Supporting Information for

Photosensitized Splitting of Thymine Dimer or Oxetane Unit by a Covalently *N*-Linked Carbazole via Electron Transfer in Different Marcus Regions

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Table of Contents

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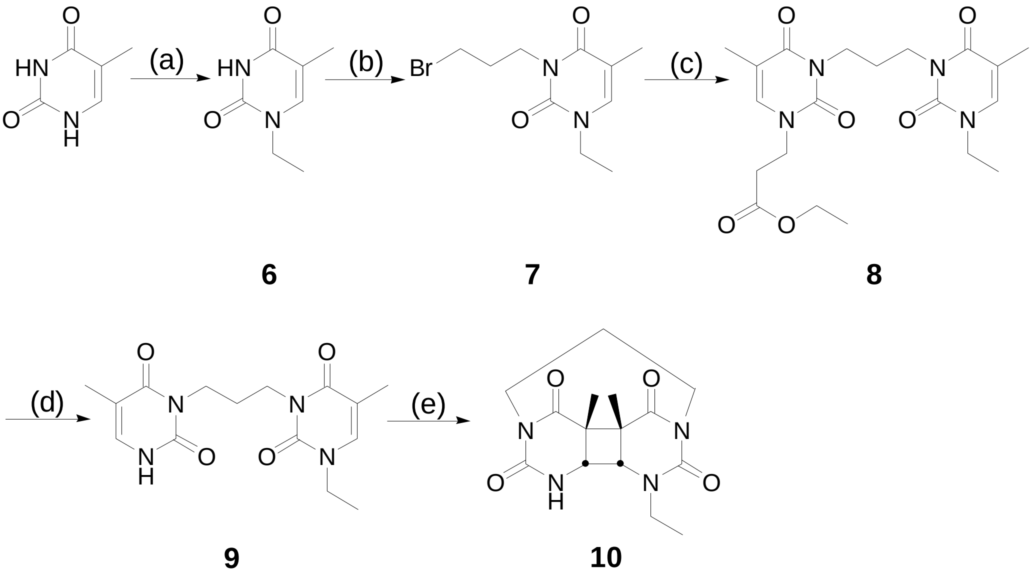
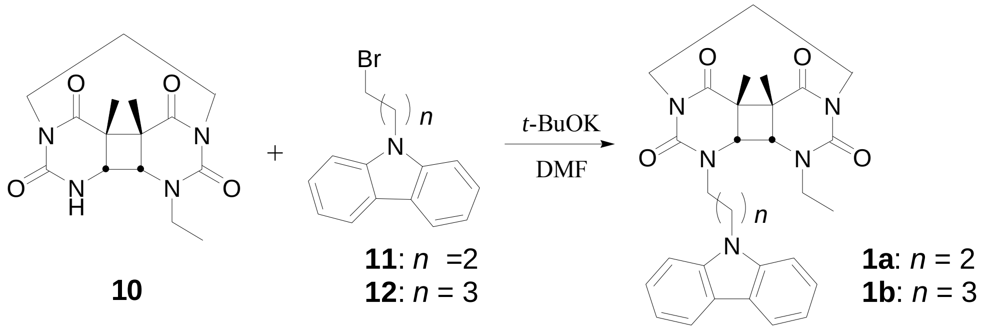
Synthesis of the Model Compounds

Model compounds were designed to be constructed with a thymine dimer unit or thymine oxetane unit covalently attached by a carbazole or indole moiety using a short aliphatic hydrocarbon as a linker.

For the preparation of independent thymine dimer unit, the detailed processing route[1] is depicted in Scheme S1. Thymine was dissolved into DMF to react with iodoethane to yield **6**. Reaction of **6** with dibromopropane gave **7**, which further reacted with ethyl thymine-1-propionate through the same reaction style as last step to afford **8** in a satisfying yield. **9** was obtained by dissolving **8** into toluene and pouring it into a refluxed toluene solution suspended with sodium to eliminate ethyl propionate. **9** was irradiated with a 300 W high pressure Hg lamp accompanying to bubble with high pure N_2 in MeCN/Acetone mixed solvent to lead to **10**.



Scheme S1 Reagents and conditions: (a) iodoethane, K_2CO_3, DMF, RT, 24h; (b) dibiomopropane, K_2CO_3, DMF, RT, 48h; (c) ethyl thymine-1-propionate, K_2CO_3, DMF, RT, 48h; (d) Metal sodium, toluene, reflux, overnight; (e) hv (>290nm), MeCN/Acetone, RT, 5h.

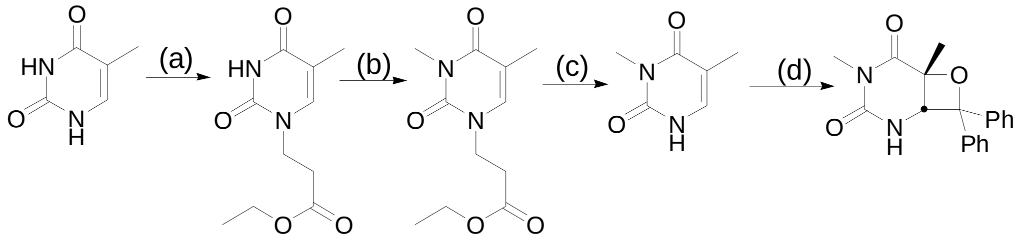


Scheme S2

The carbazole moiety was introduced onto the thymine dimer unit by a simple nucleophilic reaction[1, 2] between **10** and *N*-biomobroalkyl carbazole **11**[3] and **12**[3] with potassium *tert*-butoxide, and synthesis procedure is depicted in Scheme S2.

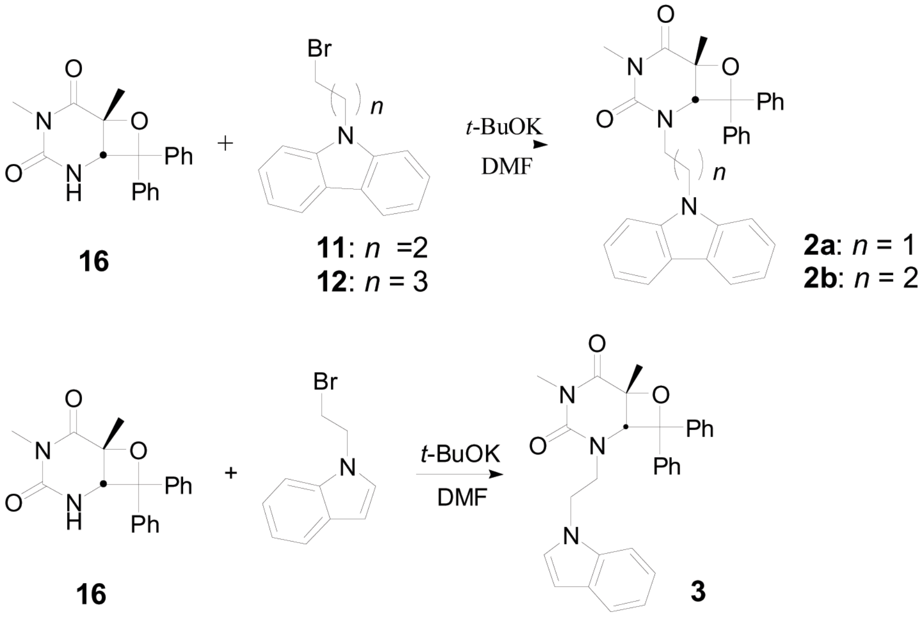
Compound **11**[3] was synthesized through the procedure where carbazole reacted with dibromoethane (also utilized as solvent) in the presence of K_2CO_3, KOH and tetrabutyl ammonium bromide (TBAB)[4], instead, **12**[3] was obtained via phase transfer catalytic reaction[5] using TBAB serving as phase-transfer catalyst in benzene/water two-phase solvent system.

For the synthesis of thymine oxetane model compounds, a similar preparation process was adopted as that for thymine dimer models. First of all, the oxetane unit was prepared in terms of the route shown in Scheme S3. Compound **13**[6] was obtained from the reaction between thymine and ethyl acrylate under refluxed condition, then **13**[6] reacted with iodomethane in the presence of K_2CO_3 (dried at 300°C for 5 h) gave **14**, which dissolved into toluene and followed the condition of step (d) displayed in Scheme S1 to yield **15**.[7] Compound **16**[2] can be obtained via a Paternò–Büchi [2+2]cycloaddition reaction of **15**[7] with benzophenone[2] at room temperature.



13 **14** **15** **16**

Scheme S3 Reagents and conditions: (a) ethyl acrylate, NaOH, hydroquinone, reflux, 24h; (b) iodomethane, K_2CO_3, DMF, RT, 9h; (c) Metal sodium, toluene, reflux, overnight; (d) benzophenone, *hv* (> 290 nm), MeCN, RT, 5h.



Scheme S4

The oxetane unit **16** was alkylated with **11** in the presence of potassium *tert*-butoxide to yield model compound **2a** (shown in Scheme S4)[2]. A same method was carried out to give model compound **2b** and **3** .

Irradiation of model compounds with a monochromatic light (328nm) yielded the corresponding photolysis products **4** and **5**, which were purified by separation from column chromatography (silica gel-H).

Characterization data of synthesized compounds

1-ethyl-5-methylpyrimidine-2,4(1H,3H)-dione (6). To a 20 mL DMF solution of thymine (1.50 g, 11.9 mmol), dry K_2CO_3 (1.64 g, 11.9 mmol) was added dropwise iodoethane (1.56 g, 10.0 mmol). The reaction mixture was stirred at room temperature for 24 h, and then diluted with 100 mL water. The organic components were extracted with CH_2Cl_2 three times (40 mL×3). Purified product **6** (0.42 g, 28%) was obtained via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:1-3:2) as white powder. R_f = 0.28 (ethyl acetate / petroleum ether 1:1). m.p. 218–220°C. IR (KBr): nu(tilde) = 3036s, 2977s, 1687s, 1474s, 1349s cm^{-1}. ^1H NMR (300Hz, $DCCl_3$): δ = 1.29-1.33 (m, 3H, CH_2CH_3), 1.93 (d, J=0.9Hz, 3H, CH_3), 3.72-3.80 (m, 2H, CH_2CH_3), 7.00 (d, J=1.2Hz, 1H, CH). ^{13}C NMR (75MHz, d_6-DMSO): δ = 164.3, 150.7, 141.1, 108.5, 42.4, 14.1, 11.9. TOFMS (EI) calcd for (M^+) $C_7H_{10}N_2O_2$: 154.0742, found 154.0736.

3-(3-bromopropyl)-1-ethyl-5-methylpyrimidine-2,4(1H,3H)-dione (7). Dibromopropane (0.77 g, 3.8 mmol), dry K_2CO_3 (0.42 g, 3.0 mmol) were added into 4 mL anhydrous DMF dissolved with **6** (0.30 g, 1.95 mmol), it was stirred at room temperature for 48 h, and then diluted with 80 mL ethyl acetate, washed with 100 mL water. Solvent was removed under reduced pressure. The crude products were purified via column chromatography (silica gel-H, ethyl acetate / petroleum ether 3:2). **7** (0.36 g, 67%) was obtained as colorless oil. R_f = 0.51 (ethyl acetate / petroleum ether 2:1). ^1H NMR (300Hz, $DCCl_3$): δ = 1.28-1.33 (m, 3H, CH_2CH_3), 1.93-1.94 (d, J=1.2Hz, 3H, CH_3), 2.18-2.27 (m, 2H, $CH_2CH_2CH_2$), 3.40-3.45 (m, 2H, $CH_2CH_2CH_2$), 3.74-3.82 (m, 2H, CH_2CH_3), 4.07-4.12 (m, 2H, $CH_2CH_2CH_2$), 6.98 (d, J=1.2Hz, 1H, CH). ^{13}C NMR (75MHz, $CDCl_3$): δ = 163.6, 151.0, 138.1, 109.7, 44.4, 40.1, 30.9, 30.4, 14.2, 12.8. TOFMS (EI) calcd for (M^+) $C_{10}H_{15}N_2O_2Br$: 274.0317, found 274.0313.

Ethyl3-(3-(3-(3-ethyl-5-methyl-2,6-dioxo-2,3-dihydropyrimidin-1(6H)-yl)propyl)-5-methyl-2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)propanoate (8). A reaction mixture of **7** (0.48 g, 1.8 mmol), ethyl thymine-1-propionate (0.73 g, 3.2 mmol) and dry K_2CO_3 (0.44 g, 3.2 mmol) in 4 mL anhydrous DMF was stirred at room temperature for 48 h. After reaction, 50 mL ethyl acetate was poured in for dilution and washed with 50 ml water. Solvent was removed under reduced pressure. The crude products were purified via column chromatography (silica gel-H, ethyl acetate / petroleum ether 3:2-3:0). **8** (0.54 g, 74%) was obtained as colorless oil. R_f= 0.69 (ethyl acetate). IR (KBr): nu(tilde) = 2975m, 1734s, 1700s, 1667s, 1466s cm^{-1}. ^{1}H NMR (300Hz, $DCCl_3$): δ = 1.20-1.31 (m, 6H, CH_3, CH_2CH_3), 1.90 (d, J=0.9Hz, 3H, CH_3), 1.91-2.04 (m, 2H, $CH_2CH_2CH_2$), 2.73-2.77 (m, 2H, CH_2), 3.72-3.79 (m, 2H, CH_2CH_3), 3.94-4.06 (m, 6H, $CH_2CH_2CH_2$, CH_2CH_2), 4.11-4.19 (m, 2H, CH_2CH_3), 6.95 (d, J=0.9Hz, 1H, CH), 7.13 (d, J=0.9Hz, 1H, CH). ^{13}C NMR (75MHz, $CDCl_3$): δ = 171.2, 163.6, 151.1, 151.0, 139.4, 137.9, 109.6, 109.2, 60.8, 45.7, 44.3, 39.1, 33.0, 26.1, 14.2, 14.0, 12.9, 12.8. TOFMS (EI) calcd for (M^+) $C_{20}H_{28}N_4O_6$: 420.2009, found 420.1949.

1-ethyl-5-methyl-3-(3-(5-methyl-2,6-dioxo-2,3-dihydropyrimidin-1(6H)-yl)propyl)pyrimidine-2,4(1H,3H)-dione (9). A solution of anhydrous toluene (20 mL) containing sodium (0.20 g, 8.7 mmol) was refluxed for 20 min to disperse sodium, then cooled to room temperature. **8** (0.54 g, 1.3 mmol) dissolved in 20 mL toluene was added and reflux was resumed for 20 h under N_2 atmosphere. 1 mL CH_3COOH and 10 ml CH_3OH were added to clear reaction mixture, solvent was removed and crude product was purified by column chromatography (silica gel-H, ethyl acetate). **9** (0.34 g, 82%) was obtained as white powder. R_f= 0.41 (ethyl acetate). m.p. 183–184°C. IR (KBr): nu(tilde) = 2973m, 2927m, 1698s, 1666s, 1630s, 1453m cm^{-1}. ^{1}H NMR (300Hz, $DCCl_3$): δ = 1.27-1.33 (m, 3H, CH_2CH_3), 1.91-1.92 (m, 6H, CH_3, CH_3), 1.96-2.06 (m, 2H, $CH_2CH_2CH_2$), 3.73-3.80 (m, 2H, CH_2CH_3), 4.00-4.07 (m, 4H, $CH_2CH_2CH_2$), 6.96-7.00 (m, 2H, CH, CH), 8.82-8.84 (d, J=4.8Hz, 1H, NH). ^{13}C NMR (75MHz, d_6-DMSO): δ = 163.7, 163.0, 151.2, 150.5, 139.7, 136.2, 107.7, 107.1, 43.5, 38.4, 37.6, 25.8, 14.0, 12.5, 12.4. TOFMS (EI) calcd for (M^+) $C_{15}H_{20}N_4O_4$: 320.1485, found 320.1491.

1-ethyl-3,3'-trimethylene-*cis,syn*-thymine dimer (10). **9** (0.14 g, 0.44 mmol) was dissolved in a mixed solution of MeCN (30 mL) and acetone (20 mL) placed in a Pyrex photochemical reactor and bubbled with N_2 for 20 min and then irradiated for 5 h with a 300W high pressure Hg lamp. During irradiation, N_2 bubbling was continued. The reaction mixture was evaporated to dryness in vacuo. The product **10** (0.10 g, 71%) as white solid was obtained via recrystallization from CH_3Cl / petroleum ether. R_f = 0.38 (ethyl acetate). m.p. 237–239°C. IR (KBr): nu(tilde) = 3114w, 2964m, 2938w, 1720s, 1690s, 1658s, 1475s cm^{-1}. ^{1}H NMR (300Hz, $DCCl_3$): δ = 1.13-1.18 (m, 3H, CH_2CH_3), 1.49 (s, 3H, CH_3), 1.50 (s, 3H, CH_3), 2.90-2.97 (m, 1H, $CH_2CH_2CH_2$), 3.78-3.99 (m, 5H, $CH_2CH_2CH_2$, CH_2CH_3), 4.16-4.23 (m, 2H, $CH_2CH_2CH_2$), 6.20 (s, 1H, NH). ^{13}C NMR (75MHz, d_6-DMSO): δ = 170.8, 170.2, 152.0, 151.1, 56.5, 51.6, 49.7, 49.2, 41.2, 40.8, 40.1, 22.2, 17.5, 17.3, 12.2. TOFMS (EI) calcd for (M^+) $C_{15}H_{20}N_4O_4$: 320.1485, found 320.1482.

9-(2-bromoethyl)-9H-carbazole (11).[3] To a mixture of K_2CO_3 (1.8 g, 0.013 mol), KOH (2.2 g, 0.04 mol) and tetrabutyl ammonium bromide(0.04 g, 0.12 mmol) in ethylene dibromide (10 mL) was added carbazole (1.0 g, 6.0 mmol) and stirred fiercely at 50°C for 5.5 h, then it was diluted with dichloromethane and washed with water three times. The solvent was removed under reduced pressure, the crude product was purified by column chromatography (silica gel-H, petroleum ether) to give **11** (0.42 g, 26%) as white powder. ^{1}H NMR (300Hz, $DCCl_3$): δ = 3.65-3.70 (m, 2H, CH_2CH_2), 4.69-4.74 (m, 2H, CH_2CH_2), 7.24-7.29 (m, 3H, carbazole-H), 7.42-7.51 (m, 3H, carbazole-H), 8.09-8.11 (d, J=7.8Hz, 2H, carbazole-H).

9-(3-bromopropyl)-9H-carbazole (12).[3] To a mixture of tetrabutyl ammonium bromide (0.10 g, 0.31 mmol) and aqueous 50% NaOH (3 mL) was added a solution of carbazole (1.0 g, 6.0 mmol) and propane dibromide (2 mL) in benzene (3 mL). The mixture was stirred at room temperature for 12 h. Water was added and the organic components were extracted with dichloromethane. Solvent was removed, purification via column chromatography (silica gel-H, petroleum ether) gave **12** (0.64 g, 37%) as light yellow powder. ^{1}H NMR (300Hz, $DCCl_3$): δ = 2.40-2.49 (m, 2H, $CH_2CH_2CH_2$), 3.37-3.41 (m, 2H, $CH_2CH_2CH_2$), 4.49-4.53 (m, 2H, $CH_2CH_2CH_2$), 7.20-7.29 (m, 3H, carbazole-H), 7.44-7.50 (m, 3H, carbazole-H), 8.09-8.11 (d, J=7.8Hz, 2H, carbazole-H).

Model compound 1a. Potassium tert-butoxide (35 mg, 0.31 mmol) was added to a 4 mL anhydrous DMF solution dissolved with **10** (50 mg, 0.16 mmol) and stirred at room temperature for 30min. Then 9-(2-bromoethyl)-9H-carbazole **11** (90 mg, 0.32 mmol) was added into, stirring was continued for 4 h. The reaction mixture was diluted with 80 mL CH_2Cl_2, washed with 80 mL water. Purified **1a** (16 mg, 20%) as white power was obtained via column chromatography(silica gel-H, ethyl acetate / petroleum ether 1:1-1:0) and following recrystallization from CH_2Cl_2 / petroleum ether. R_f = 0.57 (ethyl acetate / petroleum ether 2:1). m.p. >250°C. IR (KBr): nu(tilde) = 1695s, 1660s, 1640s, 1460s cm^{-1}. ^1H NMR (300Hz, $DCCl_3$): δ = 0.15 (s, 3H, CH_3), 1.01-1.06 (m, 3H, CH_3), 1.13 (s, 3H, CH_3), 1.58-1.60 (m, 1H, $CH_2CH_2CH_2$), 2.42-2.45 (d, J=7.2Hz, 1H, CH), 2.89-3.01 (m, 1H, CH_2CH_2), 3.41-3.43 (d, J=6.9Hz, 1H, CH), 3.94-4.81 (m, 8H, $CH_2CH_2CH_2$, CH_2CH_2), 7.23-7.30(m, 4H, carbazole-H), 7.40-7.46 (m, 2H, carbazole-H), 8.08-8.11 (d, J=7.5Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, $CDCl_3$): δ = 170.0, 169.3, 151.8, 151.3, 140.0, 126.4, 122.8, 120.7, 119.7, 108.0, 60.5, 57.7, 50.5, 50.4, 48.2, 42.8, 41.6, 41.3, 40.7, 22.8, 17.9, 16.5, 13.0. TOFMS (EI) calcd for (M^+) $C_{29}H_{31}N_5O_4$: 513.2376, found 513.2354.

Model compound 1b. Potassium tert-butoxide (35 mg, 0.31 mmol) was added to a 4 mL anhydrous DMF solution dissolved with **10** (50 mg, 0.16 mmol) and stirred at room temperature for 30min. Then 9-(3-bromopropy)-9H-carbazole **12** (90 mg, 0.30 mmol) was added into, stirring was continued for 4 h. The reaction mixture was diluted with 80 ml CH_2Cl_2, washed with 80 mL water. Purified **1b** (35 mg, 44%) as white power was obtained via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:1-1:0) and following recrystallization from CH_2Cl_2 / petroleum ether. R_f = 0.51 (ethyl acetate / petroleum ether 2:1). m.p. 212–214°C. IR (KBr): nu(tilde) = 1689s, 1667s, 1642s, 1455s cm^{-1}. ^1H NMR (300Hz, $DCCl_3$): δ = 0.85-0.90 (m, 3H, CH_3), 1.20 (s, 3H, CH_3), 1.30 (s, 3H, CH_3), 1.61-1.62 (m, 1H, $CH_2CH_2CH_2$), 1.83-1.88 (m, 1H, $CH_2CH_2CH_2$), 2.19-2.31 (m, 4H, $CH_2CH_2CH_2$), 3.17-3.19 (d, J=6.9Hz, 1H, CH), 3.29-3.31 (d, J=7.2Hz, 1H, CH), 3.76-3.94 (m, 3H, $CH_2CH_2CH_2$), 4.12-4.19 (m, 3H, $CH_2CH_2CH_2$), 4.38-4.41 (m, 2H, $CH_2CH_2CH_2$), 7.24-7.51 (m, 6H, carbazole-H), 8.11-8.14 (d, J=7.8Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, $CDCl_3$): δ = 170.0, 151.8, 151.2, 140.2, 126.0, 122.9, 120.5, 119.4, 108.7, 58.6, 57.9, 50.9, 50.2, 46.0, 43.0, 41.6, 41.3, 40.4, 27.6, 22.7, 18.1, 18.1, 13.0. TOFMS (EI) calcd for (M^+) $C_{30}H_{33}N_5O_4$: 527.2533, found 527.2510.

Ethyl 3-(5-methyl-2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)propanoate (13).[6] Thymine (5.0 g, 0.040 mol) was added to a 70 mL mixed ethanol solution of NaOH (138 mg, 3.5 mmol), hydroquinone (50 mg, 0.45 mmol), ethyl acrylate (5.3 g, 0.053 mol). The reaction mixture was refluxed for 24 h, then frozen-stored overnight. The precipitation was filtered, washed with cool ethanol two times and recrystallized from ethanol to give **13** (3.6 g, 42 %) as white needle-like solid. ^{1}H NMR (300Hz, $DCCl_3$): δ = 1.22-1.26 (m, 3H, CH_2CH_3), 1.89 (d, J=1.2Hz, 3H, CH_3), 2.72-2.76 (m, 2H, CH_2CH_2), 3.92-3.96 (m, 2H, CH_2CH_2), 4.10-4.18 (m, 2H, CH_2CH_3), 7.17 (d, J=0.9Hz, 1H, CH).

Ethyl 3-(3,5-dimethyl-2,4-dioxo-3,4-dihydropyrimidin-1(2H)-yl)propanoate (14). A mixture of **13** (0.67 g, 3.0 mmol), dry K_2CO_3 (0.63 g, 4.7 mmol), iodomethane (0.75 mL) in anhydrous DMF (4 mL) was stirred at room temperature for 9 h, and then diluted with 50 mL ethyl acetate, washed with 100 mL water and dried with Na_2SO_4. Purification via column chromatography (silica gel-H, ethyl acetate / petroleum ether 3:2) gave **14** (0.7 g, 94 %) as colorless oil. R_f = 0.55 (ethyl acetate / petroleum ether 1:1). IR (KBr): nu(tilde) = 2982w, 1734s, 1701s, 1670s, 1472s cm^{-1}. ^{1}H NMR (300Hz, $DCCl_3$): δ=1.23-1.28 (m, 3H, CH_2CH_3), 1.93 (d, J=0.9Hz, 3H, CH_3), 2.75-2.79 (m, 2H, CH_2CH_2), 3.35 (s, 3H, NCH_3), 3.96-4.00 (m, 2H, CH_2CH_2), 4.11-4.19 (m, 2H, CH_2CH_3), 7.17 (s, 1H, CH). ^{13}C NMR (75MHz, $CDCl_3$): δ = 171.1, 163.7, 151.2, 139.2, 108.9, 60.7, 45.6, 32.9, 27.5, 13.9, 12.7. TOFMS (EI) calcd for (M^+) $C_{11}H_{16}N_2O_4$: 240.1110, found 240.1082.

3,5-dimethylpyrimidine-2,4(1H,3H)-dione (15).[7] A solution of anhydrous toluene (20 mL) containing sodium (0.2 g, 8.7 mmol) was intensity refluxed for 20 min to disperse sodium, then cooled to room temperature. **14** (0.7 g, 2.9 mmol) dissolved in 10mL toluene was added and reflux was resumed for 20 h under N_2 atmosphere. 1 mL CH_3COOH and 10 ml CH_3OH were added to clear reaction mixture, solvent was removed and crude product was purified by column chromatography (silica gel-H, ethyl acetate / petroleum ether 3:1). **15** (0.3 g, 75 %) was obtained as white powder. ^{1}H NMR (300Hz, $DCCl_3$): δ = 1.90-1.91 (d, J=0.9Hz, 3H, CH_3), 3.31 (s, 3H, CH_3), 6.98-7.00 (m, 1H, CH), 9.11 (s, 1H, NH).

4,6-dimethyl-8,8-diphenyl-7-oxa-2,4-diazabicyclo[4.2.0]octane-3,5-dione (16).[2] A 40 mL CH_3CN solution of **15** (0.14 g, 1.0 mmol) and benzophenone (0.36 g, 2.0 mmol) was placed in Pyrex photochemical reactor and bubbled with N_2 for 20 min and then irradiated for 5 h with a 300W high pressure Hg lamp. During irradiation, N_2 bubbling was continued. The reaction mixture was evaporated to dryness in vacuo. The product **16** (90 mg, 28%) as white solid was obtained via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:3). ^{1}H NMR (300Hz, $DCCl_3$): δ = 1.77 (s, 3H, CH_3), 2.80 (s, 3H, NCH_3), 4.68-4.70 (d, J=4.8Hz, 1H, CH), 5.81-5.82 (d, J=3.6Hz, 1H, NH), 7.26-7.37 (m, 10H, benzene-H).

Model compound 2a. Potassium tert-butoxide (35 mg, 0.31 mmol) was added to a 4 mL anhydrous DMF solution dissolved with **16** (50 mg, 0.16 mmol) and stirred at room temperature for 30 min. Then 9-(2-bromopropy)-9H-carbazole (85 mg, 0.32 mmol) was added into, stirring was continued for 4 h. The reaction mixture was diluted with 80 ml CH_2Cl_2, washed with 80 mL water. Purified **2a** (14 mg, 18%) as white power was obtained via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:3) and following recrystalization from CH_2Cl_2 / petroleum ether. R_f= 0.63 (ethyl acetate / petroleum ether 2:1). m.p. 203–204°C. IR (KBr): nu(tilde) = 1713s, 1676s, 1462s cm^{-1}. ^{1}H NMR (300Hz, $DCCl_3$): δ = 0.30 (s, 3H, CH_3), 2.94 (s, 3H, CH_3), 3.01 (s, 1H, CH), 3.33-3.37 (m, 1H, CH_2CH_2), 4.03-4.07 (m, 1H, CH_2CH_2), 4.30-4.43 (m, 1H, CH_2CH_2), 4.86-4.91 (m, 1H, CH_2CH_2), 6.56-6.58 (d, J=7.2Hz, 2H, benzene-H), 7.00-7.32 (m, 14H, carbazole-H + benzene-H). 8.08-8.14 (d, J=7.8Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, $CDCl_3$): δ = 169.3, 151.6, 142.9, 140.2, 139.0, 128.3, 128.2, 128.1, 127.8, 126.2, 125.8, 125.2, 123.0, 121.1, 119.9, 108.6, 108.3, 90.6, 75.3, 63.8, 46.5, 40.4, 27.3, 22.4. TOFMS (EI) calcd for (M^+) $C_{33}H_{29}N_3O_3$: 515.2209, found 515.2175.

Model compound 2b. Potassium tert-butoxide (35 mg, 0.31 mmol) was added to a 4 mL anhydrous DMF solution dissolved with **16** (50 mg, 0.16 mmol) and stirred at room temperature for 30 min. Then 9-(3-bromopropy)-9H-carbazole (85 mg, 0.30 mmol) was added into, stirring was continued for 4 h. The reaction mixture was diluted with 80 ml CH_2Cl_2, washed with 80 mL water. Purified **2b** (15 mg, 19%) as white power was obtained via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:3) and following recrystallization from CH_2Cl_2 / petroleum ether. R_f= 0.65 (ethyl acetate / petroleum ether 2:1). m.p. 180–182°C. IR (KBr): nu(tilde) = 1720s, 1678s, 1473s, 1462s cm^{-1}. ^1H NMR (300Hz, $DCCl_3$): δ = 1.53 (s, 3H, CH_3), 2.20-2.24 (m, 2H, $CH_2CH_2CH_2$), 2.67 (m, 1H, $CH_2CH_2CH_2$), 2.94 (s, 3H, CH_3), 3.84-3.89 (m, 1H, $CH_2CH_2CH_2$), 4.21 (s, 1H, CH), 4.37-4.45 (m, 2H, $CH_2CH_2CH_2$), 6.70-6.73 (m, 2H, benzene-H), 6.94-7.54 (m, 14H, carbazole-H + benzene-H), 8.17-8.20 (d, J=7.5Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, $CDCl_3$): δ = 169.4, 151.6, 143.5, 140.2, 138.5, 128.4, 128.2, 128.0, 127.6, 126.0, 125.7, 124.7, 123.1, 120.7, 119.4, 108.6, 108.5, 91.3, 75.9, 64.1, 45.9, 40.2, 27.4, 27.0, 23.7. TOFMS (EI) calcd for (M^+) $C_{34}H_{31}N_3O_3$: 529.2365, found 529.2369.

Model compound 3. potassium tert-butoxide (35 mg, 0.31 mmol) and **16** (50 mg, 0.16 mmol) was added to anhydrous DMF of 4 mL and stirred at room temperature for 30 min by following addition of 1-(2-bromoethyl)-1H-indole (100 mg, 0.45 mmol), the reaction processed for another 4 h, and then diluted with 80 ml CH_2Cl_2, washed with 80 mL water. Solvent was removed and purified **3** (20 mg, 27%) as white power was obtained via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:3) and following recrystallization from CH_2Cl_2 / petroleum ether. R_f= 0.54 (ethyl acetate / petroleum ether 2:1). m.p. 159-161°C. IR (KBr): nu(tilde) = 1709s, 1673s, 1469s cm^{-1}. ^1H NMR (300MHz, $CDCl_3$): δ = 0.87 (s, 3H, CH_3), 2.93 (s, 3H, CH_3), 3.15-3.21 (m, 1H, CH_2CH_2), 3.25 (s, 1H, CH), 4.07-4.11 (m, 1H, CH_2CH_2), 4.25-4.30 (m, 1H, CH_2CH_2), 4.50-4.58 (m, 1H, CH_2CH_2) 6.58-6.59 (d, J=3Hz, 1H, benzene-H), 6.88-6.91 (m, 2H, indole-H), 7.09-7.32 (m, 12H, benzene-H + indole-H) 7.64 (d, J=8.1Hz, 1H, indole-H); ^{13}C NMR (100MHz, $CDCl_3$): δ = 169.6, 151.5, 143.1, 138.9, 136.3, 128.8, 128.5, 128.2, 128.1, 128.0, 127.4, 125.8, 125.4, 122.3, 121.8, 120.2, 108.9, 102.8, 90.9, 75.8, 64.4, 48.9 , 44.1, 27.3, 23.0. TOFMS (EI) calcd for (M^+) $C_{29}H_{27}N_3O_3$: 465.2052, found 465.2028.

Photolysis product 4a. 1a (25 mg, 0.049 mmol) was dissolved in methanol of 30 mL and irradiated with a Xe lamp for 2 h, **4a** (16 mg, 64%) was isolated via column chromatography (silica gel-H, ethyl acetate / petroleum ether 2:1) as white powder. R_f= 0.51 (ethyl acetate / petroleum ether 2:1). m.p. 201-203°C. IR (KBr): nu(tilde) = 2953w, 1693s, 1667s, 1640s, 1463s cm^{-1}. ^1H NMR (300Hz, $DCCl_3$): δ = 1.23-1.33 (m, 6H, CH_3, CH_2CH_3), 1.93 (s, 3H, CH_3), 1.93-2.07 (m, 2H, $CH_2CH_2CH_2$), 3.74-3.82 (m, 2H, CH_2CH_3), 4.00-4.13 (m, 6H, CH_2CH_2, $CH_2CH_2CH_2$), 4.62-4.66 (m, 2H, $CH_2CH_2CH_2$), 5.66 (s, 1H, CH), 6.97 (d, 1H, CH), 7.20-7.46 (m, 6H, carbazole-H), 8.06-8.08 (d, J=7.8Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, d_6-DMSO): δ = 163.0, 162.6, 150.8, 150.2, 139.9, 139.8, 139.7, 125.7, 122.2, 120.2, 119.0, 108.9, 107.7, 107.4, 47.2, 43.6, 40.5, 38.3, 25.4, 14.0, 12.6, 12.2. TOFMS (EI) calcd for (M^+) $C_{29}H_{31}N_5O_4$: 513.2376, found 513.2341.

Photolysis product 4b. 1b (25 mg, 0.047 mmol) was dissolved in methanol of 30 mL and irradiated with a Xe lamp for 2 h, **4b** (20 mg, 80%) was isolated via column chromatography (silica gel-H, ethyl acetate / petroleum ether 2:1) as white powder. IR (KBr): nu(tilde) = 2957w, 2925w, 1697s, 1667s, 1655s, 1464s cm^{-1}. R_f= 0.55 (ethyl acetate / petroleum ether 2:1). m.p. 170-172°C. ^1H NMR (300Hz, $DCCl_3$): δ = 1.24-1.26 (m, 3H, CH_2CH_3), 1.74 (s, 3H, CH_3), 1.88 (s, 3H, CH_3), 1.95-2.00 (m, 2H, $CH_2CH_2CH_2$), 2.31-2.36 (m, 2H, $CH_2CH_2CH_2$), 3.67-3.74 (m, 4H, $CH_2CH_2CH_2$), 3.98-4.04 (m, 4H, CH_2CH_3, $CH_2CH_2CH_2$), 4.40-4.44 (m, 2H, $CH_2CH_2CH_2$), 6.55 (s, 1H, CH), 6.91 (s, 1H, CH), 7.21-7.48 (m, 6H, carbazole-H), 8.08-8.11 (d, J=7.8Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, $DCCl_3$): δ = 163.7, 163.5, 151.4, 151.1, 140.1, 138.0, 137.9, 125.9, 123.0, 120.5, 119.2, 109.8, 108.5, 47.7, 44.4, 40.3, 39.3, 39.2, 28.0, 26.4, 14.3, 13.1, 12.9. TOFMS (EI) calcd for (M^+) $C_{30}H_{33}N_5O_4$: 527.2533, found 527.2482.

Photolysis product 5a. 2a (25 mg, 0.049 mmol) was dissolved in methanol of 30 mL and irradiated with a Xe lamp for 2 h, **5a** (10 mg, 63%) was isolated via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:1) as white powder. R_f= 0.65 (ethyl acetate / petroleum ether 2:1).

m.p. 209-211°C. IR (KBr): nu(tilde) = 2942w, 2924w, 1701s, 1666s, 1634s, 1464s cm^{-1}.^{1}H NMR (300Hz, $DCCl_3$): δ = 1.30 (d, J=0.9Hz, 3H, CH_3), 3.33 (s, 3H, NCH_3), 4.07-4.11 (m, 2H, CH_2CH_2), 4.63-4.67 (m, 2H, CH_2CH_2), 5.69-5.70 (d, J=0.9Hz, 1H, CH), 7.19-7.44 (m, 6H, carbazole-H), 8.05-8.07 (d, J=7.8Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, $DCCl_3$): δ = 163.5, 151.7, 140.1, 137.6, 126.1, 123.2, 120.6, 119.7, 109.4, 108.1, 48.2, 40.7, 27.8, 12.2. TOFMS (EI) calcd for (M^+) $C_{20}H_{19}N_3O_2$: 333.1477, found 333.1422.

Photolysis product 5b. 2b (25 mg, 0.047 mmol) was dissolved in methanol of 30 mL and irradiated with a Xe lamp for 2 h, **5b** (11 mg, 69%) was isolated via column chromatography (silica gel-H, ethyl acetate / petroleum ether 1:1) as white powder. R_f= 0.66 (ethyl acetate / petroleum ether 2:1). m.p. 189-190°C. IR (KBr): nu(tilde) = 2952w, 2924w, 1696s, 1666s, 1633s, 1469s cm^{-1}. ^{1}H NMR (300Hz, $DCCl_3$): δ = 1.72 (d, J=0.6Hz, 3H, CH_3), 3.29 (s, 3H, NCH_3), 4.40-4.44 (m, 2H, $CH_2CH_2CH_2$), 3.70-3.75 (m, 2H, $CH_2CH_2CH_2$), 2.30-2.39 (m, 2H, $CH_2CH_2CH_2$), 6.53 (d, J=0.6Hz, 1H, CH), 7.21-7.49 (m, 6H, carbazole-H), 8.08-8.10 (d, J=7.8Hz, 2H, carbazole-H). ^{13}C NMR (100MHz, d_6-DMSO): δ = 163.2, 150.9, 139.8, 139.4, 125.6, 122.2, 118.8, 109.1, 117.5, 46.6, 39.8, 27.6, 27.4, 12.5. TOFMS (EI) calcd for (M^+) $C_{21}H_{21}N_3O_2$: 347.1634, found 347.1607.

References

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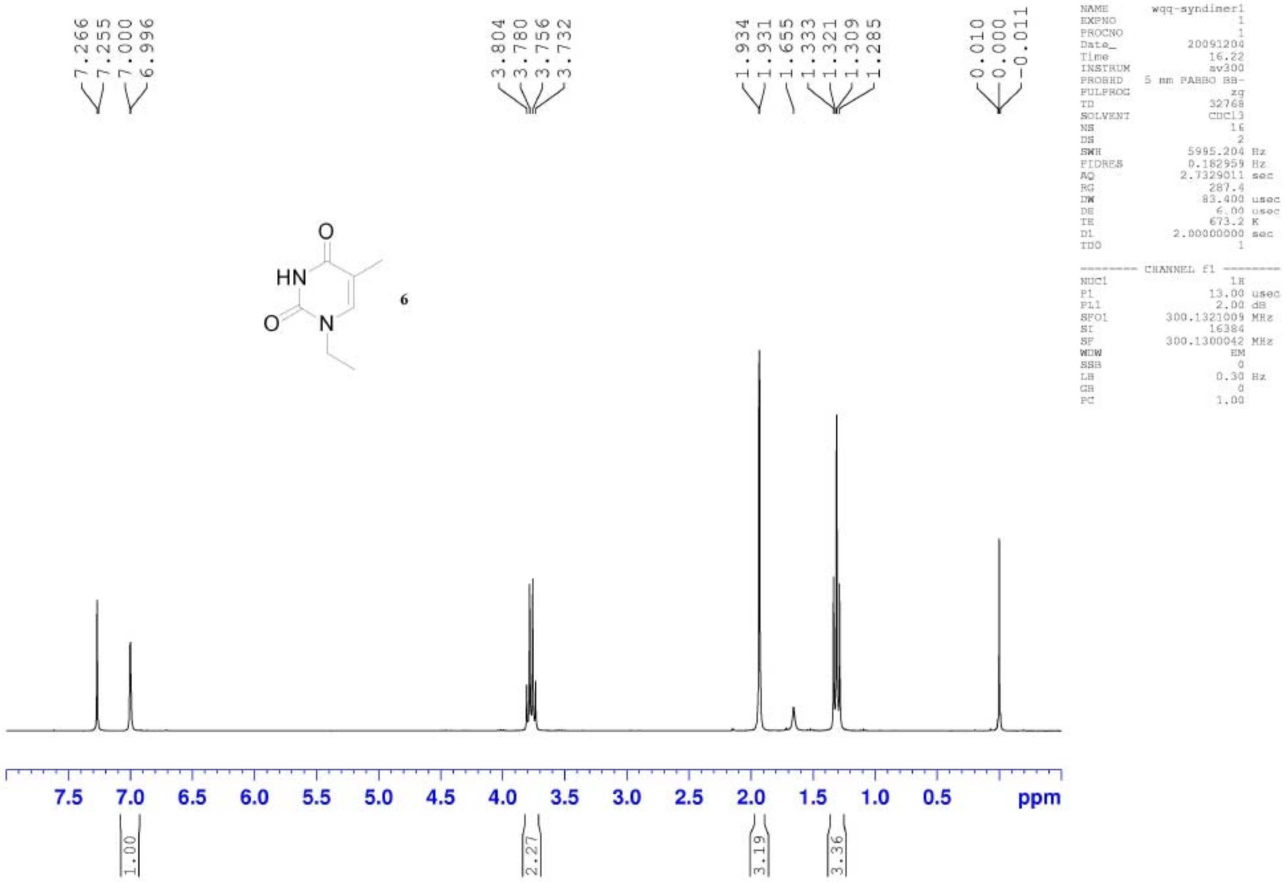

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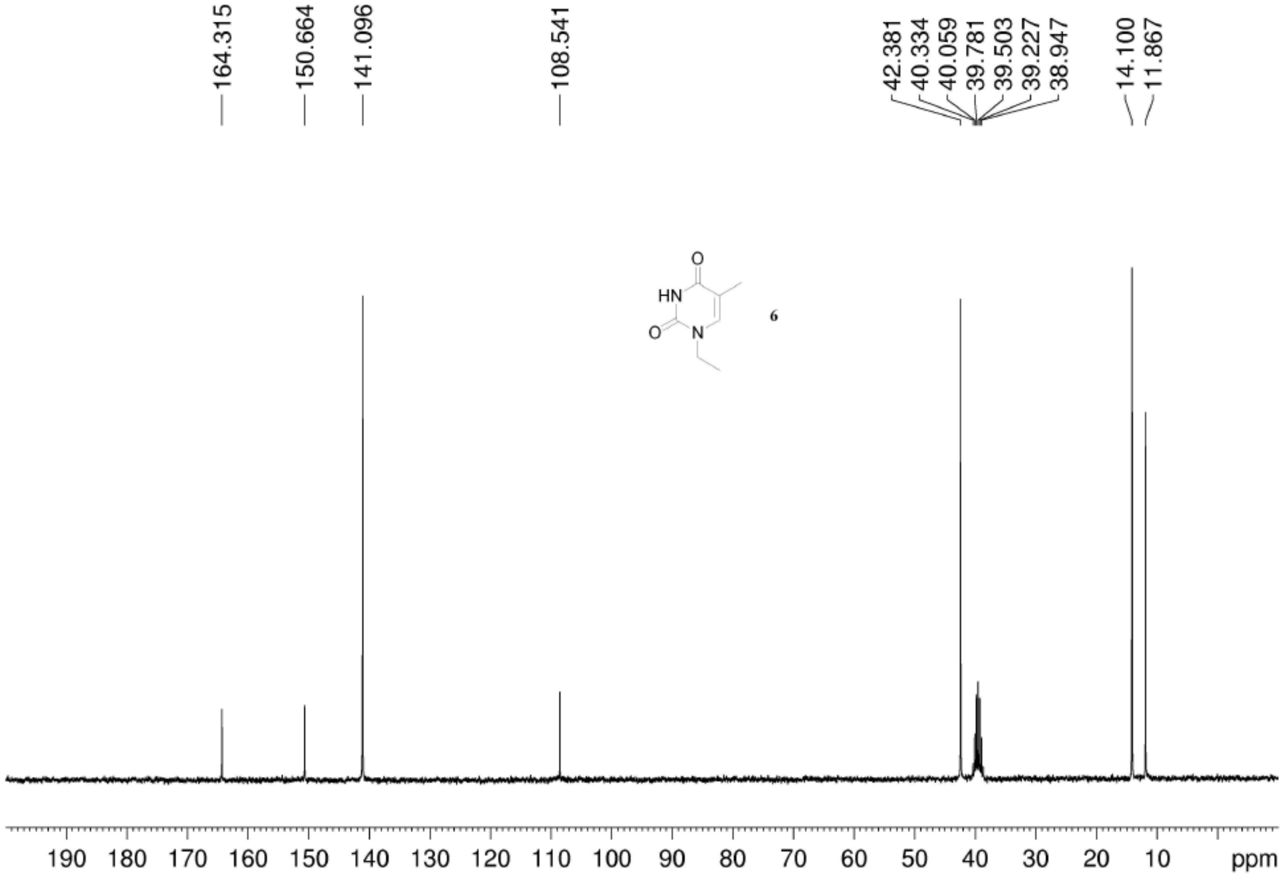

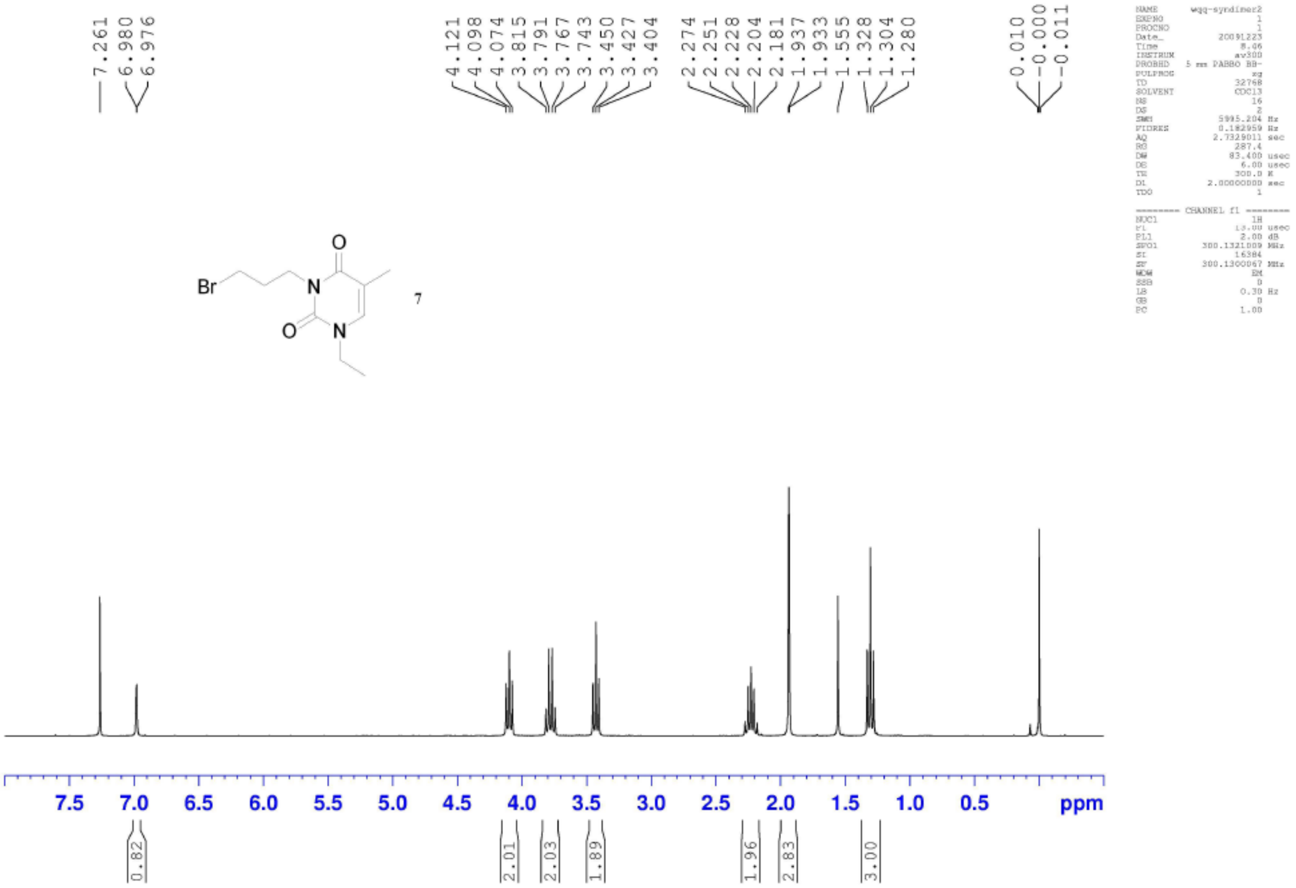
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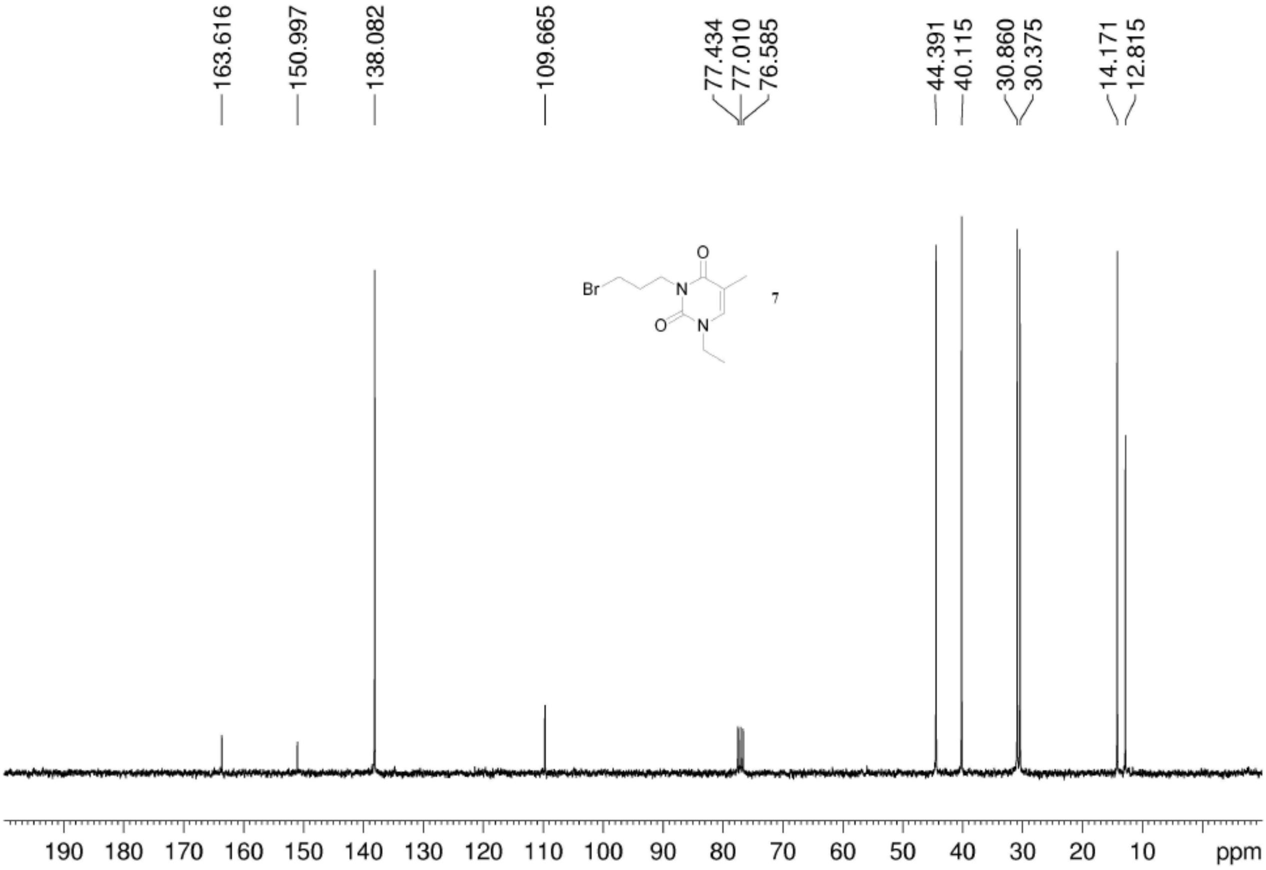
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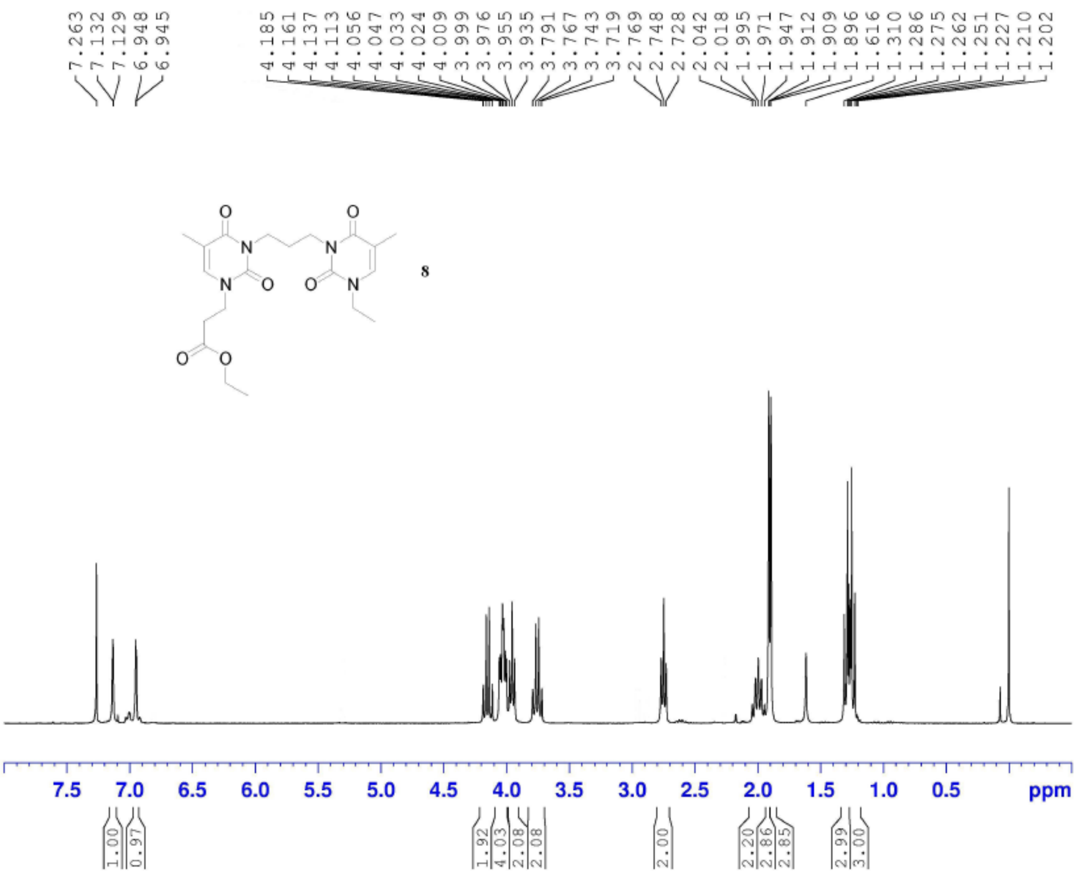

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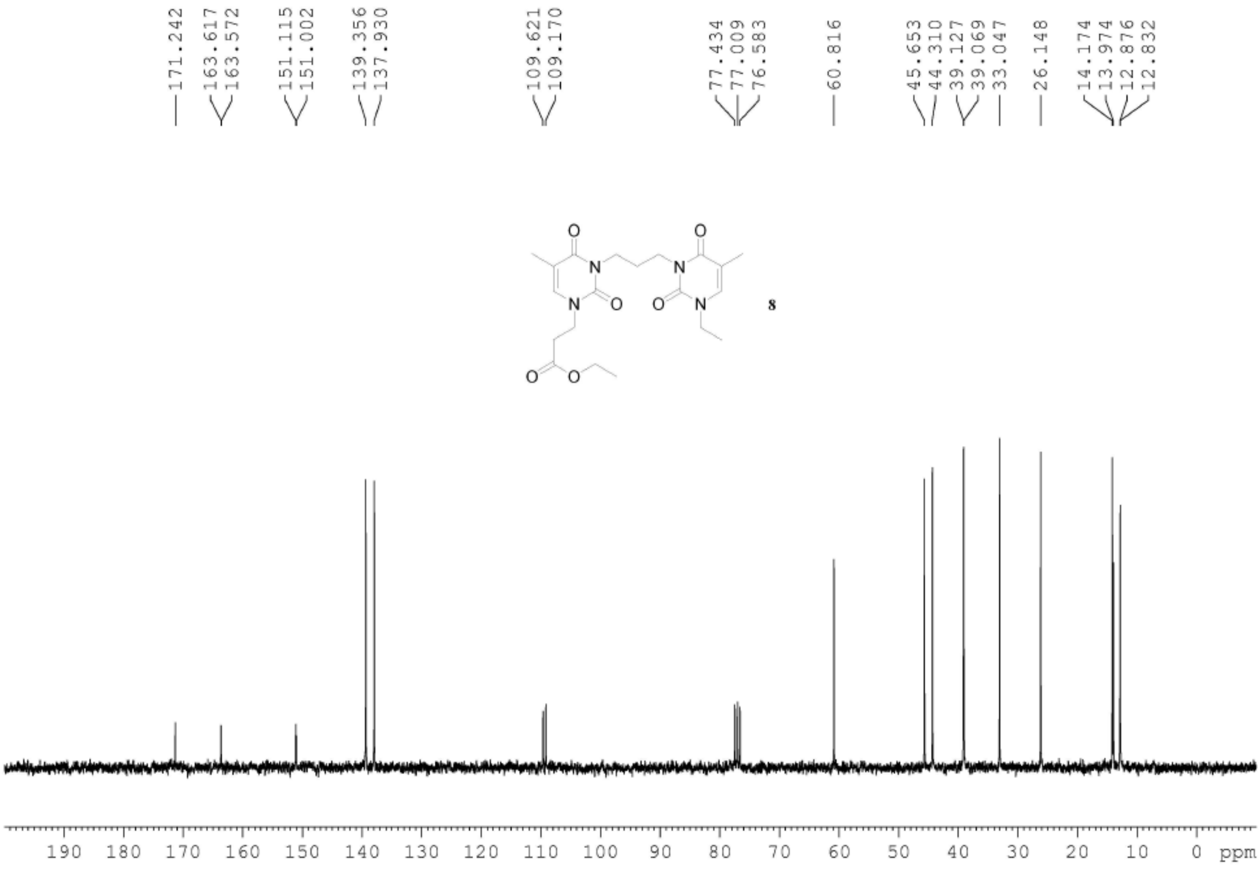

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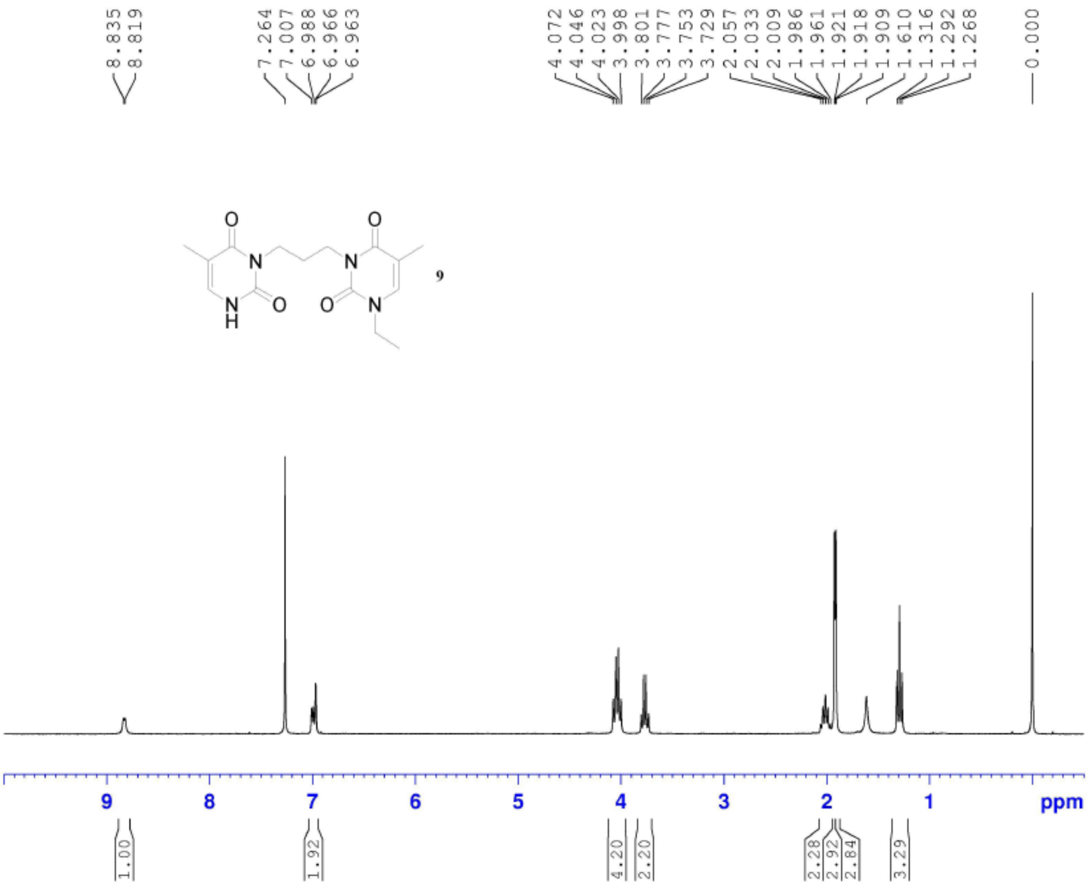

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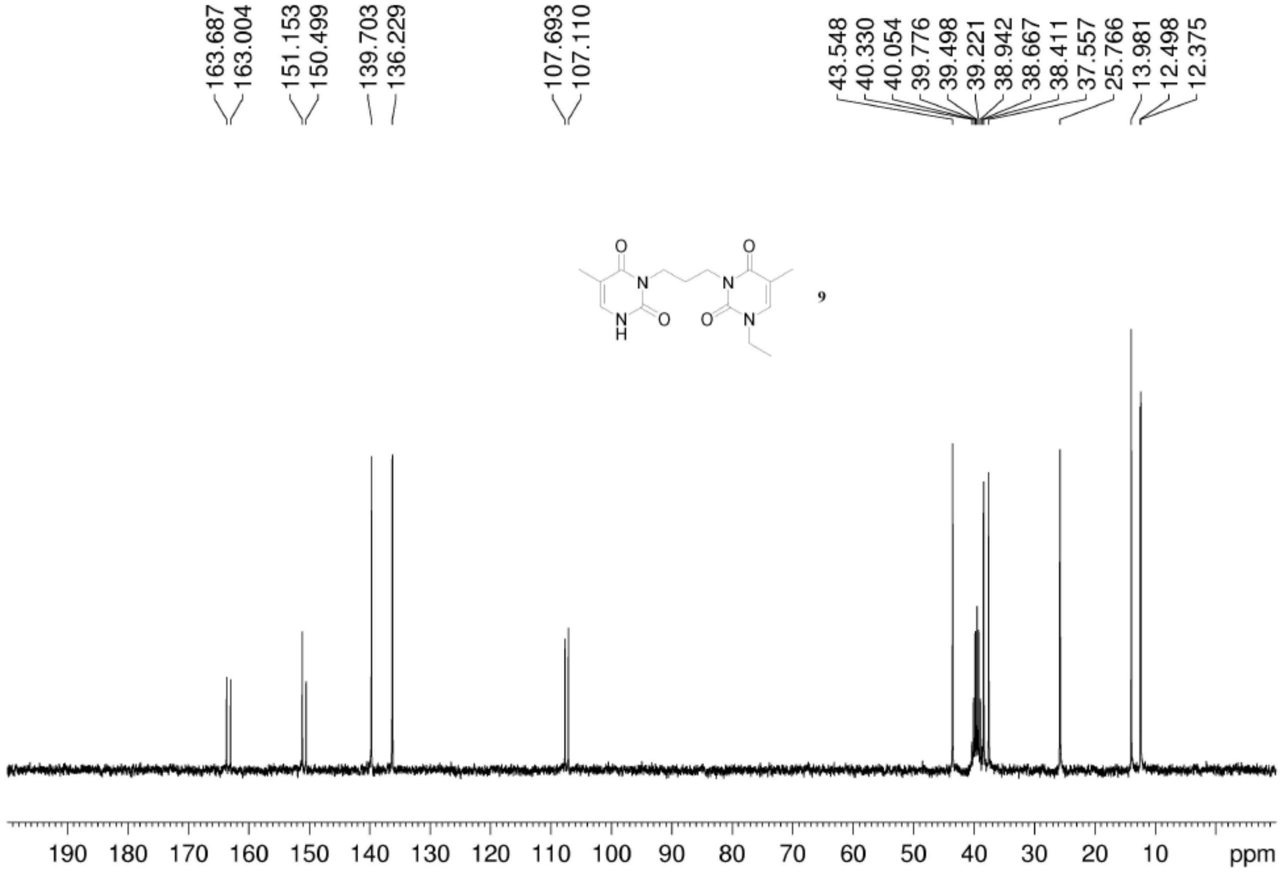

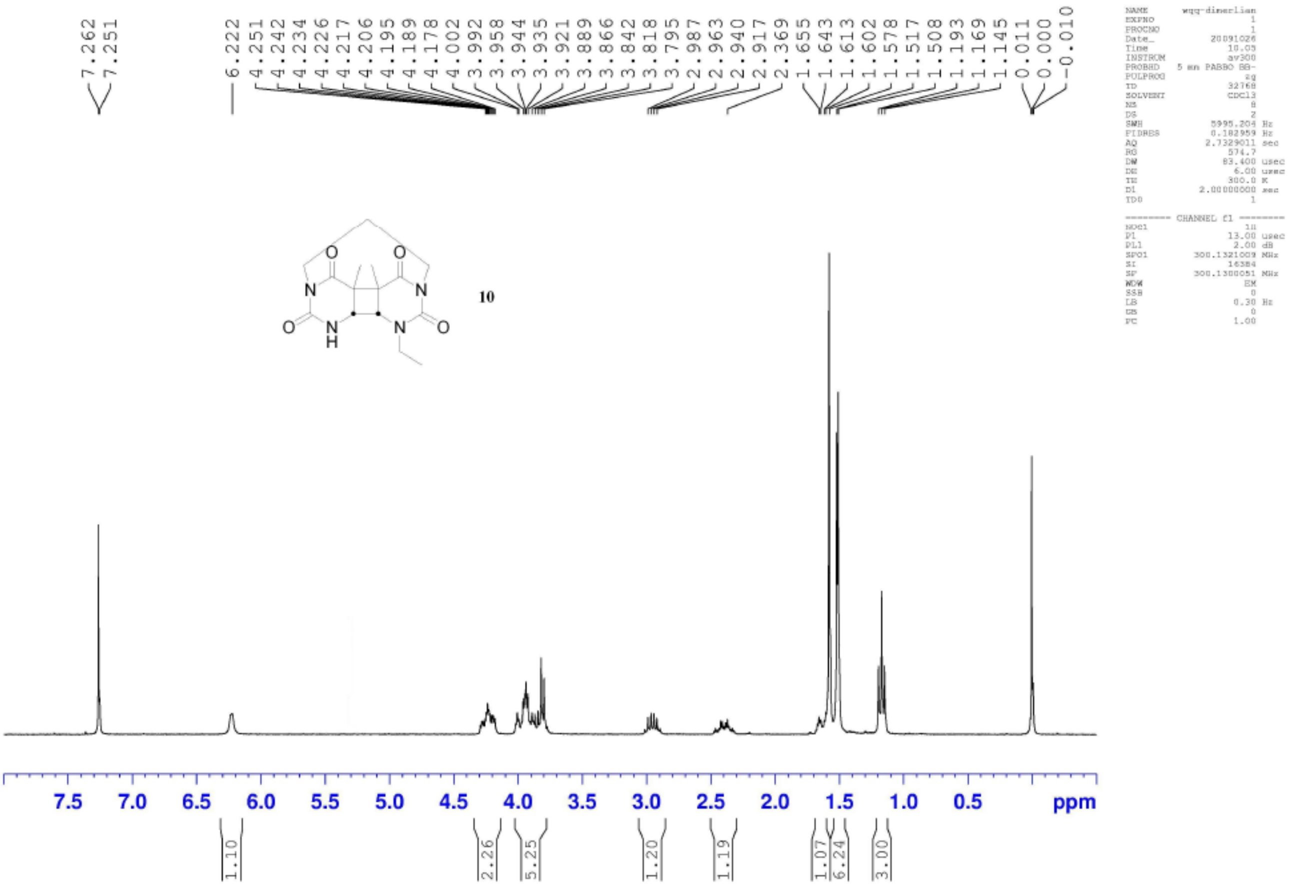
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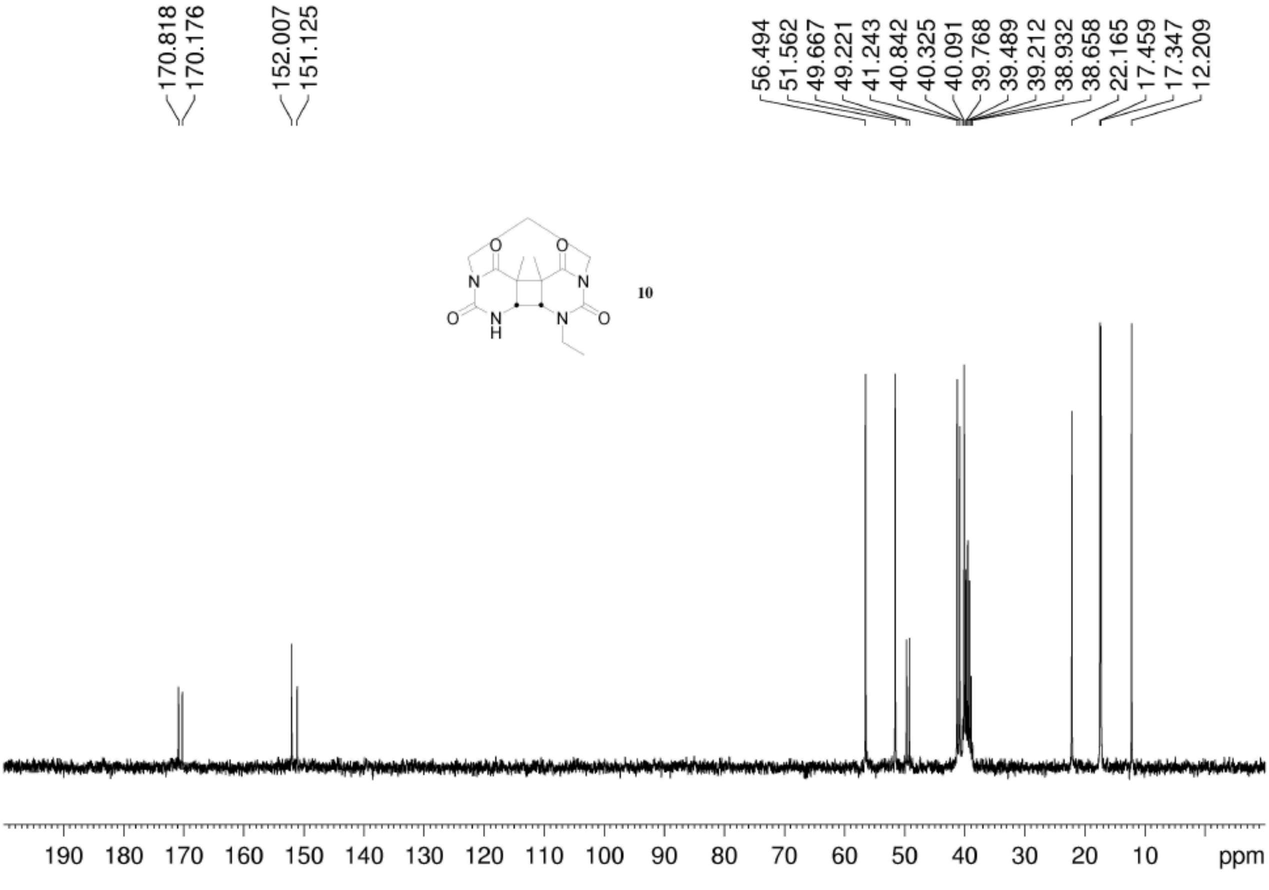
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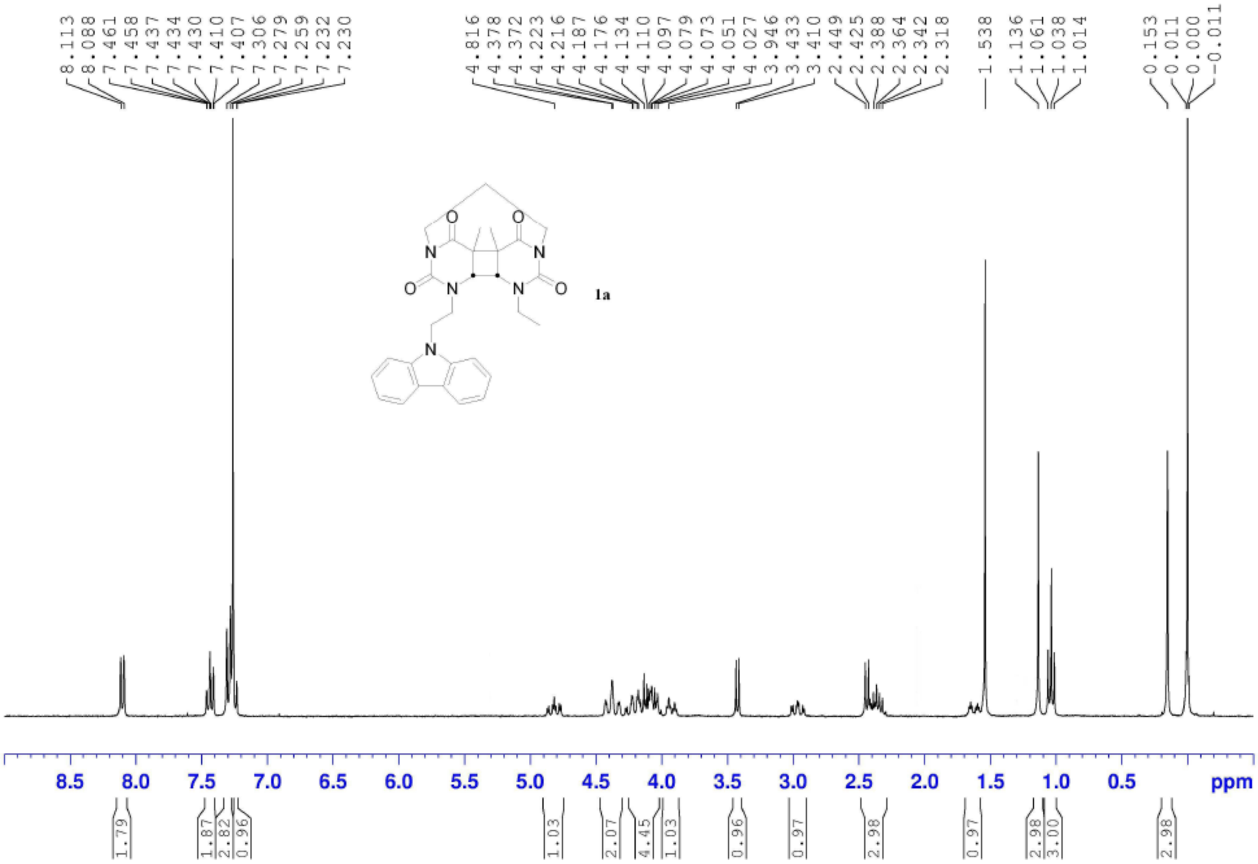
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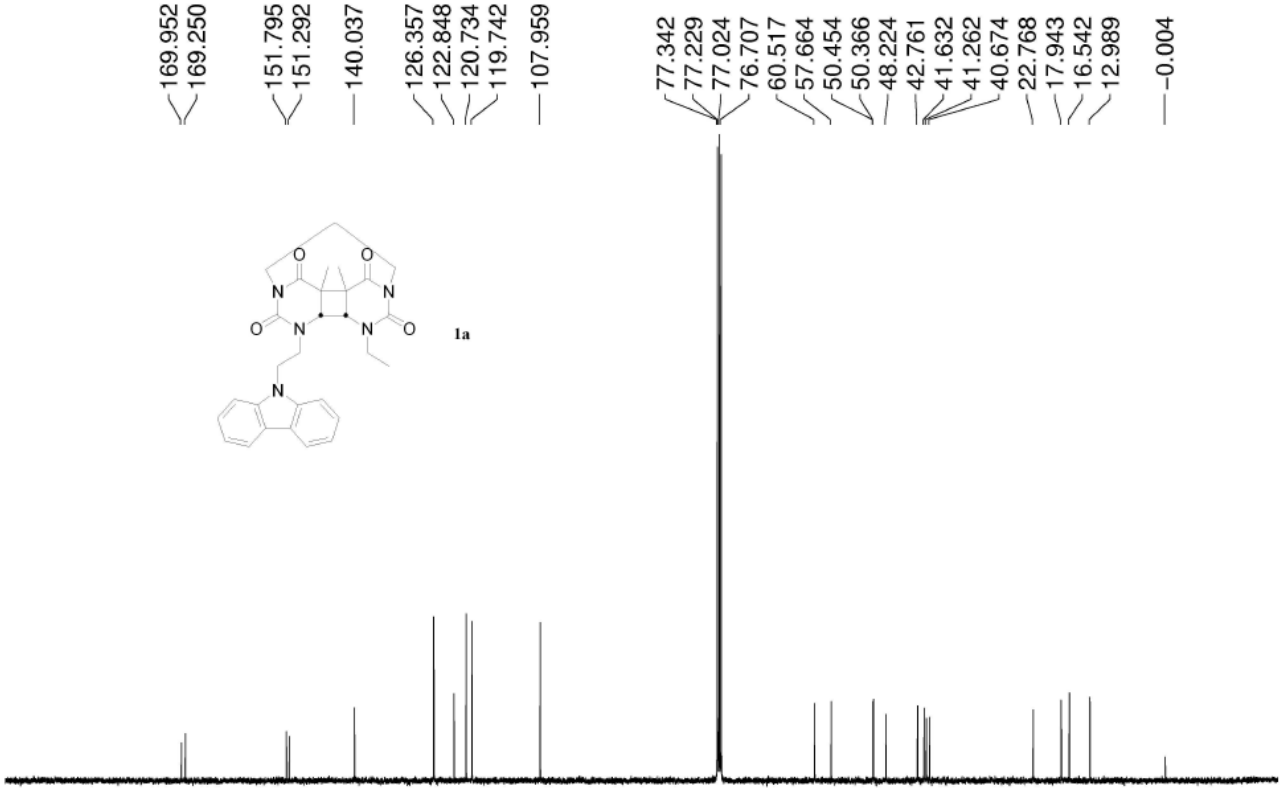
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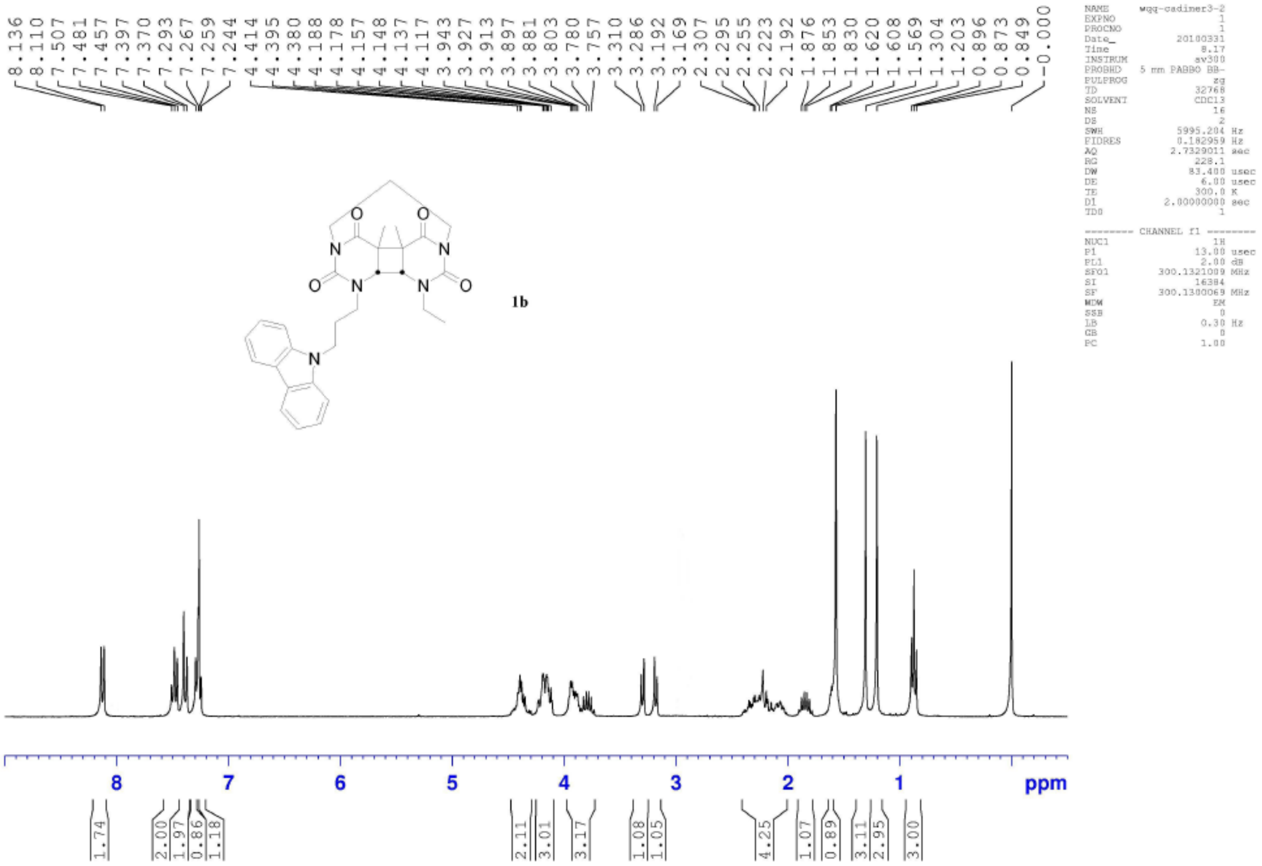
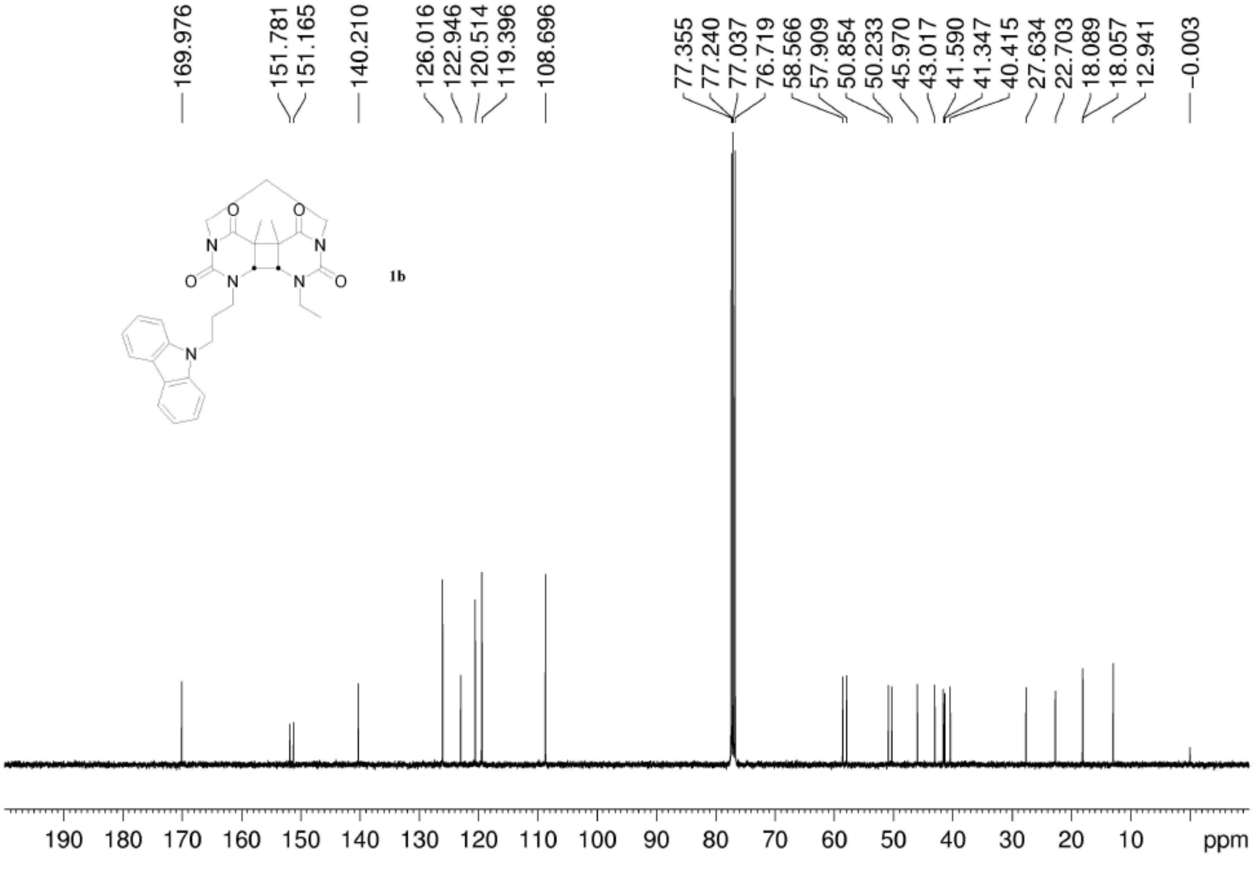
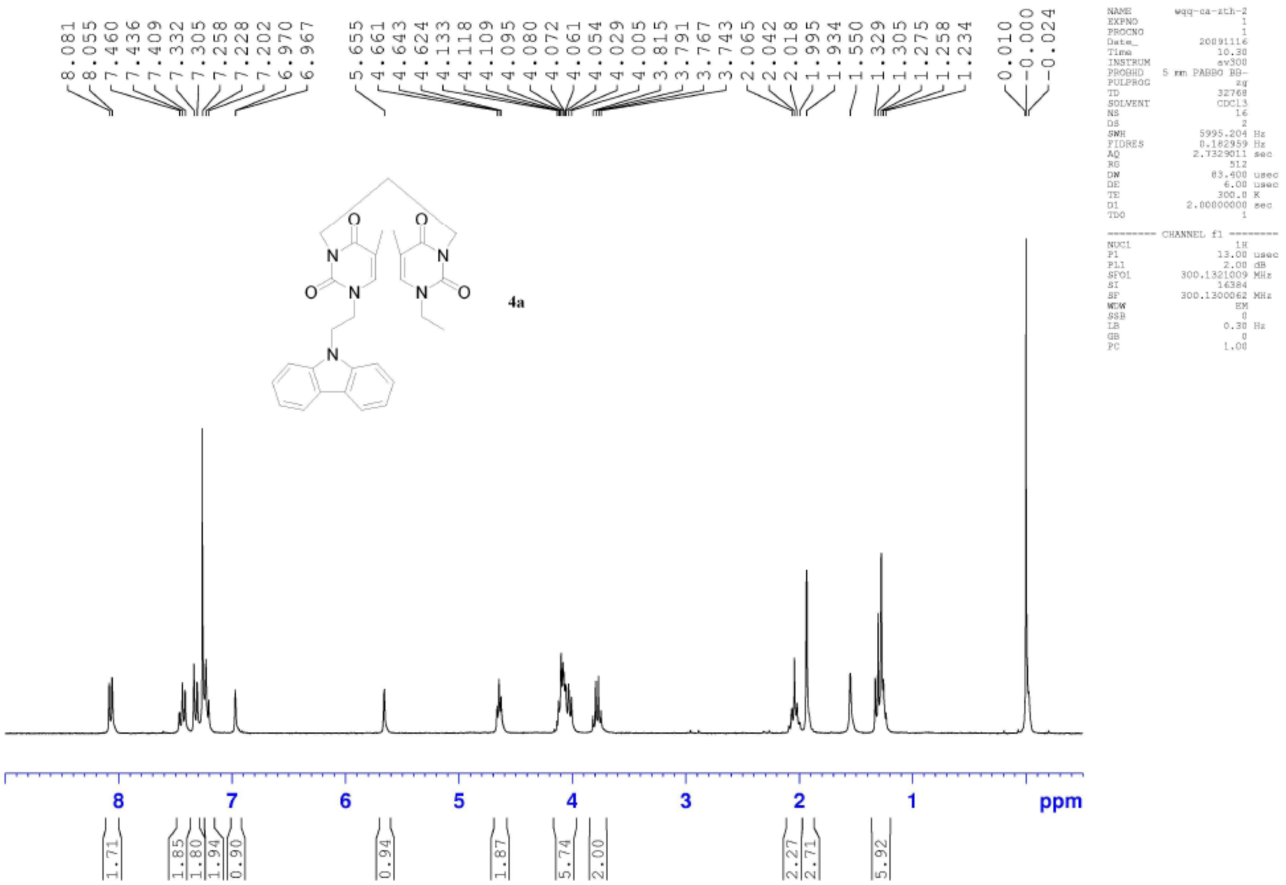
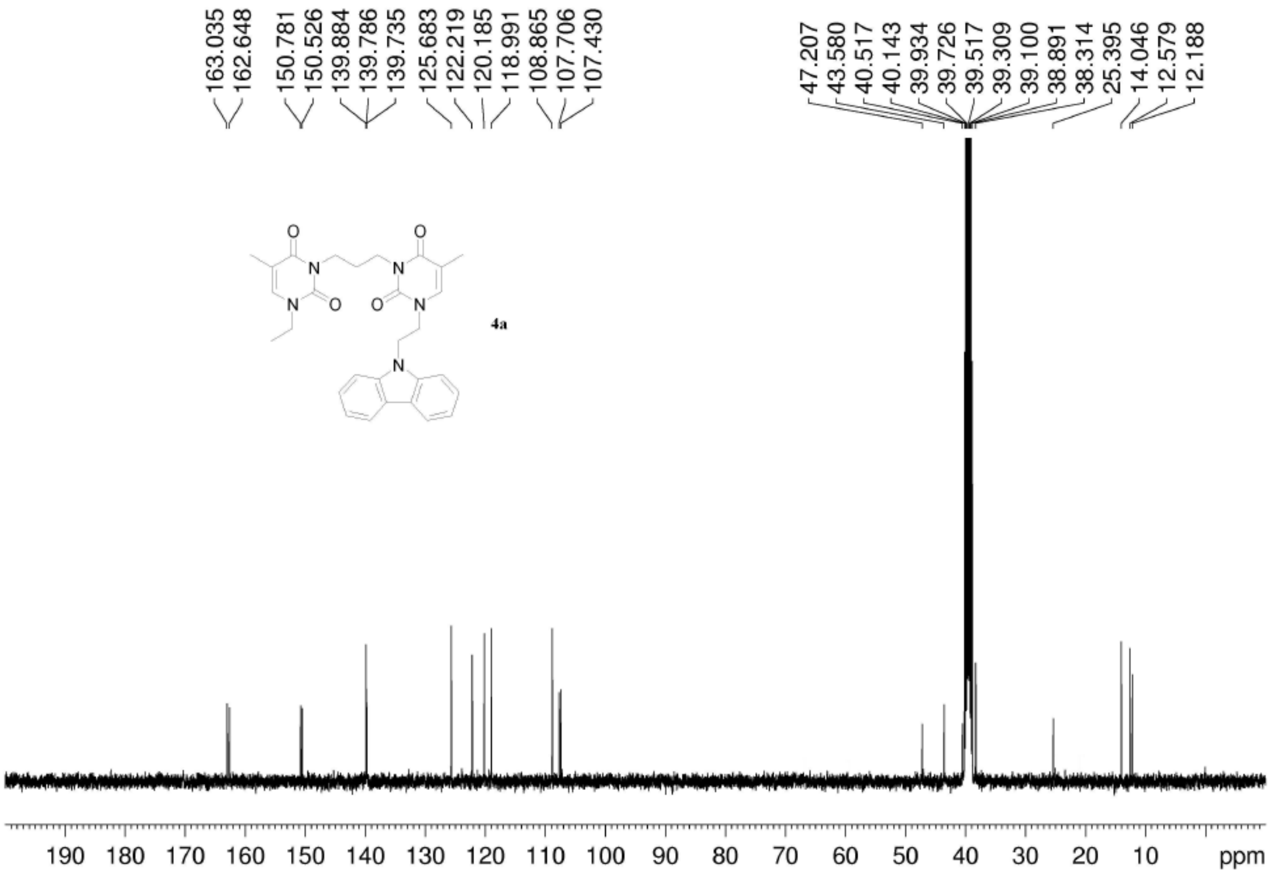
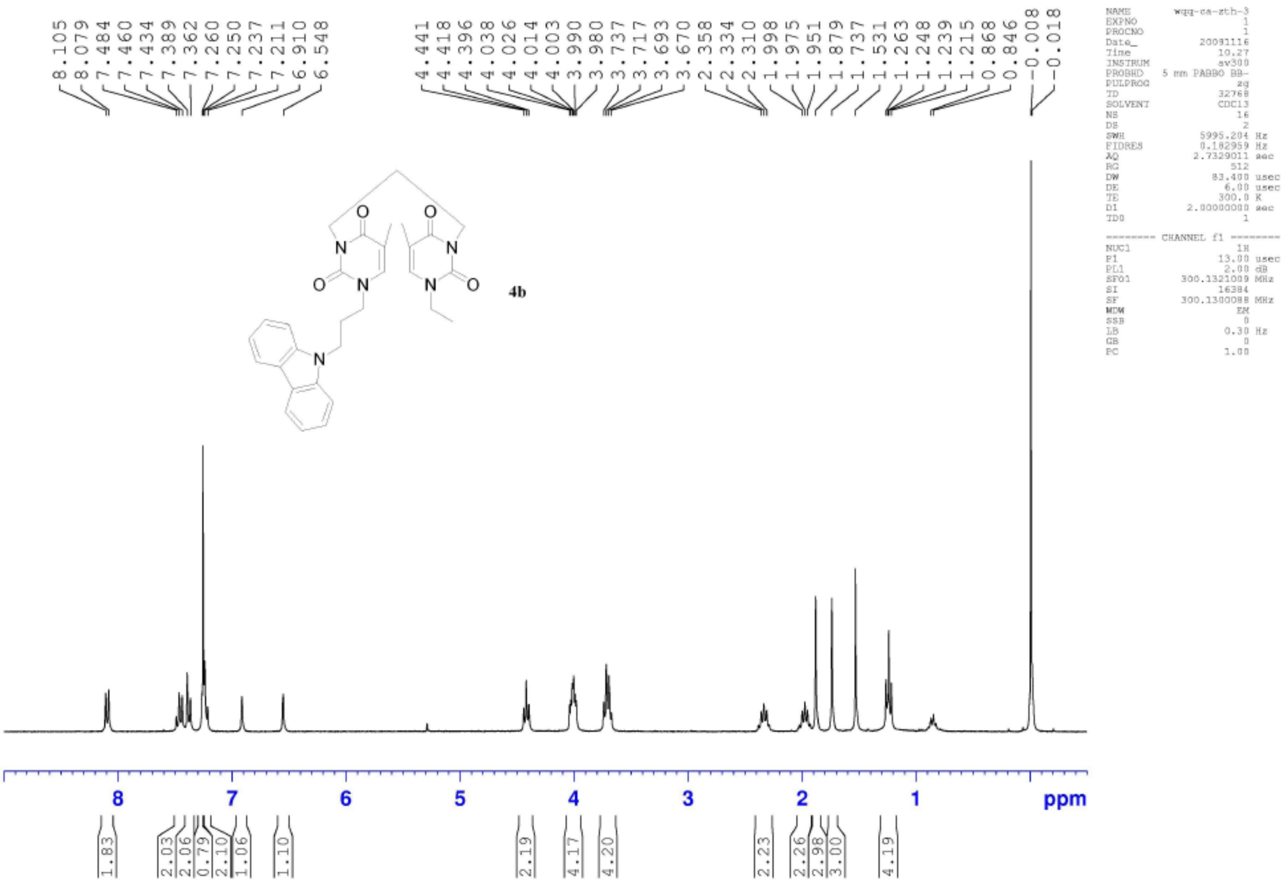
151.795
151.292
140.037
126.357
122.848
120.734
119.742
107.959
77.342
77.229
77.024
76.707
60.517
57.664
50.454
50.366
48.224
42.761
41.632
41.262
40.674
22.768
17.943
16.542
12.989
-0.004
1a
180
160
140
120
100
80
60
40
20
0
ppm


8.136
8.110
7.507
7.481
7.457
7.397
7.370
7.293
7.267
7.259
7.244
4.414
4.395
4.380
4.188
4.178
4.157
4.148
4.137
4.117
3.943
3.927
3.913
3.897
3.881
3.803
3.780
3.757
3.310
3.286
3.192
3.169
2.307
2.295
2.255
2.223
2.192
1.876
1.853
1.830
1.620
1.608
1.569
1.304
1.203
0.896
0.873
0.849
-0.000
NAME wqq-cadimer3-2
EXPNO 1
PROCNO 1
Date_ 20100331
Time 8.17
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 228.1
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1321009 MHz
SI 16384
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1b
8
7
6
5
4
3
2
1
ppm
1.74
2.00
1.97
0.86
1.18
2.11
3.01
3.17
1.08
1.05
4.25
1.07
0.89
3.11
2.95
3.00


169.976
151.781
151.165
140.210
126.016
122.946
120.514
119.396
108.696
77.355
77.240
77.037
76.719
58.566
57.909
50.854
50.233
45.970
43.017
41.590
41.347
40.415
27.634
22.703
18.089
18.057
12.941
-0.003
1b
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

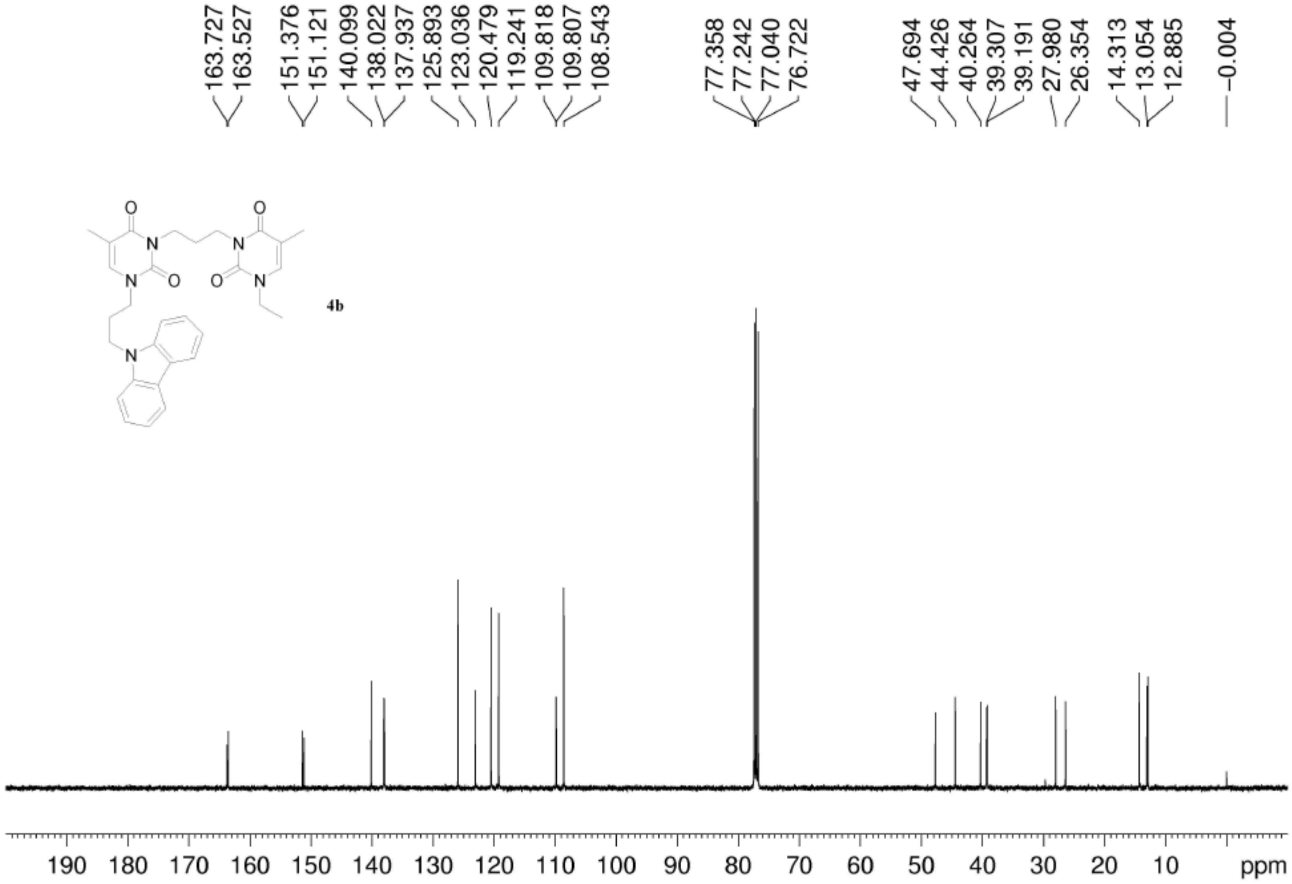

8.081
8.055
7.460
7.436
7.409
7.332
7.305
7.258
7.228
7.202
6.970
6.967
5.655
4.661
4.643
4.624
4.133
4.118
4.109
4.095
4.080
4.072
4.061
4.054
4.029
4.005
3.815
3.791
3.767
3.743
2.065
2.042
2.018
1.995
1.934
1.550
1.329
1.305
1.275
1.258
1.234
0.010
-0.000
-0.024
NAME wqq-ca-zth-2
EXPNO 1
PROCNO 1
Date_ 20091116
Time 10.30
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1321009 MHz
SI 16384
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
N
O
N
O
N
O
N
O
N
4a
8
7
6
5
4
3
2
1
ppm
1.71
1.85
1.80
1.94
0.90
0.94
1.87
5.74
2.00
2.27
2.71
5.92


163.035
162.648
150.781
150.526
139.884
139.786
139.735
125.683
122.219
120.185
118.991
108.865
107.706
107.430
47.207
43.580
40.517
40.143
39.934
39.726
39.517
39.309
39.100
38.891
38.314
25.395
14.046
12.579
12.188
O
N
O
N
O
O
N
N
N
4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



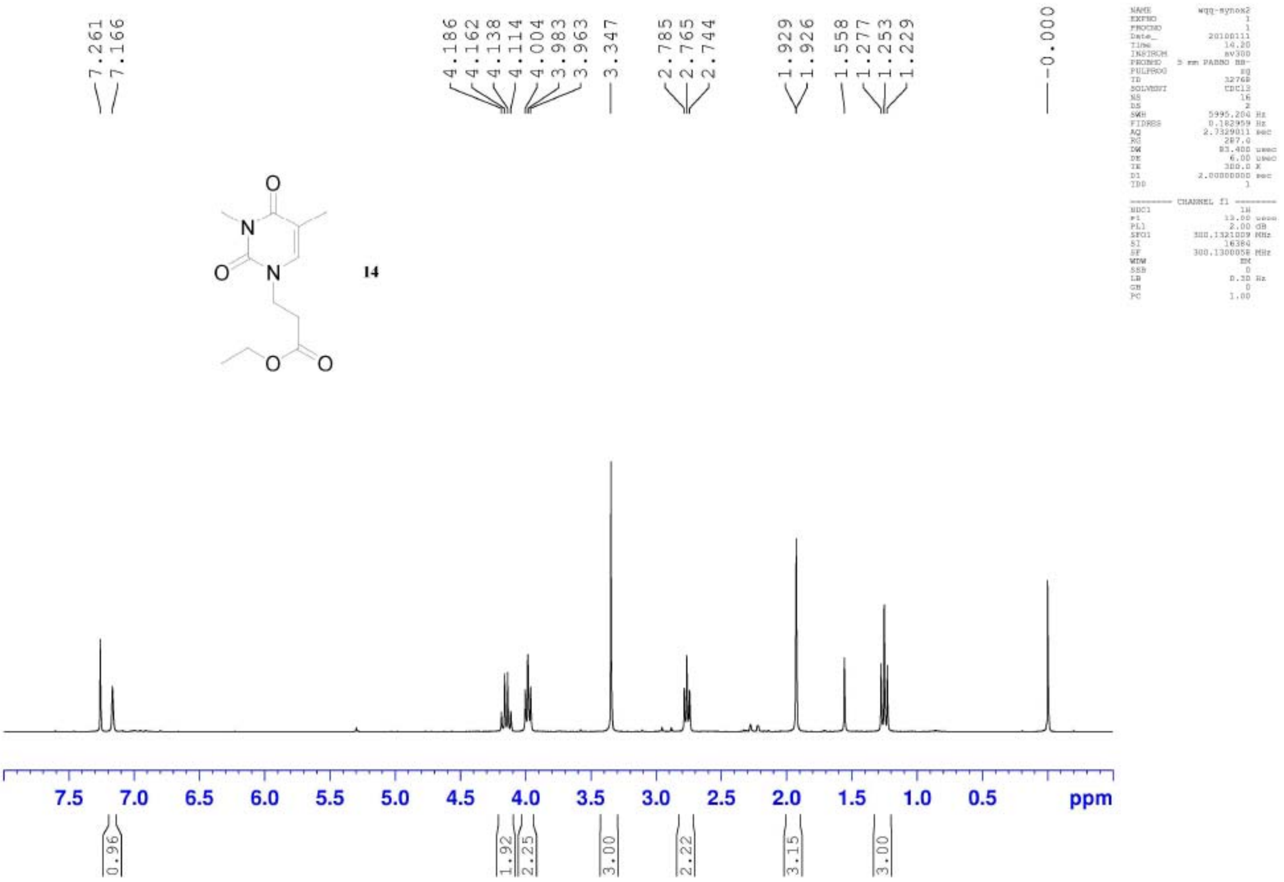
4b

8.105
8.079
7.484
7.460
7.434
7.389
7.362
7.260
7.250
7.237
7.211
6.910
6.548
4.441
4.418
4.396
4.038
4.026
4.014
4.003
3.990
3.980
3.737
3.717
3.693
3.670
2.358
2.334
2.310
1.998
1.975
1.951
1.879
1.737
1.531
1.263
1.248
1.239
1.215
0.868
0.846
-0.008
-0.018
NAME wqq-ca-zth-3
EXPNO 1
PROCNO 1
Date_ 20091116
Time 10.27
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1321009 MHz
SI 16384
SF 300.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
O
N
O
O
N
N
O
N
8
7
6
5
4
3
2
1
ppm
1.83
2.03
2.06
0.79
2.10
1.06
1.10
2.19
4.17
4.20
2.23
2.26
2.98
3.00
4.19

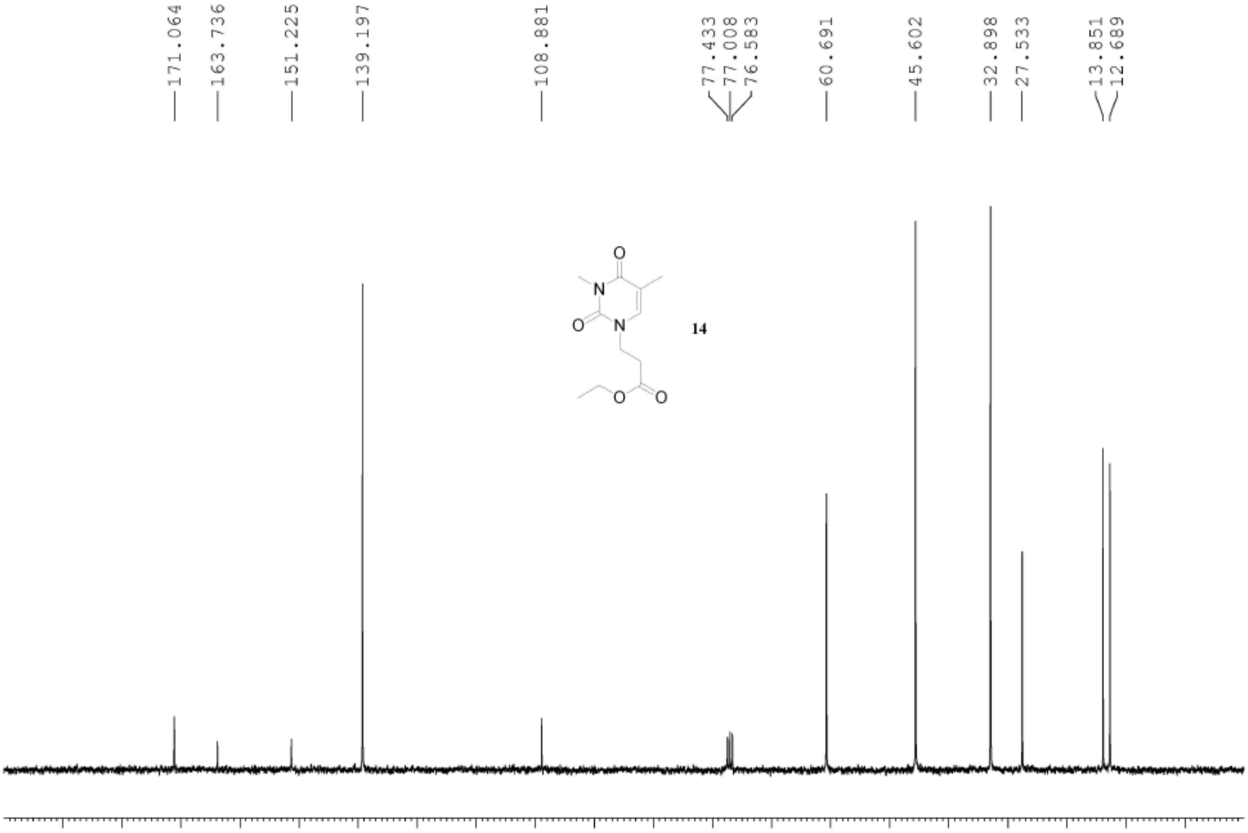


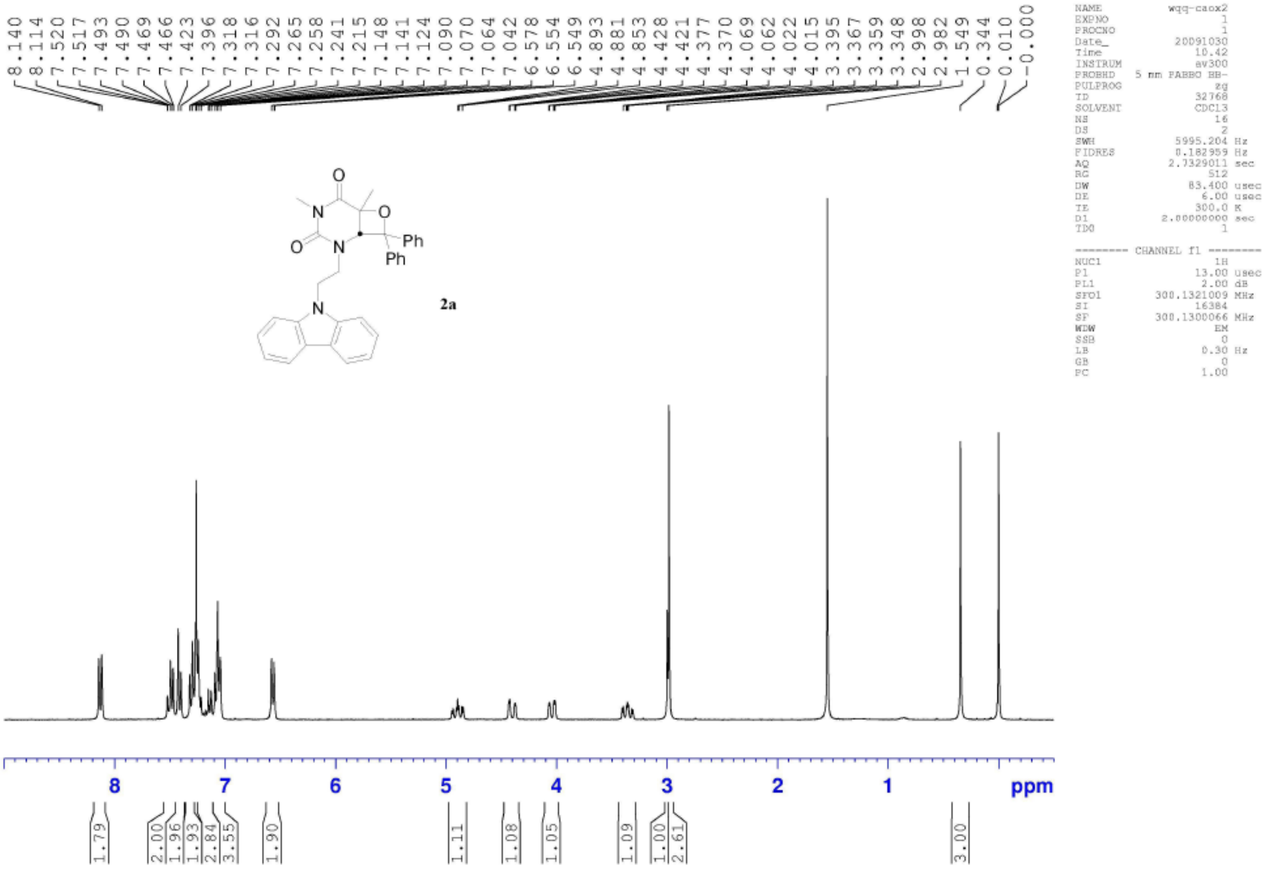
4b

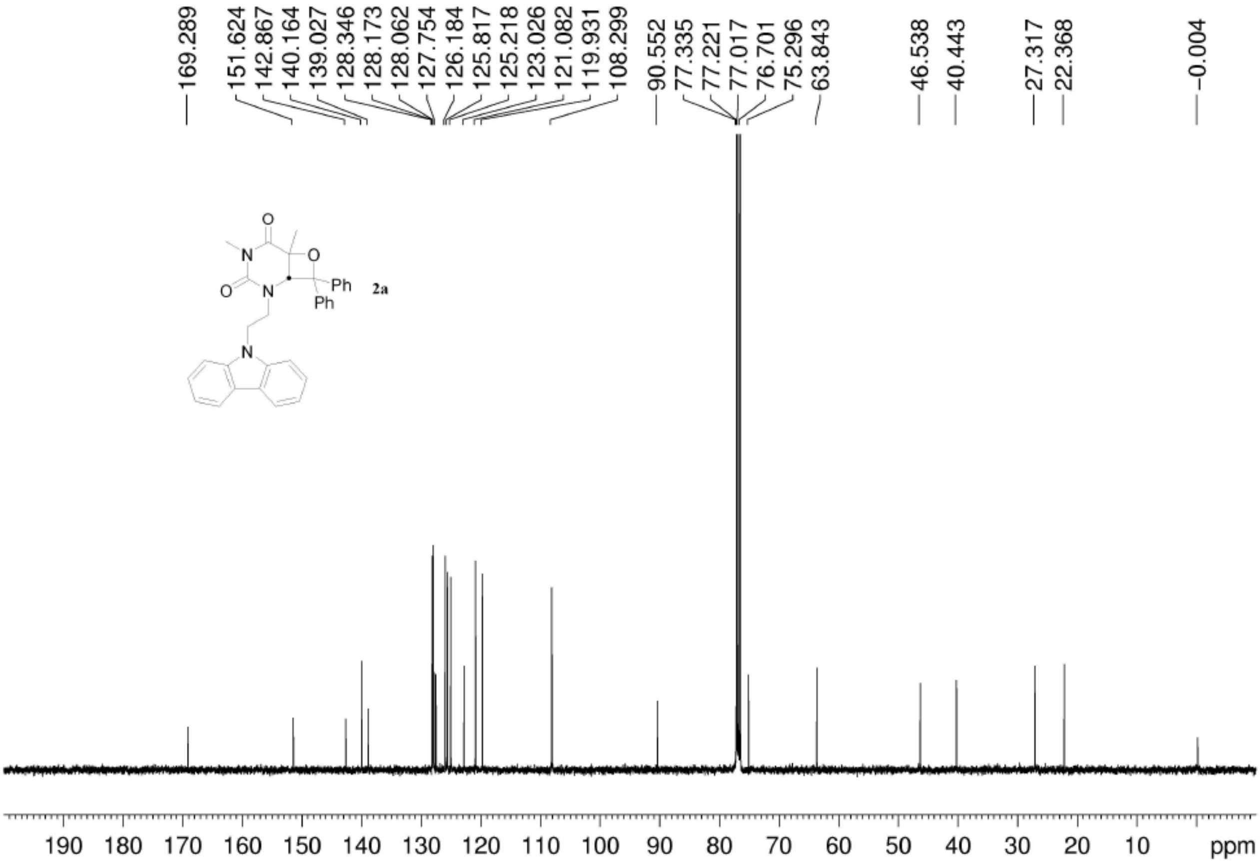
163.727
163.527
151.376
151.121
140.099
138.022
137.937
125.893
123.036
120.479
119.241
109.818
109.807
108.543
77.358
77.242
77.040
76.722
47.694
44.426
40.264
39.307
39.191
27.980
26.354
14.313
13.054
12.885
-0.004
O
N
O
N
O
O
N
N
O
N
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

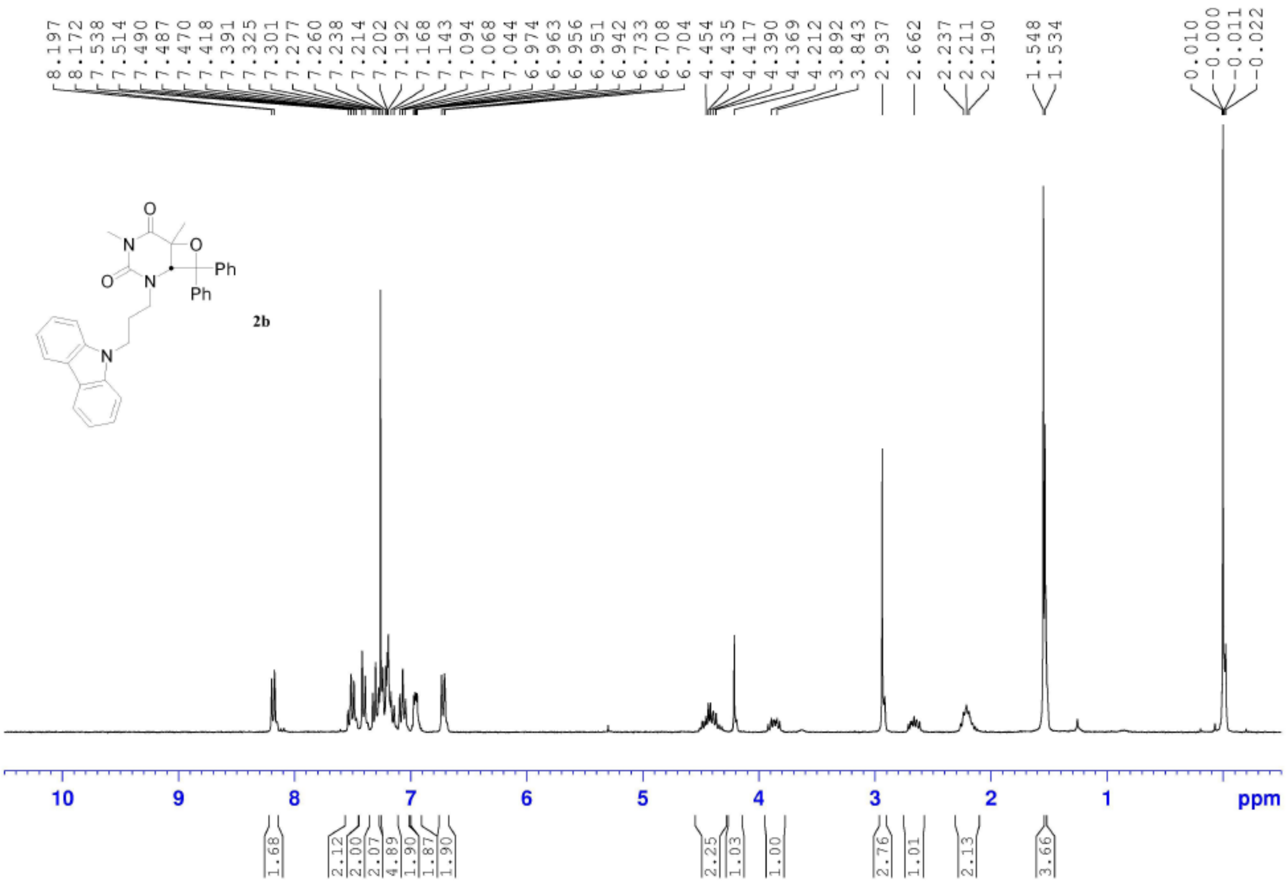


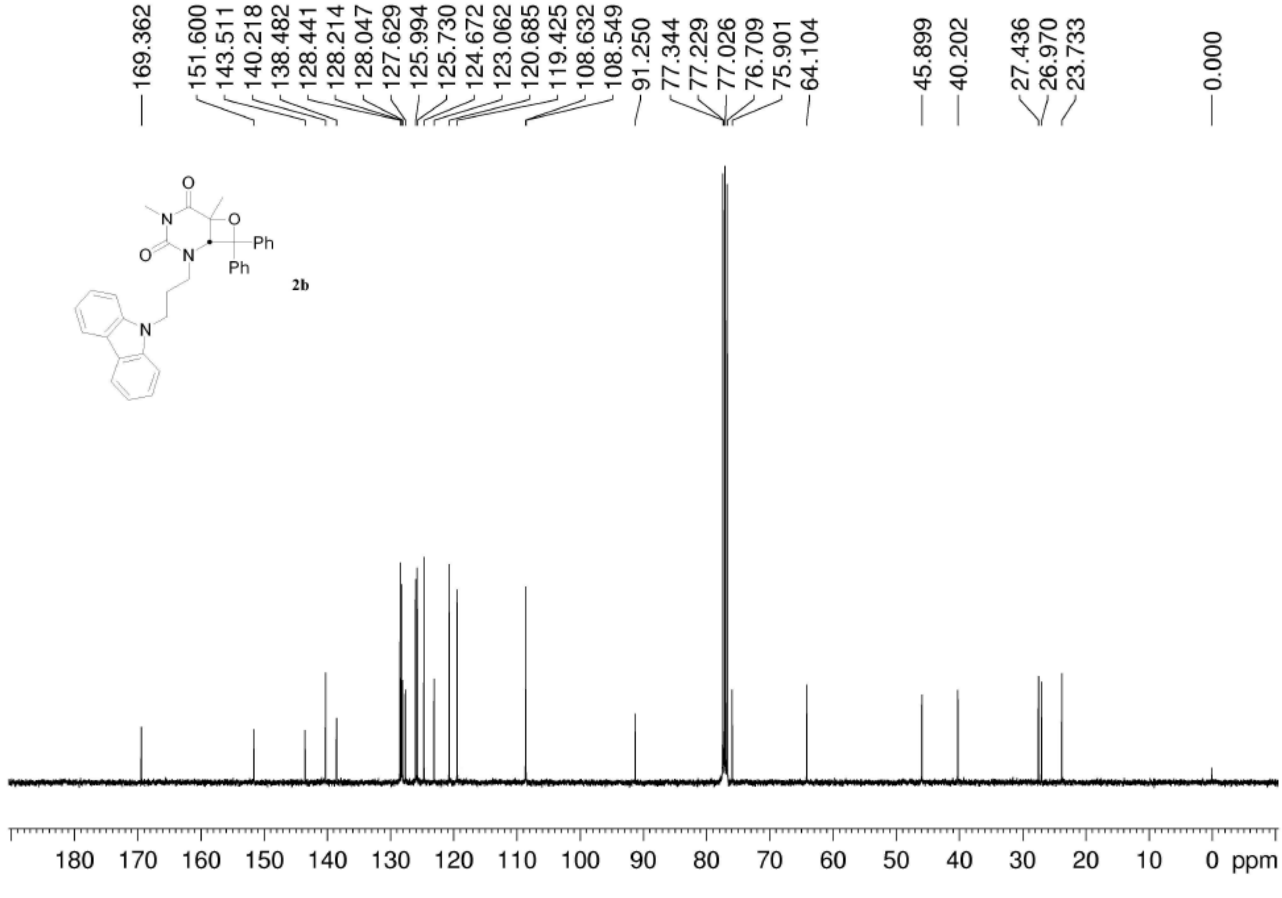
```
NAME          wqq-synox2
EXPNO                  1
PROCNO                 1
Date_           20100111
Time               14.20
INSTRUM            av300
PROBHD    5 mm PABBO BB-
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             5995.204 Hz
FIDRES          0.182959 Hz
AQ             2.7329011 sec
RG                 287.0
DW                83.400 usec
DE                  6.00 usec
TE                 300.0 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 13.00 usec
PL1                 2.00 dB
SFO1         300.1321009 MHz
SI                 16384
SF           300.1300058 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

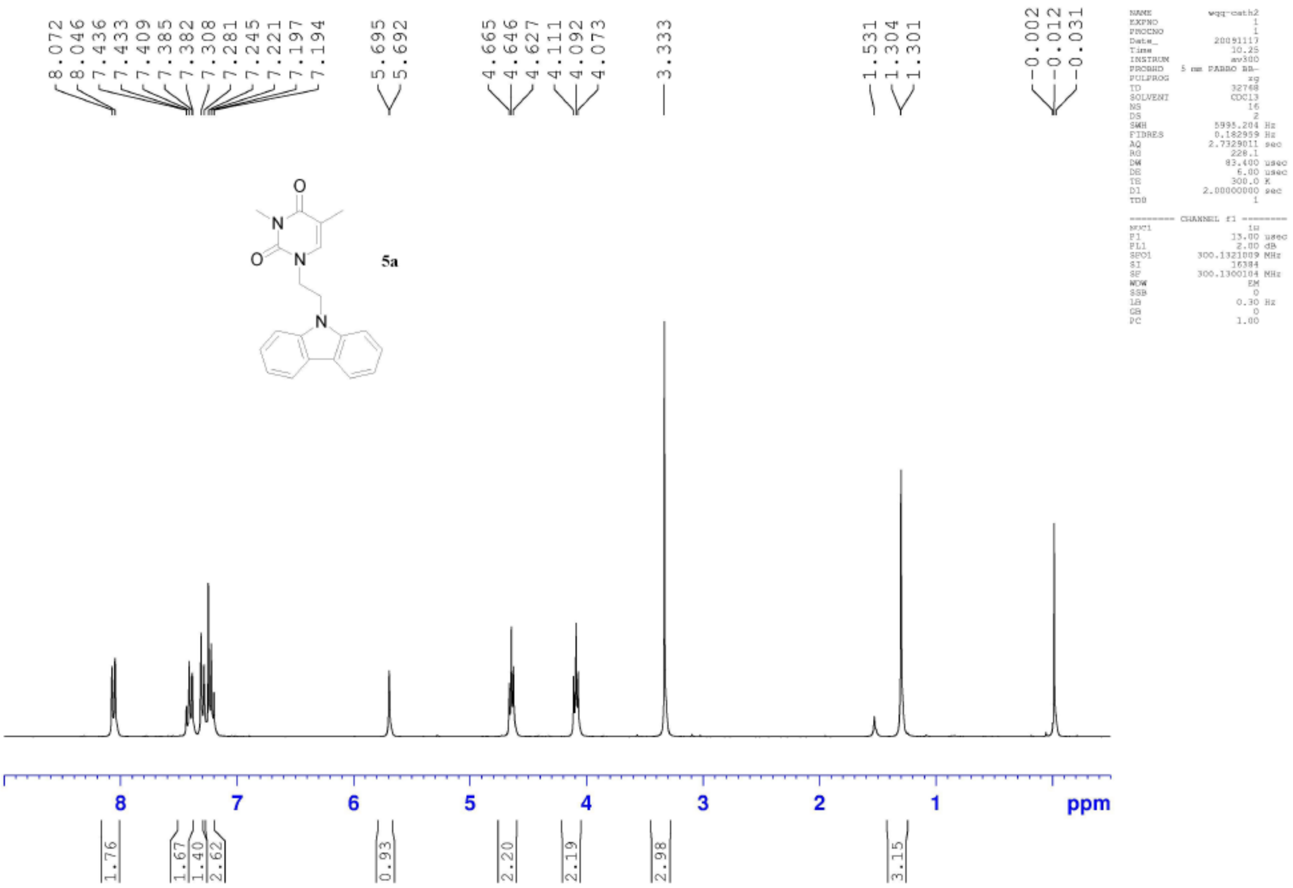



8.140
8.114
7.520
7.517
7.493
7.469
7.466
7.423
7.396
7.318
7.316
7.292
7.265
7.258
7.241
7.215
7.148
7.141
7.124
7.090
7.070
7.064
7.042
6.578
6.554
6.549
4.893
4.881
4.853
4.428
4.421
4.377
4.370
4.069
4.062
4.022
4.015
3.395
3.367
3.359
3.348
2.998
2.982
1.549
0.344
0.010
-0.000
NAME wqq-caox2
EXPNO 1
PROCNO 1
Date_ 20091030
Time 10.42
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 512
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1321009 MHz
SI 16384
SF 300.1300066 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
O
N
O
Ph
Ph
N
2a
8
7
6
5
4
3
2
1
ppm
1.79
2.00
1.96
1.93
2.84
3.55
1.90
1.11
1.08
1.05
1.09
1.00
2.61
3.00

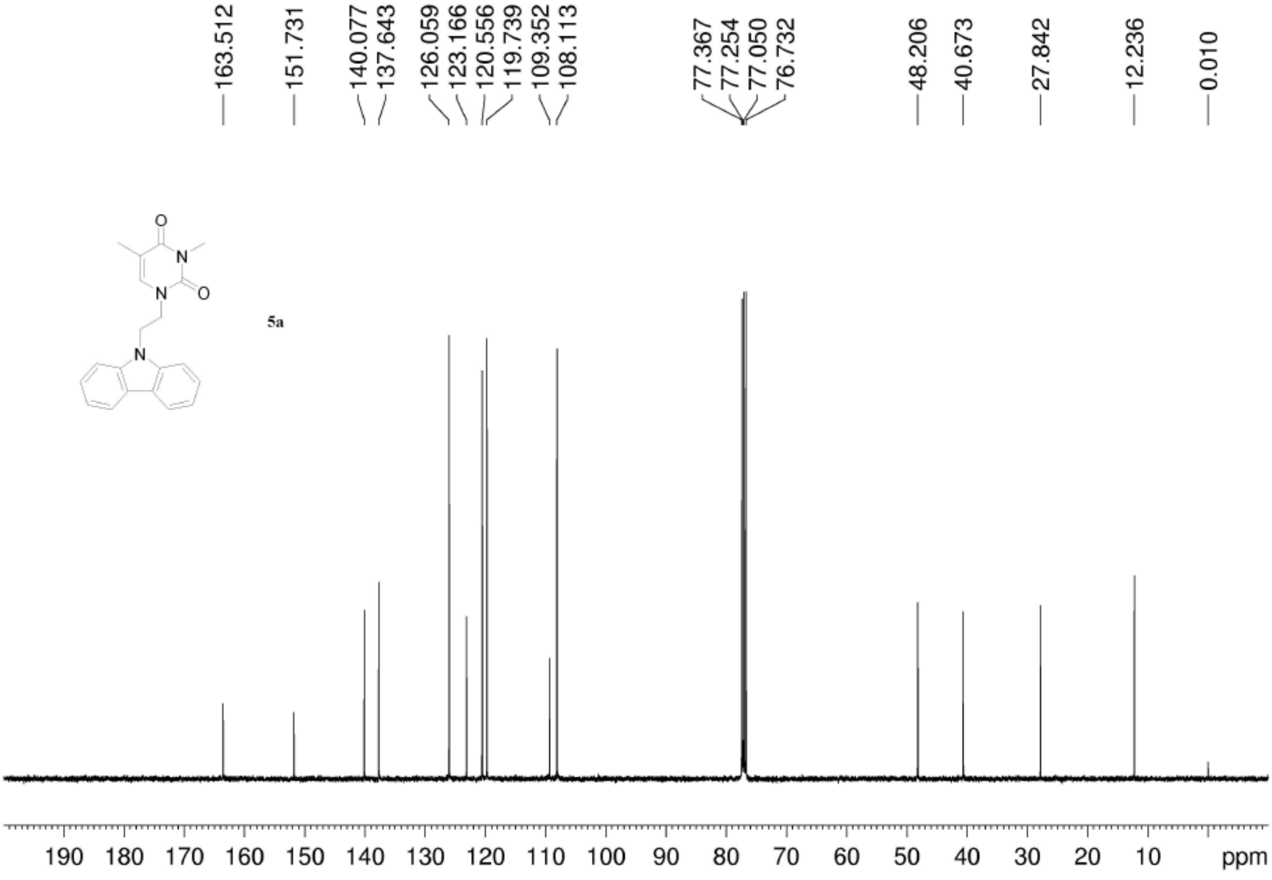

169.289
151.624
142.867
140.164
139.027
128.346
128.173
128.062
127.754
126.184
125.817
125.218
123.026
121.082
119.931
108.299
90.552
77.335
77.221
77.017
76.701
75.296
63.843
46.538
40.443
27.317
22.368
-0.004
O
N
O
N
O
Ph
Ph
N
2a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

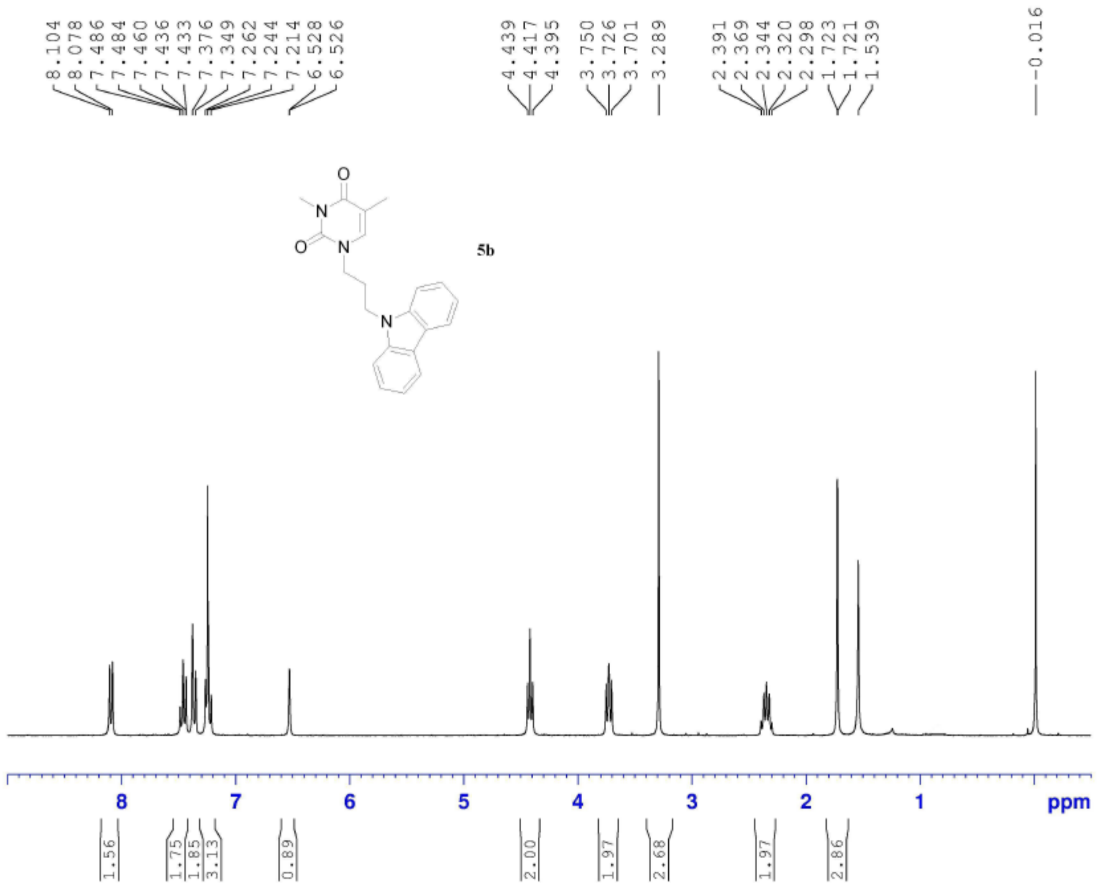

8.197
8.172
7.538
7.514
7.490
7.487
7.470
7.418
7.391
7.325
7.301
7.277
7.260
7.238
7.214
7.202
7.192
7.168
7.143
7.094
7.068
7.044
6.974
6.963
6.956
6.951
6.942
6.733
6.708
6.704
4.454
4.435
4.417
4.390
4.369
4.212
3.892
3.843
2.937
2.662
2.237
2.211
2.190
1.548
1.534
0.010
-0.000
-0.011
-0.022
O
N
O
N
O
Ph
Ph
N
2b
10
9
8
7
6
5
4
3
2
1
ppm
1.68
2.12
2.00
2.07
4.89
1.90
1.87
1.90
2.25
1.03
1.00
2.76
1.01
2.13
3.66


169.362
151.600
143.511
140.218
138.482
128.441
128.214
128.047
127.629
125.994
125.730
124.672
123.062
120.685
119.425
108.632
108.549
91.250
77.344
77.229
77.026
76.709
75.901
64.104
45.899
40.202
27.436
26.970
23.733
0.000
O
N
O
N
O
Ph
Ph
N
2b
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



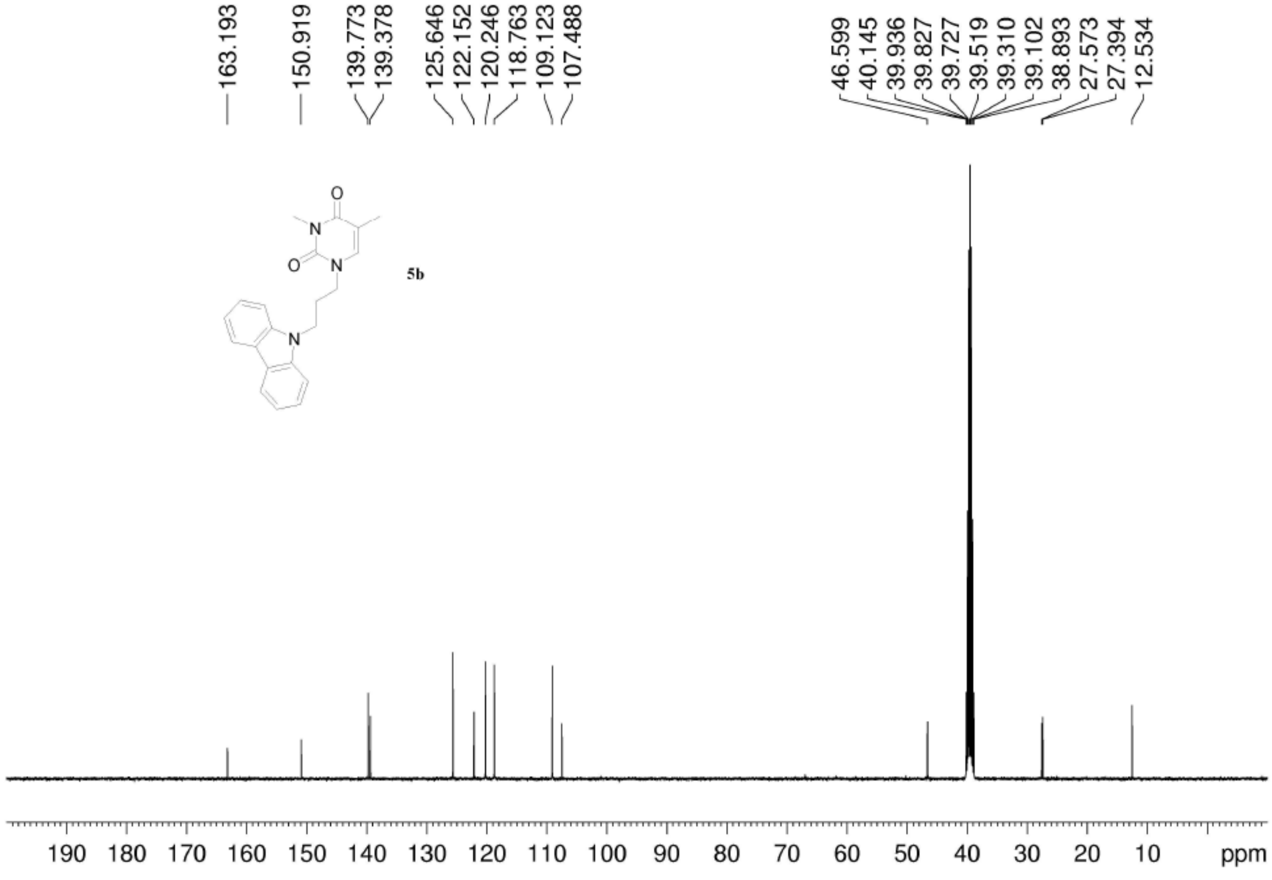
```
NAME          wqq-cath2
EXPNO                 1
PROCNO                1
Date_          20091117
Time              10.25
INSTRUM           av300
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            5995.204 Hz
FIDRES         0.182959 Hz
AQ            2.7329011 sec
RG                228.1
DW               83.400 usec
DE                 6.00 usec
TE                300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                13.00 usec
PL1                2.00 dB
SFO1        300.1321009 MHz
SI                16384
SF          300.1300104 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



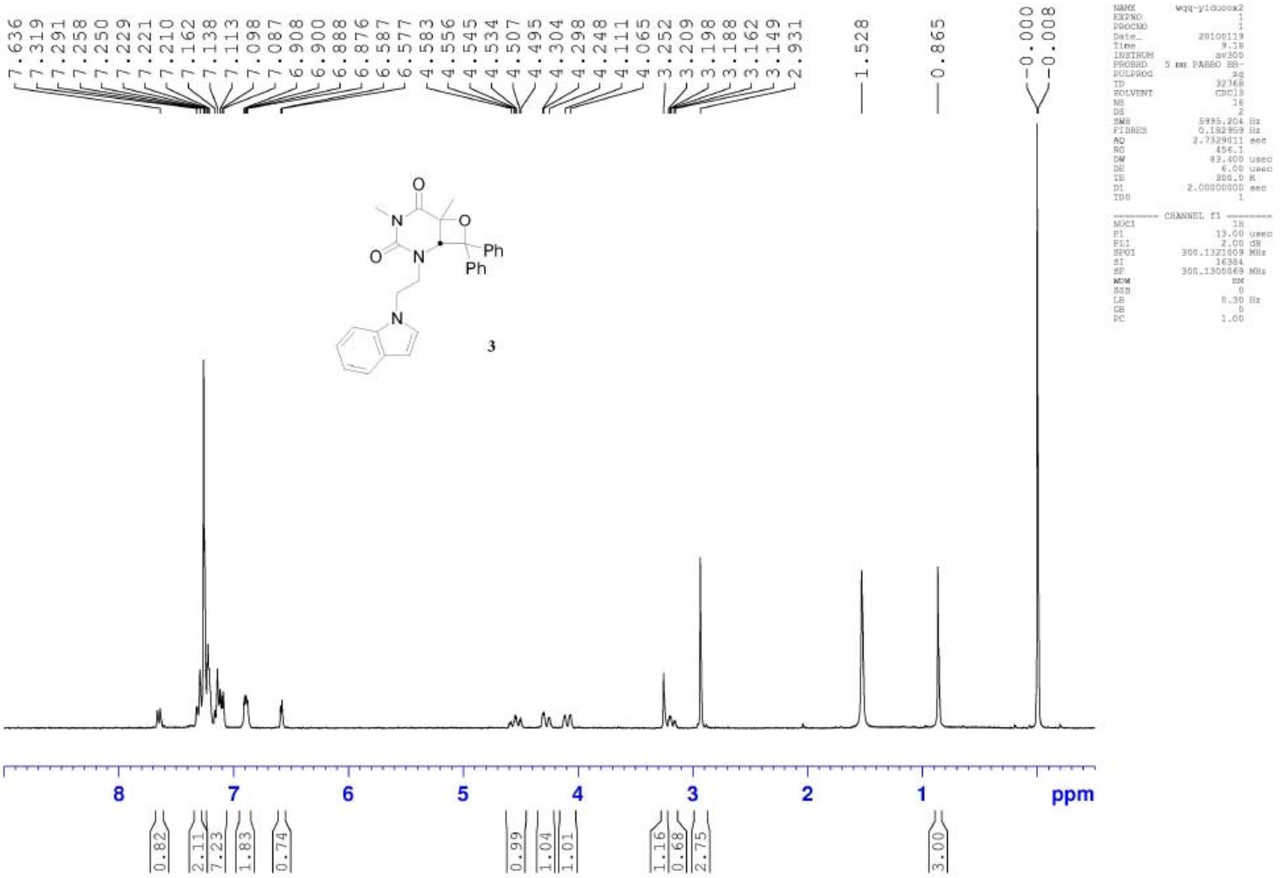


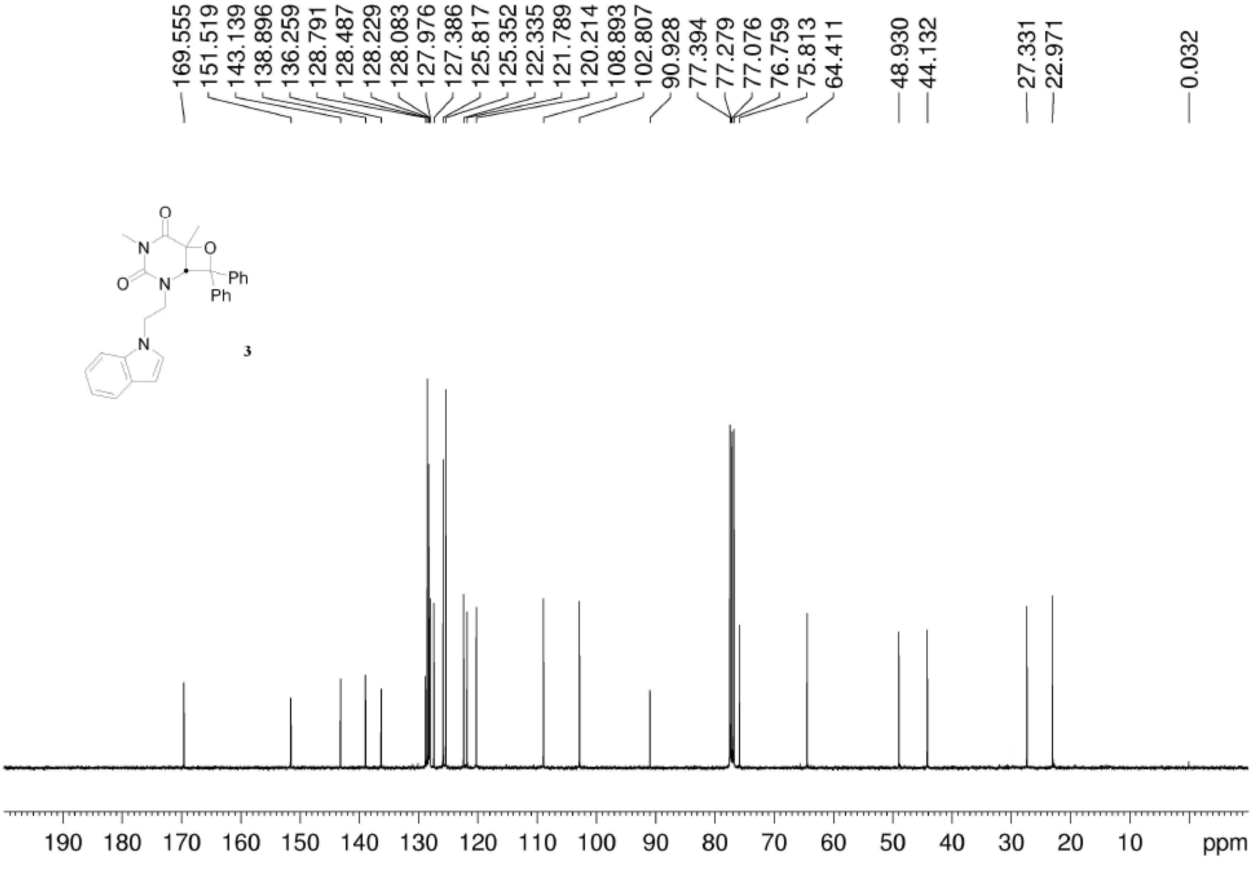
5b

```
NAME          wqq-cath
EXPNO                1
PROCNO               1
Date_         20090928
Time             11.14
INSTRUM          av300
PROBHD   5 mm PABBO BB-
PULPROG             zg
TD               32768
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           5995.204 Hz
FIDRES        0.182959 Hz
AQ           2.7329011 sec
RG               574.7
DW              83.400 usec
DE                6.00 usec
TE               296.8 K
D1          2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               13.00 usec
PL1               2.00 dB
SFO1       300.1321009 MHz
SI               16384
SF         300.1300108 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



5b


7.636
7.319
7.291
7.258
7.250
7.229
7.221
7.210
7.162
7.138
7.113
7.098
7.087
6.908
6.900
6.888
6.876
6.587
6.577
4.583
4.556
4.545
4.534
4.507
4.495
4.304
4.298
4.248
4.111
4.065
3.252
3.209
3.198
3.188
3.162
3.149
2.931
1.528
0.865
0.000
-0.008
NAME wqq-yldxoox2
EXPNO 1
PROCNO 1
Date_ 20100119
Time 9.18
INSTRUM av300
PROBHD 5 mm PABBO BB-
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 456.1
DW 83.400 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.00 usec
PL1 2.00 dB
SFO1 300.1321009 MHz
SI 16384
SF 300.1300069 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
N
O
O
N
Ph
Ph
N
3
8
7
6
5
4
3
2
1
ppm
0.82
2.11
7.23
1.83
0.74
0.99
1.04
1.01
1.16
0.68
2.75
3.00


169.555
151.519
143.139
138.896
136.259
128.791
128.487
128.229
128.083
127.976
127.386
125.817
125.352
122.335
121.789
120.214
108.893
102.807
90.928
77.394
77.279
77.076
76.759
75.813
64.411
48.930
44.132
27.331
22.971
0.032
O
N
O
O
N
Ph
Ph
N
3
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm