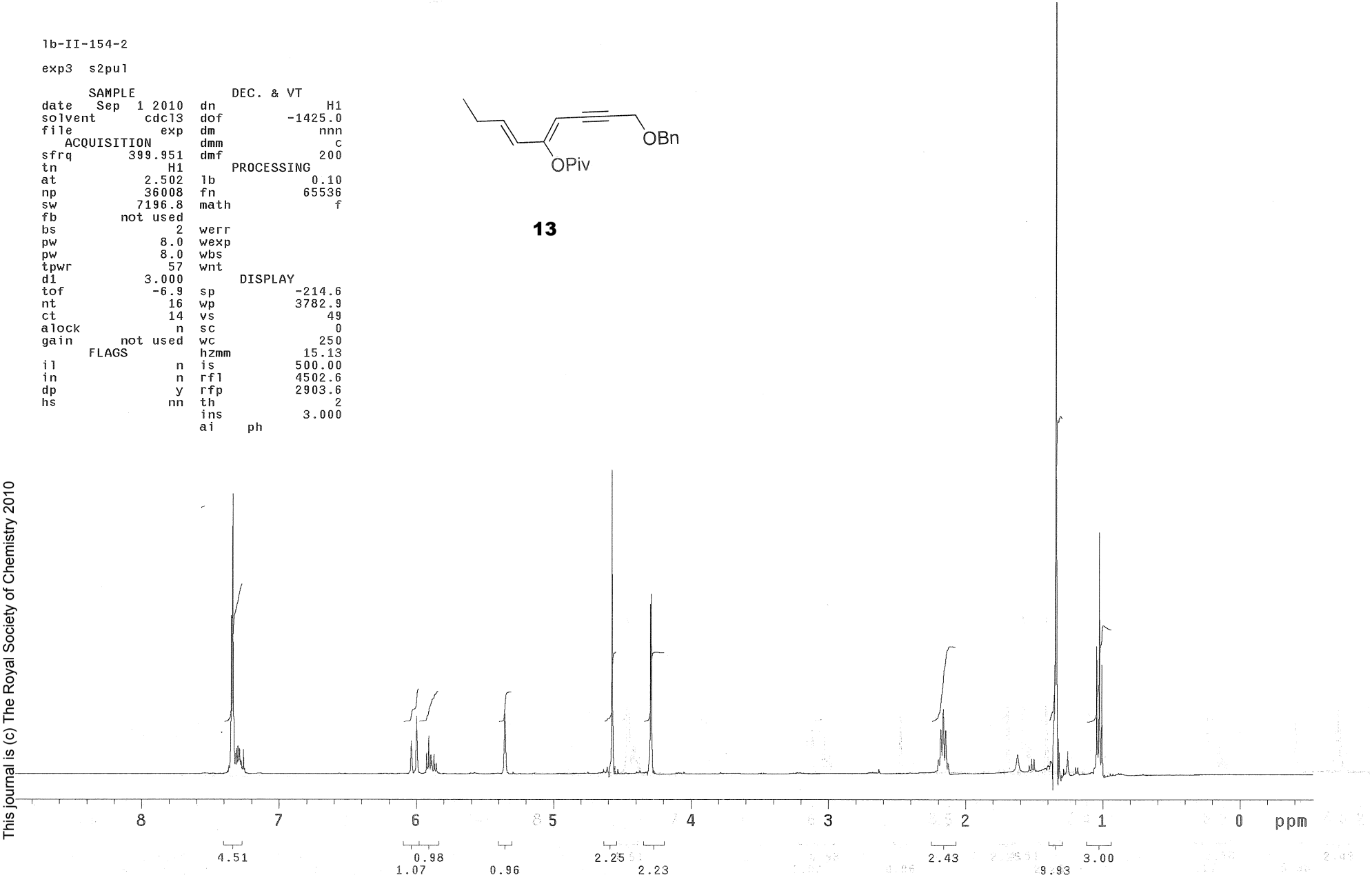
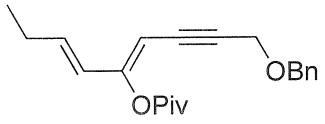


lb-II-154-1-C13

exp1 CARBON

SAMPLE		SPECIAL	
date	Sep 1 2010	temp	not used
solvent	cdcl3	gain	54
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	28258.6	pw90	9.200
at	1.300	alfa	10.000
np	73498	FLAGS	
fb	16000	il	n
bs	4	in	n
d1	3.000	dp	y
nt	10000	hs	nn
ct	792	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	lsfid	2
sfrq	125.702	fn	not used
tof	865.4	DISPLAY	
tpwr	56	sp	-736.9
pw	13.500	wp	25418.7
DECOUPLER		rfl	11038.4
dn	H1	rfp	9707.0
dof	0	rp	-124.4
dm	nny	lp	-979.5
decwave		PLOT	
dpwr	36	wc	250
dmf	11101	sc	0
		vs	722
		th	5
		ai cdc ph	



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