

Supporting Information for

Palladium-Catalyzed [2+1+1] Annulation of Norbornenes with (Z)-Bromostyrenes: Synthesis of Bismethylenecyclobutanes *via* Twofold C(sp²)-H Bond Activation

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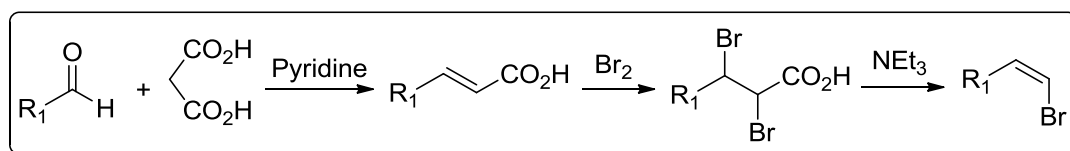
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1 General information

Experimental: All bimethylenecyclobutanation ([2+1+1] cycloaddition) reactions were carried out under an inert atmosphere of nitrogen in sealed tube. All solvents were dried by standard methods before use. All reactions were monitored by TLC with silica gel-coated plates. NMR spectra were recorded on Bruker Avance 400 (400 MHz for ^1H ; 100 MHz for ^{13}C) instruments. Chemical shifts were reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard (for CDCl_3 , ^1H NMR: 7.26 ppm, ^{13}C NMR: 77.16 ppm). Coupling constants (J) were reported in Hz. Mass spectra (EI, 70 eV) were recorded on an Agilent 5975 instrument. High resolution mass spectra (HRMS) were recorded on a Waters Micromass GCT instrument. All commercially available reagents were used as received.

2 Substrates Preparation

2.1 General Procedure for the Preparation of (Z)-Vinyl Bromides¹:



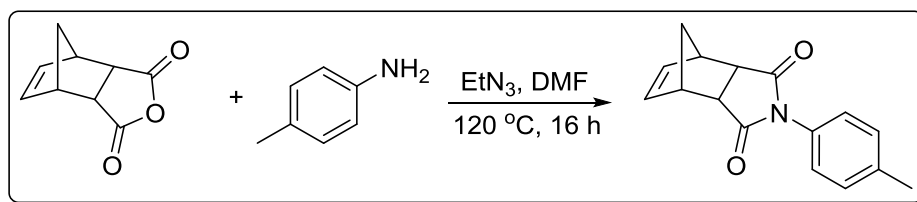
Aldehydes (50 mmol), malonic acid (50 mmol), pyridine (150 mmol), and a few drops of piperidine were added in a 50 mL three-neck flask equipped with a reflux condenser. Firstly the reaction mixture was stirred at 100 °C for 8 h and 120 °C for 2 h, then transferred to a beaker containing 10 mL concentrated hydrochloric acid and 30 mL ice water. After the mixture was cooled, the resultant precipitate was filtered, washed three times with ice water, and recrystallized with ethanol to give pure propenoic acids.

To a mixture of propenoic acids (85 mmol) and chloroform (50 mL) cooled to 0 °C, was added bromine (5.3 mL, 102mmol) dropwise and the resulting solution was stirred at this temperature for 20 min. The solution was stored in refrigerator

overnight, filtered and washed twice with cold chloroform to give the crude product 2,3-dibromopropanoic acid derivatives which was used in the next step without further purification.

Triethylamine (160 mmol, 23 mL) was added to the mixture of 2,3-dibromopropanoic acid derivatives (80 mmol) and dry DMF (40mL) at 0 °C dropwise. The solution was stirred at 0 °C for 30 min, then at room temperature for 6 h. Water (20mL) was added. The mixture was extracted with ethoxyethane (3 x 40mL). The organic layers were combined, washed with saturated potassium carbonate (2 x 40mL) and saturated sodium chloride(2 x 40mL), dried over magnesium sulfate and concentrated *in vacuo*, purified by chromatography on a column of silica gel with PE/EA=100/1 to give (z)-vinyl bromides.

2.2 The Synthesis of *Endo*-Norbornenesuccinimides (2a, 2b)²:



Triethylamine (6.6 mmol, 0.92 mL) and the desired anhydride (6 mmol) were added to a solution of 4-amino acid 6 (6 mmol) in 5 mL of N, N-dimethylformamide (DMF). The solution was heated for 16 h at 120 °C. After it returned to room temperature, the resulting mixture was treated with water, and extracted with ethyl ether and washed with 1 N HCl (20 mL). The combined organic layer was washed with brine, dried over magnesium sulfate and concentrated *in vacuo*, purified by chromatography on a column of silica gel with PE/EA=3/1 to afford the pure products as white solid(86% yield). ¹H NMR (CDCl₃, 400 MHz) δ 7.25 (d, *J* = 8.0 Hz, 2H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.28 (s, 2H), 3.52 (s, 2H), 3.43-3.44 (m, 2H), 2.38 (s, 3H), 1.80 (d, *J* = 8.8 Hz, 2H), 1.62 (d, *J* = 8.8 Hz, 2H).

3 Pd(OAc)₂-Catalyzed [2+1+1] Cycloaddition of (Z)-Vinyl Bromides and Norbornene Derivatives

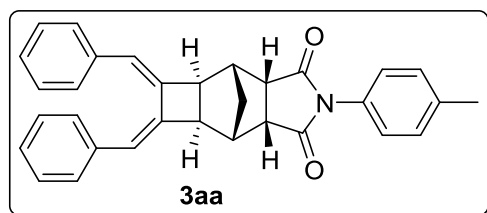
3.1 General Procedures for the [2+1+1] Cycloaddition

Synthesis of [2+1+1] Cycloaddition products 3

To a flame-dried Teflon-screw-capped tube was equipped with a magnetic stir bar, (Z)-vinyl bromides **1** (1.1 mmol, 2.2 equiv.), norbornene derivatives **2** (0.5 mmol, 1.0 equiv.), Pd(OAc)₂ (11.22 mg, 0.05 mmol, 10mol%), PPh₃ (28.85 mg, 0.11 mmol, 22 mol%), Cs₂CO₃ (488.73 mg, 1.5 mmol, 3.0 equiv.) and toluene (2.0 mL) were added sequentially under nitrogen. The tube was sealed with a Teflon lined cap, the reaction mixture was stirred at 110 °C for 12 h. After completion of the reaction, the resulting mixture was cooled down to room temperature, diluted with CH₂Cl₂ (10 mL), filtered through a short pad of silica gel and washed with EtOAc (30 mL). The filtrate was concentrated under vacuum and the residue was purified by silica gel column chromatography to afford the corresponding products **3**.

3.2 Experimental Characterization of Products

(3^aR,4S,4^aR,5Z,6Z,6^aS,7R,7^aS)-5,6-dibenzylidene-2-(*p*-tolyl)octahydro-1*H*-4,7-methanocyclobuta[*f*]isoindole-1,3(2*H*)-dione

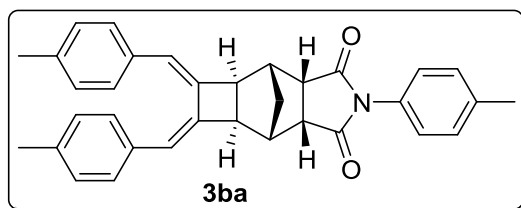


(3aa): The title compound was prepared according to the general procedure and purified by flash column chromatography on

silica gel with a gradient eluent of petroleum ether/EtOAc (10/1→3/1) to give white solid, 196.6 mg, 86 % yield. **Mp:** 264-266°C; **¹H NMR** (400 MHz, CDCl₃) δ 7.31 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 6.95 (t, *J* = 7.2 Hz, 2H), 6.75 (t, *J* = 7.6 Hz, 4H), 6.66 (d, *J* = 7.2 Hz, 4H), 6.33 (s, 2H), 3.38 (s, 2H), 3.24 (s, 2H), 3.05 (s, 2H), 2.41 (s, 3H), 2.36 (d, *J* = 11.2 Hz, 1H), 1.64 (d, *J* = 10.8 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 176.8, 140.3, 138.8, 136.8, 129.9, 129.0, 127.9, 126.6, 126.5, 126.5, 124.8, 48.0, 43.5, 43.2, 36.0, 21.2; **HRMS (EI)** calcd. for C₃₂H₂₇NO₂ [M⁺]: 457.2042,

found: 457.2039.

(3^aR,4S,4^aR,5Z,6Z,6^aS,7R,7^aS)-5,6-bis(4-methylbenzylidene)-2-(*p*-tolyl)octahydro



-1*H*-4,7-methanocyclobuta[*f*]isoindole-1

,3(2*H*)-dione (3ba): The title compound was prepared according to the general procedure and purified by flash column

chromatography on silica gel with a gradient eluent of petroleum ether/EtOAc (10/1 → 3/1) to give white solid, 201.4 mg, 83 % yield. **Mp:** 258-260°C; **¹H NMR** (400 MHz, CDCl₃) δ 7.31 (d, *J* = 7.6 Hz, 2H), 7.19 (d, *J* = 8.4 Hz, 2H), 6.55 (d, *J* = 8.4 Hz, 4H), 6.51 (d, *J* = 8.4 Hz, 4H), 6.27 (s, 2H), 3.36 (s, 2H), 3.21 (s, 2H), 3.02 (s, 2H), 2.41 (s, 3H), 2.37 (d, *J* = 10.4 Hz, 1H), 2.18 (s, 6H), 1.62 (d, *J* = 10.0 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 176.8, 139.7, 138.7, 136.4, 134.2, 129.8, 129.2, 127.8, 127.1, 126.5, 124.4, 48.0, 43.3, 43.2, 36.0, 21.1, 21.0; **HRMS (EI)** calcd. for C₃₄H₃₁NO₂ [M⁺]: 485.2355, found: 485.2358. The configuration was confirmed by X-ray analysis (Figure S1) and undoubtedly determined that bismethenylcyclobutane moiety was formed.

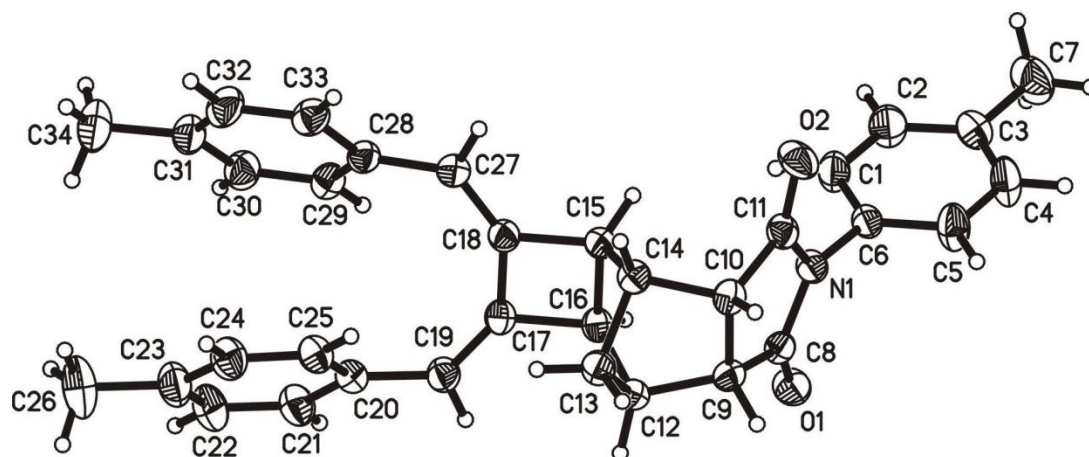
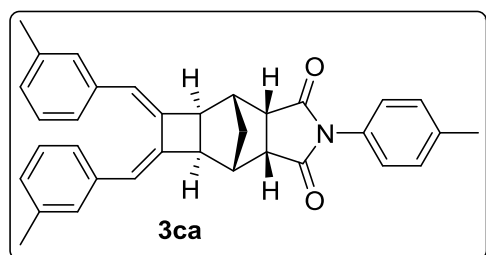


Figure S1. ORTEP drawing of product 3ba

(3^aR,4S,4^aR,5Z,6Z,6^aS,7R,7^aS)-5,6-bis(3-methylbenzylidene)-2-(*p*-tolyl)octahydro

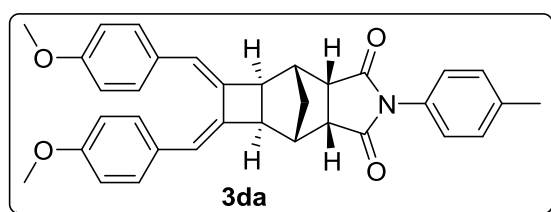


-1*H*-4,7-methanocyclobuta[*f*]isoindole-1,3(

2*H*)-dione (3ca): The title compound was prepared according to the general procedure and purified by flash column

chromatography on silica gel with a gradient eluent of petroleum ether/EtOAc (10/1 → 3/1) to give white solid, 186.8 mg, 77 % yield. **Mp:** 242-245°C; **¹H NMR** (400 MHz, CDCl₃) δ 7.31 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 6.80 (d, *J* = 4.4 Hz, 4H), 6.65 (s, 2H), 6.37 (s, 2H), 6.29 (s, 2H), 3.37 (s, 2H), 3.23 (s, 2H), 3.04 (s, 2H), 2.41 (s, 3H), 2.35 (d, *J* = 11.2 Hz, 1H), 1.83 (s, 6H), 1.63 (d, *J* = 10.8 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 176.8, 140.0, 138.7, 136.7, 136.5, 129.8, 129.2, 129.1, 127.4, 126.6, 126.5, 125.3, 124.9, 48.0, 43.5, 43.2, 36.0, 21.1, 20.7; **HRMS (EI)** calcd. for C₃₄H₃₁NO₂ [M⁺]: 485.2355, found: 485.2354.

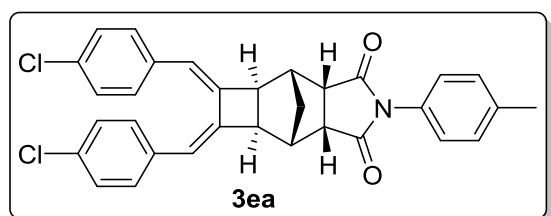
(3^aR,4S,4^aR,5Z,6Z,6^aS,7R,7^aS)-5,6-bis(4-methoxybenzylidene)-2-(*p*-tolyl)octahydro-



ro-1H-4,7-methanocyclobuta[f]isoindole-1,3(2H)-dione (3da): The title compound was prepared according to the general procedure and purified by

flash column chromatography on silica gel with a gradient eluent of petroleum ether/EtOAc (10/1 → 3/1) to give white solid, 186.2 mg, 72 % yield. **Mp:** 255-257°C; **¹H NMR** (400 MHz, CDCl₃) δ 7.31 (d, *J* = 8.4 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 6.55 (d, *J* = 8.8 Hz, 4H), 6.31 (d, *J* = 8.4 Hz, 4H), 6.25 (s, 2H), 3.68 (s, 6H), 3.36 (s, 2H), 3.21 (s, 2H), 3.01 (s, 2H), 2.41 (s, 3H), 2.34 (d, *J* = 10.8 Hz, 1H), 1.62 (d, *J* = 10.8 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 176.9, 158.9, 139.0, 138.8, 129.9, 129.8, 129.1, 129.1, 126.5, 123.5, 111.8, 54.9, 48.0, 43.3, 43.1, 36.0, 21.2; **HRMS (EI)** calcd. for C₃₄H₃₁NO₄ [M⁺]: 517.2253, found: 517.2258.

(3^aR,4S,4^aR,5Z,6Z,6^aS,7R,7^aS)-5,6-bis(4-chlorobenzylidene)-2-(*p*-tolyl)octahydro-

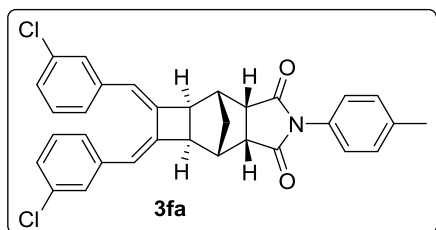


1H-4,7-methanocyclobuta[f]isoindole-1,3(2H)-dione (3ea): The title compound was prepared according to the general procedure and purified by flash

column chromatography on silica gel with a gradient eluent of petroleum ether/EtOAc (10/1 → 3/1) to give white solid, 246.8 mg, 94 % yield. **Mp:** 298-301°C; **¹H NMR** (400 MHz, CDCl₃) δ 7.31 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 6.76 (d, *J* = 8.8 Hz, 4H), 6.57 (d, *J* = 8.4 Hz, 4H), 6.26 (s, 2H), 3.37 (s, 2H), 3.22 (s, 2H), 3.03 (s,

2H), 2.41 (s, 3H), 2.30 (d, $J = 10.8$ Hz, 1H), 1.66 (d, $J = 10.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.7, 141.0, 138.9, 135.1, 132.9, 129.9, 129.0, 128.9, 126.8, 126.4, 123.7, 54.9, 48.0, 43.3, 43.1, 36.0, 21.2; **HRMS (EI)** calcd. for $\text{C}_{32}\text{H}_{25}\text{Cl}_2\text{NO}_2$ [M^+]: 525.1262, found: 525.1258.

(3^aR,4S,4^aR,5Z,6Z,6^aS,7R,7^aS)-5,6-bis(3-chlorobenzylidene)-2-(p-tolyl)octahydro-

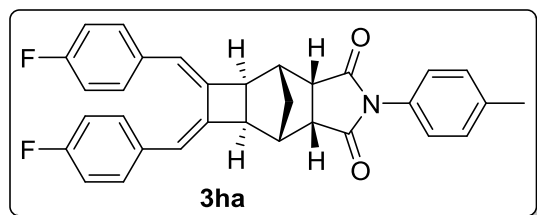


1H-4,7-methanocyclobuta[f]isoindole-1,3(2H)-

dione (3fa): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel

with a gradient eluent of petroleum ether/EtOAc (10/1→3/1) to give white solid, 238.9 mg, 91 % yield. **Mp:** 263-265°C; ^1H NMR (400 MHz, CDCl_3) δ 7.31 (d, $J = 7.6$ Hz, 2H), 7.18 (d, $J = 7.6$ Hz, 2H), 6.97 (d, $J = 8.0$ Hz, 2H), 6.79 (t, $J = 7.6$ Hz, 2H), 6.65 (d, $J = 7.6$ Hz, 2H), 6.60 (s, 2H), 6.27 (s, 2H), 3.39 (s, 2H), 3.24 (s, 2H), 3.05 (s, 2H), 2.41 (s, 3H), 2.30 (d, $J = 10.8$ Hz, 1H), 1.67 (d, $J = 10.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.7, 141.4 138.9, 138.4, 133.1, 129.9, 129.0, 128.1, 127.8, 126.8, 126.4, 125.9, 123.8, 47.9, 43.5, 43.1, 36.0, 21.2; **HRMS (EI)** calcd. for $\text{C}_{32}\text{H}_{25}\text{Cl}_2\text{NO}_2$ [M^+]: 525.1262, found: 525.1263.

(3^aR,4S,4^aR,5Z,6Z,6^aS,7R,7^aS)-5,6-bis(4-fluorobenzylidene)-2-(p-tolyl)octahydro-



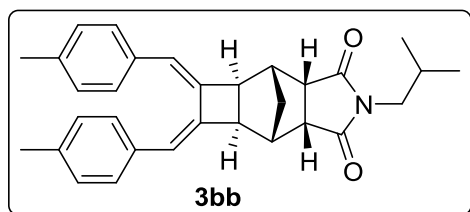
1H-4,7-methanocyclobuta[f]isoindole-1

,3(2H)-dione (3ha): The title compound was prepared according to the general procedure and purified by flash column

chromatography on silica gel with a gradient eluent of petroleum ether/EtOAc (10/1 → 3/1) to give white solid, 236.7 mg, 96 % yield. **Mp:** 297-299°C; ^1H NMR (400 MHz, CDCl_3) δ 7.31 (d, $J = 8.4$ Hz, 2H), 7.18 (d, $J = 8.4$ Hz, 2H), 6.65 (dd, $J_1 = 8.4$ Hz, $J_2 = 13.6$ Hz, 4H), 6.51 (t, $J = 8.8$ Hz, 4H), 6.26 (s, 2H), 3.38 (t, $J = 2.8$ Hz, 2H), 3.22 (s, 2H), 3.03 (s, 2H), 2.41 (s, 3H), 2.31 (d, $J = 10.8$ Hz, 1H), 1.66 (d, $J = 10.8$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 176.7, 162.0 (d, $J_{\text{C-F}} = 247.3$ Hz), 140.2, 138.9, 132.8 (d, $J_{\text{C-F}} = 3.4$ Hz), 129.9, 129.4 (d, $J_{\text{C-F}} = 8.9$ Hz), 129.0, 126.4, 123.5, 113.5 (d, $J_{\text{C-F}} = 21$ Hz), 47.9, 43.4, 43.1, 36.0, 21.2; **HRMS (EI)** calcd. for $\text{C}_{32}\text{H}_{25}\text{F}_2\text{NO}_2$ [M^+]:

493.1853, found: 493.1852.

(3^aR,4S,4^aR,6^aS,7R,7^aS)-5,6-bis(4-methylbenzylidene)-2-isobutyloctahydro-1H-4,

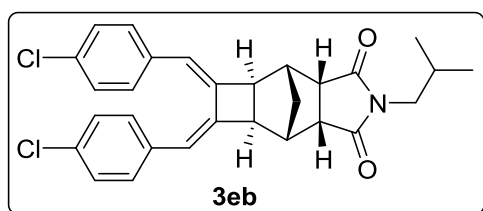


7-methanocyclobuta[f]isoindole-1,3(2H)-dione (3bb):

The title compound was prepared according to the general procedure and purified by flash column chromatography on

silica gel with a gradient eluent of petroleum ether/EtOAc (10/1→3/1) to give white solid, 180.5 mg, 80 % yield. **Mp:** 200-203°C; **¹H NMR** (400 MHz, CDCl₃) δ 6.53 (d, *J* = 8.4 Hz, 4H), 6.50 (d, *J* = 8.0 Hz, 4H), 6.24 (s, 2H), 3.34 (d, *J* = 6.0 Hz, 2H), 3.20 (s, 2H), 3.02 (s, 2H), 2.91 (s, 2H), 2.30 (d, *J* = 10.4 Hz, 1H), 2.17 (s, 6H), 2.05-2.12 (m, 1H), 1.56 (d, *J* = 10.8 Hz, 1H), 0.95 (d, *J* = 6.8 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 178.7, 140.6, 137.1, 135.0, 128.5, 127.8, 125.0, 48.7, 46.8, 44.1, 43.3, 36.8, 27.9, 21.7, 21.1; **HRMS (EI)** calcd. for C₃₁H₃₃NO₂ [M⁺]: 451.2511, found: 451.2505.

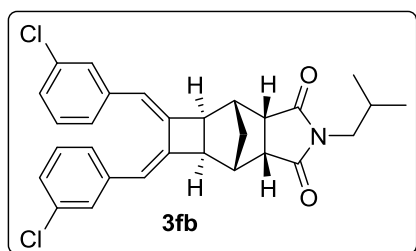
(3^aR,4S,4^aR,6^aS,7R,7^aS)-5,6-bis(4-chlorobenzylidene)-2-isobutyloctahydro-1H-4,7



-methanocyclobuta[f]isoindole-1,3(2H)-dione (3eb):

The title compound was prepared according to the general procedure and purified by flash column chromatography on

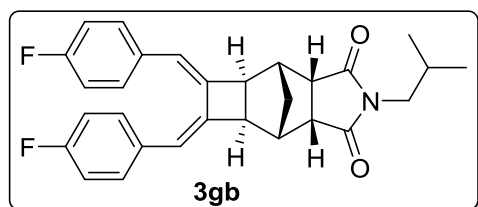
silica gel with a gradient eluent of petroleum ether/EtOAc (10/1→3/1) to give light yellow solid, 221.0 mg, 90 % yield. **Mp:** 221-224°C; **¹H NMR** (400 MHz, CDCl₃) δ 6.75 (d, *J* = 8.4 Hz, 4H), 6.54 (d, *J* = 8.4 Hz, 4H), 6.24 (s, 2H), 3.34 (d, *J* = 6.0 Hz, 2H), 3.21 (s, 2H), 3.03 (s, 2H), 2.92 (s, 2H), 2.25 (d, *J* = 10.4 Hz, 1H), 2.04-2.11 (m, 1H), 1.60 (d, *J* = 10.4 Hz, 1H), 0.95 (d, *J* = 6.4 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 177.7, 141.1, 135.2, 132.9, 128.9, 126.7, 123.5, 47.8, 46.1, 43.4, 42.5, 36.0, 27.1, 20.3; **HRMS (EI)** calcd. for C₂₉H₂₇Cl₂NO₂ [M⁺]: 491.1419, found: 491.1426.



(3^aR,4S,4^aR,6^aS,7R,7^aS)-5,6-bis(3-chlorobenzylidene)-2-isobutyloctahydro-1H-4,7-methanocyclobuta[f]isoindole-1,3(2H)-dione (3fb): The title compound was prepared according to the general

procedure and purified by flash column chromatography on silica gel with a gradient eluent of petroleum ether/EtOAc (10/1→3/1) to give white solid, 203.8 mg, 83 % yield. **Mp:** 217-221°C; **¹H NMR** (400 MHz, CDCl₃) δ 6.93 (s, 2H), 6.77 (s, 2H), 6.63 (s, 2H), 6.57 (s, 2H), 6.23 (s, 2H), 3.33 (d, *J* = 4.8 Hz, 2H), 3.21 (s, 2H), 3.04 (s, 2H), 2.93 (s, 2H), 2.24 (d, *J* = 8.8 Hz, 1H), 2.06-2.08 (m, 1H), 1.56 (d, *J* = 7.6 Hz, 1H), 0.94 (d, *J* = 3.6 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 177.8, 141.6, 138.6, 133.2, 128.2, 127.9, 126.9, 126.0, 123.8, 48.0, 46.2, 43.7, 42.6, 36.2, 27.2, 20.4; **HRMS (EI)** calcd. for C₂₉H₂₇Cl₂NO₂ [M⁺]: 491.1419, found: 491.1424.

(3^aR,4S,4^aR,6^aS,7R,7^aS)-5,6-bis(4-fluorobenzylidene)-2-isobutyloctahydro-1H-4,7

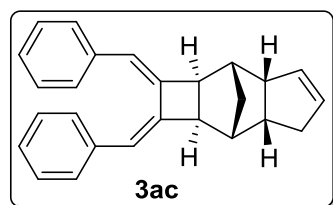


-methanocyclobuta[f]isoindole-1,3(2H)-dio

ne (3gb): The title compound was prepared according to the general procedure and purified by flash column chromatography on

silica gel with a gradient eluent of petroleum ether/EtOAc (10/1→3/1) to give white solid, 195.2 mg, 85 % yield. **Mp:** 228-232°C; **¹H NMR** (400 MHz, CDCl₃) δ 6.61 (t, *J* = 7.2 Hz, 4H), 6.48 (t, *J* = 8.2 Hz, 4H), 6.22 (s, 2H), 3.33 (d, *J* = 7.2 Hz, 2H), 3.20 (s, 2H), 3.02 (s, 2H), 2.91 (s, 2H), 2.25 (d, *J* = 10.8 Hz, 1H), 2.04-2.12 (m, 1H), 1.58 (d, *J* = 14.4 Hz, 1H), 0.94 (d, *J* = 14.4 Hz, 6H); **¹³C NMR** (100 MHz, CDCl₃) δ 177.8, 162.2 (d, *J*_{C-F} = 253.1 Hz), 140.4, 133.0 (d, *J*_{C-F} = 3.0 Hz), 129.5 (d, *J*_{C-F} = 7.7 Hz), 123.5, 113.6 (d, *J*_{C-F} = 22.1 Hz), 48.0, 46.2, 43.5, 42.7, 36.1, 27.2, 20.4; **HRMS (EI)** calcd. for C₂₉H₂₇F₂NO₂ [M⁺]: 459.2010, found: 459.2010.

(2^aR,3R,3^aR,6^aR,7S,7^aS)-1,2-bis((Z)-benzylidene)-2,2^a,3,3^a,4,6^a,7,7^a-octahydro-1H

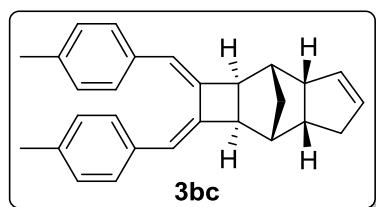


-3,7-methanocyclobuta[f]indene (3ac): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel with a eluent of petroleum ether to give

white solid, 131.1 mg, 78 % yield. **Mp:** 132-135°C; **¹H NMR** (400 MHz, CDCl₃) δ 6.81 (t, *J* = 7.2 Hz, 2H), 6.57-6.65 (m, 8H), 6.12 (s, 2H), 5.60 (s, 1H), 5.54 (s, 1H), 3.08-3.10 (m, 1H), 2.96 (d, *J* = 5.6 Hz, 1H), 2.83 (d, *J* = 6.0 Hz, 1H), 2.58-2.63 (m, 1H), 2.41 (d, *J* = 4.0 Hz, 1H), 2.01-2.30 (m, 3H), 1.98 (d, *J* = 10.4 Hz, 1H), 1.32 (d, *J*

= 10.0 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.7, 142.2, 136.7, 130.5, 129.9, 126.8, 126.8, 125.5, 125.0, 122.1, 121.9, 51.9, 44.1, 44.1, 42.3, 41.3, 41.1, 34.6, 30.7; **HRMS (EI)** calcd. for $\text{C}_{26}\text{H}_{24}$ [M^+]: 336.1878, found: 336.1874.

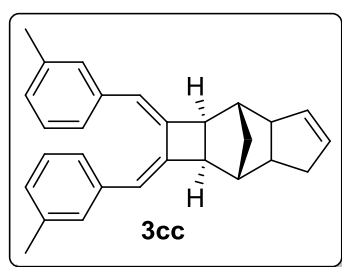
(2^aR,3R,3^aR,6^aR,7S,7^aS)-1,2-bis((Z)-4-methylbenzylidene)-2,2^a,3,3^a,4,6^a,7,7^a-octahydro-1H-3,7-methanocyclobuta[f]indene (3bc):



ydro-1H-3,7-methanocyclobuta[f]indene (3bc): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel with a eluent of petroleum ether to give pale yellow solid, 97.8 mg, 74 % yield. **Mp:** 138-142°C; ^1H

NMR (400 MHz, CDCl_3) δ 6.43 (t, J = 15.2 Hz, 8H), 6.06 (s, 2H), 5.59 (dd, J = 5.2 Hz, J = 1.6 Hz, 1H), 5.53 (dd, J = 4.8 Hz, J = 1.6 Hz, 1H), 3.06-3.09 (m, 1H), 2.92 (d, J = 6.0 Hz, 1H), 2.80 (d, J = 6.0 Hz, 1H), 2.55-2.62 (m, 1H), 2.38 (d, J = 4.0 Hz, 1H), 2.19-2.27 (m, 3H), 2.07 (s, 6H), 1.98 (d, J = 10.0 Hz, 1H), 1.30 (d, J = 10.0 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.1, 141.6, 134.7, 134.7, 134.0, 130.5, 129.8, 126.7, 126.7, 126.0, 121.7, 121.5, 51.9, 44.1, 44.0, 42.3, 41.2, 41.1, 34.5, 30.7, 20.0; **HRMS (EI)** calcd. for $\text{C}_{28}\text{H}_{28}$ [M^+]: 364.2191, found: 364.2192.

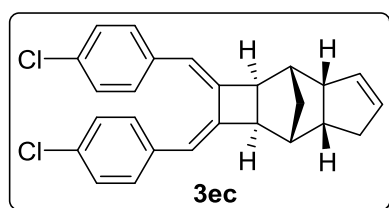
(2^aR,3R,3^aR,6^aR,7S,7^aS)-1,2-bis((Z)-3-methylbenzylidene)-2,2^a,3,3^a,4,6^a,7,7^a-octahydro-1H-3,7-methanocyclobuta[f]indene (3cc):



ydro-1H-3,7-methanocyclobuta[f]indene (3cc): The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel with a eluent of petroleum ether to give pale yellow solid, 91.2 mg, 69 % yield. **Mp:** 135-139°C; ^1H

NMR (400 MHz, CDCl_3) δ 6.65-6.72 (m, 4H), 6.56 (d, J = 7.2 Hz, 2H), 6.30 (s, 2H), 6.09 (s, 2H), 5.61 (d, J = 3.6 Hz, 1H), 5.54 (d, J = 1.6 Hz, 1H), 3.07-3.10 (m, 1H), 2.94 (d, J = 6.0 Hz, 1H), 2.82 (d, J = 4.8 Hz, 1H), 2.56-2.63 (m, 1H), 2.41 (d, J = 4.8 Hz, 1H), 2.20-2.27 (m, 3H), 1.73 (s, 6H), 1.97 (d, J = 10.4 Hz, 1H), 1.31 (d, J = 10.0 Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 142.5, 142.0, 136.6, 135.4, 130.5, 129.9, 128.2, 128.2, 125.8, 125.5, 124.2, 124.2, 122.2, 122.0, 51.9, 44.1, 44.1, 42.3, 41.3, 41.1, 34.5, 30.7, 19.9; **HRMS (EI)** calcd. for $\text{C}_{28}\text{H}_{28}$ [M^+]: 264.2191, found: 264.2197.

(2^aR,3R,3^aR,6^aR,7S,7^aS)-1,2-bis((Z)-4-chlorobenzylidene)-2,2^a,3,3^a,4,6^a,7,7^a-octahydro-1H-3,7-methanocyclobuta[f]indene (3ec):

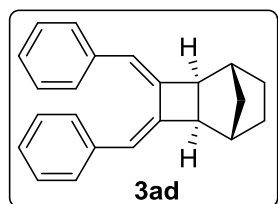


hydro-1H-3,7-methanocyclobuta[f]indene (3ec):

The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel with a eluent of petroleum ether to give pale yellow solid, 161.6 mg, 80 % yield. **Mp:** 174-179°C; **¹H**

NMR (400 MHz, CDCl₃) δ 6.72 (d, *J* = 8.4 Hz, 4H), 6.55 (d, *J* = 8.4 Hz, 4H), 6.12 (s, 2H), 5.67 (dd, *J* = 5.6 Hz, *J* = 1.6 Hz, 1H), 5.60 (dd, *J* = 4.8 Hz, *J* = 1.6 Hz, 1H), 3.15-3.16 (m, 1H), 3.01 (d, *J* = 6.0 Hz, 1H), 2.89 (d, *J* = 6.0 Hz, 1H), 2.63-2.70 (m, 1H), 2.47 (d, *J* = 4.0 Hz, 1H), 2.28-2.31 (m, 3H), 1.99 (d, *J* = 10.4 Hz, 1H), 1.81 (d, *J* = 10.0 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 144.5, 144.0, 136.1, 132.3, 132.3, 131.4, 131.0, 128.9, 128.9, 126.8, 122.0, 121.8, 52.8, 45.2, 45.1, 43.3, 42.3, 42.1, 35.6, 31.7; **HRMS (EI)** calcd. for C₂₆H₂₂Cl₂ [M⁺]: 404.1099, found: 404.1101.

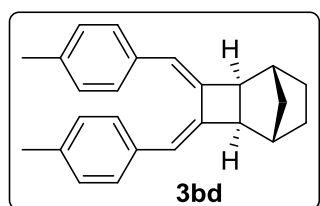
(1R,2S,5R,6S)-3,4-bis((Z)-benzylidene)tricyclo[4.2.1.0^{2,5}]nonane (3ad): The title



compound was prepared according to the general procedure and purified by flash column chromatography on silica gel with a eluent of petroleum ether to give pale yellow solid, 108.8 mg, 73 % yield. **Mp:** 88-92°C; **¹H NMR** (400 MHz,

CDCl₃) δ 6.82 (t, *J* = 6.8 Hz, 2H), 6.59-6.66 (m, 8H), 6.17 (s, 2H), 2.83 (s, 2H), 2.29 (s, 2H), 1.81 (d, *J* = 10.0 Hz, 1H), 1.49 (d, *J* = 8.4 Hz, 2H), 1.14 (d, *J* = 8.4 Hz, 2H), 1.07 (d, *J* = 10.0 Hz, 1H); **¹³C NMR** (100 MHz, CDCl₃) δ 142.1, 136.6, 126.9, 125.5, 125.0, 122.4, 47.8, 39.6, 31.3, 27.1; **HRMS (EI)** calcd. for C₂₃H₂₂ [M⁺]: 298.1722, found: 298.1725.

(1R,2S,5R,6S)-3,4-bis((Z)-4-methylbenzylidene)tricyclo[4.2.1.0^{2,5}]nonane (3bd):

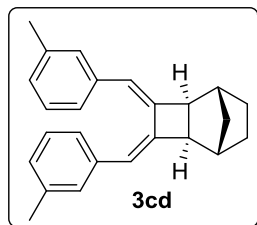


The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel with a eluent of petroleum ether to give pale yellow solid, 114.2 mg, 70 % yield. **Mp:** 102-106°C; **¹H**

NMR (400 MHz, CDCl₃) δ 6.45 (t, *J* = 8.4 Hz, 8H), 6.12 (s, 2H), 2.80 (s, 2H), 2.26 (s, 2H), 2.08 (s, 6H), 1.81 (d, *J* = 10.0 Hz, 1H), 1.48 (d, *J* = 8.4 Hz, 2H), 1.10 (dd, *J* = 7.2

Hz, $J = 1.6$ Hz, 2H), 1.06 (d, $J = 10.0$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 141.6, 134.8, 134.0, 126.7, 126.1, 122.0, 47.7, 39.6, 31.3, 27.1, 20.0; HRMS (EI) calcd. for $\text{C}_{25}\text{H}_{26}$ [M^+]: 326.2035, found: 326.2036.

(1R,2S,5R,6S)-3,4-bis((Z)-3-methylbenzylidene)tricyclo[4.2.1.0^{2,5}]nonane (3cd):



The title compound was prepared according to the general procedure and purified by flash column chromatography on silica gel with a eluent of petroleum ether to give pale yellow solid, 106.0 mg, 65 % yield. **Mp:** 95-99°C; ^1H NMR (400

MHz, CDCl_3) δ 6.66-6.72 (m, 4H), 6.58 (d, $J = 7.2$ Hz, 2H), 6.32 (s, 2H), 6.15 (s, 2H), 2.82 (s, 2H), 2.28 (s, 2H), 1.80 (d, $J = 10.4$ Hz, 1H), 1.74 (s, 6H), 1.50 (d, $J = 10.0$ Hz, 2H), 1.14 (dd, $J = 7.2$ Hz, $J = 1.6$ Hz, 2H), 1.07 (d, $J = 10.4$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 141.9, 136.5, 135.4, 128.3, 125.9, 125.6, 124.3, 122.5, 47.8, 39.6, 31.3, 27.1, 19.9; HRMS (EI) calcd. for $\text{C}_{25}\text{H}_{26}$ [M^+]: 326.2035, found: 326.2032.

4 References

- (1) (a) Sun, M.-M.; Wu, H.-D.; Zheng, J.-N.; Bao, W.-L. *Adv. Synth. Catal.* **2012**, *354*, 835. (b) Kuang, C.; Senboku, H.; Tokuda, M. *Tetrahedron Lett.* **2001**, *42*, 3893.
- (2) Lanier, M.; Schade, D.; Willems, E.; Tsuda, M.; Spiering, S.; Kalisiak, J.; Mercola, M.; Cashman, J. R. *J. Med. Chem.* **2012**, *55*, 697.
- (3) Goll, J. M.; Fillion, E. *Organometallics.* **2008**, *27*, 3622.
- (4) (a) Stille, J. R.; Santarsiero, B. D.; Grubbs, R. H. *J. Org. Chem.* **1990**, *55*, 843. (a) Wilson, G. O.; Caruso, M. M.; Schelkopf, S. R.; Sottos, N. R.; White, S. R.; Moore, J. S. *Appl. Mater. Interfaces.* **2011**, *3*, 3072.

5 Copies for ^1H NMR and ^{13}C NMR

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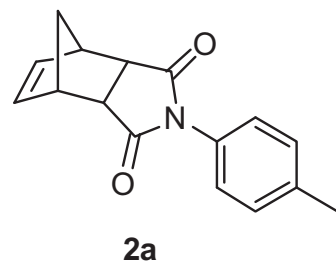
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3.434
3.430

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1.636
1.614

0.031



2.05 2.00

2.01

2.01 2.03

3.08

1.03 1.02

8

7

6

5

4

3

2

1

PPM

USER: root -- DATE: May 19 16:32:45 2012 (UT+8h) nmr@NMR

F1: 400.130

F2: 1.000

SW1: 11990

OF1: 3604.3

PTS1d: 32768

EX: zg30

PW: 12.5 usec

PD: 1.5 sec

NA: 8

LB: 0.3

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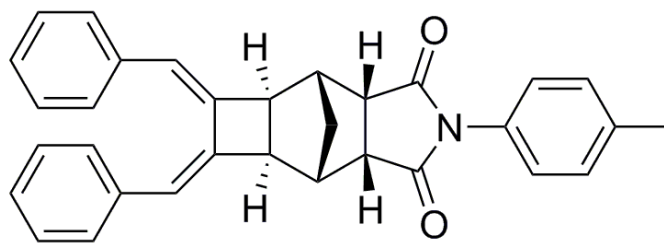
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6.671
6.653
6.330

3.379
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3.047

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2.344

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1.631
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0.019



2.08
2.05
2.07
4.06
4.09
2.00

2.06
2.02
2.03

3.03
1.15

1.01

8

7

6

5

4

3

2

1

PPM

USER: root -- DATE: May 23 14:56:23 2012 (UT+8h) nmr@NMR

F1: 400.130

F2: 1.000

SW1: 11990

OF1: 3598.3

PTS1d: 32768

EX: zg30

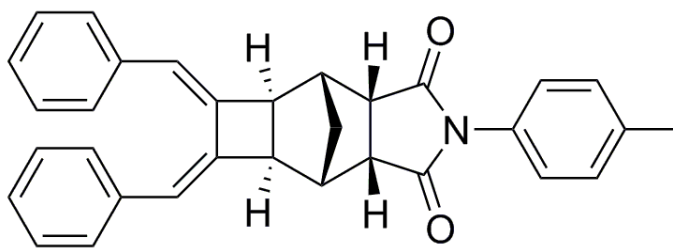
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PD: 1.5 sec

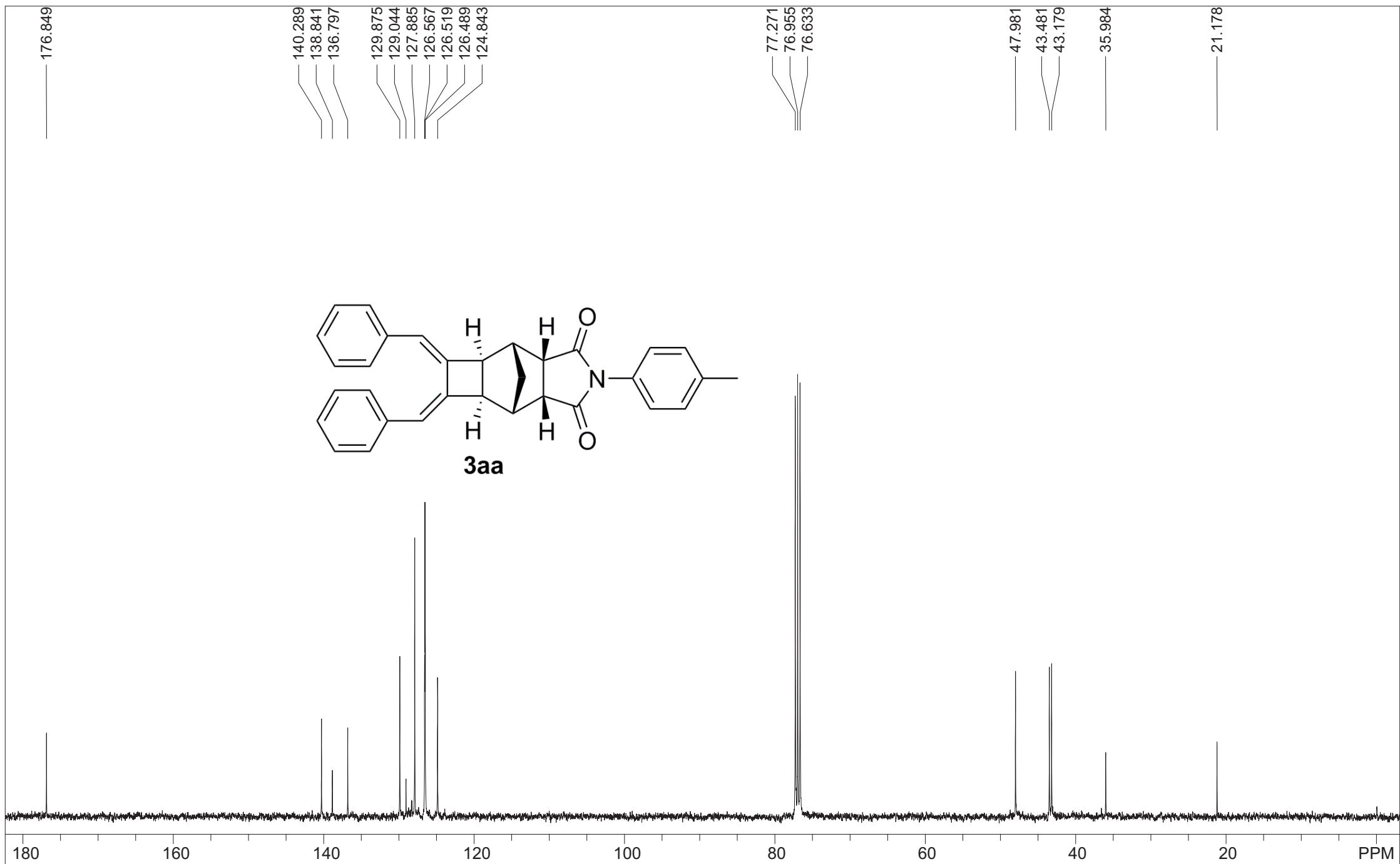
NA: 8

LB: 0.3

Nuts - \$mjpg0114501_74148.1



3aa



USER: root -- DATE: May 30 19:12:16 2012 (UT+8h) nmr@NMR

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Nuts - \$mjpg0114501-C_20591.1

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7.175

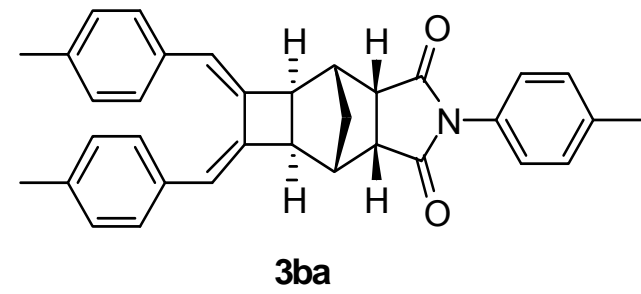
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1.636
1.610

0.017



2.03
2.05

4.08
4.04
2.00

2.02
2.08
2.01

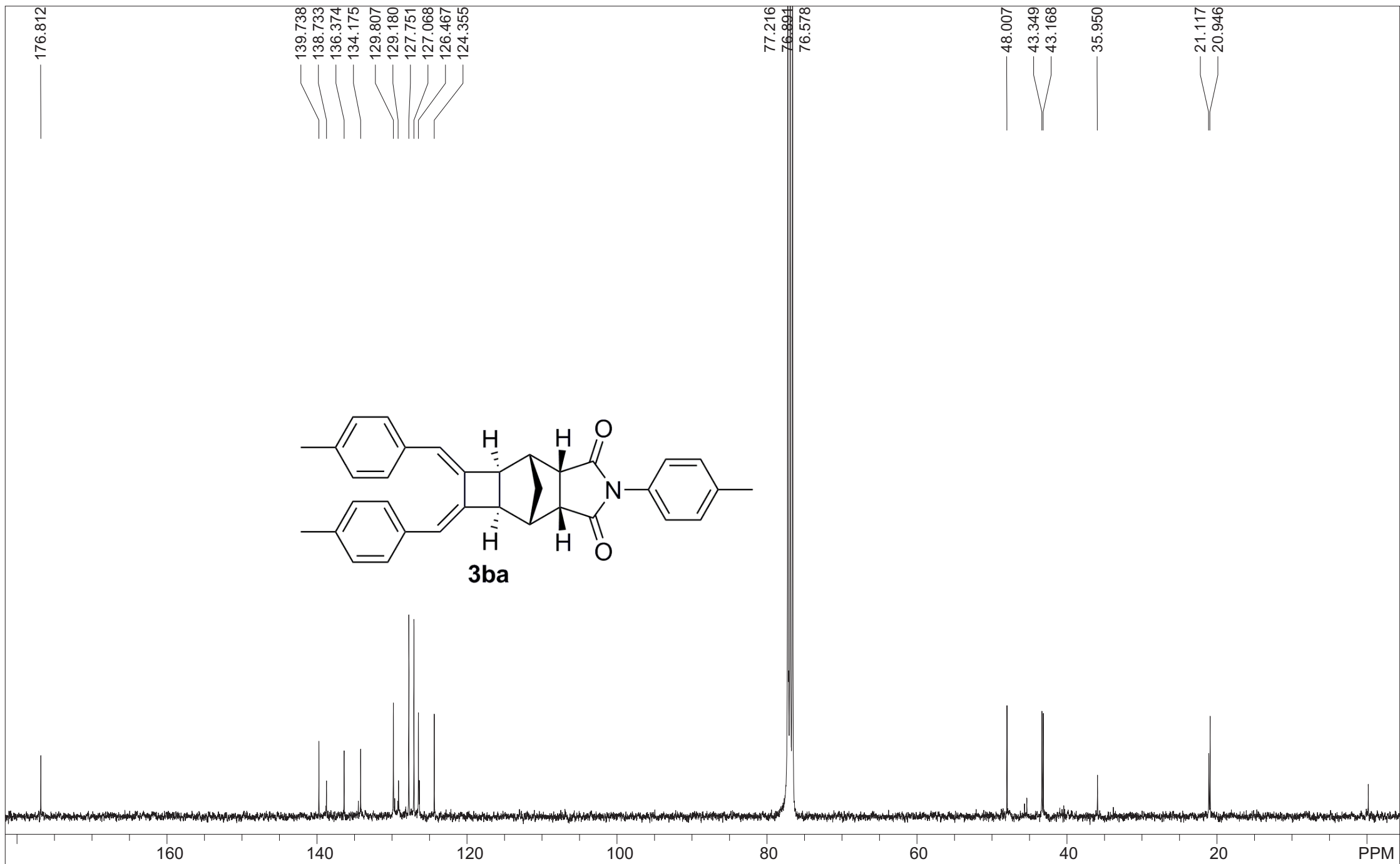
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8 7 6 5 4 3 2 1 PPM

USER: root -- DATE: Oct 8 13:49:25 2012 (UT+8h) nmr@NMR

F1: 400.130	F2: 1.000	SW1: 11990	OF1: 3591.1	PTS1d: 32768
EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 8	LB: 0.3
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USER: root -- DATE: Oct 11 10:05:17 2012 (UT+8h) nmr@NMR

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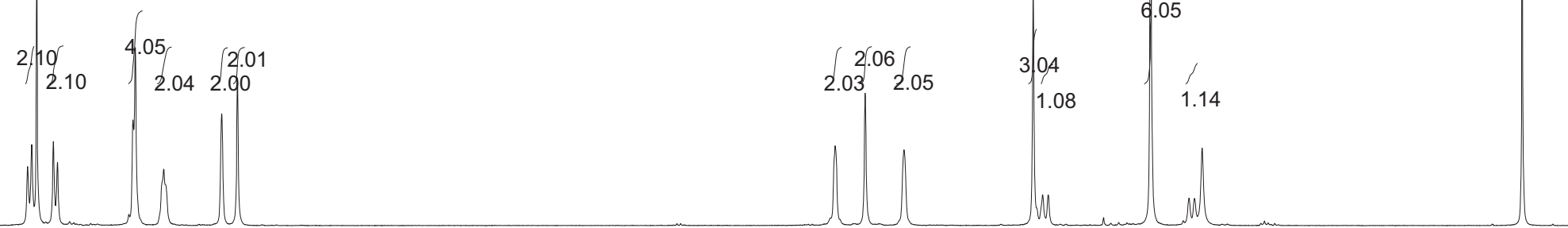
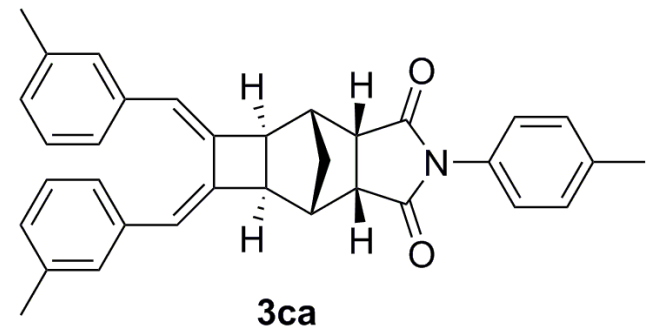
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6.654
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3.036

2.406
2.360
2.332

1.832
1.644
1.617
1.580

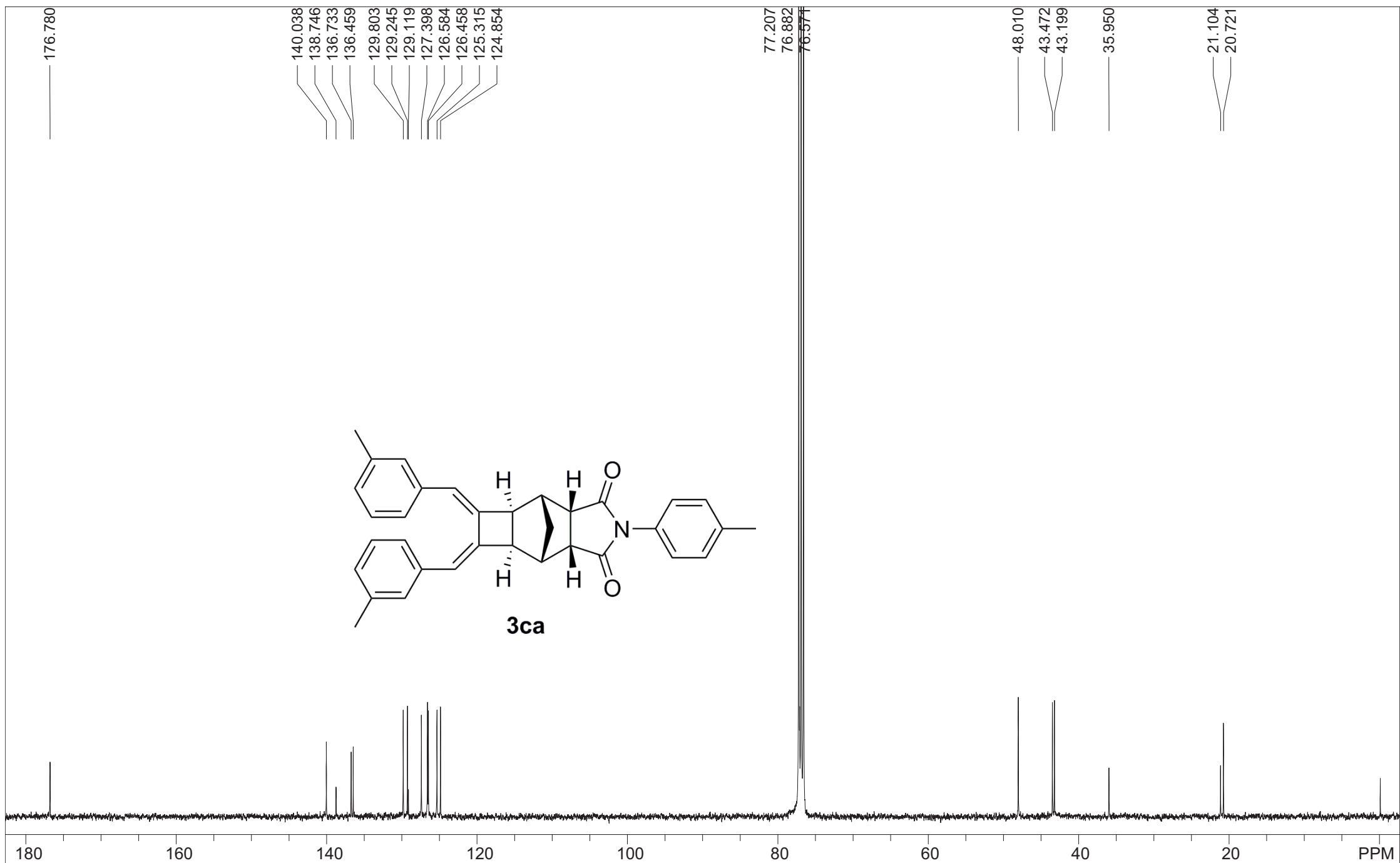
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8 7 6 5 4 3 2 1 0 PPM

USER: root -- DATE: Jun 12 20:32:26 2012 (UT+8h) nmr@NMR

F1: 400.130	F2: 1.000	SW1: 11990	OF1: 3597.6	PTS1d: 32768
EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 8	LB: 0.3
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USER: root -- DATE: Jun 15 10:42:40 2012 (UT+8h) nmr@NMR

F1: 100.613	F2: 1.000	SW1: 25126	OF1: 11056.6	PTS1d: 32768
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Nuts - \$mjq0114901-2-C_20722.1

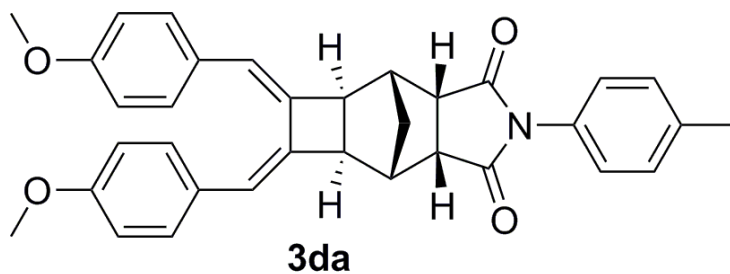
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3.013

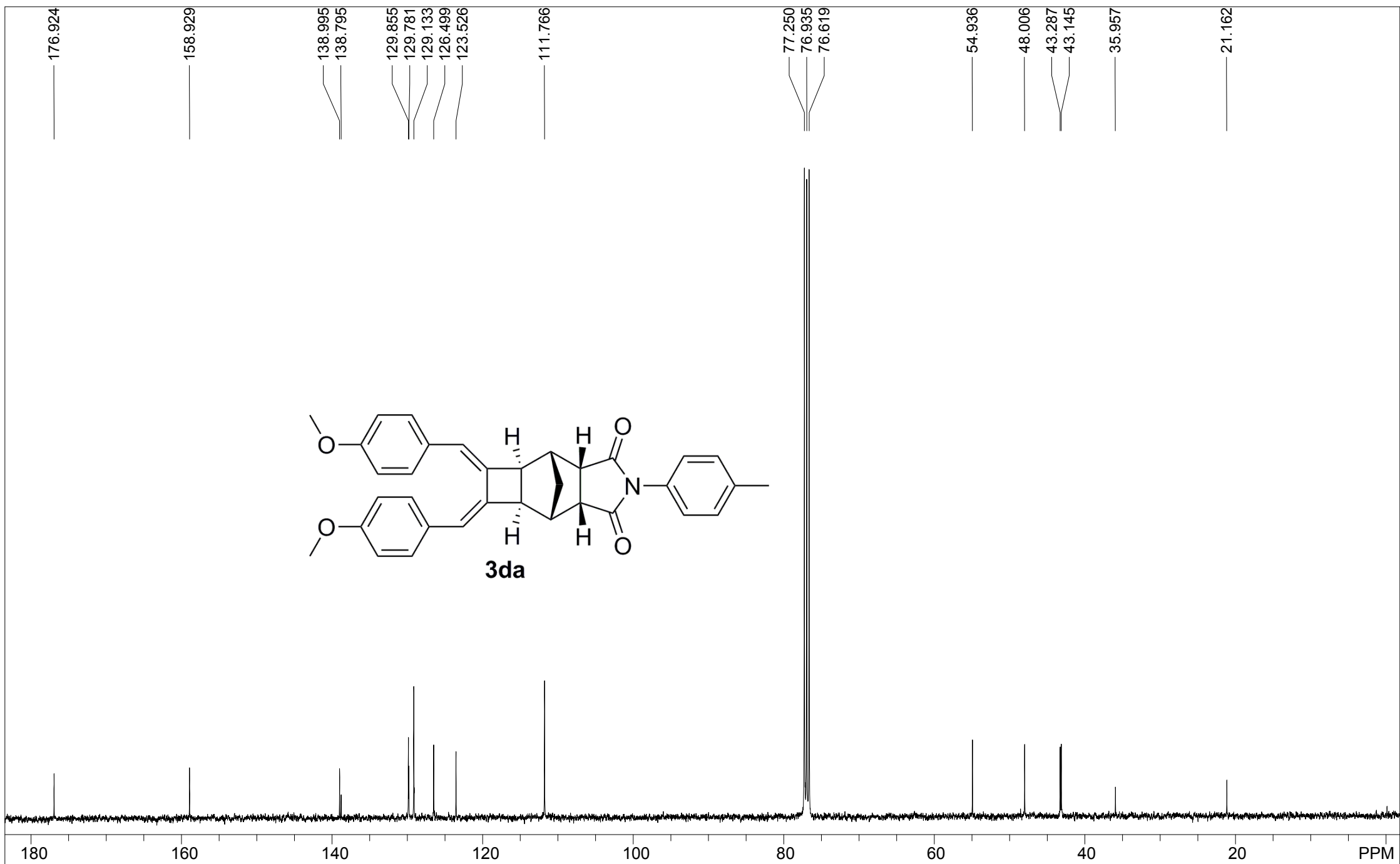
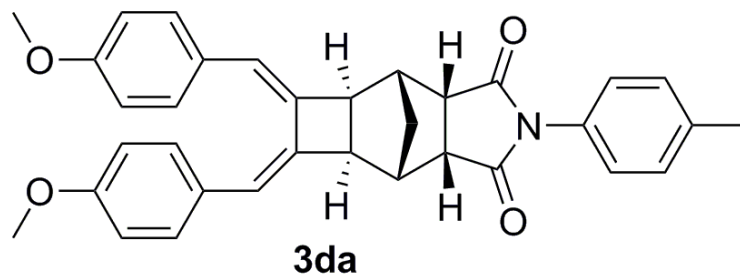
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0.018



USER: root -- DATE: Nov 8 12:58:55 2012 (UT+8h) nmr@NMR

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EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 16	LB: 0.3
				Nuts - \$mjpg0123301_84352.1



USER: root -- DATE: Nov 8 17:57:24 2012 (UT+8h) nmr@NMR

F1: 100.613	F2: 1.000	SW1: 25126	OF1: 11056.6	PTS1d: 32768
EX: zgdc30	PW: 7.5 usec	PD: 1.0 sec	NA: 822	LB: 2.0

Nuts - \$mjpg0123301-C 21626.1

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6.774
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6.261

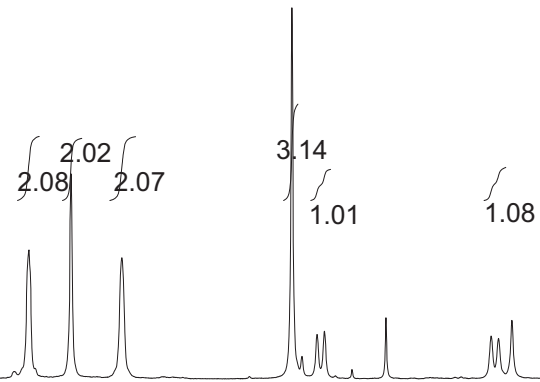
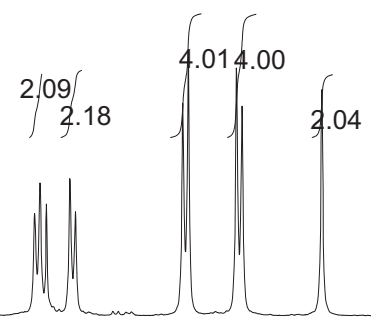
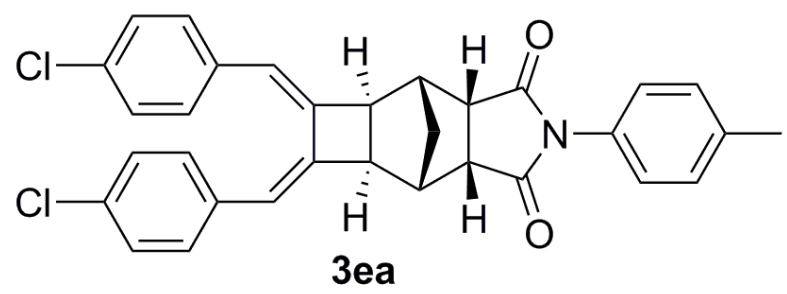
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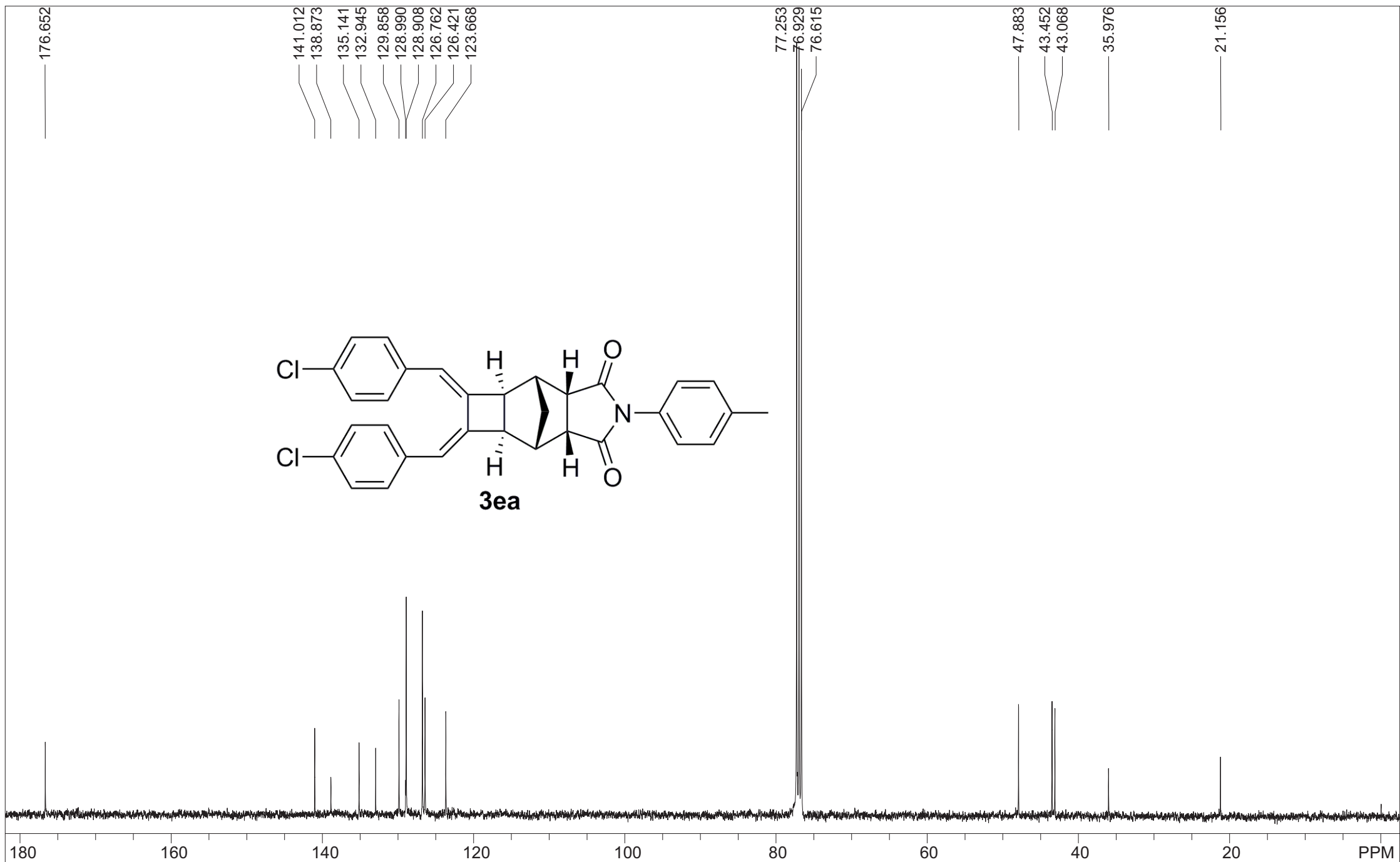
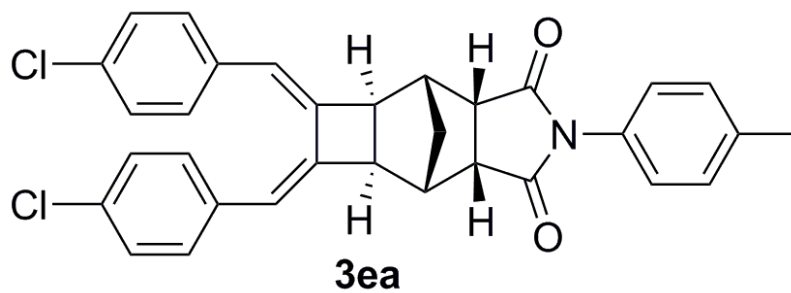
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8 7 6 5 4 3 2 1 PPM

USER: root -- DATE: Sep 27 15:41:47 2012 (UT+8h) nmr@NMR

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EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 8	LB: 0.3
Nuts - \$mjpg0119801_81195.1				



USER: root -- DATE: Sep 28 12:52:08 2012 (UT+8h) nmr@NMR

F1: 100.613	F2: 1.000	SW1: 25126	OF1: 11056.6	PTS1d: 32768
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Nuts - \$mjpg0119801-C_21340.1

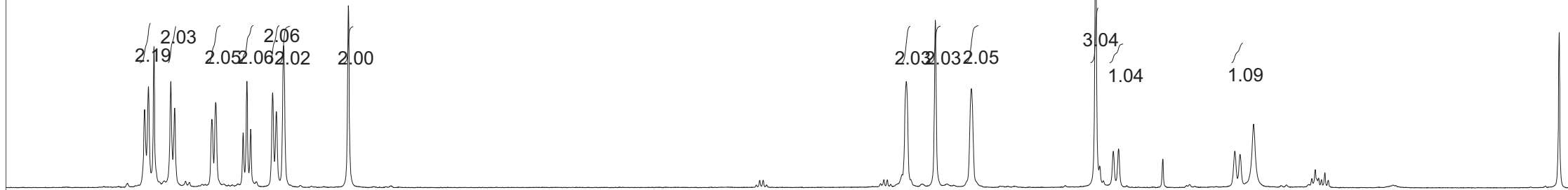
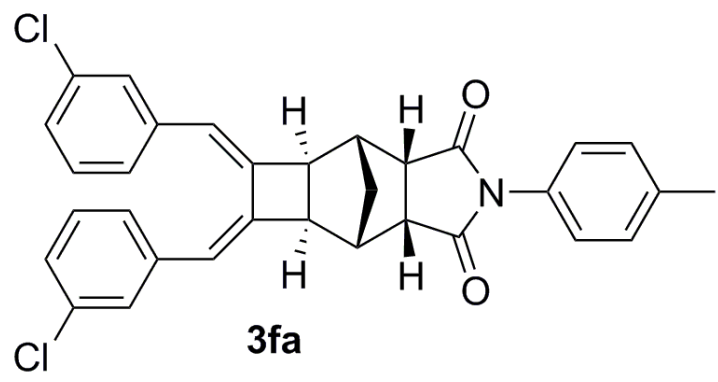
7.322
7.303
7.273
7.186
7.167
6.975
6.955
6.812
6.793
6.774
6.661
6.642
6.603
6.269

3.386
3.236
3.049

2.408
2.316
2.289

1.688
1.661
1.592

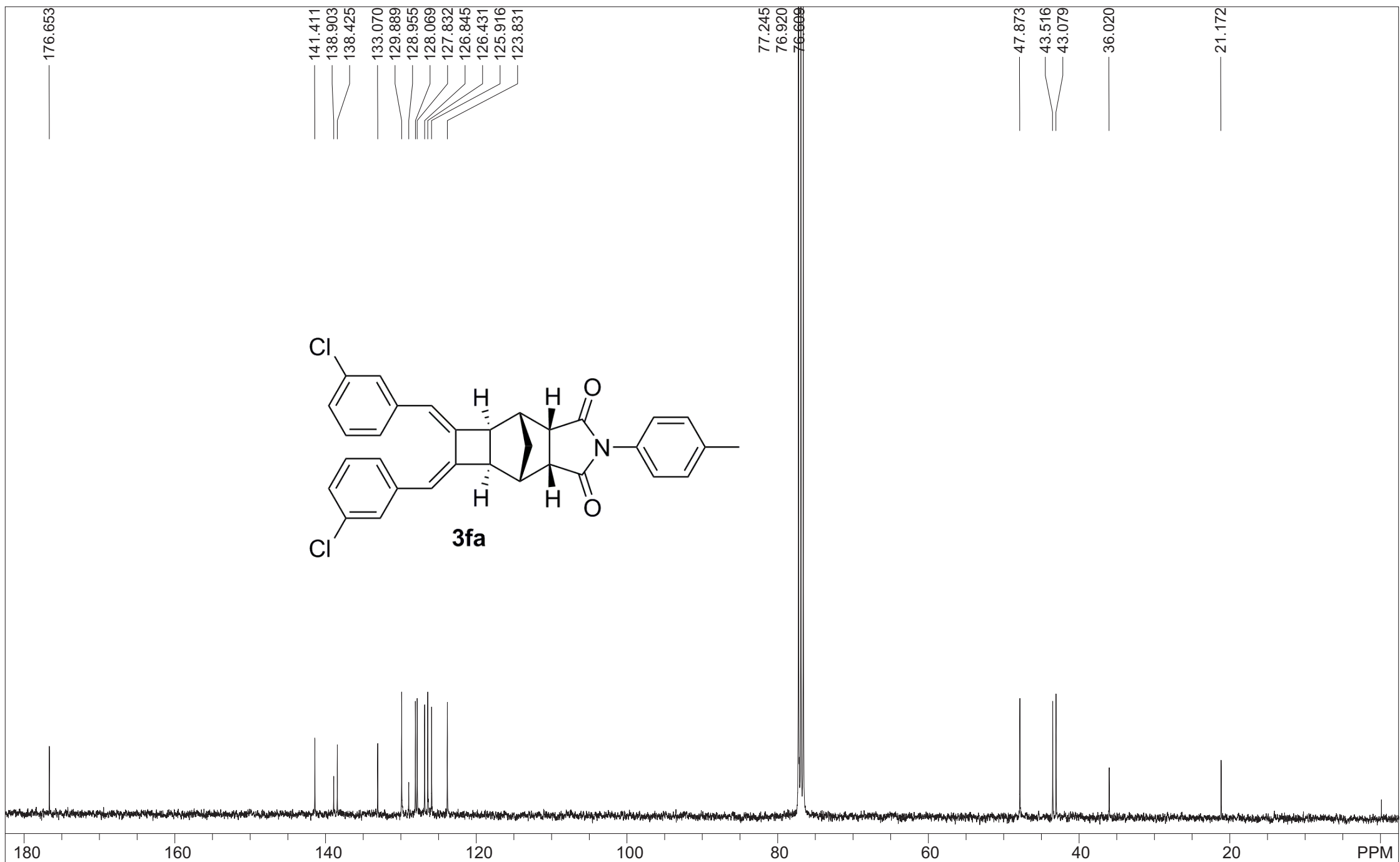
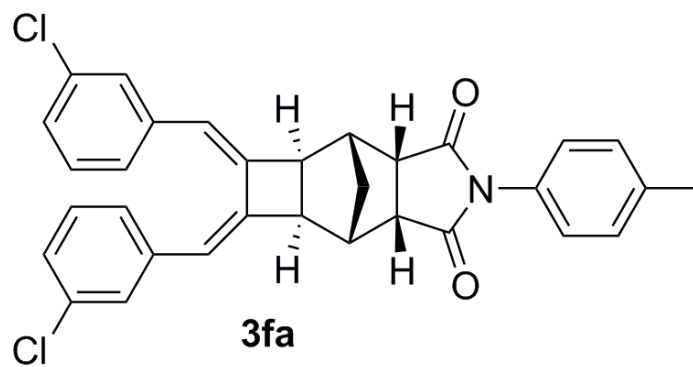
0.012



8 7 6 5 4 3 2 1 PPM

USER: root -- DATE: May 31 21:16:33 2012 (UT+8h) nmr@NMR

F1: 400.130	F2: 1.000	SW1: 11990	OF1: 3597.6	PTS1d: 32768
EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 8	LB: 0.3
Nuts - \$mjpg0114702_75007.1				



USER: root -- DATE: Jun 2 12:51:01 2012 (UT+8h) nmr@NMR

F1: 100.613	F2: 1.000	SW1: 25126	OF1: 11056.6	PTS1d: 32768
EX: zgdc30	PW: 7.5 usec	PD: 1.0 sec	NA: 1525	LB: 2.0

Nuts - \$mjpg0114702-C_20625.1

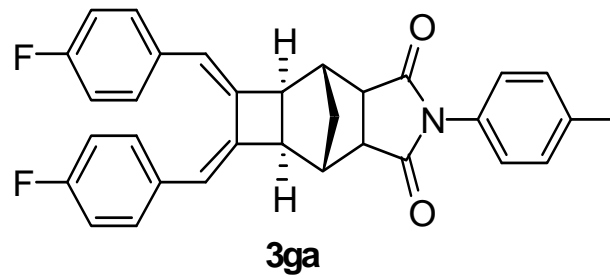
7.323
7.302
7.276
7.192
7.171
6.663
6.650
6.643
6.629
6.531
6.509
6.488
6.262

3.384
3.377
3.372
3.217
3.030

2.408
2.325
2.298

1.669
1.642
1.592

0.016



2.01
2.04
4.02
4.03
2.00

2.07
2.02
2.06

3.09
1.02

1.09

8 7 6 5 4 3 2 1 0 PPM

USER: root -- DATE: May 22 14:54:52 2012 (UT+8h) nmr@NMR

F1: 400.130	F2: 1.000	SW1: 11990	OF1: 3598.3	PTS1d: 32768
EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 8	LB: 0.3
				Nuts - \$mjpg0114401_74038.1

176.746

163.227

160.754

140.208

138.873

132.851

132.817

129.873

129.474

129.385

128.997

126.443

123.511

113.654

113.444

77.258

76.940

76.615

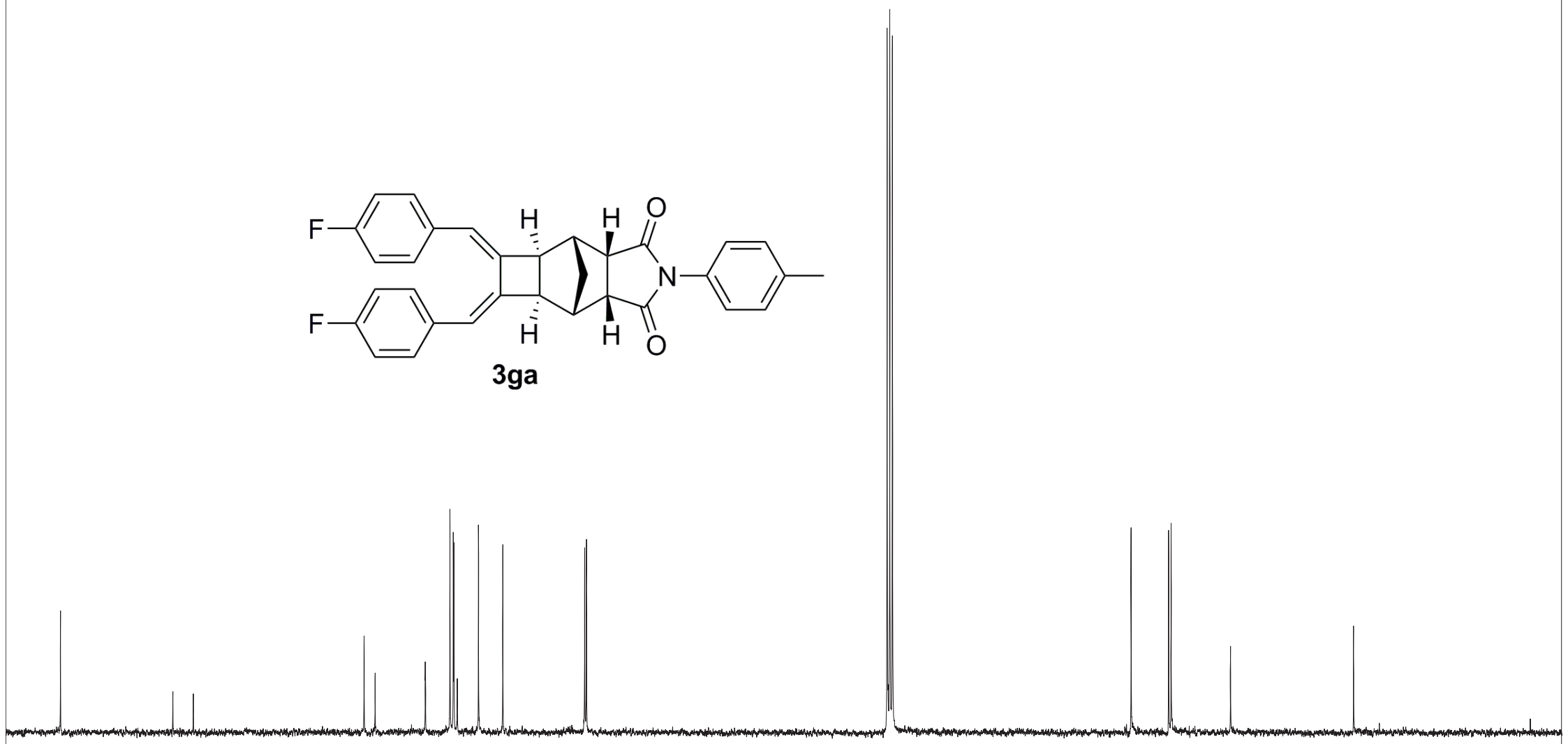
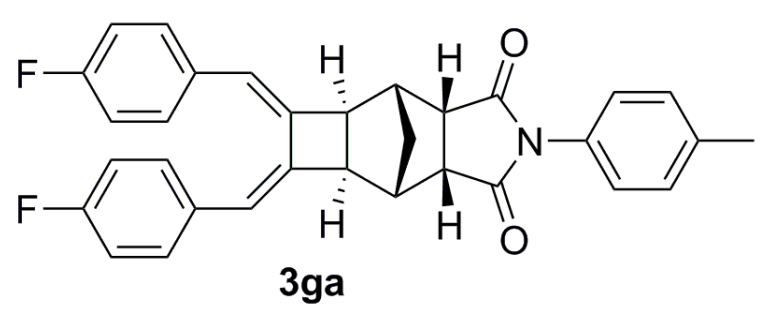
47.905

43.404

43.102

35.960

21.167

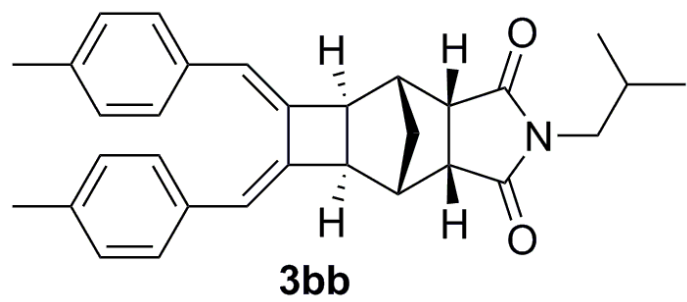


180 160 140 120 100 80 60 40 20 PPM

USER: root -- DATE: May 30 18:53:22 2012 (UT+8h) nmr@NMR

F1: 100.613	F2: 1.000	SW1: 25126	OF1: 11056.6	PTS1d: 32768
EX: zgdc30	PW: 7.5 usec	PD: 1.0 sec	NA: 1620	LB: 2.0

Nuts - \$mjpg0114401-C_20590.1



6.545
6.524
6.507
6.487
6.243

3.349
3.332
3.199
3.023
2.911

2.312
2.286
2.173
2.120
2.103
2.087
2.069
2.052
1.573
1.546

0.962
0.945

4.09
4.11
2.00

2.02
2.03
2.07
2.00

1.04
6.06
1.08

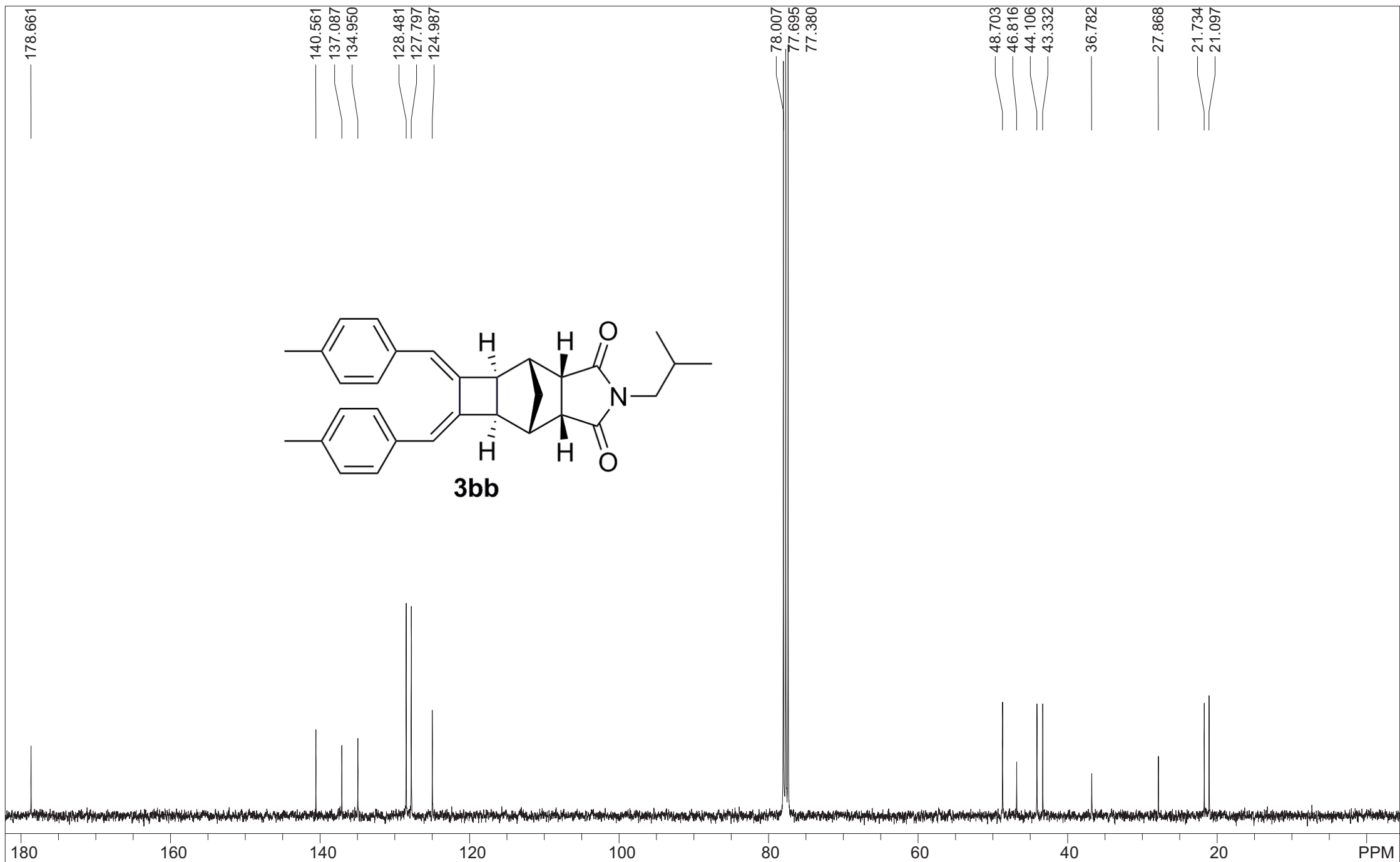
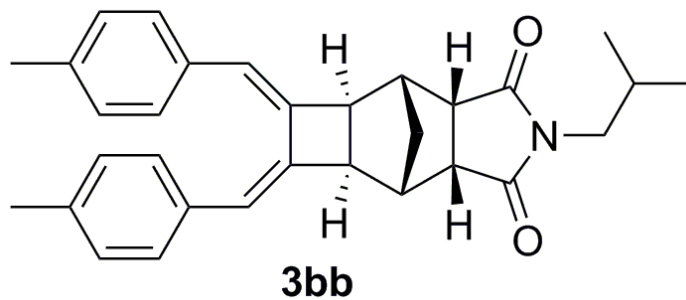
1.01

6.14

8 7 6 5 4 3 2 1 PPM

USER: root -- DATE: Dec 14 17:13:44 2012 (UT+8h) nmr@NMR

F1: 400.130	F2: 1.000	SW1: 11990	OF1: 3598.0	PTS1d: 32768
EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 8	LB: 0.3
				Nuts - \$mjq0203002_87058.1

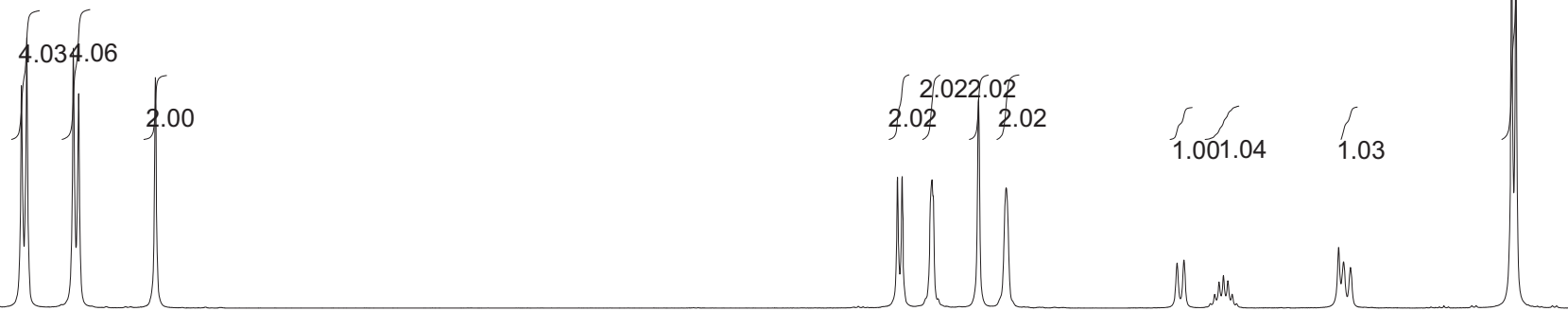
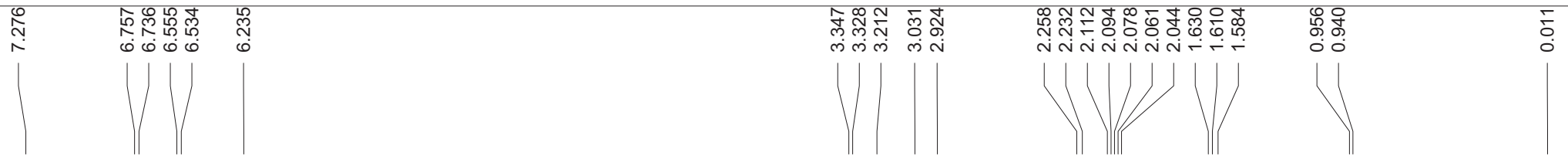
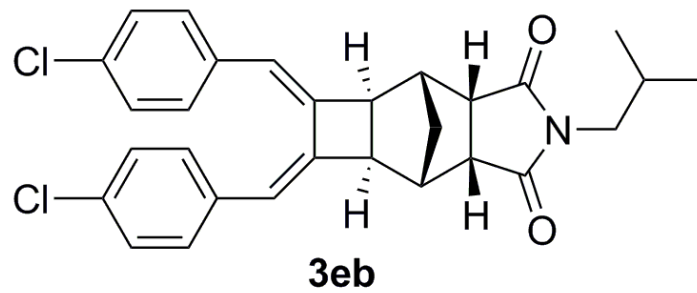


180 160 140 120 100 80 60 40 20 PPM

USER: root -- DATE: Dec 17 16:47:40 2012 (UT+8h) nmr@NMR

F1: 100.613	F2: 1.000	SW1: 25126	OF1: 11132.9	PTS1d: 32768
EX: zgdc30	PW: 7.5 usec	PD: 1.0 sec	NA: 553	LB: 2.0

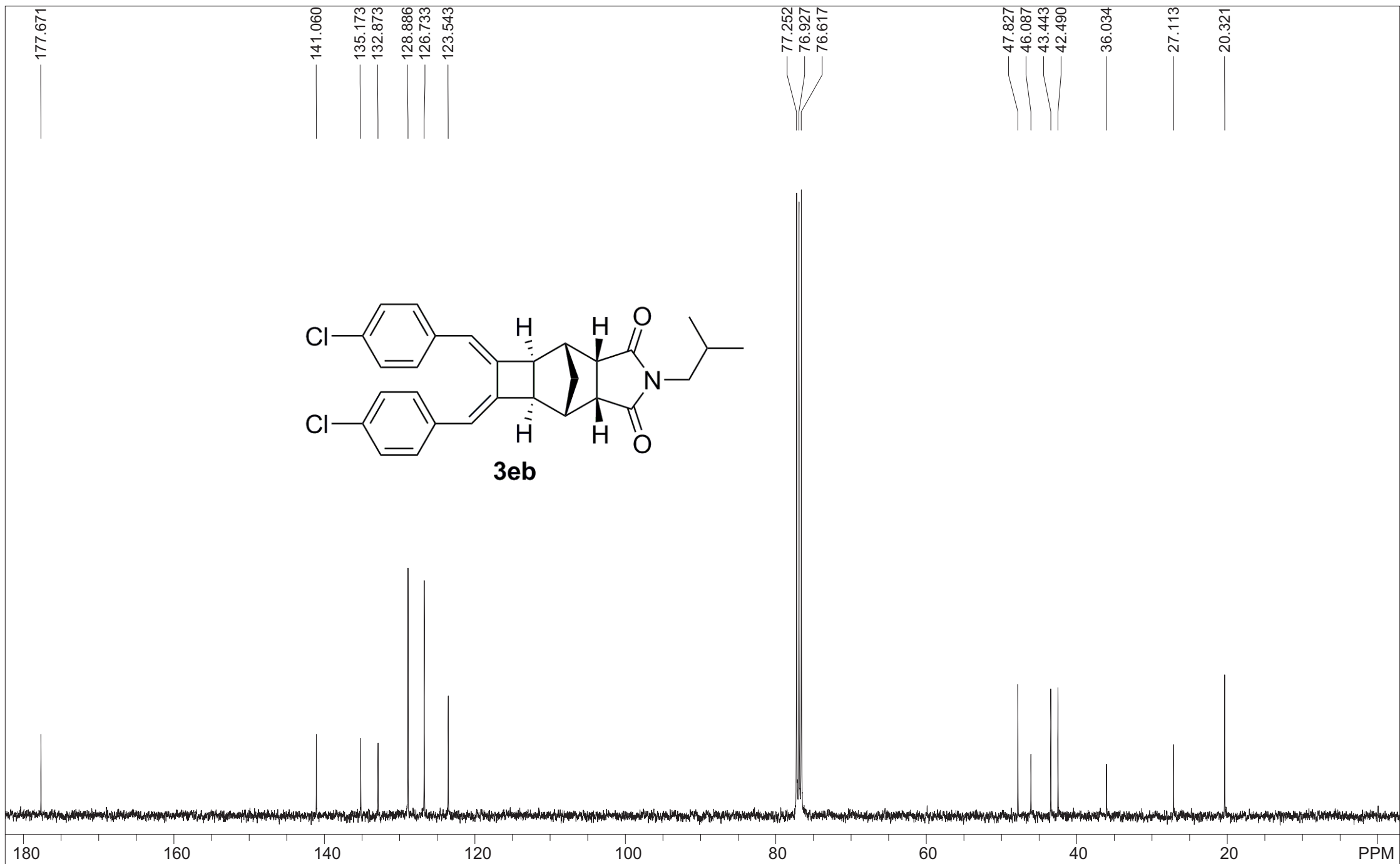
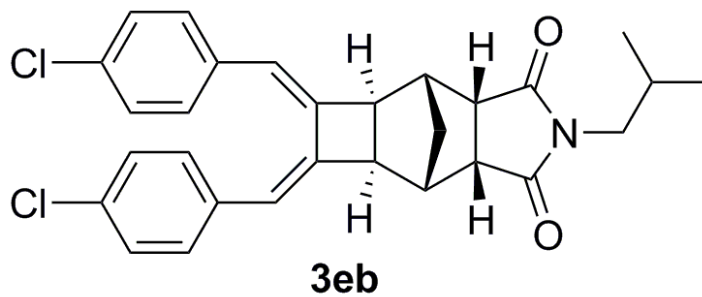
Nuts - \$mjpg0203002-C_21934.1



USER: root -- DATE: Nov 10 19:03:07 2012 (UT+8h) nmr@NMR

F1: 400.130	F2: 1.000	SW1: 11990	OF1: 3598.3	PTS1d: 32768
EX: zg30	PW: 12.5 usec	PD: 1.5 sec	NA: 8	LB: 0.3

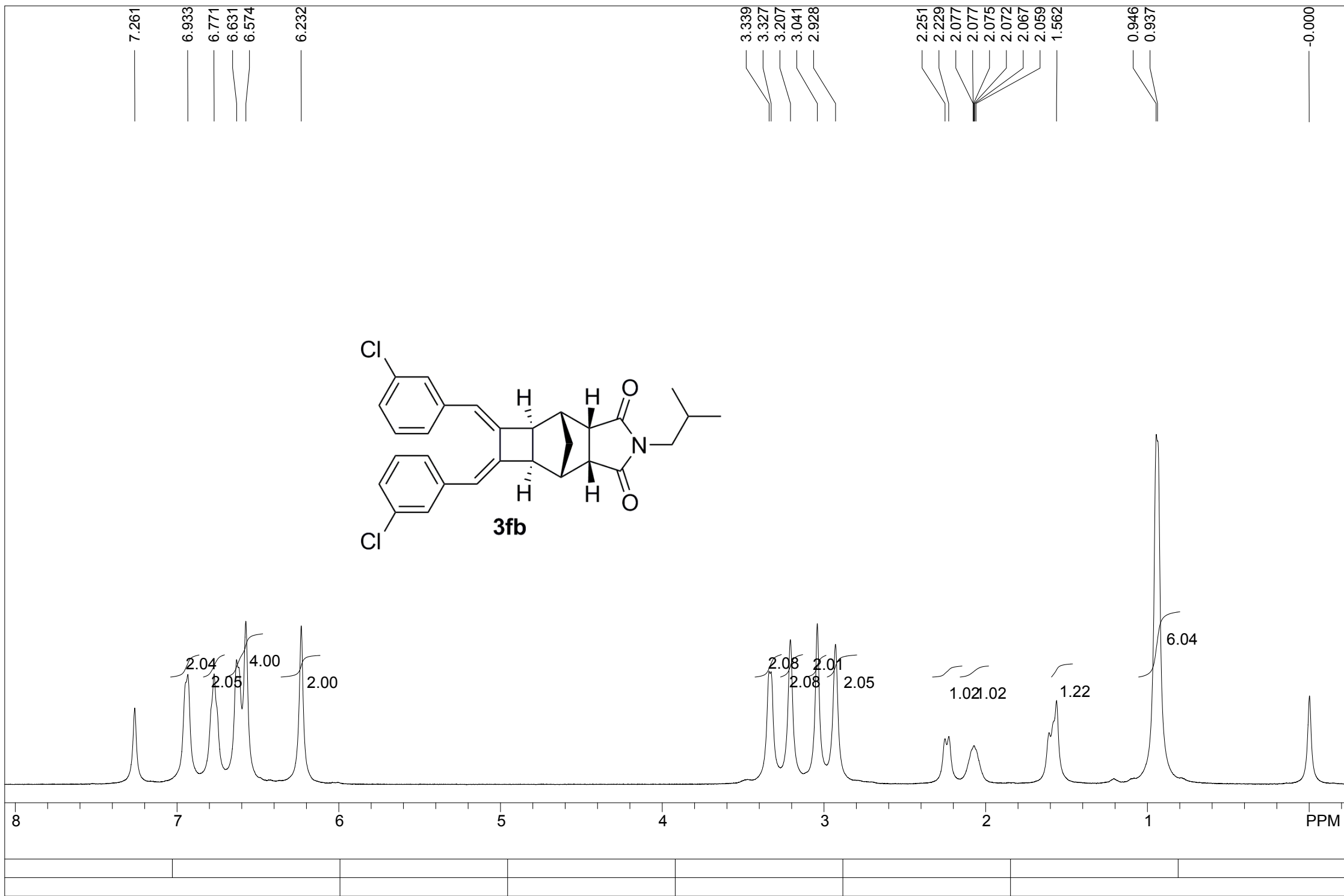
Nuts - \$mjpg0123501_84555.1

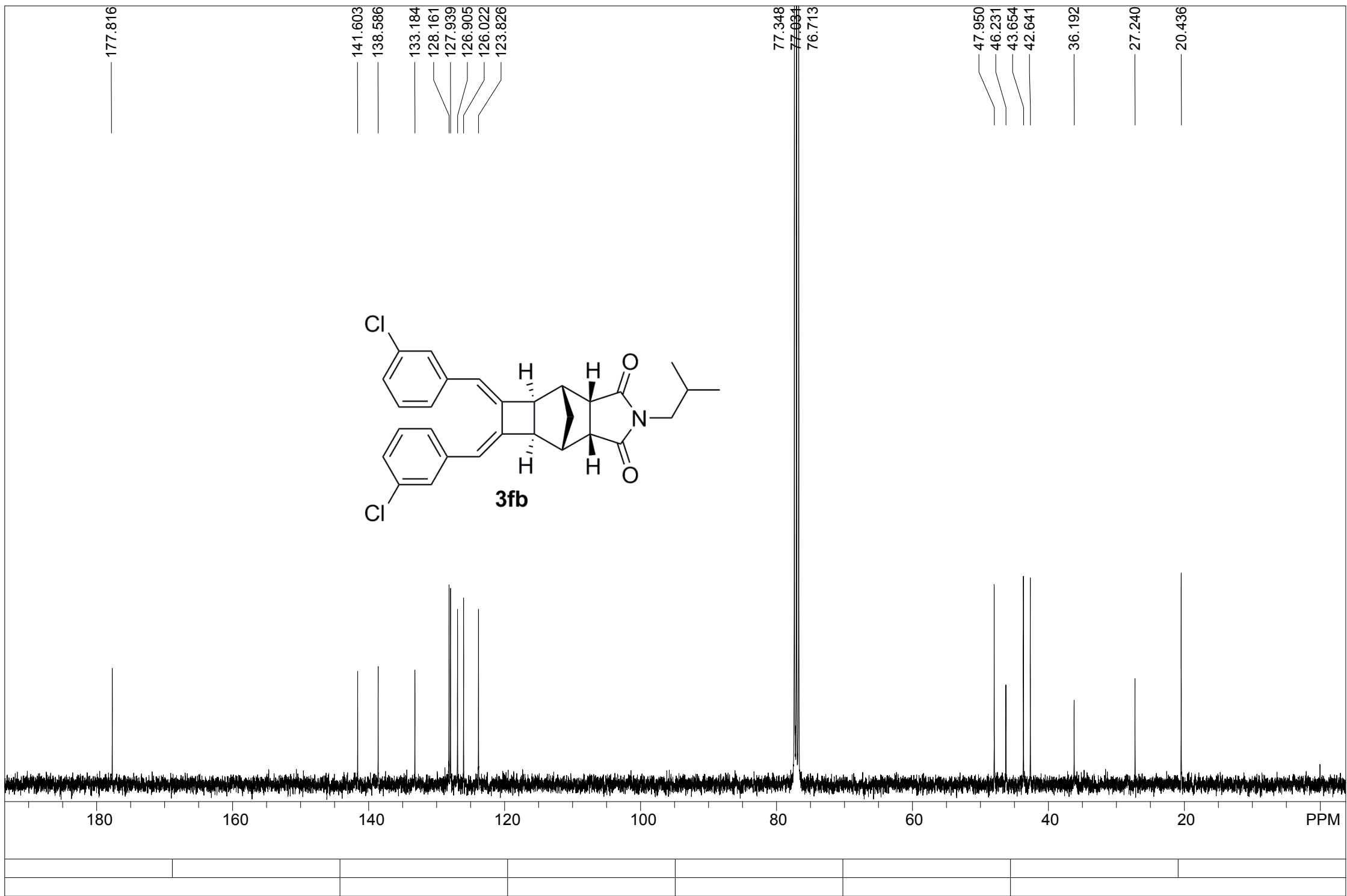


USER: root -- DATE: Nov 12 13:21:08 2012 (UT+8h) nmr@NMR

F1: 100.613	F2: 1.000	SW1: 25126	OF1: 11056.6	PTS1d: 32768
EX: zgdc30	PW: 7.5 usec	PD: 1.0 sec	NA: 339	LB: 2.0

Nuts - \$mjpg0123501-C_21653.1





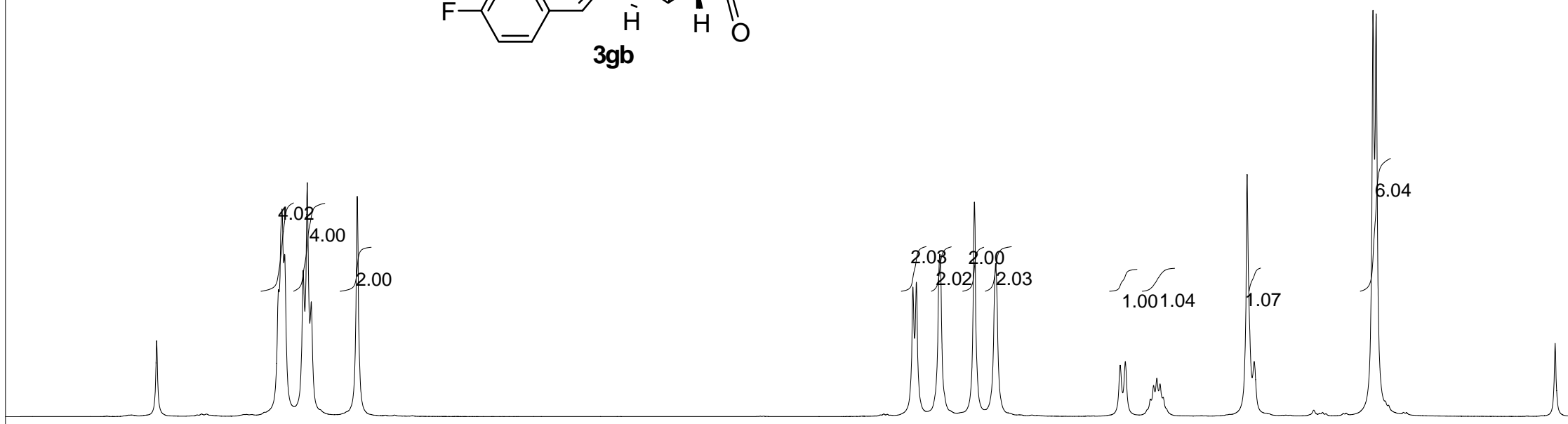
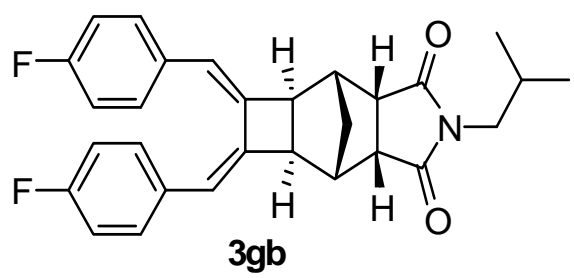
6.631
6.613
6.599
6.503
6.482
6.461
6.222

3.337
3.319
3.197
3.017
2.907

2.260
2.233
2.120
2.103
2.087
2.070
2.053
2.037
1.600
1.564

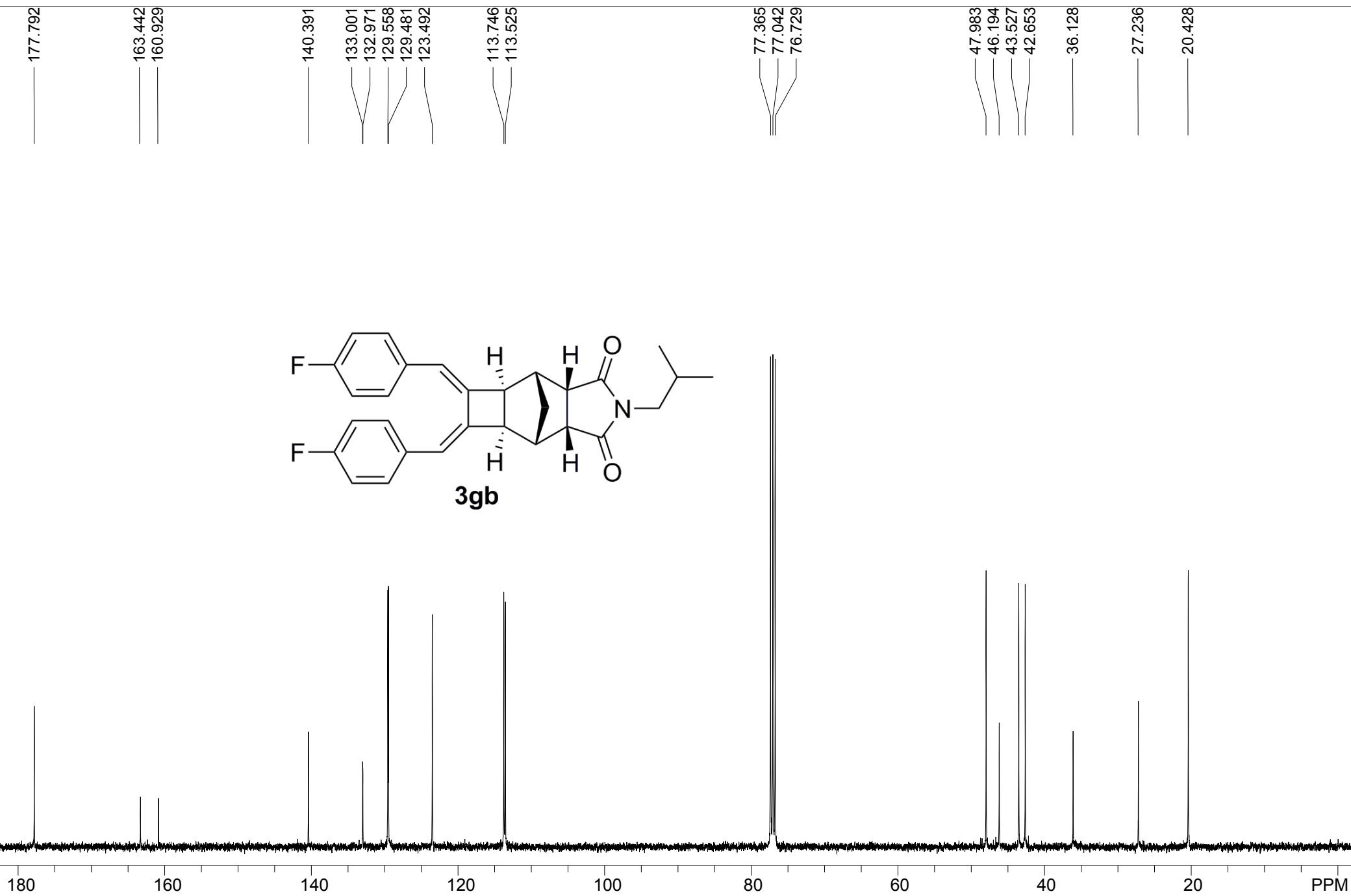
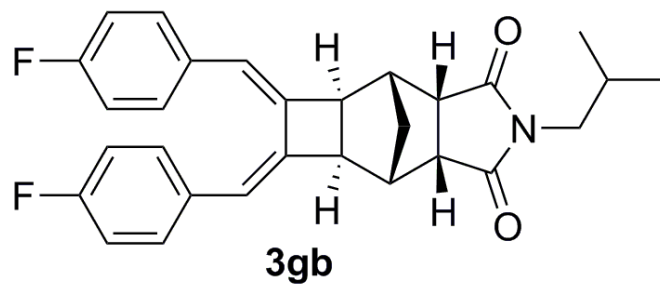
0.946
0.930

-0.000



8 7 6 5 4 3 2 1 PPM

spect, CDCl3,		USER: nmrsu -- DATE: Sat Jun 21 13:26:55 2014			
F1: 400.132	F2: 1.000	SW1: 8013	OF1: 2462.8	PTS1d: 65536	
EX: zg30	PW: 9.8 usec	PD: 1.0 sec	NA: 64	LB: 0.0	Nuts - \$pdata

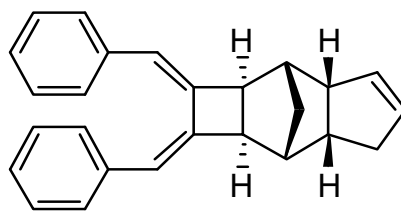


6.825
6.807
6.789
6.648
6.629
6.610
6.590
6.572
6.112

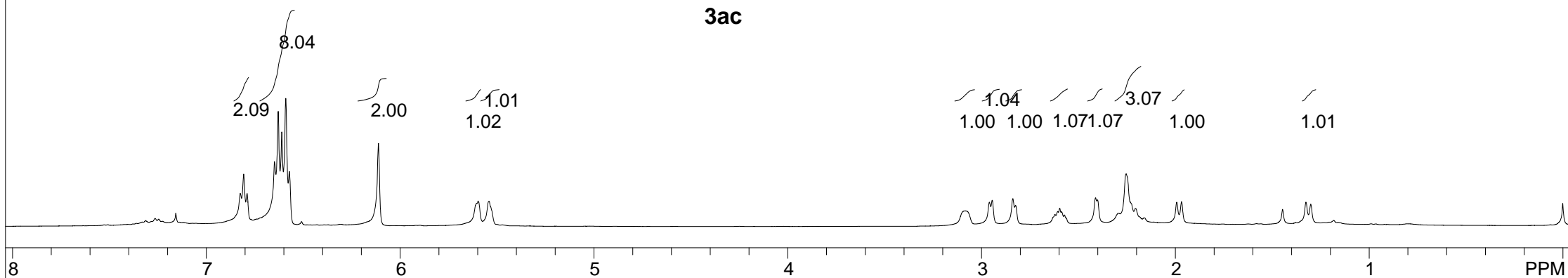
5.606
5.597
5.543
5.541

3.096
3.090
3.084
3.079
3.072
2.960
2.945
2.839
2.825
2.621
2.607
2.596
2.587
2.572
2.563
2.411
2.402
2.295
2.253
2.231
2.206
2.202
1.993
1.968
1.325
1.300

-0.001



3ac



spect. CDCl3.

USER: nmrsu -- DATE: Tue Oct 07 08:42:09 2014

F1: 400.132

F2: 1.000

SW1: 8013

OF1: 2420.0

PTS1d: 65536

EX: zg30

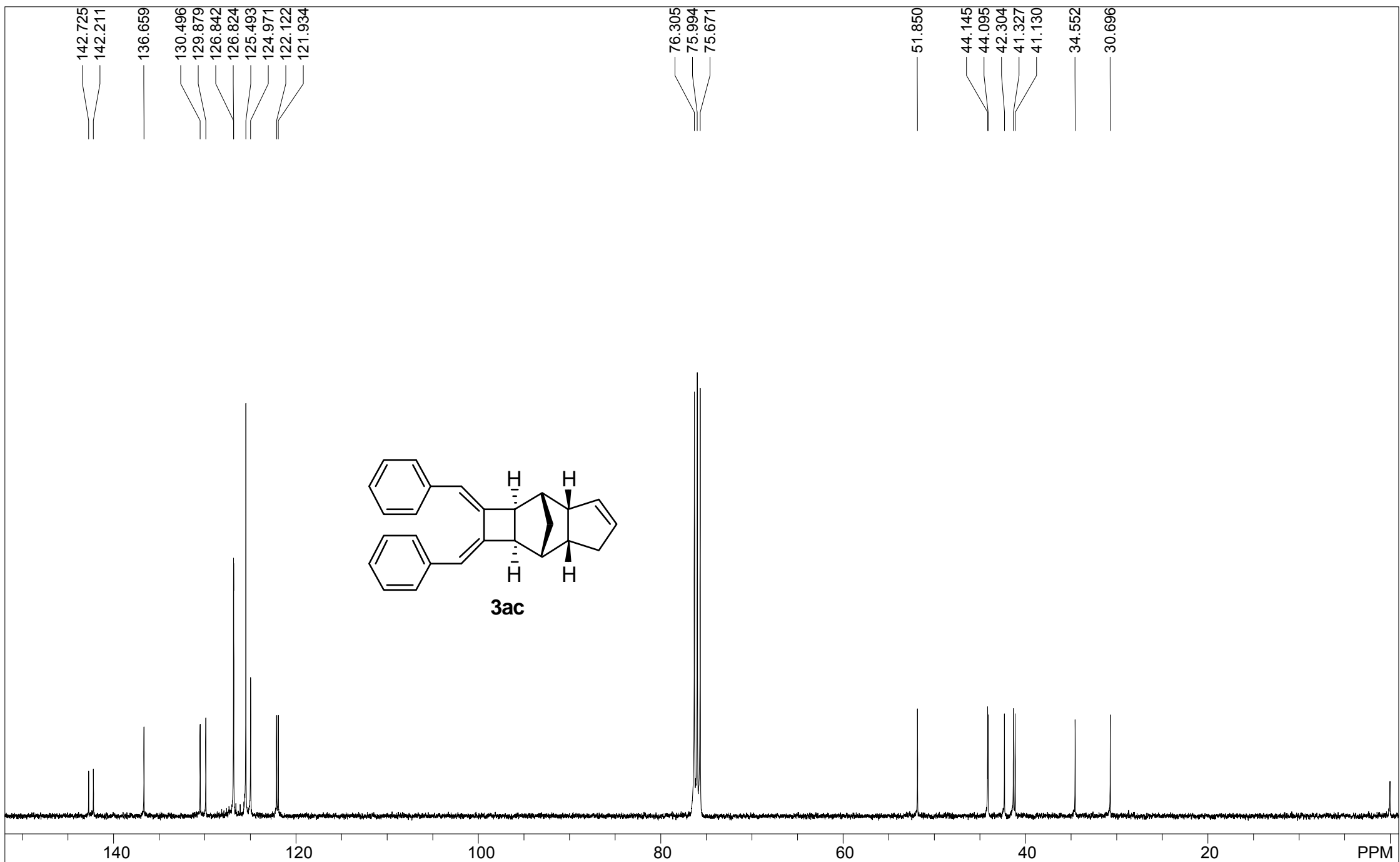
PW: 9.8 usec

PD: 1.0 sec

NA: 16

LB: 0.0

Nuts - \$pdata



spect, CDC13,

USER: nmrsu -- DATE: Sat Jun 14 19:04:24 2014

F1: 100.623

F2: 1.000

SW1: 24038

OF1: 9958.3

PTS1d: 32768

EX: zgdc30

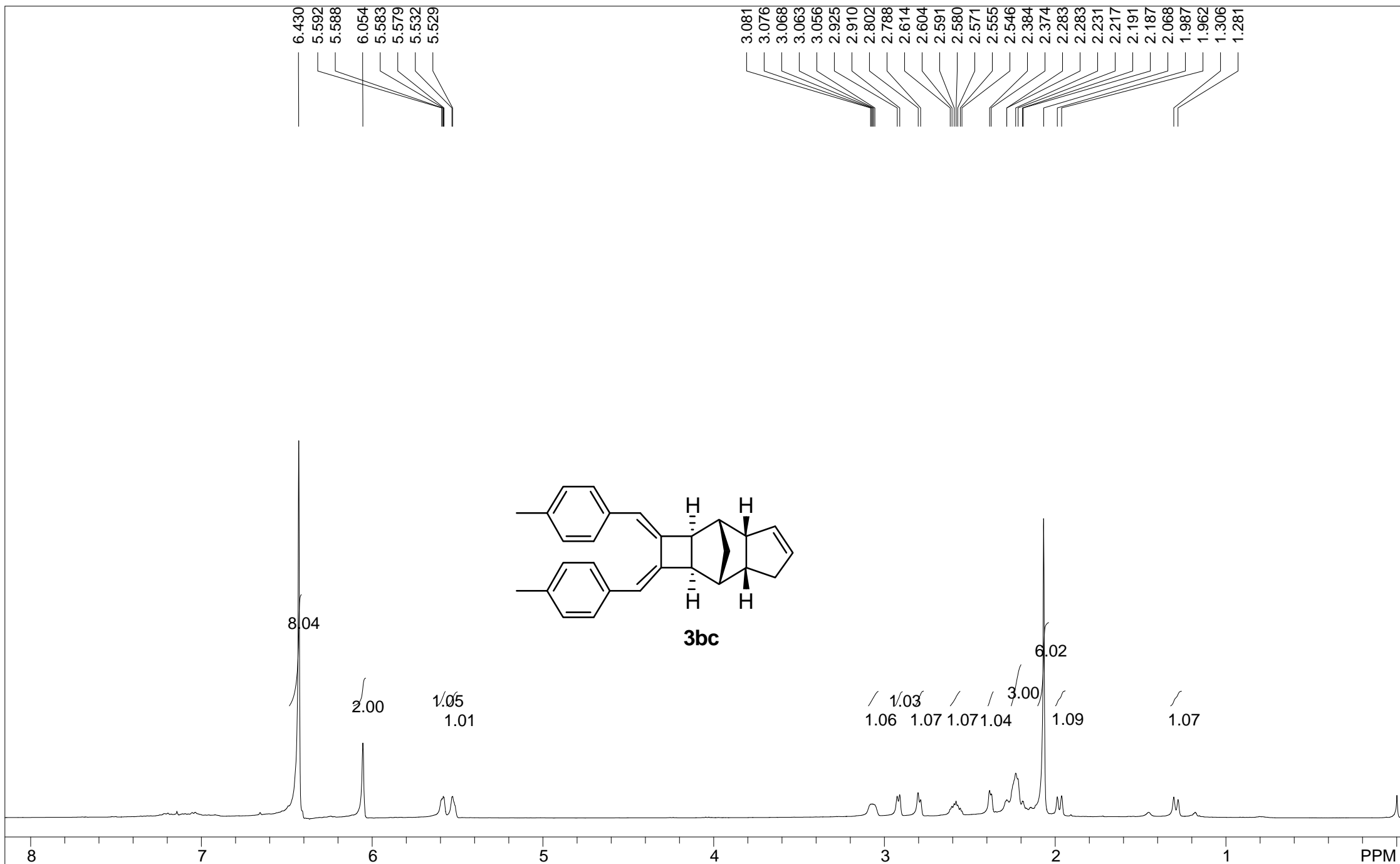
PW: 9.5 usec

PD: 1.2 sec

NA: 1024

LB: 0.0

Nuts - \$pdata

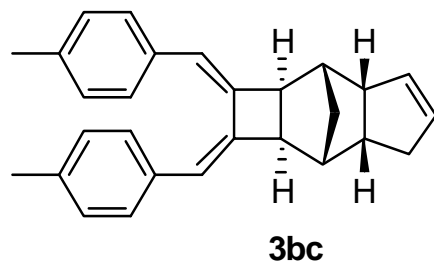


spect. CDCl ₃ .		USER: nmrsu -- DATE: Tue Oct 07 08:48:46 2014				
F1: 400.132	F2: 1.000	SW1: 8013		OF1: 2414.5		PTS1d: 65536
EX: zg30		PW: 9.8 usec	PD: 1.0 sec	NA: 16	LB: 0.0	Nuts - \$pdata

142.148
141.637
134.680
134.654
134.015
130.538
126.695
126.682
126.040
121.651
121.460

76.305
75.988
75.670

51.858
44.057
43.990
42.262
41.152
41.129
34.541
30.690
20.008



160 140 120 100 80 60 40 20 PPM

spect, CDC13,

USER: nmrsu -- DATE: Fri Jun 13 18:53:14 2014

F1: 100.623

F2: 1.000

SW1: 24039

OF1: 9957.0

PTS1d: 32768

EX: zgdc30

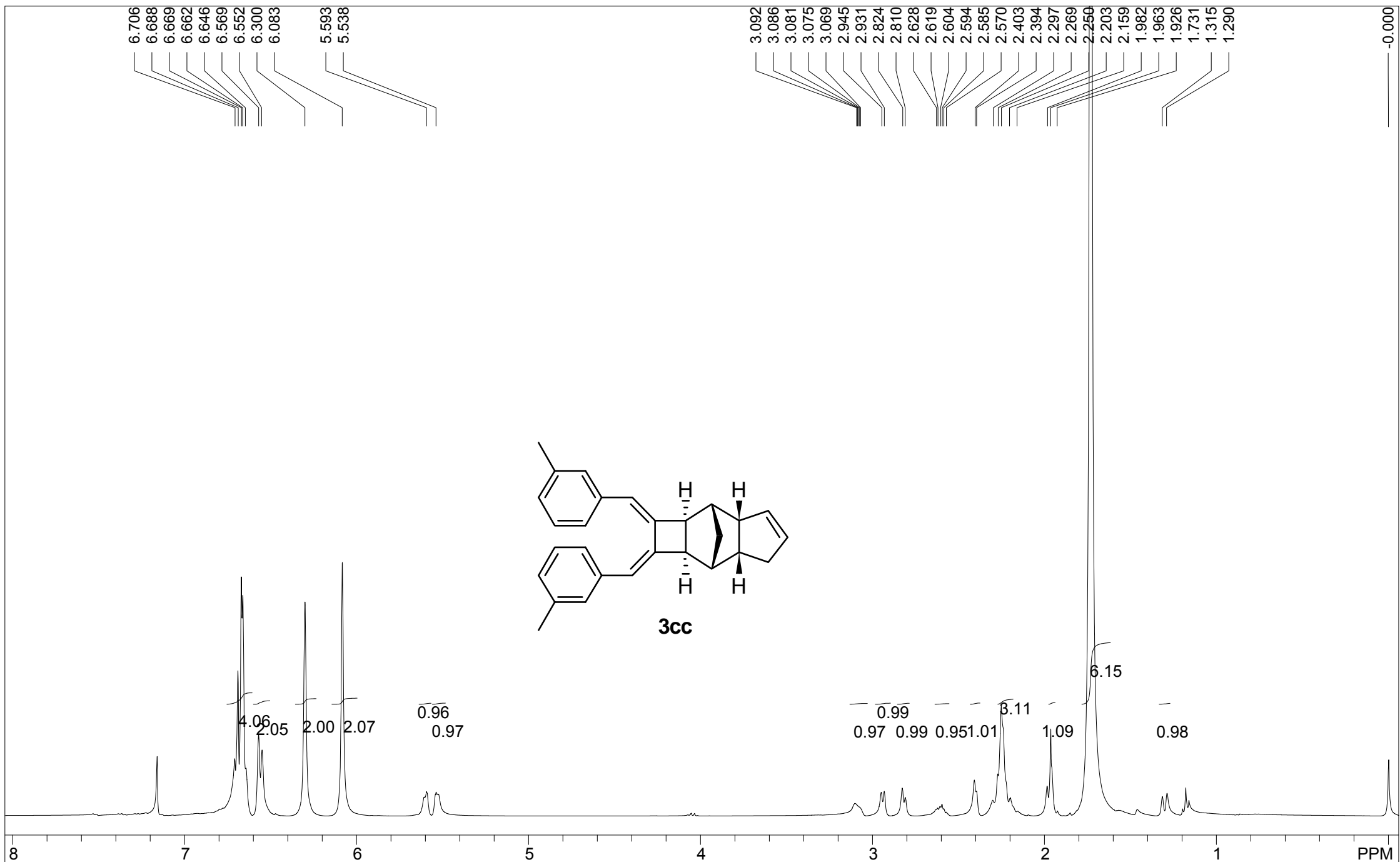
PW: 9.5 usec

PD: 1.2 sec

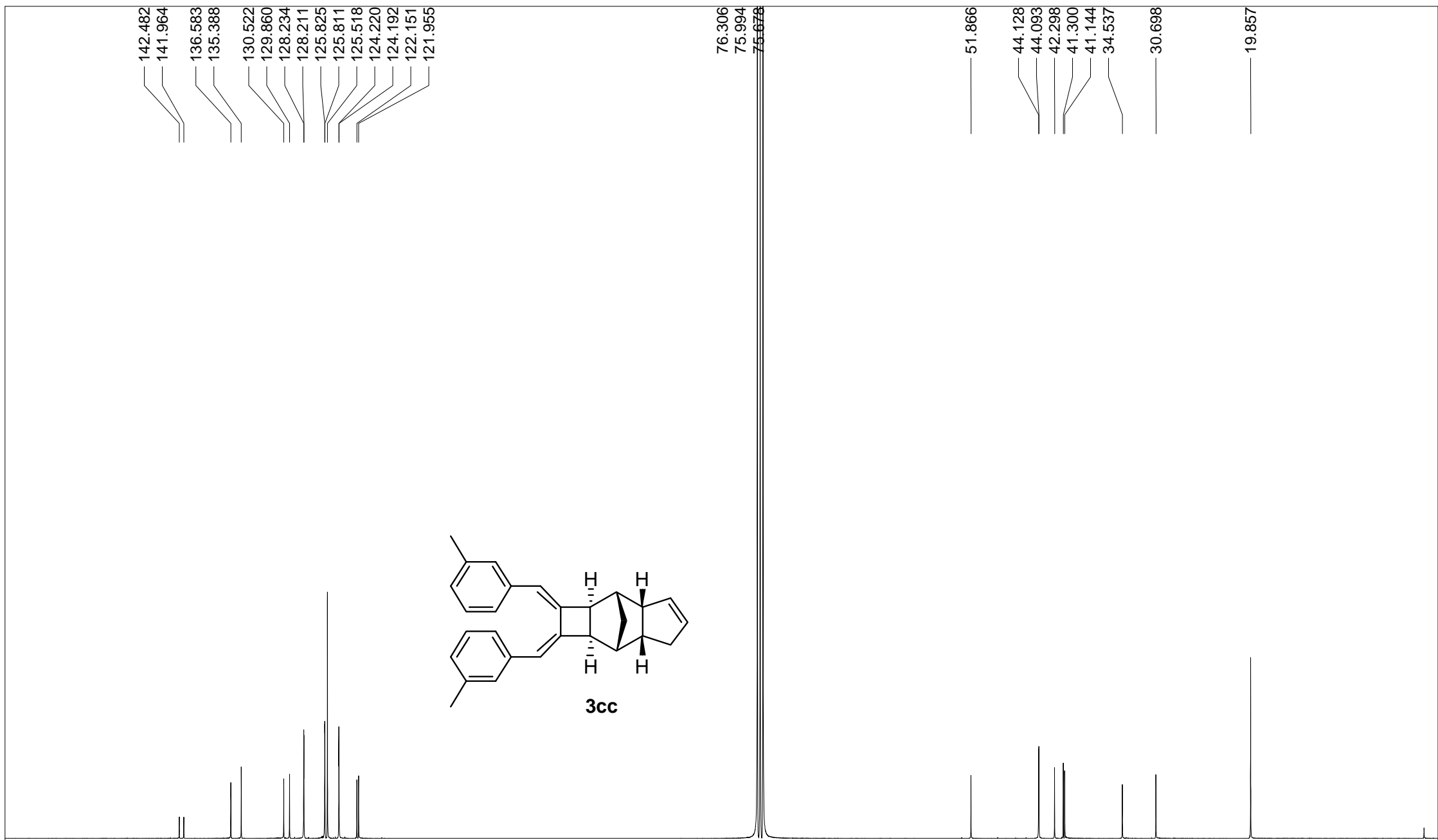
NA: 512

LB: 0.0

Nuts - \$pdata



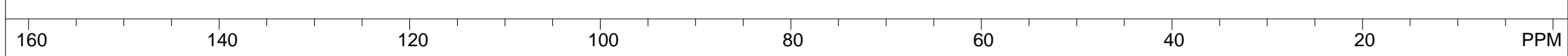
spect. CDCl ₃ .		USER: nmrsu -- DATE: Wed Oct 08 09:30:43 2014	
F1: 400.132	F2: 1.000	SW1: 8013	OF1: 2421.3
EX: zg30	PW: 9.8 usec	PD: 1.0 sec	NA: 64
		LB: 0.0	PTS1d: 65536
			Nuts - \$pdata



142.482
141.964
136.583
135.388
130.522
129.860
128.234
128.211
125.825
125.811
125.518
124.220
124.192
122.151
121.955

76.306
75.994
75.678

51.866
44.128
44.093
42.298
41.300
41.144
34.537
30.698
19.857

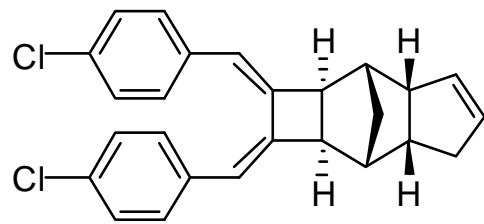


spect, CDC13,		USER: nmrsu -- DATE: Fri Jun 13 19:49:04 2014				
F1: 100.623	F2: 1.000	SW1: 24038		OF1: 9958.5	PTS1d: 32768	
EX: zgdc30		PW: 9.5 usec	PD: 1.2 sec	NA: 1024	LB: 0.0	
					Nuts - \$pdata	

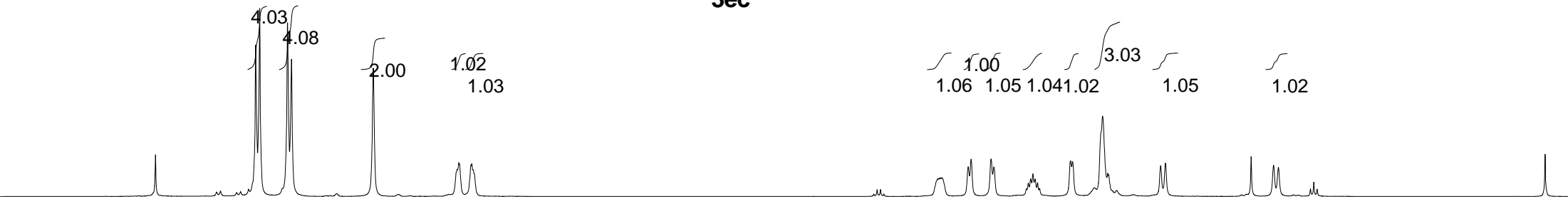
6.730
6.709
6.564
6.543
5.679
6.116
5.669
5.665
5.606
5.602
5.594

3.171
3.165
3.158
3.152
3.145
3.010
2.995
2.890
2.875
2.706
2.696
2.684
2.672
2.660
2.648
2.637
2.475
2.465
2.308
2.282
2.276
2.006
1.980
1.416
1.391

-0.001



3ec

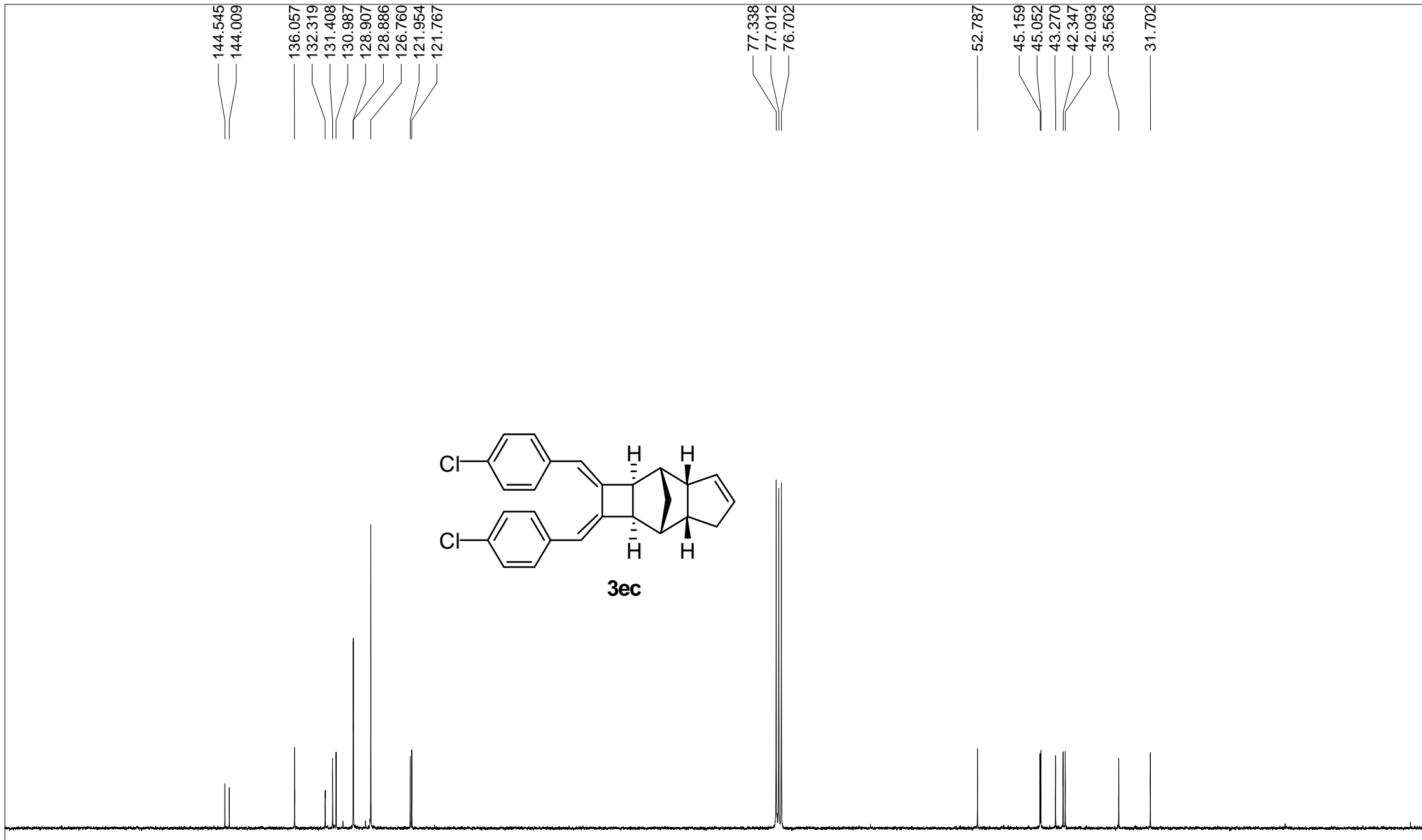
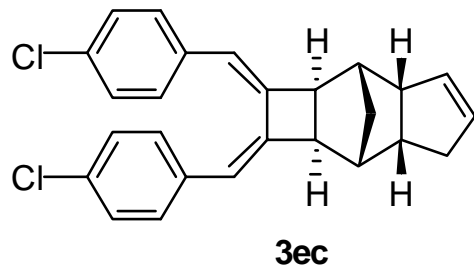


8 7 6 5 4 3 2 1 PPM

spect. CDCl₃, USER: nmrsu -- DATE: Sat Mar 15 14:32:09 2014

F1: 400.132	F2: 1.000	SW1: 8013	OF1: 2458.6	PTS1d: 65536
EX: zg30	PW: 9.8 usec	PD: 1.0 sec	NA: 16	LB: 0.0

144.545
144.009
136.057
132.319
131.408
130.987
128.907
128.886
126.760
121.954
121.767
77.338
77.012
76.702
52.787
45.159
45.052
43.270
42.347
42.093
35.563
31.702



160

140

120

100

80

60

40

20

PPM

spect, CDC13,

USER: nmrsu -- DATE: Sat Mar 15 21:17:02 2014

F1: 100.623

F2: 1.000

SW1: 24038

OF1: 10062.5

PTS1d: 32768

EX: zgdc30

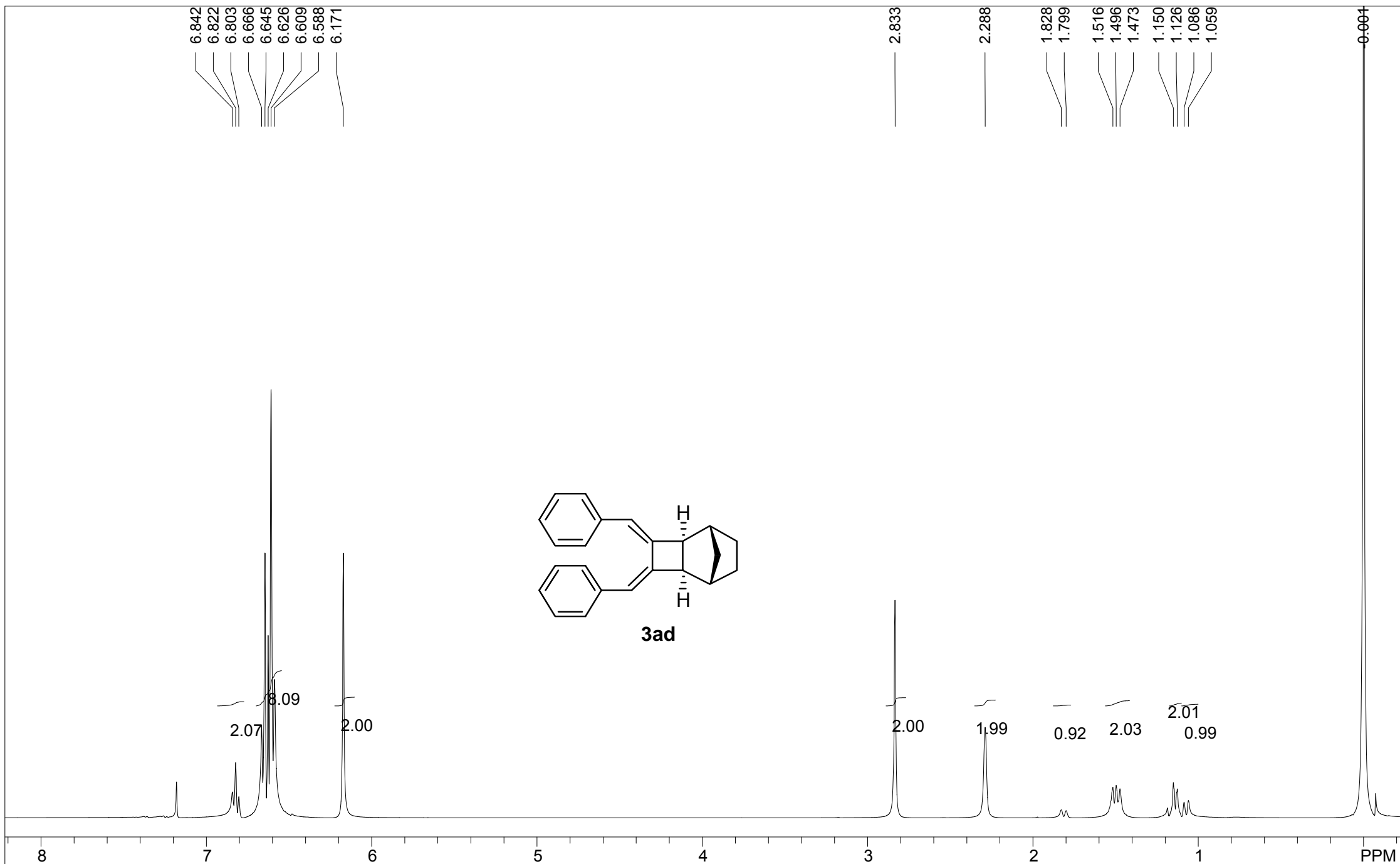
PW: 9.5 usec

PD: 1.2 sec

NA: 1024

LB: 0.0

Nuts - \$pdata



spect, CDCl ₃ ,		USER: nmrsu -- DATE: Tue Jun 10 18:34:38 2014				
F1: 400.132	F2: 1.000	SW1: 8013		OF1: 2429.1		PTS1d: 65536
EX: zg30		PW: 9.8 usec	PD: 1.0 sec	NA: 16	LB: 0.0	Nuts - \$pdata

142.105
136.611
126.862
125.499
125.036
122.421

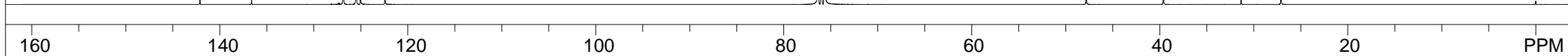
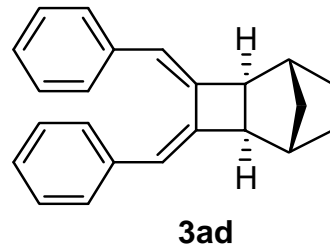
76.311
75.983
75.675

47.813

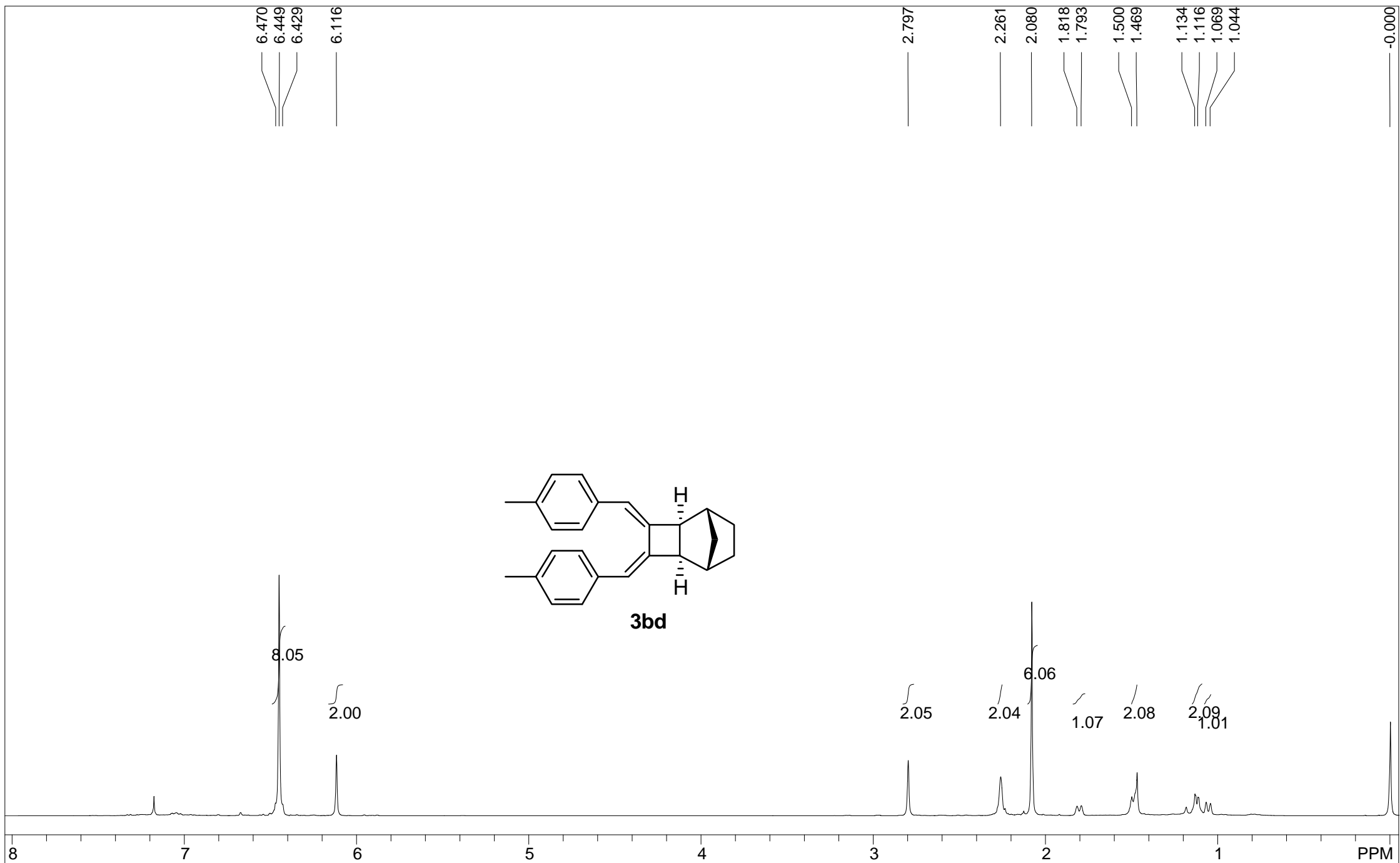
39.607

31.345

27.101



spect, CDC13,		USER: nmrsu -- DATE: Tue May 27 15:45:57 2014					
F1: 100.623	F2: 1.000	SW1: 24038		OF1: 9957.6		PTS1d: 32768	
EX: zgdc30		PW: 9.5 usec	PD: 1.2 sec	NA: 512	LB: 0.0		Nuts - \$pdata



spect. CDCl₃.

USER: nmrsu -- DATE: Tue Jun 10 12:50:55 2014

F1: 400.132 F2: 1.000

SW1: 8013

OF1: 2427.7

PTS1d: 65536

EX: zg30

PW: 9.8 usec

PD: 1.0 sec

NA: 16

LB: 0.0

Nuts - \$pdata

141.564
134.760
133.976
126.729
126.060
121.964

76.313
75.987
75.678

47.700

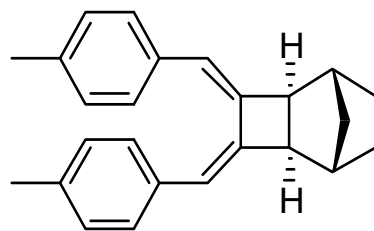
39.581

31.320

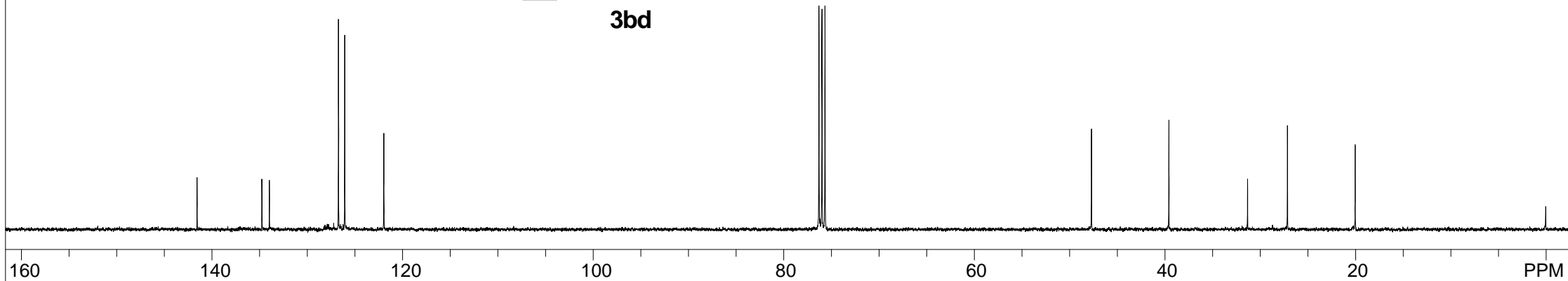
27.127

20.021

0.012



3bd



spect, CDC13,

USER: nmrsu -- DATE: Tue Jun 10 19:01:21 2014

F1: 100.623 F2: 1.000

SW1: 24038

OF1: 9958.7

PTS1d: 32768

EX: zgdc30

PW: 9.5 usec

PD: 1.2 sec

NA: 512

LB: 0.0

Nuts - \$pdata

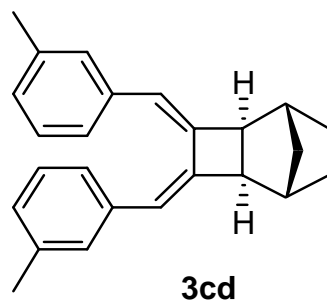
6.724
6.705
6.687
6.677
6.659
6.592
6.574
6.317
6.146

2.820

2.282

1.815
1.789
1.739
1.512
1.492
1.474
1.147
1.143
1.129
1.125
1.078
1.052

-0.000
-0.070



4.08
2.06 2.03 2.00

2.06

2.03

6.06
1.01 2.08

2.01
1.05

8 7 6 5 4 3 2 1 PPM

spect. CDC13.

USER: nmrsu -- DATE: Thu Jun 12 20:00:36 2014

F1: 400.132 F2: 1.000

SW1: 8013

OF1: 2431.7

PTS1d: 65536

EX: zg30

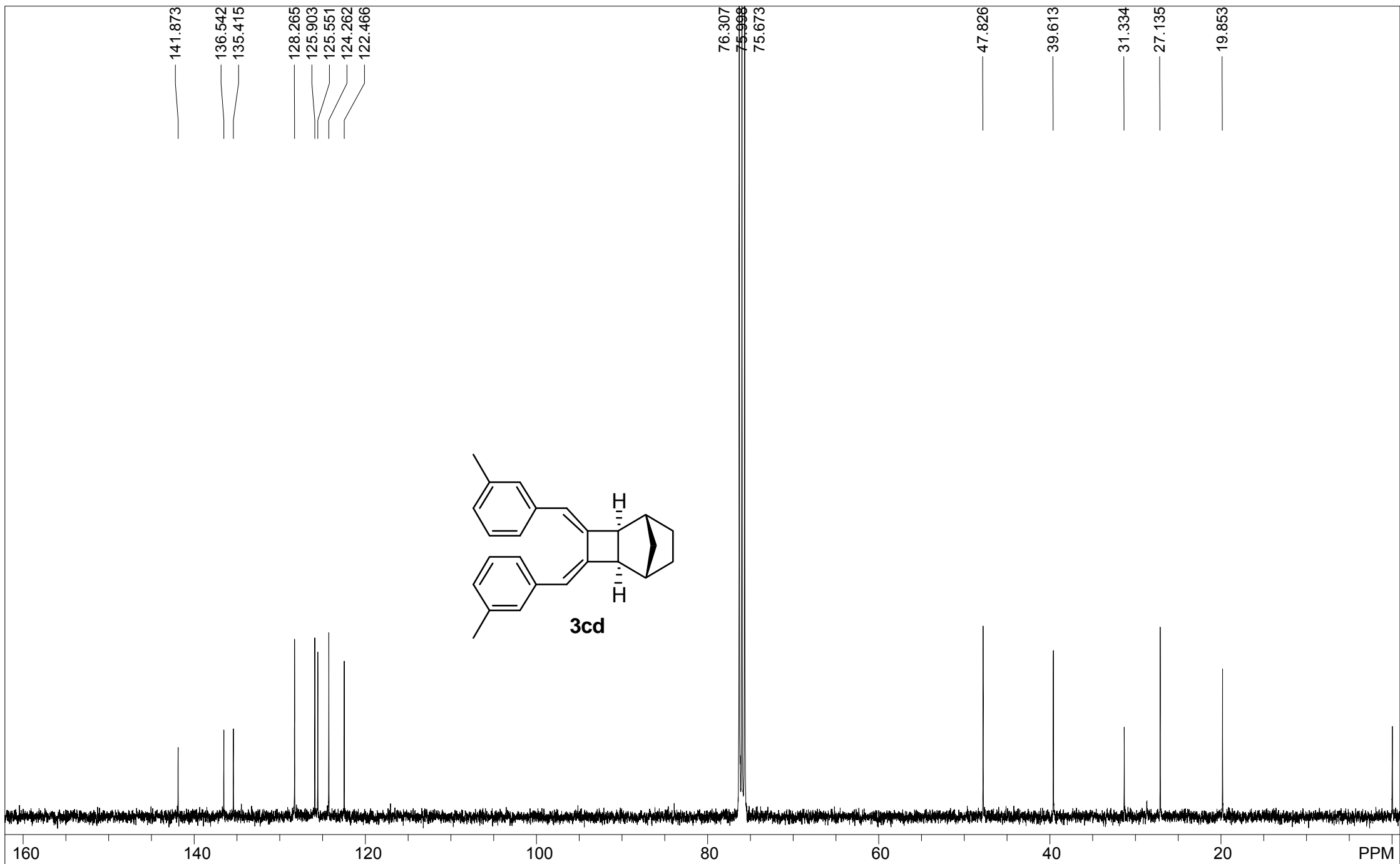
PW: 9.8 usec

PD: 1.0 sec

NA: 64

LB: 0.0

Nuts - \$pdata



spect, CDCl₃,

USER: nmrsu -- DATE: Thu Jun 12 20:46:31 2014

F1: 100.623 F2: 1.000

SW1: 24038

OF1: 9960.1

PTS1d: 32768

EX: zgdc30

PW: 9.5 usec

PD: 1.2 sec

NA: 1024

LB: 0.0

Nuts - \$pdata