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Supporting Information (SI)

A Green Synthesis of Highly Substituted 1,5-Diketones

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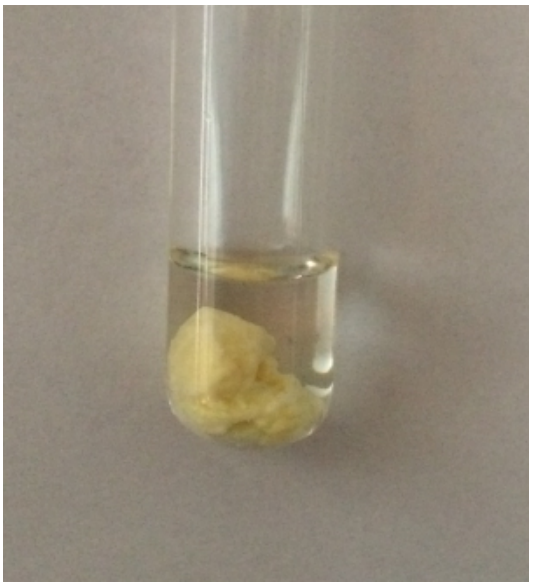
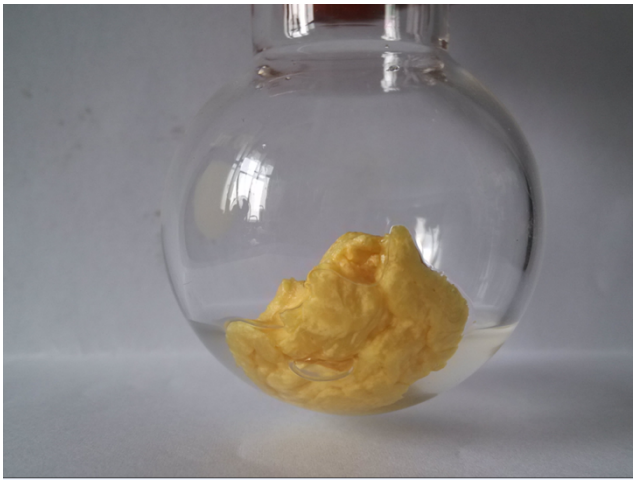


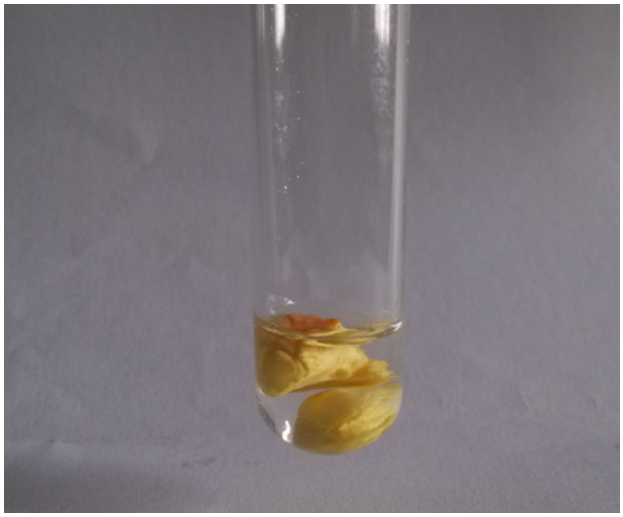
Picture 1. Synthesis of **3a** using TBAB as PTC at 200 mg scale in the test tube at 2.5 h.

Reaction conditions: acetophenone (200 mg), isobutylaldehyde (1 equiv), KOH (2 equiv), TBAB (0.06 equiv), water (1 mL), rt, 2.5 h.



Picture 2. Synthesis of **3a** using TBAB as PTC at 2000 mg scale in a 100 mL round-bottom flask at 2.5 h.

Reaction conditions: acetophenone (2000 mg), isobutylaldehyde (1 equiv), KOH (2 equiv), TBAB (0.06 equiv), water (10 mL), rt, 2.5h.



Picture 3. Synthesis of **3a** using Aliquat 336 as PTC at 200 mg scale in the test tube at 1 h.

Reaction conditions: acetophenone (200 mg), isobutylaldehyde (1 equiv), KOH (2 equiv), Aliquat 336 (0.1 equiv), water (1 mL), rt, 1h.

Picture 4. Synthesis of **3a** using Aliquat 336 as PTC at 2000 mg scale in a 100 mL round-bottom flask at 1 h.

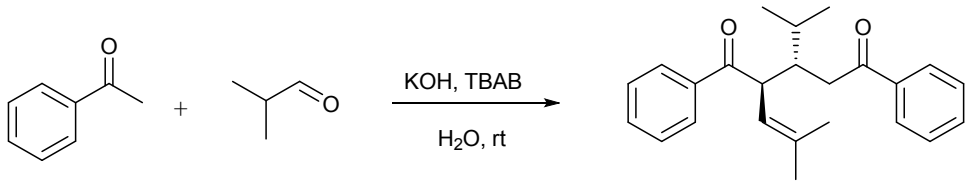
Reaction conditions: acetophenone (2000 mg), isobutylaldehyde (1 equiv), KOH (2 equiv), Aliquat 336 (0.1 equiv), water (10 mL), rt, 1h.

General Experimental Information

All of the chemicals were obtained from commercial sources or prepared according to standard methods. NMR spectra were recorded with a 400 or 600 MHz spectrometer for ^{1}H NMR, a 100 or 151 MHz spectrometer for ^{13}C NMR and a 377 MHz spectrometer for ^{19}F NMR. TMS was used as an internal standard. Chemical shifts (δ) were reported relative to TMS (^{1}H) or CDCl3 (^{13}C). Multiplicities were reported as follows: singlet (s), doublet (d), triplet (t), quartet (q), multiplet (m), dd (doublet of doublets) and dt (doublet of triplets). Coupling constants were reported in Hertz (Hz). Melting points were recorded with a micro melting point apparatus. Infrared analyses (KBr pellet) were performed by FT-IR. X-ray structural analyses were conducted on the XtaLAB mini diffractometer (600 W, SHINE, CCD, 75 mn, 0.1 electorns/pixel/sec). High resolution mass spectra (HRMS) were recorded on a QTOF mass analyzer with electrospray ionization (ESI).

General procedure for all products

Typical 200 mg scale condensation procedure using the synthesis of 3a as an example



1 **2** **3a**

Acetophenone (200 mg), isobutylaldehyde (1 equiv), KOH (2 equiv), TBAB (0.06 equiv) and water (1 mL) were added to a 10 mL test tube. After the mixture was stirred at rt for 2.5 h and TLC indicated the completion of the reaction, the crude product aggregated into a hard clump (SI, S2, Picture 1). The reaction was then stopped and the aqueous KOH was decanted. The crude product in the test tube was washed with distilled water (3 x 2 mL), and then crystallized with methanol (1 mL) to give a white product **3a** (279 mg, 96%, mp: 142.1-143.5 ^{o}C).

The same procedure was used in preparing **3a** using Aliquat 336 as PTC (SI, S3, Picture 3).

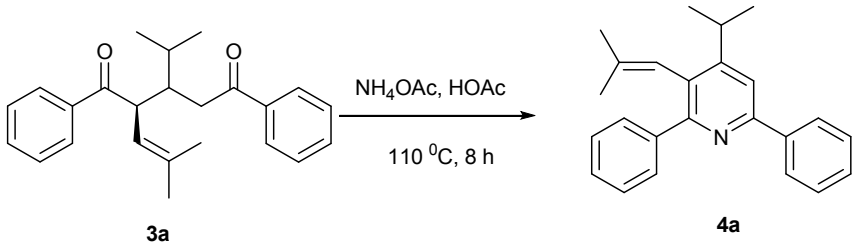
Synthesis of 3a in 13.9 g scale

The synthesis of **3a** starting from 10 g of acetophenone was performed in a 100 mL round-bottom flask using the same procedure as above to yield **3a** (13.9 g, 96%, t: 2.5 h).

Recycling procedure in the synthesis of 3a

In a 100 mL round-bottom flask, a mixture of acetophenone (2000 mg), isobutylaldehyde (1 equiv), Aliquat 336 (0.1 equiv), KOH (2 equiv) and water (10 mL) was stirred at rt. After TLC indicated the completion of the reaction, the crude product aggregated into a hard clump (SI, S3, Picture 4). The reaction was then stopped and the aqueous KOH was decanted. The crude product was washed with distilled water (3 x 5 mL), and crystallized with methanol (10 mL) to yield **3a** (2.75 g, 95%). In the second cycle, a new mixture of acetophenone (2000 mg), isobutylaldehyde (1 equiv), Aliquat 336 (0.1 equiv) and the aqueous KOH used in the first reaction was subject to the as above conditions to yield **3a** (2.68 g, 92%). The reaction was repeated in this manner five times. The reaction times ranged from 1-17 h and the yields from 95-84%.

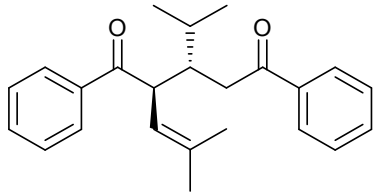
Typical 300 mg scale synthesis procedure using the synthesis of 4a as an example



3a **4a**

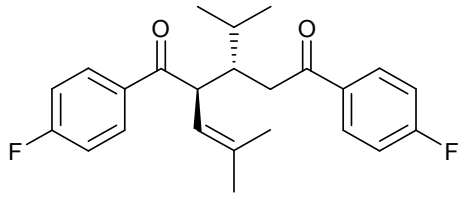
In a 25 mL round-bottom flask, a mixture of **3a** (300 mg), NH_4OAc (6 equiv) and acetic acid (5 mL) was refluxed for 8 h. The acetic acid was then distilled off. The residue was dissolved in ethyl acetate (5 mL), washed with water (2 x 5 mL), and dried over Na_2SO_4. The ethyl acetate was then evaporated and the product was purified by column chromatography on silica gel (petroleum ether/ ethyl acetate=10:1) to give **4a** (203 mg, 72%, mp: 62.6-63.5 ^oC).

Analytical data for all products



3a

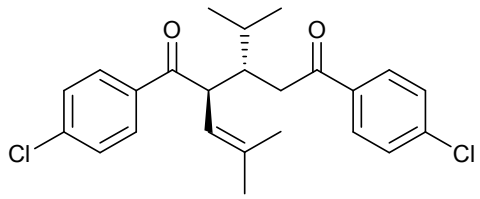
***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-diphenyl-l,5-pentanedione (3a):**[1] 279 mg, 96% yield; white solid; mp: 142-144 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.01 – 7.91 (m, 4H), 7.55 (dd, *J* = 7.6, 5.6 Hz, 2H), 7.47 (t, *J* = 7.5 Hz, 4H), 5.03 (d, *J* = 10.4 Hz, 1H), 4.41 (t, *J* = 10.1 Hz, 1H), 3.14 (m, 1H), 3.05 (dd, *J* = 16.6, 7.0 Hz, 1H), 2.74 (dd, *J* = 16.6, 4.7 Hz, 1H), 1.79 (m, 1H), 1.68 (d, *J* = 0.7 Hz, 3H), 1.39 (s, 3H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.89 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.2, 199.8, 137.6, 137.5, 135.8, 132.9, 132.6, 128.6, 128.4, 128.2, 127.9, 123.6, 50.2, 42.4, 36.4, 29.9, 25.7, 21.5, 18.4, 18.1; **IR** (KBr) ν: 3061, 2961, 2928, 2872, 1679, 1589, 1445, 1272, 1027, 753, 698 cm^{-1}.



3b

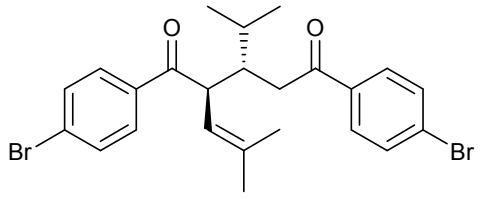
***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-bis(4-fluorophenyl)-l,5-pentanedione (3b):** 250 mg, 90% yield; white solid; mp: 133-135 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 5.1 Hz, 4H), 7.13 (s, 4H), 4.97 (d, *J* = 9.9 Hz, 1H), 4.33 (t, *J* = 9.4 Hz, 1H), 3.09 (s, 1H), 2.99 (dd, *J* = 16.4, 6.5 Hz, 1H), 2.69 (d, *J* = 16.2 Hz, 1H), 1.74 (s, 1H), 1.67 (s, 3H), 1.37 (s, 3H), 0.95 (d, *J* = 5.9 Hz, 3H), 0.86 (d, *J* = 6.0 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.6, 198.1, 166.9-164.4 (d, J_{CF} = 254.6 Hz), 166.7-164.2 (d, J_{CF} = 254.1 Hz), 135.9, 134.0 (d, J_{CF} = 3.0 Hz), 133.8 (d, J_{CF} = 2.4 Hz), 130.8-130.7 (d, J_{CF} = 9.2 Hz), 130.5-130.4 (d, J_{CF} = 9.2 Hz), 123.3, 115.8-115.4 (t, J_{CF} = 21.9 Hz), 50.1, 42.3, 36.3, 29.9, 25.7, 21.4, 18.4, 18.1; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -105.6, -106.1; **IR** (KBr) ν: 3078, 2963, 2923, 1673, 1597,1504, 1224, 1153, 834, 617, 592 cm^{-1}. **HRMS** (ESI) found: m/z 407.1798 $[M+Na]^+$; calcd. for

$C_{24}H_{26}F_2O_2Na^+$ 407.1793.



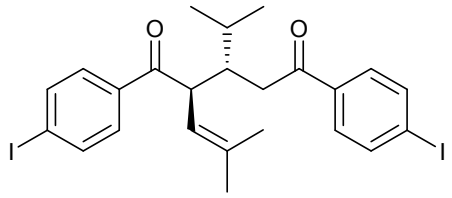
3c

***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-bis(4-chlorophenyl)-l,5-pentanedione (3c):**[2] 238 mg, 88% yield; white solid; mp: 160-163 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.89 (q, *J* = 4.2 Hz, 4H), 7.45 (dd, *J* = 8.6, 2.2 Hz, 4H), 4.97 (d, *J* = 10.4 Hz, 1H), 4.32 (t, *J* = 9.7 Hz, 1H), 3.09 (m, 1H), 2.98 (dd, *J* = 16.8, 7.1 Hz, 1H), 2.70 (dd, *J* = 16.8, 4.5 Hz, 1H), 1.75 (m, 1H), 1.67 (s, 3H), 1.40 (s, 3H), 0.96 (d, *J* = 6.8 Hz, 3H), 0.87 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.8, 198.4, 139.3, 139.0, 136.1, 135.8, 135.7, 129.6, 129.3, 129.0, 128.8, 123.2, 50.2, 42.3, 36.3, 29.9, 25.7, 21.4, 18.4, 18.1; **IR** (KBr) ν: 3067, 2961, 2926, 1674, 1589, 1396, 1271, 1216, 1091, 980, 825, 743 cm^{-1}.



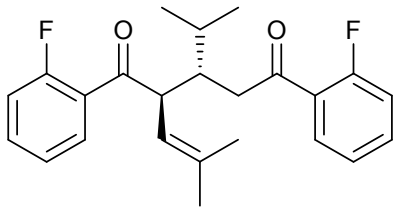
3d

***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-bis(4-bromophenyl)-l,5-pentanedione (3d):**[2] 221 mg, 87% yield; white solid; mp: 155-157 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.79 (s, 4H), 7.60 (s, 4H), 4.94 (d, *J* = 9.8 Hz, 1H), 4.30 (t, *J* = 9.3 Hz, 1H), 3.08 (s, 1H), 2.96 (dd, *J* = 16.3, 6.0 Hz, 1H), 2.67 (d, *J* = 16.7 Hz, 1H), 1.73 (s, 1H), 1.65 (s, 3H), 1.37 (s, 3H), 0.94 (d, *J* = 4.8 Hz, 3H), 0.85 (d, *J* = 5.1 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ 200.1, 198.7, 136.22, 136.20, 136.0, 132.0, 131.8, 129.7, 129.5, 128.1, 127.8, 123.2, 50.2, 42.3, 36.3, 29.9, 25.7, 21.5, 18.5, 18.1; **IR** (KBr) ν: 3063, 2957, 2923, 1683, 1582, 1223, 1031, 977, 778, 742, 678 cm^{-1}.



3e

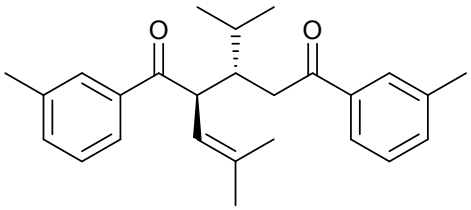
***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-bis(4-iodophenyl)-l,5-pentanedione (3e):** 200 mg, 82% yield; white solid; mp: 177-178 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 7.7 Hz, 4H), 7.70 – 7.59 (m, 4H), 4.96 (d, *J* = 10.4 Hz, 1H), 4.30 (t, *J* = 9.7 Hz, 1H), 3.15 – 3.02 (m, 1H), 2.96 (dd, *J* = 16.8, 7.1 Hz, 1H), 2.68 (dd, *J* = 16.8, 4.4 Hz, 1H), 1.78 – 1.70 (m, 1H), 1.66 (s, 3H), 1.40 (s, 3H), 0.95 (d, *J* = 6.7 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.3, 198.9, 138.0, 137.8, 136.8, 136.6, 136.2, 129.6, 129.4, 123.2, 100.9, 100.5, 50.1, 42.3, 36.3, 29.9, 25.7, 21.5, 18.5, 18.1; **IR** (KBr) ν: 2959, 2923, 1670, 1558, 1391, 1268, 1001, 773, 709 cm^{-1}. **HRMS** (ESI) found: m/z 622.9915 $[M+Na]^+$; calcd. for $C_{24}H_{26}I_2O_2Na^+$ 622.9914.



3f

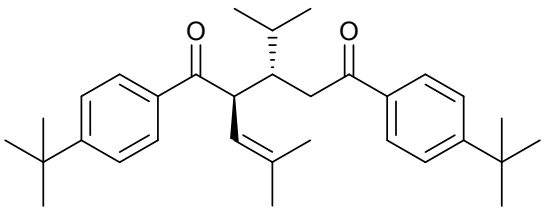
***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-bis(2-fluorophenyl)-l,5-pentanedione (3f):** 228 mg, 82% yield; white solid; mp: 73-74 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.77 (td, J = 7.6, 1.8 Hz, 1H), 7.69 (td, J = 7.6, 1.7 Hz, 1H), 7.53 – 7.43 (m, 2H), 7.22 (td, J = 8.1, 0.8 Hz, 2H), 7.13 (m, 2H), 4.95 (dd, J = 10.4, 1.1 Hz, 1H), 4.38 (dd, J = 10.2, 8.9 Hz, 1H), 3.10 – 3.01 (m, 1H), 2.96 (ddd, J = 17.6, 6.9, 2.3 Hz, 1H), 2.82 (ddd, J = 17.6, 4.6, 2.3 Hz, 1H), 1.88 – 1.75 (m, 1H), 1.58 (s, 3H), 1.48 (d, J = 0.8 Hz, 3H), 0.98 (d, J = 6.8 Hz, 3H), 0.88 (d, J = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.4 (d, J_{CF} = 3.8 Hz), 198.5 (d, J_{CF} = 3.9 Hz), 162.6-160.0 (d, J_{CF} = 253.5 Hz), 162.2-159.6 (d, J_{CF} = 252.8 Hz), 137.2, 133.9-133.8 (d, J_{CF} = 9.0 Hz), 133.8-133.7 (d, J_{CF} = 9.0 Hz), 130.9 (d, J_{CF} = 2.9 Hz), 130.8-130.7 (d, J_{CF} = 2.8 Hz), 127.3-127.2 (d,

J_{CF} = 13.0 Hz), 126.7-126.6 (d, J_{CF} = 13.3 Hz), 124.4 (d, J_{CF} = 3.4 Hz), 124.3 (d, J_{CF} = 3.4 Hz), 122.4, 116.6-116.4 (d, J_{CF} = 23.9 Hz), 116.6-116.3 (d, J_{CF} = 23.8 Hz), 54.3 (d, J_{CF} = 6.0 Hz), 41.7, 41.6, 30.0, 25.9, 21.4, 18.30 (d, J_{CF} = 2.0 Hz), 17.9; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -109.7, -111.6; **IR** (KBr) ν: 3082, 2961, 2929, 1683, 1610, 1480, 1451, 1372, 1276, 1001, 982, 759 cm^{-1}; **HRMS** (ESI) found: m/z 407.1795 $[M+Na]^+$; calcd. for $C_{24}H_{26}F_2O_2Na^+$ 407.1793.



3g

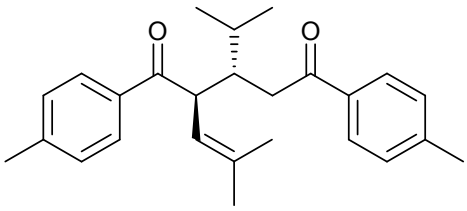
***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-di*m*-tolyl-l,5-pentanedione (3g):** 244 mg, 87% yield; white solid; mp: 101-102 °C ; ^{1}H N**MR** (400 MHz, $CDCl_3$) δ 7.84 – 7.70 (m, 4H), 7.43 – 7.32 (m, 4H), 5.03 (d, *J* = 10.4 Hz, 1H), 4.40 (dd, *J* = 10.2, 9.2 Hz, 1H), 3.15 – 3.07 (m, 1H), 3.03 (dd, *J* = 16.5, 6.8 Hz, 1H), 2.74 (dd, *J* = 16.5, 4.8 Hz, 1H), 2.44 (d, *J* = 5.2 Hz, 6H), 1.83 – 1.76 (m, 1H), 1.70 (d, *J* = 1.1 Hz, 3H), 1.42 (d, *J* = 1.0 Hz, 3H), 0.97 (d, *J* = 6.8 Hz, 3H), 0.89 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.48, 200.16, 138.40, 138.15, 137.72, 137.51, 135.61, 133.64, 133.32, 128.84, 128.51, 128.46, 128.30, 125.33, 125.14, 123.68, 50.23, 42.44, 36.56, 29.89, 25.72, 21.48, 21.43, 21.40, 18.44, 18.14; **IR** (KBr) ν: 2963, 1674, 1422, 1371, 1268, 1152, 1088, 986, 741, 688 cm^{-1}; **HRMS** (ESI) found: m/z 399.2298 $[M+Na]^+$; calcd. for $C_{26}H_{32}O_2Na^+$ 399.2295.



3h

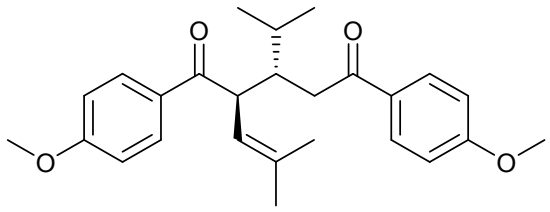
***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-bis(4-*tert*-butylphenyl)-l,5-pentanedione (3h):** 212 mg, 81% yield;

white solid; mp: 156-158 °C ; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.4 Hz, 2H), 7.48 (q, *J* = 8.5 Hz, 4H), 5.06 (d, *J* = 10.3 Hz, 1H), 4.40 (t, *J* = 9.7 Hz, 1H), 3.11 (m, 1H), 3.02 (dd, *J* = 16.5, 6.5 Hz, 1H), 2.74 (dd, *J* = 16.5, 5.1 Hz, 1H), 1.81 – 1.74 (m, 1H), 1.71 (s, 3H), 1.42 (s, 3H), 1.36 (d, 18H), 0.96 (d, *J* = 6.8 Hz, 3H), 0.88 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.8, 199.6, 156.5, 156.1, 135.5, 135.1, 134.7, 128.2, 127.9, 125.6, 125.3, 123.7, 49.9, 42.4, 36.5, 35.1, 35.0, 31.12, 31.10, 29.9, 25.7, 21.5, 18.5, 18.2; **IR** (KBr) ν: 3059, 2958, 2871, 1676, 1604, 1462, 1369, 1270, 1191, 1108, 982, 830 cm^{-1}. **HRMS** (ESI) found: m/z 483.3227 $[M+Na]^+$; calcd. for $C_{32}H_{44}O_2Na^+$ 483.3234.



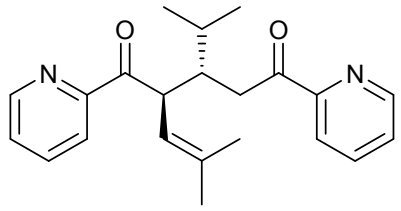
3i

***anti*-3-Isopropyl-2-(2-methylpropeny1)-l,5-di*p*-tolyl-l,5-pentanedione (3i):**[3] 213 mg, 76% yield; white solid; mp: 128-130 °C ; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 23.5, 8.0 Hz, 4H), 7.25 (dd, *J* = 11.9, 6.2 Hz, 4H), 5.00 (d, *J* = 10.4 Hz, 1H), 4.36 (t, *J* = 9.8 Hz, 1H), 3.10 (s, 1H), 2.99 (dd, *J* = 16.6, 6.9 Hz, 1H), 2.69 (dd, *J* = 16.6, 4.7 Hz, 1H), 2.40 (d, 6H), 1.80 – 1.72 (m, 1H), 1.66 (s, 3H), 1.36 (s, 3H), 0.94 (d, *J* = 6.8 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ 200.7, 199.3, 143.6, 143.2, 135.4, 135.1, 134.9, 129.3, 129.1, 128.3, 128.0, 124.0, 50.1, 42.5, 36.1, 29.8, 25.6, 21.5, 18.4, 18.0; **IR** (KBr) ν: 3036, 2961, 2924, 1676, 1606, 1373, 1181, 974, 809, 737 cm^{-1}.



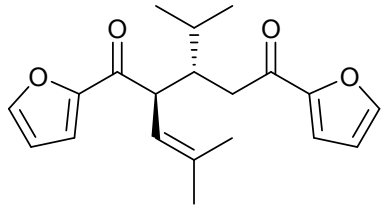
3j

***anti*-3-Isopropyl-2-(2-methylpropeny1)-1,5-bis(4-methoxyphenyl)-l,5-pentanedione (3j):**[2] 209 mg, 77% yield; white solid; mp: 116-117 °C ; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.95 (dd, *J* = 16.1, 8.8 Hz, 4H), 6.94 (dd, *J* = 8.9, 2.1 Hz, 4H), 5.05 (d, *J* = 10.4 Hz, 1H), 4.34 (t, *J* = 9.7 Hz, 1H), 3.88 (d, 6H), 3.14 – 3.03 (m, 1H), 2.99 (dd, *J* = 16.5, 6.7 Hz, 1H), 2.69 (dd, *J* = 16.5, 5.0 Hz, 1H), 1.82 – 1.72 (m, 1H), 1.69 (s, 3H), 1.41 (s, 3H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.87 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 199.8, 198.5, 163.3, 163.0, 135.2, 130.7, 130.5, 130.4, 130.2, 123.9, 113.8, 113.5, 55.43, 55.42, 49.8, 42.5, 36.2, 29.8, 25.7, 21.4, 18.4, 18.1; **IR** (KBr) ν: 3069, 2961, 2932, 1673, 1600, 1509, 1259, 1171, 1029, 837, 598 cm^{-1}.



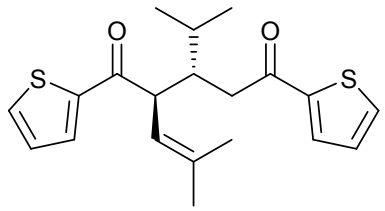
3k

***anti*-3-Isopropyl-2-(2-methylpropeny1)-1,5-di(pyridin-2-yl)-l,5-pentanedione (3k):** 252 mg, 87% yield; white solid; mp: 73-75 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.85 – 8.49 (m, 2H), 8.13 – 7.93 (m, 2H), 7.91 – 7.68 (m, 2H), 7.56 – 7.34 (m, 2H), 5.27 (t, *J* = 10.2 Hz, 1H), 4.87 (d, *J* = 10.5 Hz, 1H), 3.50 (dd, *J* = 17.1, 8.0 Hz, 1H), 3.10 (m, 1H), 2.84 (dd, *J* = 17.1, 3.9 Hz, 1H), 1.81 – 1.69 (m, 1H), 1.62 (s, 3H), 1.20 (s, 3H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.91 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ 202.7, 201.6, 153.8, 153.4, 148.9, 148.6, 136.8, 136.74, 136.71, 126.9, 126.7, 123.6, 122.3, 121.8, 48.1, 43.1, 34.7, 30.0, 25.5, 21.6, 18.3, 17.7; **IR** (KBr) ν: 3055, 2959, 2925, 1694, 1580, 1437, 1329, 991, 768, 745 cm^{-1}. **HRMS** (ESI) found: m/z 373.1890 $[M+Na]^+$; calcd. for $C_{22}H_{26}N_2O_2Na^+$ 373.1886.



3l

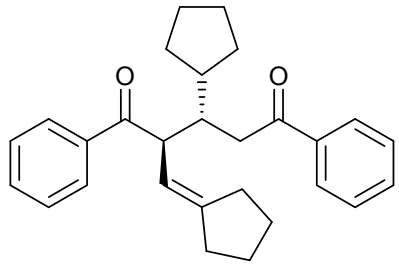
***anti*-3-Isopropyl-2-(2-methylpropeny1)-1,5-di(2-furanyl)-l,5-pentanedione (3l):** 277 mg, 93% yield; white solid; mp: 155-156 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.57 (d, *J* = 15.3 Hz, 2H), 7.21 (d, *J* = 3.5 Hz, 1H), 7.15 (d, *J* = 3.5 Hz, 1H), 6.62 – 6.47 (m, 2H), 5.02 (d, *J* = 10.5 Hz, 1H), 4.16 (t, *J* = 10.2 Hz, 1H), 3.02 (m, 1H), 2.85 (dd, *J* = 16.4, 7.4 Hz, 1H), 2.59 (dd, *J* = 16.4, 4.8 Hz, 1H), 1.76 (m, 1H), 1.68 (s, 3H), 1.42 (s, 3H), 0.93 (d, *J* = 6.8 Hz, 3H), 0.87 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ 189.8, 189.1, 153.3, 152.8, 146.3, 145.9, 136.7, 122.8, 117.1, 116.0, 112.3, 112.1, 51.1, 41.9, 36.3, 29.5, 25.5, 21.4, 18.3, 17.5; **IR** (KBr) ν: 3126, 3097, 2962, 1659, 1464, 1390, 1160, 1045, 765, 593 cm^{-1}. **HRMS** (ESI) found: m/z 351.1569 $[M+Na]^+$; calcd. for $C_{20}H_{24}O_4Na^+$ 351.1567.



3m

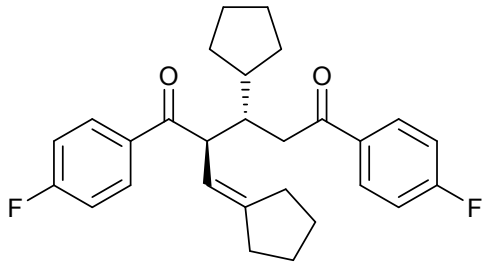
***anti*-3-Isopropyl-2-(2-methylpropeny1)-1,5-di(2-thiophenyl)-l,5-pentanedione (3m):** 254 mg, 89% yield; white solid; mp: 158-160 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 (d, *J* = 3.7 Hz, 1H), 7.68 (d, *J* = 3.7 Hz, 1H), 7.62 (d, *J* = 4.9 Hz, 1H), 7.60 (d, *J* = 4.9 Hz, 1H), 7.13 (q, *J* = 5.2 Hz, 2H), 5.07 (d, *J* = 10.5 Hz, 1H), 4.14 (t, *J* = 10.0 Hz, 1H), 3.07 (m, 1H), 2.89 (dd, *J* = 16.4, 7.2 Hz, 1H), 2.69 (dd, *J* = 16.4, 4.7 Hz, 1H), 1.81 (m, 1H), 1.70 (s, 3H), 1.40 (s, 3H), 0.95 (d, *J* = 6.8 Hz, 3H), 0.90 (d, *J* = 6.9 Hz, 3H); **^{13}C NMR** (151 MHz, $CDCl_3$) δ 193.6, 192.5, 145.0, 144.6, 136.2, 133.7, 133.1, 131.5, 131.1, 128.3, 128.0, 123.5, 52.4, 42.6, 37.3, 29.6, 25.6, 21.5, 18.4, 17.7; **IR** (KBr) ν: 3107, 2960, 1646, 1413, 1371, 1271, 1063, 851, 721 cm^{-1}. **HRMS** (ESI)

found: m/z 383.1115 $[M+Na]^+$; calcd. for $C_{20}H_{24}O_2S_2Na^+$ 383.1110.



3n

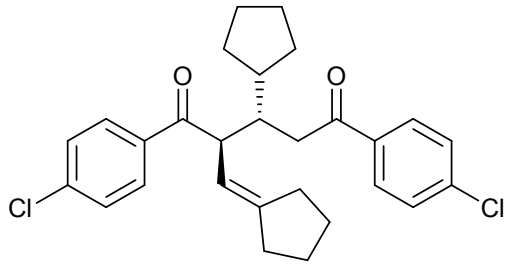
***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-l,5-diphenyl-l,5-pentanedione (3n):** 277 mg, 83% yield; white solid; mp: 102-105 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 7.7 Hz, 2H), 7.92 (d, *J* = 7.7 Hz, 2H), 7.58 – 7.51 (m, 2H), 7.51 – 7.37 (m, 4H), 5.24 (d, *J* = 10.1 Hz, 1H), 4.34 (q, *J* = 7.7, 1H), 3.19 (dd, *J* = 17.2, 5.7 Hz, 1H), 3.06 (m, 1H), 2.88 (dd, *J* = 17.2, 4.9 Hz, 1H), 2.27 (m, 1H), 2.22 – 2.13 (m, 1H), 2.13 – 2.03 (m, 1H), 1.93 (m, 2H), 1.80 – 1.73 (m, 1H), 1.63 – 1.41 (m, 8H), 1.31 – 1.15 (m, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.6, 199.7, 147.7, 137.6, 137.5, 132.8, 132.6, 128.6, 128.5, 128.3, 127.9, 118.2, 52.3, 44.1, 40.8, 38.3, 33.8, 31.6, 29.7, 29.4, 26.2, 25.8, 25.2, 25.1; **IR** (KBr) ν: 3061, 2951, 2868, 1681, 1597, 1447, 1272, 1215, 992, 885, 753, 696 cm^{-1}. **HRMS** (ESI) found: m/z 423.2300 $[M+Na]^+$; calcd. for $C_{28}H_{32}O_2Na^+$ 423.2295.



3o

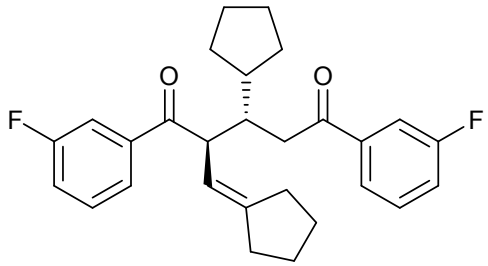
***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-l,5-bis(4-fluorophenyl)-l,5-pentanedione (3o):** 256 mg, 81% yield; white solid; mp: 124-125 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99 (m, 4H), 7.14 (q, *J* = 8.7 Hz, 4H), 5.26 (d, *J* = 10.1 Hz, 1H), 4.31 (dd, *J* = 10.1, 7.2 Hz, 1H), 3.18 (dd, *J* = 17.3, 5.7 Hz, 1H), 3.08 – 2.95 (m, 1H), 2.87 (dd, *J* = 17.3, 4.9 Hz, 1H), 2.28 (m, 1H), 2.22 – 2.07 (m, 2H), 2.05 – 1.88 (m, 2H),

1.85 – 1.70 (m, 1H), 1.48 (m, 9H), 1.22 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.0, 197.9, 166.9-164.4 (d, J_{CF} = 254.5 Hz), 166.8-164.2 (d, J_{CF} = 254.1 Hz), 147.8, 134.0 (d, J_{CF} = 3.0 Hz), 133.8(d, J_{CF} = 2.6 Hz), 130.9-130.8 (d, J_{CF} = 9.2 Hz), 130.5-130.4 (d, J_{CF} = 9.2 Hz), 117.8, 115.8-115.6 (d, J_{CF} = 21.7 Hz), 115.6-115.4 (d, J_{CF} = 21.7 Hz), 52.0, 44.1, 40.8, 38.2, 33.8, 31.6, 29.9, 29.4, 26.2, 25.9, 25.2, 25.1; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -105.7, -106.0; **IR** (KBr) ν: 3072, 2952, 2869, 1682, 1597, 1505, 1409, 1230, 1156, 990, 840, 599 cm^{-1}. **HRMS** (ESI) found: m/z 459.2107 $[M+Na]^+$; calcd. for $C_{28}H_{30}F_2O_2Na^+$ 459.2106.



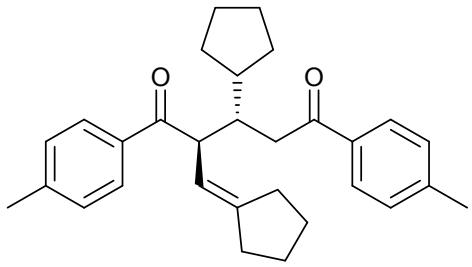
3p

***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-l,5-bis(4-chlorophenyl)-l,5-pentanedione (3p):** 252 mg, 83% yield; white solid; mp: 146-148 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90 (q, J = 8.5 Hz, 4H), 7.44 (t, J = 8.4 Hz, 4H), 5.24 (d, J = 10.2 Hz, 1H), 4.29 (dd, J = 10.1, 7.2 Hz, 1H), 3.16 (dd, J = 17.3, 5.8 Hz, 1H), 3.08 – 2.94 (m, 1H), 2.87 (dd, J = 17.3, 4.8 Hz, 1H), 2.27 (m, 1H), 2.22 – 2.06 (m, 2H), 2.04 – 1.88 (m, 2H), 1.83 – 1.71 (m, 1H), 1.63 – 1.34 (m, 9H), 1.20 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.3, 198.3, 148.1, 139.3, 139.1, 135.8, 135.7, 129.7, 129.3, 129.0, 128.8, 117.7, 52.1, 44.1, 40.8, 38.3, 33.8, 31.7, 29.9, 29.4, 26.2, 25.9, 25.2, 25.1; **IR** (KBr) ν: 3067, 2952, 2868, 1681, 1589, 1399, 1092, 990, 767, 530 cm^{-1}. **HRMS** (ESI) found: m/z 491.1515 $[M+Na]^+$; calcd. for $C_{28}H_{30}Cl_2O_2Na^+$ 491.1515.



3q

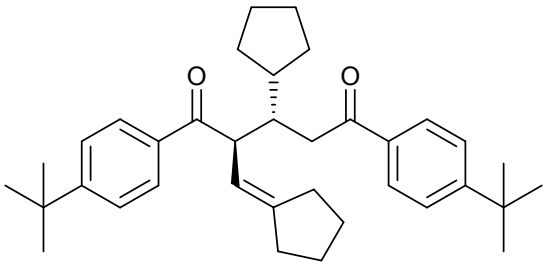
***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-1,5-bis(3-fluorophenyl)-l,5-pentanedione (3q):** 228 mg, 72% yield; white solid; mp: 100-101 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.75 (m, 2H), 7.63 (t, *J* = 11.2 Hz, 2H), 7.51 – 7.37 (m, 2H), 7.35 – 7.13 (m, 2H), 5.23 (d, *J* = 10.1 Hz, 1H), 4.29 (dd, *J* = 10.2, 7.2 Hz, 1H), 3.18 (dd, *J* = 17.3, 5.9 Hz, 1H), 3.08 – 2.99 (m, 1H), 2.87 (dd, *J* = 17.3, 4.8 Hz, 1H)., 2.34 – 2.25 (m, 1H), 2.23 – 2.10 (m, 2H), 2.01 – 1.91 (m, 2H), 1.77 (m, 1H), 1.65 – 1.44 (m, 8H), 1.40 – 1.30 (m, 1H), 1.29 – 1.14 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.2, 198.2, 164.2-161.7 (d, J_{CF} = 247.8 Hz), 164.1-161.6 (d, J_{CF} = 247.5 Hz), 148.3, 139.7-139.6 (t, J_{CF} = 5.5 Hz), 130.3-130.1 (t, J_{CF} = 8.7 Hz), 123.9 (d, J_{CF} = 2.8 Hz), 123.6 (d, J_{CF} = 2.9 Hz), 120.0-119.6 (t, J_{CF} = 21.1 Hz), 117.6, 115.2-115.0 (d, J_{CF} = 22.3 Hz), 114.8-114.6 (d, J_{CF} = 22.2 Hz), 52.4, 44.1, 40.8, 38.4, 33.8, 31.7, 29.9, 29.4, 26.2, 25.8, 25.2, 25.1; **^{19}F NMR** (377 MHz, $CDCl_3$) δ -111.8, -112.0; **IR** (KBr) ν: 2952, 2867, 1680, 1641, 1587, 1437, 1402, 1267, 1145, 864, 678 cm^{-1}. **HRMS** (ESI) found: m/z 459.2100 $[M+Na]^+$; calcd. for $C_{28}H_{30}F_2O_2Na^+$ 459.2106.



3r

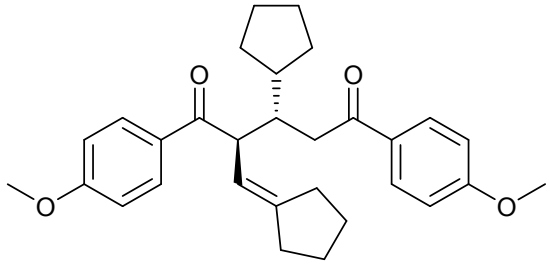
***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-l,5-di*p*-tolyl-l,5-pentanedione (3r):** 249 mg, 78% yield; white solid; mp: 128-130 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 8.1 Hz, 2H), 7.84 (d, *J* = 8.1 Hz, 2H), 7.26 (m, 4H), 5.27 (d, *J* = 10.2 Hz, 1H), 4.34 (dd, *J* = 10.0, 7.5 Hz, 1H), 3.17 (dd, *J* = 17.1, 5.5 Hz, 1H), 3.04 (m, 1H), 2.88

(dd, J = 17.1, 5.2 Hz, 1H), 2.42 (d, 6H), 2.29 (m, 1H), 2.23 – 2.09 (m, 2H), 2.00 – 1.92 (m, 2H), 1.81 – 1.73 (m, 1H), 1.62 – 1.42 (m, 8H), 1.38 – 1.29 (m, 1H), 1.21 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.2, 199.4, 147.4, 143.5, 143.2, 135.2, 135.0, 129.3, 129.1, 128.5, 128.0, 118.4, 52.1, 44.1, 40.8, 38.2, 33.8, 31.6, 29.7, 29.3, 26.2, 25.9, 25.2, 25.1, 21.6, 21.5; **IR** (KBr) ν: 3030, 2951, 2867, 1676, 1607, 1405, 1273, 1205, 1179, 997, 810, 758 cm^{-1}. **HRMS** (ESI) found: m/z 451.2610 $[M+Na]^+$; calcd. for $C_{30}H_{36}O_2Na^+$ 451.2608.



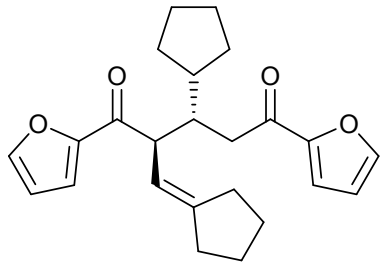
3s

***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-l,5-bis(4-*tert*-butylphenyl)-l,5-pentanedione (3s):** 209 mg, 72% yield; white solid; mp: 168-170 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.95 (d, J = 8.4 Hz, 2H), 7.88 (d, J = 8.3 Hz, 2H), 7.47 (q, J =8.4 Hz, 4H), 5.27 (d, J = 10.2 Hz, 1H), 4.34 (dd, J = 10.1, 7.5 Hz, 1H), 3.19 (dd, J = 17.0, 5.6 Hz, 1H), 3.12 – 3.00 (m, 1H), 2.86 (dd, J = 17.0, 5.2 Hz, 1H), 2.33 – 2.08 (m, 3H), 2.01 – 1.89 (m, 2H), 1.83 – 1.74 (m, 1H), 1.62 – 1.43 (m, 8H), 1.35 (d, 18H), 1.32 – 1.16 (m, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.1, 199.5, 156.4, 156.2, 147.3, 135.1, 134.8, 128.3, 127.9, 125.6, 125.3, 118.5, 52.2, 44.1, 40.9, 38.2, 35.1, 35.0, 33.7, 31.6, 31.1, 29.6, 29.3, 26.2, 25.8, 25.3, 25.2; **IR** (KBr) ν: 3040, 2957, 2868, 1679, 1604, 1463, 1407, 1269, 1222, 1108, 990, 774, 547 cm^{-1}. **HRMS** (ESI) found: m/z 535.3544 $[M+Na]^+$; calcd. for $C_{36}H_{48}O_2Na^+$ 535.3547.



3t

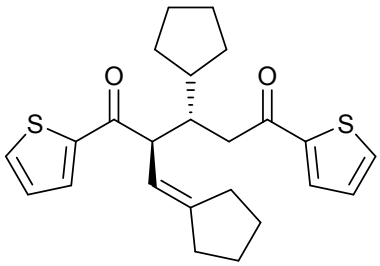
***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-1,5-bis(4-methoxyphenyl)-l,5-pentanedione (3t):** 227 mg, 74% yield; white solid; mp: 136-137 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.8 Hz, 2H), 7.92 (d, *J* = 8.8 Hz, 2H), 6.96 (d, *J* = 8.8 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H).5.29 (d, *J* = 10.2 Hz, 1H), 4.31 (dd, *J* = 10.1, 7.4 Hz, 1H), 3.88 (d, 6H), 3.15 (dd, *J* = 16.9, 5.4 Hz, 1H), 3.08 – 2.94 (m, 1H), 2.86 (dd, *J* = 17.0, 5.3 Hz, 1H), 2.36 – 2.24 (m, 1H), 2.16 (m, 2H), 2.03 – 1.88 (m, 2H), 1.76 (m, 1H), 1.64 – 1.33 (m, 9H), 1.29 – 1.10 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 200.2, 198.3, 163.3, 163.1, 147.1, 130.7, 130.6, 130.4, 130.1, 118.5, 113.8, 113.5, 55.4, 51.9, 44.1, 40.9, 38.0, 33.8, 31.6, 29.7, 29.3, 26.3, 25.9, 25.2, 25.1; **IR** (KBr) ν: 2949, 2867, 1673, 1600, 1574, 1509, 1459, 1259, 1170, 1030, 837, 737 cm^{-1}. **HRMS** (ESI) found: m/z 483.2507 $[M+Na]^+$; calcd. for $C_{30}H_{36}O_4Na^+$ 483.2506.



3u

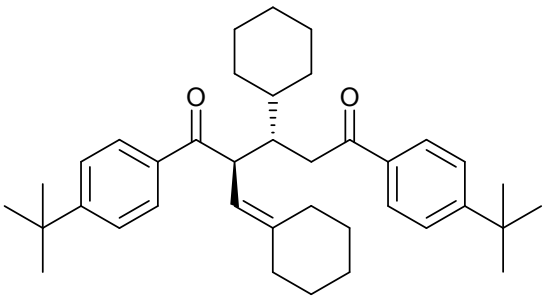
***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-1,5-di(2-furanyl)-l,5-pentanedione (3u):** 294 mg, 85% yield; white solid; mp: 138-139 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.61 (s, 1H), 7.55 (s, 1H), 7.24 (d, *J* = 3.4 Hz, 1H), 7.16 (d, *J* = 3.4 Hz, 1H), 6.54 (m, 2H), 5.22 (d, *J* = 10.4 Hz, 1H), 4.06 (dd, *J* = 10.2, 7.8 Hz, 1H), 3.11 – 2.90 (m, 2H), 2.73 (dd, *J* = 19.0, 6.9 Hz, 1H), 2.27 (m, 2H), 2.19 – 2.05 (m, 1H), 1.93 (m, 2H), 1.78 – 1.70 (m, 1H), 1.65 – 1.34 (m, 9H), 1.28 – 1.16 (m, 2H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 190.1, 188.9, 153.2, 152.9, 148.4, 146.4, 145.8, 117.7, 117.2, 116.1, 112.3,

112.1, 53.6, 43.6, 40.2, 38.0, 33.7, 31.3, 29.2, 29.1, 26.2, 26.0, 25.3, 25.2; **IR** (KBr) ν: 3133, 2951, 2867, 1671, 1565, 1466, 1392, 1160, 990, 733, 594 cm^{-1}. **HRMS** (ESI) found: m/z 403.1884 $[M+Na]^+$; calcd. for $C_{24}H_{28}O_4Na^+$ 403.1880.



3v

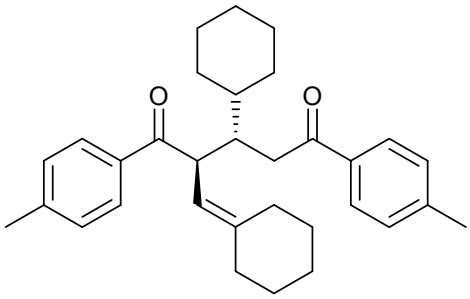
***anti*-3-Cyclopentyl-2-(cyclopentylidenemethyl)-1,5-di(2-thiophenyl)-l,5-pentanedione (3v):** 265 mg, 81% yield; white solid; mp: 152-154 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.81 (d, *J* = 3.6 Hz, 1H), 7.71 (d, *J* = 3.6 Hz, 1H), 7.63 (dd, *J* = 14.8, 4.9 Hz, 2H), 7.14 (dt, *J* = 12.9, 4.3 Hz, 2H), 5.27 (d, *J* = 10.3 Hz, 1H), 4.09 (q, *J* = 10.1, 7.9 Hz, 1H), 3.17 – 2.98 (m, 2H), 2.86 (dd, *J* = 15.3, 3.3 Hz, 1H), 2.29 (m, 2H), 2.19 – 2.06 (m, 1H), 2.06 – 1.84 (m, 2H), 1.83 – 1.73 (m, 1H), 1.56 (m, 8H), 1.38 – 1.18 (m, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 194.0, 192.4, 148.1, 144.9, 144.8, 133.7, 133.0, 131.6, 131.1, 128.2, 128.0, 118.3, 54.8, 43.7, 41.1, 39.2, 33.8, 31.4, 29.3, 29.2, 26.2, 25.9, 25.3, 25.2; **IR** (KBr) ν: 3105, 2950, 2867, 1649, 1517, 1415, 1215, 1062, 856, 726 cm^{-1}. **HRMS** (ESI) found: m/z 435.1424 $[M+Na]^+$; calcd. for $C_{24}H_{28}O_2S_2Na^+$ 435.1423.



3w

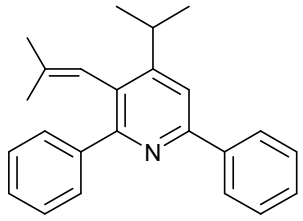
***anti*-3-Cyclohexyl-2-(cyclohexylidenemethyl)-1,5-bis(4-*tert*-butylphenyl)-l,5-pentanedione (3w):** 236 mg, 77% yield; white solid; mp: 174-176 °C; **^{1}H NMR** (400

MHz, $CDCl_3$) δ 7.90 (dd, *J* = 8.1, 5.9 Hz, 4H), 7.47 (d, *J* = 8.4 Hz, 4H), 5.03 (d, *J* = 10.4 Hz, 1H), 4.53 (dd, *J* = 10.1, 8.3 Hz, 1H), 3.06 (m, 2H), 2.86 (m, 1H), 2.23 (s, 2H), 1.91 (m, 1H), 1.87 – 1.54 (m, 7H), 1.44 (m, 4H), 1.36 (s, 18H), 1.25 – 0.99 (m, 7H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.1, 199.5, 156.3, 156.2, 143.1, 135.0, 134.8, 128.2, 128.0, 125.5, 125.3, 119.9, 48.1, 41.1, 40.8, 37.6, 37.1, 35.1, 35.0, 31.9, 31.12, 31.11, 29.5, 29.3, 27.8, 27.3, 26.8, 26.7, 26.6; **IR** (KBr) ν: 2961, 2925, 2853, 1678, 1605, 1446, 1403, 1270, 1236, 1107, 936, 830, 718 cm^{-1}. **HRMS** (ESI) found: m/z 563.3861 $[M+Na]^+$; calcd. for $C_{38}H_{52}O_2Na^+$ 563.3860.



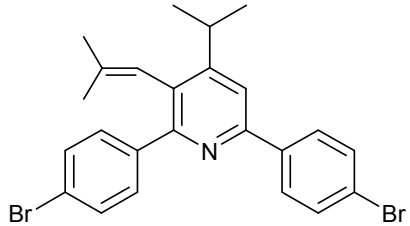
3x

***anti*-3-Cyclohexyl-2-(cyclohexylidenemethyl)-1,5-di*p*-tolyl-l,5-pentanedione (3x):** 252 mg, 74% yield; white solid; mp: 129-130 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.86 (t, *J* = 8.4 Hz, 4H), 7.26 (d, *J* = 7.3 Hz, 4H), 5.02 (d, *J* = 10.4 Hz, 1H), 4.52 (dd, *J* = 10.2, 8.3 Hz, 1H), 3.08 – 2.98 (m, 2H), 2.85 (m, 1H), 2.42 (s, 6H), 2.28 – 2.15 (m, 2H), 1.96 – 1.88 (m, 1H), 1.72 (m, 6H), 1.47 – 1.01 (m, 12H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 201.1, 199.3, 143.5, 143.2, 143.1, 135.0, 134.9, 129.3, 129.1, 128.3, 128.1, 120.0, 48.1, 41.1, 40.8, 37.6, 37.2, 31.9, 29.5, 29.3, 28.0, 27.4, 26.74, 26.70, 26.6, 21.6; **IR** (KBr) ν: 3031, 2926, 2852, 1680, 1607, 1572, 1447, 1408, 1273, 1179, 977, 786, 736 cm^{-1}. **HRMS** (ESI) found: m/z 479.2921 $[M+Na]^+$; calcd. for $C_{32}H_{40}O_2Na^+$ 479.2921.



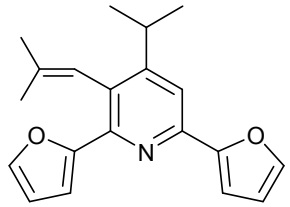
4a

4-Isopropyl-3-(2-methyl-1-propenyl)-2,6-diphenylpyridine (4a):[4] 203 mg, 72% yield; white solid; mp: 63-64 °C; **^{1}H NMR** (400 MHz, CDCl3) δ 8.16 – 8.09 (m, 2H), 7.67 (dd, *J* = 11.5, 4.6 Hz, 3H), 7.49 (dd, *J* = 10.2, 4.7 Hz, 2H), 7.38 (m, 4H), 6.21 (s, 1H), 3.24 (m, 1H), 1.75 (d, *J* = 1.0 Hz, 3H), 1.28 (s, 6H), 1.21 (s, 3H); **^{13}C NMR** (101 MHz, CDCl3) δ 157.94, 157.77, 155.34, 142.04, 139.92, 136.70, 129.75, 129.60, 128.59, 128.55, 127.42, 127.31, 126.95, 126.86, 120.81, 115.27, 30.10, 25.11, 22.96, 22.81, 19.34; **IR** (KBr) ν: 3064, 2966, 2929, 1587, 1488, 1438, 1369, 1009, 834, 733 cm^{-1}.



4b

4-Isopropyl-3-(2-methyl-1-propenyl)-2,6-bis(4-bromophenyl)pyridine (4b):[5] 253 mg, 88% yield; white solid; mp: 126-129 °C; **^{1}H NMR** (400 MHz, CDCl3) δ 7.98 (d, *J* = 8.5 Hz, 2H), 7.60 (d, *J* = 8.4 Hz, 3H), 7.53 (q, *J* = 8.6 Hz, 4H), 6.19 (s, 1H), 3.23 (m, 1H), 1.77 (s, 3H), 1.27 (d, *J* = 8.6 Hz, 6H), 1.20 (s, 3H); **^{13}C NMR** (151 MHz, CDCl3) δ 158.30, 156.84, 154.25, 140.75, 138.50, 137.36, 131.75, 131.42, 130.66, 129.93, 128.50, 123.12, 121.85, 120.33, 115.30, 30.14, 25.22, 22.92, 22.81, 19.40; **IR** (KBr) ν: 2963, 2925, 1645, 1586, 1487, 1436, 1368, 1071, 1008, 833 cm^{-1}.



4c

4-Isopropyl-3-(2-methyl-1-propenyl)-2,6-di(2-furanyl)pyridine (4c): 211 mg, 75% yield; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.56 (m, 3H), 7.13 (dd, *J* = 3.3, 0.6 Hz, 1H), 6.89 (dd, *J* = 3.4, 0.6 Hz, 1H), 6.55 (dd, *J* = 3.4, 1.8 Hz, 1H), 6.48 (dd, *J* = 3.4, 1.8 Hz, 1H), 6.20 (s, 1H), 3.21 (m, 1H), 1.96 (d, *J* = 1.1 Hz, 3H), 1.39 (d, *J* = 0.7 Hz, 3H), 1.27 (d, *J* = 6.5 Hz, 3H), 1.20 (d, *J* = 6.8 Hz, 3H); **^{13}C NMR** (101 MHz, $CDCl_3$) δ 157.97, 153.97, 153.31, 147.69, 147.32, 142.94, 137.42, 128.24, 120.66, 113.61, 111.92, 111.77, 111.22, 108.38, 29.74, 25.19, 23.25, 22.24, 19.09; **IR** (KBr) ν: 3118, 2966, 2929, 2871, 1605, 1565, 1492, 1224, 1051, 1006, 932, 737 cm^{-1}. **HRMS** (ESI) found: m/z 330.1460 $[M+Na]^+$; calcd. for $C_{20}H_{21}NO_2Na^+$ 330.1465.

References

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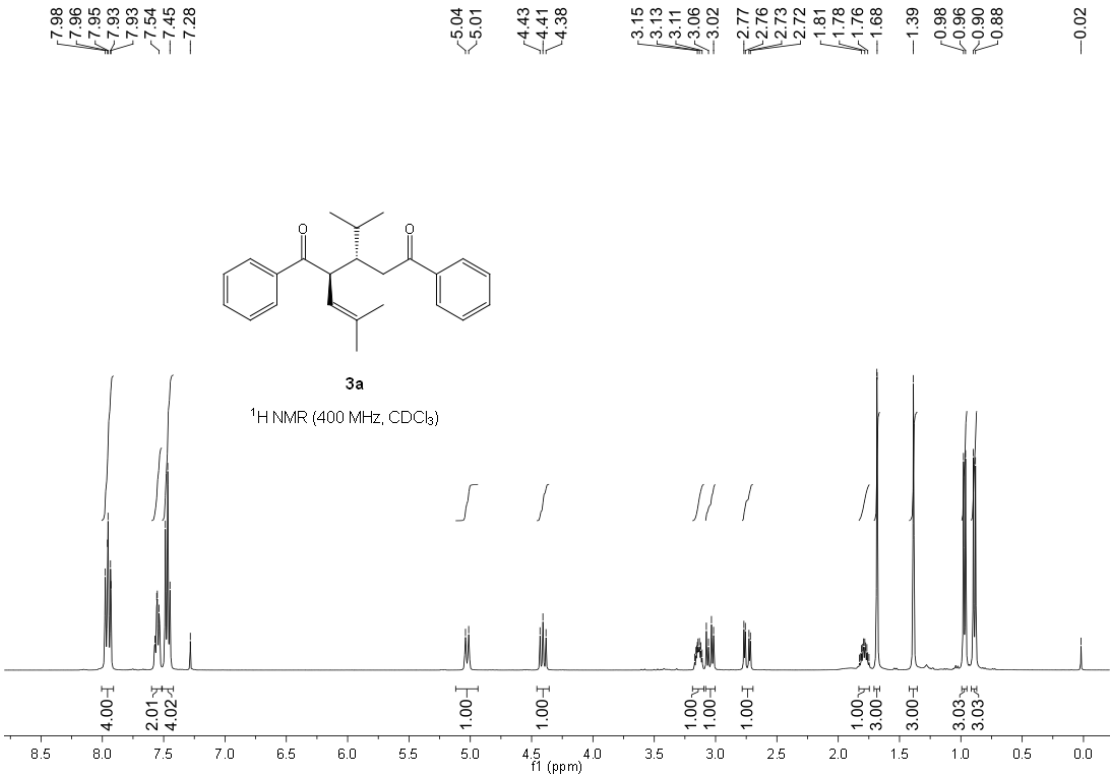
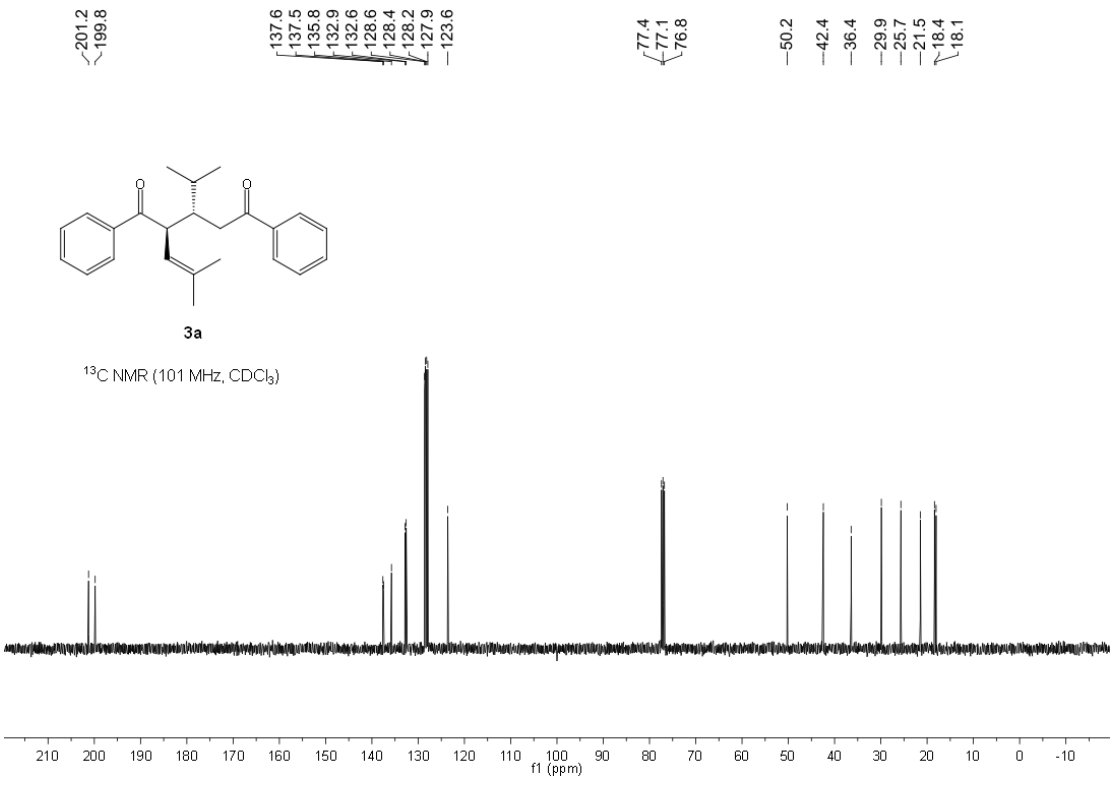
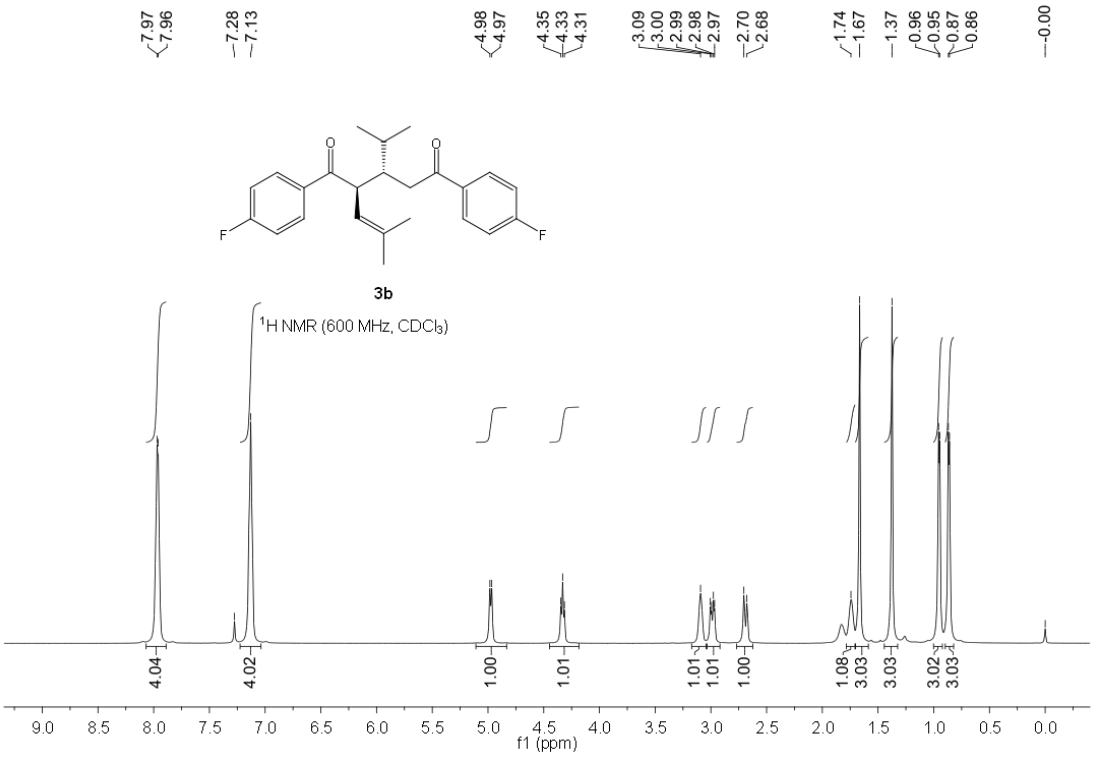
2. L. S. Novikov and I. G. Tishchenko, *Vestsi Akad. Navuk BSSR, Ser. Khim. Navuk*, 1970, **5**, 79-82.
3. I. G. Tishchenko, L. S. Novikov and L. S. Stanishevskii, *Vestsi Akad. Navuk BSSR, Ser. Khim. Navuk*, 1968, **4**, 90-94.
4. L. S. Novikov, V. A. Mezhentsev and I. G. Tishchenko, *Chem. Heterocycl. Compd. (New York, NY, U.S.)*, 1979, **15**, 1367-1367.
5. I. G. Tishchenko, V. A. Mezhentsev, L. S. Novikov and E. V. Volkov, *Vestsi Akad. Navuk BSSR, Ser. Khim. Navuk*, 1984, **4**, 92-95.

NMR spectra of the products:



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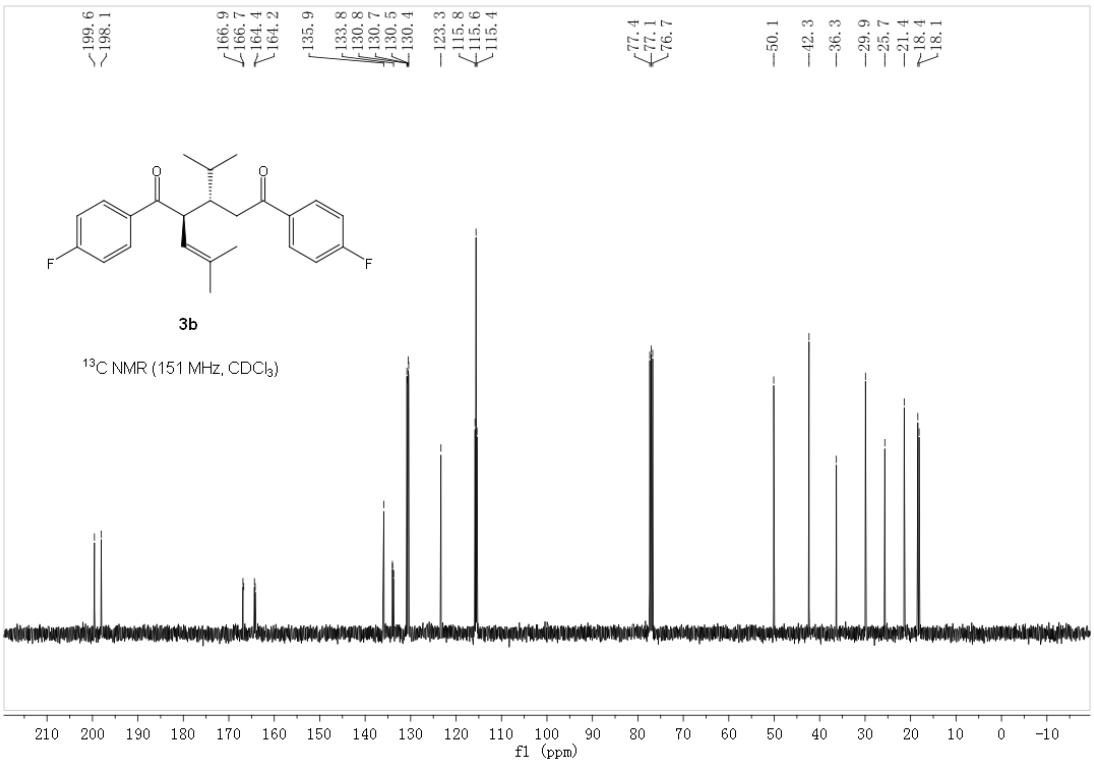
7.96
7.28
7.13
4.98
4.97
4.35
4.33
4.31
3.09
3.00
2.99
2.98
2.97
2.70
2.68
1.74
1.67
1.37
0.96
0.95
0.87
0.86
0.00
O
O
F
F
3b
1H NMR (600 MHz, CDCl3)
4.04
4.02
1.00
1.01
1.01
1.01
1.00
1.08
3.03
3.03
3.02
3.03
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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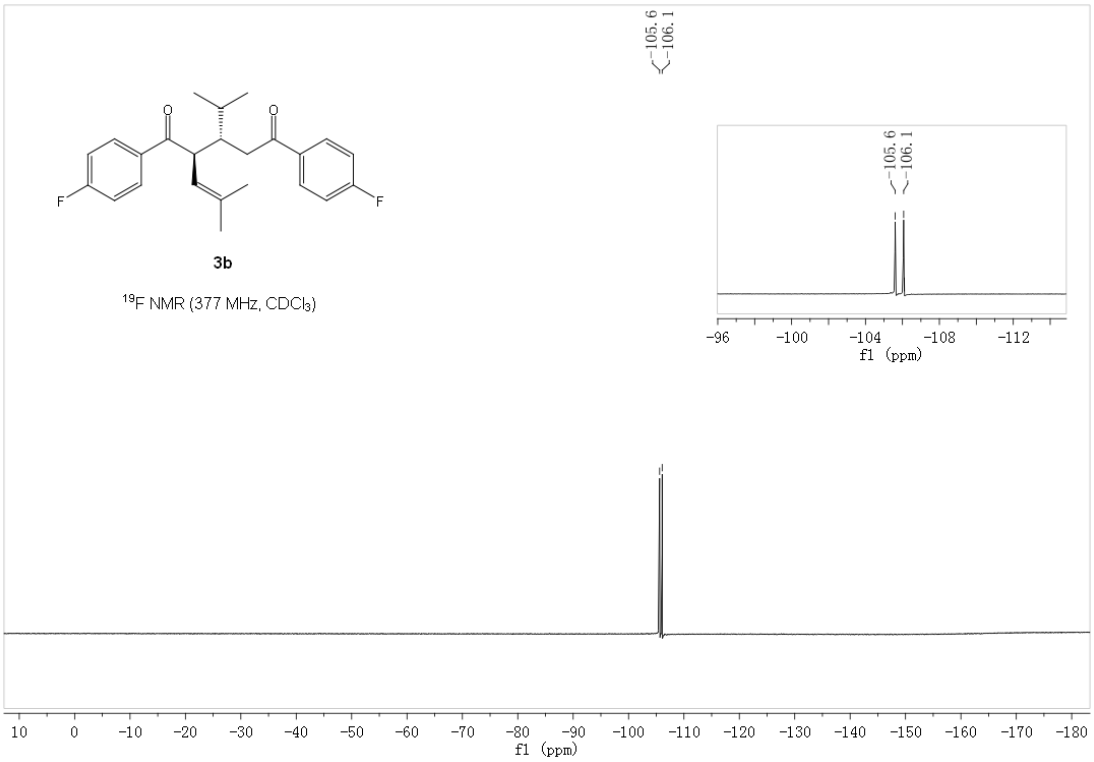
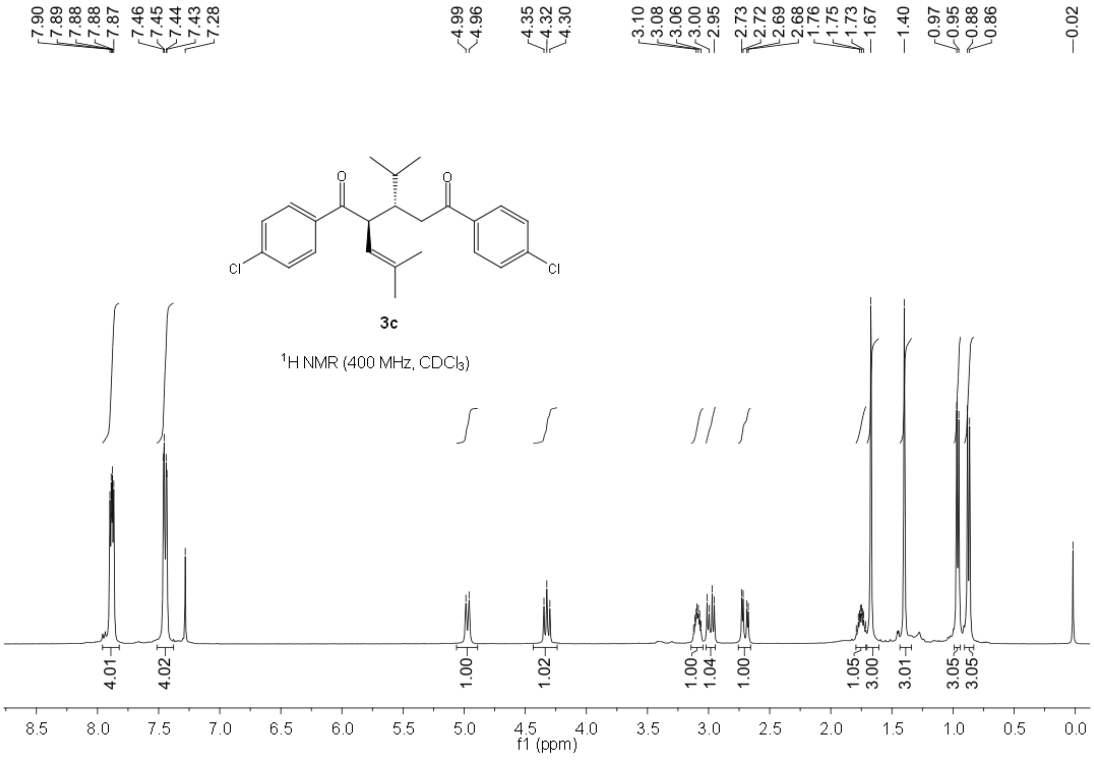
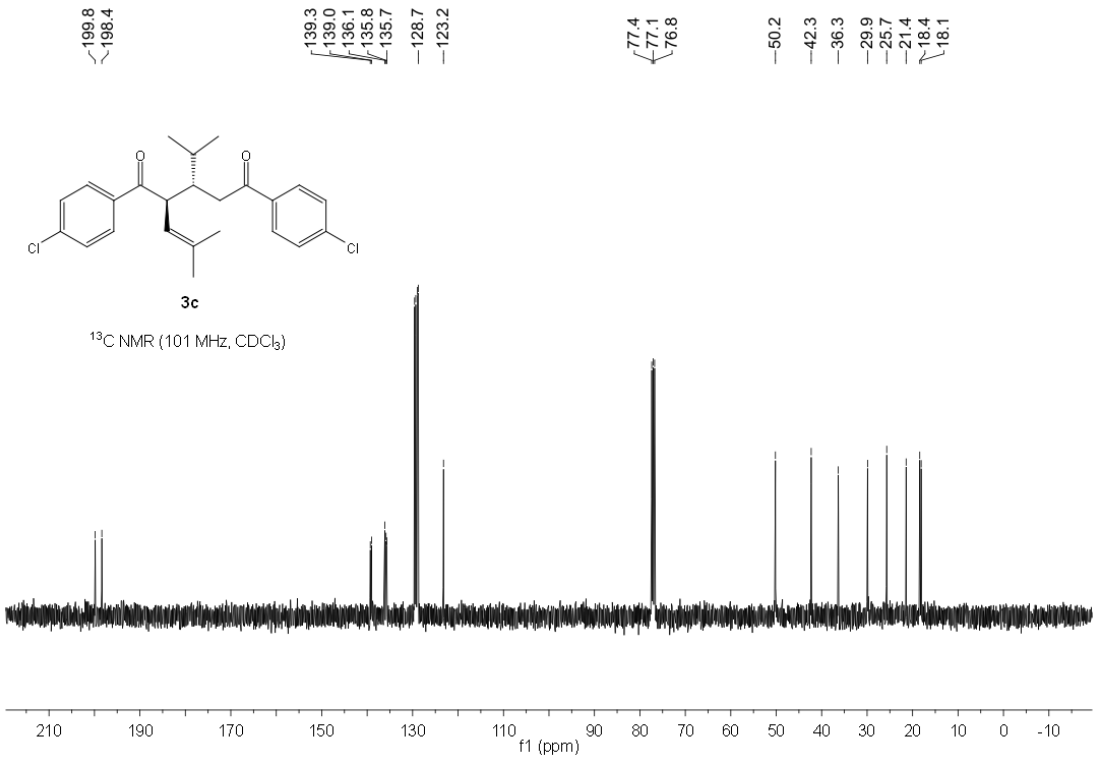
166.9
166.7
164.4
164.2
135.9
133.8
130.8
130.7
130.5
130.4
123.3
115.8
115.6
115.4
77.4
77.1
76.7
50.1
42.3
36.3
29.9
25.7
21.4
18.4
18.1
O
O
F
F
3b
13C NMR (151 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)


105.6
106.1
3b
19F NMR (377 MHz, CDCl3)
105.6
106.1
−96 −100 −104 −108 −112
f1 (ppm)
10 0 −10 −20 −30 −40 −50 −60 −70 −80 −90 −100 −110 −120 −130 −140 −150 −160 −170 −180
f1 (ppm)


7.90 7.89 7.88 7.88 7.87 7.46 7.45 7.44 7.43 7.28
4.99 4.96 4.35 4.32 4.30
3.10 3.08 3.06 3.00 2.95 2.73 2.72 2.69 2.68 1.76 1.75 1.73 1.67 1.40 0.97 0.95 0.88 0.86 0.02
3c
1H NMR (400 MHz, CDCl3)
4.01 4.02 1.00 1.02 1.00 1.04 1.00 1.05 3.00 3.01 3.05 3.05
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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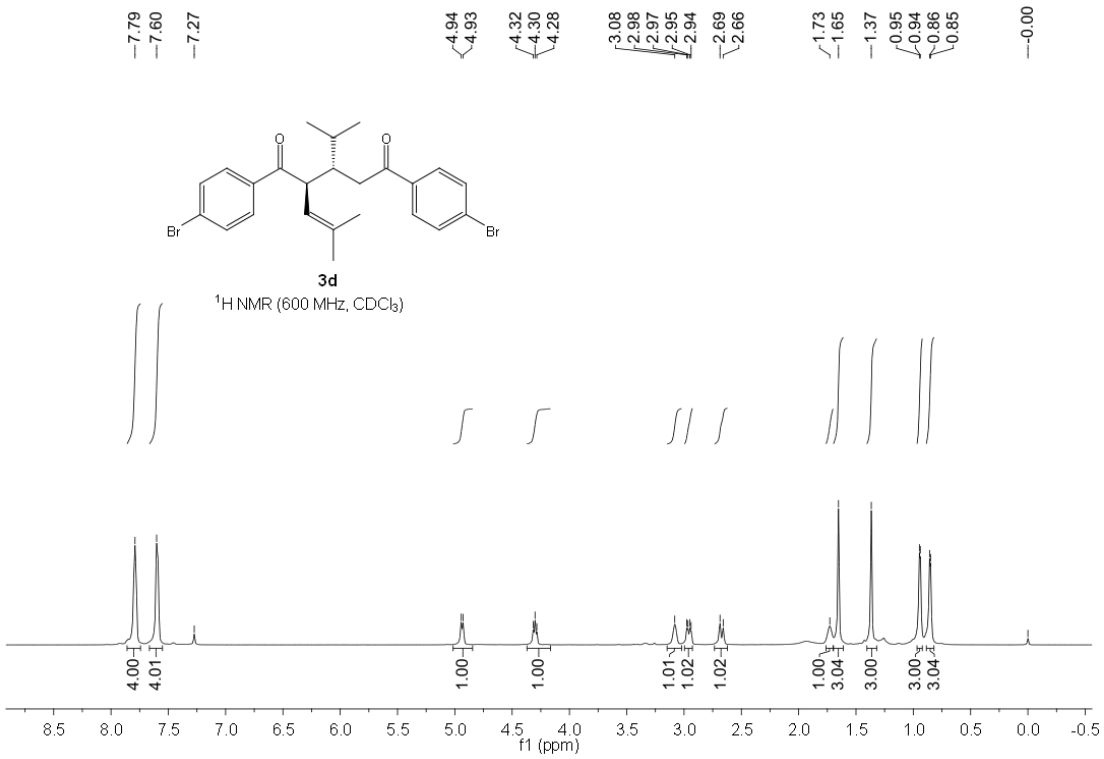
123.2
77.4
77.1
76.8
50.2
42.3
36.3
29.9
25.7
21.4
18.4
18.1
O
O
Cl
Cl
3c
13C NMR (101 MHz, CDCl3)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.60
7.27
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4.32
4.30
4.28
3.08
2.98
2.97
2.95
2.94
2.69
2.66
1.73
1.65
1.37
0.95
0.94
0.86
0.85
0.00
O
O
Br
Br
3d
1H NMR (600 MHz, CDCl3)
4.00
4.01
1.00
1.00
1.01
1.02
1.02
1.00
3.04
3.00
3.00
3.04
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
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2.0
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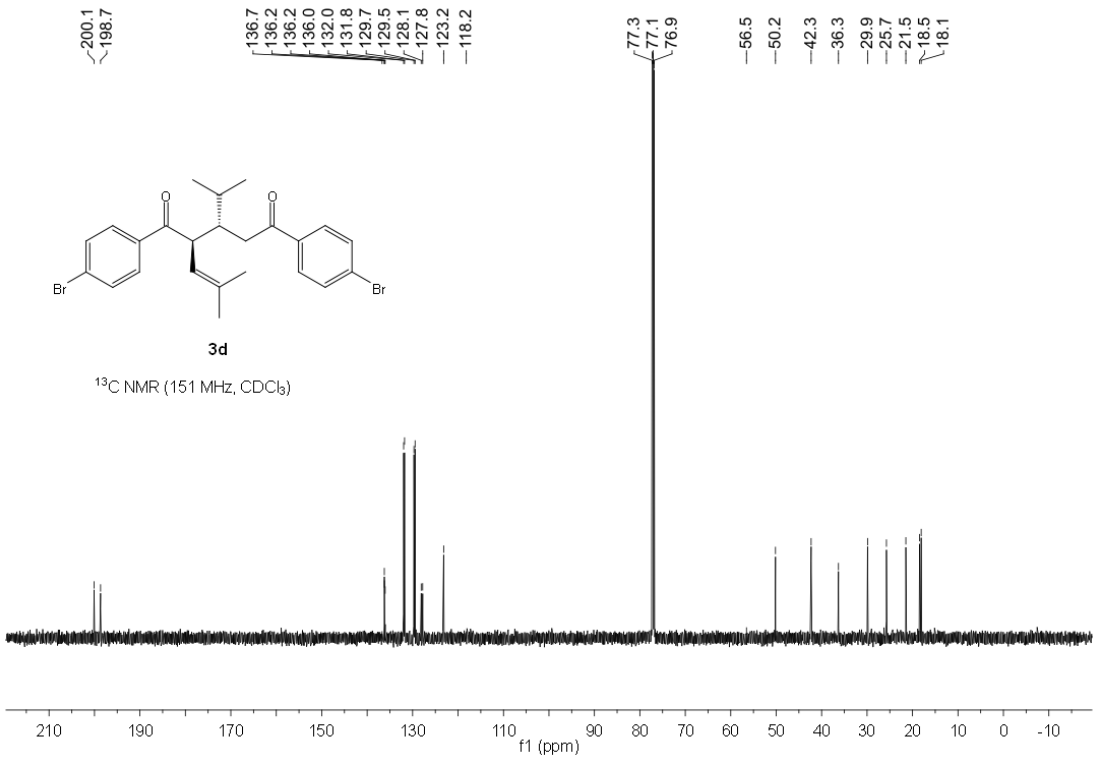
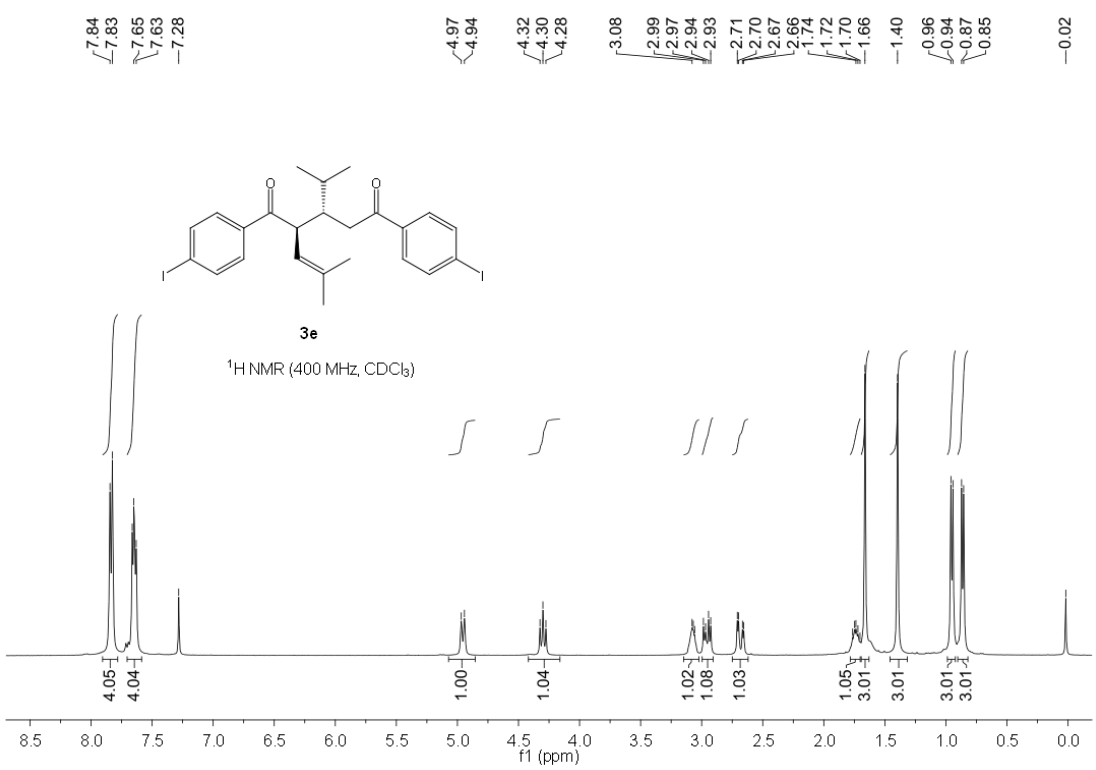
0.5
0.0
-0.5
f1 (ppm)



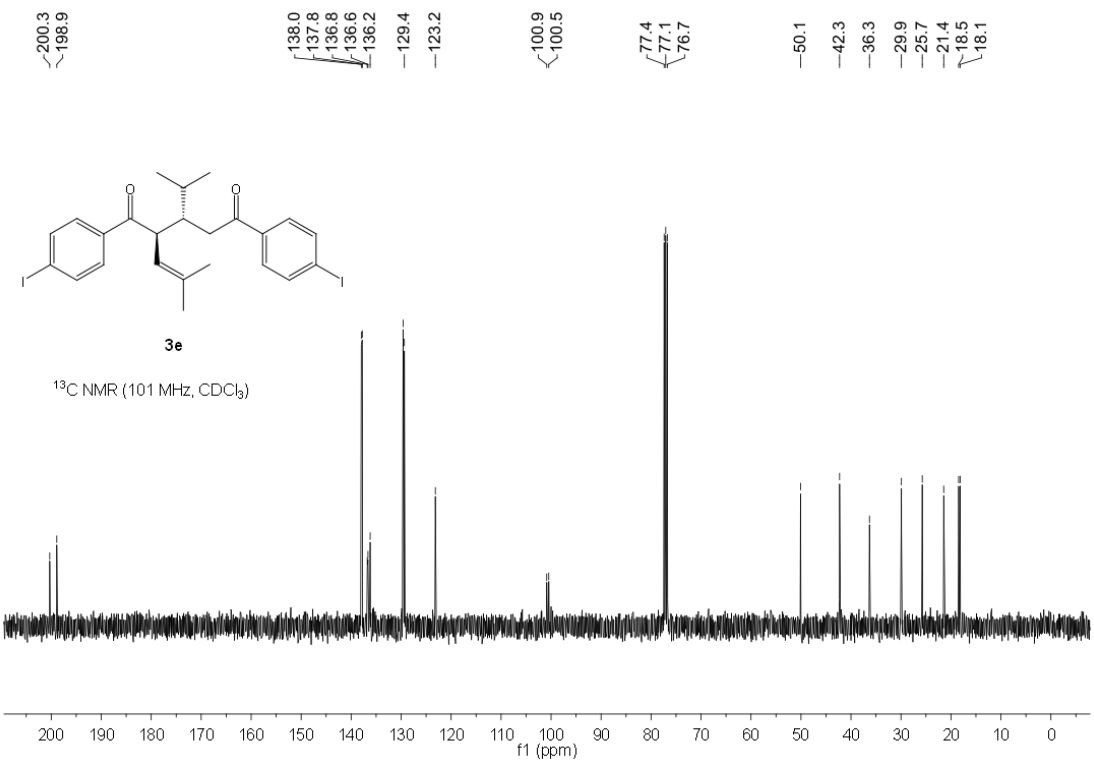
3d

^{13}C NMR (151 MHz, $CDCl_3$)



3e

^{1}H NMR (400 MHz, $CDCl_3$)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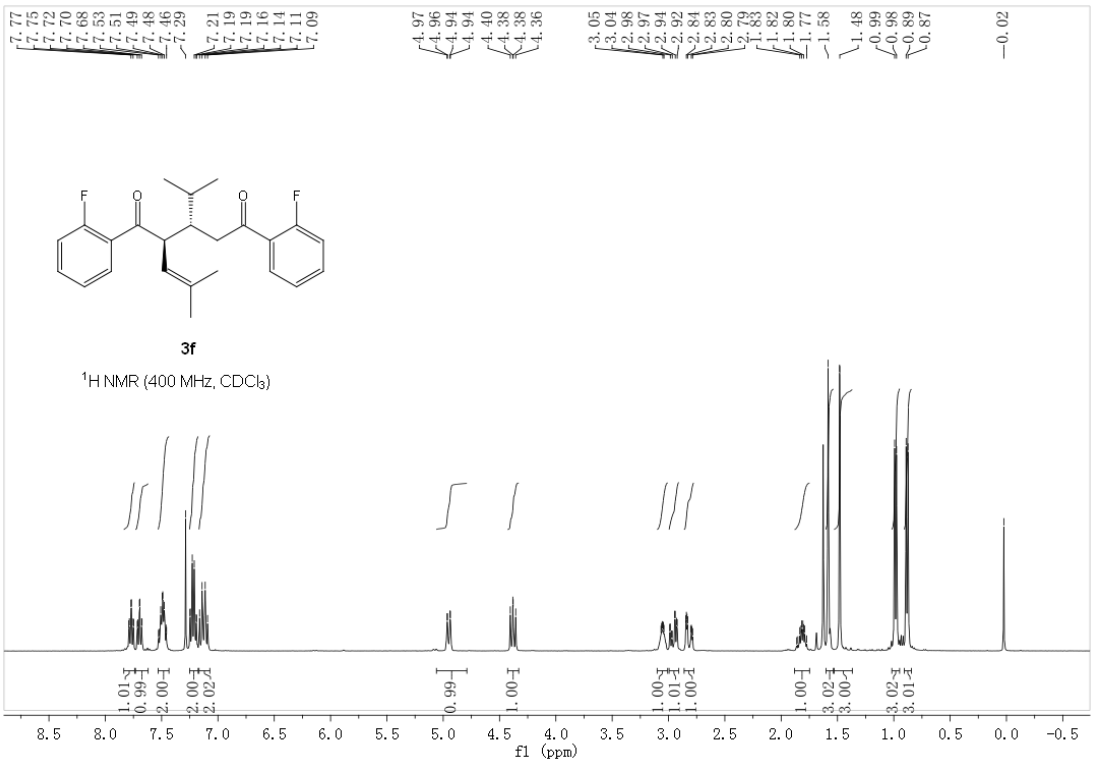
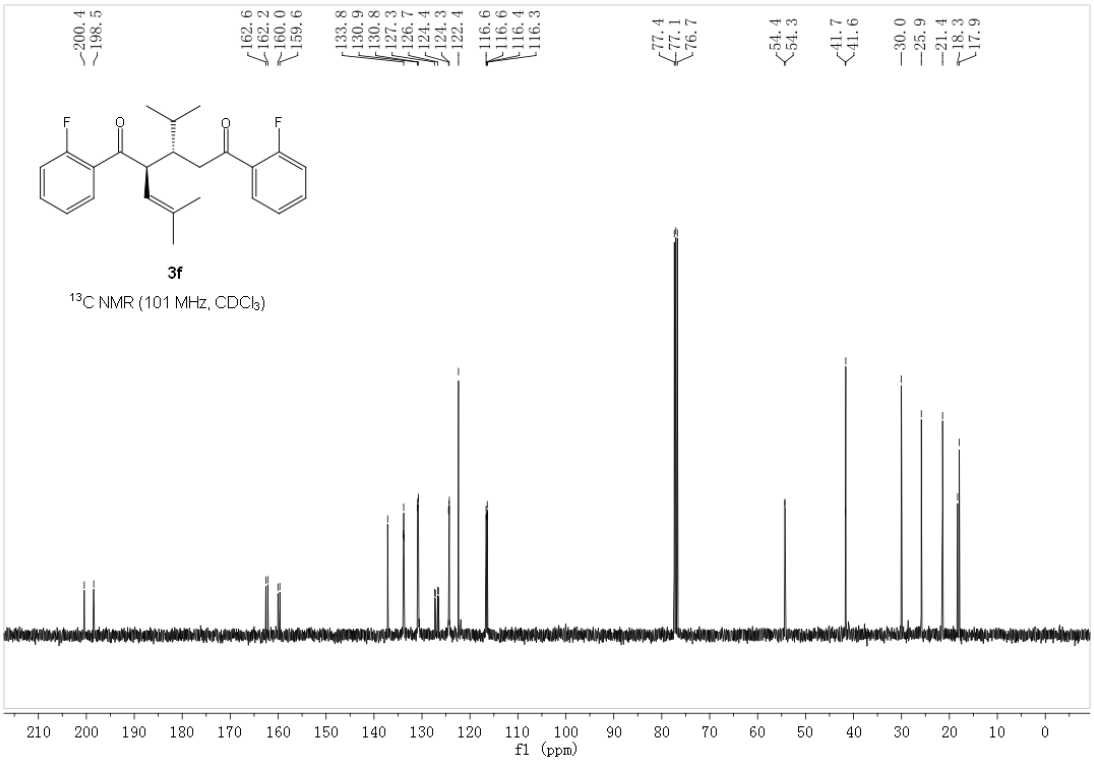
136.2
129.4
123.2
100.9
100.5
77.4
77.1
76.7
50.1
42.3
36.3
29.9
25.7
21.4
18.5
18.1
3e
13C NMR (101 MHz, CDCl3)
f1 (ppm)


3f
1H NMR (400 MHz, CDCl3)
f1 (ppm)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127.3
126.7
124.4
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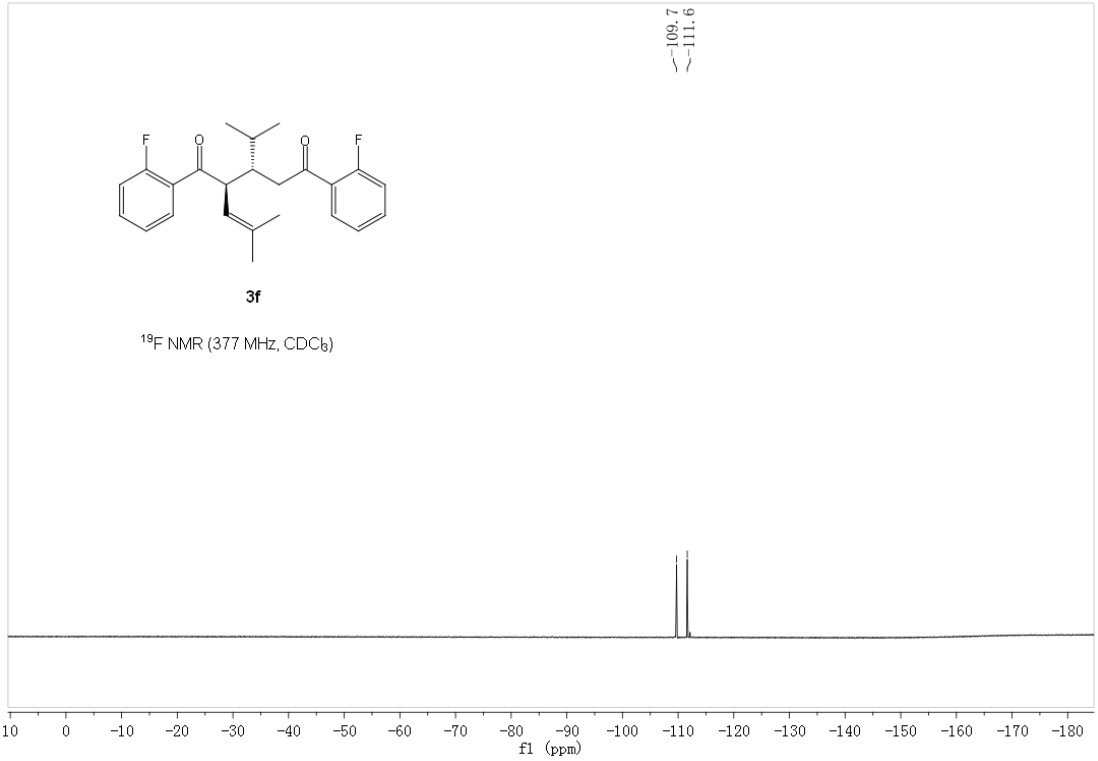
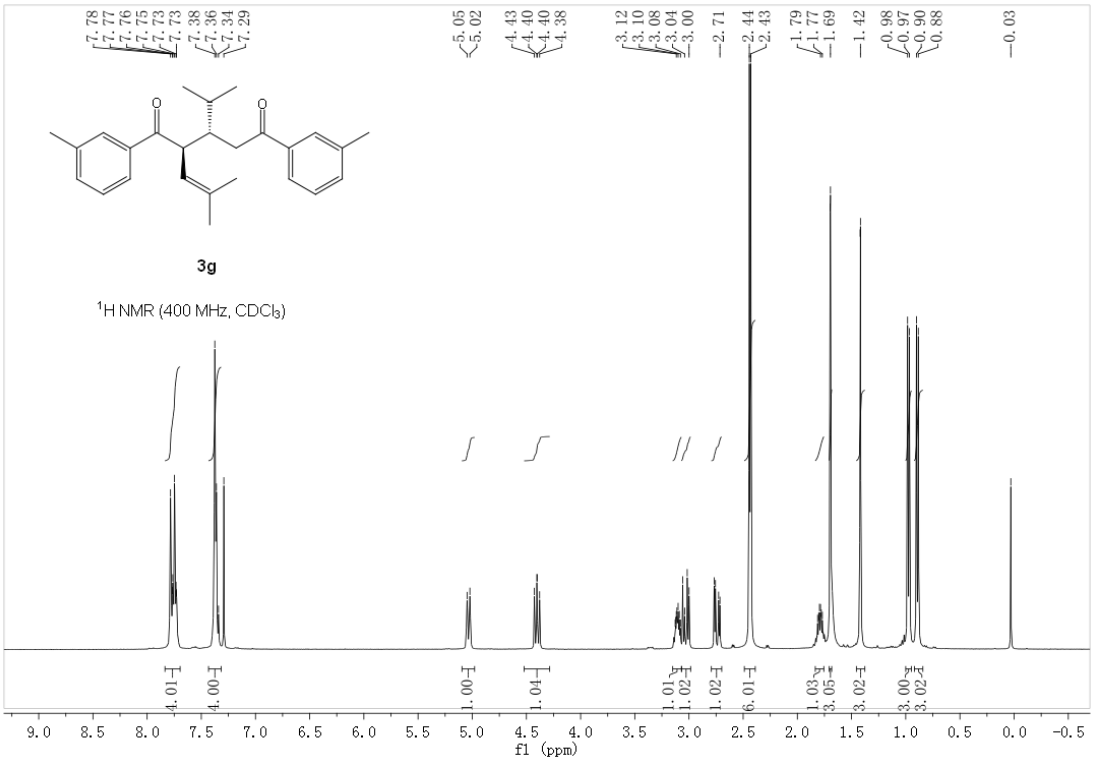
122.4
116.6
116.6
116.4
116.3
77.4
77.1
76.7
54.4
54.3
41.7
41.6
30.0
25.9
21.4
18.3
17.9
F
O
O
F
3f
13C NMR (101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


109.7
111.6
F
O
O
F
3f
19F NMR (377 MHz, CDCl3)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180
f1 (ppm)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3.08
3.04
3.00
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2.44
2.43
1.79
1.77
1.69
1.42
0.98
0.97
0.90
0.88
0.03
3g
1H NMR (400 MHz, CDCl3)
4.01
4.00
1.00
1.04
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1.02
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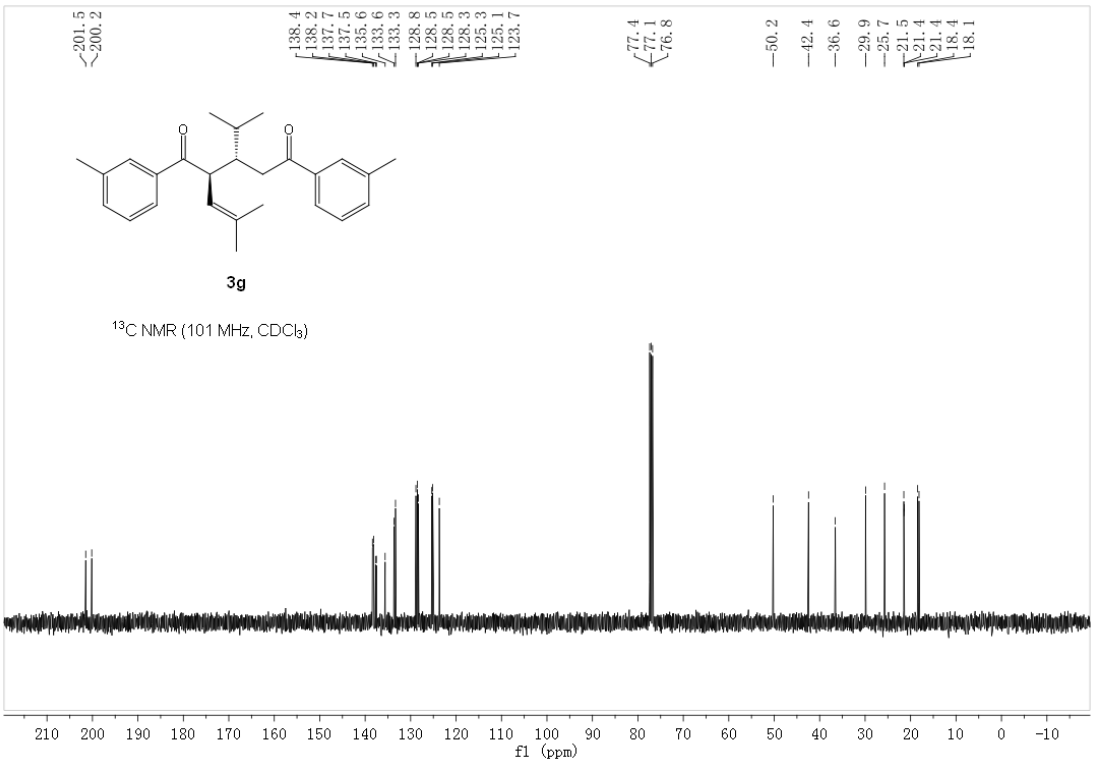
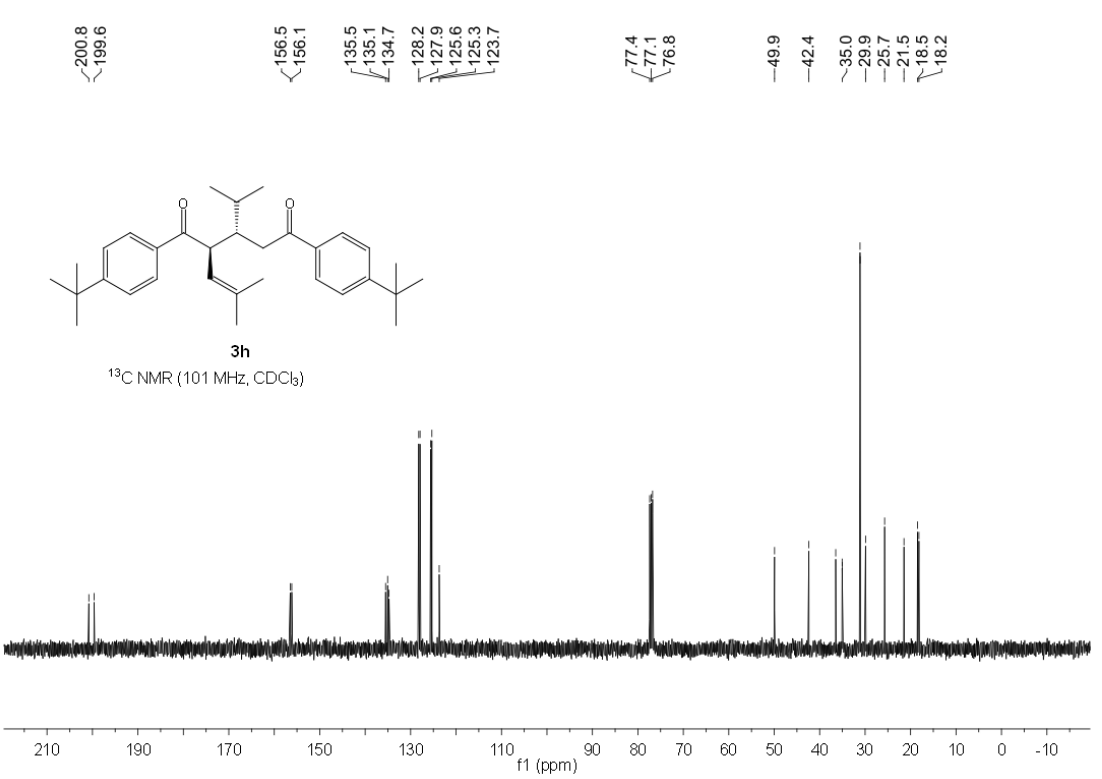
3.05
3.02
3.00
3.02
f1 (ppm)


201.5
200.2
138.4
138.2
137.7
137.5
135.6
133.6
133.3
128.8
128.5
128.5
128.3
125.3
125.1
123.7
77.4
77.1
76.8
50.2
42.4
36.6
29.9
25.7
21.5
21.4
21.4
18.4
18.1
3g
13C NMR (101 MHz, CDCl3)
f1 (ppm)

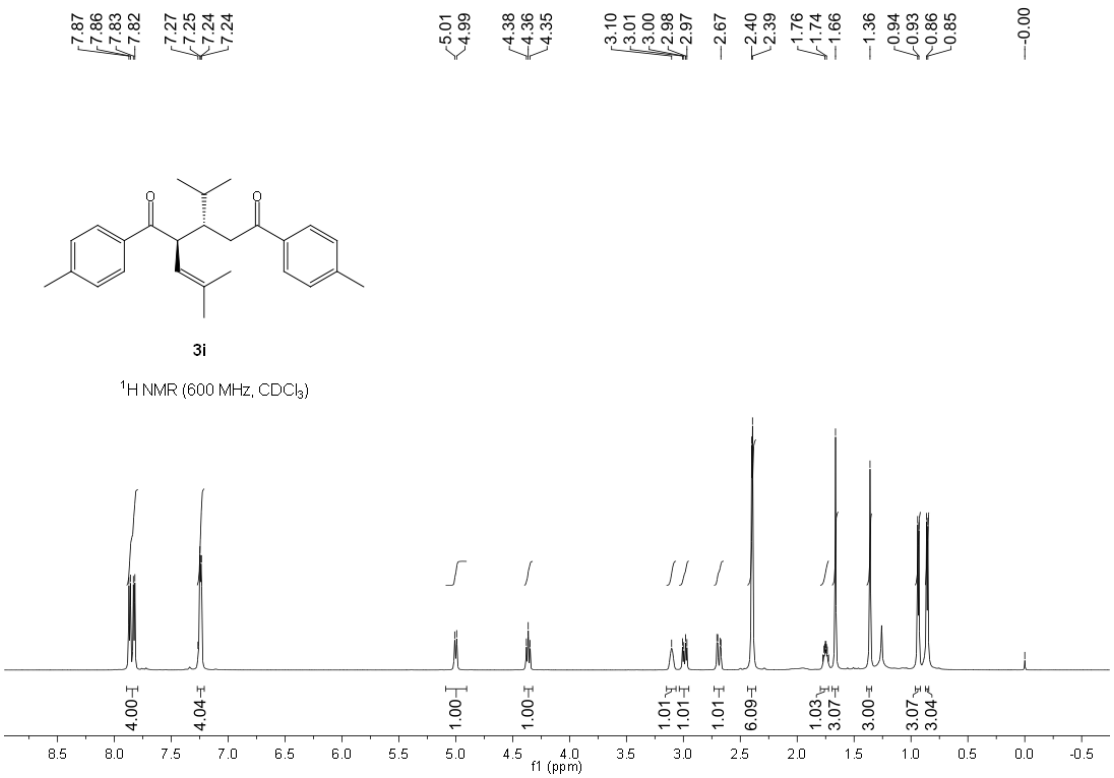
3h

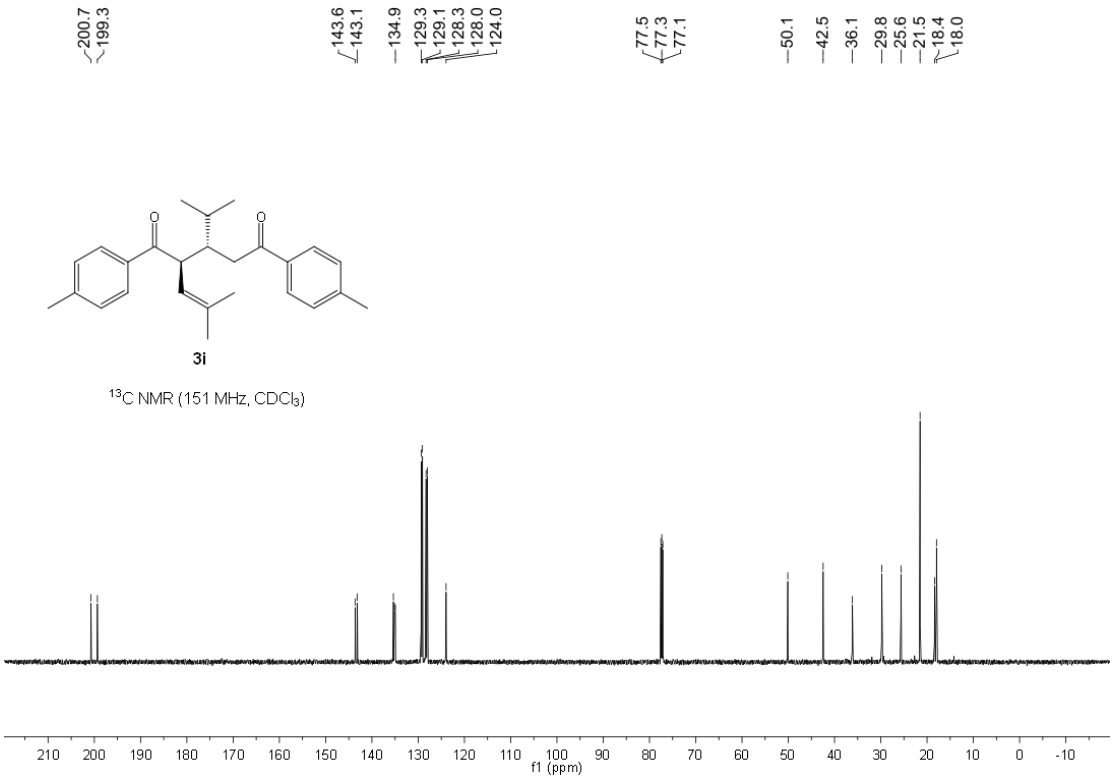
^{1}H NMR (400 MHz, $CDCl_3$)

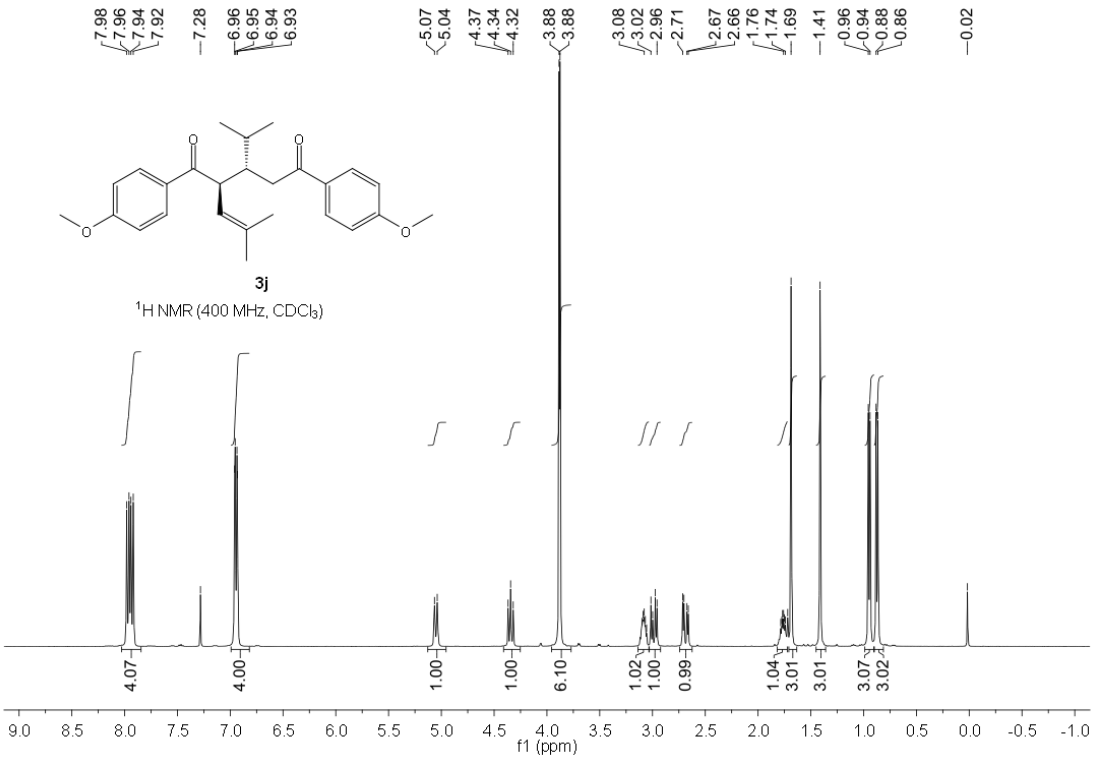


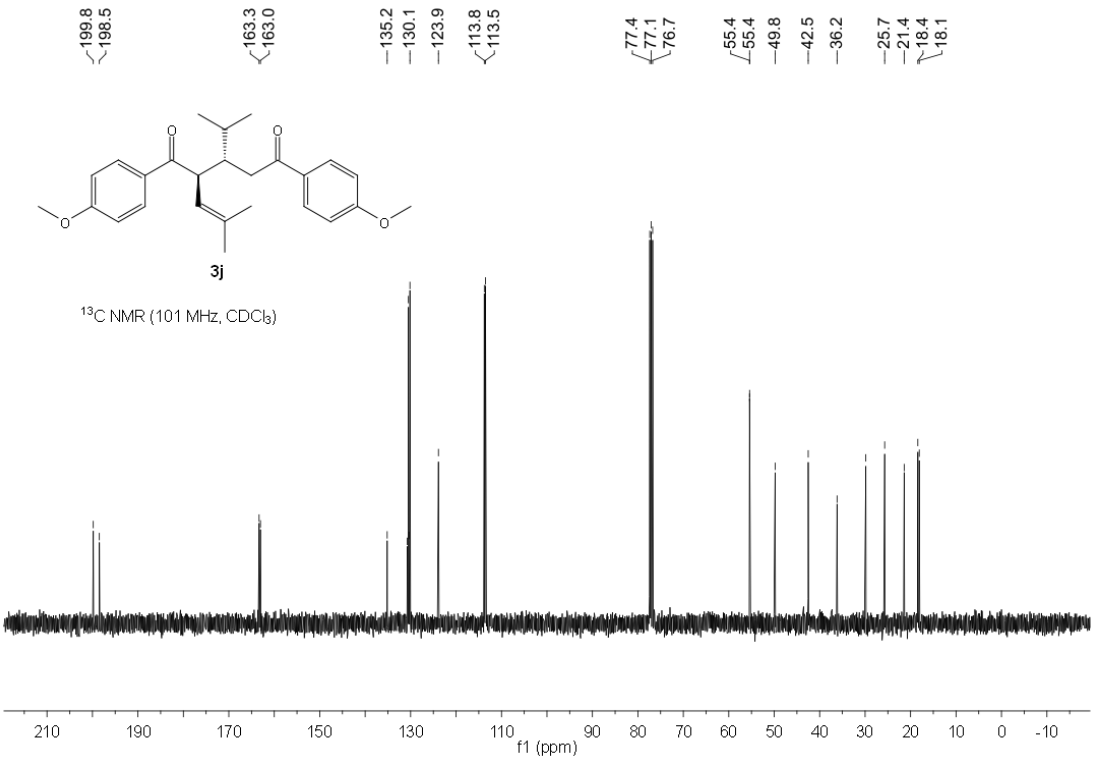
3h

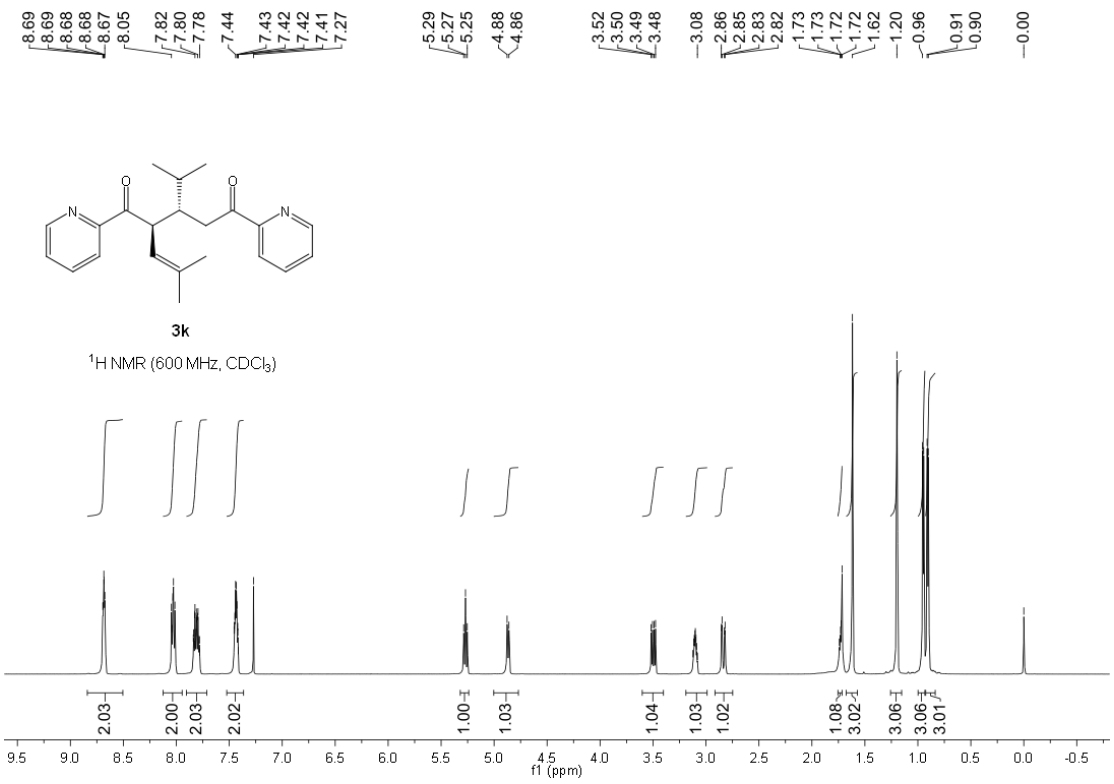
^{13}C NMR (101 MHz, $CDCl_3$)


7.87
7.86
7.83
7.82
7.27
7.25
7.24
7.24
5.01
4.99
4.38
4.36
4.35
3.10
3.01
3.00
2.98
2.97
2.67
2.40
2.39
1.76
1.74
1.66
1.36
0.94
0.93
0.86
0.85
0.00
O
O
3i
1H NMR (600 MHz, CDCl3)
4.00
4.04
1.00
1.00
1.01
1.01
1.01
6.09
1.03
3.07
3.00
3.07
3.04
f1 (ppm)


200.7
199.3
143.6
143.1
134.9
129.3
129.1
128.3
128.0
124.0
77.5
77.3
77.1
50.1
42.5
36.1
29.8
25.6
21.5
18.4
18.0
O
O
3i
13C NMR (151 MHz, CDCl3)
f1 (ppm)

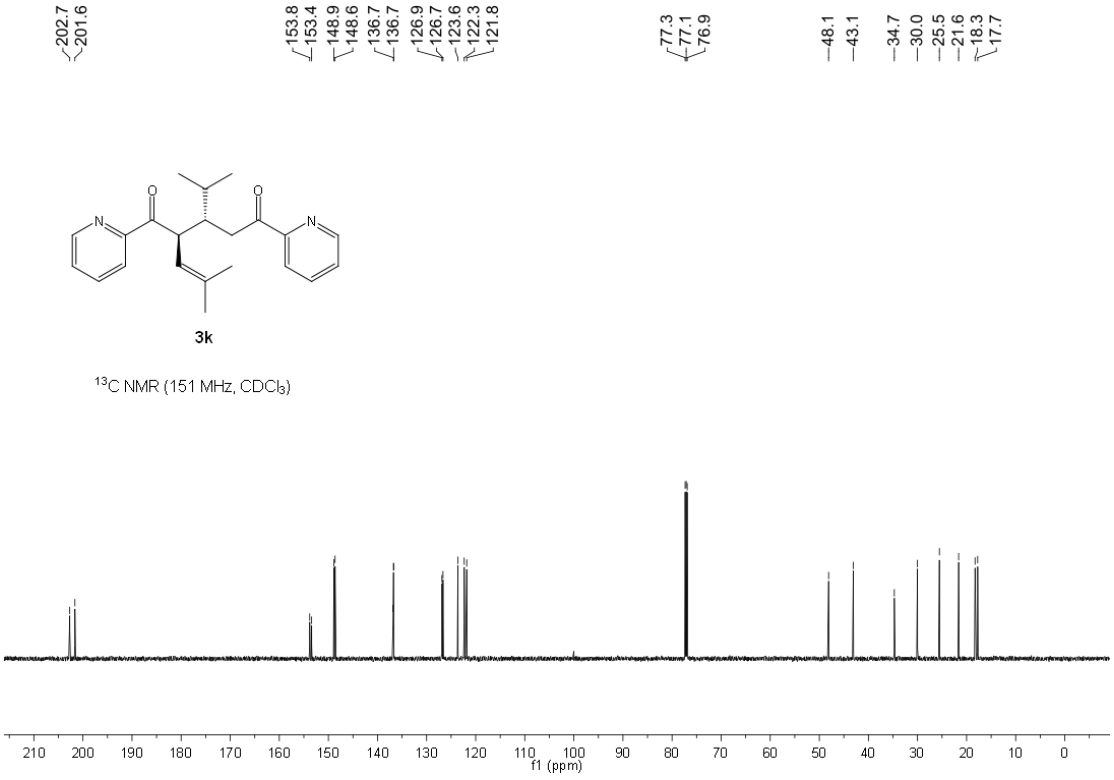

7.98
7.96
7.94
7.92
7.28
6.96
6.95
6.94
6.93
5.07
5.04
4.37
4.34
4.32
3.88
3.08
3.02
2.96
2.71
2.67
2.66
1.76
1.74
1.69
1.41
0.96
0.94
0.88
0.86
0.02
3j
1H NMR (400 MHz, CDCl3)
4.07
4.00
1.00
1.00
6.10
1.02
1.00
0.99
1.04
3.01
3.01
3.07
3.02
f1 (ppm)


199.8
198.5
163.3
163.0
135.2
130.1
123.9
113.8
113.5
77.4
77.1
76.7
55.4
55.4
49.8
42.5
36.2
25.7
21.4
18.4
18.1
3j
13C NMR (101 MHz, CDCl3)
f1 (ppm)



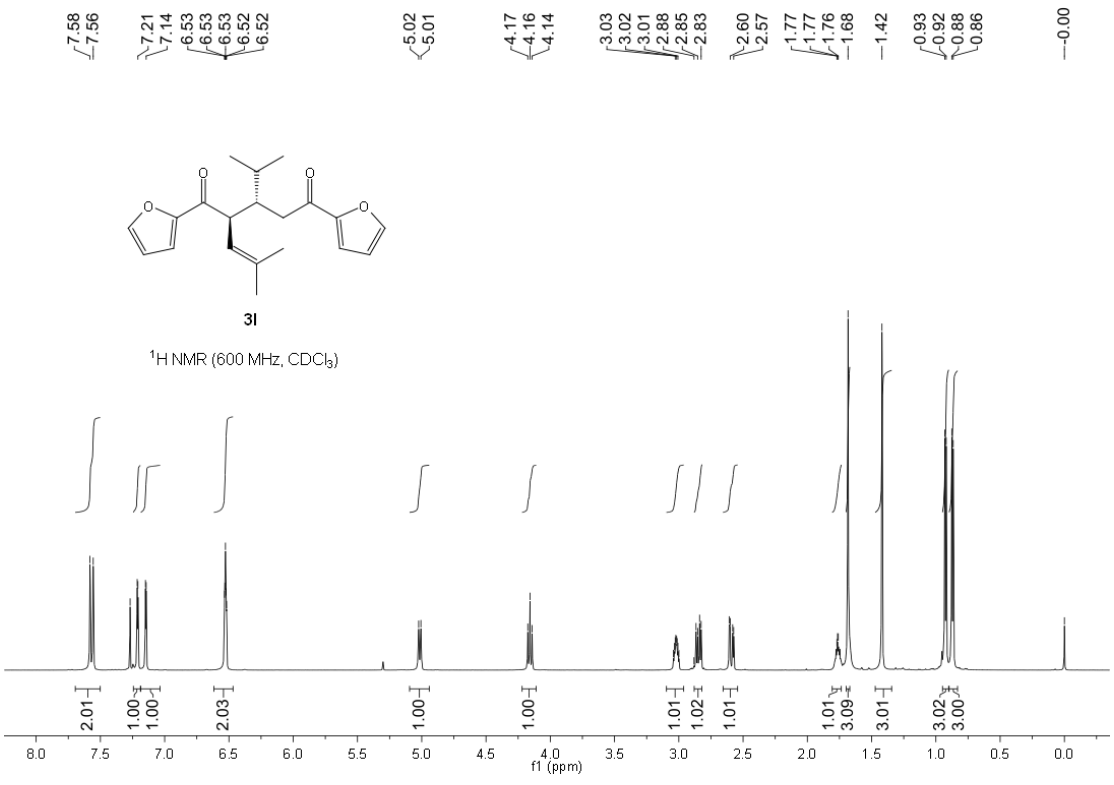
3k

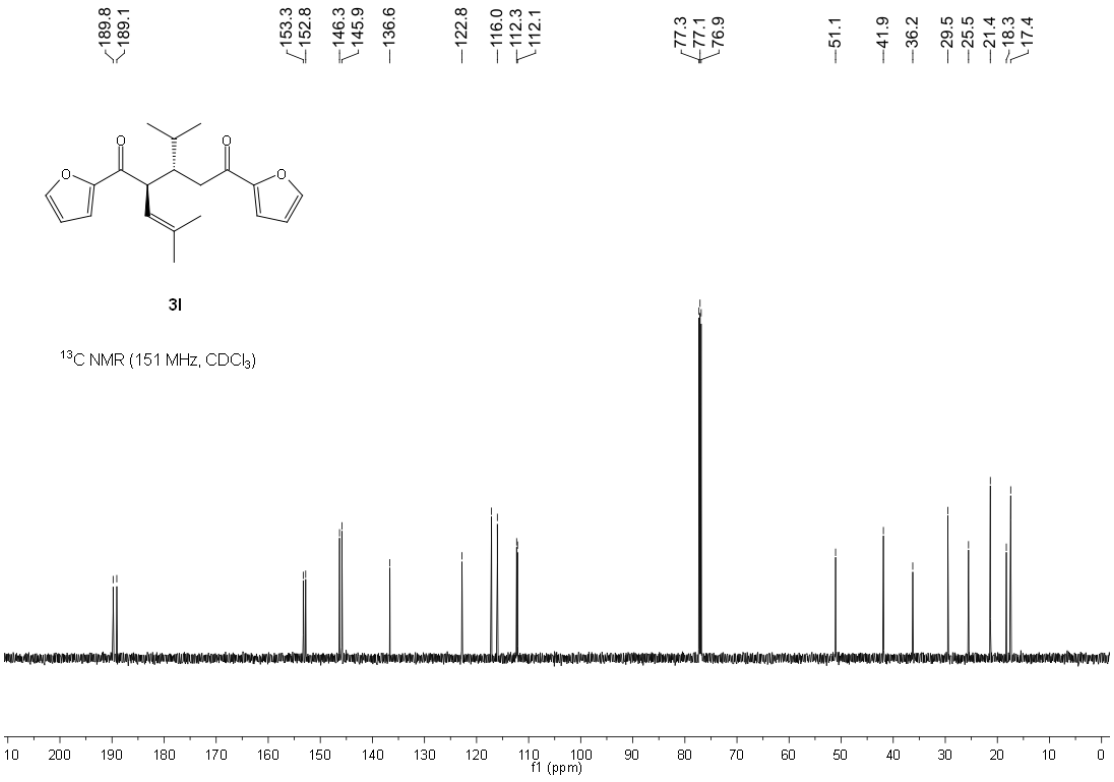
^{1}H NMR (600 MHz, $CDCl_3$)

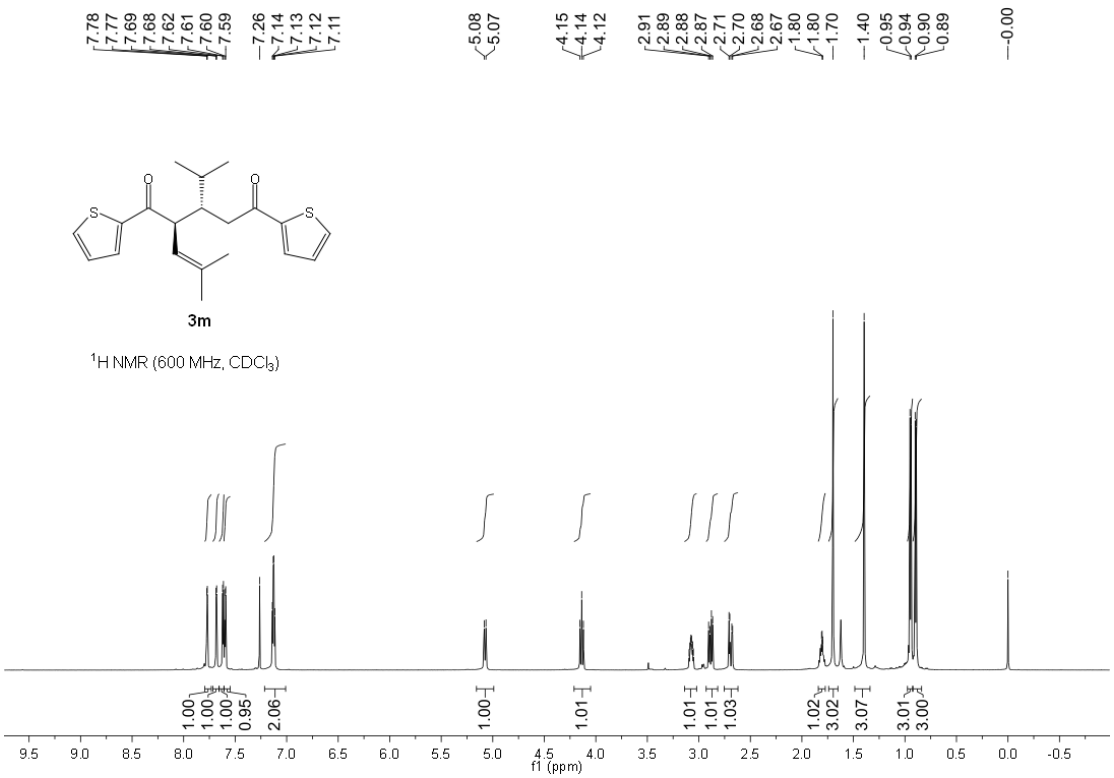


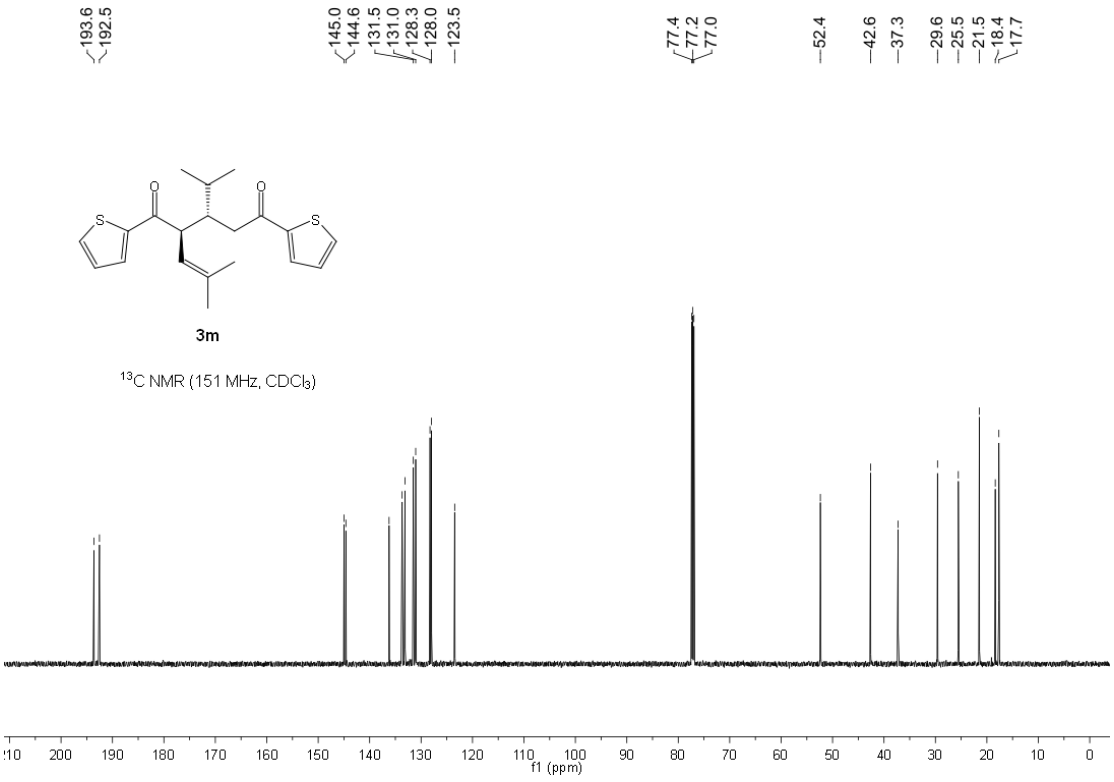
3k

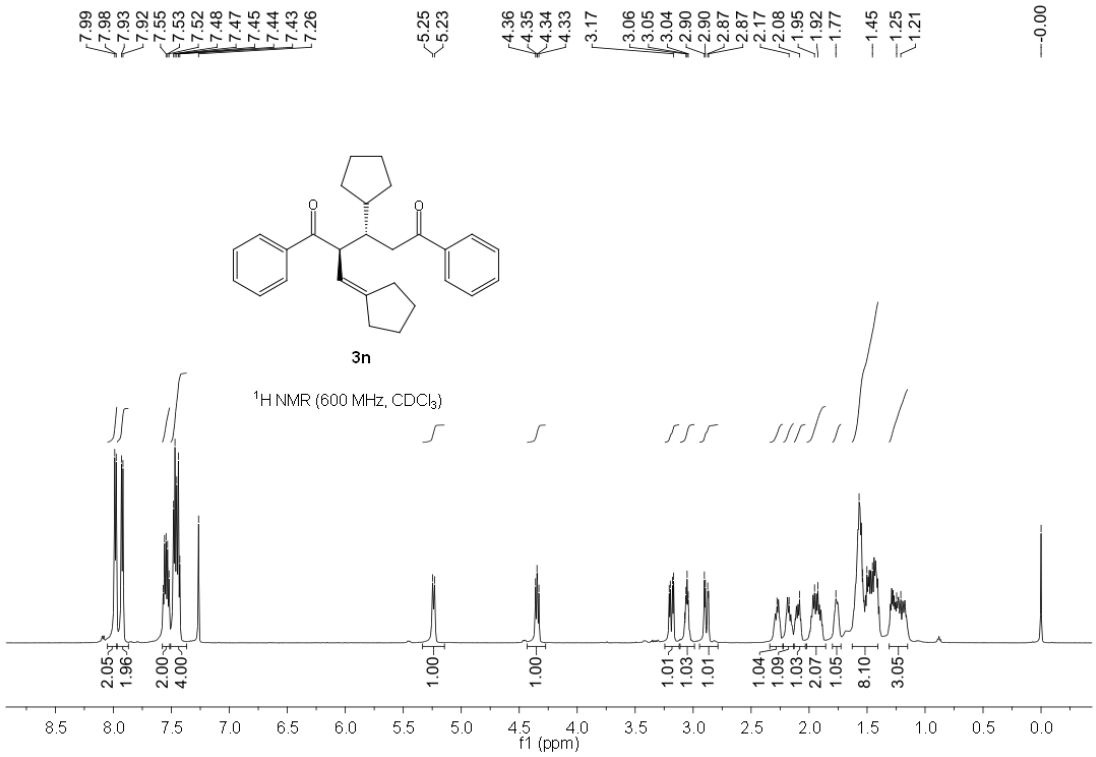
^{13}C NMR (151 MHz, $CDCl_3$)

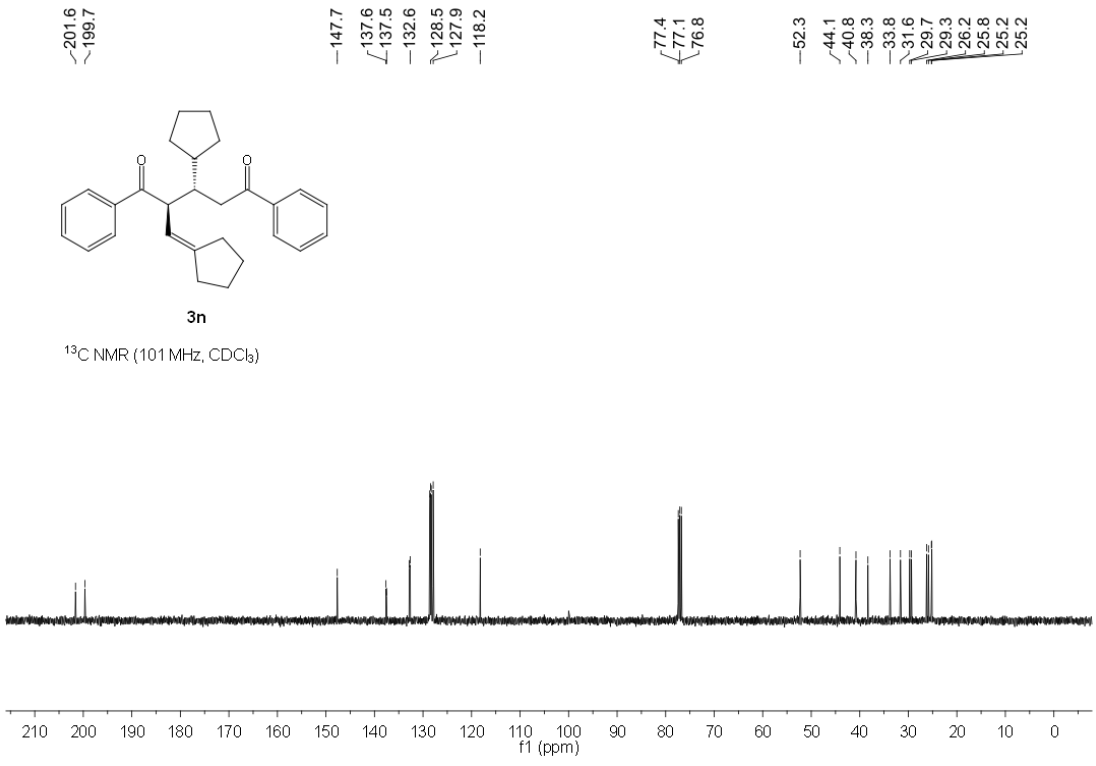

7.58
7.56
7.21
7.14
6.53
6.53
6.53
6.52
6.52
5.02
5.01
4.17
4.16
4.14
3.03
3.02
3.01
2.88
2.85
2.83
2.60
2.57
1.77
1.77
1.76
1.68
1.42
0.93
0.92
0.88
0.86
0.00
O
O
O
O
3l
^{1}H NMR (600 MHz, $CDCl_3$)
2.01
1.00
1.00
2.03
1.00
1.00
1.01
1.02
1.01
1.01
3.09
3.01
3.02
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

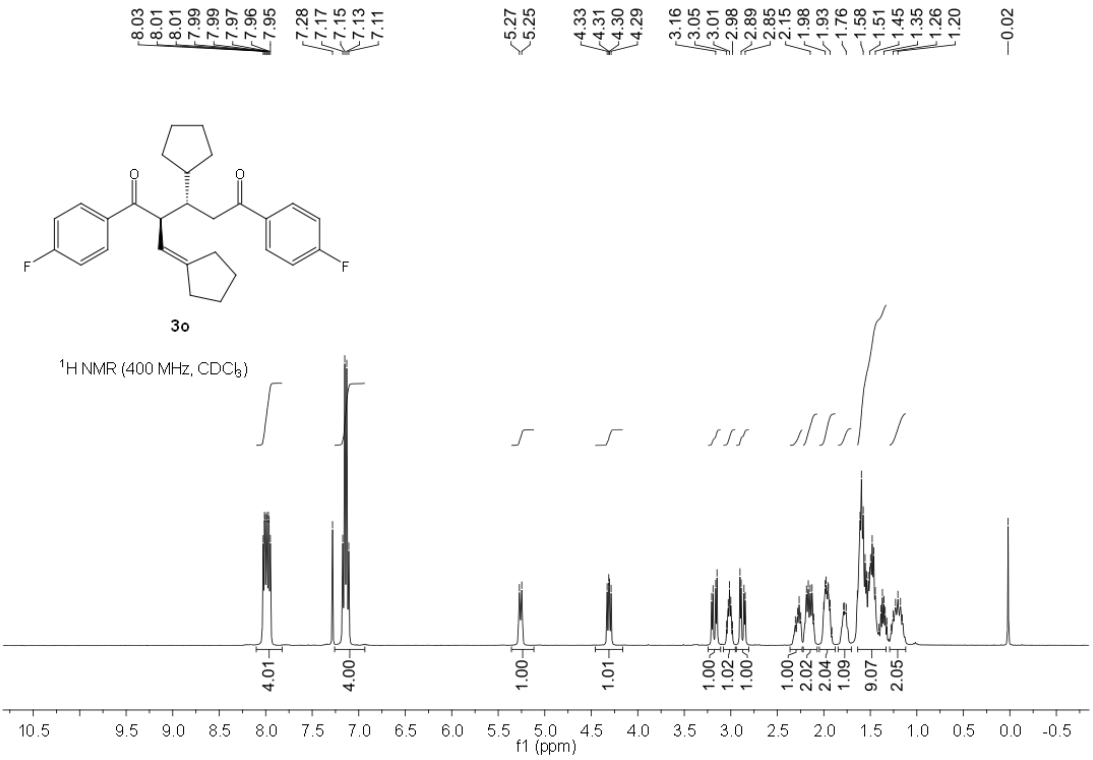

189.8
189.1
153.3
152.8
146.3
145.9
136.6
122.8
116.0
112.3
112.1
77.3
77.1
76.9
51.1
41.9
36.2
29.5
25.5
21.4
18.3
17.4
O
O
O
O
3l
^{13}C NMR (151 MHz, $CDCl_3$)
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

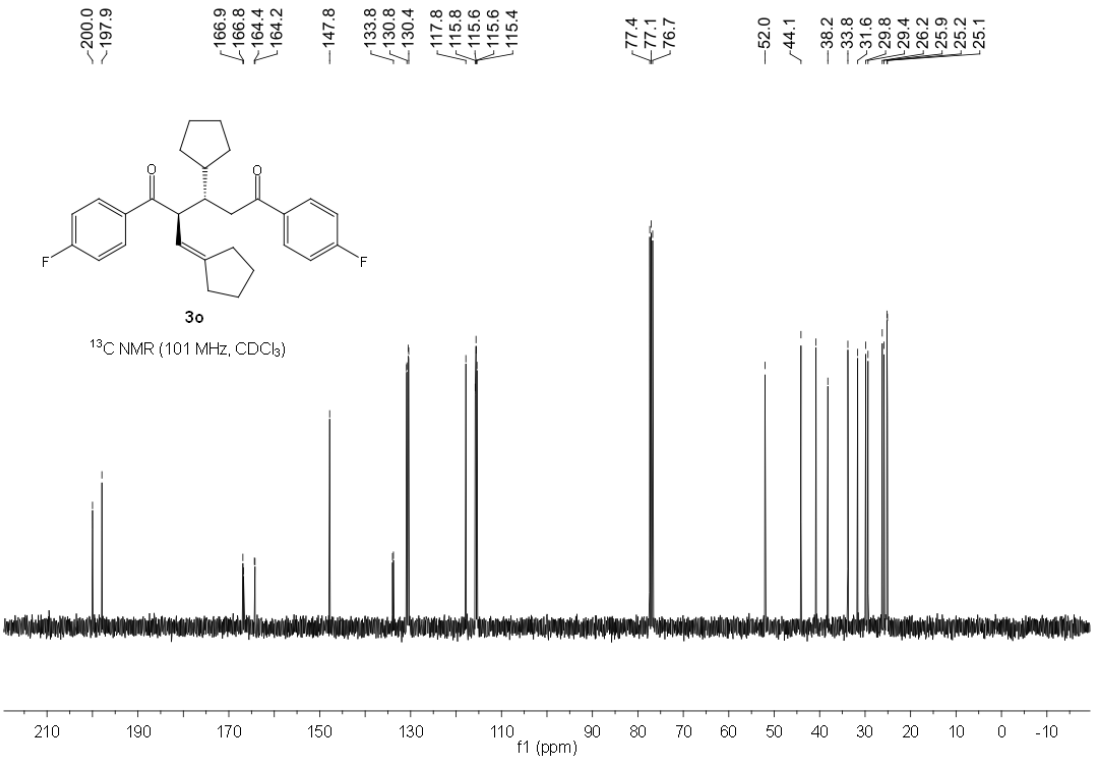

7.78
7.77
7.69
7.68
7.62
7.61
7.60
7.59
7.26
7.14
7.13
7.12
7.11
5.08
5.07
4.15
4.14
4.12
2.91
2.89
2.88
2.87
2.71
2.70
2.68
2.67
1.80
1.80
1.70
1.40
0.95
0.94
0.90
0.89
0.00
3m
1H NMR (600 MHz, CDCl3)
1.00
1.00
1.00
0.95
2.06
1.00
1.01
1.01
1.01
1.03
1.02
3.02
3.07
3.01
3.00
f1 (ppm)

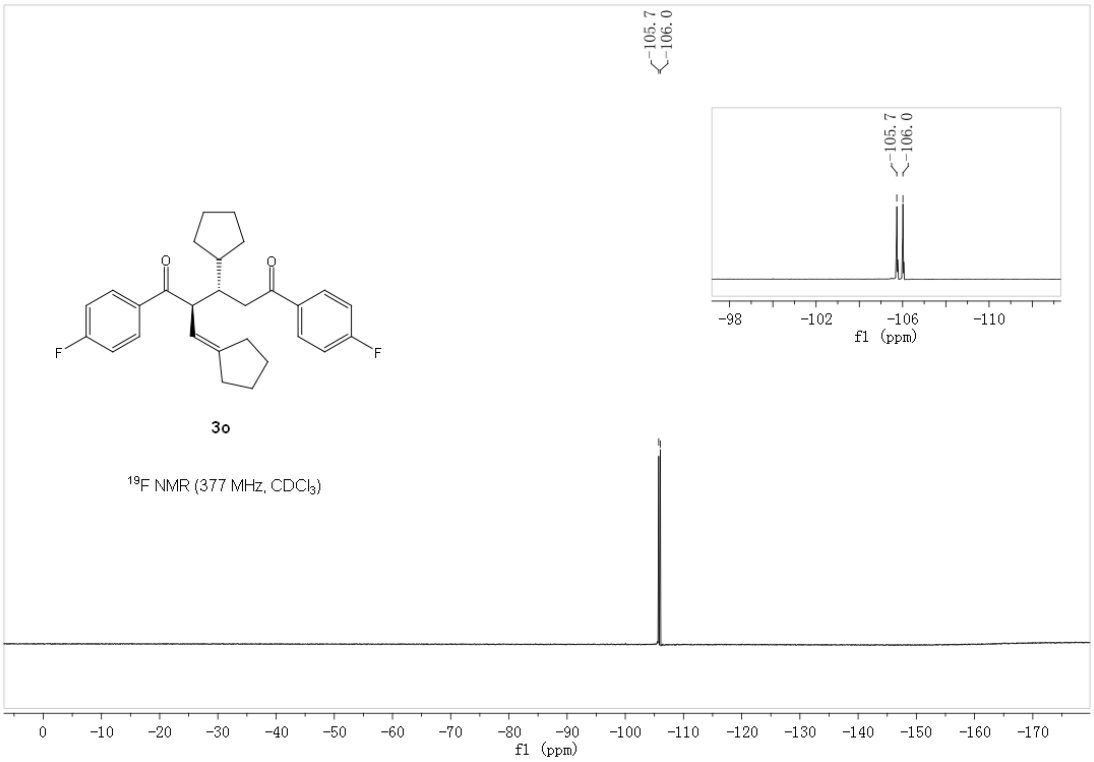

193.6
192.5
145.0
144.6
131.5
131.0
128.3
128.0
123.5
77.4
77.2
77.0
52.4
42.6
37.3
29.6
25.5
21.5
18.4
17.7
3m
13C NMR (151 MHz, CDCl3)
f1 (ppm)

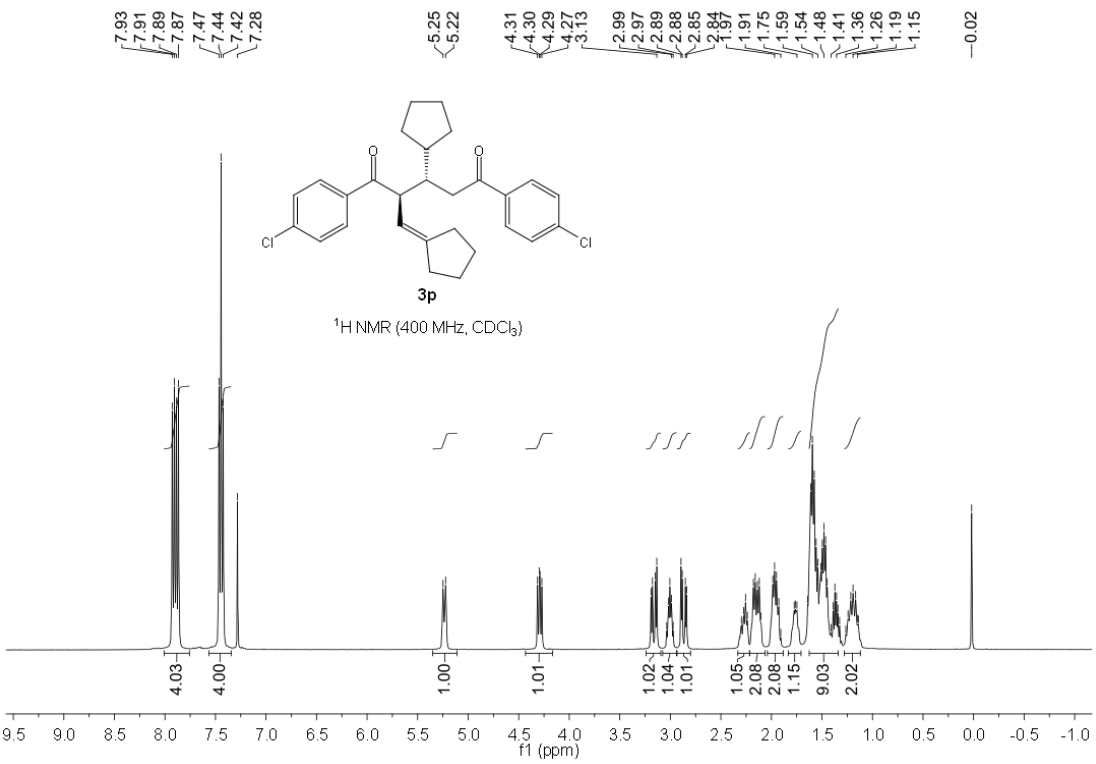

7.99
7.98
7.93
7.92
7.55
7.53
7.52
7.48
7.47
7.45
7.44
7.43
7.26
5.25
5.23
4.36
4.35
4.34
4.33
3.17
3.06
3.05
3.04
2.90
2.90
2.87
2.87
2.17
2.08
1.95
1.92
1.77
1.45
1.25
1.21
0.00
O
O
3n
1H NMR (600 MHz, CDCl3)
2.05
1.96
2.00
4.00
1.00
1.00
1.01
1.03
1.01
1.04
1.09
1.03
2.07
1.05
8.10
3.05
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

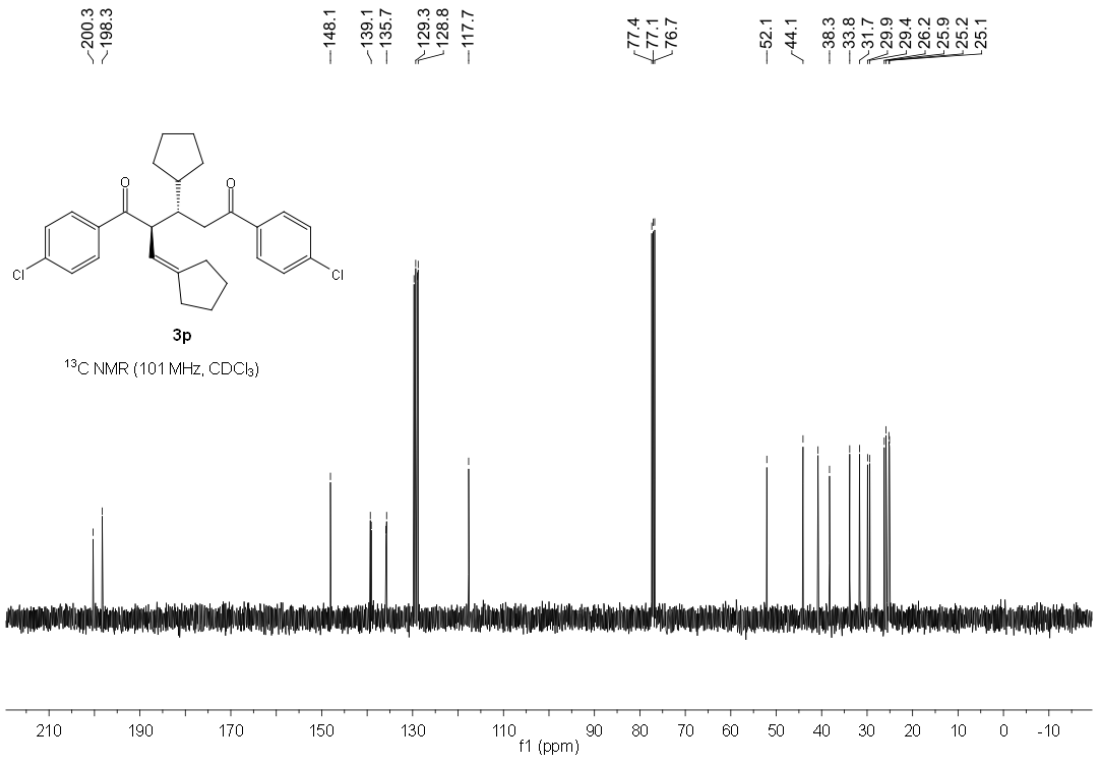

201.6
199.7
147.7
137.6
137.5
132.6
128.5
127.9
118.2
77.4
77.1
76.8
52.3
44.1
40.8
38.3
33.8
31.6
29.7
29.3
26.2
25.8
25.2
25.2
O
O
3n
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.03
8.01
8.01
7.99
7.99
7.97
7.96
7.95
7.28
7.17
7.15
7.13
7.11
5.27
5.25
4.33
4.31
4.30
4.29
3.16
3.05
3.01
2.98
2.89
2.85
2.15
1.98
1.93
1.76
1.58
1.51
1.45
1.35
1.26
1.20
0.02
O
O
F
F
3o
1H NMR (400 MHz, CDCl3)
4.01
4.00
1.00
1.01
1.00
1.02
1.00
1.00
2.02
2.04
1.09
9.07
2.05
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


200.0
197.9
166.9
166.8
164.4
164.2
147.8
133.8
130.8
130.4
117.8
115.8
115.6
115.6
115.4
77.4
77.1
76.7
52.0
44.1
38.2
33.8
31.6
29.8
29.4
26.2
25.9
25.2
25.1
O
O
F
F
3o
13C NMR (101 MHz, CDCl3)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

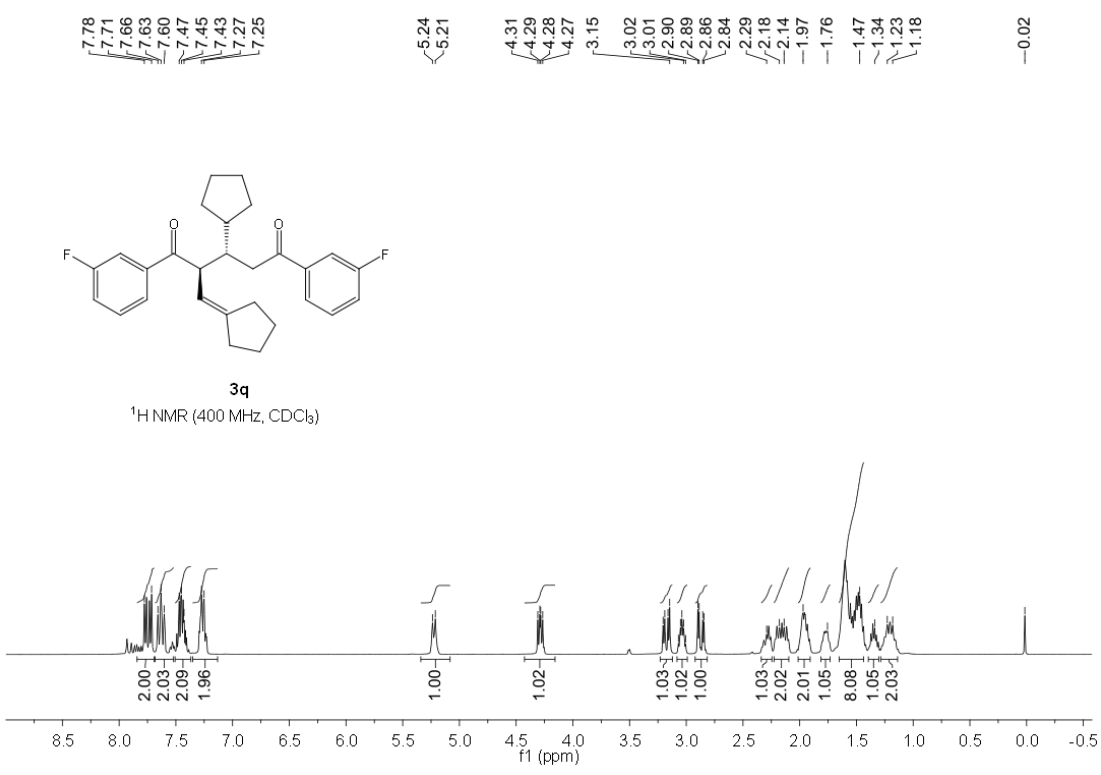

-105.7
-106.0
-105.7
-106.0
-98
-102
-106
-110
f1 (ppm)
F
F
O
O
3o
^{19}F NMR (377 MHz, $CDCl_3$)
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
f1 (ppm)


7.93
7.91
7.89
7.87
7.47
7.44
7.42
7.28
5.25
5.22
4.31
4.30
4.29
4.27
3.13
2.99
2.97
2.89
2.88
2.85
2.84
1.97
1.91
1.75
1.59
1.54
1.48
1.41
1.36
1.26
1.19
1.15
0.02
Cl
Cl
O
O
3p
^{1}H NMR (400 MHz, $CDCl_3$)
4.03
4.00
1.00
1.01
1.02
1.04
1.01
1.05
2.08
2.08
1.15
9.03
2.02
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)



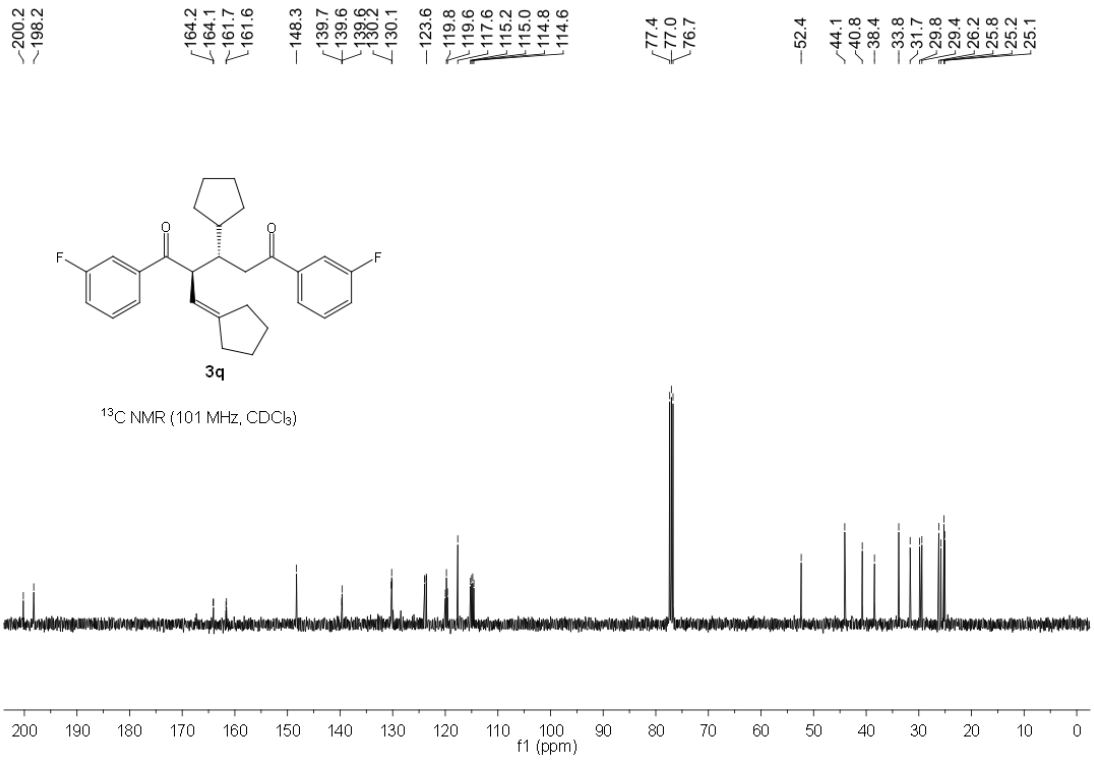
3p

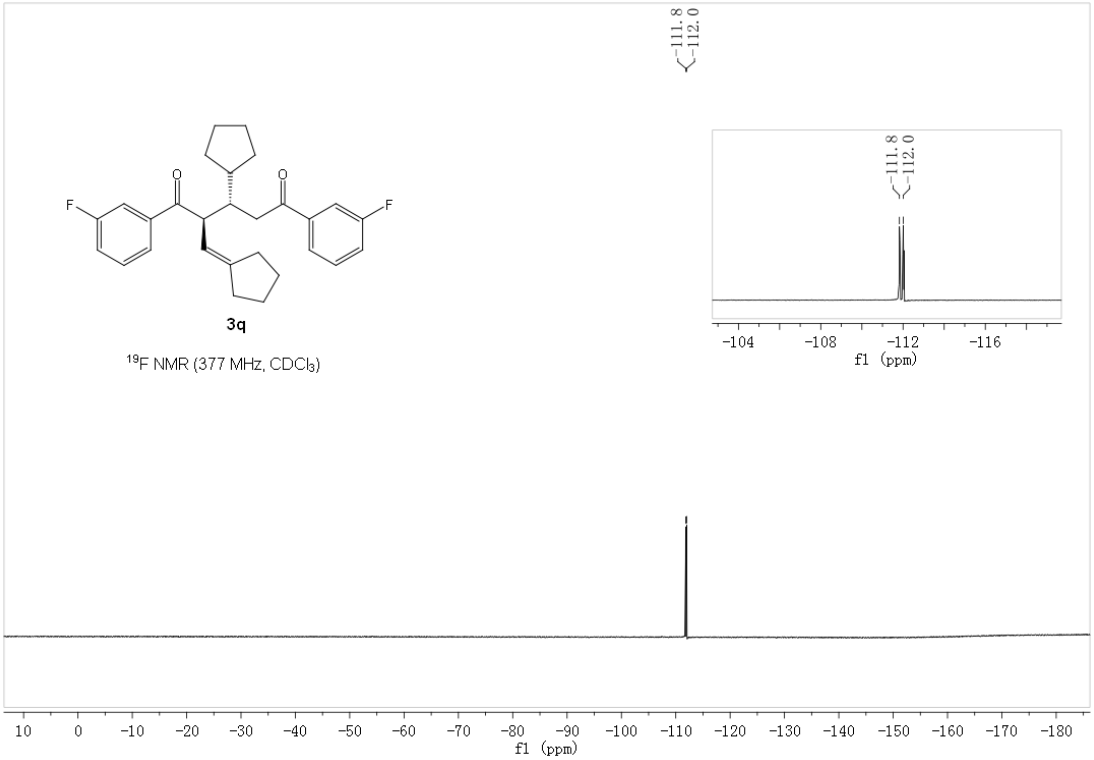
^{13}C NMR (101 MHz, $CDCl_3$)

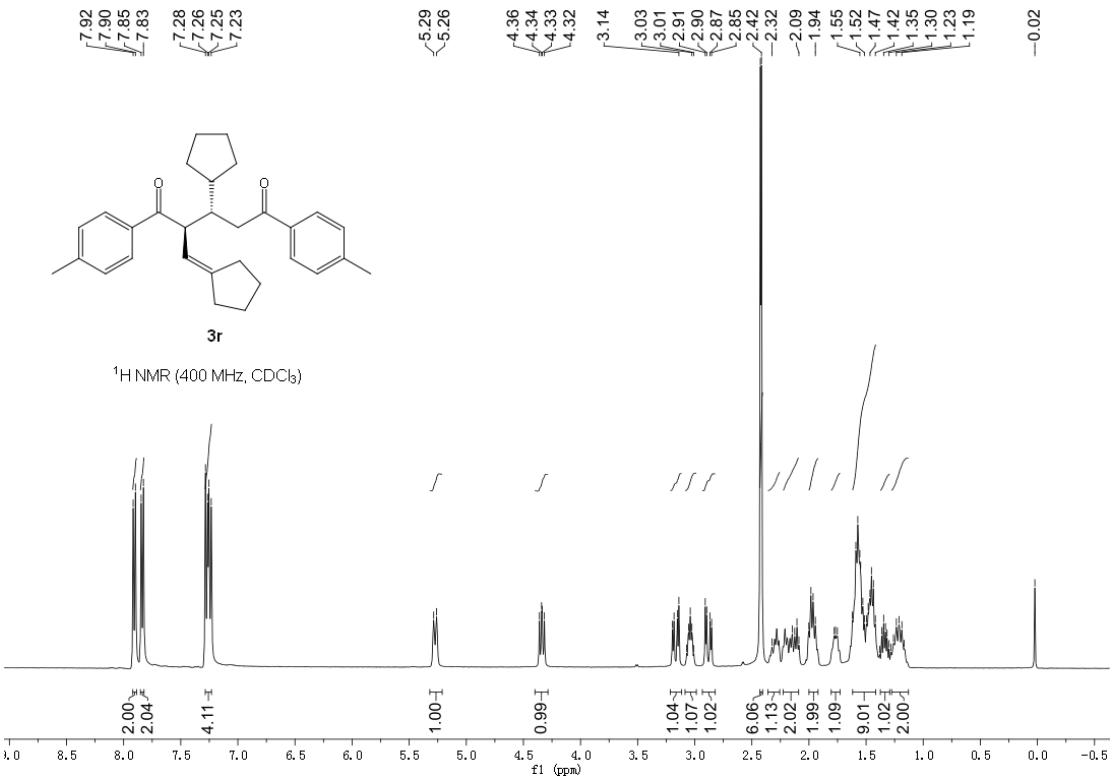


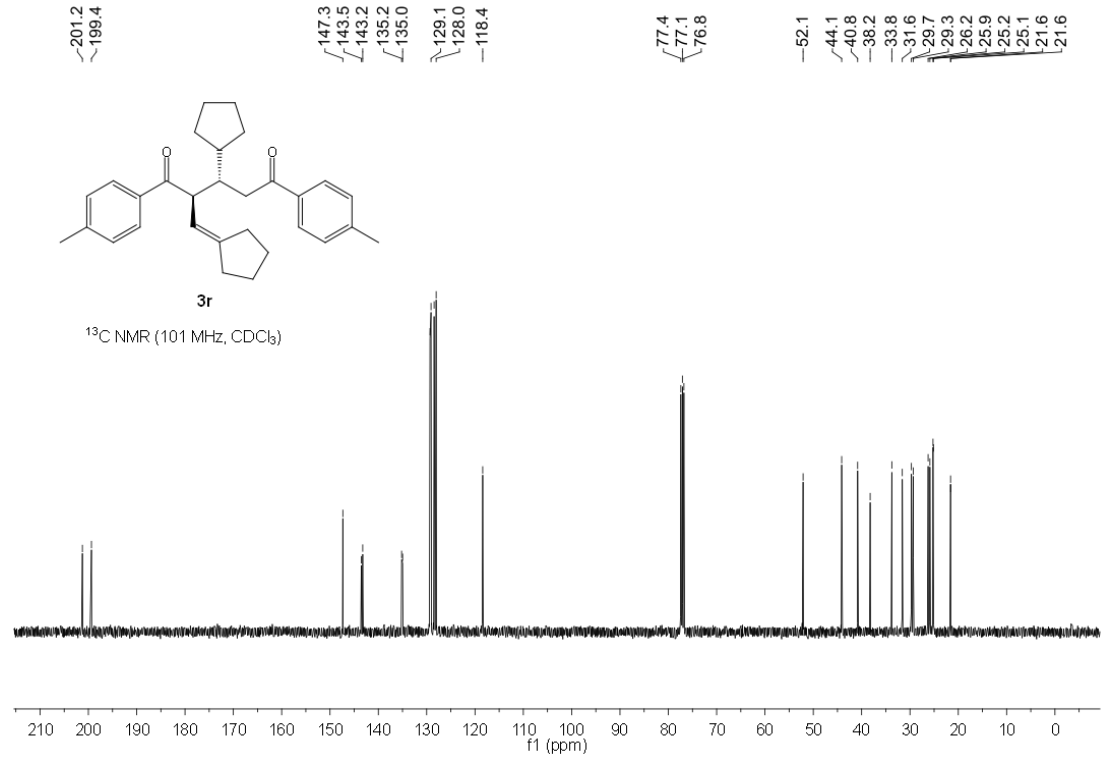
3q

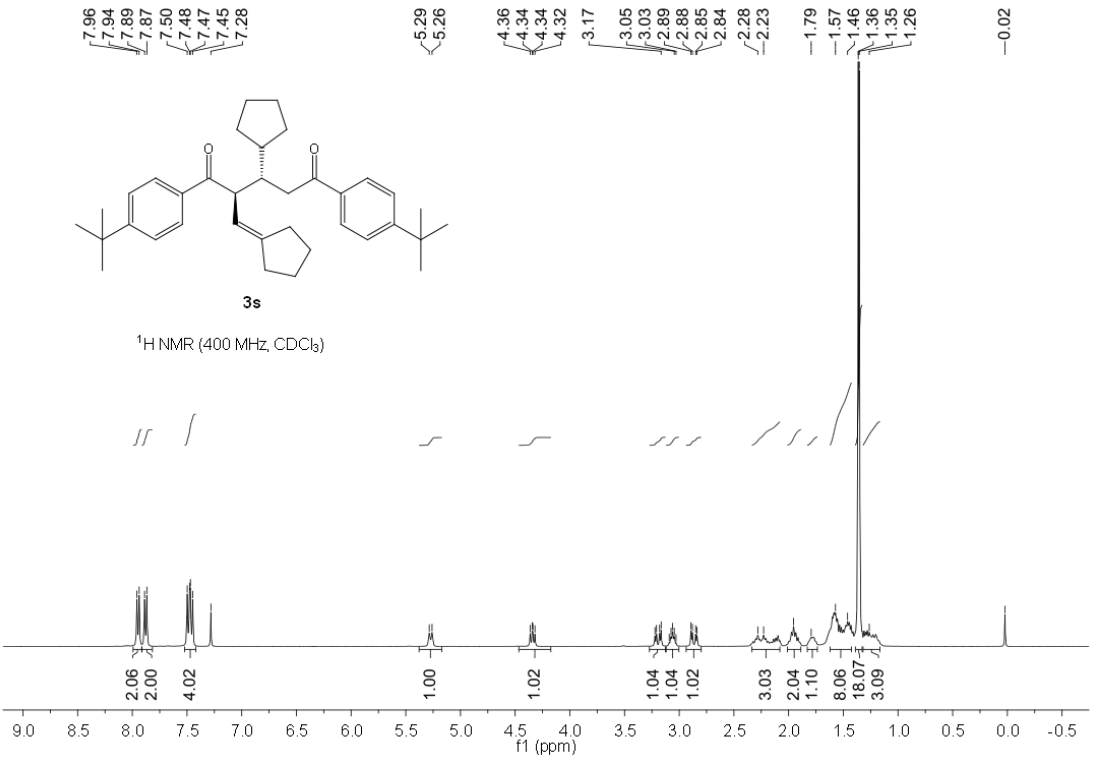
^{1}H NMR (400 MHz, $CDCl_3$)

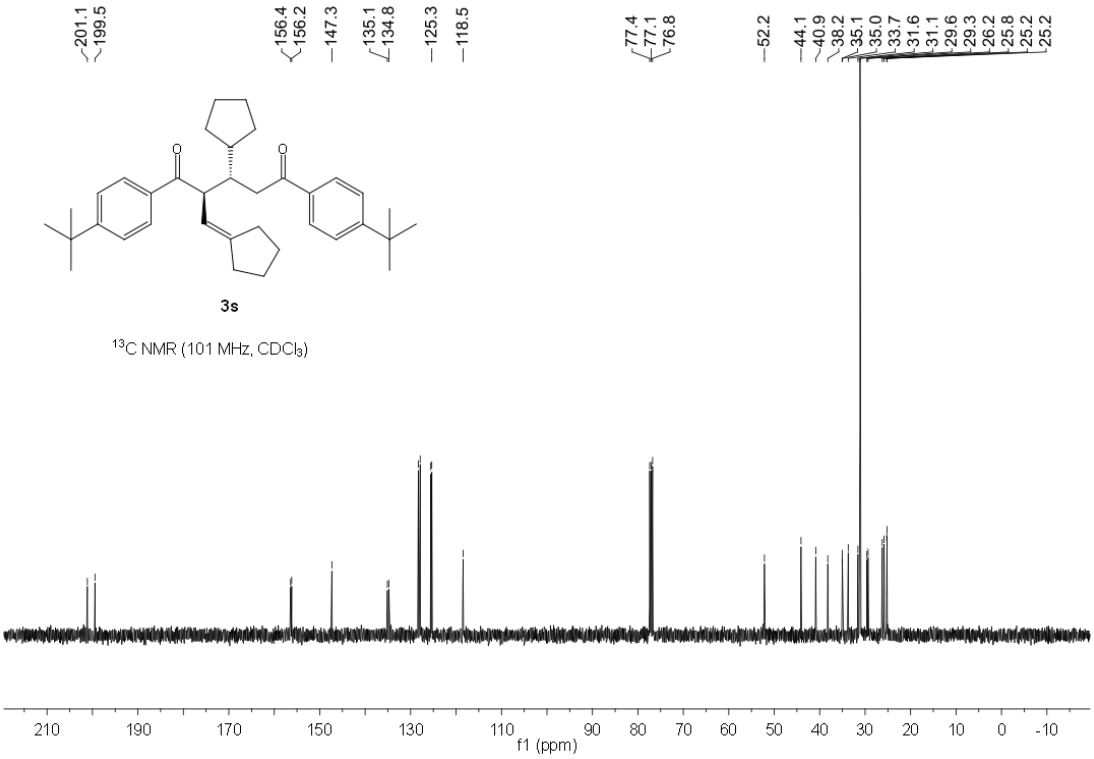

200.2
198.2
164.2
164.1
161.7
161.6
148.3
139.7
139.6
139.6
130.2
130.1
123.6
119.8
119.6
117.6
115.2
115.0
114.8
114.6
77.4
77.0
76.7
52.4
44.1
40.8
38.4
33.8
31.7
29.8
29.4
26.2
25.8
25.2
25.1
F
O
O
F
3q
13C NMR (101 MHz, CDCl3)
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

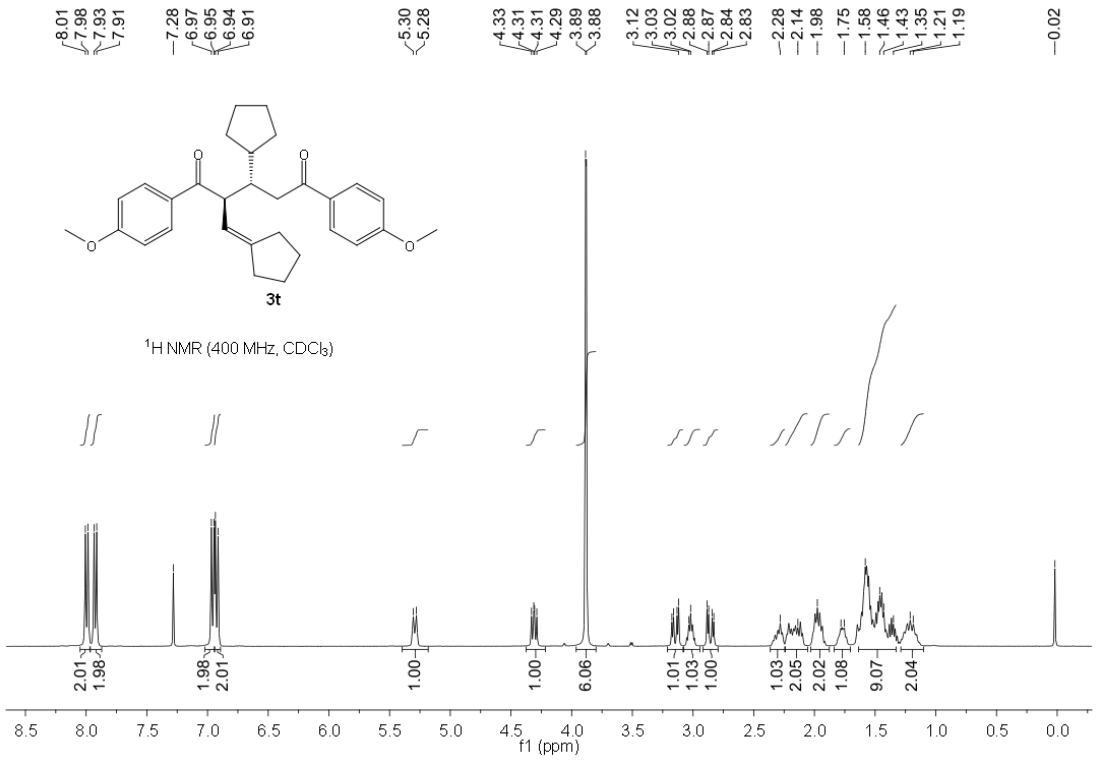

-111.8
-112.0
F
O
O
F
3q
19F NMR (377 MHz, CDCl3)
-104 -108 -112 -116
f1 (ppm)
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180
f1 (ppm)

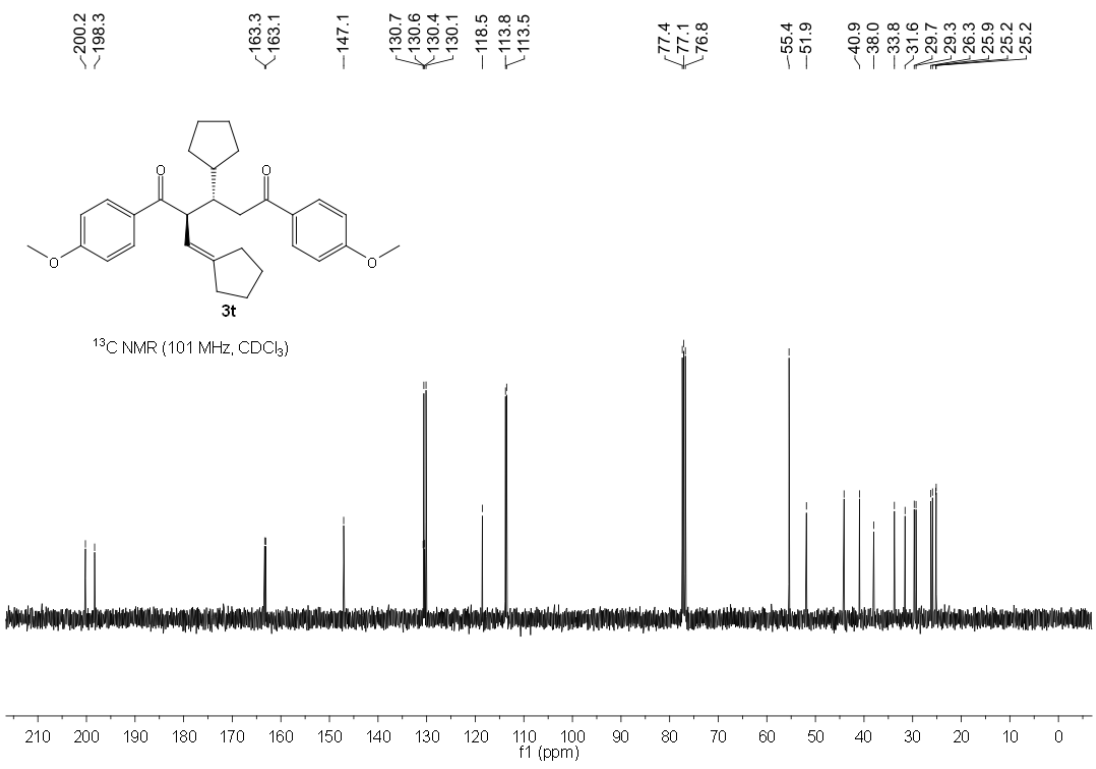

7.92
7.90
7.85
7.83
7.28
7.26
7.25
7.23
5.29
5.26
4.36
4.34
4.33
4.32
3.14
3.03
3.01
2.91
2.90
2.87
2.85
2.42
2.32
2.09
1.94
1.55
1.52
1.47
1.42
1.35
1.30
1.23
1.19
0.02
O
O
3r
1H NMR (400 MHz, CDCl3)
2.00
2.04
4.11
1.00
0.99
1.04
1.07
1.02
6.06
1.13
2.02
1.99
1.09
9.01
1.02
2.00
f1 (ppm)

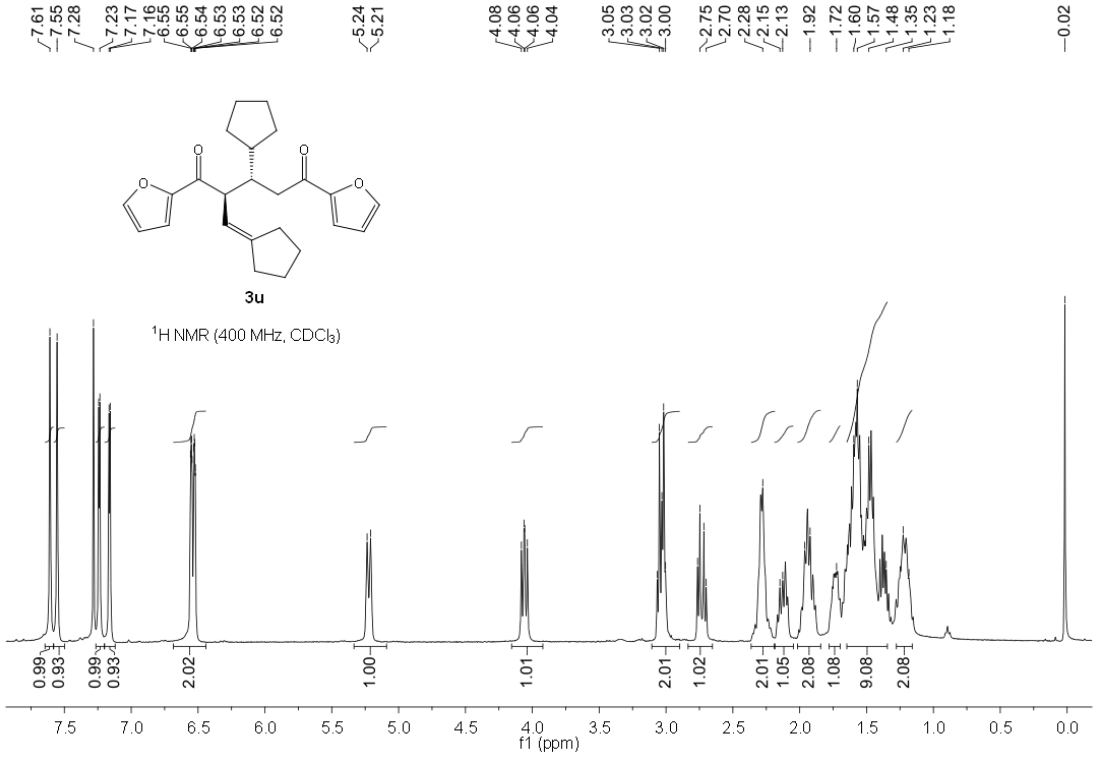

201.2
199.4
147.3
143.5
143.2
135.2
135.0
129.1
128.0
118.4
77.4
77.1
76.8
52.1
44.1
40.8
38.2
33.8
31.6
29.7
29.3
26.2
25.9
25.2
25.1
21.6
21.6
O
O
3r
13C NMR (101 MHz, CDCl3)
f1 (ppm)

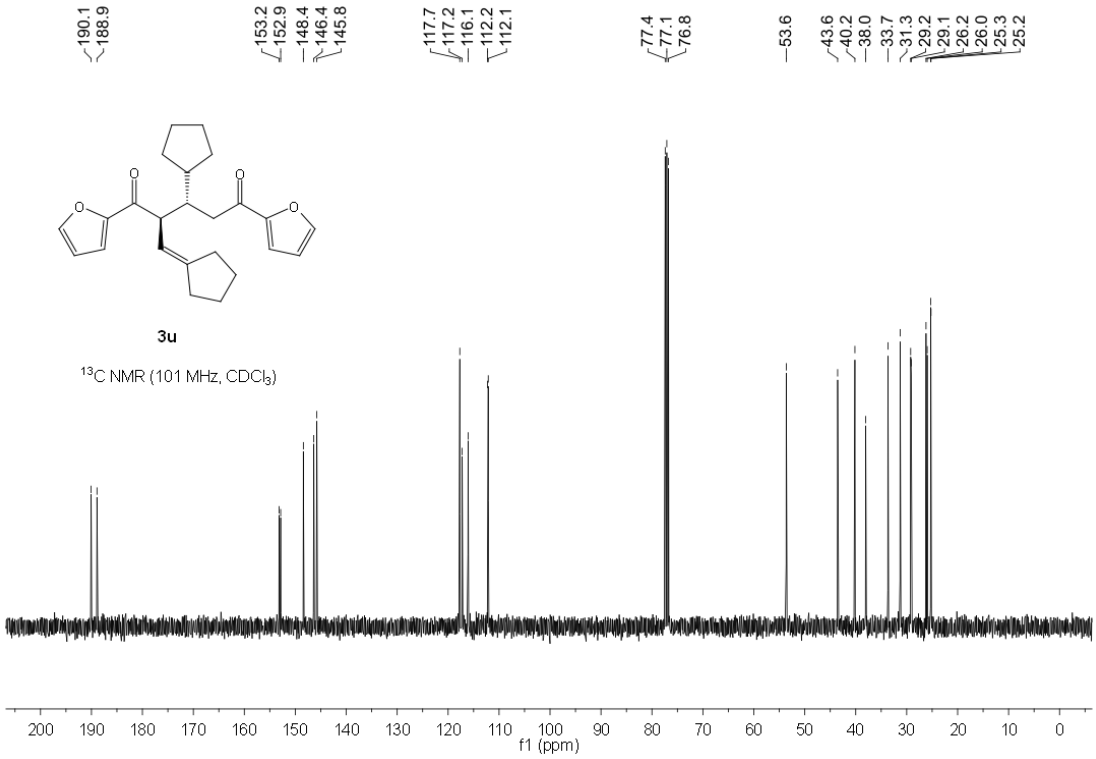

7.96
7.94
7.89
7.87
7.50
7.48
7.47
7.45
7.28
5.29
5.26
4.36
4.34
4.34
4.32
3.17
3.05
3.03
2.89
2.88
2.85
2.84
2.28
2.23
1.79
1.57
1.46
1.36
1.35
1.26
0.02
3s
1H NMR (400 MHz, CDCl3)
2.06
2.00
4.02
1.00
1.02
1.04
1.04
1.02
3.03
2.04
1.10
8.06
18.07
3.09
f1 (ppm)

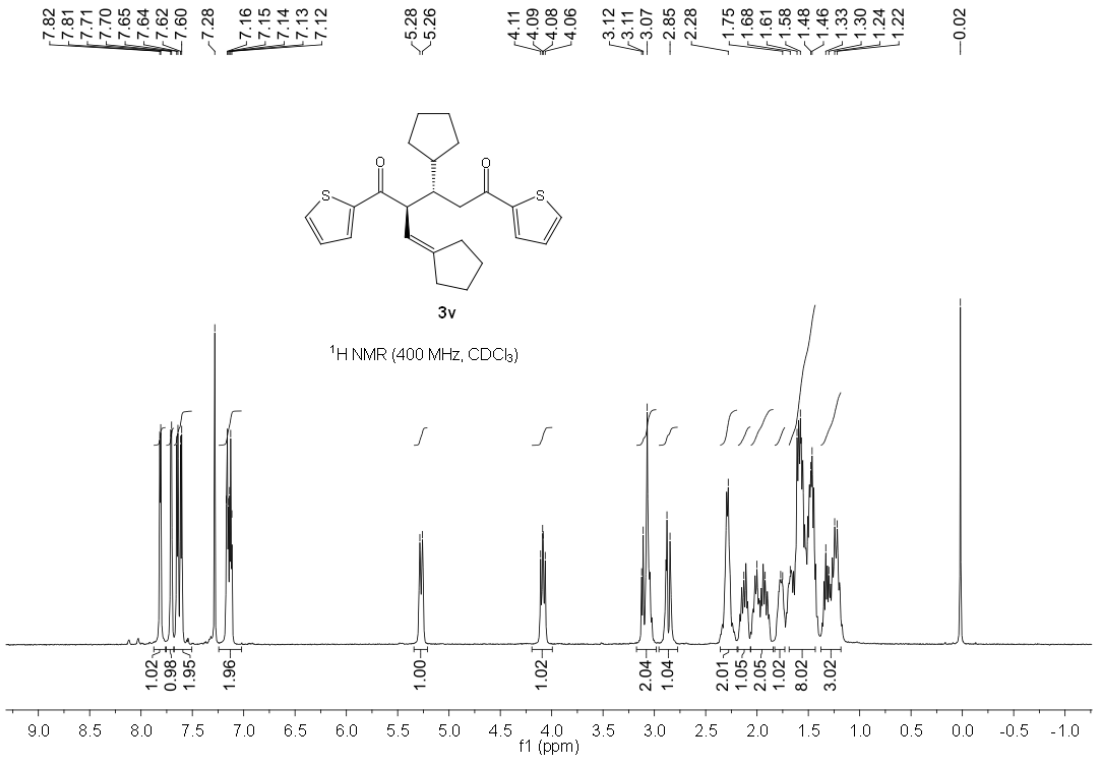

201.1
199.5
156.4
156.2
147.3
135.1
134.8
125.3
118.5
77.4
77.1
76.8
52.2
44.1
40.9
38.2
35.1
35.0
33.7
31.6
31.1
29.6
29.3
26.2
25.8
25.2
25.2
3s
13C NMR (101 MHz, CDCl3)
f1 (ppm)

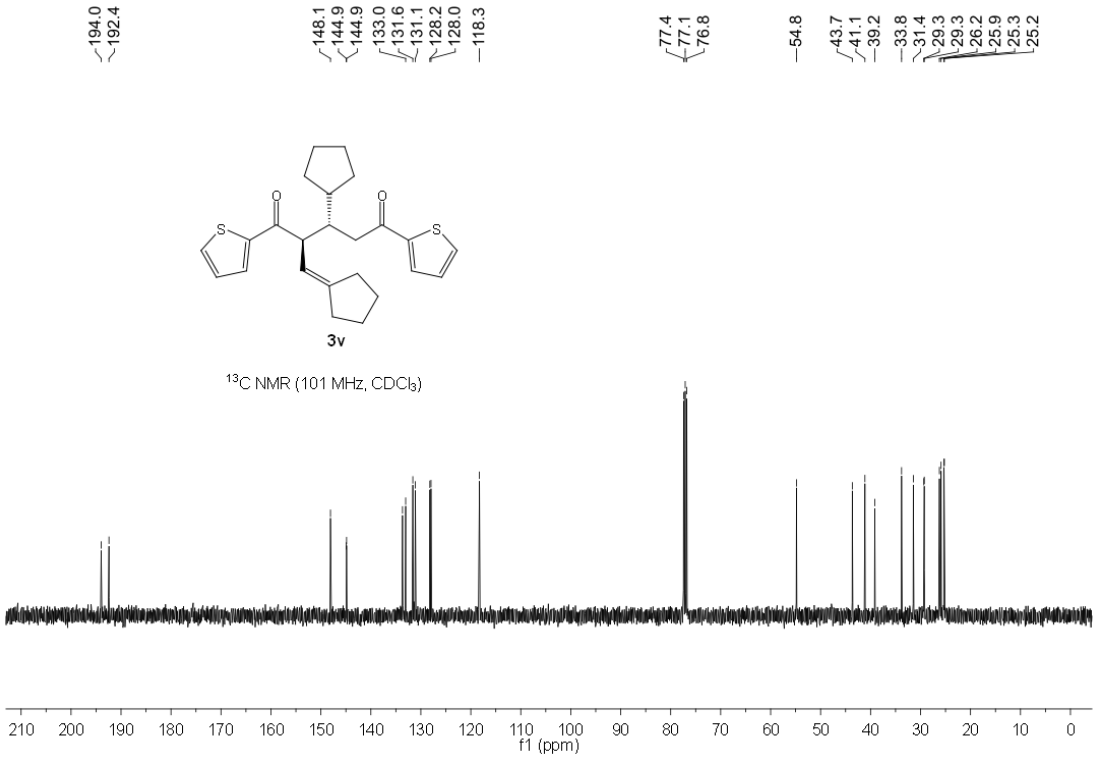

8.01
7.98
7.93
7.91
7.28
6.97
6.95
6.94
6.91
5.30
5.28
4.33
4.31
4.31
4.29
3.89
3.88
3.12
3.03
3.02
2.88
2.87
2.84
2.83
2.28
2.14
1.98
1.75
1.58
1.46
1.43
1.35
1.21
1.19
0.02
3t
1H NMR (400 MHz, CDCl3)
2.01
1.98
1.98
2.01
1.00
1.00
6.06
1.01
1.03
1.00
1.03
2.05
2.02
1.08
9.07
2.04
f1 (ppm)

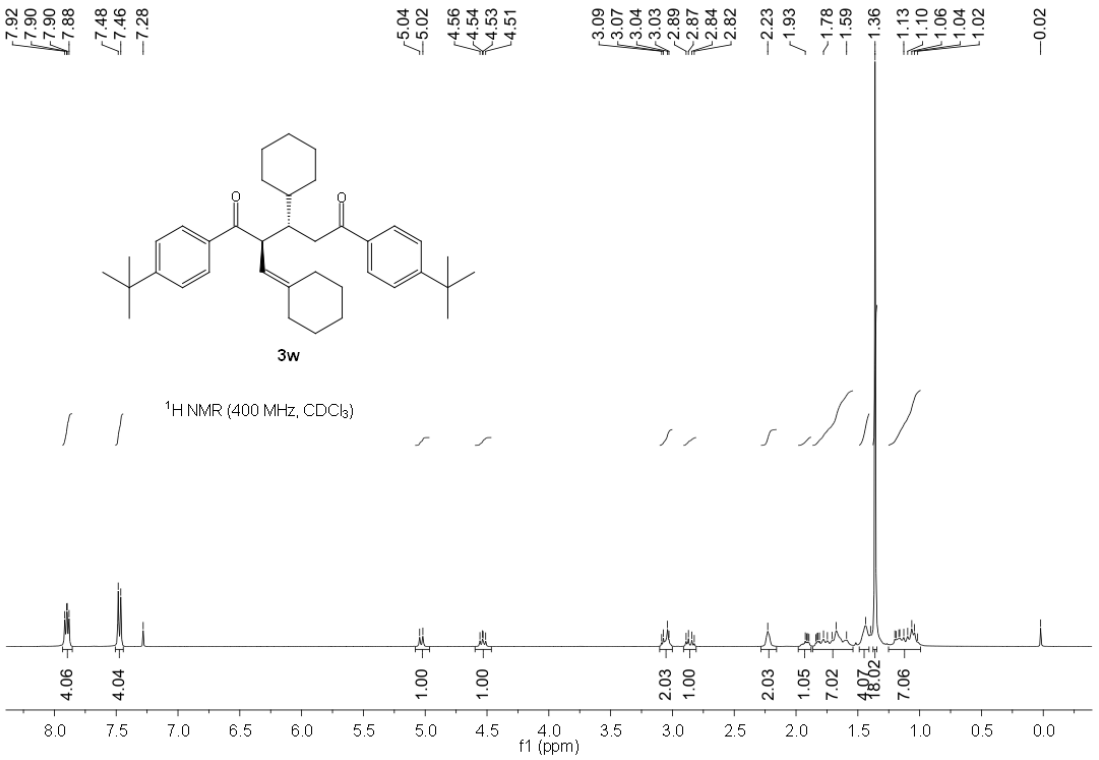

200.2
198.3
163.3
163.1
147.1
130.7
130.6
130.4
130.1
118.5
113.8
113.5
77.4
77.1
76.8
55.4
51.9
40.9
38.0
33.8
31.6
29.7
29.3
26.3
25.9
25.2
25.2
3t
13C NMR (101 MHz, CDCl3)
f1 (ppm)

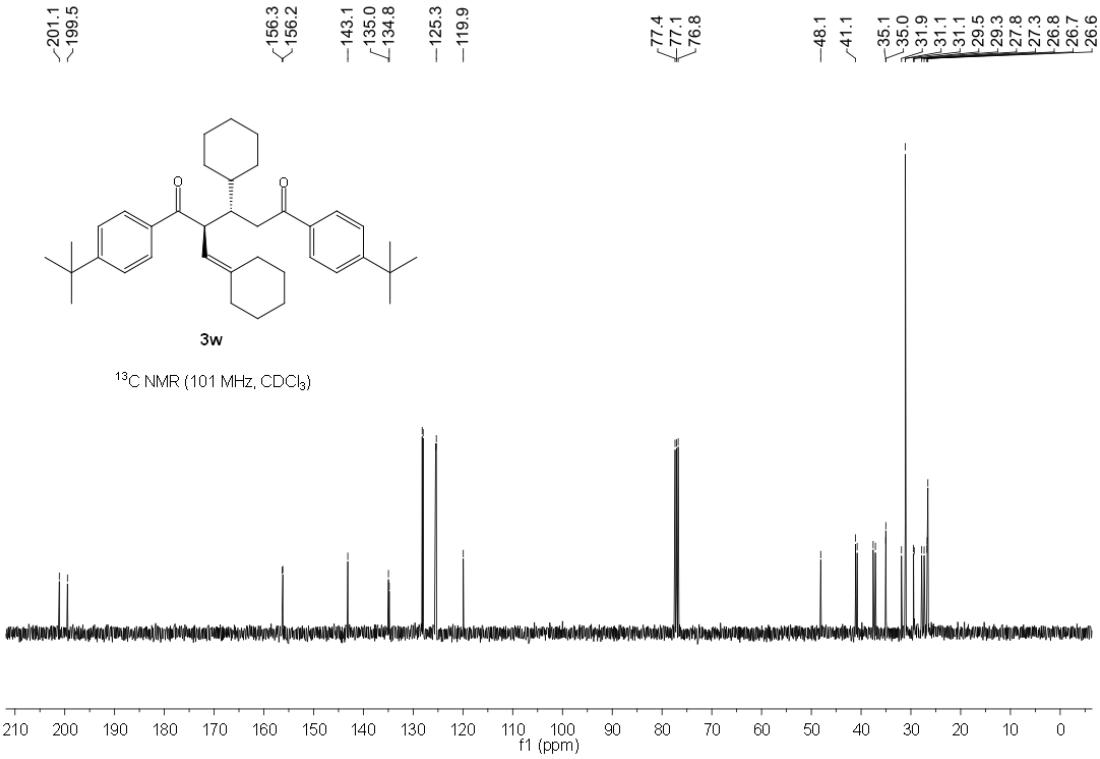

7.61
7.55
7.28
7.23
7.17
7.16
6.55
6.54
6.53
6.52
6.52
5.24
5.21
4.08
4.06
4.06
4.04
3.05
3.03
3.02
3.00
2.75
2.70
2.28
2.15
2.13
1.92
1.72
1.60
1.57
1.48
1.35
1.23
1.18
0.02
3u
1H NMR (400 MHz, CDCl3)
0.99
0.93
0.99
0.93
2.02
1.00
1.01
2.01
1.02
2.01
1.05
2.08
1.08
9.08
2.08
f1 (ppm)

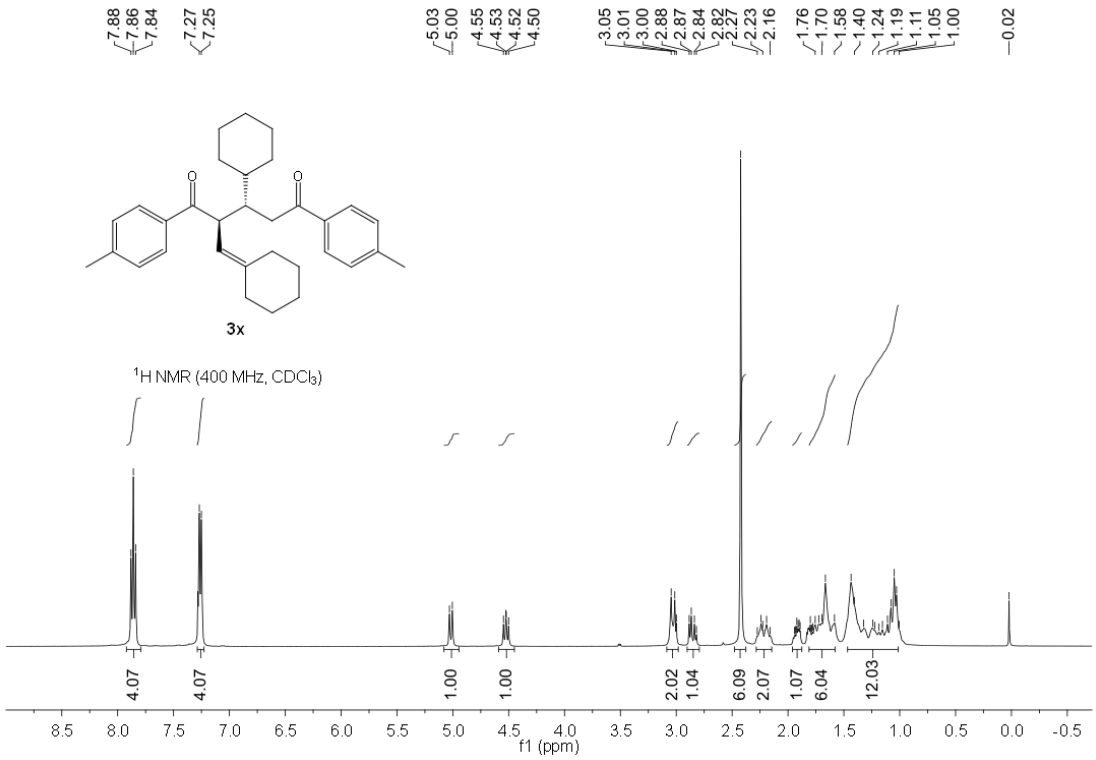

190.1
188.9
153.2
152.9
148.4
146.4
145.8
117.7
117.2
116.1
112.2
112.1
77.4
77.1
76.8
53.6
43.6
40.2
38.0
33.7
31.3
29.2
29.1
26.2
26.0
25.3
25.2
3u
13C NMR (101 MHz, CDCl3)
f1 (ppm)

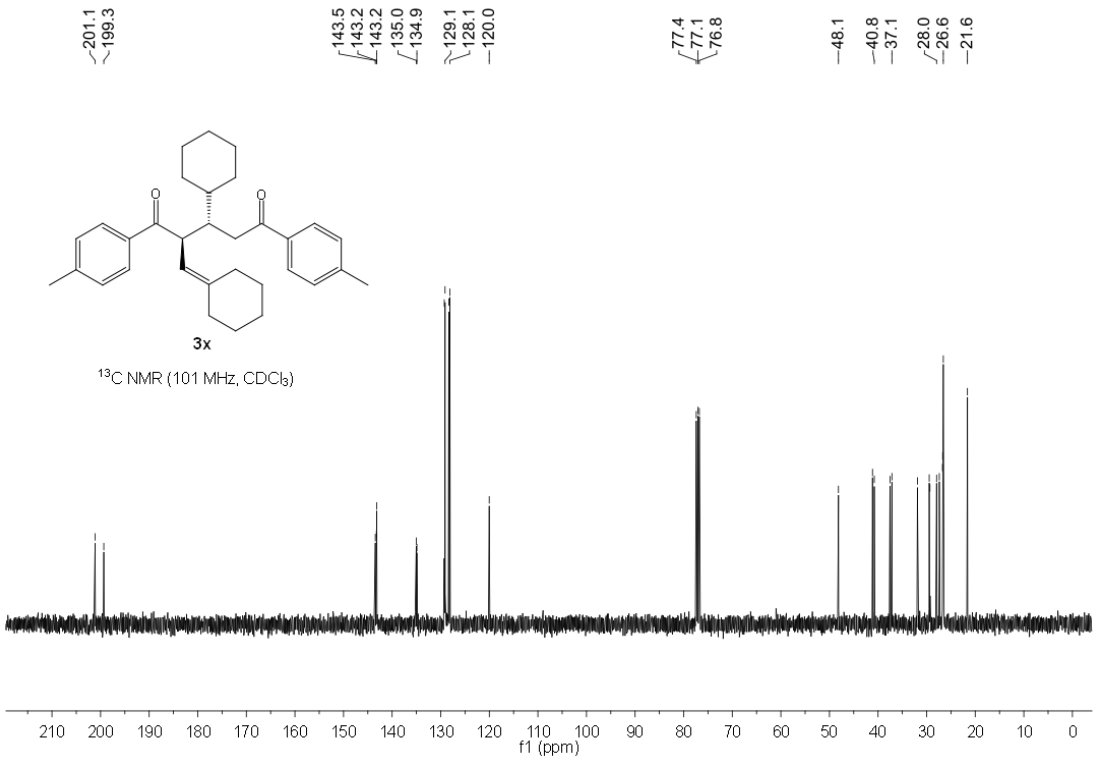

7.82
7.81
7.71
7.70
7.65
7.64
7.62
7.60
7.28
7.16
7.15
7.14
7.13
7.12
5.28
5.26
4.11
4.09
4.08
4.06
3.12
3.11
3.07
2.85
2.28
1.75
1.68
1.61
1.58
1.48
1.46
1.33
1.30
1.24
1.22
0.02
O
O
S
S
3v
1H NMR (400 MHz, CDCl3)
1.02
0.98
1.95
1.96
1.00
1.02
2.04
1.04
2.01
1.05
2.05
1.02
8.02
3.02
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)

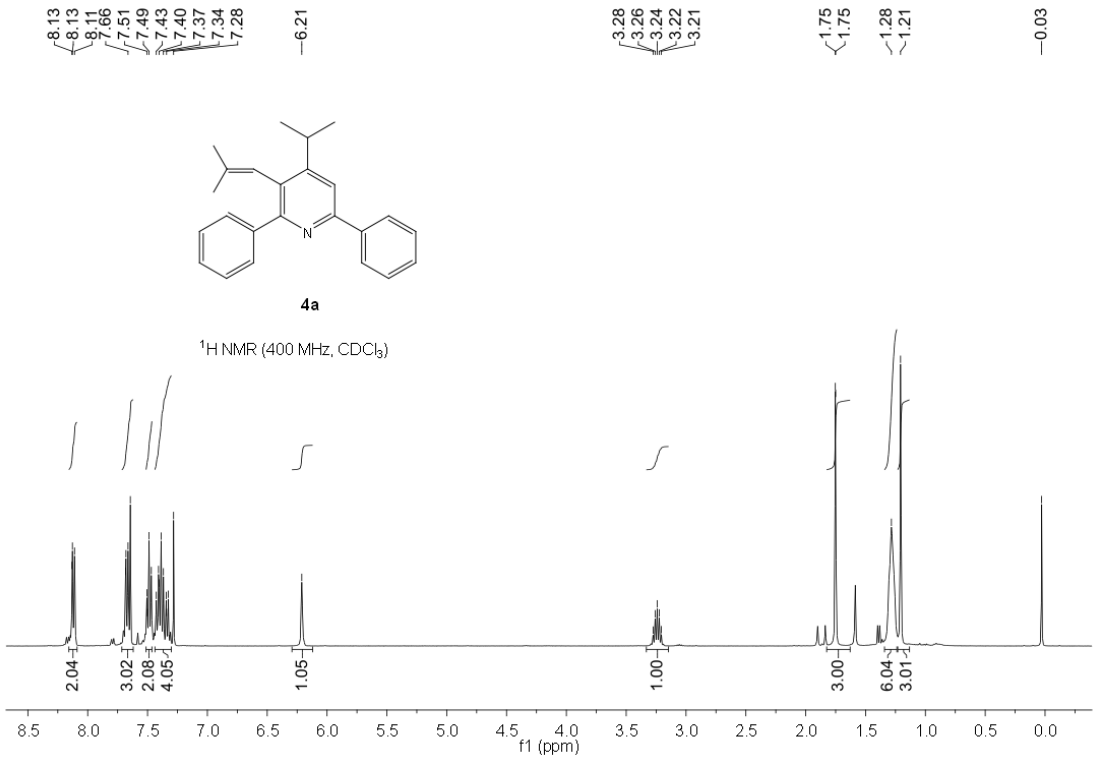

194.0
192.4
148.1
144.9
144.9
133.0
131.6
131.1
128.2
128.0
118.3
77.4
77.1
76.8
54.8
43.7
41.1
39.2
33.8
31.4
29.3
29.3
26.2
25.9
25.3
25.2
O
O
S
S
3v
13C NMR (101 MHz, CDCl3)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

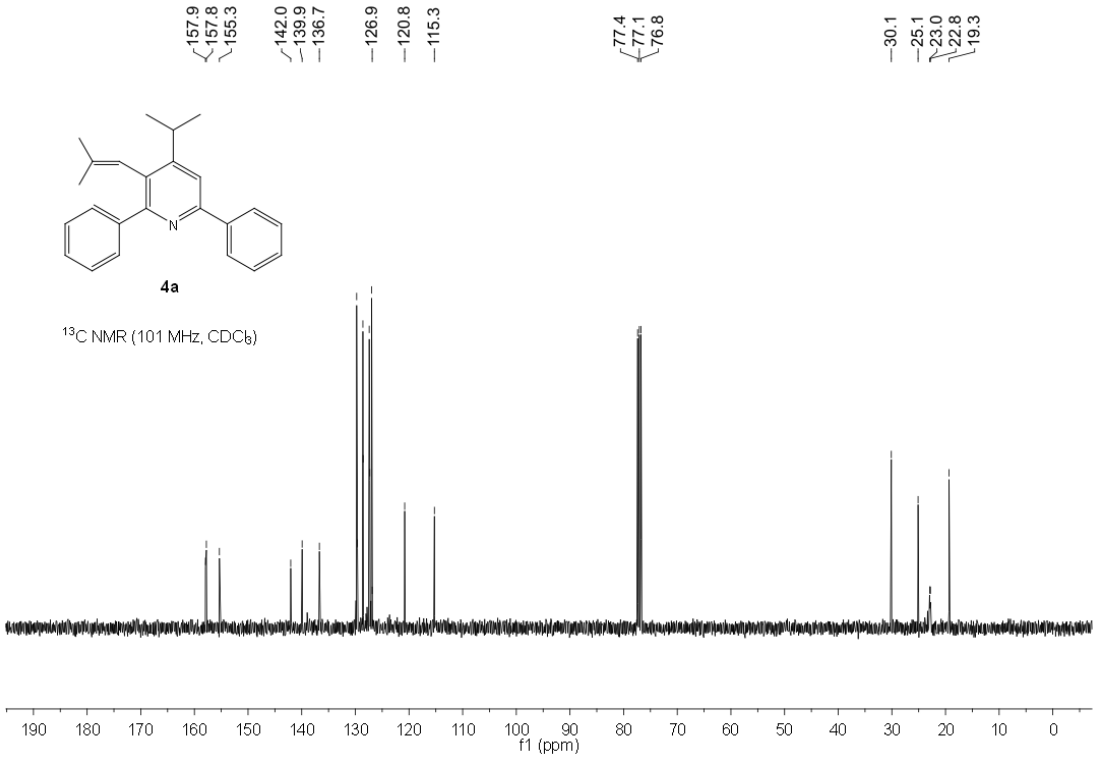

7.92
7.90
7.90
7.88
7.48
7.46
7.28
5.04
5.02
4.56
4.54
4.53
4.51
3.09
3.07
3.04
3.03
2.89
2.87
2.84
2.82
2.23
1.93
1.78
1.59
1.36
1.13
1.10
1.06
1.04
1.02
0.02
3w
1H NMR (400 MHz, CDCl3)
4.06
4.04
1.00
1.00
2.03
1.00
2.03
1.05
7.02
4.07
18.02
7.06
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

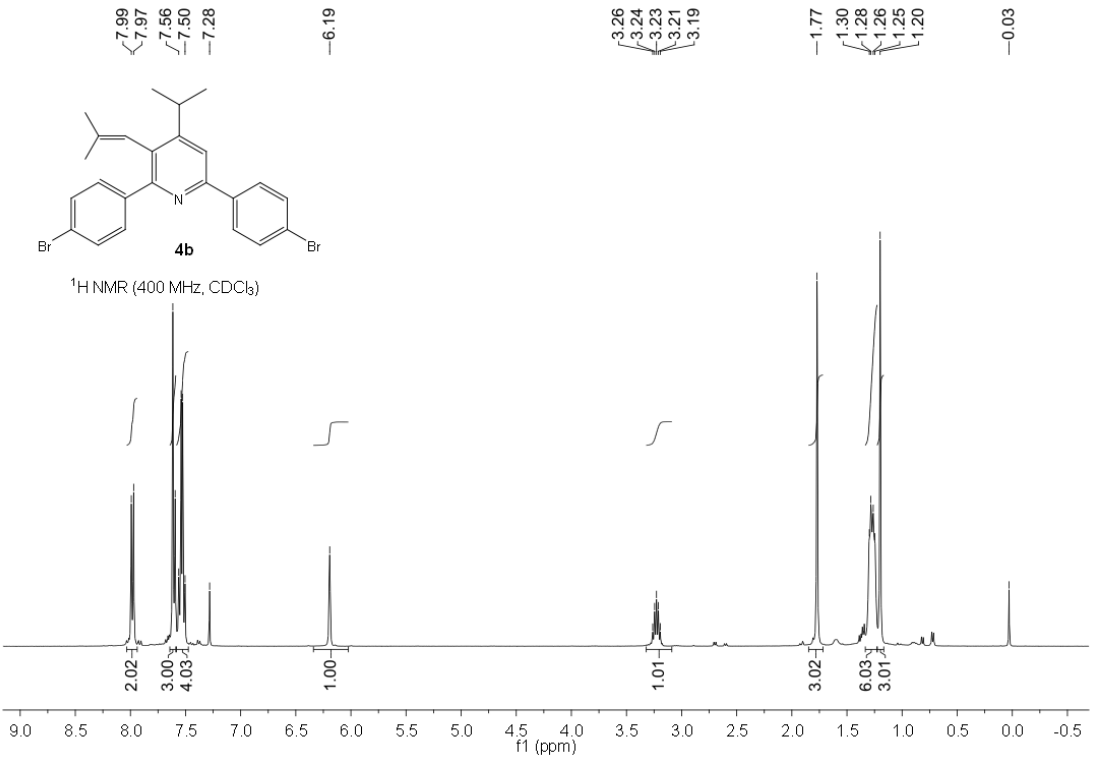

201.1
199.5
156.3
156.2
143.1
135.0
134.8
125.3
119.9
77.4
77.1
76.8
48.1
41.1
35.1
35.0
31.9
31.1
31.1
29.5
29.3
27.8
27.3
26.8
26.7
26.6
3w
13C NMR (101 MHz, CDCl3)
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

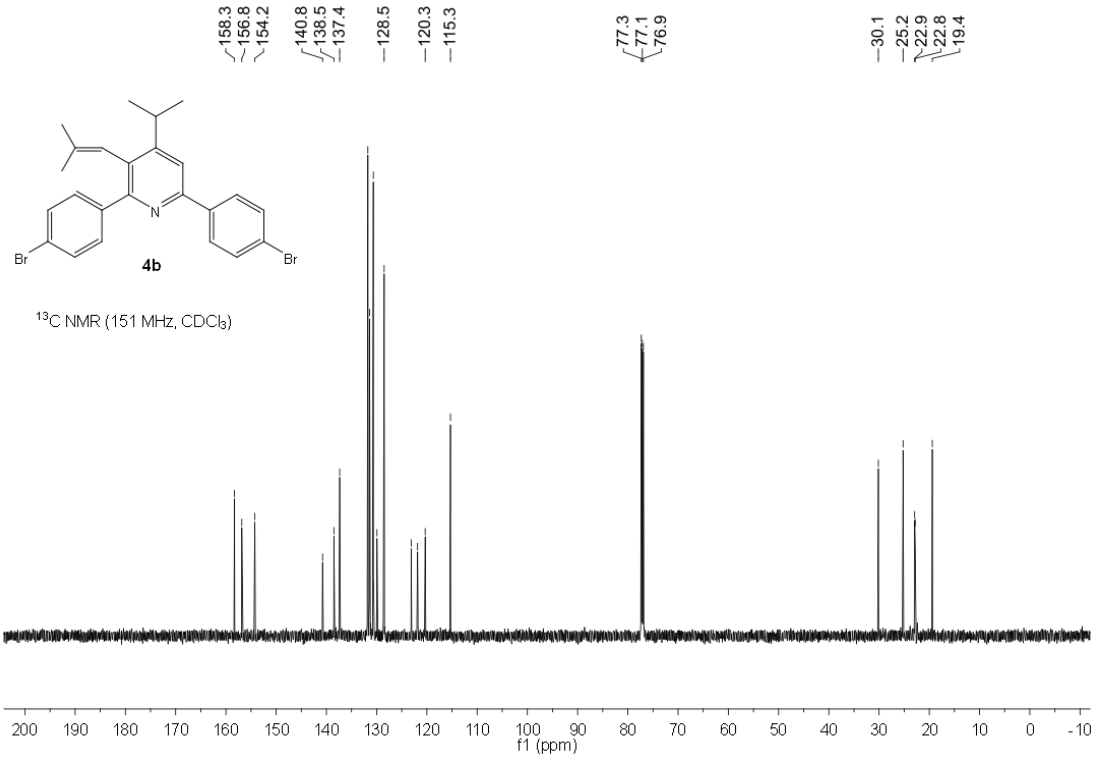

7.88
7.86
7.84
7.27
7.25
5.03
5.00
4.55
4.53
4.52
4.50
3.05
3.01
3.00
2.88
2.87
2.84
2.82
2.27
2.23
2.16
1.76
1.70
1.58
1.40
1.24
1.19
1.11
1.05
1.00
0.02
3x
1H NMR (400 MHz, CDCl3)
4.07
4.07
1.00
1.00
2.02
1.04
6.09
2.07
1.07
6.04
12.03
f1 (ppm)

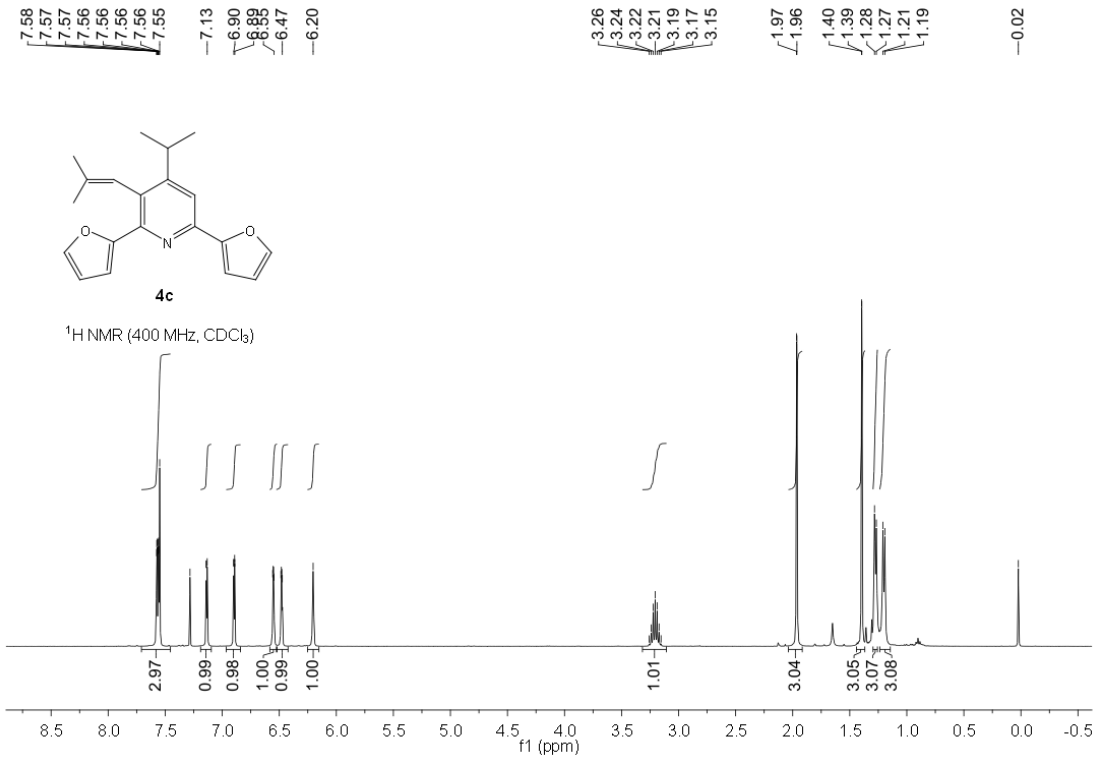

201.1
199.3
143.5
143.2
143.2
135.0
134.9
129.1
128.1
120.0
77.4
77.1
76.8
48.1
40.8
37.1
28.0
26.6
21.6
3x
13C NMR (101 MHz, CDCl3)
f1 (ppm)

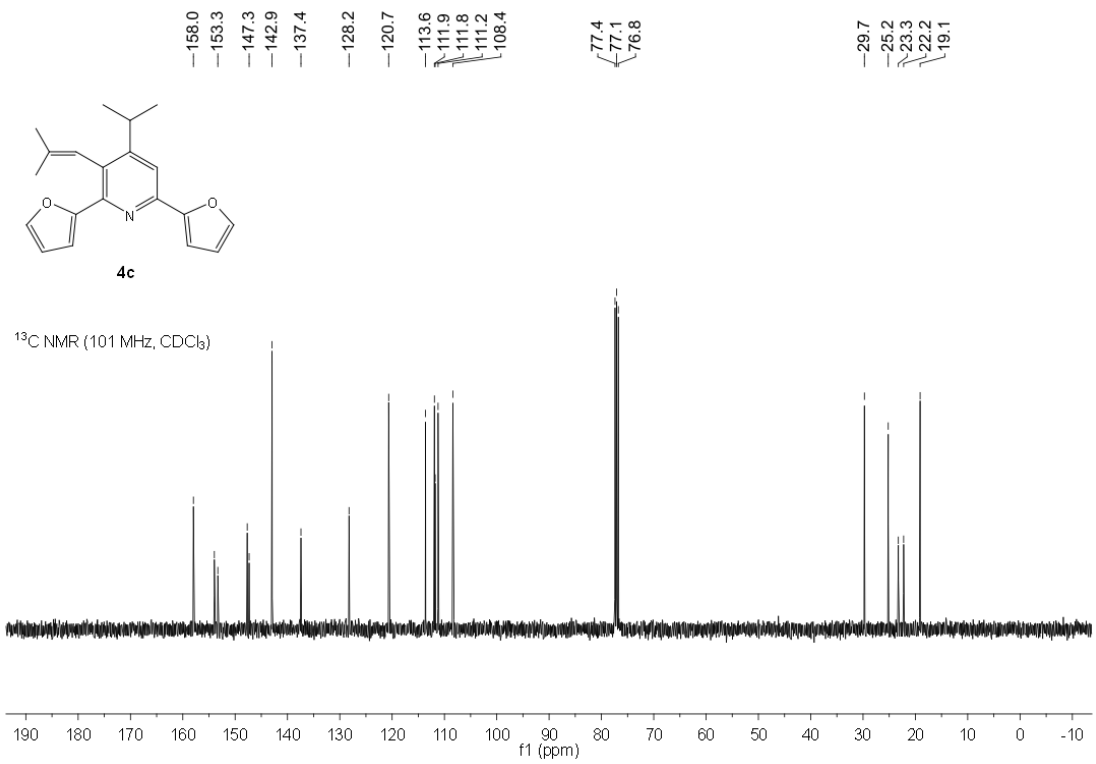

8.13
8.13
8.11
7.66
7.51
7.49
7.43
7.40
7.37
7.34
7.28
6.21
3.28
3.26
3.24
3.22
3.21
1.75
1.75
1.28
1.21
0.03
4a
1H NMR (400 MHz, CDCl3)
2.04
3.02
2.08
4.05
1.05
1.00
3.00
6.04
3.01
f1 (ppm)


157.9
157.8
155.3
142.0
139.9
136.7
126.9
120.8
115.3
77.4
77.1
76.8
30.1
25.1
23.0
22.8
19.3
4a
13C NMR (101 MHz, CDCl3)
f1 (ppm)


7.99
7.97
7.56
7.50
7.28
6.19
3.26
3.24
3.23
3.21
3.19
1.77
1.30
1.28
1.26
1.25
1.20
0.03
Br
Br
4b
^{1}H NMR (400 MHz, CDCl$_3$)
2.02
3.00
4.03
1.00
1.01
3.02
6.03
3.01
f1 (ppm)

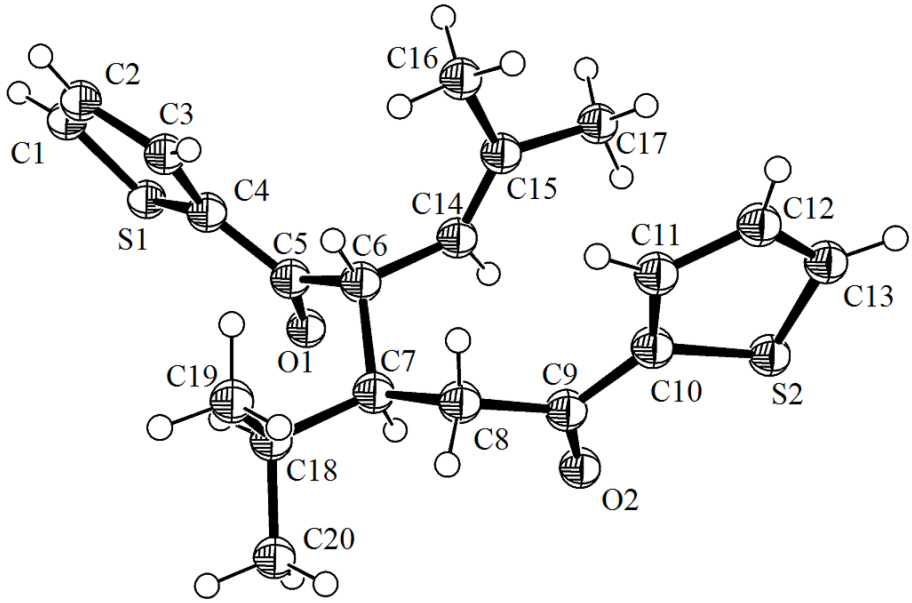

158.3
156.8
154.2
140.8
138.5
137.4
128.5
120.3
115.3
77.3
77.1
76.9
30.1
25.2
22.9
22.8
19.4
Br
Br
4b
^{13}C NMR (151 MHz, CDCl$_3$)
f1 (ppm)


7.58
7.57
7.57
7.56
7.56
7.56
7.56
7.55
7.13
6.90
6.89
6.55
6.47
6.20
3.26
3.24
3.22
3.21
3.19
3.17
3.15
1.97
1.96
1.40
1.39
1.28
1.27
1.21
1.19
0.02
4c
1H NMR (400 MHz, CDCl3)
2.97
0.99
0.98
1.00
0.99
1.00
1.01
3.04
3.05
3.07
3.08
f1 (ppm)


158.0
153.3
147.3
142.9
137.4
128.2
120.7
113.6
111.9
111.8
111.2
108.4
77.4
77.1
76.8
29.7
25.2
23.3
22.2
19.1
4c
13C NMR (101 MHz, CDCl3)
f1 (ppm)

X-Ray crystallographic analysis for product 3m:

CCDC 1063937 (**3m**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk /data_request/cif.



3m