Supporting Information

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Content

A: Experimental section

1. General Remarks

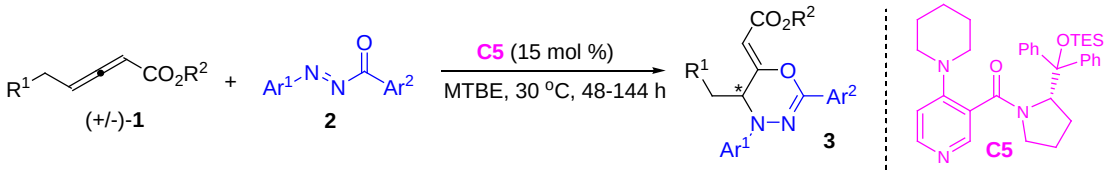
All reactions were carried out with dry, freshly distilled solvents in anhydrous conditions. All chemicals were used without further purification as commercially available unless otherwise noted. All reactions were performed under an atmosphere of dry nitrogen. Thin-layer chromatography (TLC) was performed on silica gel plates (60F-254) using UV-light (254 and 365 nm). Flash chromatography was conducted on silica gel (300–400 mesh). NMR (400 or 500 MHz for ^{1}H NMR, 100 or 126 MHz for ^{13}C NMR) spectra were recorded in $CDCl_3$ with TMS as the internal standard. Chemical shifts are reported in ppm and coupling constants are given in Hz. Data for ^{1}H NMR are recorded as follows: chemical shift (ppm), multiplicity (s, singlet; d, doublet; t, triplet; q,quarter; m, multiplet), coupling constant (Hz), integration. Data for ^{13}C NMR are reported in terms of chemical shift (δ, ppm). High resolution mass spectral (HRMS) analyses were measured using ESI techniques.

Both allenoates[1] and *N*-acyldiazenes[2] were prepared according to the reported procedure. And the *L*-proline-derived DMAP catalysts were synthesized according to Chen and Ouyang's work.[3]

Reference：

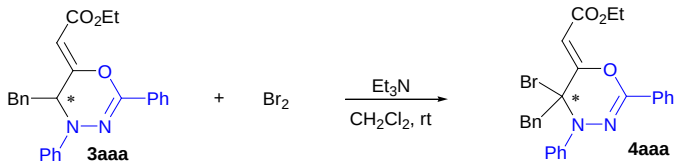
1. S. Xu, L. Zhou, S. Zeng, R. Ma, Z. Wang, Z. He. *Org. Lett.* 2009, **11**, 3498-3501.
2. J. E. Taylor, D. S. B. Daniels, A. D. Smith. *Org. Lett.* 2013, **15**, 23, 6058-6061.
3. G. Zhan, M. Shi, Q. He, W. Lin, Q. Ouyang, W. Du, Y-C. Chen. *Angew. Chem. Int. Ed*. 2016, **55**, 2147-2151.

2. General procedure for asymmetric [2+4] cycloadditions of allenoates 1 with *N*-acyldiazenes 2:



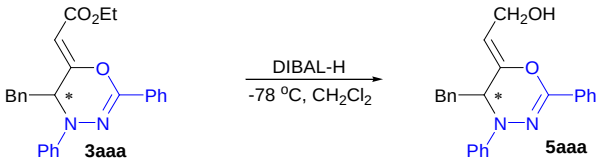
To a stirred solution of allenoates **1** (0.30 mmol) with *N*-acyldiazenes **2** (0.20mmol) in 1.5 mL of MTBE was added **C5** (0.03 mmol). The reaction mixture was stirred at 30 °C for 48-144 h. Then after removal of the solvent, the crude residue was purified by column chromatography on silica gel to give the pure product **3**.

3. Bromination of product 3aaa



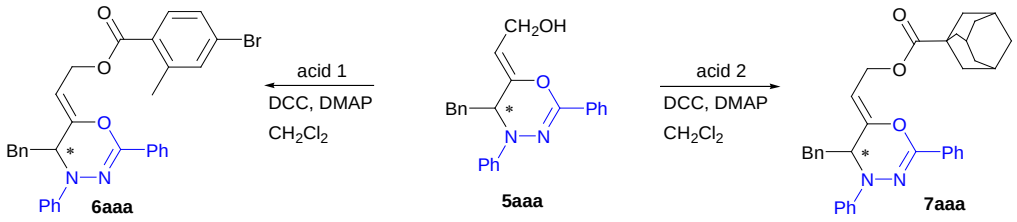
To a suspension of **3aaa** (0.1 mmol, 76% ee) in dichloromethane (2.0 mL) at 0 °C, a solution of Br_2 (0.11 mmol) in dichloromethane (1.0 mL) was added dropwise. The reaction mixture was stirred for about 30 minutes before removal of the solvent under reduced pressure. The residue was redissolved in Et_2O (2.0 mL) and cooled to 0 °C. Then a solution of Et_3N (0.13 mmol) in Et_2O (1.0 mL) was added dropwise. The reaction mixture was stirred for 12 h at room temperature. After removal the solvent under reduced pressure, the crude product was subjected to column chromatography purification to provide **4aaa** as yellow oil in 62% yield (30.4 mg, 76% ee).

4. Reduction of product 3aaa



To a suspension of **3aaa** (0.1 mmol, 76% ee) in anhydrous dichloromethane (2.0 mL) at -78 °C, DIBAL-H (0.4 mmol) was added dropwise. Then the reaction mixture was stirred at -78 °C for 14 h. Then MeOH (1.0 mL) was added to quench the reaction. After removal the solvent under reduced pressure, the crude product was subjected to column chromatography purification to provide **5aaa** as colorless oil in 88% yield (32.5 mg, 76% ee).

5. Esterification of product 5aaa

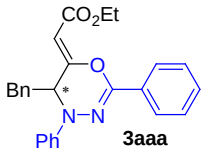


To a solution of **5aaa** (0.1 mmol, 76% ee) in anhydrous dichloromethane (2.0 mL) at room temperature, 4-bromo-2-methylbenzoic acid (0.11 mmol), DCC (0.11 mmol), and DMAP (0.02 mmol) were added. Then the reaction mixture was stirred at room temperature for 24 h. After removal the solvent under reduced pressure, the crude product was subjected to column chromatography purification to provide **6aaa** as colorless oil in 78% yield (44.2 mg, 76% ee).

To a solution of **5aaa** (0.1 mmol, 76% ee) in anhydrous dichloromethane (2.0 mL) at room temperature, 1-adamantanecarboxylic acid (0.11 mmol), DCC (0.11 mmol), and DMAP (0.02 mmol) were added. Then the reaction mixture was stirred at room temperature for 48 h. After removal the solvent under reduced pressure, the crude product was subjected to column chromatography purification to provide **7aaa** as colorless oil in 68% yield (36.4 mg, 76% ee).

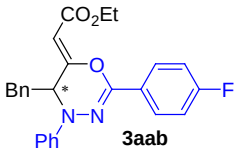
B: Characterization of the products

(*Z*)-Ethyl 2-(5-benzyl-2,4-diphenyl-4*H*-1,3,4-oxadiazin-6(5*H*)-ylidene)acetate (3aaa): pale yellow solid (67.2 mg, 82% yield). m.p. 103.5-105.1 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.22-8.08 (m, 2H), 7.52-7.44 (m, 3H), 7.37 (t, *J* = 7.9 Hz, 2H), 7.32-7.27 (m, 4H), 7.22 (dd, *J* = 21.4, 7.1 Hz, 3H), 6.99 (t, *J* = 7.1 Hz, 1H), 4.78 (s, 1H), 4.72 (dd, *J* = 9.5, 4.7 Hz, 1H), 4.29-4.12 (m, 2H), 2.97 (ddd, *J* = 23.2, 13.6, 7.2 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.4, 152.9, 144.5, 141.9, 136.2, 130.0, 129.9, 129.5, 129.4, 128.7, 128.4, 127.1, 125.7, 120.9, 114.0, 98.3, 60.1, 56.1, 33.9, 14.3. HRMS (ESI) calcd for $C_{26}H_{25}N_2O_3$ $[M+H]^+$: 413.1860; Found: 4131852. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.264 min, t_R (minor) = 8.353 min, 71% ee. $[\alpha]^{20}_D$ = -42.672 (c = 1.00, CH_2Cl_2).



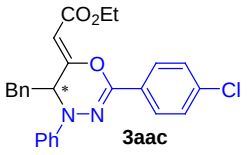
3aaa

(*Z*)-Ethyl 2-(5-benzyl-2-(4-fluorophenyl)-4-phenyl-4*H*-1,3,4-oxadiazin-6(5*H*)-ylidene)acetate (3aab): pale yellow solid (62.1 mg, 72% yield), m.p. 103.2-104.6 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.21-8.06 (m, 2H), 7.39 (t, *J* = 7.9 Hz, 2H), 7.31 (t, *J* = 7.3 Hz, 4H), 7.25 (d, *J* = 7.1 Hz, 1H), 7.22-7.13 (m, 4H), 7.00 (t, *J* = 7.2 Hz, 1H), 4.81 (s, 1H), 4.73 (dd, *J* = 9.5, 4.7 Hz, 1H), 4.32-4.12 (m, 2H), 2.97 (ddd, *J* = 23.1, 13.6, 7.1 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4, 164.0 (d, *J* = 248.4 Hz), 153.0, 144.5, 141.2, 136.2, 129.5, 129.4, 128.7, 127.7 (d, *J* = 8.5 Hz), 127.2, 126.24 (d, *J* = 3.1 Hz), 121.0, 115.6 (d, *J* = 21.9 Hz), 114.1, 98.4, 77.4, 77.1, 76.8, 60.2, 56.1, 34.1, 14.4. ^{19}F NMR (471 MHz, CDCl3) δ -110.6. HRMS (ESI) calcd for $C_{26}H_{24}FN_2O_3$ $[M+H]^+$: 431.1765; Found: 431.1762. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.144 min, t_R (minor) = 6.934 min, 72% ee. $[\alpha]^{20}_D$ = -50.483 (c = 1.00, CH_2Cl_2).



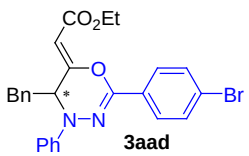
3aab

(*Z*)-Ethyl 2-(5-benzyl-2-(4-chlorophenyl)-4-phenyl-4*H*-1,3,4-oxadiazin-6(5*H*)-ylidene)acetate (3aac): pale yellow solid (65.3 mg, 73% yield), m.p. 123.2-124.7 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07-8.00 (m, 2H), 7.45-7.40 (m, 2H), 7.36 (dd, *J* = 8.7, 7.3 Hz, 2H), 7.29-7.25 (m, 4H), 7.22 (d, *J* = 7.3 Hz, 1H), 7.17-7.13 (m, 2H), 6.98 (t, *J* = 7.3 Hz, 1H), 4.78 (s, 1H), 4.70 (dd, *J* = 9.5, 4.7 Hz, 1H), 4.26-4.12 (m, 2H), 2.94 (ddd, *J* = 23.2, 13.7, 7.1 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.3, 152.7, 144.3, 141.0, 136.1, 135.8, 129.4, 129.4, 128.7, 128.7, 128.5, 127.1, 126.9, 121.1, 114.0, 98.5, 60.1, 56.0, 34.1, 14.3. HRMS (ESI) calcd for $C_{26}H_{24}ClN_2O_3$ $[M+H]^+$: 447.1470; Found: 447.1466. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.355 min, t_R (minor) = 7.973 min, 71% ee. $[\alpha]^{20}_D$ = -18.229 (c = 1.00, CH_2Cl_2).



3aac

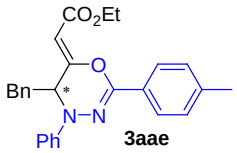
(*Z*)-Ethyl 2-(5-benzyl-2-(4-bromophenyl)-4-phenyl-4H-1,3,4-oxadiazin-6(5*H*)-ylidene)acetate (3aad): pale yellow solid (64.3 mg; 66% yield), m.p. 111.2-113.7 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05-7.90 (m, 2H), 7.67-7.52 (m, 2H), 7.44-7.14 (m, 10H), 7.00 (t, *J* = 7.2 Hz, 1H), 4.81 (s, 1H), 4.73 (dd, *J* = 9.4, 4.7 Hz, 1H), 4.32-4.11 (m, 2H), 2.97 (ddd, *J* = 23.0, 13.6, 7.1 Hz, 2H), 1.32 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3, 152.6, 144.3, 141.1, 136.0, 131.6, 129.4, 129.0, 128.7,



3aad

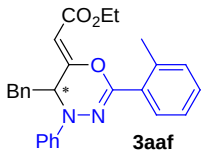
127.1, 124.2, 121.1, 114.0, 98.5, 60.1, 56.1, 34.1, 14.3. HRMS (ESI) calcd for $C_{26}H_{24}BrN_2O_3$ $[M+H]^+$: 491.0965; Found: 491.0960. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.582 min, t_R (minor) = 8.588 min, 70% ee. $[\alpha]^{20}_D$ = -80.663 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl 2-(5-benzyl-4-phenyl-2-(*p*-tolyl)-4*H*-1,3,4-oxadiazin- 6(5*H*)-ylidene)acetate (3aae): pale yellow oil (64.3 mg; 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04 (d, *J* = 8.0 Hz, 2H), 7.40-7.17 (m, 12H), 6.98 (t, *J* = 7.2 Hz, 1H), 4.76 (s, 1H), 4.71 (dd, *J* = 9.5, 4.7 Hz, 1H), 4.29-4.15 (m, 2H), 2.96 (ddd, *J* = 23.2, 13.6, 7.2 Hz, 2H), 2.45 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.4, 153.1, 144.6, 142.2, 140.1, 136.3, 129.5, 129.3, 129.2 128.6, 127.3, 127.1, 125.7, 120.8, 114.0, 98.2, 60.1, 56.2, 33.8, 21.5, 14.3. HRMS (ESI) calcd for $C_{26}H_{27}N_2O_3$ $[M+H]^+$: 427.2016; Found: 427.2014. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.155 min, t_R (minor) = 7.87 min, 70% ee. $[\alpha]^{20}_D$ = -81.081 (c = 1.00, CH_2Cl_2).



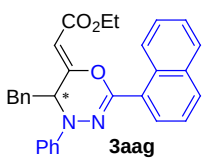
3aae

(*Z*)-Ethyl 2-(5-benzyl-4-phenyl-2-(o-tolyl)-4*H*-1,3,4-oxadiazin- 6(5*H*)-ylidene)acetate (3aaf): pale yellow oil (75.0 mg; 88% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 6.0, 3.2 Hz, 1H), 7.37-7.15 (m, 13H), 6.96 (t, *J* = 7.2 Hz, 1H), 4.73 (s, 1H), 4.70 (dd, *J* = 9.5, 4.7 Hz, 1H), 4.18 (tt, *J* = 7.8, 5.4 Hz, 2H), 2.96 (ddd, *J* = 23.2, 13.6, 7.1 Hz, 2H), 2.74 (s, 3H), 1.27 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.4, 153.2, 144.6, 142.4, 137.3, 136.2, 131.6, 129.5, 129.4, 128.9, 128.7, 128.4, 127.1, 126.0, 120.9, 114.0, 98.0, 60.1, 55.8, 33.5, 23.1, 14.3. HRMS (ESI) calcd for $C_{26}H_{27}N_2O_3$ $[M+H]^+$: 427.2016; Found: 427.2015. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.15 min, t_R (minor) = 9.707 min, 71% ee. $[\alpha]^{20}_D$ = -51.144 (c = 1.00, CH_2Cl_2).



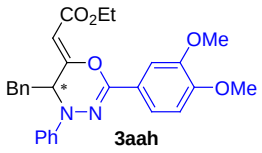
3aaf

(*Z*)-Ethyl 2-(5-benzyl-2-(naphthalen-1-yl)-4-phenyl-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene) acetate (3aag): pale yellow solid (71.0 mg; 77% yield), m.p. 108.3-110.1 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.20 (d, *J* = 8.7 Hz, 1H), 8.35 (d, *J* = 7.3 Hz, 1H), 7.93 (dd, *J* = 17.3, 8.1 Hz, 2H), 7.59 (tt, *J* = 18.5, 7.5 Hz, 3H), 7.41-7.35 (m, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.29 (t, *J* = 7.3 Hz, 2H), 7.22 (dd, *J* = 15.2, 6.3 Hz, 3H), 6.99 (t, *J* = 7.1 Hz, 1H), 4.91-4.68 (m, 2H), 4.31-4.07 (m, 2H), 3.07 (dd, *J* = 13.5, 7.1 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.5, 153.1, 144.5, 142.0, 136.2, 134.1, 130.9, 130.3, 129.5, 128.8, 128.7, 127.6, 127.2, 127.1, 126.3, 126.0, 125.1, 121.1, 114.0, 98.2, 60.1, 55.9, 33.9, 14.3. HRMS (ESI) calcd for $C_{30}H_{29}N_2O_3$ $[M+H]^+$: 463.2016; Found: 463.2011. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 6.461 min, t_R (minor) = 8.548 min, 70% ee. $[\alpha]^{20}_D$ = -11.601 (c = 1.00, CH_2Cl_2).



3aag

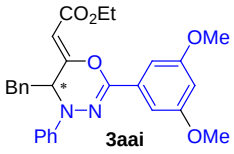
(*Z*)-Ethyl 2-(5-benzyl-2-(3,4-dimethoxyphenyl)-4-phenyl-4*H*-1,3,4-oxadiazin-6(5*H*)-ylidene) acetate (3aah): yellow oil (66.7 mg; 71% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.68 (d, *J* = 2.0 Hz, 1H), 7.41-7.18 (m, 11H), 6.97 (d, *J* = 8.3 Hz, 2H), 4.78 (s, 1H), 4.70 (dd, *J* = 9.5, 4.8 Hz, 1H), 4.28-4.14 (m, 2H), 4.04 (s, 3H), 3.98 (s, 3H), 2.97 (ddd, *J* = 23.1, 13.6, 7.1



3aah

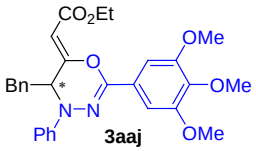
Hz, 2H), 1.31 (t, *J* = 7.1 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.3, 153.1, 150.8, 148.9, 144.6, 142.1, 136.3, 129.4, 129.3, 128.7, 127.0, 122.8, 120.8, 119.1, 114.0, 110.7, 108.6, 98.1, 60.0, 56.3, 56.1, 56.0, 34.0, 14.3. HRMS (ESI) calcd for $C_{28}H_{29}N_2O_5$ $[M+H]^+$: 473.2071; Found: 473.2071. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 9.73 min, t_R (minor) = 12.267 min, 74% ee. $[\alpha]^{20}_D$ = -91.105 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl 2-(5-benzyl-2-(3,5-dimethoxyphenyl)-4-phenyl-4*H*-1,3,4-oxadiazin-6(5*H*)-ylidene) acetate (3aai): yellow oil (63.4 mg; 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40-7.14 (m, 12H), 6.97 (t, *J* = 7.2 Hz, 1H), 6.56 (t, *J* = 2.3 Hz, 1H), 4.75 (s, 1H), 4.69 (dd, *J* = 9.5, 4.8 Hz, 1H), 4.20 (dt, *J* = 7.1, 2.4 Hz, 2H), 3.89 (s, 6H), 2.94 (ddd, *J* = 23.2, 13.6, 7.2 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.3, 160.8, 152.5, 144.4, 141.7, 136.2, 131.9, 129.5, 129.4, 128.7, 127.1, 121.0, 114.1, 103.8, 102.3, 98.4, 60.1, 56.2, 55.5, 34.0, 14.3. HRMS (ESI) calcd for $C_{28}H_{29}N_2O_5$ $[M+H]^+$: 473.2071; Found: 473.2069. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 6.756 min, t_R (minor) = 8.195 min, 72% ee. $[\alpha]^{20}_D$ = -85.065 (c = 1.00, CH_2Cl_2).



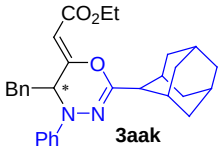
3aai

(*Z*)-Ethyl 2-(5-benzyl-4-phenyl-2-(3,4,5-trimethoxyphenyl)-4*H*-1,3,4-oxadiazin-6(5*H*)-ylidene) acetate (3aaj): yellow oil (77.0 mg; 77% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 (s, 2H), 7.35 (dd, *J* = 8.5, 7.4 Hz, 2H), 7.29 (t, *J* = 7.3 Hz, 2H), 7.26-7.22 (m, 3H), 7.21- 7.17 (m, 2H), 6.97 (t, *J* = 7.3 Hz, 1H), 4.77 (s, 1H), 4.68 (dd, *J* = 9.5, 4.7 Hz, 1H), 4.18 (dd, *J* = 7.1, 4.7 Hz, 2H), 4.00 (s, 6H), 3.92 (s, 3H), 2.95 (ddd, *J* = 23.2, 13.7, 7.2 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.1, 153.2, 152.7, 144.4, 141.8, 139.8, 136.3, 129.5, 129.4, 128.7, 127.1, 125.4, 121.0, 114.0, 103.1, 98.3, 61.0, 59.9, 56.3, 56.2, 34.1, 14.4. HRMS (ESI) calcd for $C_{29}H_{31}N_2O_6$ $[M+H]^+$: 503.2177; Found: 503.2173. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 11.078 min, t_R (minor) = 12.725 min, 78% ee. $[\alpha]^{20}_D$ = -101.07 (c = 1.00, CH_2Cl_2).



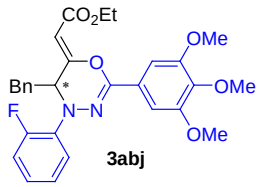
3aaj

(*Z*)-Ethyl-2-(2-((3r,5r,7r)-adamantan-1-yl)-5-benzyl-4-phenyl-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3aak): pale yellow solid (82.0 mg; 87% yield), m.p. 103.2-104.3 °C. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34-7.25 (m, 4H), 7.20 (dd, *J* = 24.2, 7.6 Hz, 3H), 7.11 (d, *J* = 7.0 Hz, 2H), 6.90 (t, *J* = 7.2 Hz, 1H), 4.58 (s, 1H), 4.54 (dd, *J* = 9.8, 4.7 Hz, 1H), 4.22-4.06 (m, 2H), 2.80 (ddd, *J* = 23.4, 13.5, 7.3 Hz, 2H), 2.10 (d, *J* = 13.1 Hz, 9H), 1.79 (s, 6H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.6, 153.7, 151.3, 145.1, 136.4, 129.4, 129.2, 128.6, 126.9, 120.3, 113.8, 97.5, 59.9, 56.0, 39.2, 37.8, 36.7, 32.3, 28.0, 14.3. HRMS (ESI) calcd for $C_{30}H_{35}N_2O_3$ $[M+H]^+$: 471.2642; Found: 471.2642. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 97.5/2.5, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.193 min, t_R (minor) = 6.91 min, 75% ee. $[\alpha]^{20}_D$ = -99.510 (c = 1.00, CH_2Cl_2).



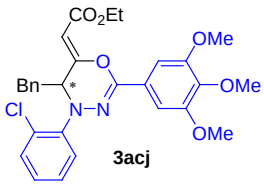
3aak

(*Z*)-Ethyl-2-(5-benzyl-4-(2-fluorophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene) acetate (3abj): yellow oil (85.1 mg; 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64



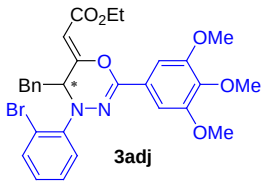
(td, *J* = 8.6, 1.5 Hz, 1H), 7.38 (s, 2H), 7.21 (t, *J* = 7.2 Hz, 2H), 7.15 (ddd, *J* = 12.2, 5.4, 1.3 Hz, 2H), 7.10-6.97 (m, 4H), 4.71 (s, 1H), 4.65 (ddd, *J* = 9.1, 5.3, 1.6 Hz, 1H), 4.14 (dddd, *J* = 18.0, 10.9, 7.1, 3.8 Hz, 2H), 3.97 (s, 6H), 3.90 (s, 3H), 2.89 (d, *J* = 2.7 Hz, 2H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.1, 153.6, 153.2, 152.7 (d, *J* = 245.7 Hz), 143.3, 140.0, 136.2, 133.8, 133.7, 129.3, 128.5, 126.9, 125.3, 124.9 (d, *J* = 3.3 Hz), 124.0 (d, *J* = 7.8 Hz), 122.5, 116.5 (d, *J* = 20.2 Hz), 103.2, 98.3, 61.0, 59.9, 59.3, 59.2, 56.3, 34.3, 14.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -123.5. HRMS (ESI) calcd for $C_{29}H_{30}FN_2O_6$ $[M+H]^+$: 521.2083; Found: 521.2082. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 98/2, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 10.71 min, t_R (minor) = 14.188 min, 75% ee. $[\alpha]^{20}_D$ = -63.4687 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl-2-(5-benzyl-4-(2-chlorophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene) acetate (3acj): yellow oil (89.7 mg; 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66



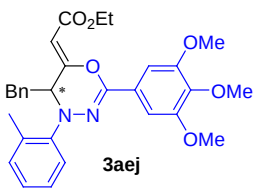
(dd, *J* = 8.1, 1.4 Hz, 1H), 7.44-7.38 (m, 3H), 7.34 (td, *J* = 8.1, 1.4 Hz, 1H), 7.24-7.11 (m, 4H), 7.05-6.95 (m, 2H), 4.72 (s, 1H), 4.66 (dt, *J* = 17.0, 8.5 Hz, 1H), 4.25-4.09 (m, 2H), 3.98 (s, 6H), 3.92 (s, 3H), 2.89 -2.74 (m, 2H), 1.27 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 153.9, 153.2, 144.0, 143.3, 140.0, 136.3, 130.6, 129.2, 128.5, 127.7, 126.8, 126.2, 126.0, 125.2, 103.2, 98.0, 60.9, 59.9, 58.7, 56.3, 32.6, 14.3. HRMS (ESI) calcd for $C_{29}H_{30}ClN_2O_6$ $[M+H]^+$: 537.1787; Found: 537.1785. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 98/2, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 11.983 min, t_R (major) =13.467 min, 78% ee. $[\alpha]^{20}_D$ = -139.58 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl-2-(5-benzyl-4-(2-bromophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3adj): yellow oil (94.2 mg; 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.66



(ddd, *J* = 9.5, 8.0, 4.0 Hz, 2H), 7.46-7.37 (m, 3H), 7.27-7.18 (m, 3H), 7.15-7.05 (m, 1H), 7.01 (d, *J* = 7.5 Hz, 2H), 4.74 (t, *J* = 5.3 Hz, 1H), 4.72-4.65 (m, 1H), 4.24-4.14 (m, 2H), 4.00 (d, *J* = 1.6 Hz, 6H), 3.94 (d, *J* = 1.8 Hz, 3H), 2.85 (dd, *J* = 19.0, 6.8 Hz, 2H), 1.29 (ddd, *J* = 7.1, 4.5, 1.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 154.0, 153.2, 144.6, 144.0, 140.0, 136.3, 133.8, 129.2, 128.5, 128.3, 126.9, 126.7, 126.5, 125.2, 116.3, 103.1, 97.9, 61.0, 59.9, 58.9, 56.3, 32.0, 14.3. HRMS (ESI) calcd for $C_{29}H_{30}BrN_2O_6$ $[M+H]^+$: 581.1282; Found: 581.1281. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 98/2, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 10.365 min, t_R (minor) = 13.814 min, 76% ee. $[\alpha]^{20}_D$ = -133.740 (c = 1.00, CH_2Cl_2).

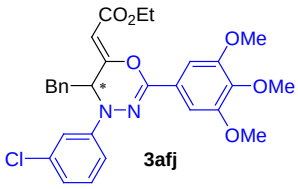
(*Z*)-Ethyl-2-(5-benzyl-4-(o-tolyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3aej): yellow oil (83.4 mg; 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (d, *J* = 7.9



Hz, 1H), 7.41 (s, 2H), 7.37-7.13 (m, 7H), 7.00 (d, *J* = 7.3 Hz, 2H), 4.65 (s, 1H), 4.26-4.15 (m, 2H), 4.00 (s, 6H), 3.94 (d, *J* = 0.4 Hz, 3H), 2.89 (dd, *J* = 11.1, 5.6 Hz, 2H), 2.34 (s, 3H), 1.30 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 154.3, 153.2, 144.8, 143.0, 139.8, 136.6, 131.5, 130.5, 129.2, 128.5, 126.8, 125.5, 125.3, 124.5, 102.9, 97.3, 60.9, 59.9, 59.6,

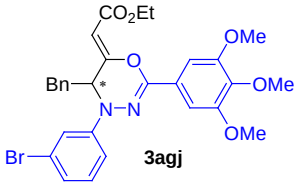
56.3, 31.8, 18.2, 14.4. HRMS (ESI) calcd for $C_{30}H_{31}N_2O_6$ $[M+H]^+$: 517.2333; Found: 517.2332. HPLC analysis [Daicel Chiralpak IC-3 column, n-hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 22.5 min, t_R (major) = 24.362 min, 78% ee. $[\alpha]^{20}_D$ = -119.251 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl-2-(5-benzyl-4-(3-chlorophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3afj): yellow oil (81.1 mg; 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 (s, 2H), 7.30-7.15 (m, 7H), 7.01 (dd, *J* = 8.3, 2.1 Hz, 1H), 6.90 (dd, *J* = 7.9, 1.0 Hz, 1H), 4.84 (s, 1H), 4.64 (dd, *J* = 8.9, 5.4 Hz, 1H), 4.18 (dd, *J* = 7.1, 2.3 Hz, 2H), 4.00 (s, 6H), 3.92 (s, 3H), 2.94 (ddd, *J* = 22.5, 13.6, 7.1 Hz, 2H), 1.27 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.9, 153.3, 152.4, 145.6, 142.4, 135.9, 135.2, 130.2, 129.4, 128.7, 127.3, 125.0, 120.6, 114.2, 111.6, 103.3, 98.6, 61.0, 60.0, 56.3, 56.1, 34.5, 14.3. HRMS (ESI) calcd for $C_{29}H_{30}ClN_2O_6$ $[M+H]^+$: 537.1787; Found: 537.1785. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 98/2, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 17.11 min, t_R (minor) = 19.818 min, 74% ee. $[\alpha]^{20}_D$ = -86.687 (c = 1.00, CH_2Cl_2).



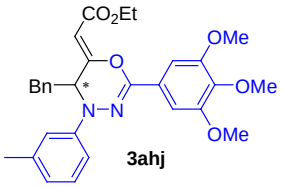
3afj

(*Z*)-Ethyl-2-(5-benzyl-4-(3-bromophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3agj): yellow oil (94.2 mg; 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 2H), 7.38 (s, 1H), 7.29 (ddd, *J* = 11.1, 9.4, 7.0 Hz, 3H), 7.19 (dd, *J* = 9.8, 8.1 Hz, 3H), 7.07 (t, *J* = 7.1 Hz, 2H), 4.87 (d, *J* = 0.7 Hz, 1H), 4.66 (dd, *J* = 8.7, 5.5 Hz, 1H), 4.27-4.16 (m, 2H), 4.03 (s, 6H), 3.95 (d, *J* = 0.8 Hz, 3H), 2.97 (ddd, *J* = 22.5, 13.6, 7.2 Hz, 2H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.9, 153.2, 152.4, 145.7, 142.4, 140.1, 135.8, 130.4, 129.4, 128.8, 127.3, 125.0, 123.6, 123.4, 117.1, 112.1, 103.3, 98.6, 61.0, 60.0, 56.3, 56.0, 34.5, 14.3. HRMS (ESI) calcd for $C_{29}H_{30}BrN_2O_6$ $[M+H]^+$: 581.1282; Found: 581.1279. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 98/2, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 17.801 min, t_R (minor) = 21.201 min, 75% ee. $[\alpha]^{20}_D$ = -84.760 (c = 1.00, CH_2Cl_2).



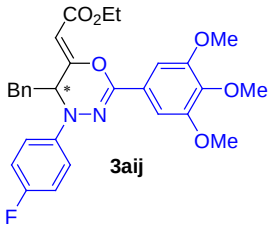
3agj

(*Z*)-Ethyl-2-(5-benzyl-4-(m-tolyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3ahj): yellow oil (87.2 mg; 84% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (s, 2H), 7.32 -7.12 (m, 6H), 7.00 (d, *J* = 5.6 Hz, 2H), 6.76 (d, *J* = 7.4 Hz, 1H), 4.75 (s, 1H), 4.64 (dd, *J* = 9.3, 4.9 Hz, 1H), 4.15 (dt, *J* = 7.1, 4.0 Hz, 2H), 3.97 (s, 6H), 3.89 (s, 3H), 2.92 (dd, *J* = 24.0, 7.1 Hz, 2H), 2.34 (s, 3H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 153.2, 152.8, 144.5, 141.8, 139.8, 139.2, 136.4, 129.4, 129.1, 128.7, 127.1, 125.5, 121.9, 114.9, 111.2, 103.2, 98.1, 61.0, 59.9, 56.4, 56.3, 34.2, 21.8, 14.4. HRMS (ESI) calcd for $C_{30}H_{33}N_2O_6$ $[M+H]^+$: 517.2333; Found: 517.2332. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 5.989 min, t_R (minor) = 14.448 min, 77% ee. $[\alpha]^{20}_D$ = -112.963 (c = 1.00, CH_2Cl_2).



3ahj

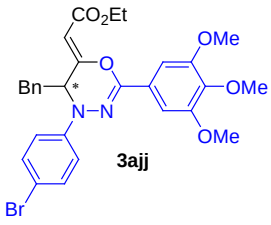
(*Z*)-Ethyl-2-(5-benzyl-4-(4-fluorophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3aij): yellow oil (86.7 mg; 83% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (s,



3aij

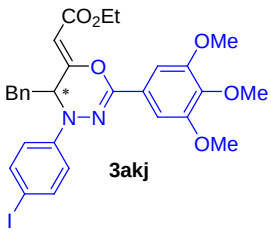
2H), 7.28-7.19 (m, 3H), 7.12 (dd, *J* = 9.9, 5.5 Hz, 4H), 7.00 (t, *J* = 8.7 Hz, 2H), 4.77 (s, 1H), 4.57 (dd, *J* = 9.0, 5.2 Hz, 1H), 4.15 (qd, *J* = 7.1, 3.0 Hz, 2H), 3.97 (s, 6H), 3.89 (s, 3H), 2.89 (ddd, *J* = 22.7, 13.6, 8.8 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 157.8 (d, *J* = 238 Hz), 153.2, 152.7, 142.1, 141.1 (d, *J* = 2.1 Hz), 139.9, 136.1, 129.4, 128.7, 127.1, 125.3, 115.9 (d, *J* = 23.2 Hz), 115.7 (d, *J* = 8.1 Hz), 103.1, 98.2, 61.0, 59.9, 56.9, 56.3, 33.9, 14.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -123.3. HRMS (ESI) calcd for $C_{29}H_{30}FN_2O_6$ $[M+H]^+$: 521.2083; Found: 521.2083. HPLC analysis [Daicel Chiralpak ID-3 column, n-hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 21.68 min, t_R (major) = 22.662 min, 79% ee. $[\alpha]^{20}_D$ = -93.037 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl-2-(5-benzyl-4-(4-bromophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3ajj): yellow oil (89.7 mg; 77% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (t, *J* = 5.1 Hz, 4H), 7.28-7.19 (m, 3H), 7.13 (d, *J* = 6.8 Hz, 2H), 7.04 (d, *J* = 7.9 Hz, 2H), 4.79 (d, *J* = 1.2 Hz, 1H), 4.59 (dd, *J* = 8.1, 5.7 Hz, 1H), 4.22-4.09 (m, 2H), 3.97 (d, *J* = 1.1 Hz, 6H), 3.89 (d, *J* = 1.2 Hz, 3H), 2.90 (qd, *J* = 13.6, 7.2 Hz, 2H), 1.23 (td, *J* = 7.1, 1.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.0, 153.2, 152.5, 143.6, 142.2, 140.0, 135.9, 132.1, 129.4, 128.7, 127.2, 125.1, 115.5, 113.1, 103.2, 98.5, 61.0, 60.0, 56.3, 56.2, 34.3, 14.3. HRMS (ESI) calcd for $C_{29}H_{30}BrN_2O_6$ $[M+H]^+$: 581.1282; Found: 581.1282. HPLC analysis [Daicel Chiralpak IC-3 column, n-hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 39.081 min, t_R (major) = 42.242 min, 80% ee. $[\alpha]^{20}_D$ = -85.448 (c = 1.00, CH_2Cl_2).



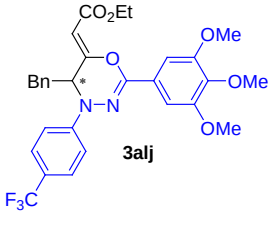
3ajj

(*Z*)-Ethyl-2-(5-benzyl-4-(4-iodophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3akj): yellow oil (97.4 mg; 78% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 (d, *J* = 8.8 Hz, 2H), 7.36 (s, 2H), 7.29-7.21 (m, 3H), 7.13 (d, *J* = 7.0 Hz, 2H), 6.94 (d, *J* = 8.8 Hz, 2H), 4.80 (s, 1H), 4.59 (dd, *J* = 8.9, 5.3 Hz, 1H), 4.16 (qd, *J* = 7.1, 2.6 Hz, 2H), 3.97 (s, 6H), 3.89 (s, 3H), 2.90 (qd, *J* = 13.6, 7.1 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 164.0, 153.3, 152.4, 144.2, 142.3, 140.1, 138.0, 135.9, 129.4, 128.8, 127.2, 125.1, 116.0, 103.2, 98.5, 82.9, 61.0, 60.0, 56.3, 56.0, 34.3, 14.3. HRMS (ESI) calcd for $C_{29}H_{30}IN_2O_6$ $[M+H]^+$: 629.1143; Found: 629.1143. HPLC analysis [Daicel Chiralpak IC-3 column, n-hexane/2-propanol = 98/2, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 88.805 min, t_R (major) = 94.628 min, 78% ee. $[\alpha]^{20}_D$ = -44.304 (c = 1.00, CH_2Cl_2).



3akj

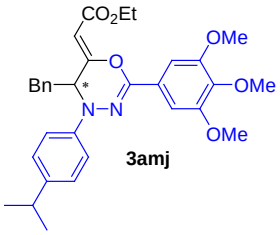
(*Z*)-Ethyl-2-(5-benzyl-4-(4-(trifluoromethyl)phenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6H-1,3,4-oxadiazin-6-ylidene) acetate (3alj): yellow oil (85.0 mg; 75% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.7 Hz, 2H), 7.40 (s, 2H), 7.24 (ddd, *J* = 14.4, 8.2, 2.8 Hz, 5H), 7.18-7.13 (m, 2H), 4.86 (s, 1H), 4.71 (dd, *J* = 8.7, 5.5 Hz, 1H), 4.18 (qd, *J* = 7.1, 2.0 Hz, 2H), 2.95 (qd, *J* = 13.7, 7.1 Hz, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 153.3, 152.2, 146.8, 142.7, 140.2, 135.7, 129.4, 128.8, 127.3, 126.6 (q, *J* = 3.8 Hz), 124.9, 124.5 (q, *J* = 269.1 Hz), 122.2 (q, *J* = 32.5 Hz), 113.1, 103.4, 98.9, 61.0,



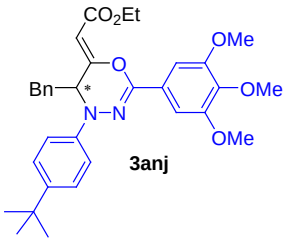
3alj

60.1, 56.3, 55.7, 34.7, 14.3. ^{19}F NMR (471 MHz, $CDCl_3$) δ -61.5. HRMS (ESI) calcd for $C_{30}H_{30}F_3N_2O_6$ $[M+H]^+$: 571.2051; Found: 571.2049. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 8.021 min, t_R (major) = 8.813 min, 72% ee. $[\alpha]^{20}_D$ = -79.201 (c = 1.00, CH_2Cl_2).

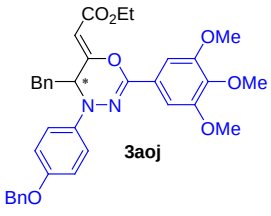
(*Z*)-Ethyl-2-(5-benzyl-4-(4-isopropylphenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3amj): yellow oil (77.9 mg; 72% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (s, 2H), 7.35-7.19 (m, 9H), 4.77 (s, 1H), 4.67 (dd, *J* = 9.6, 4.5 Hz, 1H), 4.27-4.13 (m, 2H), 4.02 (s, 6H), 3.95 (s, 3H), 3.09-2.85 (m, 3H), 1.29 (t, *J* = 5.6 Hz, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 153.2, 152.8, 142.5, 141.7, 141.6, 139.7, 136.4, 129.4, 128.7, 127.2, 127.0, 125.5, 114.3, 103.0, 98.1, 61.0, 59.9, 56.5, 56.3, 33.9, 33.3, 24.2, 24.1, 14.4. HRMS (ESI) calcd for $C_{32}H_{37}N_2O_6$ $[M+H]^+$: 545.2646; Found: 545.2646. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 8.472 min, t_R (minor) = 14.456 min, 73% ee. $[\alpha]^{20}_D$ = -80.492 (c = 1.00, CH_2Cl_2).



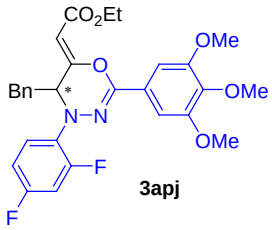
(*Z*)-Ethyl-2-(5-benzyl-4-(4-(tert-butyl)phenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3anj): colorless oil (84.5 mg; 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ7.42-7.35 (m, 4H), 7.32-7.23 (m, 3H), 7.19 (d, *J* = 7.2 Hz, 4H), 4.73 (d, *J* = 1.3 Hz, 1H), 4.64 (dd, *J* = 9.1, 3.8 Hz, 1H), 4.22-4.09 (m, 2H), 3.99 (d, *J* = 1.0 Hz, 6H), 3.94-3.89 (m, 3H), 3.04-2.83 (m, 2H), 1.33 (d, *J* = 1.2 Hz, 9H), 1.28-1.23 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.2, 153.2, 152.8, 143.9, 142.1, 141.6, 139.7, 136.4, 129.4, 128.7, 127.0, 126.2, 125.5, 113.9, 103.0, 98.1, 61.0, 59.9, 56.4, 56.3, 34.1, 33.9, 31.4, 14.4. HRMS (ESI) calcd for $C_{33}H_{39}N_2O_6$ $[M+H]^+$: 559.2803; Found: 559.2800. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 7.862 min, t_R (minor) = 13.153 min, 72% ee. $[\alpha]^{20}_D$ = -79.553 (c = 1.00, CH_2Cl_2).



(*Z*)-Ethyl-2-(5-benzyl-4-(4-(benzyloxy)phenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3aoj): yellow oil (84.5 mg; 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ7.44 -7.20 (m, 10H), 7.14 (d, *J* = 8.9 Hz, 4H), 6.96 (d, *J* = 9.1 Hz, 2H), 5.04 (s, 2H), 4.72 (s, 1H), 4.55 (dd, *J* = 9.4, 4.8 Hz, 1H), 4.20-4.10 (m, 2H), 3.97 (s, 6H), 3.89 (s, 3H), 2.89 (ddd, *J* = 23.0, 13.6, 7.1 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 153.7, 153.2, 153.0, 141.7, 139.7, 138.9, 137.2, 136.4, 129.4, 128.7, 128.6, 127.9, 127.4, 127.0, 125.5, 116.0, 115.9, 103.0, 97.9, 70.5, 61.0, 59.9, 57.3, 56.3, 33.7, 14.4. HRMS (ESI) calcd for $C_{36}H_{37}N_2O_7$ $[M+H]^+$: 609.2596; Found: 609.2594. HPLC analysis [Daicel Chiralpak IC-3 column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 40.142 min, t_R (major) = 44.565 min, 73% ee. $[\alpha]^{20}_D$ = -43.204 (c = 1.00, CH_2Cl_2).



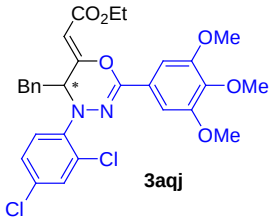
(*Z*)-Ethyl-2-(5-benzyl-4-(2,4-difluorophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3apj): colorless oil (99.2 mg; 92% yield). ^{1}H NMR (500 MHz, $CDCl_3$)



3apj

δ7.60 (td, *J* = 9.3, 5.9 Hz, 1H), 7.38 (s, 2H), 7.26-7.16 (m, 3H), 7.08-7.00 (m, 2H), 6.99-6.76 (m, 2H), 4.75 (s, 1H), 4.59-4.49 (m, 1H), 4.22-4.10 (m, 2H), 3.99 (s, 6H), 3.92 (s, 3H), 2.92-2.85 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.0, 158.7 (dd, *J* = 245.8, 11.5 Hz), 153.6, 153.2, 151.6 (d, *J* = 12.0 Hz), 143.6, 140.1, 136.1, 130.4 (dd, *J* = 8.2, 3.4 Hz), 129.3, 128.5, 126.9, 125.1, 123.5 (dd, *J* = 9.2, 3.0 Hz), 111.6 (dd, *J* = 22.0, 3.5 Hz), 104.8 (dd, *J* = 26.3, 22.1 Hz), 103.2, 98.3, 61.0, 59.9, 59.4, 59.3, 56.3, 34.0, 14.3. ^{19}F NMR (376 MHz, $CDCl_3$) δ -116.8, -119.3. HRMS (ESI) calcd for $C_{29}H_{29}F_2N_2O_6$ $[M+H]^+$: 539.1988; Found: 539.1988. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 99/1, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 30.227 min, t_R (minor) = 33.246 min, 75% ee. $[\alpha]^{20}_D$ = -58.692 (c = 1.00, CH_2Cl_2).

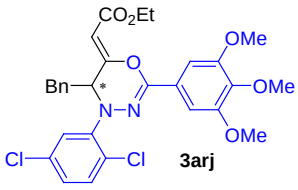
(*Z*)-Ethyl-2-(5-benzyl-4-(2,4-dichlorophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3aqj): colorless oil (106.4 mg; 93% yield). ^{1}H NMR (400 MHz, $CDCl_3$)



3aqj

δ7.57 (d, *J* = 8.7 Hz, 1H), 7.42 (d, *J* = 2.3 Hz, 1H), 7.37 (s, 2H), 7.29 (dd, *J* = 8.7, 2.3 Hz, 1H), 7.25-7.17 (m, 3H), 7.03-6.94 (m, 2H), 5.29 (s, 1H), 4.74 (s, 1H), 4.63 (dd, *J* = 9.7, 5.0 Hz, 1H), 4.18 (d, *J* = 7.2 Hz, 2H), 3.98 (s, 6H), 3.92 (s, 3H), 2.91-2.75 (m, 2H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.0, 153.7, 153.2, 144.4, 142.1, 140.2, 136.0, 130.4, 130.2, 129.2, 128.6, 127.9, 126.9, 126.6, 126.4, 125.0, 103.2, 98.1, 61.0, 59.9, 58.6, 56.3, 32.7, 14.3. HRMS (ESI) calcd for $C_{29}H_{29}Cl_2N_2O_6$ $[M+H]^+$: 571.1397; Found: 571.1396. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 98/2, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) =10.989 min, t_R (major) = 15.664 min, 75% ee. $[\alpha]^{20}_D$ = -96.001 (c = 1.00, CH_2Cl_2).

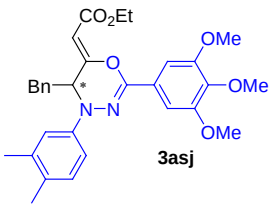
(*Z*)-Ethyl-2-(5-benzyl-4-(2,5-dichlorophenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3arj): yellow oil (87.5 mg; 77% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37



3arj

(s, 2H), 7.30-7.20 (m, 5H), 7.17-7.11 (m, 2H), 6.91 (dd, *J* = 8.9, 2.8 Hz, 1H), 4.86 (s, 1H), 4.58 (dd, *J* = 8.4, 5.8 Hz, 1H), 4.17 (qd, *J* = 7.1, 1.5 Hz, 2H), 3.98 (s, 6H), 3.90 (s, 3H), 3.01-2.80 (m, 2H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.9, 153.3, 152.3, 144.0, 142.7, 140.2, 135.7, 133.1, 130.6, 129.4, 128.8, 127.3, 124.8, 123.5, 115.6, 113.0, 103.4, 98.7, 61.0, 60.1, 56.3, 56.1, 34.7, 14.3. HRMS (ESI) calcd for $C_{29}H_{29}Cl_2N_2O_6$ $[M+H]^+$: 571.1397; Found: 571.1392. HPLC analysis [Daicel Chiralpak IC-3 column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 20.453 min, t_R (major) = 28.365 min, 74% ee. $[\alpha]^{20}_D$ = -77.265 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl-2-(5-benzyl-4-(3,4-dimethylphenyl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3asj): yellow oil (92.1 mg; 87% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ

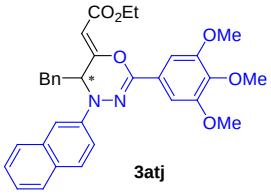


3asj

7.38 (s, 2H), 7.23 (ddd, *J* = 32.7, 19.4, 7.3 Hz, 5H), 7.08 (d, *J* = 8.2 Hz, 1H), 7.00 (s, 1H), 6.94 (dd, *J* = 8.2, 2.0 Hz, 1H), 4.73 (s, 1H), 4.60 (dd, *J* = 9.4, 4.7 Hz, 1H), 4.18 (d, *J* = 3.3 Hz, 2H), 3.98 (s, 6H), 3.89 (s, 3H), 2.91 (ddd, *J* = 23.0, 13.6, 7.1 Hz, 2H), 2.26 (s, 3H), 2.21 (s, 3H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.2, 153.2, 152.9, 142.6, 141.6,

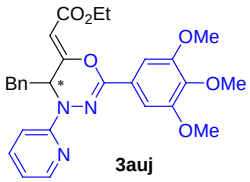
139.7, 137.5, 136.5, 130.3, 129.5, 129.4, 128.7, 127.0, 125.6, 115.9, 111.8, 103.1, 97.9, 61.0, 59.9, 56.7, 56.3, 33.9, 20.3, 18.9, 14.4. HRMS (ESI) calcd for $C_{31}H_{35}N_2O_6$ $[M+H]^+$: 531.2490; Found: 531.2487. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 6.696 min, t_R (minor) = 18.255 min, 76% ee. $[\alpha]^{20}_D$ = -98.419 (c = 1.00, CH_2Cl_2).

(*Z*)-Ethyl-2-(5-benzyl-4-(naphthalen-2-yl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3atj): yellow solid (83.7 mg; 76% yield), m.p. 65.2-66.3 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.73 (m, 3H), 7.65 (dd, *J* = 9.0, 2.1 Hz, 1H), 7.52-7.45 (m, 3H), 7.44-7.22 (m, 8H), 4.92-4.82 (m, 2H), 4.25 (d, *J* = 2.9 Hz, 2H), 4.05 (s, 6H), 3.96 (s, 3H), 3.05 (ddd, *J* = 22.9, 13.6, 7.1 Hz, 2H), 1.30 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.1, 153.3, 152.7, 142.3, 142.2, 139.9, 136.3, 134.2, 129.5, 129.4, 129.0, 128.7, 127.6, 127.2, 126.9, 126.6, 125.4, 123.8, 116.2, 108.8, 103.2, 98.4, 61.0, 60.0, 56.5, 56.4, 34.3, 14.4. HRMS (ESI) calcd for $C_{44}H_{39}N_2O_6$ $[M+H]^+$: 691.2803; Found: 691.2804. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/ 2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 12.132 min, t_R (minor) = 20.642 min, 87% ee. $[\alpha]^{20}_D$ = -54.108 (c = 1.00, CH_2Cl_2).



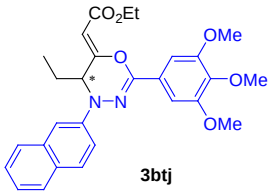
3atj

(*Z*)-Ethyl-2-(5-benzyl-4-(pyridin-2-yl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3auj): yellow oil (74.3 mg; 74% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.25 (d, *J* = 3.9 Hz, 1H), 7.71-7.58 (m, 1H), 7.52 (d, *J* = 8.5 Hz, 1H), 7.42 (s, 2H), 7.28-7.26 (m, 4H), 7.21 (dd, *J* = 9.0, 4.8 Hz, 1H), 6.83 (dd, *J* = 6.6, 5.4 Hz, 1H), 5.68 (dd, *J* = 9.8, 4.4 Hz, 1H), 4.81 (s, 1H), 4.24-4.09 (m, 2H), 4.01 (s, 6H), 3.93 (s, 3H), 3.05 (dd, *J* = 13.2, 4.4 Hz, 1H), 2.87 (dd, *J* = 13.1, 9.9 Hz, 1H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 164.1, 155.3, 153.5, 153.2, 147.3, 142.1, 140.0, 137.7, 136.6, 129.6, 128.4, 126.8, 125.2, 115.7, 109.0, 103.3, 98.7, 61.0, 59.8, 56.3, 52.5, 35.3, 14.3. HRMS (ESI) calcd for $C_{28}H_{30}N_3O_6$ $[M+H]^+$: 504.2129; Found: 504.2128. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 280 nm]: t_R (major) = 6.267 min, t_R (minor) = 8.909 min, 82% ee. $[\alpha]^{20}_D$ = -48.276 (c = 1.00, CH_2Cl_2).



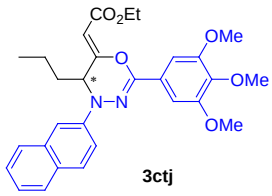
3auj

(*Z*)-Ethyl-2-(5-ethyl-4-(naphthalen-2-yl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3btj): colorless oil (75.0 mg, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 9.0 Hz, 1H), 7.76 (t, *J* = 7.4 Hz, 2H), 7.68 (dd, *J* = 9.1, 2.0 Hz, 1H), 7.47-7.39 (m, 3H), 7.39-7.29 (m, 2H), 5.25 (s, 1H), 4.58 (dd, *J* = 9.3, 5.3 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 4.00 (s, 6H), 3.92 (s, 3H), 1.80 (dd, *J* = 14.8, 7.4 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H), 1.07 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.3, 153.5, 153.2, 142.4, 141.7, 139.7, 134.2, 129.3, 128.9, 127.6, 126.8, 126.6, 125.4, 123.7, 116.1, 108.3, 103.0, 97.9, 61.0, 60.1, 56.3, 55.6, 20.6, 14.4, 10.8. HRMS (ESI) calcd for $C_{28}H_{31}N_2O_6$ $[M+H]^+$: 491.2177; Found: 491.2176. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/ 2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 11.093 min, t_R (minor) = 19.962 min, 75% ee. $[\alpha]^{20}_D$ = -54.108 (c = 1.00, CH_2Cl_2).



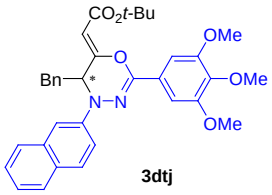
3btj

(*Z*)-Ethyl-2-(4-(naphthalen-2-yl)-5-propyl-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3ctj): colorless oil (73.9 mg, 73% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (d, *J* = 9.0 Hz, 1H), 7.76 (t, *J* = 7.5 Hz, 2H), 7.70-7.64 (m, 1H), 7.46-7.40 (m, 3H), 7.39-7.30 (m, 2H), 5.24 (s, 1H), 4.67 (dd, *J* = 9.2, 5.2 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 4.00 (s, 6H), 3.91 (s, 3H), 1.72 (dd, *J* = 9.4, 5.7 Hz, 2H), 1.55-1.39 (m, 2H), 1.33 (t, *J* = 7.1 Hz, 4H), 0.96 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 164.4, 153.9, 153.2, 142.4, 141.6, 139.8, 134.2, 129.3, 128.9, 127.6, 126.8, 126.6, 125.4, 123.7, 116.1, 108.3, 103.0, 97.7, 61.0, 60.1, 56.3, 53.9, 29.3, 19.5, 14.4, 13.7. HRMS (ESI) calcd for $C_{29}H_{33}N_2O_6$ $[M+H]^+$: 505.2333; Found: 505.2333. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 9.362 min, t_R (minor) = 19.838 min, 82% ee. $[\alpha]^{20}_D$ = -101.070 (c = 1.00, CH_2Cl_2).



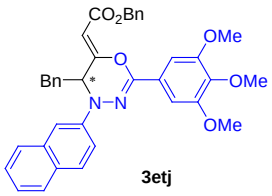
3ctj

(*Z*)-*tert*-Butyl-2-(5-benzyl-4-(naphthalen-2-yl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3dtj): colorless oil (84.0 mg, 72% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ7.83- 7.69 (m, 3H), 7.57 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.47-7.42 (m, 3H), 7.35-7.28 (m, 4H), 7.25-7.22 (m, 3H), 4.83 (s, 1H), 4.79 (dd, *J* = 8.8, 5.2 Hz, 1H), 4.02 (s, 6H), 3.93 (s, 3H), 3.06 (dd, *J* = 13.7, 5.2 Hz, 1H), 2.97 (dd, *J* = 13.7, 8.9 Hz, 1H), 1.47 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.4, 153.2, 151.7, 142.4, 142.3, 139.8, 136.5, 134.2, 129.5, 129.2, 128.9, 128.7, 127.6, 127.1, 126.9, 126.6, 125.5, 123.7, 116.1, 108.7, 103.3, 100.0, 80.2, 61.0, 56.5, 56.4, 34.6, 28.2. HRMS (ESI) calcd for $C_{35}H_{37}N_2O_6$ $[M+H]^+$: 581.2646; Found: 581.2645. HPLC analysis [Daicel Chiralpak AD-H column, n-hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 4.862 min, t_R (minor) = 10.439 min, 76% ee. $[\alpha]^{20}_D$ = -47.172 (c = 1.00, CH_2Cl_2).



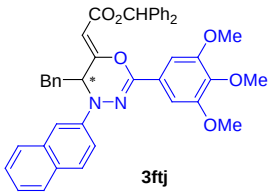
3dtj

(*Z*)-Benzyl-2-(5-benzyl-4-(naphthalen-2-yl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3etj): colorless oil (100.0 mg, 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ7.88-7.72 (m, 3H), 7.65 (d, *J* = 9.0 Hz, 1H), 7.49-7.23 (m, 15H), 5.21 (s, 2H), 4.93 (d, *J* = 1.8 Hz, 1H), 4.89-4.83 (m, 1H), 3.97 (dd, *J* = 14.7, 1.8 Hz, 9H), 3.10 (dd, *J* = 13.6, 3.2 Hz, 1H), 3.06-2.95 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 163.8, 153.2, 142.2, 142.1, 139.9, 136.2, 134.2, 129.5, 129.4, 129.0, 128.7, 128.6, 128.5, 128.1, 127.8, 127.7, 127.2, 126.9, 126.8, 126.6, 125.2, 123.8, 116.1, 108.9, 103.2, 97.9, 65.6, 61.0, 56.4, 56.2, 34.2. HRMS (ESI) calcd for $C_{38}H_{35}N_2O_6$ $[M+H]^+$: 615.2490; Found: 615.2487. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 85/15, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 20.211 min, t_R (minor) = 27.733 min, 81% ee. $[\alpha]^{20}_D$ = -77.794 (c = 1.00, CH_2Cl_2).



3etj

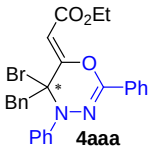
(*Z*)-Benzhydryl-2-(5-benzyl-4-(naphthalen-2-yl)-2-(3,4,5-trimethoxyphenyl)-4,5-dihydro-6*H*-1,3,4-oxadiazin-6-ylidene)acetate (3ftj): yellow oil (113.6 mg, 82% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ7.88-7.75 (m, 3H), 7.65 (dd, *J* = 9.0, 2.4 Hz, 1H), 7.51-7.46 (m, 1H), 7.43 (s, 3H), 7.40-7.23 (m, 16H), 6.99 (s, 1H), 5.00 (s, 1H), 4.88 (dd, *J* = 9.3, 5.0 Hz, 1H), 3.94 (s, 9H), 3.12 (dd, *J* = 13.6, 4.9 Hz, 1H), 2.99 (dd, *J* = 13.6, 9.3 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8, 153.4, 153.3,



3ftj

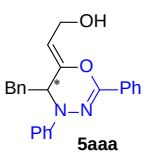
142.2, 140.4, 140.3, 139.9, 136.3, 134.2, 129.5, 129.4, 129.0, 128.7, 128.5, 128.4, 127.9, 127.8, 127.7, 127.2, 127.1, 127.0, 126.6, 125.2, 123.8, 116.1, 108.9, 103.2, 98.1, 61.0, 56.5, 56.2, 34.2. HRMS (ESI) calcd for $C_{44}H_{39}N_2O_6$ $[M+H]^+$: 691.2803; Found: 691.2804. HPLC analysis [Daicel Chiralpak ID-3 column, n-hexane/2-propanol = 80/20, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (minor) = 13.188 min, t_R (major) = 15.296 min, 74% ee. $[\alpha]^{20}_D$ = -68.326 (c = 1.00, CH_2Cl_2).

(Z)-ethyl 2-(5-benzyl-5-bromo-4,5-dihydro-2,4-diphenyl-1,3,4-oxadiazin-6-ylidene)acetate (4aaa): yellow oil (30.4 mg, 62% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (dd, *J* = 6.5, 2.8 Hz, 2H), 7.50-7.39 (m, 3H), 7.31 (d, *J* = 8.9 Hz, 2H), 7.20-7.04 (m, 7H), 6.48 (t, *J* = 7.0 Hz, 1H), 5.63 (s, 1H), 4.15-4.02 (m, 2H), 2.98 (ddd, *J* = 44.1, 13.5, 7.0 Hz, 2H), 1.23 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.2, 158.5, 143.9, 142.4, 135.9, 131.8, 130.0, 129.9, 129.8, 128.5, 128.3, 126.8, 125.3, 115.6, 112.6, 98.2, 60.3, 50.9, 34.1, 14.3. HRMS (ESI) calcd for $C_{26}H_{24}BrN_2O_3$ $[M+H]^+$: 491.0965; Found: 491.0963. HPLC analysis [Daicel Chiralpak OD-H column, n-hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, λ = 254 nm]: t_R (major) = 4.034 min, t_R (minor) = 4.786 min, 76% ee. $[\alpha]^{20}_D$ = -76.251 (c = 1.00, CH_2Cl_2).



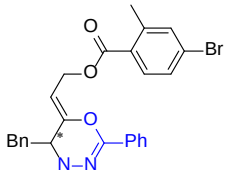
4aaa

(Z)-2-(5-benzyl-2,4-diphenyl-4,5-dihydro-6H-1,3,4-oxadiazin-6-ylidene)ethan-1-ol (5aaa): colorless oil (32.5 mg, 88% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, *J* = 7.4, 2.1 Hz, 2H), 7.51-7.19 (m, 13H), 6.98 (t, *J* = 6.8 Hz, 1H), 4.70 (dd, *J* = 10.3, 4.4 Hz, 1H), 4.57 (t, *J* = 7.0 Hz, 1H), 4.34 (dd, *J* = 6.6, 2.6 Hz, 2H), 3.03 (dd, *J* = 13.4, 4.3 Hz, 1H), 2.86 (dd, *J* = 13.4, 10.4 Hz, 1H), 1.20 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 144.9, 143.0, 141.2, 137.3, 130.8, 129.6, 129.6, 129.3, 128.5, 128.4, 126.9, 125.4, 120.4, 113.7, 107.4, 56.2, 56.0, 34.0. HRMS (ESI) calcd for $C_{24}H_{23}N_2O_2$ $[M+H]^+$: 371.1754; Found: 371.1755. HPLC conditions: Daicel Chiralpak AD-H column, n-hexane/2-propanol = 95/5, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (minor) =10.354 min, t_R (major) = 19.686 min, 76% ee. $[\alpha]^{20}_D$ = -54.325(c = 1.00, CH_2Cl_2).



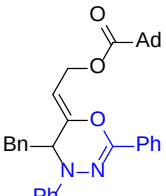
5aaa

(Z)-2-(5-benzyl-2,4-diphenyl-4,5-dihydro-6H-1,3,4-oxadiazin-6-ylidene)ethyl 4-bromo-2-methylbenzoate(6aaa): colorless oil (44.2 mg, 78% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04 (dd, *J* = 7.6, 1.7 Hz, 2H), 7.75 (d, *J* = 8.4 Hz, 1H), 7.50-7.33 (m, 9H), 7.22-7.12 (m, 5H), 6.97 (t, *J* = 6.8 Hz, 1H), 5.03 (dd, *J* = 12.3, 7.5 Hz, 1H), 4.91 (dd, *J* = 12.3, 7.1 Hz, 1H), 4.71 (dd, *J* = 10.5, 4.1 Hz, 1H), 4.63 (t, *J* = 7.3 Hz, 1H), 2.99 (dd, *J* = 13.5, 4.0 Hz, 1H), 2.86 (dd, *J* = 13.4, 10.6 Hz, 1H), 2.56 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.6, 145.2, 144.8, 142.6, 141.2, 137.0, 134.6, 132.3, 130.7, 129.7, 129.6, 129.4, 129.0, 128.9, 128.5, 128.5, 128.3, 126.8, 125.4, 120.5, 113.7, 102.3, 57.9, 56.2, 33.9, 21.7. HRMS (ESI) calcd for $C_{32}H_{28}BrN_2O_3$ $[M+H]^+$: 567.1278; Found: 567.1283. HPLC conditions: Daicel Chiralpak ID-3 column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (minor) =5.204 min, t_R (major) = 5.978 min, 76% ee. $[\alpha]^{20}_D$ = -95.420(c = 1.00, CH_2Cl_2).



6aaa

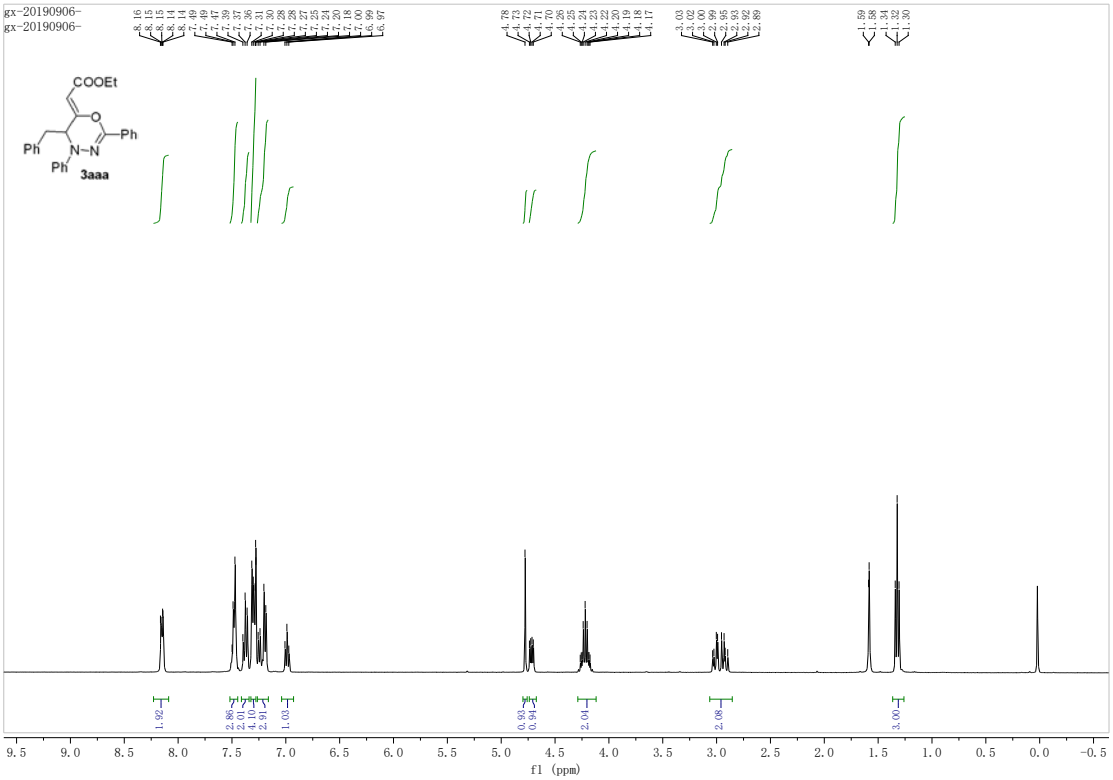
(Z)-2-(5-benzyl-2,4-diphenyl-4,5-dihydro-6H-1,3,4-oxadiazin-6-ylidene)ethyl (3s)-adamantane-1-carboxylate(7aaa): colorless oil (36.4 mg, 68% yield). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.09-7.99 (m, 2H), 7.53-7.17 (m, 13H), 6.98 (t, *J* = 6.7 Hz, 1H), 4.86- 4.65 (m, 3H), 4.55 (t, *J* = 6.5 Hz, 1H), 3.05-2.79 (m, 2H), 1.84 (m, 15H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.6, 144.86,

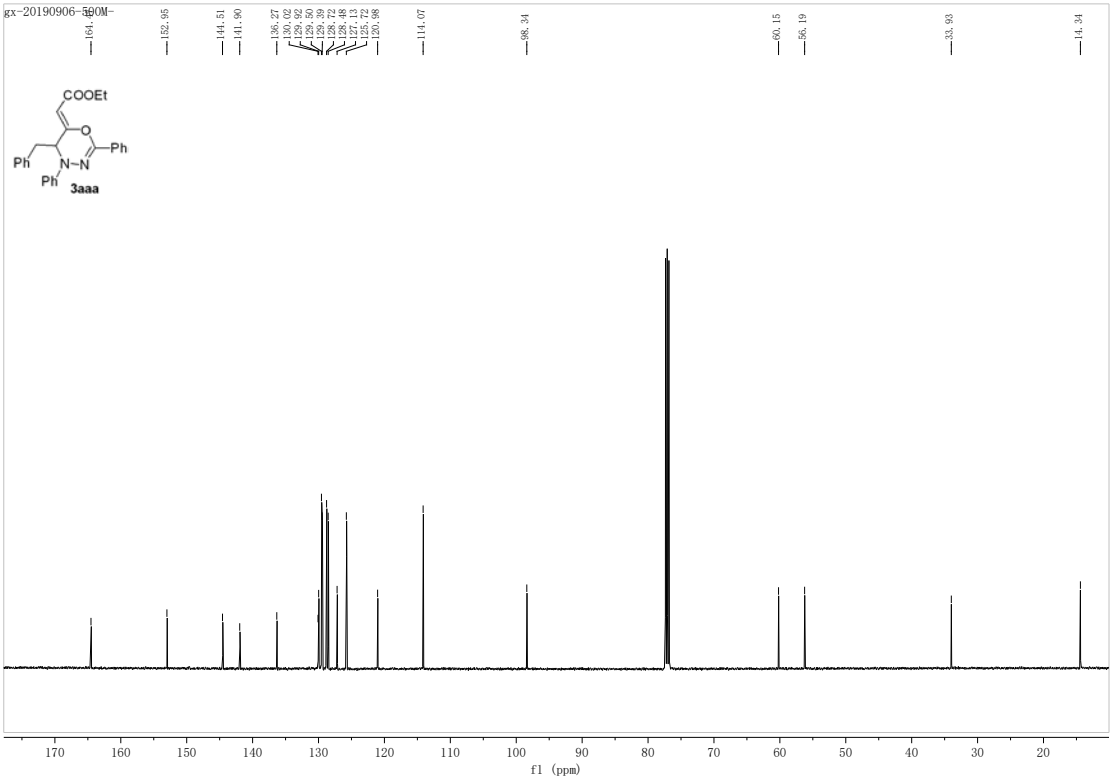


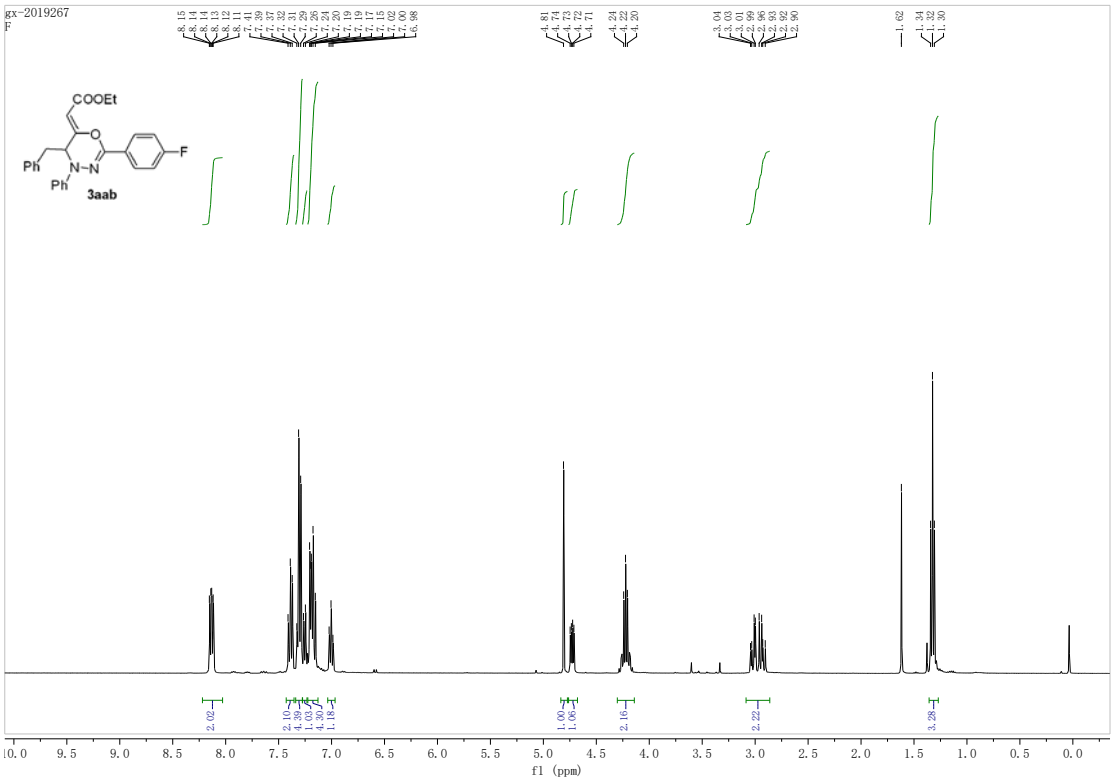
7aaa

144.6, 141.2, 137.1, 130.7, 129.6, 129.6, 129.4, 128.5, 128.4, 126.8, 125.4, 120.4, 113.7, 102.9, 57.3, 56.1, 40.7, 38.8, 36.5, 34.0, 27.9. HRMS (ESI) calcd for $C_{35}H_{37}N_2O_3$ $[M+H]^+$: 533.2799; Found: 533.2804. HPLC conditions: Daicel Chiralpak OD-H column, n-hexane/2-propanol = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 4.885 min, t_R (minor) = 13.151 min, 76% ee. $[\alpha]^{20}_D$ = -46.625(c = 1.00, CH_2Cl_2).

C: Copies of NMR Analysis



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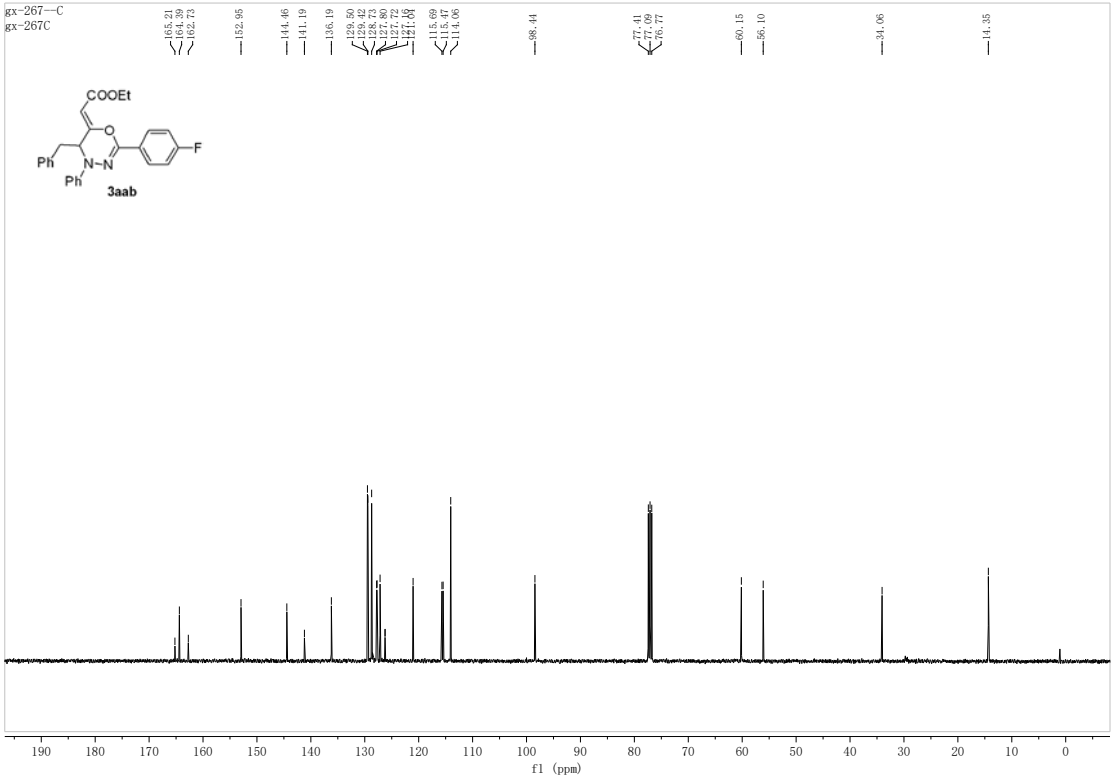
N-N
Ph
3aab
f1 (ppm)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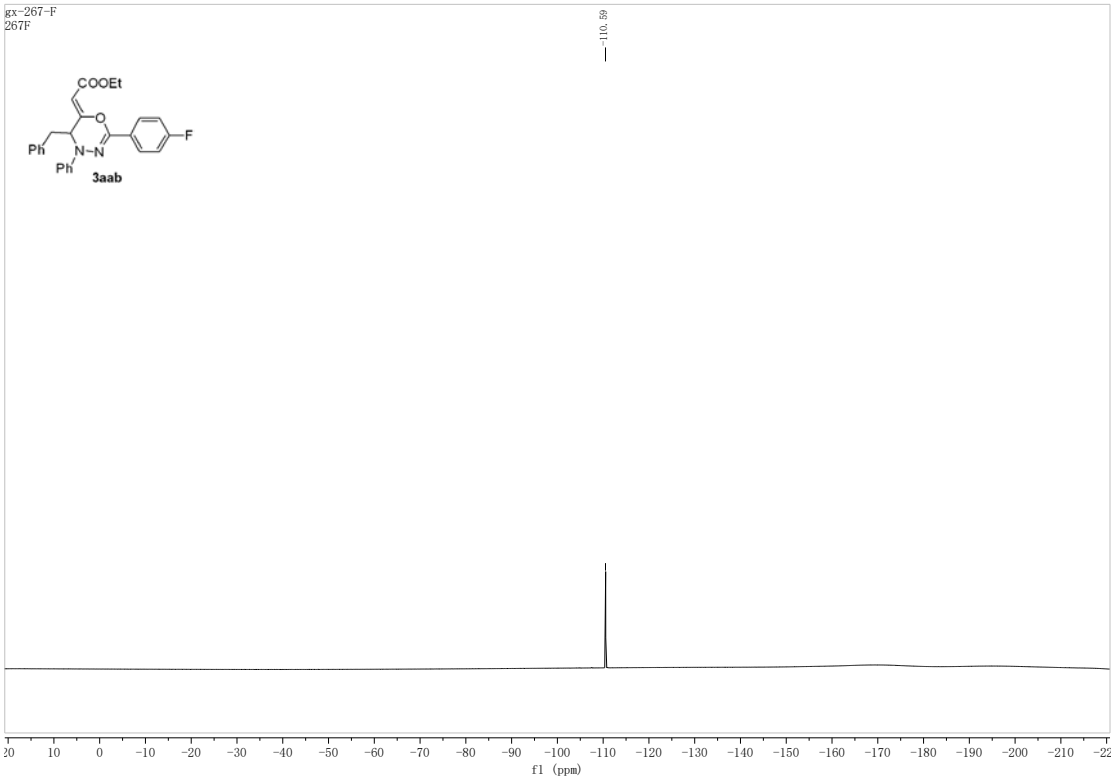
gx-267C
COOEt
F
Ph
N-N
Ph
3aab
f1 (ppm)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Ph
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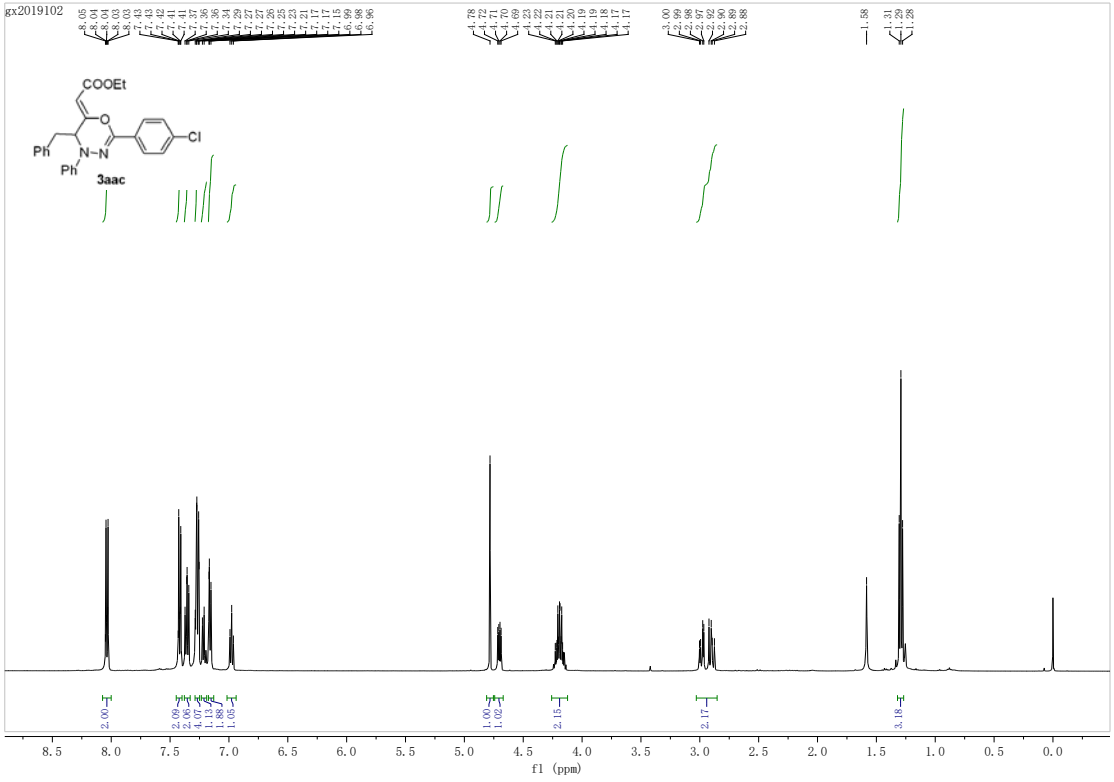
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Cl
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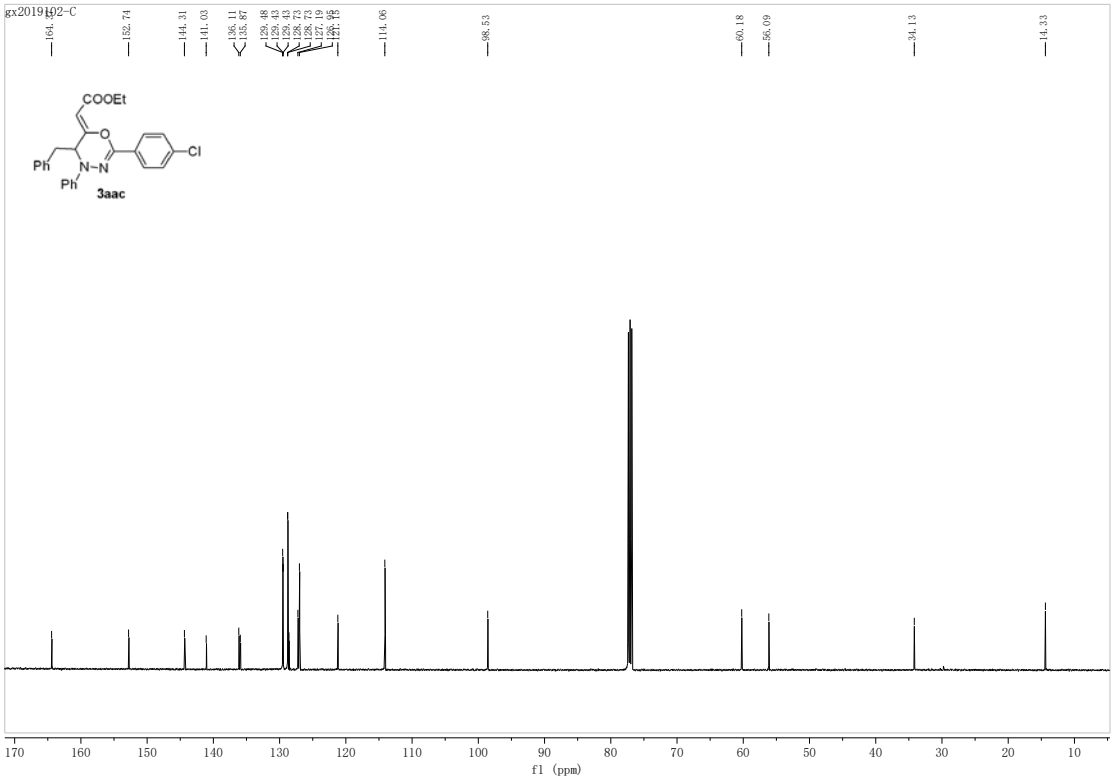
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14.33
COOEt
O
Ph
N-N
Ph
Cl
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100
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60
50
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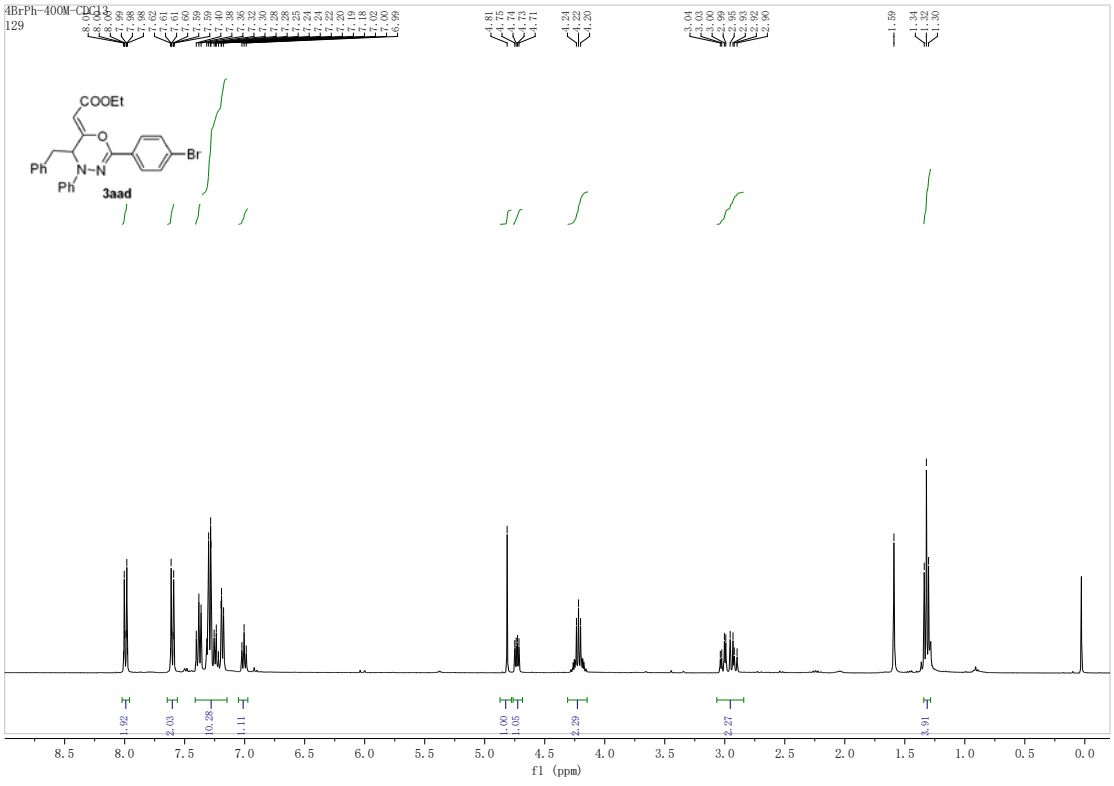
10
f1 (ppm)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7.28
7.25
7.24
7.22
7.20
7.19
7.18
7.02
7.00
6.99
4.81
4.75
4.74
4.73
4.71
4.24
4.22
4.20
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3.03
3.00
2.99
2.95
2.93
2.92
2.90
1.59
1.34
1.32
1.30
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O
Ph
N-N
Ph
Br
3aad
1.92
2.03
10.28
1.11
1.00
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2.27
3.91
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7.0
6.5
6.0
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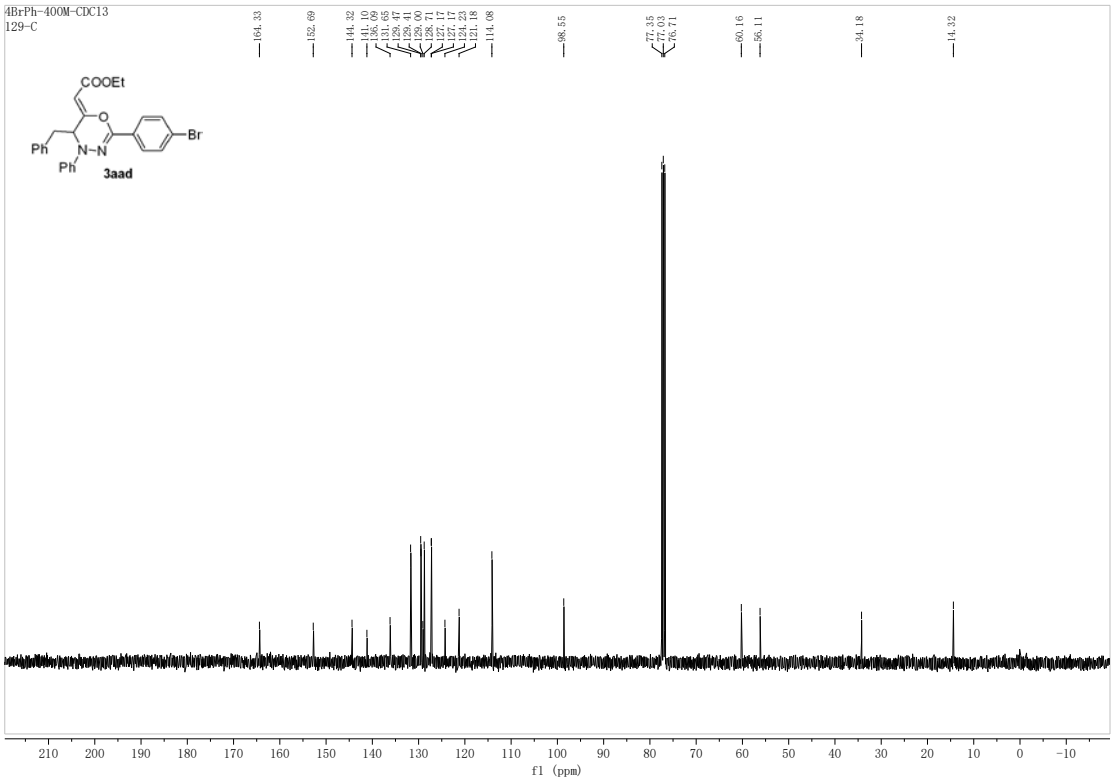
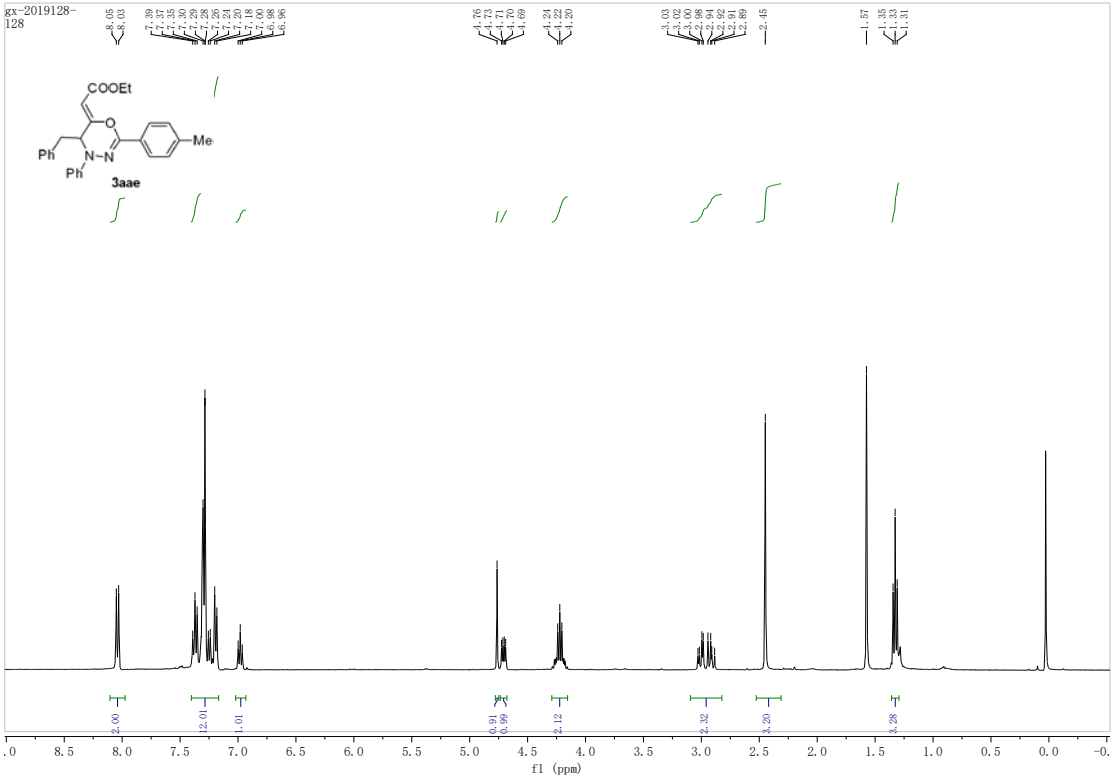
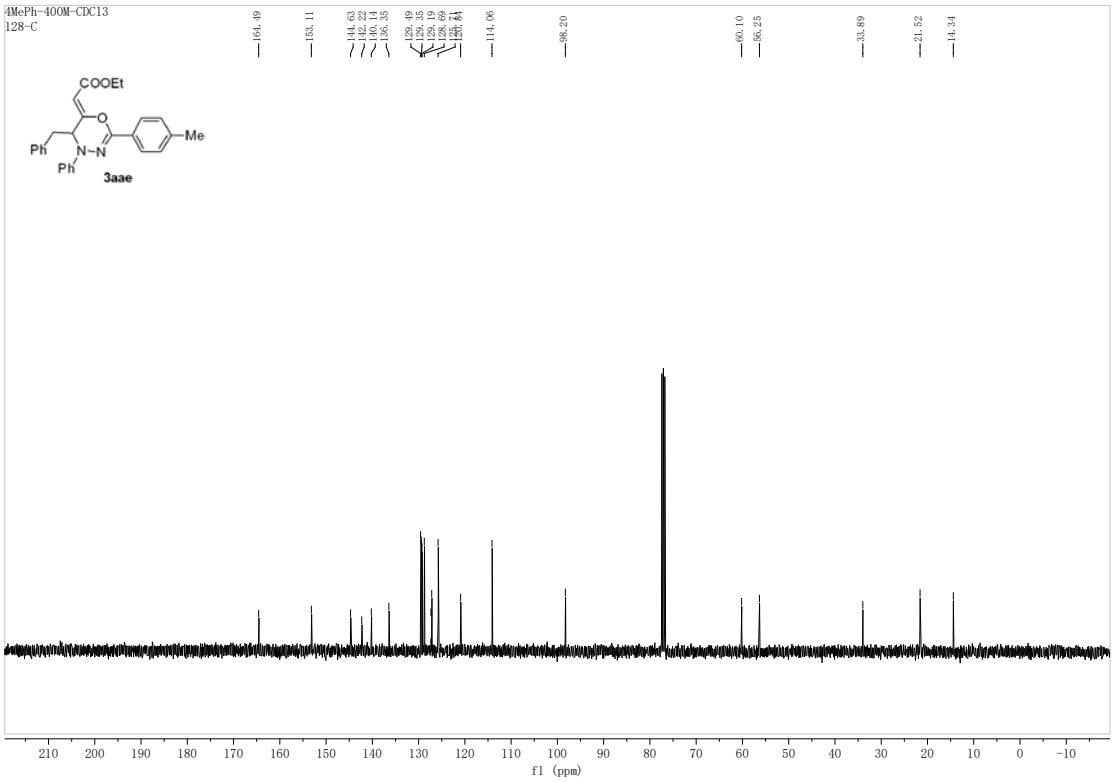
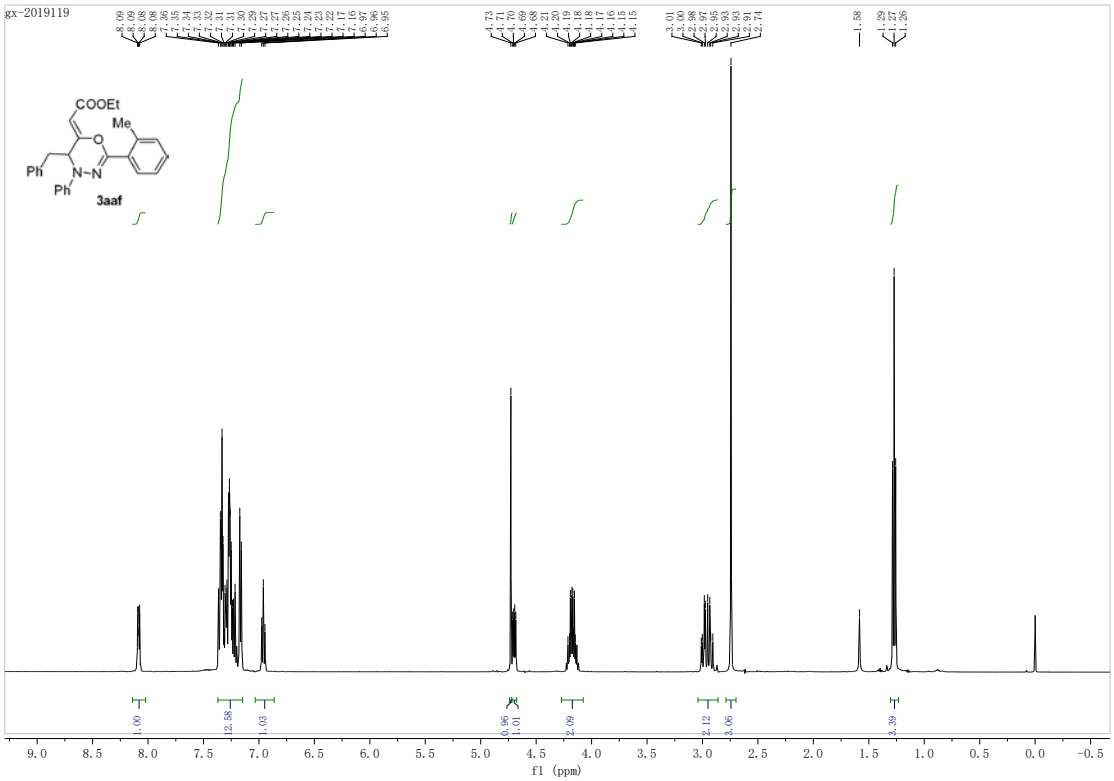
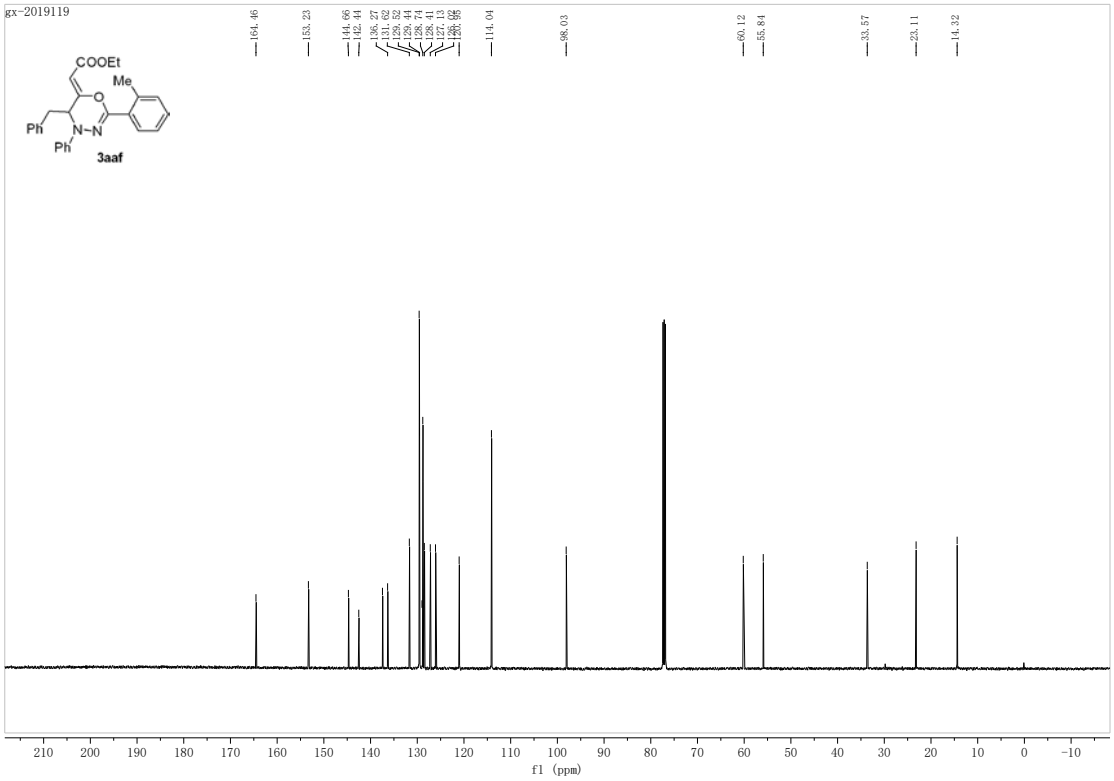
1.0
0.5
0.0
f1 (ppm)

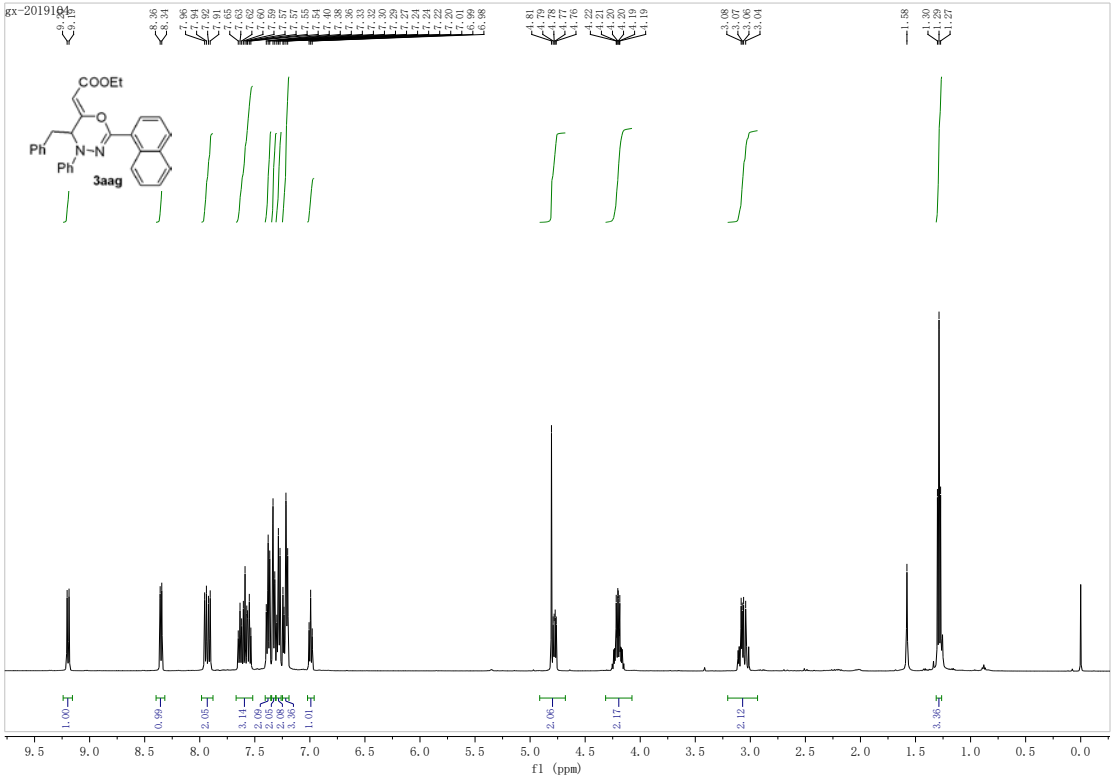

4BrPh-400M-CDC13
129-C
COOEt
Ph
N-N
Ph
O
Br
3aad
f1 (ppm)

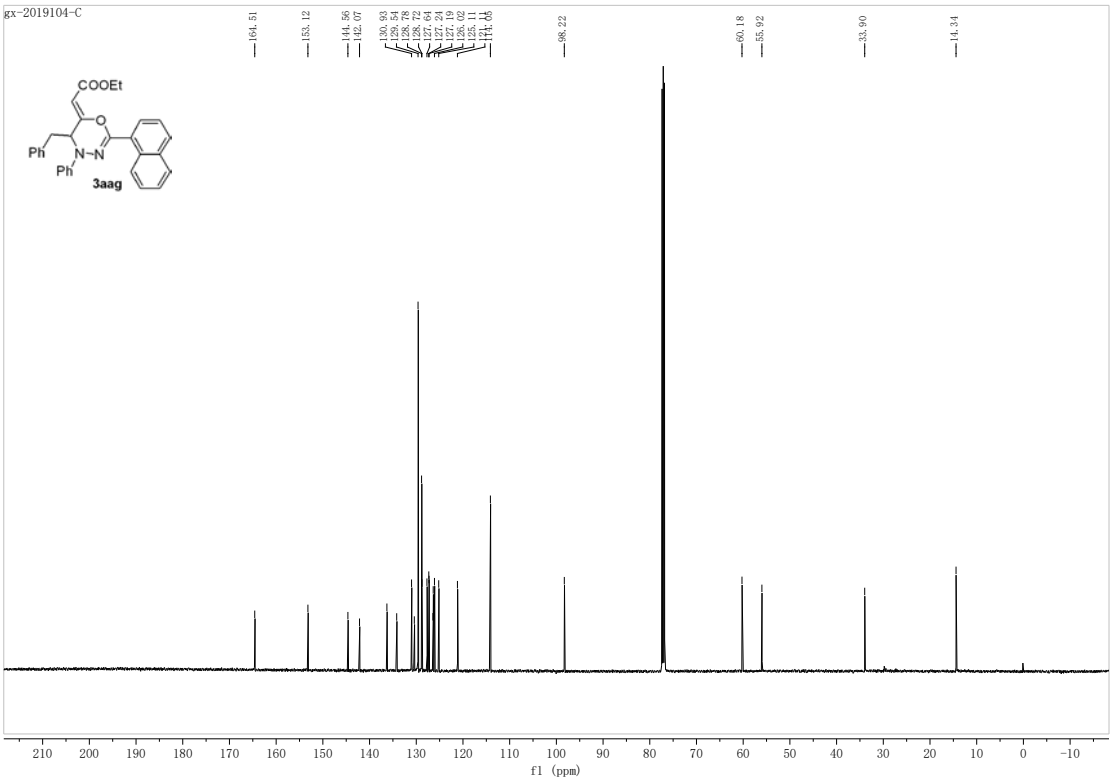

gx-2019128-
128
COOEt
Ph
N-N
Ph
O
Me
3aae
f1 (ppm)

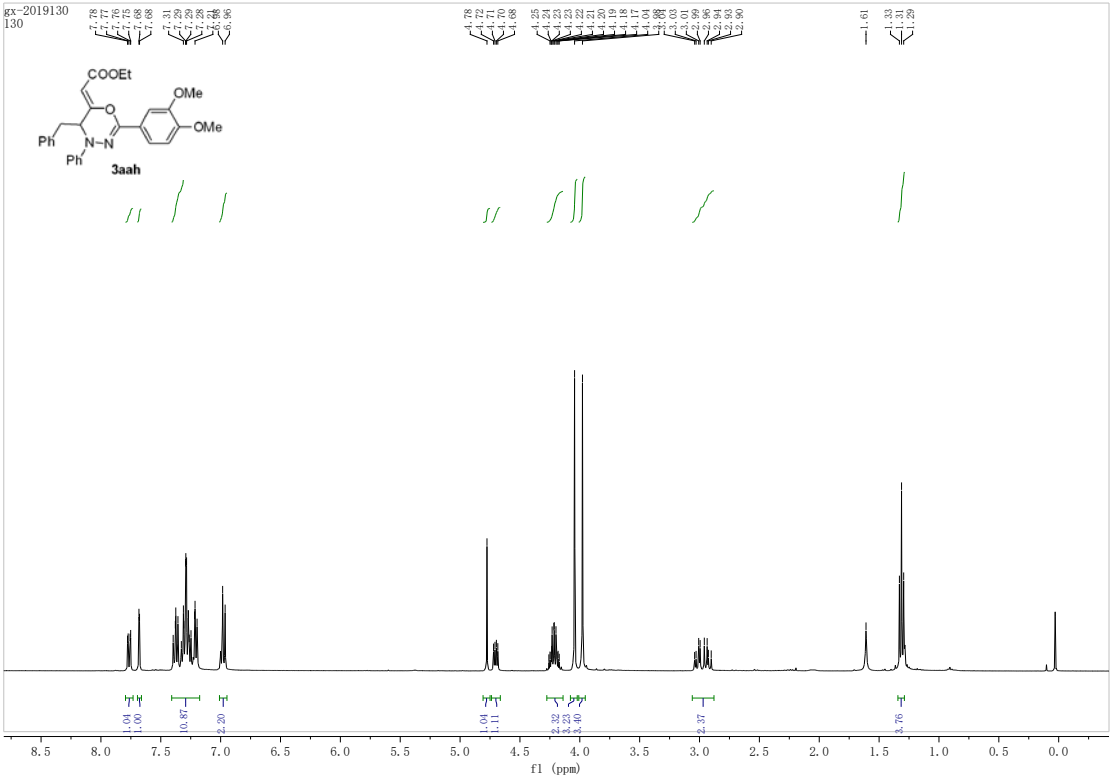

4MePh-400M-CDC13
128-C
164.49
153.11
144.63
142.22
140.14
136.35
129.49
129.35
129.19
128.69
114.06
98.20
60.10
56.25
33.89
21.52
14.34
COOEt
Me
Ph
N-N
Ph
3aae
f1 (ppm)

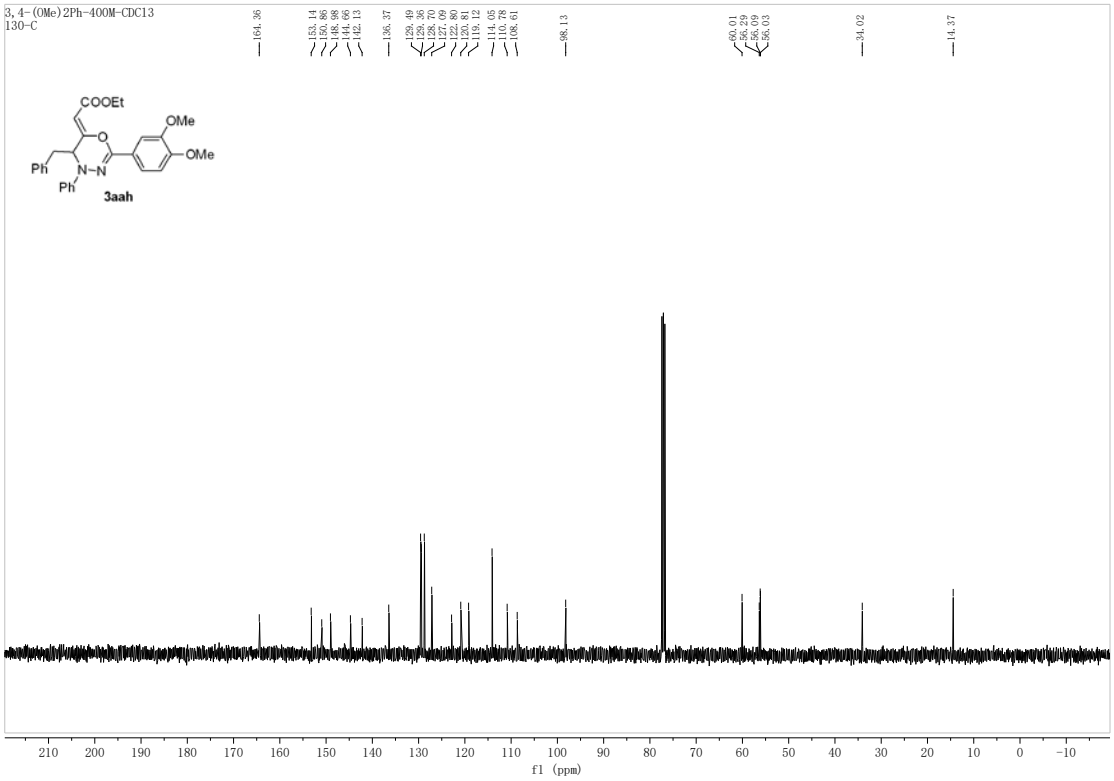

gx-2019119
COOEt
Me
Ph
N-N
Ph
3aaf
1.00
2.58
1.03
0.96
1.01
2.09
2.12
3.06
3.39
f1 (ppm)

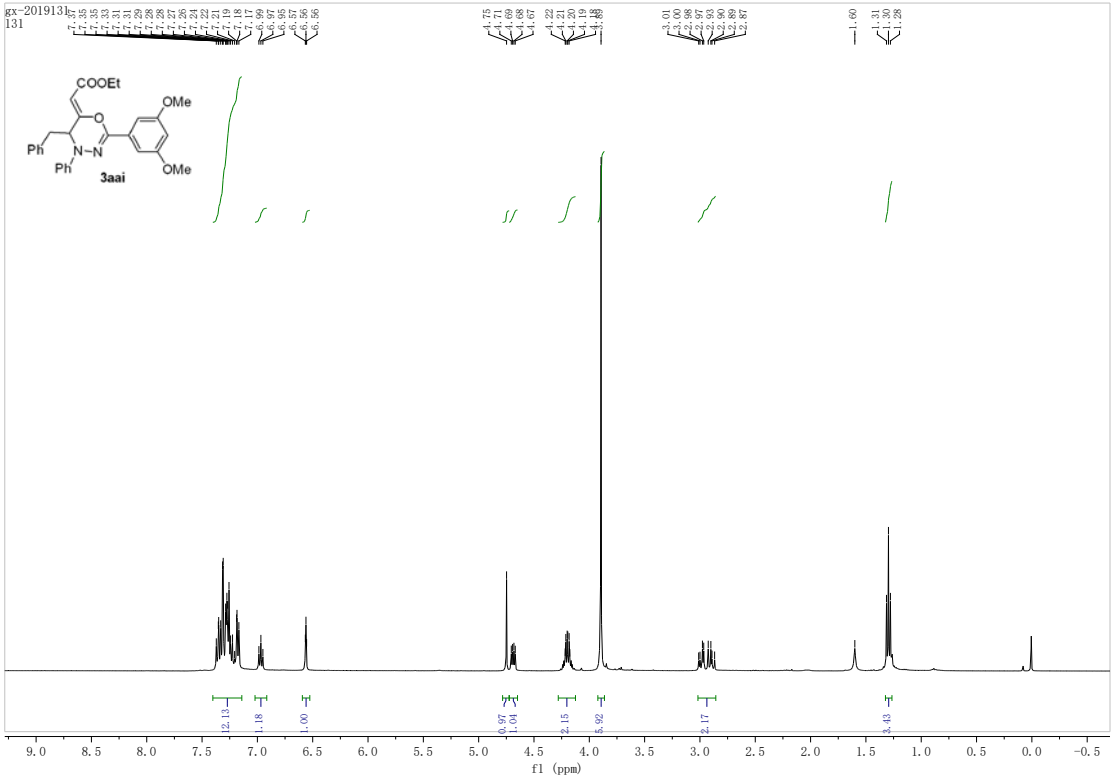

gx-2019119
COOEt
Me
Ph
N-N
Ph
3aaf
164.46
153.23
144.66
142.44
136.27
131.62
129.52
129.44
128.74
128.41
127.13
114.04
98.03
60.12
55.84
33.57
23.11
14.32
f1 (ppm)

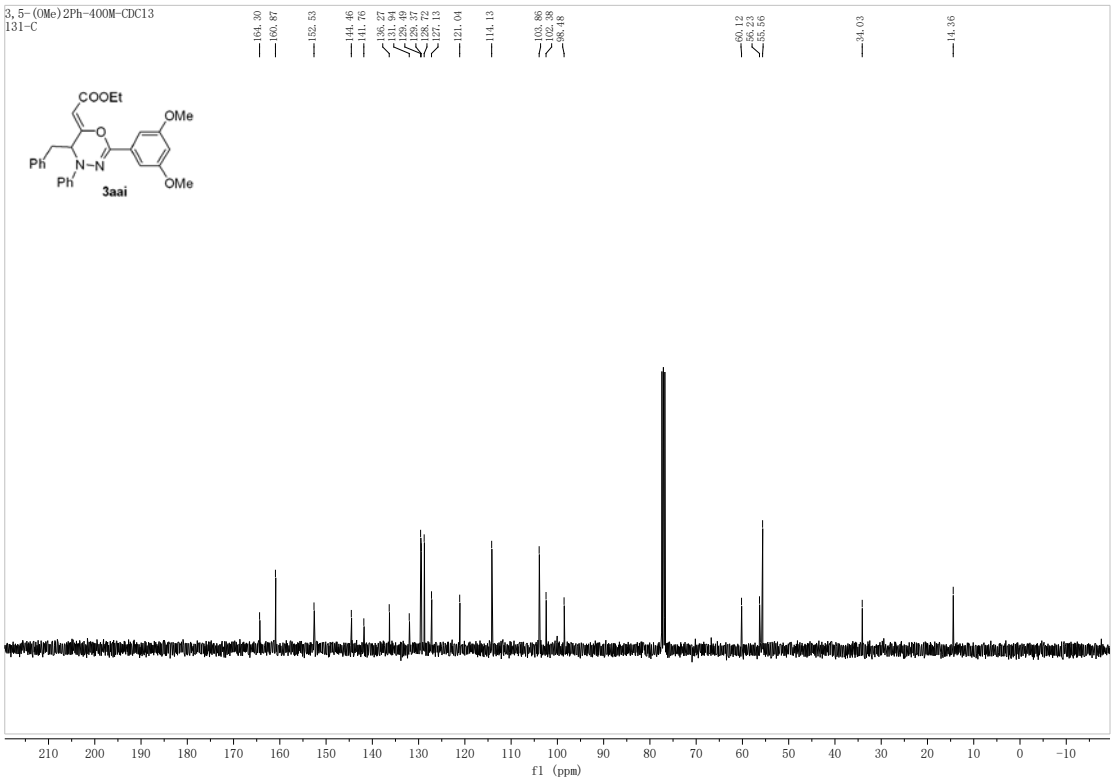

gx-2019104
COOEt
Ph
N-N
Ph
3aag
f1 (ppm)

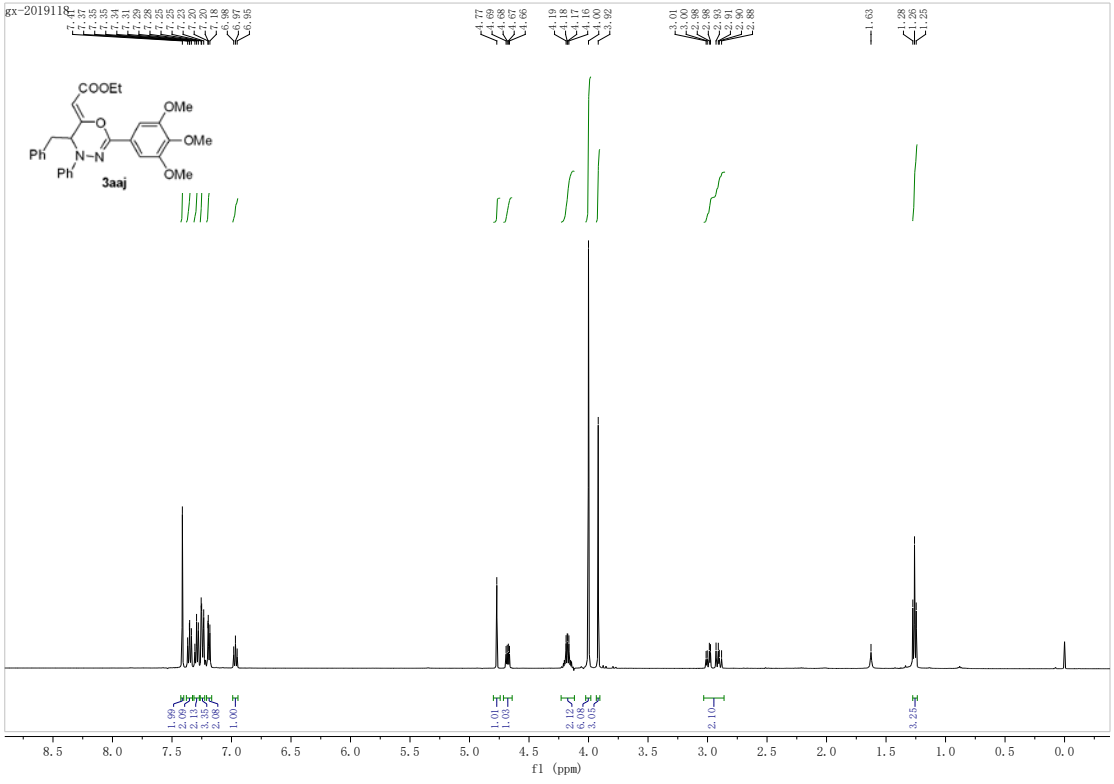

gx-2019104-C
COOEt
Ph
Ph
3aag
f1 (ppm)

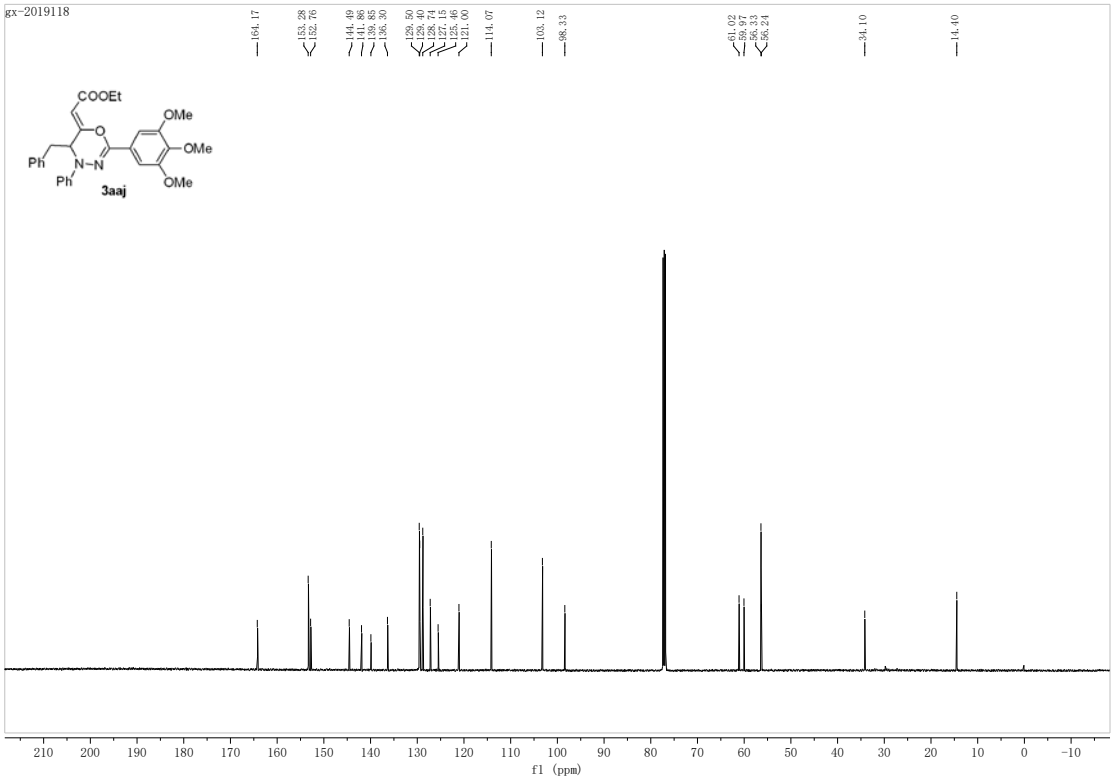

gx-2019130
130
COOEt
OMe
OMe
Ph
Ph
3aah
f1 (ppm)

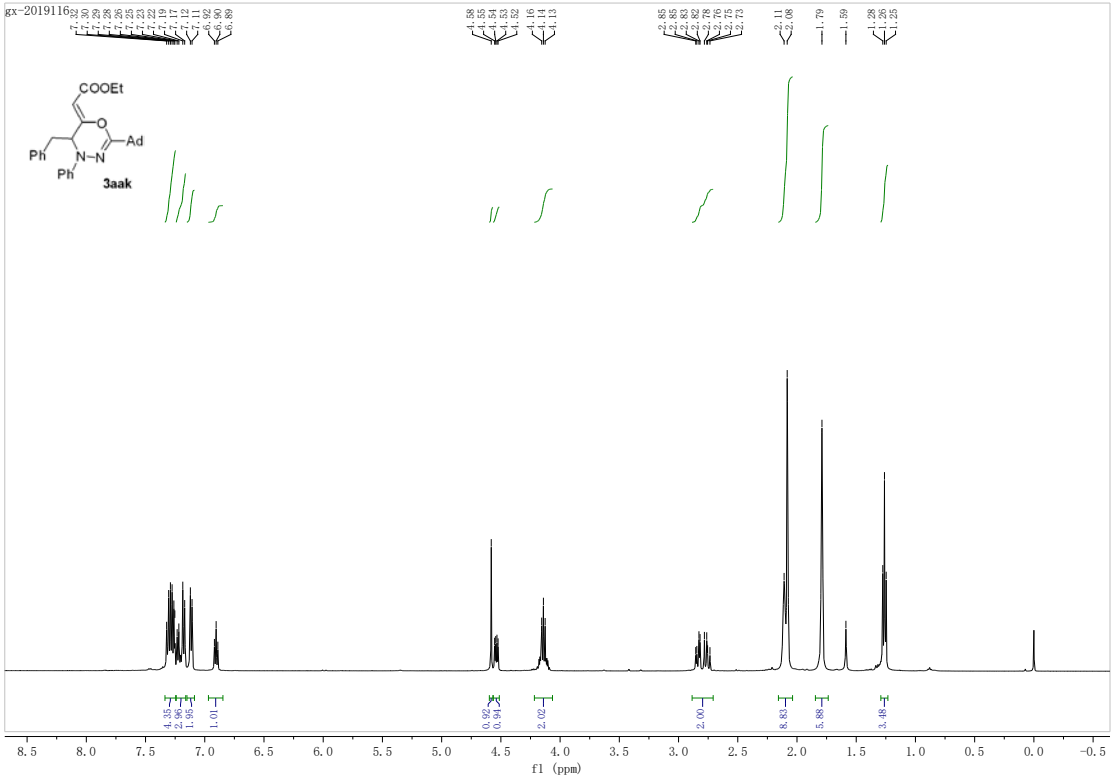

3,4-(OMe)2Ph-400M-CDC13
130-C
164.36
153.14
151.98
148.98
144.66
142.13
136.37
129.49
129.35
128.70
127.09
122.80
120.81
114.12
114.05
110.78
108.61
98.13
60.01
56.09
56.03
34.02
14.37
COOEt
OMe
OMe
Ph
N-N
Ph
3aah
f1 (ppm)

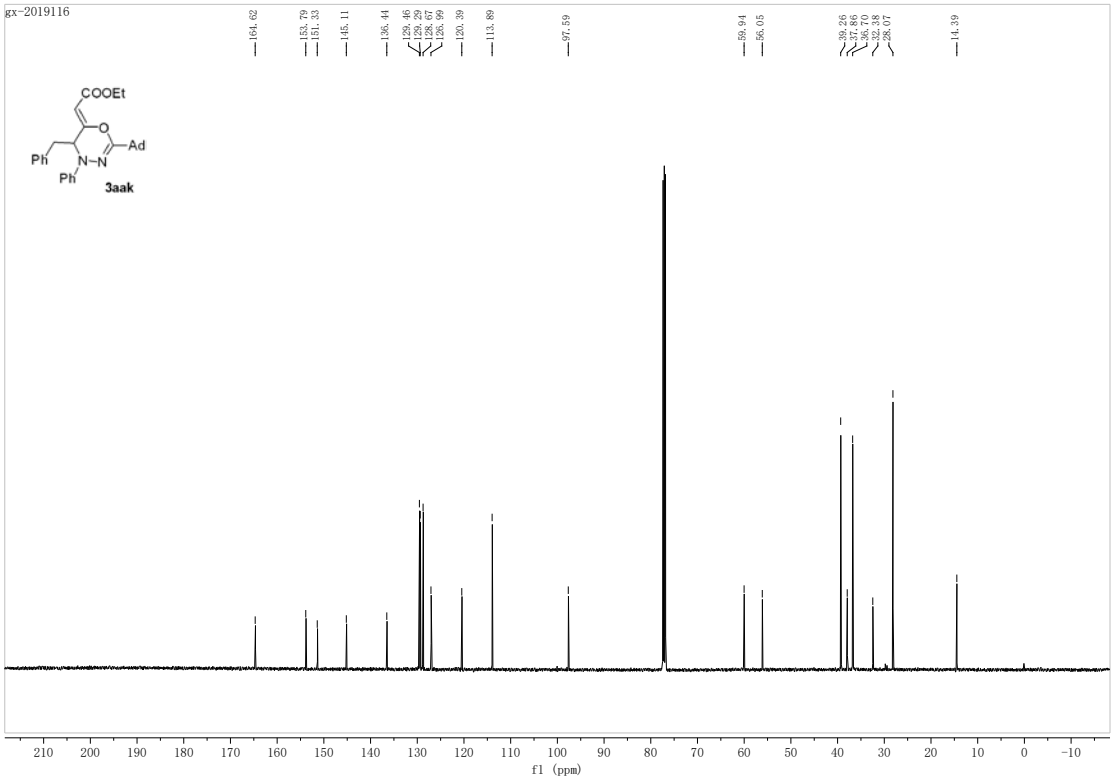

gx-201913l
131
7.35
7.33
7.31
7.29
7.28
7.27
7.26
7.24
7.22
7.21
7.19
7.18
7.17
6.99
6.97
6.95
6.57
6.56
6.56
4.75
4.71
4.69
4.68
4.67
4.22
4.21
4.20
4.19
4.18
3.01
3.00
2.98
2.97
2.93
2.90
2.89
2.87
1.60
1.31
1.30
1.28
COOEt
OMe
OMe
Ph
N-N
Ph
3aai
12.13
1.18
1.00
0.97
1.04
2.15
5.92
2.17
8.43
f1 (ppm)

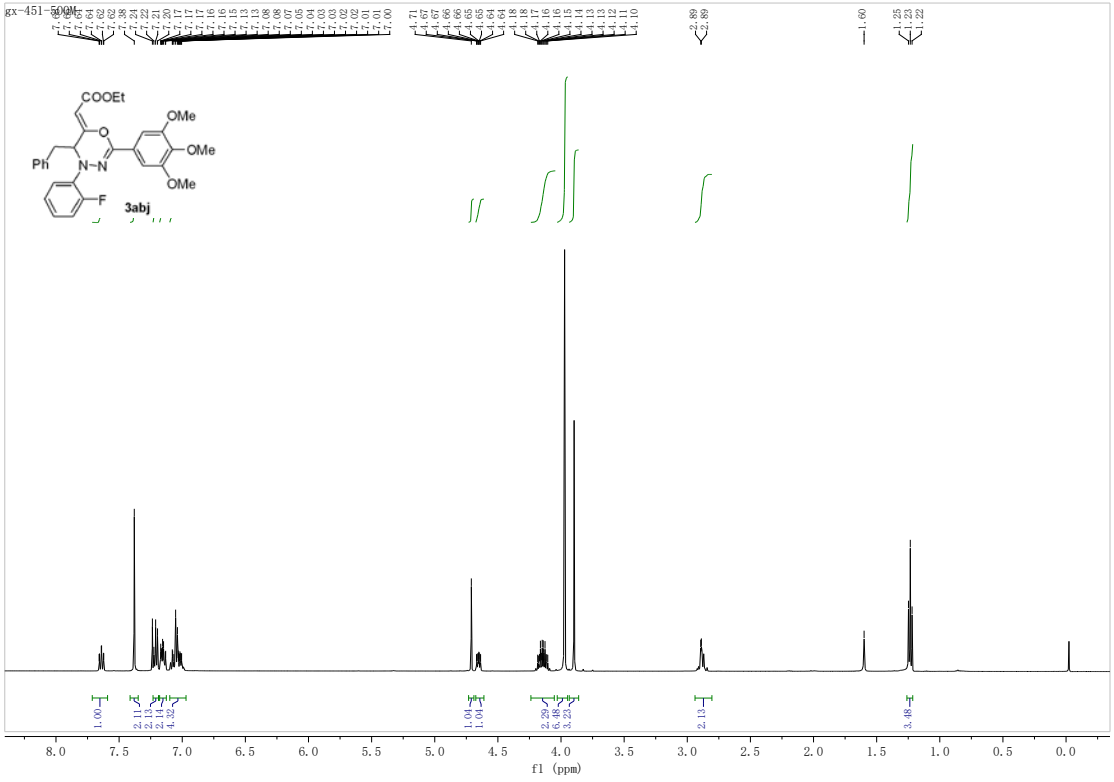

3,5-(OMe)2Ph-400M-CDC13
131-C
164.30
160.87
152.53
144.46
141.76
136.27
131.64
129.49
129.37
128.72
127.13
121.04
114.13
103.86
102.38
98.48
60.12
56.23
55.56
34.03
14.36
COOEt
OMe
Ph
N-N
Ph
OMe
3aai
f1 (ppm)

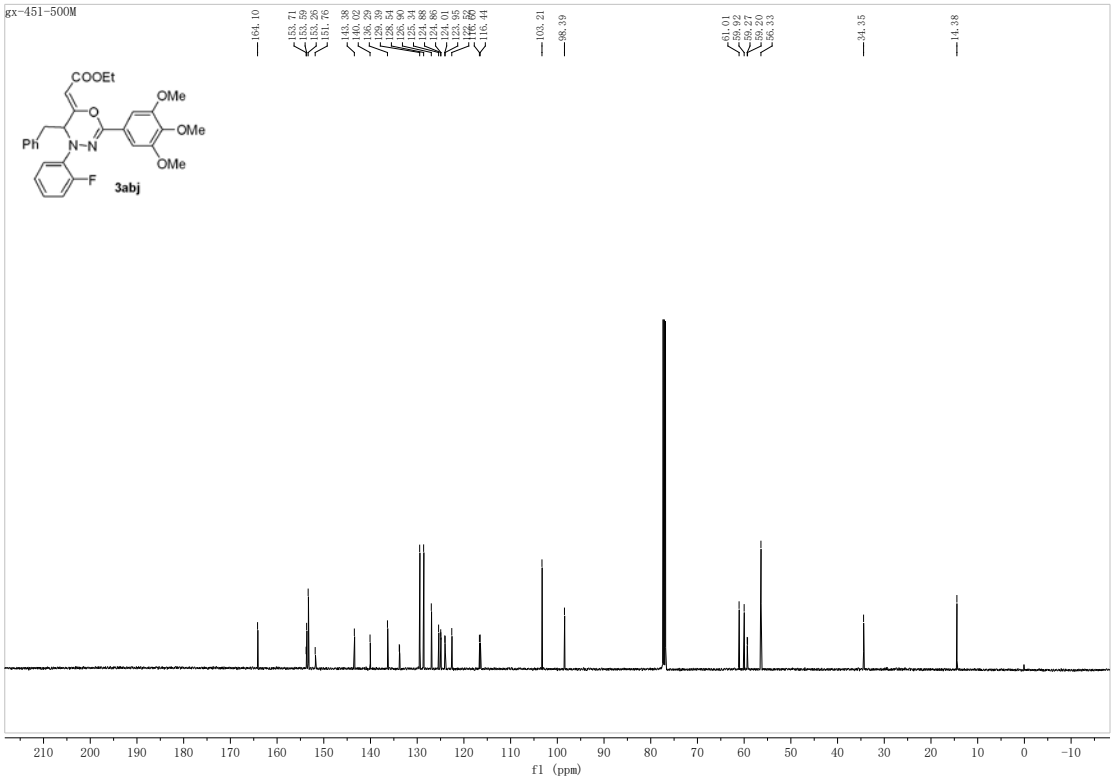

gx-2019118
7.97
7.35
7.34
7.31
7.29
7.28
7.25
7.23
7.20
7.18
6.98
6.97
6.95
4.69
4.68
4.67
4.66
4.19
4.18
4.17
4.16
4.00
3.92
3.01
3.00
2.98
2.93
2.91
2.90
2.88
1.63
1.28
1.25
COOEt
OMe
OMe
Ph
N-N
Ph
OMe
3aaj
1.99
2.09
2.13
3.35
2.08
1.00
1.01
1.03
2.12
6.08
3.05
2.10
3.25
f1 (ppm)

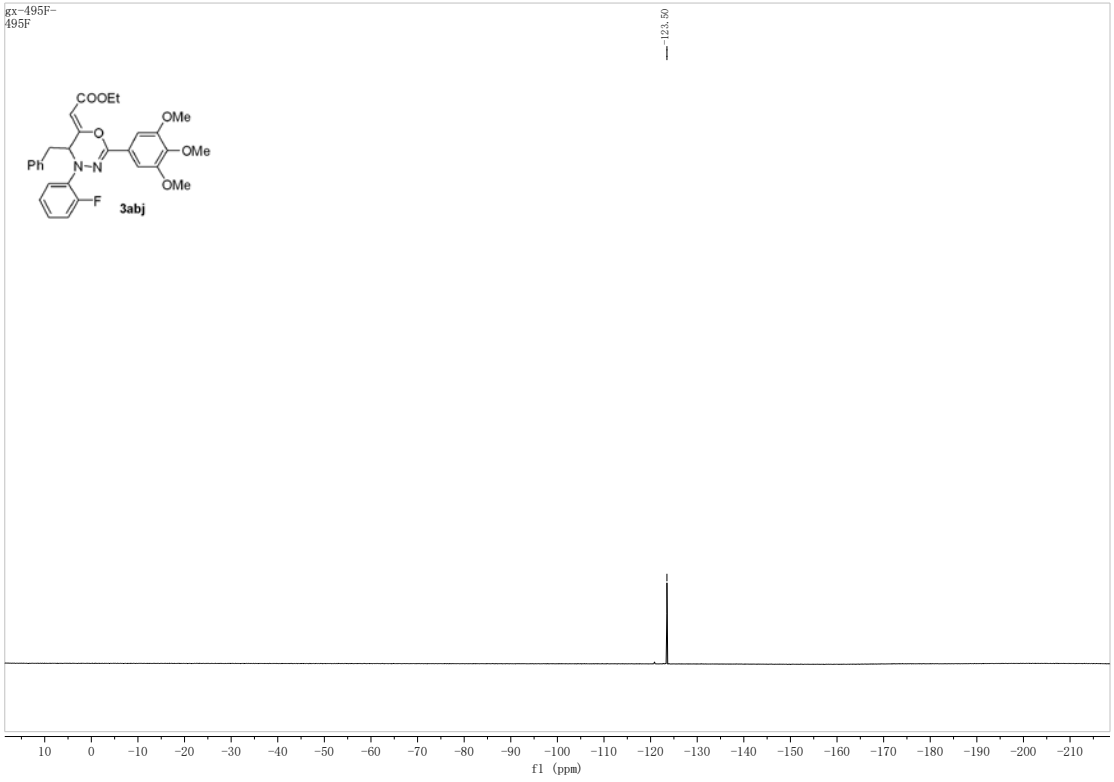

gx-2019118
COOEt
OMe
OMe
OMe
Ph
N-N
Ph
3aaj
f1 (ppm)

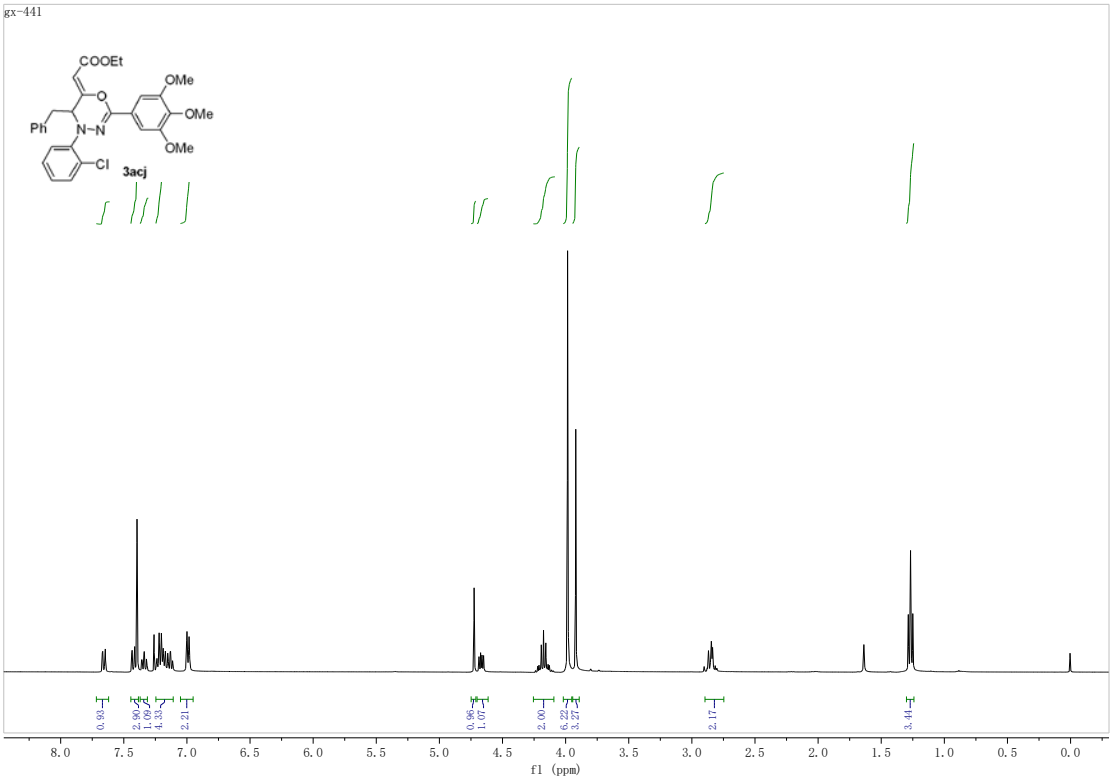

gx-2019116
COOEt
Ad
Ph
N-N
Ph
3aak
f1 (ppm)

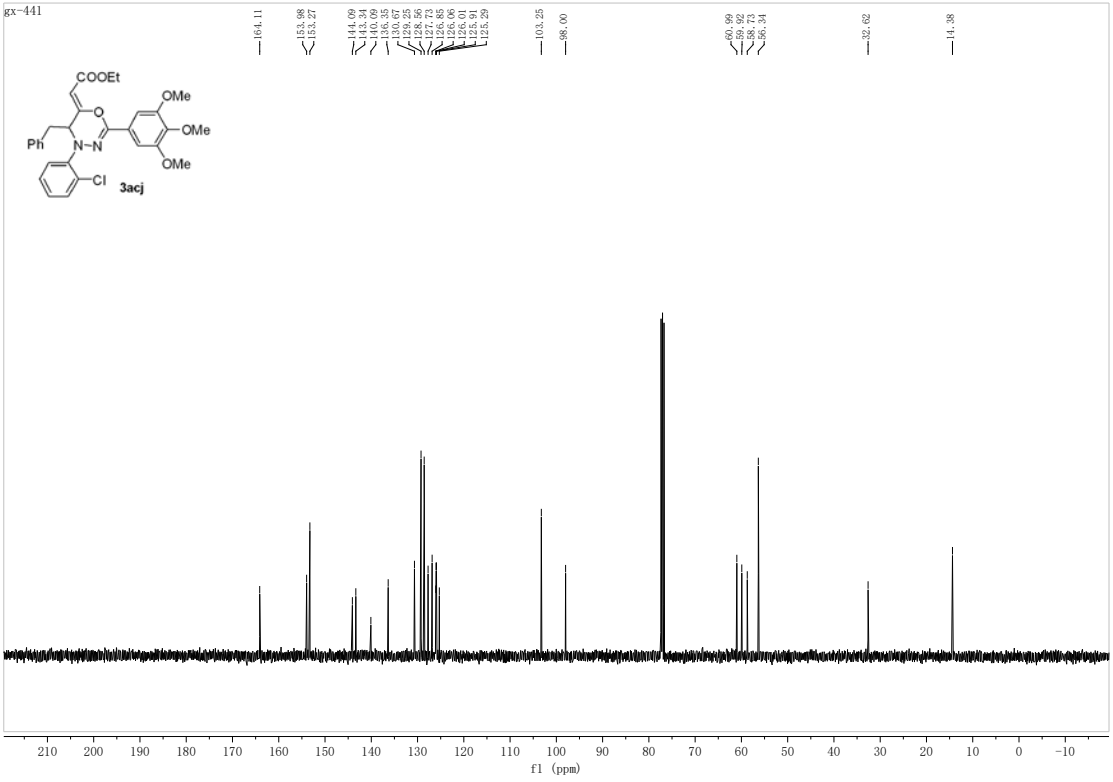

gx-2019116
164.62
153.79
151.33
145.11
136.44
129.46
129.29
128.67
126.99
120.39
113.89
97.59
59.94
56.05
39.26
37.85
36.70
32.38
28.07
14.39
COOEt
Ad
Ph
Ph
3aak
f1 (ppm)

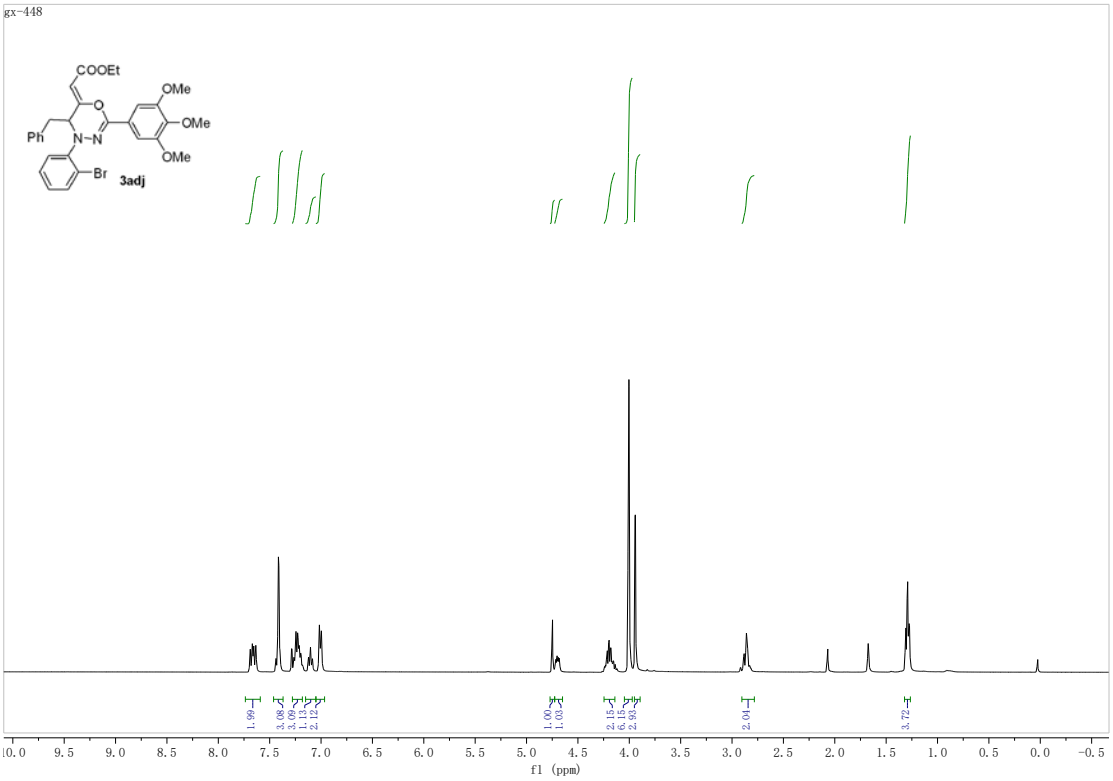

gx-451-500M
COOEt
OMe
OMe
OMe
Ph
F
3abj
f1 (ppm)

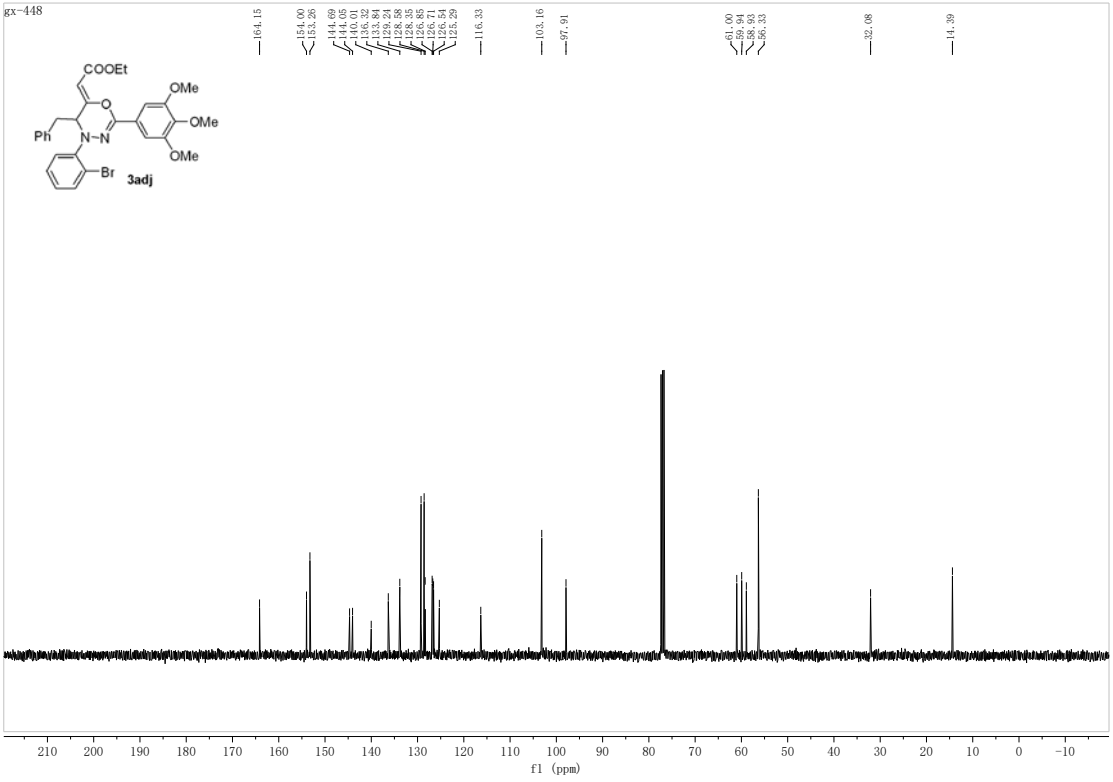

gx-451-500M
164.10
153.71
153.59
153.26
151.76
143.38
140.02
136.29
129.39
128.54
126.90
125.34
124.88
124.86
124.01
123.95
116.44
103.21
88.39
61.01
59.92
59.27
59.20
56.33
34.35
14.38
COOEt
OMe
OMe
OMe
Ph
N
N
O
F
3abj
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

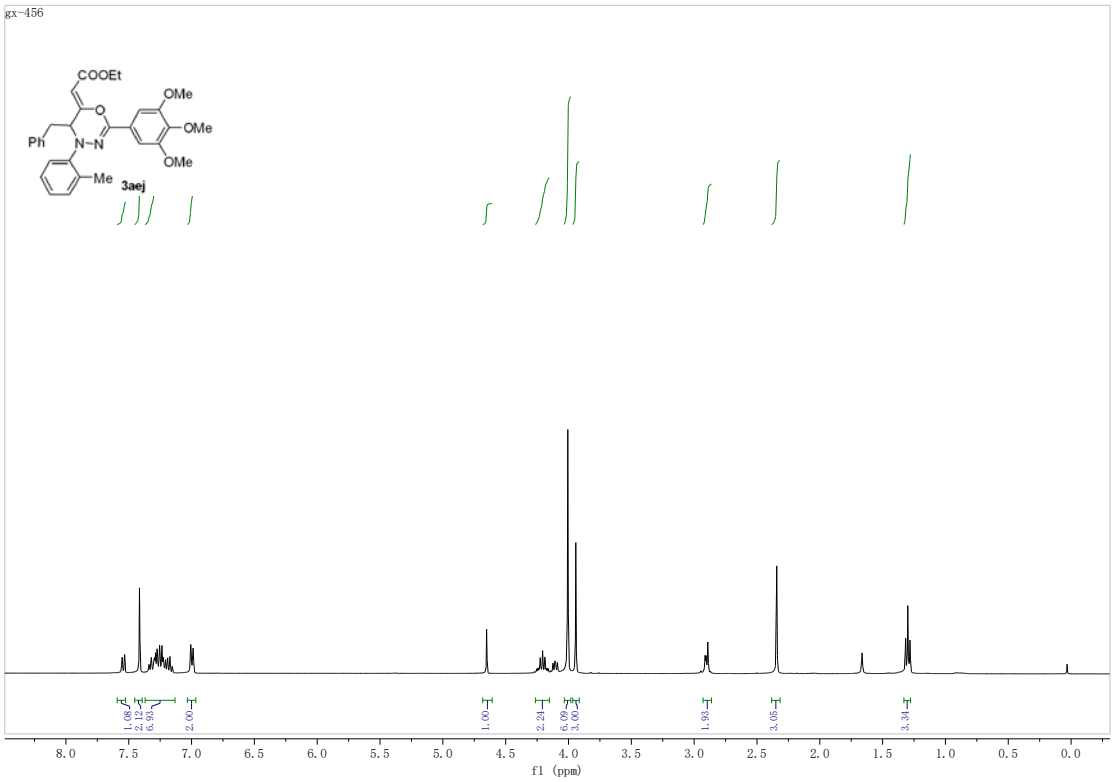

gx-495F-
495F
-123.50
COOEt
OMe
OMe
OMe
Ph
N
N
O
F
3abj
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

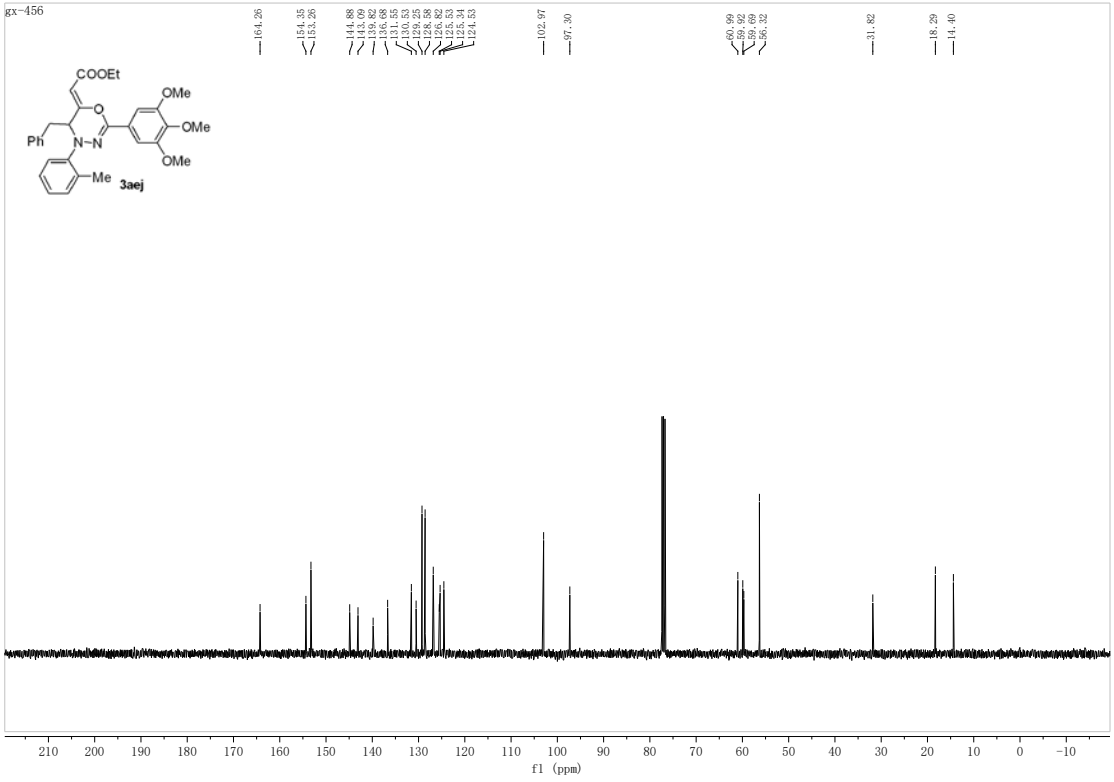

gx-441
COOEt
OMe
OMe
OMe
Ph
N-N
Cl
3acj
0.93
2.90
1.09
4.33
2.21
0.96
1.07
2.00
6.22
3.27
2.17
3.44
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

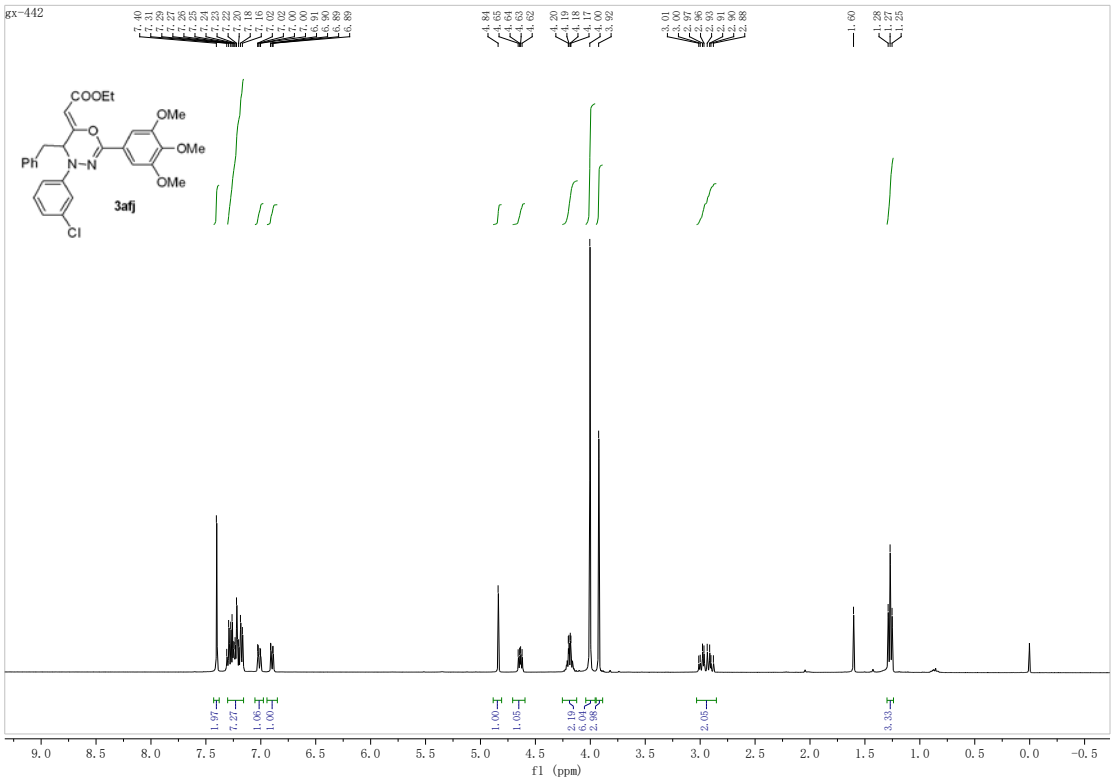

gx-441
164.11
153.98
153.27
144.09
142.34
140.09
136.35
130.67
129.35
128.56
127.73
126.85
126.06
125.01
125.91
125.29
103.25
98.00
60.99
59.92
58.73
56.34
22.62
14.38
COOEt
OMe
OMe
OMe
Ph
N-N
Cl
3acj
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

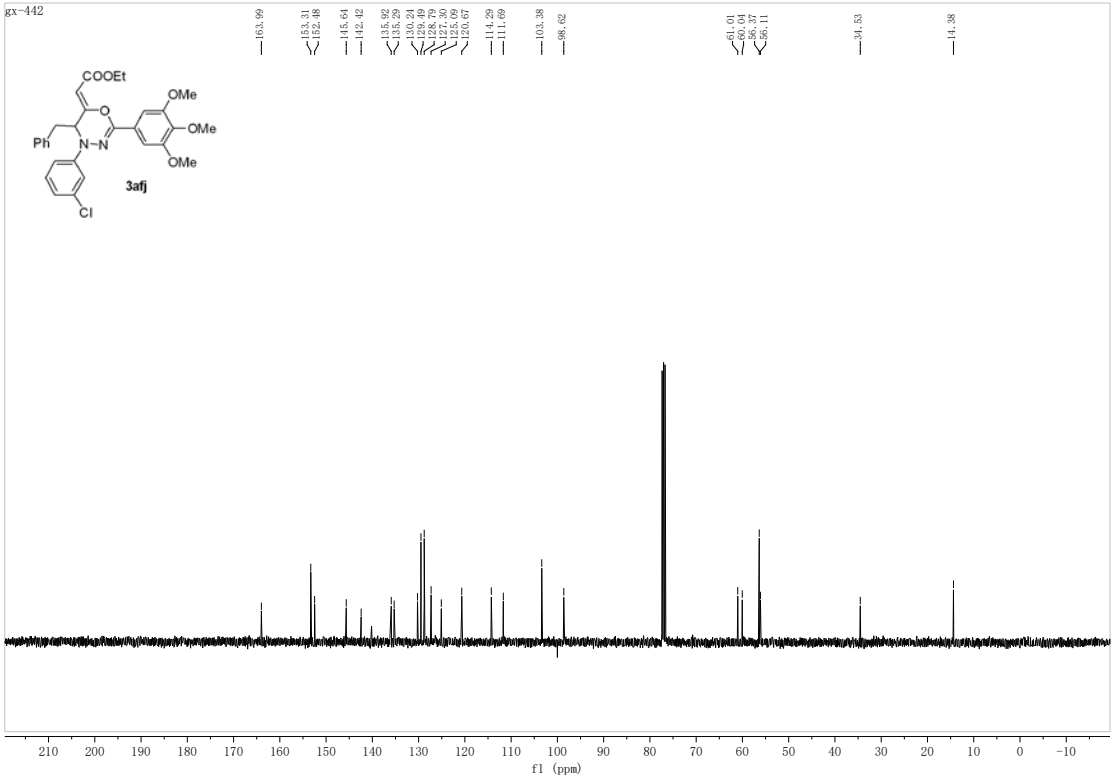

gx-448
COOEt
OMe
OMe
OMe
Ph
N-N
Br
3adj
1.99
3.06
3.09
1.13
2.12
1.00
1.02
2.15
6.15
2.93
2.04
3.72
f1 (ppm)

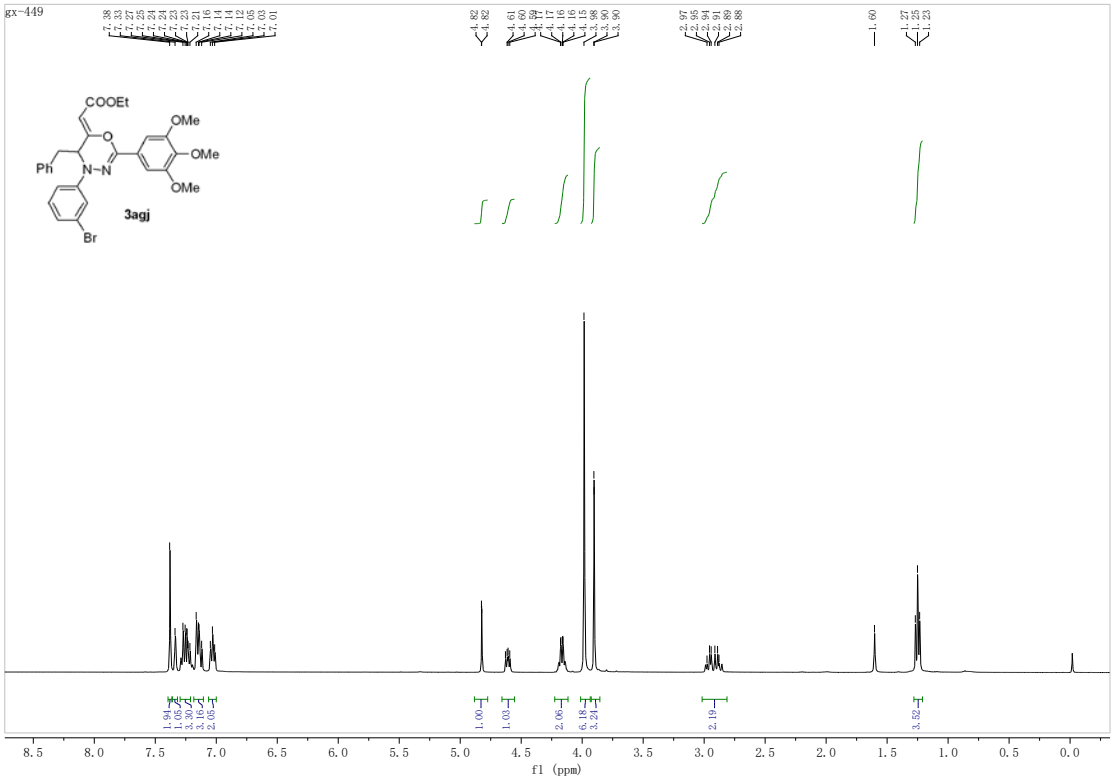

gx-448
164.15
154.00
153.26
144.69
144.05
140.01
136.22
133.84
129.24
128.58
128.35
126.65
126.71
126.54
125.29
116.33
103.16
97.91
61.00
59.94
58.93
56.33
32.08
14.39
COOEt
OMe
OMe
OMe
Ph
N-N
Br
3adj
f1 (ppm)

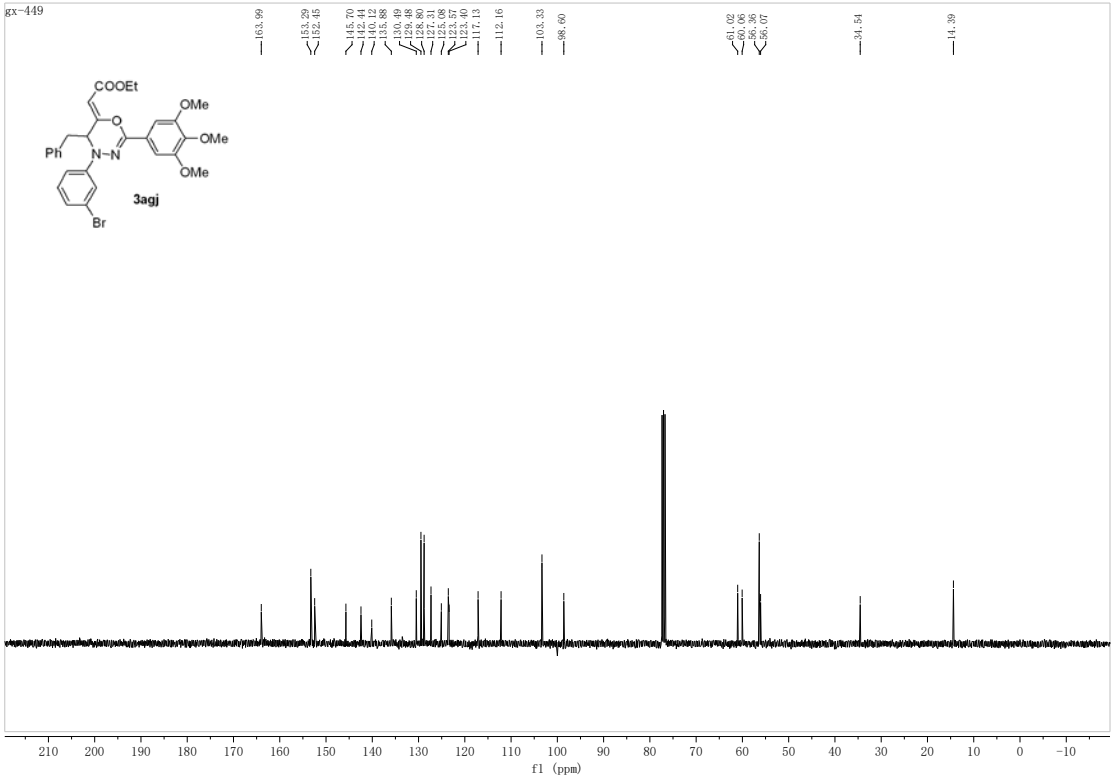

gx-456
COOEt
OMe
OMe
OMe
Ph
N-N
Me
3aej
1.08
2.12
6.93
2.00
1.00
2.24
6.09
3.00
1.93
3.05
3.34
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

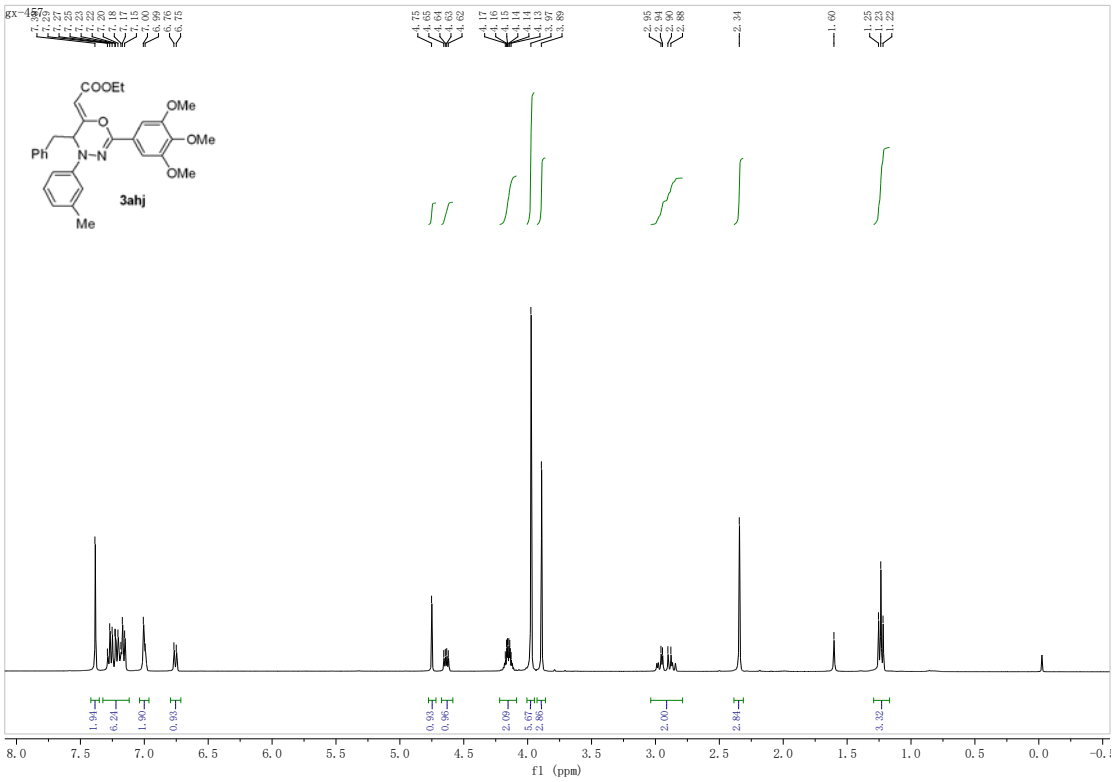

gx-456
164.26
154.35
153.26
144.88
143.09
139.82
136.68
131.15
130.53
129.25
128.58
126.82
125.53
125.34
124.53
102.97
97.30
60.99
59.92
59.69
56.32
31.82
18.29
14.40
COOEt
OMe
OMe
OMe
Ph
N-N
Me
3aej
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

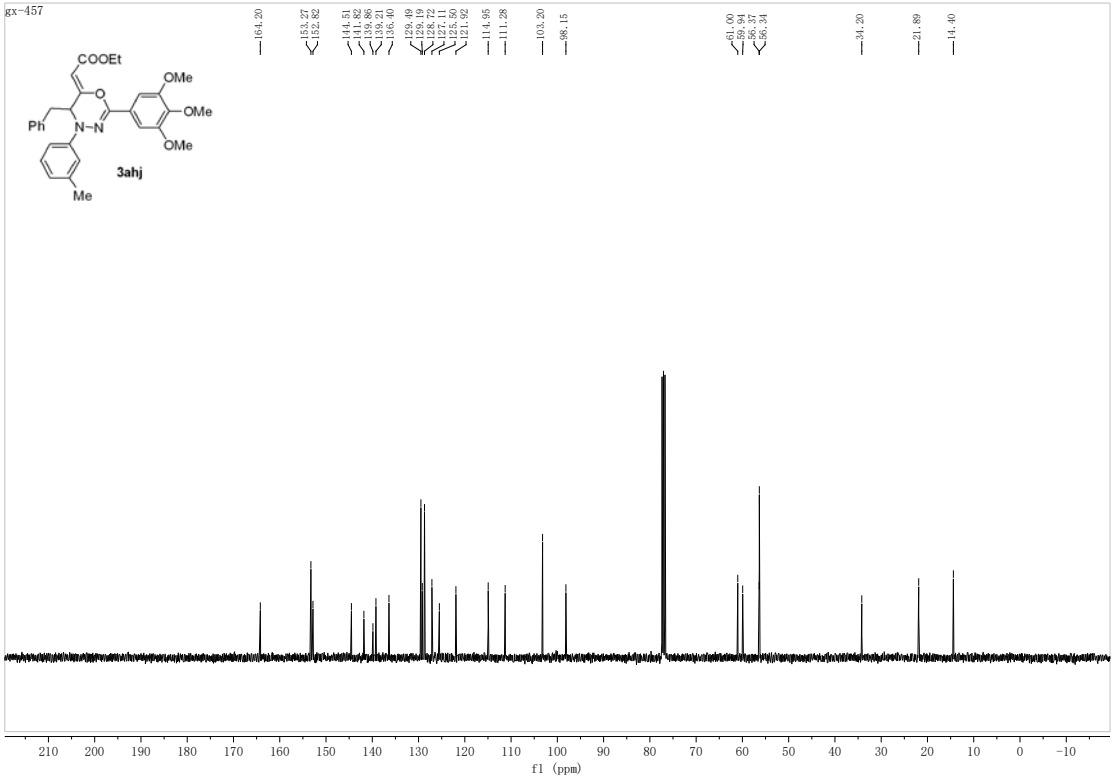

gx-442
COOEt
OMe
OMe
OMe
Ph
N-N
Cl
3afj
f1 (ppm)

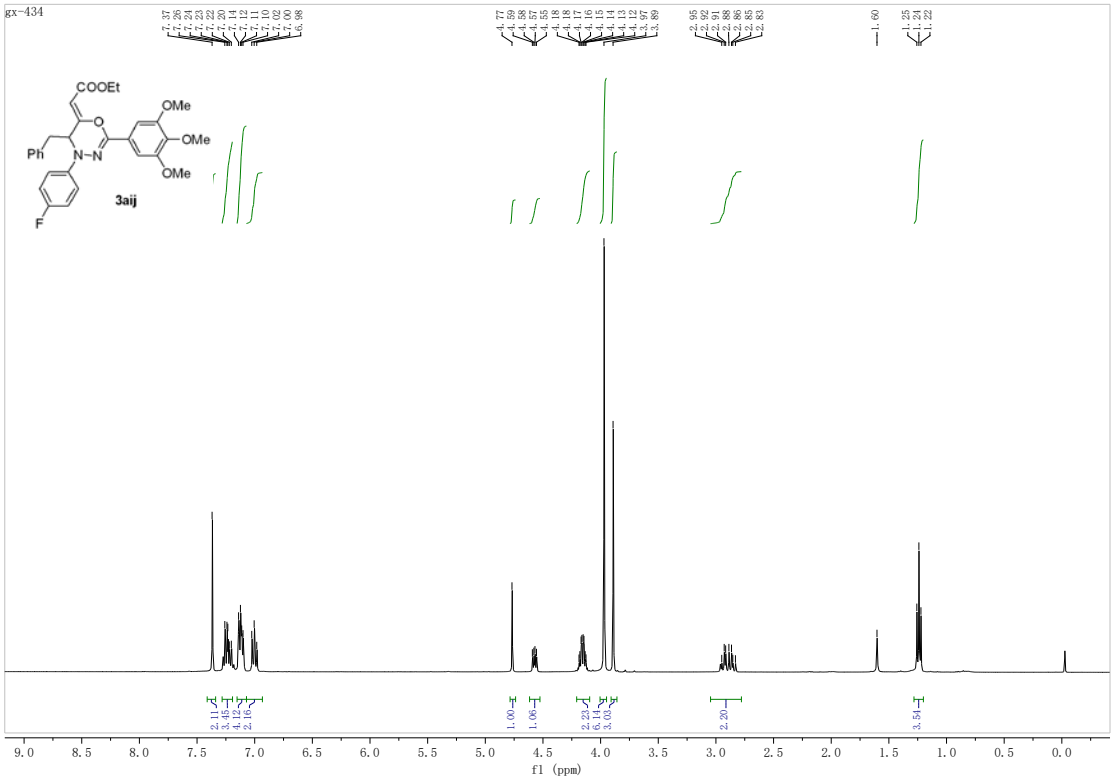

gx-442
COOEt
OMe
OMe
OMe
Ph
N-N
Cl
3afj
f1 (ppm)

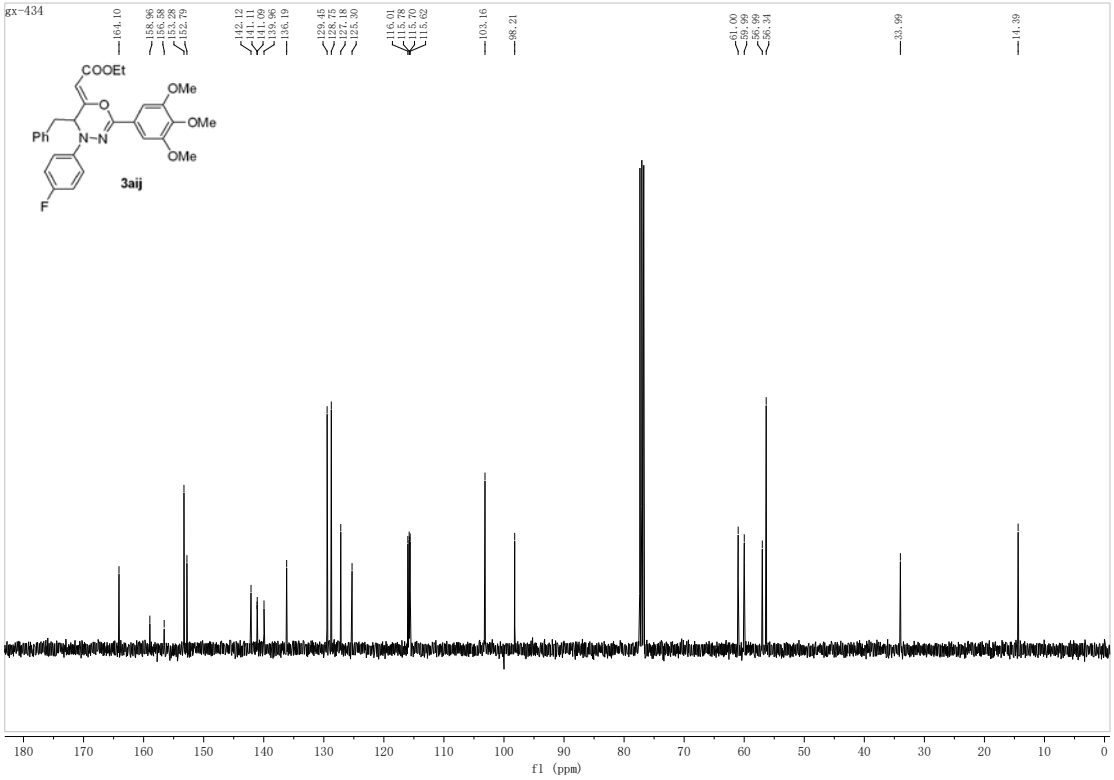

gx-449
COOEt
OMe
OMe
OMe
Ph
N-N
O
Br
3agj
f1 (ppm)

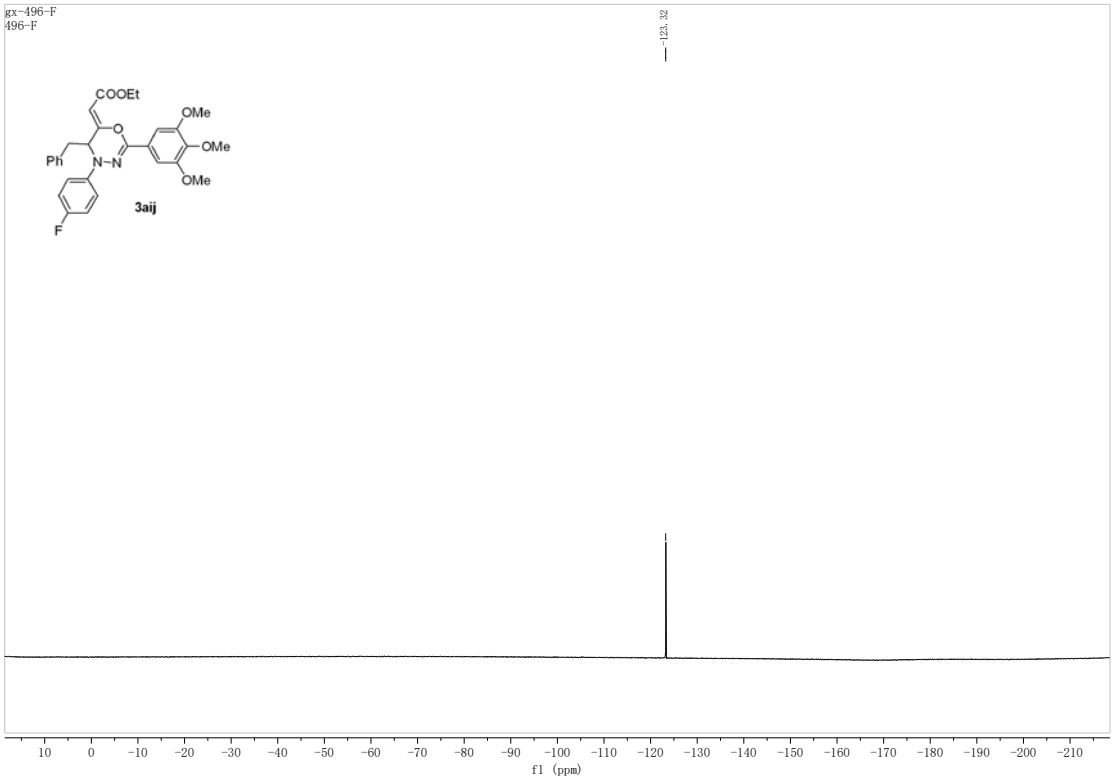

gx-449
COOEt
OMe
OMe
OMe
Ph
N-N
O
Br
3agj
f1 (ppm)

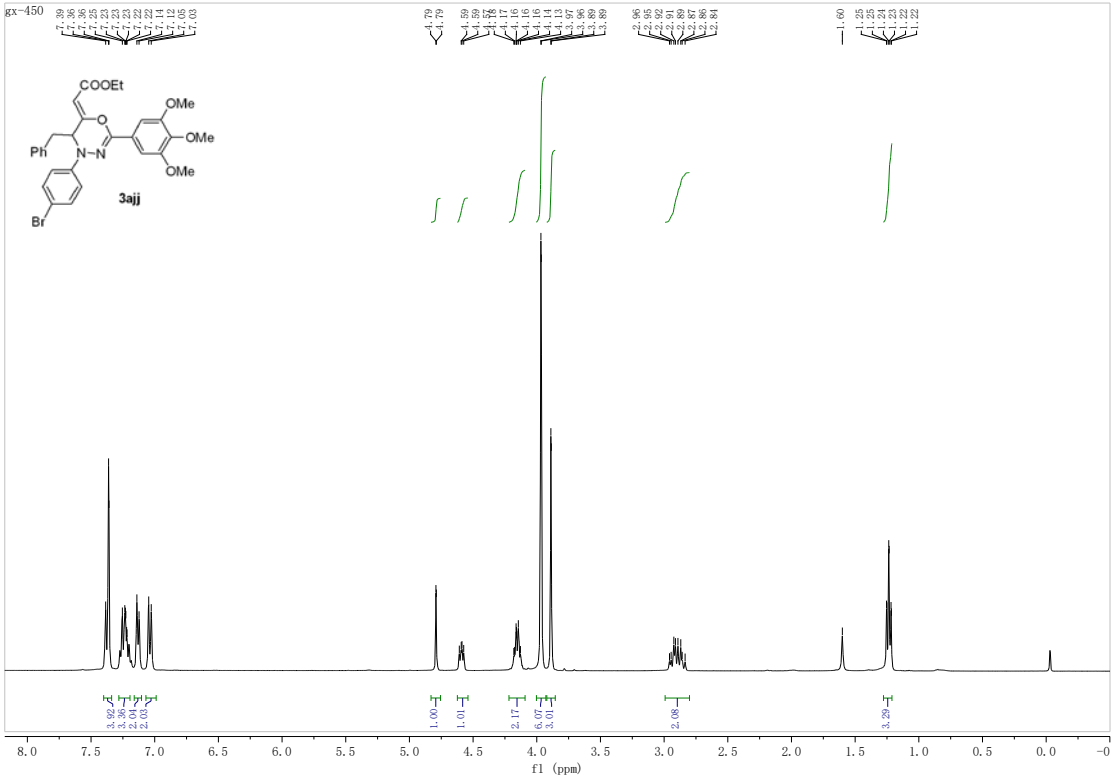

gx-457
COOEt
OMe
OMe
OMe
Ph
N-N
O
Me
3ahj
f1 (ppm)

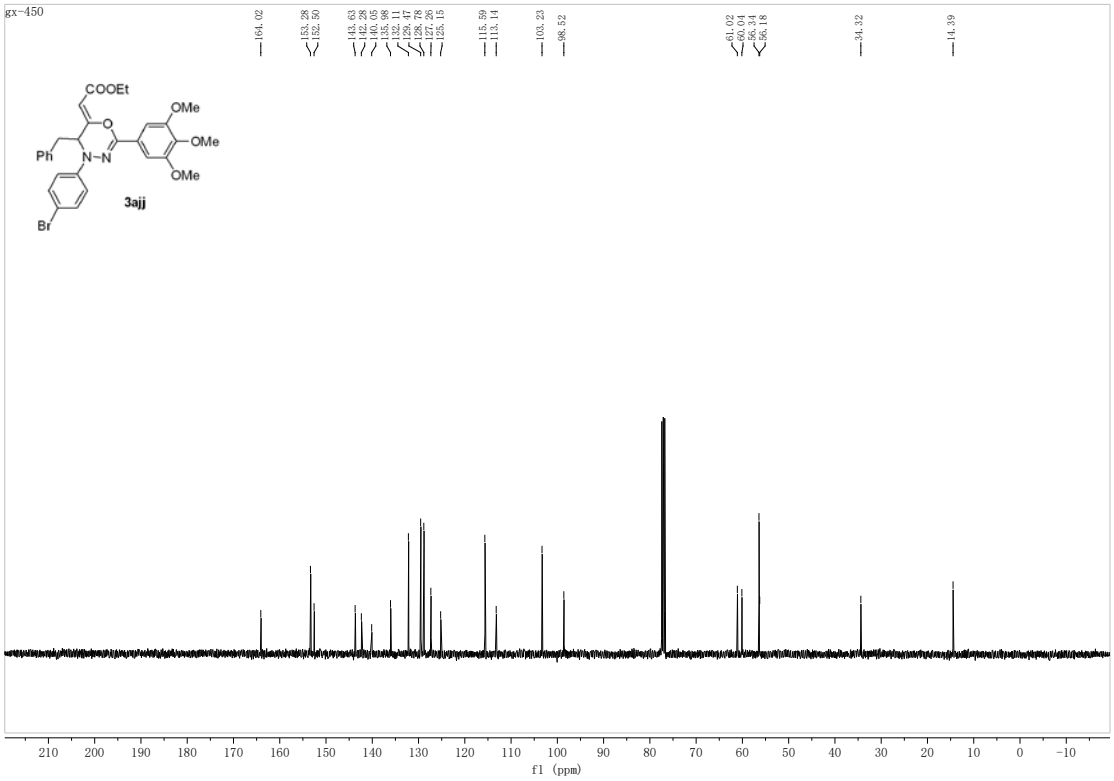

gx-457
COOEt
OMe
OMe
OMe
Ph
N-N
O
Me
3ahj
f1 (ppm)

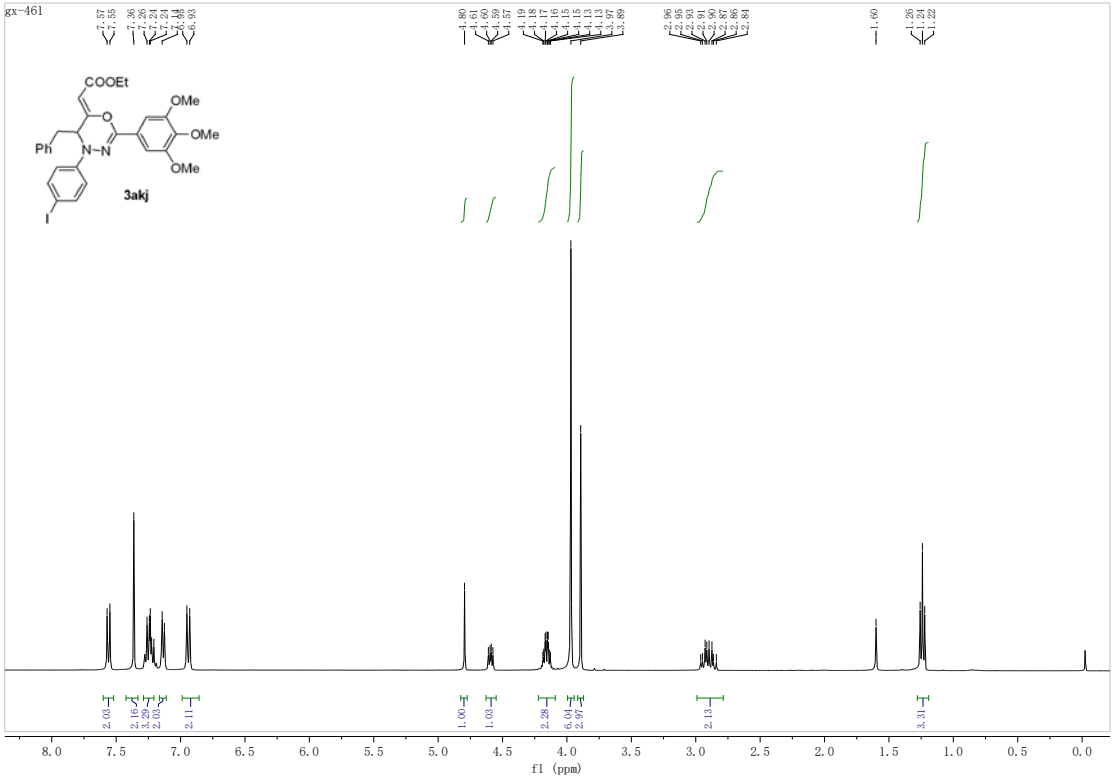

gx-434
COOEt
OMe
OMe
OMe
Ph
N-N
F
3aij
f1 (ppm)

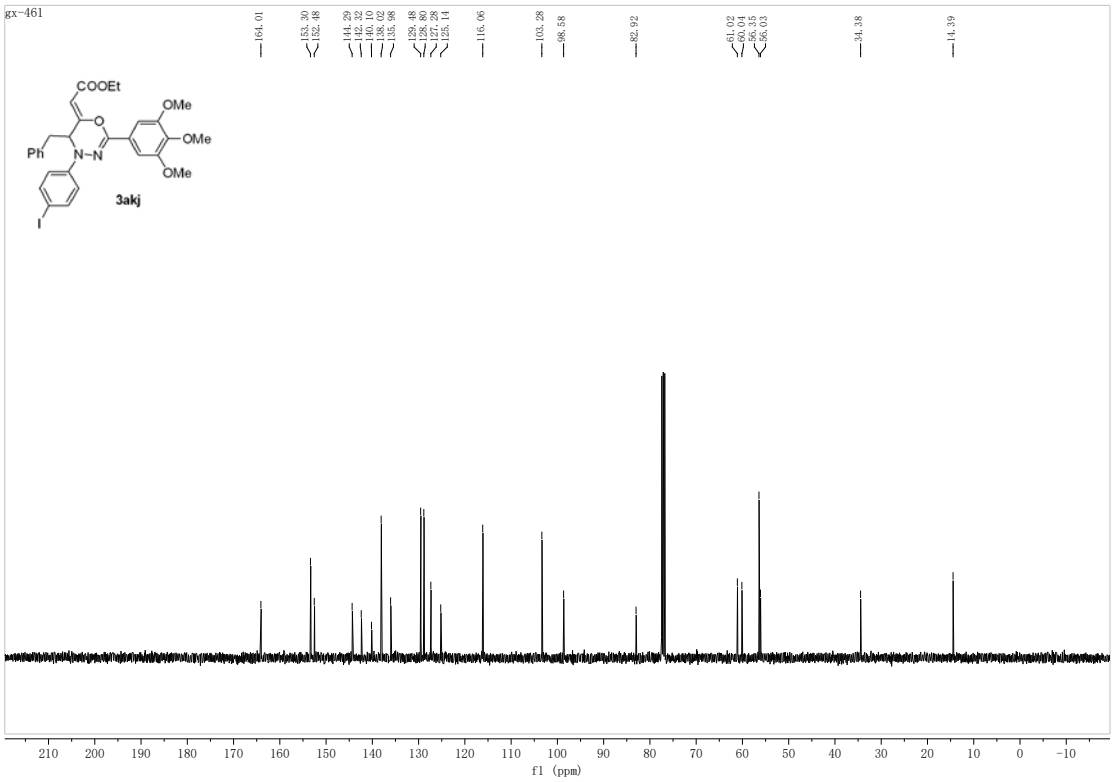

gx-434
COOEt
OMe
OMe
OMe
Ph
N-N
F
3aij
f1 (ppm)

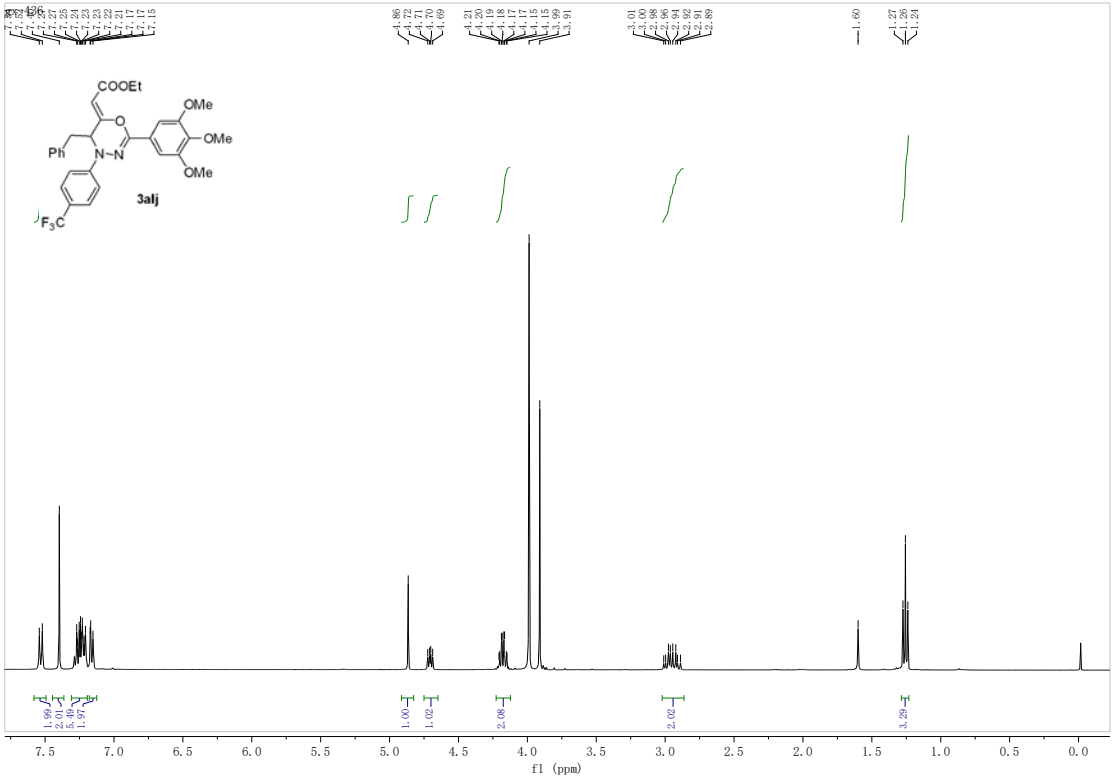

gx-496-F
496-F
-123.32
COOEt
OMe
OMe
OMe
Ph
N-N
3aij
F
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)

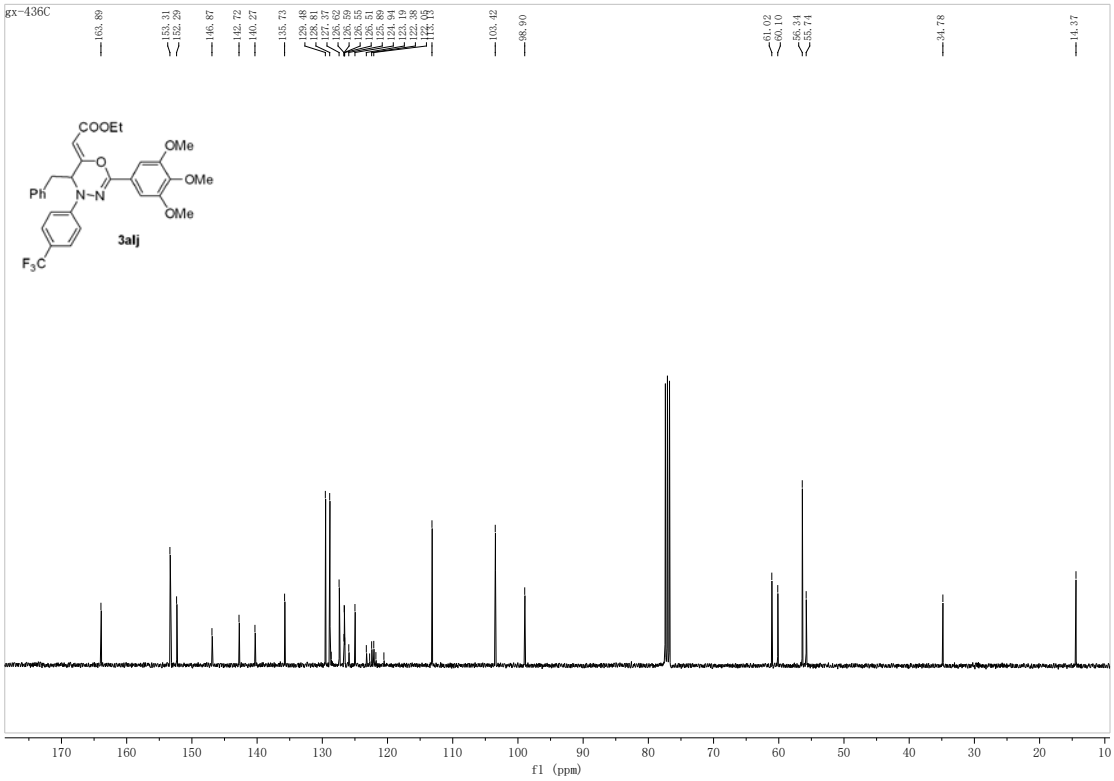

gx-450
7.39 7.38 7.36 7.25 7.23 7.23 7.23 7.22 7.22 7.14 7.12 7.05 7.03
4.79 4.79 4.59 4.58 4.57 4.18 4.17 4.16 4.16 4.16 4.14 4.13 3.97 3.96 3.89 3.89
2.96 2.92 2.91 2.89 2.87 2.86 2.84
1.60
1.25 1.25 1.24 1.23 1.22 1.22
COOEt
OMe
OMe
OMe
Ph
N-N
3ajj
Br
3.92 3.36 2.04 2.03
1.00 1.01 2.17 6.07 3.01 2.08 3.29
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)


gx-450
164.02
153.28
152.50
143.63
142.28
140.05
135.98
132.11
129.47
128.78
127.26
125.15
115.59
113.14
103.23
88.52
61.02
60.04
56.34
56.18
34.32
14.39
COOEt
OMe
OMe
OMe
Ph
N-N
O
Br
3ajj
f1 (ppm)


gx-461
7.57
7.55
7.36
7.29
7.24
6.94
6.93
4.80
4.61
4.60
4.59
4.57
4.19
4.18
4.17
4.16
4.15
4.13
4.13
3.97
3.89
2.96
2.95
2.93
2.91
2.90
2.87
2.86
2.84
1.60
1.25
1.24
1.22
2.03
2.16
3.29
2.03
2.11
1.00
1.03
2.28
6.04
2.97
2.13
3.31
COOEt
OMe
OMe
OMe
Ph
N-N
O
I
3akj
f1 (ppm)


gx-461
COOEt
OMe
OMe
OMe
Ph
N-N
I
3akj
f1 (ppm)


COOEt
OMe
OMe
OMe
Ph
N-N
F3C
3alj
f1 (ppm)


gx-436C
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135.73
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128.81
127.37
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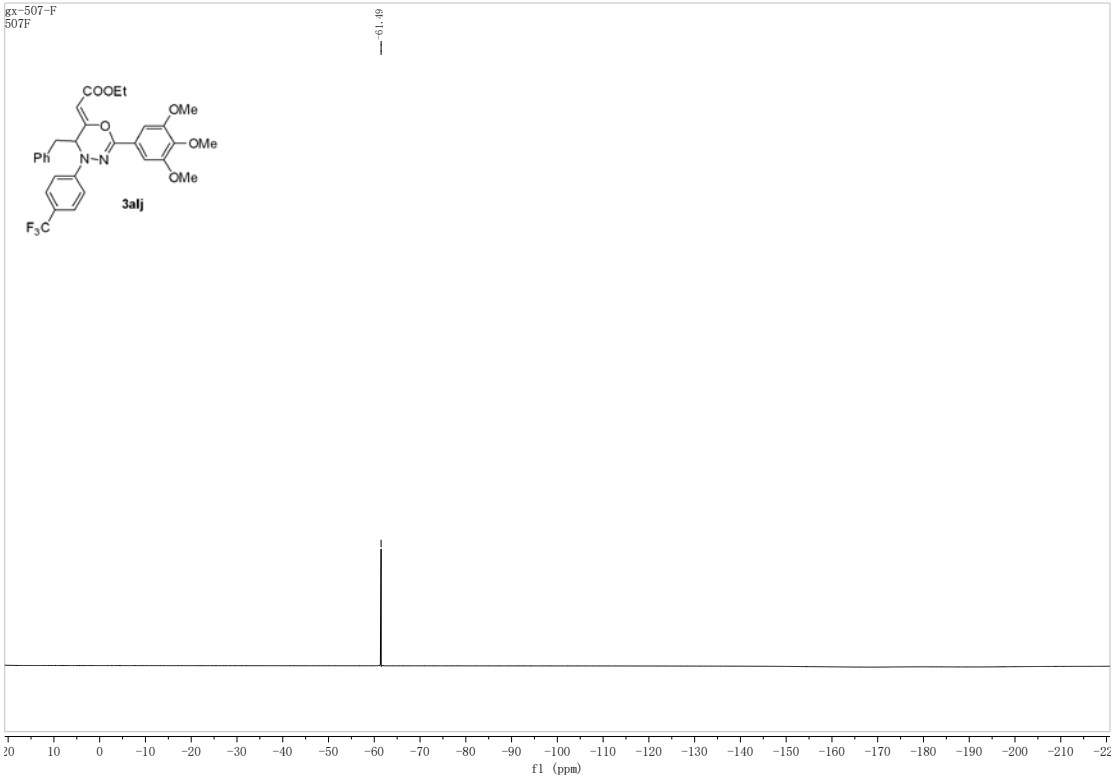
126.55
126.61
125.89
124.94
123.19
122.38
103.42
98.90
61.02
60.10
56.34
55.74
34.78
14.37
COOEt
OMe
OMe
OMe
Ph
N-N
O
F3C
3alj
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


gx-507-F
507F
-61.49
COOEt
OMe
OMe
OMe
Ph
N-N
O
F3C
3alj
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
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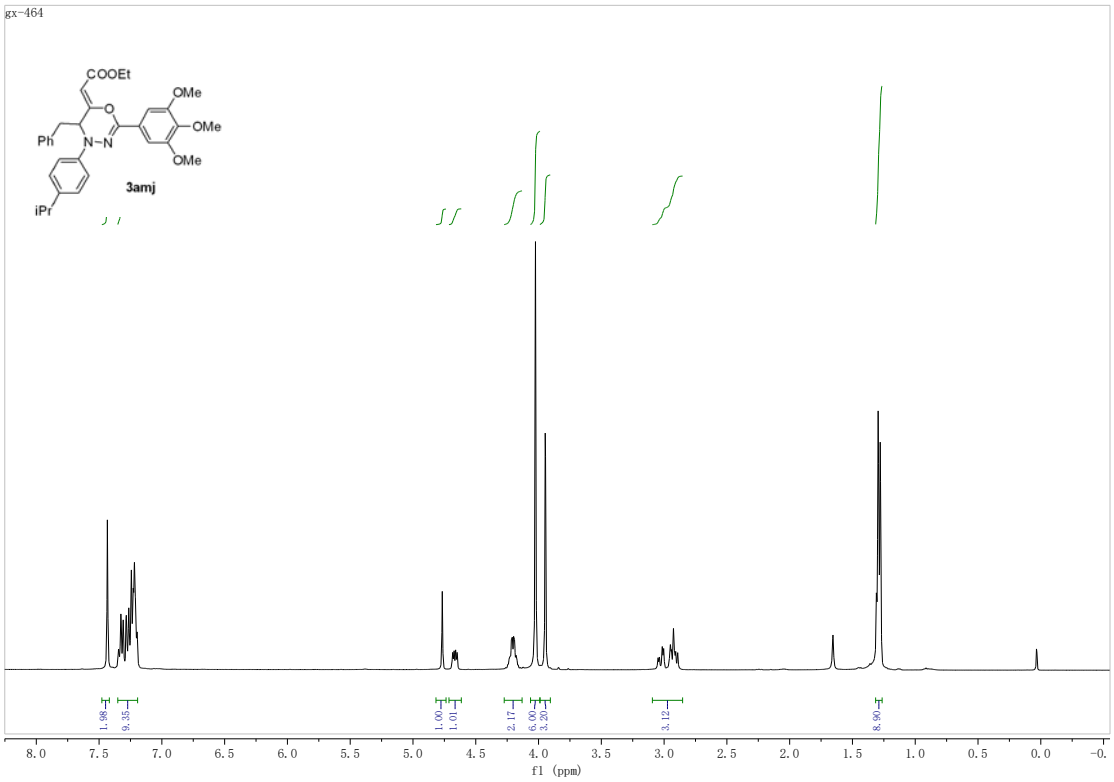
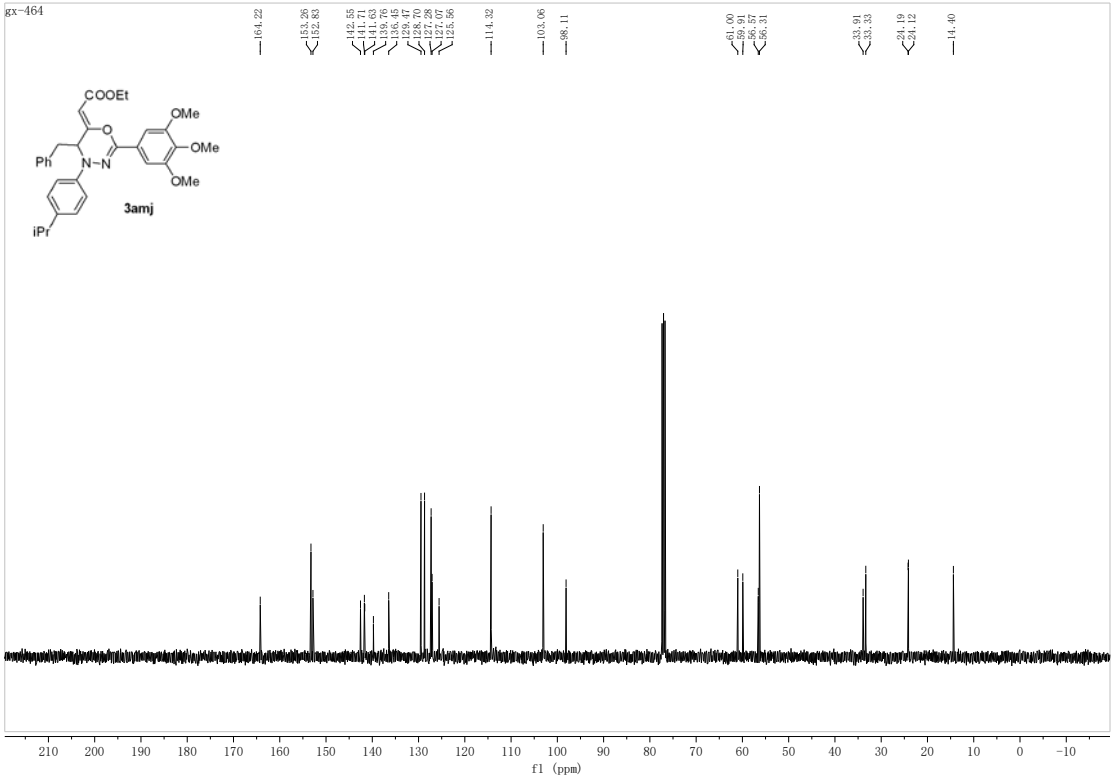
-180
-190
-200
-210
-22
f1 (ppm)


gx-464
COOEt
OMe
OMe
OMe
Ph
N-N
O
iPr
3amj
1.98
9.35
1.00
1.01
2.17
6.00
3.20
3.12
8.90
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


gx-464
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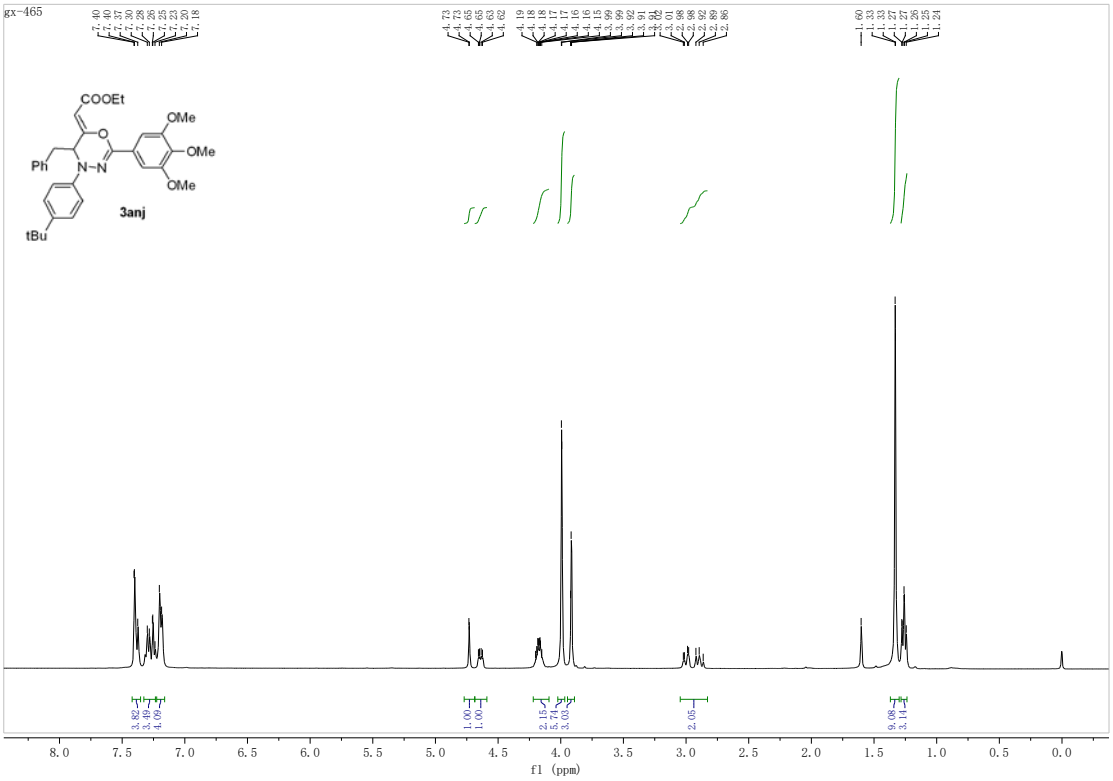
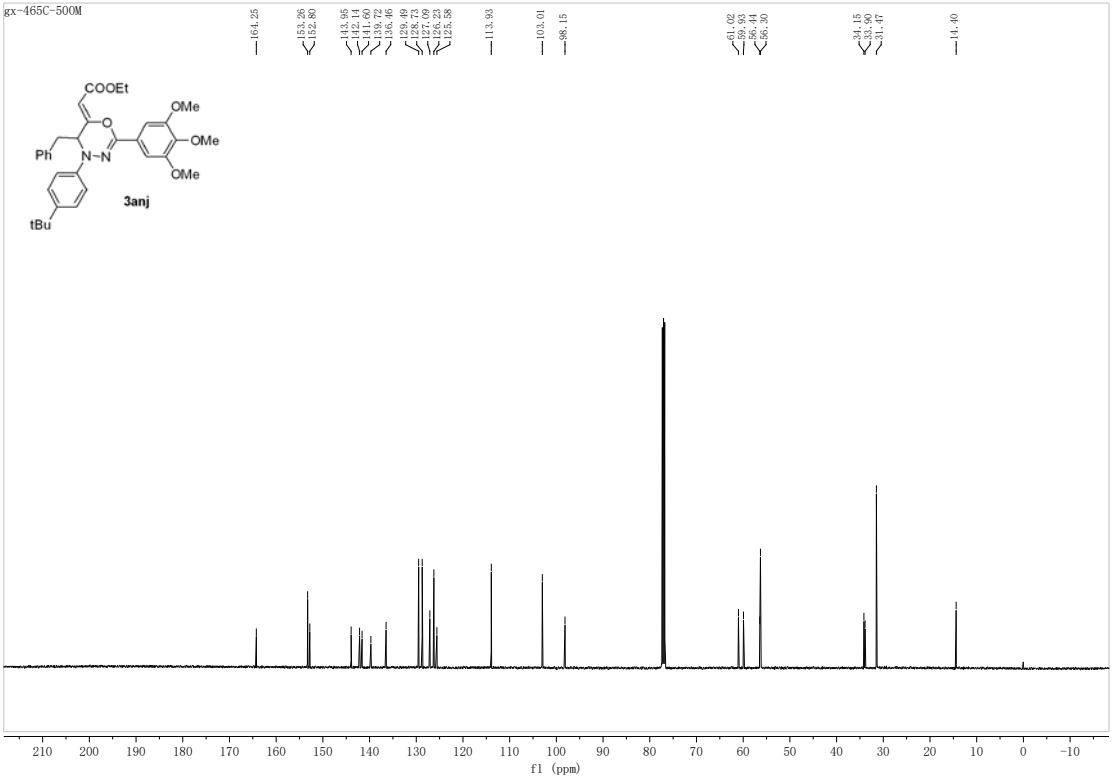
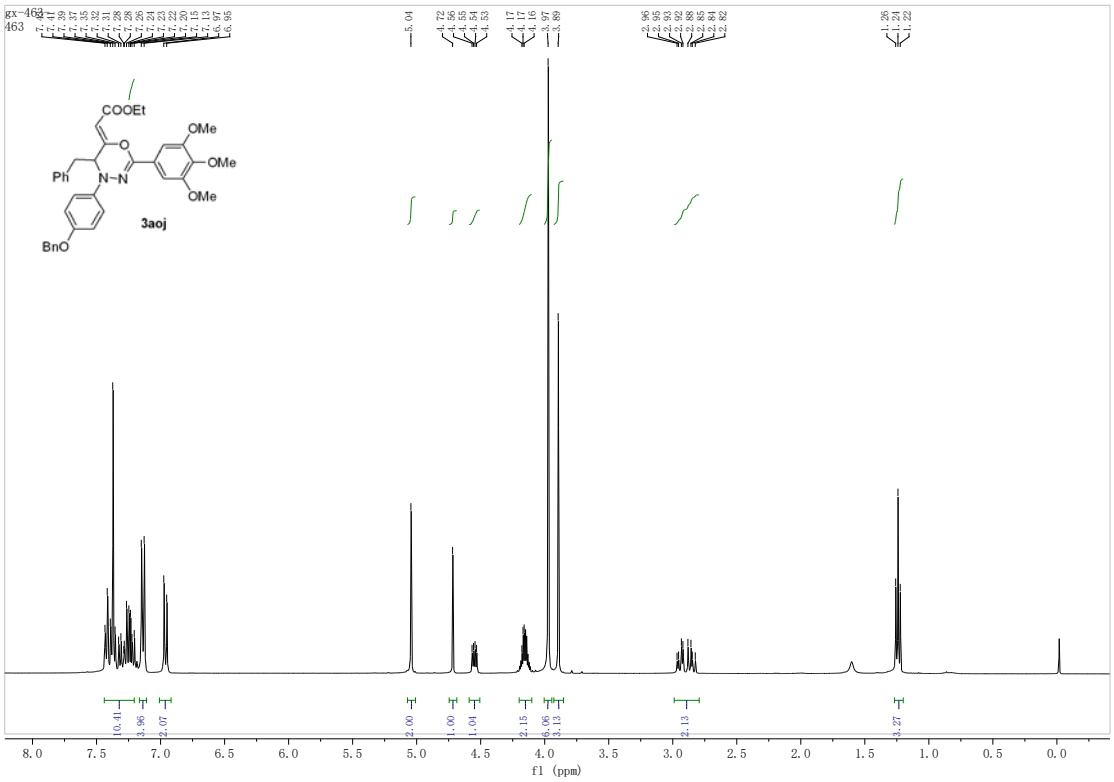
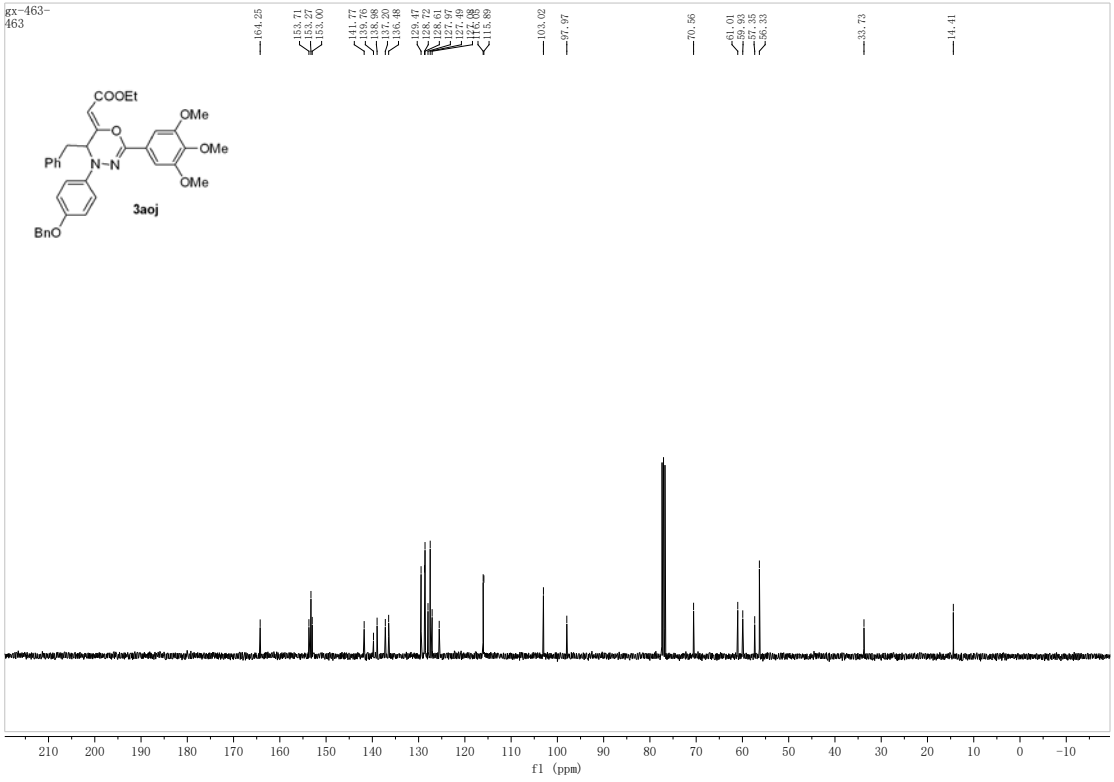
152.83
142.55
141.71
141.63
139.76
135.45
129.47
128.70
127.28
127.07
125.86
114.32
103.06
98.11
61.00
59.91
56.57
56.31
33.91
33.33
21.19
20.12
14.40
COOEt
OMe
OMe
OMe
Ph
N-N
O
iPr
3amj
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


gx-465
COOEt
OMe
OMe
OMe
Ph
N-N
tBu
3anj
f1 (ppm)


gx-465C-500M
COOEt
OMe
OMe
OMe
Ph
N-N
tBu
3anj
f1 (ppm)


gx-463
463
COOEt
OMe
OMe
OMe
Ph
N-N
O
BnO
3aoj
5.04
4.72
4.56
4.55
4.54
4.53
4.17
4.17
4.16
3.97
3.89
2.96
2.95
2.93
2.92
2.88
2.85
2.84
2.82
1.26
1.24
1.22
10.41
3.96
2.07
2.00
1.00
1.04
2.15
6.06
3.13
2.13
3.27
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


gx-463-
463
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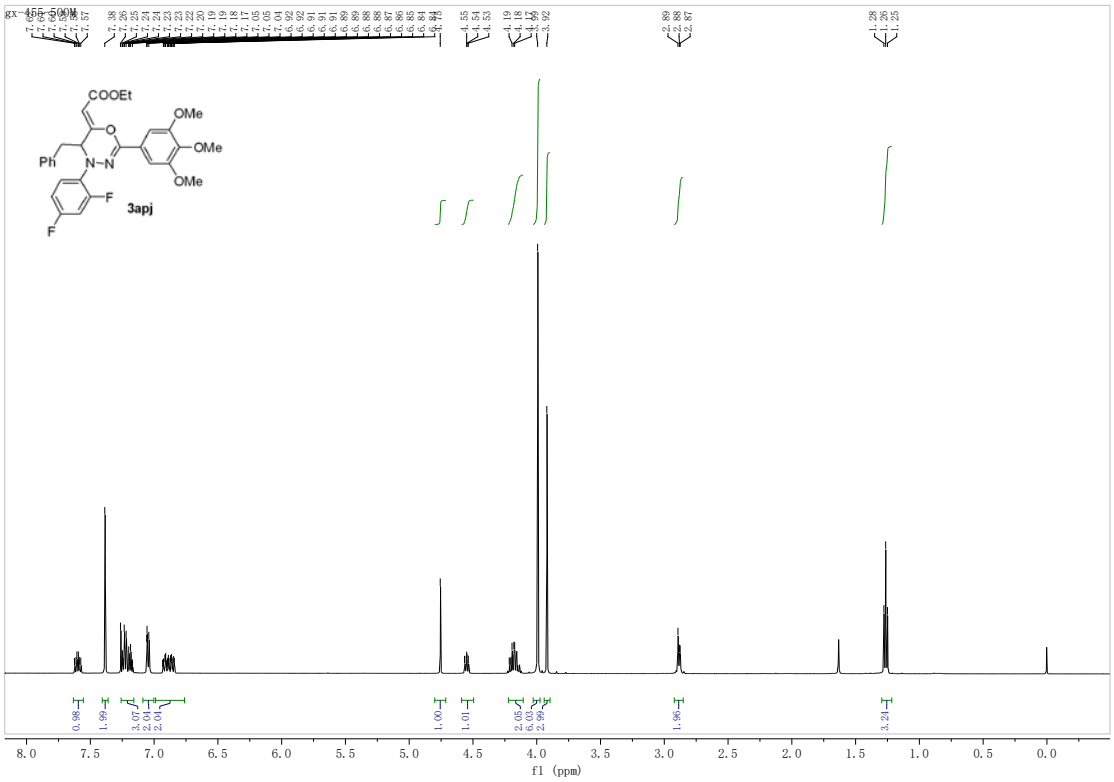
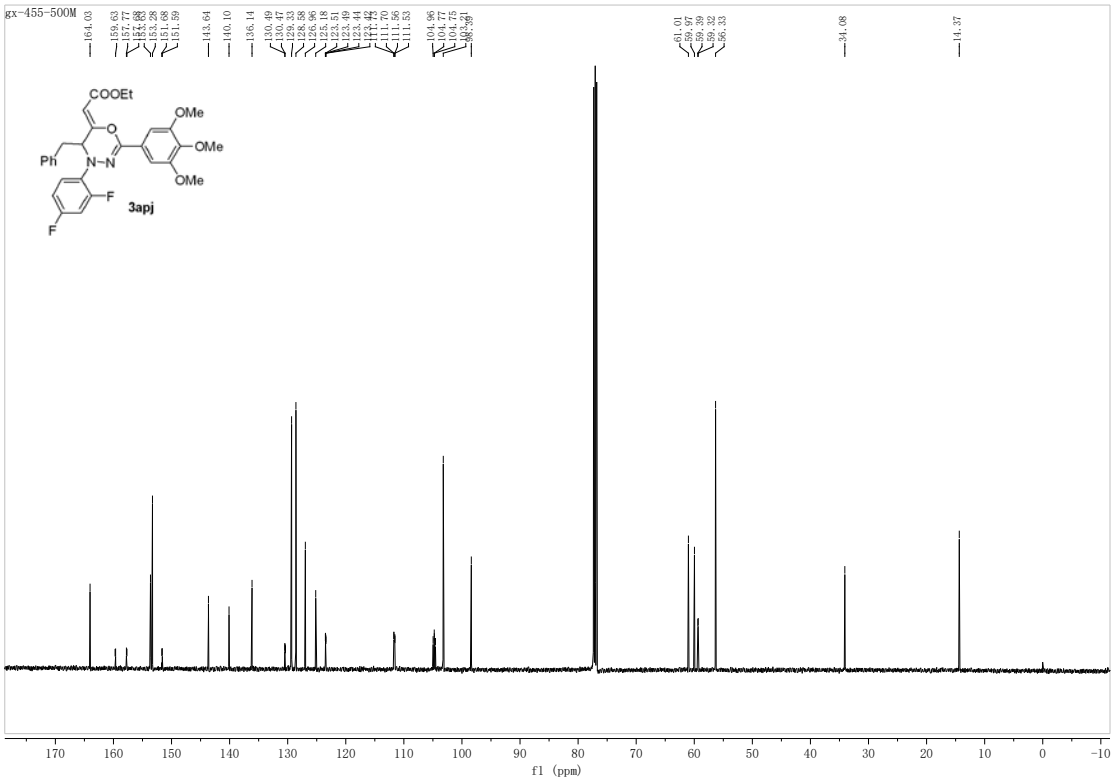
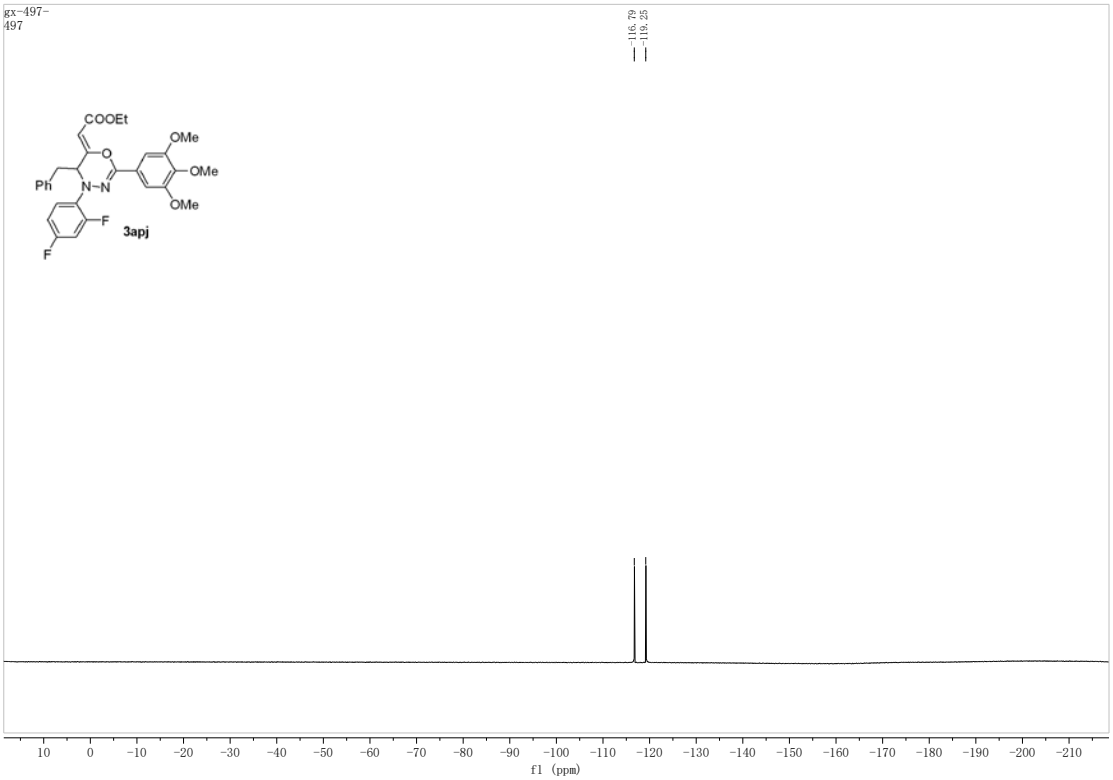
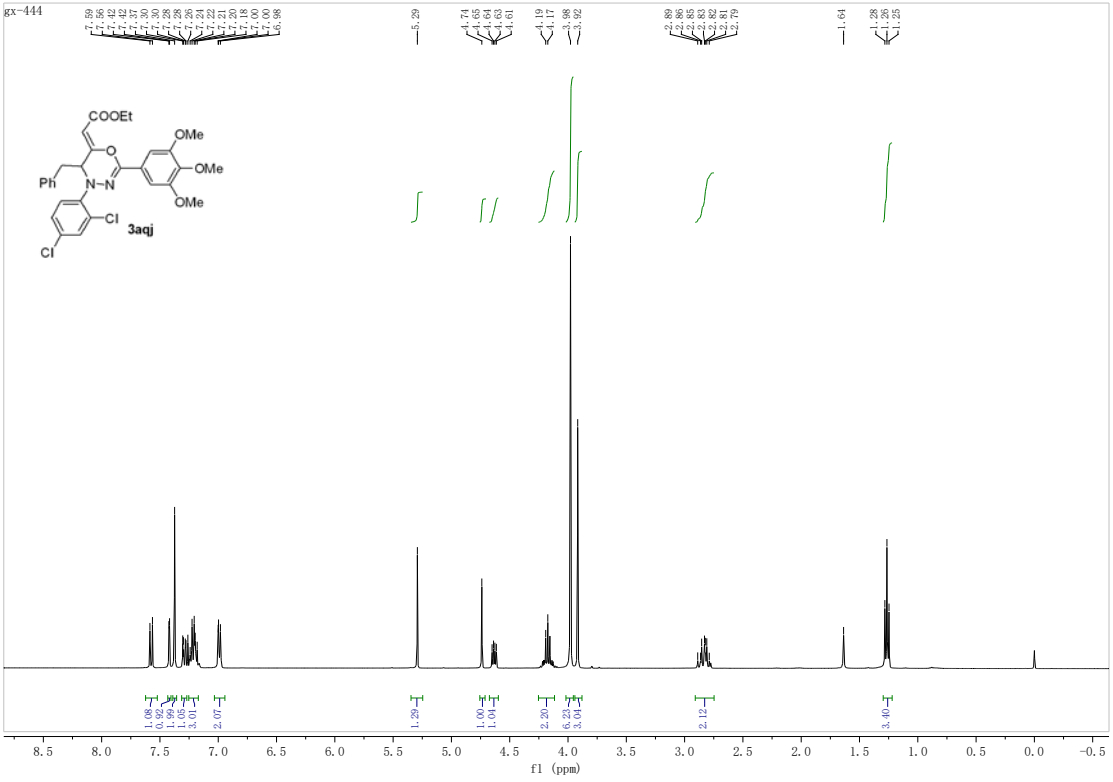
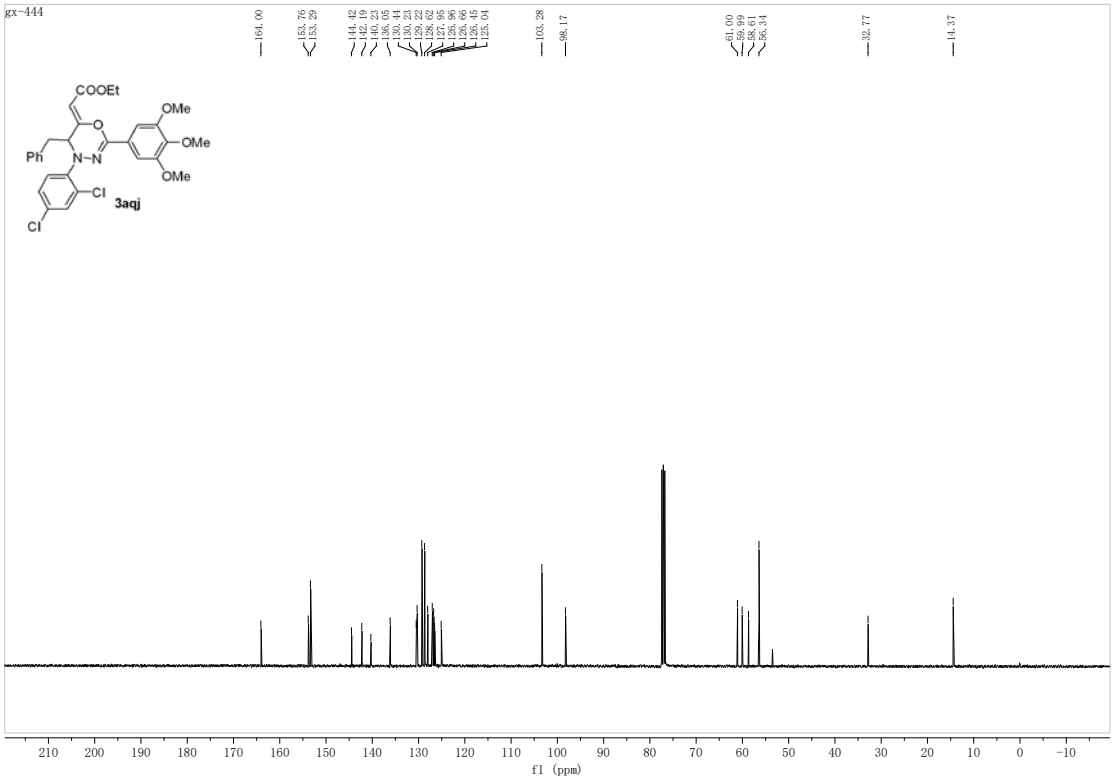
153.71
153.37
153.00
141.77
139.76
138.98
137.20
135.48
129.47
128.72
128.61
127.97
127.49
117.08
115.50
115.89
103.02
97.97
70.56
61.01
59.93
57.35
56.33
33.73
14.41
COOEt
OMe
OMe
OMe
Ph
N-N
O
BnO
3aoj
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)


gx-455-500M
COOEt
OMe
OMe
OMe
Ph
N-N
F
F
3apj
f1 (ppm)


gx-455-500M
COOEt
OMe
OMe
OMe
Ph
N-N
F
F
3apj
f1 (ppm)


gx-497-
497
-116.79
-119.25
COOEt
OMe
OMe
OMe
Ph
N-N
O
F
F
3apj
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)


gx-444
7.59
7.38
7.42
7.42
7.37
7.30
7.28
7.28
7.26
7.24
7.22
7.21
7.20
7.18
7.00
7.00
6.98
5.29
4.74
4.65
4.64
4.63
4.61
4.19
4.17
3.98
3.92
2.89
2.86
2.85
2.83
2.82
2.81
2.79
1.64
1.28
1.26
1.25
COOEt
OMe
OMe
OMe
Ph
N-N
O
Cl
Cl
3aqj
1.08
0.92
1.09
1.05
3.01
2.07
1.29
1.00
1.04
2.20
6.23
3.04
2.12
3.40
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)


gx-444
164.00
153.76
153.29
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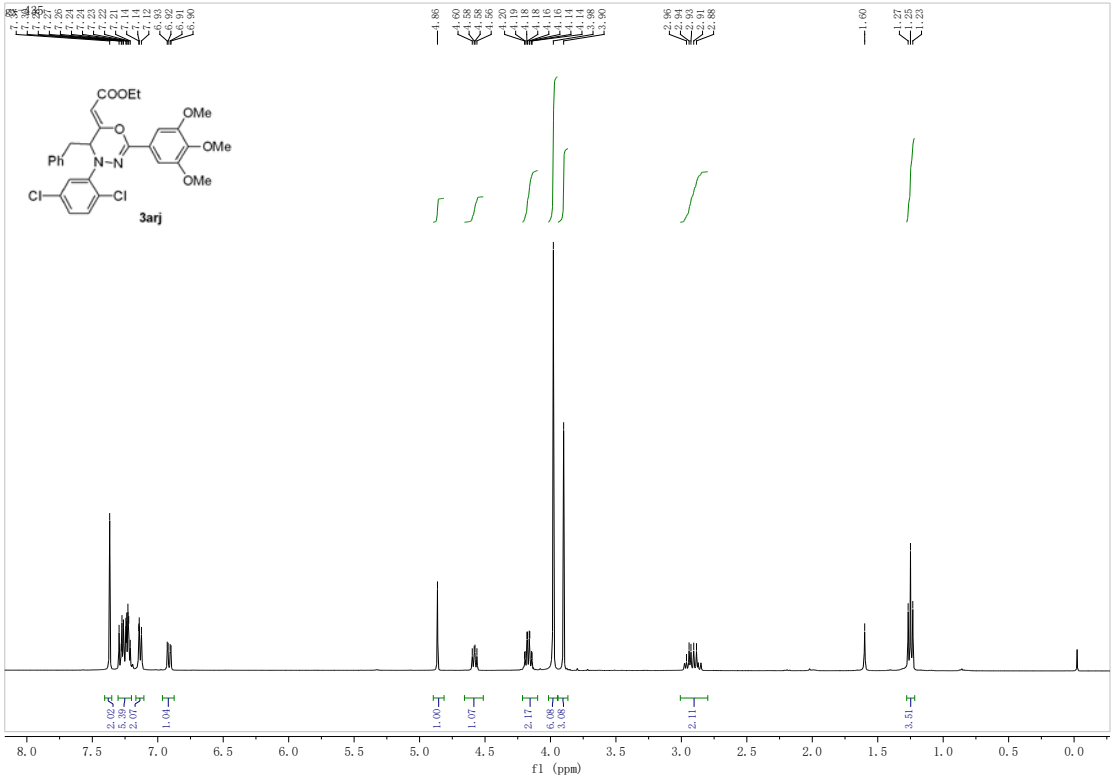
142.19
140.23
136.05
130.44
129.23
128.62
127.95
126.96
126.65
125.45
125.04
103.28
98.17
61.00
59.99
58.61
56.34
32.77
14.37
COOEt
OMe
OMe
OMe
Ph
N-N
Cl
Cl
3aqj
f1 (ppm)


gx-435
4.86
4.60
4.58
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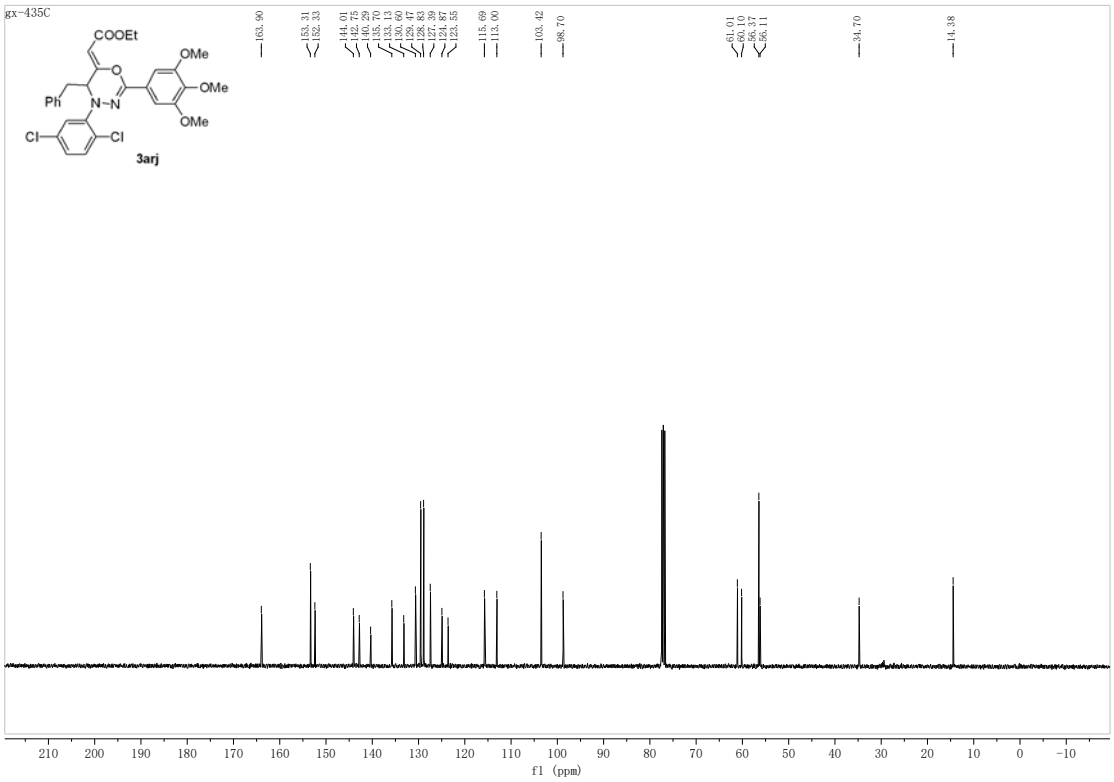
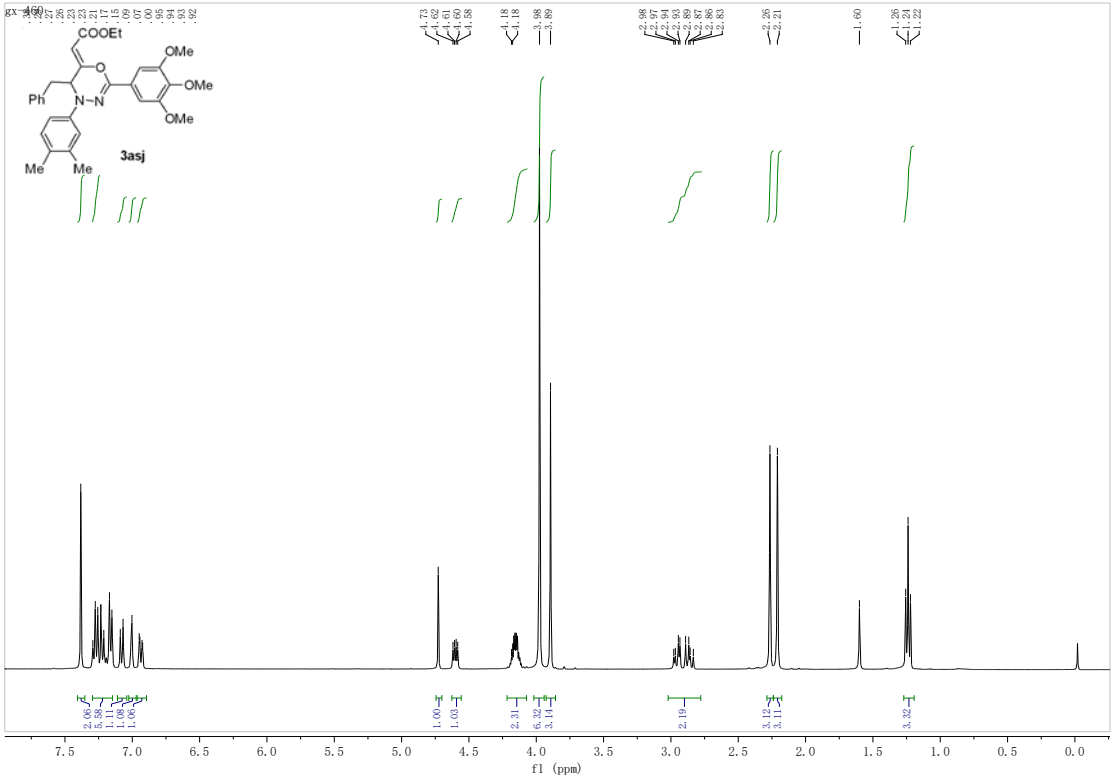
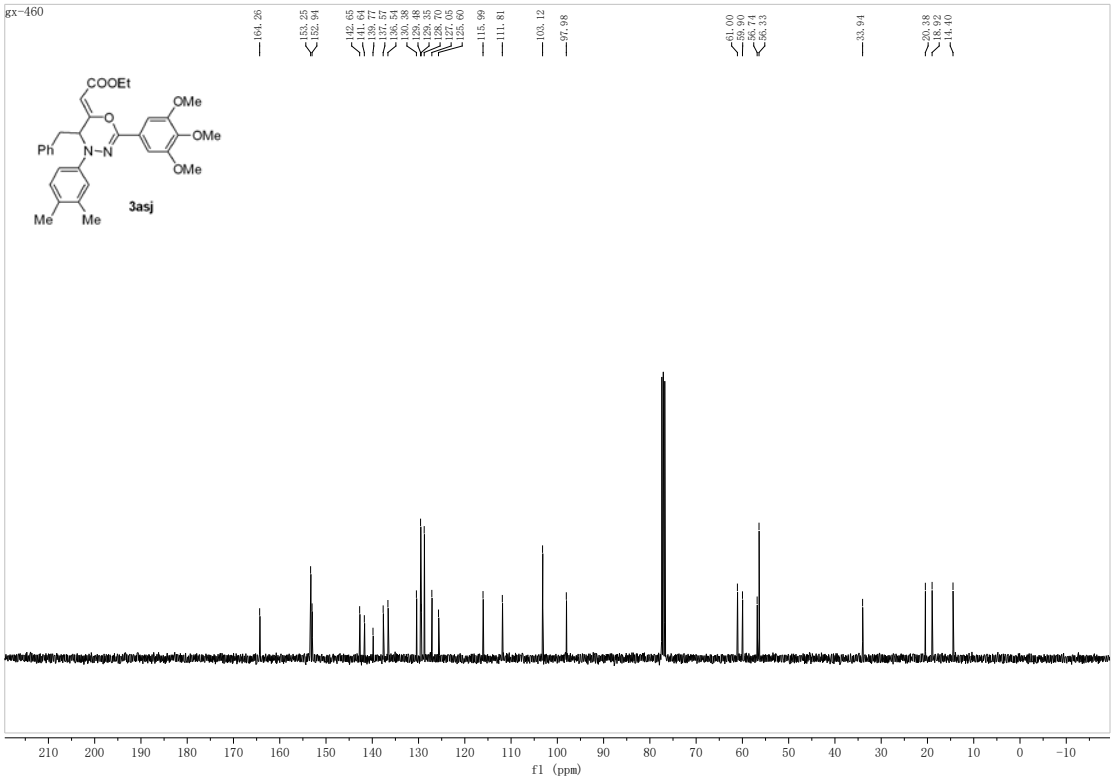
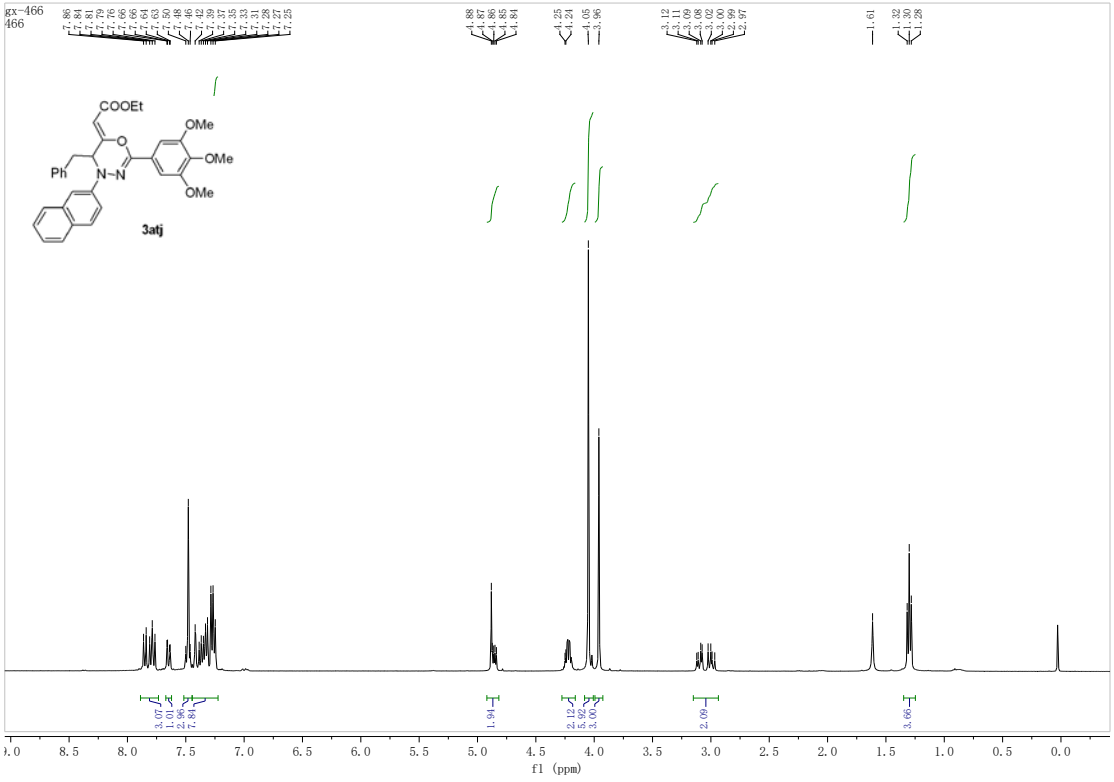
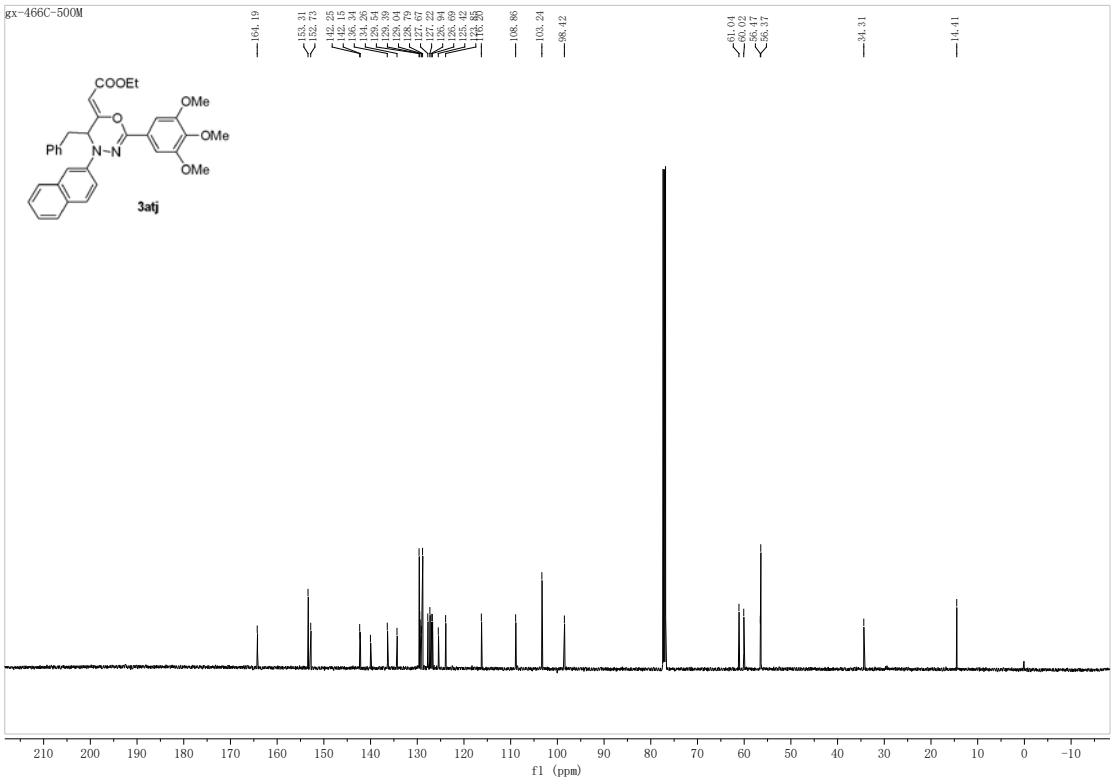
4.20
4.19
4.18
4.16
4.14
3.98
3.96
2.96
2.94
2.93
2.91
2.88
1.60
1.27
1.25
1.23
COOEt
OMe
OMe
OMe
Ph
N-N
Cl
Cl
3arj
2.02
5.39
2.07
1.04
1.00
1.07
2.17
6.08
3.08
2.11
3.51
f1 (ppm)

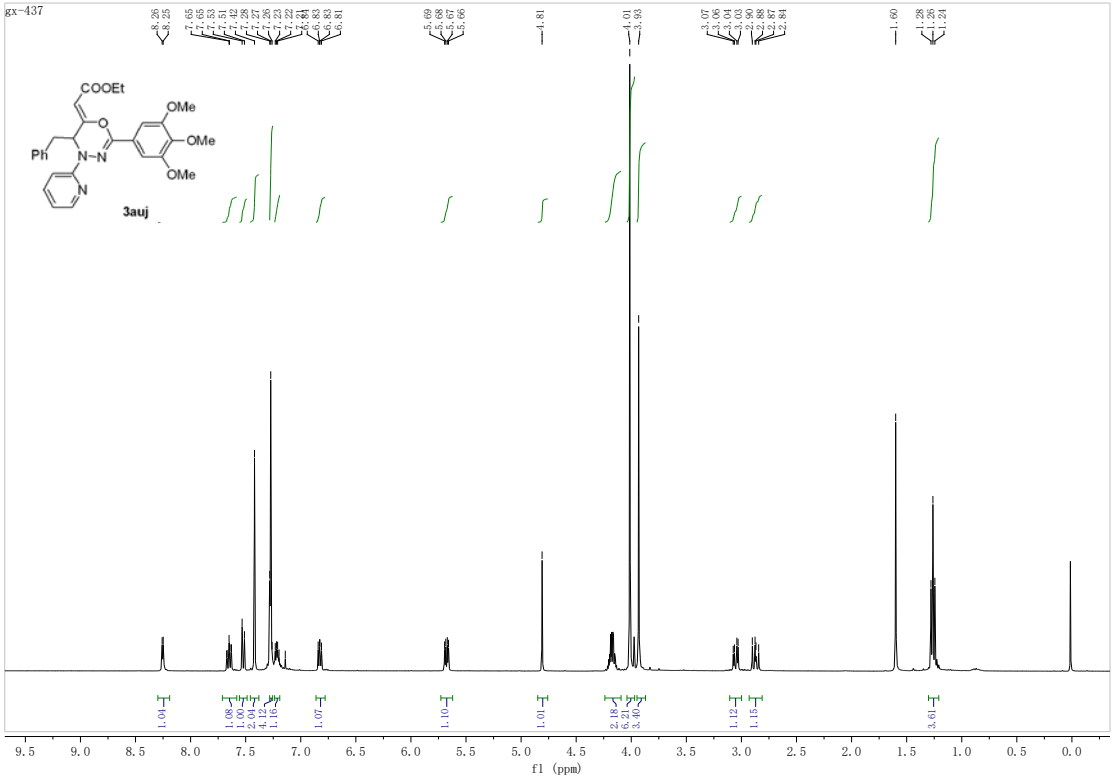

gx-435C
COOEt
OMe
OMe
OMe
Ph
N-N
Cl
Cl
3arj
163.90
153.31
152.33
144.01
142.75
140.29
135.70
133.13
130.60
128.47
128.05
127.39
124.87
123.55
115.69
113.00
103.42
88.70
61.01
60.10
56.37
56.11
34.70
14.38
f1 (ppm)

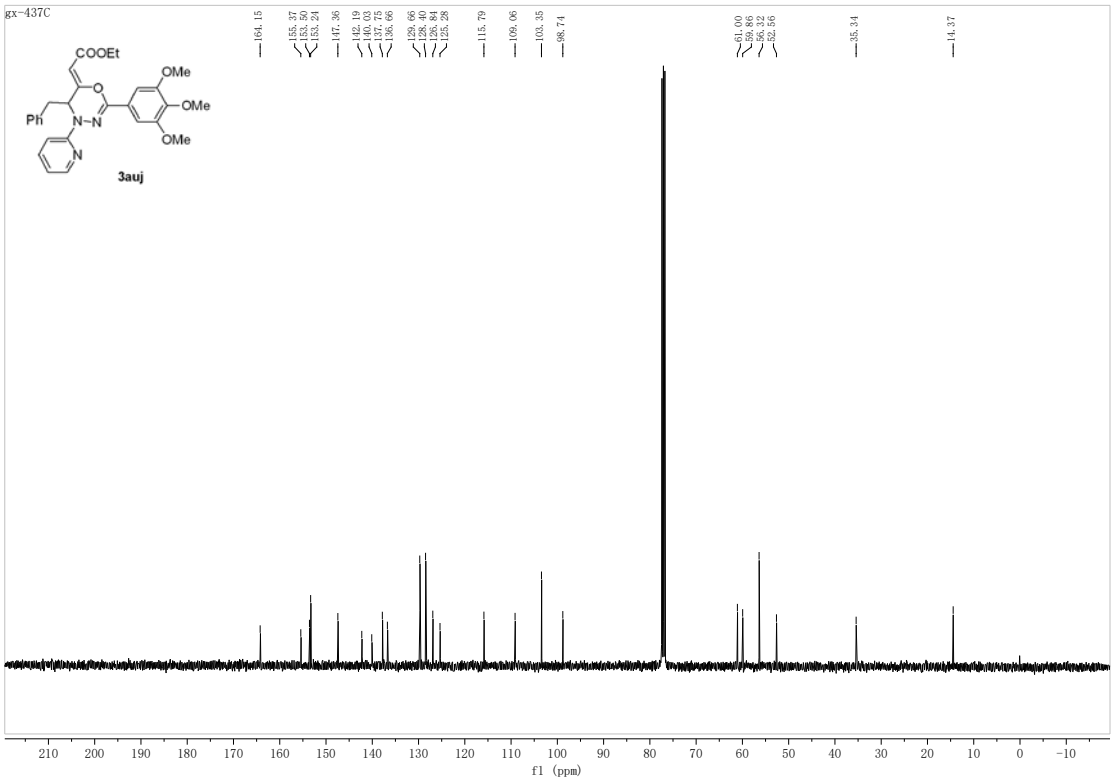

gx-460
COOEt
OMe
OMe
OMe
Ph
N-N
Me
Me
3asj
f1 (ppm)


gx-460
COOEt
OMe
OMe
OMe
Ph
N-N
Me
Me
3asj
f1 (ppm)


gx-466
466
COOEt
OMe
OMe
OMe
Ph
N-N
3atj
f1 (ppm)


gx-466C-500M
COOEt
OMe
OMe
OMe
Ph
N-N
3atj
f1 (ppm)


gx-437
COOEt
OMe
OMe
OMe
Ph
N-N
N
3auj
f1 (ppm)


gx-437C
COOEt
OMe
OMe
OMe
Ph
N-N
N
3auj
164.15
155.37
153.50
153.24
147.36
142.19
140.03
137.75
136.66
129.66
128.40
125.84
125.28
115.79
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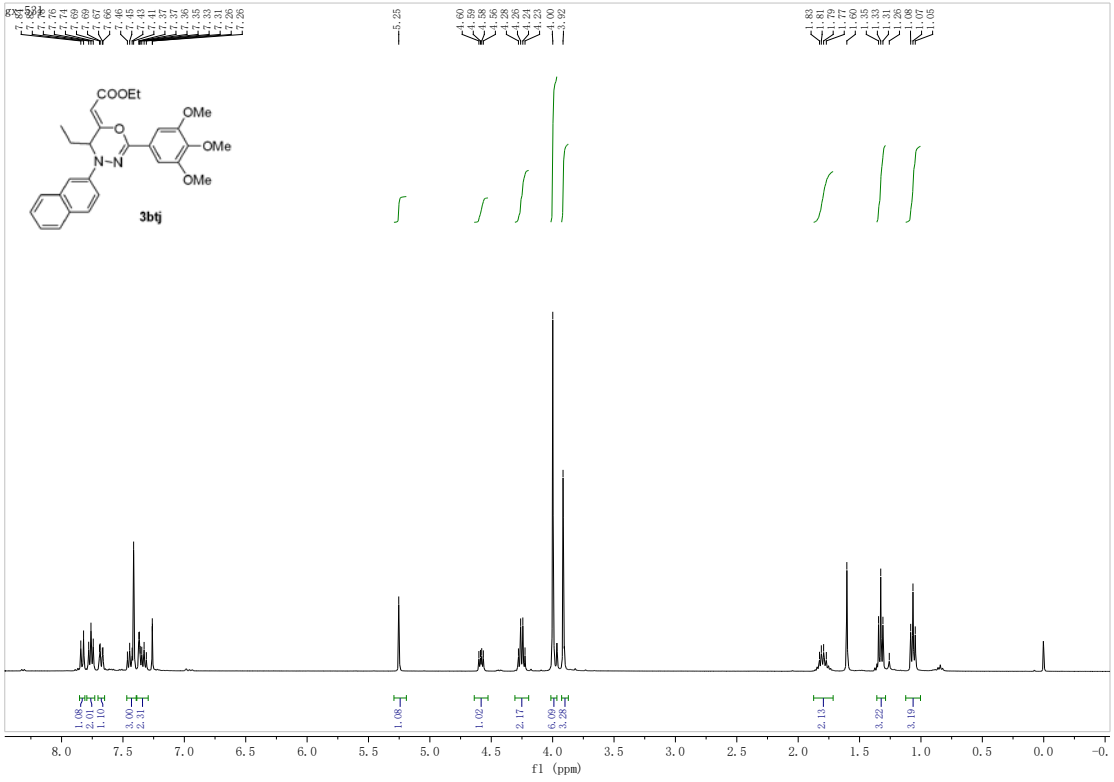
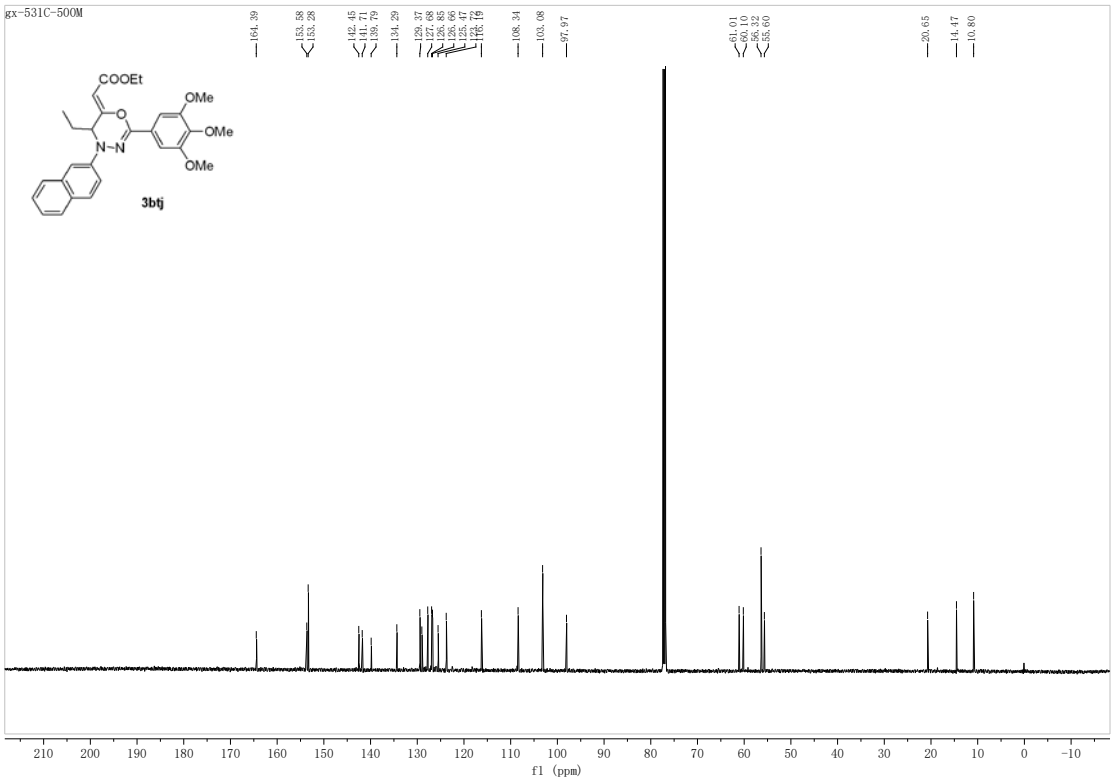
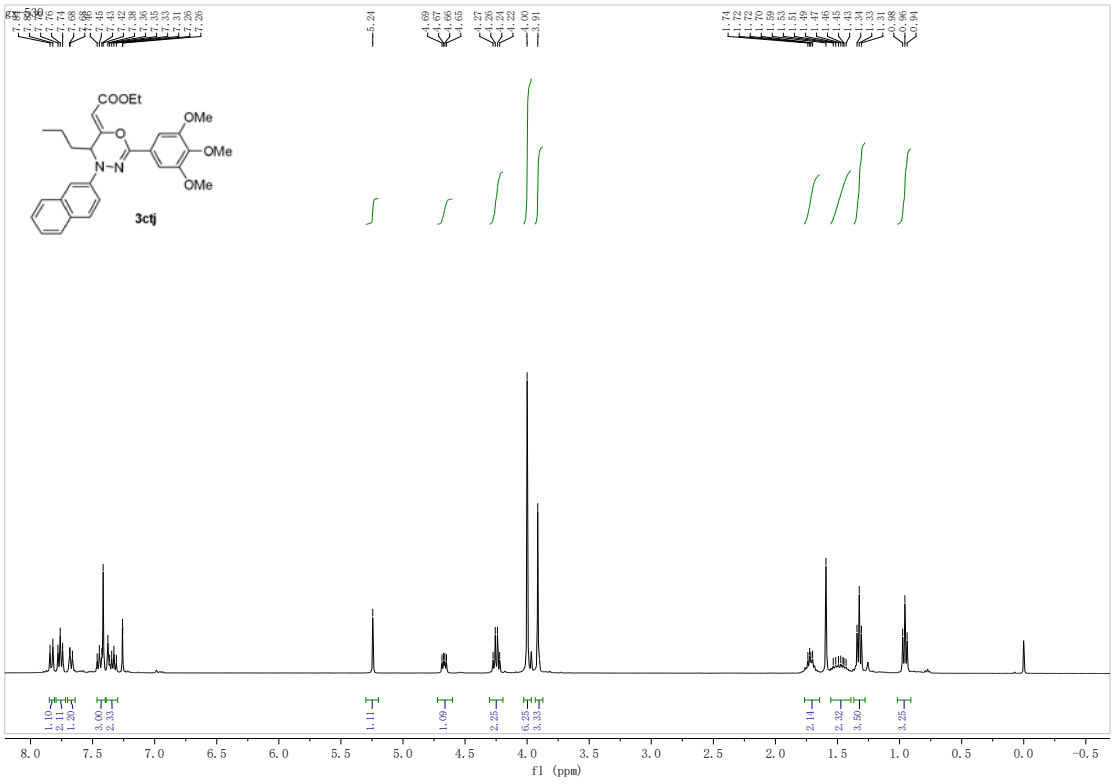
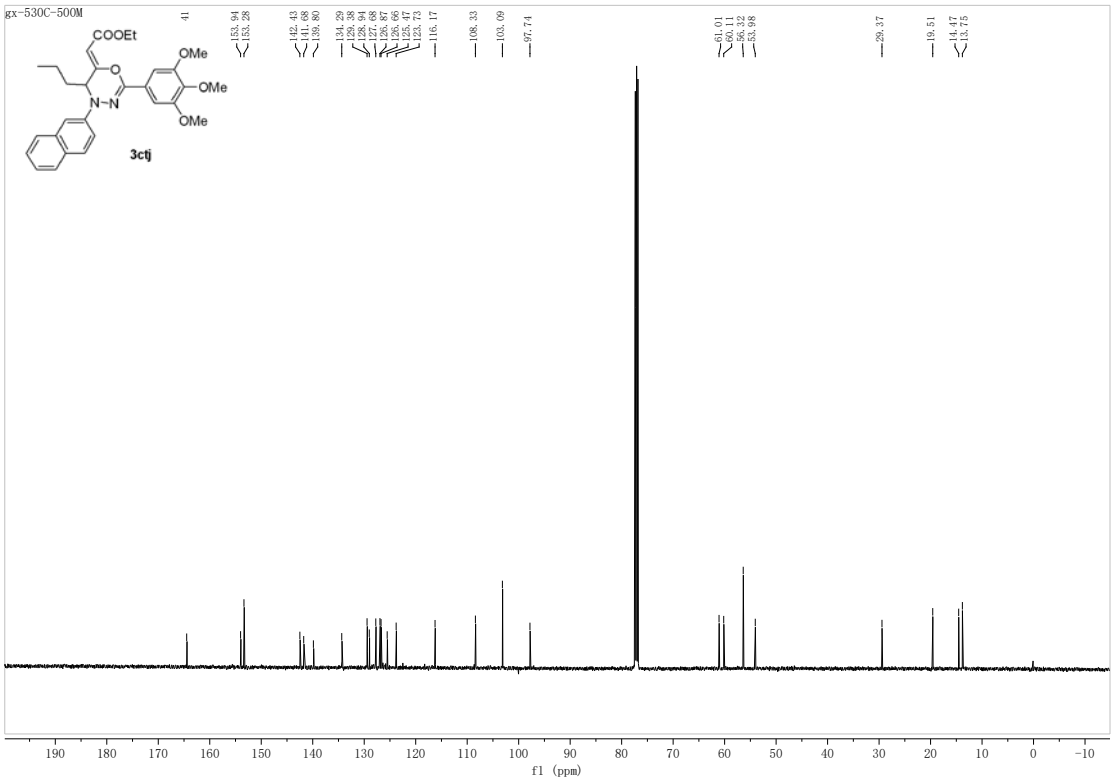
103.35
88.74
61.00
59.86
55.32
52.56
35.34
14.37
f1 (ppm)


COOEt
OMe
OMe
OMe
N-N
3btj
5.25
4.60
4.59
4.58
4.26
4.24
4.23
4.00
3.92
1.83
1.81
1.79
1.77
1.60
1.35
1.33
1.31
1.08
1.07
1.05
f1 (ppm)


gx-531C-500M
COOEt
OMe
OMe
OMe
N-N
3btj
f1 (ppm)


COOEt
OMe
OMe
OMe
N-N
3ctj
f1 (ppm)


gx-530C-500M
COOEt
OMe
OMe
OMe
N-N
O
3ctj
41
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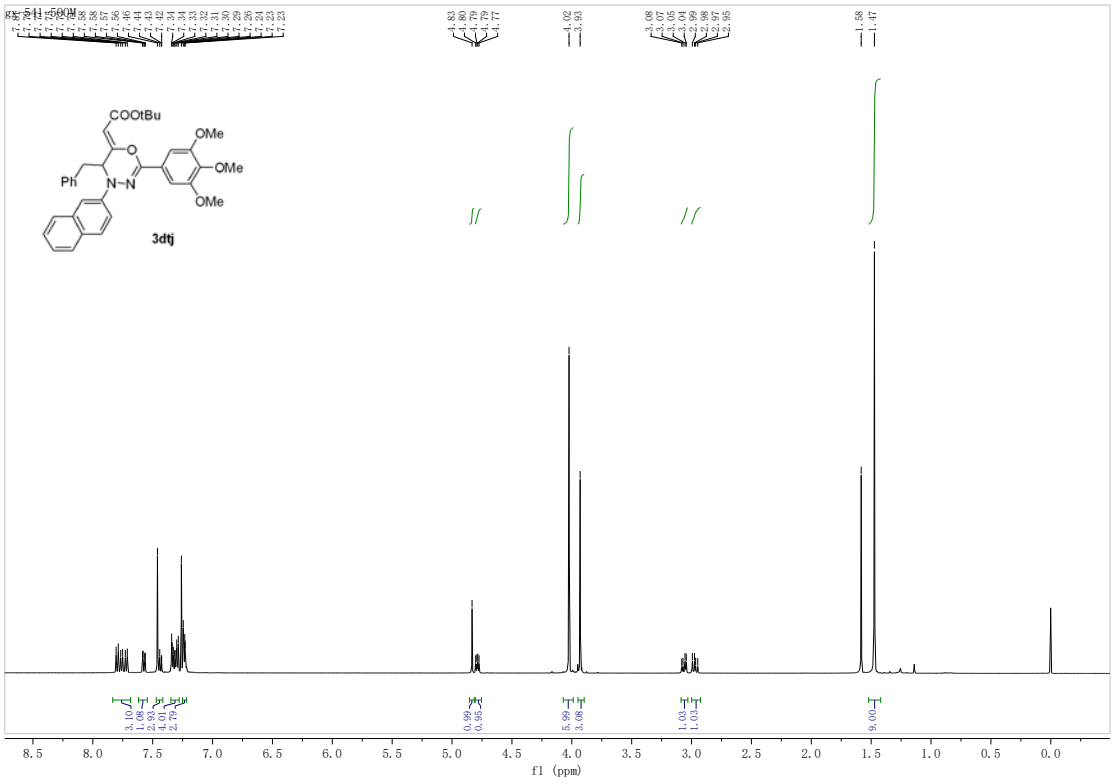
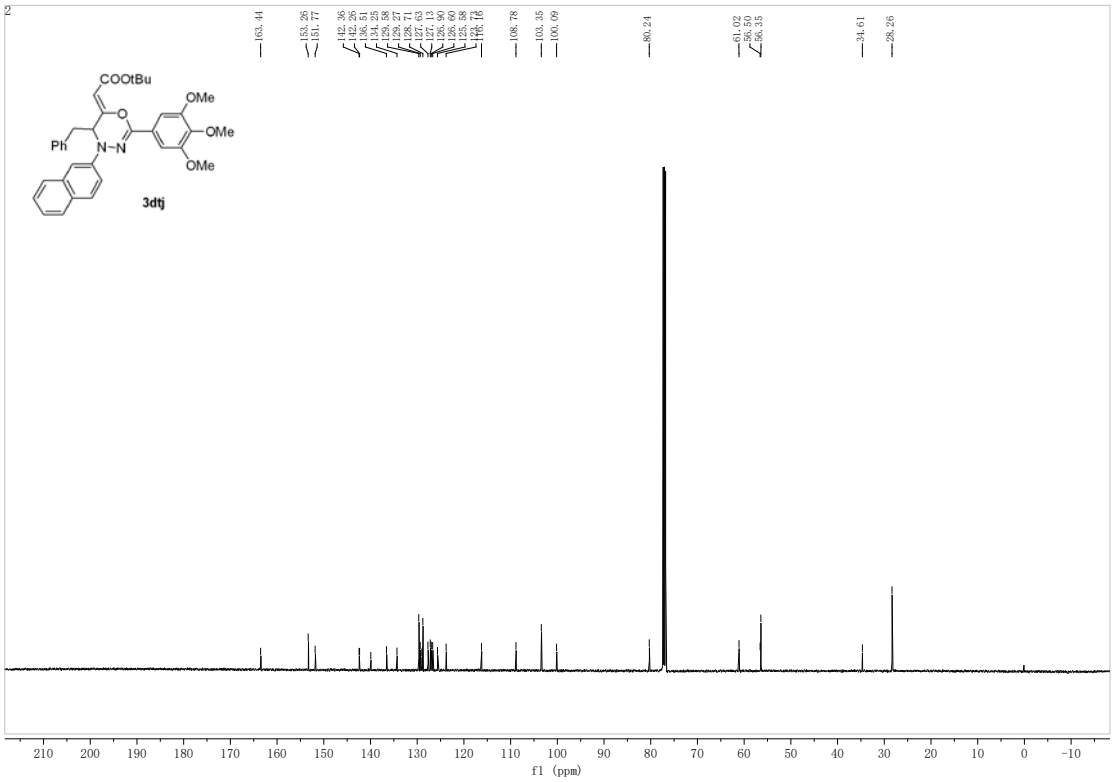
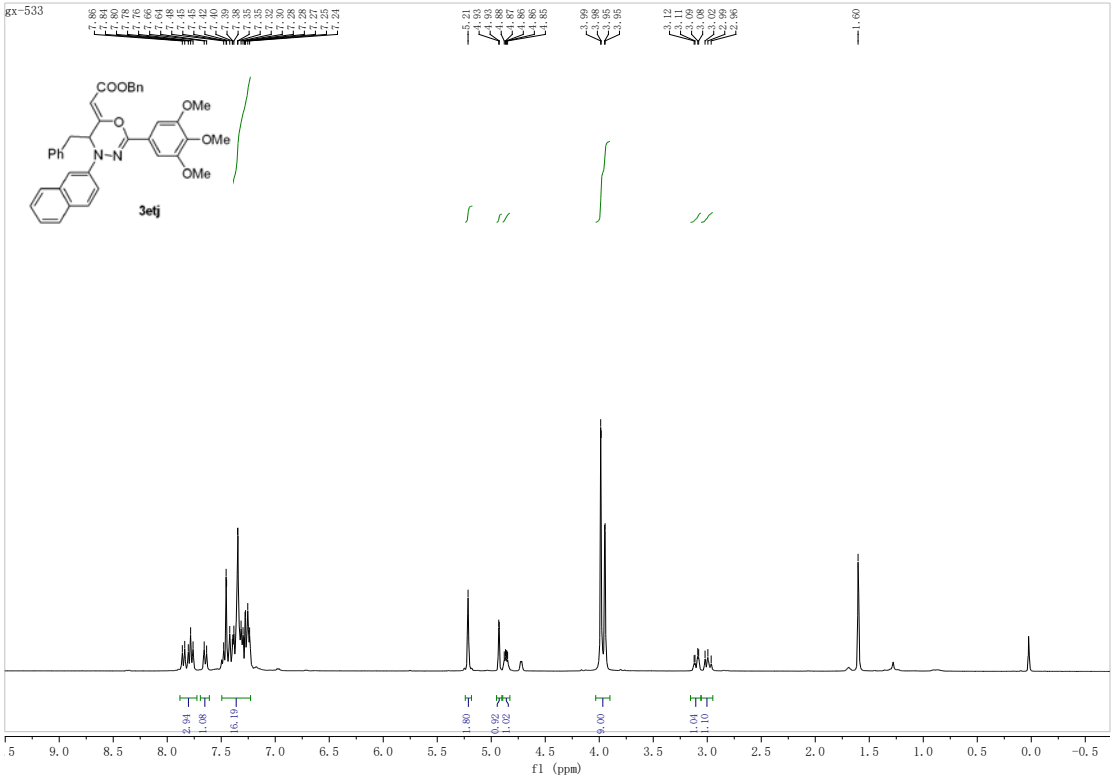
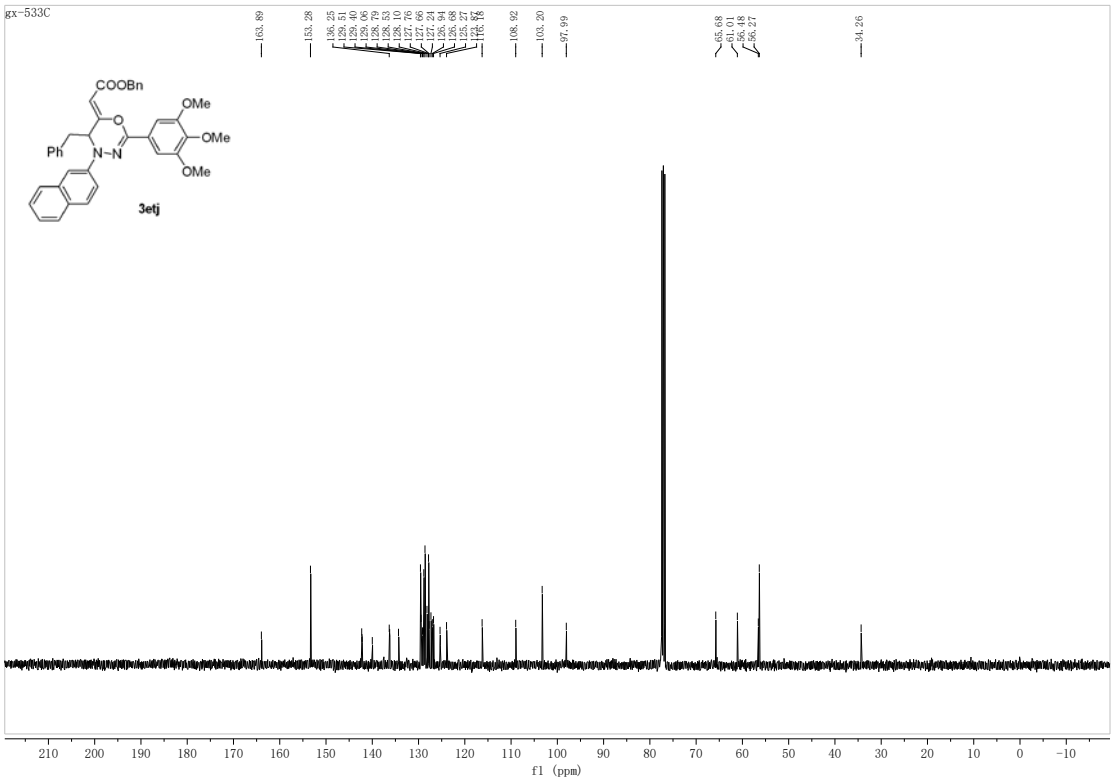
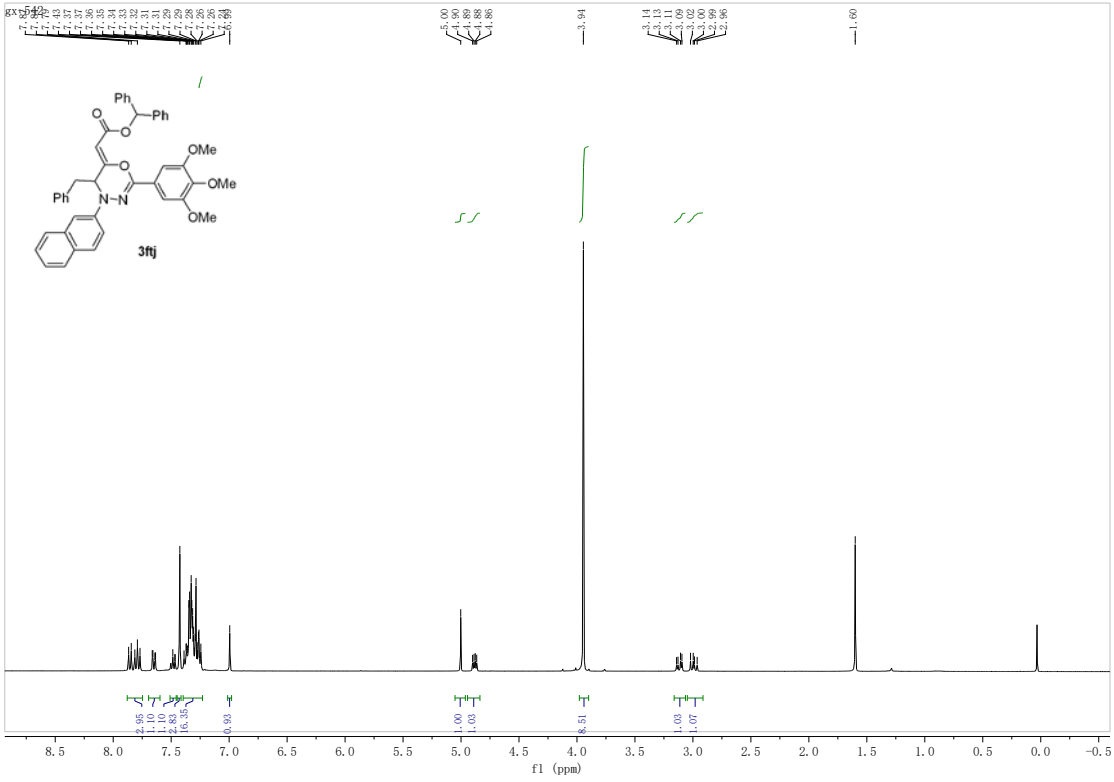
153.28
142.43
141.68
139.80
134.29
129.38
128.94
127.68
126.66
125.47
123.73
116.17
108.33
103.09
97.74
61.01
60.11
56.12
53.98
29.37
19.51
14.47
13.75
f1 (ppm)

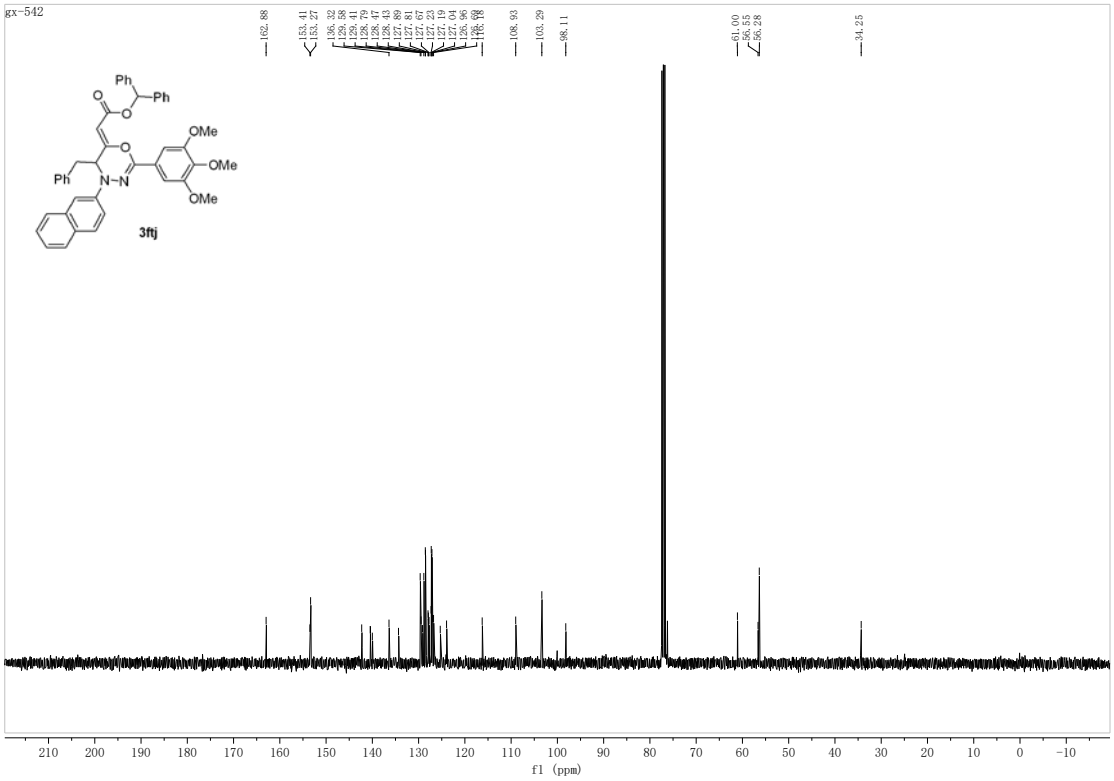

COOtBu
OMe
OMe
OMe
Ph
N-N
O
3dtj
4.83
4.80
4.79
4.79
4.77
4.02
3.93
3.08
3.07
3.05
3.04
2.99
2.98
2.97
2.95
1.68
1.47
3.10
1.08
2.93
4.01
2.79
0.99
0.95
5.99
3.08
1.03
1.03
9.00
f1 (ppm)

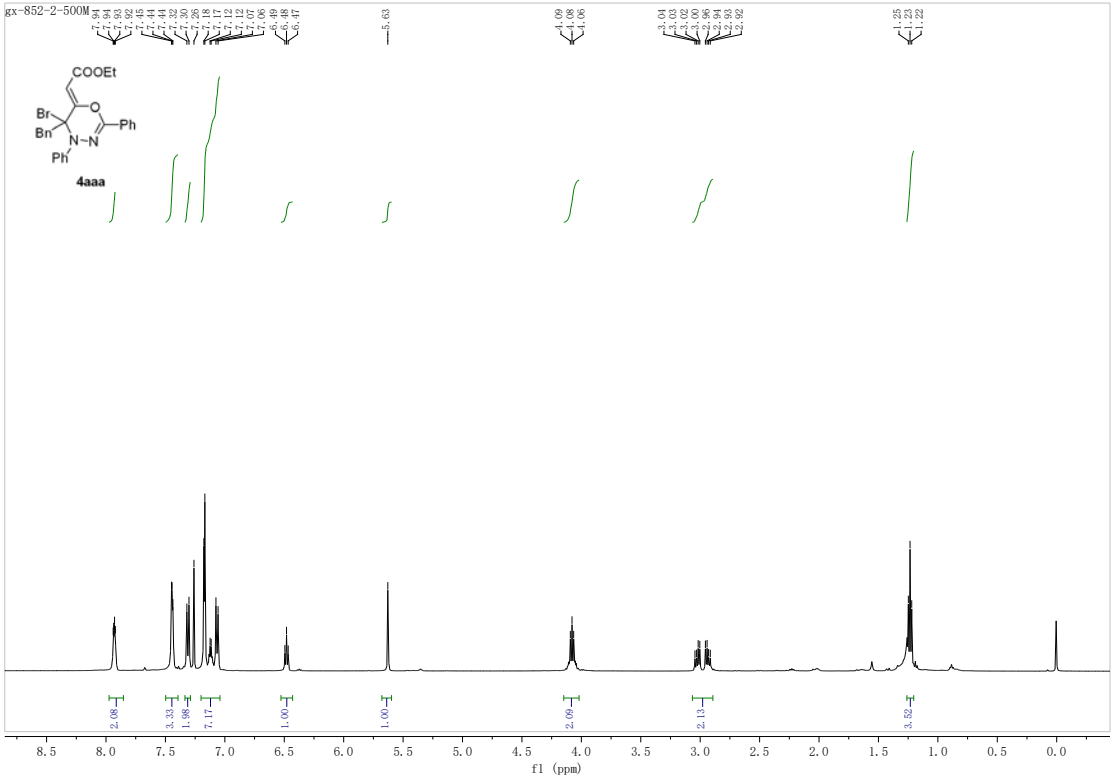

2
COOtBu
OMe
OMe
OMe
Ph
N-N
3dtj
f1 (ppm)

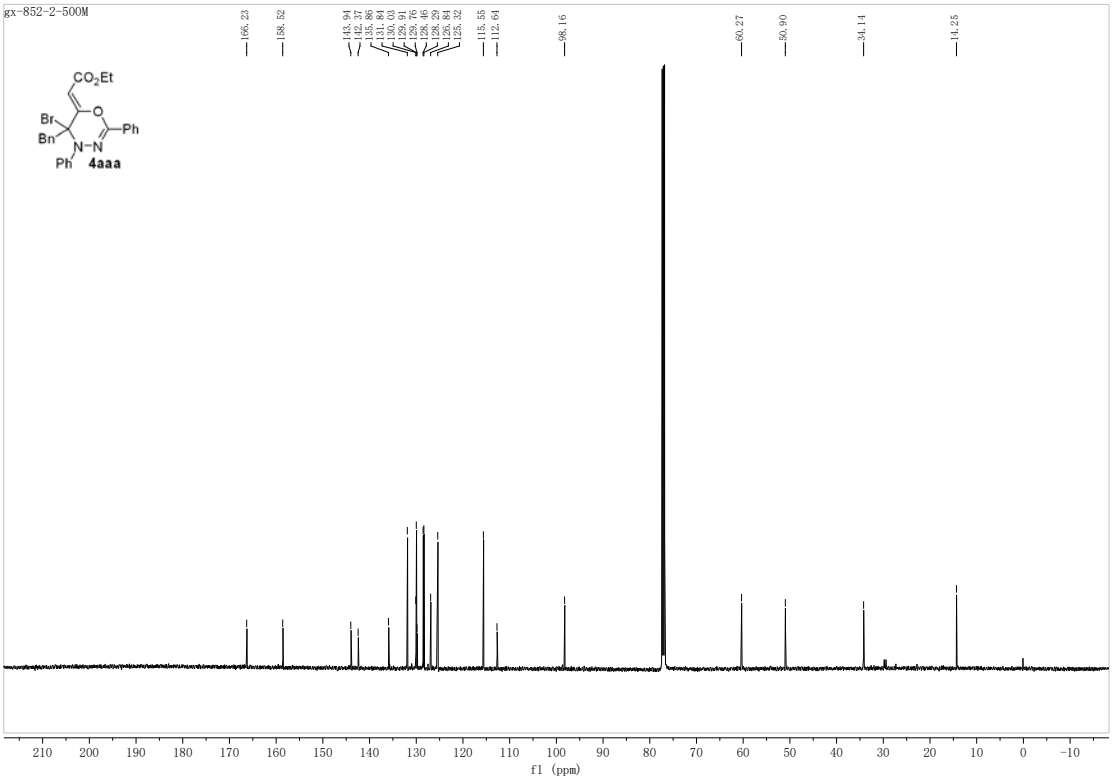

gx-533
COOBn
OMe
OMe
OMe
Ph
N-N
3etj
f1 (ppm)

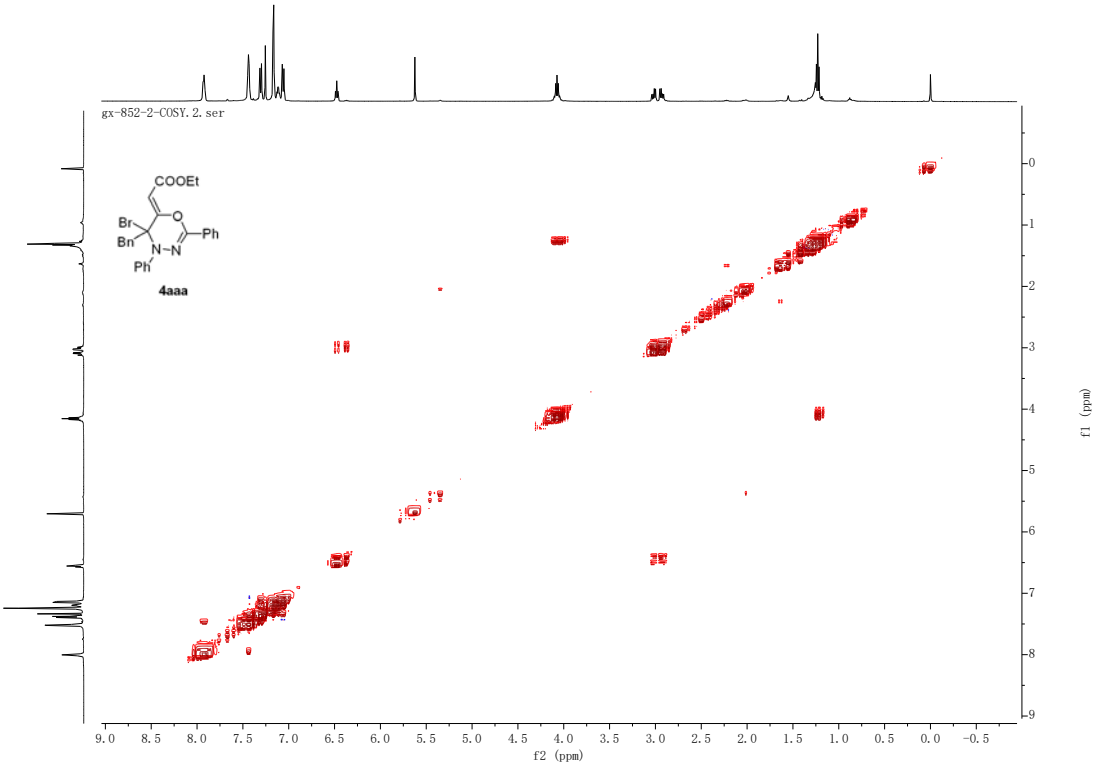

gx-533C
163.89
153.28
136.25
129.51
129.40
129.05
128.79
128.53
128.10
127.76
127.66
127.24
126.94
126.68
125.27
123.87
116.18
108.92
103.20
97.99
65.68
57.01
56.18
56.27
34.26
COOBn
OMe
OMe
OMe
Ph
N-N
3etj
f1 (ppm)

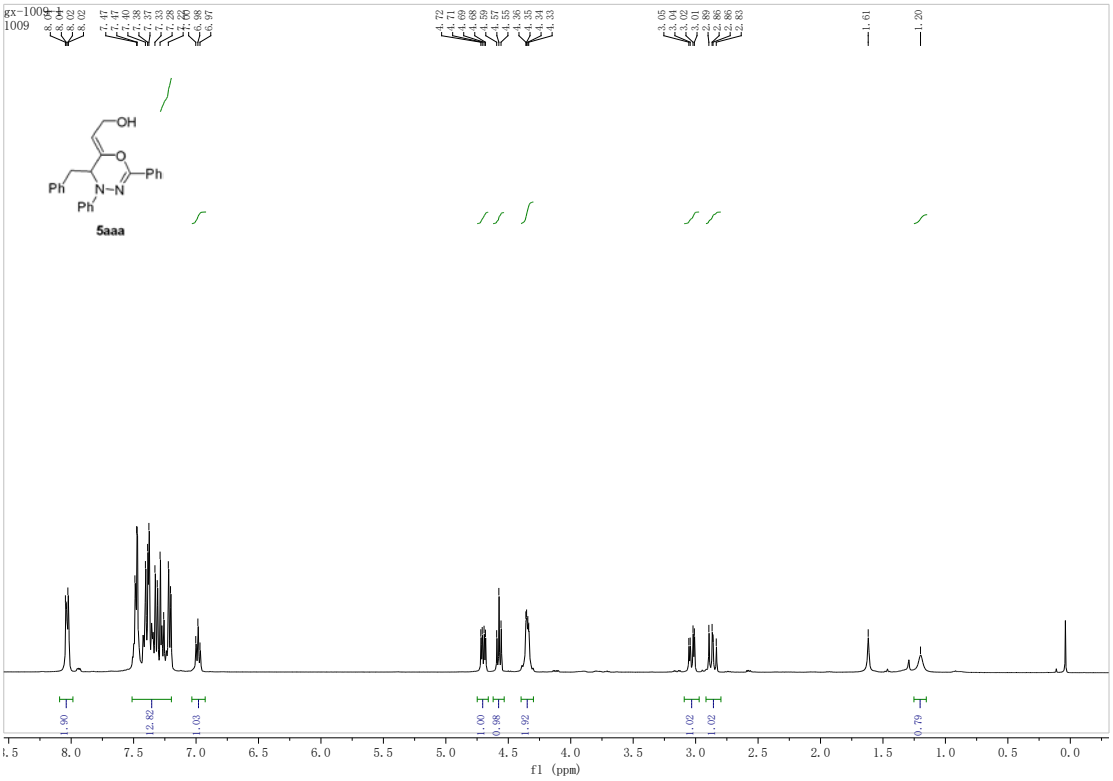

gx-542
5.00
4.90
4.89
4.88
4.86
3.94
3.14
3.13
3.11
3.09
3.02
3.00
2.99
2.96
1.60
Ph
Ph
OMe
OMe
OMe
Ph
N-N
3ftj
2.95
1.10
1.10
2.83
16.35
0.93
1.00
1.03
8.51
1.03
1.07
f1 (ppm)

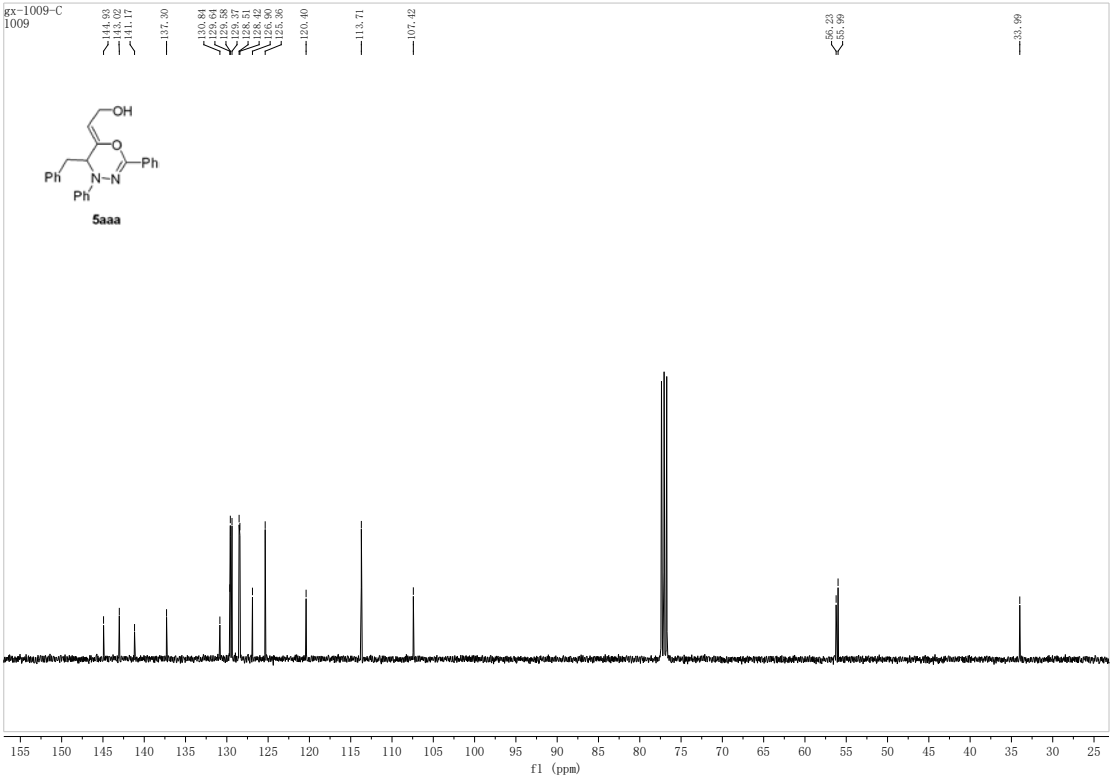

gx-542
Ph
Ph
O
O
OMe
OMe
OMe
Ph
N-N
3ftj
f1 (ppm)

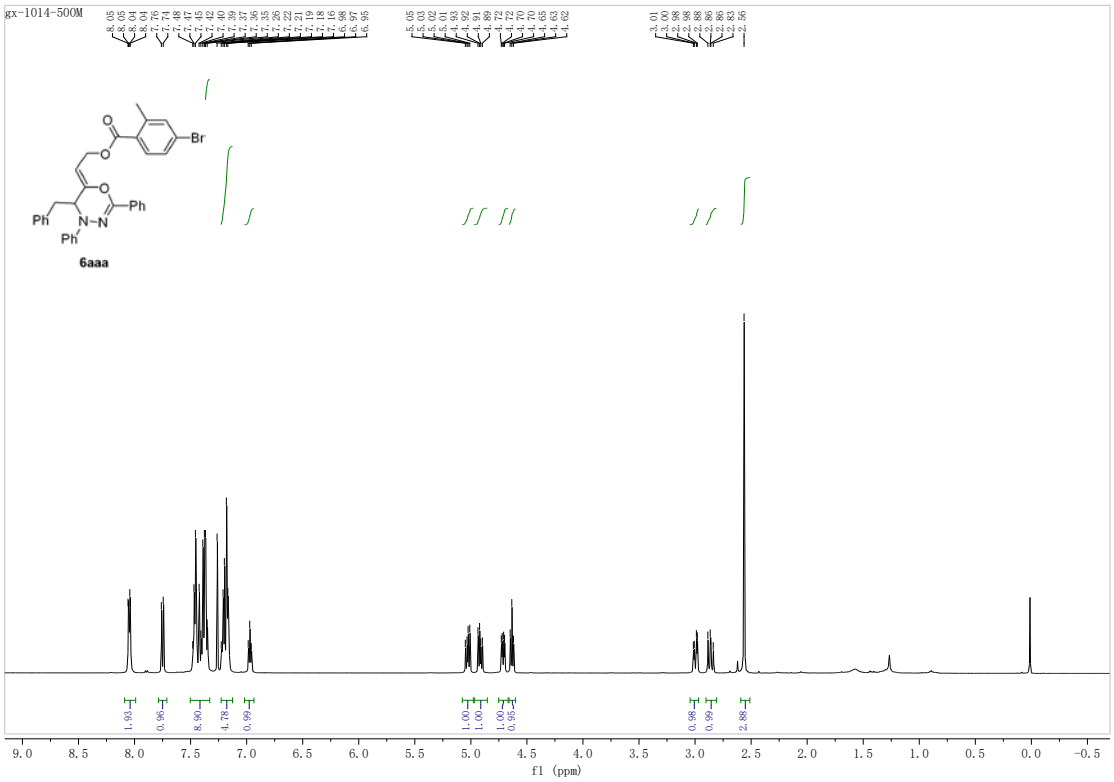

gx-852-2-500M
COOEt
Br
Bn
Ph
N-N
Ph
4aaa
f1 (ppm)

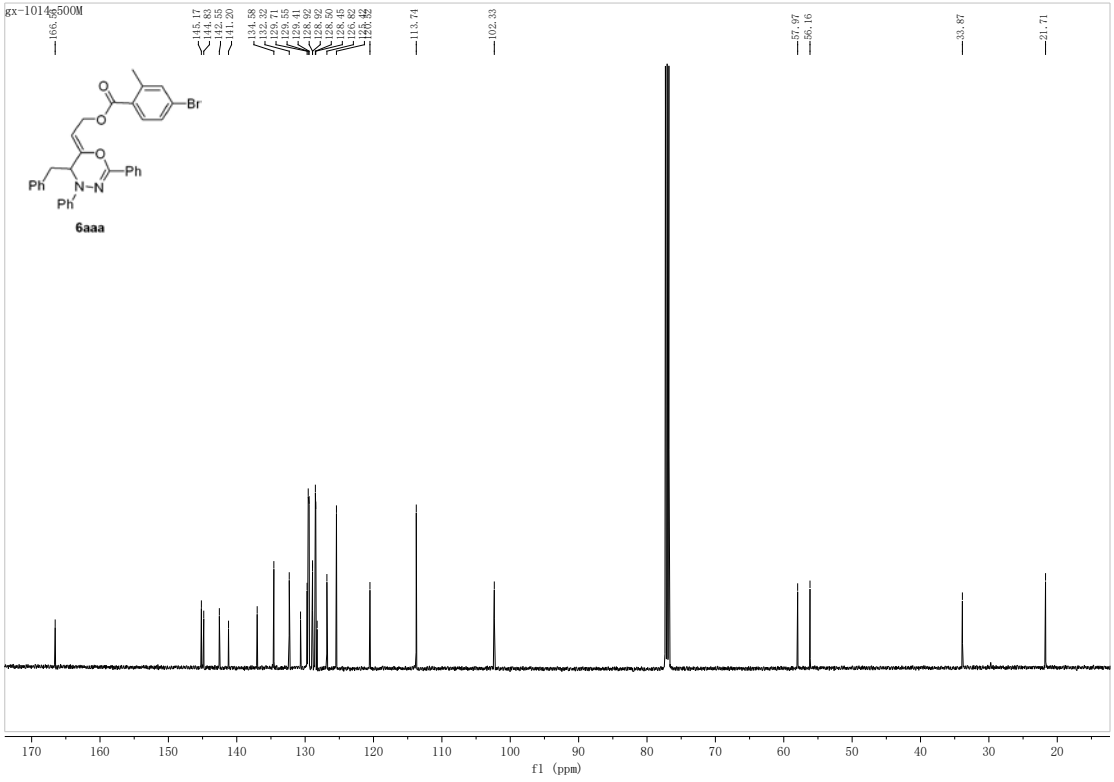

gx-852-2-500M
166.23
158.52
143.94
142.37
135.86
131.84
130.03
129.91
129.76
128.46
128.29
126.84
125.32
115.55
112.64
98.16
60.27
50.90
34.14
14.25
CO2Et
Br
Bn
N-N
Ph
Ph
4aaa
f1 (ppm)

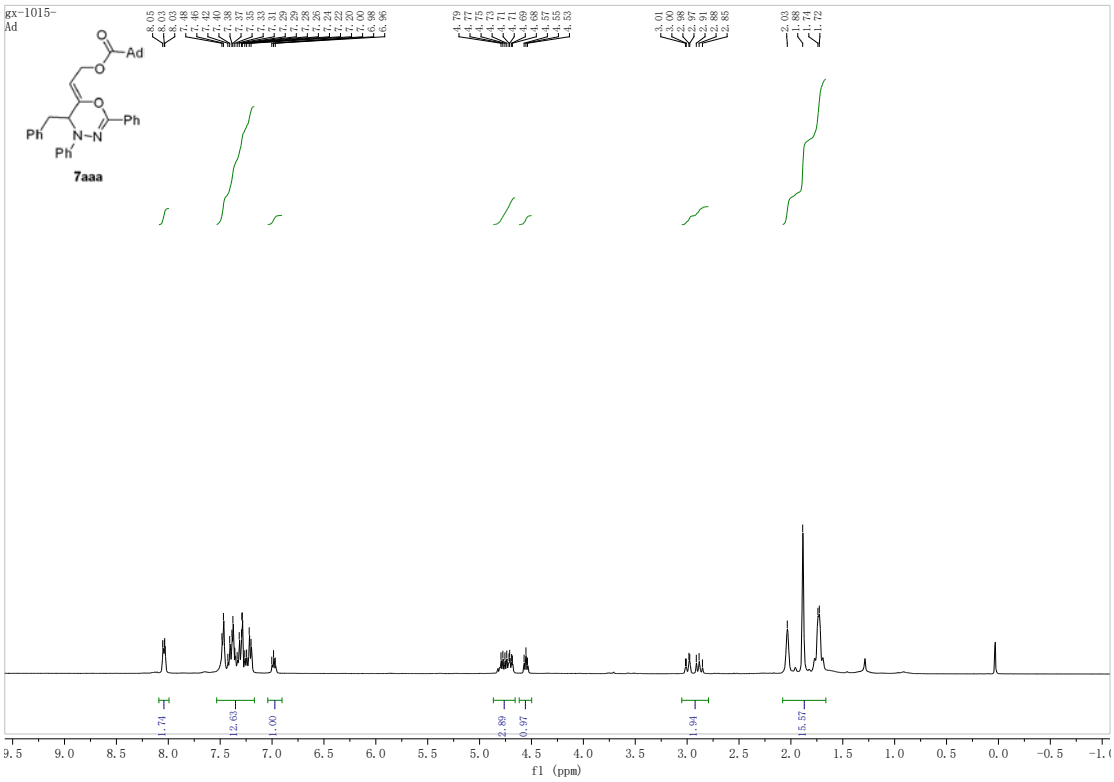

gx-852-2-COSY.2.ser
COOEt
Br
Bn
N-N
Ph
Ph
4aaa
f2 (ppm)
f1 (ppm)

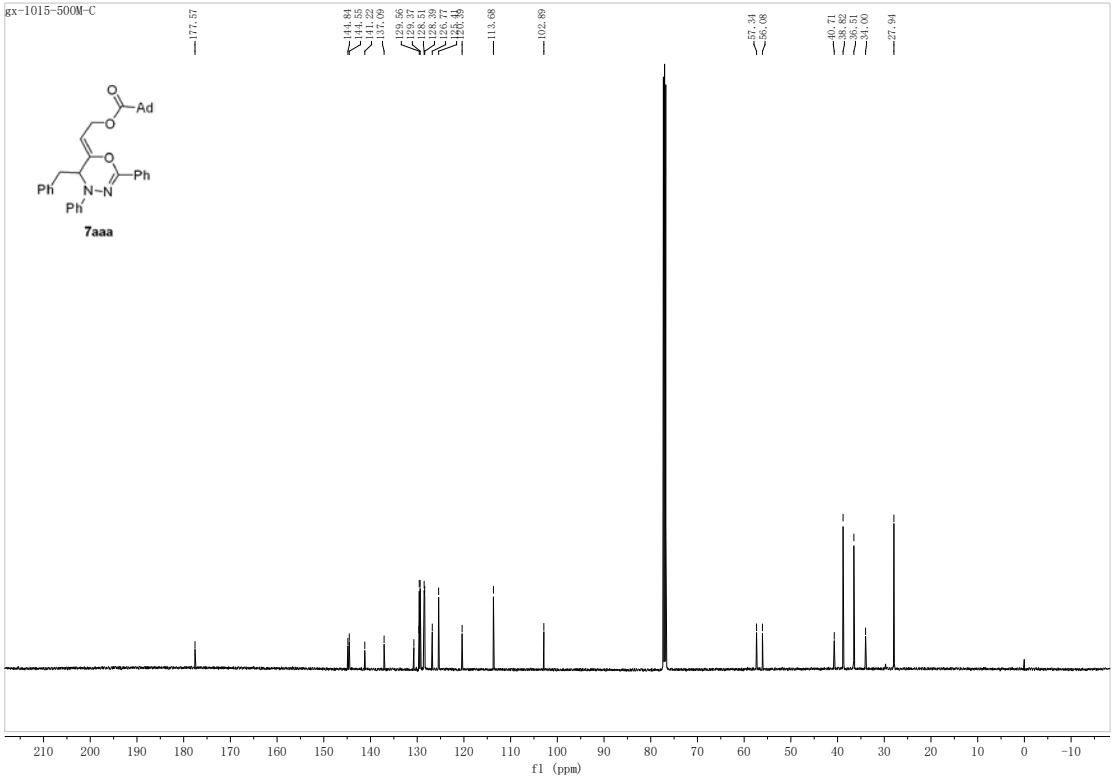

gx-1009-1
1009
OH
O
Ph
Ph
N-N
Ph
5aaa
1.90
12.82
1.03
1.00
0.98
1.92
1.02
1.02
0.79
f1 (ppm)


gx-1009-C
1009
OH
O
Ph
Ph
N-N
Ph
5aaa
f1 (ppm)


gx-1014-500M
6aaa
f1 (ppm)

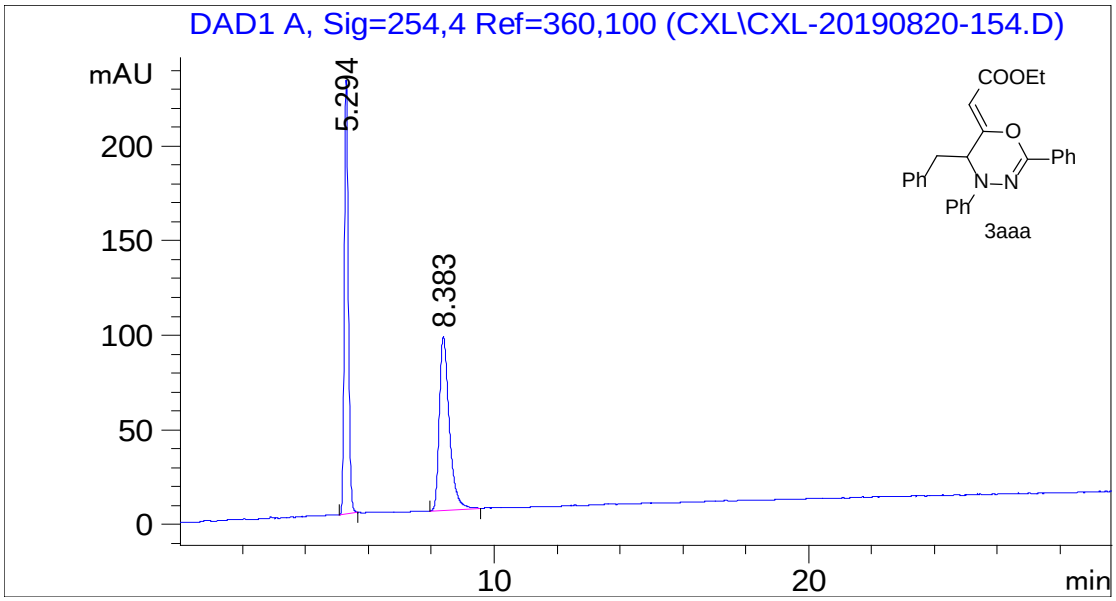

gx-1014-c500M
6aaa
f1 (ppm)


gx-1015-
Ad
7aaa
f1 (ppm)

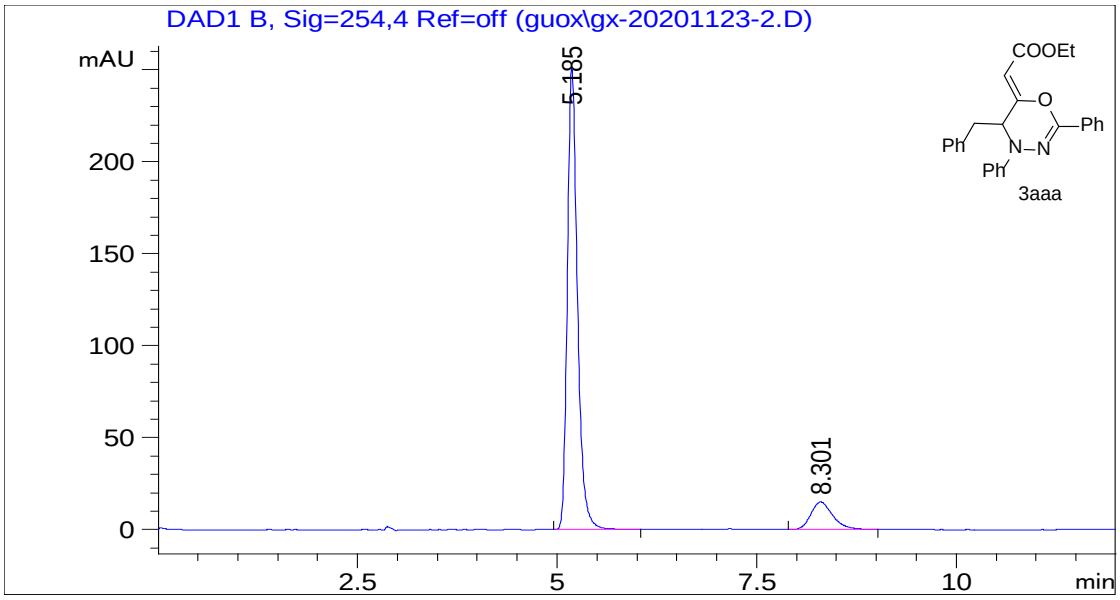

gx-1015-500M-C
7aaa
f1 (ppm)

D: HPLC Analysis

3aaa: OD-H, 90/10, 1.0 ml/min, 254 nm

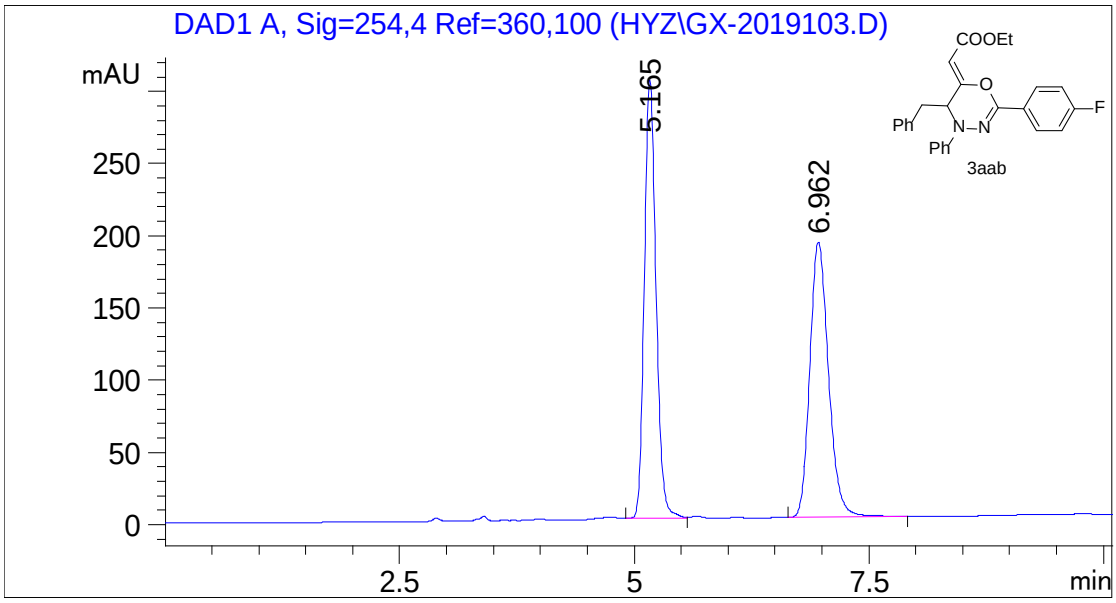


#	Time	Area	Height	Width	Area%	Symmetry
1	5.294	2035.9	229.6	0.1375	50.036	0.823
2	8.383	2033	91.9	0.3361	49.964	0.623

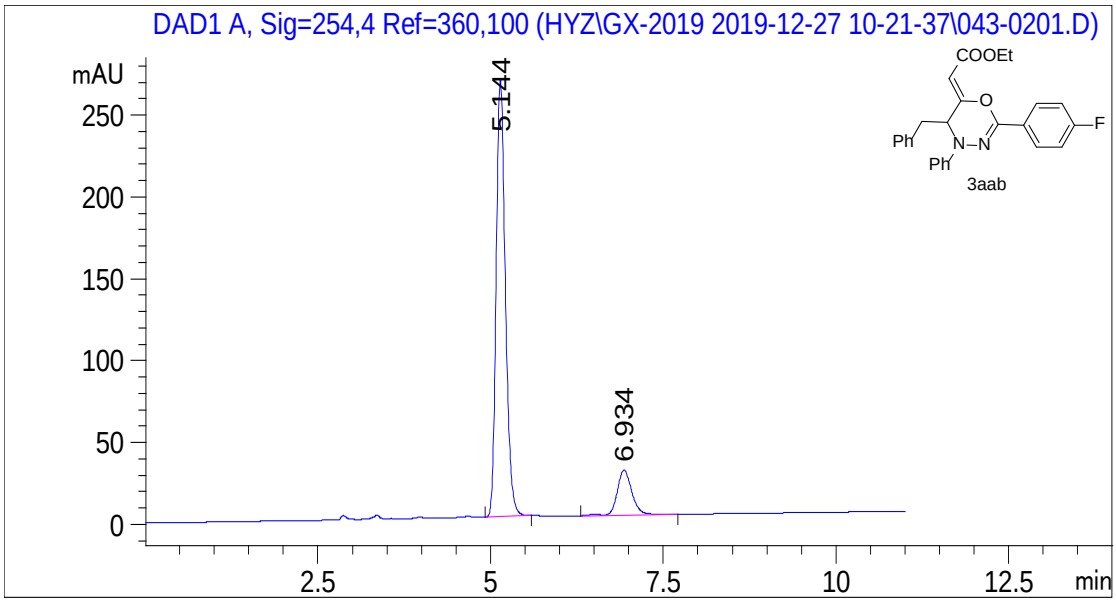


#	Time	Area	Height	Width	Area%	Symmetry
1	5.264	3732.2	420.8	0.1478	85.443	0.857
2	8.353	635.9	28.4	0.3725	14.557	0.727

3aab: OD-H, 90/10, 1.0 ml/min, 254 nm

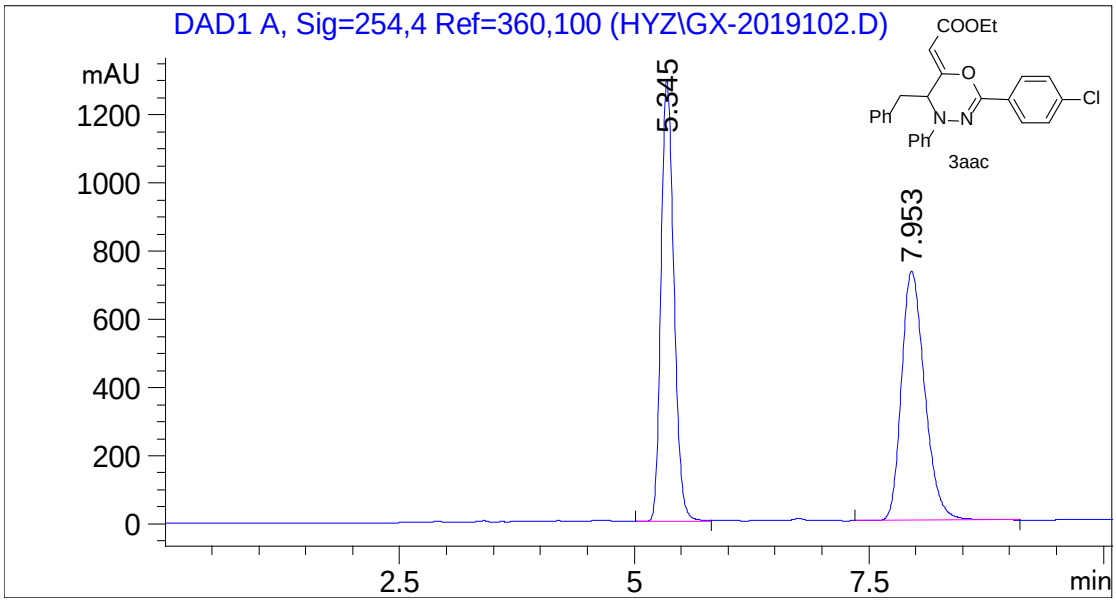


#	Time	Area	Height	Width	Area%	Symmetry
1	5.165	2636.7	303.7	0.1333	49.891	0.811
2	6.962	2648.3	190.7	0.214	50.109	0.772

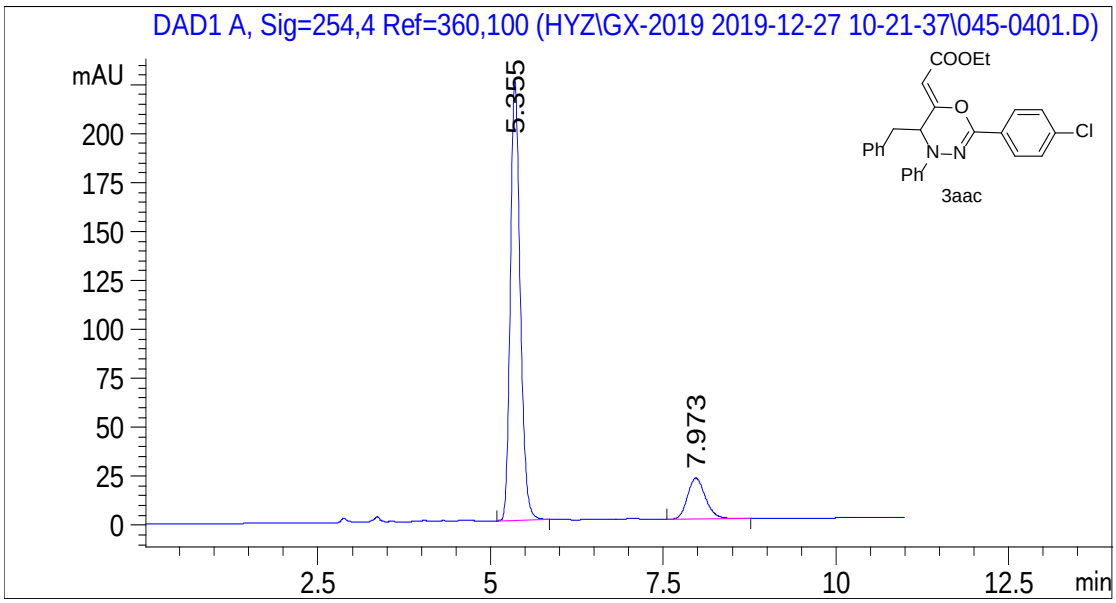


#	Time	Area	Height	Width	Area%	Symmetry
1	5.144	2470.5	267.3	0.1418	85.831	0.771
2	6.934	407.8	27.4	0.2279	14.169	0.825

3aac: OD-H, 90/10, 1.0 ml/min, 254 nm

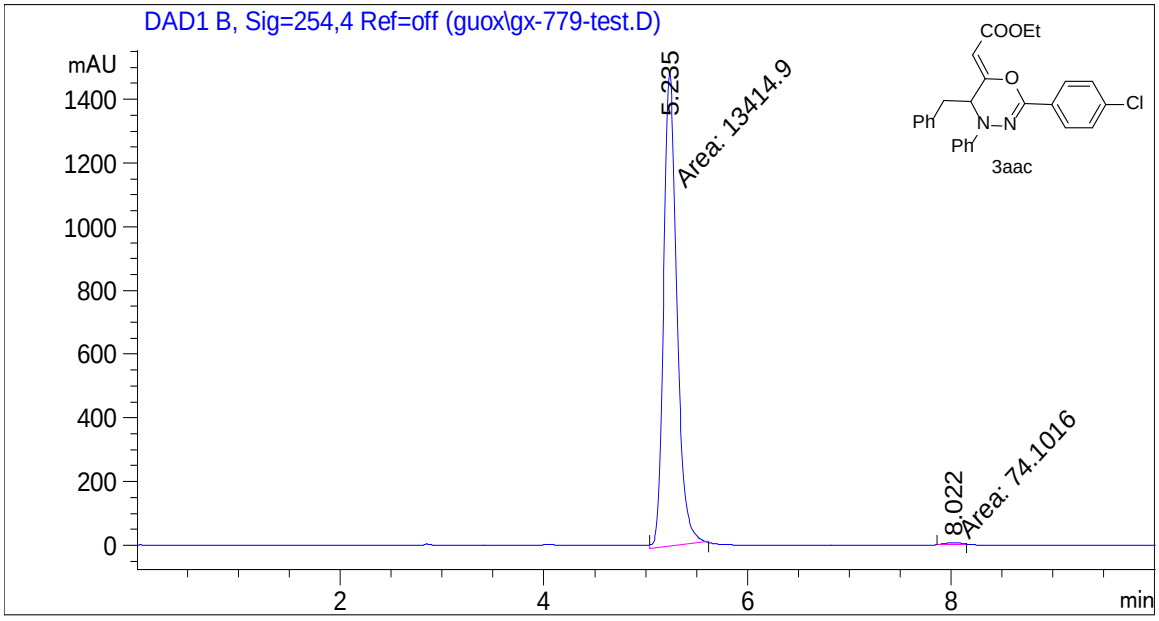


#	Time	Area	Height	Width	Area%	Symmetry
1	5.345	12336	1294.5	0.1492	49.431	0.784
2	7.953	12620	732.1	0.2651	50.569	0.719



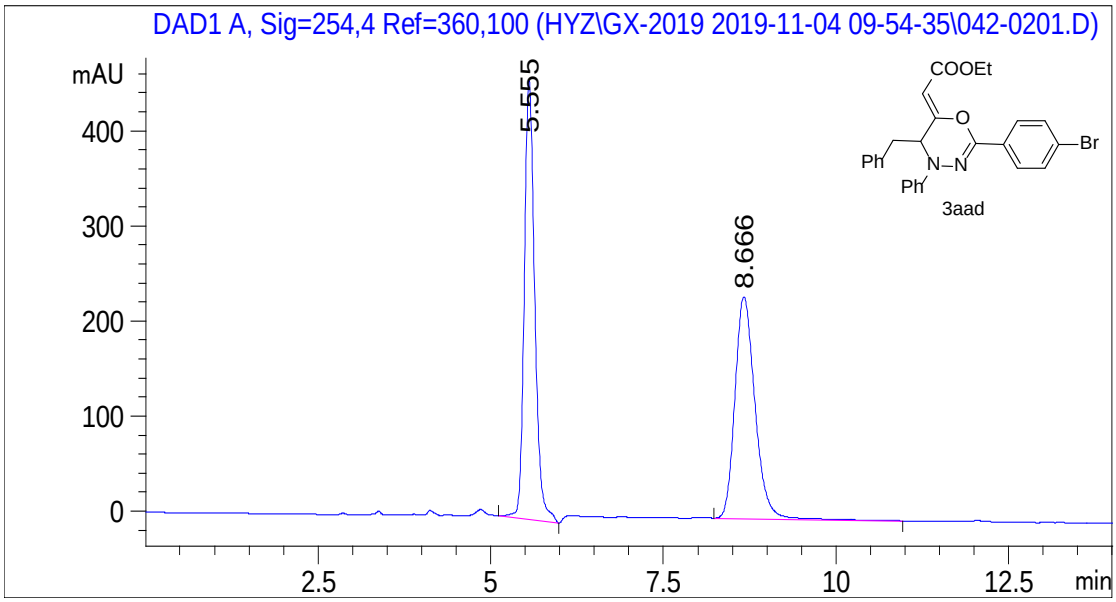
#	Time	Area	Height	Width	Area%	Symmetry
1	5.355	2227.2	224.5	0.1537	85.481	0.782
2	7.973	378.3	20.9	0.279	14.519	0.802

The mother liquor after crystal of **3aac**:

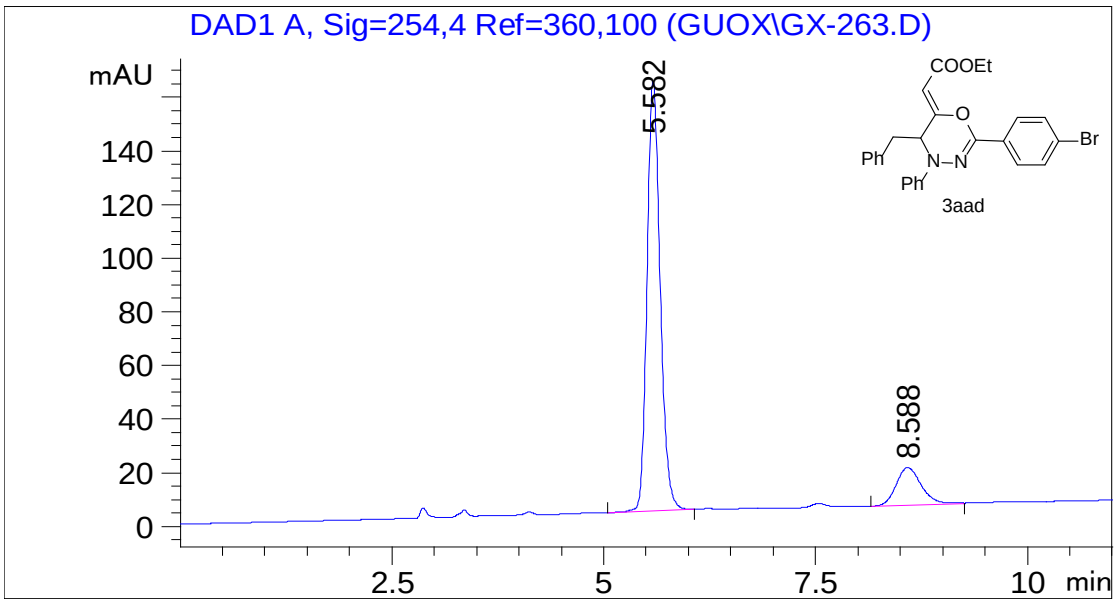


#	Time	Area	Height	Width	Area%	Symmetry
1	5.235	13414.9	1484.1	0.1506	99.451	0.785
2	8.022	74.1	6.4	0.1938	0.549	0.972

3aad: OD-H, 90/10, 1.0 ml/min, 254 nm

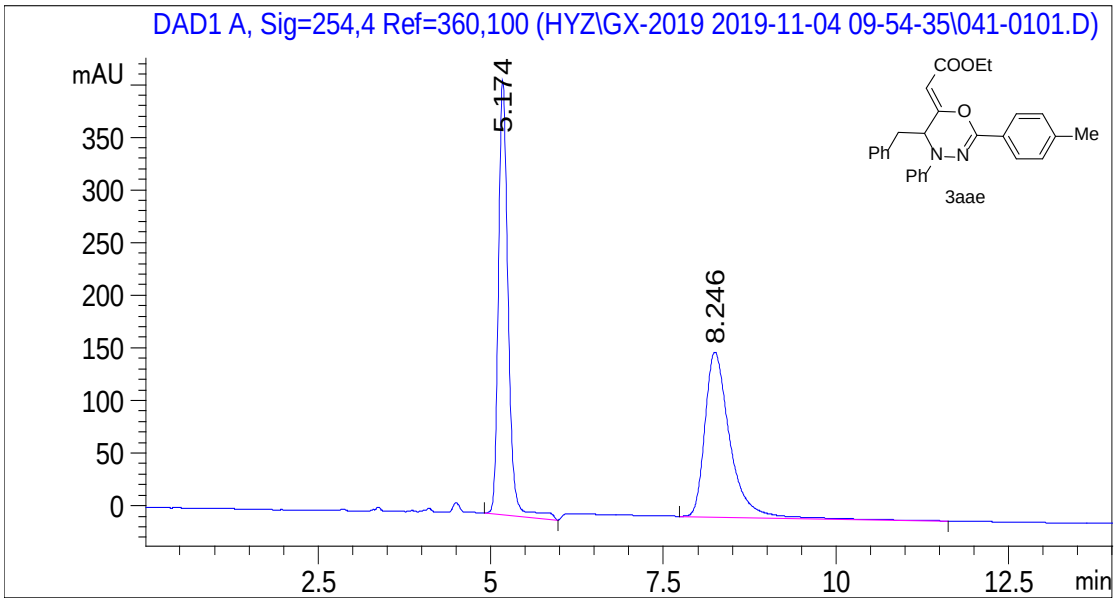


#	Time	Area	Height	Width	Area%	Symmetry
1	5.555	4855.4	461.9	0.1606	50.716	0.784
2	8.666	4718.4	233.1	0.3101	49.284	0.741

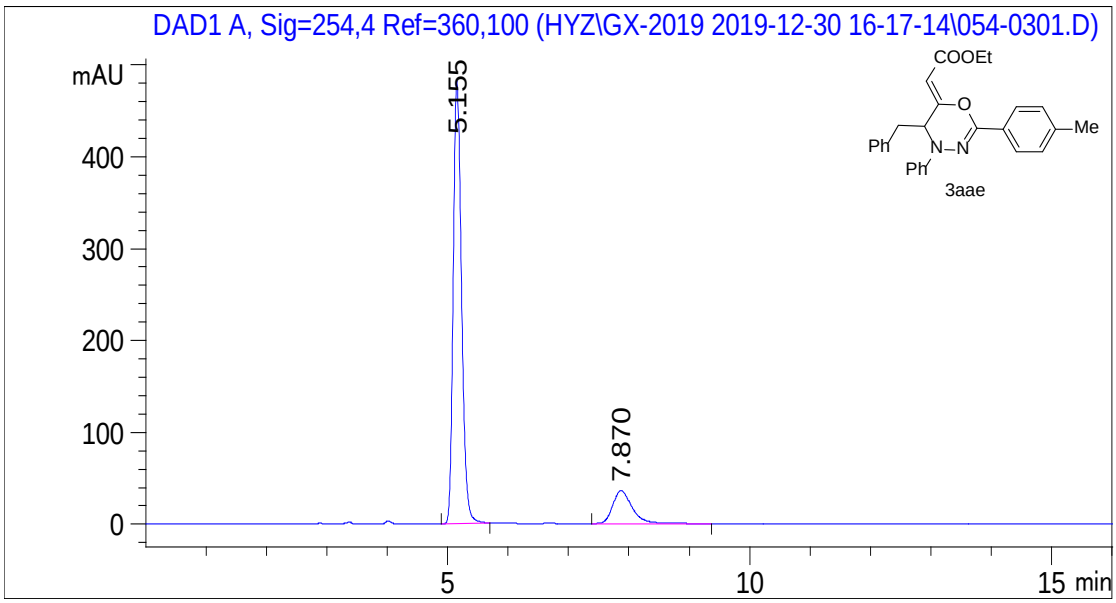


#	Time	Area	Height	Width	Area%	Symmetry
1	5.582	1730.1	160.4	0.1658	85.247	0.792
2	8.588	282.9	13.9	0.3131	14.753	0.814

3aae: OD-H, 90/10, 1.0 ml/min, 254 nm

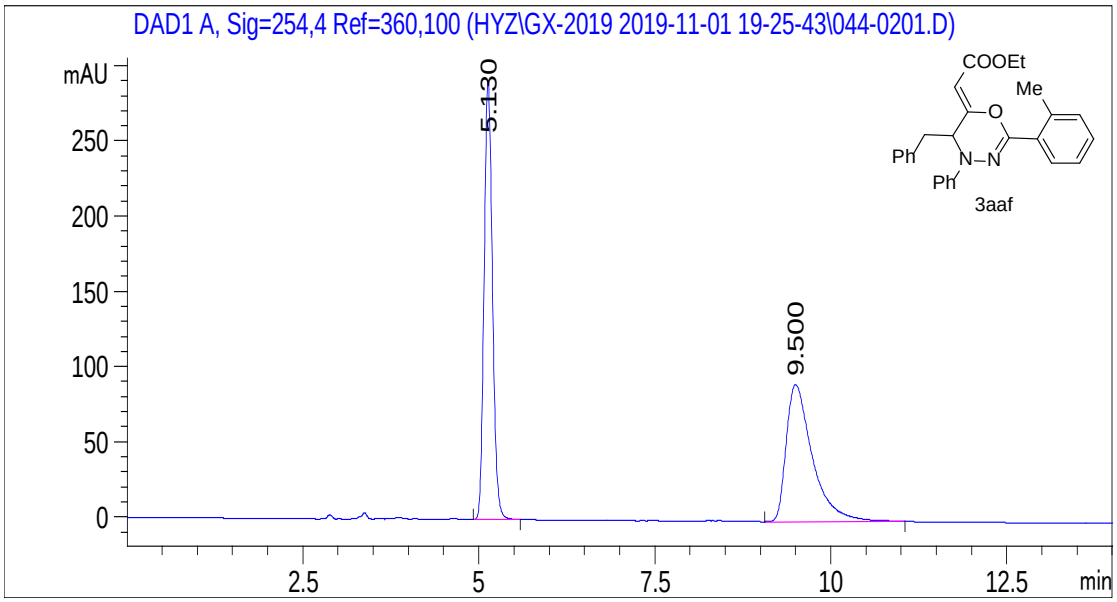


#	Time	Area	Height	Width	Area%	Symmetry
1	5.174	4061.6	412.8	0.1488	50.779	0.72
2	8.246	3936.9	156.6	0.3794	49.221	0.601

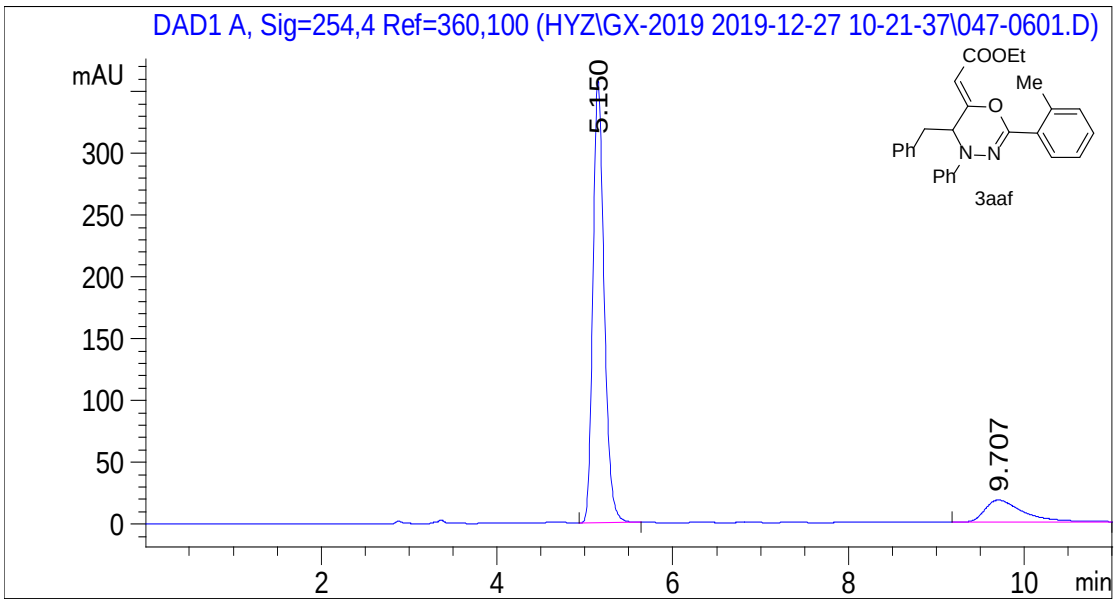


#	Time	Area	Height	Width	Area%	Symmetry
1	5.155	4557.4	482	0.1443	85.101	0.761
2	7.87	797.9	35.9	0.3371	14.899	0.694

3aaf: OD-H, 90/10, 1.0 ml/min, 254 nm

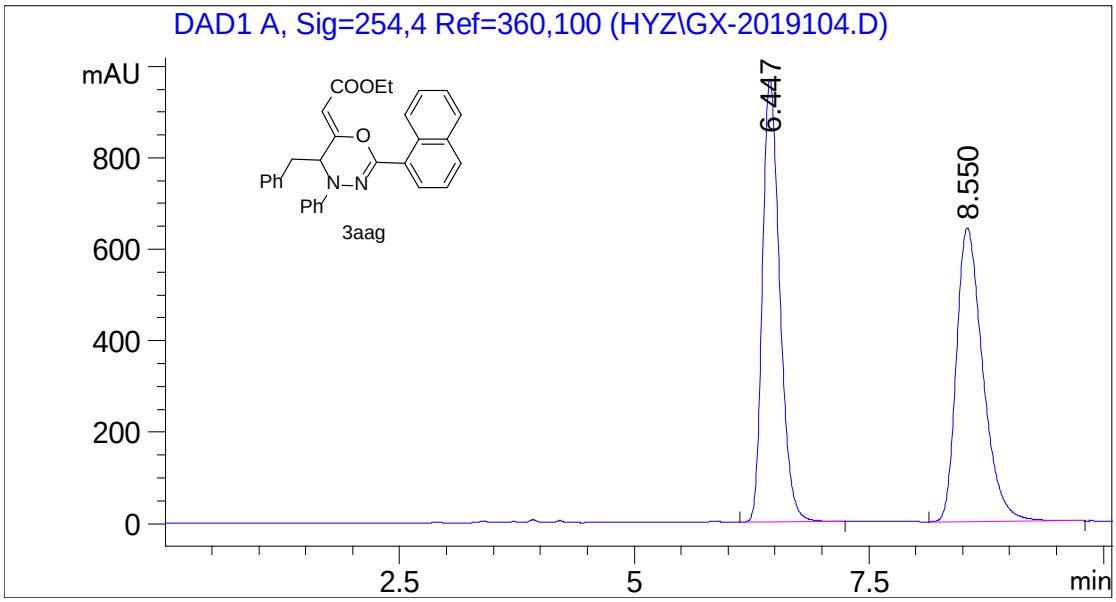


#	Time	Area	Height	Width	Area%	Symmetry
1	5.13	2404.7	292.5	0.1279	50.423	0.833
2	9.5	2364.3	91	0.389	49.577	0.505

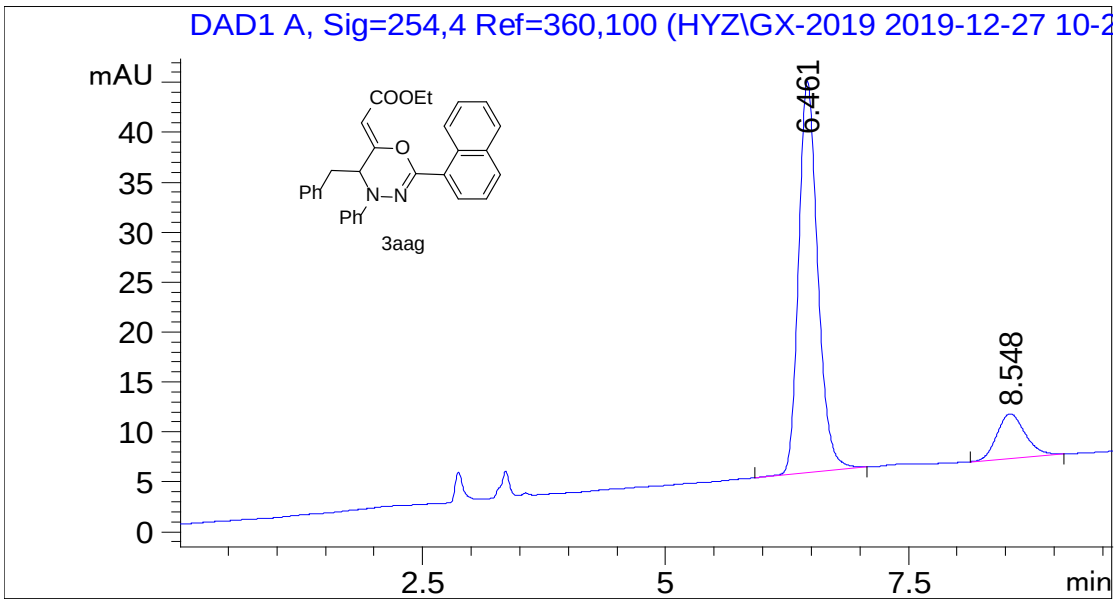


#	Time	Area	Height	Width	Area%	Symmetry
1	5.15	3165	357.9	0.1352	85.474	0.766
2	9.707	537.9	17.8	0.4479	14.526	0.509

3aag: OD-H, 90/10, 1.0 ml/min, 254 nm

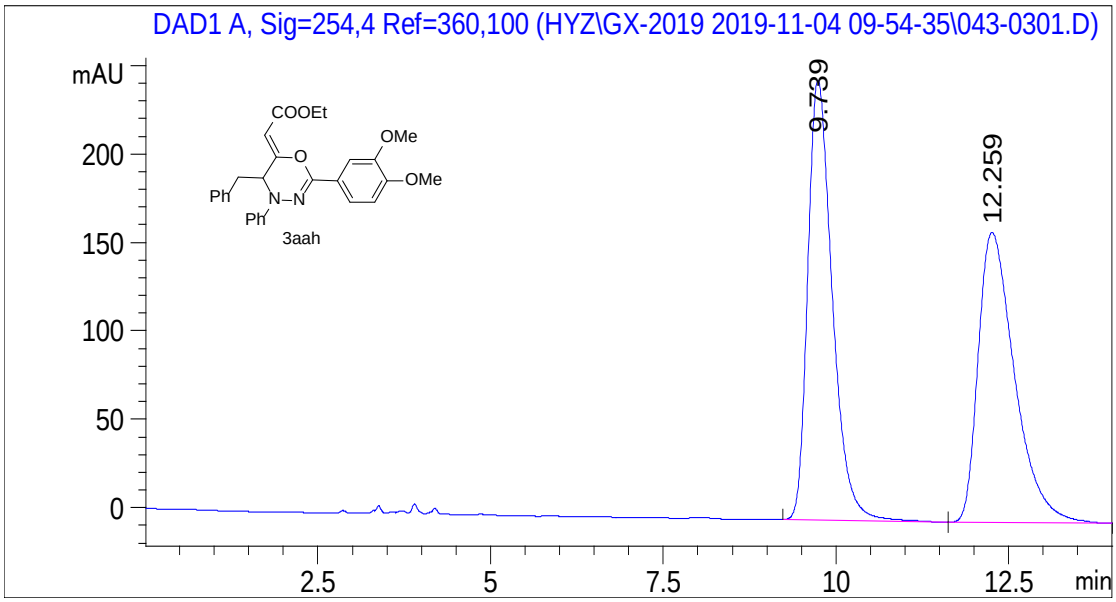


#	Time	Area	Height	Width	Area%	Symmetry
1	6.447	12425.2	966.3	0.2	49.714	0.707
2	8.55	12568.2	641.9	0.3004	50.286	0.646

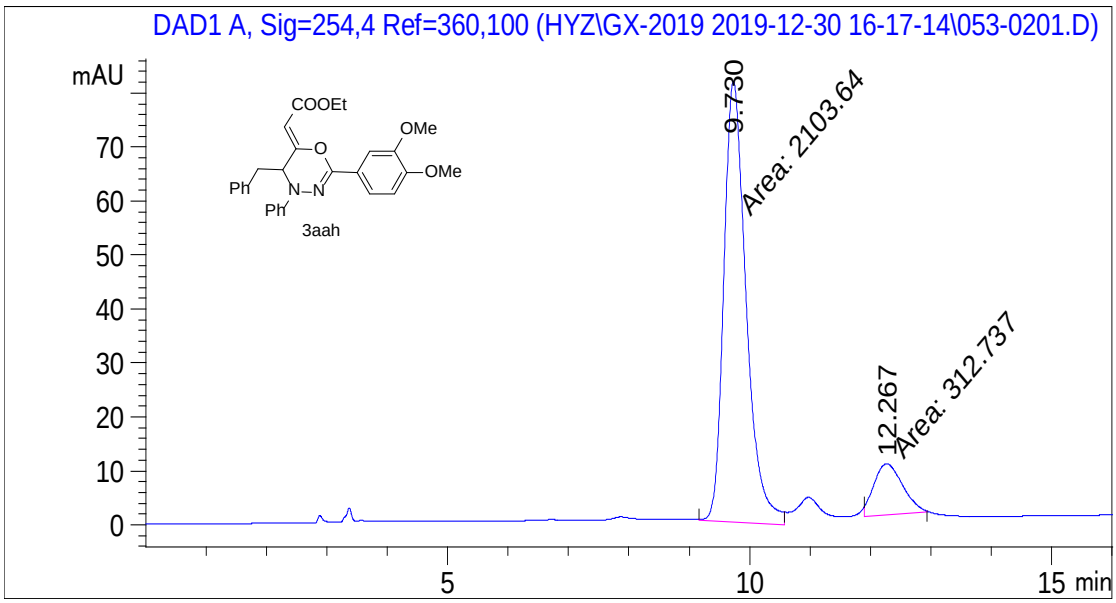


#	Time	Area	Height	Width	Area%	Symmetry
1	6.461	535.9	39.3	0.2092	85.184	0.776
2	8.548	90.3	4.5	0.3118	14.816	0.83

3aah: OD-H, 90/10, 1.0 ml/min, 254 nm

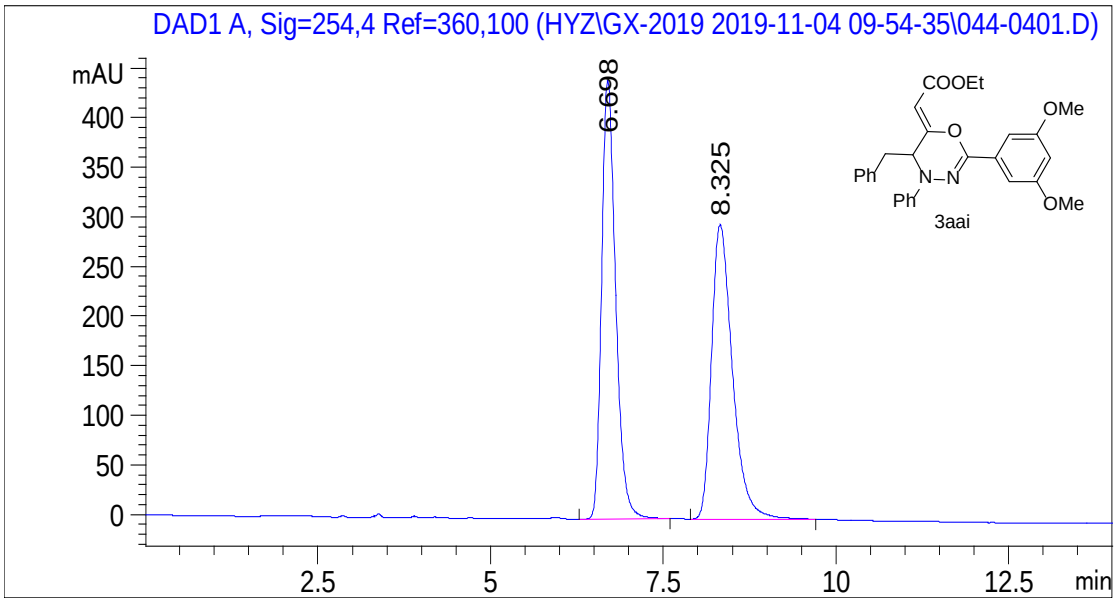


#	Time	Area	Height	Width	Area%	Symmetry
1	9.739	6164.5	248.9	0.3789	50.531	0.653
2	12.259	6035	164.3	0.5645	49.469	0.567

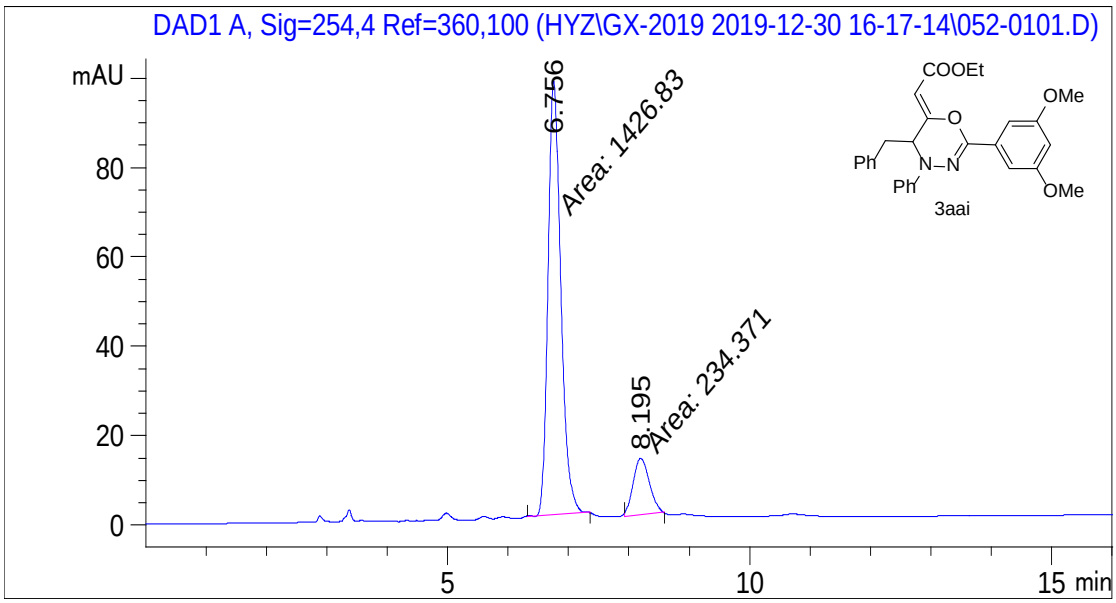


#	Time	Area	Height	Width	Area%	Symmetry
1	9.73	2103.6	81.8	0.4286	87.058	0.712
2	12.267	312.7	9.4	0.5527	12.942	0.788

3aai: OD-H, 90/10, 1.0 ml/min, 254 nm

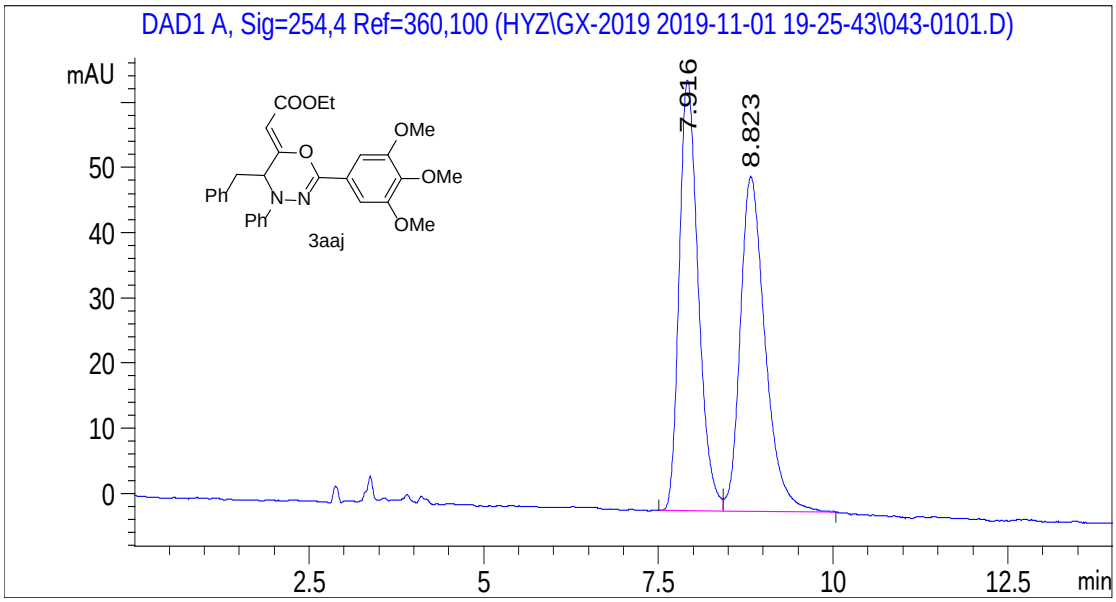


#	Time	Area	Height	Width	Area%	Symmetry
1	6.698	6265.3	443	0.217	50.000	0.709
2	8.325	6265.2	296.9	0.3202	50.000	0.644

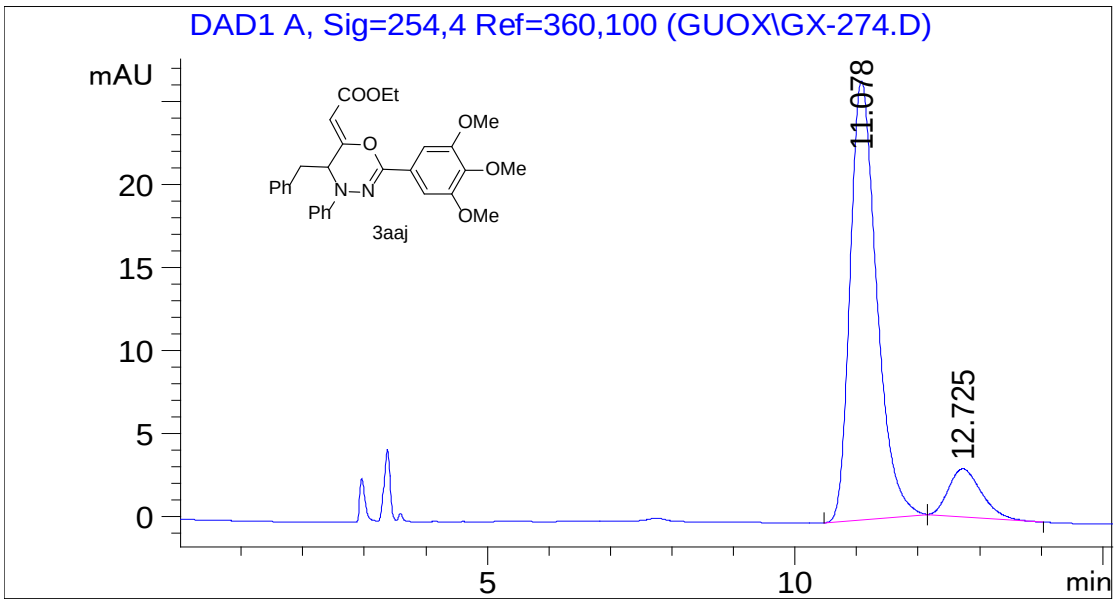


#	Time	Area	Height	Width	Area%	Symmetry
1	6.756	1426.8	97.1	0.245	85.891	0.769
2	8.195	234.4	12.6	0.3107	14.109	0.863

3aaj: OD-H, 95/5, 1.0 ml/min, 254 nm

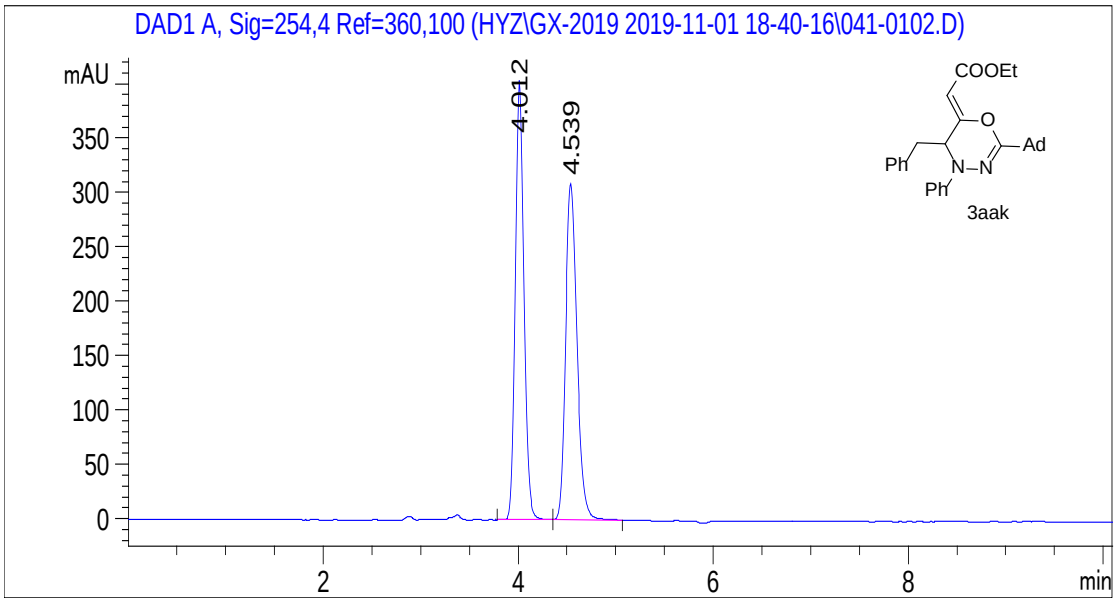


#	Time	Area	Height	Width	Area%	Symmetry
1	7.916	1256.4	66.1	0.2918	49.610	0.72
2	8.823	1276.2	51.4	0.3719	50.390	0.688

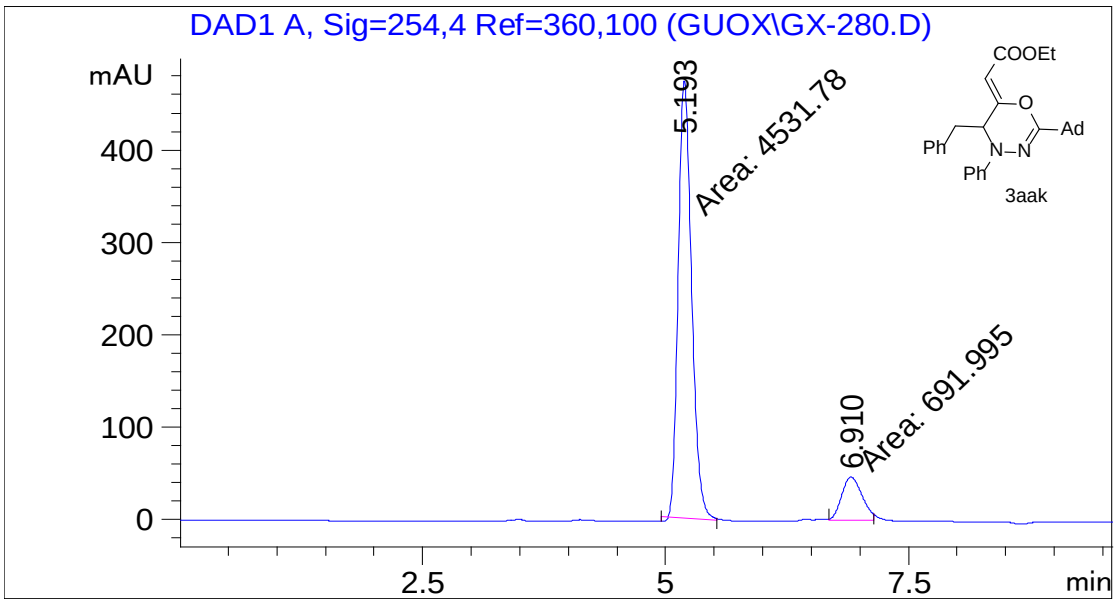


#	Time	Area	Height	Width	Area%	Symmetry
1	11.078	792.9	26.4	0.4602	89.118	0.687
2	12.725	96.9	2.9	0.5624	10.882	0.717

3aak: OD-H, 97.5/2.5, 1.0 ml/min, 254 nm

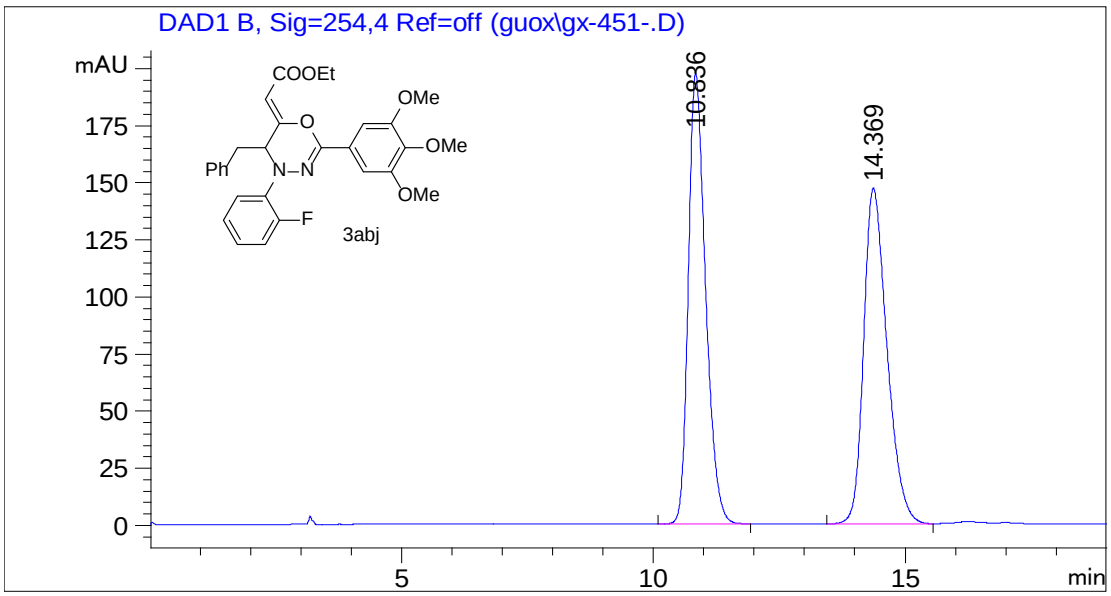


#	Time	Area	Height	Width	Area%	Symmetry
1	4.012	2506	404.7	0.0963	50.136	0.852
2	4.539	2492.5	308.8	0.1242	49.864	0.76

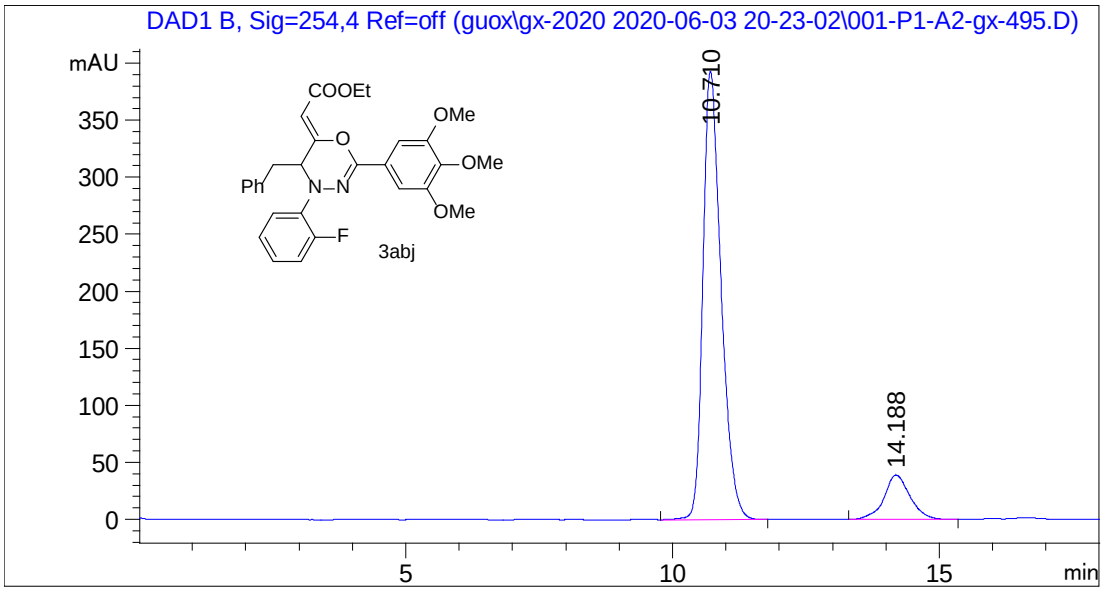


#	Time	Area	Height	Width	Area%	Symmetry
1	5.193	4531.8	474	0.1593	87.253	0.78
2	6.91	692	47.1	0.2449	12.747	0.822

3abj: AD-H, 98/2, 1.0 ml/min, 254 nm

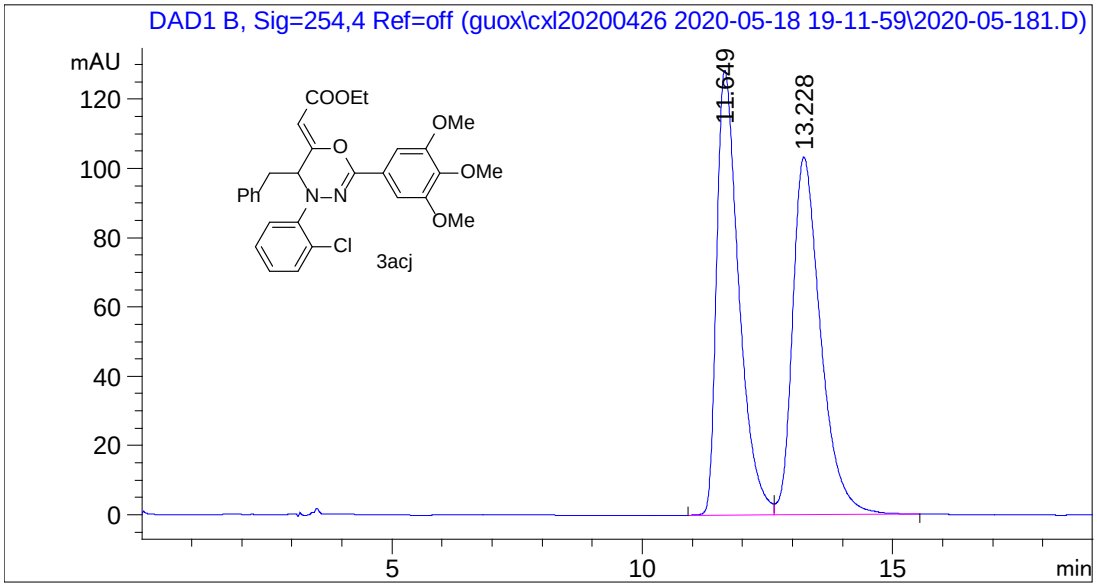


#	Time	Area	Height	Width	Area%	Symmetry
1	10.836	4703.1	197.5	0.3618	49.868	0.687
2	14.369	4728	147.1	0.487	50.132	0.674

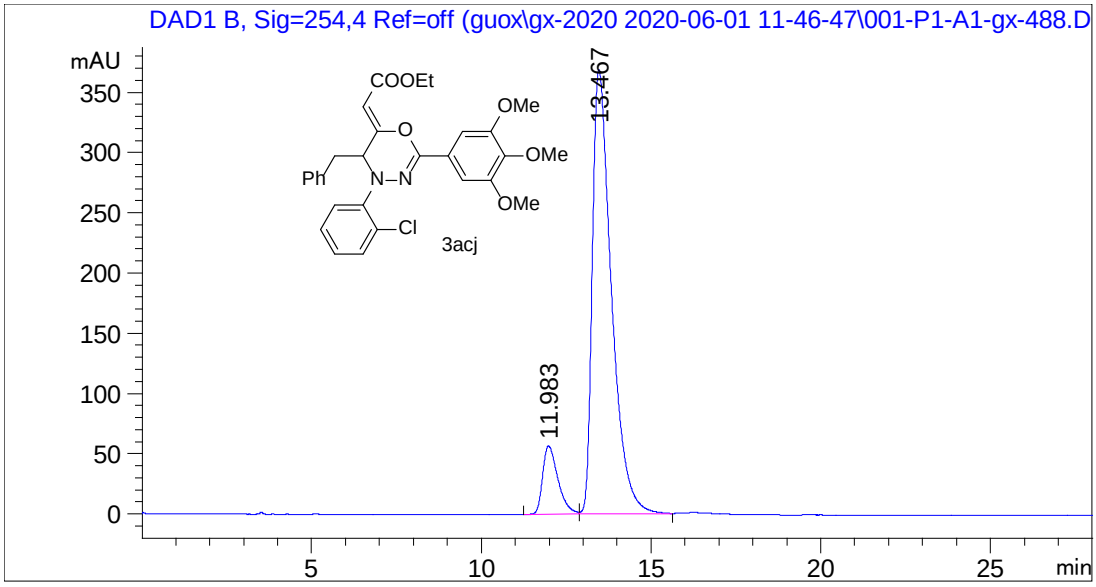


#	Time	Area	Height	Width	Area%	Symmetry
1	10.71	9304.6	393.2	0.3581	87.562	0.659
2	14.188	1321.7	38.9	0.5039	12.438	0.839

3acj: OD-H, 98/2, 1.0 ml/min, 254 nm

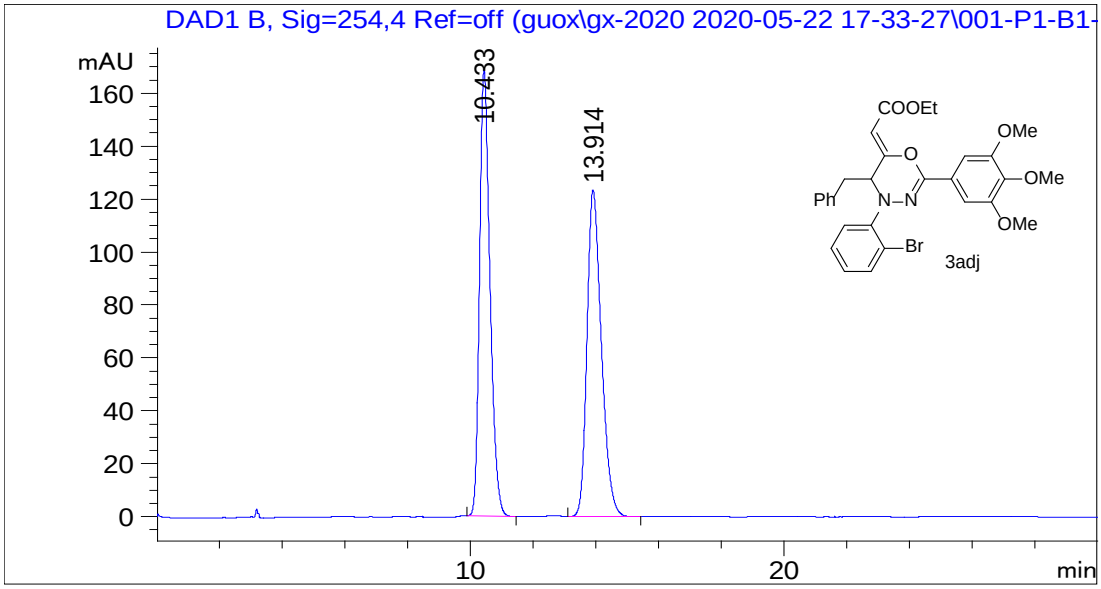


#	Time	Area	Height	Width	Area%	Symmetry
1	11.649	3962.5	128.4	0.466	49.640	0.532
2	13.228	4020	103.2	0.5884	50.360	0.621

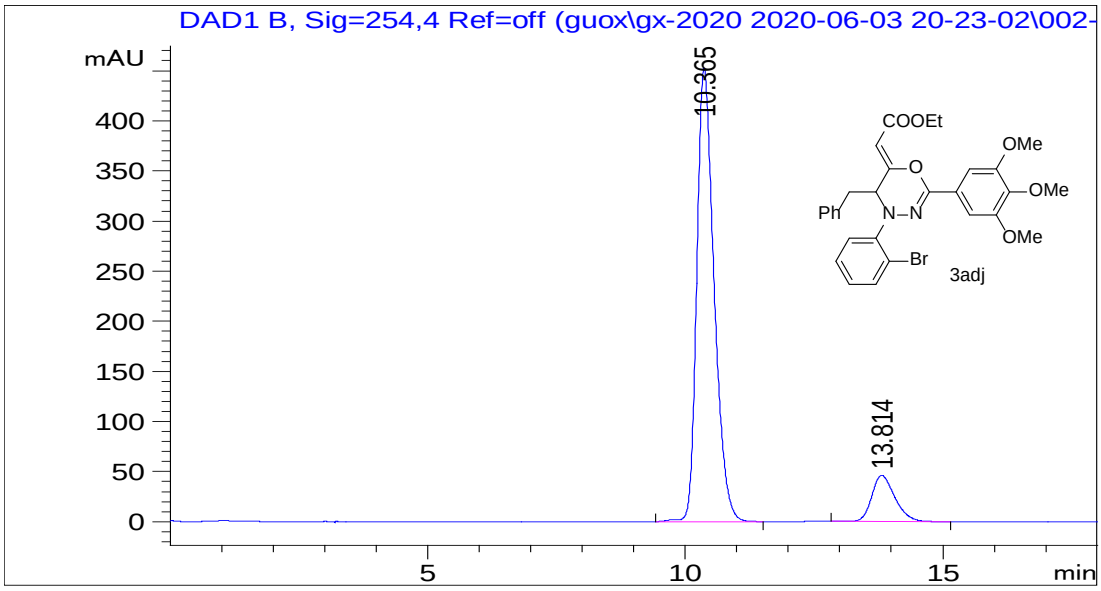


#	Time	Area	Height	Width	Area%	Symmetry
1	11.983	1786.6	56.9	0.4765	11.029	0.595
2	13.467	14412.1	369.1	0.5835	88.971	0.489

3adj: AD-H, 98/2, 1.0 ml/min, 254 nm

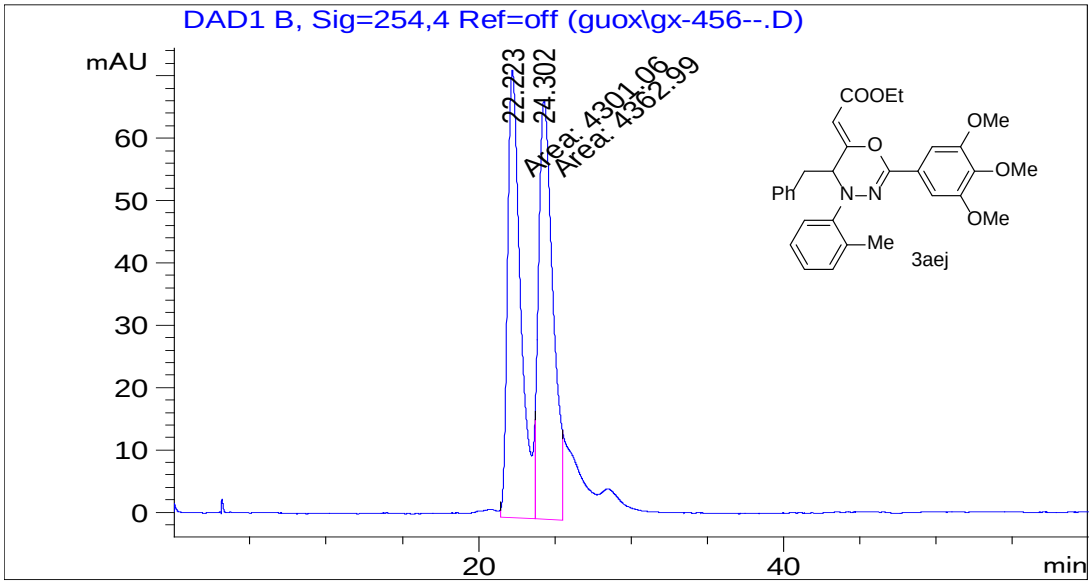


#	Time	Type	AreaHeight	Width	Area%	Symmetry
1	10.433	3911.8	168.4	0.3549	49.861	0.686
2	13.914	3933.6	123.4	0.4859	50.139	0.691

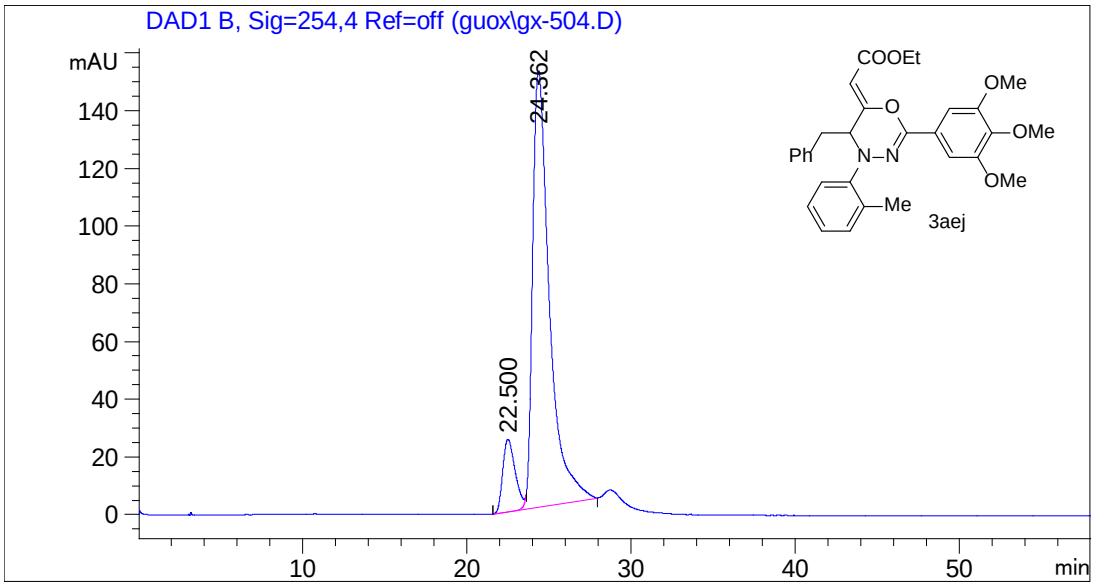


#	Time	Area	Height	Width	Area%	Symmetry
1	10.365	10521.1	452.2	0.3534	87.983	0.671
2	13.814	1437	46.1	0.4736	12.017	0.72

3aej: IC-3, 95/5, 1.0 ml/min, 254 nm

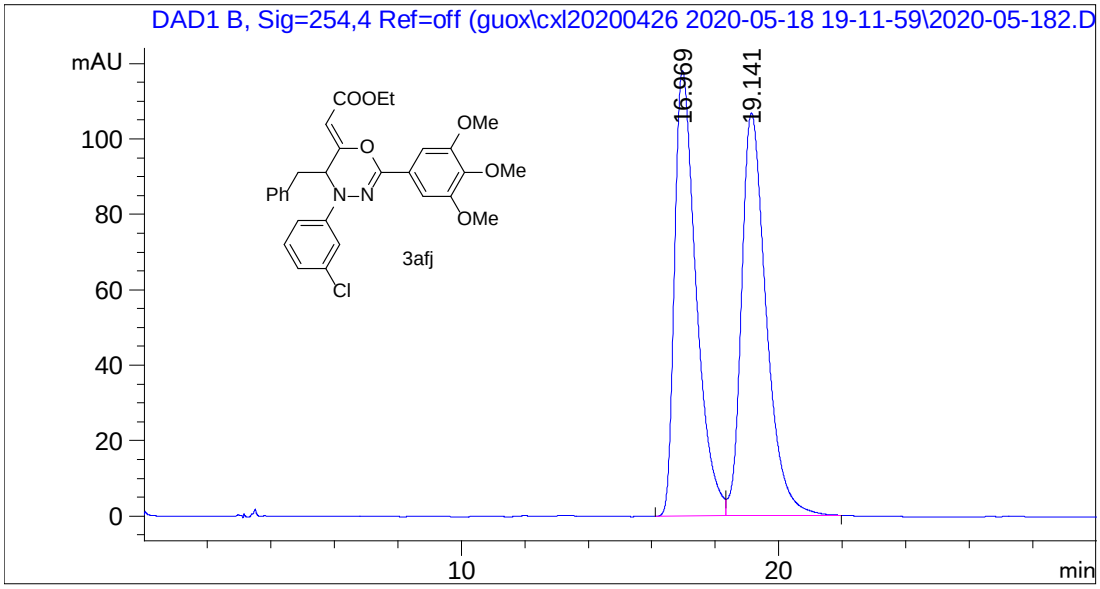


#	Time	Area	Height	Width	Area%	Symmetry
1	22.223	4301.1	71.8	0.9988	49.643	0
2	24.302	4363	67.3	1.0813	50.357	0.593

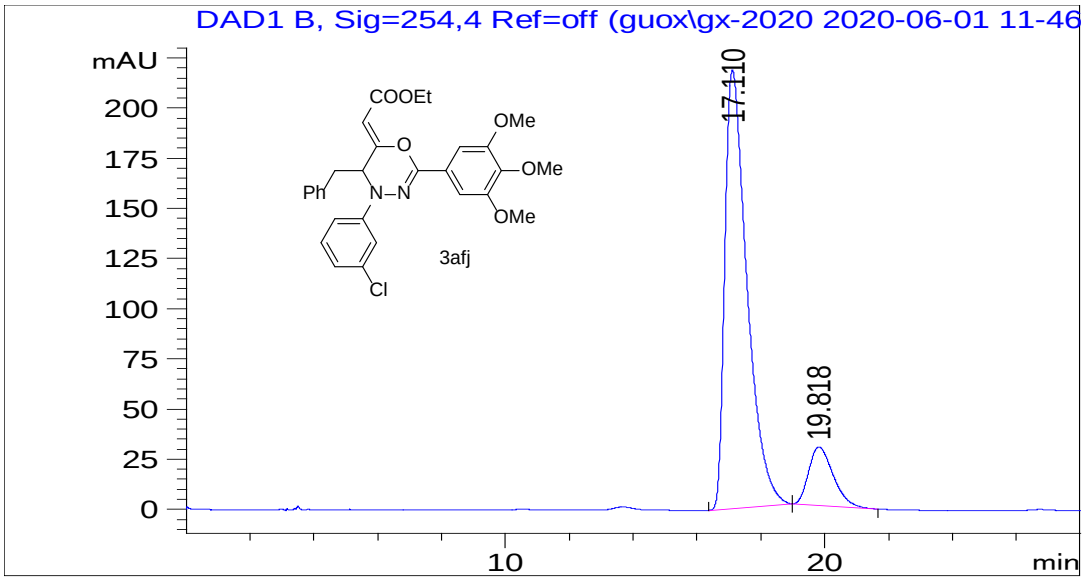


#	Time	Area	Height	Width	Area%	Symmetry
1	22.5	1313.3	25.1	0.7944	10.866	0.68
2	24.362	10773.2	151.2	1.0672	89.134	0.894

3afj: OD-H, 98/2, 1.0 ml/min, 254 nm

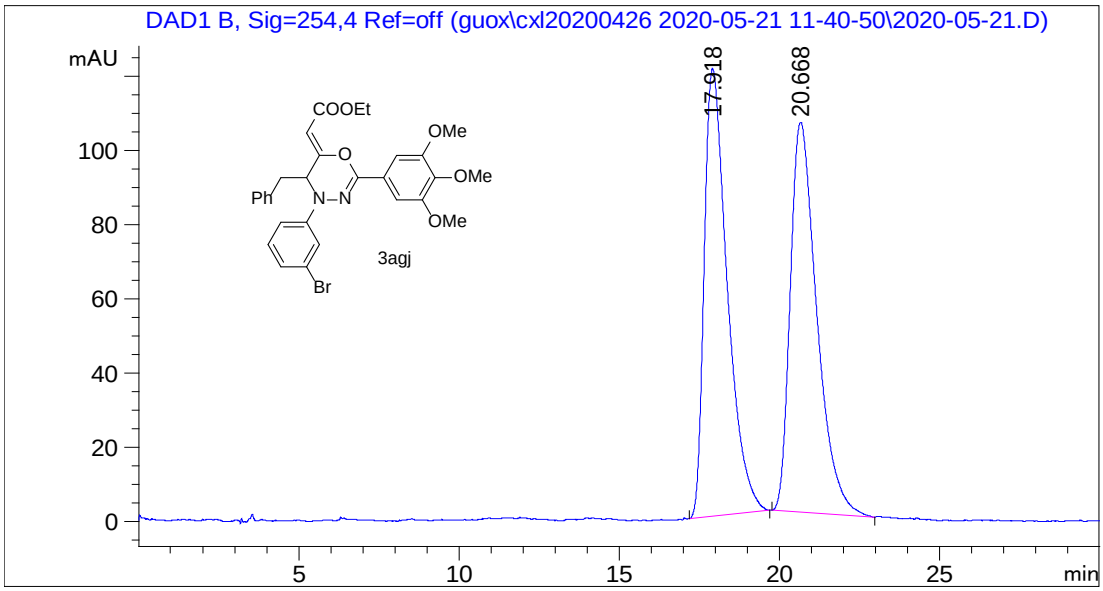


#	Time	Area	Height	Width	Area%	Symmetry
1	16.969	5592.8	118.2	0.7141	49.272	0.535
2	19.141	5758	106.8	0.8128	50.728	0.623

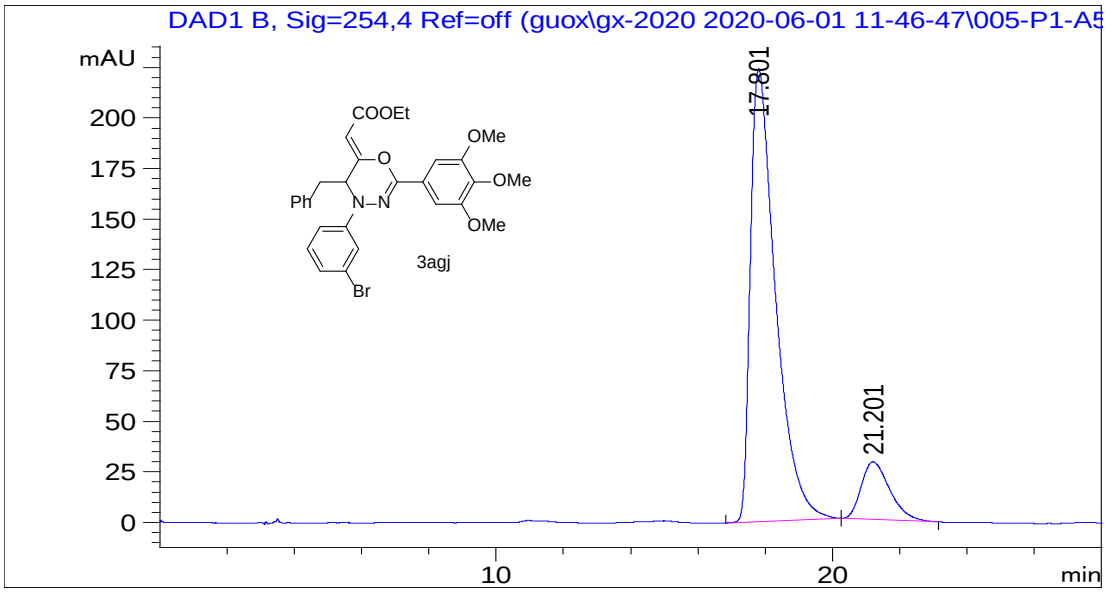


#	Time	Area	Height	Width	Area%	Symmetry
1	17.11	10511	218.7	0.7248	87.094	0.478
2	19.818	1557.6	29.2	0.8064	12.906	0.678

3agj: AD-H, 98/2, 1.0 ml/min, 254 nm

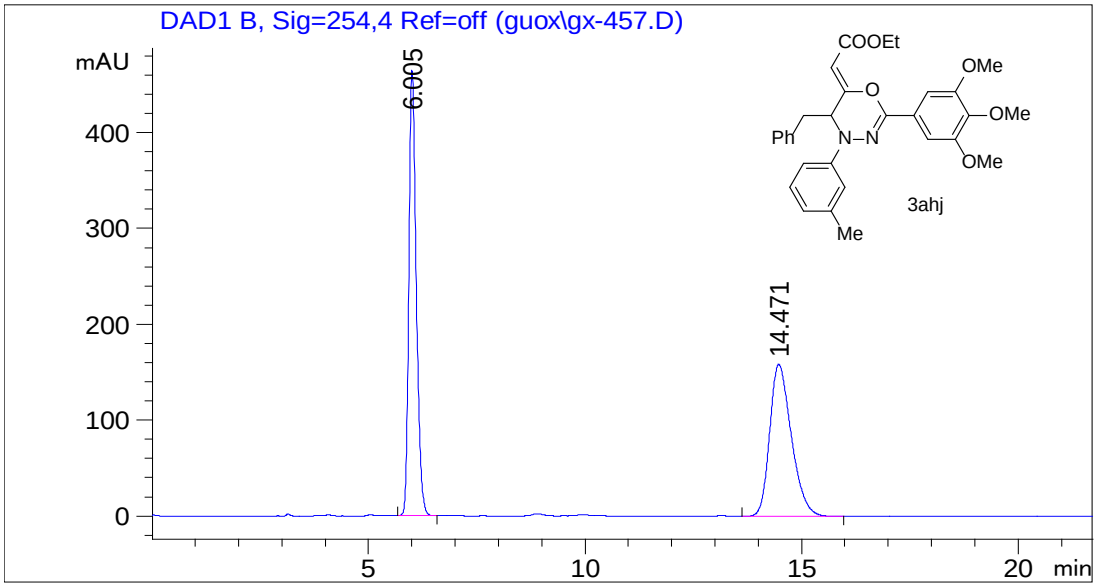


#	Time	Area	Height	Width	Area%	Symmetry
1	17.918	6038.5	120.6	0.722	49.878	0.525
2	20.668	6068.1	105	0.8641	50.122	0.594

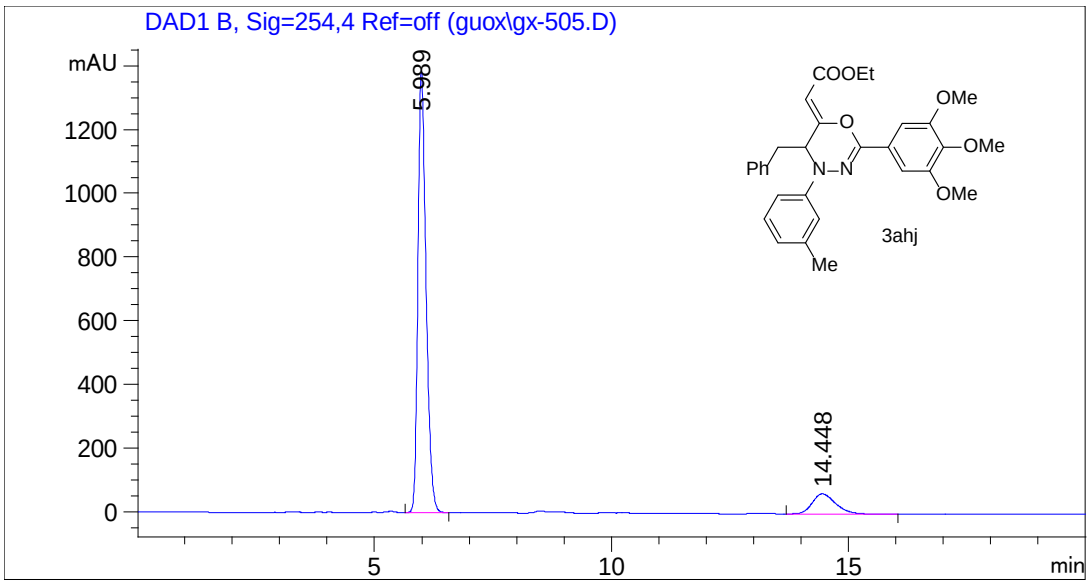


#	Time	Area	Height	Width	Area%	Symmetry
1	17.801	11627.3	224	0.7816	87.321	0.442
2	21.201	1688.2	28.5	0.8945	12.679	0.668

3ahj: AD-H, 90/10, 1.0 ml/min, 254 nm

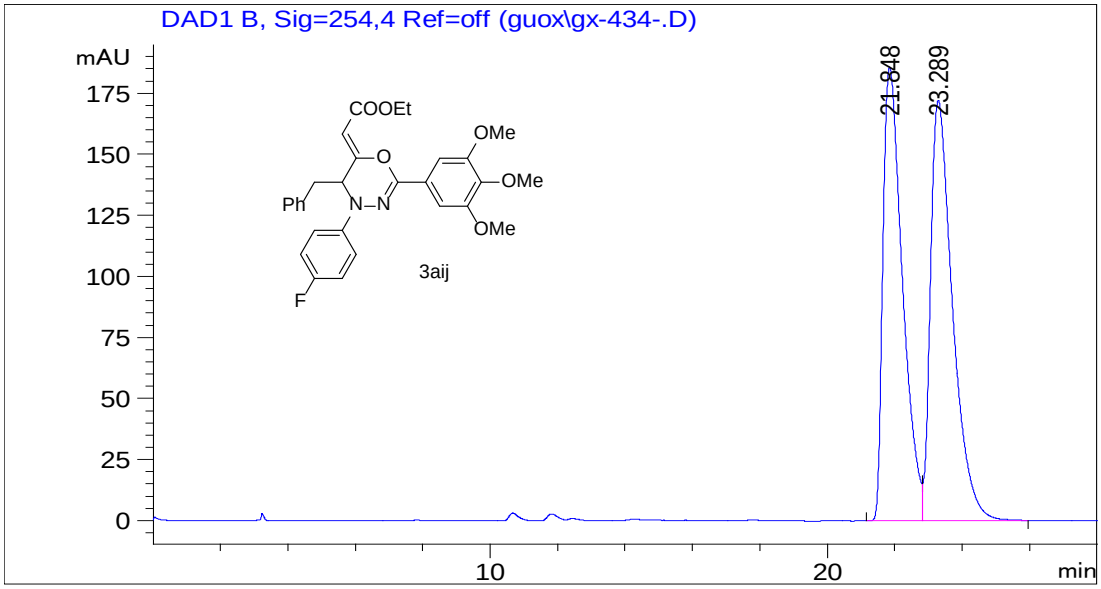


#	Time	Area	Height	Width	Area%	Symmetry
1	6.005	5516.4	464.7	0.1805	49.992	0.702
2	14.471	5518.3	158.5	0.5322	50.008	0.679

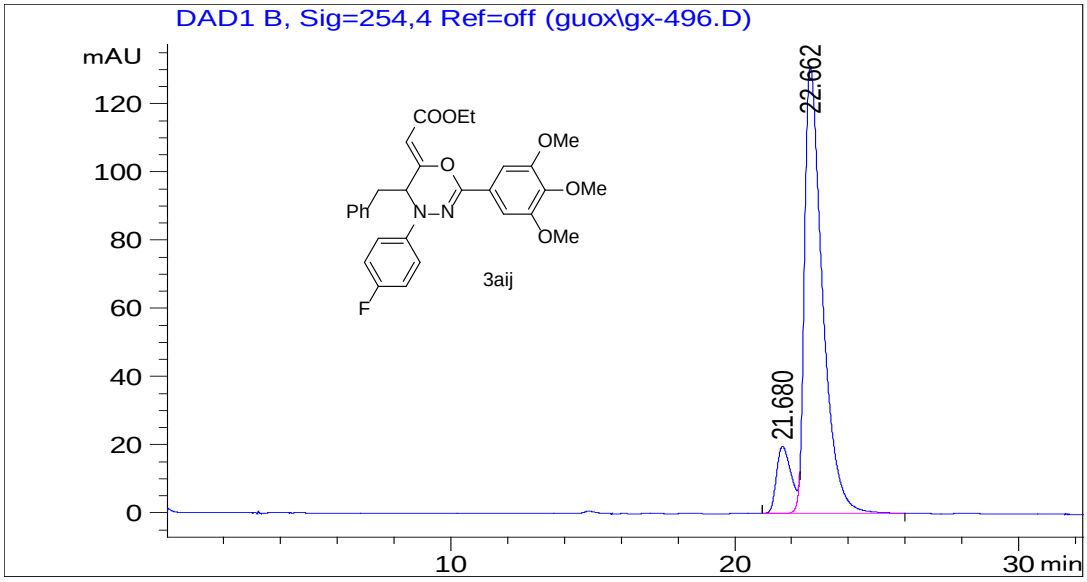


#	Time	Area	Height	Width	Area%	Symmetry
1	5.989	16665.2	1387.4	0.1821	88.495	0.677
2	14.448	2166.7	63	0.5233	11.505	0.725

3aij: ID-H, 95/5, 1.0 ml/min, 254 nm

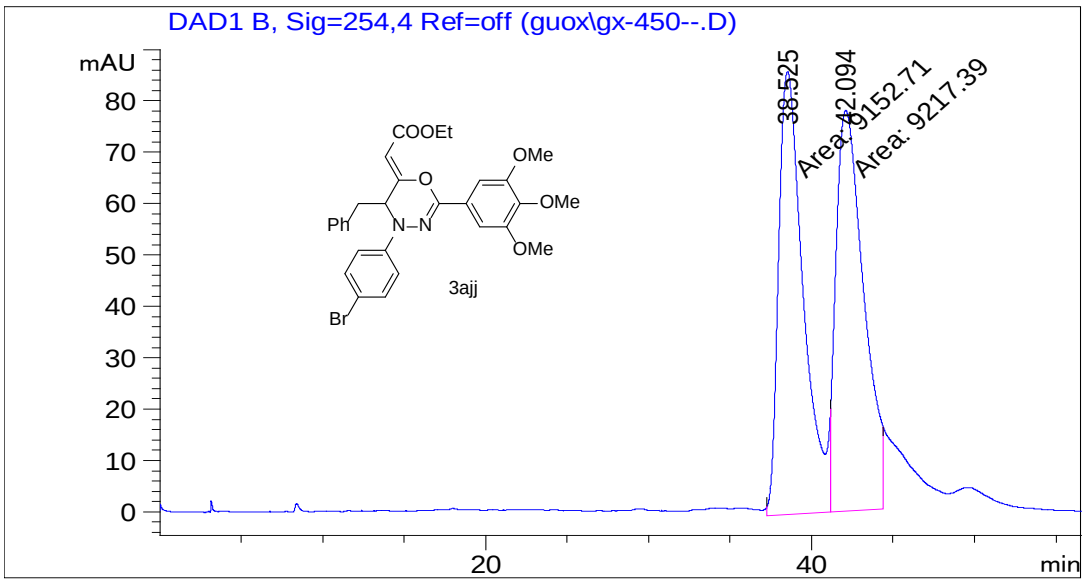


#	Time	Area	Height	Width	Area%	Symmetry
1	21.848	7472.2	185.7	0.6113	48.783	0.483
2	23.289	7845	172.2	0.6875	51.217	0.499

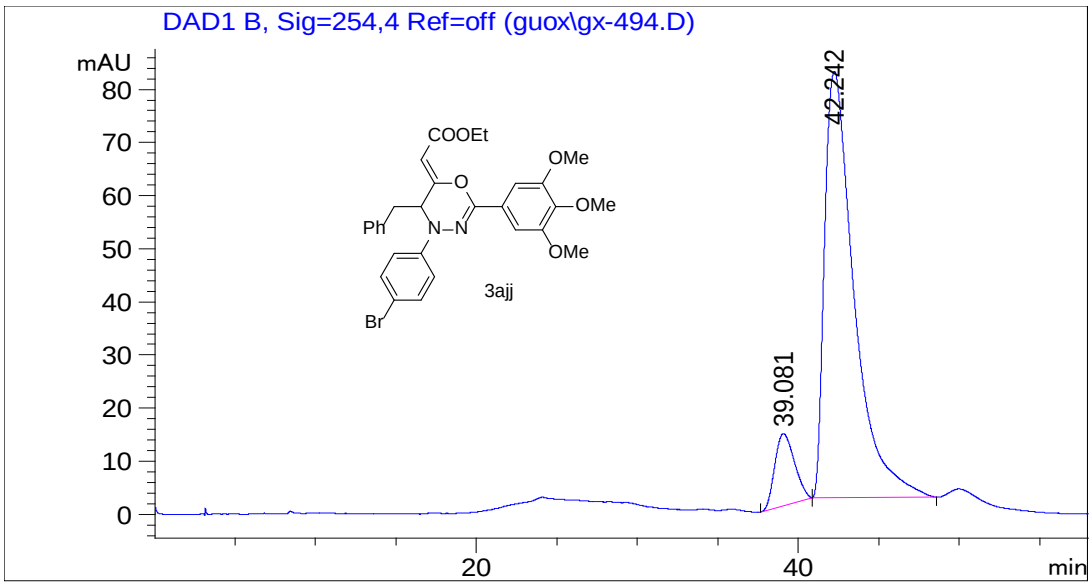


#	Time	Area	Height	Width	Area%	Symmetry
1	21.68	683.8	19.6	0.5261	10.585	0.713
2	22.662	5776.1	131.1	0.6561	89.415	0.833

3ajj: IC-3, 95/5, 1.0 ml/min, 254 nm

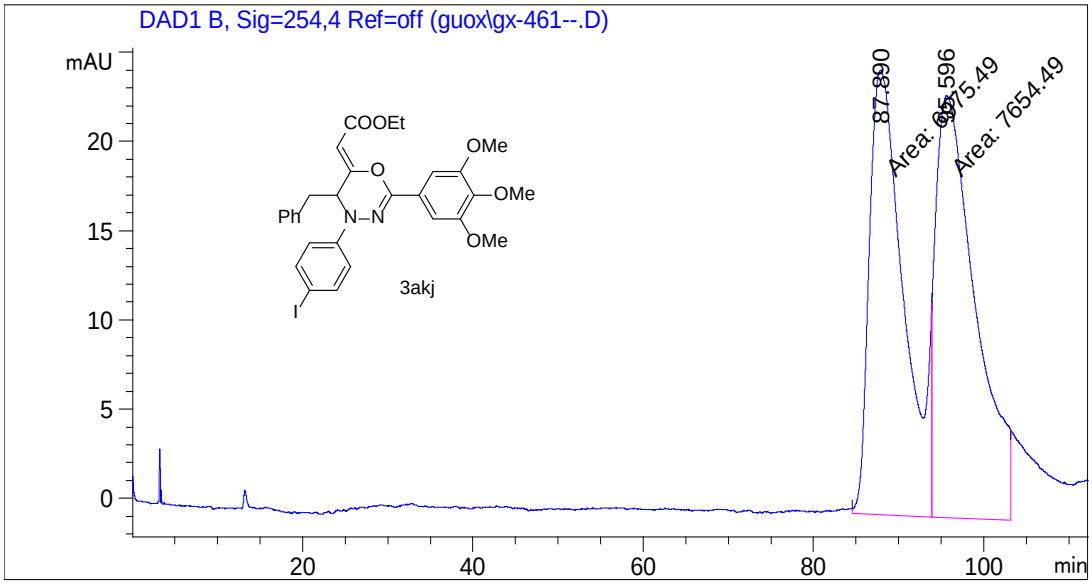


#	Time	Area	Height	Width	Area%	Symmetry
1	38.525	9152.7	86.2	1.7698	49.824	0
2	42.094	9217.4	78	1.9703	50.176	0.512

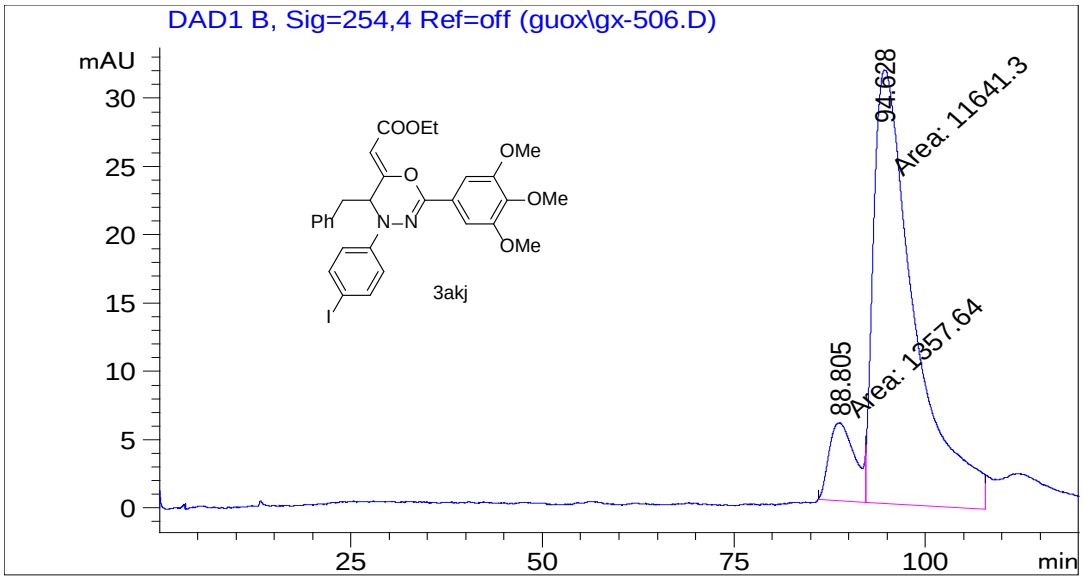


#	Time	Area	Height	Width	Area%	Symmetry
1	39.081	1155.5	13.6	1.1036	10.141	0.748
2	42.242	10239.1	80.3	1.7974	89.859	0.451

3akj: IC-3, 98/2, 1.0 ml/min, 254 nm

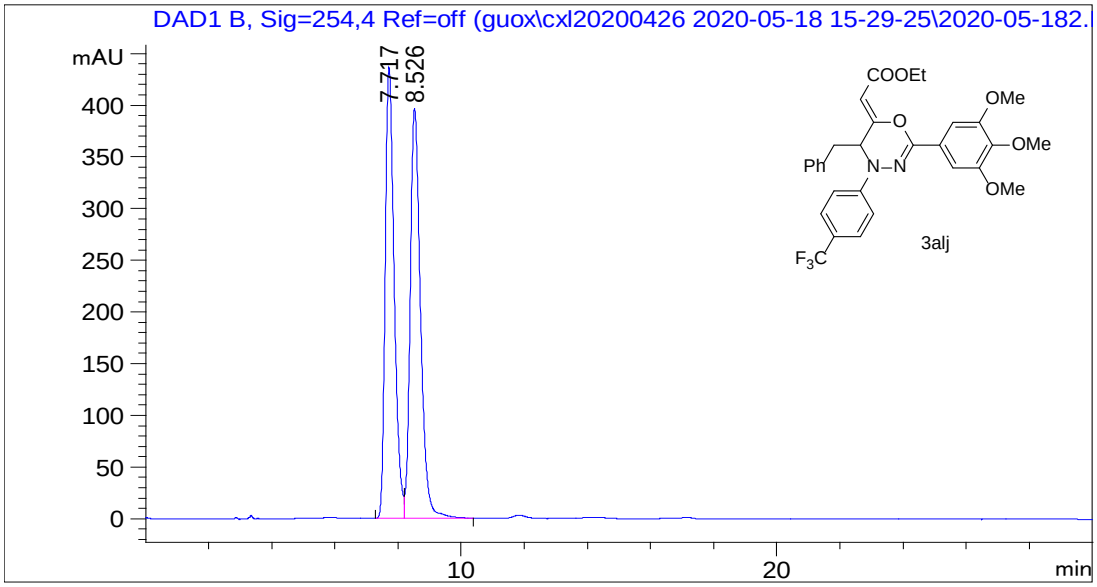


#	Time	Area	Height	Width	Area%	Symmetry
1	87.89	6975.5	24.9	4.6651	47.679	0
2	95.596	7654.5	23.6	5.3949	52.321	0.347

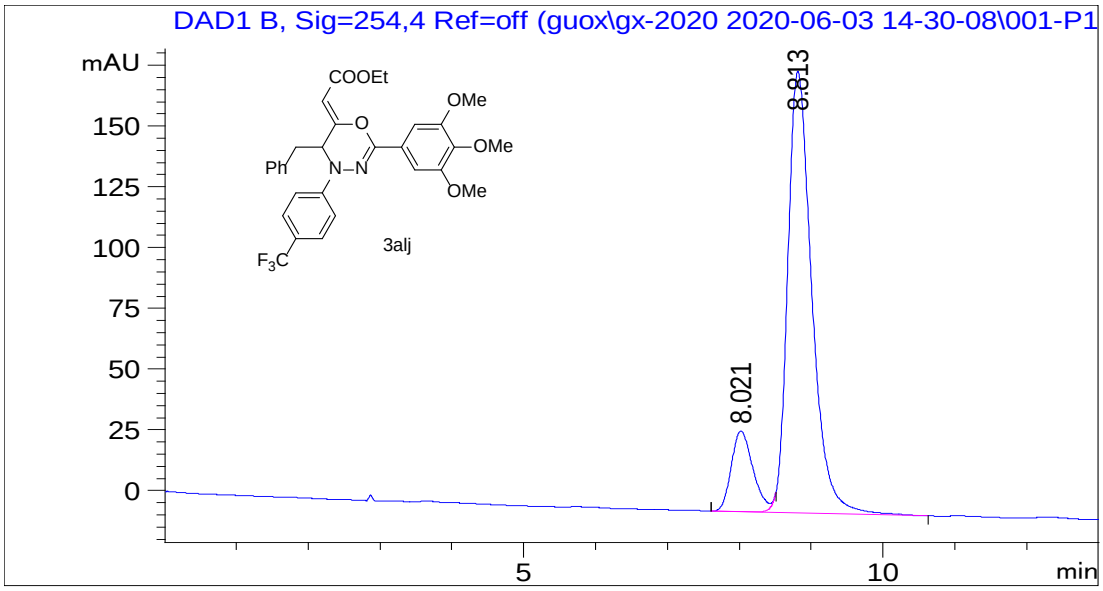


#	Time	Area	Height	Width	Area%	Symmetry
1	88.805	1357.6	5.7	3.9521	10.944	0
2	94.628	11641.3	31.8	6.1087	89.056	0.348

3alj: OD-H, 90/10, 1.0 ml/min, 254 nm

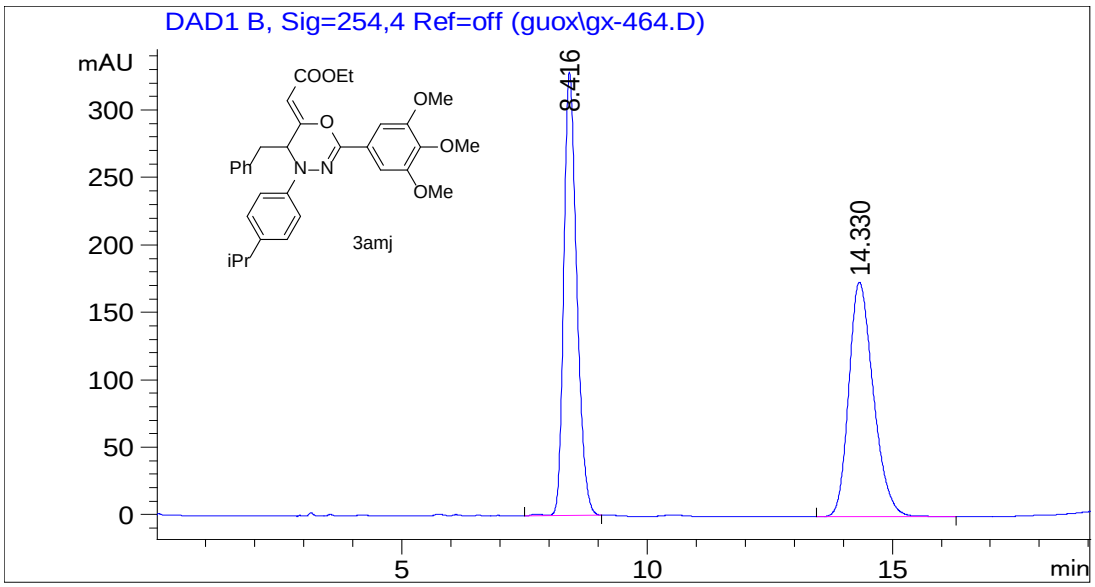


#	Time	Area	Height	Width	Area%	Symmetry
1	7.717	8621.1	436.2	0.3046	48.916	0.679
2	8.526	9003.3	396.7	0.3468	51.084	0.675

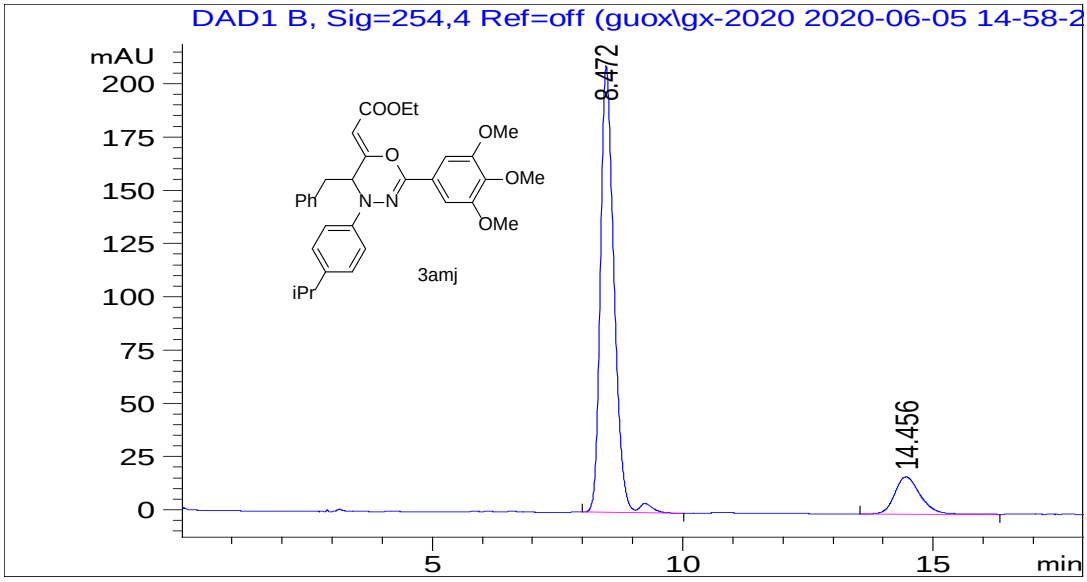


#	Time	Area	Height	Width	Area%	Symmetry
1	8.021	696.2	33.3	0.3202	14.065	0.755
2	8.813	4253.7	181.9	0.3569	85.935	1.478

3amj: AD-H, 90/10, 1.0 ml/min, 254 nm

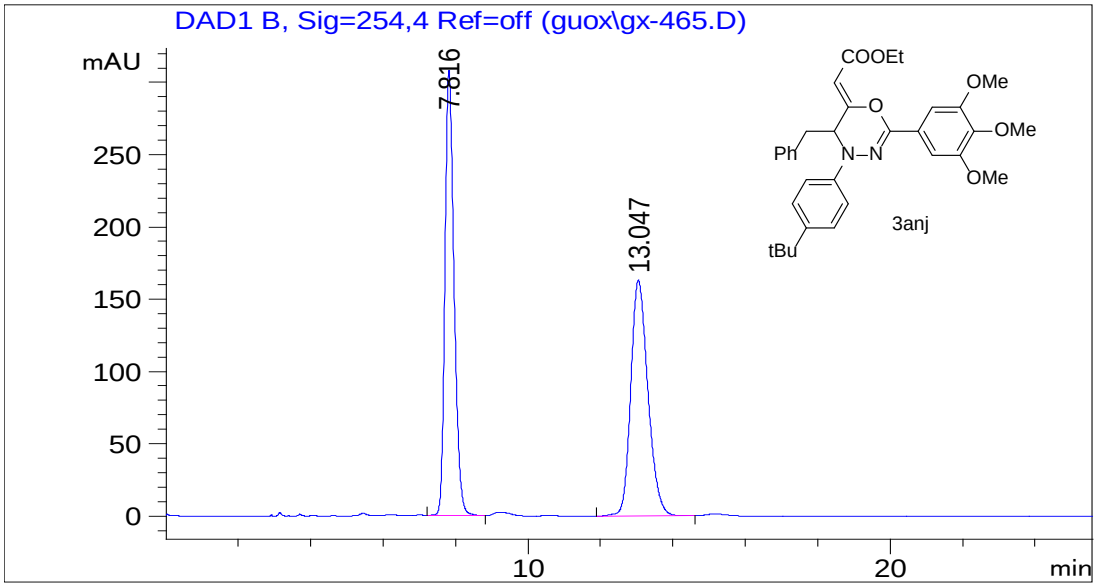


#	Time	Area	Height	Width	Area%	Symmetry
1	8.416	6059.7	329.2	0.2801	49.934	0.723
2	14.33	6075.8	173.9	0.5316	50.066	0.716

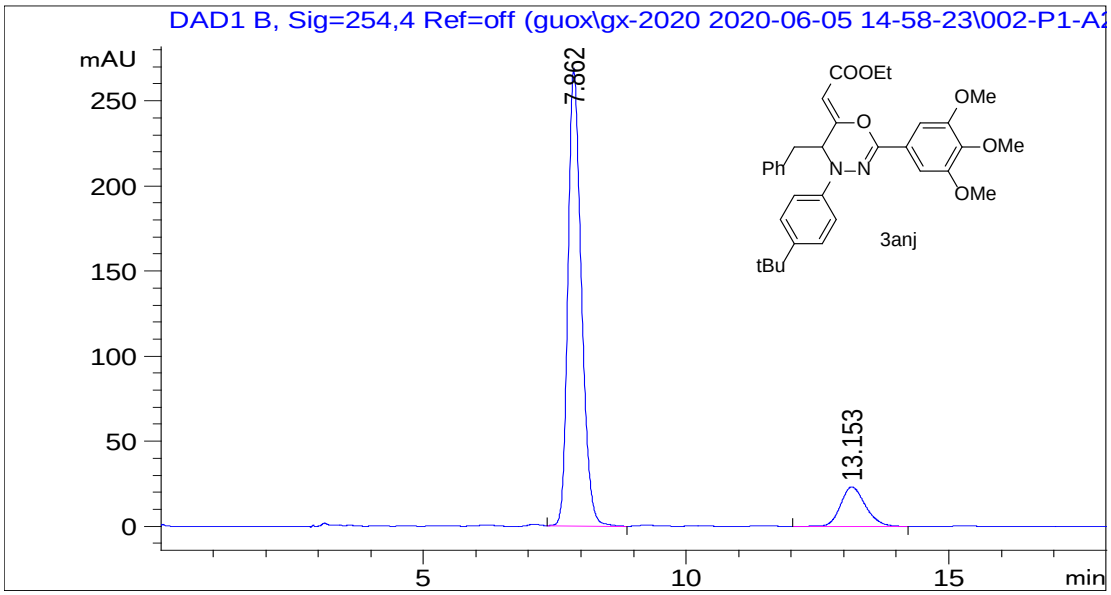


#	Time	Area	Height	Width	Area%	Symmetry
1	8.472	3993.4	209.4	0.2842	86.533	0.715
2	14.456	621.5	17.5	0.5355	13.467	0.735

3anj: AD-H, 90/10, 1.0 ml/min, 254 nm

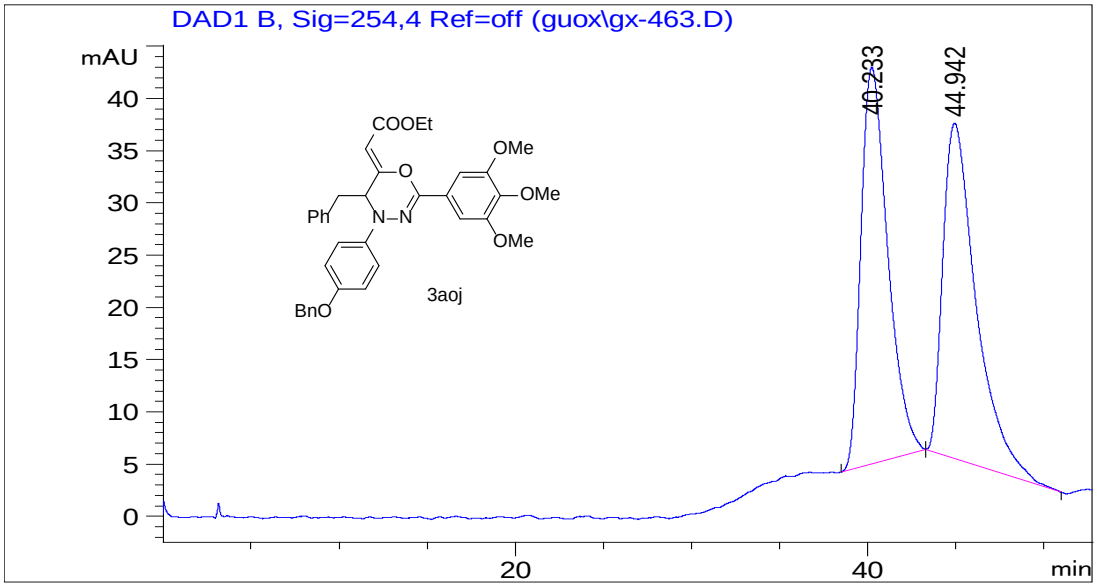


#	Time	Area	Height	Width	Area%	Symmetry
1	7.816	5383.3	308.2	0.2678	50.047	0.717
2	13.047	5373.3	163.1	0.5043	49.953	0.756

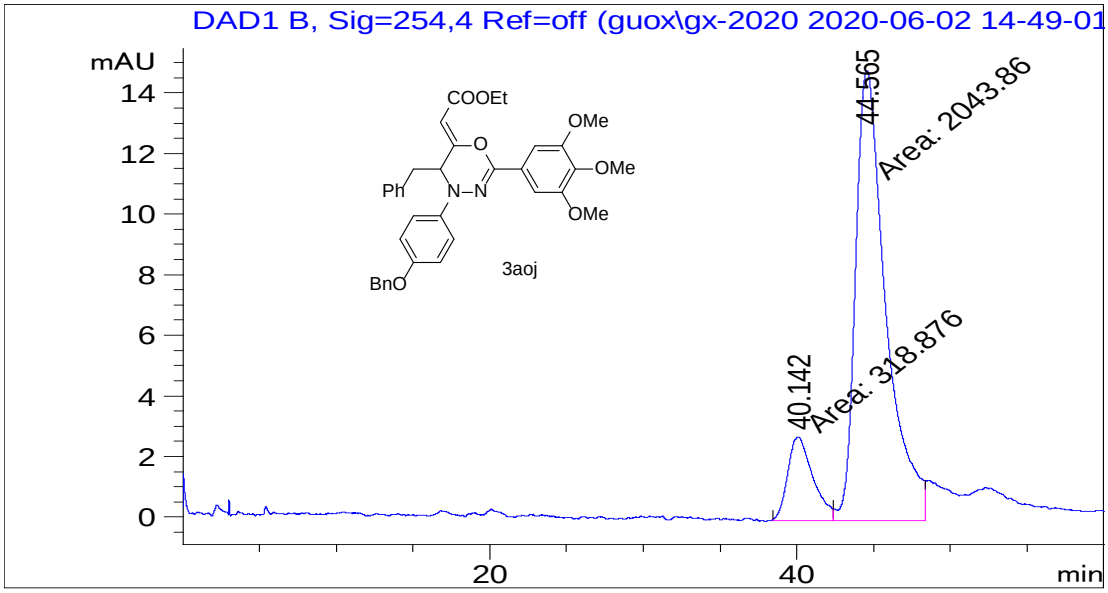


#	Time	Area	Height	Width	Area%	Symmetry
1	7.862	4760.8	268.2	0.2711	86.193	0.716
2	13.153	762.6	23.2	0.5061	13.807	0.802

3aoj: IC-3, 90/10, 1.0 ml/min, 254 nm

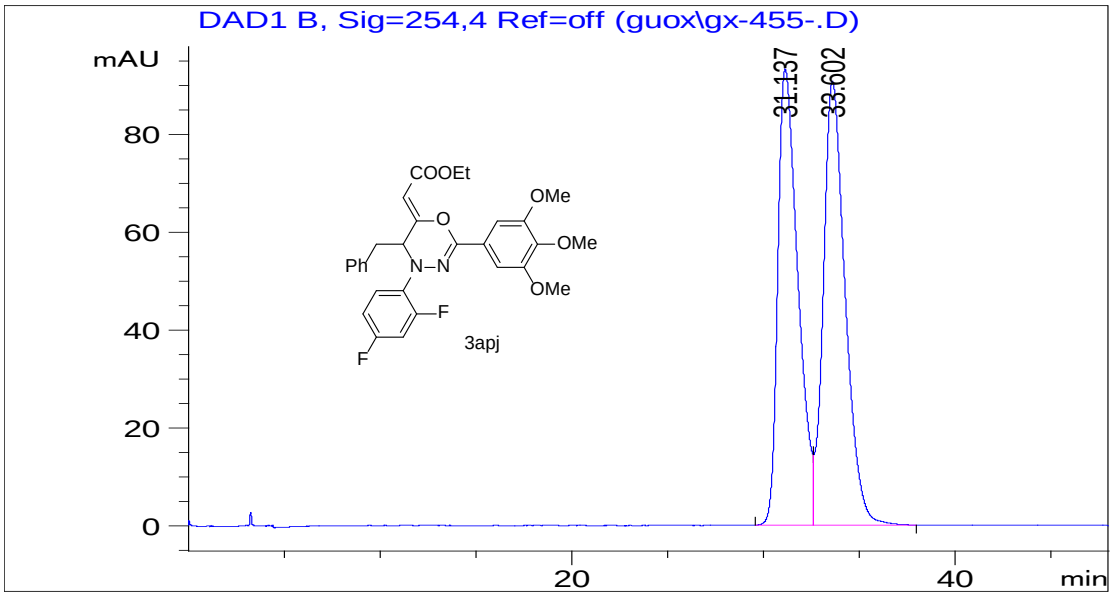


#	Time	Area	Height	Width	Area%	Symmetry
1	40.233	4021.4	37.9	1.4966	48.796	0.634
2	44.942	4219.8	32.1	1.7614	51.204	0.502

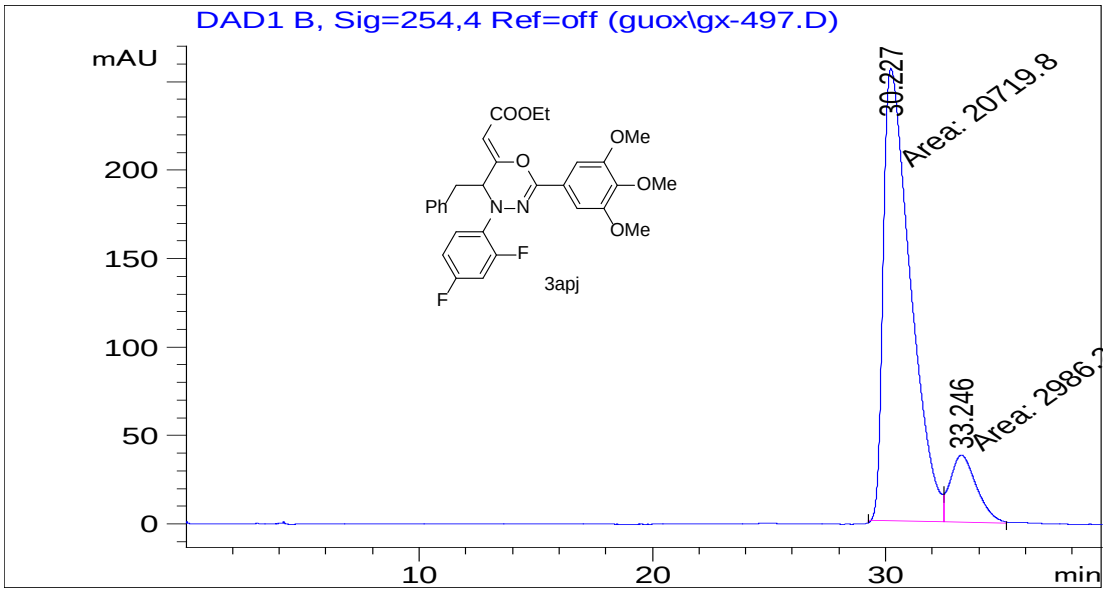


#	Time	Area	Height	Width	Area%	Symmetry
1	40.142	318.9	2.8	1.9216	13.496	0.767
2	44.565	2043.9	14.9	2.2932	86.504	0.579

3apj: AD-H, 99/1, 1.0 ml/min, 254 nm

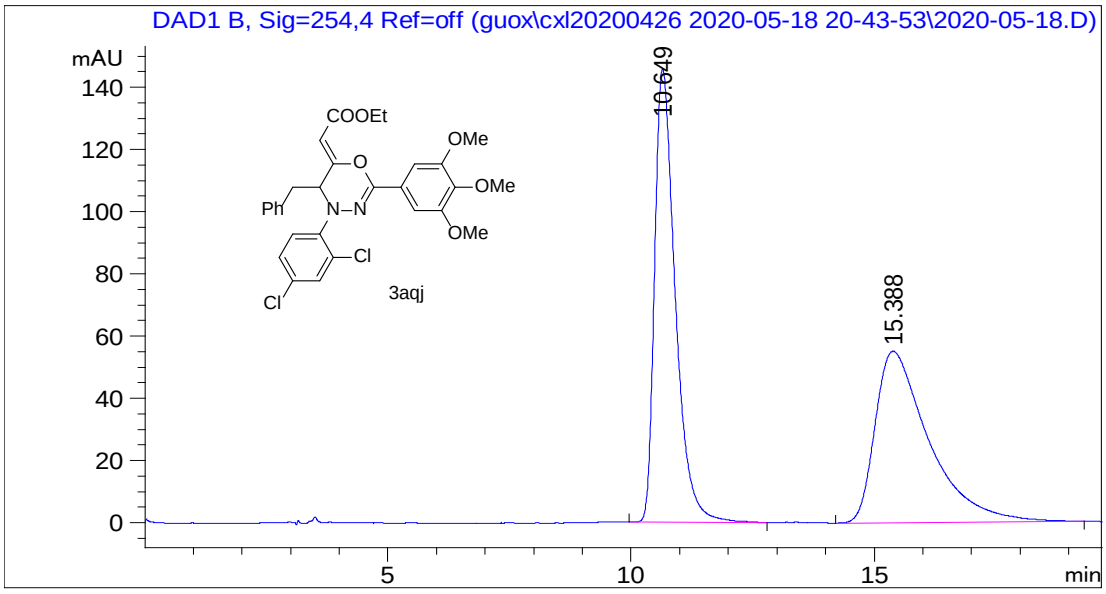


#	Time	Area	Height	Width	Area%	Symmetry
1	31.137	6811.6	93.2	1.091	48.603	0.548
2	33.602	7203.1	90.7	1.1742	51.397	0.676

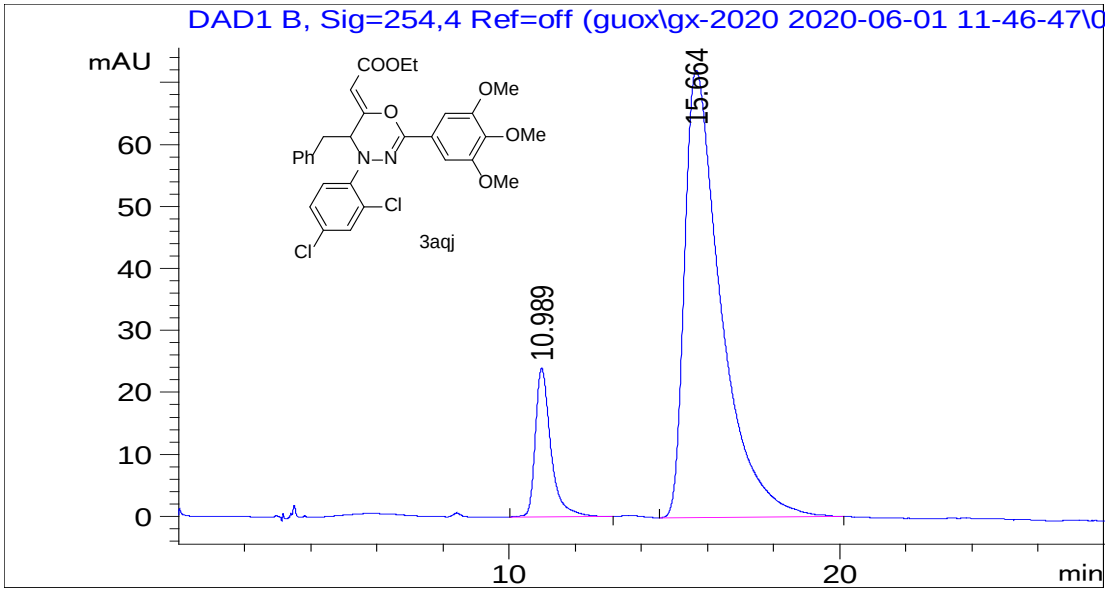


#	Time	Area	Height	Width	Area%	Symmetry
1	30.227	20719.8	255.5	1.3515	87.403	0
2	33.246	2986.3	38	1.3112	12.597	0.704

3aqj: OD-H, 98/2, 1.0 ml/min, 254 nm

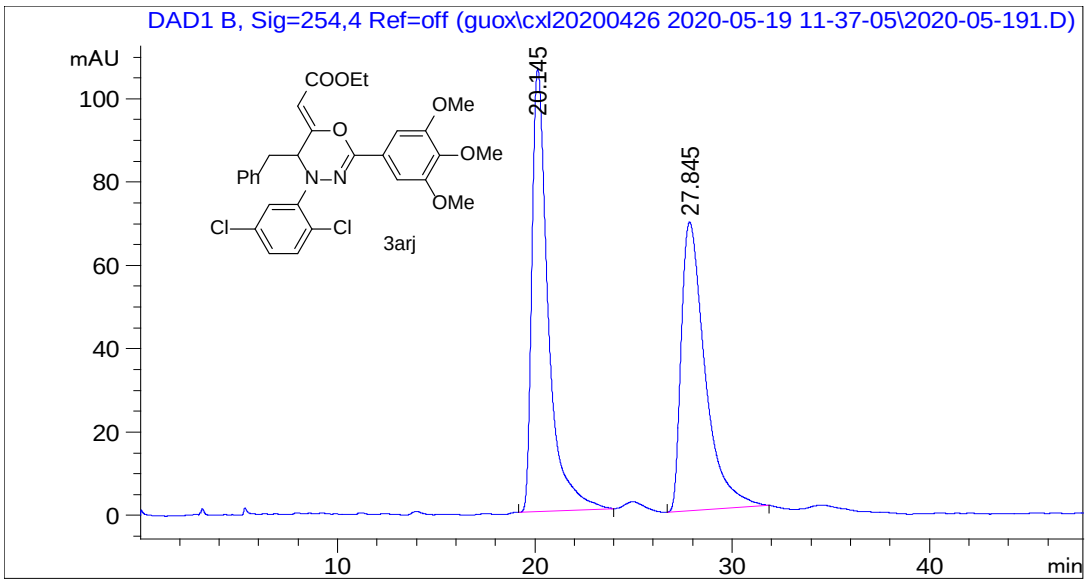


#	Time	Area	Height	Width	Area%	Symmetry
1	10.649	4291.8	145.8	0.4475	50.261	0.578
2	15.388	4247.3	55.1	1.1227	49.739	0.466

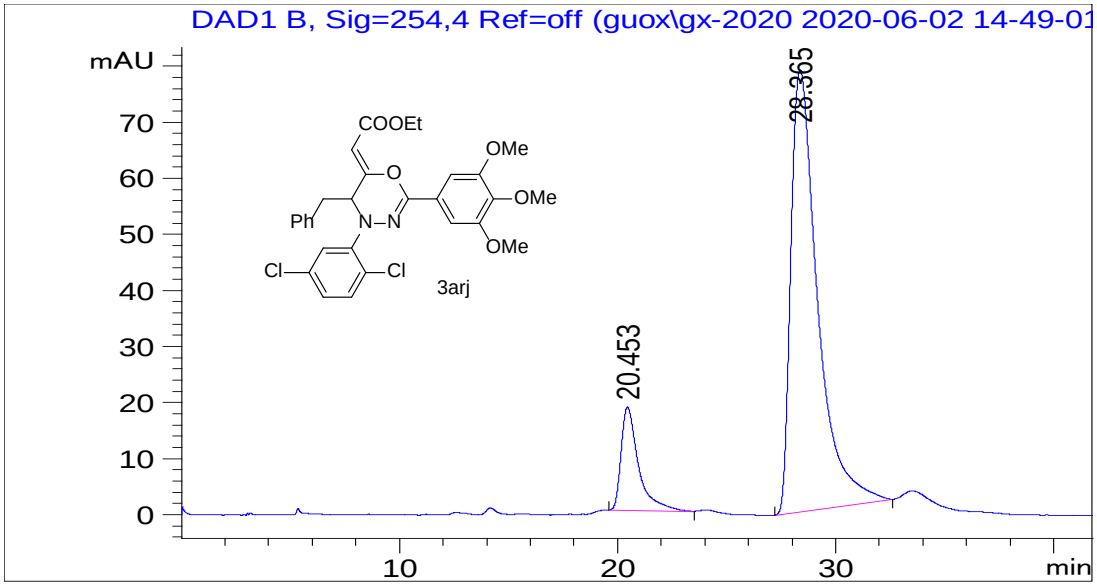


#	Time	Area	Height	Width	Area%	Symmetry
1	10.989	780.8	24	0.49	12.372	0.628
2	15.664	5530.7	72.2	1.118	87.628	0.431

3arj: IC-3, 90/10, 1.0 ml/min, 254 nm

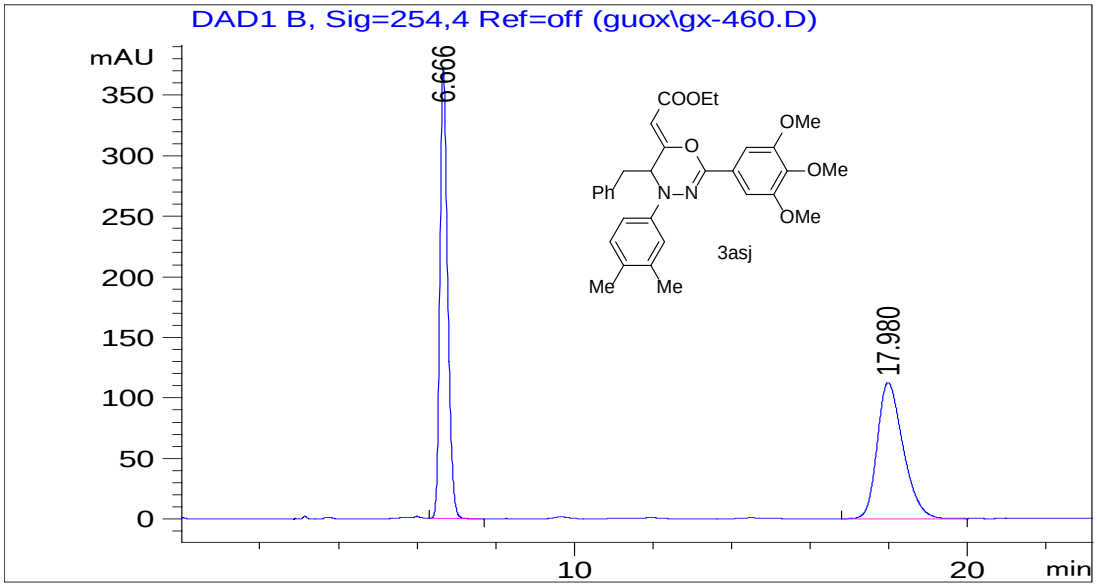


#	Time	Area	Height	Width	Area%	Symmetry
1	20.145	6112.7	106.4	0.8522	51.142	0.495
2	27.845	5839.6	69.4	1.2581	48.858	0.493

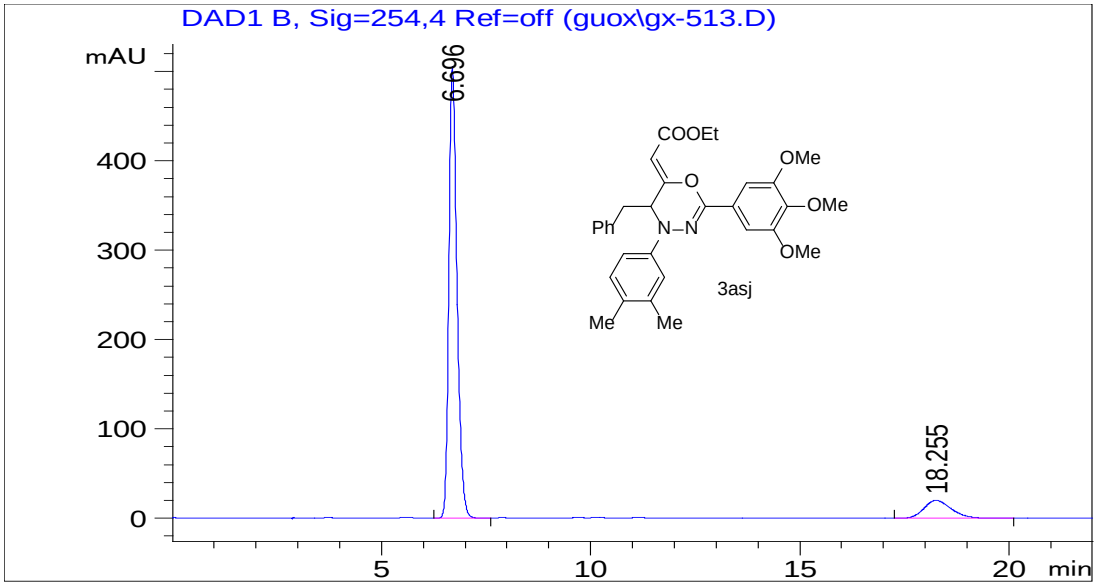


#	Time	Area	Height	Width	Area%	Symmetry
1	20.453	1016.5	18.4	0.7994	13.112	0.526
2	28.365	6736.1	78.9	1.2518	86.888	0.479

3asj: AD-H, 90/10, 1.0 ml/min, 254 nm

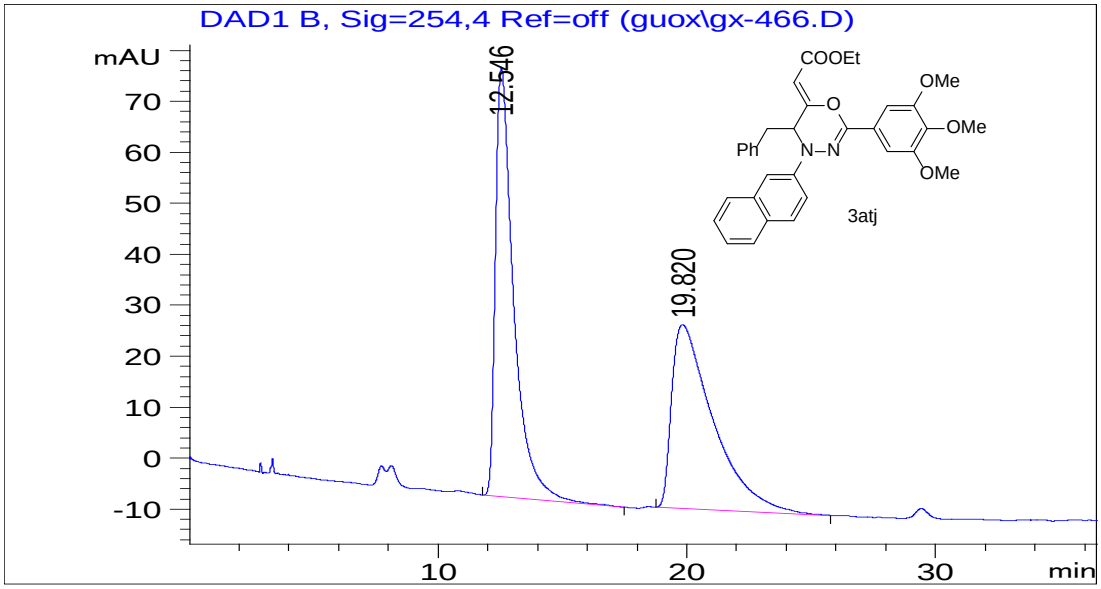


#	Time	Area	Height	Width	Area%	Symmetry
1	6.666	5115	372.7	0.2081	49.885	0.708
2	17.98	5138.6	112.5	0.6949	50.115	0.689

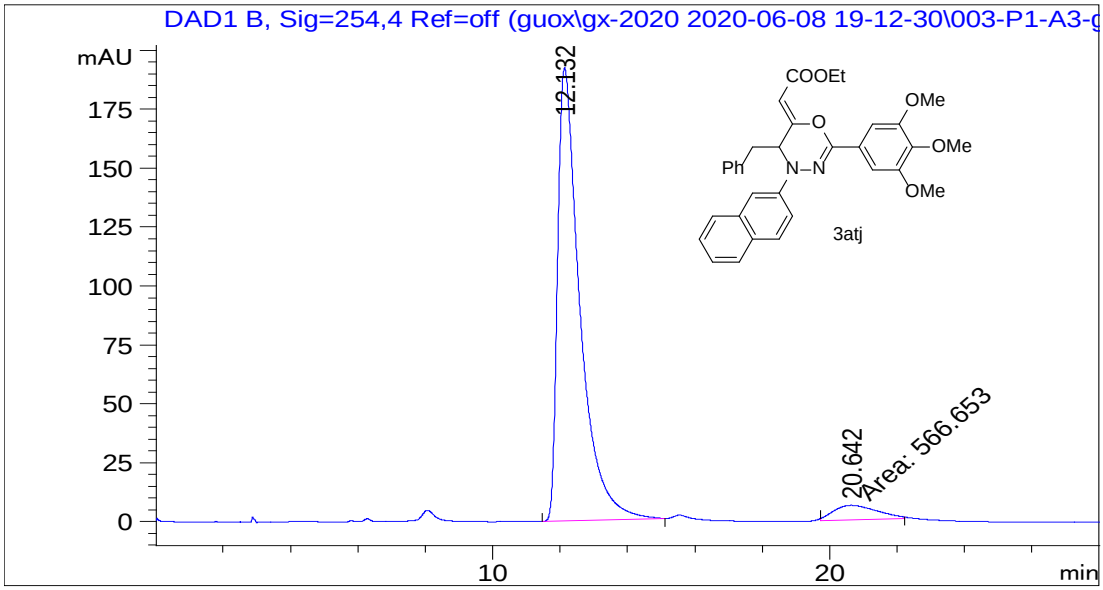


#	Time	Area	Height	Width	Area%	Symmetry
1	6.696	6855.9	505.4	0.2043	88.178	0.714
2	18.255	919.1	20.1	0.6927	11.822	0.75

3atj: OD-H, 90/10, 1.0 ml/min, 254 nm

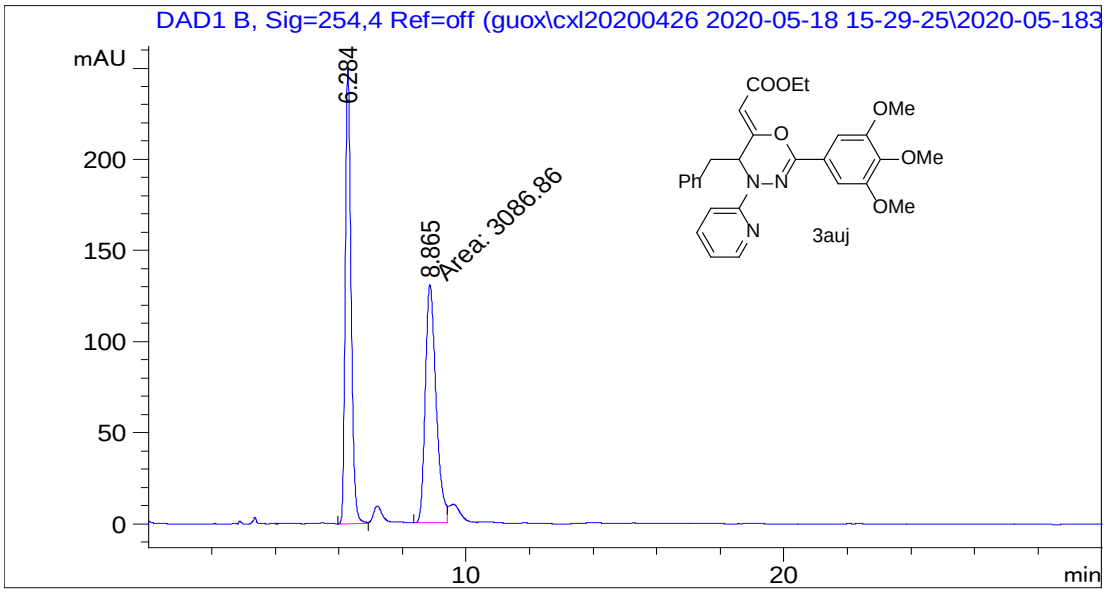


#	Time	Area	Height	Width	Area%	Symmetry
1	12.546	4290.9	84.2	0.7465	50.638	0.433
2	19.82	4182.8	36.1	1.6108	49.362	0.321

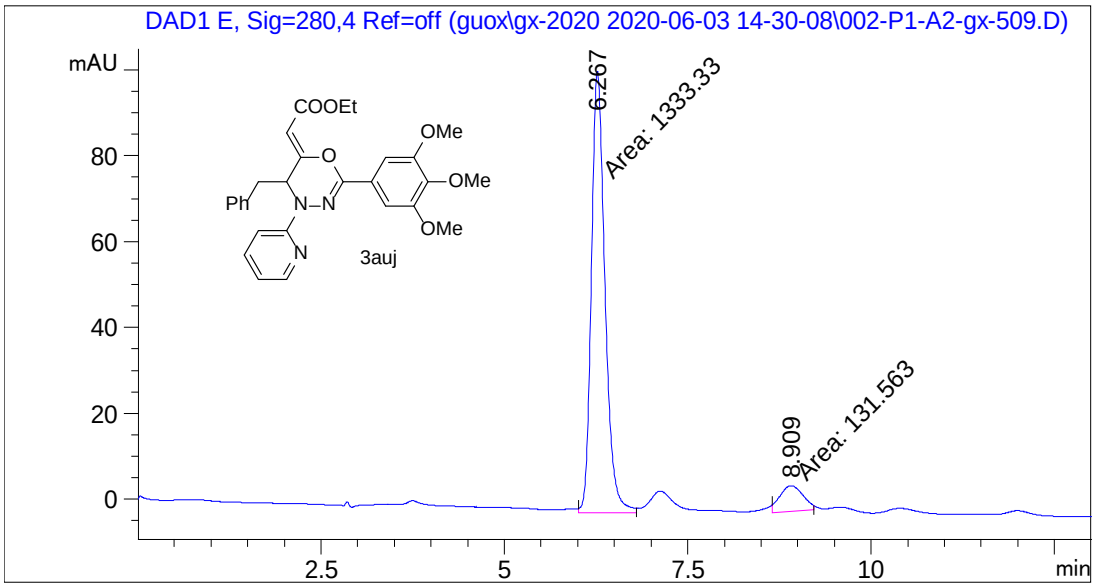


#	Time	Area	Height	Width	Area%	Symmetry
1	12.132	8215.9	192.3	0.6766	93.480	0.406
2	20.642	566.7	6.3	1.5005	6.520	0.698

3auj: OD-H, 90/10, 1.0 ml/min, 254 nm

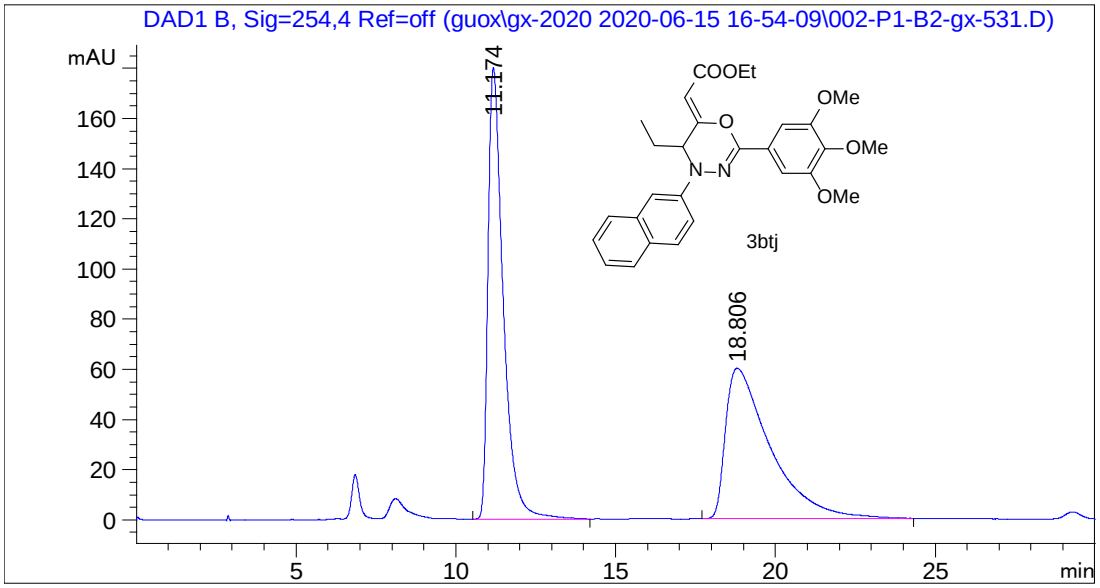


#	Time	Area	Height	Width	Area%	Symmetry
1	6.284	3175.7	249.6	0.1965	50.709	0.745
2	8.865	3086.9	130.7	0.3938	49.291	0.714

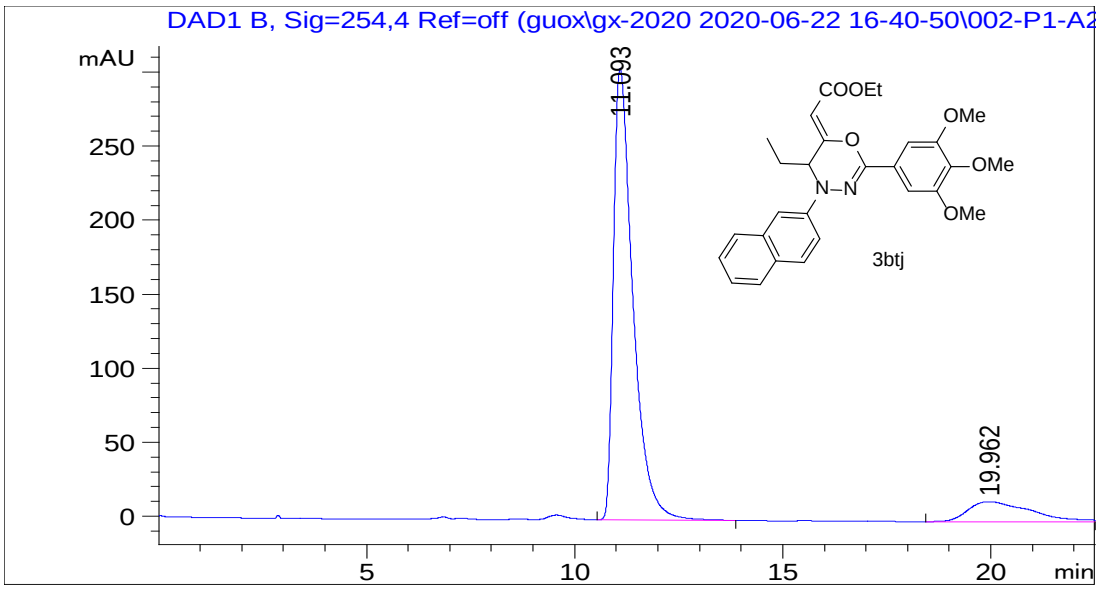


#	Time	Area	Height	Width	Area%	Symmetry
1	6.267	1333.3	102.9	0.216	91.019	0.752
2	8.909	131.6	6	0.367	8.981	0.96

3btj: OD-H, 90/10, 1.0 ml/min, 254 nm

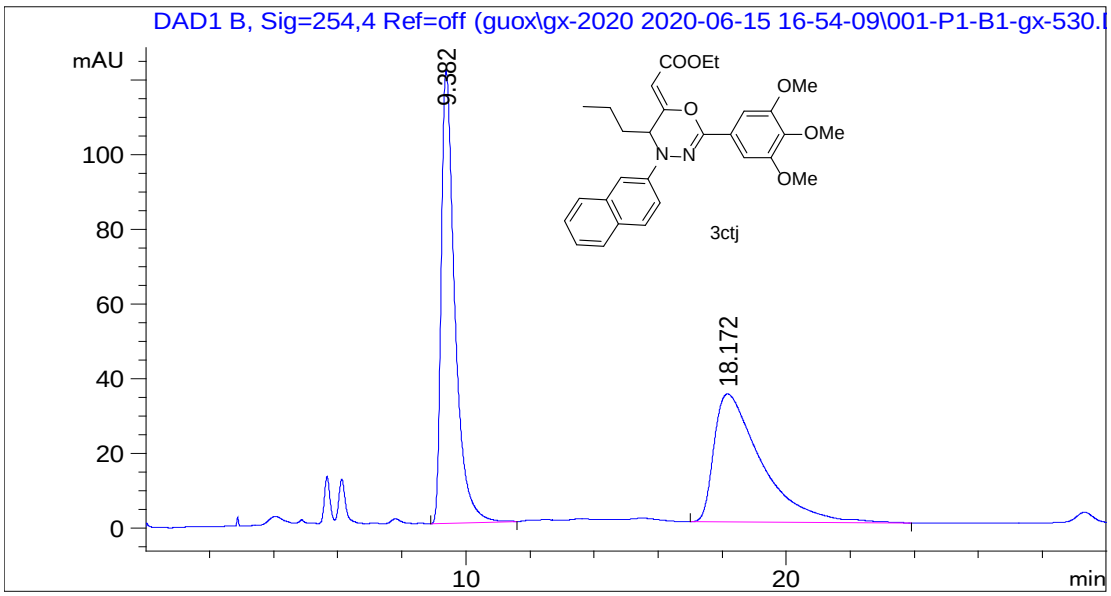


#	Time	Area	Height	Width	Area%	Symmetry
1	11.174	5967.5	180	0.4968	50.594	0.493
2	18.806	5827.4	60	1.3619	49.406	0.327

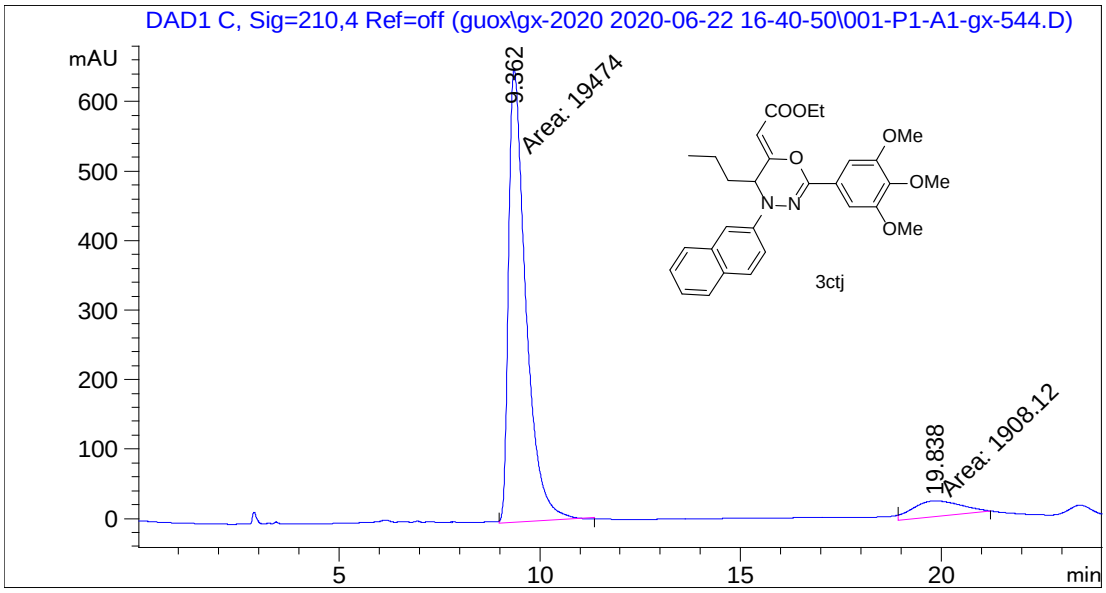


#	Time	Area	Height	Width	Area%	Symmetry
1	11.093	9920.7	304.7	0.4878	87.671	0.475
2	19.962	1395.1	13.5	1.3177	12.329	0.477

3ctj: OD-H, 90/10, 1.0 ml/min, 254 nm

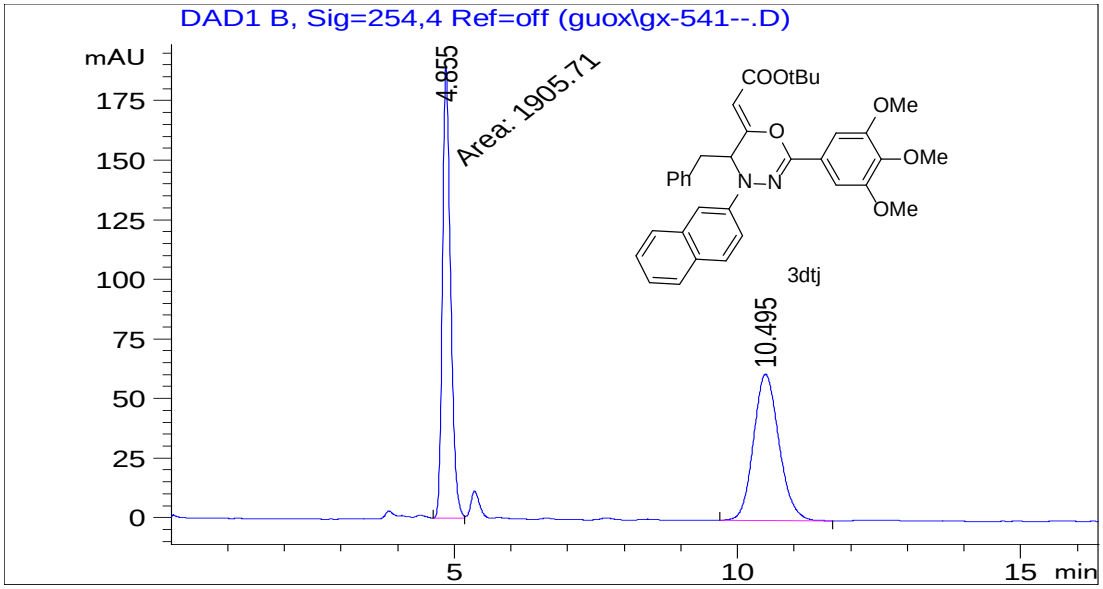


#	Time	Area	Height	Width	Area%	Symmetry
1	9.382	3595.1	121.3	0.4416	50.515	0.501
2	18.172	3521.9	34.3	1.4227	49.485	0.373

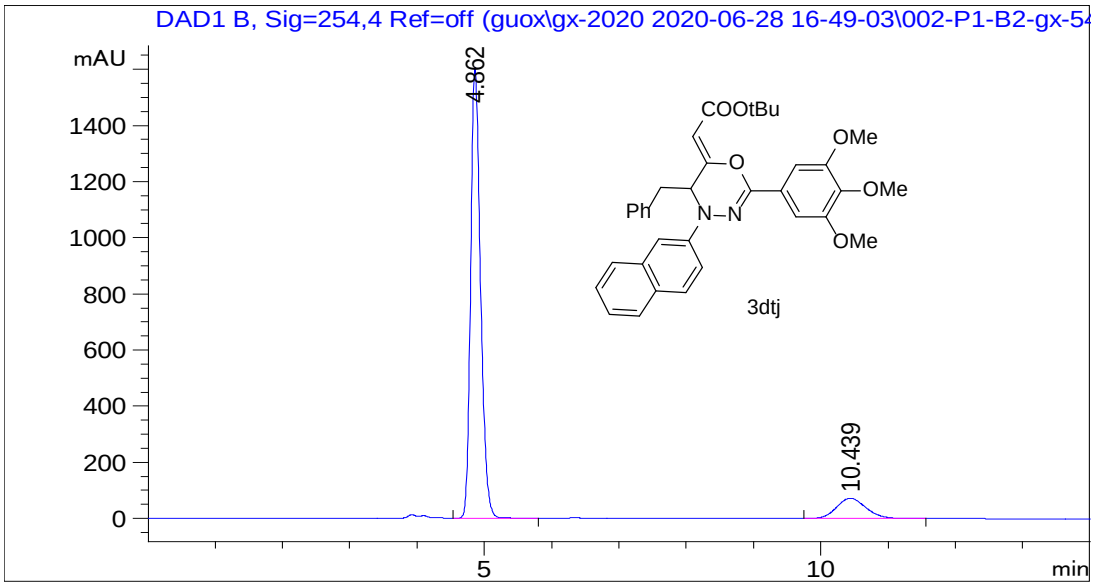


#	Time	Area	Height	Width	Area%	Symmetry
1	9.362	19474	652.9	0.4971	91.076	0.461
2	19.838	1908.1	22.7	1.3999	8.924	0.897

3dtj: AD-H, 80/20, 1.0 ml/min, 254 nm

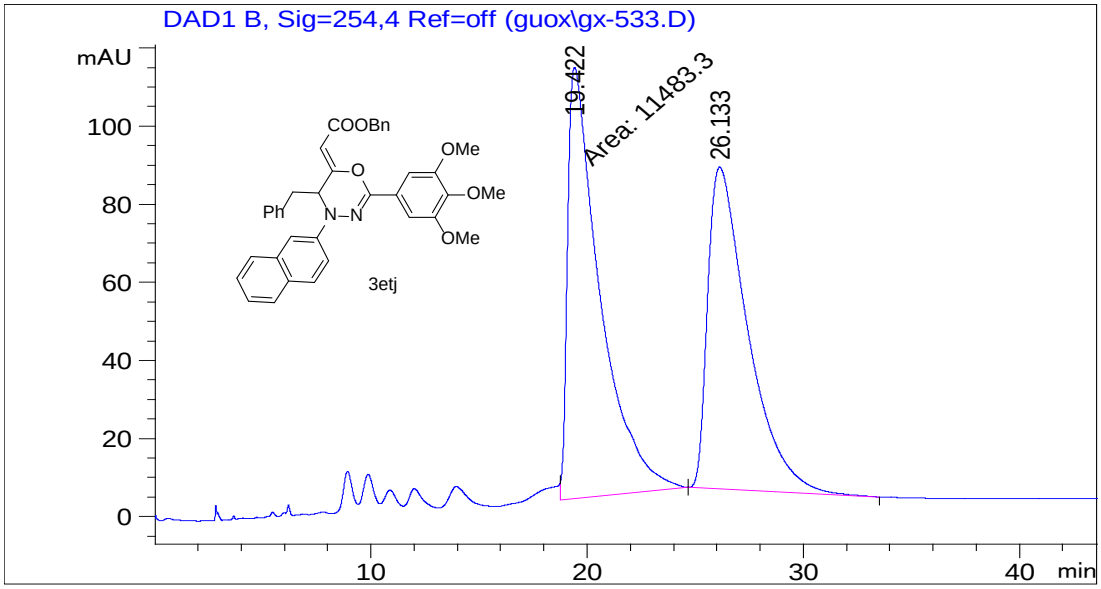


#	Time	Area	Height	Width	Area%	Symmetry
1	4.855	1905.7	189.5	0.1676	49.856	0.743
2	10.495	1916.7	61.3	0.4847	50.144	0.847

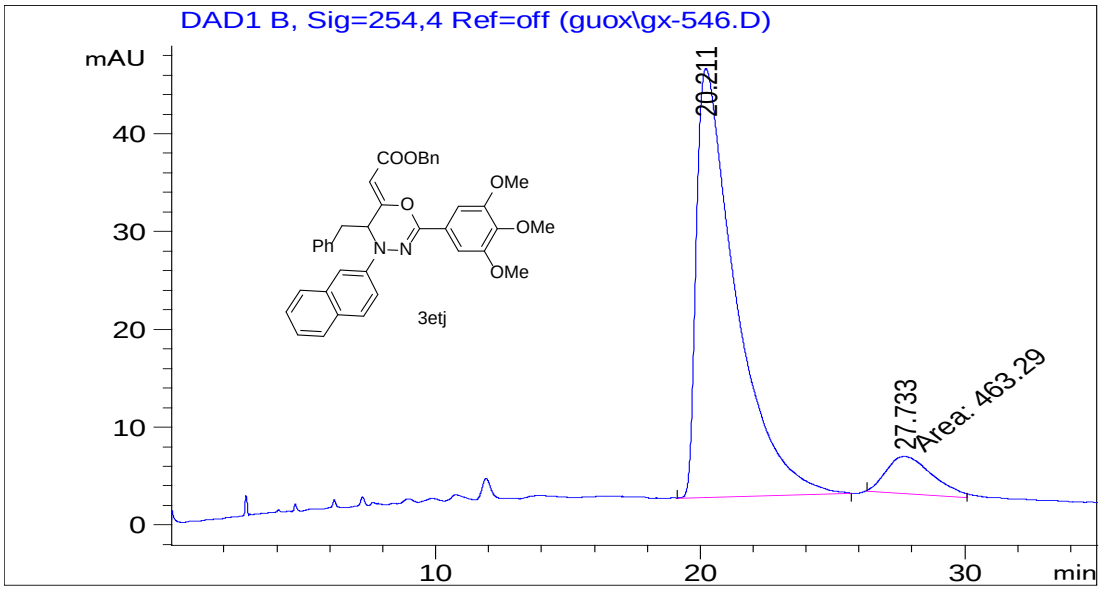


#	Time	Area	Height	Width	Area%	Symmetry
1	4.862	16382	1605.9	0.157	88.206	0.726
2	10.439	2190.5	71.9	0.4712	11.794	0.838

3etj: OD-H, 85/15, 1.0 ml/min, 254 nm

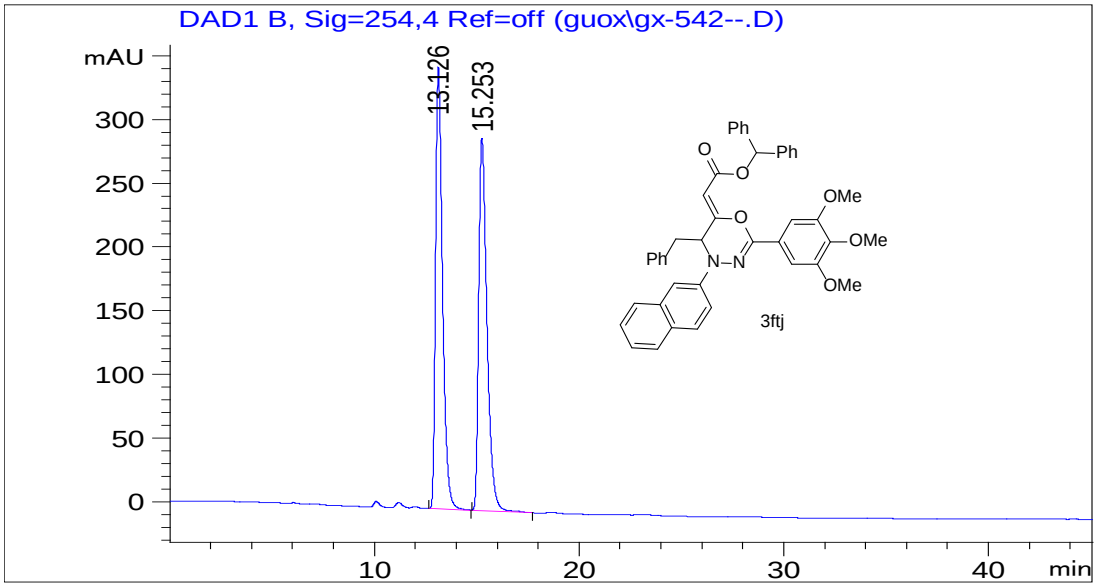


#	Time	Area	Height	Width	Area%	Symmetry
1	19.422	11483.3	110.5	1.7317	51.895	0.266
2	26.133	10644.7	82.5	1.8018	48.105	0.392

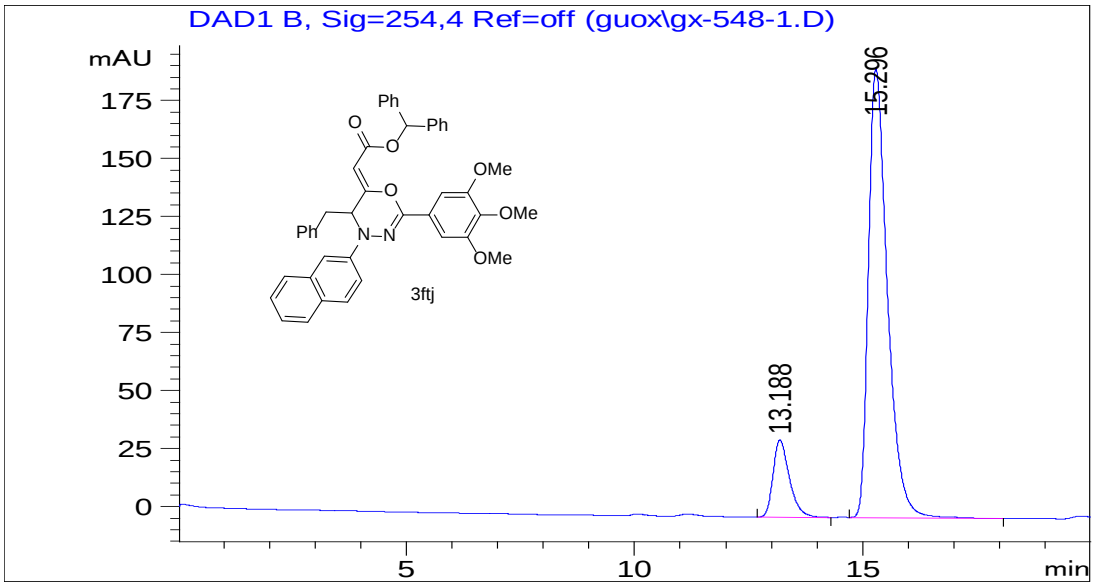


#	Time	Area	Height	Width	Area%	Symmetry
1	20.211	4520.2	43.8	1.4071	90.704	0.294
2	27.733	463.3	3.8	2.018	9.296	0.622

3ftj: ID-3, 80/20, 1.0 ml/min, 254 nm

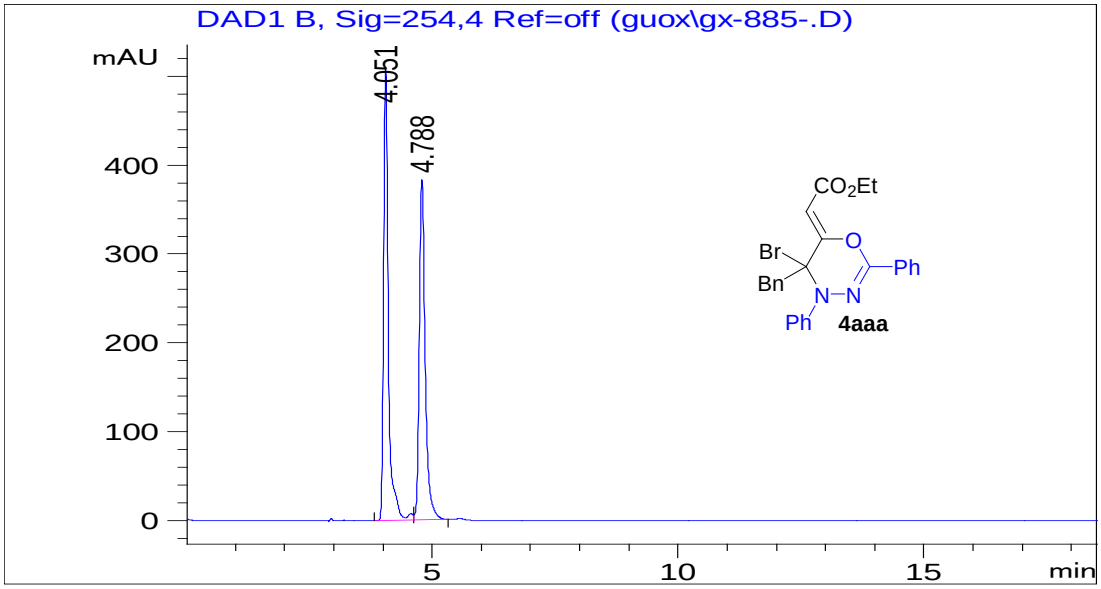


#	Time	Area	Height	Width	Area%	Symmetry
1	13.126	8187.9	346.5	0.3577	49.967	0.605
2	15.253	8198.6	292.4	0.4251	50.033	0.573

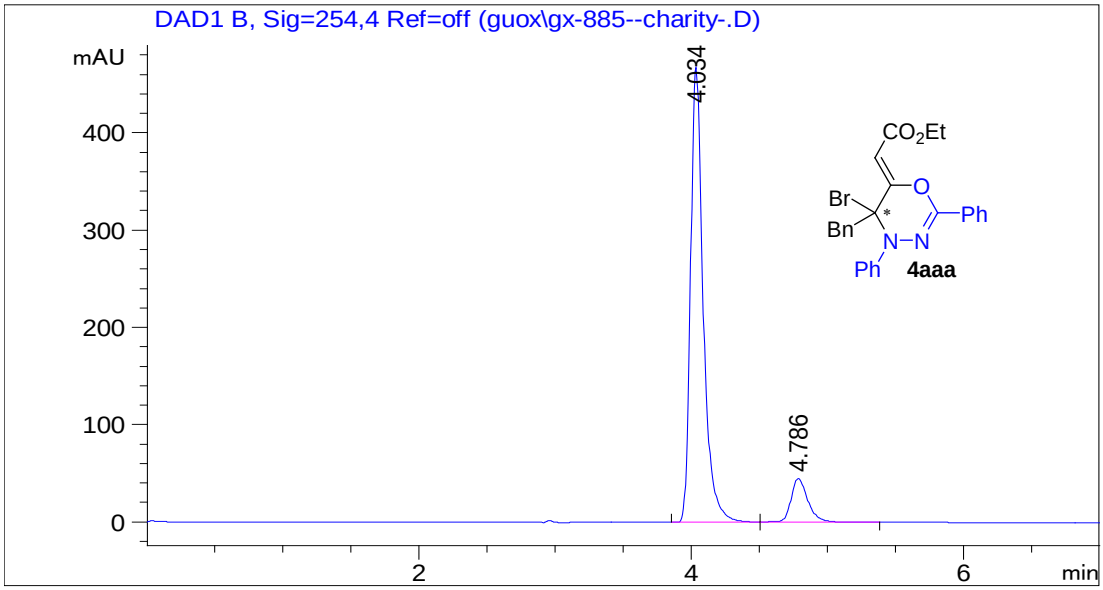


#	Time	Area	Height	Width	Area%	Symmetry
1	13.188	829.6	33.3	0.3787	12.877	0.664
2	15.296	5613	193.8	0.4399	87.123	0.599

4aaa: OD-H, 95/5, 1.0 ml/min, 254 nm

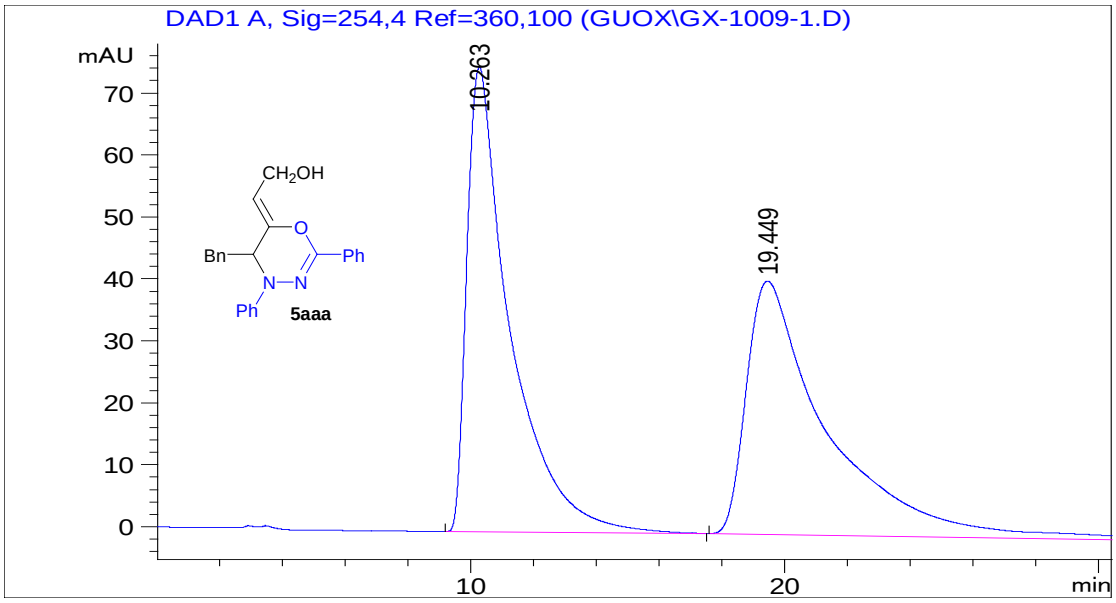


#	Time	Area	Height	Width	Area%	Symmetry
1	4.051	3455.1	510.4	0.0997	51.580	0.625
2	4.788	3243.4	383.5	0.1287	48.420	0.666

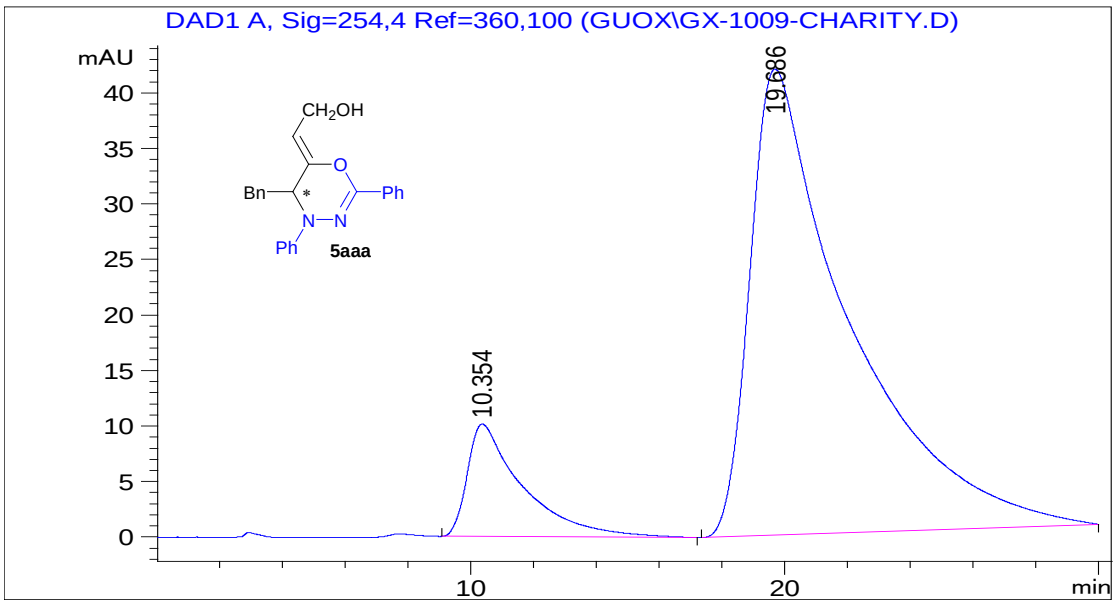


#	Time	Area	Height	Width	Area%	Symmetry
1	4.034	3032	467.5	0.0977	88.042	0.677
2	4.786	392.4	45.1	0.1296	11.958	0.745

5aaa: AD-H, 95/5, 1.0 ml/min, 254 nm

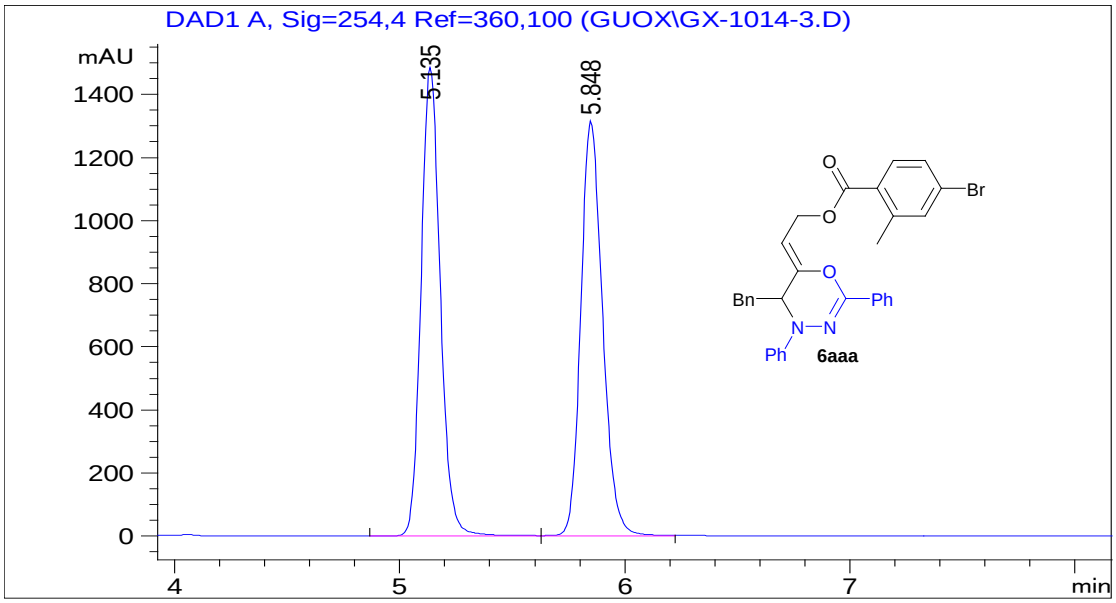


#	Time	Area	Height	Width	Area%	Symmetry
1	10.263	7183	75	1.3594	49.275	0.359
2	19.449	7394.5	40.9	2.4701	50.725	0.316

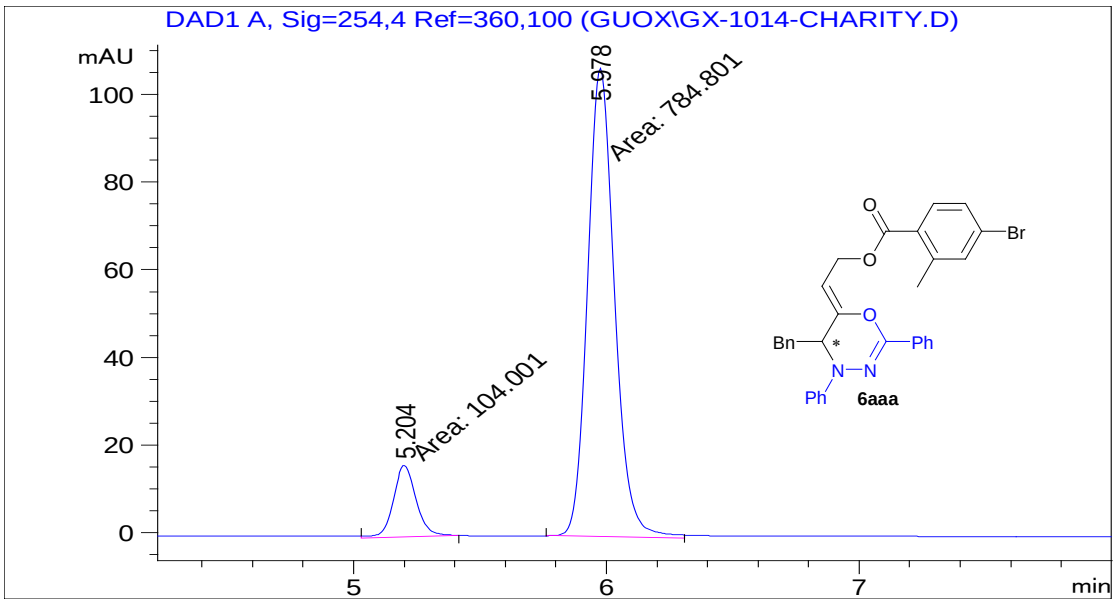


#	Time	Area	Height	Width	Area%	Symmetry
1	10.354	1240.8	10.1	1.6526	11.845	0.342
2	19.686	9233.9	42	2.9519	88.155	0.313

6aaa: ID-3, 90/10, 1.0 ml/min, 254 nm

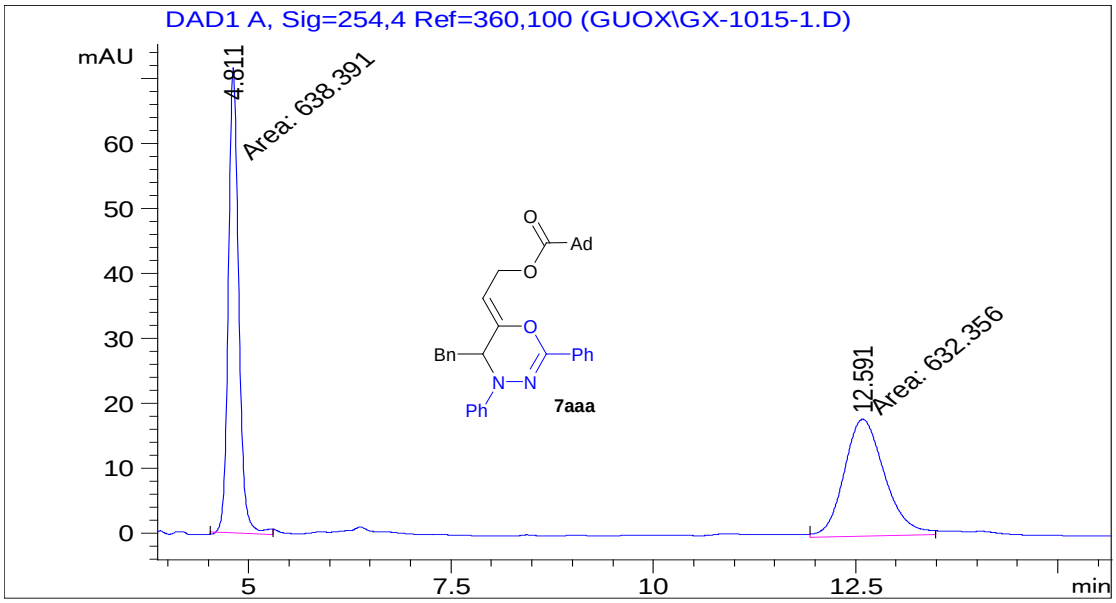


#	Time	Area	Height	Width	Area%	Symmetry
1	5.135	8795.1	1484.9	0.0911	49.866	0.846
2	5.848	8842.4	1315.4	0.1044	50.134	0.787

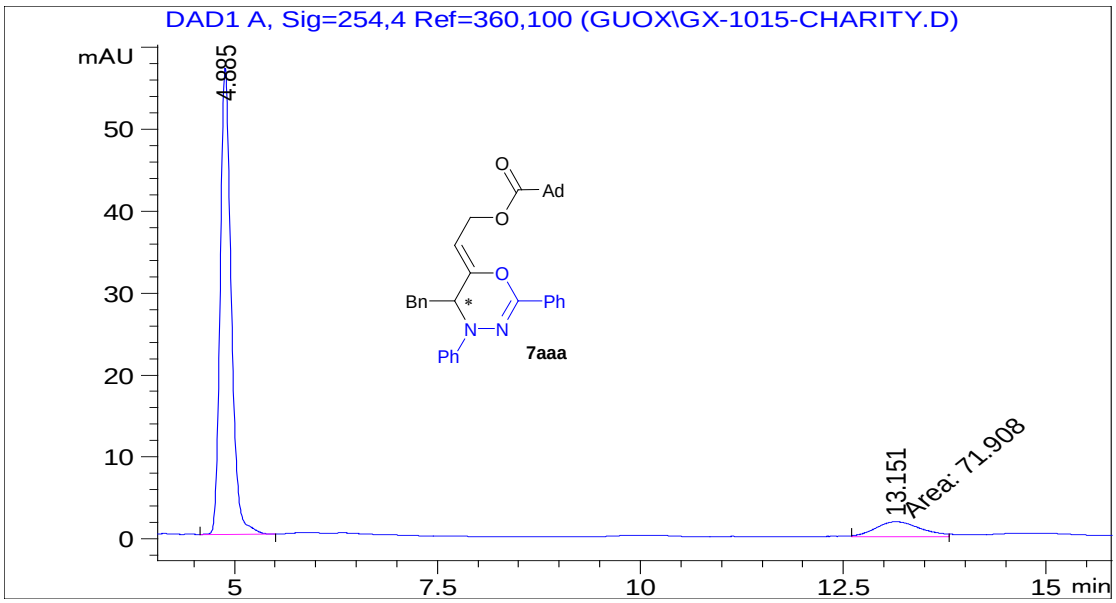


#	Time	Area	Height	Width	Area%	Symmetry
1	5.204	104	16.4	0.1059	11.701	0.929
2	5.978	784.8	107	0.1223	88.299	0.843

7aaa: OD-H, 90/10, 1.0 ml/min, 254 nm



#	Time	Area	Height	Width	Area%	Symmetry
1	4.811	638.4	71.7	0.1483	50.237	0.824
2	12.591	632.4	18	0.5846	49.763	0.819



#	Time	Area	Height	Width	Area%	Symmetry
1	4.885	537.9	57	0.1442	88.208	0.746
2	13.151	71.9	1.8	0.6516	11.792	0.808

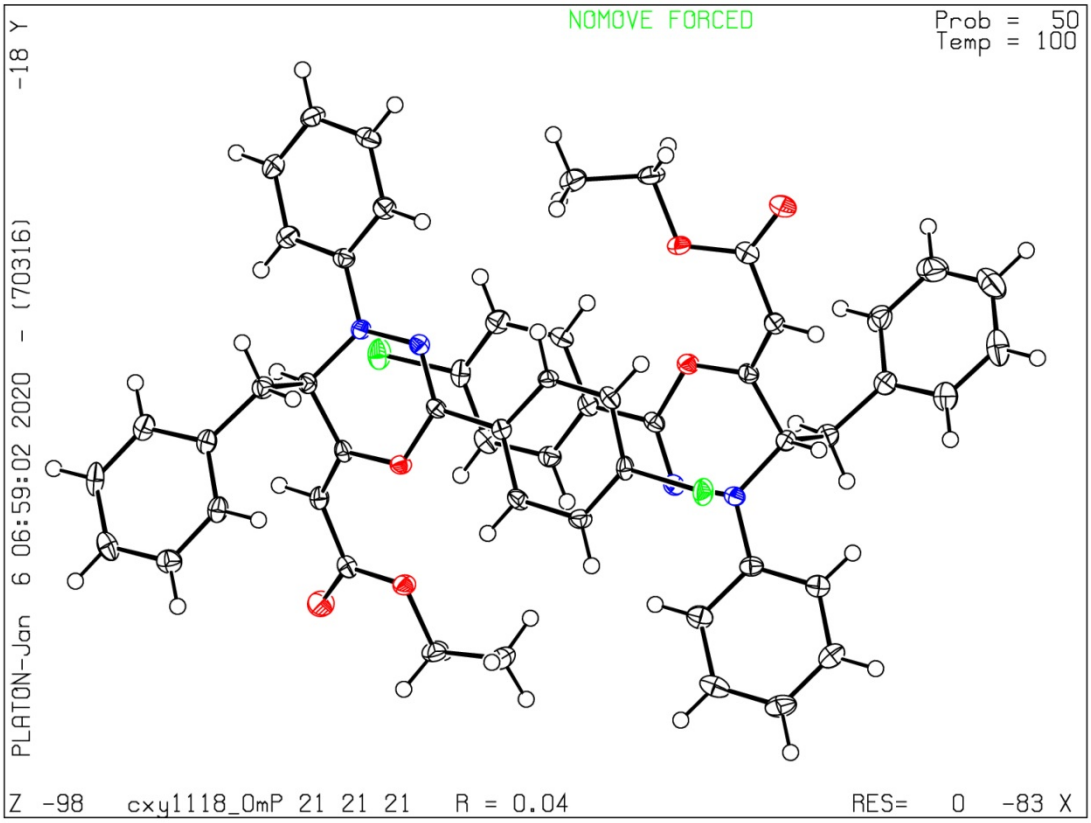
D: X-Ray Analysis

cxy1118_0m (3aac CCDC 2046544)

Crystal data and structure refinement for cxy1118_0m.

Identification code	cxy1118_0m
Empirical formula	$C_{26}H_{23}ClN_2O_3$
Formula weight	446.91
Temperature/K	100
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	10.234(3)
b/Å	14.235(5)
c/Å	30.529(12)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	4448(3)
Z	8
ρ_{calc}g/cm^3	1.335
μ/mm^{-1}	0.203
F(000)	1872.0
Crystal size/mm^3	0.41 × 0.36 × 0.36
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.792 to 56.576
Index ranges	$-9 \le h \le 13$, $-18 \le k \le 18$, $-29 \le l \le 40$
Reflections collected	22253
Independent reflections	10880 [R_{int} = 0.0421, R_{sigma} = 0.0654]
Data/restraints/parameters	10880/0/580
Goodness-of-fit on F^2	1.020
Final R indexes [I>=2σ (I)]	R_1 = 0.0427, wR_2 = 0.0901
Final R indexes [all data]	R_1 = 0.0554, wR_2 = 0.0958
Largest diff. peak/hole / e Å^{-3}	0.37/-0.24
Flack parameter	-0.04(3)

cxy1118_0m (3aac CCDC 2046544)



cxy3100_0m (3aak CCDC 2046545)

Crystal data and structure refinement for cxy3100_0m.

Identification code	cxy3100_0m
Empirical formula	$C_{30}H_{34}N_2O_3$
Formula weight	470.59
Temperature/K	100
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	10.6498(4)
b/Å	10.9500(4)
c/Å	21.8176(8)
α/°	90
β/°	104.078(2)
γ/°	90
Volume/Å^3	2467.85(16)
Z	4
ρ_{calc}g/cm^3	1.267
μ/mm^{-1}	0.645
F(000)	1008.0
Crystal size/mm^3	0.35 × 0.28 × 0.21
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection/°	8.356 to 136.68
Index ranges	$-12 \leq h \leq 12$, $-13 \leq k \leq 13$, $-20 \leq l \leq 26$
Reflections collected	23342
Independent reflections	4523 [R_{int} = 0.0322, R_{sigma} = 0.0216]
Data/restraints/parameters	4523/0/318
Goodness-of-fit on F^2	1.030
Final R indexes [I>=2σ (I)]	R_1 = 0.0359, wR_2 = 0.0848
Final R indexes [all data]	R_1 = 0.0389, wR_2 = 0.0864
Largest diff. peak/hole / e Å^{-3}	0.22/-0.20

cxy3100_0m (3aak CCDC 2046545)