

Supporting Information

Cycloaddition of *N*-arylnitrones with Donor-Acceptor Oxiranes via C–C Bond Cleavage to Construct 1,5,2-Dioxazinanes

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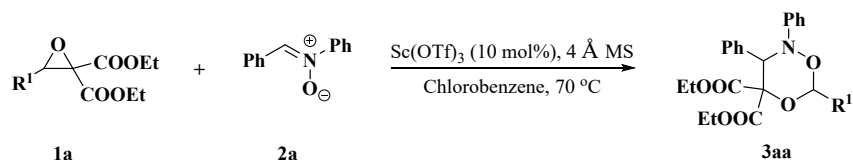
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1. General information

^1H NMR spectra were taken on a Bruker AVANCE III 600 or 400 MHz NMR spectrometer. The chemical shifts are reported in ppm downfield to the CDCl_3 resonance ($\delta = 7.27$) and CD_3OD ($\delta = 3.31$). Spectra are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment. $^{13}\text{C}\{^1\text{H}\}$ NMR data were collected at 150 and 100 MHz with complete proton decoupling. The chemical shifts are reported in ppm downfield to the central CDCl_3 resonance ($\delta = 77.0$). High-resolution mass spectra were performed on a microTOF-Q II instrument with an ESI source. Melting points were measured with a RD-II melting point apparatus and are uncorrected. Unless otherwise noted, all reagents and solvents obtained from commercial sources were used without further purification. Deuterated solvents were purchased from Sigma-Aldrich. Column chromatography was performed on silica gel (200–300 mesh) using petroleum ether /ethyl acetate. All yields were referred to isolated yields (average of two runs) of compounds. Starting materials such as *N*-arylnitrones (**2a–2n**)¹ and donor-acceptor oxiranes **1**² were separately synthesized according to the corresponding literature procedures.

2. General procedure

2.1 The procedure for $\text{Sc}(\text{OTf})_3$ -catalyzed cycloaddition of *N*-arylnitrones and donor-acceptor oxiranes



The mixture of donor-acceptor oxirane **1a** (0.14 mmol, 1.4 equiv), *N*-phenylnitronium **2a** (0.1 mmol), $\text{Sc}(\text{OTf})_3$ (0.01 mmol, 10 mol%) and PhCl (0.8 mL) was stirred at 70 °C (oil bath) under air atmosphere. Subsequently, the reaction was monitored by TLC. Upon completion of the consumption of the *N*-phenylnitronium **2a**, the reaction mixture

was directly purified by silica gel column chromatography to give the cycloaddition product **3aa**.

2.2 Operational procedure for 3.0 mmol-scale preparation of **3aa**

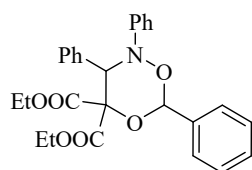
The mixture of donor-acceptor oxirane **1a** (4.2 mmol, 1.4 equiv), *N*-phenylnitrone **2a** (3 mmol), Sc(OTf)₃ (0.3 mmol, 10 mol%) and PhCl (25 mL) was stirred at 70 °C (oil bath) under air atmosphere. Subsequently, the reaction was monitored by TLC. Upon completion of the consumption of the *N*-phenylnitrone **2a**, the reaction mixture was directly purified by silica gel column chromatography to give the cycloaddition product **3aa** (1.148 g, 83% yield).

2.3 Operational procedure for the synthesis of compound **4**

The reaction system of compound **3ga** (49.5 mg, 0.1 mmol) and KOH (0.15 mmol) in MeOH (1.0 mL) was stirred at 25 °C for 12 h. Finally, the reaction mixture was purified by silica gel column chromatography to produce the corresponding product **4** (36.8 mg, 90% yield).

3. Characterization data of products **3**

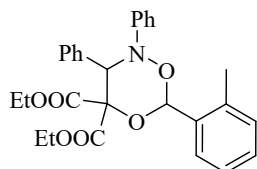
Diethyl 2,3,6-triphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (**3aa**)



White solid, mp 103–104 °C, 40 mg, yield: 87%. Eluent: petroleum ether/ethyl acetate = 30:1; ¹H NMR (400 MHz, CDCl₃): δ 7.76 (d, *J* = 6.7 Hz, 2H), 7.47–7.43 (m, 5H), 7.21–7.10 (m, 5H), 6.99 (d, *J* = 7.7 Hz, 2H), 6.91 (t, *J* = 7.3 Hz, 1H), 6.32 (s, 1H), 5.64 (s, 1H), 4.49–4.39 (m, 2H), 3.98–3.90 (m, 2H), 1.39 (t, *J* = 7.1 Hz, 3H), 0.85 (t, *J* = 7.1 Hz, 3H) ppm; ¹³C{¹H} NMR (100 MHz, CDCl₃): δ 167.4, 164.9, 147.9, 135.4, 132.5, 131.0, 129.8, 128.6, 128.5, 128.3, 127.6, 127.0, 123.1, 116.8,

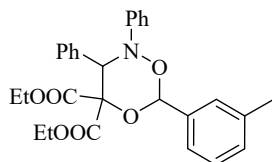
100.6, 84.0, 67.2, 63.1, 62.3, 14.3, 13.5 ppm. **HRMS** (ESI) m/z : $[M + H]^+$ calcd for $C_{27}H_{28}NO_6^+$, 462.1911; found, 462.1910.

Diethyl 2,3-diphenyl-6-(*o*-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (3ba)



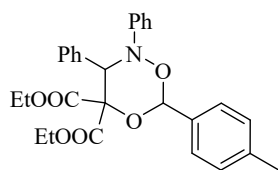
Yellow solid, mp 79–80 °C, 26 mg, yield: 54%. Eluent: petroleum ether/ethyl acetate = 30:1; 1H NMR (600 MHz, $CDCl_3$): δ 7.96–7.95 (m, 1H), 7.41–7.39 (m, 2H), 7.30–7.26 (m, 2H), 7.17–7.13 (m, 1H), 7.11–7.08 (m, 3H), 7.07–7.04 (m, 2H), 6.92–6.90 (m, 2H), 6.85–6.82 (m, 1H), 6.36 (s, 1H), 5.57 (s, 1H), 4.42–4.36 (m, 2H), 3.91–3.86 (m, 2H), 2.39 (s, 3H), 1.33 (t, $J = 7.2$ Hz, 3H), 0.78 (t, $J = 7.2$ Hz, 3H) ppm; $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): δ 166.7, 164.0, 147.1, 135.9, 132.6, 131.6, 130.1, 129.6, 128.7, 127.7, 126.7, 125.4, 125.3, 122.1, 115.6, 97.5, 83.1, 66.2, 62.1, 61.2, 18.1, 13.4, 12.6 ppm. **HRMS** (ESI) m/z : $[M + H]^+$ calcd for $C_{28}H_{30}NO_6^+$, 476.2068; found, 476.2060.

Diethyl 2,3-diphenyl-6-(*m*-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (3ca)



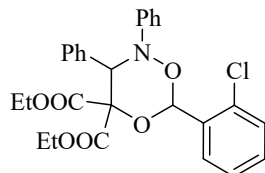
White solid, mp 79–81 °C, 37 mg, yield: 78%. Eluent: petroleum ether/ethyl acetate = 30:1; 1H NMR (400 MHz, $CDCl_3$): δ 7.61 (d, $J = 6.3$ Hz, 2H), 7.53 (d, $J = 6.8$ Hz, 2H), 7.43 (t, $J = 7.7$ Hz, 1H), 7.32 (t, $J = 7.5$ Hz, 1H), 7.22–7.16 (m, 5H), 7.05 (d, $J = 8.0$ Hz, 2H), 6.98 (t, $J = 7.3$ Hz, 1H), 6.32 (s, 1H), 5.68 (s, 1H), 4.57–4.44 (m, 2H), 4.05–3.97 (m, 2H), 2.48 (s, 3H), 1.46 (t, $J = 7.9$ Hz, 3H), 0.91 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 167.5, 164.9, 147.9, 138.3, 135.3, 132.5, 131.0, 130.5, 128.6, 128.5, 128.3, 127.6, 126.5, 124.0, 123.1, 116.7, 100.7, 84.0, 67.0, 63.1, 62.3, 21.6, 14.3, 13.5 ppm. **HRMS** (ESI) m/z : $[M + H]^+$ calcd for $C_{28}H_{30}NO_6^+$, 476.2068; found, 476.2070.

Diethyl 2,3-diphenyl-6-(p-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (3da)



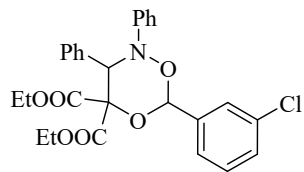
White solid, mp 79–80 °C, 40 mg, yield: 84%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.66 (d, $J = 7.9$ Hz, 2H), 7.48 (d, $J = 8.0$ Hz, 2H), 7.31 (d, $J = 7.9$ Hz, 2H), 7.20–7.13 (m, 5H), 7.00 (d, $J = 8.4$ Hz, 2H), 6.93 (t, $J = 7.3$ Hz, 1H), 6.29 (s, 1H), 5.63 (s, 1H), 4.52–4.42 (m, 2H), 4.00–3.94 (m, 2H), 2.42 (s, 3H), 1.42 (t, $J = 7.1$ Hz, 3H), 0.87 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.4, 164.8, 147.8, 139.6, 132.4, 132.3, 130.9, 129.1, 128.5, 128.2, 127.5, 126.8, 122.9, 116.6, 100.6, 83.9, 66.9, 62.9, 62.2, 21.4, 14.2, 13.4 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{28}\text{H}_{30}\text{NO}_6^+$, 476.2068; found, 476.2064.

Diethyl 6-(2-chlorophenyl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3ea)



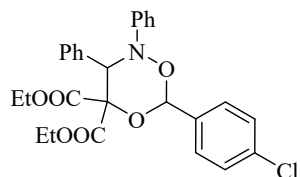
Light yellow solid, mp 139–141 °C, 33 mg, yield: 66%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (400 MHz, CDCl_3): δ 8.17 (d, $J = 7.4$ Hz, 1H), 7.54–7.42 (m, 5H), 7.23–7.19 (m, 5H), 7.05 (d, $J = 8.2$ Hz, 2H), 6.96 (t, $J = 7.3$ Hz, 1H), 6.66 (s, 1H), 5.73 (s, 1H), 4.58–4.41 (m, 2H), 4.05–3.97 (m, 2H), 1.45 (t, $J = 7.1$ Hz, 3H), 0.91 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): δ 167.3, 164.8, 147.6, 133.8, 132.8, 132.5, 131.0, 130.9, 129.7, 128.6, 128.4, 127.7, 127.3, 122.8, 116.3, 97.9, 84.1, 66.4, 63.2, 62.4, 14.3, 13.5 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{ClNO}_6^+$, 496.1521; found, 496.1520.

Diethyl 6-(3-chlorophenyl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3fa)



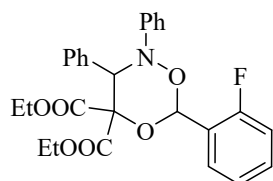
White solid, mp 134–136 °C, 39 mg, yield: 79%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.75 (s, 1H), 7.65 (d, $J = 7.3$ Hz, 1H), 7.45–7.40 (m, 4H), 7.21–7.13 (m, 5H), 6.98 (d, $J = 8.6$ Hz, 2H), 6.96 (t, $J = 7.3$ Hz, 1H), 6.29 (s, 1H), 5.61 (s, 1H), 4.52–4.43 (m, 2H), 4.02–3.93 (m, 2H), 1.43 (t, $J = 7.1$ Hz, 3H), 0.86 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.1, 164.6, 147.6, 137.1, 134.4, 132.1, 130.8, 129.8, 129.2, 128.5, 128.3, 127.6, 127.0, 125.0, 123.2, 116.7, 99.5, 83.8, 67.2, 63.1, 62.3, 14.2, 13.4 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{ClNO}_6^+$, 496.1521; found, 496.1523.

Diethyl 6-(4-chlorophenyl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3g)



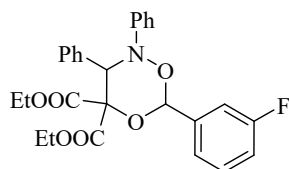
White solid, mp 137–139 °C, 41 mg, yield: 82%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.70 (d, $J = 8.4$ Hz, 2H), 7.46 (d, $J = 8.4$ Hz, 2H), 7.41 (d, $J = 7.2$ Hz, 2H), 7.20–7.17 (m, 3H), 7.14–7.11 (m, 2H), 6.97–6.93 (m, 3H), 6.29 (s, 1H), 5.60 (s, 1H), 4.50–4.44 (m, 2H), 4.00–3.94 (m, 2H), 1.41 (t, $J = 7.2$ Hz, 3H), 0.85 (t, $J = 6.6$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.2, 164.7, 147.7, 135.6, 133.8, 132.2, 130.8, 128.7, 128.6, 128.3, 128.2, 127.6, 123.2, 116.8, 99.7, 83.9, 67.2, 63.1, 62.3, 14.3, 13.4 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{ClNO}_6^+$, 496.1521; found, 496.1518.

Diethyl 6-(2-fluorophenyl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3ha)



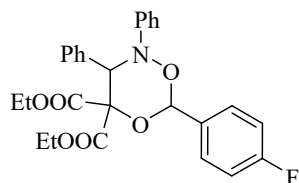
White solid, mp 121–123 °C, 31 mg, yield: 64%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 8.03 (t, $J = 7.4$ Hz, 1H), 7.51 (d, $J = 7.1$ Hz, 2H), 7.47–7.44 (m, 1H), 7.33 (t, $J = 7.5$ Hz, 1H), 7.20–7.16 (m, 5H), 7.15 (t, $J = 9.2$ Hz, 1H), 6.99 (d, $J = 8.6$ Hz, 2H), 6.93 (t, $J = 7.4$ Hz, 1H), 6.61 (s, 1H), 5.68 (s, 1H), 4.55–4.50 (m, 1H), 4.45–4.39 (m, 1H), 4.02–3.93 (m, 2H), 1.43 (t, $J = 7.1$ Hz, 3H), 0.87 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 165.9 (d, $J = 381.6$ Hz), 160.6 (d, $J = 249.5$ Hz), 147.5, 132.3, 131.6, 131.5, 130.8, 128.5, 128.3, 127.6, 124.4, 124.4, 123.0, 116.5, 95.6, 84.0, 66.8, 63.1, 62.3, 14.1, 13.4 ppm. ^{19}F NMR (564 MHz, CDCl_3) δ -112.68 ppm; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{FNO}_6^+$, 480.1817; found, 480.1816.

Diethyl 6-(3-fluorophenyl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3ia)



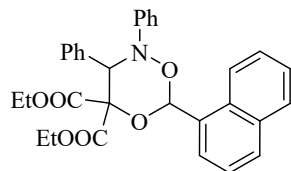
White solid, mp 101–102 °C, 36 mg, yield: 75%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.54–7.48 (m, 2H), 7.47–7.44 (m, 1H), 7.42 (d, $J = 7.8$ Hz, 2H), 7.21–7.17 (m, 3H), 7.16–7.13 (m, 3H), 6.98 (d, $J = 7.8$ Hz, 2H), 6.94 (t, $J = 7.2$ Hz, 1H), 6.31 (s, 1H), 5.62 (s, 1H), 4.52–4.43 (m, 2H), 4.02–3.93 (m, 2H), 1.42 (t, $J = 7.2$ Hz, 3H), 0.86 (t, $J = 6.6$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 165.9 (d, $J = 381.9$ Hz), 162.8 (d, $J = 244.8$ Hz), 147.7, 137.6, 137.5, 132.2, 130.8, 130.2, 130.1, 130.1, 128.6, 128.3, 127.6, 123.2, 122.6, 122.5, 116.8, 116.7, 116.6, 116.5, 114.0, 113.9, 99.6, 99.5, 83.9, 67.3, 63.1, 62.3, 14.2, 13.4 ppm. ^{19}F NMR (564 MHz, CDCl_3) δ -112.40 ppm; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{FNO}_6^+$, 480.1817; found, 280.1814.

Diethyl 6-(4-fluorophenyl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3ja)



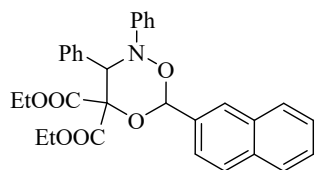
White solid, mp 98–100 °C, 38 mg, yield: 80%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.76–7.74 (m, 2H), 7.45 (d, $J = 7.1\text{Hz}$, 2H), 7.20–7.13 (m, 7H), 6.98 (d, $J = 8.6\text{ Hz}$, 2H), 6.95 (t, $J = 7.4\text{ Hz}$, 1H), 6.30 (s, 1H), 5.63 (s, 1H), 4.52–4.43 (m, 2H), 4.02–3.93 (m, 2H), 1.42 (t, $J = 7.1\text{ Hz}$, 3H), 0.87 (t, $J = 7.1\text{ Hz}$, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 165.9 (d, $J = 381.8\text{ Hz}$), 163.5 (d, $J = 246.8\text{ Hz}$), 147.7, 137.5, 132.2, 130.8, 130.2, 128.9, 128.8, 128.5, 128.3, 127.5, 123.1, 116.7, 115.5, 115.4, 99.9, 83.9, 67.0, 63.0, 62.3, 14.2, 13.4 ppm. ^{19}F NMR (564 MHz, CDCl_3) δ -112.52 ppm; **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{FNO}_6^+$, 480.1817; found, 480.1818.

Diethyl 6-(naphthalen-1-yl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3ka)

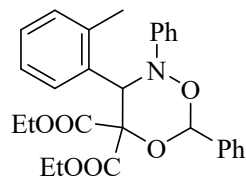


White solid, mp 122–123 °C, 42 mg, yield: 81%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 8.34–8.32 (m, 2H), 8.00 (d, $J = 8.2\text{ Hz}$, 1H), 7.94–7.92 (m, 1H), 7.68 (t, $J = 7.4\text{ Hz}$, 1H), 7.57–7.52 (m, 4H), 7.23–7.15 (m, 5H), 7.07 (d, $J = 8.6\text{ Hz}$, 2H), 7.01 (s, 1H), 6.97 (t, $J = 7.3\text{ Hz}$, 1H), 5.77 (s, 1H), 4.58–4.50 (m, 2H), 4.06–3.98 (m, 2H), 1.47 (t, $J = 7.1\text{ Hz}$, 3H), 0.91 (t, $J = 7.1\text{ Hz}$, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.5, 164.9, 147.9, 133.6, 132.3, 131.1, 130.9, 130.7, 130.2, 128.6, 128.5, 128.3, 127.6, 126.6, 125.8, 125.3, 124.3, 123.8, 122.9, 116.4, 97.9, 84.1, 67.1, 63.2, 62.3, 14.3, 13.4 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{31}\text{H}_{30}\text{NO}_6^+$, 512.2068; found, 512.2070.

Diethyl 6-(naphthalen-2-yl)-2,3-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate

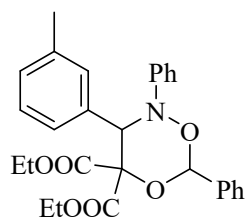
(3la)

White solid, mp 118–119 °C, 41 mg, yield: 80%. Eluent: petroleum ether/ethyl acetate = 30:1; ¹H NMR (600 MHz, CDCl₃): δ 8.30 (s, 1H), 8.00–7.97 (m, 2H), 7.92–7.91 (m, 1H), 7.88 (dd, *J* = 8.5, 1.5 Hz, 1H), 7.57–7.55 (m, 2H), 7.54 (d, *J* = 7.0 Hz, 2H), 7.24–7.15 (m, 5H), 7.06 (d, *J* = 7.7 Hz, 2H), 6.97 (t, *J* = 7.3 Hz, 1H), 6.52 (s, 1H), 5.70 (s, 1H), 4.56–4.47 (m, 2H), 4.06–3.97 (m, 2H), 1.46 (t, *J* = 7.1 Hz, 3H), 0.90 (t, *J* = 7.1 Hz, 2H) ppm; ¹³C{¹H} NMR (150 MHz, CDCl₃): δ 167.4, 164.8, 147.8, 133.9, 132.9, 132.6, 132.3, 130.9, 128.6, 128.3, 128.3, 127.7, 127.6, 126.7, 126.5, 126.3, 124.0, 123.1, 116.7, 100.5, 84.0, 67.2, 63.0, 62.3, 14.3, 13.4 ppm. **HRMS** (ESI) *m/z*: [M + H]⁺ calcd for C₃₁H₃₀NO₆⁺, 512.2068; found, 512.2065.

Diethyl 2,6-diphenyl-3-(o-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (3ab)

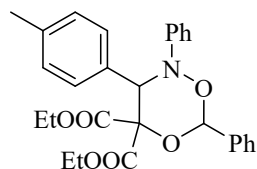
White solid, mp 99–100 °C, 30 mg, yield: 62%. Eluent: petroleum ether/ethyl acetate = 30:1; ¹H NMR (600 MHz, CDCl₃): δ 7.67 (d, *J* = 7.8 Hz, 2H), 7.48 (d, *J* = 7.8 Hz, 2H), 7.31–7.29 (m, 2H), 7.20–7.13 (m, 5H), 7.00–6.99 (m, 2H), 6.94–6.91 (t, *J* = 7.2 Hz, 1H), 6.29 (s, 1H), 5.64 (s, 1H), 4.51–4.43 (m, 2H), 4.00–3.95 (m, 2H), 2.43 (s, 3H), 1.41 (t, *J* = 6.6 Hz, 3H), 0.86 (t, *J* = 6.6 Hz, 3H) ppm. ¹³C{¹H} NMR (150 MHz, CDCl₃): δ 167.4, 164.8, 147.8, 139.6, 132.4, 132.3, 130.9, 129.1, 128.5, 128.2, 127.5, 126.8, 122.9, 116.6, 100.6, 83.9, 66.9, 62.9, 62.2, 21.4, 14.2, 13.4 ppm. **HRMS** (ESI) *m/z*: [M + H]⁺ calcd for C₂₈H₃₀NO₆⁺, 476.2068; found, 476.2066.

Diethyl 2,6-diphenyl-3-(m-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (3ac)



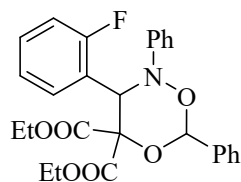
White solid, mp 102–104 °C, 39 mg, yield: 81%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.78 (d, $J = 6.8$ Hz, 2H), 7.51–7.47 (m, 3H), 7.35 (d, $J = 7.7$ Hz, 1H), 7.28 (s, 1H), 7.21 (t, $J = 7.8$ Hz, 2H), 7.06–6.99 (m, 4H), 6.94 (t, $J = 7.3$ Hz, 1H), 6.32 (s, 1H), 5.63 (s, 1H), 4.52–4.42 (m, 2H), 4.01 (q, $J = 7.1$ Hz, 2H), 2.21 (s, 3H), 1.43 (t, $J = 7.1$ Hz, 3H), 0.90 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.4, 164.8, 147.8, 136.7, 135.3, 132.2, 131.7, 129.7, 128.9, 128.5, 127.9, 127.4, 126.9, 122.9, 116.6, 100.6, 84.0, 66.8, 62.9, 62.2, 21.3, 14.2, 13.4 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{28}\text{H}_{30}\text{NO}_6^+$, 476.2068; found, 476.2068.

Diethyl 2,6-diphenyl-3-(p-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (3ad)



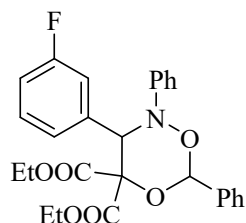
White solid, mp 131–133 °C, 42 mg, yield: 87%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.79 (d, $J = 7.7$ Hz, 2H), 7.51–7.47 (m, 3H), 7.39 (d, $J = 6.6$ Hz, 2H), 7.21 (t, $J = 7.7$ Hz, 2H), 7.03 (d, $J = 8.1$ Hz, 2H), 6.97–6.92 (m, 3H), 6.32 (s, 1H), 5.64 (s, 1H), 4.52–4.43 (m, 2H), 4.01 (q, $J = 7.1$ Hz, 2H), 2.22 (s, 3H), 1.43 (t, $J = 7.2$ Hz, 3H), 0.93 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.4, 164.8, 147.8, 137.8, 135.3, 130.7, 129.6, 129.3, 128.5, 128.4, 128.3, 126.9, 122.9, 116.5, 100.5, 84.0, 66.7, 62.9, 62.2, 21.1, 14.2, 13.5 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{28}\text{H}_{30}\text{NO}_6^+$, 476.2068; found, 476.2069.

Diethyl 3-(2-fluorophenyl)-2,6-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3ae)



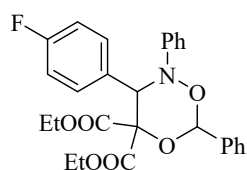
White solid, mp 100–102 °C, 32 mg, yield: 66%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 8.16–8.14 (m, 1H), 7.78–7.77 (m, 2H), 7.52–7.48 (m, 3H), 7.21–7.17 (m, 3H), 7.06–7.05 (m, 3H), 6.94 (t, $J = 7.2$ Hz, 1H), 6.72 (t, $J = 9.0$ Hz, 1H), 6.34 (s, 1H), 6.21 (s, 1H), 4.49–4.44 (m, 2H), 4.00–3.97 (m, 2H), 1.40 (t, $J = 7.2$ Hz, 3H), 0.92 (t, $J = 6.6$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 165.6 (d, $J = 378.2$ Hz), 161.1 (d, $J = 246.2$ Hz), 147.5, 135.2, 132.1, 130.1, 129.9, 129.7, 128.5, 128.4, 127.9, 127.0, 126.7, 123.7, 123.2, 123.0, 120.3, 120.2, 116.4, 114.5, 114.3, 100.4, 63.0, 62.3, 57.3, 14.2, 13.3 ppm. ^{19}F NMR (564 MHz, CDCl_3) d -115.2 ppm; **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{FNO}_6^+$, 480.1817; found, 480.1813.

Diethyl 3-(3-fluorophenyl)-2,6-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3af)



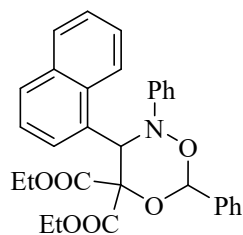
White solid, mp 103–105 °C, 39 mg, yield: 81%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.75–7.74 (m, 2H), 7.50–7.49 (m, 3H), 7.30–7.28 (m, 1H), 7.21–7.18 (m, 3H), 7.12–7.08 (m, 1H), 7.00–6.96 (m, 2H), 6.95 (t, $J = 1.8$ Hz, 1H), 6.89 (td, $J = 8.7, 1.8$ Hz, 1H), 6.30 (s, 1H), 5.62 (s, 1H), 4.51–4.44 (m, 2H), 4.01 (q, $J = 7.2$ Hz, 2H), 1.41 (t, $J = 7.2$ Hz, 3H), 0.92 (t, $J = 7.2$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 165.9 (d, $J = 368.6$ Hz), 161.9 (d, $J = 244.1$ Hz), 147.6, 135.1, 134.8, 134.7, 129.8, 128.9, 128.8, 128.7, 128.6, 128.5, 126.8, 126.7, 126.7, 123.3, 118.0, 117.8, 116.7, 116.6, 115.3, 115.2, 100.6, 83.7, 66.6, 63.1, 62.4, 14.2, 13.5 ppm. ^{19}F NMR (564 MHz, CDCl_3) δ -113.26 ppm; **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{FNO}_6^+$, 480.1817; found, 480.1816.

Diethyl 3-(4-fluorophenyl)-2,6-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3ag)



White solid, mp 117–119 °C, 41 mg, yield: 86%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.75–7.74 (m, 2H), 7.51–7.44 (m, 5H), 7.21–7.19 (m, 2H), 6.98–6.94 (m, 3H), 6.84 (t, J = 8.4 Hz, 2H), 6.31 (s, 1H), 5.61 (s, 1H), 4.51–4.44 (m, 2H), 4.02–3.97 (m, 2H), 1.41 (t, J = 7.2 Hz, 3H), 0.91 (t, J = 7.2 Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 166.0 (d, J = 371.6 Hz), 162.6 (d, J = 245.9 Hz), 147.7, 135.2, 132.6, 132.5, 129.7, 128.6, 128.5, 128.3, 128.2, 126.8, 123.3, 123.2, 116.7, 114.6, 114.5, 100.6, 100.5, 83.8, 66.5, 63.0, 62.3, 14.2, 13.5 ppm. ^{19}F NMR (564 MHz, CDCl_3) δ -113.39 ppm; HRMS (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{FNO}_6^+$, 480.1817; found, 480.1814.

Diethyl 3-(naphthalen-1-yl)-2,6-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate(3ah)

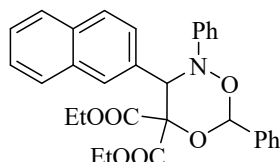


White solid, mp 123–125°C, 38 mg, yield: 74%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 8.53 (dd, J = 7.4, 1.0 Hz, 1H), 8.03 (d, J = 8.6 Hz, 1H), 7.86 (d, J = 7.0 Hz, 2H), 7.72 (d, J = 8.2 Hz, 1H), 7.70 (d, J = 7.4 Hz, 1H), 7.55–7.49 (m, 3H), 7.44 (t, J = 7.8 Hz, 1H), 7.40–7.37 (m, 1H), 7.35–7.32 (m, 1H), 7.22–7.15 (m, 1H), 7.05–7.00 (m, 3H), 6.79–6.76 (m, 1H), 6.73 (s, 1H), 6.42 (s, 1H), 4.59–4.48 (m, 2H), 3.71–3.65 (m, 1H), 3.63–3.58 (m, 1H), 1.47 (t, J = 7.14 Hz, 3H), 0.42 (t, J = 7.1 Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.8, 164.5, 147.7, 135.4, 133.1, 132.9, 130.9, 129.7, 129.2, 128.9, 128.8, 128.5, 128.4, 128.3,

127.5, 126.9, 125.8, 125.0, 124.9, 123.5, 123.1, 117.5, 116.7, 100.6, 84.2, 63.1, 62.0, 59.0, 14.3, 12.9 ppm. **HRMS** (ESI) m/z : $[M + H]^+$ calcd for $C_{31}H_{30}NO_6^+$, 512.2068; found, 512.2066.

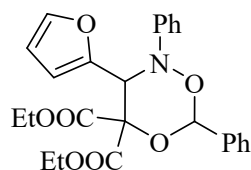
Diethyl 3-(naphthalen-2-yl)-2,6-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate

(3ai)



White solid, mp 113–115 °C, 39 mg, yield: 76%. Eluent: petroleum ether/ethyl acetate = 30:1; 1H NMR (600 MHz, $CDCl_3$): δ 8.05 (s, 1H), 7.87 (d, $J = 6.8$ Hz, 2H), 7.75–7.73 (m, 2H), 7.64 (s, 2H), 7.56–7.50 (m, 3H), 7.43–7.41 (m, 2H), 7.20 (t, $J = 8.5$ Hz, 2H), 7.07 (d, $J = 7.7$ Hz, 2H), 6.93 (t, $J = 7.3$ Hz, 1H), 6.42 (s, 1H), 5.87 (s, 1H), 4.57–4.47 (m, 2H), 3.97–3.89 (m, 2H), 1.47 (t, $J = 7.1$ Hz, 3H), 0.81 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): δ 167.4, 164.8, 147.8, 135.3, 132.9, 132.5, 130.6, 130.1, 129.8, 128.6, 128.6, 128.5, 128.3, 127.4, 127.0, 126.9, 126.1, 125.7, 123.1, 116.6, 100.7, 84.1, 67.1, 63.0, 62.3, 14.3, 13.4 ppm. **HRMS** (ESI) m/z : $[M + H]^+$ calcd for $C_{31}H_{30}NO_6^+$, 512.2068; found, 512.2067.

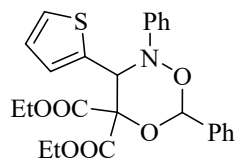
Diethyl 3-(furan-2-yl)-2,6-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (3aj)



White solid, mp 130–132 °C, 29 mg, yield: 65%. Eluent: petroleum ether/ethyl acetate = 30:1; 1H NMR (600 MHz, $CDCl_3$): δ 7.80–7.79 (m, 2H), 7.55–7.51 (m, 3H), 7.33–7.28 (m, 3H), 7.26 (s, 1H), 7.06 (t, $J = 7.2$ Hz, 2H), 6.66–6.60 (m, 1H), 6.34 (s, 1H), 5.90 (s, 1H), 4.55–4.48 (m, 2H), 4.25–4.13 (m, 2H), 1.47 (t, $J = 7.2$ Hz, 3H), 1.11 (t, $J = 7.2$ Hz, 3H) ppm; $^{13}C\{^1H\}$ NMR (150 MHz, $CDCl_3$): δ 166.6, 164.4, 147.8, 147.2, 141.8, 135.1, 129.7, 128.6, 128.4, 127.0, 123.4, 116.5, 111.8, 110.4, 100.4, 83.1, 63.0, 62.4, 61.5, 14.2, 13.8 ppm. **HRMS** (ESI) m/z : $[M + H]^+$ calcd for

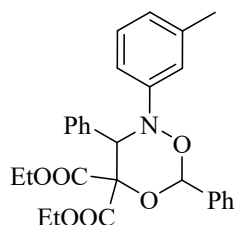
C₂₅H₂₆NO₇⁺, 452.1704; found, 452.1700.

Diethyl 2,6-diphenyl-3-(thiophen-2-yl)-1,5,2-dioxazinane-4,4-dicarboxylate (3ak)



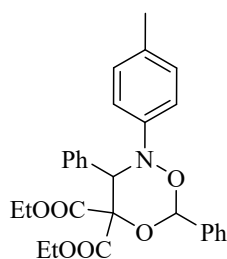
White solid, mp 139–140 °C, 36 mg, yield: 76%. Eluent: petroleum ether/ethyl acetate = 30:1; ¹H NMR (600 MHz, CDCl₃): δ 7.85 (d, *J* = 7.8 Hz, 2H), 7.53–7.50 (m, 3H), 7.25 (t, *J* = 7.6 Hz, 2H), 7.21 (d, *J* = 5.0 Hz, 1H), 7.08 (d, *J* = 8.6 Hz, 2H), 7.04 (d, *J* = 3.5 Hz, 1H), 6.99 (t, *J* = 6.7 Hz, 1H), 6.86–6.84 (m, 1H), 6.31 (s, 1H), 6.01 (s, 1H), 4.54–4.45 (m, 2H), 4.13–4.03 (m, 2H), 1.45 (t, *J* = 7.1 Hz, 3H), 1.05 (t, *J* = 7.1 Hz, 3H) ppm; ¹³C{¹H} NMR (150 MHz, CDCl₃): δ 166.8, 164.4, 147.6, 134.9, 131.9, 129.8, 129.8, 128.6, 128.4, 127.3, 127.2, 125.2, 123.2, 116.4, 100.8, 84.1, 63.8, 63.0, 62.4, 14.2, 13.6 ppm. **HRMS** (ESI) *m/z*: [M + H]⁺ calcd for C₂₅H₂₆NO₆S⁺, 468.1475; found, 468.1474.

Diethyl 3,6-diphenyl-2-(*m*-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (3al)



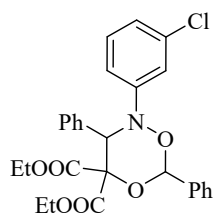
White solid, mp 114–115 °C, 36 mg, yield: 76%. Eluent: petroleum ether/ethyl acetate = 30:1; ¹H NMR (600 MHz, CDCl₃): δ 7.80 (d, *J* = 7.6 Hz, 2H), 7.54–7.50 (m, 5H), 7.22–7.17 (m, 3H), 7.11 (t, *J* = 7.8 Hz, 1H), 6.85 (s, 1H), 6.82 (d, *J* = 8.1 Hz, 1H), 6.78 (d, *J* = 7.4 Hz, 1H), 6.33 (s, 1H), 5.66 (s, 1H), 4.55–4.45 (m, 2H), 4.04–3.96 (m, 2H), 2.27 (s, 3H), 1.45 (t, *J* = 7.2 Hz, 3H), 0.90 (t, *J* = 7.1 Hz, 3H) ppm; ¹³C{¹H} NMR (150 MHz, CDCl₃): δ 167.4, 164.8, 147.7, 138.3, 135.3, 132.4, 130.9, 129.6, 128.5, 128.3, 128.2, 127.5, 126.9, 123.8, 117.2, 113.7, 100.6, 83.9, 66.9, 63.0, 62.2, 21.5, 14.2, 13.4 ppm. **HRMS** (ESI) *m/z*: [M + H]⁺ calcd for C₂₈H₃₀NO₆⁺, 476.2068; found, 476.2065.

Diethyl 3,6-diphenyl-2-(*p*-tolyl)-1,5,2-dioxazinane-4,4-dicarboxylate (**3am**)



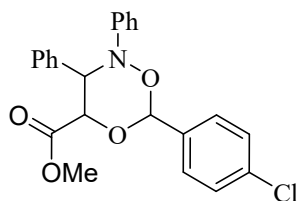
White solid, mp 117–119 °C, 43 mg, yield: 90%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.76–7.75 (m, 2H), 7.49–7.46 (m, 5H), 7.20–7.12 (m, 3H), 6.98–6.96 (m, 2H), 6.86–6.85 (m, 2H), 6.31 (s, 1H), 5.56 (s, 1H), 4.47–4.43 (m, 2H), 3.99–3.94 (m, 2H), 2.22 (s, 3H), 1.40 (t, $J = 7.2$ Hz, 3H), 0.86 (t, $J = 7.2$ Hz, 3H) ppm. $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.6, 165.0, 145.6, 135.6, 132.7, 132.6, 131.2, 129.2, 128.3, 127.7, 127.1, 117.0, 100.7, 84.1, 67.4, 63.1, 62.4, 20.8, 14.4, 13.6 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{28}\text{H}_{30}\text{NO}_6^+$, 476.2068; found, 476.2069.

Diethyl 2-(3-chlorophenyl)-3,6-diphenyl-1,5,2-dioxazinane-4,4-dicarboxylate (**3an**)



White solid, mp 139–141 °C, 37 mg, yield: 74%. Eluent: petroleum ether/ethyl acetate = 30:1; ^1H NMR (600 MHz, CDCl_3): δ 7.77 (d, $J = 8.0$ Hz, 2H), 7.52–7.48 (m, 5H), 7.21–7.16 (m, 3H), 7.13 (t, $J = 8.1$ Hz, 1H), 7.01 (t, $J = 2.0$ Hz, 1H), 6.91–6.89 (m, 2H), 6.28 (s, 1H), 5.64 (s, 1H), 4.52–4.42 (m, 2H), 4.02–3.94 (m, 2H), 1.42 (t, $J = 7.1$ Hz, 3H), 0.88 (t, $J = 7.1$ Hz, 3H) ppm; $^{13}\text{C}\{^1\text{H}\}$ NMR (150 MHz, CDCl_3): δ 167.2, 164.5, 148.9, 134.9, 134.5, 132.0, 130.8, 129.8, 129.6, 128.5, 128.4, 127.7, 126.9, 122.9, 116.2, 114.5, 100.7, 83.8, 66.6, 63.1, 62.3, 14.2, 13.4 ppm. **HRMS** (ESI) m/z : $[\text{M} + \text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{ClNO}_6^+$, 496.1521; found, 496.1522.

4. Characterization data of product 4

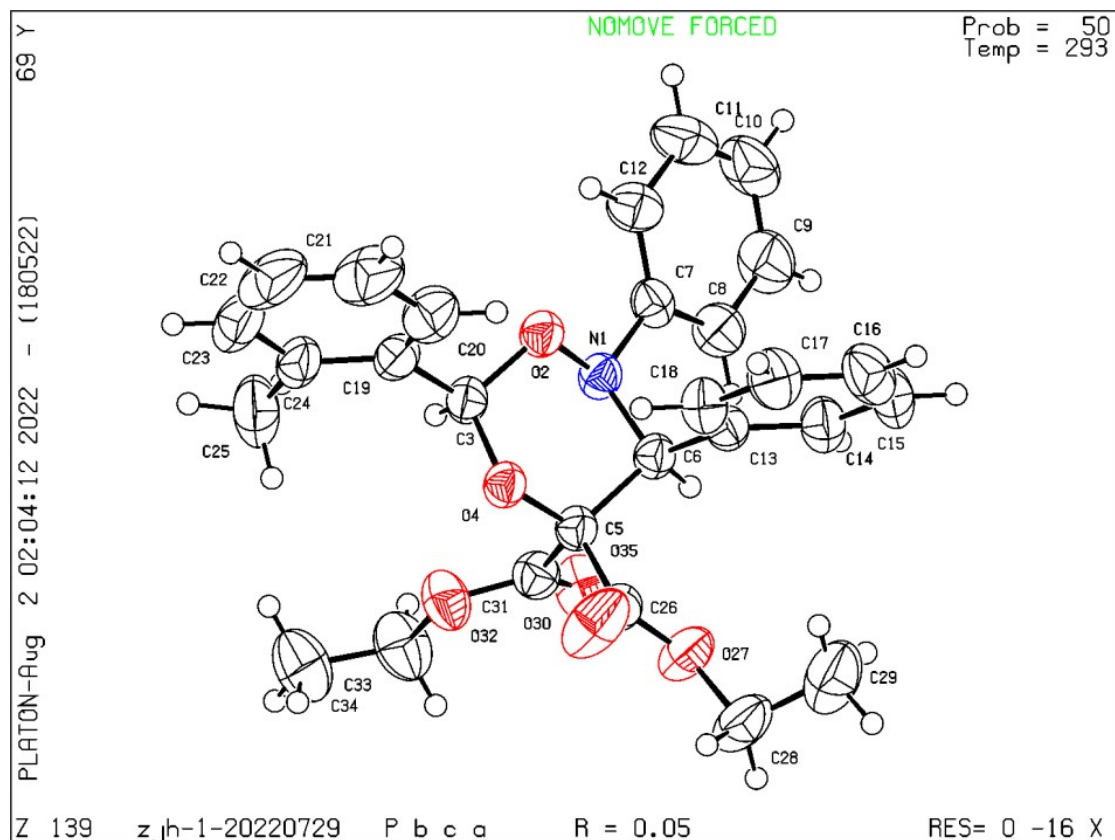


Dark brown solid, mp 117–119 °C, 36.8 mg, yield: 90%. Silica gel TLC R_f = 0.15 (EA:CH₃OH = 9:1); **¹H NMR** (600 MHz, CD₃OD) δ 7.81–7.80 (m, 2H), 7.53–7.48 (m, 5H), 7.17–7.15 (m, 2H), 7.08–7.05 (m, 3H), 6.94–6.93 (m, 2H), 6.90–6.87 (m, 1H), 6.18 (s, 1H), 5.72 (s, 1H), 3.92 (s, 1H) ppm; **¹³C{¹H} NMR** (150 MHz, CD₃OD) δ 172.2, 150.0, 136.5, 136.4, 135.4, 132.6, 130.1, 129.7, 129.5, 128.7, 128.2, 123.7, 117.8, 101.3, 87.5, 68.9 ppm. **HRMS** (ESI) m/z : [M + H]⁺ calcd for C₂₃H₂₁ClNO₄⁺, 410.1154; found, 410.1151.

5. References

- [1] L. Zheng, F. Gao, C. Yang, G.-L. Gao, Y. Zhao, Y. Gao, W. Xia, *Org. Lett.* **2017**, *19*, 5086–5089.
- [2] G. V. Kryshstal, G. M. Zhdankina, S. G. Zlotin, *Mendeleev Commun.* **2013**, *1*, 24–25.

6. Crystal structure of compound 3ba (CCDC 2203061)

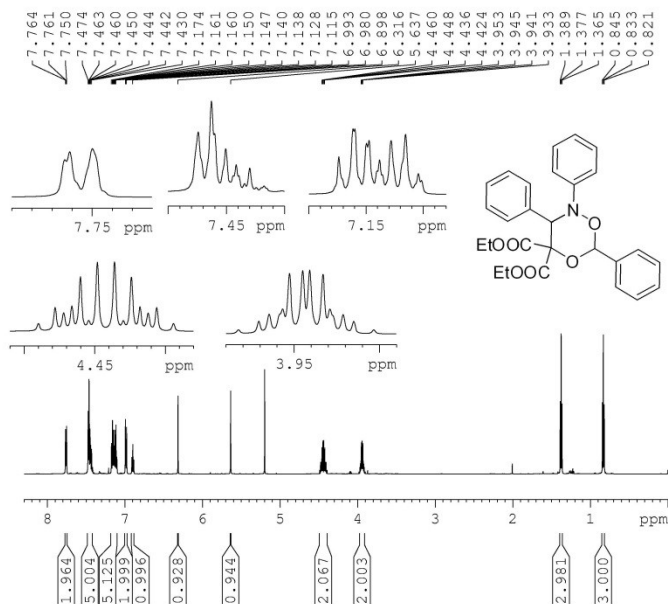


CCDC number	2203061
Identification code	3ba
Empirical formula	C ₂₈ H ₂₉ NO ₆
Formula weight	475.52
Temperature/K	293
Crystal system	orthorhombic
Space group	Pbca
a/Å	9.66800(10)
b/Å	16.9490(2)
c/Å	30.1249(4)
α/°	90
β/°	90

$\gamma/^\circ$	90
Volume/ \AA^3	4936.36(10)
Z	8
$\rho_{\text{calc}}/\text{g/cm}^3$	1.280
μ/mm^{-1}	0.735
F(000)	2016.0
Crystal size/ mm^3	$0.13 \times 0.07 \times 0.06$
Radiation	CuK α ($\lambda = 1.54184$)
2Θ range for data collection/ $^\circ$	10.438 to 143.05
Index ranges	$-11 \leq h \leq 11, -20 \leq k \leq 20, -35 \leq l \leq 37$
Reflections collected	37505
Independent reflections	4780 [$R_{\text{int}} = 0.0245, R_{\text{sigma}} = 0.0127$]
Data/restraints/parameters	4780/0/319
Goodness-of-fit on F^2	1.055
Final R indexes [$I \geq 2\sigma(I)$]	$R_1 = 0.0484, wR_2 = 0.1281$
Final R indexes [all data]	$R_1 = 0.0542, wR_2 = 0.1331$
Largest diff. peak/hole / $e \text{\AA}^{-3}$	0.25/-0.19

7. ¹H-, ¹³C- and ¹⁹F-NMR spectra of products 3

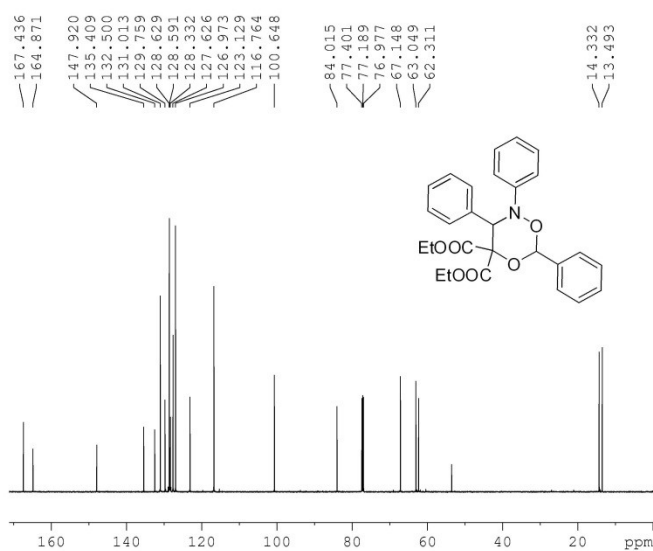
3aa



```

NAME          YXZ
EXPNO         651
PROCNO        1
Date_         20180118
Time          10.47
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12019.230 Hz
FIDRES        0.183399 Hz
AQ            2.7263477 sec
RG            17.26
DW            41.600 usec
DE            6.50 usec
TE            298.0 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
SF01          600.1337060 MHz
NUC1          1H
P1            9.90 usec
SI            65536
SF            600.1300437 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

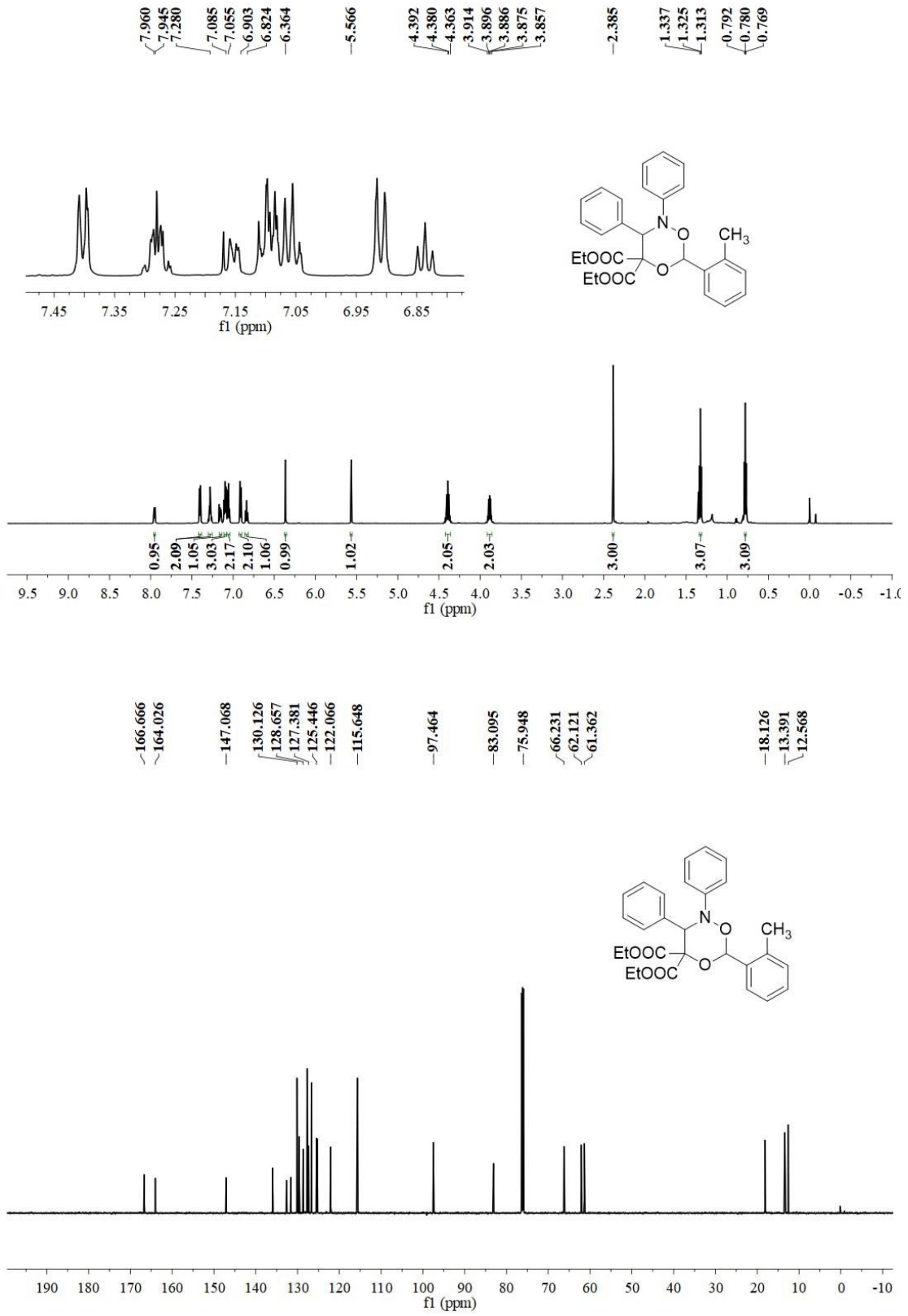


```

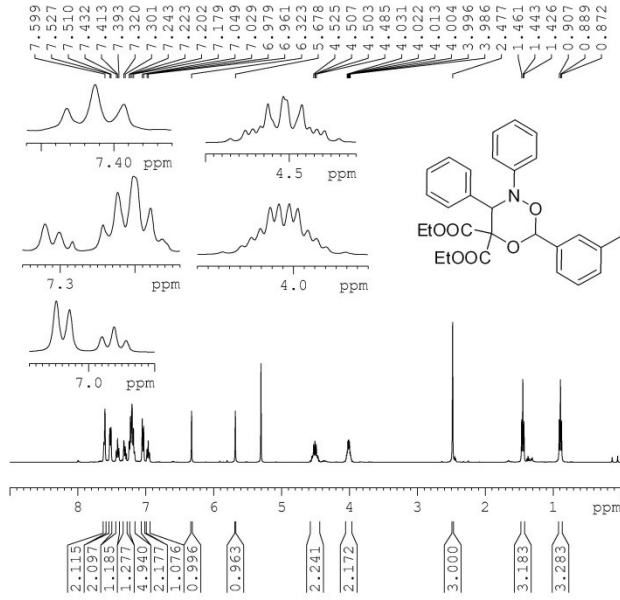
NAME          YXZ
EXPNO         652
PROCNO        1
Date_         20180118
Time          11.01
INSTRUM       spect
PROBHD        5 mm PABBO BB/
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            256
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG            194.06
DW            13.867 usec
DE            6.50 usec
TE            298.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
SF01          150.9178981 MHz
NUC1          13C
P1            11.50 usec
SI            32768
SF            150.9028085 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

3ba



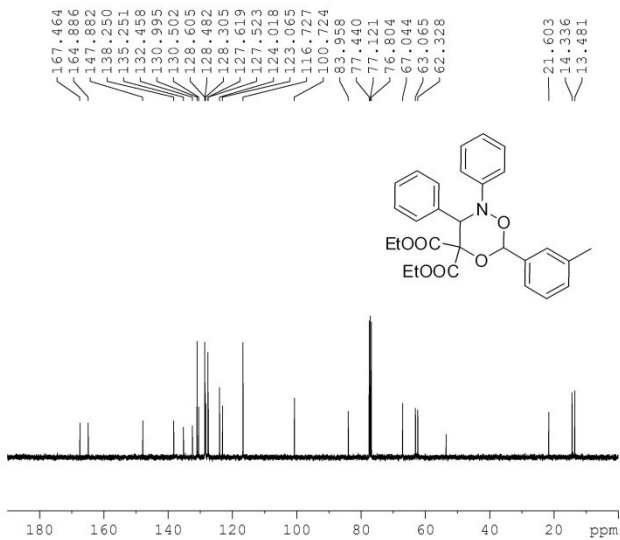
3ca



```

NAME          19
EXPNO         1
PROCNO        1
Date_         20180324
Time          8.07
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            45.2
DW            60.800 usec
DE            6.50 usec
TE            289.5 K
D1            1.00000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
F1            14.00 usec
PL1           -3.13 dB
PL1W          20.50172997 W
SF01          400.1324710 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
    
```



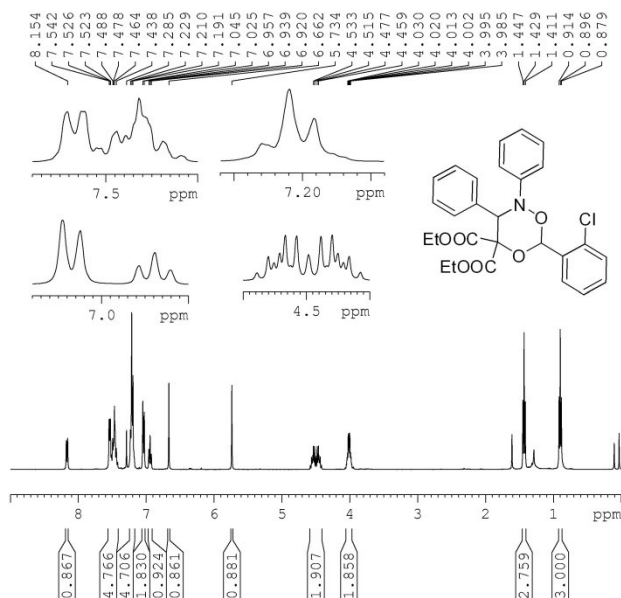
```

NAME          19
EXPNO         2
PROCNO        1
Date_         20180324
Time          8.12
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zpgg30
TD            65536
SOLVENT       CDCl3
NS            64
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            32800
DW            20.800 usec
DE            6.50 usec
TE            291.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
F1            10.00 usec
PL1           -2.04 dB
PL1W          55.04534149 W
SF01          100.6238364 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
PCPD2         80.00 usec
    
```

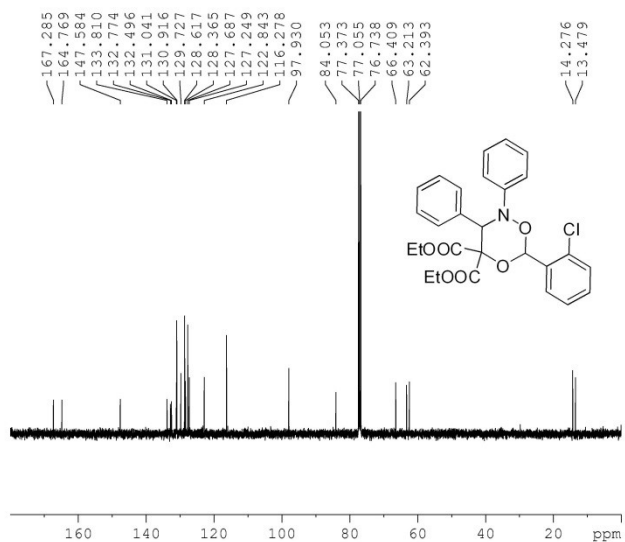

3ea



```

NAME          20
EXPNO         1
PROCNO        1
Date_         20180324
Time          8.16
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            8
DS            0
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            90.5
DW            60.800 usec
DE            6.50 usec
TE            289.9 K
D1            1.0000000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          1H
P1            14.00 usec
PL1           -3.13 dB
PL1W          20.50172997 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
    
```



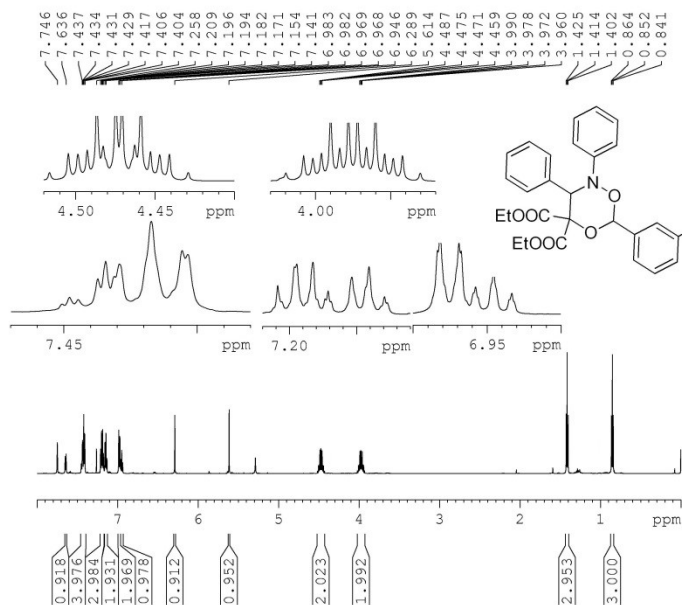
```

NAME          20
EXPNO         2
PROCNO        1
Date_         20180324
Time          8.26
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            160
DS            4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG            32800
DW            20.800 usec
DE            6.50 usec
TE            291.4 K
D1            2.0000000 sec
D11           0.0300000 sec
TD0           1

===== CHANNEL f1 =====
NUC1          13C
P1            10.00 usec
PL1           -2.04 dB
PL1W          55.04534149 W
SFO1          100.6238364 MHz

===== CHANNEL f2 =====
CPDPRG2       waltz16
NUC2          1H
FCPD2         80.00 usec
    
```

3fa

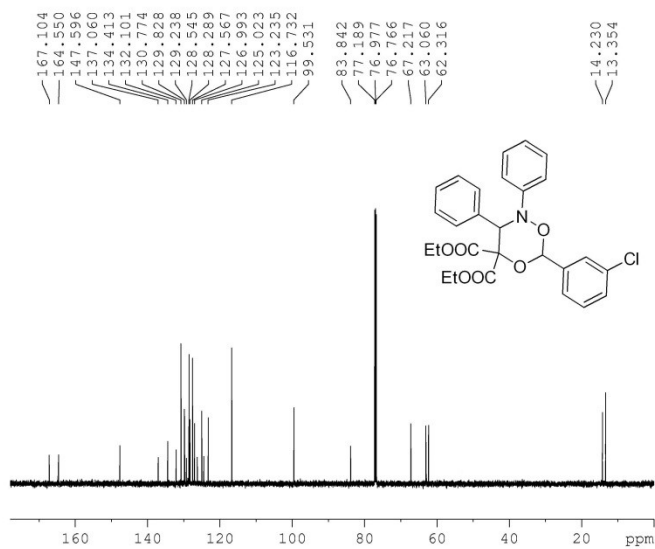


```

NAME                HHG
EXPNO                2018011971
PROCNO               1
Date_                20180119
Time                 9.42
INSTRUM              spect
PROBHD               5 mm PABBO BB-
PULPROG              zg30
TD                   65536
SOLVENT              CDCl3
NS                    16
DS                     2
SWH                  12335.526 Hz
FIDRES               0.188225 Hz
AQ                   2.6564426 sec
RG                     40.3
DW                   40.533 usec
DE                     6.50 usec
TE                   291.4 K
D1                    1.00000000 sec
    
```

```

===== CHANNEL f1 =====
NUC1                  1H
P1                     9.88 usec
SI                    65536
SF                    600.1800153 MHz
WDW                     EM
SSB                      0
LB                      0.30 Hz
GB                      0
PC                       1.00
    
```



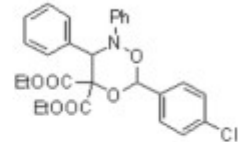
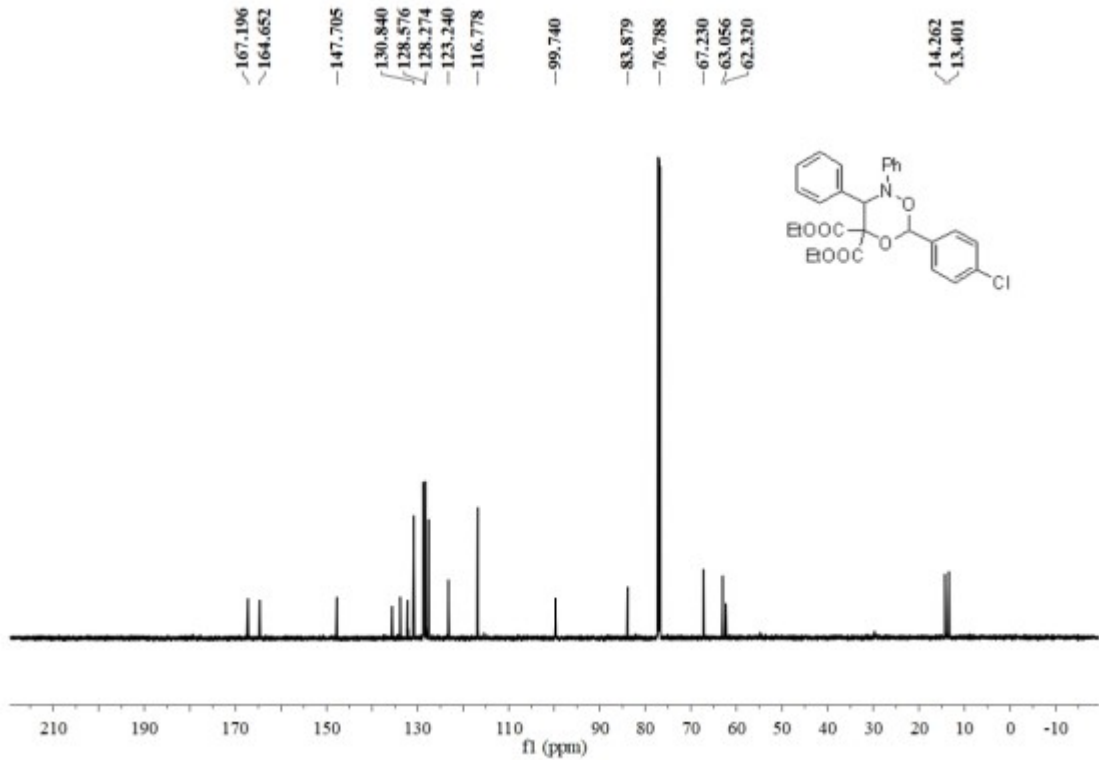
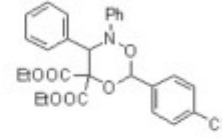
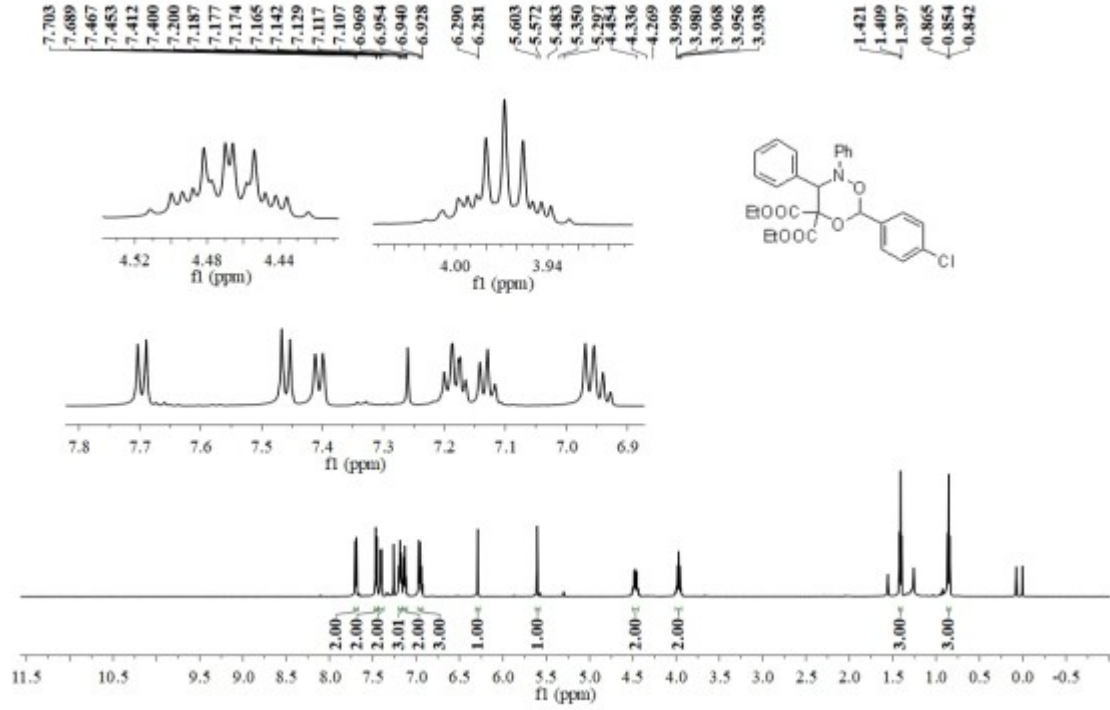
```

NAME                YXZ
EXPNO                18011971
PROCNO               1
Date_                20180119
Time                 9.45
INSTRUM              spect
PROBHD               5 mm PABBO BB-
PULPROG              zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     57
DS                      4
SWH                  36057.691 Hz
FIDRES               0.550197 Hz
AQ                   0.9088159 sec
RG                     2050
DW                   13.867 usec
DE                     6.50 usec
TE                   291.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
    
```

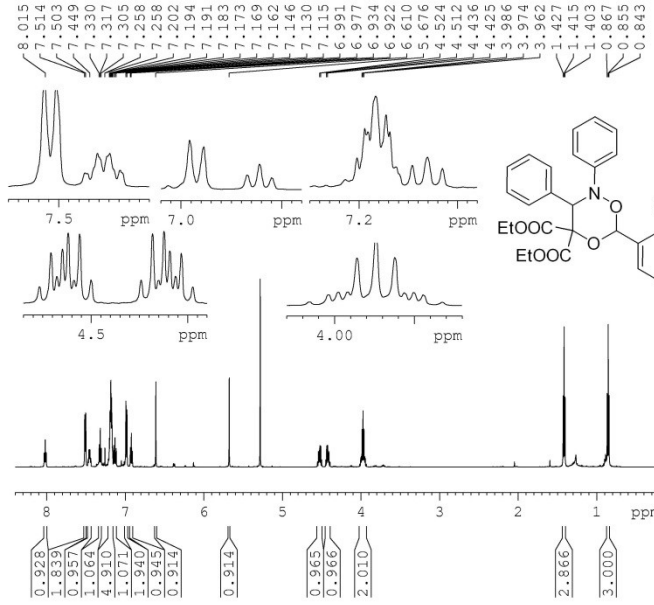
```

===== CHANNEL f1 =====
NUC1                  13C
P1                    15.64 usec
SI                    32768
SF                    150.9153956 MHz
WDW                     EM
SSB                      0
LB                      1.00 Hz
GB                      0
PC                       1.40
    
```


3ga



3ha

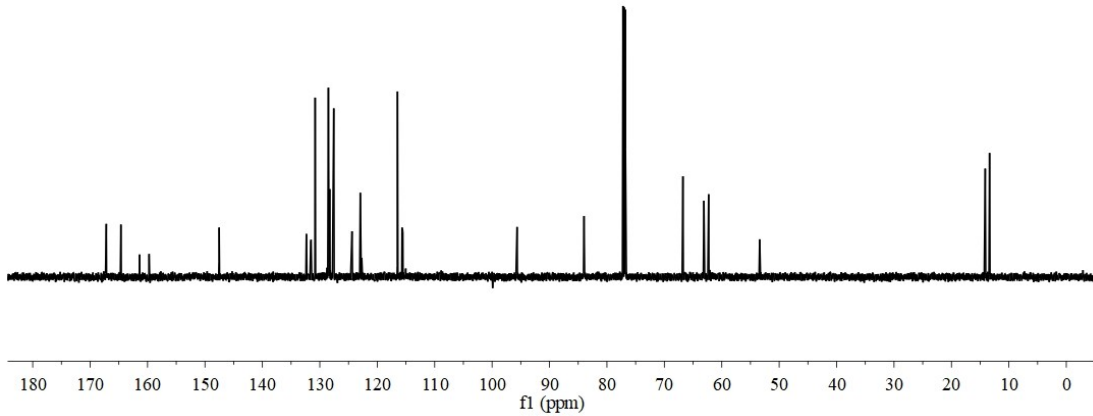
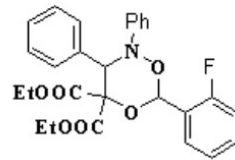


```

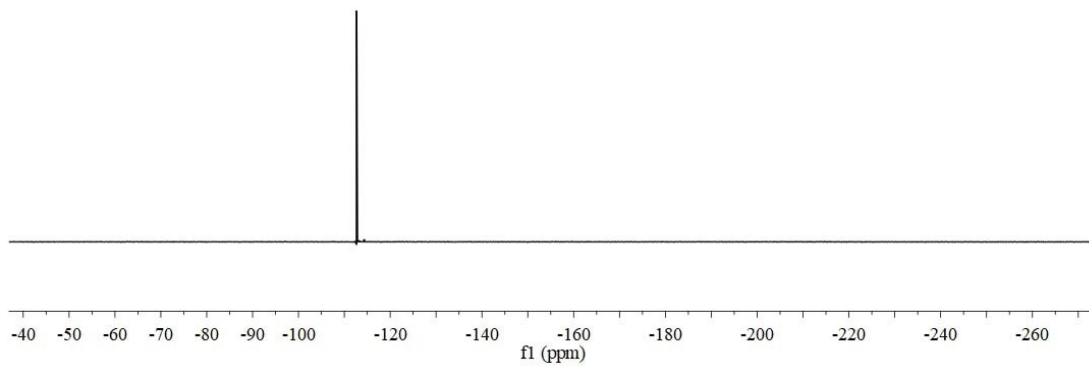
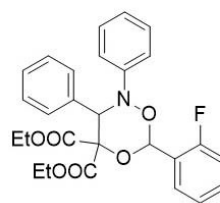
NAME          HHG
EXPNO         2018011972
PROCNO        1
Date_         20180119
Time          9.53
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            40.3
DW            40.533 usec
DE            6.50 usec
TE            291.6 K
D1            1.00000000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

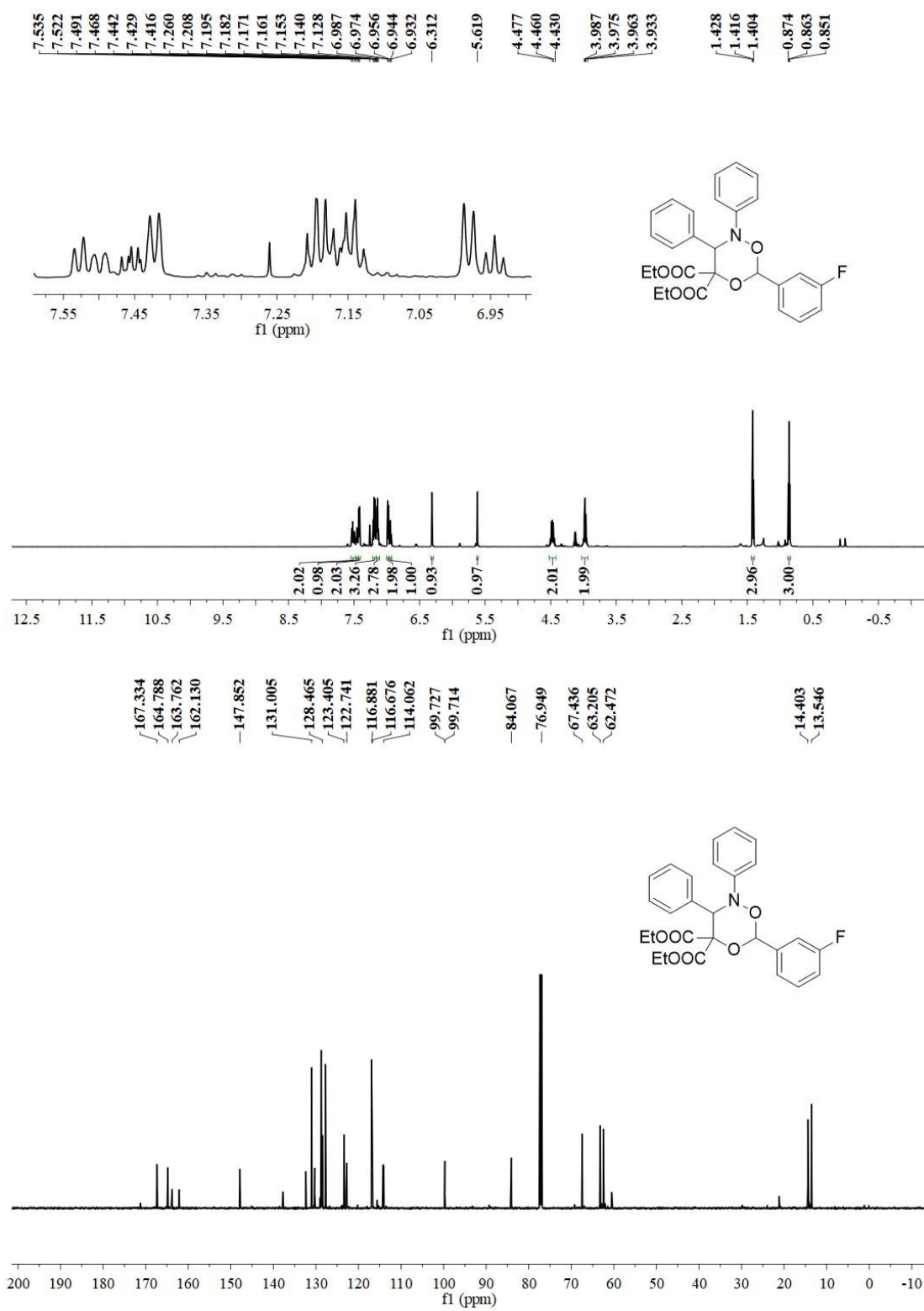
167.183
164.639
161.395
159.732
147.529
128.506
128.267
124.388
122.952
122.614
116.475
115.680
115.542
95.680
95.646
83.990
77.202
76.779
66.761
63.110
62.287
53.396
14.115
13.361



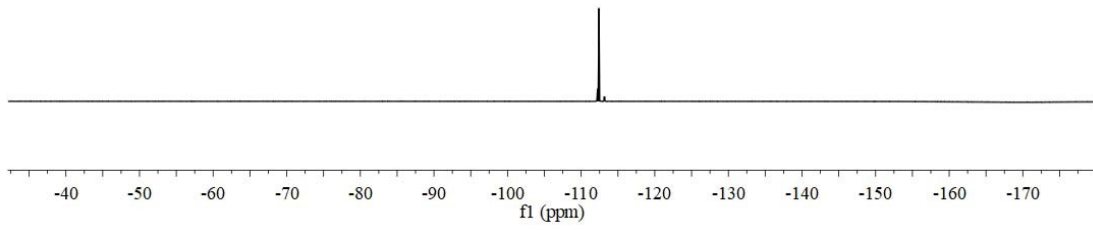
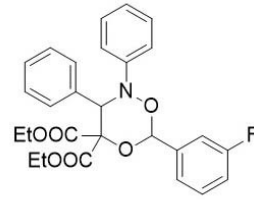
--112.679



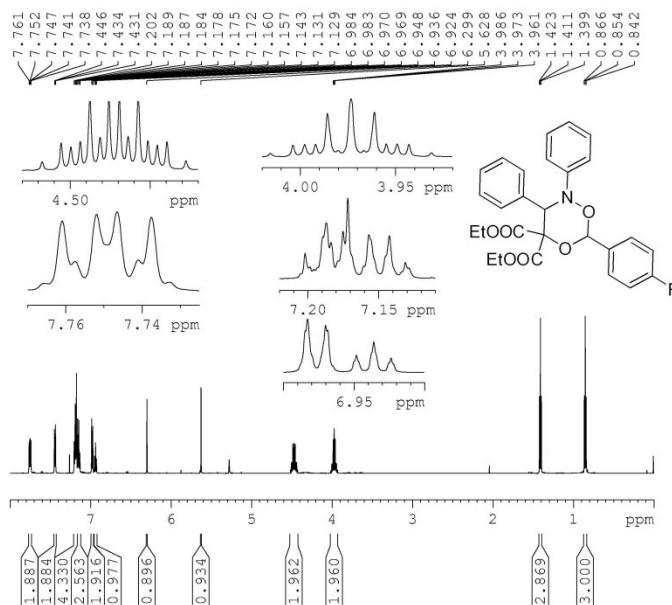
3ia



-112.403



3ja

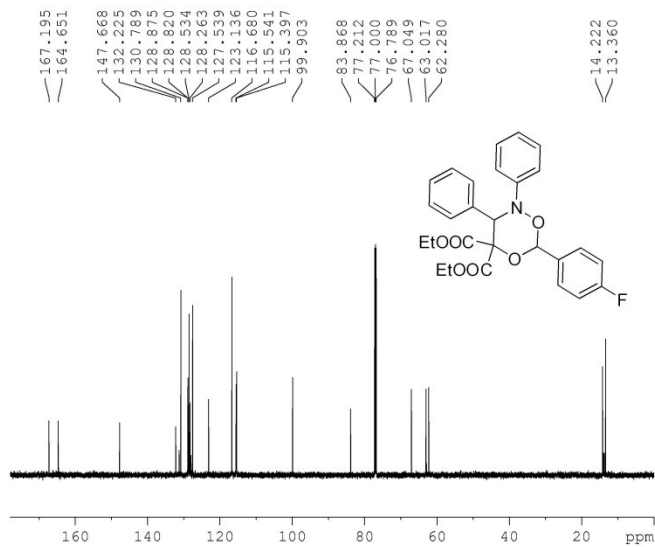


```

NAME          HHG
EXPNO         2018011973
PROCNO        1
Date_         20180119
Time          10.01
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            40.3
DW            40.533 usec
DE            6.50 usec
TE            291.6 K
D1            1.00000000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



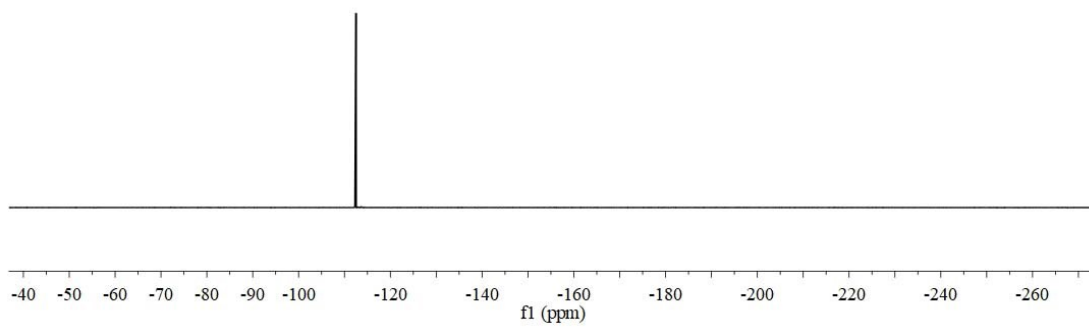
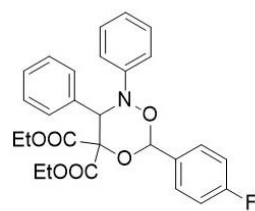
```

NAME          YXZ
EXPNO         18011973
PROCNO        1
Date_         20180119
Time          10.04
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            36
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG            2050
DW            13.867 usec
DE            6.50 usec
TE            291.8 K
D1            2.00000000 sec
D11           0.03000000 sec
  
```

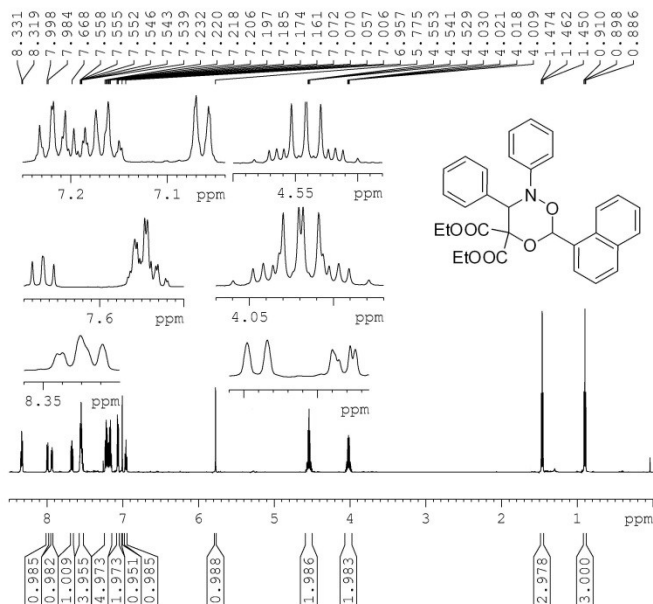
```

===== CHANNEL f1 =====
NUC1          13C
P1            15.64 usec
SI            32768
SF            150.9153956 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

-112.520



3ka



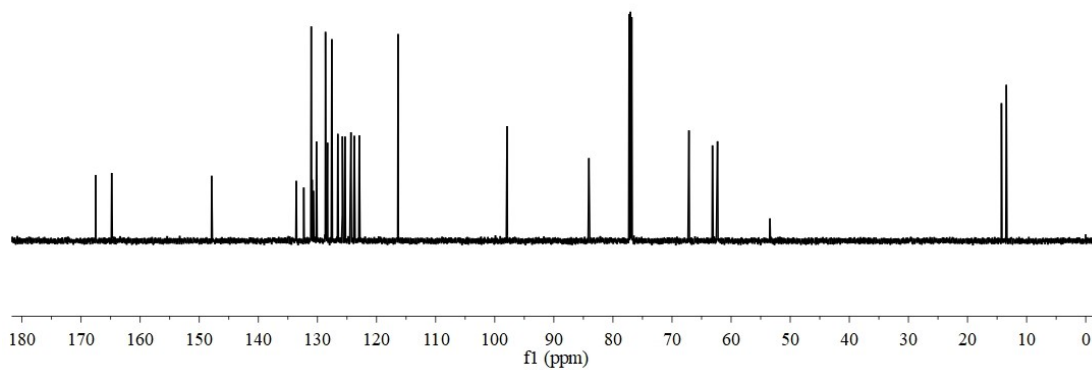
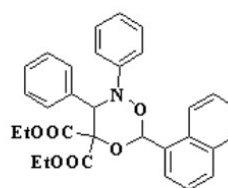
```

NAME          HHG
EXPNO         2018011975
PROCNO        1
Date_         20180119
Time          10.19
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            28.5
DW            40.533 usec
DE            6.50 usec
TE            291.6 K
D1            1.00000000 sec
    
```

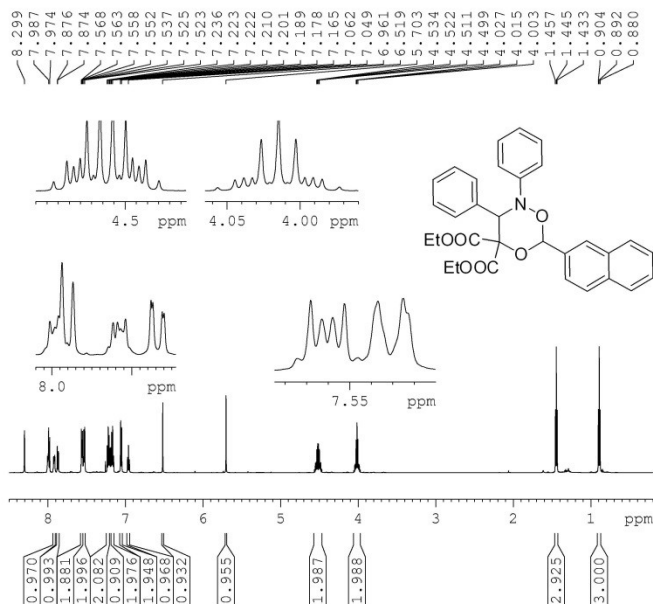
```

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

¹³C NMR chemical shifts (ppm): 167.518, 164.814, 147.897, 133.583, 128.639, 127.566, 125.328, 122.921, 116.351, 97.908, 84.068, 77.035, 76.823, 67.133, 63.155, 62.302, 14.256, 13.431.



3la

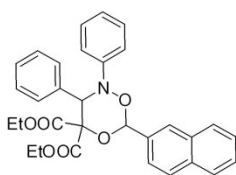


```

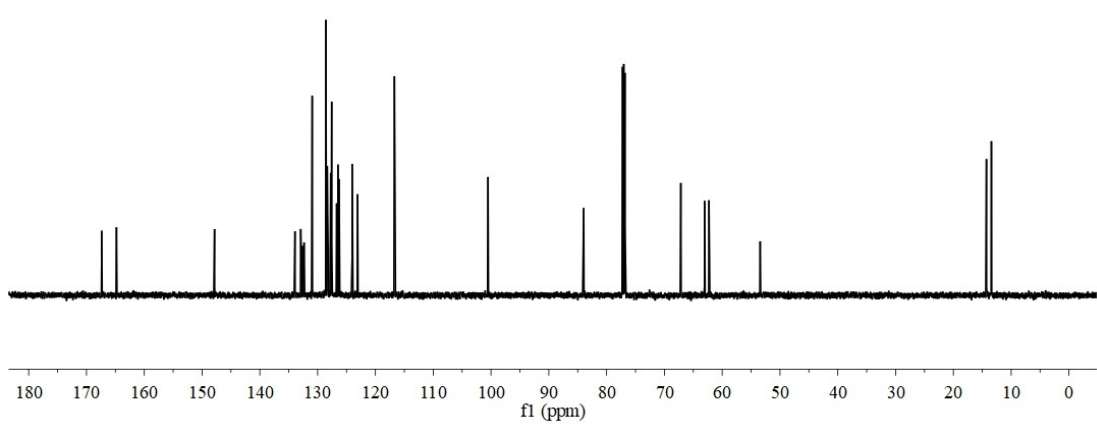
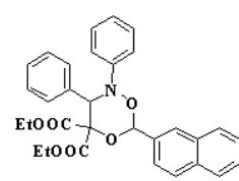
NAME          HHG
EXPNO         2018011976
PROCNO        1
Date_         20180119
Time          10.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            28.5
DW            40.533 usec
DE            6.50 usec
TE            291.6 K
D1            1.0000000 sec
    
```

```

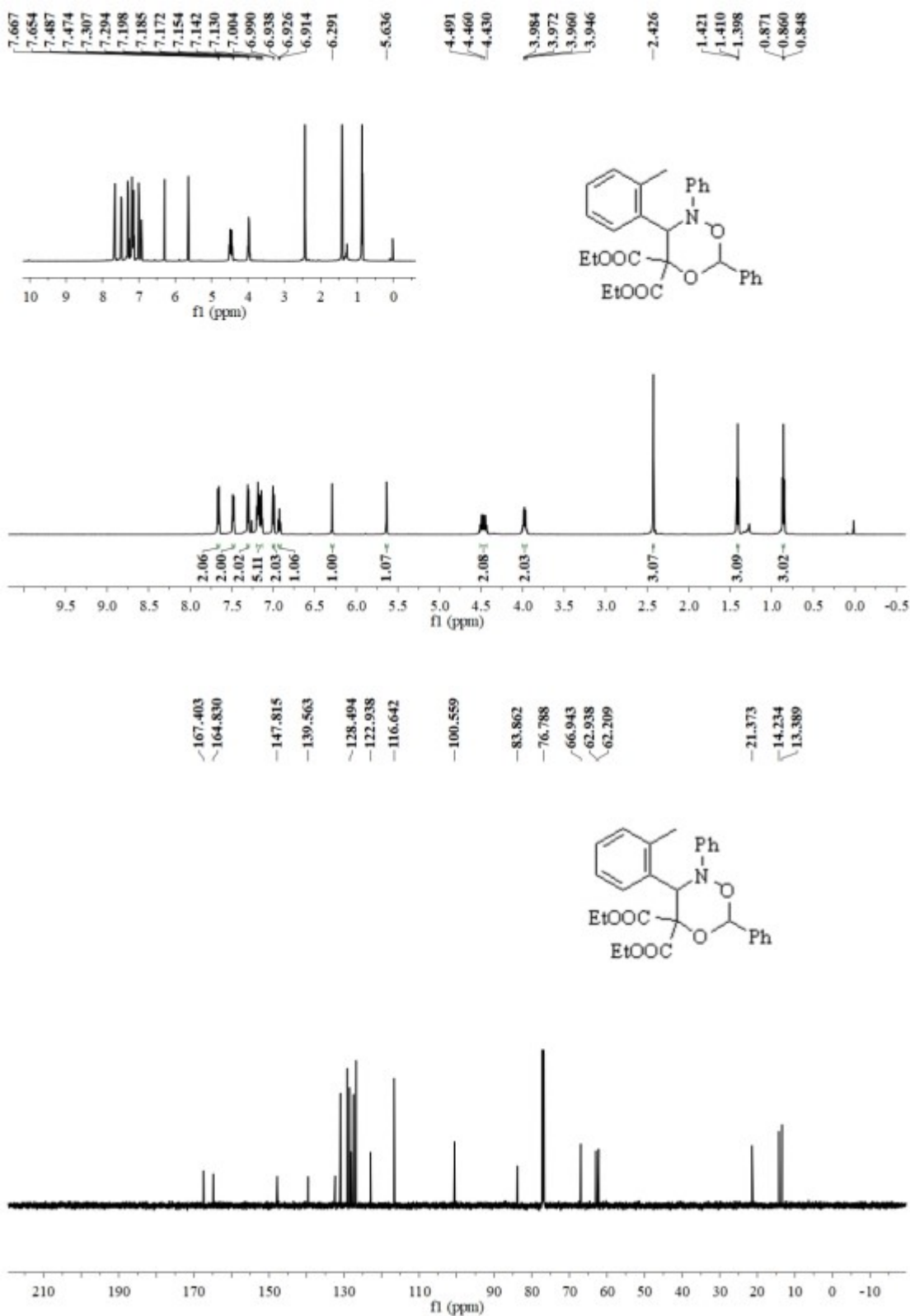
===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



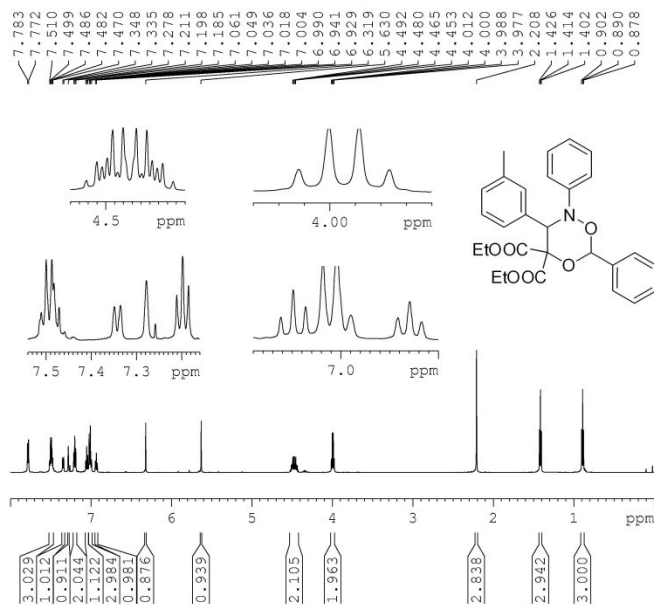
~167.376
 ~164.810
 -147.835
 132.639
 128.570
 127.738
 126.478
 -123.089
 -116.719
 -100.520
 -83.961
 77.034
 76.822
 -67.157
 63.044
 62.292
 -53.431
 14.277
 13.420



3ab



3ac

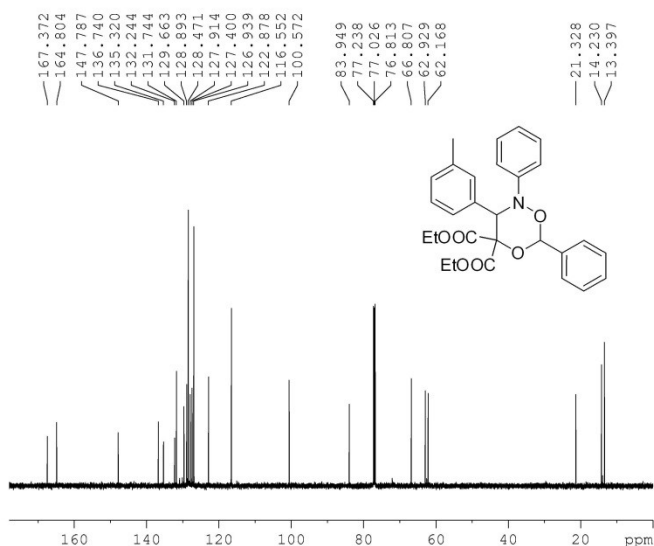


```

NAME          HHG
EXPNO         2018011978
PROCNO        1
Date_         20180119
Time          10.51
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            28.5
DW            40.533 usec
DE            6.50 usec
TE            291.7 K
D1            1.00000000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



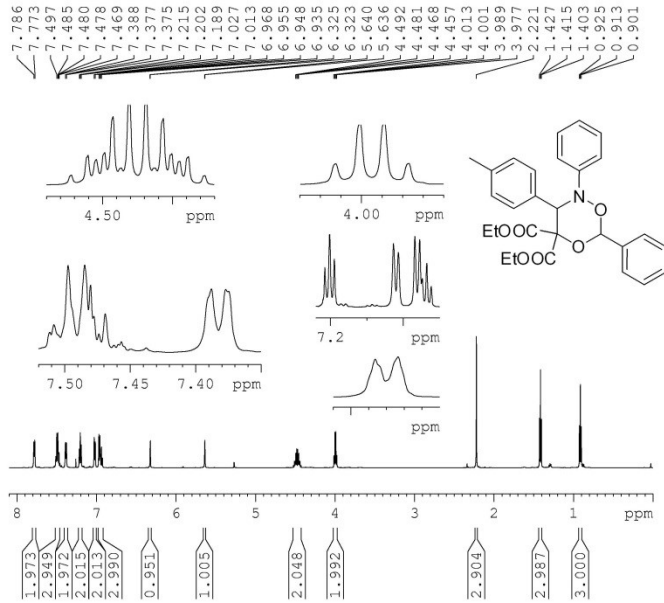
```

NAME          YXZ
EXPNO         18011978
PROCNO        1
Date_         20180119
Time          10.54
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            38
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG            2050
DW            13.867 usec
DE            6.50 usec
TE            292.1 K
D1            2.00000000 sec
D11           0.03000000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            15.64 usec
SI            32768
SF            150.9153956 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```

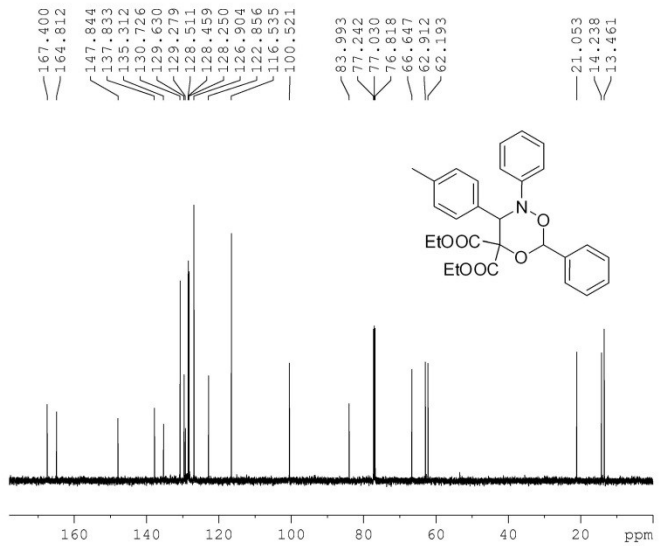
3ad



```

NAME          HHG
EXPNO         2018011979
PROCNO        1
Date_         20180119
Time          11.00
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            28.5
DW            40.533 usec
DE            6.50 usec
TE            291.7 K
D1            1.00000000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

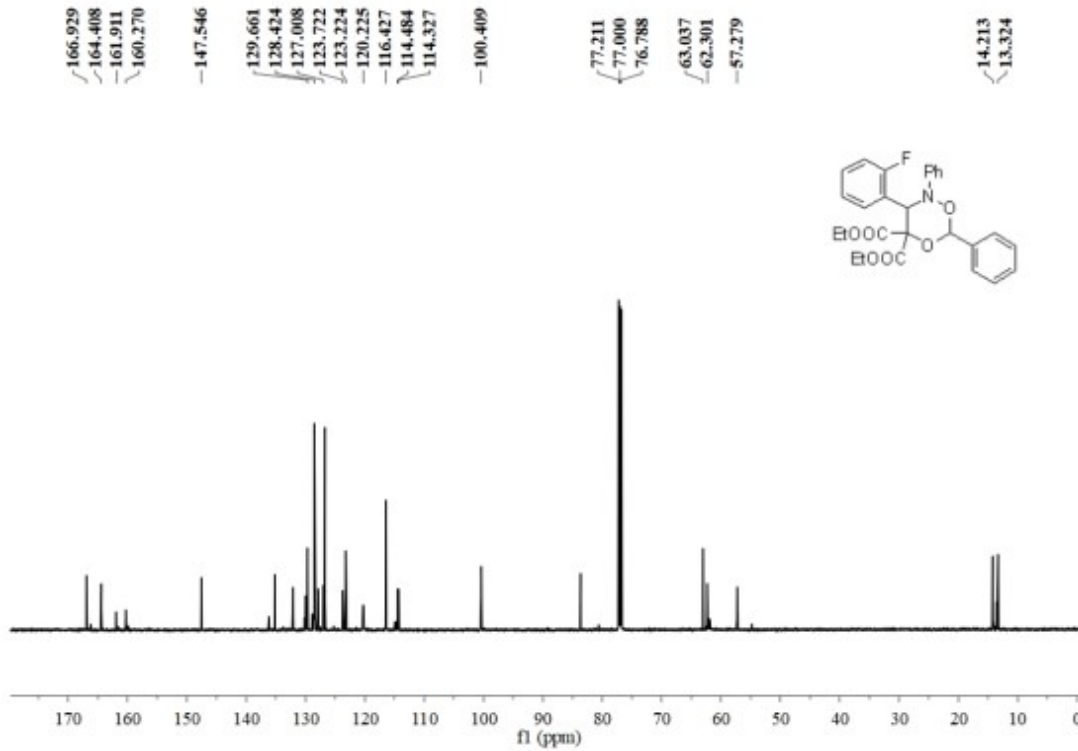
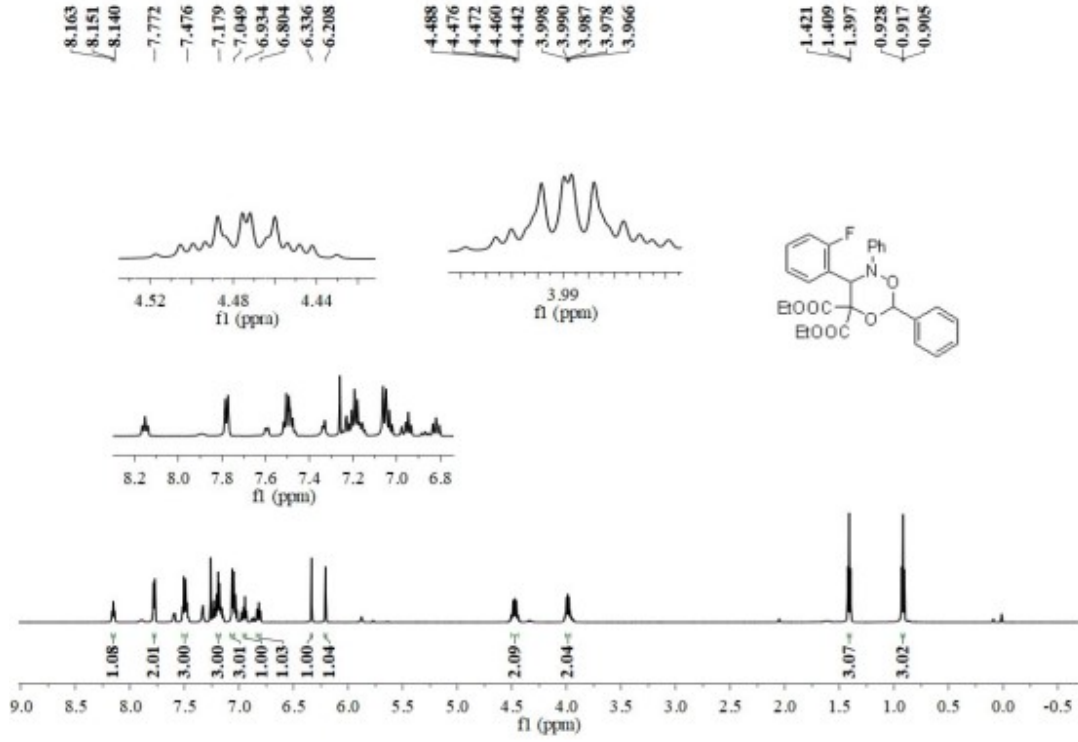


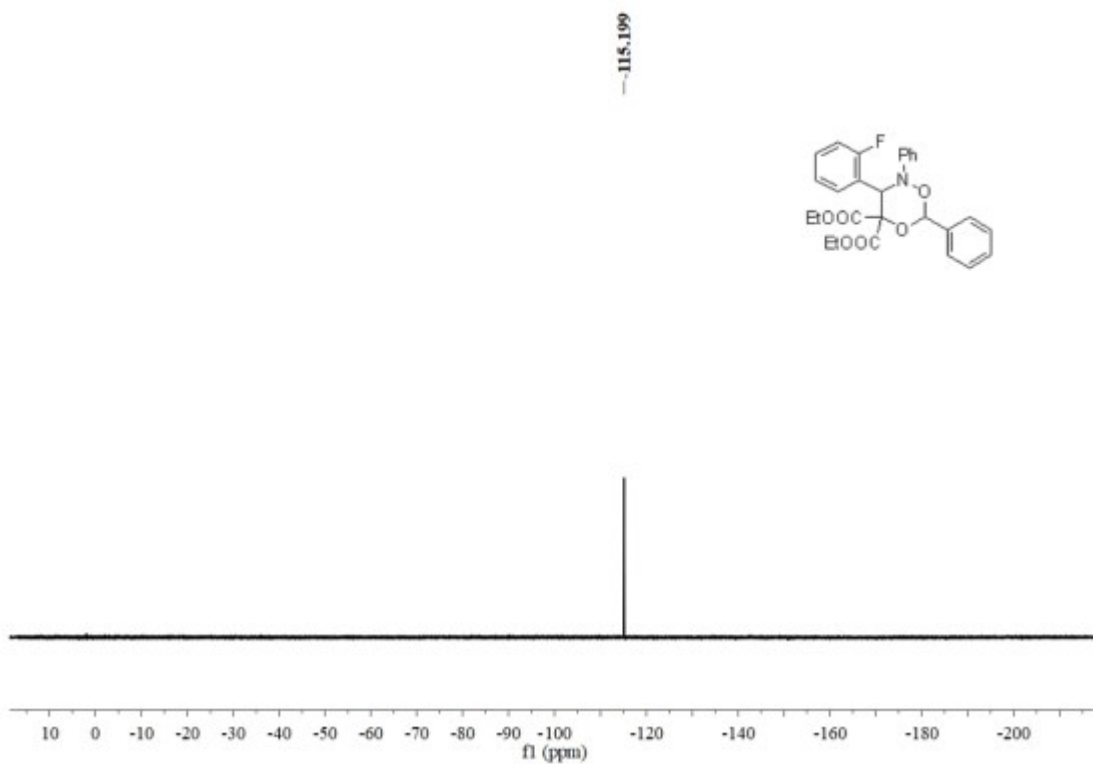
```

NAME          YXZ
EXPNO         18011979
PROCNO        1
Date_         20180119
Time          11.02
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            26
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG            2050
DW            13.867 usec
DE            6.50 usec
TE            291.9 K
D1            2.00000000 sec
D11           0.03000000 sec

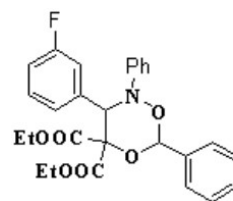
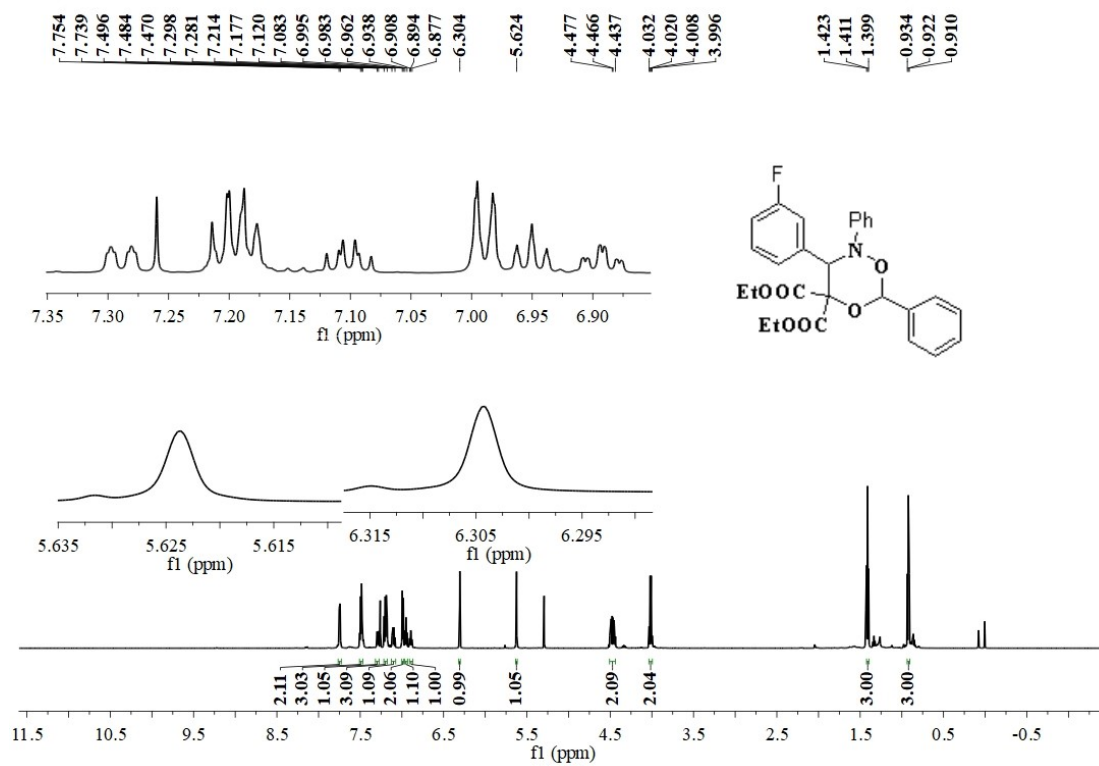
===== CHANNEL f1 =====
NUC1          13C
P1            15.64 usec
SI            32768
SF            150.9153956 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

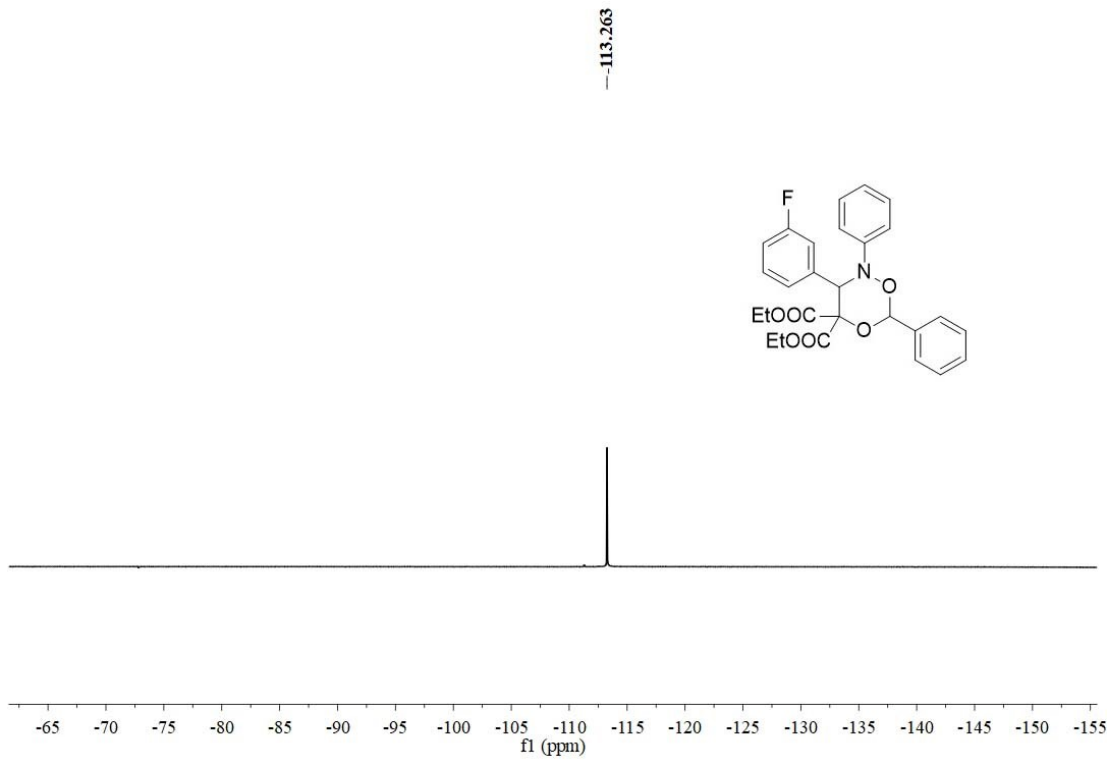
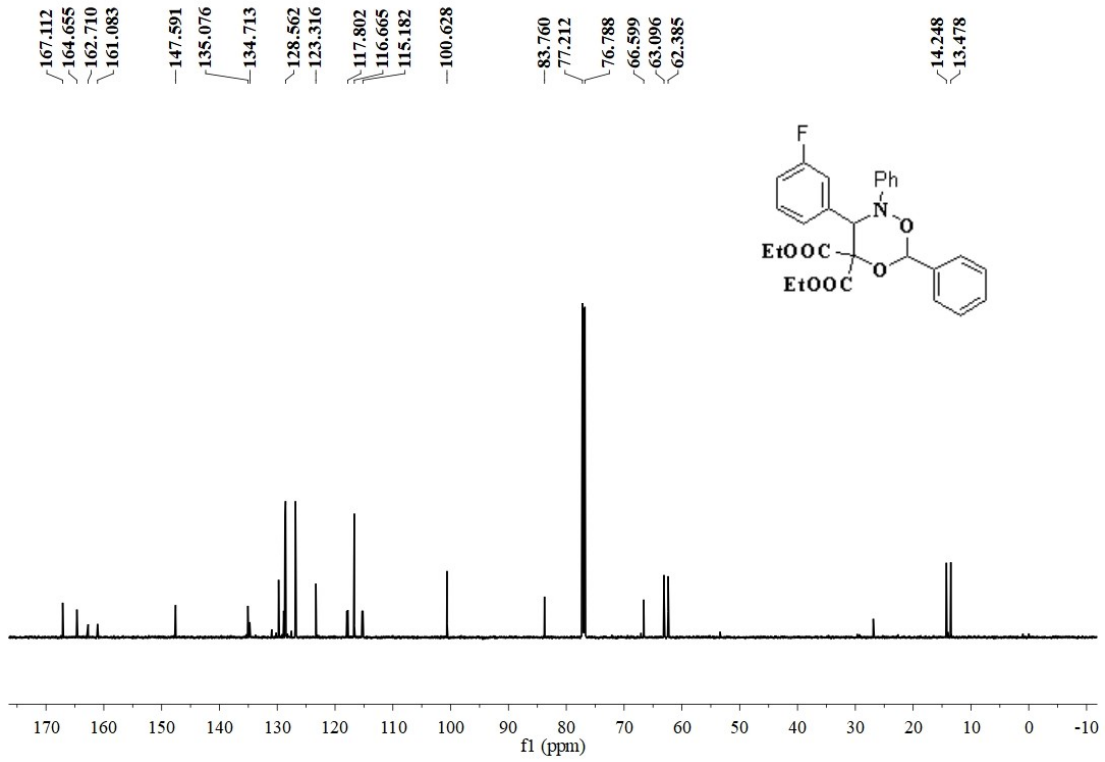
3ae



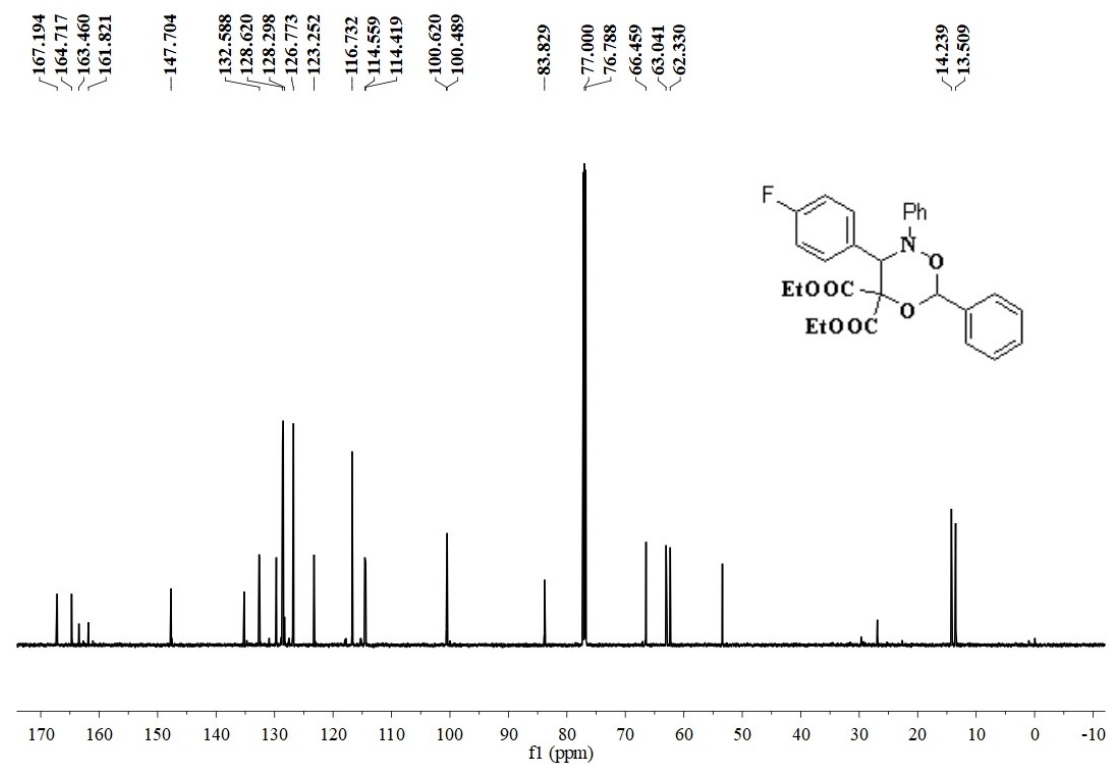
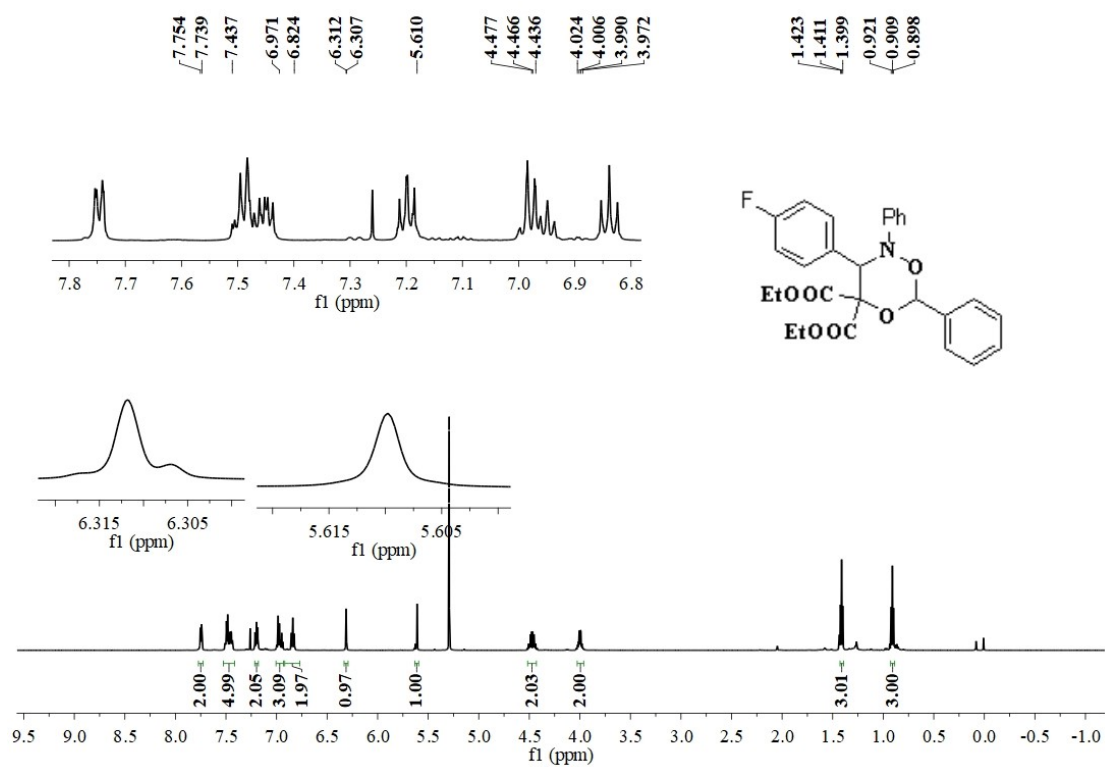


3af

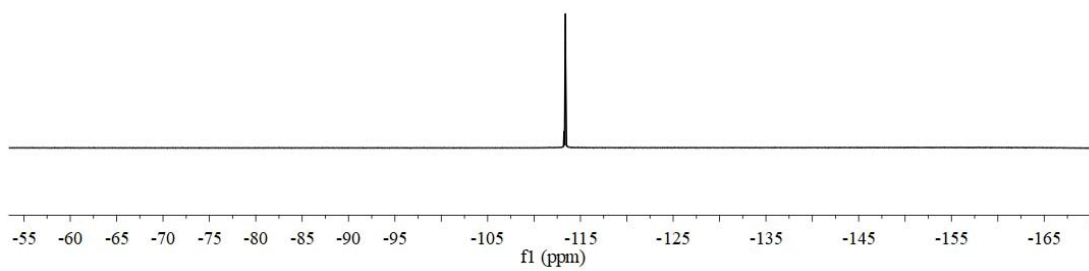
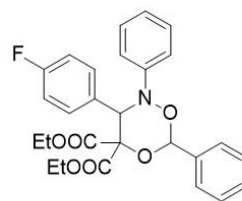




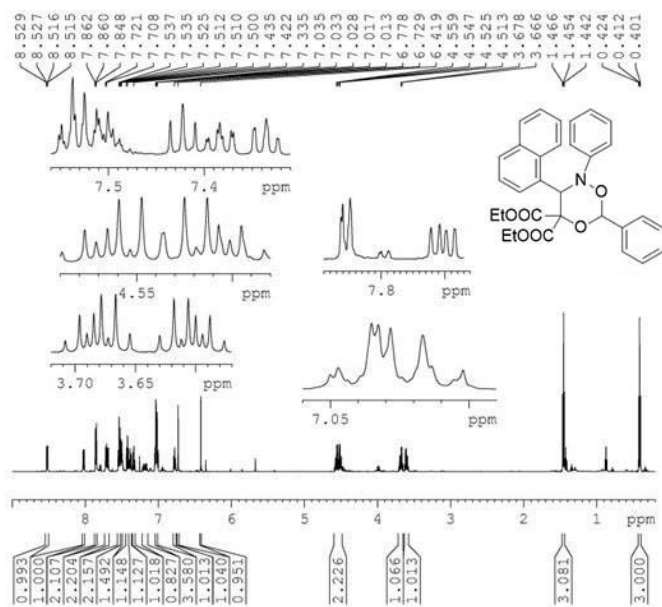
3ag



-113.385



3ah



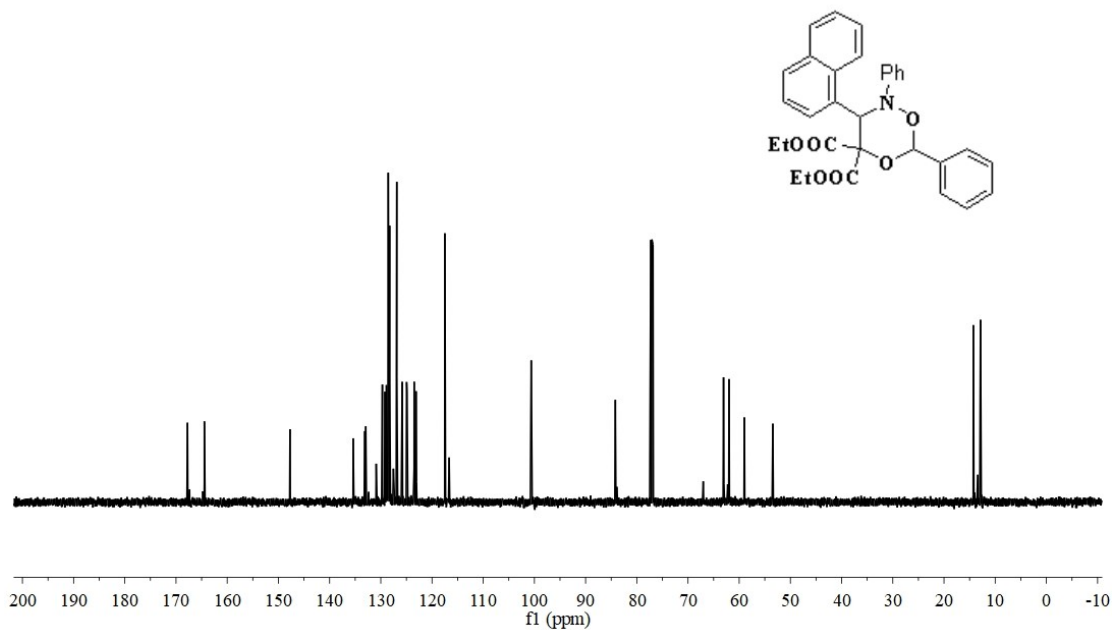
```

NAME          HHG
EXPNO         2018011983
PROCNO        1
Date_         20180119
Time          18.54
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            28.5
DW            40.533 usec
DE            6.50 usec
TE            292.3 K
D1            1.00000000 sec
    
```

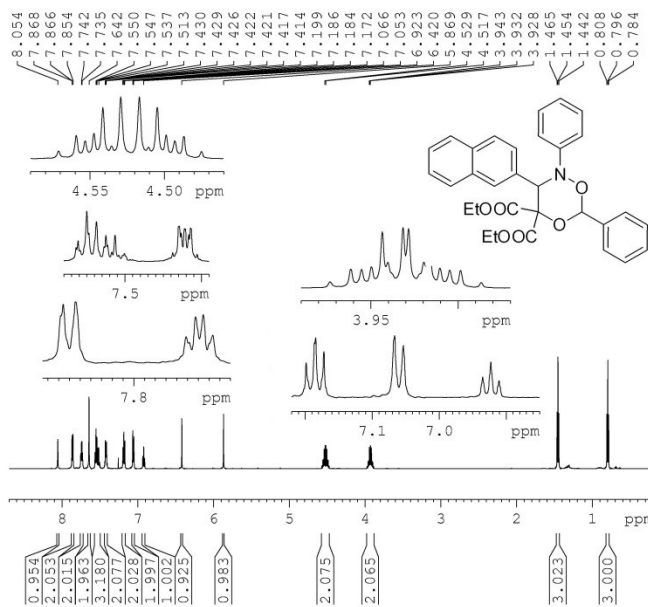
```

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

¹³C NMR chemical shifts (ppm): 167.757, 164.461, 147.728, 128.894, 128.489, 126.875, 124.884, 123.025, 117.483, 116.654, 100.618, 100.555, 84.195, 77.048, 76.836, 63.057, 61.983, 58.988, 53.432, 14.269, 12.869.



3ai



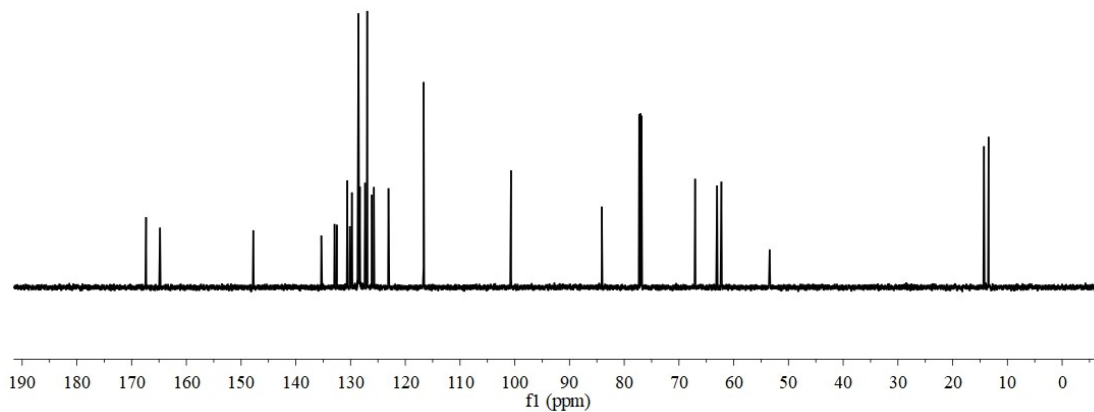
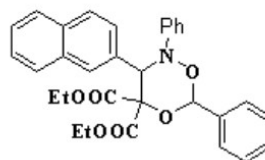
```

NAME          HHG
EXPNO         2018011984
PROCNO        1
Date_         20180119
Time          18.44
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            28.5
DW            40.533 usec
DE            6.50 usec
TE            292.3 K
D1            1.0000000 sec
    
```

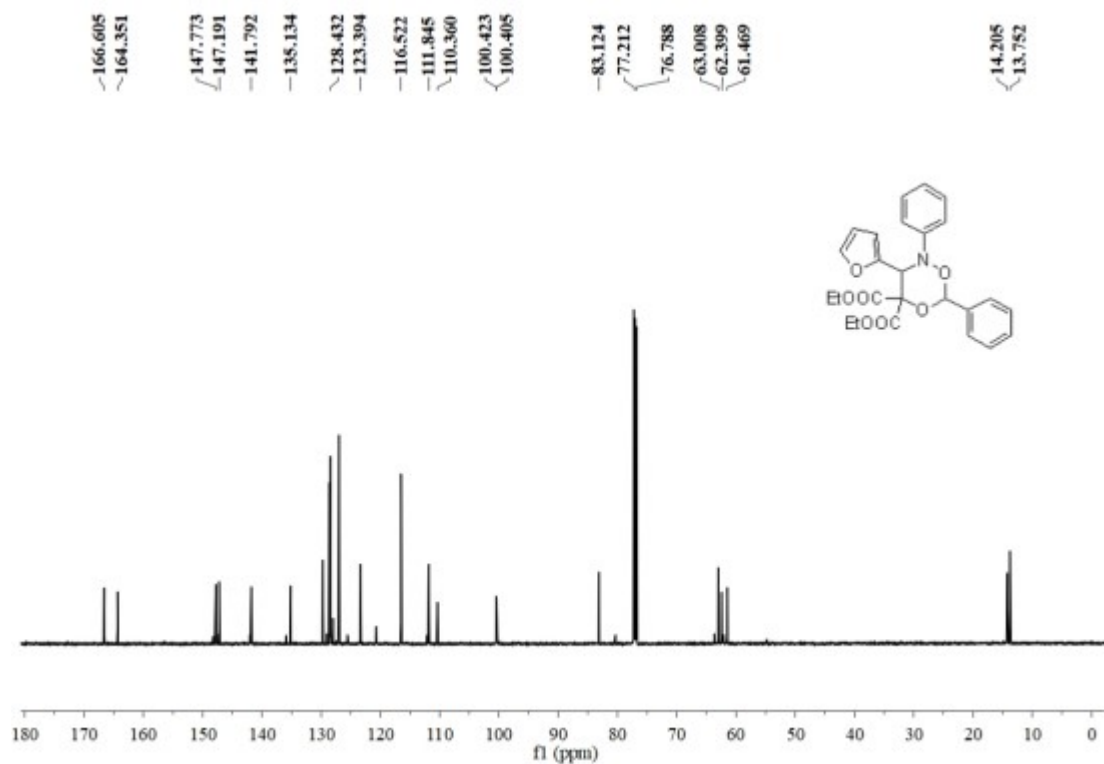
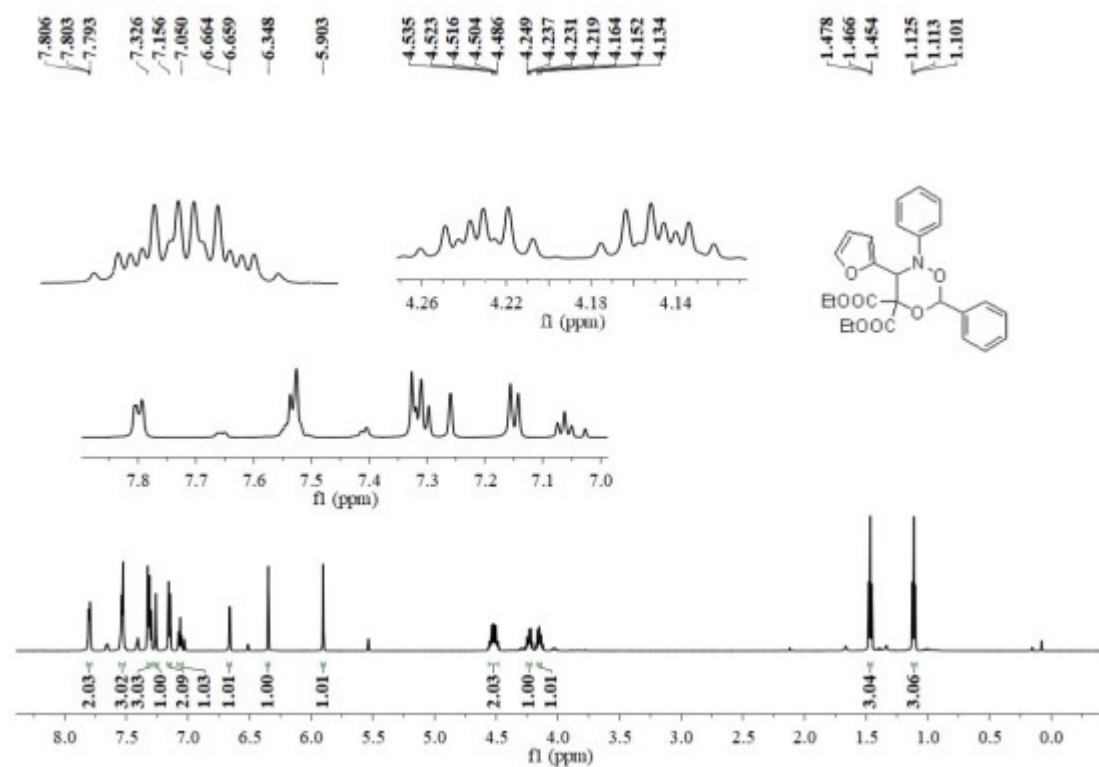
```

===== CHANNEL f1 =====
NUC1           1H
P1             9.88 usec
SI            65536
SF            600.1800153 MHz
WDW            EM
SSB            0
LB            0.30 Hz
GB            0
PC            1.00
    
```

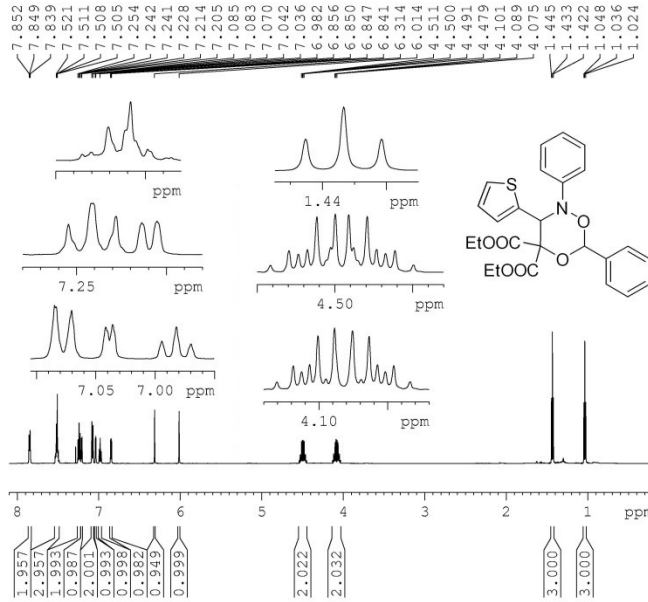
~167.559
 ~164.805
 -147.779
 -130.613
 -128.585
 -128.292
 -126.959
 -123.069
 -116.645
 -100.678
 -84.097
 77.056
 76.845
 67.053
 63.038
 62.251
 -53.440
 14.279
 13.441



3aj



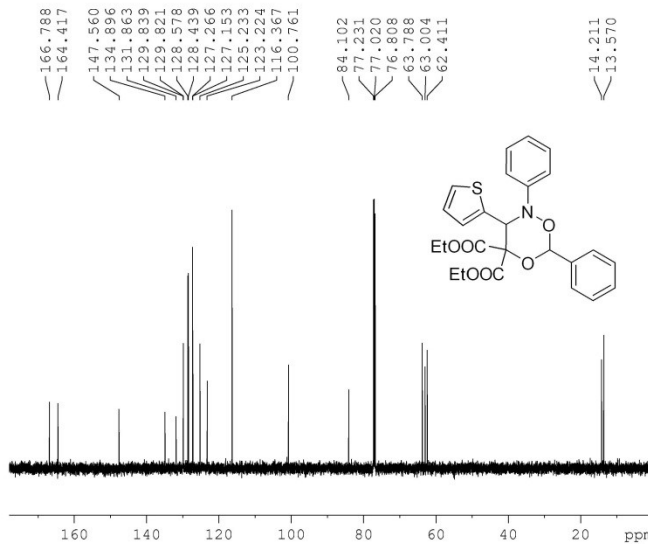
3ak



```

NAME          HHG
EXPNO         2018011986
PROCNO        1
Date_         20180119
Time          18.11
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            32
DW            40.533 usec
DE            6.50 usec
TE            292.1 K
D1            1.00000000 sec

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```

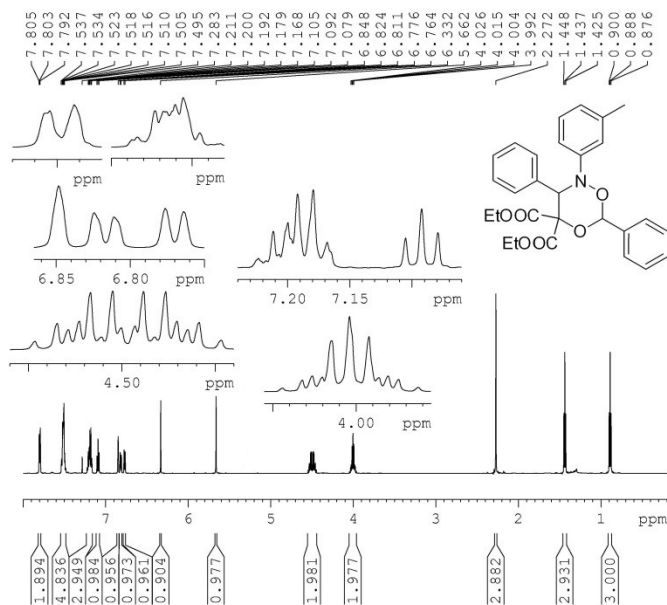


```

NAME          YXZ
EXPNO         18011986
PROCNO        1
Date_         20180119
Time          18.20
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            9
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG            2050
DW            13.867 usec
DE            6.50 usec
TE            292.4 K
D1            2.00000000 sec
D11           0.03000000 sec

===== CHANNEL f1 =====
NUC1          13C
P1            15.64 usec
SI            32768
SF            150.9153956 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

3aI

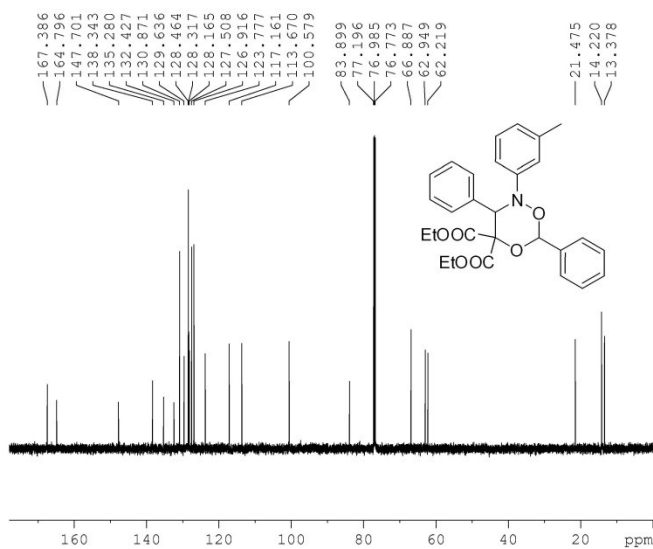


```

NAME          HHG
EXPNO         2018011987
PROCNO        1
Date_         20180119
Time          17.51
INSTRUM       spect
PROBHD        5 mm FABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDCl3
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            32
DW            40.533 usec
DE            6.50 usec
TE            292.1 K
D1            1.00000000 sec
    
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800000 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
    
```



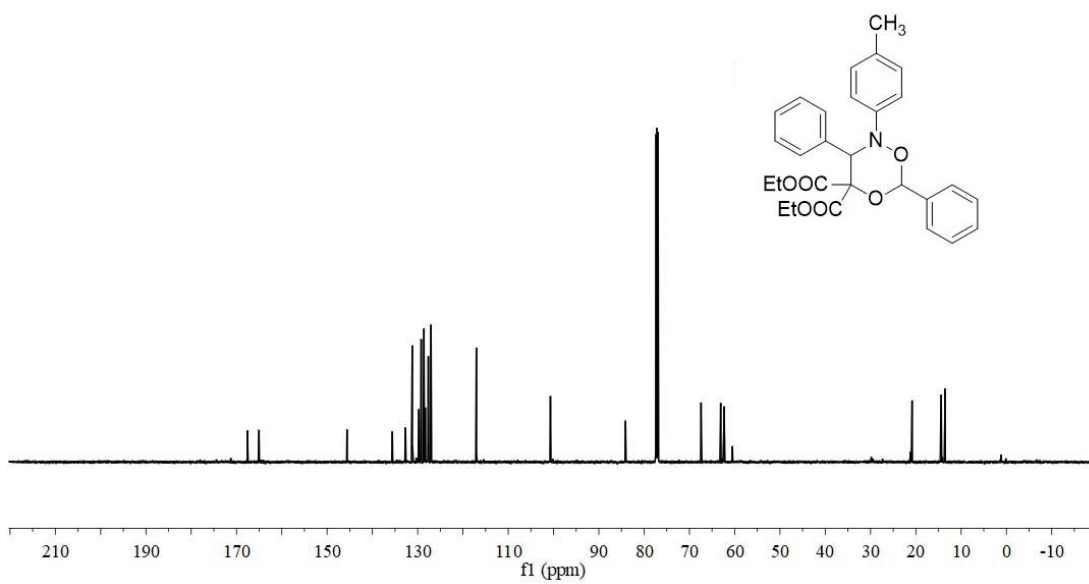
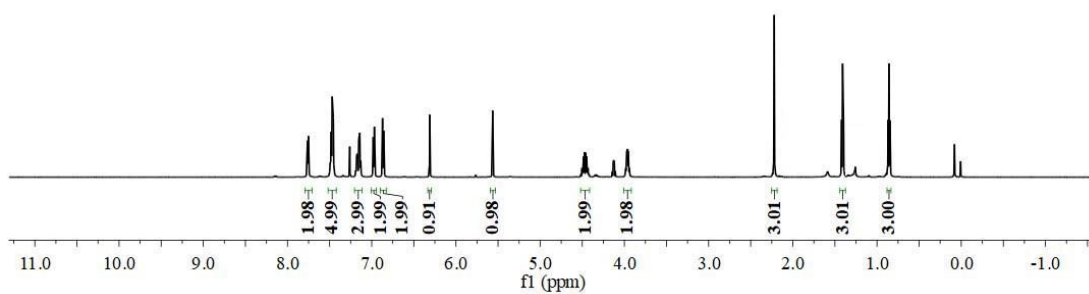
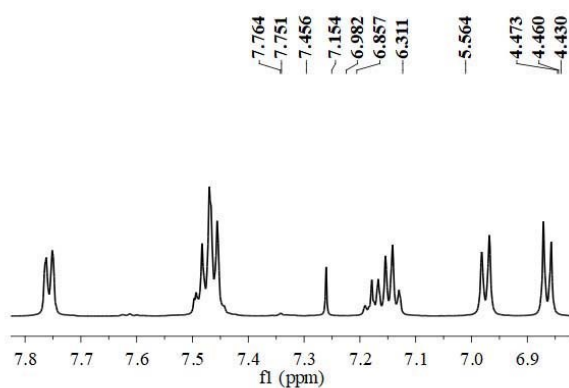
```

NAME          YXZ
EXPNO         18011987
PROCNO        1
Date_         20180119
Time          18.25
INSTRUM       spect
PROBHD        5 mm FABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDCl3
NS            31
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG            2050
DW            13.867 usec
DE            6.50 usec
TE            292.5 K
D1            2.00000000 sec
D11           0.03000000 sec
    
```

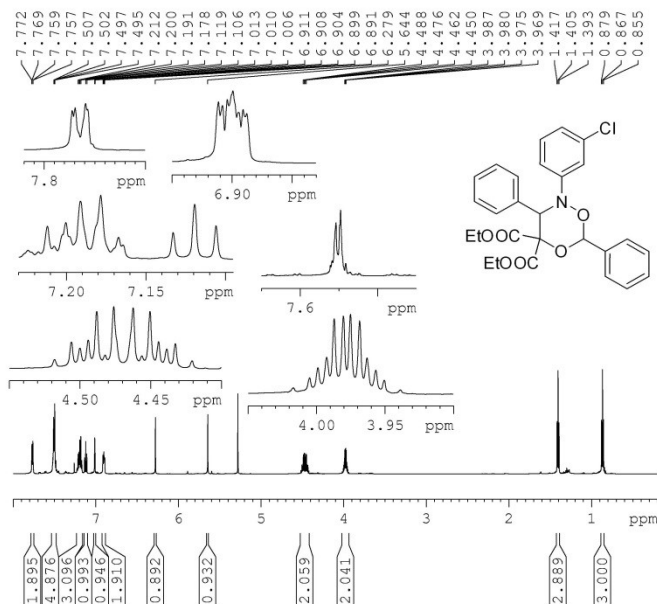
```

===== CHANNEL f1 =====
NUC1          13C
P1            15.64 usec
SI            32768
SF            150.9153956 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
    
```

3am



3an

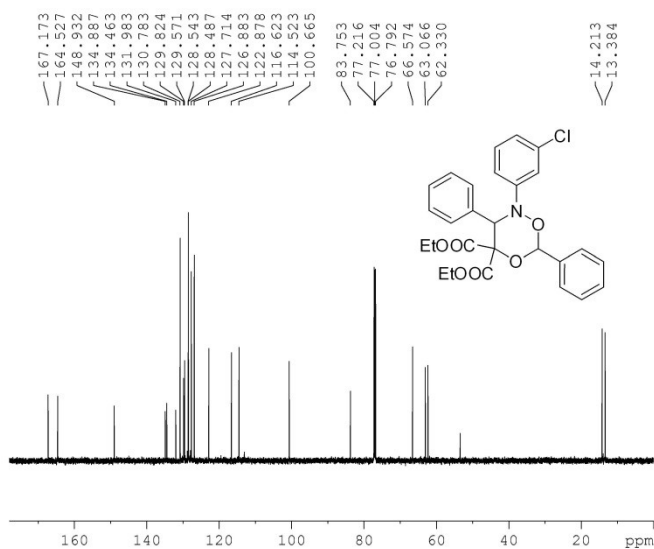


```

NAME          HHG
EXPNO         2018011989
PROCNO        1
Date_         20180119
Time          17.14
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            2
SWH           12335.526 Hz
FIDRES        0.188225 Hz
AQ            2.6564426 sec
RG            28.5
DW            40.533 usec
DE            6.50 usec
TE            292.1 K
D1            1.00000000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          1H
P1            9.88 usec
SI            65536
SF            600.1800153 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



```

NAME          XYZ
EXPNO         170411089
PROCNO        1
Date_         20180119
Time          17.18
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zgpg30
TD            65536
SOLVENT       CDC13
NS            48
DS            4
SWH           36057.691 Hz
FIDRES        0.550197 Hz
AQ            0.9088159 sec
RG            2050
DW            13.867 usec
DE            6.50 usec
TE            292.5 K
D1            2.00000000 sec
D11           0.03000000 sec
  
```

```

===== CHANNEL f1 =====
NUC1          13C
P1            15.64 usec
SI            32768
SF            150.9153956 MHz
WDW           EM
SSB           0
LB            1.00 Hz
GB            0
PC            1.40
  
```


8. ¹H- and ¹³C-NMR spectra of product 4

