Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Synthesis of Spiro[2.5]octa-4,7-dien-6-one with Consecutive Quarternary Centers via 1,6-Conjugate Addition Induced Dearomatization of *para*-Quinone Methides

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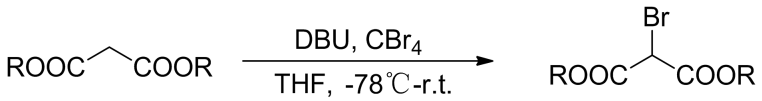
1. General Information

Reagents and Solvents: All solvents were purified and dried according to standard methods. PE refers to petroleum ether (b.p. 60 - 90 °C) and EA refers to ethyl acetate. Malontes **2a, 2b**, **2h** were commercially available, **2g** was synthesized according reported procedure[1].

Chromatography: Flash column chromatography was carried out using commercially available 200-300 mesh under pressur and conducted by eluting with PE/EA listed as volume/volume ratios.

Data collection: ^{1}H and ^{13}C NMR spectra were collected on BRUKER AV - 300 (300 MHz) spectrometer using $CDCl_3$ as solvent. Chemical shifts of ^{1}H NMR were recorded in parts per million (ppm, δ) relative to tetramethylsilane (δ = 0.00 ppm) with the solvent resonance as an internal standard ($CDCl_3$: δ = 7.26 ppm). Data are reported as follows: chemical shift in ppm (δ), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constant (Hz), and integration. Chemical shifts of ^{13}C NMR were reported in ppm with the solvent as the internal standard ($CDCl_3$: δ = 77.0 ppm). Infrared spectra (IR) were recorded on a Thermo Scientific iS10 FT/IR spectrometer. Absorptions are reported in reciprocal centimeters. High Resolution Mass measurement was performed on Agilent QTOF 6520 mass spectrometer with electron spray ionization (ESI) as the ion source. Melting point (m.p.) was measured on a microscopic melting point apparatus.

2. Preparation of Bromomalonates[2, 3]

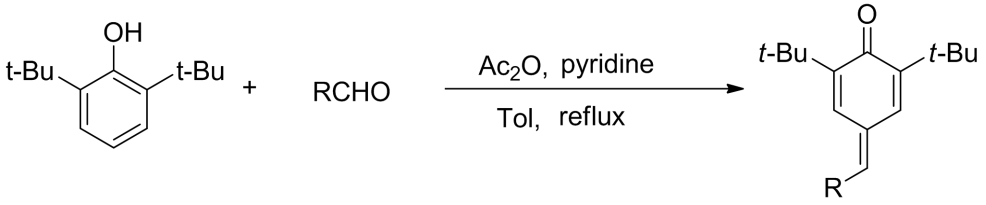


2c, 2d, 2e, 2f, 2h, 2i, 2j

Malonates (1 equiv) was dissolved in THF at 0 ºC and then DBU (1 equiv) was added. The solution was stirred for 1h at room temperature before cooled to -78ºC. After that, CBr_4 (1 equiv) was added. The mixture was stirred until completed (detected by TLC). After quenching with NH_4Cl, the phases were separated and diluted with CH_2Cl_2. The aqueous layer was washed twice with CH_2Cl_2, and combined organic layers were washed with brine. Then the organic layer was dried over anhydrous sodium sulfate and concentrated under vaccum. The crude product was purified by flash chromatography (PE/EA = 100:1 – 20:1) to obtain the pure product as colorless oil.

3. Preparation of *p*-QMs

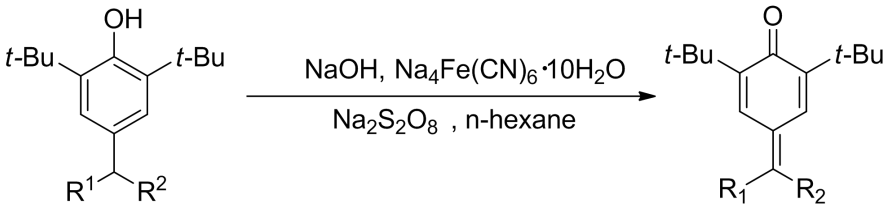
1a-1n was synthesized according to the following procedure[4]:



1a - 1n

In a Dean-Stark apparatus, a solution of phenols (1 equiv) and the corresponding aldehydes (1 equiv) in toluene was heated to reflux. Piperidine (2 equiv) was added dropwise within 1 h. The reaction mixture was continued to reflux for 3 hrs. After cooling just below the boiling point of the reaction mixture, acetic anhydride (2 equiv) was added and stirring was continued for 15 min. Then the reaction mixture was poured into water and extracted with CH_2Cl_2. The combined organic phase was dried over anhydrous Na_2SO_4, and solvents were removed under reduced pressure. The crude products were purified by flash column chromatography and further recrystallized from *n*-hexane, affording the desired *p*-QMs **1a-1n**.

1o and **1p** were synthesized according to the following procedure[5]:

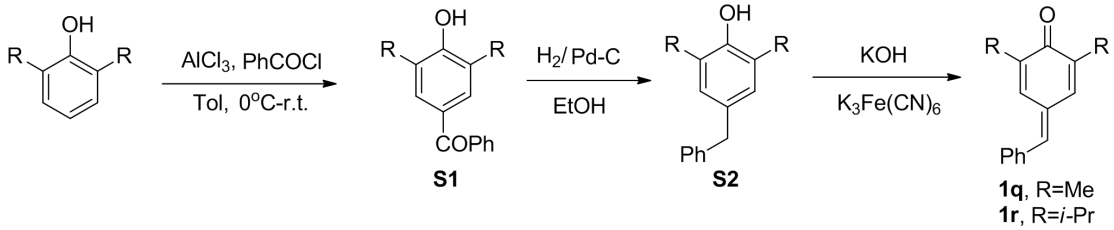


1o R^1=Me, R^2=H

1p R^1=Me, R^2=Et

NaOH (2.1 equiv), $Na_4Fe(CN)_6$ •$10H_2O$ (0.4 equiv) were dissolved in water, and a solution of substituted 2,6-di-*tert*-butylphenol (1 equiv) in n-hexane was added. Under Ar atmosphere, a solution of $Na_2S_2O_8$ (1.05 equiv) in water was slowly added. The mixture was stirred for 4 hrs, and then poured into separated funnel. Organic phase was separated and dried over anhydrous Na_2SO_4. The solvents were removed under reduced pressure and recrystallized from n-hexane to obtain the pure products **1o** and **1**p.

1q and **1r** were synthesized according to the following procedure[6]:



1q, R=Me
1r, R=*i*-Pr

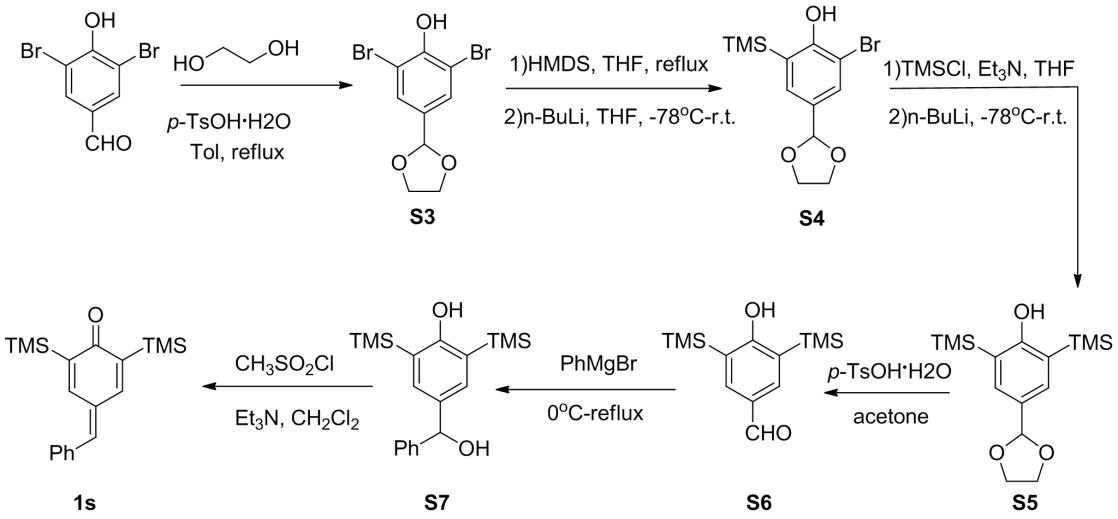
Phenols (1 equiv) were dissolved in toluene, and benzoyl chloride (1.8 equiv) was added dropwise. Then, aluminium chloride (0.4 equiv) was slowly added and the mixture was stirred at

room temperature for 7 hrs. After completion, solvents were evaporated and directly subjected to flash chromatography to obtain the pure product **S1**.

Palladium (10% by weight on carbon powder) (5% equiv) was added in one portion to a solution of **S1** (1.0 equiv) in ethanol under an atmosphere of H_2 (balloon). The reaction mixture was stirred vigorously for 3 hrs at 23 °C and then filtered through a pad of celite. The solvent was removed by rotary evaporation, and the residue was purified by silica gel flash column chromatography to afford **S2**.

Potassium ferricyanide (4 equiv) and potassium hydroxide (4.2 equiv) in water was added in one portion to a solution of **S2** (1 equiv) in hexane under an Ar atmosphere. The reaction mixture was stirred vigorously for 1 h at 23 °C. The aqueous layer was separated and extracted with hexanes. The combined organic layers were washed with brine and dried over anhydrous sodium sulfate. The solids were removed by filtration and the solution was concentrated by rotary evaporation. The residue was purified by silica gel flash column chromatography to afford **1q** and **1r**.

1s was synthesized according to the following procedure[4a,7]:



In a Deal-Stark apparatus, 3,5-dibromo-4-hydroxybenzaldehyde (8.9 mmol, 2.5 g) and PTSA (0.45 mmol, 86 mg) were dissolved in toluene (30 mL) under Ar atmosphere and heated to reflux. To the stirred solution was added glycol (18 mmol, 1 mL). After stirred overnight, K_2CO_3 (1.8 mmol, 250 mg) was added and stirring was continued for 1 h. Then, the mixture was filtered and the solvents were removed under reduce pressued. The residue was purified by chromotogrphy to obtain the pure product **S3**.

A mixture of **S3** (1.9 g, 5.86 mmol), HMDS (2.46 mL, 11.72 mmol) in anhydours THF (15 mL) was refluxed for 5 hrs. Solvents were then removed under reduced pressure to obtain the crude intermediate (2.47 g). Then the intermediate was dissolved in THF (17 mL), and cooled to -78 °C. To the stirred solution was slowly added 1.6M n-BuLi (5.86 mmol, 3.7 mL). After 1h, the reaction was moved to room tepreature and stirring was continued for 2hrs. After quenching with saturated NH_4Cl, the mixture was extracted with EtOAc, washed with brine, and dried over anhydrous sodium sulfate. The solids were removed by filtration and the solution was concentrated by rotary evaporation. The residue was purified by silica gel flash column

chromatography to afford product **S4**.

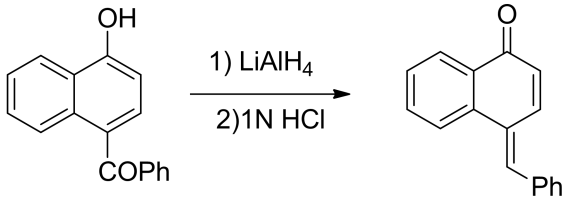
A mixture of **S4** (4.92 mmol, 1.56 g), TMSCl (7.38 mmol, 0.9 mL), Et_3N (7.38 mmol, 1.0 mL) in THF (15 mL) was stirred for 1h and then cooled to -78 °C. 1.6M n-BuLi (4.92 mmol, 3.1 mL) was slowly added and the mixture was continued to stir overnight. After quenching with saturated NH_4Cl, the mixture was extracted with EtOAc, washed with brine, and dried over anhydrous sodium sulfate. The solids were removed by filtration and the solution was concentrated by rotary evaporation. The residue was purified by silica gel flash column chromatography to afford product **S5**.

S5 (3.5 mmol, 1.1 g) was dissolved in acetone, and PTSA (0.35 mmol, 67 mg) was added. The mixture was stirred for 1 h and diluted with EtOAc and H_2O. The mixture was extracted with EtOAc, washed with brine, and dried over anhydrous sodium sulfate. The solids were removed by filtration and the solution was concentrated by rotary evaporation. The residue was purified by silica gel flash column chromatography to afford product **S6** (710 mg, 75% yield).

S6 (2.7 mmol, 710 mg) was dissolved in anhydrous THF (15 mL) at 0°C under Ar atmosphere. Then PhMgBr (1M in THF, 11mmol) was slowly added. Then the reaction mixture was refluxed for 30 min and quenched with saturated NH_4Cl. After extraction with EtOAc, the mixture was washed with brine, and dried over anhydrous sodium sulfate. The solids were removed by filtration and the solution was concentrated by rotary evaporation. The residue was purified by silica gel flash column chromatography to afford product **S7**.

A mixture of **S7** (2.9 mmol, 1 g), Et_3N (6.4 mmol, 0.9 mL) in dry CH_2Cl_2 (30mL) was slowly added CH_3SO_2Cl (3.2 mmol, 0.3 mL) at 0°C. The mixture was then warmed to room temperature and stirred for 1hr. The mixture was diluted with CH_2Cl_2 and water, then the mixture was extracted with CH_2Cl_2, washed with brine, and dried over anhydrous sodium sulfate. The mixture was filtrated and concentrated by rotary evaporation. The crude product **1s** was used directly without further purification.

1t was synthesized according to the following procedure[4a]:

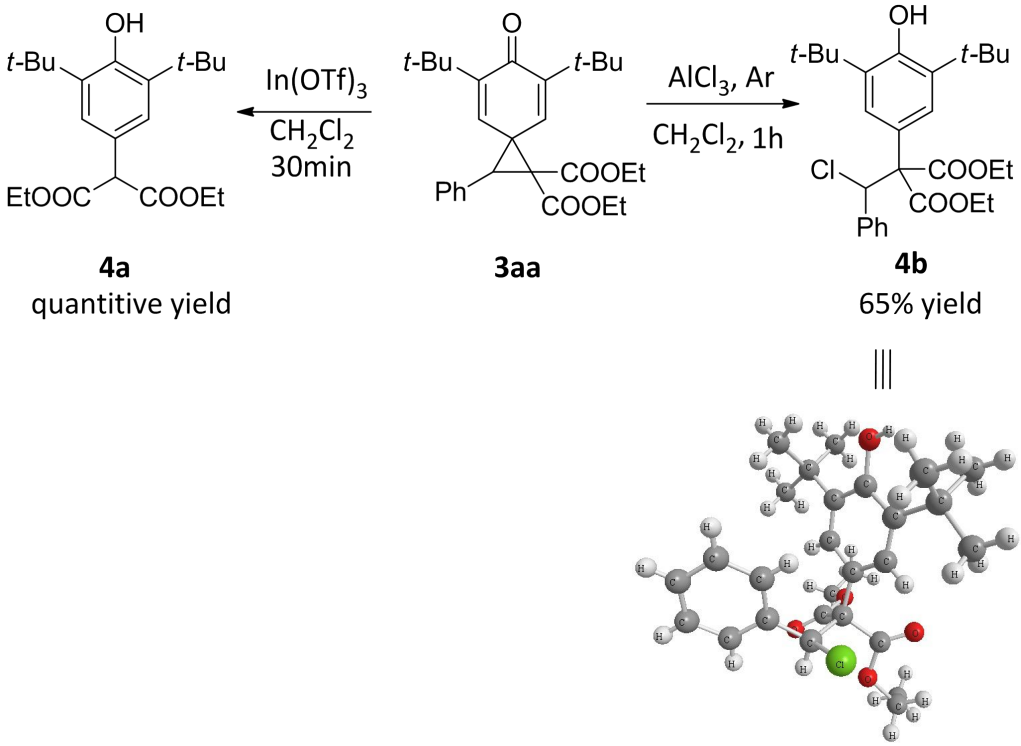


S8 **1t**

S8 was synthesized in the same manner as **S1** described before.

Treatment of **S8** (1 equiv) in THF with $LiAlH_4$ (1.5 equiv) was performed under Ar at 0°C. The mixture was slowly warmed to room temperature and stirred overnight. After quenching with 1N HCl, the mixture was extracted with EtOAc, and washed with brine. The combined organic phase was dried over anhydrous Na_2SO_4, and the filtrate was concentrated under reduced pressure. The residue obtained was purified by flash column chromatography on silica gel to afford the pure product **1t**.

4. Procedures for ring-open reactions



4a
quantitive yield

3aa

4b
65% yield

4a: In a 10 mL test tube was added **3aa** (0.05 mmol, 22.6 mg), $In(OTf)_3$ (0.01 mmol, 5.6 mg) and 0.1 mL CH_2Cl_2. The mixture was stirred at room temperature for 30 min until the color turned red. Then the solvent was removed under reduced pressure and the residue was purified by flash column chromatography (PE / EA = 20 : 1) on silica gel to afford **4a** as a colorless solid in quantitive yield.

White solid, m.p. 58 – 59 °C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.17 (s, 2H), 5.22 (s, 1H), 4.51 (s, 1H), 4.26 – 4.15 (m ,4H), 1.43 (s, 18H), 1.27 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 168.7, 153.8, 135.8, 126.0, 123.4, 61.6, 57.9, 34.3, 30.3, 14.1 ppm. **IR(KBr):** $\tilde{\nu}$ = 3608, 1949, 1753, 1722, 1629, 1436, 1218, 1151, 1036 cm^{-1}. **HRMS** (ESI): *m/z* calculated for $[C_{21}H_{33}O_5+H]^+$: 365.2323; found: 365.2322.

4b: In a 25 mL round bottom was added **3aa** (0.05 mmol, 22.6 mg), $AlCl_3$ (0.25 mmol, 34 mg) and 5 mL CH_2Cl_2 under Ar atmosphere. The mixture was stirred at room temperature for 1 h, and water was added to quench the reaction. Then the mixture was diluted with CH_2Cl_2 and washed with water. The combined organic phase was washed with brine. Then solvents were removed under reduced pressure and the residue was purified by flash column chromatography (PE / EA = 20 : 1) on silica gel to afford **4b** as a colorless solid (16mg, 65% yield).

White solid, m.p.153 - 155 °C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.15 – 7.12 (m, 1H), 7.06 – 7.01 (m, 2H), 6.92 (s, 2H), 6.83 – 6.81 (m, 2H), 5.95 (s, 1H), 5.23 (s, 1H), 4.46 – 4.30 (m, 2H), 4.23 (q, *J* = 7.1 Hz, 2H), 1.36 (t, *J* = 7.1 Hz, 3H), 1.32 (s, 18H), 1.22 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ =169.2, 167.8, 153.7, 137.0, 134.3, 130.0, 128.0, 127.6, 126.8, 122.6, 69.6, 65.0, 62.1, 62.0, 34.2, 30.2, 14.1, 14.0 ppm. **IR(KBr):** $\tilde{\nu}$ = 3610, 1865, 1762, 1731, 1630,

1439, 1249, 1182, 707 cm^{-1}. **HRMS** (ESI): *m/z* calculated for $[C_{28}H_{38}ClO_5+H]^+$: 489.2402; found: 489.2402.

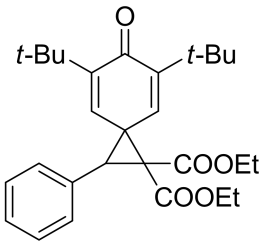
5. General procedure for the synthesis of spiro-compounds

a) General procedure

In a 10 mL test tube was sequencially added *p*-QMs **1** (0.1 mmol, 1equiv), Cs_2CO_3 (0.2 mmol, 2 equiv), **2** (0.15 mmol, 1.5 equiv) and CH_2Cl_2 (0.5 mL). Then, the tube was sealed and stirred overnight. After the reaction was completed (detected by TLC), solvent was directly removed under reduced pressure and the crude mixture was purified by flash column chromatography on silica gel to afford the pure product.

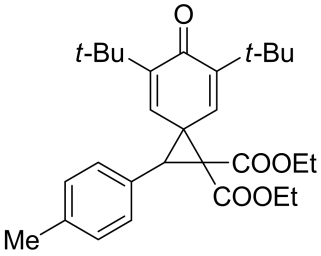
b) Characterization of the products

Diethyl 5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3aa)



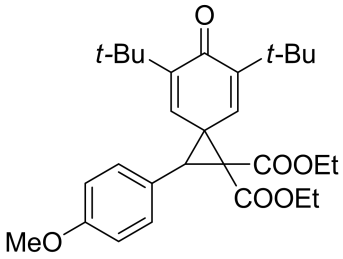
Light yellow solid, m.p. 155 - 156°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.38 - 7.31 (m, 3H), 7.26 - 7.24 (m, 2H), 6.79 (d, *J* = 2.8, 1H), 6.31 (d, *J* = 2.8, 1H), 4.39 – 4.28 (m, 2H), 4.20 (q, *J* = 7.1, 2H), 3.95 (s, 1H), 1.36 (t, *J* = 7.1 Hz, 3H), 1.28 (s, 9H), 1.26 (t, *J* = 7.1 Hz, 3H), 1.23 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.8, 167.3, 165.2, 150.3, 148.3, 137.3, 136.4, 132.2, 129.9, 128.3, 127.9, 62.5, 62.0, 50.2, 41.9, 37.9, 35.5, 35.2, 29.3, 29.3, 14.2, 14.0 ppm. **IR(KBr):** $\tilde{\nu}$ = 3009, 2957, 2867, 1734, 1648, 1621, 1239, 744, 701 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{28}H_{37}O_5+H]^+$: 453.2636; found: 453.2635.

Diethyl 5,7-di-*tert*-butyl-6-oxo-2-(p-tolyl)spiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ba)



White solid, m.p. 147 - 148°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.07 (m, 4H), 6.74 (d, *J* = 2.8, 1H), 6.23 (d, *J* = 2.8 Hz, 1H), 4.27 (m, 2H), 4.12 (q, *J* = 7.1 Hz, 2H), 3.85 (s, 1H), 2.30 (t, *J* = 7.1 Hz, 3H), 1.23 - 1.20 (m, 12H), 1.17 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.9, 167.4, 165.2, 150.2, 148.2, 137.6, 137.4, 136.6, 129.7, 129.1, 128.9, 62.4, 61.9, 50.2, 41.8, 37.9, 35.5, 35.2, 29.3, 29.2, 21.2, 14.2, 14.0 ppm. **IR(KBr):** $\tilde{\nu}$ = 2985, 2955, 2861, 1748, 1727, 1649, 1245 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{29}H_{39}O_5+H]^+$: 467.2792; found: 467.2788.

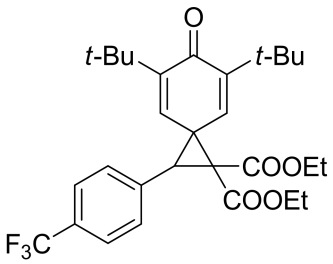
Diethyl 5,7-di-*tert*-butyl-2-(4-methoxyphenyl)-6-oxospiro[2.5]octa-4,7-diene-1,1 -dicarboxylate (3ca)



White solid, m.p. 148 - 149°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.12 (d, *J* = 8.4 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.71 (d, *J* = 2.7 Hz, 1H), 6.25 (d, *J* = 2.7 Hz, 1H), 4.33 - 4.22 (m, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 3.85 (s, 1H), 3.78 (s, 3H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.25 - 1.23 (m, 11H), 1.20 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.9, 167.3, 165.1, 159.0, 150.1, 148.2, 137.4, 136.6, 131.0, 124.0, 113.6, 62.4, 61.9, 55.2, 50.3, 41.4, 38.0, 35.5, 35.2, 29.3, 29.2, 14.2, 14.0 ppm. **IR(KBr):** $\tilde{\nu}$ = 2991, 2958, 2861, 1736, 1647, 1621, 1518, 1255, 1085,

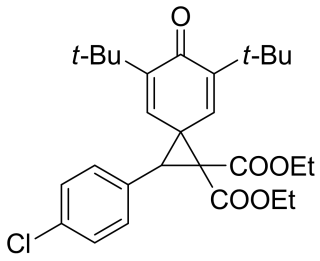
850 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{29}H_{39}O_6+H]^+$: 483.2741; found: 483.2737.

Diethyl 5,7-di-*tert*-butyl-6-oxo-2-(4-(trifluoromethyl)phenyl)spiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3da)



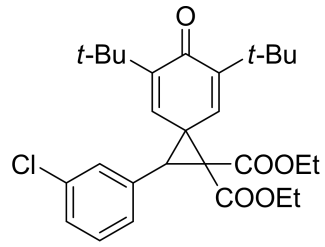
Light yellow solid, m.p. 135 - 136°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.57 (d, *J* = 8.2, 2H), 7.36 (d, *J* = 8.0 Hz, 2H), 6.63 (d, *J* = 2.7 Hz, 1H), 6.26 (d, *J* = 2.7 Hz, 1H), 4.39 - 4.25 (m, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 3.87(s, 1H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.24 - 1.22 (m, 12H), 1.95 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.6, 167.0, 164.9, 150.6, 149.0, 136.7, 136.4, 135.2, 130.3, 129.9, 125.7, 125.2 (q, $J_{C\text{-}F}$ = 3.6 Hz), 62.7, 62.2, 49.7, 41.2, 37.7, 35.5, 35.2, 29.3, 29.3, 14.1, 13.9 ppm. **IR(KBr):** $\tilde{\nu}$ = 2997, 2956, 2861, 1741, 1729, 1652, 1326, 1121, 1066 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{29}H_{36}F_3O_5+H]$: 521.2509; found: 521.2508 .

Diethyl 5,7-di-*tert*-butyl-2-(4-chlorophenyl)-6-oxospiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ea)



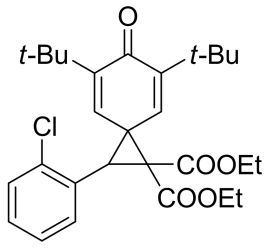
White solid, m.p. 150 - 151°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.30 (d, *J* = 8.3, 2H), 7.11 (d, *J* =8.3 Hz, 2H), 6.68 (d, *J* = 2.7 Hz, 1H), 6.28 (d, *J* = 2.7 Hz, 1H), 4.39-4.25 (m, 2H), 4.18 (q, *J* = 7.1 Hz, 2H), 3.85 (s, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.27 - 1.25 (m ,12H), 1.21 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 167.1, 164.9, 150.4, 148.7, 136.9, 135.7, 133.8, 131.3, 130.7, 128.5, 62.6, 62.1, 49.9, 41.1, 37.8, 35.5, 35.2, 29.3, 29.2, 14.1, 14.0 ppm. **IR(KBr)**: $\tilde{\nu}$ = 2958, 2861, 1734, 1648, 1621, 1399, 1246, 995 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{28}H_{36}ClO_5+H]^+$: 487.2246; found: 487.2248.

Diethyl 5,7-di-*tert*-butyl-2-(3-chlorophenyl)-6-oxospiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3fa)



White solid, m.p. 142 - 143°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.27 - 7.24 (m, 3H), 7.10 - 7.19 (m, 1H), 6.67 (d, *J* = 2.8 Hz, 1H), 6.24 (d, *J* = 2.8 Hz, 1H), 4.36 - 4.25 (m, 2H), 4.22 - 4.13 (qd, *J* = 7.1, 2.5 Hz, 2H), 3.85 (s, 1H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.25 (t, *J* = 7.1 Hz, 3H), 1.24 (s, 9H), 1.21 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 167.0, 164.8, 150.5, 148.7, 136.7, 135.7, 134.2, 134.1, 130.2, 129.5, 128.2, 128.1, 62.6, 62.2, 49.8, 41.0, 37.6, 35.5, 35.2, 29.3, 29.2, 14.2, 14.1 ppm. **IR(KBr):** $\tilde{\nu}$ = 2985, 2958, 2861, 1730, 1649, 1631, 1267, 1243, 933, 682 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{28}H_{36}ClO_5+H]^+$: 487.2246; found: 487.2241.

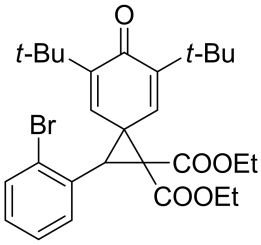
Diethyl 5,7-di-*tert*-butyl-2-(2-chlorophenyl)-6-oxospiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ga)



White solid, m.p. 108 - 109°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.38 (d, *J* = 7.5, 1H), 7.27 - 7.16 (m, 3H), 6.69 (d, *J* = 2.7 Hz, 1H), 6.28 (d, *J* = 2.7 Hz, 1H), 4.40 - 4.24 (m, 2H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 1H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.24 (s, 9H), 1.22 (t, *J* = 7.1 Hz, 3H), 1.17 (s,

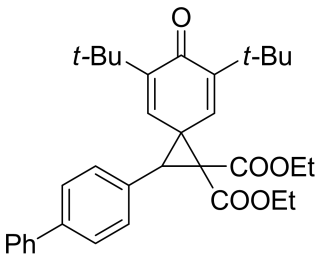
9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 167.1, 165.3, 150.5, 149.0, 137.3, 136.1, 135.1, 130.9, 130.5, 129.6, 129.2, 126.3, 62.4, 62.1, 49.1, 41.5, 38.6, 35.5, 35.2, 29.3, 29.1, 14.2, 13.8 ppm. **IR(KBr):** $\tilde{\nu}$ = 3003, 2961, 2861, 1733, 1649, 1620, 1257, 1240, 760 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{28}H_{36}ClO_5+H]^+$: 487.2246; found: 487.2245.

Diethyl 2-(2-bromophenyl)-5,7-di-*tert*-butyl-6-oxospiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ha)



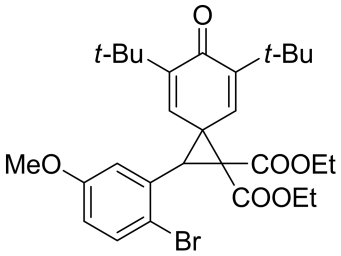
White solid, m.p. 126 - 127°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.58 (d, *J* = 7.7 Hz, 1H), 7.26 - 7.14 (m, 3H), 6.65 (d, *J* = 2.7 Hz, 1H), 6.32 (d, *J* = 2.7 Hz, 1H), 4.38 - 4.26 (m, 2H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 1H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.26-1.21 (m, 12H), 1.16 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 167.0, 165.3, 150.7, 149.0, 137.3, 135.1, 133.0, 132.7, 130.5, 129.4, 126.9, 126.3, 62.5, 62.2, 49.3, 43.6, 38.9, 35.4, 35.2, 29.3, 29.1, 14.2, 13.9 ppm. **IR(KBr):** $\tilde{\nu}$ = 2985, 2961, 2867, 1753, 1731, 1654, 1624, 1242, 751 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{28}H_{36}BrO_5+H]^+$: 531.1741; found: 531.1739.

Diethyl 2-([1,1'-biphenyl]-4-yl)-5,7-di-*tert*-butyl-6-oxospiro[2.5]octa-4,7-diene-1,1 -dicarboxylate (3ia)



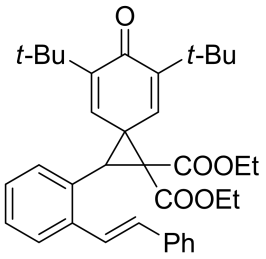
White solid, m.p. 147 - 148°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.60 – 7.53 (m, 4H), 7.46 – 7.25 (m, 5H), 6.80 (d, *J* = 2.8 Hz, 1H), 6.31 (d, *J* = 2.8 Hz, 1H), 4.37 – 4.16 (m, 4H), 3.94 (s, 1H), 1.33 (t, *J* = 7.2 Hz, 3H), 1.27 – 1.25 (m, 12H), 1.23 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.9, 167.3, 165.2, 150.4, 148.5, 140.7, 140.3, 137.2, 136.3, 131.3, 130.3, 128.8, 127.5, 127.0, 126.9 ,62.6, 62.1, 50.2, 41.7, 38.0, 35.5, 35.2, 29.4, 29.3, 14.2, 14.0 ppm. **IR(KBr)**: $\tilde{\nu}$ = 2962, 2867, 1729, 1647, 1637, 1618, 1253, 1089 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{34}H_{41}O_5+H]^+$: 529.2949; found: 529.2943.

Diethyl 2-(2-bromo-5-methoxyphenyl)-5,7-di-*tert*-butyl-6-oxospiro[2.5]octa-4,7-diene-1,1 -dicarboxylate (3ja)



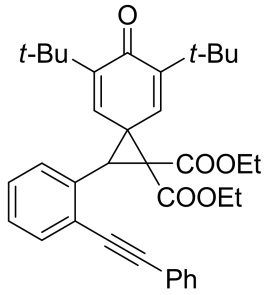
White solid, m.p. 131 - 132°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ= 7.45 (d, *J* = 8.8 Hz, 1H), 6.83 (d, *J* = 2.2 Hz, 1H), 6.72 (dd, *J* = 8.6, 2.7 Hz, 1H), 6.61 (d, *J* = 2.7 Hz, 1H), 6.29 (d, *J* = 2.7 Hz, 1H), 4.39 - 4.13 (m, 4H), 3.75 (s, 1H), 3.74 (s, 3H), 1.32 (t, *J* = 7.1 Hz, 3H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.23 (s, 9H), 1.16 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.8, 167.0, 165.3, 158.5, 150.7, 149.0, 137.2, 135.1, 133.6, 133.5, 133.4, 116.8, 114.7, 62.5, 62.2, 55.4, 49.3, 43.6, 38.8, 35.4, 35.2, 29.4, 29.1, 14.2, 13.9 ppm. **IR(KBr):** $\tilde{\nu}$ = 3001, 2959, 2855, 1735, 1654, 1624, 1245, 1165 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{29}H_{38}BrO_6+H]^+$: 561.1846; found: 561.1847.

(E)-diethyl 5,7-di-*tert*-butyl-6-oxo-2-(2-styrylphenyl)spiro[2.5]octa-4,7-diene-1,1 -dicarboxylate (3ka)



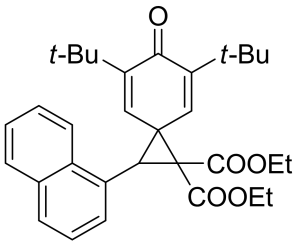
White solid, m.p. 137 - 138°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.71 (d, *J* = 7.8 Hz, 1H), 7.38 - 7.20 (m , 8H), 7.11 (d, *J* = 16.2 Hz, 1H), 6.97 (d, *J* = 16.2 Hz, 1H), 6.87 (d, *J* = 2.8 Hz, 1H), 6.22 (d, *J* = 2.8 Hz, 1H), 4.44 - 4.25 (m ,2H), 4.18 - 3.96 (m, 2H), 3.94 (s, 1H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.27 - 1.20 (m, 12H), 1.09 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.8, 167.6, 165.3, 151.0, 148.7, 137.8, 137.6, 137.0, 135.9, 130.5, 129.9, 128.6, 128.3, 127.8, 127.1, 126.6, 125.2, 125.1, 62.5, 62.2, 50.5, 41.7, 38.7, 35.5, 35.2, 29.4, 29.1, 14.2, 13.6 ppm. **IR(KBr):** $\tilde{\nu}$ = 2985, 2960, 2867, 1731, 1660, 1630, 1246, 1092, 759, 699 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{36}H_{43}O_5+H]^+$: 555.3105; found: 555.3106.

Diethyl 5,7-di-*tert*-butyl-6-oxo-2-(2-(phenylethynyl)phenyl)spiro[2.5]octa-4,7-diene-1,1 -dicarboxylate (3la)



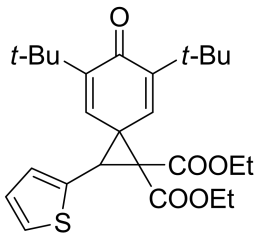
Brown solid, m.p. 120 - 121°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.58 - 7.55 (m, 1H), 7.46 - 7.43 (m, 2H), 7.31 - 7.26 (m , 5H), 7.18 (d, *J* = 6.4 Hz, 1H), 6.86 (d, *J* = 2.7 Hz, 1H), 6.28 (d, *J* = 2.7 Hz, 1H), 4.32 - 4.07 (m , 4H), 4.14 (s, 1H), 1.26 (t, *J* = 7.1 Hz, 3H), 1.18 (s, 9H), 1.17 (s, 9H), 1.09 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.9 ,167.3, 165.5, 150.4, 148.6, 137.9, 135.7, 134.4, 132.7, 131.7, 129.5, 128.4, 128.2, 127.8, 125.0, 123.0, 94.6, 87.1, 62.3, 62.0, 49.6, 42.4, 38.9, 35.5, 35.1, 29.7, 29.2, 14.2, 13.7 ppm. **IR(KBr):** $\tilde{\nu}$ = 3050, 2958, 2861, 1744, 1729, 1650, 1244, 756, 691 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{36}H_{41}O_5+H]^+$: 553.2949; found: 553.2943.

Diethyl 5,7-di-*tert*-butyl-2-(naphthalen-1-yl)-6-oxospiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ma)



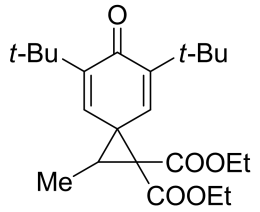
White solid, m.p. 128 - 129°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.86 - 7.78 (m, 3H), 7.49 - 7.36 (m, 5H), 6.78 (d, *J* = 2.7 Hz, 1H), 6.34 (d, *J* = 2.7 Hz, 1H), 4.74 - 4.26 (m, 2H), 4.18 (s, 1H), 4.10 - 3.95 (m, 2H), 1.38 (t, *J* = 7.1 Hz, 3H), 1.32 (s, 9H), 1.09 - 1.03 (m, 12H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 167.5, 165.3, 151.0, 148.4, 137.4, 136.4, 133.6, 132.8, 128.9, 128.6, 127.2, 126.3, 126.0, 124.7, 124.2, 62.6, 62.0, 50.1, 41.1, 38.4, 35.3, 29.4, 28.9, 14.3, 13.7 ppm. **IR(KBr):** $\tilde{\nu}$ = 2958, 2861, 1732, 1647, 1618, 1265, 1245, 1231, 771 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{32}H_{39}O_5+H]^+$: 503.2792; found: 503.2797.

Diethyl 5,7-di-*tert*-butyl-6-oxo-2-(thiophen-2-yl)spiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3na)



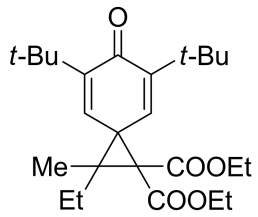
White solid, m.p. 149 - 150°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.26 - 7.25 (m, 2H), 6.96 - 6,94 (m, 1H), 6.84 (d, *J* = 2.9 Hz, 1H), 6.31 (d, *J* = 2.9 Hz, 1H), 4.37 - 4.13 (m, 4H), 3.92 (s, 1H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.26 (t, *J* = 7.1 Hz, 3H), 1.23 (s, 9H), 1.22 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 166.7, 164.7, 150.5, 148.8, 136.2, 135.7, 134.3, 128.5, 126.7, 126.0, 63.0, 62.2, 50.9, 38.6, 37.1, 35.5, 35.2, 29.3, 29.2, 14.2, 13.9 ppm. **IR(KBr):** $\tilde{\nu}$ = 3002, 2955, 2867, 1749, 1729, 1450, 1621, 1257, 1243, 1230, 1088 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{26}H_{35}O_5S+H]^+$: 459.2200; found: 459.2198.

Diethyl 5,7-di-*tert*-butyl-2-methyl-6-oxospiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3oa)



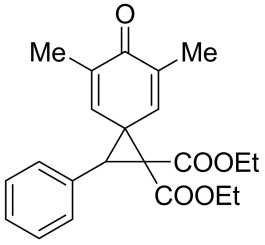
White solid, m.p. 93 - 94°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 6.67 (d, *J* = 2.7 Hz, 1H), 6.33 (d, *J* = 2.7 Hz, 1H), 4.28 - 4.13 (m, 4H), 2.63 (q, *J* = 6.7 Hz, 1H), 1.38 (d, *J* = 6.7 Hz, 3H), 1.29 (t, *J* = 7.2 Hz, 3H), 1.28 - 1.23 (m, 12H), 1.19 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 167.4, 165.6, 149.9, 149.5, 137.9, 134.7, 62.3, 61.9, 49.9, 37.8, 35.5, 35.1, 33.8, 29.3, 29.2, 14.1, 10.3 ppm. **IR(KBr):** $\tilde{\nu}$ = 2955, 2914, 2862, 1731, 1646, 1256, 1238, 919 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{23}H_{35}O_5+H]^+$: 391.2479; found: 391.2475.

Diethyl 5,7-di-*tert*-butyl-2-ethyl-2-methyl-6-oxospiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3pa)



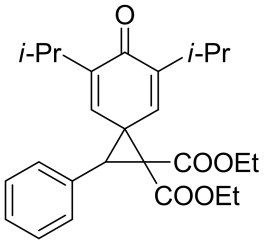
White solid, m.p. 90 - 91°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 6.83 (d, *J* = 3.0 Hz, 1H), 6.73 (d, *J* = 3.0 Hz, 1H), 4.22 (q, *J* = 7.1 Hz, 4H), 1.90 (m ,2H), 1.51 (s, 3H), 1.30 – 1.24 (m , 24H), 0.95 (t, *J* = 7.3 Hz, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.2, 166.4, 166.3, 149.3, 149.1, 136.7, 61.9, 61.8, 54.4, 44.7, 39.7, 35.5, 35.4, 30.3, 29.4, 29.3, 26.8, 16.8, 14.1, 10.5 ppm. **IR(KBr):** $\tilde{\nu}$ = 3003, 2961, 2873, 1736, 1647, 1620, 1459, 1387, 1094, 1023 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{25}H_{39}O_5+H]^+$: 419.2792; found: 419.2794.

Diethyl 5,7-dimethyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3qa)



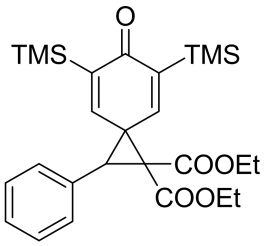
White solid, m.p. 87 - 88°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.31 - 7.29 (m, 3H), 7.22 - 7.20 (m, 2H), 6.81 (d, *J* = 1.3 Hz, 1H), 6.41 (d, *J* = 1.3 Hz, 1H), 4.37 - 4.27 (m ,2H), 4.25 - 4.16 (m , 2H), 3.93 (s, 1H), 1.96 (dd, *J* = 3.7, 1.0, 2H), 1.29 (t, *J* = 7.1 Hz, 3H), 1.26 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 186.6, 167.1, 165.0, 140.8, 139.5, 138.3, 136.9, 132.1, 129.8, 128.4, 128.0, 62.6, 62.2, 50.1, 41.9, 38.1, 16.8, 16.4, 14.0, 13.9 ppm. **IR(KBr)**: $\tilde{\nu}$ = 2982, 2920, 1732, 1660, 1633, 1370, 1243, 1092, 748, 700 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{22}H_{25}O_5+H]^+$: 369.1697; found: 369.1699.

Diethyl 5,7-diisopropyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ra)



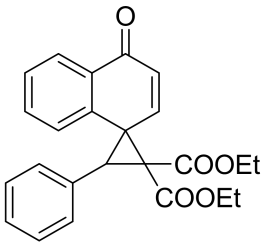
White solid, m.p. 76 - 77°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.31 - 7.28 (m, 3H), 7.21 - 7.19 (m, 2H), 6.73 (d, *J* = 3.0, 1H), 6.27 (d, *J* = 3.0 Hz, 1H), 4.36 – 4.23 (m, 2H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.96 (s, 1H), 3.11 (m, 2H), 1.31 (t, *J* = 7.1 Hz, 3H), 1.23 (t, *J* = 7.1 Hz, 3H), 1.09 - 0.99 (m, 12H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 184.6, 167.2, 165.1, 148.5, 146.6, 137.3, 136.6, 132.2, 129.9, 128.3, 127.9, 62.6, 62.1, 50.4, 42.1, 38.0, 26.8, 26.7, 22.0, 21.9, 21.8, 21.7, 14.1, 14.0 ppm. **IR(KBr)**: $\tilde{\nu}$ = 2979, 2956, 2867, 1749, 1725, 1655, 1627, 1251, 702 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{26}H_{33}O_5+H]^+$: 425.2323; found: 425.2321.

Diethyl 6-oxo-2-phenyl-5,7-bis(trimethylsilyl)spiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3sa)



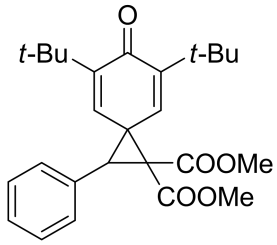
White solid, m.p. 150 - 151°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.27 – 7.24 (m, 3H), 7.19 – 7.07 (m, 2H), 7.07 (d, *J* = 3.1 Hz, 1H), 6.60 (d, *J* = 3.1 Hz, 1H), 4.26 (q, *J* = 7.1 Hz, 2H), 4.14 (qd, *J* = 7.1, 2.0 Hz, 2H), 3.93 (s, 1H), 1.27 (t, *J* = 7.1 Hz, 3H), 1.18 (t, *J* = 7.1 Hz, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 190.1, 165.4, 163.3, 150.8, 149.9, 143.5, 141.5, 130.3, 128.2, 126.8, 126.5, 61.1, 60.7, 49.7, 41.2, 37.1, 12.6, 12.4, -3.0, -3.1 ppm. **IR(KBr):** $\tilde{\nu}$ = 2985, 2961, 1734, 1615, 1599, 1244, 860, 838. **HRMS (ESI)**: *m/z* calculated for $[C_{26}H_{37}O_5Si_2+H]^+$: 485.2174; found: 485.2171.

Diethyl 4'-oxo-3-phenyl-4'H-spiro[cyclopropane-1,1'-naphthalene]-2,2-dicarboxylate (3ta)



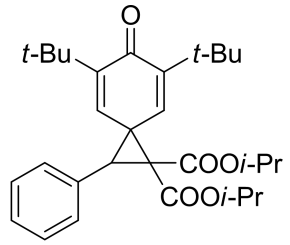
White solid, m.p. 163 - 164°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 8.31-8.28 (m, 1H), 7.54 – 7.71 (m, 9H), 6.63 (d, *J* = 10.6 Hz, 1H), 4.44 (s, 1H), 4.22 – 4.15 (m, 2H), 4.15 – 3.78 (m, 2H), 1.24 (t, *J* = 7.2 Hz, 3H), 0.83 (t, *J* = 7.2 Hz, 3H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 184.8, 165.8, 164.9, 145.6, 138.5, 133.8, 132.1, 131.7, 129.6, 129.1, 128.7, 128.0, 127.7, 127.5, 122.8, 62.4, 62.1, 54.2, 40.9, 37.6, 13.9, 13.5 ppm. **IR(KBr):** $\tilde{\nu}$ = 2991, 2949, 1741, 1727, 1658, 1246, 1217, 1187, 844, 762, 703. **HRMS (ESI)**: *m/z* calculated for $[C_{24}H_{23}O_5+H]^+$: 391.1540; found: 391.1539.

Dimethyl 5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ab)



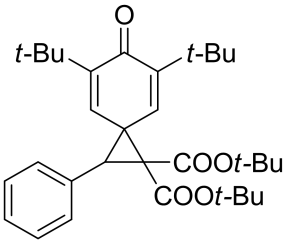
White solid, m.p. 158 - 159°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.34 - 7.28 (m, 3H), 7.20 - 7.17 (m ,2H), 6.72 (d, *J* = 3.0, 1H), 6.26 (d, *J* = 3.0, 1H), 3.92 (s, 1H), 3.82 (s, 3H), 3.70 (s, 3H), 1.23 (s, 9H), 1.19 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ =185.7, 167.7, 165.5, 150.5, 148.5, 137.1, 136.1, 132.1, 129.8, 128.3, 127.9, 53.3, 52.8, 49.8, 42.1, 37.9, 35.5, 35.2, 29.3, 29.2 ppm. **IR(KBr):** $\tilde{\nu}$ = 2991, 2955, 2855, 1752, 1735, 1650, 1622, 1437, 1250, 1088, 702 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{26}H_{33}O_5+H]^+$: 425.2323; found: 425.2322.

Diisopropyl 5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ac)



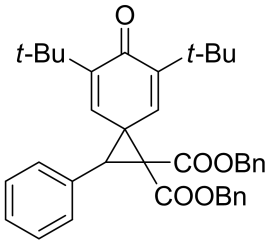
White solid, m.p. 180 - 181°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.30 – 7.27 (m, 3H), 7.24 – 7.21 (m, 2H), 6.80 (d, *J* = 2.9 Hz, 1H), 6.26 (d, *J* = 2.9 Hz, 1H), 5.16 (m, 1H), 5.03 (m, *J* = 6.3 Hz, 1H), 3.90 (s, 1H), 1.32 – 1.20 (m, 30H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 186.0, 166.9, 164.6, 150.2, 148.1, 137.4, 136.7, 132.4, 129.9, 128.2, 127.8, 70.5, 69.9, 50.6, 41.7, 37.8, 35.5, 35.2, 29.3, 29.3, 21.9, 21.7, 21.6, 21.5 ppm. **IR(KBr):** $\tilde{\nu}$ = 2956, 2861, 1726, 1647, 1620, 1256, 1109, 1084, 930, 745, 701. **HRMS (ESI)**: *m/z* calculated for $[C_{30}H_{41}O_5+H]^+$: 481.2949; found: 481.2947.

Di-tert-butyl 5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ad)



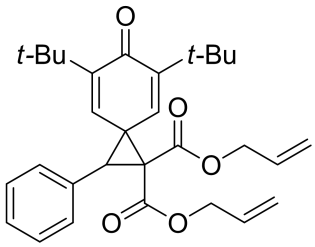
Yellow solid, m.p. 157 - 158°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.28 - 7.26 (m, 5H), 6.73 (d, *J* = 2.7 Hz, 1H), 6.33 (d, *J* = 2.7 Hz, 1H), 3.81 (s, 1H), 1.53 (s, 9H), 1.43 (s, 9H), 1.26 (s, 9H), 1.21 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 186.2, 166.4, 163.8, 149.9, 147.6, 137.6, 137.4, 132.7, 130.1, 128.0, 127.7, 83.0, 82.8, 52.3, 41.4, 37.7, 35.4, 35.2, 29.4, 29.3, 28.0, 27.9 ppm. **IR(KBr):** $\tilde{\nu}$ = 3000, 2957, 2867, 1723, 1648, 1618, 1371, 1303, 699, 507, 463 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{32}H_{44}NaO_5+Na]^+$: 531.3081; found: 531.3080.

Dibenzyl 5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ae)

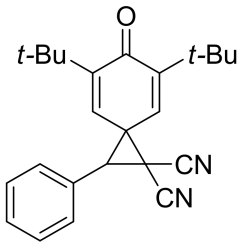


White solid, m.p. 147 - 148°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.31 - 7.14 (m, 15H), 6.74 (d, *J* = 2.8 Hz, 1H), 6.21 (d, *J* = 2.8 Hz, 1H), 5.19 (s, 2H), 5.05 (dd, J = 28.5, 12 Hz, 2H), 3.93 (s, 1H), 1.17 (s, 9H), 1.14 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.7, 167.2, 165.0, 150.4, 148.5, 137.0, 136.1, 134.9, 134.6, 132.1, 129.9, 128.8, 128.7, 128.6, 128.6, 128.3, 127.9, 68.3, 67.9, 50.1, 42.1, 38.3, 35.5, 35.1, 29.3, 29.2 ppm. **IR(KBr):** $\tilde{\nu}$ = 2955, 2920, 1730, 1648, 1618, 1369, 1170, 699 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{38}H_{40}NaO_5+Na]^+$: 599.2768; found: 599.2765.

Diallyl 5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3af)

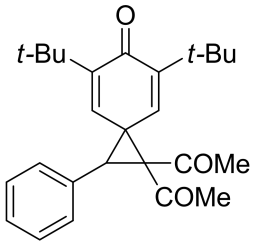


White solid. m.p. 118 - 119°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.31 – 7.28 (m, 3H), 7.27 – 7.20 (m, 2H), 6.75 (d, *J* = 2.9 Hz, 1H), 6.28 (d, *J* = 2.9 Hz. 1H), 5.93 – 5.82 (m, 2H), 5.40 – 5.22 (m, 4H), 4.75 – 4.72 (m, 2H), 4.60 – 4.58 (m, 2H), 3.94 (s, 1H), 1.25 (s, 9H), 1.20 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.8, 167.0, 164.8, 150.4, 148.5, 137.1, 136.1, 132.1, 131.2, 131.1, 129.9, 128.3, 128.0, 119.6, 119.5, 67.1, 66.7, 49.9, 42.1, 38.1, 35.5, 35.2, 29.3, 29.2 ppm. **IR(KBr):** $\tilde{\nu}$ = 2944 1736, 1647, 1620, 1231, 1171, 963, 916, 700 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{30}H_{36}O_5+H]^+$: 477.2636; found: 477.2625.



5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarbonitrile (3ag)

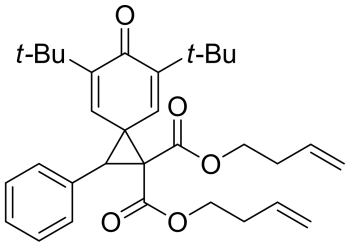
Yellow solid, m.p. 165 - 166°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.52 - 7.49 (m, 3H), 7.38 – 7.34 (m, 2H), 6.42 (d, *J* = 3.0 Hz, 1H), 6.12 (d, *J* = 3.0 Hz, 1H), 3.82 (s, 1H), 1.38 (s, 9H), 1.24 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 184.4, 153.7, 153.5, 134.1, 131.1, 129.7, 129.6, 129.3, 128.3, 113.2, 111.1, 44.1, 40.5, 35.9, 35.7, 29.2, 29.1, 20.2 ppm. **IR(KBr):** $\tilde{\nu}$ = 2956, 2919, 2865, 2239, 1659, 1625, 1448, 1381, 913, 696 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{24}H_{27}N_2O+H]^+$: 359.2118; found: 359.2109.



1,1'-(5,7-di-*tert*-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-diyl)diethanone (3ah)

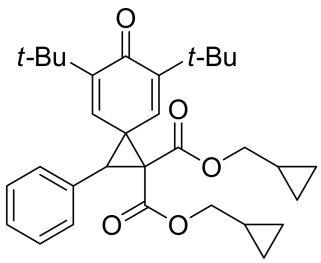
White solid. m.p. 129 - 130°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.29 – 7.24 (m, 3H), 7.00 (m, 2H), 6.69 (d, *J* = 3.1 Hz, 1H), 5.92 (d, *J* = 3.1 Hz, 1H), 4.35 (d, *J* = 1.0 Hz, 1H), 2.46 (s, 3H), 1.92 (s, 3H), 1.27 (s, 9H), 0.86 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 194.6, 185.2, 167.7, 146.3, 146.0, 137.4, 136.8, 128.4, 127.4, 114.4, 84.5, 58.5, 34.3, 34.0, 29.2, 28.9, 28.3, 14.8 ppm. **IR(KBr):** $\tilde{\nu}$ = 2958, 2914, 1673, 1650, 1600, 1365, 1275, 1261, 931, 750 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{26}H_{32}O_3+H]^+$: 393.2419; found: 393.2424.

Di(but-3-en-1-yl) 5,7-di-tert-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3ai)



White solid. m.p. 83 - 84°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.29 – 7.28 (m, 3H), 7.24 – 7.17 (m, 2H), 6.73 (d, *J* = 3.0 Hz, 1H), 6.25 (d, *J* = 3.0 Hz, 1H), 5.76 - 5.68 (m, 2H), 5.15 – 5.04 (m, 4H), 4.33 – 4.20 (m, 2H), 4.17 – 4.08 (m, 2H), 3.89 (s, 1H), 2.45 – 2.31 (m, 4H), 1.23 (s, 9H), 1.19 (s, 9H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.8, 167.2, 165.1, 150.4, 148.4, 137.2, 136.2, 133.4, 133.2, 132.1, 129.9, 128.3, 127.9, 117.7, 117.6, 65.6, 65.2, 50.1, 42.1, 38.0, 35.5, 35.2, 32.8, 32.6, 29.3, 29.3 ppm. **IR(KBr):** $\tilde{\nu}$ = 2954, 1759, 1733, 1648, 1628, 1230, 1247, 1188, 930, 704 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $[C_{32}H_{40}O_5+H]^+$: 505.2949; found: 505.2941.

Bis(cyclopropylmethyl) 5,7-di-tert-butyl-6-oxo-2-phenylspiro[2.5]octa-4,7-diene-1,1-dicarboxylate (3aj)



White solid. m.p. 158 - 159°C. **^{1}H NMR** (300 MHz, $CDCl_3$) δ = 7.30 - 7.27 (m, 5H), 6.80 (s, 1H), 6.30 (s, 1H), 4.09 (d, *J* = 7.1 Hz, 2H), 4.03 - 3.91 (m, 3H), 1.26 (s, 9H), 1.22 (s, 9H), 1.10 (m, 2H), 0.62 - 0.55 (m, 4H), 0.34 - 0.33 (m, 2H), 0.25 (m, 2H) ppm. **^{13}C NMR** (75 MHz, $CDCl_3$) δ = 185.9, 167.5, 165.4, 150.3, 148.3, 137.4, 136.6, 132.3, 129.9, 128.3, 127.9, 71.4, 71.0, 50.3, 41.9, 38.0, 35.5, 35.2, 29.3, 29.3, 9.7, 9.6, 3.6, 3.5, 3.4 ppm. **IR(KBr):** $\tilde{\nu}$ = 2953, 1734, 1619, 1230, 1255, 1238, 1171, 958, 706 cm^{-1}. **HRMS (ESI)**: *m/z* calculated for $C_{32}H_{40}O_5$: 505.2949; found: 505.2941 $[M+H]^+$.

6. References

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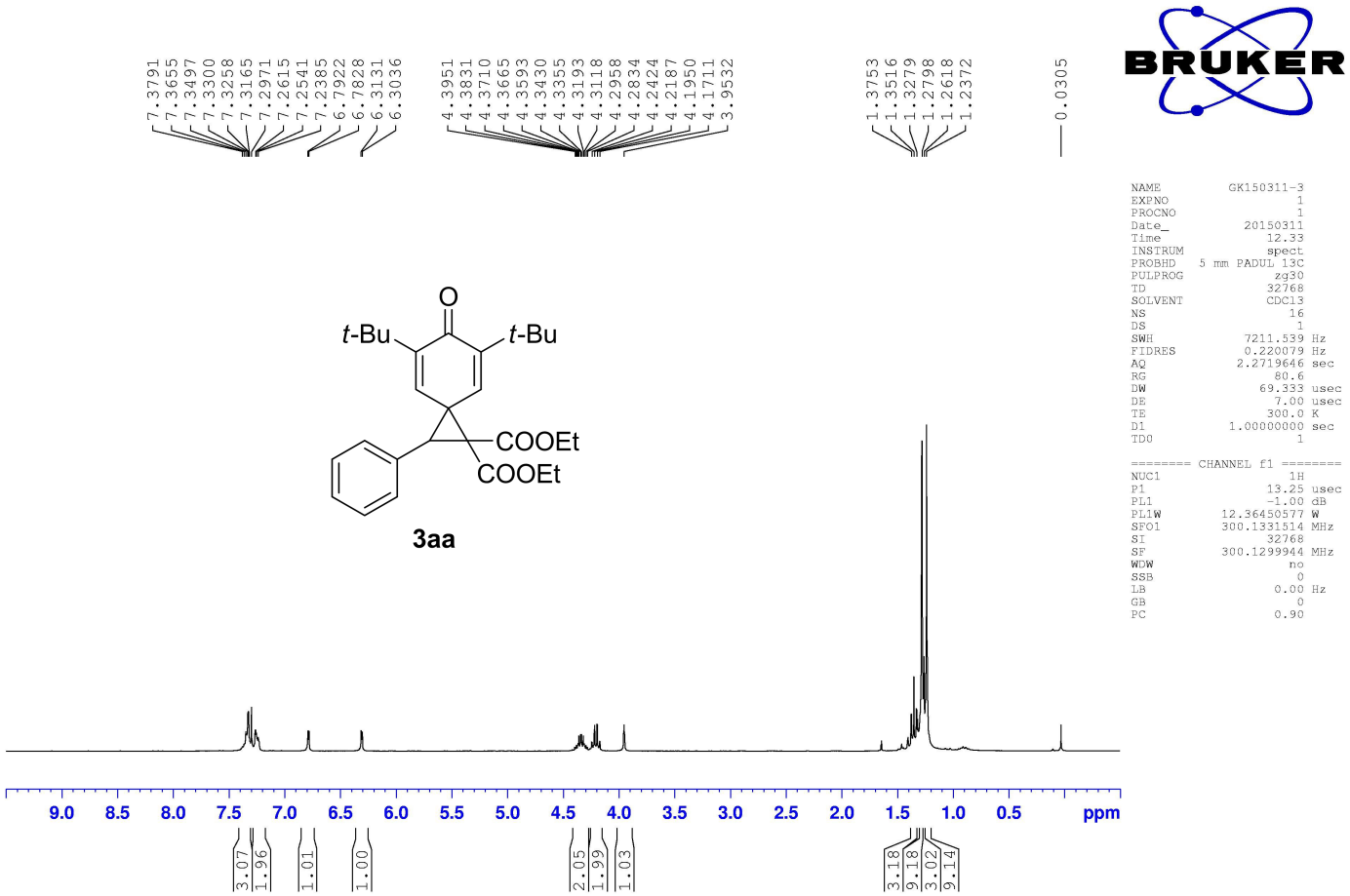
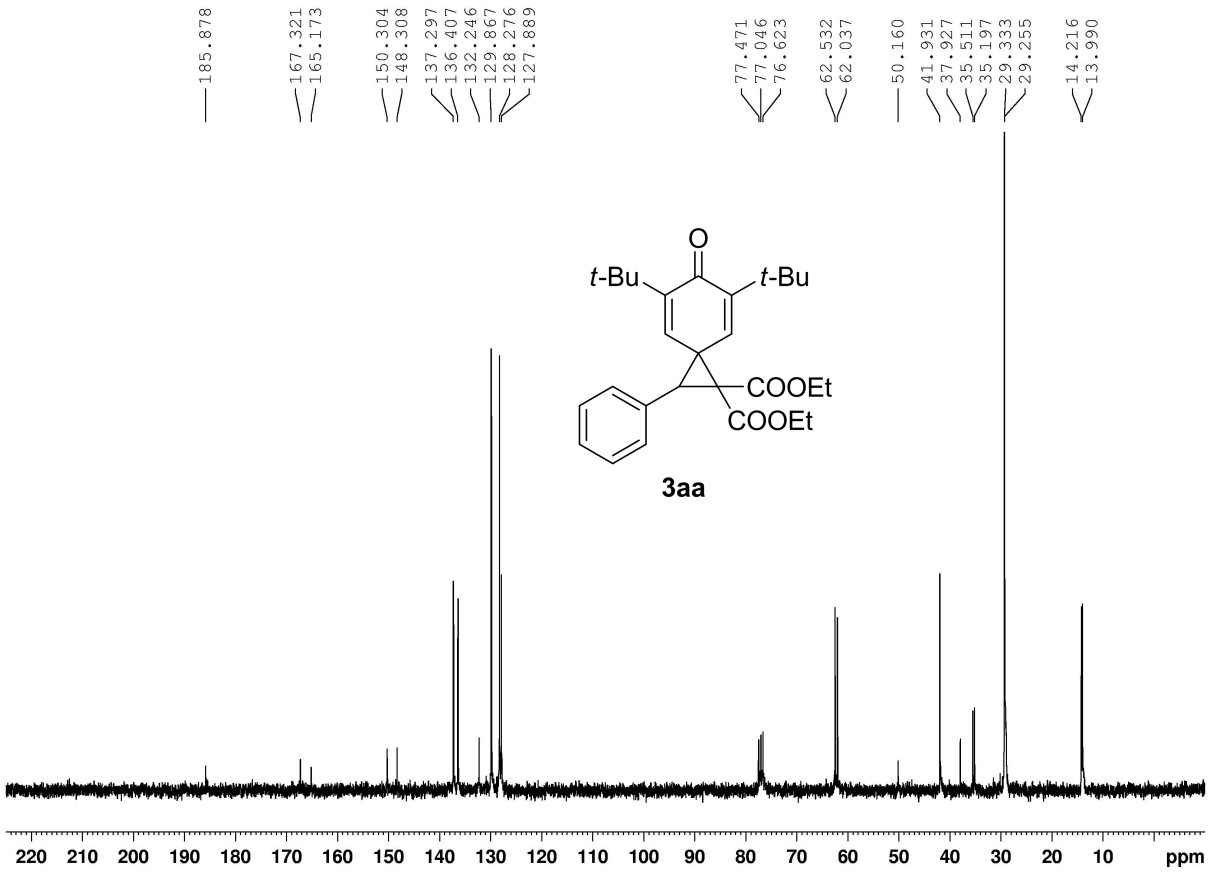
7. ^{1}H and ^{13}C NMR Specra of Title Compounds





BRUKER

NAME	GK150311-C
EXPNO	1
PROCNO	1
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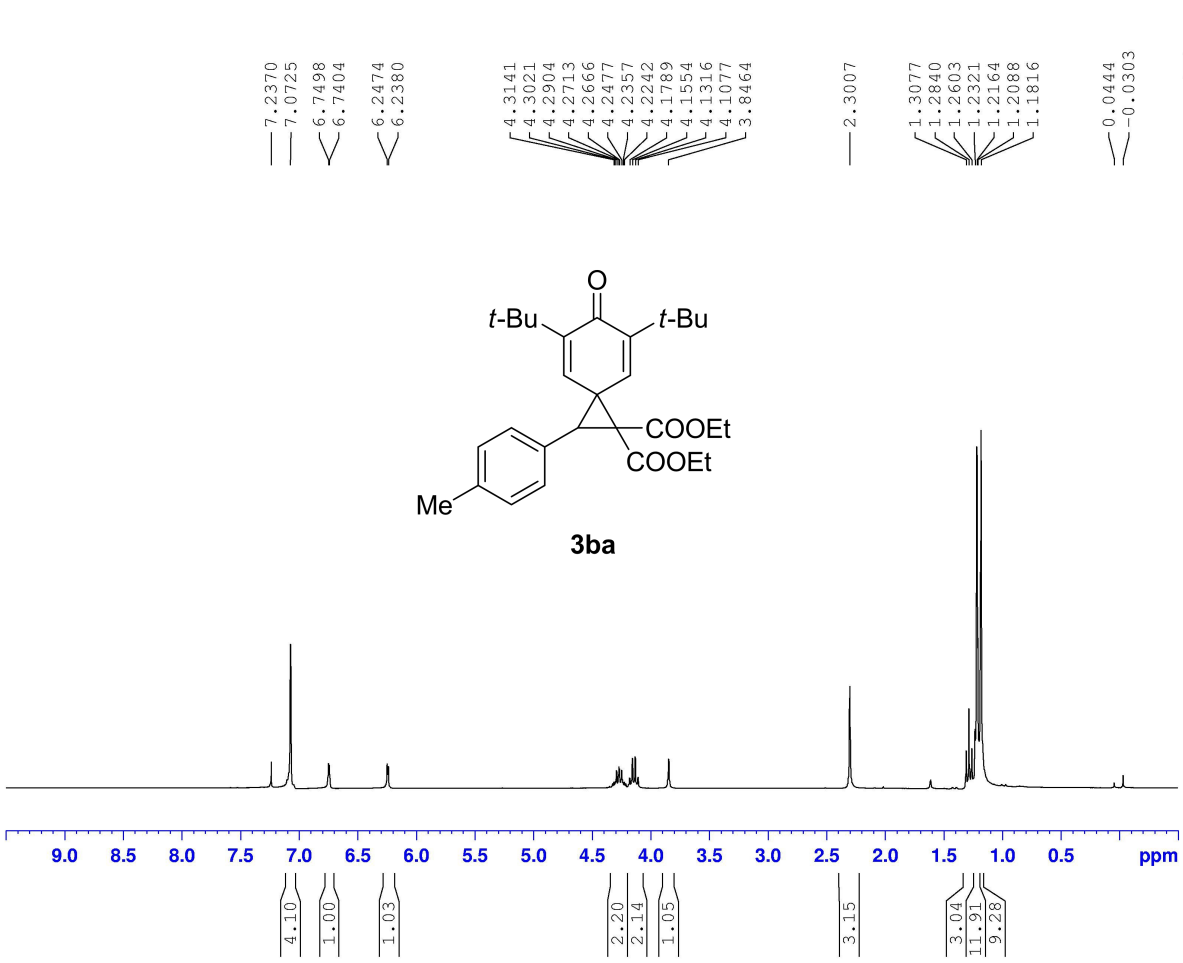
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	255
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	40.3
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

NAME	GK150325-2
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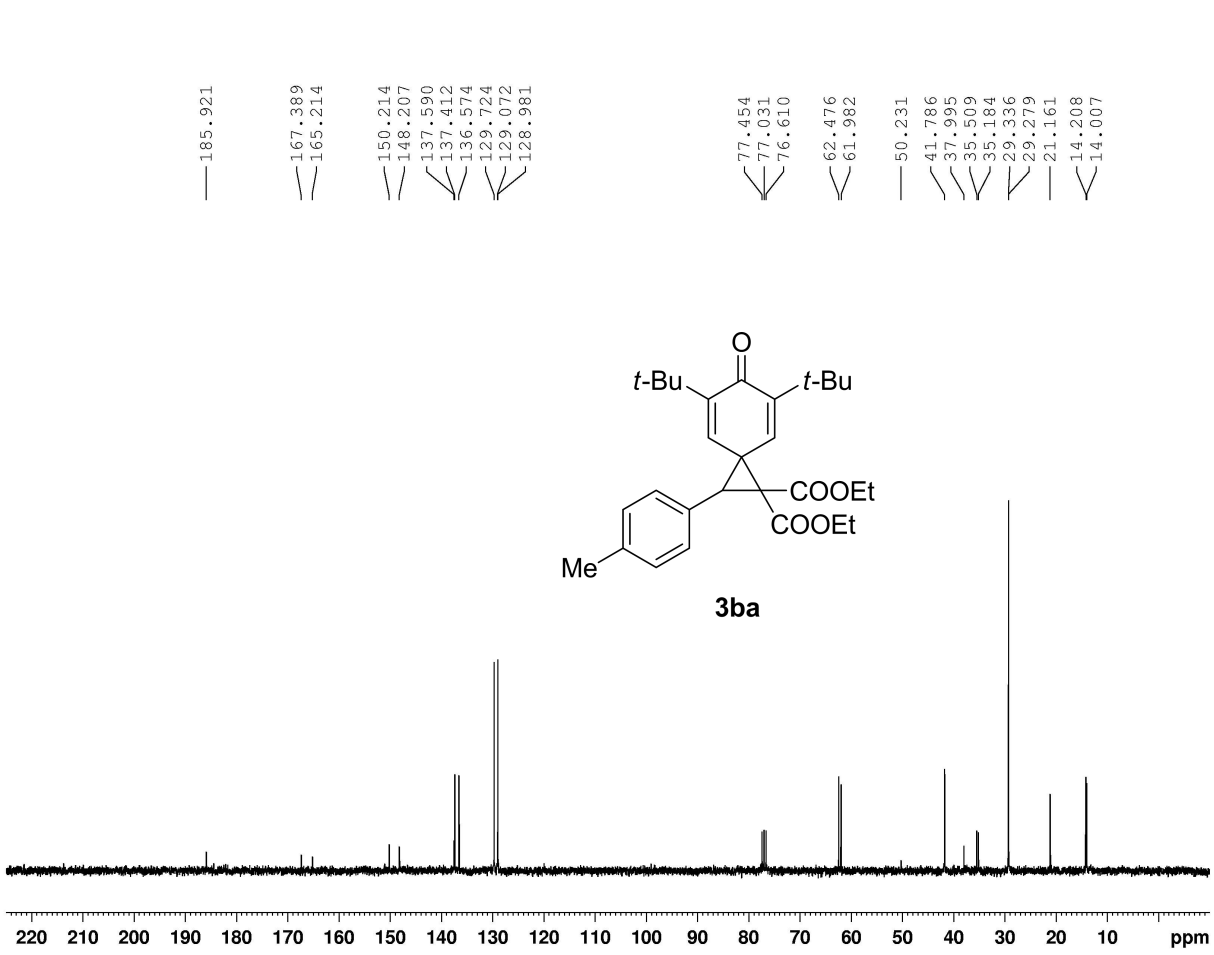
PROCNO	1
Date_	20120403
Time	13.47
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	1
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	71.8
DW	69.333 usec
DE	7.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.25 usec
PL1	-1.00 dB
PL1W	12.36450577 W
SFO1	300.1331514 MHz
SI	32768
SF	300.1300125 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.90



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PROCNO	1
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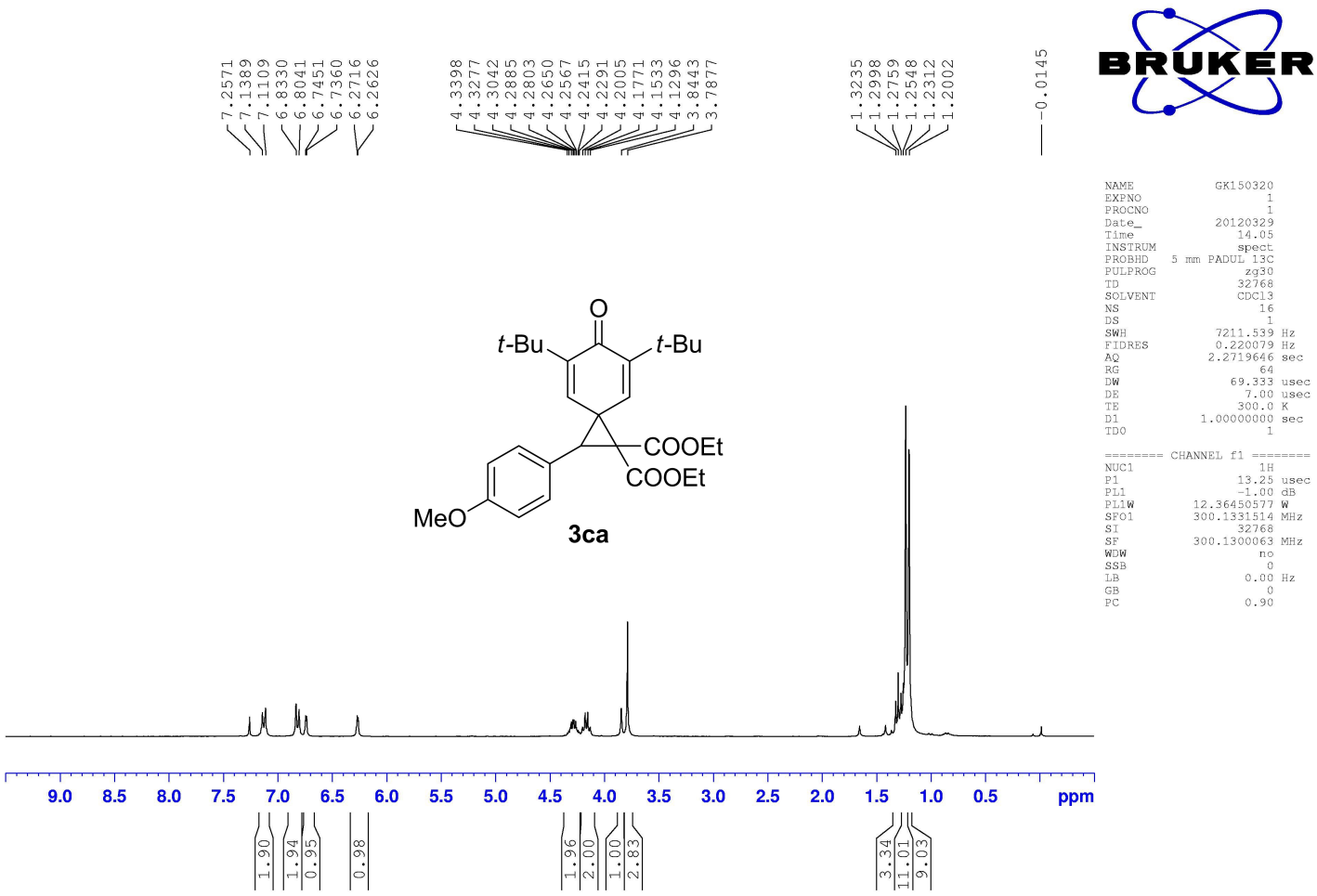
Time	13.50
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	212
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	36
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

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PROCNO               1
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PULPROG           zg30
TD               32768
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NS                  16
DS                   1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719646 sec
RG                  64
DW               69.333 usec
DE                7.00 usec
TE               300.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               13.25 usec
PL1              -1.00 dB
PL1W       12.36450577 W
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GB                   0
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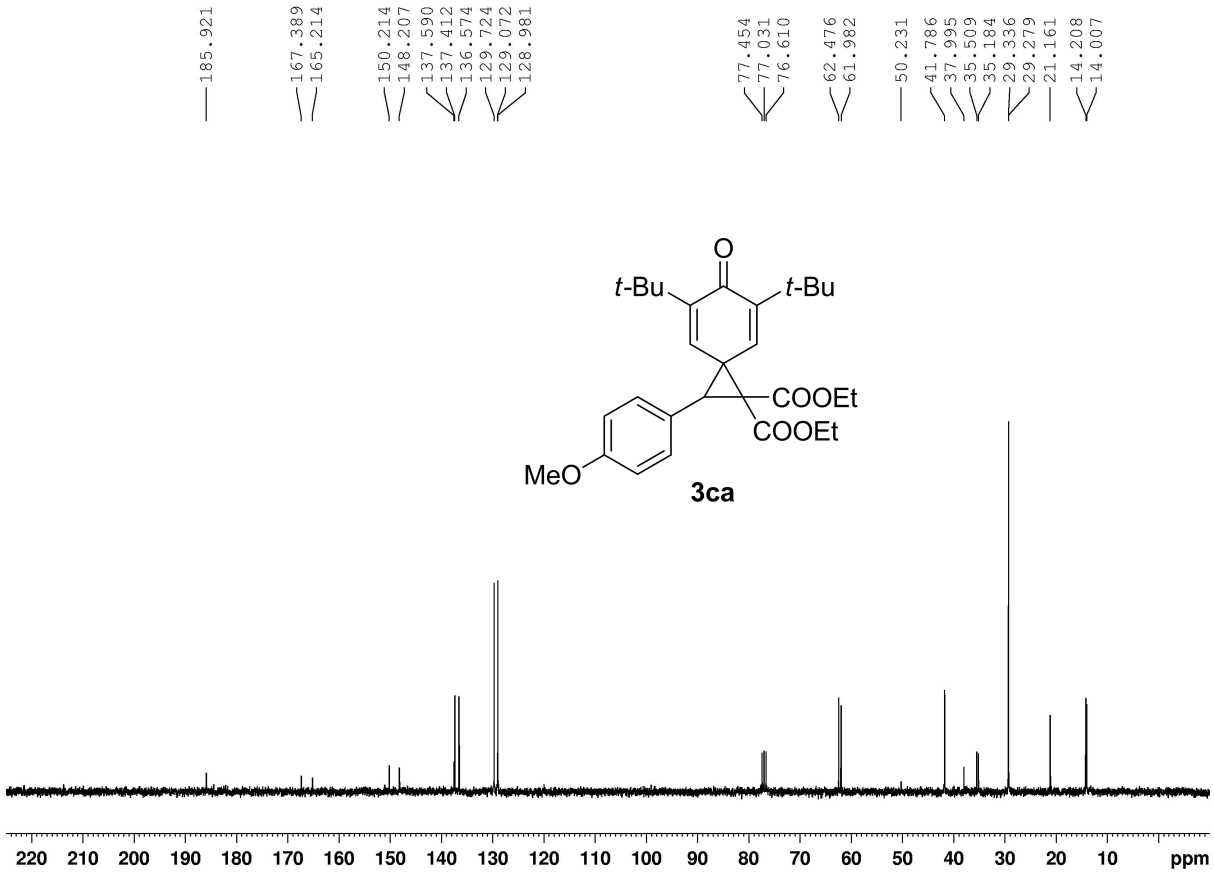

BRUKER

NAME	GK150325-2-C
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PROCNO	1
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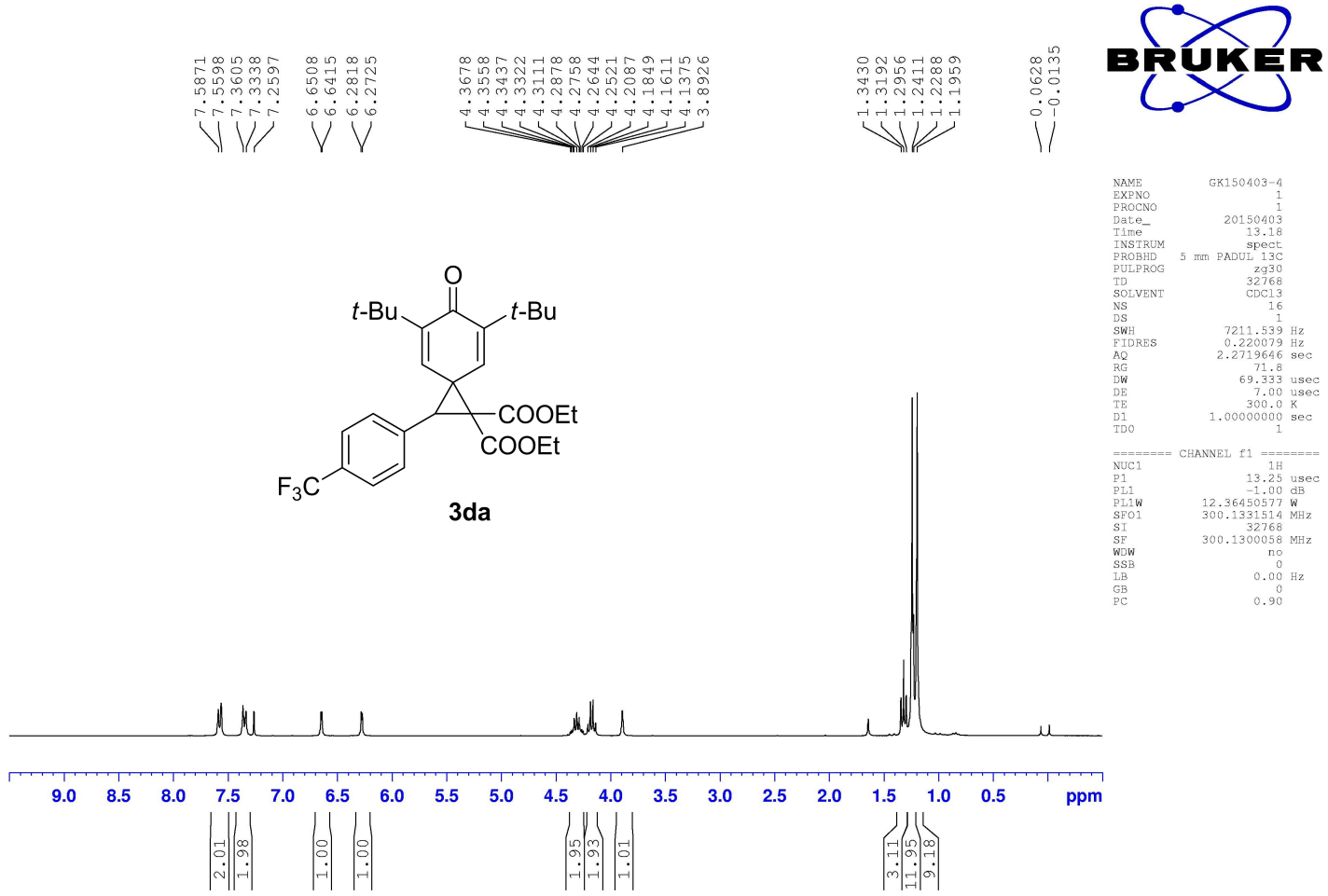
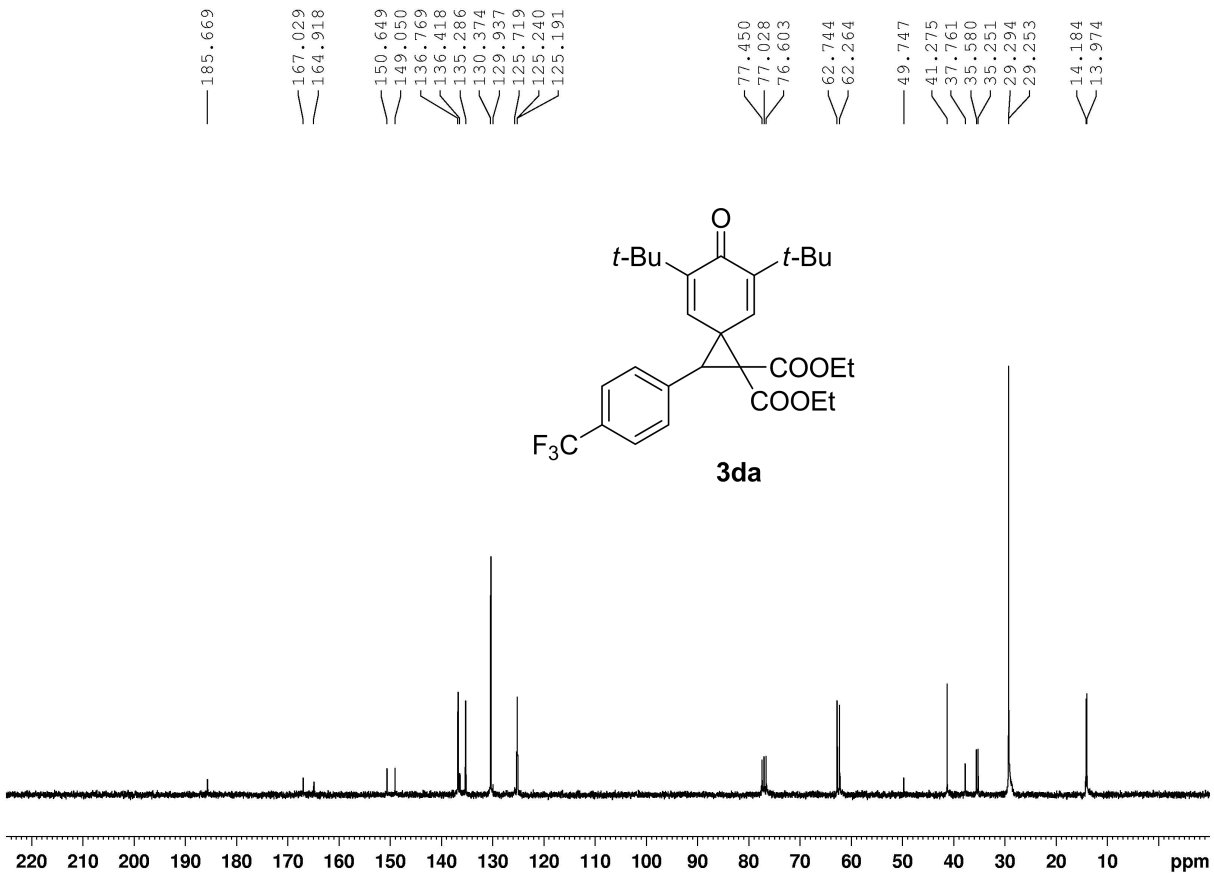
SOLVENT	CDCl3
NS	212
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	36
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



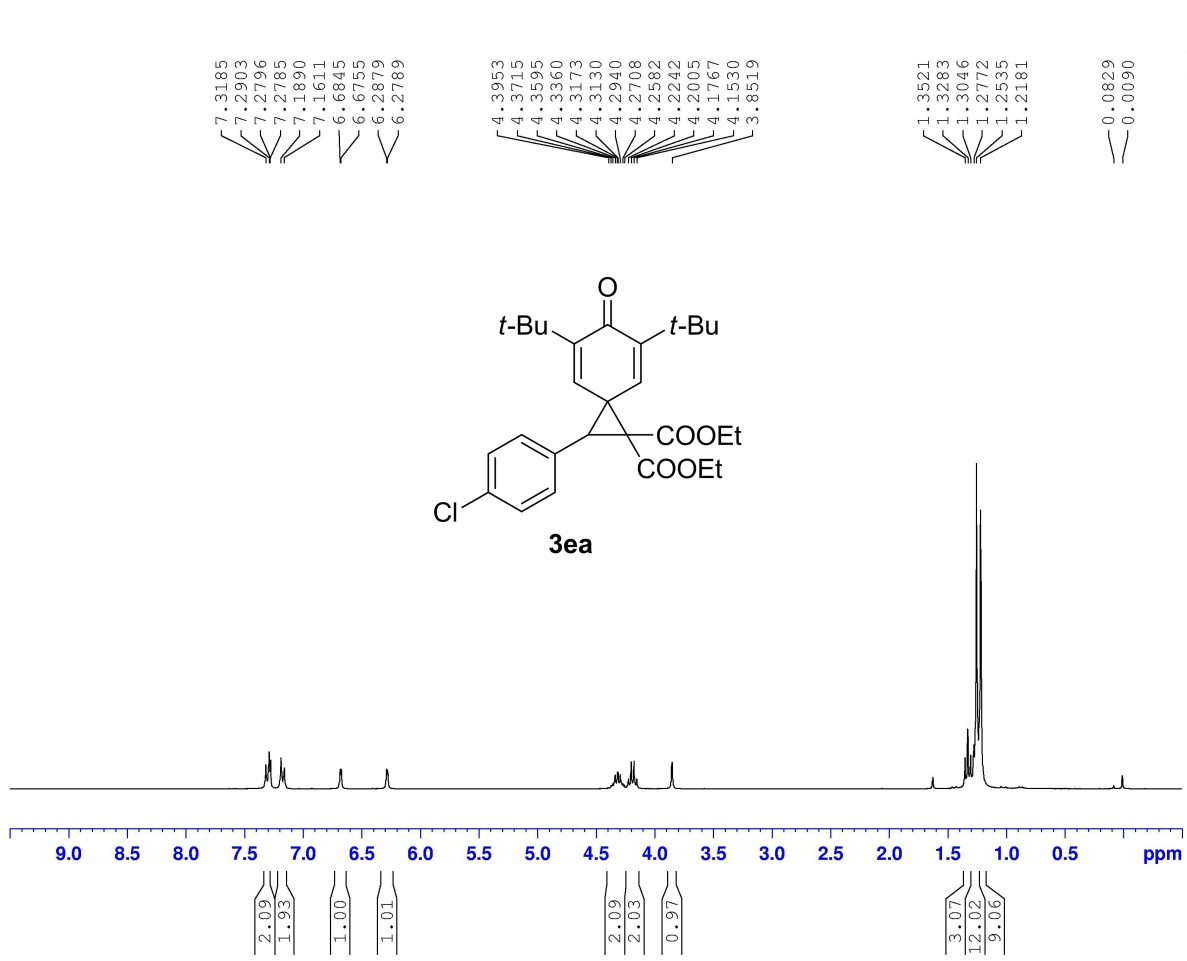

```
NAME          GK150403-4
EXPNO                  1
PROCNO                 1
Date_           20150403
Time               13.18
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719646 sec
RG                  71.8
DW             69.333 usec
DE               7.00 usec
TE               300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1             13.25 usec
PL1              -1.00 dB
PL1W       12.36450577 W
SFO1       300.1331514 MHz
SI                 32768
SF         300.1300058 MHz
WDW                   no
SSB                    0
LB              0.00 Hz
GB                     0
PC                  0.90
```



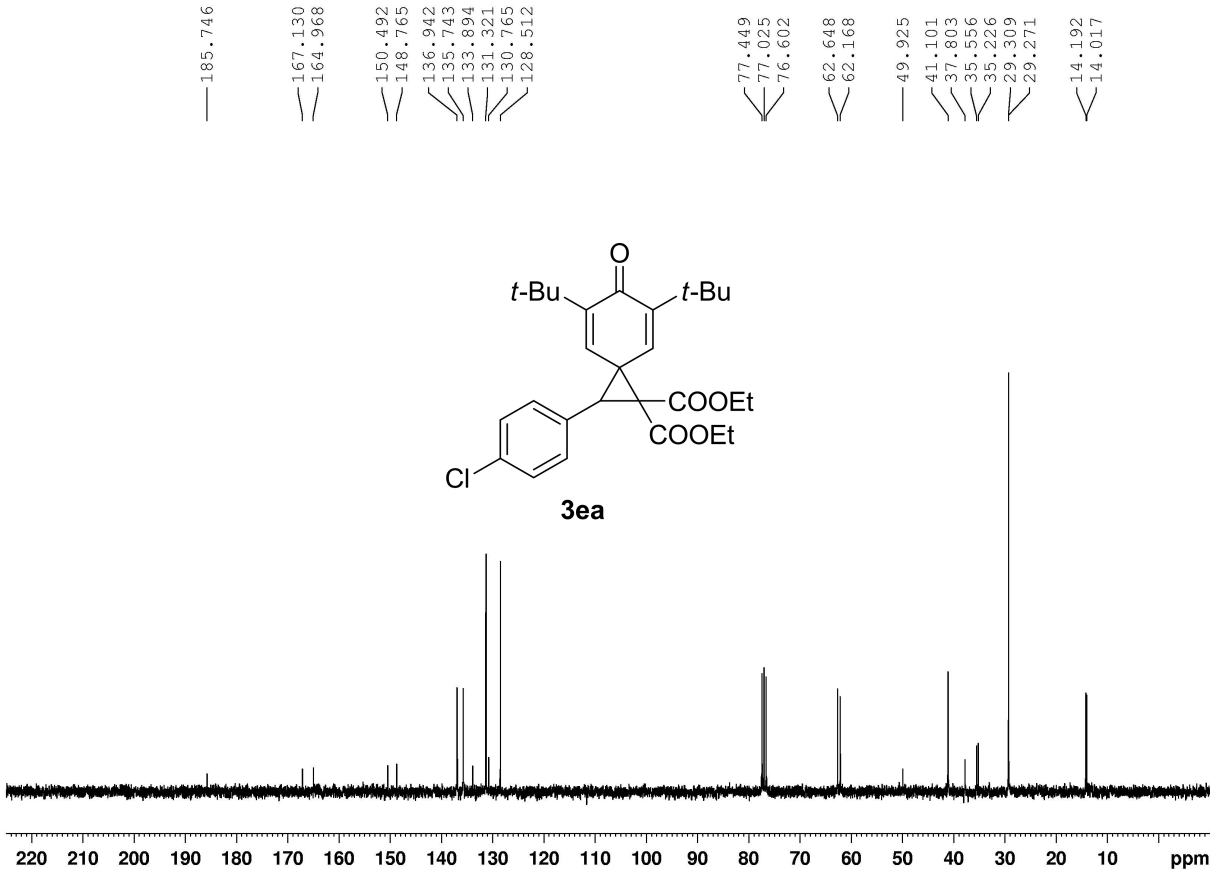
BRUKER

NAME	GK-150412-4-C
EXPNO	1
PROCNO	1
Date_	20150412
Time	12.29
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	526
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



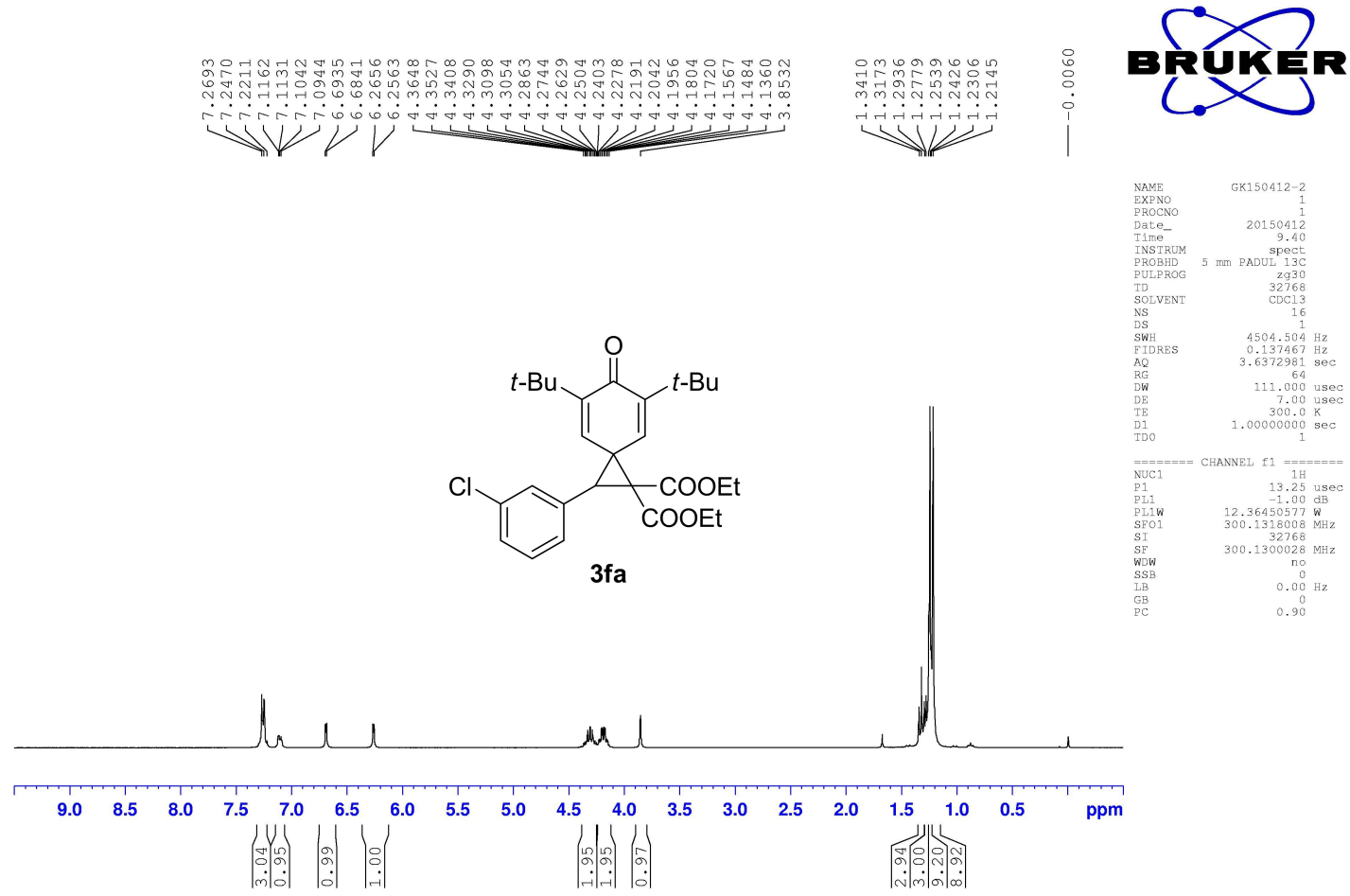
BRUKER

NAME	GK150325-1
EXPNO	1
PROCNO	1
Date_	20120403
Time	13.30
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	1
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	71.8
DW	69.333 usec
DE	7.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.25 usec
PL1	-1.00 dB
PL1W	12.36450577 W
SFO1	300.1331514 MHz
SI	32768
SF	300.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.90



BRUKER

NAME	GK150325-1-C
EXPNO	1
PROCNO	1
Date_	20120403
Time	13.36
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	189
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	36
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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7.2211
7.1162
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7.0944
6.6935
6.6841
6.2656
6.2563
4.3648
4.3527
4.3408
4.3290
4.3098
4.3054
4.2863
4.2744
4.2629
4.2504
4.2403
4.2278
4.2191
4.2042
4.1956
4.1804
4.1720
4.1567
4.1484
4.1360
3.8532
1.3410
1.3173
1.2936
1.2779
1.2539
1.2426
1.2306
1.2145
0.0060
BRUKER
NAME GK150412-2
EXPNO 1
PROCNO 1
Date_ 20150412
Time 9.40
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 4504.504 Hz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 64
DW 111.000 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.25 usec
PL1 -1.00 dB
PL1W 12.36450577 W
SFO1 300.1318008 MHz
SI 32768
SF 300.1300028 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.90
O
t-Bu
t-Bu
Cl
COOEt
COOEt
3fa
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
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ppm
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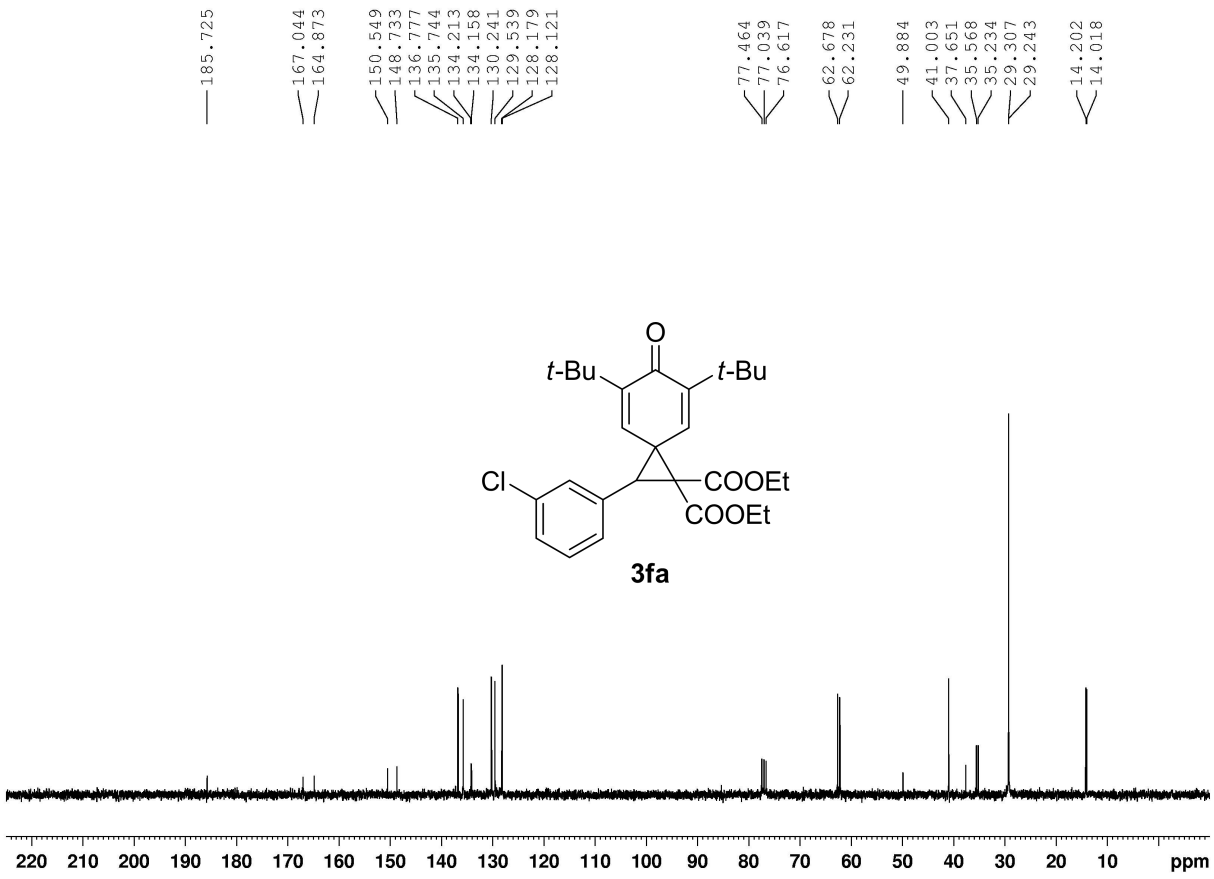
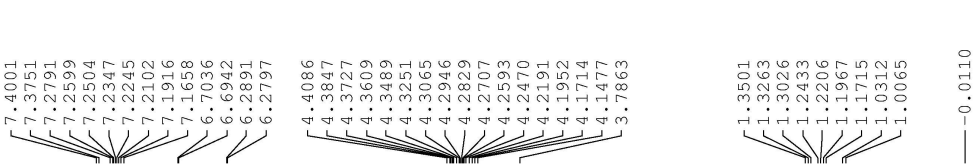
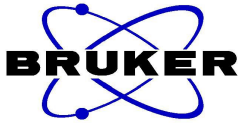
1.95
0.97
2.94
3.00
9.20
8.92

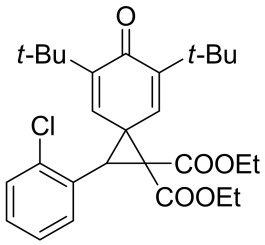


BRUKER

```
NAME        GK-150412-2-C
EXPNO                   1
PROCNO                  1
Date_            20150412
Time                9.42
INSTRUM            spect
PROBHD    5 mm PADUL 13C
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                     98
DS                      4
SWH           19531.250 Hz
FIDRES         0.298023 Hz
AQ             1.6777716 sec
RG                  45.2
DW                25.600 usec
DE                 7.00 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.40 usec
PL1                 -1.00 dB
PL1W         42.37451935 W
SFO1          75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                14.62 dB
PL2W         12.36450577 W
PL12W         0.33898211 W
SFO2         300.1312005 MHz
SI                  32768
SF           75.4677490 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

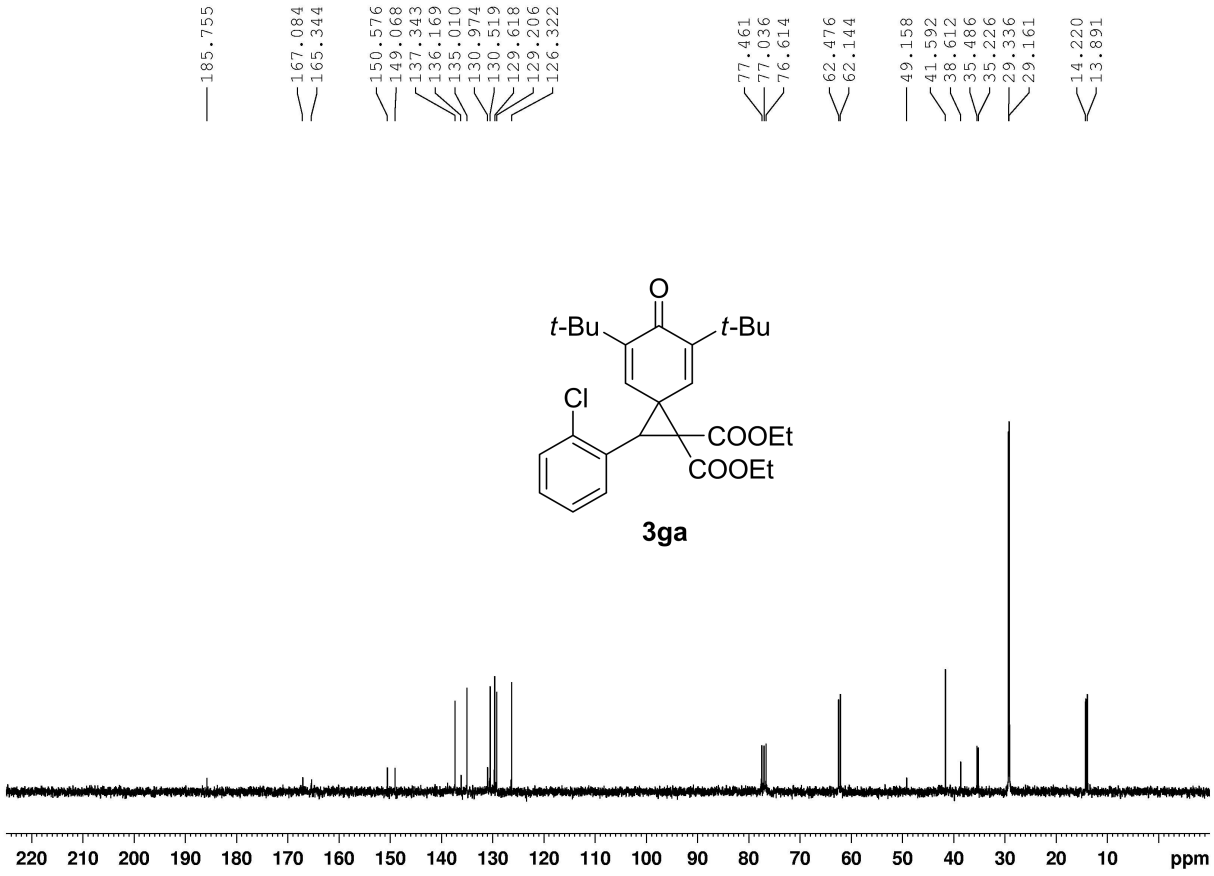






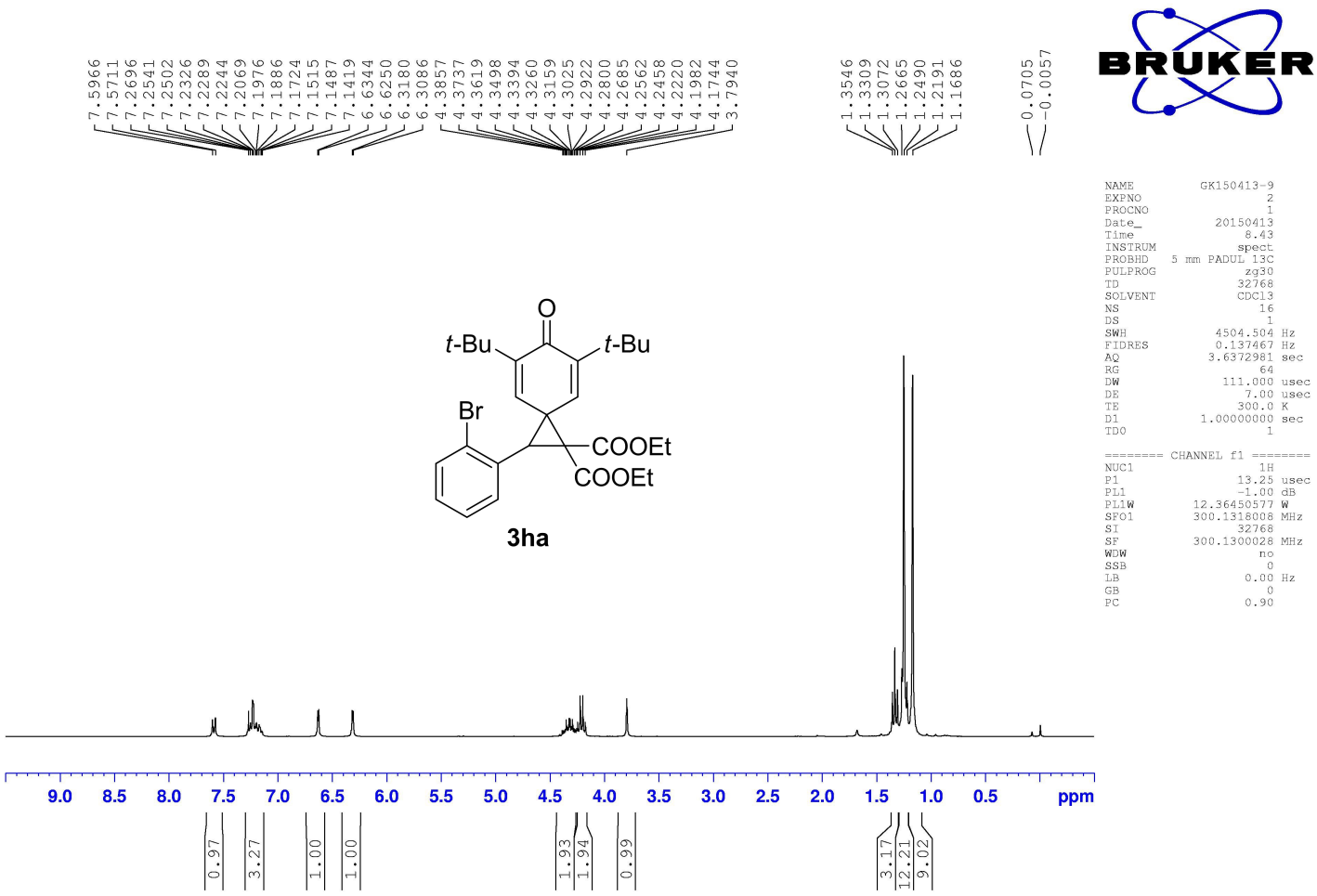
3ga

```
NAME            GK150403-3
EXPNO                    1
PROCNO                   1
Date_             20150403
Time                 13.15
INSTRUM              spect
PROBHD   5 mm PADUL 13C
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       1
SWH               7211.539 Hz
FIDRES            0.220079 Hz
AQ                2.2719646 sec
RG                    71.8
DW                 69.333 usec
DE                   7.00 usec
TE                  300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  13.25 usec
PL1                 -1.00 dB
PL1W          12.36450577 W
SFO1          300.1331514 MHz
SI                   32768
SF            300.1300058 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    0.90
```

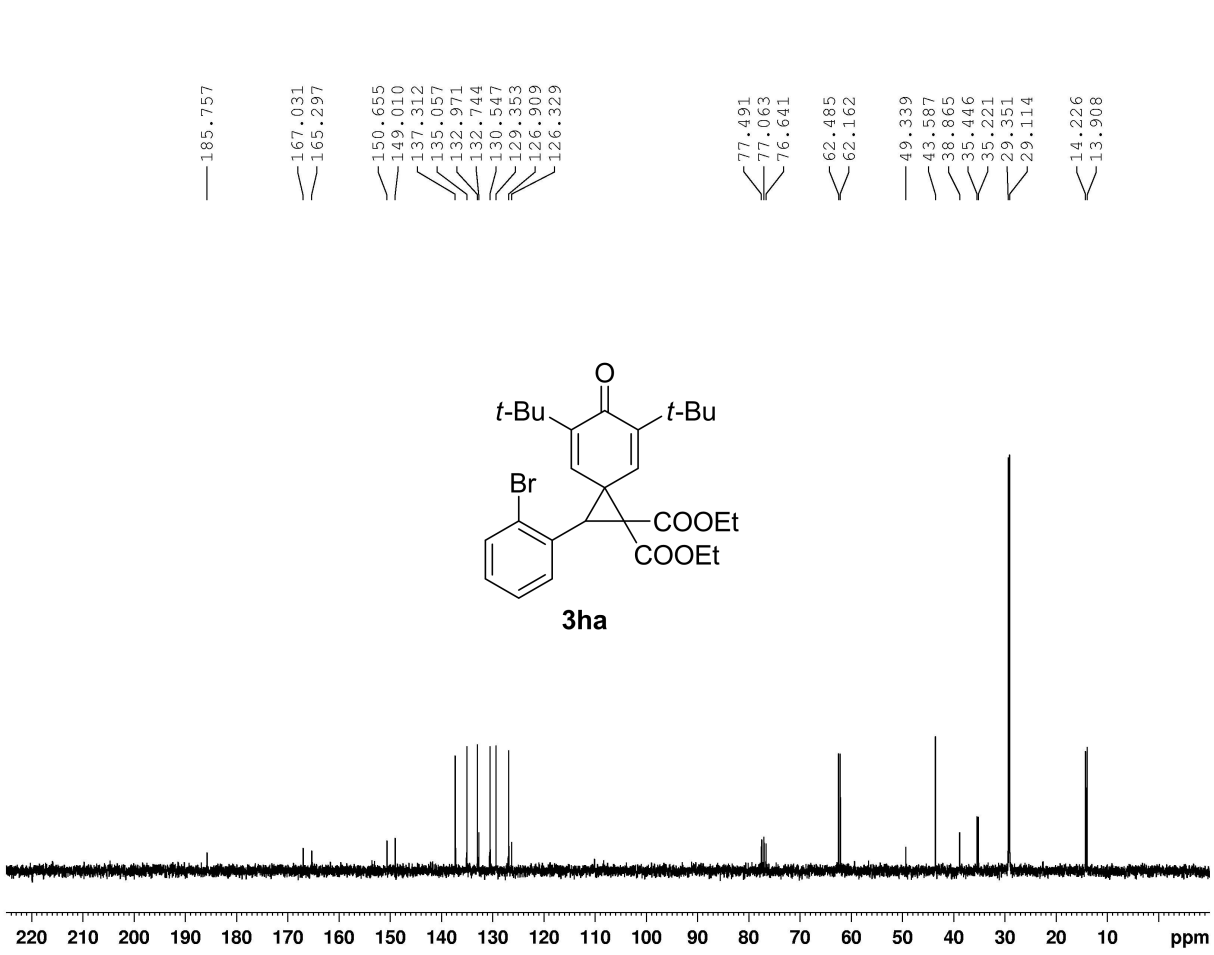


BRUKER

NAME	GK-150412-3-C
EXPNO	1
PROCNO	1
Date_	20150412
Time	11.21
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	170
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

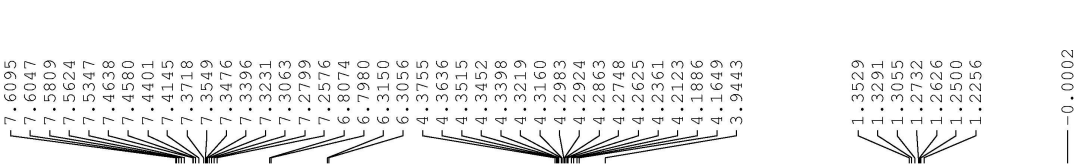


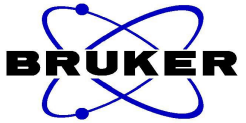

```
NAME          GK150413-9
EXPNO                  2
PROCNO                 1
Date_           20150413
Time                8.43
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH           4504.504 Hz
FIDRES        0.137467 Hz
AQ            3.6372981 sec
RG                    64
DW             111.000 usec
DE               7.00 usec
TE               300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W        12.36450577 W
SFO1        300.1318008 MHz
SI                 32768
SF          300.1300028 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                0.90
```

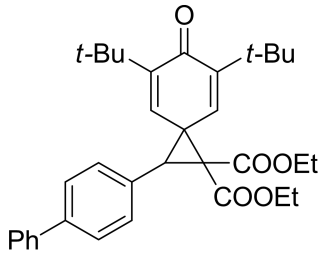


BRUKER

NAME	GK-150526-1-C
EXPNO	1
PROCNO	1
Date_	20150526
Time	6.28
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	25
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

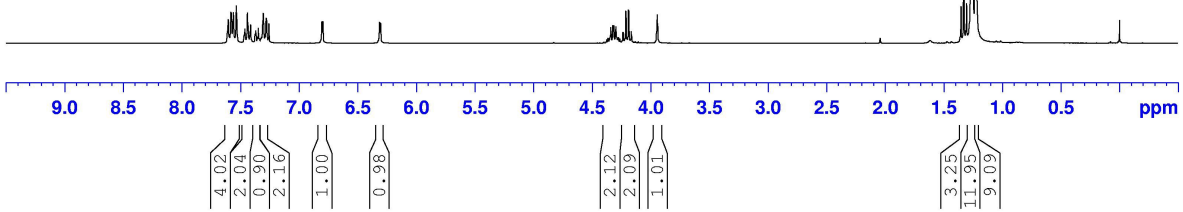


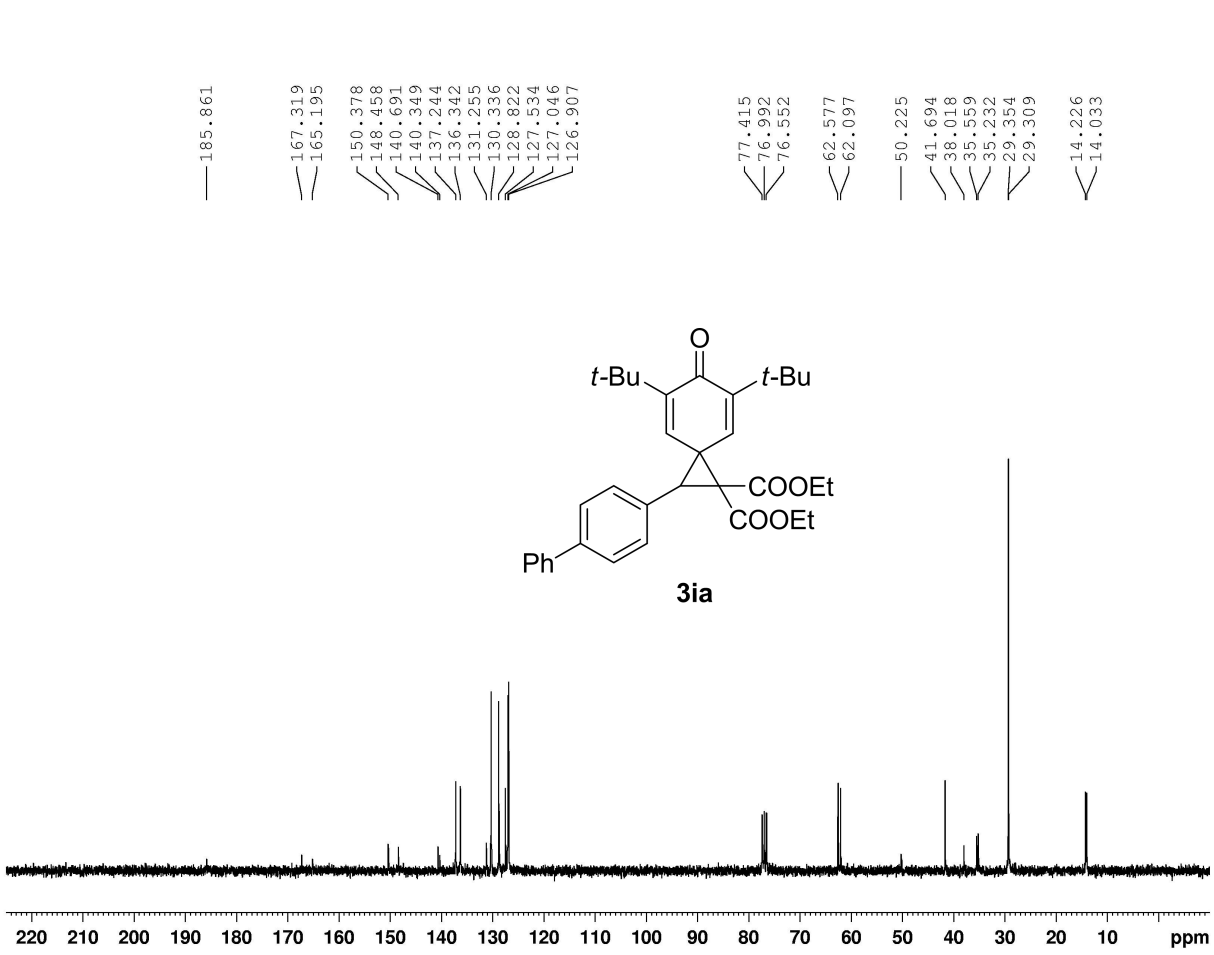




3ia

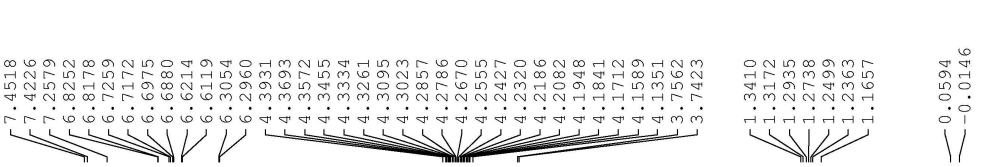
```
NAME          GK150504-1
EXPNO                  1
PROCNO                 1
Date_           20150504
Time               12.52
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719646 sec
RG                  71.8
DW             69.333 usec
DE               7.00 usec
TE              300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1             13.25 usec
PL1              -1.00 dB
PL1W       12.36450577 W
SFO1       300.1331514 MHz
SI               32768
SF         300.1300064 MHz
WDW                  no
SSB                   0
LB             0.00 Hz
GB                    0
PC                 0.90
```

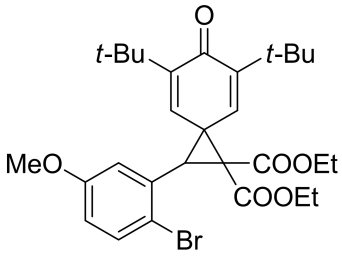




BRUKER

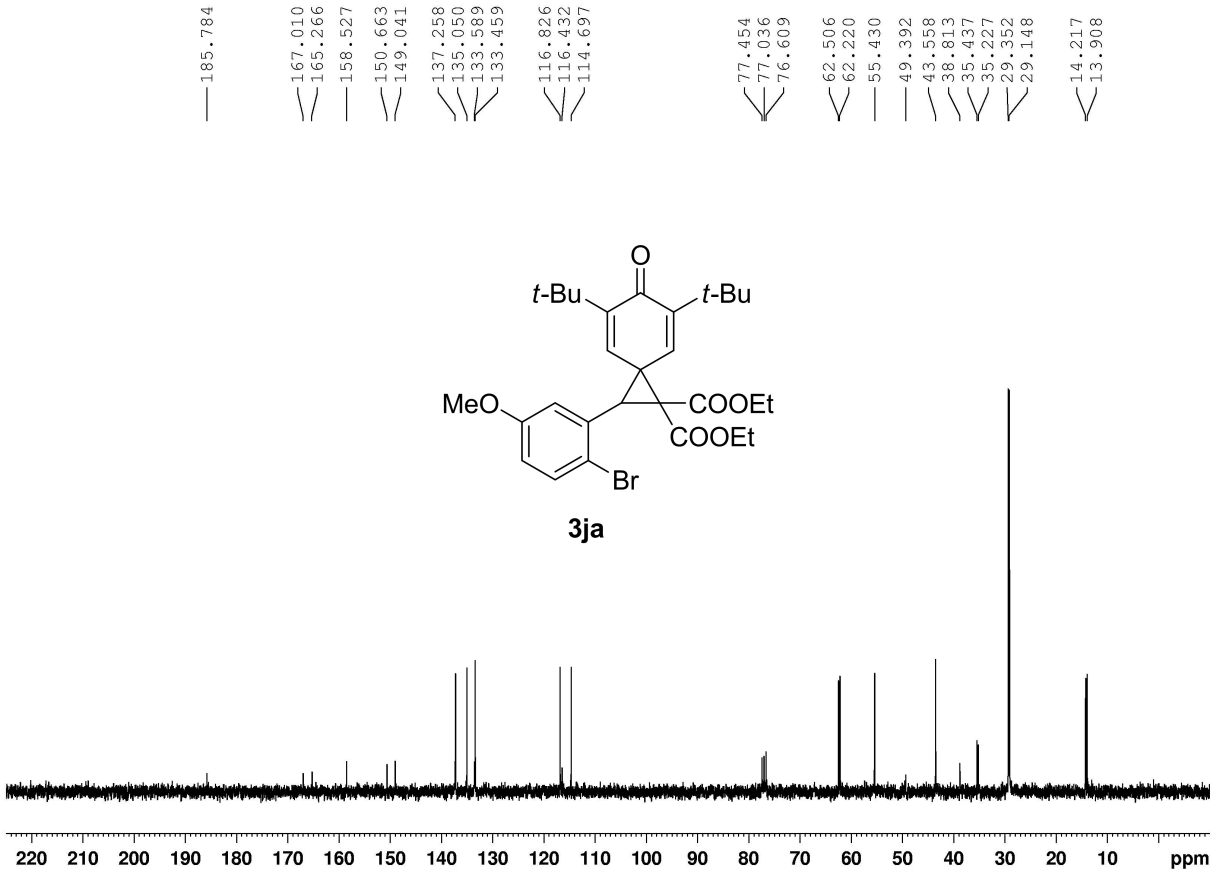
```
NAME          GK-150506-1-C
EXPNO                     1
PROCNO                    1
Date_              20150506
Time                   6.30
INSTRUM               spect
PROBHD    5 mm PADUL 13C
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                      320
DS                        4
SWH              19531.250 Hz
FIDRES           0.298023 Hz
AQ               1.6777716 sec
RG                     45.2
DW                  25.600 usec
DE                   7.00 usec
TE                   300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.40 usec
PL1                   -1.00 dB
PL1W           42.37451935 W
SFO1            75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   -1.00 dB
PL12                  14.62 dB
PL2W           12.36450577 W
PL12W           0.33898211 W
SFO2           300.1312005 MHz
SI                    32768
SF             75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```





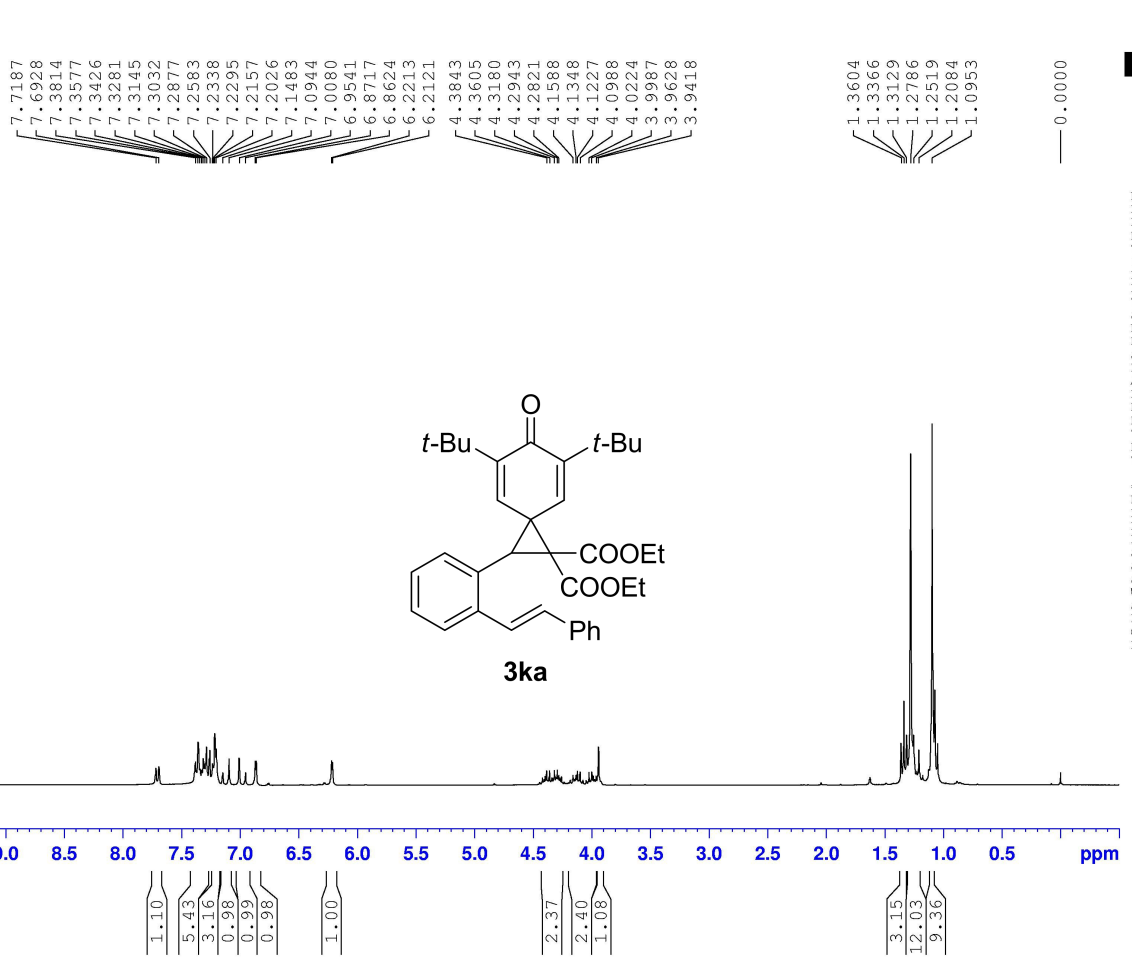
3ja

```
NAME          GK150417-5
EXPNO                  1
PROCNO                 1
Date_           20150417
Time               13.23
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719646 sec
RG                    57
DW                69.333 usec
DE                 7.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W        12.36450577 W
SFO1        300.1331514 MHz
SI                 32768
SF          300.1300063 MHz
WDW                   no
SSB                    0
LB                 0.00 Hz
GB                     0
PC                 0.90
```



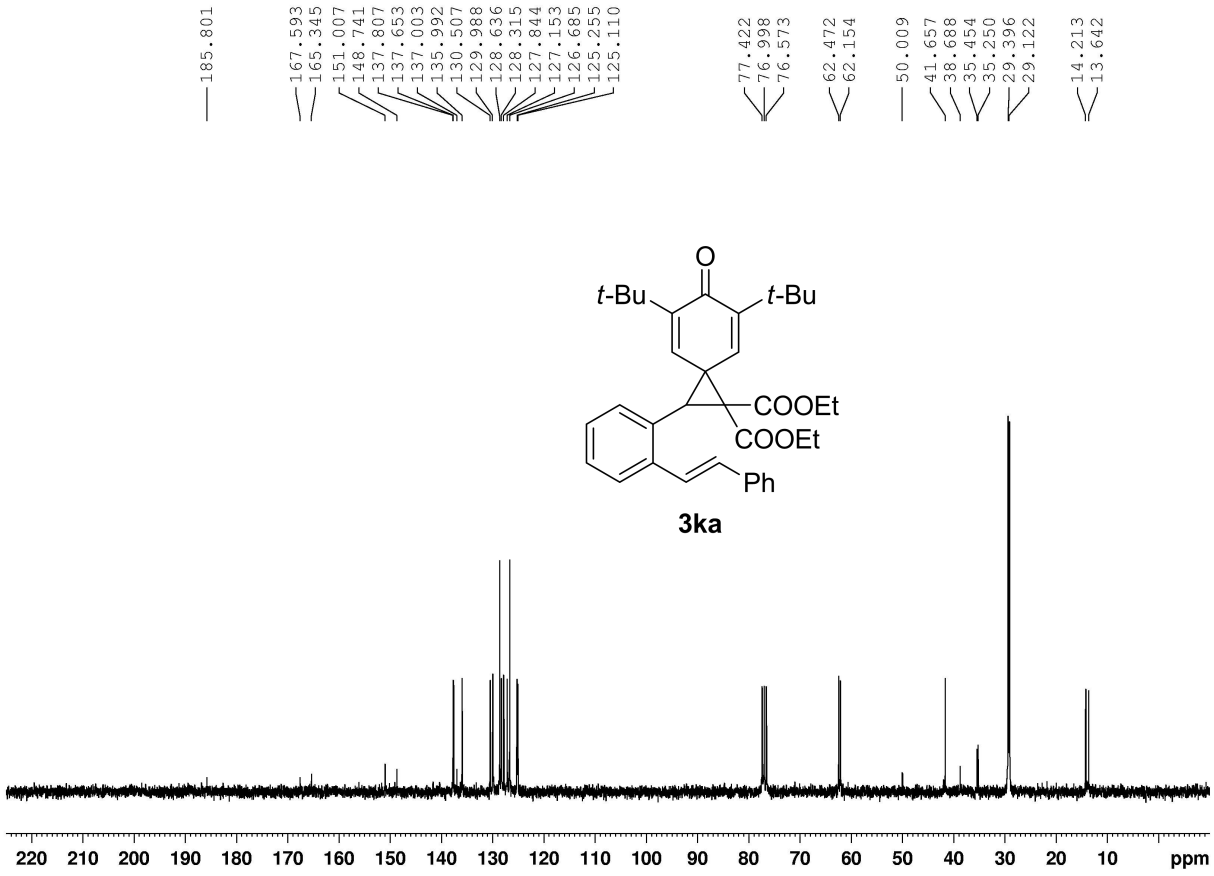
BRUKER

NAME	GK-150506-5-C
EXPNO	1
PROCNO	1
Date_	20150506
Time	7.47
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	69
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



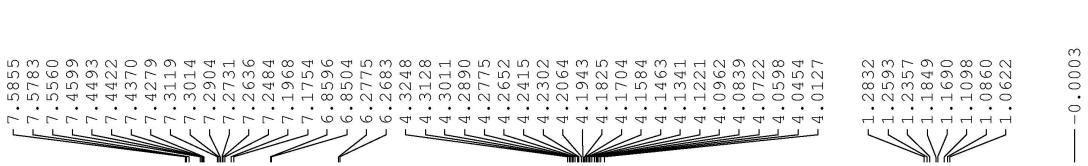
BRUKER

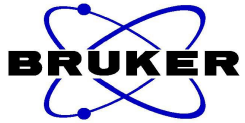
NAME	GK150428-14
EXPNO	1
PROCNO	1
Date_	20150428
Time	7.44
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	1
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	57
DW	69.333 usec
DE	7.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.25 usec
PL1	-1.00 dB
PL1W	12.36450577 W
SFO1	300.1331514 MHz
SI	32768
SF	300.1300063 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.90

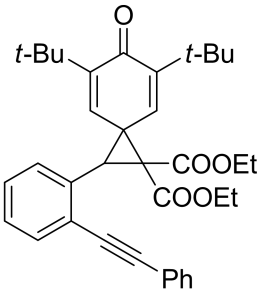


BRUKER

NAME	GK-150515-14-C
EXPNO	1
PROCNO	1
Date_	20150515
Time	6.58
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	624
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

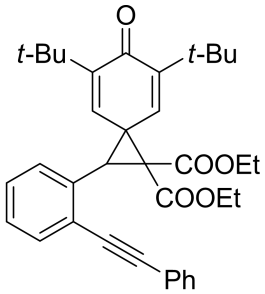




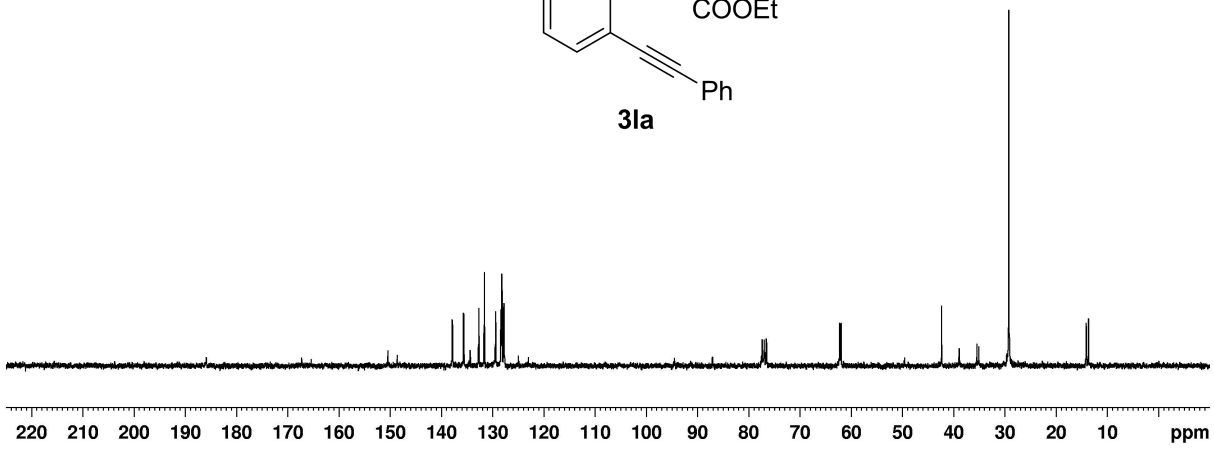


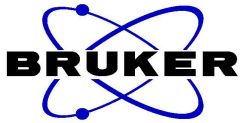
3la

```
NAME          GK150428-3
EXPNO                  1
PROCNO                 1
Date_           20150428
Time                6.45
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719646 sec
RG                  80.6
DW               69.333 usec
DE                7.00 usec
TE               300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W        12.36450577 W
SFO1        300.1331514 MHz
SI                 32768
SF          300.1300047 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                0.90
```

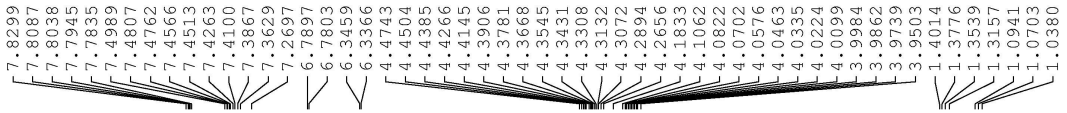


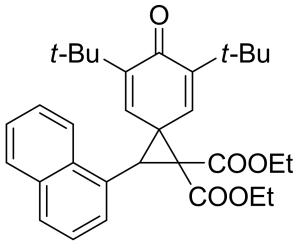
3la



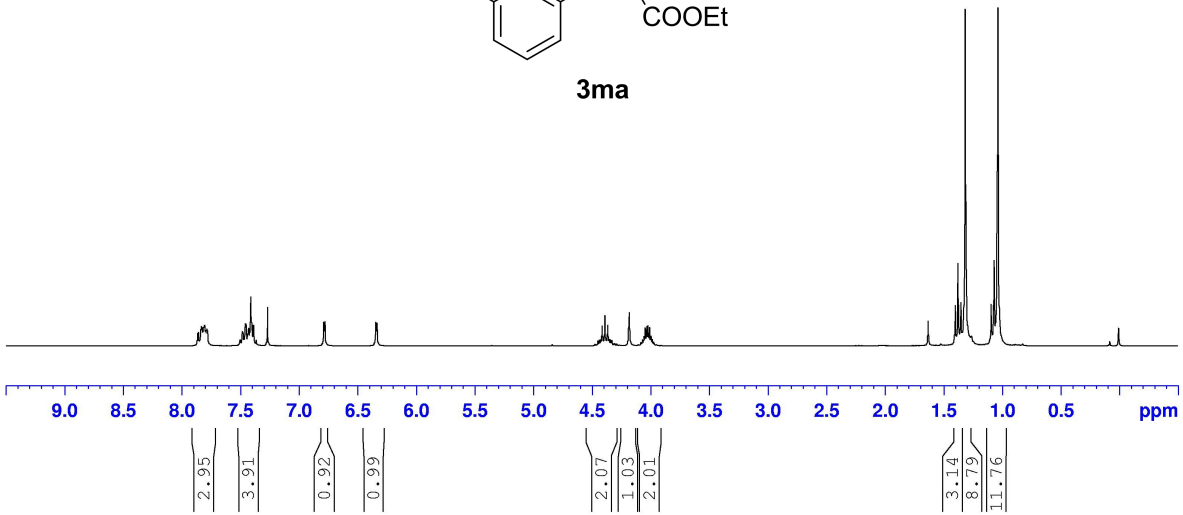



```
NAME          GK-150506-3-C
EXPNO                     1
PROCNO                    1
Date_              20150506
Time                   7.12
INSTRUM               spect
PROBHD    5 mm PADUL 13C
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                      726
DS                        4
SWH              19531.250 Hz
FIDRES           0.298023 Hz
AQ               1.6777716 sec
RG                     45.2
DW                  25.600 usec
DE                   7.00 usec
TE                   300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.40 usec
PL1                   -1.00 dB
PL1W           42.37451935 W
SFO1            75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   -1.00 dB
PL12                  14.62 dB
PL2W           12.36450577 W
PL12W           0.33898211 W
SFO2           300.1312005 MHz
SI                    32768
SF             75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

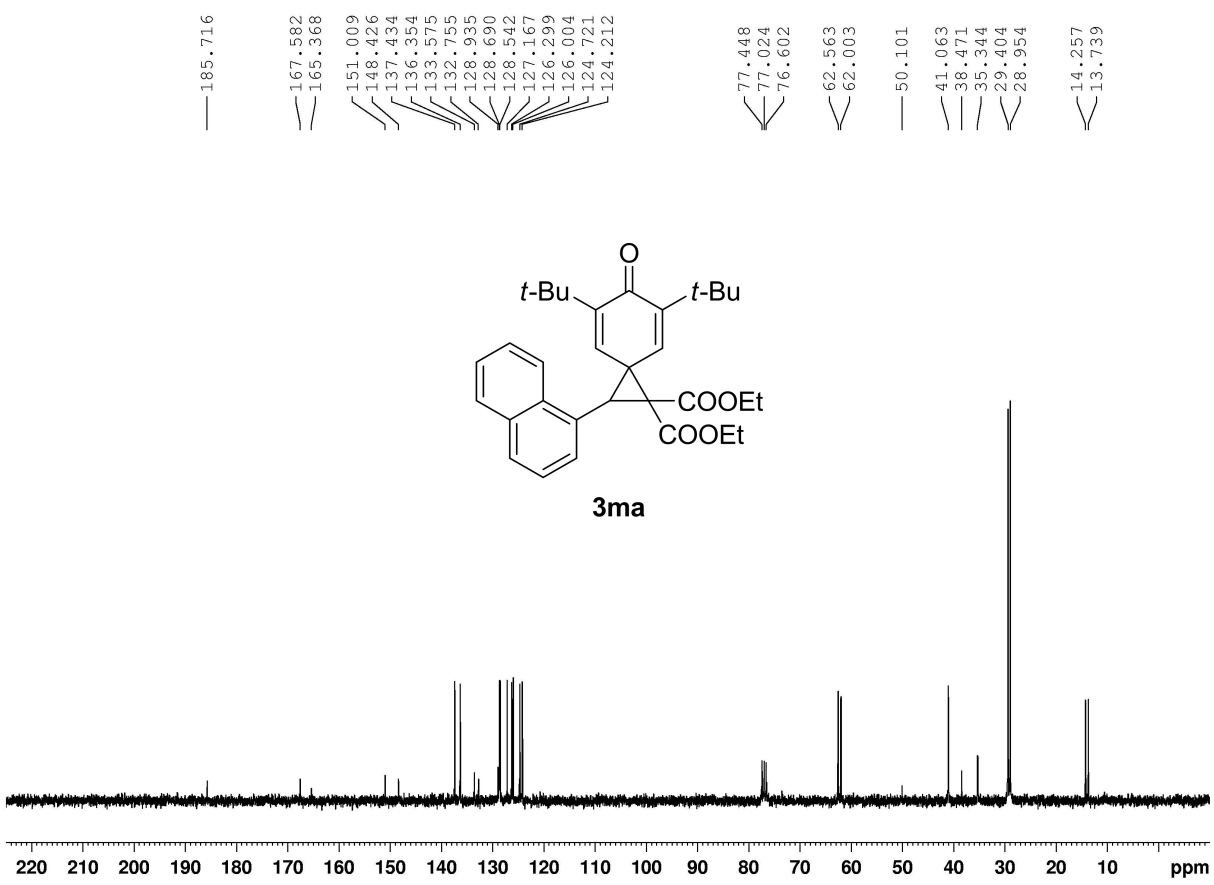




3ma

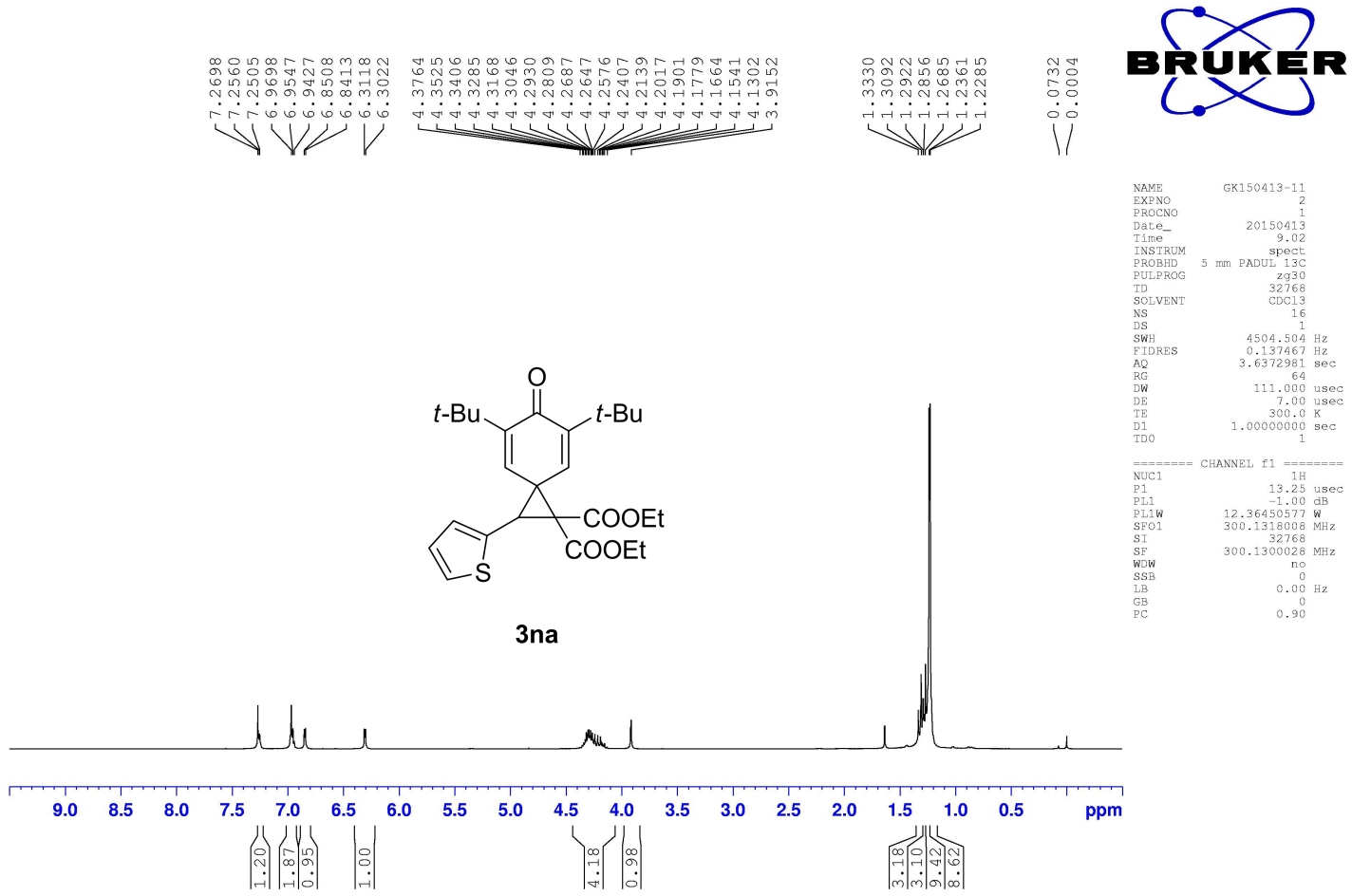


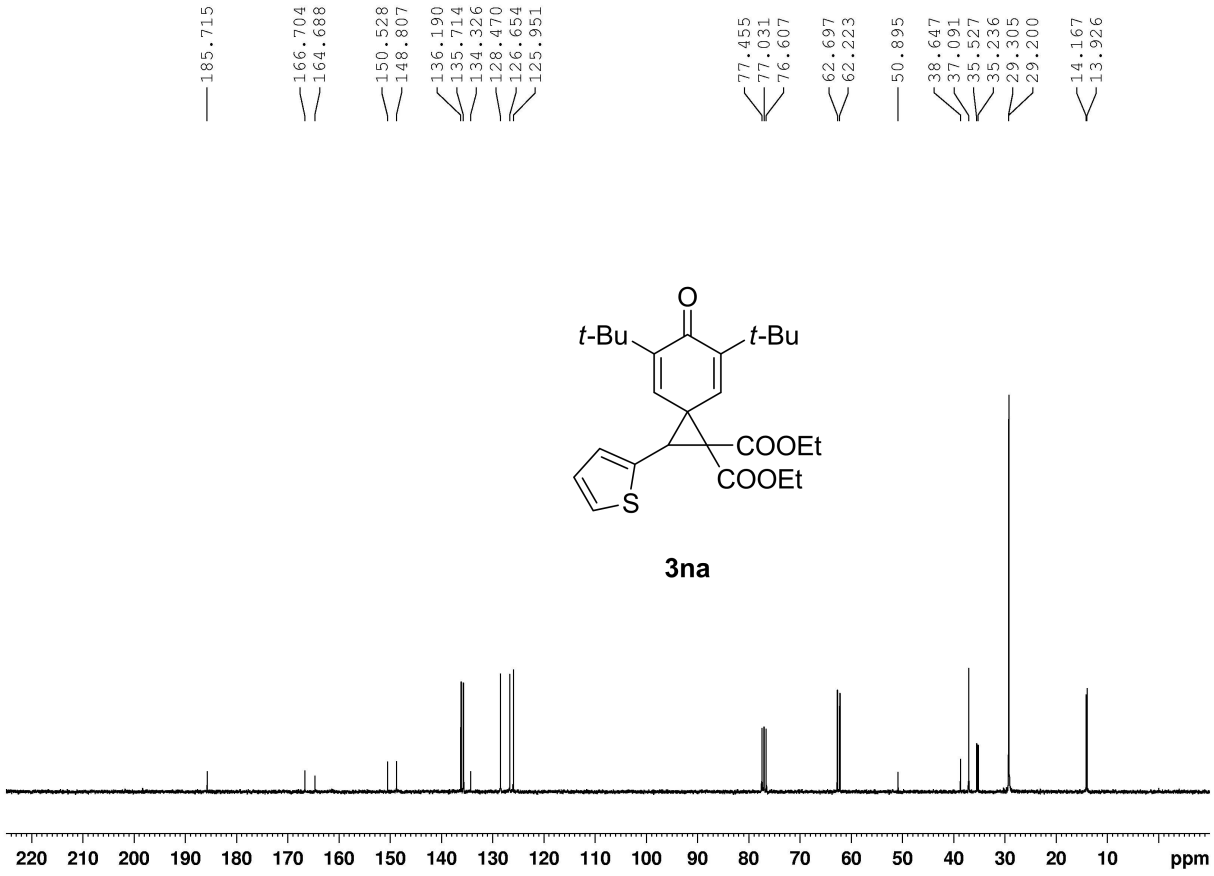

```
NAME          GK150413-10
EXPNO                   2
PROCNO                  1
Date_            20150413
Time                 8.54
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      1
SWH             4504.504 Hz
FIDRES          0.137467 Hz
AQ              3.6372981 sec
RG                     64
DW                111.000 usec
DE                  7.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.25 usec
PL1                -1.00 dB
PL1W        12.36450577 W
SFO1        300.1318008 MHz
SI                  32768
SF          300.1300028 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                 0.90
```



BRUKER

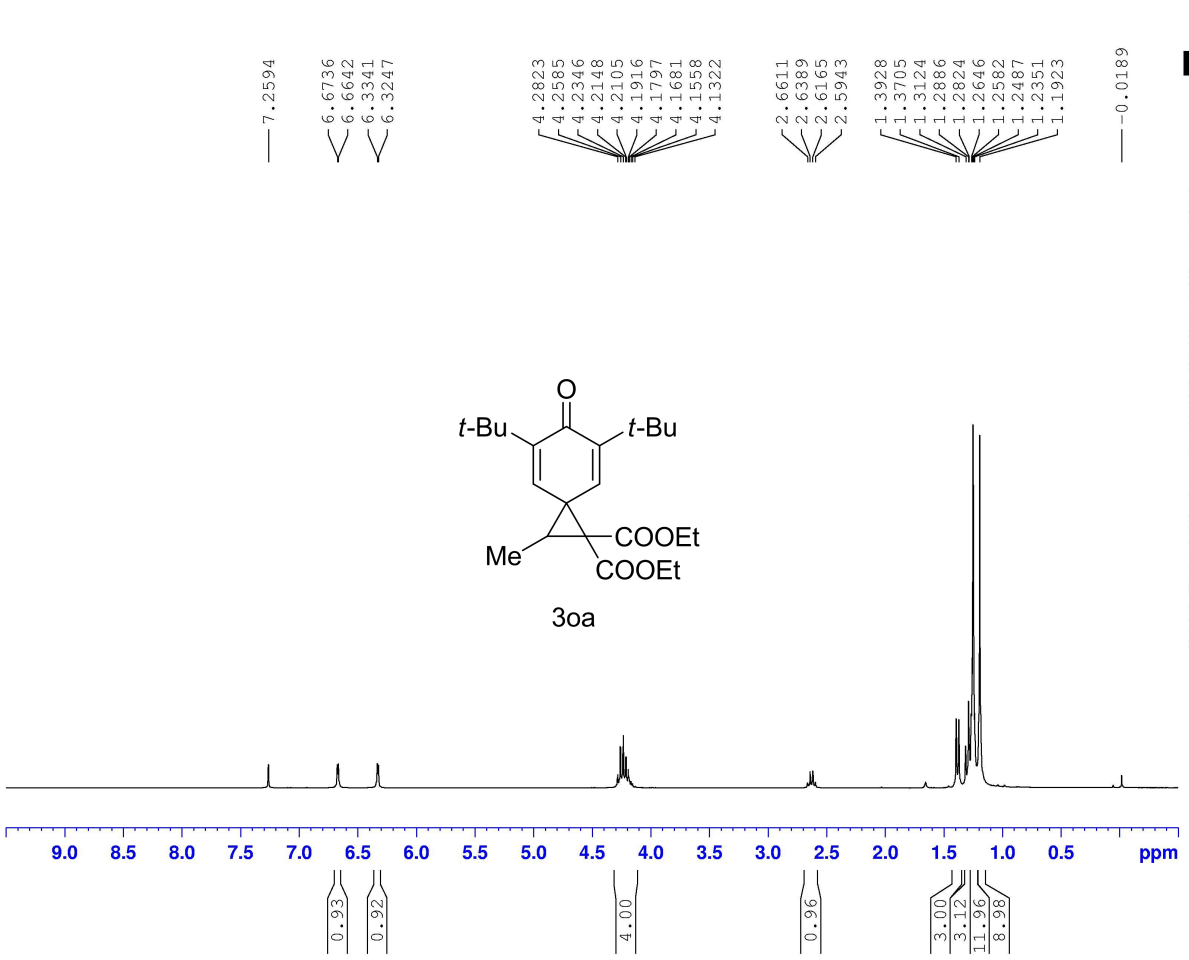
NAME	GK-150515-10-C
EXPNO	1
PROCNO	1
Date_	20150515
Time	6.17
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	191
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40


7.2698
7.2560
7.2505
6.9698
6.9547
6.9427
6.8508
6.8413
6.3118
6.3022
4.3764
4.3525
4.3406
4.3285
4.3168
4.3046
4.2930
4.2809
4.2687
4.2647
4.2576
4.2407
4.2139
4.2017
4.1901
4.1779
4.1664
4.1541
4.1302
3.9152
1.3330
1.3092
1.2922
1.2856
1.2685
1.2361
1.2285
0.0732
0.0004
BRUKER
NAME GK150413-11
EXPNO 2
PROCNO 1
Date_ 20150413
Time 9.02
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 4504.504 Hz
FIDRES 0.137467 Hz
AQ 3.6372981 sec
RG 64
DW 111.000 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.25 usec
PL1 -1.00 dB
PL1W 12.36450577 W
SFO1 300.1318008 MHz
SI 32768
SF 300.1300028 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.90
O
t-Bu
t-Bu
COOEt
COOEt
S
3na
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 ppm
1.20
1.87
0.95
1.00
4.18
0.98
3.18
3.10
9.42
8.62

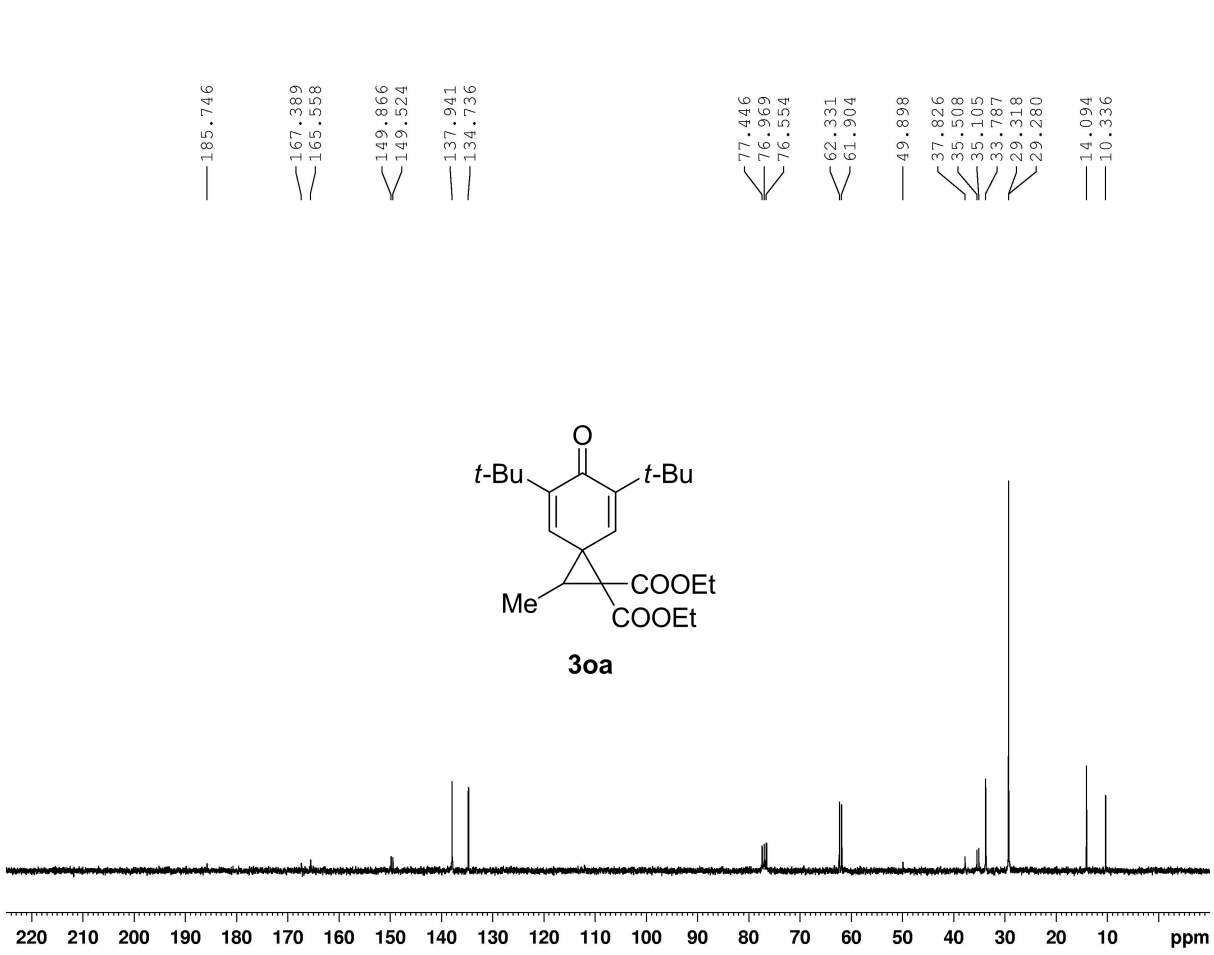


3na

```
NAME          GK150529-1-C
EXPNO                    1
PROCNO                   1
Date_             20150529
Time                 7.57
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     500
DS                       4
SWH           19531.250 Hz
FIDRES         0.298023 Hz
AQ             1.6777716 sec
RG                    40.3
DW                 25.600 usec
DE                  7.00 usec
TE                 300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.40 usec
PL1                  -1.00 dB
PL1W          42.37451935 W
SFO1           75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 14.62 dB
PL2W          12.36450577 W
PL12W          0.33898211 W
SFO2          300.1312005 MHz
SI                   32768
SF            75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

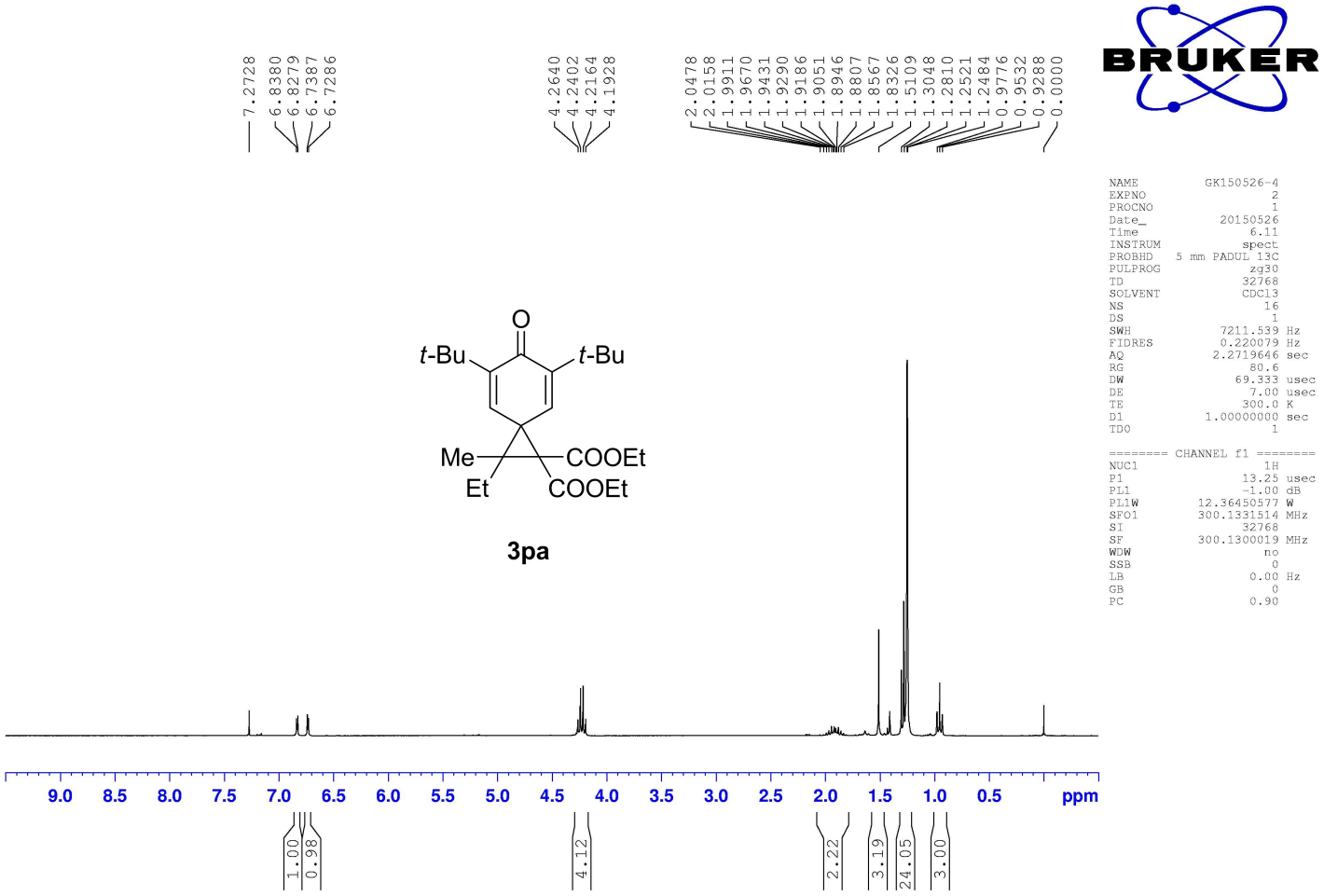



```
NAME          GK150422-2
EXPNO                  1
PROCNO                 1
Date_           20150422
Time               13.09
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719646 sec
RG                    64
DW               69.333 usec
DE                7.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W       12.36450577 W
SFO1       300.1331514 MHz
SI                 32768
SF         300.1300059 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                0.90
```



BRUKER

NAME	GK-150506-2-C
EXPNO	1
PROCNO	1
Date_	20150506
Time	6.58
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	298
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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6.8279
6.7387
6.7286
4.2640
4.2402
4.2164
4.1928
2.0478
2.0158
1.9911
1.9670
1.9431
1.9290
1.9186
1.9051
1.8946
1.8807
1.8567
1.8326
1.5109
1.3048
1.2810
1.2521
1.2484
0.9776
0.9532
0.9288
0.0000
BRUKER
NAME GK150526-4
EXPNO 2
PROCNO 1
Date_ 20150526
Time 6.11
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719646 sec
RG 80.6
DW 69.333 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.25 usec
PL1 -1.00 dB
PL1W 12.36450577 W
SFO1 300.1331514 MHz
SI 32768
SF 300.1300019 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.90
O
t-Bu
t-Bu
Me
COOEt
Et
COOEt
3pa
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
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0.5
ppm
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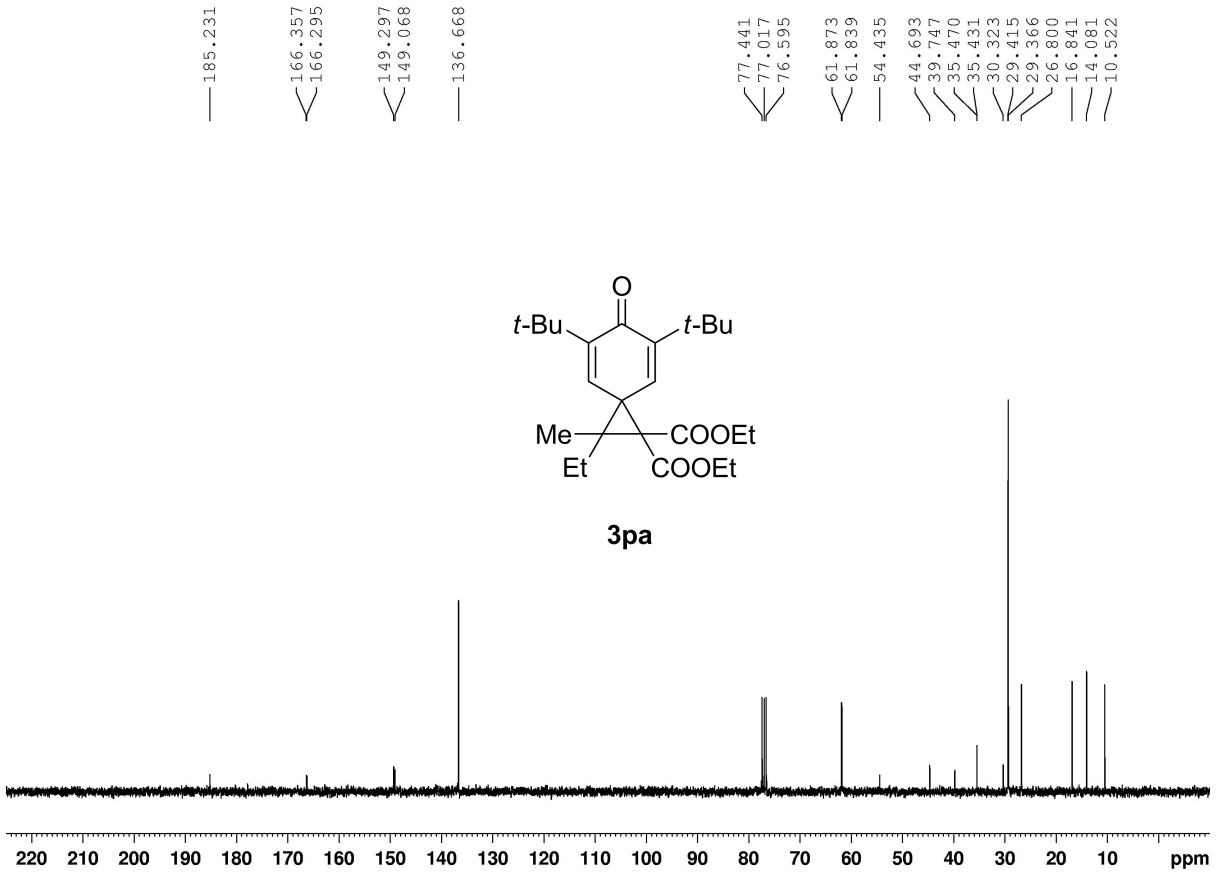
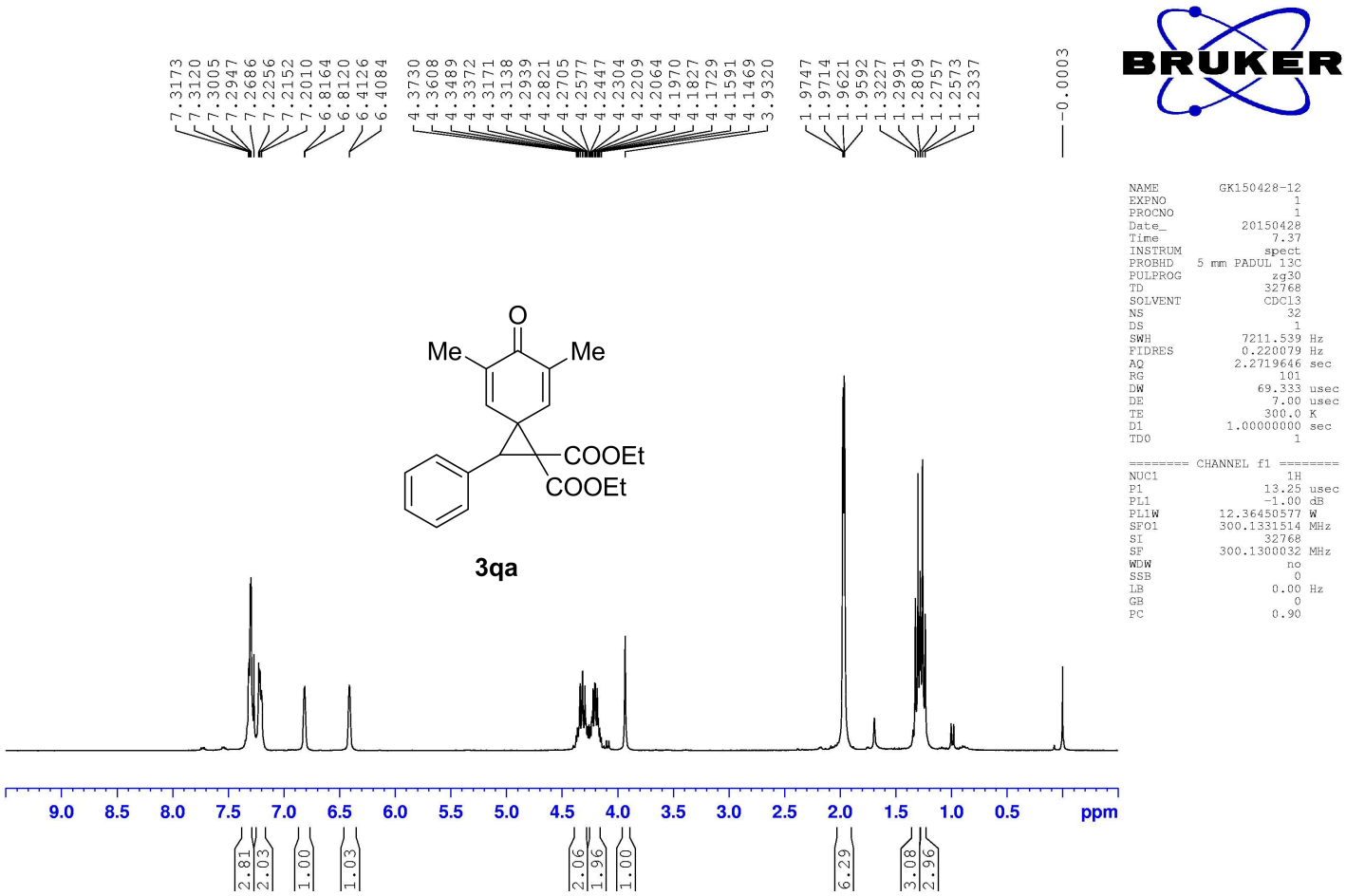
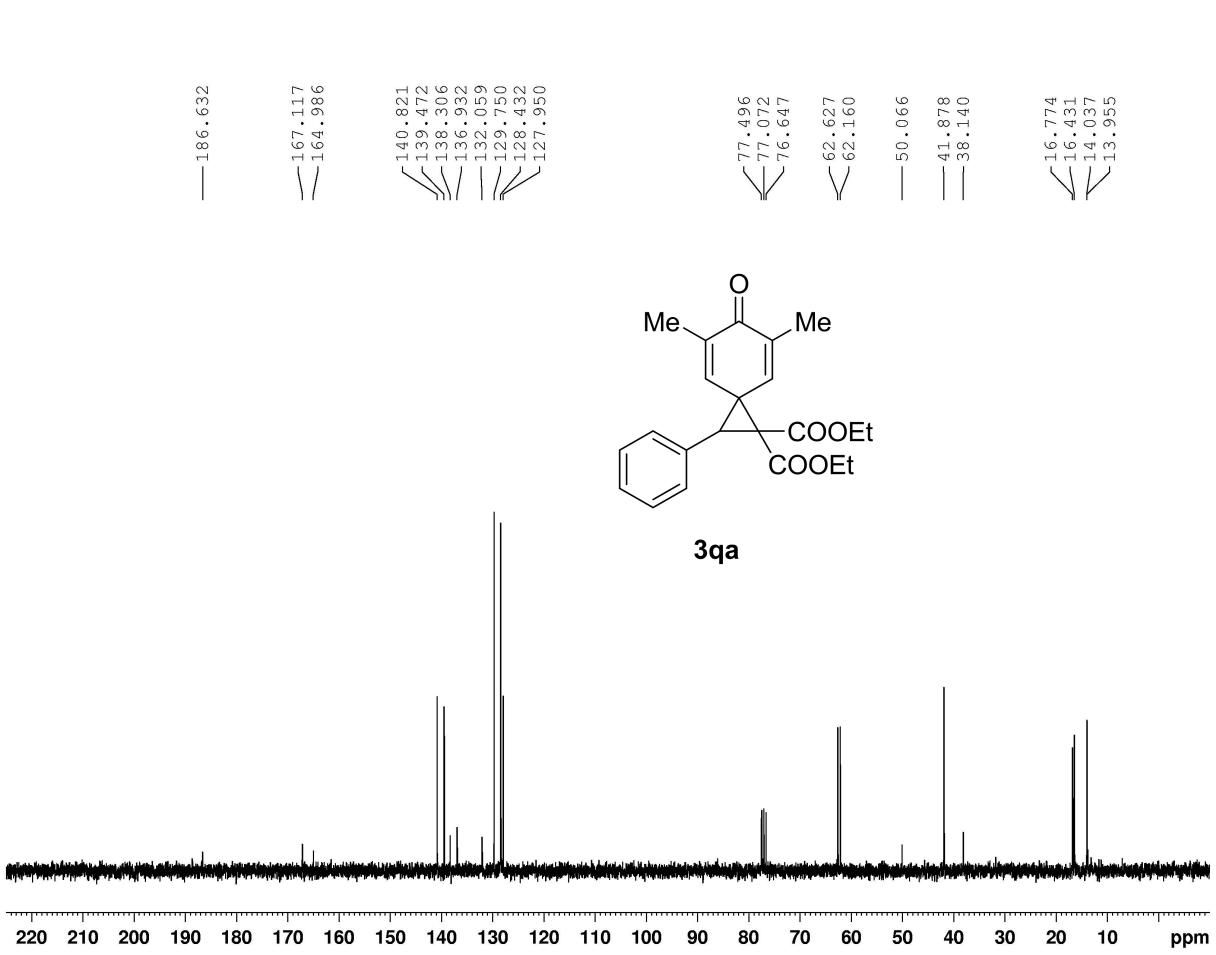
4.12
2.22
3.19
24.05
3.00




```
NAME          GK-150526-4-C
EXPNO                     1
PROCNO                    1
Date_              20150526
Time                   6.16
INSTRUM               spect
PROBHD    5 mm PADUL 13C
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                      177
DS                        4
SWH              19531.250 Hz
FIDRES           0.298023 Hz
AQ               1.6777716 sec
RG                     45.2
DW                  25.600 usec
DE                   7.00 usec
TE                  300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.40 usec
PL1                   -1.00 dB
PL1W           42.37451935 W
SFO1            75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                 80.00 usec
PL2                   -1.00 dB
PL12                  14.62 dB
PL2W           12.36450577 W
PL12W           0.33898211 W
SFO2           300.1312005 MHz
SI                    32768
SF             75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

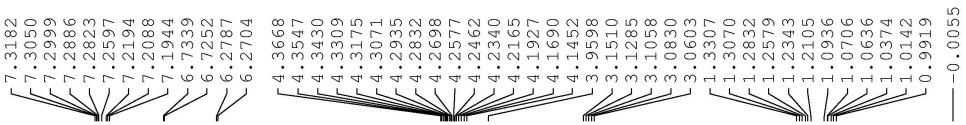


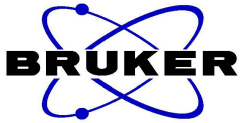

```
NAME          GK150428-12
EXPNO                   1
PROCNO                  1
Date_            20150428
Time                 7.37
INSTRUM             spect
PROBHD   5 mm PADUL 13C
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     32
DS                      1
SWH             7211.539 Hz
FIDRES          0.220079 Hz
AQ              2.2719646 sec
RG                    101
DW                69.333 usec
DE                 7.00 usec
TE                300.0 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 13.25 usec
PL1                -1.00 dB
PL1W        12.36450577 W
SFO1        300.1331514 MHz
SI                  32768
SF          300.1300032 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                 0.90
```

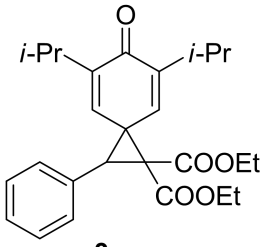


BRUKER

NAME	GK-150525-12-C
EXPNO	1
PROCNO	1
Date_	20150526
Time	6.04
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	42
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

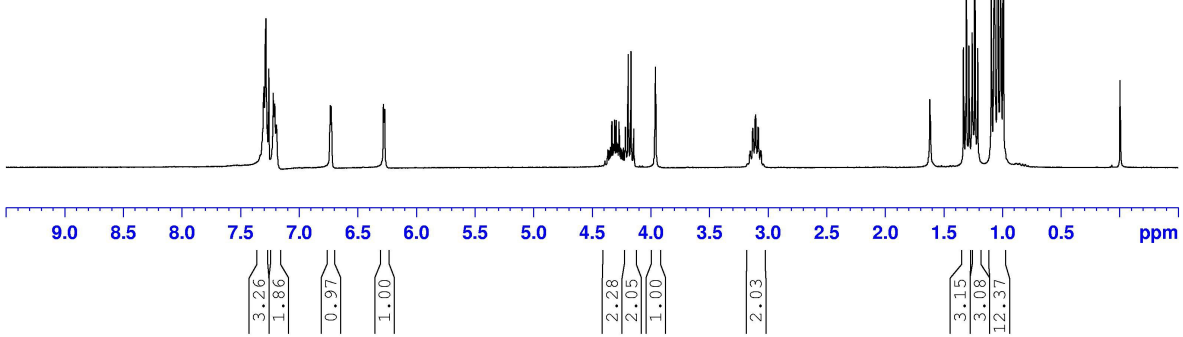


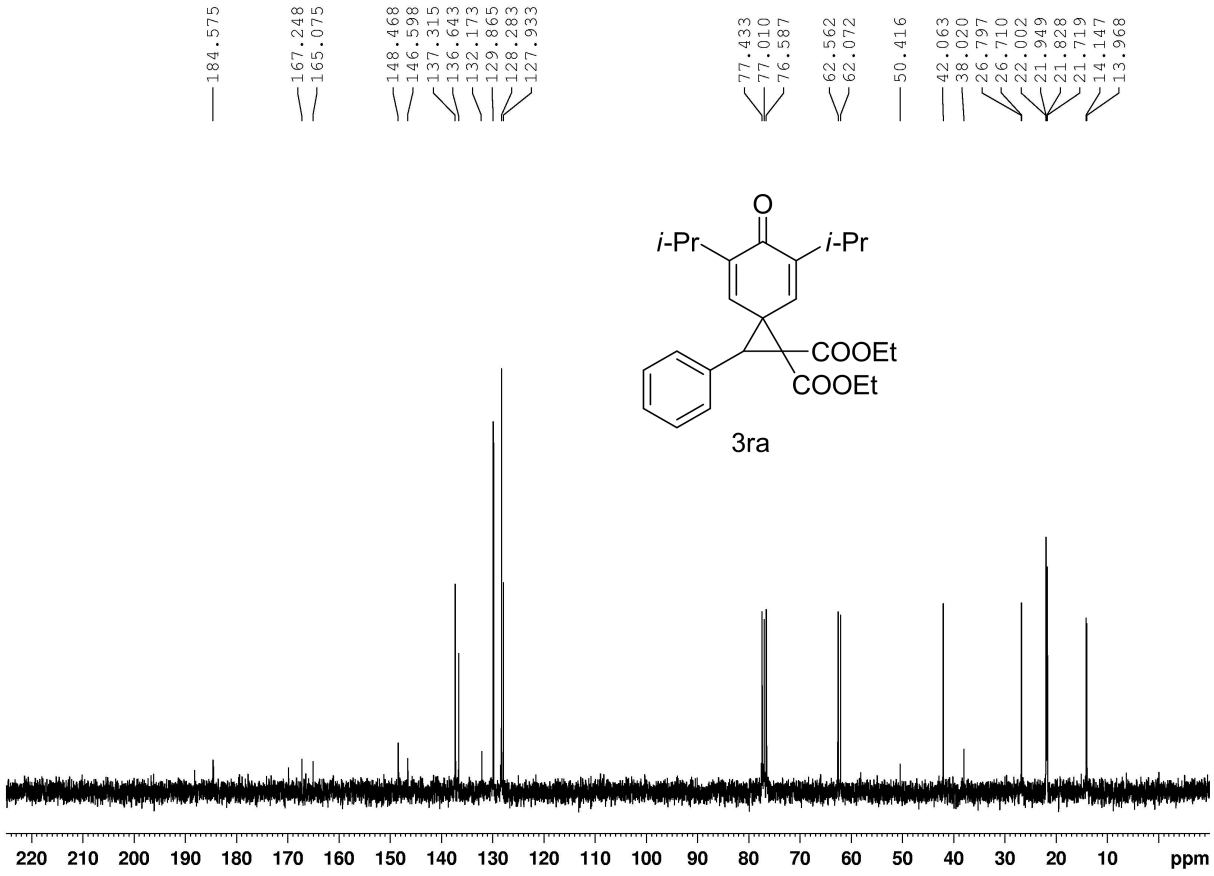




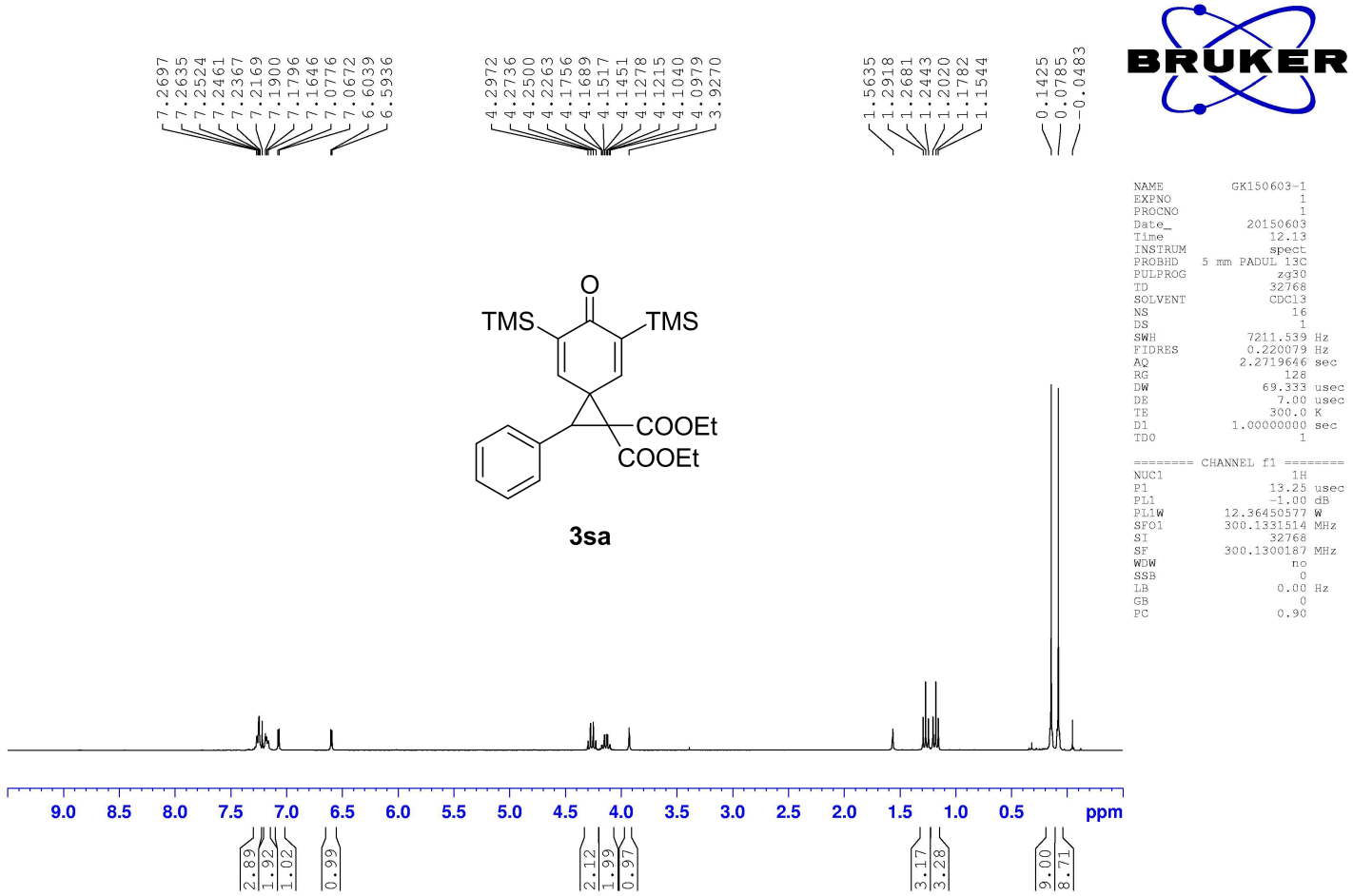
3ra

```
NAME          GK150518-3
EXPNO                  1
PROCNO                 1
Date_           20150518
Time               13.21
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719646 sec
RG                   114
DW               69.333 usec
DE                7.00 usec
TE               300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1                -1.00 dB
PL1W        12.36450577 W
SFO1        300.1331514 MHz
SI                 32768
SF          300.1300060 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                0.90
```






```
NAME          GK150518-3-C
EXPNO                    1
PROCNO                   1
Date_             20150518
Time                 13.23
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG                zgdc
TD                    65536
SOLVENT               CDCl3
NS                      176
DS                        4
SWH              19531.250 Hz
FIDRES           0.298023 Hz
AQ               1.6777716 sec
RG                     45.2
DW                  25.600 usec
DE                   7.00 usec
TE                   300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.40 usec
PL1                  -1.00 dB
PL1W           42.37451935 W
SFO1            75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 14.62 dB
PL2W           12.36450577 W
PL12W           0.33898211 W
SFO2           300.1312005 MHz
SI                   32768
SF              75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```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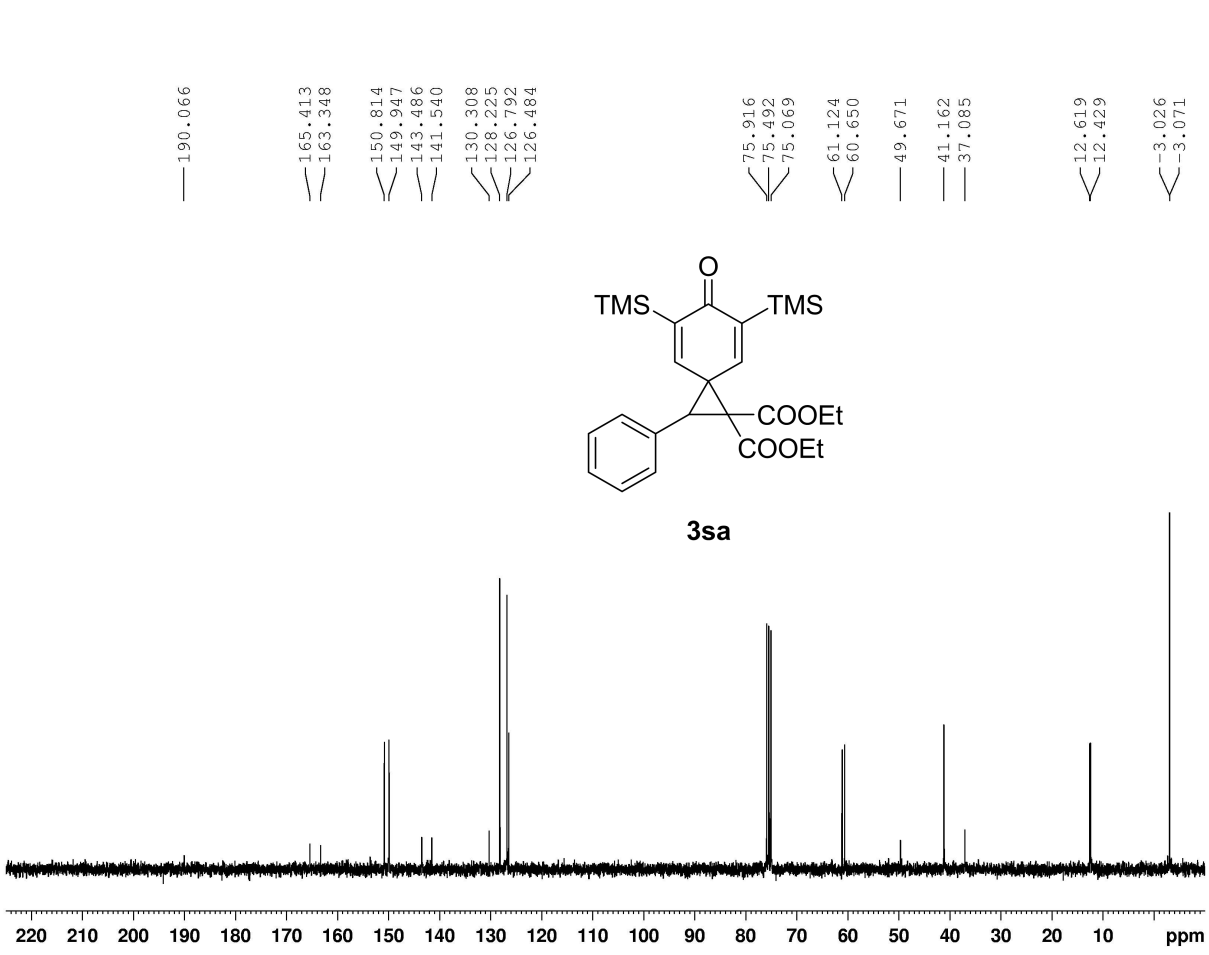
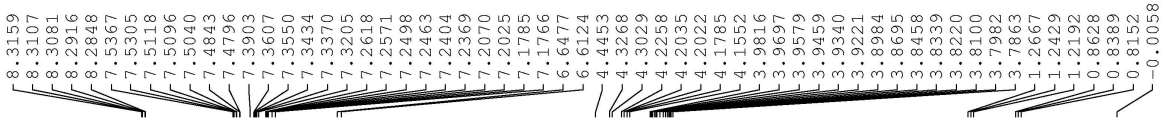
7.2635
7.2524
7.2461
7.2367
7.2169
7.1900
7.1796
7.1646
7.0776
7.0672
6.6039
6.5936
4.2972
4.2736
4.2500
4.2263
4.1756
4.1689
4.1517
4.1451
4.1278
4.1215
4.1040
4.0979
3.9270
1.5635
1.2918
1.2681
1.2443
1.2020
1.1782
1.1544
0.1425
0.0785
-0.0483
BRUKER
NAME GK150603-1
EXPNO 1
PROCNO 1
Date_ 20150603
Time 12.13
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719646 sec
RG 128
DW 69.333 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.25 usec
PL1 -1.00 dB
PL1W 12.36450577 W
SFO1 300.1331514 MHz
SI 32768
SF 300.1300187 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.90
O
TMS
TMS
COOEt
COOEt
3sa
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
ppm
2.89
1.92
1.02
0.99
2.12
1.99
0.97
3.17
3.28
9.00
8.71



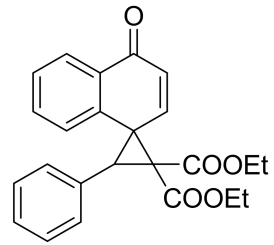
BRUKER

NAME	GK-150605-C
EXPNO	1
PROCNO	1
Date_	20150605
Time	5.48
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	DMSO
NS	383
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.30

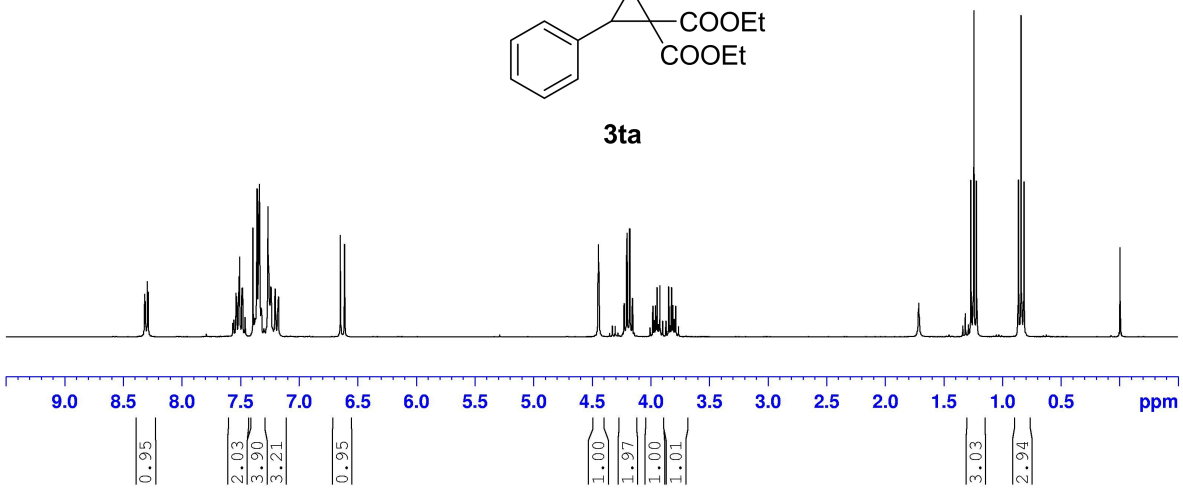


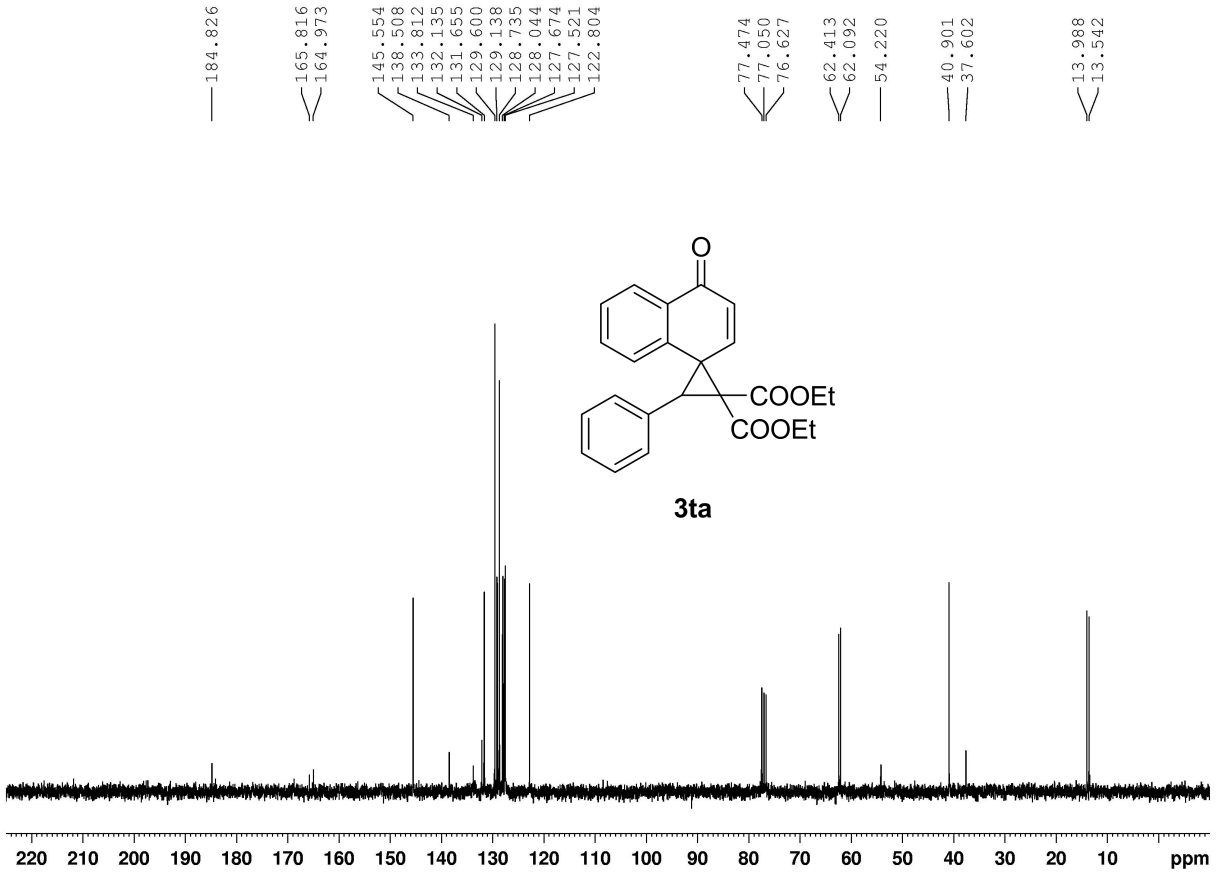

```
NAME          GK  150529-2
EXPNO                    1
PROCNO                   1
Date_             20150529
Time                 12.24
INSTRUM              spect
PROBHD    5 mm PADUL 13C
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       1
SWH               7211.539 Hz
FIDRES            0.220079 Hz
AQ                2.2719646 sec
RG                     114
DW                  69.333 usec
DE                   7.00 usec
TE                  300.0 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   13.25 usec
PL1                  -1.00 dB
PL1W          12.36450577 W
SFO1          300.1331514 MHz
SI                   32768
SF            300.1300053 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    0.90
```



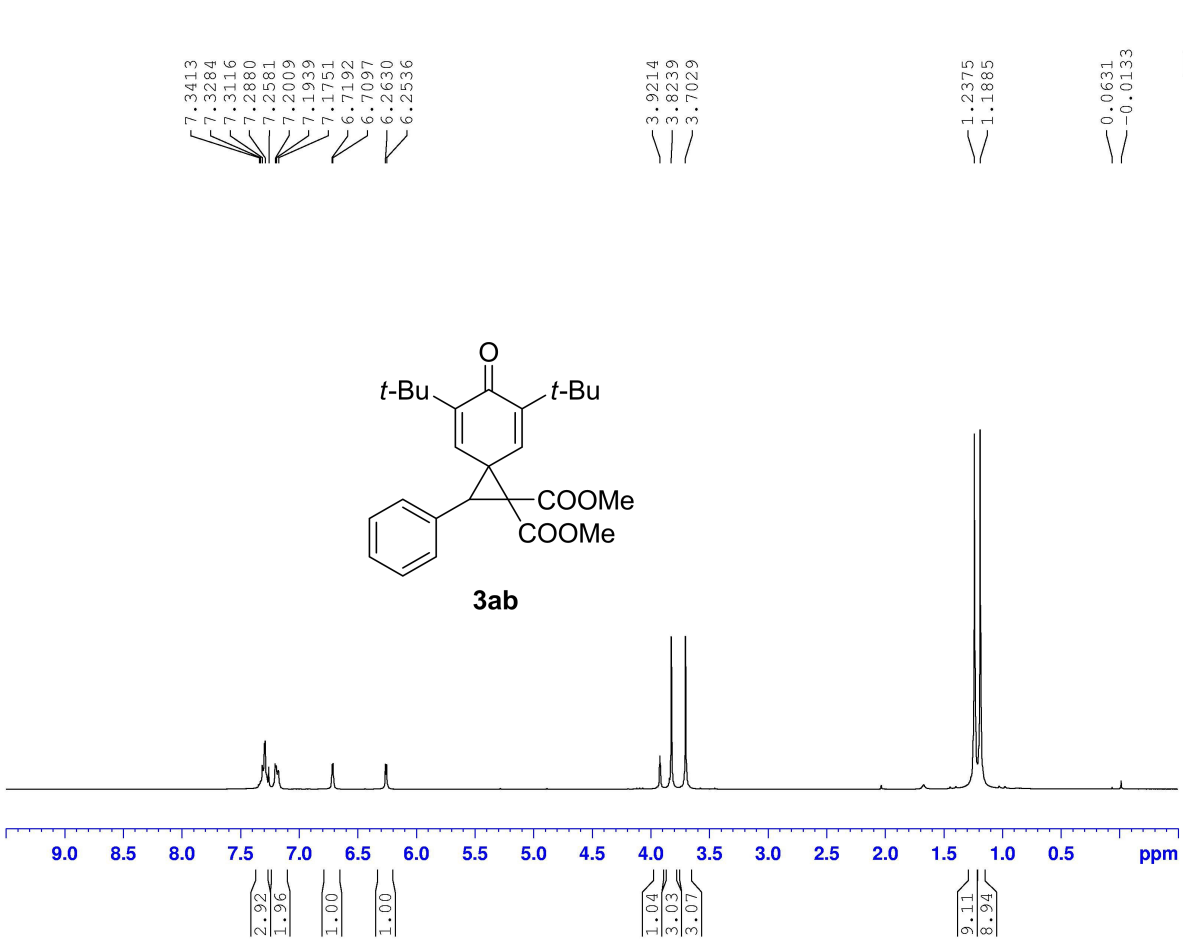
3ta





BRUKER

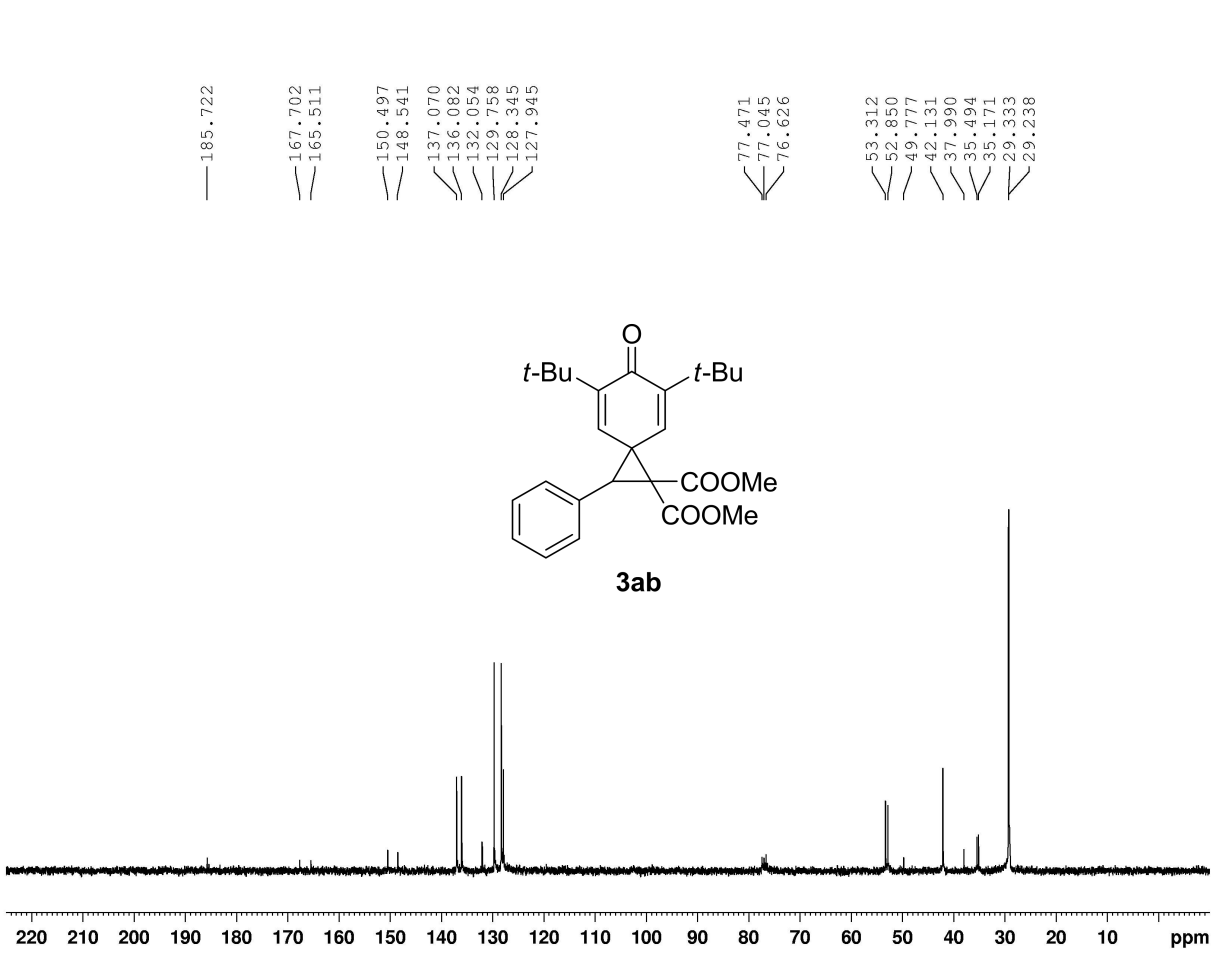
NAME	GK150531-2-C
EXPNO	1
PROCNO	1
Date_	20150531
Time	12.32
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	142
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	40.3
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



NAME	GK150417-4
EXPNO	1
PROCNO	1
Date_	20150417
Time	13.19
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	1
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	57
DW	69.333 usec
DE	7.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

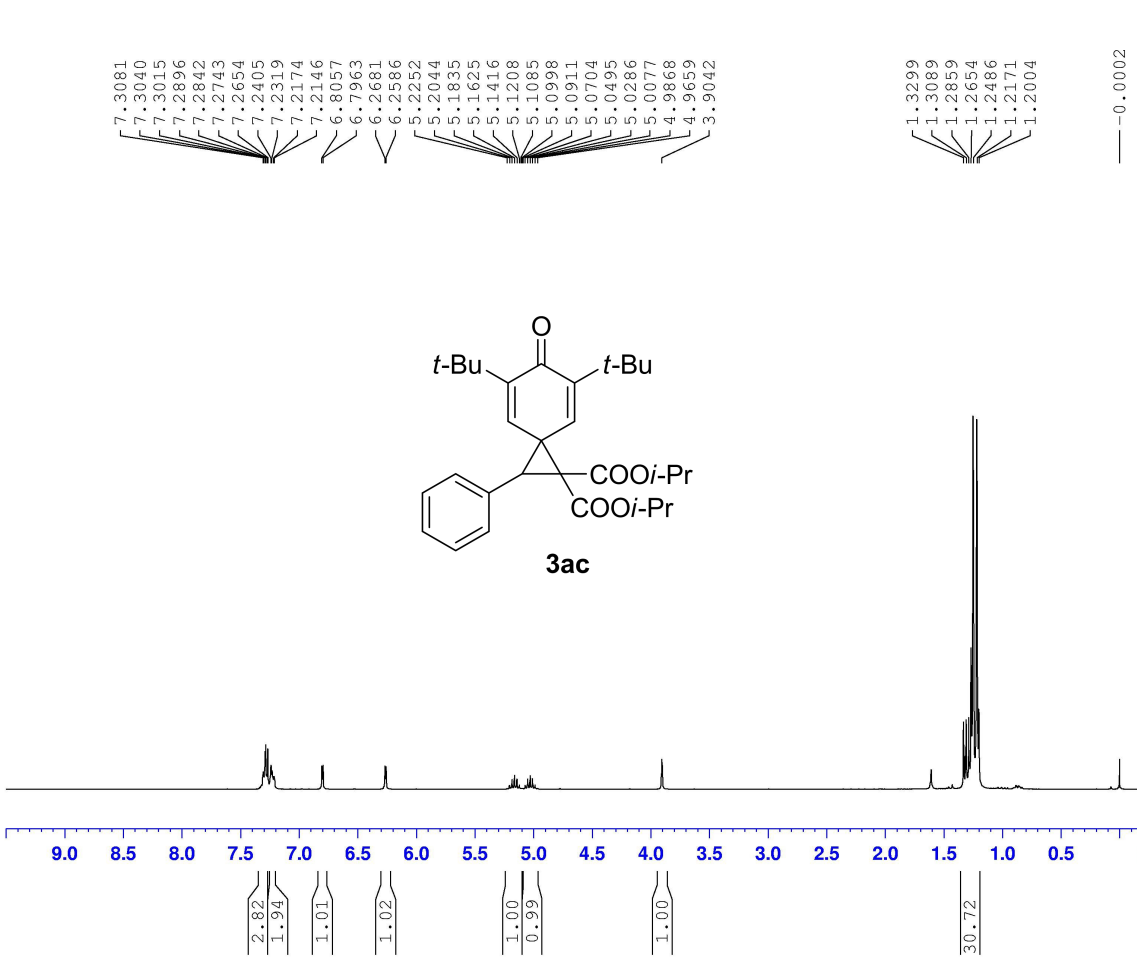
======== CHANNEL f1 ========

NUC1	1H
P1	13.25 usec
PL1	-1.00 dB
PL1W	12.36450577 W
SFO1	300.1331514 MHz
SI	32768
SF	300.1300063 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.90



BRUKER

NAME	GK-150506-6-C
EXPNO	1
PROCNO	1
Date_	20150506
Time	7.53
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	205
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40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7.2896
7.2842
7.2743
7.2654
7.2405
7.2319
7.2174
7.2146
6.8057
6.7963
6.2681
6.2586
5.2252
5.2044
5.1835
5.1625
5.1416
5.1208
5.1085
5.0998
5.0911
5.0704
5.0495
5.0286
5.0077
4.9868
4.9659
3.9042
1.3299
1.3089
1.2859
1.2654
1.2486
1.2171
1.2004
0.0002
BRUKER
NAME GK150605-2
EXPNO 1
PROCNO 1
Date_ 20150605
Time 6.15
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 1
SWH 7211.539 Hz
FIDRES 0.220079 Hz
AQ 2.2719646 sec
RG 114
DW 69.333 usec
DE 7.00 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 13.25 usec
PL1 -1.00 dB
PL1W 12.36450577 W
SFO1 300.1331514 MHz
SI 32768
SF 300.1300041 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 0.90
O
t-Bu
t-Bu
COOi-Pr
COOi-Pr
3ac
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
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0.5
ppm
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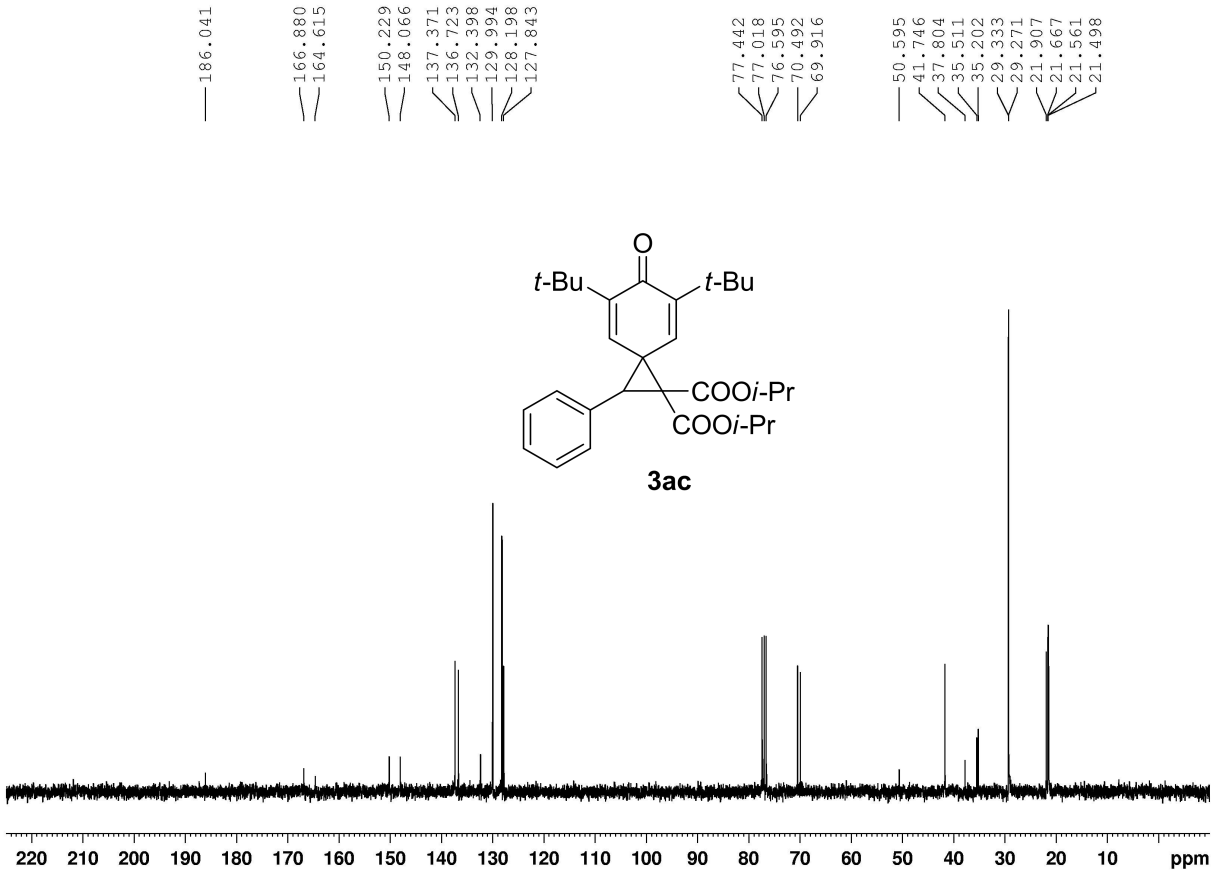
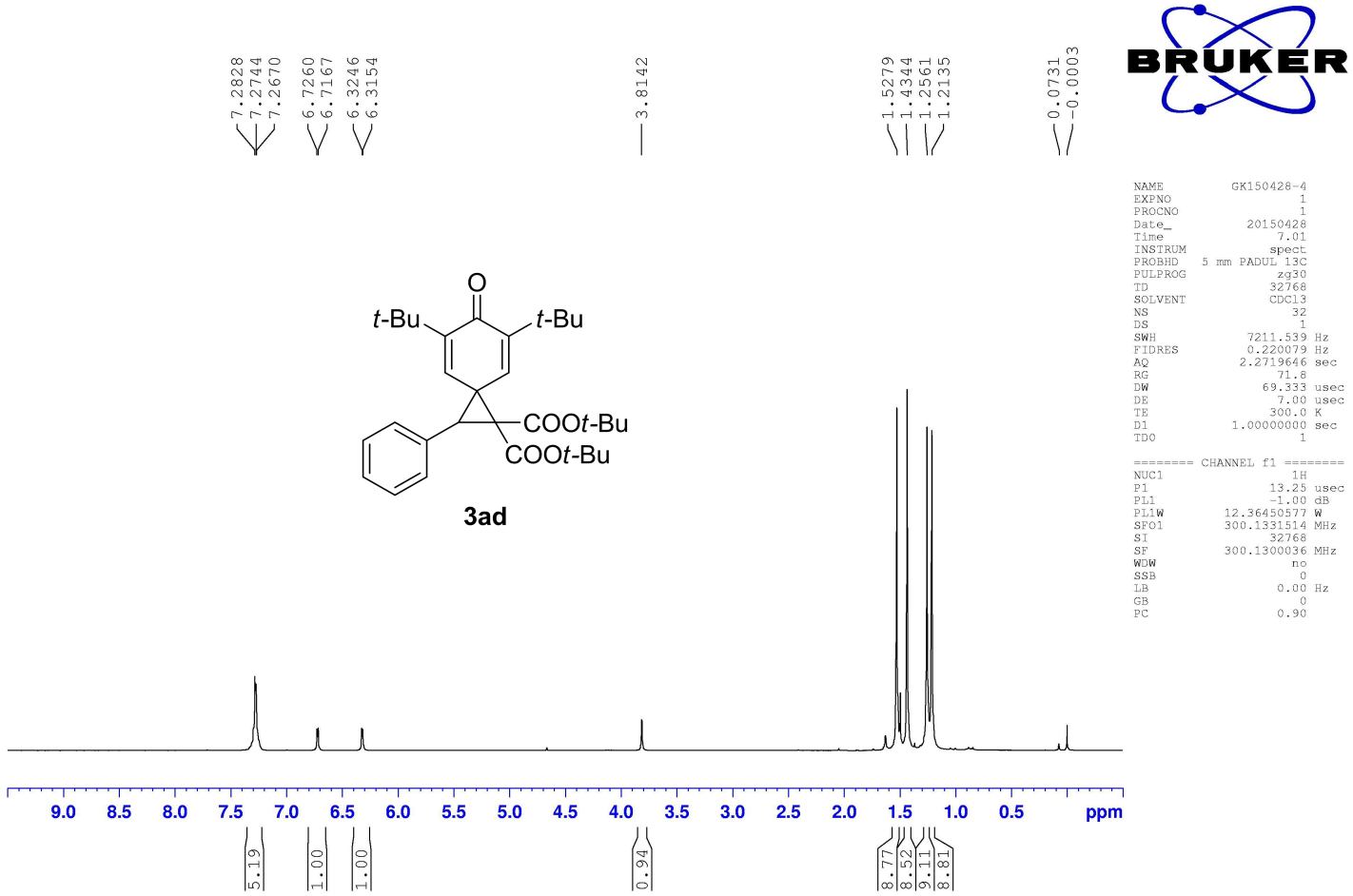
1.01
1.02
1.00
0.99
1.00
30.72



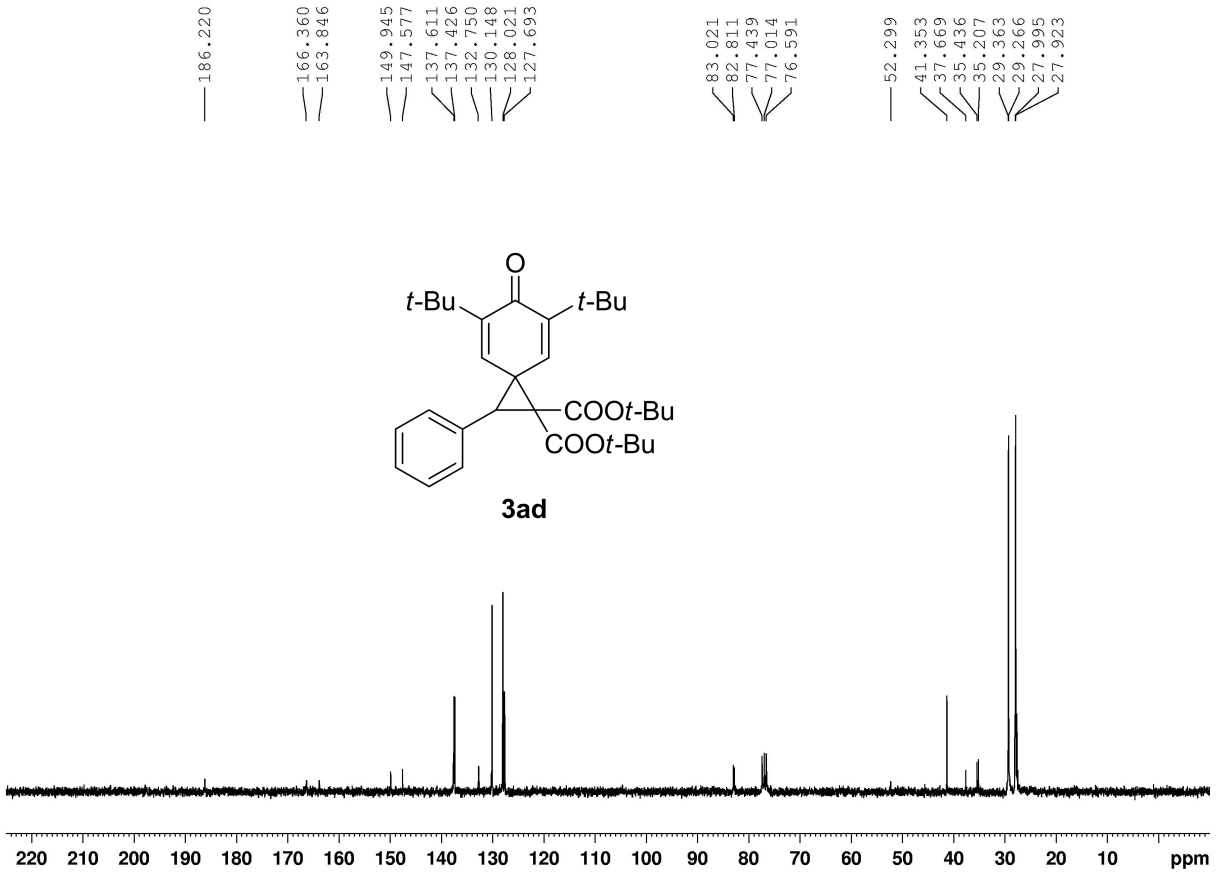
BRUKER

NAME	GK-150605-2-C
EXPNO	1
PROCNO	1
Date_	20150605
Time	6.21
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	246
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

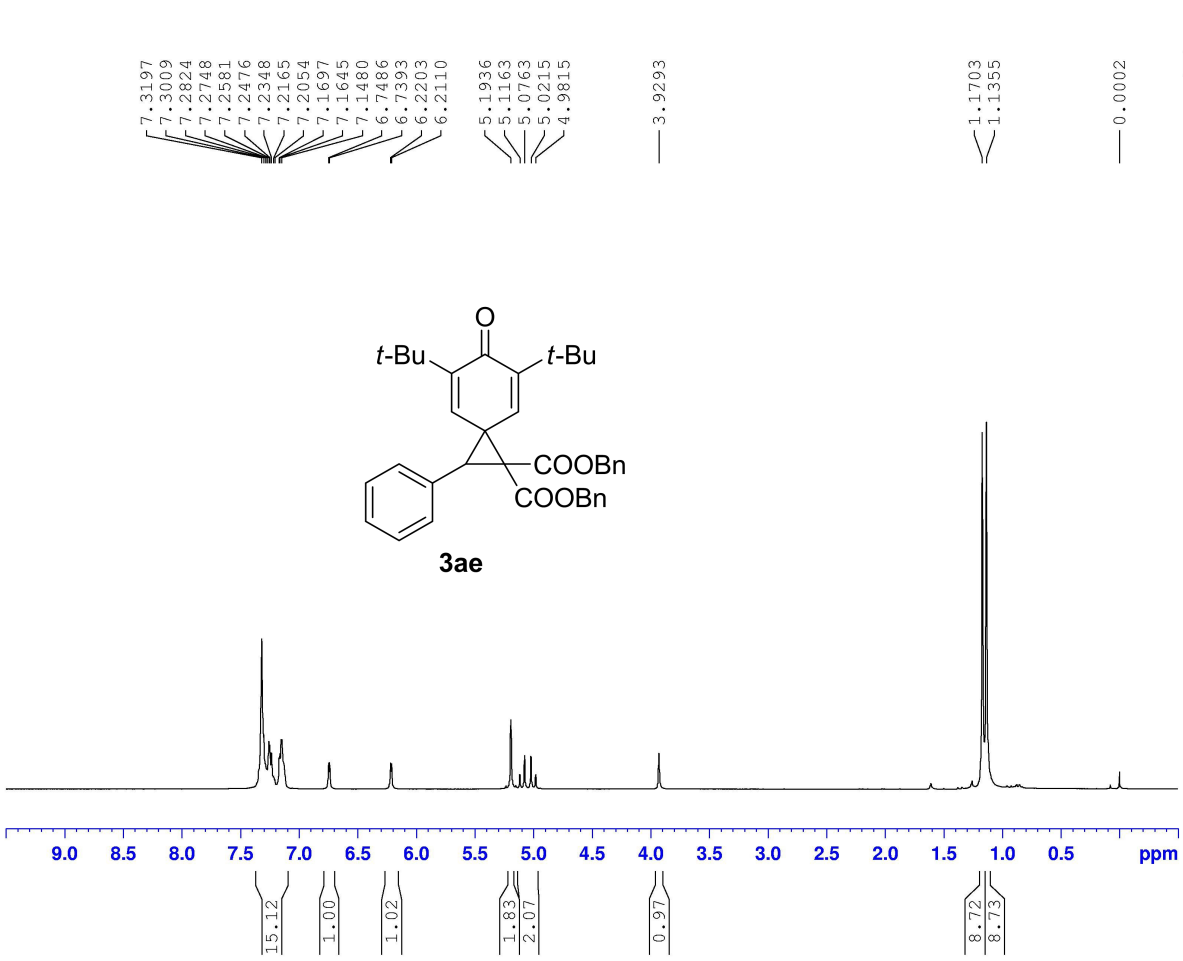


BRUKER

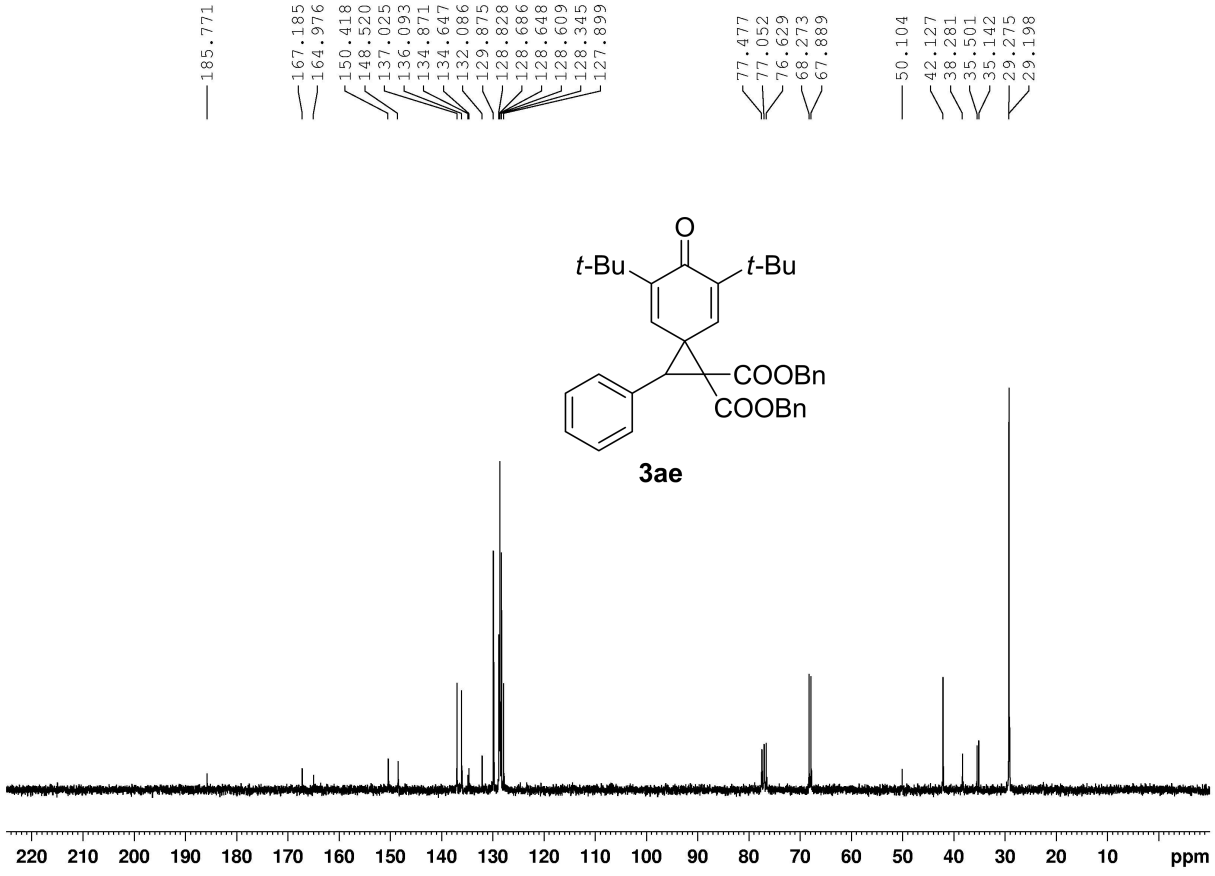
NAME	GK150428-4
EXPNO	1
PROCNO	1
Date_	20150428
Time	7.01
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	32
DS	1
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	71.8
DW	69.333 usec
DE	7.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.25 usec
PL1	-1.00 dB
PL1W	12.36450577 W
SFO1	300.1331514 MHz
SI	32768
SF	300.1300036 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.90




```
NAME        GK-150506-4-C
EXPNO                   1
PROCNO                  1
Date_            20150506
Time                 7.30
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zgdc
TD                  65536
SOLVENT             CDC13
NS                    416
DS                      4
SWH           19531.250 Hz
FIDRES         0.298023 Hz
AQ             1.6777716 sec
RG                   45.2
DW                25.600 usec
DE                 7.00 usec
TE                 300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.40 usec
PL1                -1.00 dB
PL1W         42.37451935 W
SFO1          75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.62 dB
PL2W         12.36450577 W
PL12W         0.33898211 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677490 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

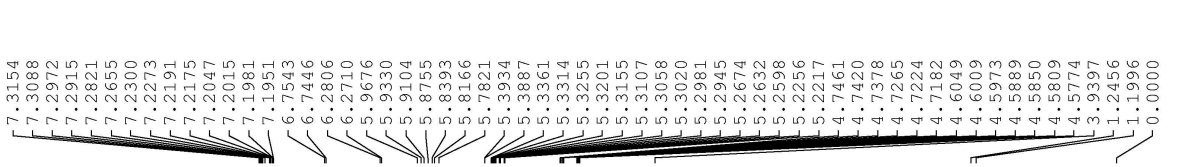


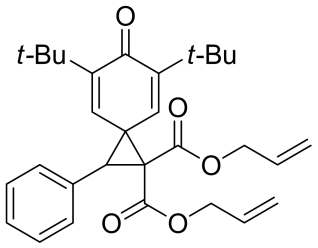

```
NAME          GK150428-8
EXPNO                  1
PROCNO                 1
Date_           20150428
Time                7.21
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     1
SWH            7211.539 Hz
FIDRES         0.220079 Hz
AQ             2.2719646 sec
RG                  71.8
DW                69.333 usec
DE                 7.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W        12.36450577 W
SFO1        300.1331514 MHz
SI                 32768
SF          300.1300094 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                 0.90
```



BRUKER

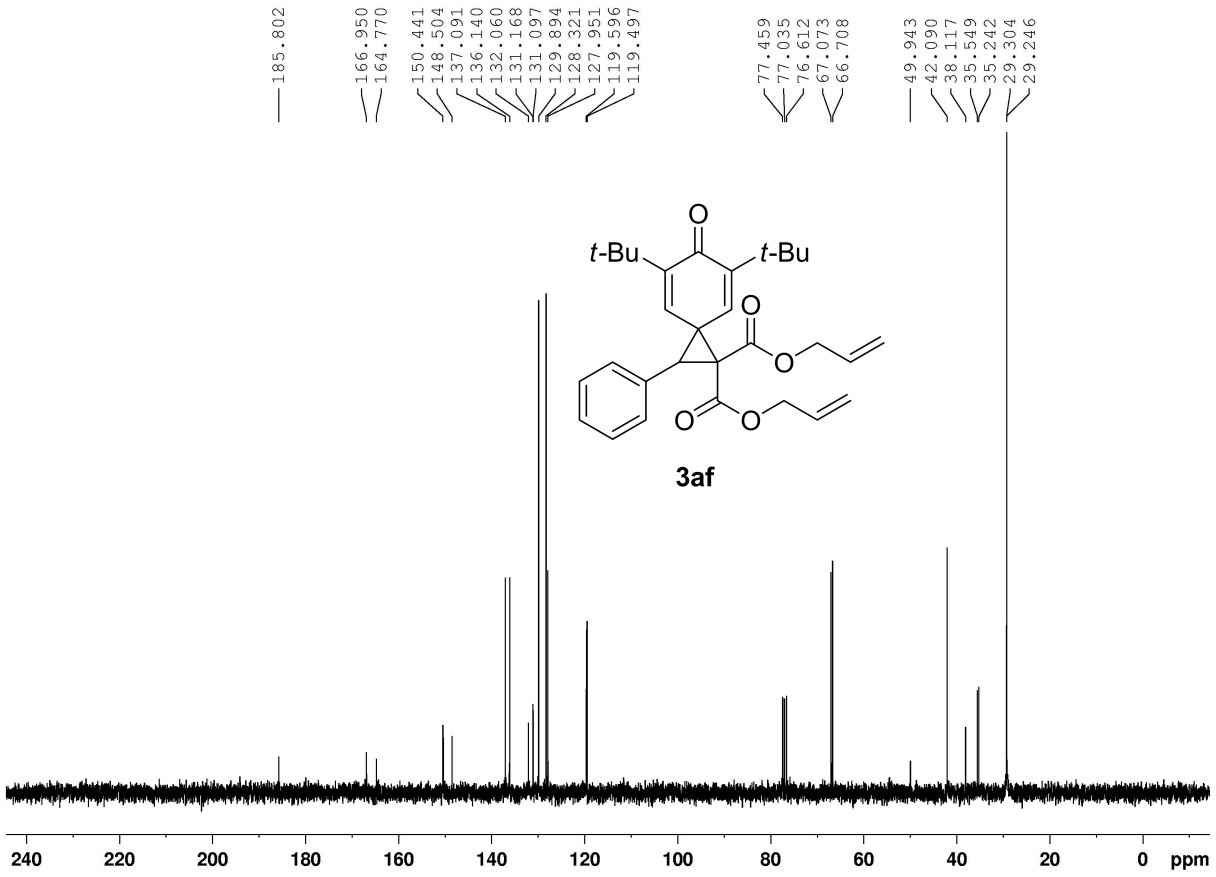
NAME	GK-150506-8-C
EXPNO	1
PROCNO	1
Date_	20150506
Time	8.03
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	294
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





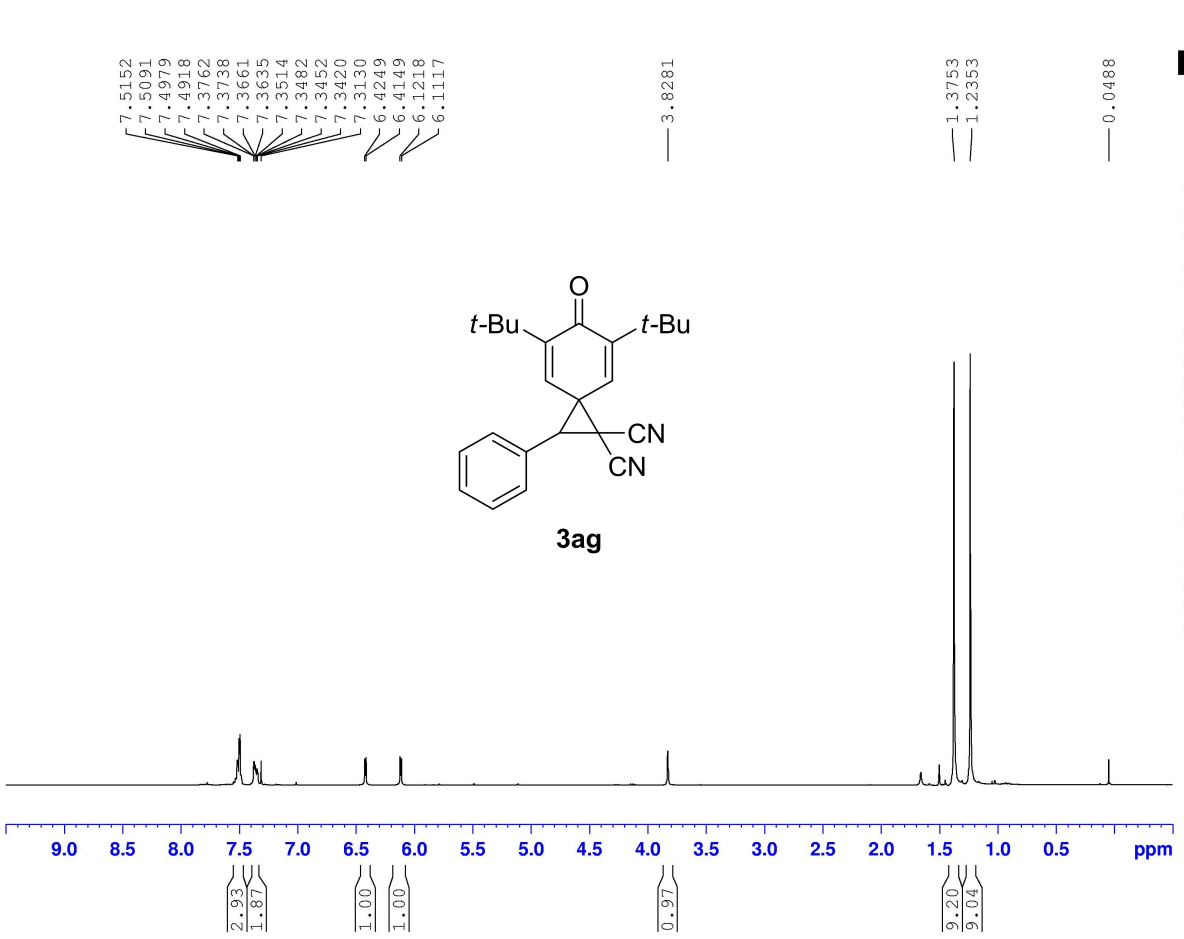
3af

```
NAME          GK150819-1
EXPNO                  1
PROCNO                 1
Date_           20150819
Time                6.13
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    32
DS                     1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ            2.2719646 sec
RG                    64
DW               69.333 usec
DE                7.00 usec
TE               300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W        12.36450577 W
SFO1        300.1331514 MHz
SI                 32768
SF          300.1300041 MHz
WDW                   no
SSB                    0
LB                0.00 Hz
GB                     0
PC                0.90
```

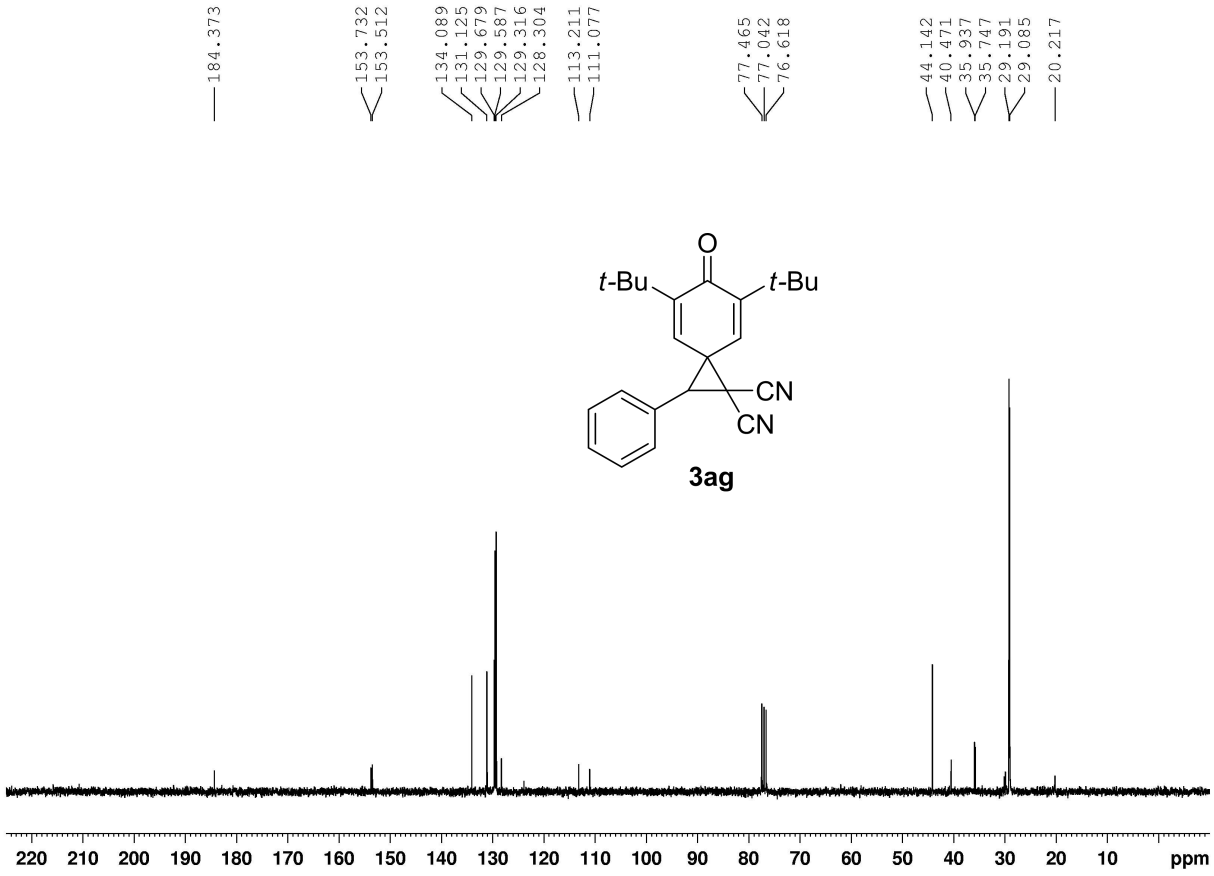


BRUKER

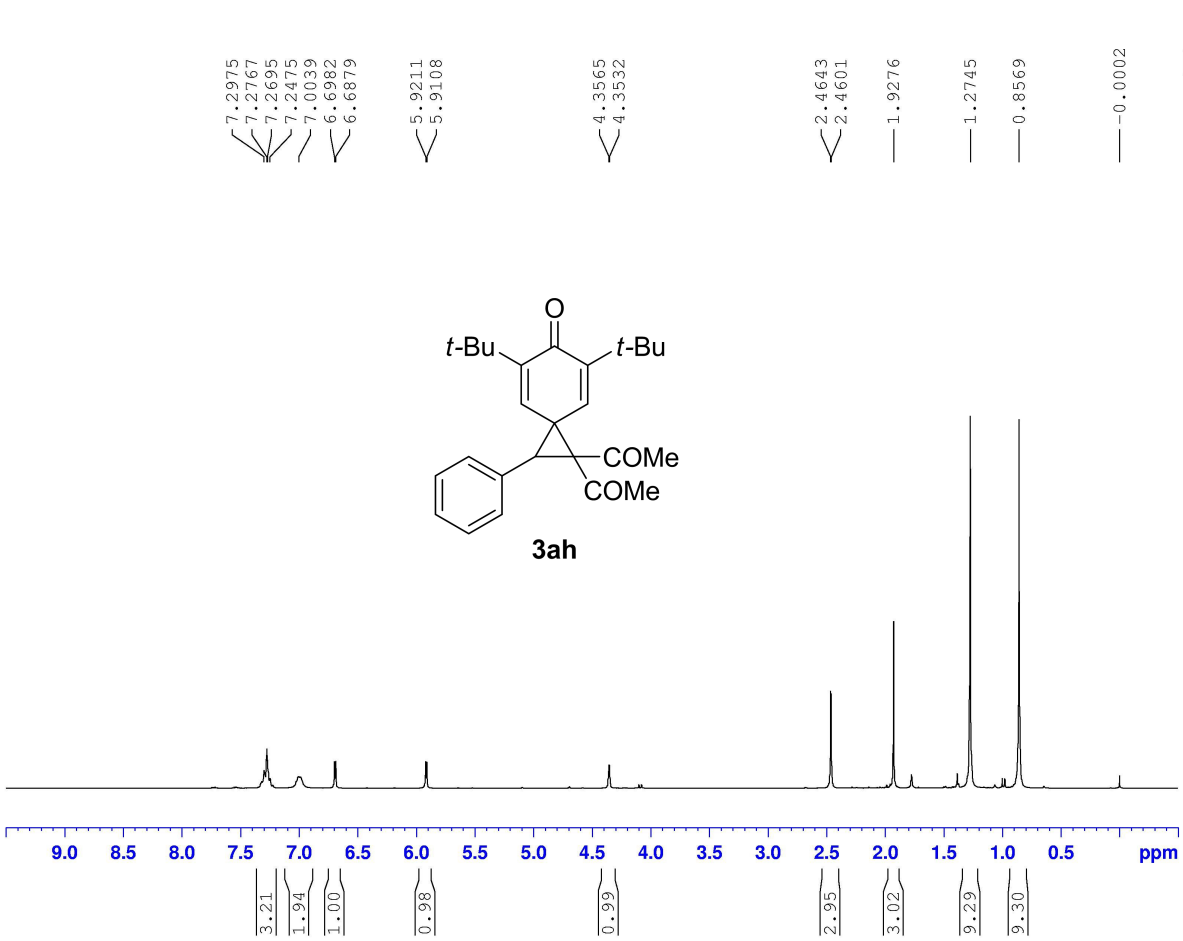
NAME	GK150815-1-C
EXPNO	1
PROCNO	1
Date_	20150819
Time	6.19
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	73
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



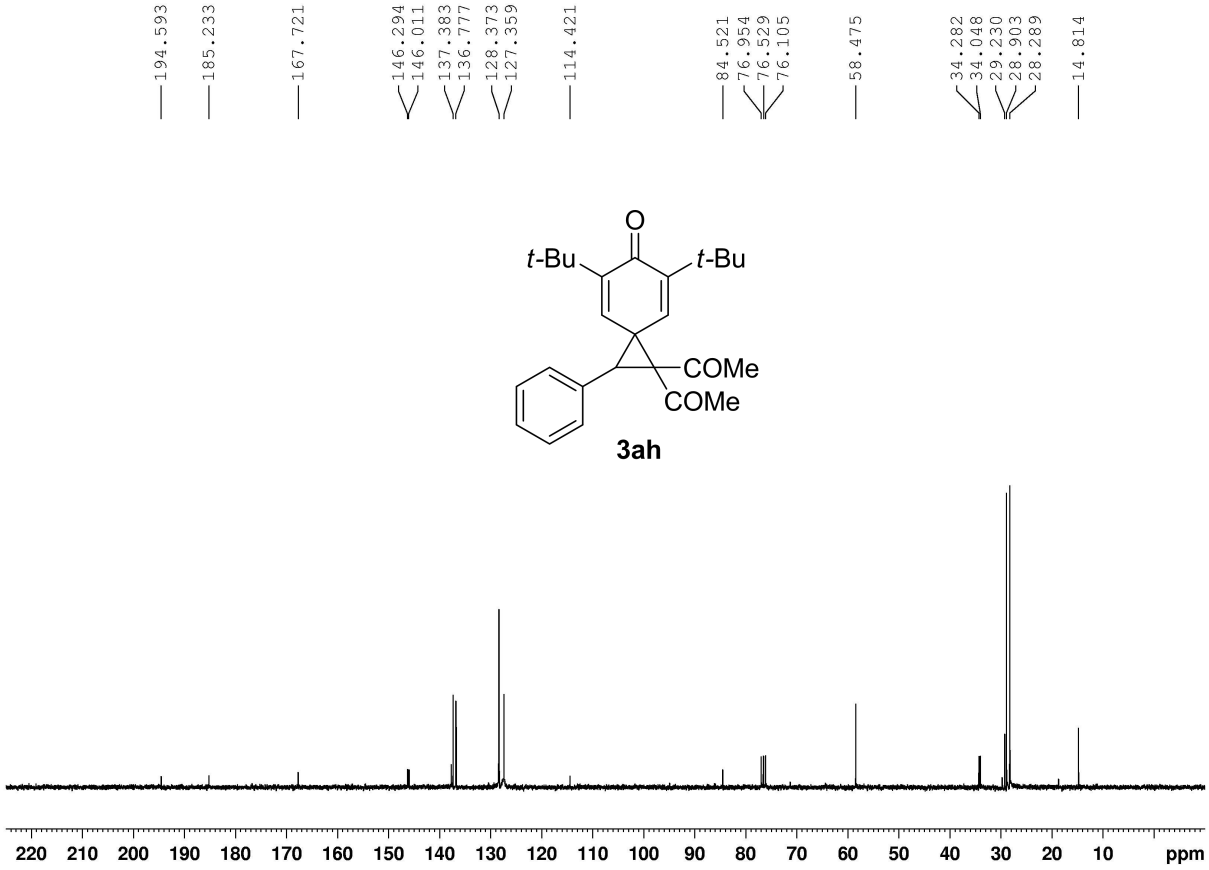

```
NAME          GK20150812-1
EXPNO                    1
PROCNO                   1
Date_             20150812
Time                 12.05
INSTRUM              spect
PROBHD   5 mm PADUL 13C
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      16
DS                       1
SWH              7211.539 Hz
FIDRES           0.220079 Hz
AQ               2.2719646 sec
RG                    90.5
DW                69.333 usec
DE                 7.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                  13.25 usec
PL1                 -1.00 dB
PL1W         12.36450577 W
SFO1         300.1331514 MHz
SI                   32768
SF           300.1299899 MHz
WDW                    no
SSB                     0
LB                 0.00 Hz
GB                      0
PC                   0.90
```



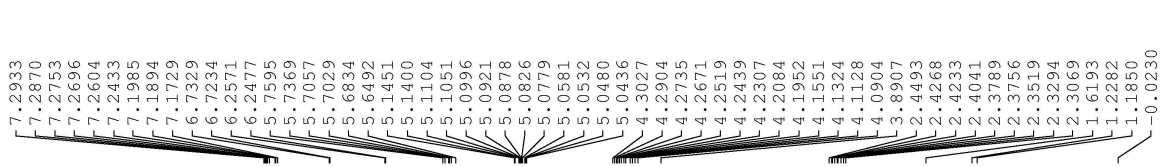

```
NAME      GK20150812-1-C
EXPNO                 1
PROCNO                1
Date_          20150812
Time              12.39
INSTRUM           spect
PROBHD   5 mm PADUL 13C
PULPROG             zgdc
TD                65536
SOLVENT           CDCl3
NS                  195
DS                    4
SWH           19531.250 Hz
FIDRES        0.298023 Hz
AQ            1.6777716 sec
RG                 45.2
DW               25.600 usec
DE                7.00 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.40 usec
PL1                -1.00 dB
PL1W        42.37451935 W
SFO1         75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.62 dB
PL2W        12.36450577 W
PL12W        0.33898211 W
SFO2        300.1312005 MHz
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

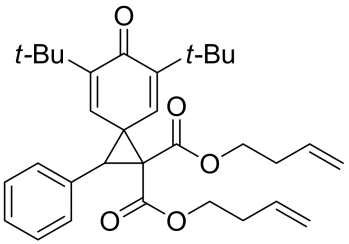



```
NAME          GK150816-3
EXPNO                  1
PROCNO                 1
Date_           20150816
Time                9.06
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH           6188.119 Hz
FIDRES        0.188846 Hz
AQ            2.6477044 sec
RG                    57
DW               80.800 usec
DE                7.00 usec
TE                300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W        12.36450577 W
SFO1        300.1318534 MHz
SI                 32768
SF          300.1300006 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



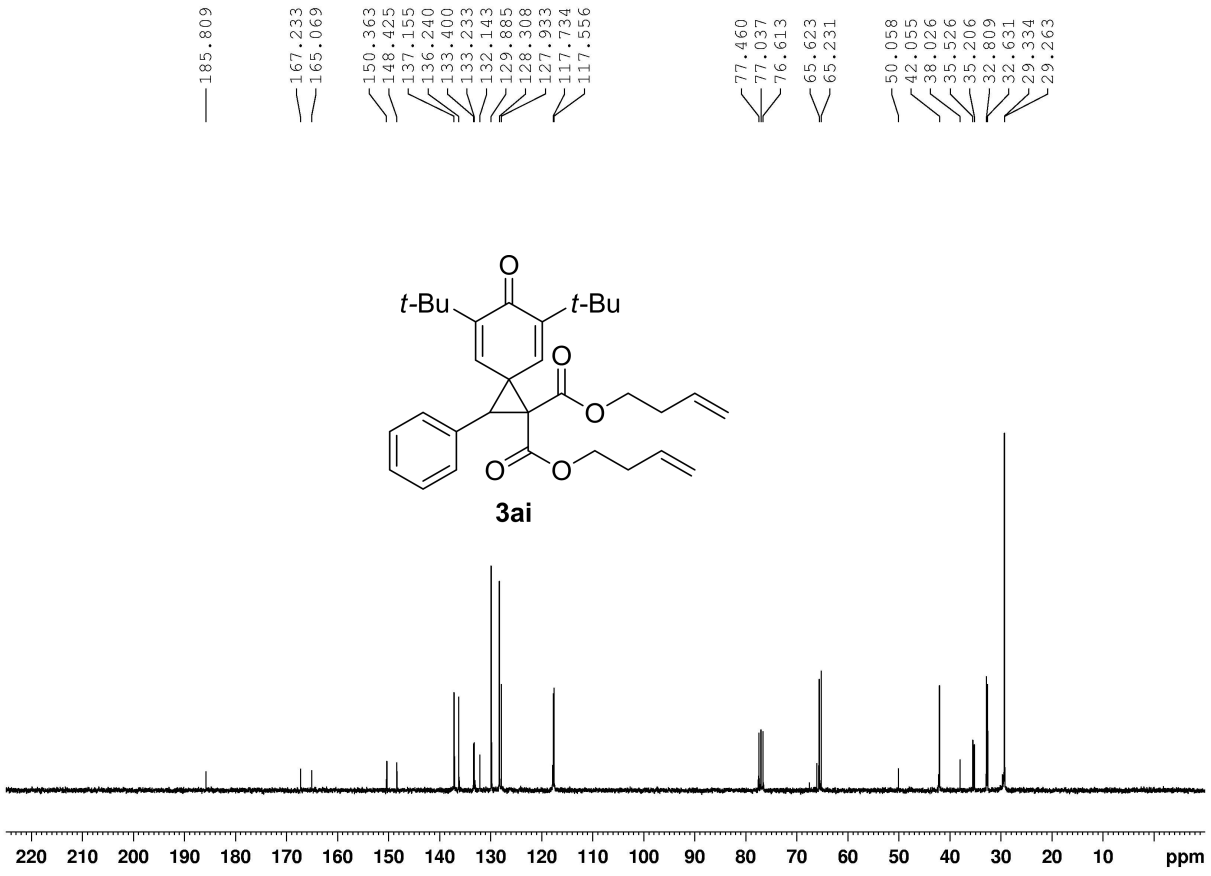

```
NAME          GK150817-1-C
EXPNO                    1
PROCNO                   1
Date_             20150817
Time                 13.48
INSTRUM              spect
PROBHD   5 mm PADUL 13C
PULPROG               zgdc
TD                   65536
SOLVENT              CDCl3
NS                     258
DS                       4
SWH              19531.250 Hz
FIDRES           0.298023 Hz
AQ               1.6777716 sec
RG                    25.4
DW                  25.600 usec
DE                   7.00 usec
TE                  300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.40 usec
PL1                  -1.00 dB
PL1W           42.37451935 W
SFO1            75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 14.62 dB
PL2W           12.36450577 W
PL12W           0.33898211 W
SFO2           300.1312005 MHz
SI                   32768
SF              75.4677867 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.50
```





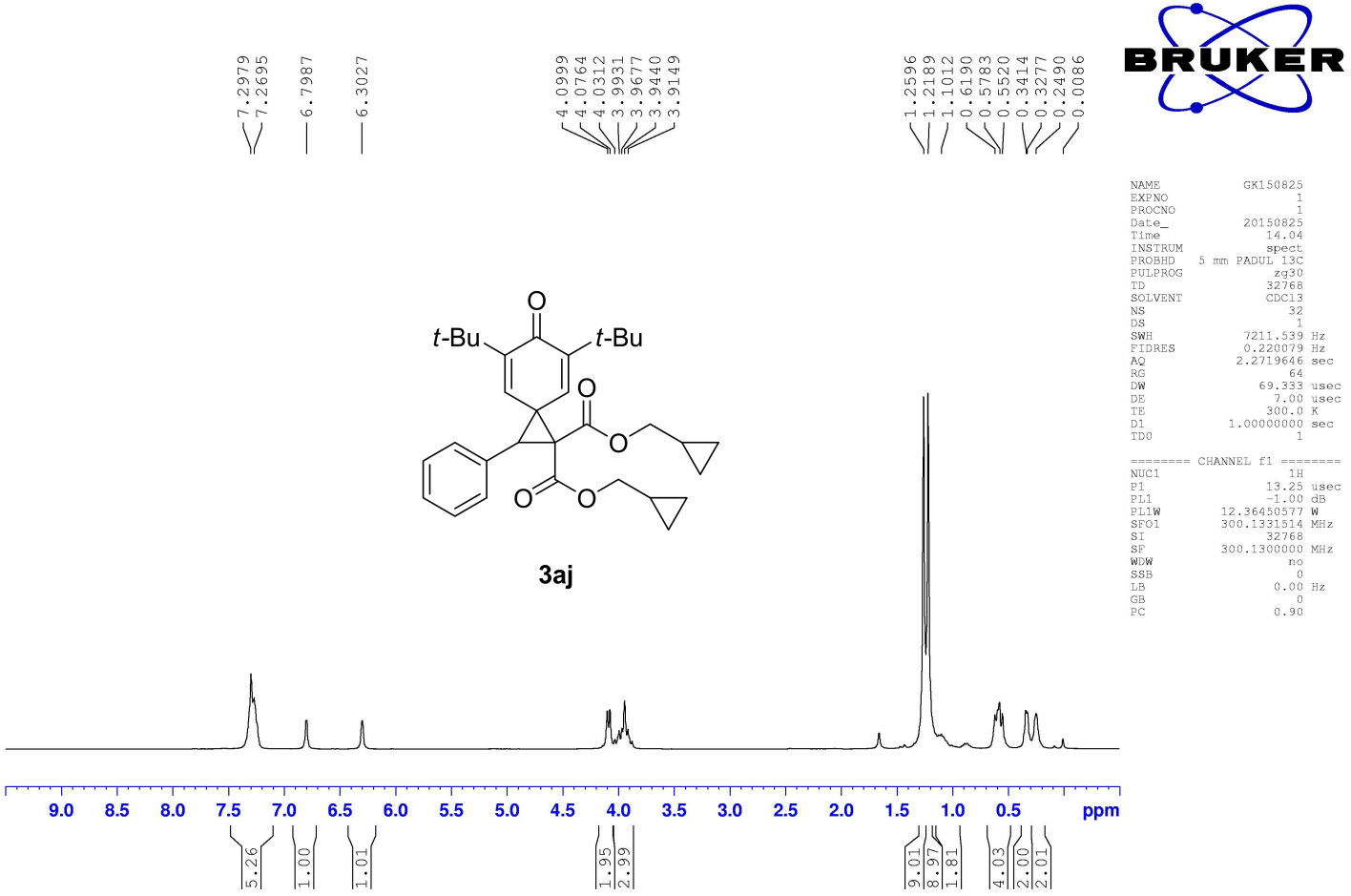
3ai

```
NAME          GK150821-1
EXPNO                  2
PROCNO                 1
Date_           20150821
Time               13.34
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    16
DS                     1
SWH           7211.539 Hz
FIDRES        0.220079 Hz
AQ           2.2719646 sec
RG                  71.8
DW             69.333 usec
DE               7.00 usec
TE               300.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                13.25 usec
PL1               -1.00 dB
PL1W       12.36450577 W
SFO1       300.1331514 MHz
SI                 32768
SF         300.1300109 MHz
WDW                   EM
SSB                    0
LB              0.30 Hz
GB                     0
PC                0.90
```

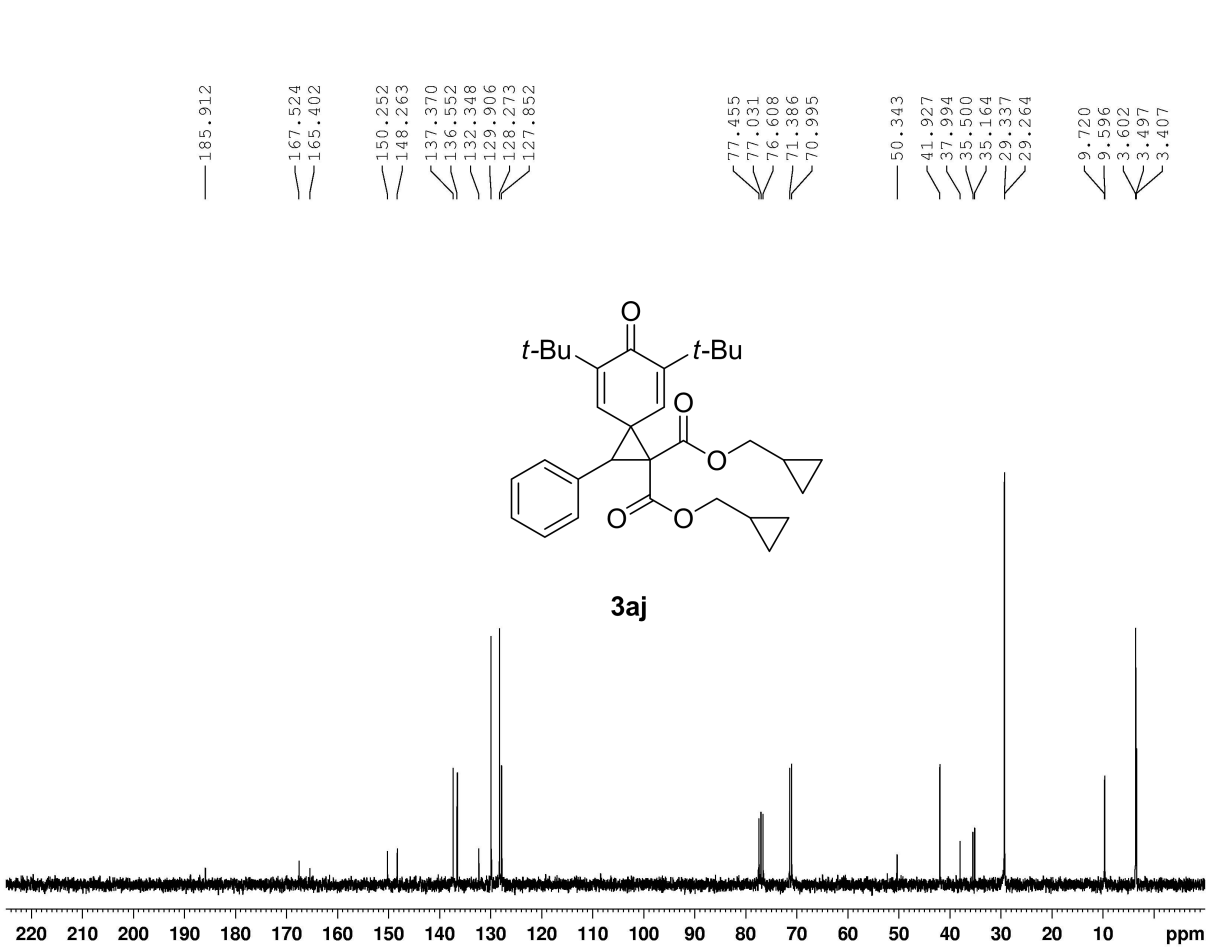


BRUKER

NAME	GK-150821-1-C
EXPNO	1
PROCNO	1
Date_	20150821
Time	18.07
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDC13
NS	427
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	25.4
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

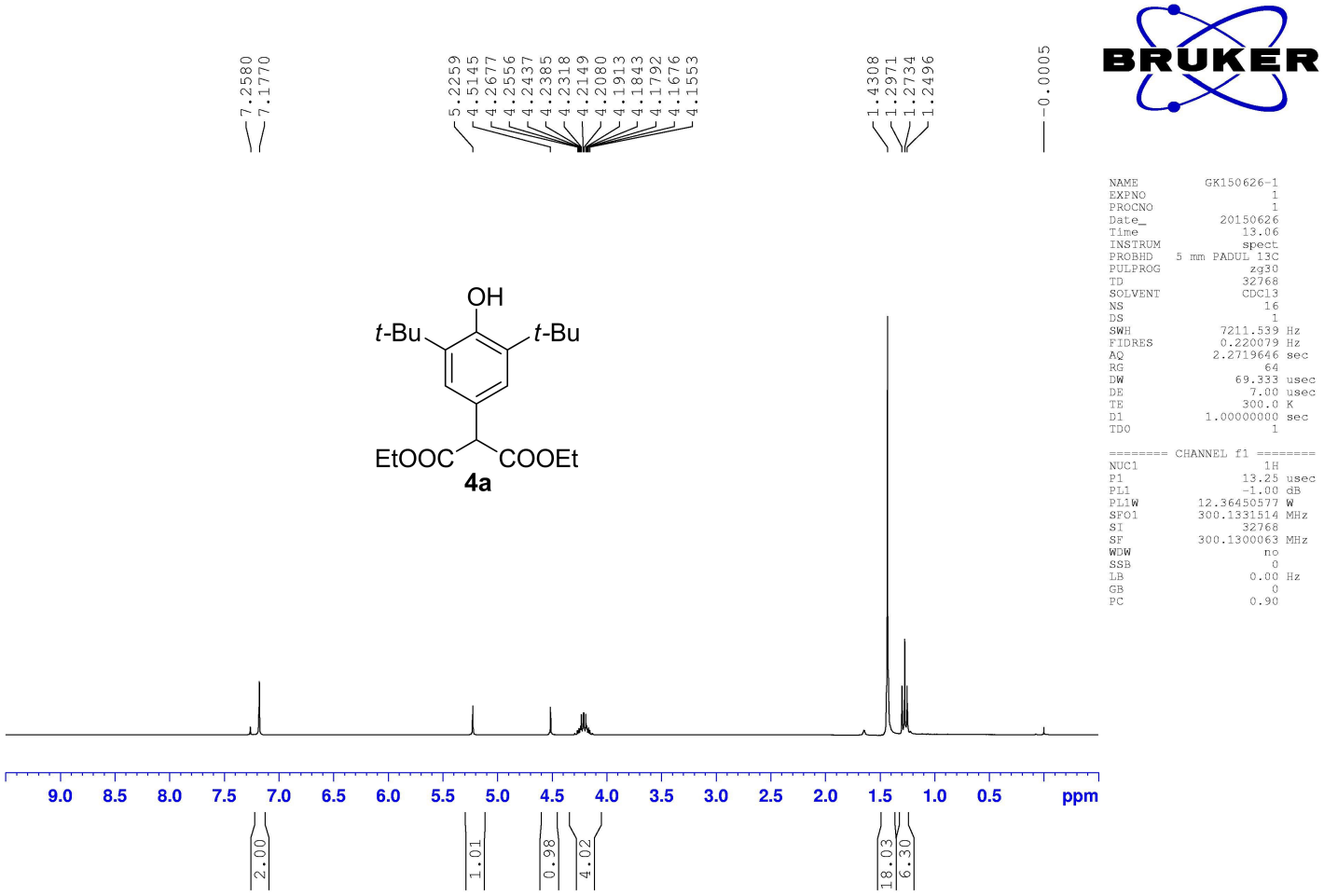



```
NAME              GK150825
EXPNO                    1
PROCNO                   1
Date_             20150825
Time                 14.04
INSTRUM              spect
PROBHD   5 mm PADUL 13C
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       1
SWH               7211.539 Hz
FIDRES            0.220079 Hz
AQ                2.2719646 sec
RG                      64
DW                69.333 usec
DE                 7.00 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                 13.25 usec
PL1                -1.00 dB
PL1W          12.36450577 W
SFO1          300.1331514 MHz
SI                   32768
SF            300.1300000 MHz
WDW                     no
SSB                      0
LB                0.00 Hz
GB                       0
PC                0.90
```



BRUKER

NAME	GK150825-C
EXPNO	1
PROCNO	1
Date_	20150825
Time	14.06
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgdc
TD	65536
SOLVENT	CDCl3
NS	96
DS	4
SWH	19531.250 Hz
FIDRES	0.298023 Hz
AQ	1.6777716 sec
RG	45.2
DW	25.600 usec
DE	7.00 usec
TE	300.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.40 usec
PL1	-1.00 dB
PL1W	42.37451935 W
SFO1	75.4764278 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	80.00 usec
PL2	-1.00 dB
PL12	14.62 dB
PL2W	12.36450577 W
PL12W	0.33898211 W
SFO2	300.1312005 MHz
SI	32768
SF	75.4677490 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.50

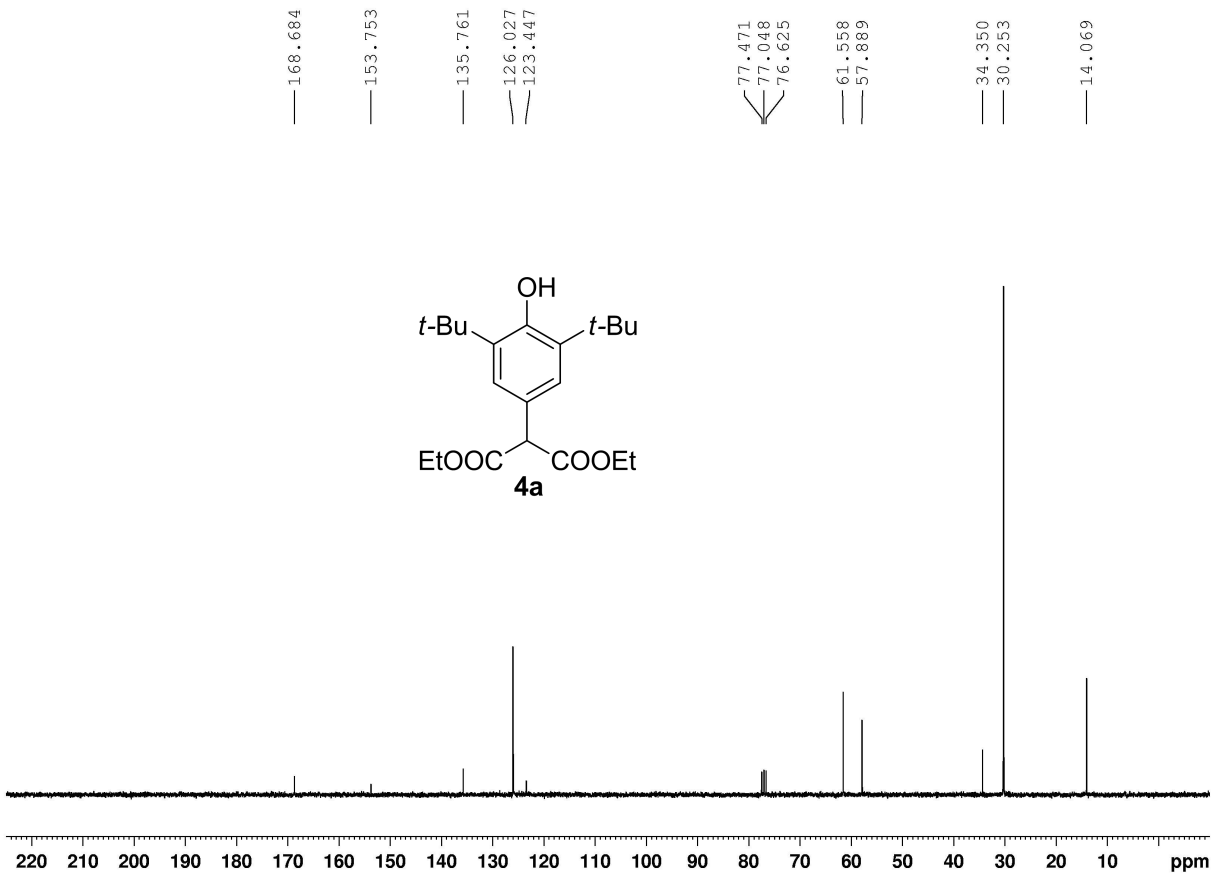


BRUKER

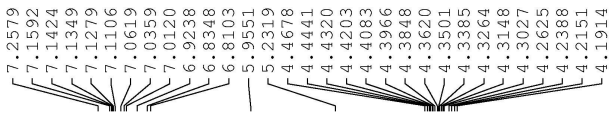
NAME	GK150626-1
EXPNO	1
PROCNO	1
Date_	20150626
Time	13.06
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	1
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	64
DW	69.333 usec
DE	7.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1

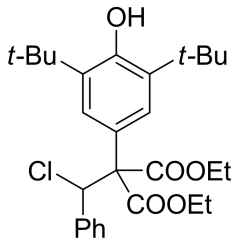
======== CHANNEL f1 ========

NUC1	1H
P1	13.25 usec
PL1	-1.00 dB
PL1W	12.36450577 W
SFO1	300.1331514 MHz
SI	32768
SF	300.1300063 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.90



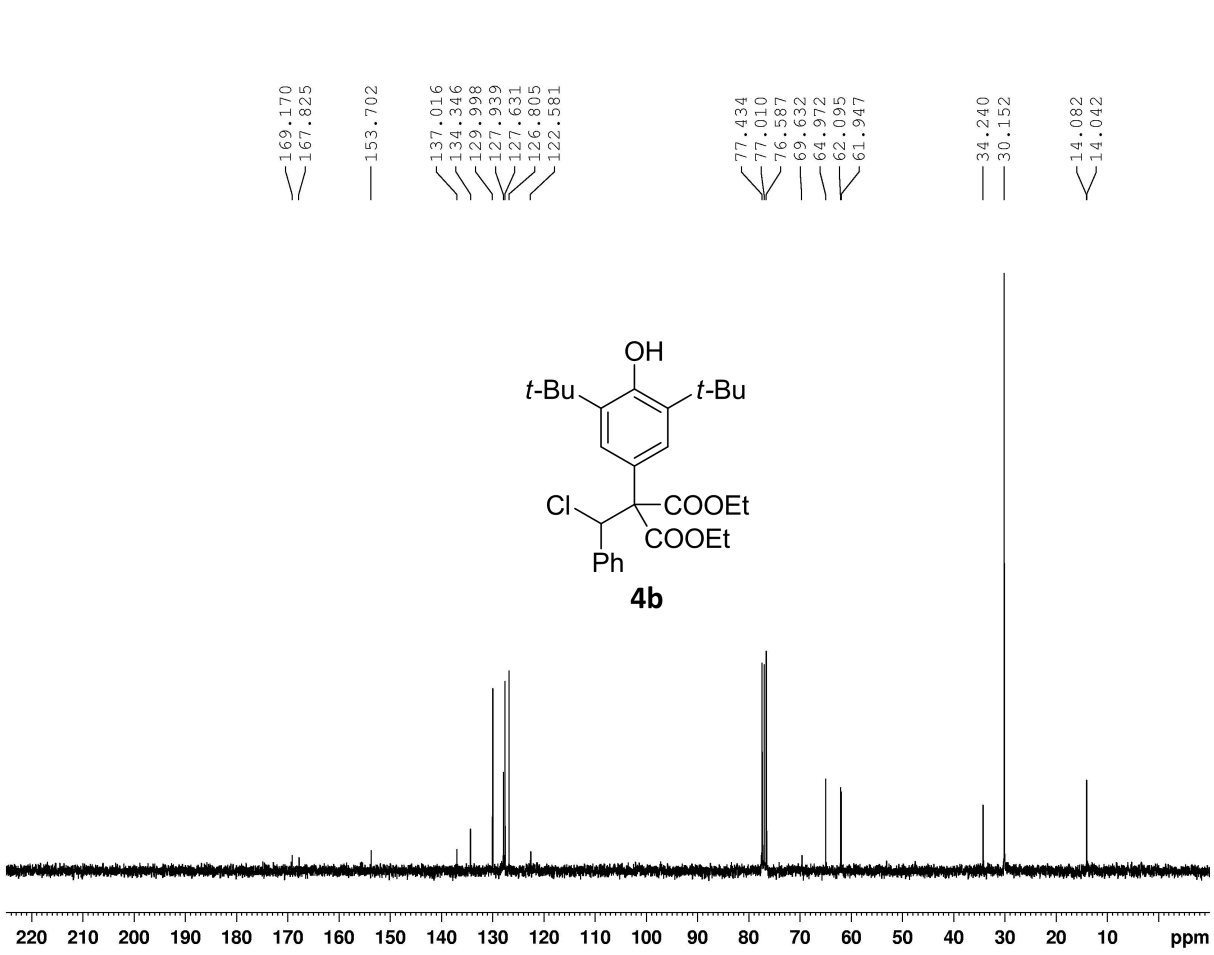

```
NAME      GK150629-1-1-C
EXPNO                  1
PROCNO                 1
Date_           20150310
Time                3.13
INSTRUM            spect
PROBHD   5 mm PADUL 13C
PULPROG              zgdc
TD                 65536
SOLVENT            CDCl3
NS                    74
DS                     4
SWH           19531.250 Hz
FIDRES         0.298023 Hz
AQ             1.6777716 sec
RG                  45.2
DW                25.600 usec
DE                 7.00 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.40 usec
PL1                -1.00 dB
PL1W         42.37451935 W
SFO1          75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               14.62 dB
PL2W         12.36450577 W
PL12W         0.33898211 W
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677490 MHz
WDW                  EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```





4b

NAME	GK150626-2
EXPNO	1
PROCNO	1
Date_	20150626
Time	13.10
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	32768
SOLVENT	CDCl3
NS	16
DS	1
SWH	7211.539 Hz
FIDRES	0.220079 Hz
AQ	2.2719646 sec
RG	64
DW	69.333 usec
DE	7.00 usec
TE	300.0 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	13.25 usec
PL1	-1.00 dB
PL1W	12.36450577 W
SFO1	300.1331514 MHz
SI	32768
SF	300.1300063 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	0.90




```
NAME          GK150701-1 C
EXPNO                   1
PROCNO                  1
Date_            20150701
Time                12.48
INSTRUM             spect
PROBHD    5 mm PADUL 13C
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                    451
DS                      4
SWH            19531.250 Hz
FIDRES          0.298023 Hz
AQ             1.6777716 sec
RG                   40.3
DW                 25.600 usec
DE                  7.00 usec
TE                 300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.40 usec
PL1                 -1.00 dB
PL1W         42.37451935 W
SFO1          75.4764278 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                14.62 dB
PL2W         12.36450577 W
PL12W         0.33898211 W
SFO2         300.1312005 MHz
SI                  32768
SF           75.4677490 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.10
```