Electronic Supplementary Information

Chemoselective Phosphine-Catalyzed Cascade Annulations between Two Different Activated Alkenes: Highly Diastereoselective Syntheses of PolySubstituted Cyclohexanes and Cyclopentenes

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Contents

1. **General Remarks**

Unless otherwise noted, all reactions were carried out in nitrogen atmosphere. Methyl vinyl ketone from commercial sources were used without further purification. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker AV 400 in $CDCl_3$ with tetramethylsilane (TMS) as the internal standard. High resolution ESI mass spectra were acquired with IonSpec QFT-ESI instrument. X-ray crystal diffraction data were collected on a Nonius Kappa CCD diffractometer with Mo Kα radiation (λ = 0.7107 Å) at room temperature. Column chromatography was performed on silica gel (200-300 mesh) using a mixture of petroleum ether/ethyl acetate as eluant.

2. **Typical Preparation of Activated Alkenes 1**[1]

According to a reported procedure,[1] to a mixture of the benzaldehyde (0.53 g, 5.0 mmol) and methyl 2-cyanoacetate (0.50 g, 5.0 mmol) was added commercial grade lithium bromide (0.09 g, 1.0 mmol) at room temperature. After being stirred for 5 min, the resulting mixture was heated at 80 °C in a heating mantle for 10 min. It was then stirred and allowed to cool to room temperature when it solidified. Dichloromethane (10 mL) and water (10 mL) was then added into it. The organic layer was separated and concentrated on a rotary evaporator. The crude product was then recrystallized from ethanol to give **1a** (0.89 g, 95% yield) as a white solid. Other doubly activated alkenes **1b-o** were readily prepared in high yields from the corresponding aryl aldehydes and methyl 2-cyanoacetate or malononitrile by following the above typical procedure.

3. **General Procedures for the Phosphine-Catalyzed Annulations**

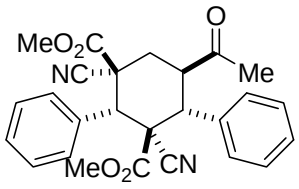
General Procedure for the PPh_3-Catalyzed [2 + 2 + 2] Annulation of **1** and **2a**: Under N_2 atmosphere and at room temperature, to a stirred solution of activated alkene **1** (1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) in 1,4-dioxane (3.0 mL) was added PPh_3 (26 mg, 0.1 mmol). The resulting reaction mixture was further stirred for 24 h. After concentration on a rotary evaporator, the residue was subjected to column chromatography isolation on silica gel (gradient eluant: petroleum ether/ethyl acetate 6:1-3:1) to afford the polysubstituted cyclohexane **3** as a single diastereomer.

General Procedure for the PBu_3-Catalyzed [2 + 2 + 1] Annulation of **1** and **2**: Under N_2 atmosphere and at room temperature, to a stirred solution of activated alkene **1** (1.0 mmol), α,β-unsaturated ketone **2** (0.75 mmol), and hydroquinone (55 mg, 0.5 mmol) in 1,4-dioxane (3.0 mL) was added PBu_3 (23 μL, 0.1 mmol) by the means of microsyringe. The resulting reaction mixture was further stirred till the activated alkene **1** was completely consumed, as monitored by TLC. After concentration on a rotary evaporator,

[1] D. Prajapati, K. C. Lekhok, J. S. Sandhu and A. C. Ghosh, *J. Chem. Soc. Perkin Trans. 1*, 1996, 959.

the residue was isolated by column chromatography on silica gel (gradient eluant: petroleum ether/ethyl acetate 8:1-5:1) to afford the polysubstituted cyclopentene **4** as a single diastereomer.

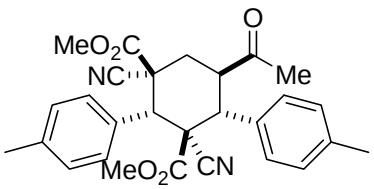
4. **Analytical Data for Compounds 3 and 4**



3a

Table 2, entry 1. Following the general procedure, the activated alkene **1a** (187 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3a** (186 mg, 84% yield).

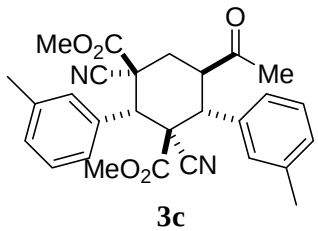
3a: white crystalline solid, mp 234-236 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.67 (m, 2H), 7.39-7.28 (m, 8H), 4.00 (td, *J* = 12.0, 3.2 Hz, 1H), 3.85 (s, 1H), 3.71 (d, *J* = 12.0 Hz, 1H), 3.56 (s, 3H), 3.22 (s, 3H), 2.58 (dd, *J* = 14.0, 3.2 Hz, 1H), 2.41 (dd, *J* = 14.0, 12.0 Hz, 1H), 2.07 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 206.5, 167.1, 166.4, 134.8, 132.6, 129.6, 129.5, 129.01, 128.98, 128.95, 128.7, 116.7, 115.4, 56.7, 54.1, 53.5, 51.4, 50.7, 49.1, 47.3, 37.8, 31.1; HRMS (ESI) calcd for $C_{26}H_{24}N_2O_5Na^+$ requires 467.1577, found 467.1583.



3b

Table 2, entry 2. Following the general procedure, the activated alkene **1b** (201 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3b** (184 mg, 78% yield).

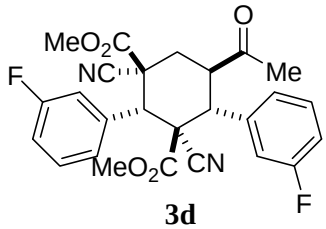
3b: white crystalline solid, mp 238-240 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.54 (d, *J* = 8.4 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.13 (m, 4H), 3.96 (td, *J* = 12.0, 3.2 Hz, 1H), 3.81 (s, 1H), 3.65 (d, *J* = 12.0 Hz, 1H), 3.58 (s, 3H), 3.27 (s, 3H), 2.53 (dd, *J* = 14.0, 3.2 Hz, 1H), 2.40 (dd, *J* = 14.0, 12.0 Hz, 1H), 2.31 (s, 3H), 2.30 (s, 3H), 2.07 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 206.7, 167.2, 166.6, 139.4, 138.8, 131.8, 129.69, 129.65, 129.57, 129.3, 128.5, 116.8, 115.5, 57.0, 54.1, 53.5, 51.1, 50.4, 49.2, 47.4, 37.9, 31.2, 21.2, 21.1; HRMS (ESI) calcd for $C_{28}H_{28}N_2O_5Na^+$ requires 495.1890, found 495.1895.



3c

Table 2, entry 3. Following the general procedure, the activated alkene **1c** (201 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3c** (198 mg, 84% yield).

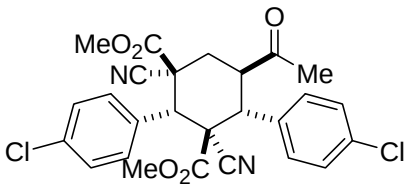
3c: white crystalline solid, mp 191-192 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.51 (d, J = 7.6 Hz, 1H), 7.41 (s, 1H), 7.24-7.11 (m, 6H), 3.98 (td, J = 12.0, 2.8 Hz, 1H), 3.79 (s, 1H), 3.65 (d, J = 12.0 Hz, 1H), 3.57 (s, 3H), 3.26 (s, 3H), 2.56 (dd, J = 14.0, 2.8 Hz, 1H), 2.42 (dd, J = 14.0, 12.0 Hz, 1H), 2.34 (s, 3H), 2.33 (s, 3H), 2.08 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 206.7, 167.2, 166.5, 138.7, 138.6, 134.7, 132.5, 130.3, 130.1, 129.8, 129.3, 128.89, 128.79, 126.5, 125.7, 116.8, 115.4, 56.7, 54.1, 53.5, 51.4, 50.6, 49.1, 47.2, 37.9, 31.2, 21.5, 21.4; HRMS (ESI) calcd for $C_{28}H_{28}N_2O_5Na^+$ requires 495.1890, found 495.1892.



3d

Table 2, entry 4. Following the general procedure, the activated alkene **1d** (205 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3d** (228 mg, 95% yield).

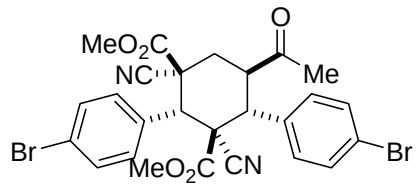
3d: white crystalline solid, mp 227-228 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.51 (d, J = 8.0 Hz, 1H), 7.40-7.31 (m, 3H), 7.19 (d, J = 8.0 Hz, 1H), 7.11-7.00 (m, 3H), 3.95 (td, J = 12.0, 3.2 Hz, 1H), 3.84 (s, 1H), 3.71 (d, J = 12.0 Hz, 1H), 3.62 (s, 3H), 3.32 (s, 3H), 2.61 (dd, J = 14.0, 3.2 Hz, 1H), 2.37 (dd, J = 14.0, 12.0 Hz, 1H), 2.13 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 206.0, 166.8, 166.2, 162.7 (d, J_{CF} = 246.4 Hz, 1C), 162.5 (d, J_{CF} = 246.7 Hz, 1C), 137.1 (d, J_{CF} = 7.1 Hz, 1C), 134.6 (d, J_{CF} = 7.3 Hz, 1C), 130.8 (t, J_{CF} = 8.9 Hz, 2C), 130.7 (d, J_{CF} = 9.2 Hz, 1C), 125.2 (d, J_{CF} = 3.0 Hz, 1C), 124.2, 117.0 (d, J_{CF} = 8.3 Hz, 1C), 116.8 (d, J_{CF} = 10.0 Hz, 1C), 116.3 (d, J_{CF} = 7.4 Hz, 1C), 115.8 (d, J_{CF} = 11.7 Hz, 1C), 115.7, 114.9, 56.2, 54.4, 53.8, 50.8, 50.0, 48.8, 47.1, 37.8, 31.1; HRMS (ESI) calcd for $C_{26}H_{22}F_2N_2O_5Na^+$ requires 503.1389, found 503.1387.



3e

Table 2, entry 5. Following the general procedure, the activated alkene **1e** (221mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3e** (182 mg, 71% yield).

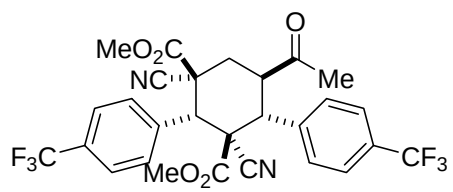
3h: white crystalline solid, mp 260-262 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS):δ 7.62 (d, *J* = 8.4 Hz, 2H), 7.33 (m, 6H), 3.92 (td, *J* = 12.0, 3.2 Hz, 1H), 3.83 (s, 1H), 3.68 (d, *J* = 12.0 Hz, 1H), 3.62 (s, 3H), 3.30 (s, 3H), 2.58 (dd, *J* = 14.0, 3.2 Hz, 1H), 2.35 (dd, *J* = 14.0, 12.0 Hz, 1H), 2.10 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 205.9, 166.8, 166.3, 135.9, 135.2, 133.2, 130.9, 129.9, 129.4, 129.33, 129.29, 116.4, 115.1, 56.3, 54.4, 53.9, 50.6, 49.8, 48.8, 47.2, 37.8, 31.1; HRMS (ESI) calcd for $C_{26}H_{22}Cl_2N_2O_5Na^+$ requires 535.0798, found 535.0799.



3f

Table 2, entry 6. Following the general procedure, the activated alkene **1f** (265mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3f** (264 mg, 88% yield).

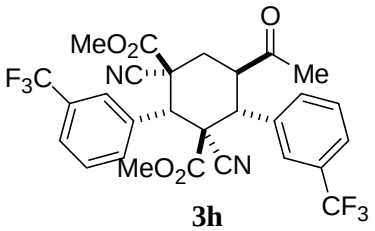
3f: white crystalline solid, mp 274-276 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.55 (d, *J* = 8.4 Hz, 2H), 7.49 (m, 4H), 7.24 (d, *J* = 8.4 Hz, 2H), 3.92 (td, *J* = 12.0, 3.0 Hz, 1H), 3.81 (s, 1H), 3.66 (d, *J* = 12.0 Hz, 1H), 3.62 (s, 3H), 3.30 (s, 3H), 2.58 (dd, *J* = 14.0, 3.0 Hz, 1H), 2.35 (dd, *J* = 14.0, 12.0 Hz, 1H), 2.10 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 205.9, 166.8, 166.3, 133.8, 132.4, 132.3, 131.4, 131.2, 130.2, 124.2, 123.4, 116.4, 115.1, 56.2, 54.4, 53.9, 50.7, 49.9, 48.8, 47.2, 37.8, 31.1; HRMS (ESI) calcd for $C_{26}H_{22}Br_2N_2O_5Na^+$ requires 622.9788, found 622.9796.



3g

Table 2, entry 7. Following the general procedure, the activated alkene **1g** (255 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3g** (264 mg, 91% yield).

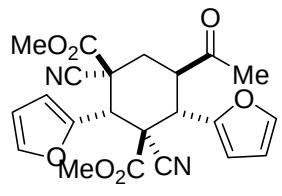
3g: white crystalline solid, mp 233-235 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.83 (d, *J* = 8.0 Hz, 2H), 7.64 (d, *J* = 8.0 Hz, 2H), 7.62 (d, *J* = 8.0 Hz, 2H), 7.51 (d, *J* = 8.0 Hz, 2H), 4.01 (td, *J* = 12.0, 2.8 Hz, 1H), 3.95 (s, 1H), 3.80 (d, *J* = 12.0 Hz, 1H), 3.62 (s, 3H), 3.27 (s, 3H), 2.65 (dd, *J* = 13.6, 2.8 Hz, 1H), 2.38 (dd, *J* = 13.6, 12.0 Hz, 1H), 2.13 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 205.6, 166.7, 166.1, 138.7, 136.3, 131.9 (q, J_{CF} = 32.6 Hz, 1C), 131.3 (q, J_{CF} = 32.7 Hz, 1C), 130.1, 129.1, 126.1 (t, J_{CF} = 2.5 Hz, 1C), 123.6 (q, J_{CF} = 270.6 Hz, 1C), 123.6 (q, J_{CF} = 270.6 Hz, 1C), 116.2, 114.9, 56.0, 54.5, 53.9, 50.9, 50.1, 48.6, 47.2, 37.9, 31.0; HRMS (ESI) calcd for $C_{28}H_{22}F_6N_2O_5Na^+$ requires 603.1325, found 603.1334.



3h

Table 2, entry 8. Following the general procedure, the activated alkene **1h** (255 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3h** (270 mg, 93% yield).

3h: white crystalline solid, mp 215-216 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 8.22 (d, *J* = 8.0 Hz, 1H), 7.67-7.49 (m, 7H), 4.03 (td, *J* = 12.0, 2.8 Hz, 1H), 3.94 (s, 1H), 3.81 (d, *J* = 13.6 Hz, 1H), 3.62 (s, 3H), 3.28 (s, 3H), 2.65 (dd, *J* = 13.6, 2.8 Hz, 1H), 2.38 (dd, *J* = 13.6, 12.0 Hz, 1H), 2.15 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 205.6, 166.6, 166.1, 135.9, 133.6, 132.1, 131.41 (q, J_{CF} = 32.6 Hz, 1C), 131.39 (q, J_{CF} = 32.6 Hz, 1C), 130.0, 129.7, 127.2 (q, J_{CF} = 3.7 Hz, 1C), 126.6 (q, J_{CF} = 4.3 Hz, 1C), 126.0 (q, J_{CF} = 3.7 Hz, 1C), 123.6 (q, J_{CF} = 270.8 Hz, 1C), 123.5 (q, J_{CF} = 270.8 Hz, 1C), 116.2, 114.7, 56.4, 54.4, 53.9, 50.8, 50.1, 48.8, 47.0, 37.8, 31.0; HRMS (ESI) calcd for $C_{28}H_{22}F_6N_2O_5Na^+$ requires 603.1325, found 603.1327.

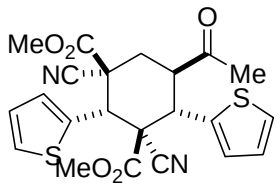


3i

Table 2, entry 9. Following the general procedure, the activated alkene **1i** (177 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3i** (169 mg, 80% yield).

3i: white crystalline solid, mp 186-188 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS):δ 7.39 (s, 2H), 6.74 (d, *J* = 3.2 Hz, 1H), 6.36 (m, 1H), 6.32 (m, 1H), 6.28 (d, *J* = 3.2 Hz, 1H), 4.11 (s, 1H), 3.86 (td, *J* = 12.0, 3.2

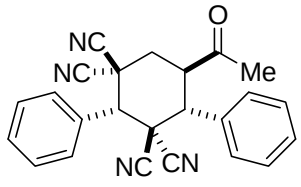
Hz, 1H), 3.80 (s, 1H), 3.77 (s, 3H), 3.62 (s, 3H), 2.52 (dd, *J* = 14.0, 3.2 Hz, 1H), 2.28 (dd, *J* = 14.0, 12.0 Hz, 1H), 2.14 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 206.2, 167.1, 166.6, 148.3, 146.3, 143.5, 143.2, 115.8, 114.2, 111.0, 110.9, 110.7, 110.6, 54.6, 54.5, 54.2, 48.0, 46.0, 45.2, 44.4, 36.9, 30.3; HRMS (ESI) calcd for $C_{22}H_{20}N_2O_7Na^+$ requires 447.1163, found 447.1161.



3j

Table 2, entry 10. Following the general procedure, the activated alkene **1j** (193 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **3j** (193 mg, 85% yield).

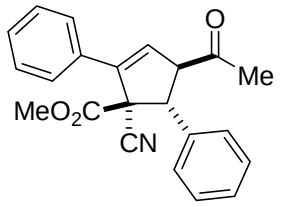
3j: white crystalline solid, mp 228-230 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS):δ 7.62 (d, *J* = 4.0 Hz, 1H), 7.29 (d, *J* = 5.2 Hz, 1H), 7.24 (d, *J* = 5.2 Hz, 1H), 7.15 (d, *J* = 4.0 Hz, 1H), 7.02 (t, *J* = 4.0 Hz, 1H), 6.97 (t, *J* = 4.0 Hz, 1H), 4.16 (s, 1H), 3.96 (d, *J* = 12.0 Hz, 1H),3.86 (td, *J* = 12.0, 3.2 Hz, 1H), 3.68 (d, *J* = 12.0 Hz, 1H), 3.62 (s, 3H), 3.46 (s, 3H), 2.55 (dd, *J* = 14.0, 3.2 Hz, 1H), 2.33 (dd, *J* = 14.0, 12.0 Hz, 1H), 2.12 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 206.0, 167.0, 166.4, 136.4, 132.9, 128.8, 127.7, 127.5, 127.3, 126.8, 126.1, 116.3, 115.2, 58.3, 54.4, 54.0, 50.1, 48.6, 46.8, 45.9, 37.6, 31.1; HRMS (ESI) calcd for $C_{22}H_{20}N_2O_5\ S_2Na^+$ requires 479.0706, found 479.0701.



3k

Table 2, entry 11. Following the general procedure, the activated alkene **1k** (154 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) employed to give **3k** (71 mg, 38% yield).

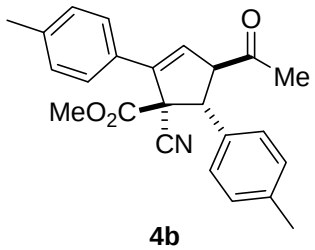
3k: white crystalline solid, mp 157-159 ºC; ^{1}H NMR (C_2D_6OS, 400 MHz, TMS): δ 7.83 (d, *J* = 6.4 Hz, 2H), 7.63 (d, *J* = 6.8 Hz, 3H), 7.55 (d, *J* = 6.8 Hz, 2H), 7.45 (m, 3H), 4.90 (s, 1H), 4.02 (d, *J* = 12.0 Hz, 1H), 3.93 (td, *J* = 12.0, 2.8 Hz, 1H), 3.51 (s, 1H), 3.42 (dd, *J* = 13.6, 2.8 Hz, 1H), 2.20 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 205.3, 134.1, 132.3, 130.9, 129.7, 129.4, 129.1, 128.9, 128.8, 113.7, 113.0, 112.9, 111.9, 49.2, 47.8, 45.4, 44.9, 36.7, 35.5, 30.2; HRMS (ESI) calcd for $C_{24}H_{18}N_4ONa^+$ requires 401.1373, found 403.1371.



4a

Table 3, entry 1. Following the general procedure, the activated alkene **1a** (187 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4a** (193 mg, 95% yield).

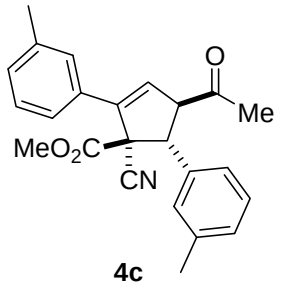
4a: white crystalline solid, mp 115-117 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.48 (d, J = 7.1 Hz, 2H), 7.36 (m, 8H), 6.52 (s, 1H), 4.49 (d, J = 9.2 Hz, 1H), 4.36 (d, J = 9.2 Hz, 1H), 3.72 (s, 3H), 2.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 204.4, 168.1, 141.4, 135.3, 132.3, 130.6, 129.1, 129.1, 129.0, 128.9, 128.6, 126.1, 116.4, 62.0, 61.8, 56.6, 54.0, 29.5; HRMS (ESI) calcd for $C_{22}H_{19}NO_3Na^+$ requires 368.1257, found 368.1250.



4b

Table 3, entry 2. Following the general procedure, the activated alkene **1b** (201 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4b** (179 mg, 96% yield).

4b: white crystalline solid, mp 185-187 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.37 (d, J = 7.4 Hz, 2H), 7.27 (s, 2H), 7.21 (d, J = 7.4 Hz, 2H), 7.16 (d, J = 7.4 Hz, 2H), 6.46 (s, 1H), 4.43 (d, J = 9.2 Hz, 1H), 4.34 (d, J = 9.2 Hz, 1H), 3.74 (s, 3H), 2.35 (s, 6H), 2.18 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 204.7, 168.3, 141.4, 139.1, 138.8, 133.0, 132.1, 129.8, 129.6, 129.4, 128.4, 126.0, 116.5, 62.1, 61.7, 56.5, 53.9, 29.5, 21.3, 21.3; HRMS (ESI) calcd for $C_{24}H_{23}NO_3Na^+$ requires 396.1570, found 396.1570.

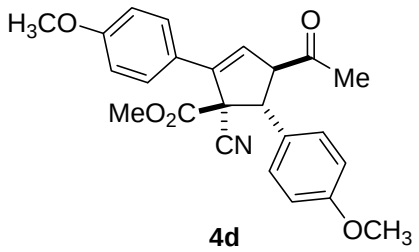


4c

Table 3, entry 3. Following the general procedure, the activated alkene **1c** (201 mg, 1.0 mmol) and

methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4c** (171 mg, 92% yield).

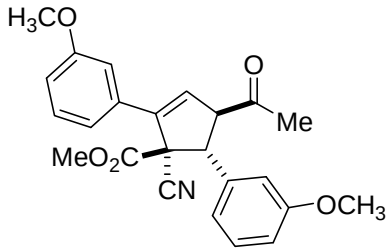
4c: white crystalline solid, mp 127-129 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.29-724 (m, 4H), 7.20-7.14 (m, 4H), 6.49 (d, *J* = 2.0 Hz, 1H), 4.44 (d, *J* = 9.0 Hz, 1H), 4.35 (dd, *J* = 9.0, 2.0 Hz, 1H), 3.74 (s, 3H), 2.37 (s, 3H), 2.36 (s, 3H), 2.19 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 204.6, 168.2, 141.6, 138.7, 138.7, 135.12, 132.3, 130.3, 129.9, 129.7, 129.3, 128.9, 128.7, 126.9, 125.5, 123.1, 116.4, 62.0, 61.7, 56.6, 53.9, 29.4, 21.5, 21.5; HRMS (ESI) calcd for $C_{24}H_{23}NO_3Na^+$ requires 396.1570, found 396.1571.



4d

Table 3, entry 4. Following the general procedure, the activated alkene **1l** (217 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4d** (146 mg, 72% yield).

4d: white crystalline solid, mp 150-152 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.41 (d, *J* = 8.0 Hz, 2H), 7.31 (d, *J* = 8.0 Hz, 2H), 6.91 (d, *J* = 8.0 Hz, 2H), 6.87 (d, *J* = 8.0 Hz, 2H), 6.39 (s, 1H), 4.42 (d, *J* = 8.8 Hz, 1H), 4.29 (d, *J* = 8.8 Hz, 1H), 3.79 (s, 6H), 3.74 (s, 3H), 2.17 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 204.9, 168.4, 160.1, 159.8, 140.8, 129.7, 128.5, 127.4, 127.2, 124.8, 116.7, 114.4, 114.2, 62.2, 61.9, 56.2, 55.3, 55.3, 54.0, 29.4; HRMS (ESI) calcd for $C_{24}H_{23}NO_5Na^+$ requires 428.1468, found 428.1463.

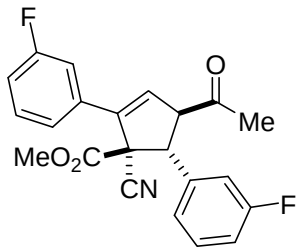


4e

Table 3, entry 5. Following the general procedure, the activated alkene **1m** (217 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4e** (182 mg, 90% yield).

4e: white crystalline solid, mp 129-131 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.36-7.27 (m, 2H), 7.06 (d, *J* = 7.7 Hz, 1H), 7.02-6.95 (m, 2H), 6.93-6.86 (m, 3H), 6.51 (d, *J* = 2.0 Hz, 1H), 4.44 (d, *J* = 9.2 Hz, 1H), 4.35 (dd, *J* = 9.2, 2.0 Hz, 1H), 3.82 (s, 3H), 3.81 (s, 3H) 3.76 (s, 3H), 2.20 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 204.4, 168.1, 159.8, 159.8, 141.4, 136.6, 133.5, 130.7, 130.1, 129.9, 120.7,

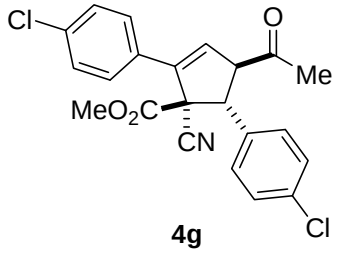
118.4, 116.3, 114.8, 114.6, 114.0, 111.7, 62.0, 61.6, 56.6, 55.3, 54.0, 29.4; HRMS (ESI) calcd for $C_{24}H_{23}NO_5Na^+$ requires 428.1468, found 428.1463.



4f

Table 3, entry 6. Following the general procedure, the activated alkene **1d** (205 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4f** (149 mg, 78% yield).

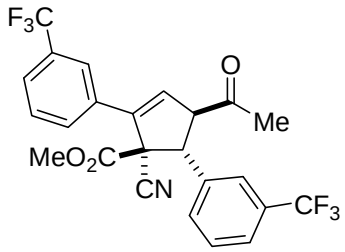
4f: white crystalline solid, mp 143-145 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.42-7.31 (m, 2H), 7.26-7.16 (m, 3H), 7.10-7.04 (m, 3H), 6.55 (d, J = 1.6 Hz, 1H), 4.51 (d, J = 8.8 Hz, 1H), 4.32 (dd, J = 8.8, 1.6 Hz, 1H), 3.78 (s, 3H), 2.24 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 203.5, 167.7, 162.9 (d, J_{CF} = 246.1 Hz, 1C), 162.8 (d, J_{CF} = 245.6 Hz, 1C), 140.3 (d, J_{CF} = 2.4 Hz, 1C), 137.6 (d, J_{CF} = 7.2 Hz, 1C), 134.2 (d, J_{CF} = 7.8 Hz, 1C), 131.6, 130.7 (d, J_{CF} = 8.2 Hz, 1C), 130.6 (d, J_{CF} = 8.4 Hz, 1C), 124.2 (d, J_{CF} = 3.0 Hz, 1C), 121.9, 116.4, 116.2 (d, J_{CF} = 4.1 Hz, 1C), 115.9 (d, J_{CF} = 9.9 Hz, 1C), 115.6 (d, J_{CF} = 22.0 Hz, 1C), 113.3 (d, J_{CF} = 22.7 Hz, 1C), 61.9, 61.6, 55.7, 54.2, 29.4; HRMS (ESI) calcd for $C_{22}H_{19}NO_3Na^+$ requires 404.1069, found 404.1066.



4g

Table 3, entry 7. Following the general procedure, the activated alkene **1e** (221 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4g** (185 mg, 89% yield).

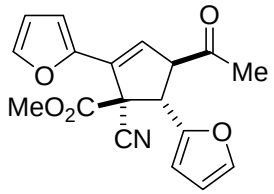
4g: white crystalline solid, mp 181-183 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.42-7.31 (m, 8H), 6.50 (d, J = 2.2 Hz, 1H), 4.48 (d, J = 9.1 Hz, 1H), 4.30 (dd, J = 9.1, 2.2 Hz, 1H), 3.75 (s, 3H), 2.22 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 203.7, 167.6, 140.4, 135.2, 135.0, 133.6, 130.9, 130.6, 129.9, 129.4, 129.2, 127.5, 116.1, 61.9, 61.7, 55.6, 54.2, 29.5; HRMS (ESI) calcd for $C_{22}H_{17}Cl_2NO_3Na^+$ requires 436.0477, found 436.0469.



4h

Table 3, entry 8. Following the general procedure, activated alkene **1h** (255 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4h** (142 mg, 59% yield).

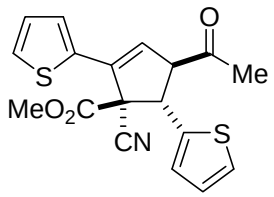
4h: white crystalline solid, mp 139-141 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.74-7.50 (m, 8H), 6.64 (s, 1H), 4.63 (d, J = 8.8 Hz, 1H), 4.38 (d, J = 8.8 Hz, 1H), 3.78 (s, 3H), 2.28 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 203.0, 167.4, 140.2, 136.2, 132.8, 132.0, 131.8, 131.5 (q, J_{CF} = 32.7 Hz, 1C), 131.3 (q, J_{CF} = 32.7 Hz, 1C), 129.7, 129.6, 129.2, 125.9 (q, J_{CF} = 37.0 Hz, 1C), 125.5 (q, J_{CF} = 37.0 Hz, 1C), 123.8 (q, J_{CF} = 270.7 Hz, 1C), 123.3 (q, J_{CF} = 38.4 Hz, 1C), 115.7, 62.0, 61.6, 55.6, 54.3, 29.4; HRMS (ESI) calcd for $C_{24}H_{17}F_6NO_3Na^+$ requires 504.1005, found 504.1002.



4i

Table 3, entry 9. Following the general procedure, activated alkene **1i** (177 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4i** (146 mg, 90% yield).

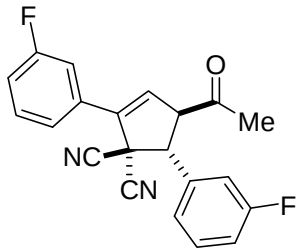
4i: reddish solid, mp 124-126 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.43 (m, 2H), 6.49 (m, 2H), 6.42-6.36 (m, 3H), 4.65 (d, J = 8.0 Hz, 1H), 4.31 (dd, J = 8.0, 2.5 Hz, 1H), 3.89 (s, 3H), 2.30 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 203.4, 167.7, 149.3, 146.7, 143.6, 143.3, 130.8, 126.6, 115.1, 111.7, 110.7, 109.2, 109.0, 61.3, 59.3, 54.3, 48.7, 28.8; HRMS (ESI) calcd for $C_{18}H_{15}NO_5Na^+$ requires 348.0842, found 348.0845.



4j

Table 3, entry 10. Following the general procedure, the activated alkene **1j** (193 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4j** (178 mg, 99% yield).

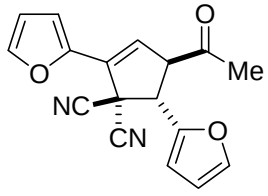
4j: reddish solid, mp 76-79 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.29 (s, 2H), 7.12 (s, 2H), 7.04-6.99 (m, 2H), 6.39 (s, 1H), 4.81 (d, J = 8.2 Hz, 1H), 4.28 (d, J = 8.2 Hz, 1H), 3.86 (s, 3H), 2.26 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 203.5, 167.7, 137.9, 135.1, 134.9, 128.5, 127.9, 127.5, 127.2, 126.6, 125.8, 125.7, 115.5, 63.7, 62.7, 54.3, 51.1, 29.3; HRMS (ESI) calcd for $C_{18}H_{15}NS_2O_3Na^+$ requires 380.0386, found 380.0382.



4k

Table 3, entry 11. Following the general procedure, the activated alkene **1n** (172 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4k** (88 mg, 51% yield).

4k: white crystalline solid, mp 154-156 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.43 (m, 1H), 7.25 (m, 2H), 7.14 (m, 2H), 6.95 (t, J = 8.4 Hz, 1H), 6.85 (d, J = 7.7 Hz, 1H), 6.76 (d, J = 9.3 Hz, 1H), 6.70 (s, 1H), 4.68 (d, J = 9.2 Hz, 1H), 3.94 (d, J = 9.3 Hz, 1H), 2.36 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 193.8, 163.03 (d, J_{CF} = 246.2 Hz, 1C), 162.96 (d, J_{CF} = 247.2 Hz, 1C), 152.9, 140.6 (d, J_{CF} = 7.3 Hz, 1C), 134.5 (d, J_{CF} = 7.8 Hz, 1C), 131.2 (d, J_{CF} = 7.8 Hz, 1C), 130.9 (d, J_{CF} = 8.9 Hz, 1C), 130.8, 124.4 (d, J_{CF} = 3.1 Hz, 1C), 122.9 (d, J_{CF} = 3.8 Hz, 1C), 117.0 (d, J_{CF} = 20.9 Hz, 1C), 115.8 (d, J_{CF} = 22.4 Hz, 1C), 115.2 (d, J_{CF} = 20.9 Hz, 1C), 114.3 (d, J_{CF} = 21.8 Hz, 1C), 113.5, 111.2, 63.6, 53.7, 45.6, 27.9; HRMS (ESI) calcd for $C_{21}H_{14}N_2F_2ONa^+$ requires 371.0966, found 371.0958.



4l

Table 3, entry 12. Following the general procedure, the activated alkene **1o** (144 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4l** (131 mg, 90% yield).

4l: reddish solid, mp 110-113 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.51 (s, 2H), 6.82 (d, J = 3.5 Hz, 1H), 6.55 (d, J = 3.3 Hz, 1H), 6.52 (dd, J = 3.5, 1.8 Hz, 1H), 6.50 (d, J = 2.6 Hz, 1H), 6.45 (dd, J = 3.3, 1.8 Hz, 1H), 4.71 (d, J = 8.3 Hz, 1H), 4.45 (dd, J = 8.3, 2.6 Hz, 1H), 2.33 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 202.0, 146.9, 145.2, 144.5, 144.1, 128.1, 126.7, 113.9, 112.1, 111.9, 111.1, 110.6, 110.6,

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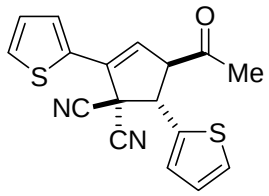
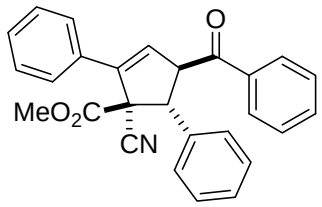


4m

Table 3, entry 13. Following the general procedure, the activated alkene **1p** (160 mg, 1.0 mmol) and methyl vinyl ketone **2a** (53 mg, 0.75 mmol) were employed to give **4m** (155 mg, 96% yield).

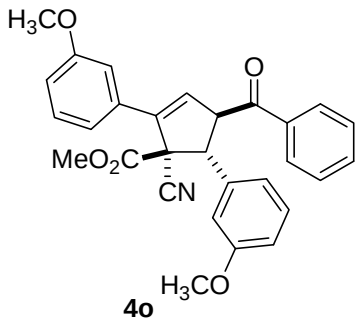
4m: reddish solid, mp 146-148 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 7.46 (s, 1H), 7.39 (d, *J* = 3.9 Hz, 2H), 7.30 (s, 1H), 7.11 (br s, 2H), 6.38 (s, 1H), 4.84 (d, *J* = 8.8 Hz), 4.36 (d, *J* = 8.8 Hz), 2.31 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 202.2, 135.4, 133.0, 132.5, 128.3, 128.2, 128.1, 127.9, 127.7, 127.0, 126.8, 114.1, 112.2, 62.8, 52.2, 48.8, 29.7; HRMS (ESI) calcd for $C_{17}H_{12}N_2S_2ONa^+$ requires 347.0283, found 347.0285.



4n

Table 3, entry 14. Following the general procedure, the activated alkene **1a** (187 mg, 1.0 mmol) and phenyl vinyl ketone **2b** (99 mg, 0.75 mmol) were employed to give **4n** (103 mg, 52% yield).

4n: white crystalline solid, mp 143-145 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 8.03 (d, *J* = 7.3 Hz, 2H), 7.62 (t, *J* = 7.4 Hz, 1H), 7.54-7.42 (m, 6H), 7.40-7.31 (m, 6H), 6.48 (d, *J* = 2.2 Hz, 1H), 5.24 (dd, *J* = 9.1, 2.2 Hz, 1H), 4.94 (d, *J* = 9.1 Hz, 1H), 3.76 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 195.2, 168.1, 141.7, 135.6, 135.1, 134.0, 132.3, 131.2, 129.3, 129.1, 129.0, 128.9, 128.8, 128.7, 128.6, 126.1, 116.7, 61.4, 56.7, 56.0, 54.0; HRMS (ESI) calcd for $C_{27}H_{21}NO_3Na^+$ requires 430.1414, found 430.1411.



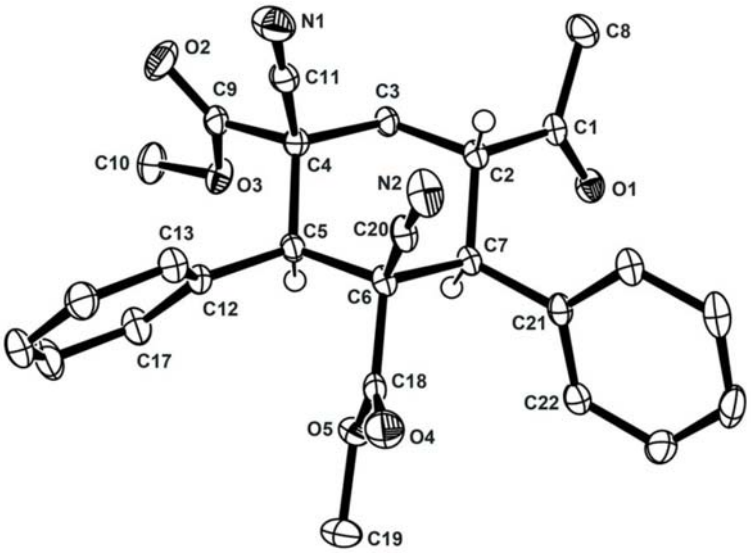
4o

Table 3, entry 15. Following the general procedure, the activated alkene **1m** (217 mg, 1.0 mmol) and

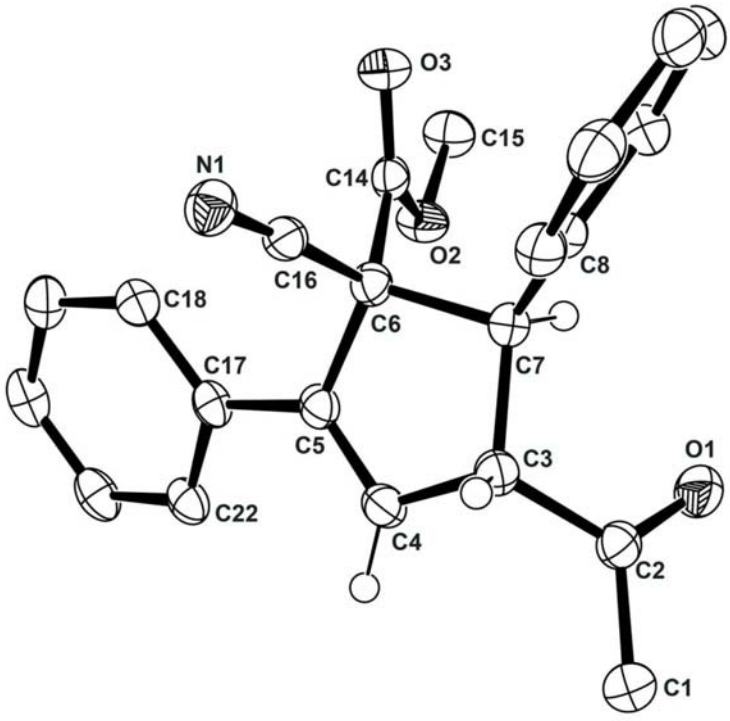
phenyl vinyl ketone **2b** (99 mg, 0.75 mmol) were employed to give **4o** (200 mg, 86% yield).

4o: white crystalline solid, mp 139-141 °C; ^{1}H NMR ($CDCl_3$, 400 MHz, TMS): δ 8.03 (d, *J* = 7.9 Hz, 2H), 7.63-7.47 (m, 3H), 7.28 (m, 2H), 7.02 (m, 2H), 6.97 (s, 1H), 6.94 (s, 1H), 6.86 (d, *J* = 8.4 Hz, 2H), 6.46 (s, 1H), 5.22 (d, *J* = 9.2 Hz, 1H), 4.91 (d, *J* = 9.2 Hz, 1H), 3.78 (s, 3H), 3.77 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz, TMS): δ 195.2, 168.1, 159.7, 141.6, 136.6, 135.5, 133.9, 133.6, 131.4, 129.91, 129.89, 129.0, 128.75, 128.69, 120.8, 118.4, 116.6, 114.8, 114.6, 113.8, 111.6, 61.4, 56.5, 56.0, 55.3, 55.2, 53.9; HRMS (ESI) calcd for $C_{29}H_{25}NO_5Na^+$ requires 490.1625, found 490.1620.

5. **ORTEP drawings for 3a and 4a**

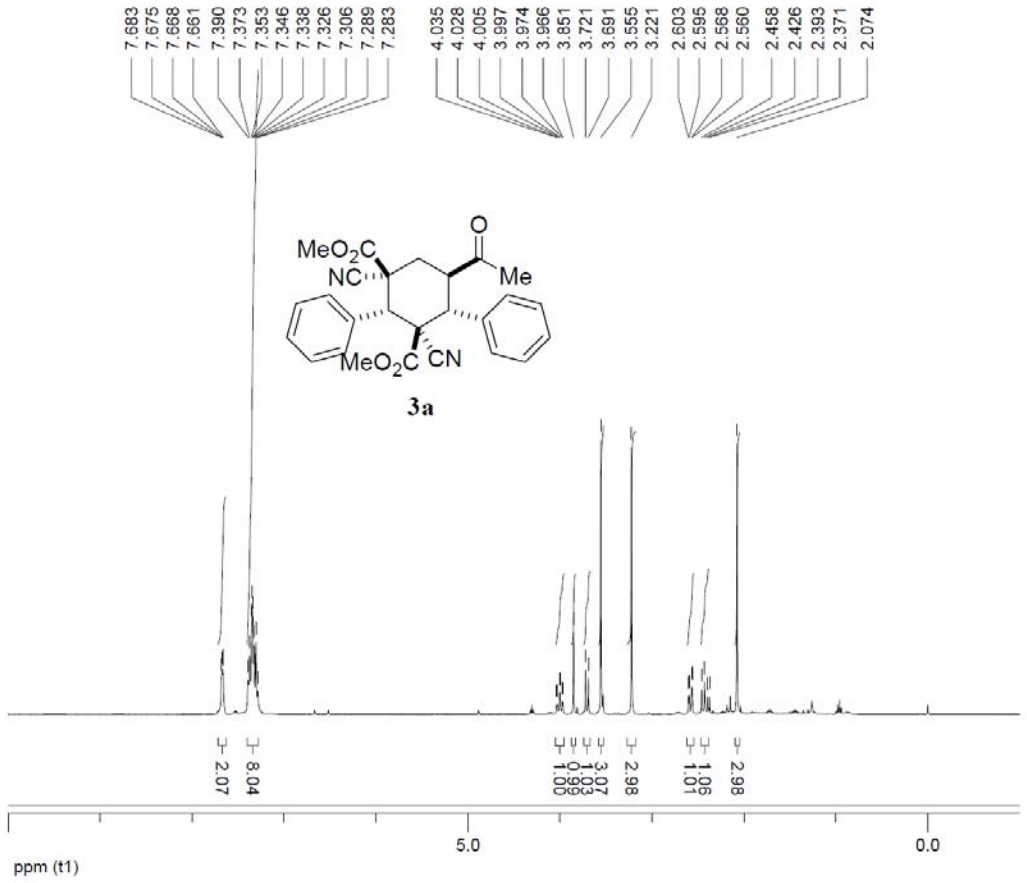


Scheme 1 ORTEP drawing for the [2 + 2 + 2] annulation product **3a**



Scheme 2 ORTEP drawing for the [2 + 2 + 1] annulation product **4a**

6. ^{1}H NMR and ^{13}C NMR Spectra of 3 and 4



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22 Jul 2010
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1r-H.mrc

Spectrum Title:
PROTON

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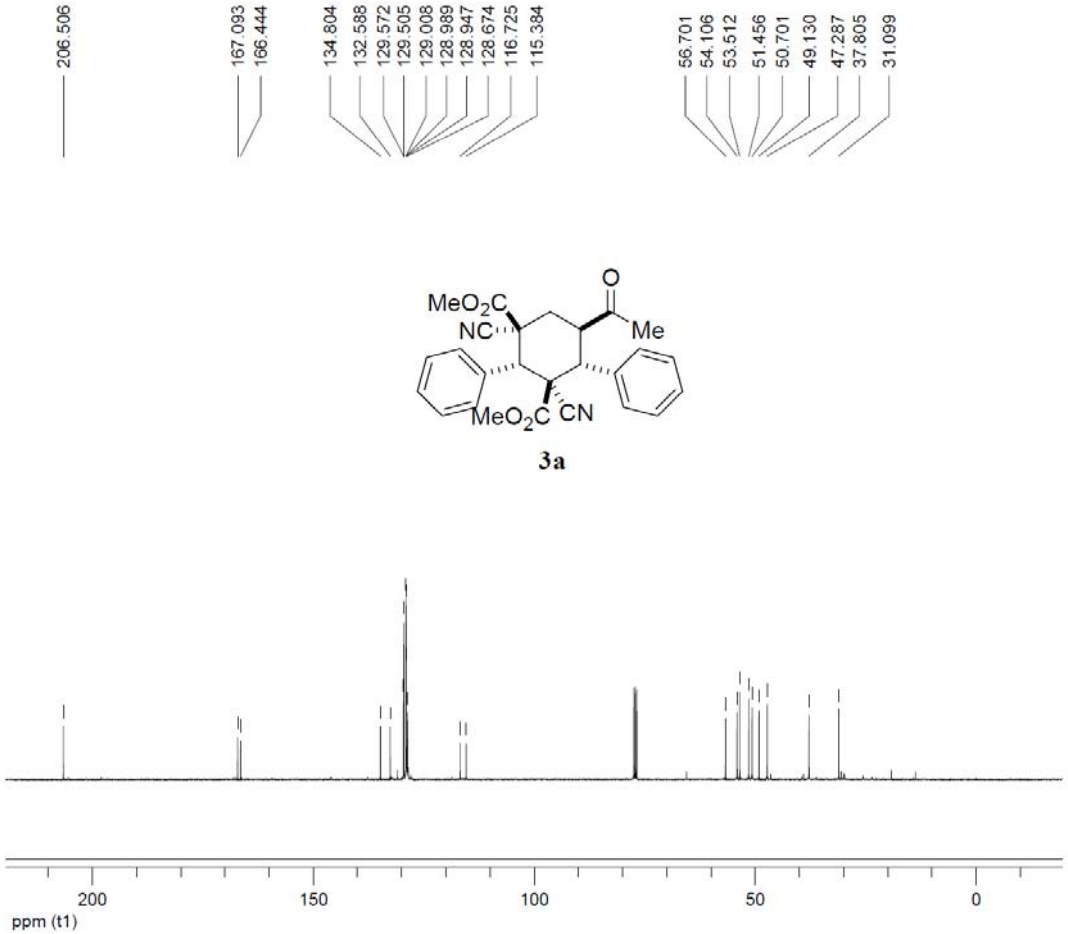

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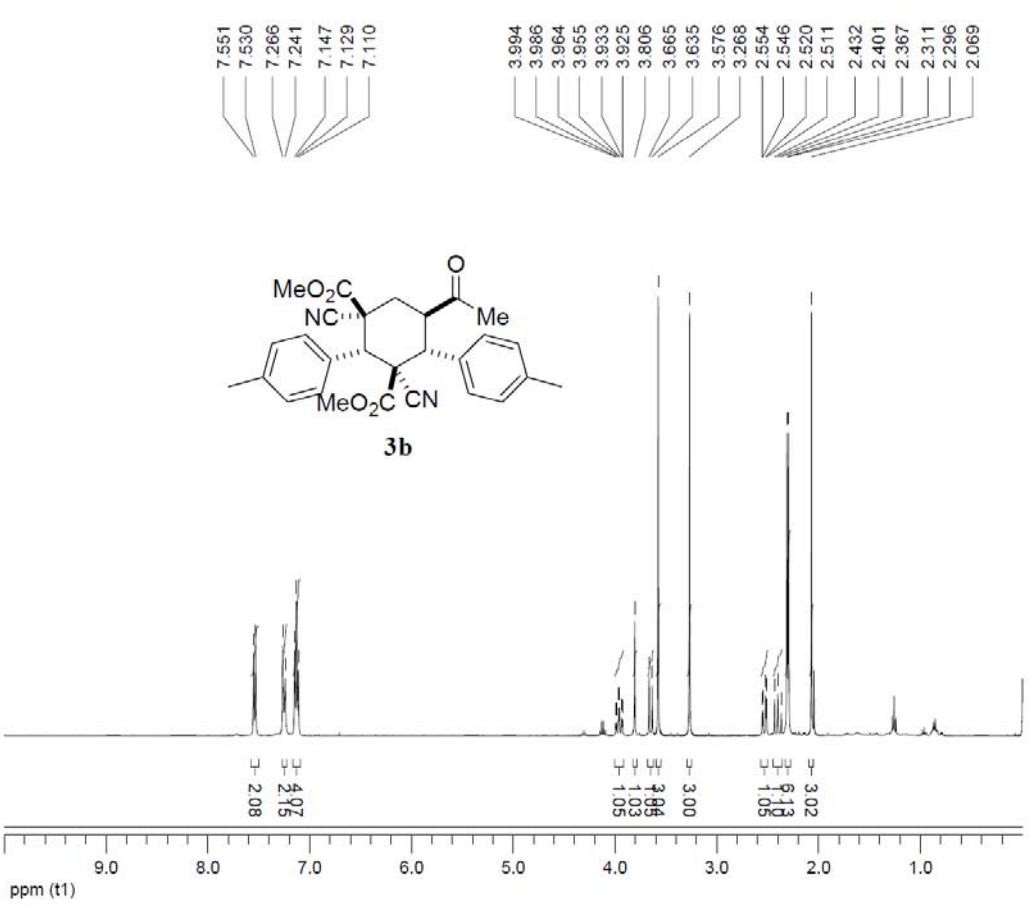

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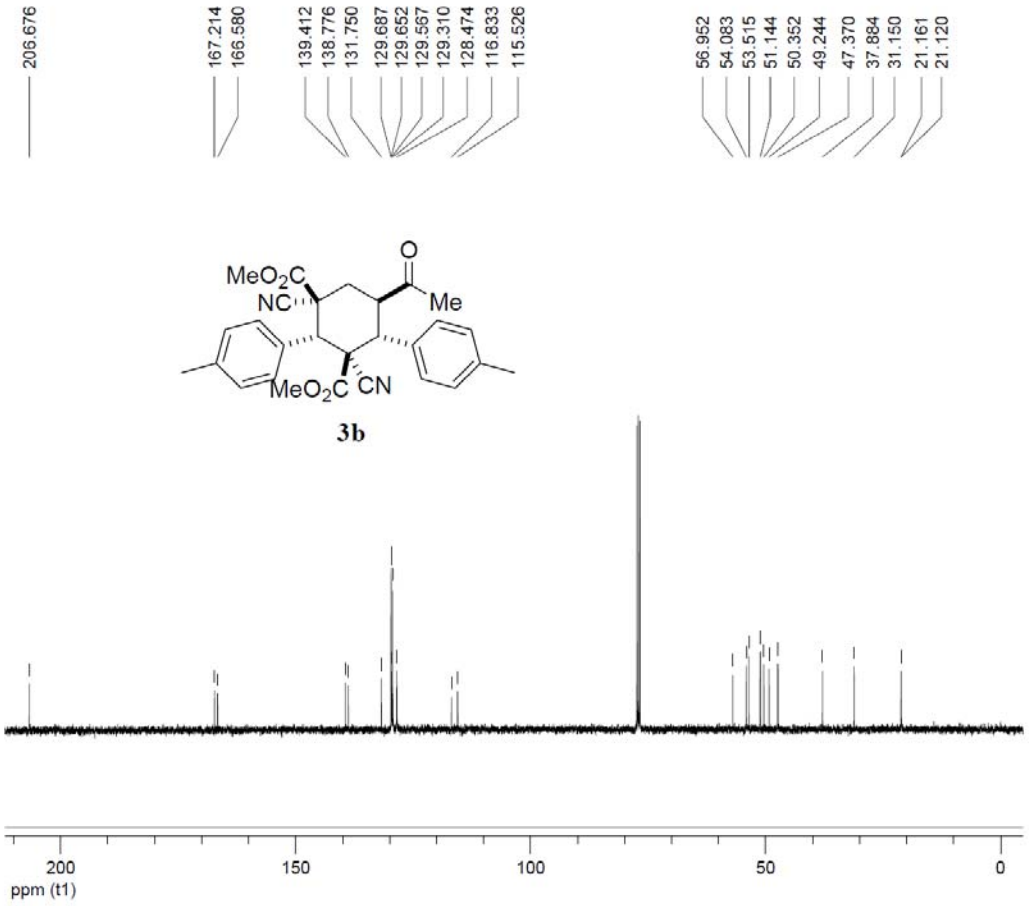
(f1) 32768
Acquisition Time (sec):
(f1) 3.9846
Spectral Width (ppm):
(f1) 20.553
Pulse Program:
Unknown



Date:
23 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
(f1) 100.613
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.3631
Spectral Width (ppm):
(f1) 238.921
Pulse Program:
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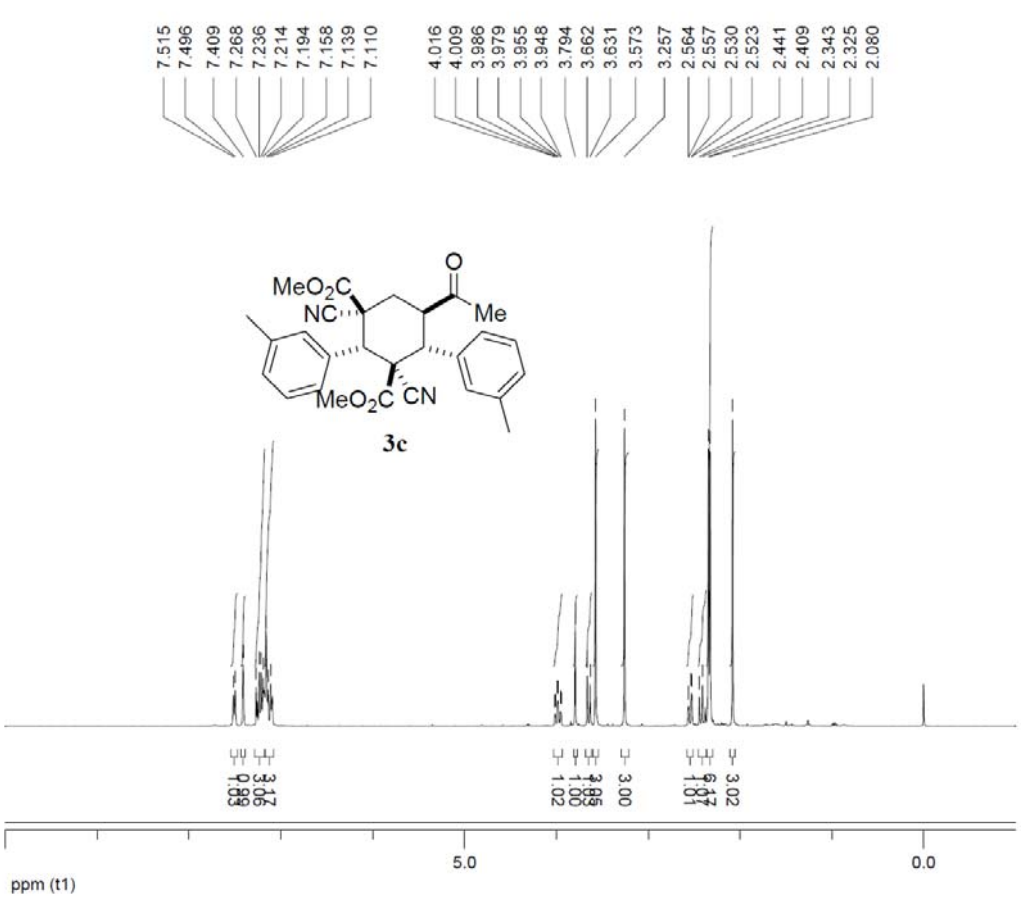

Date:
23 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
(f1) 400.130
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 3.9846
Spectral Width (ppm):
(f1) 20.553
Pulse Program:
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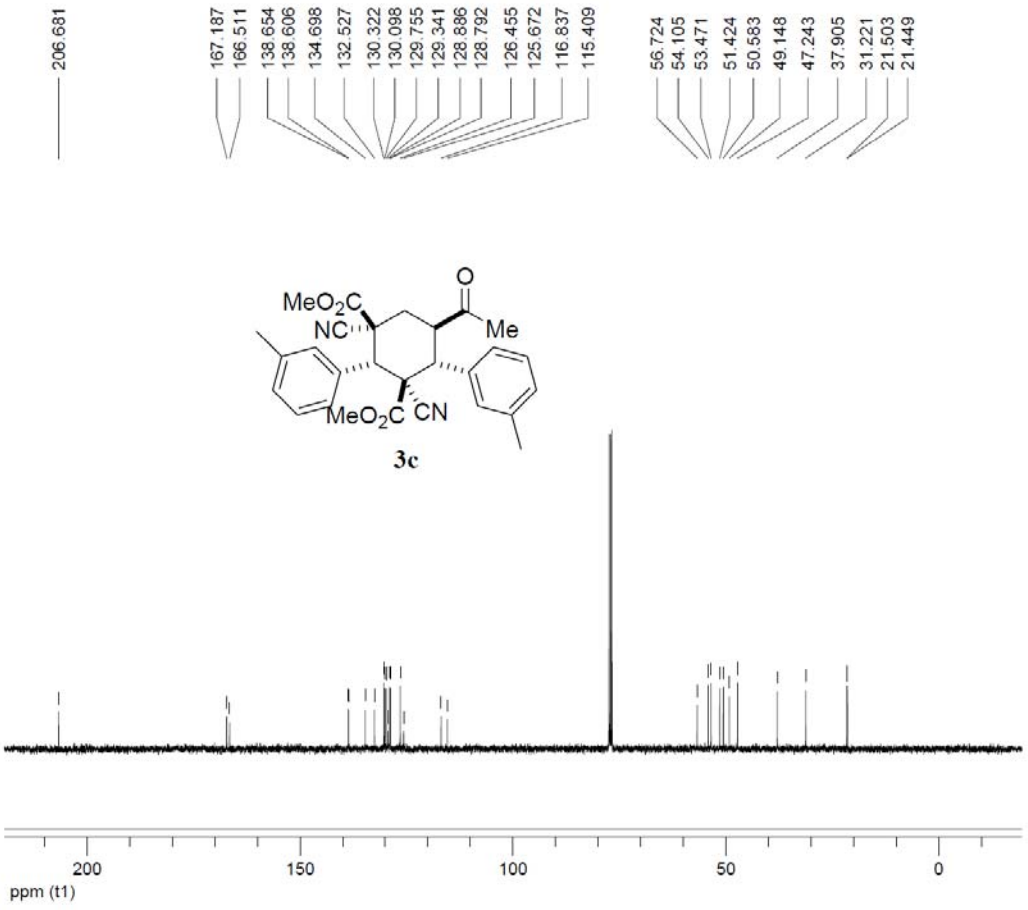

Date:
23 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
(f1) 100.613
Original Points Count:
(f1) 32768
Actual Points Count:
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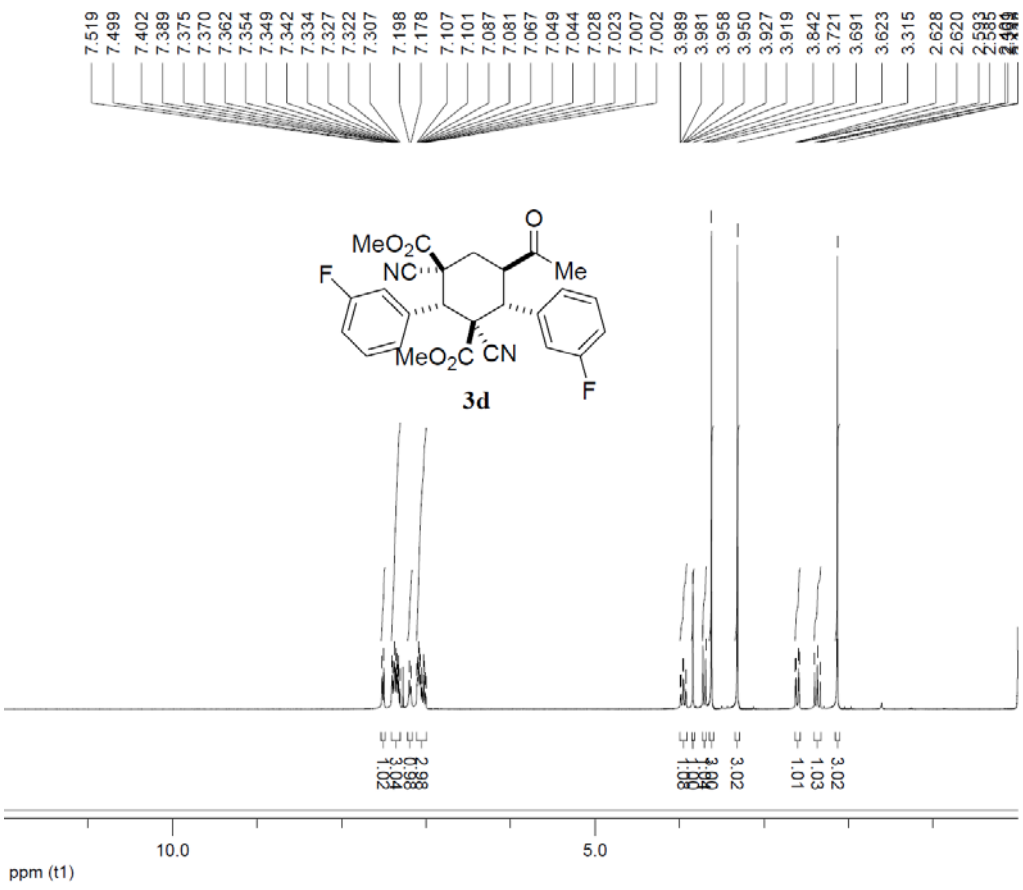
Acquisition Time (sec):
(f1) 1.3631
Spectral Width (ppm):
(f1) 238.921
Pulse Program:
Unknown



Date:
23 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
(f1) 400.130
Original Points Count:
(f1) 32768
Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
(f1) 20.553
Pulse Program:
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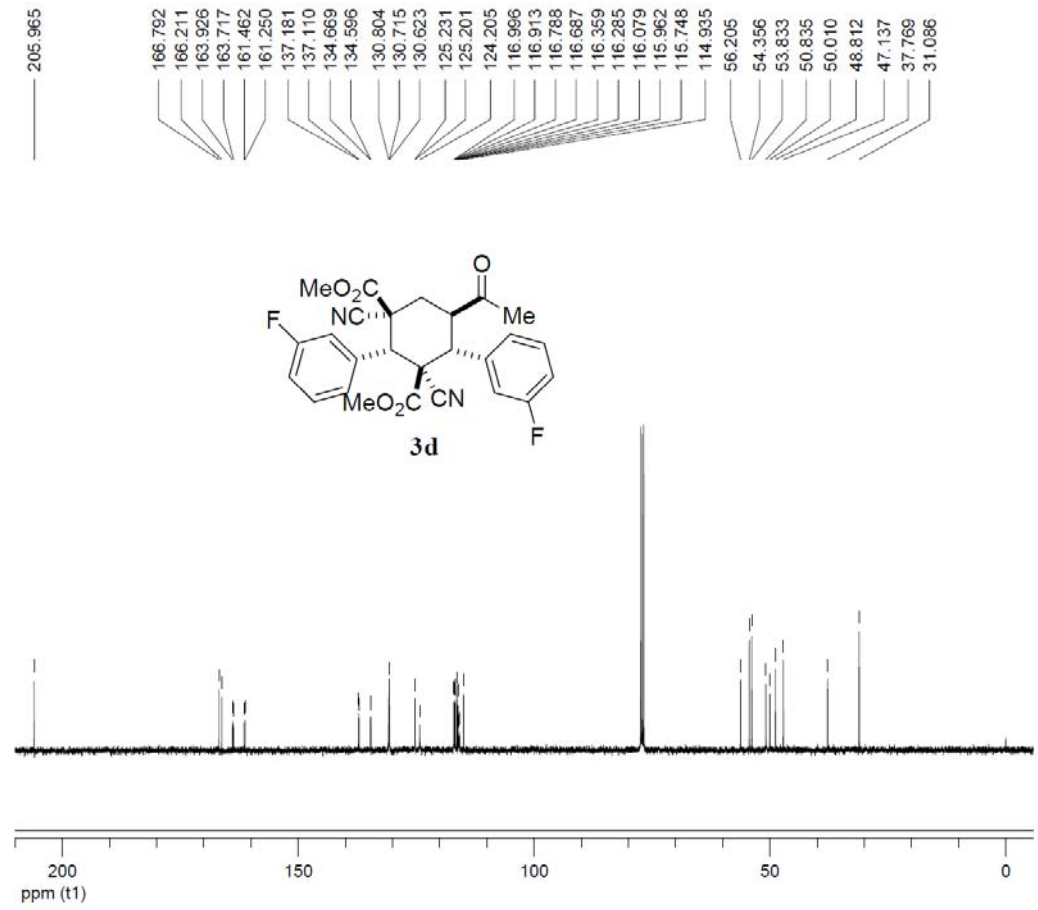

Date:
23 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
(f1) 100.613
Original Points Count:
(f1) 32768
Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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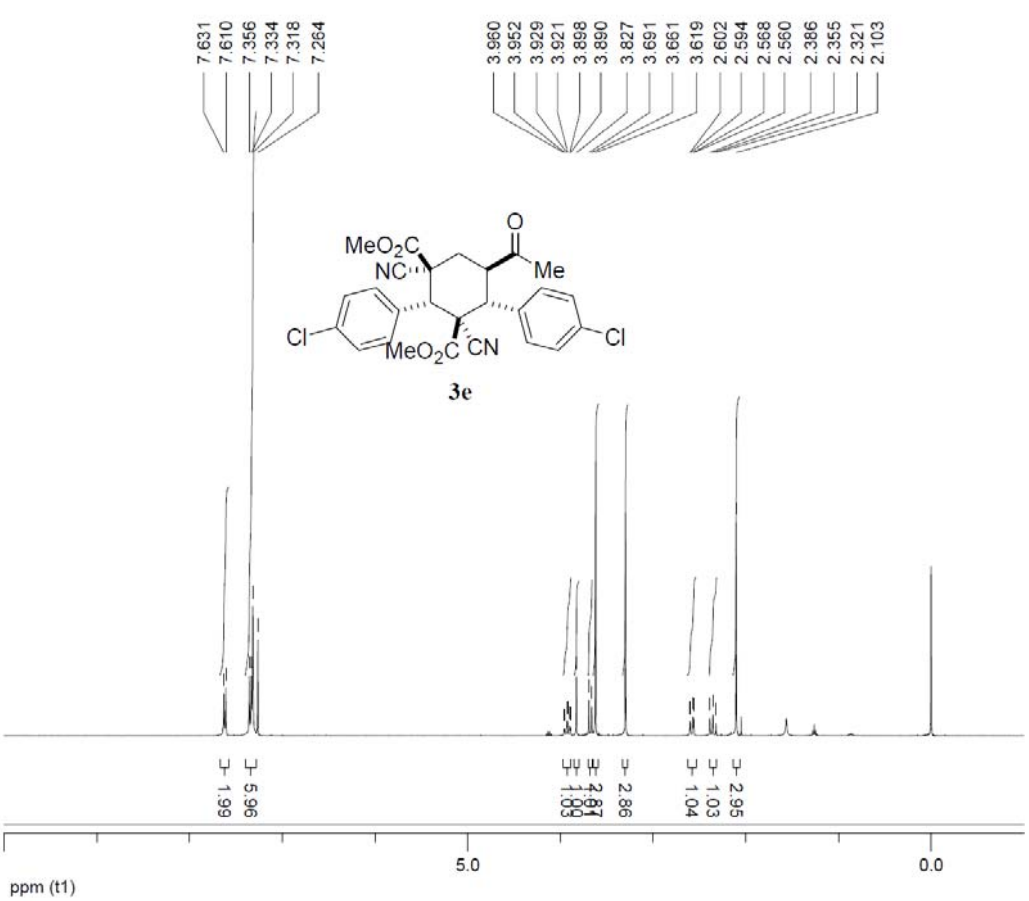

Date:
25 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
(f1) 400.130
Original Points Count:
(f1) 32768
Actual Points Count:
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Acquisition Time (sec):
(f1) 3.9846
Spectral Width (ppm):
(f1) 20.553
Pulse Program:
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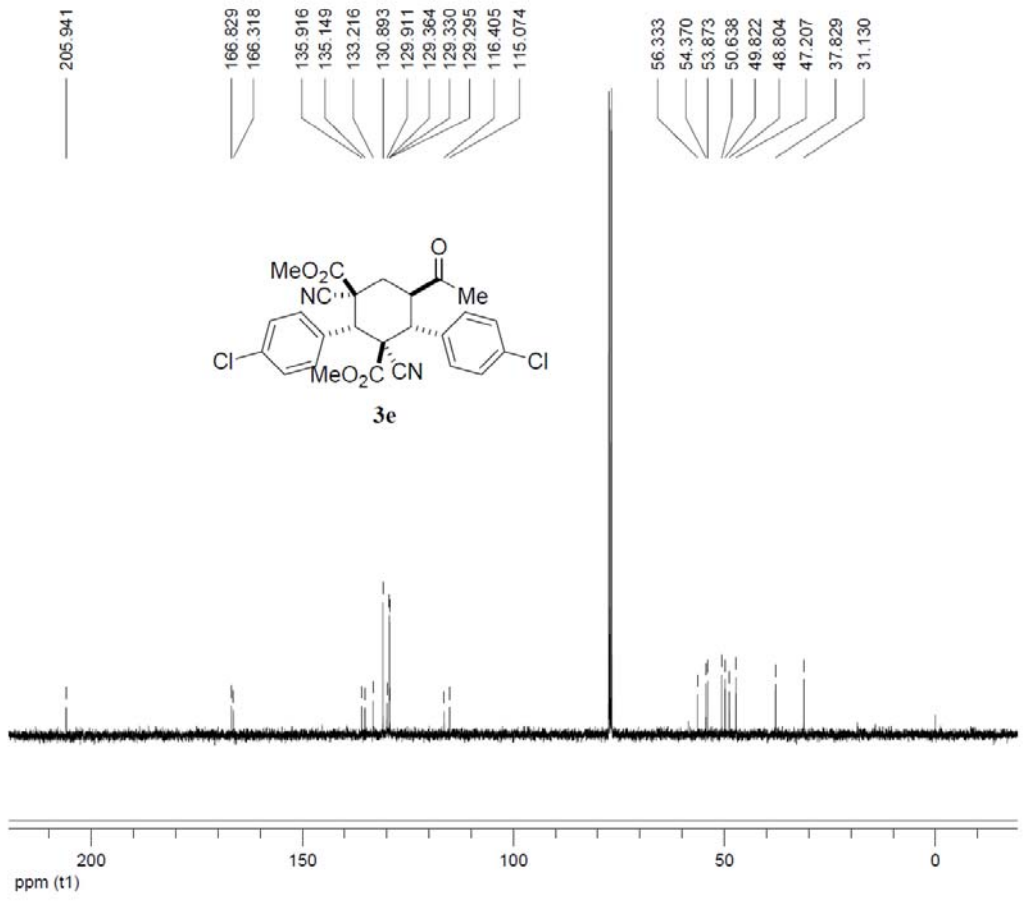

Date:
25 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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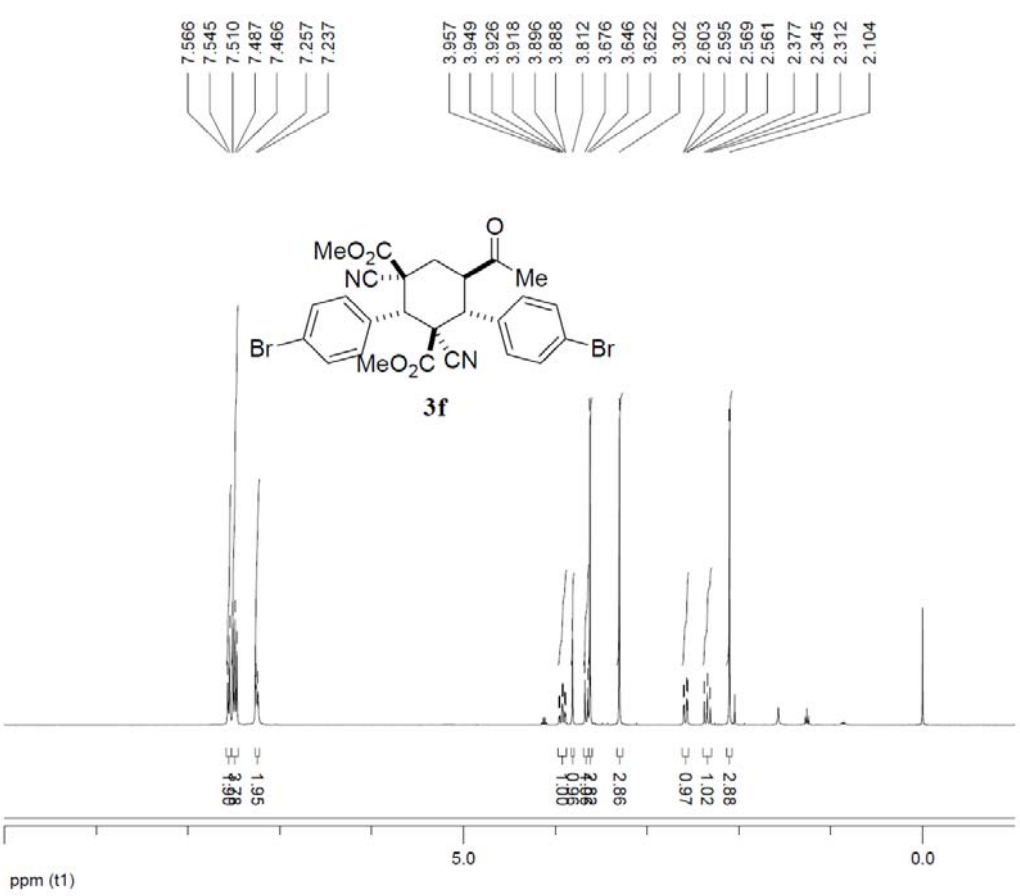

Date:
23 Jul 2010
Document's Title:
h.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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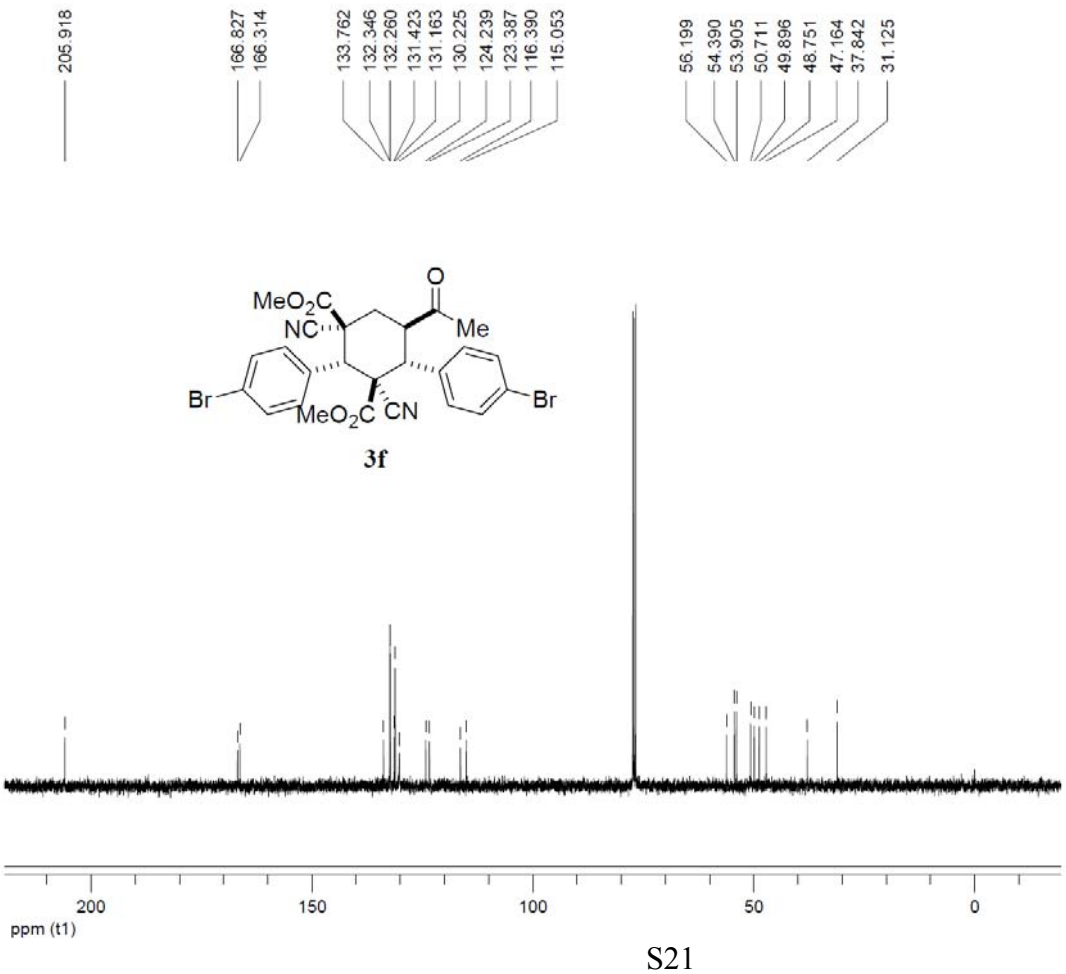

Date:
23 Jul 2010
Document's Title:
c.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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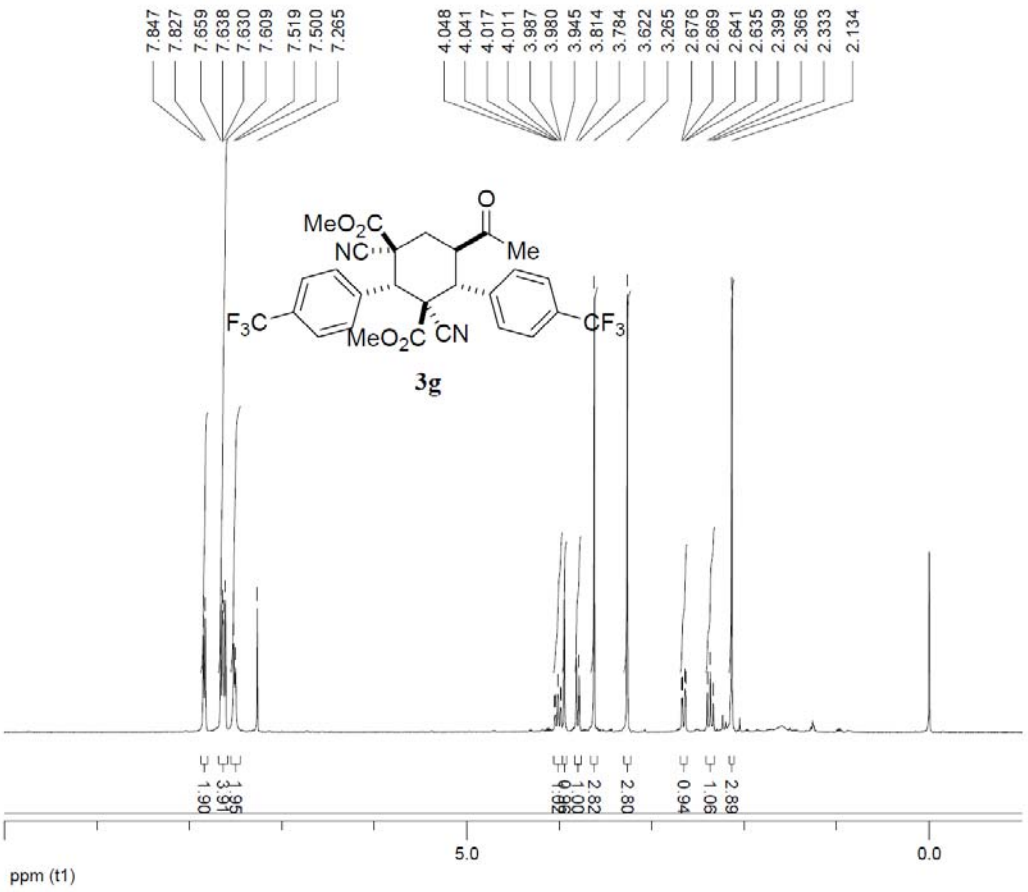

Date:
23 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
(f1) 20.553
Pulse Program:
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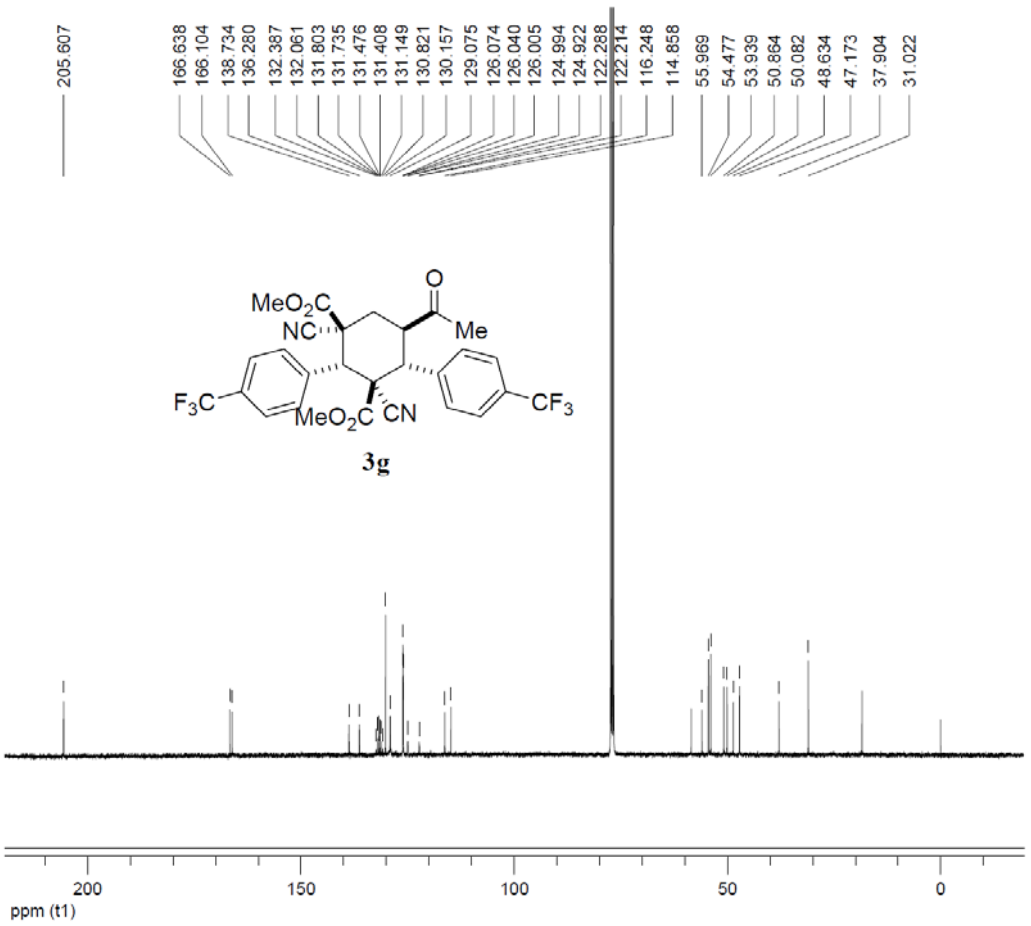

Date:
23 Jul 2010
Document's Title:
c.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
(f1) 100.613
Original Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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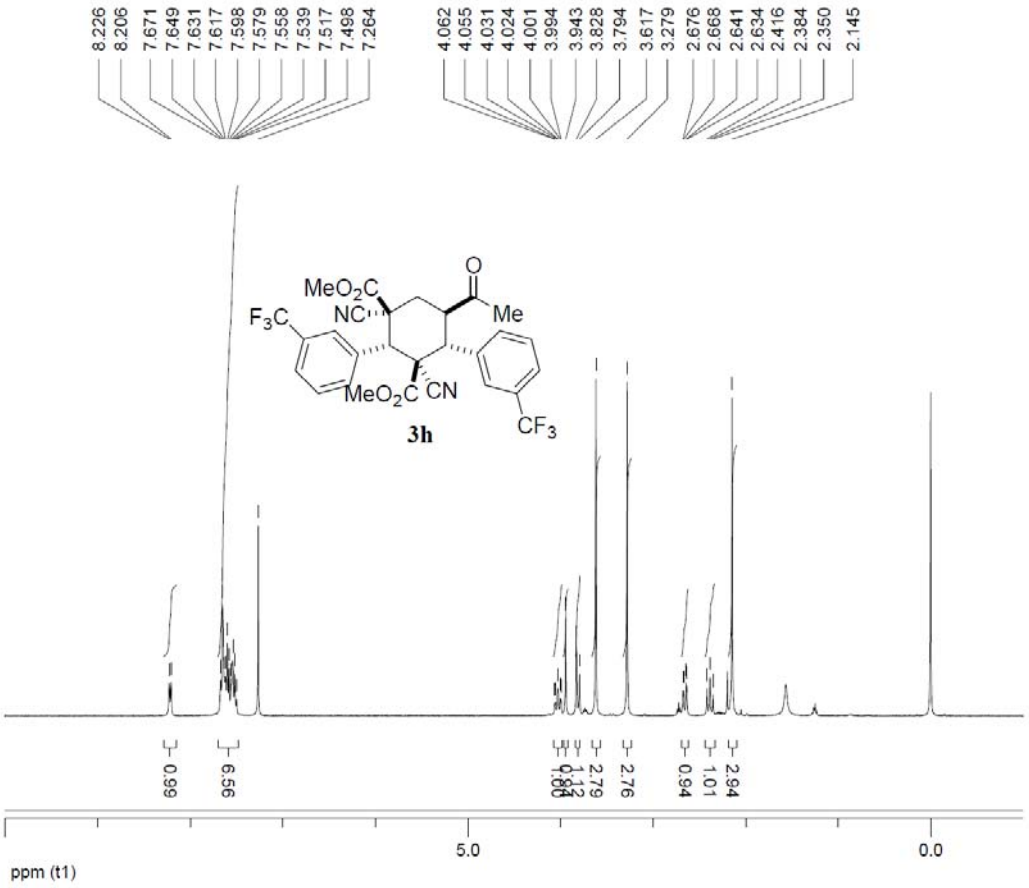

Date:
25 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
(f1) 32768
Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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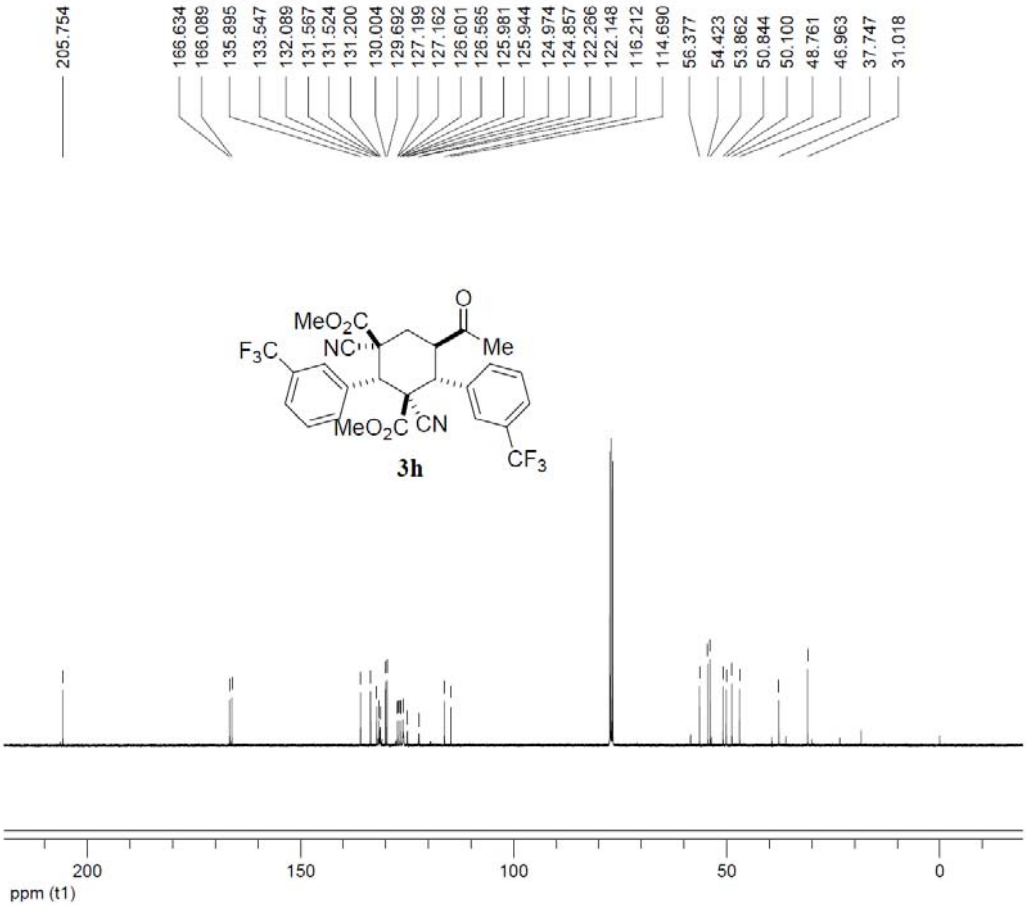

Date:
25 Jul 2010
Document's Title:
c.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
(f1) 100.613
Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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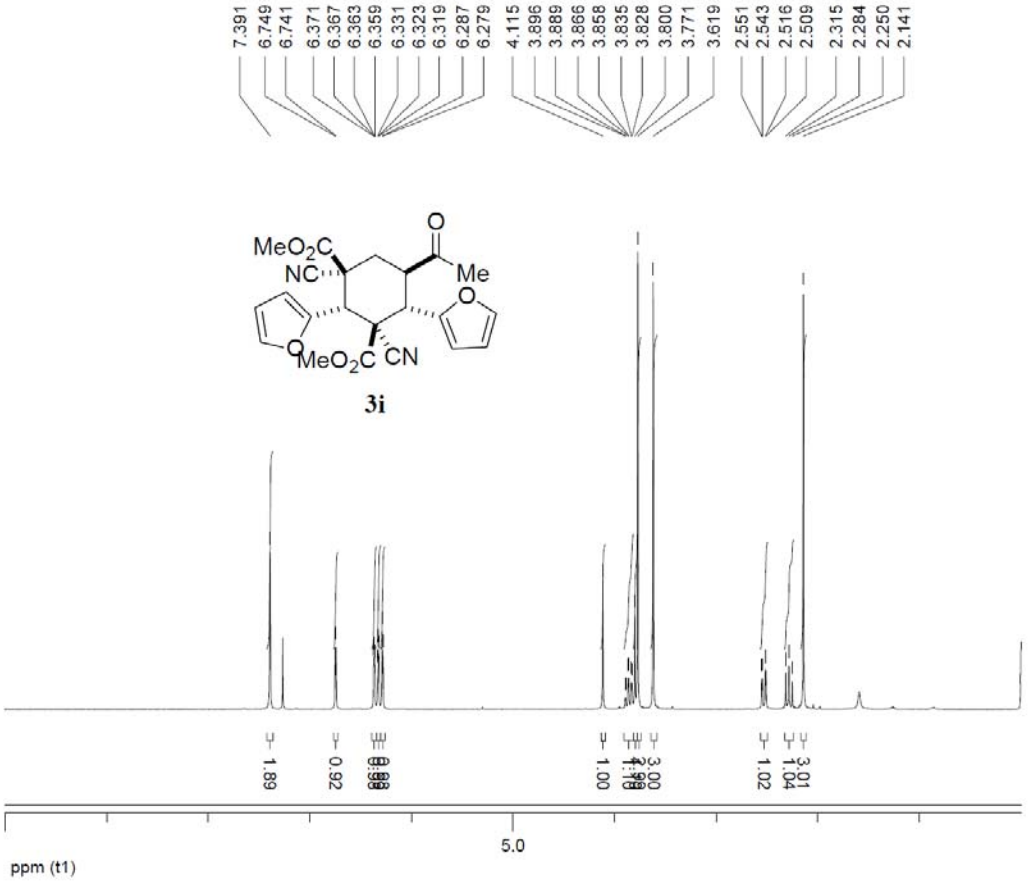

Date:
23 Jul 2010
Document's Title:
1r.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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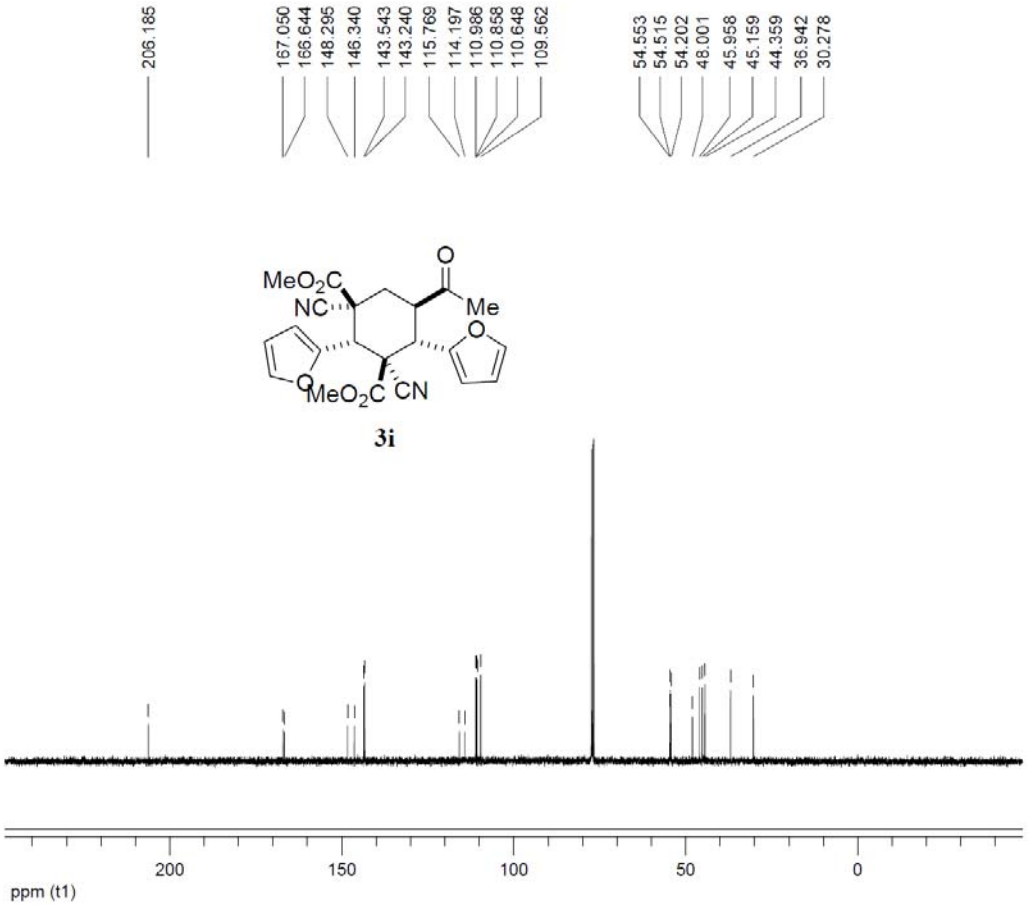

Date:
23 Jul 2010
Document's Title:
1r.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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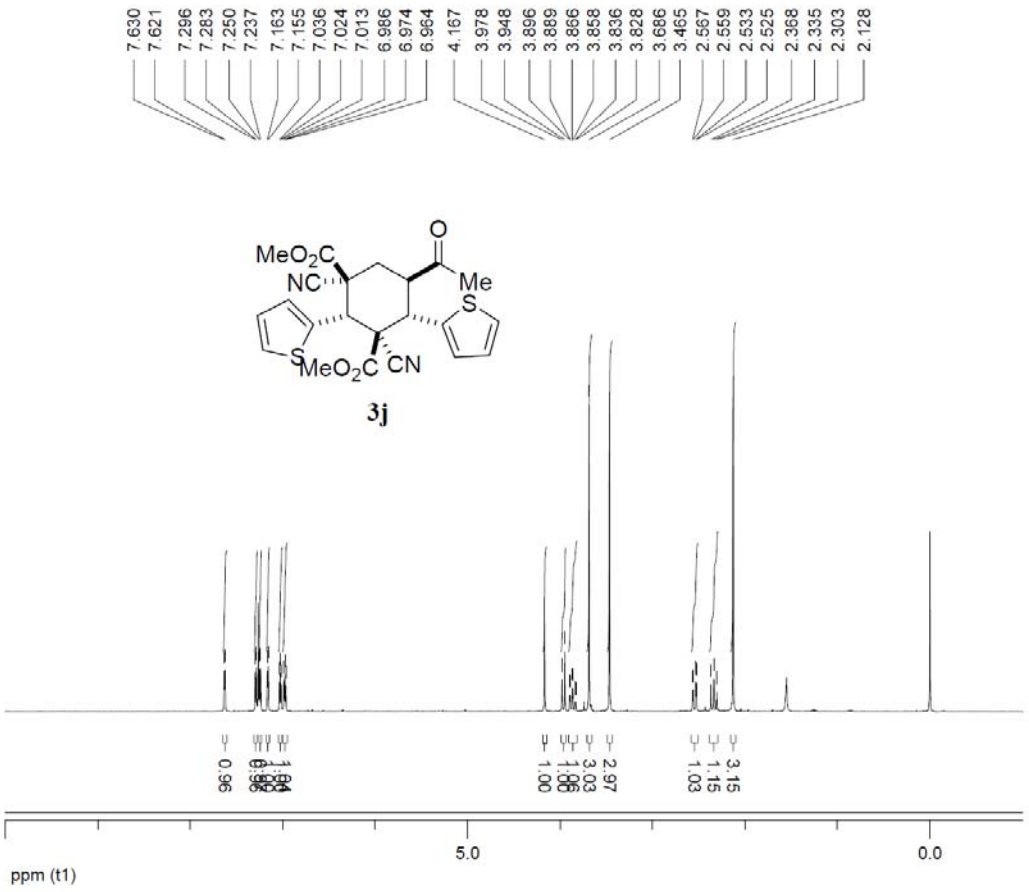

Date:
23 Jul 2010
Document's Title:
1r.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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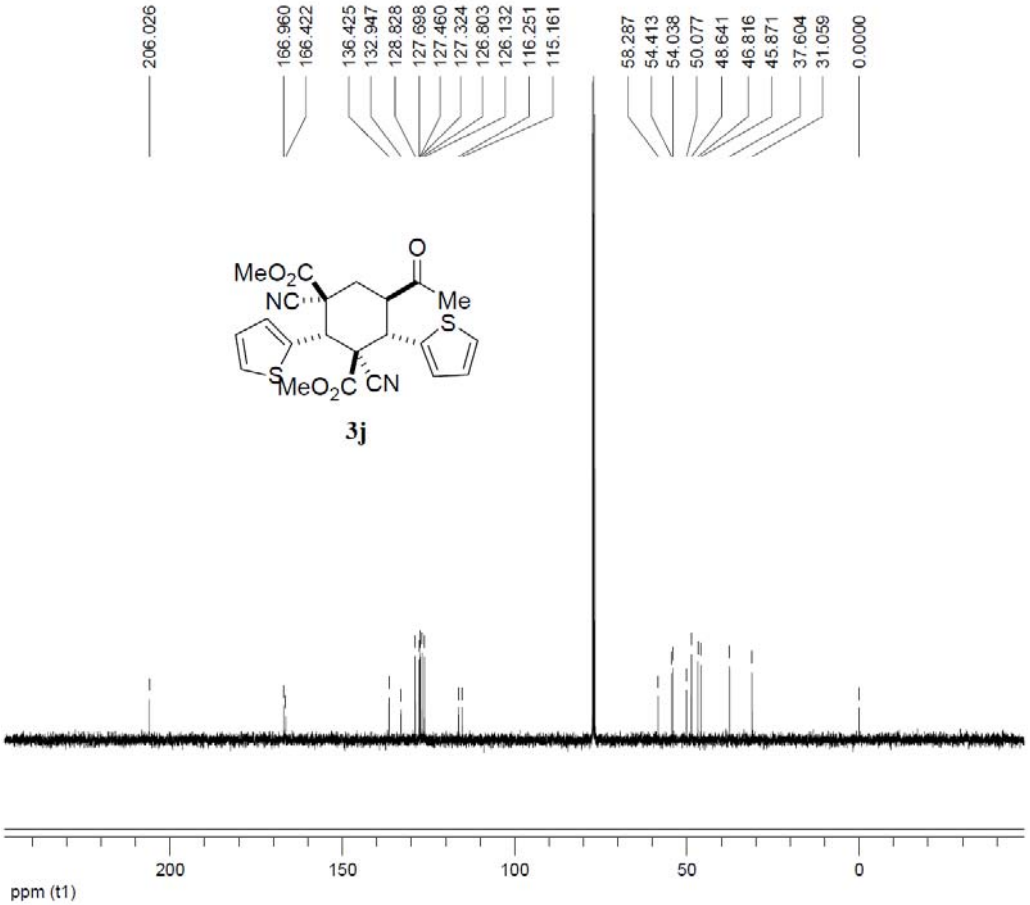

Date:
23 Jul 2010
Document's Title:
1r.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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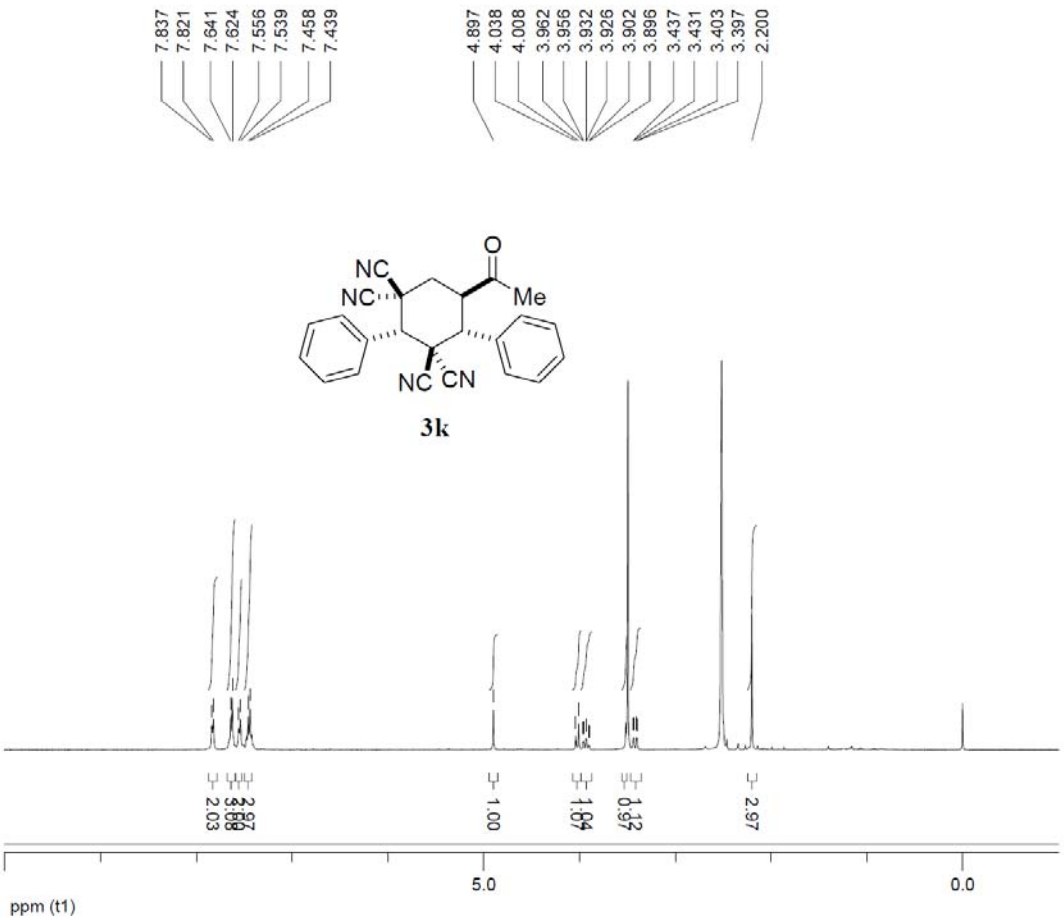

Date:
23 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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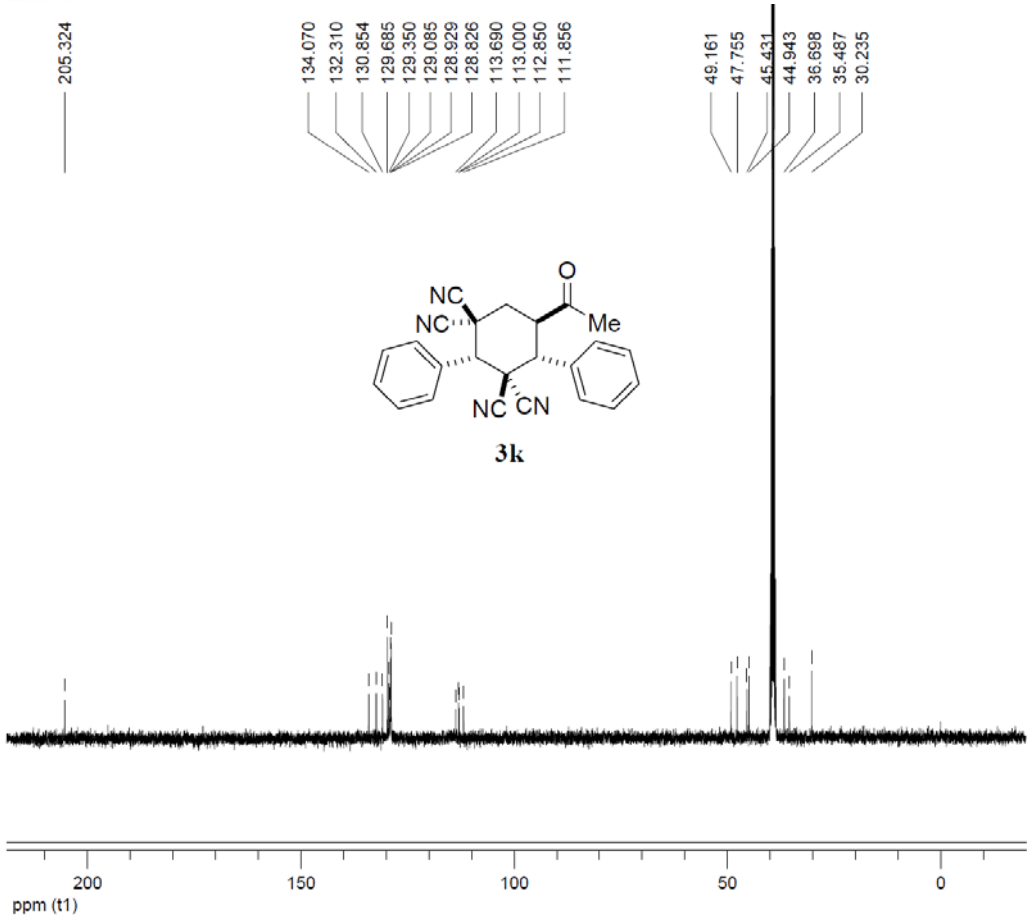

Date:
23 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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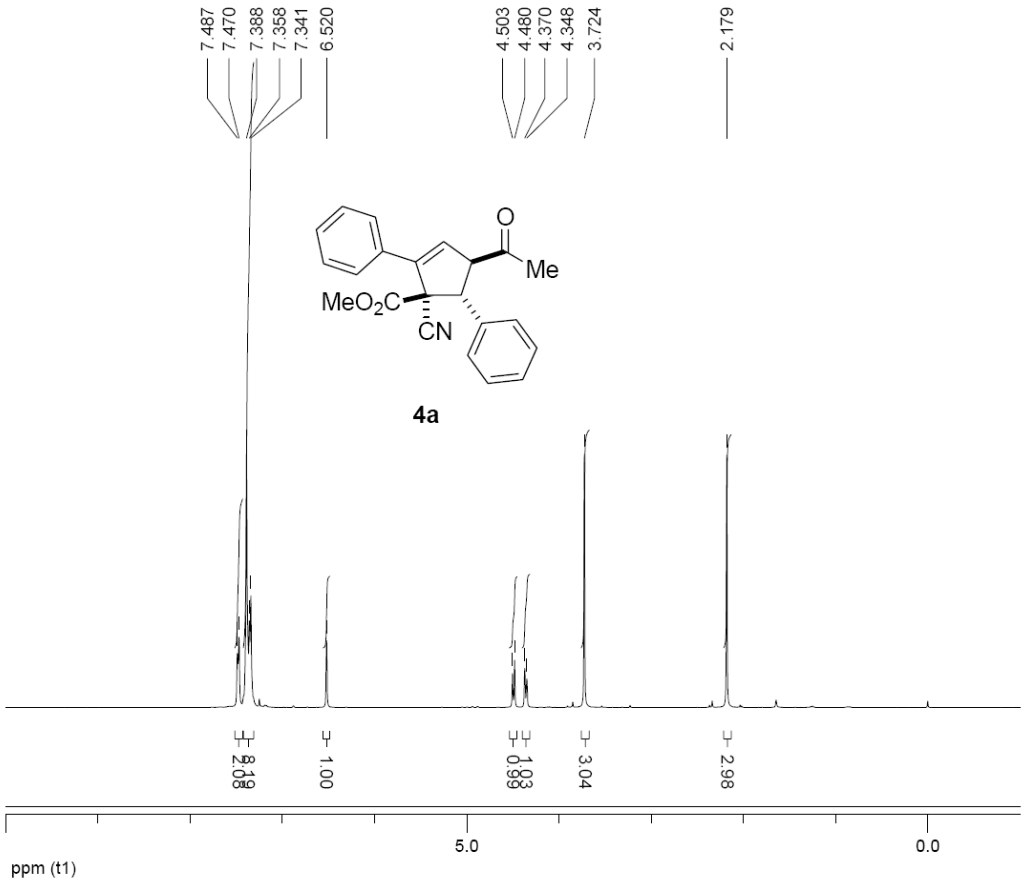

Date:
20 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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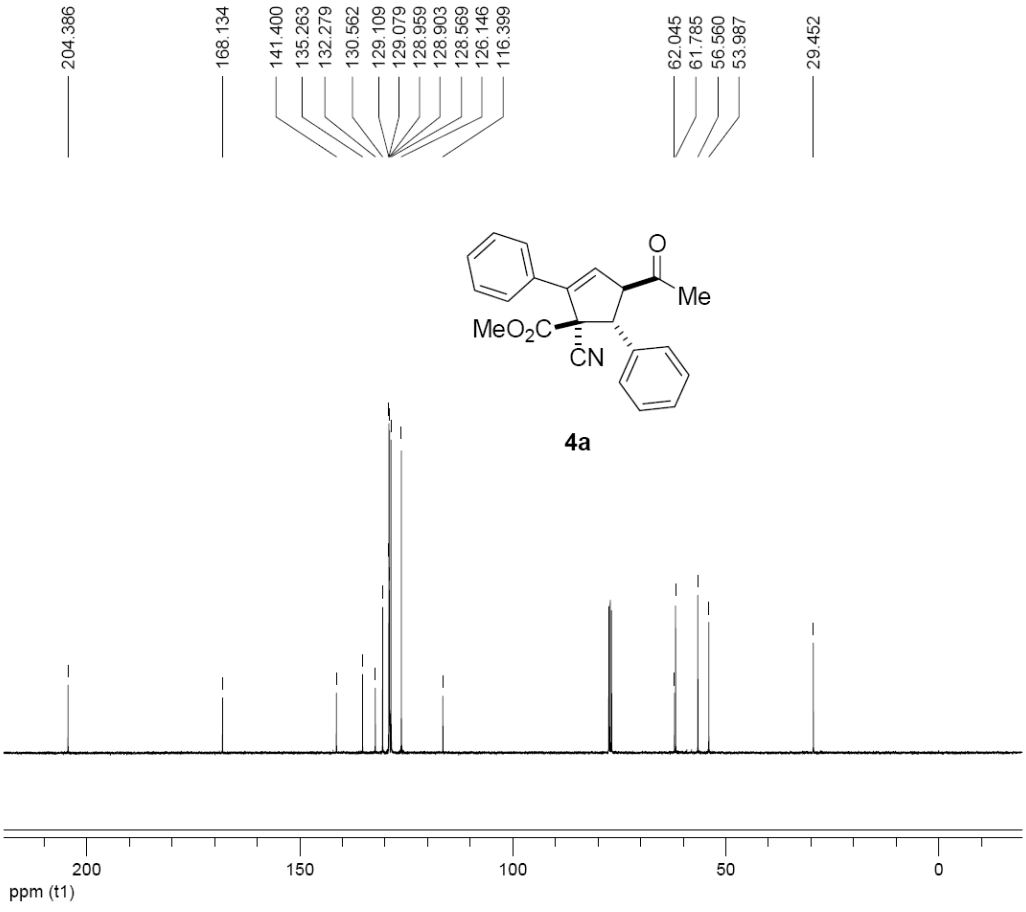

Date:
20 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Spectral Width (ppm):
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Pulse Program:
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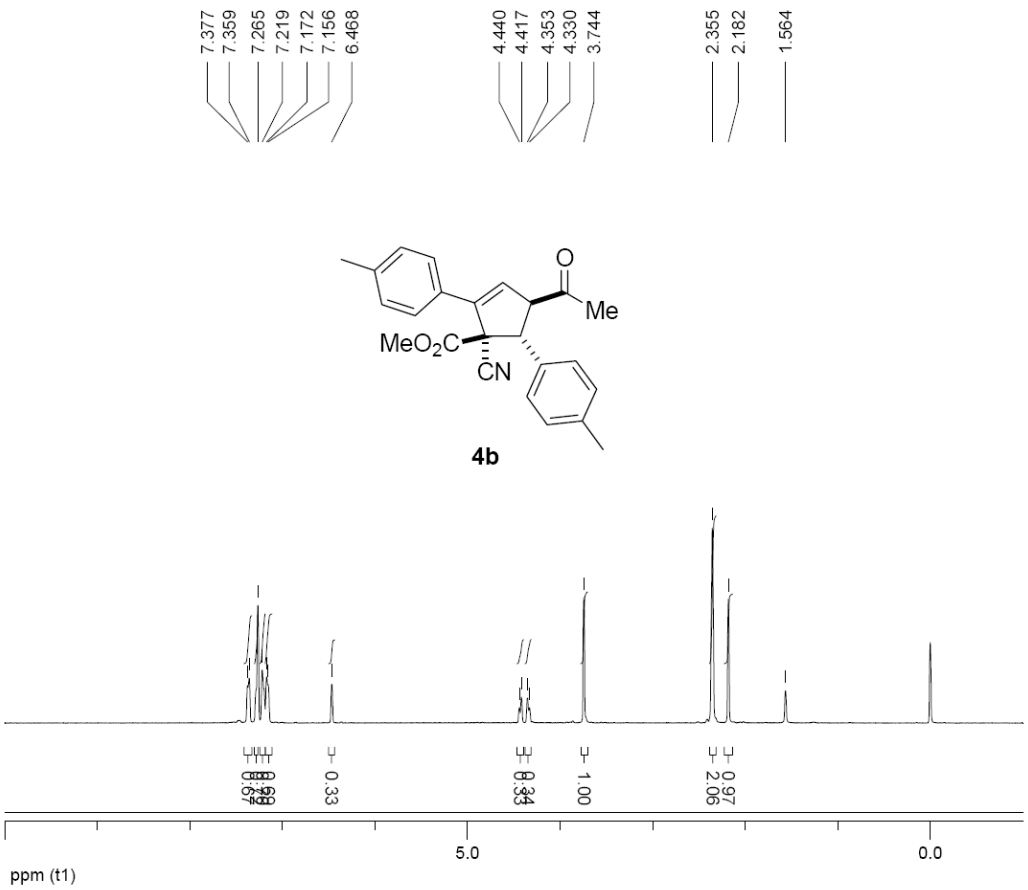

Date:
21 Jul 2010
Document's Title:
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Spectrum Title:
PROTON

Frequency (MHz):
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Spectral Width (ppm):
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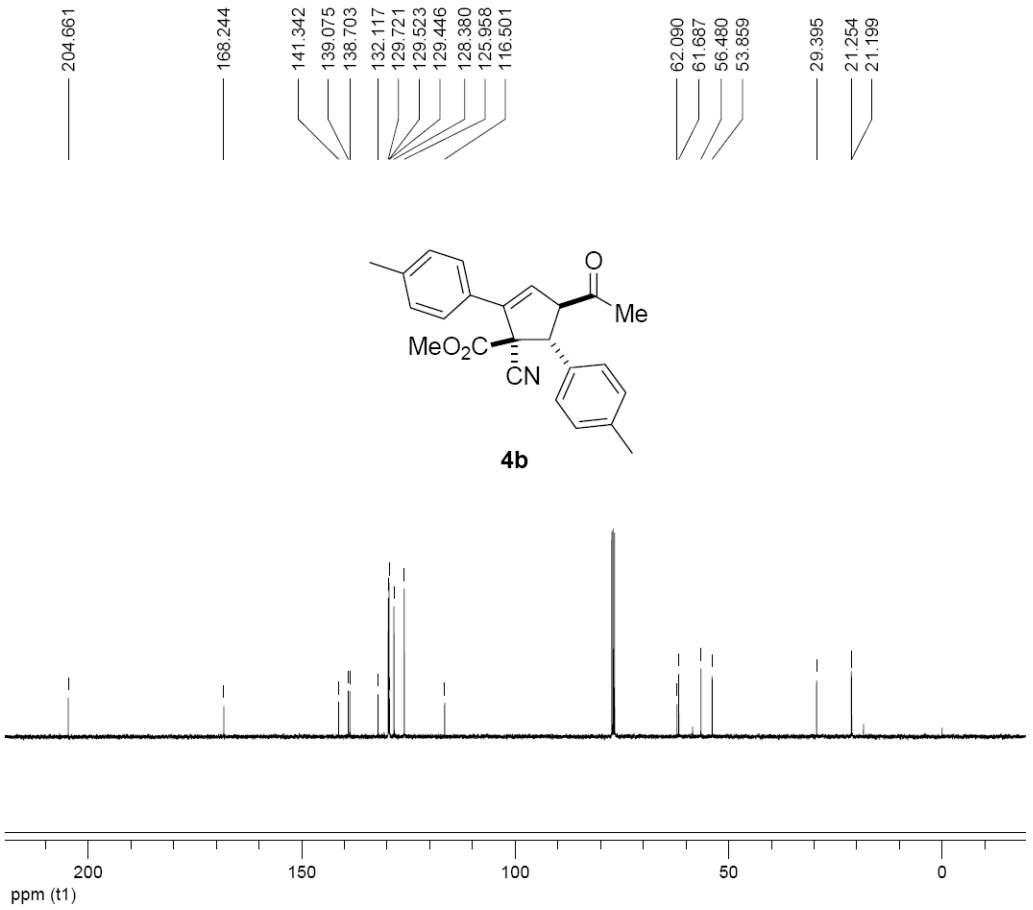

Date:
20 Jul 2010
Document's Title:
c.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Actual Points Count:
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Spectral Width (ppm):
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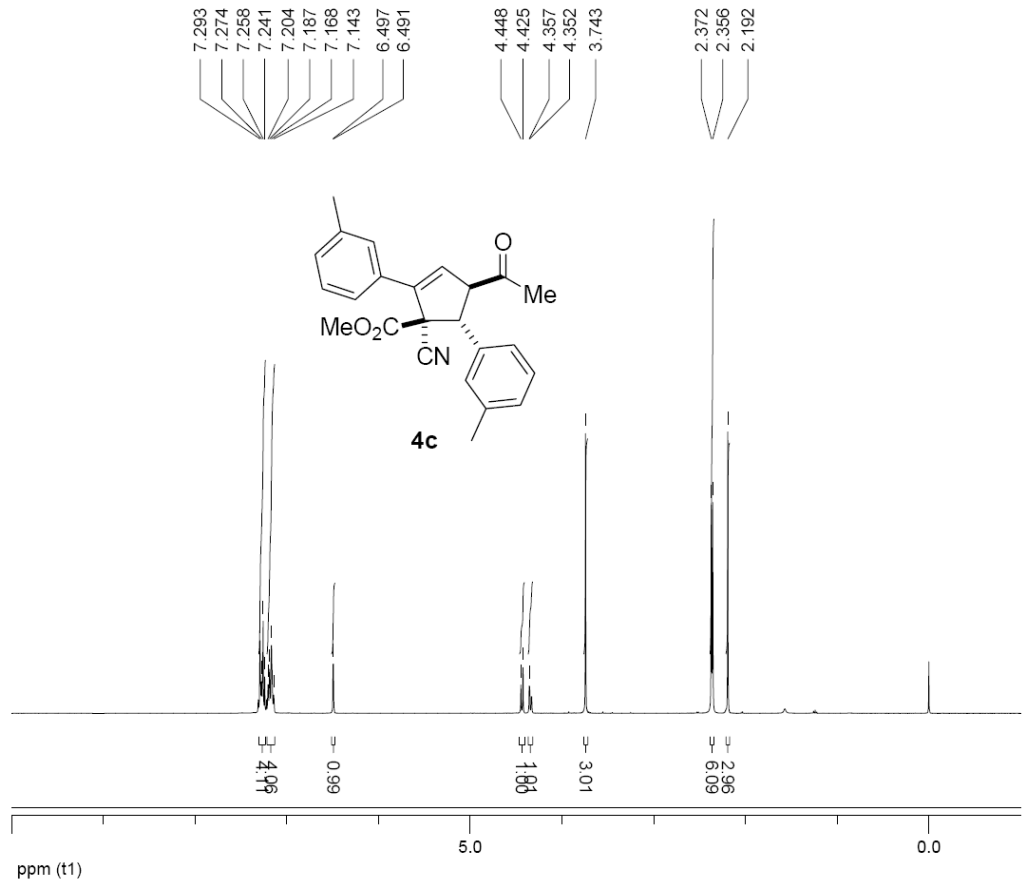

Date:
20 Jul 2010
Document's Title:
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Spectrum Title:
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Frequency (MHz):
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Spectral Width (ppm):
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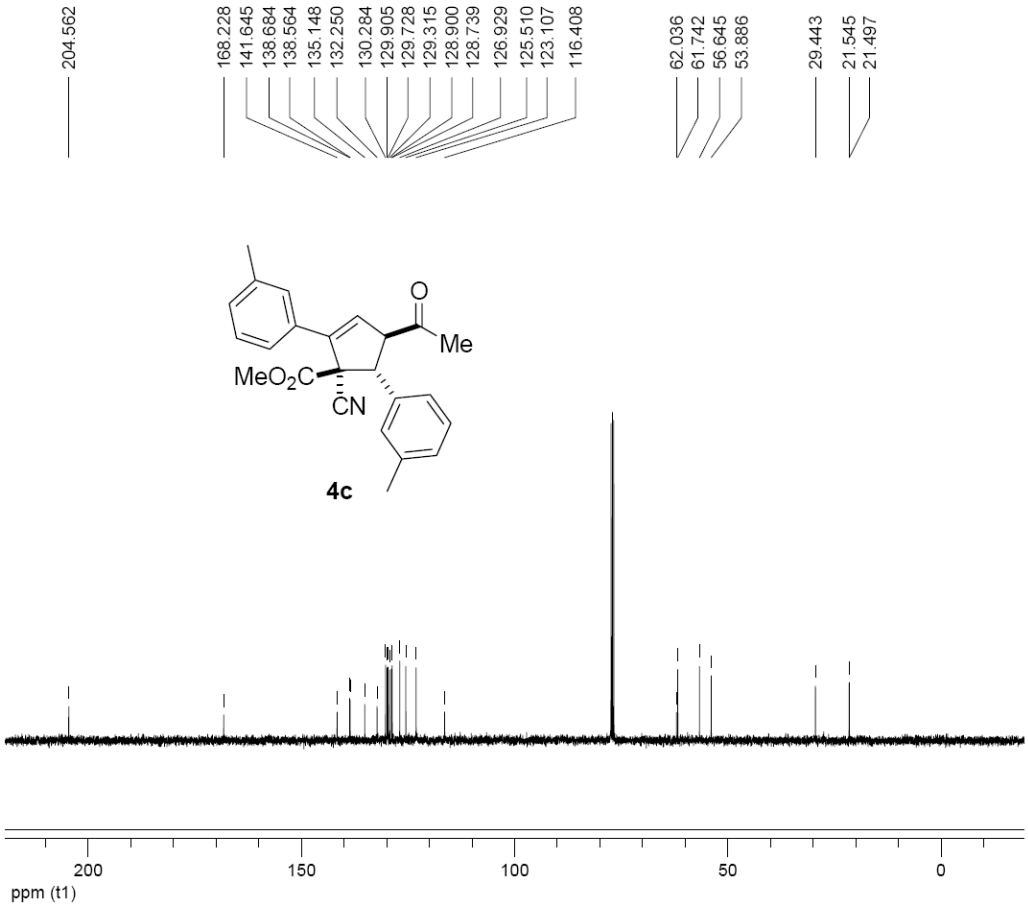

Date:
20 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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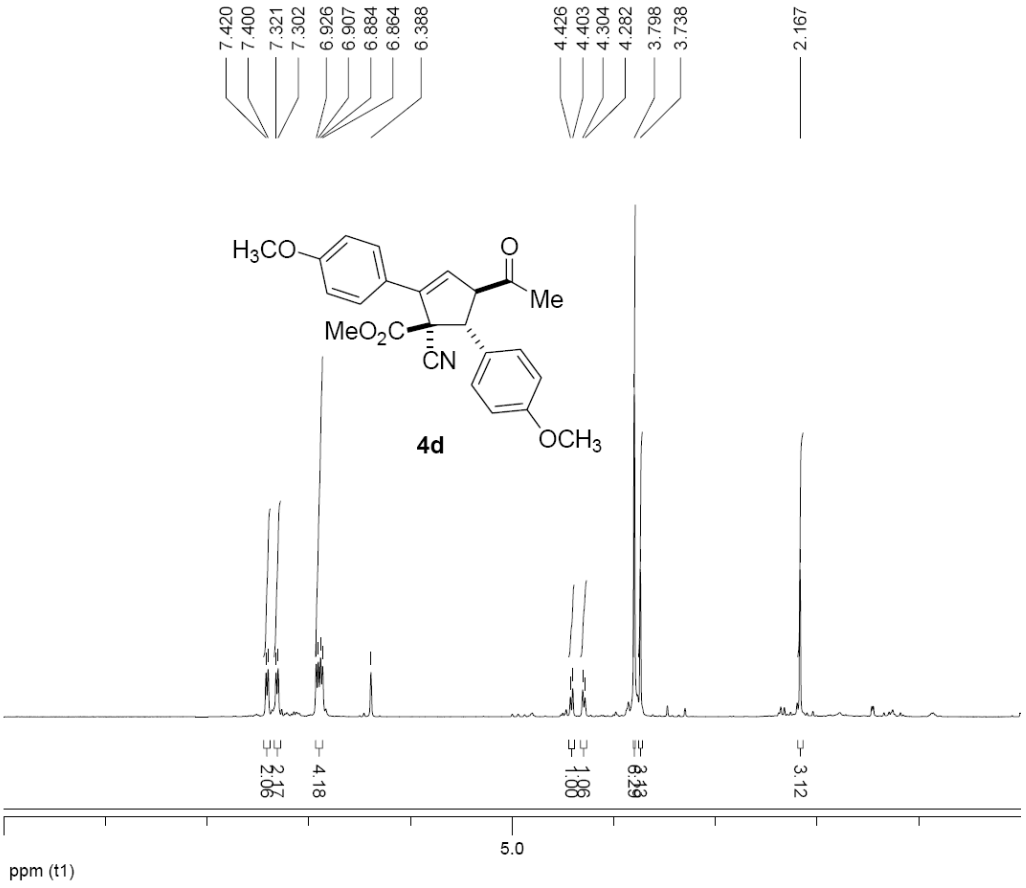

Date:
20 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
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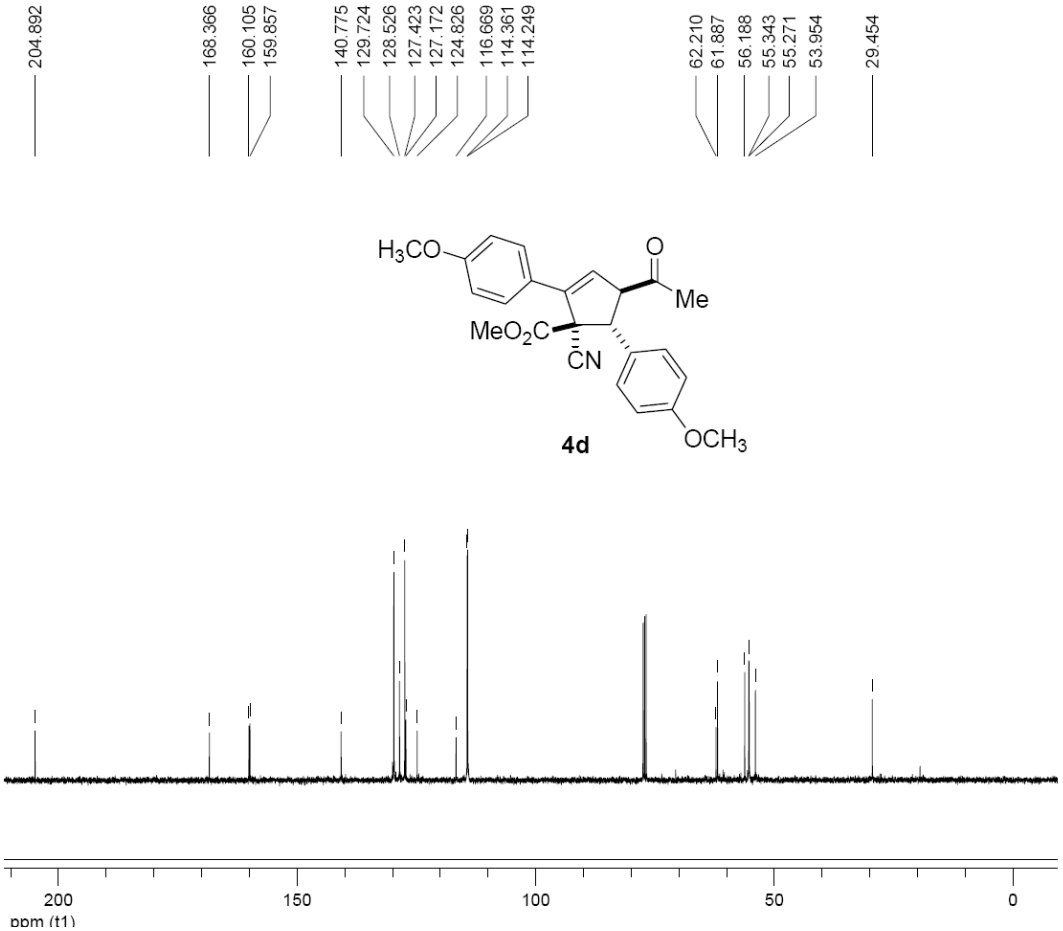

Date:
20 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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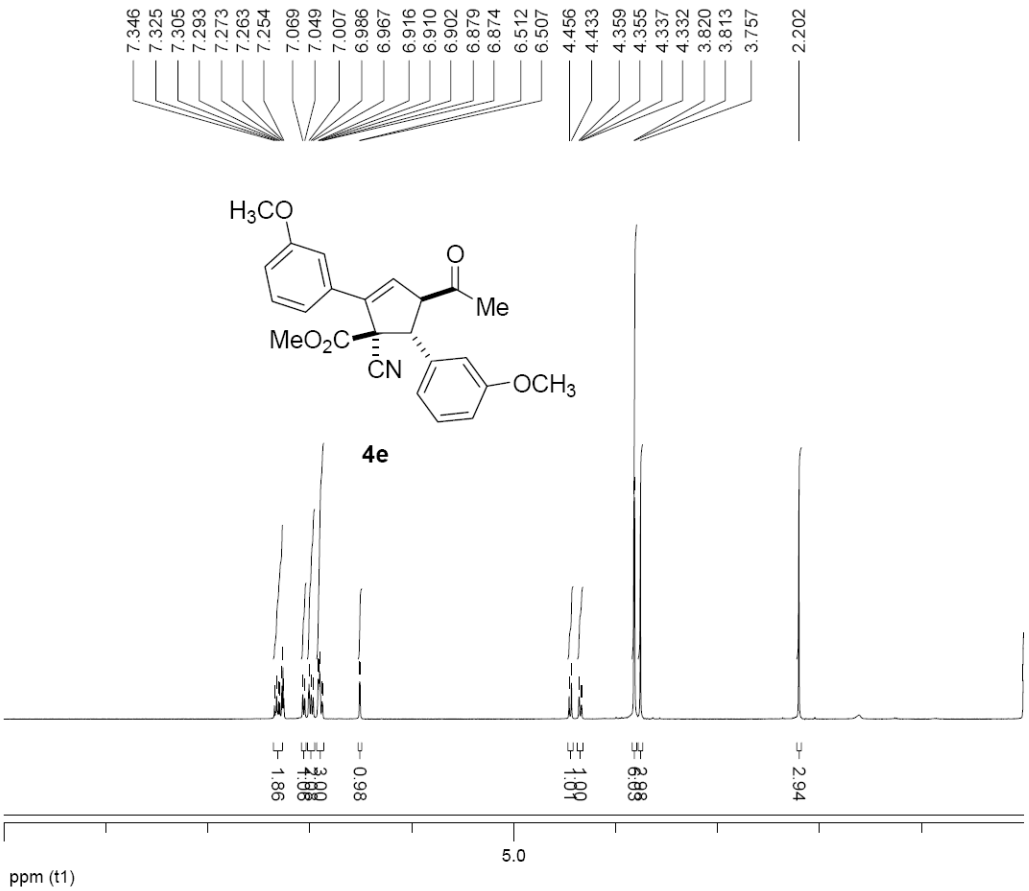

Date:
20 Jul 2010
Document's Title:
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Spectrum Title:
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Frequency (MHz):
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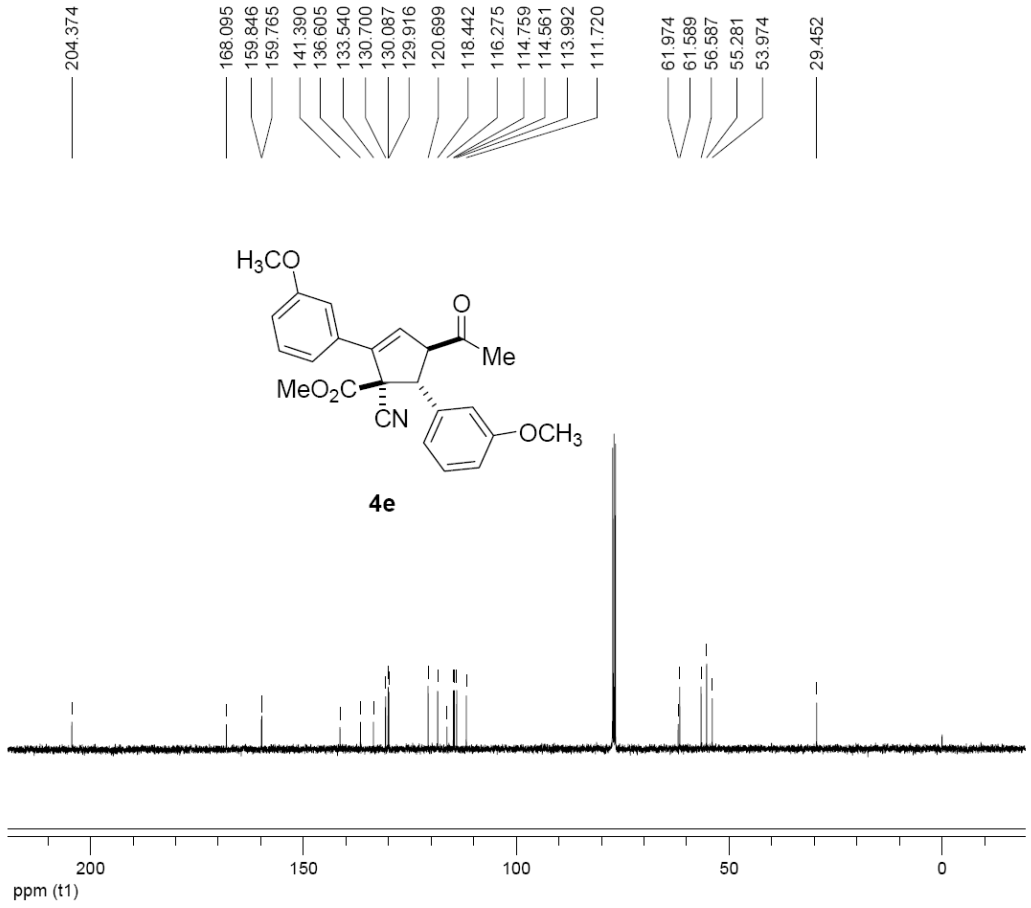

Date:
20 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

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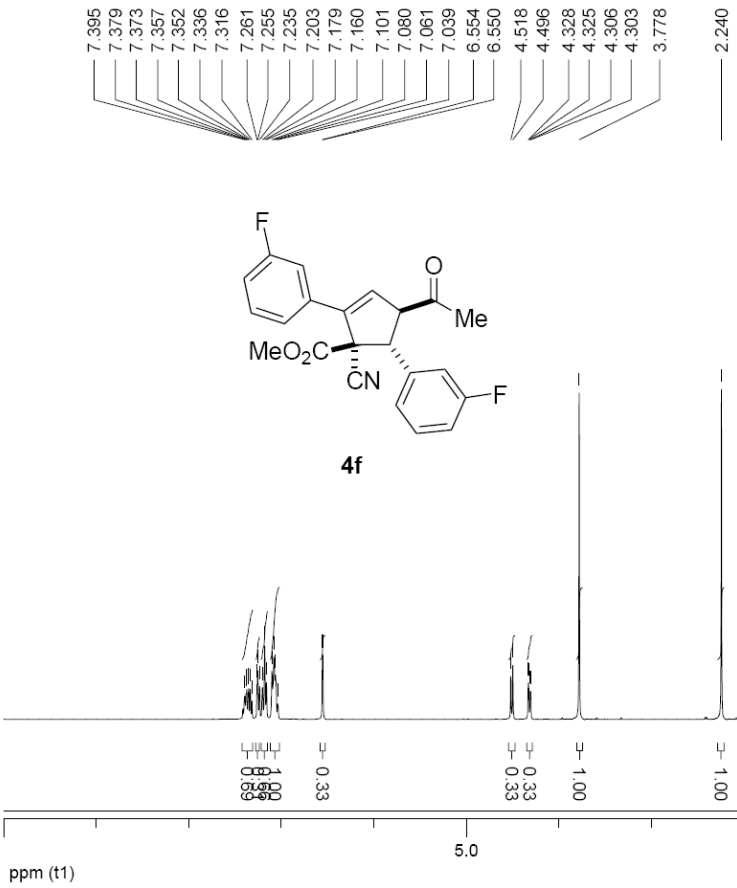

Date:
20 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Spectral Width (ppm):
(f1) 20.553
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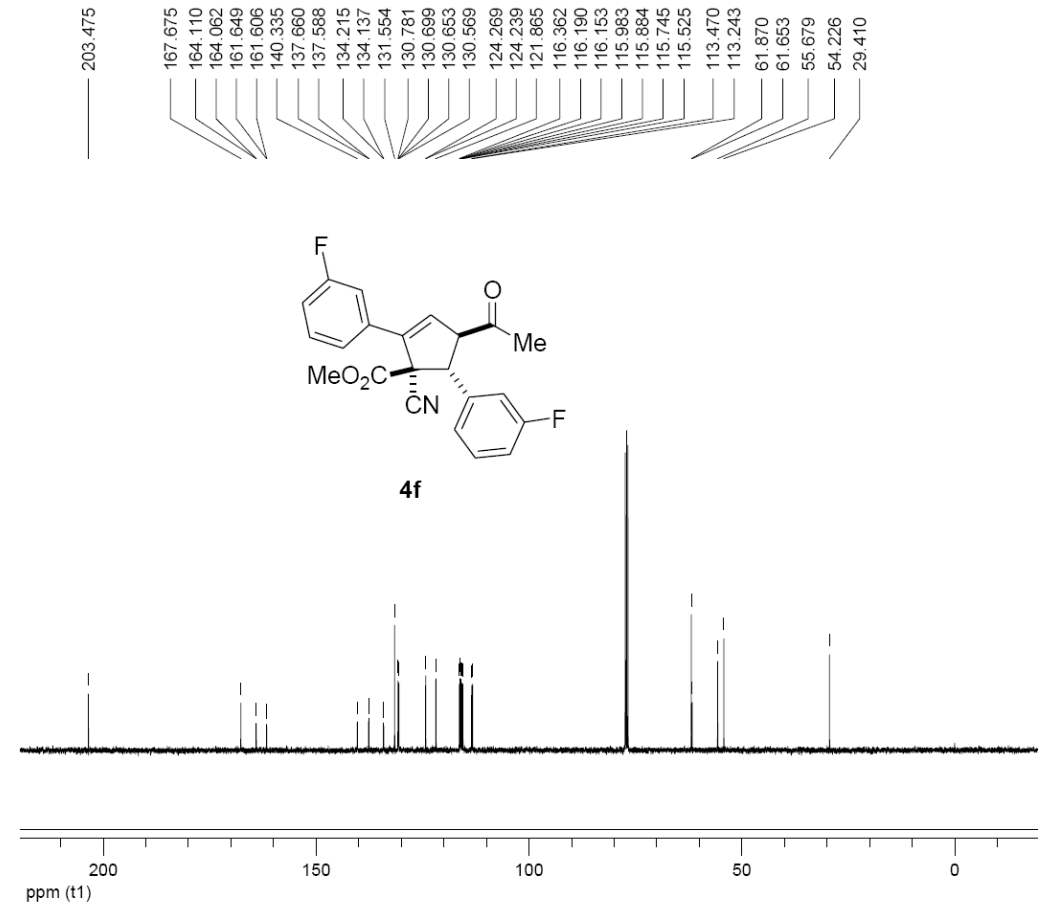

Date:
20 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Spectral Width (ppm):
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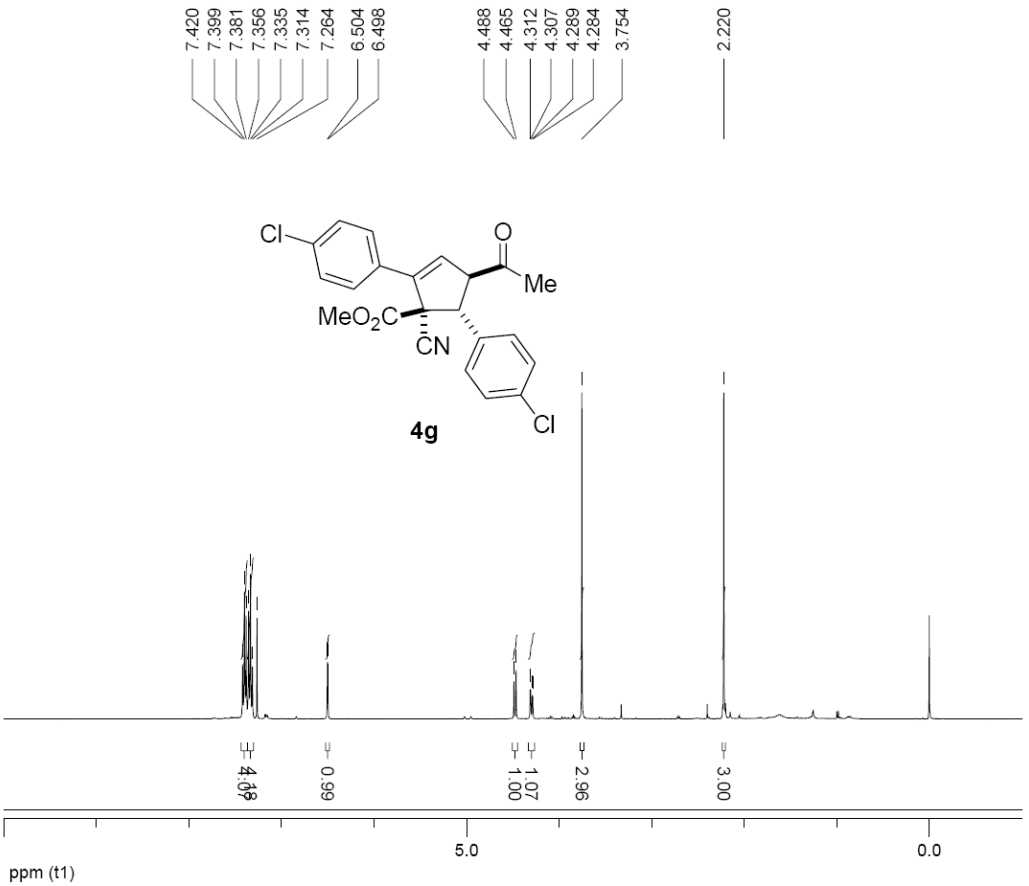

Date:
21 Jul 2010
Document's Title:
h.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Actual Points Count:
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Acquisition Time (sec):
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Spectral Width (ppm):
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Pulse Program:
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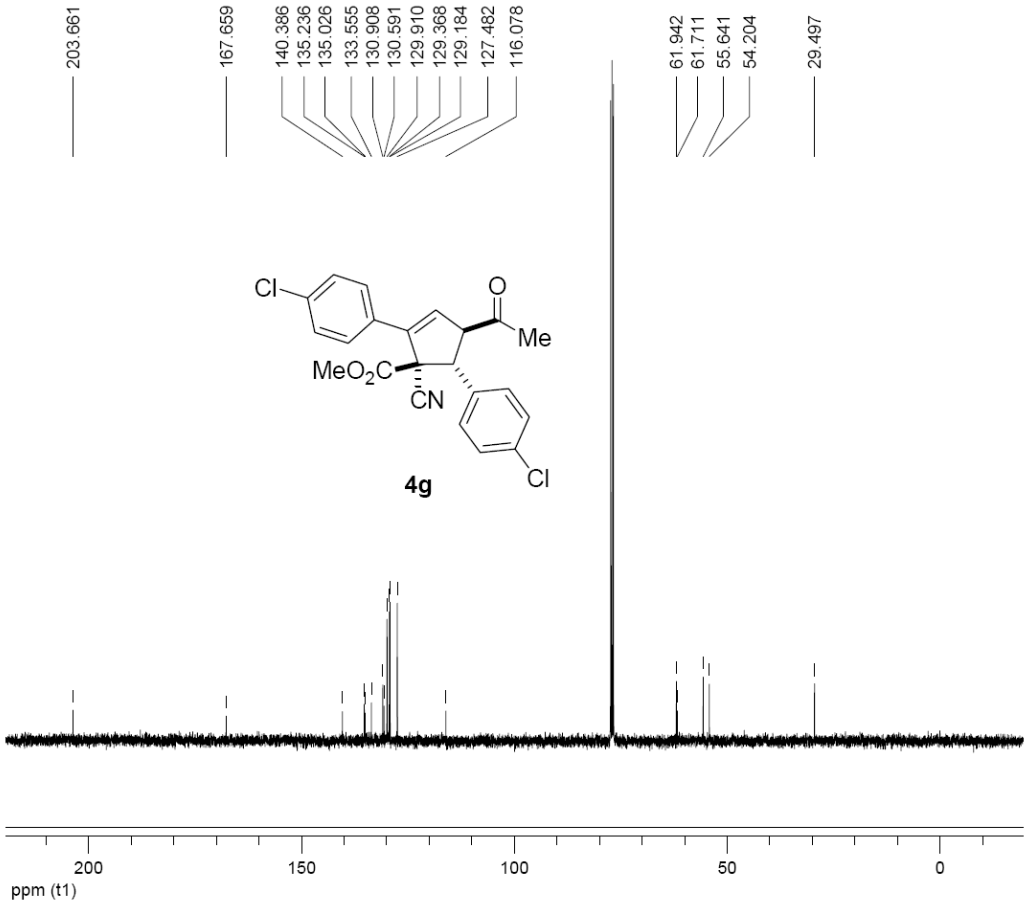
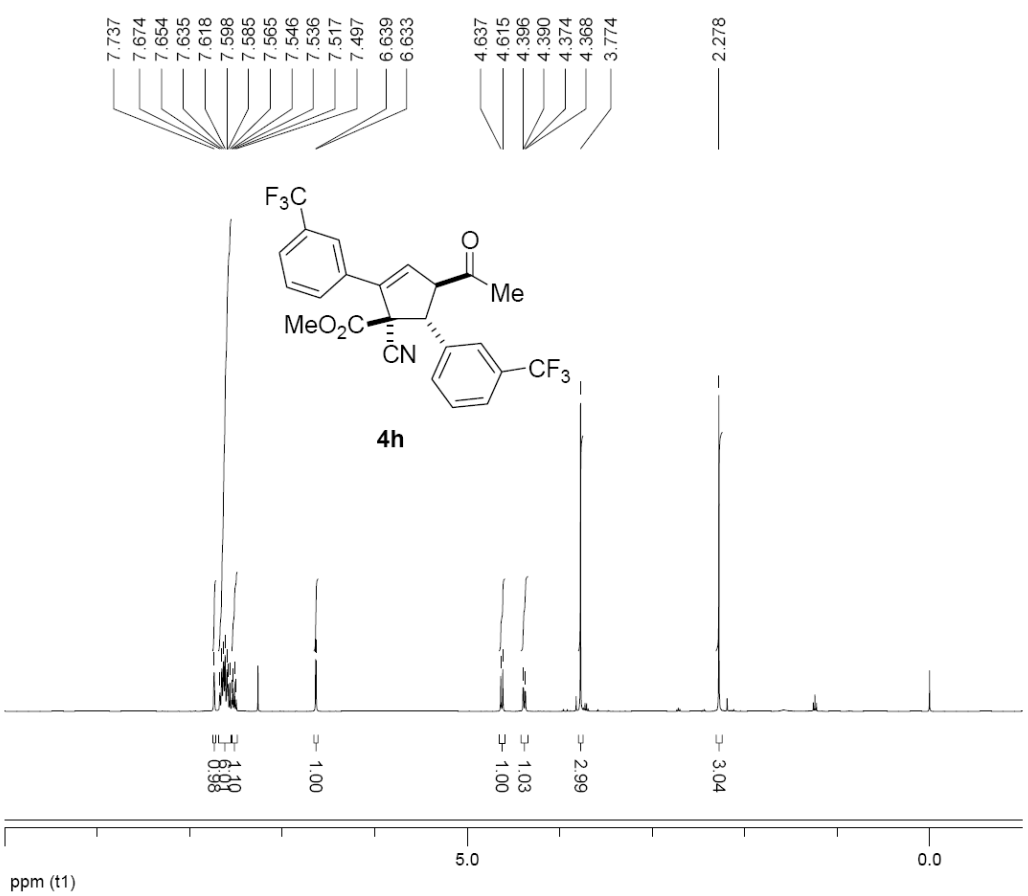


Date:
20 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

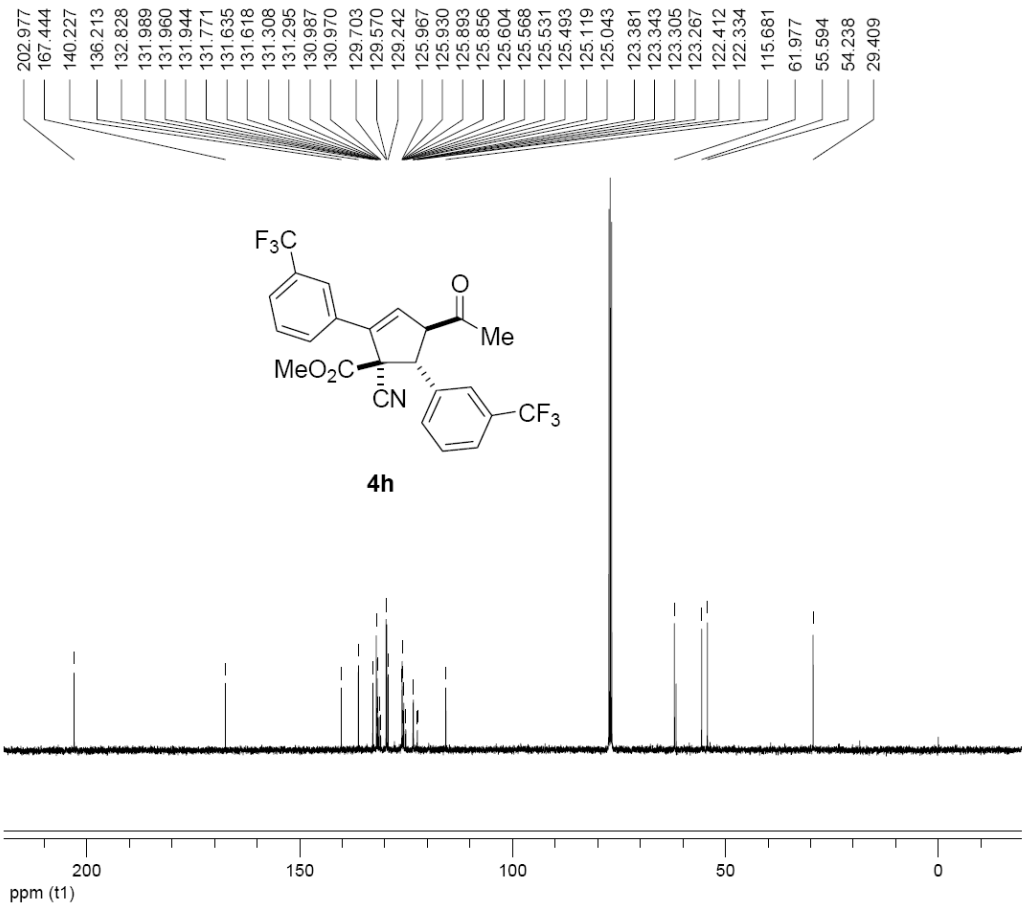
Frequency (MHz):
(f1) 100.613
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 1.3631
Spectral Width (ppm):
(f1) 238.921
Pulse Program:
Unknown



Date:
20 Jul 2010
Document's Title:
1r.mrc

Spectrum Title:
PROTON

Frequency (MHz):
(f1) 400.130
Original Points Count:
(f1) 32768
Actual Points Count:
(f1) 32768
Acquisition Time (sec):
(f1) 3.9846
Spectral Width (ppm):
(f1) 20.553
Pulse Program:
Unknown



Date:
20 Jul 2010
Document's Title:
c.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Spectral Width (ppm):
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Pulse Program:
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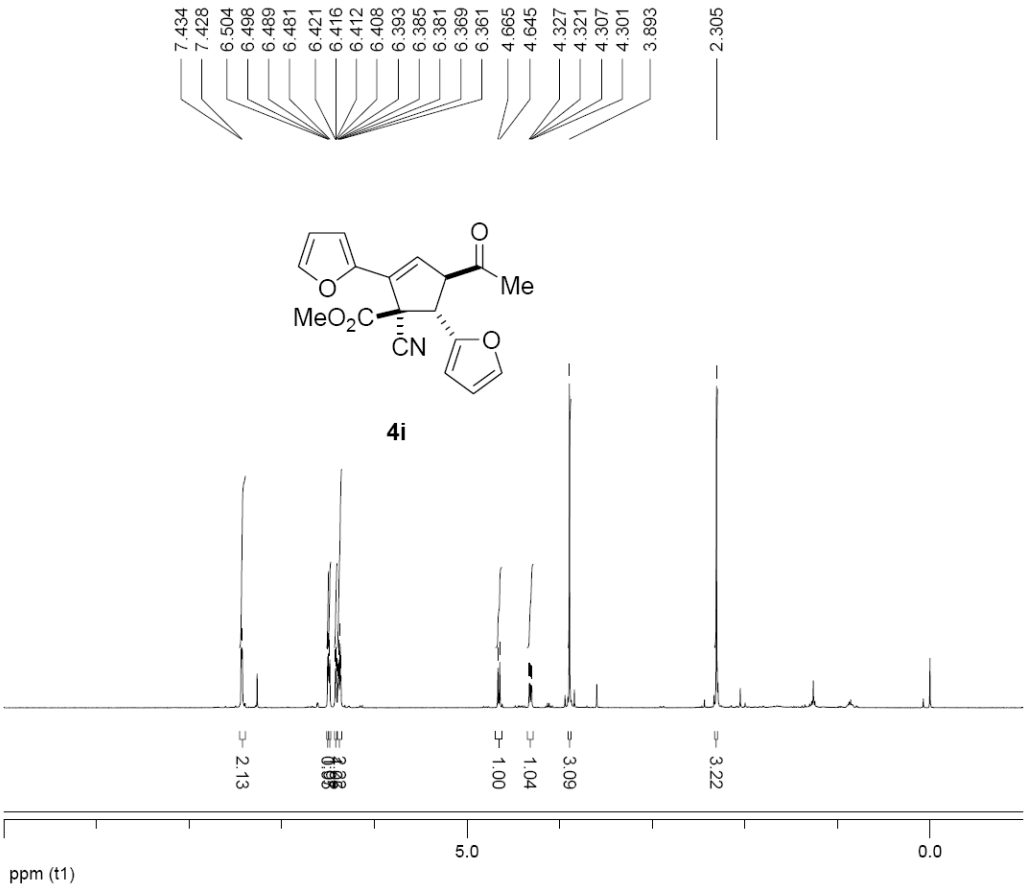

4i

Date:
20 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Original Points Count:
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Actual Points Count:
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Spectral Width (ppm):
(f1) 20.553
Pulse Program:
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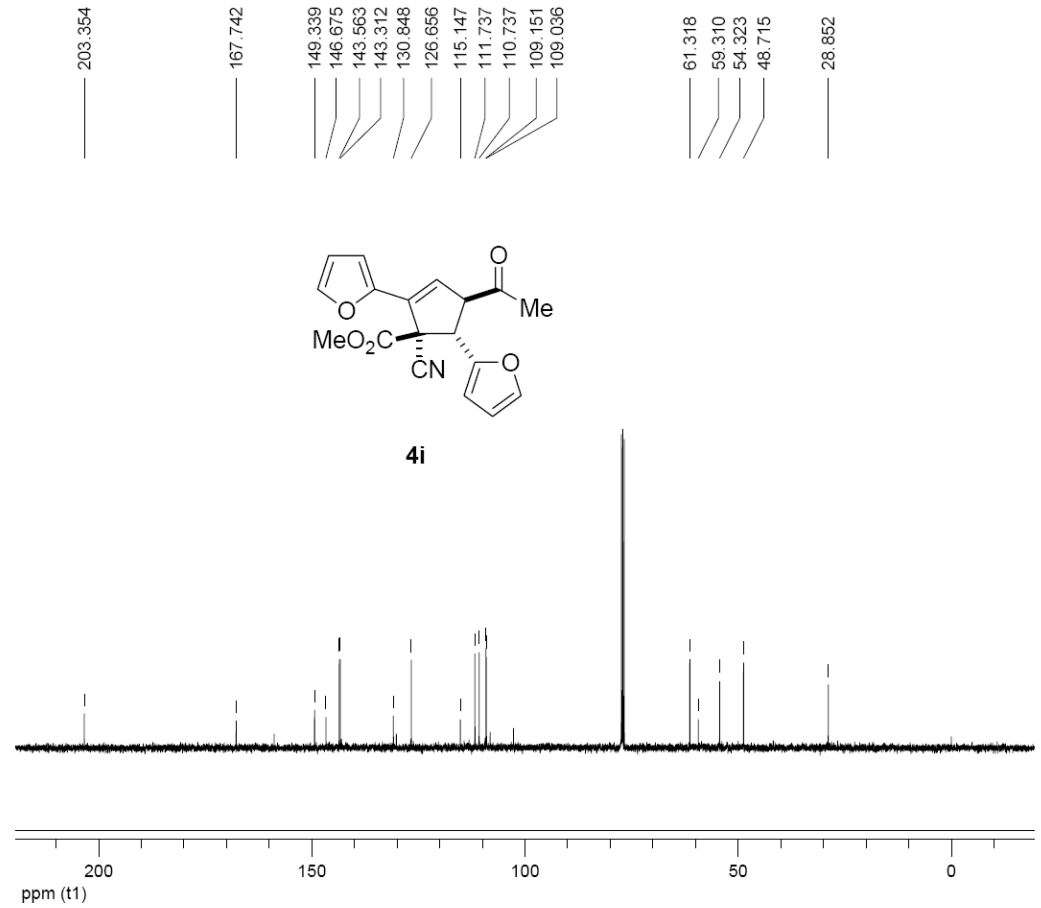

4i

Date:
20 Jul 2010
Document's Title:
C.mrc

Spectrum Title:
C13CPD

Frequency (MHz):
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Original Points Count:
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Spectral Width (ppm):
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Pulse Program:
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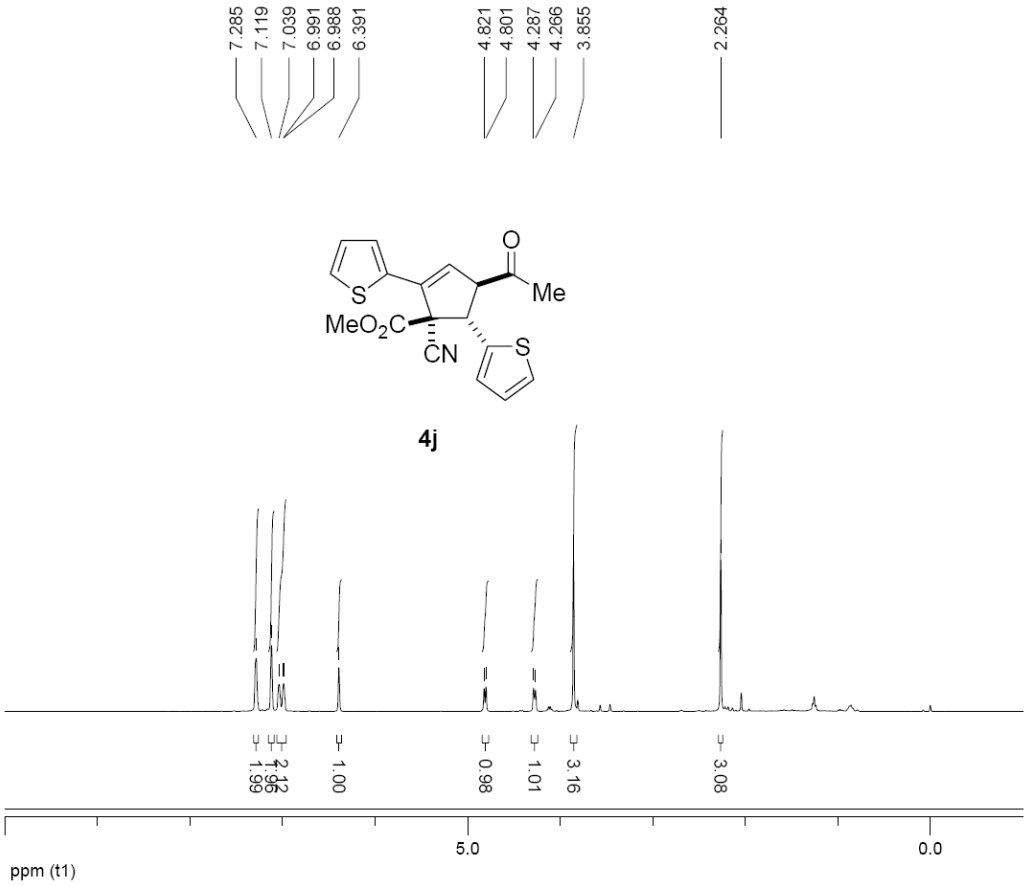

4j

Date: 20 Jul 2010
Document's Title: H.mrc

Spectrum Title: PROTON

Frequency (MHz): (f1) 400.130
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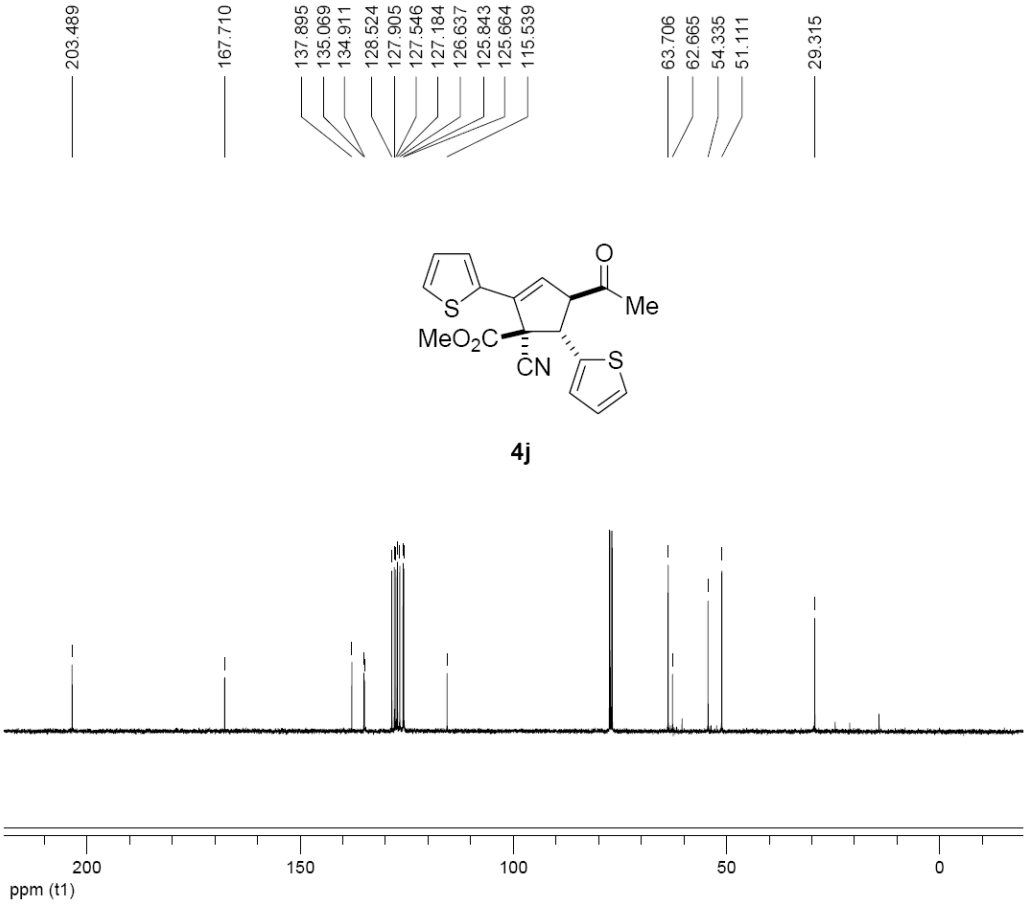
Spectral Width (ppm): (f1) 20.553
Pulse Program: Unknown



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Date: 20 Jul 2010
Document's Title: C.mrc

Spectrum Title: C13CPD

Frequency (MHz): (f1) 100.613
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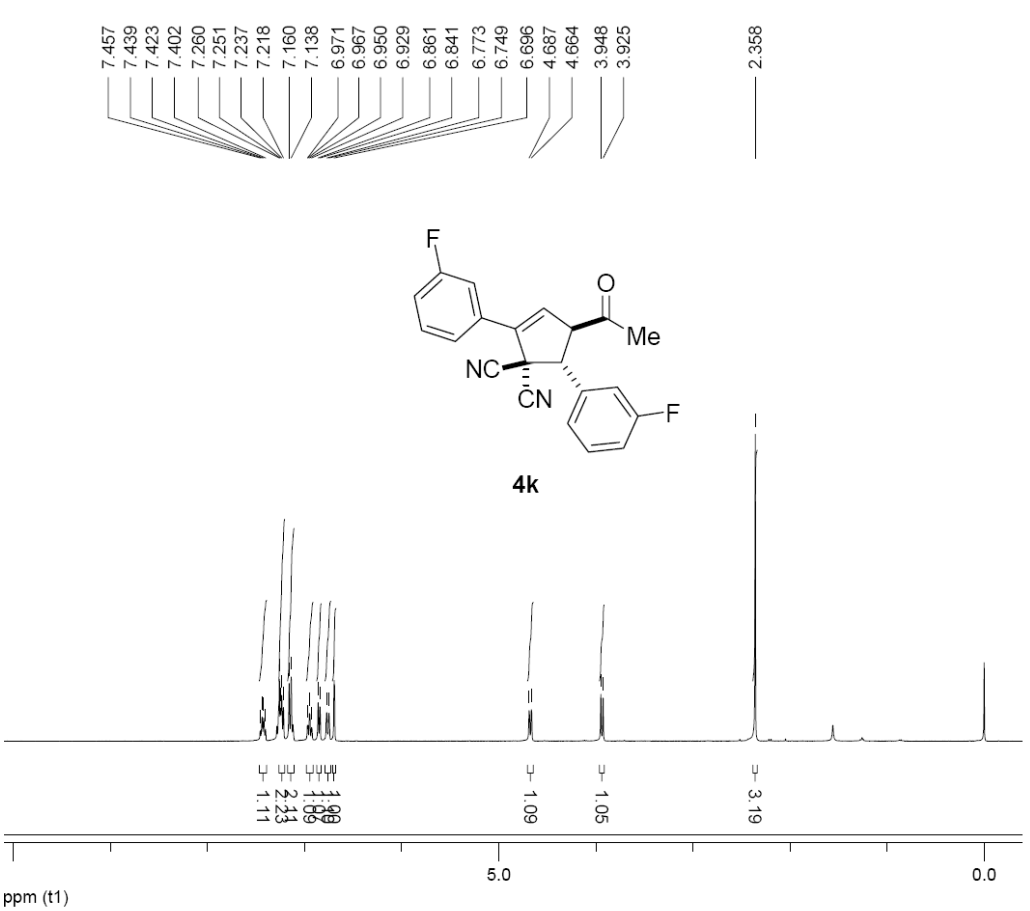
Pulse Program: Unknown



Date:
20 Jul 2010
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Spectrum Title:
PROTON

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(f1) 400.130
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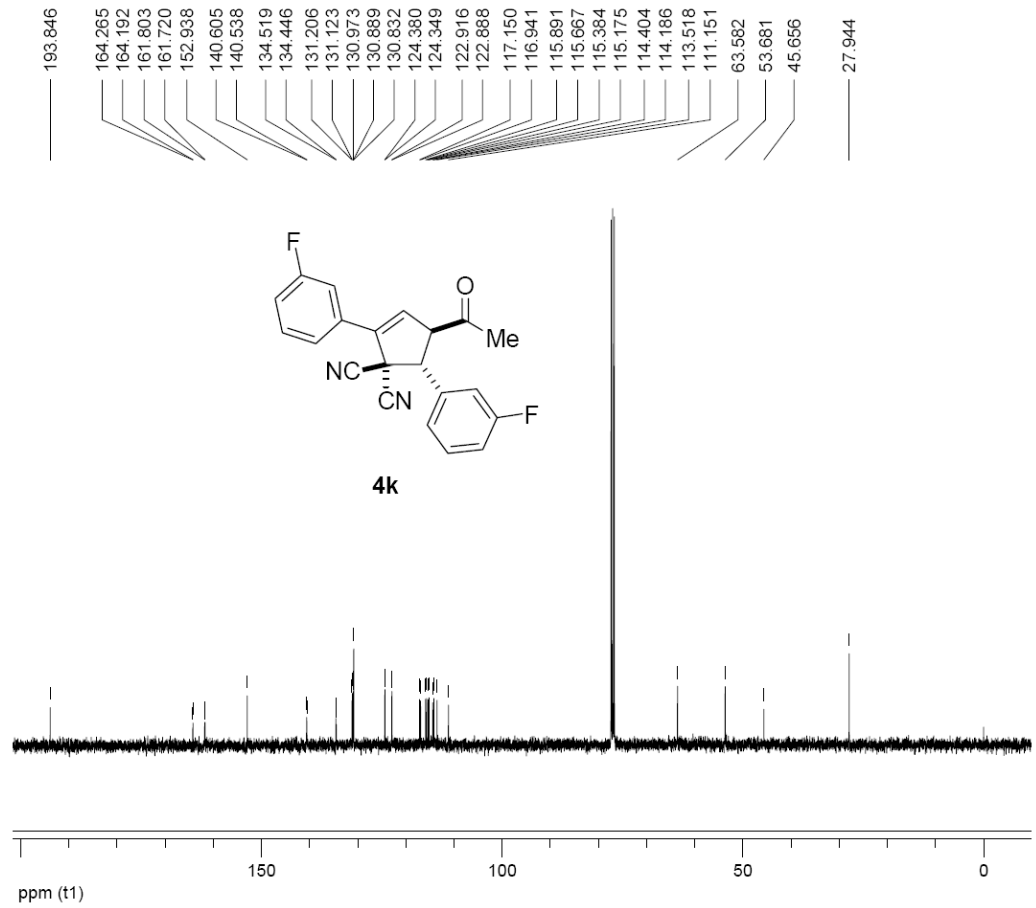

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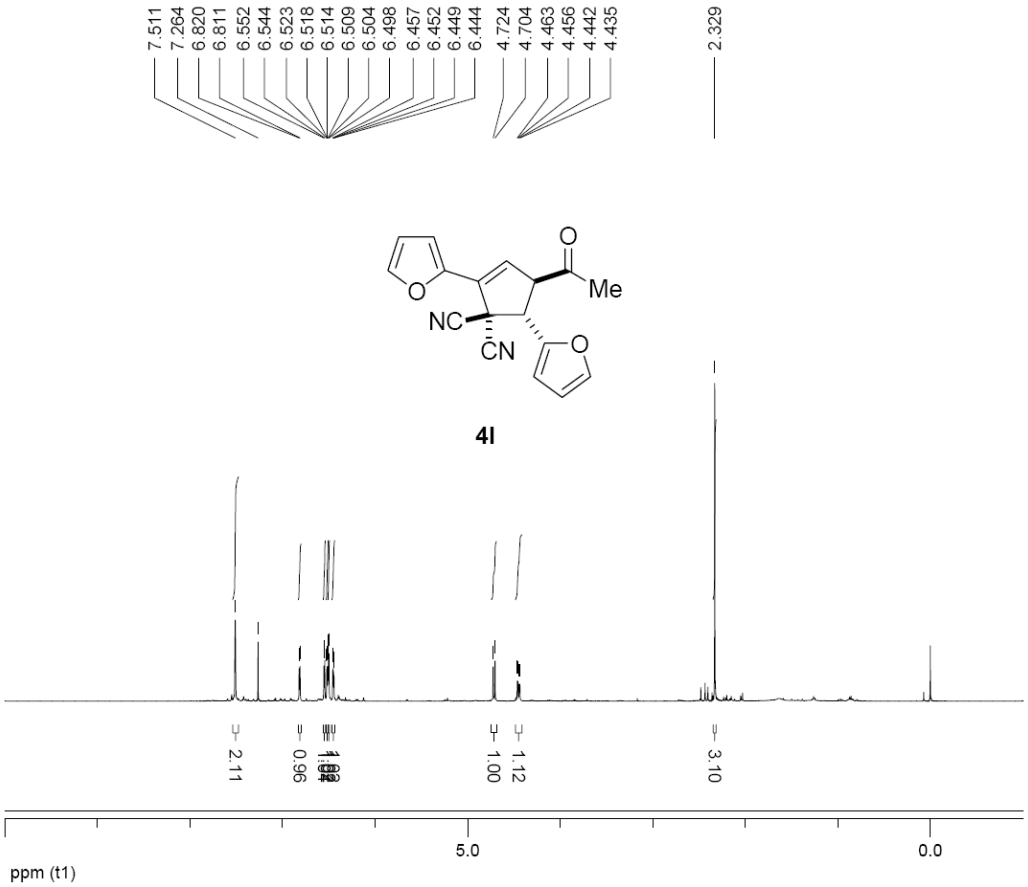

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Date:
20 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
PROTON

Frequency (MHz):
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Spectral Width (ppm):
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Pulse Program:
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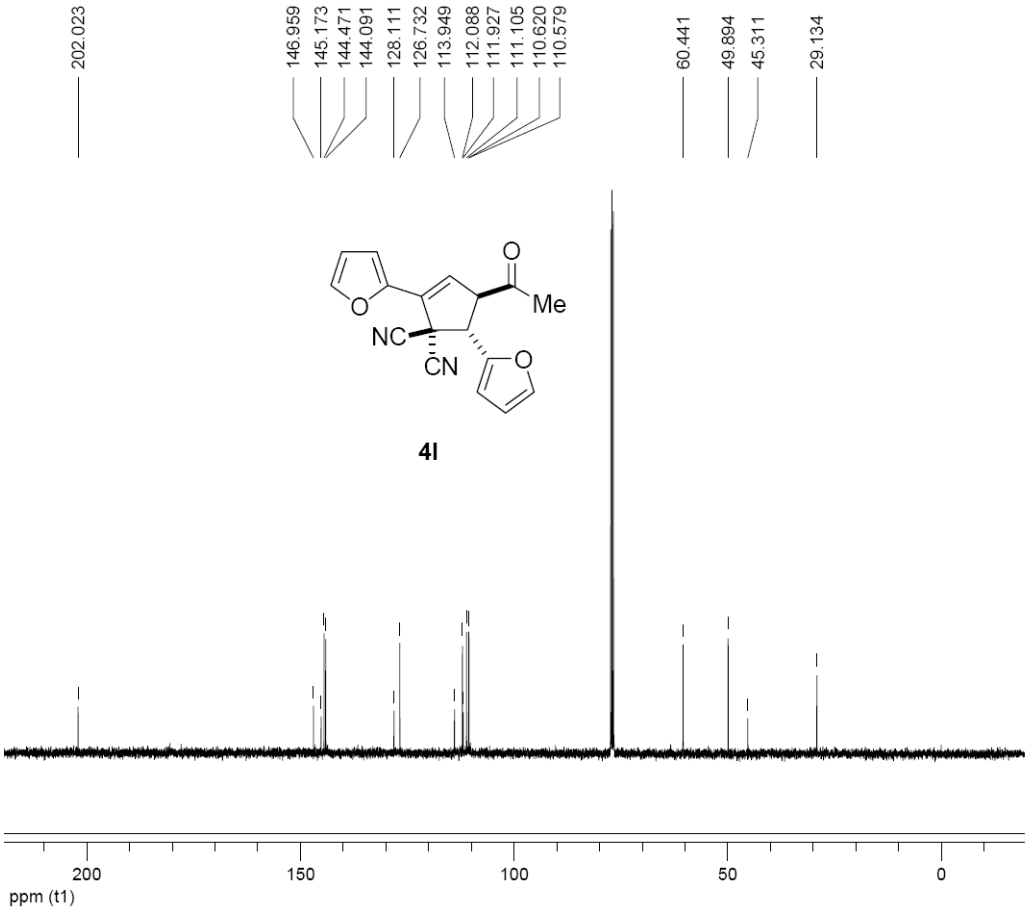

4l

Date:
20 Jul 2010
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C.mrc

Spectrum Title:
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(f1) 100.613
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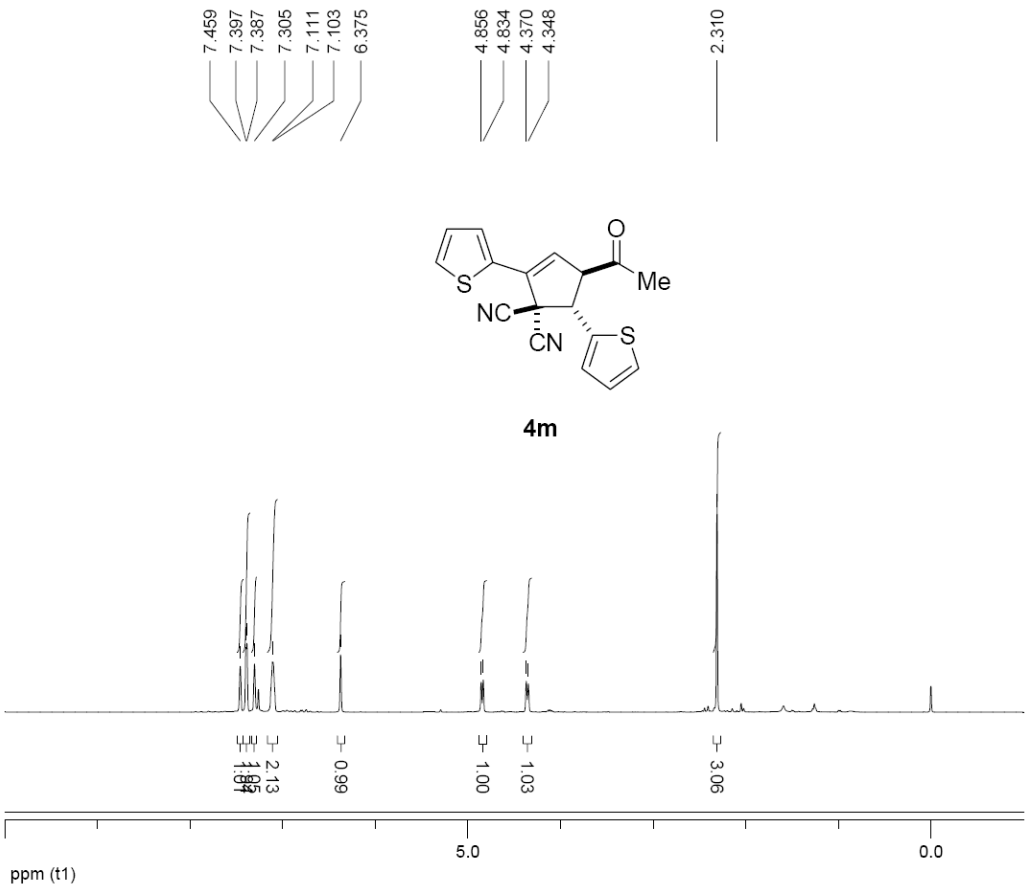

Date:
21 Jul 2010
Document's Title:
H.mrc

Spectrum Title:
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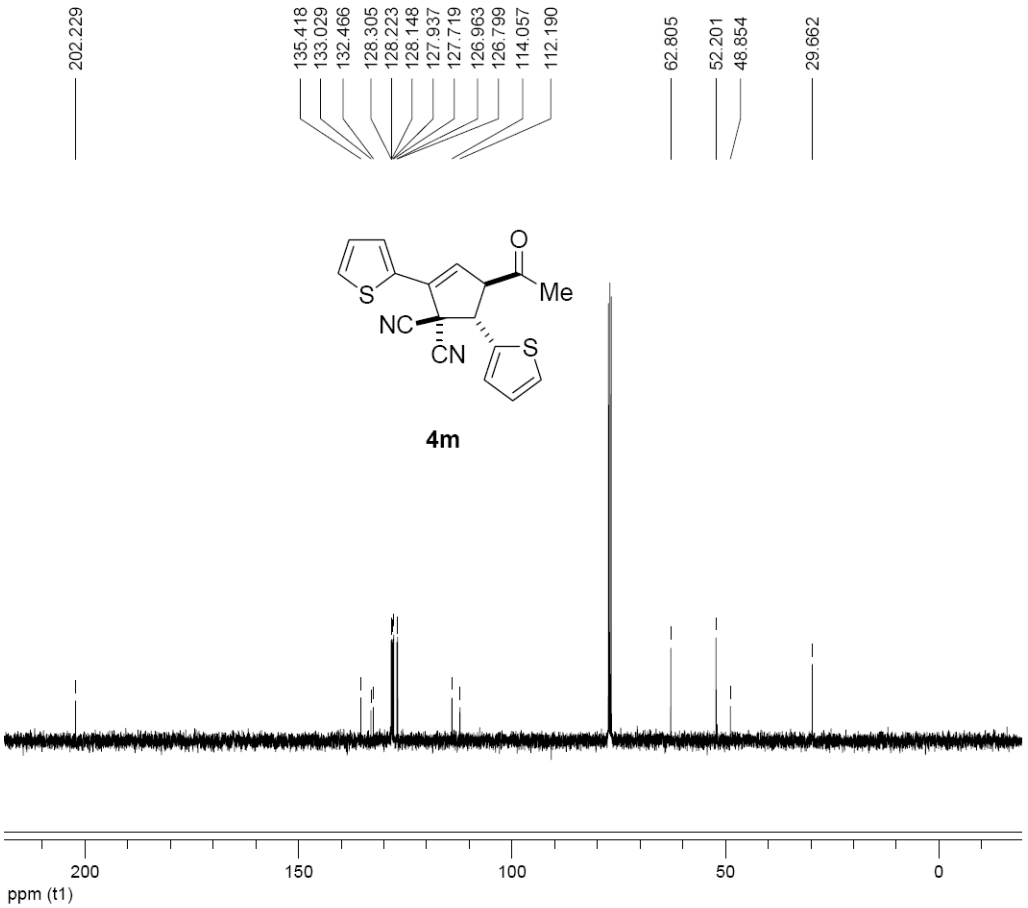

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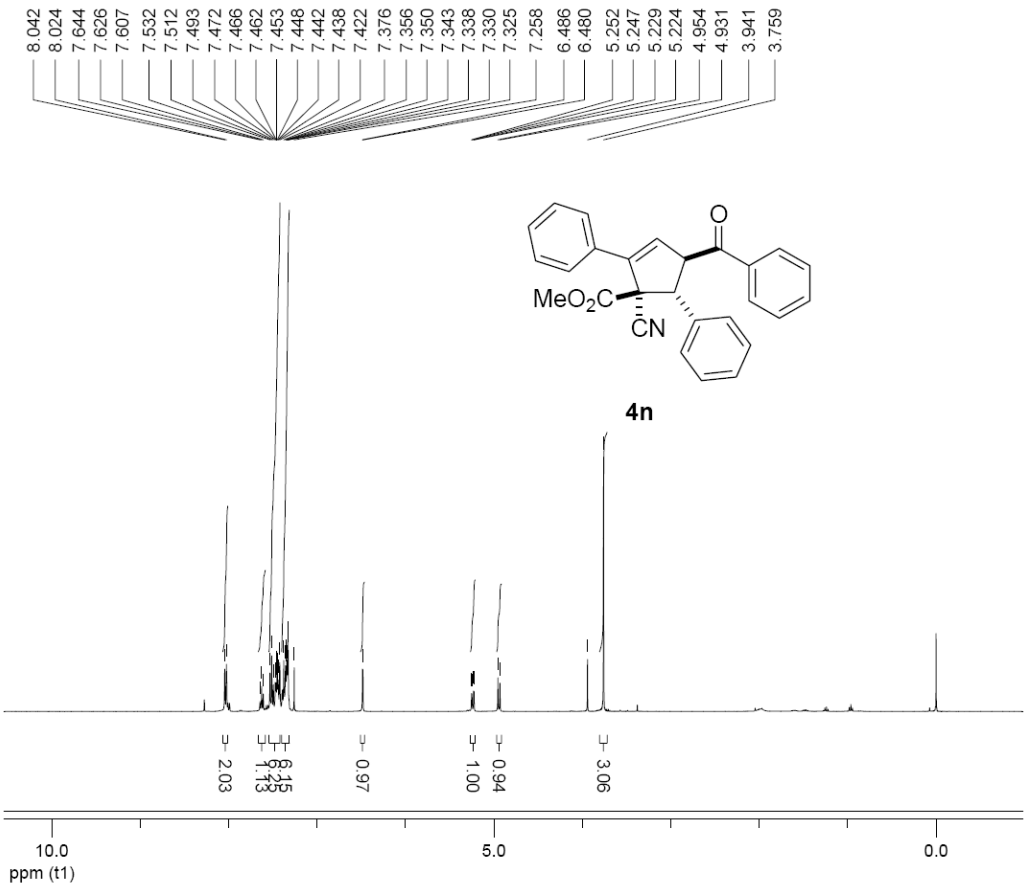

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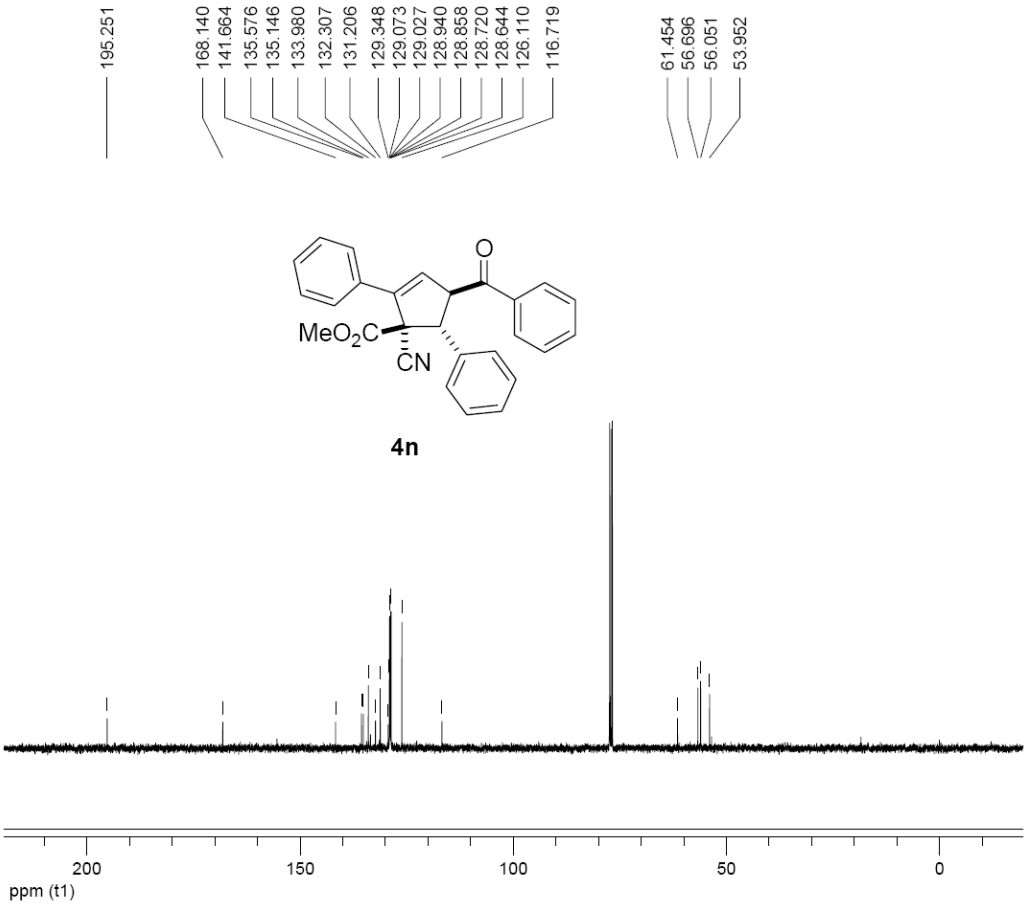

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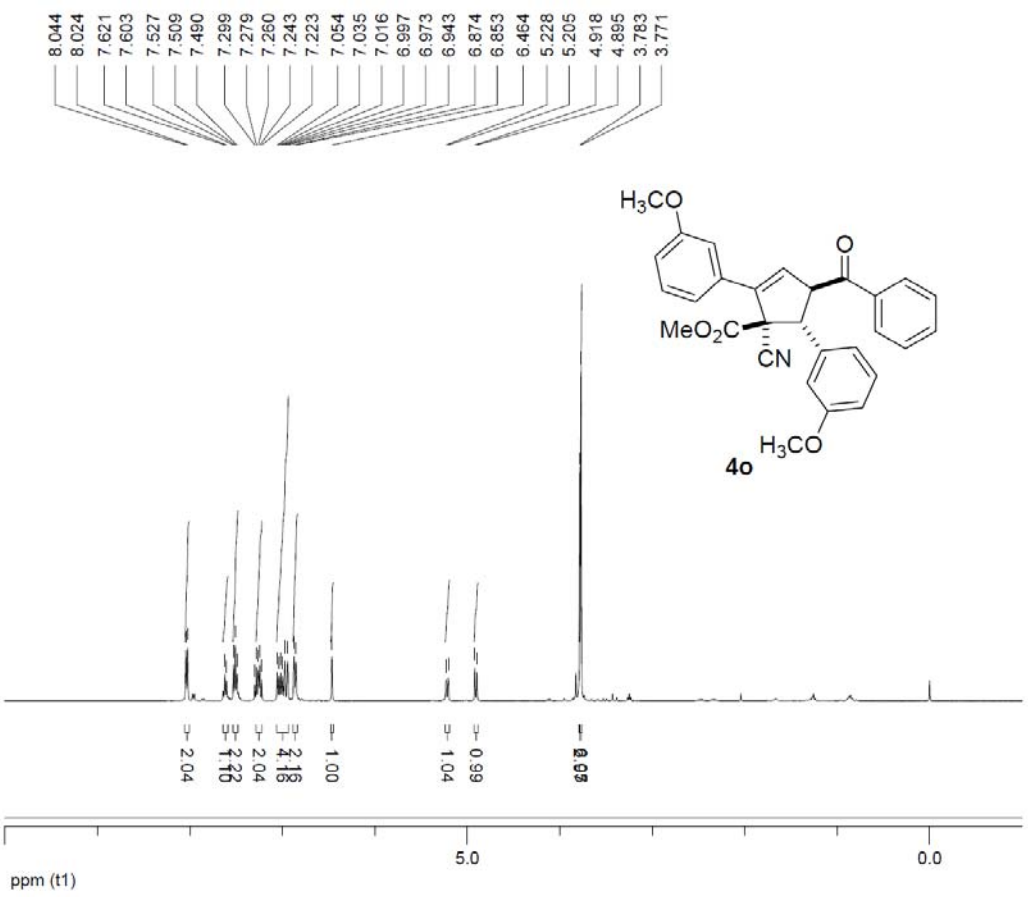

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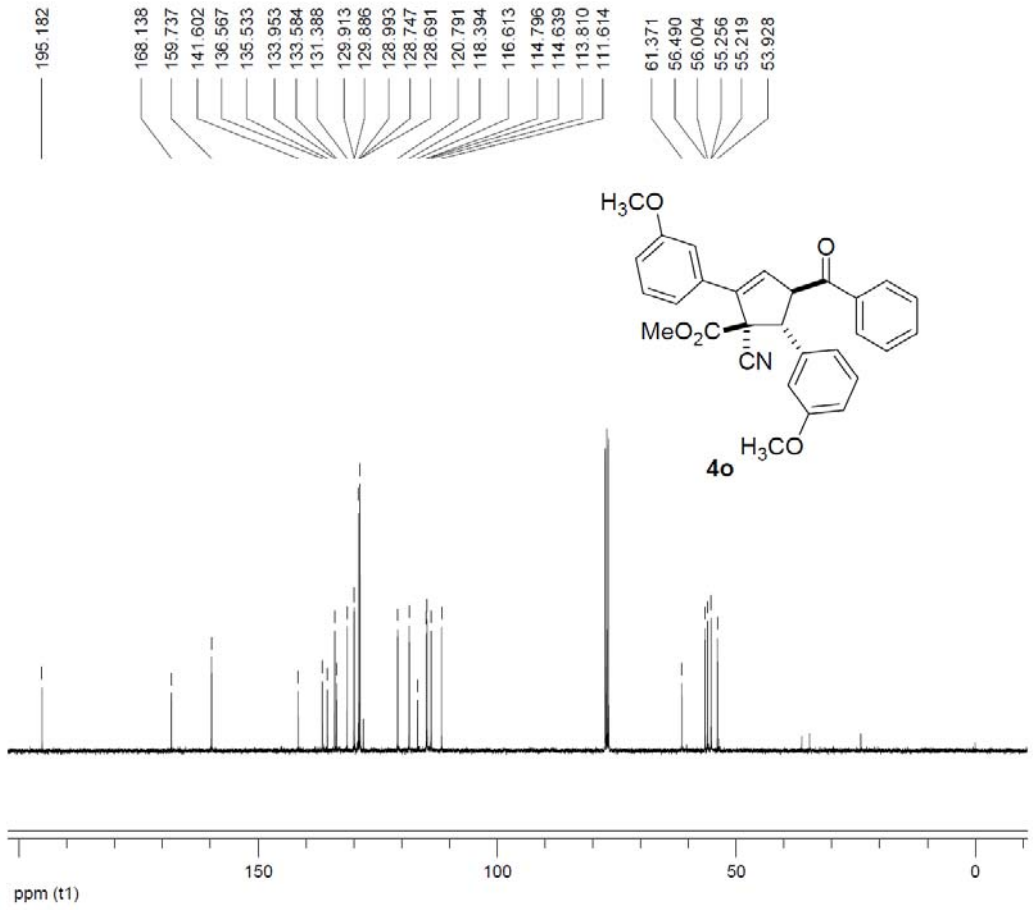

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