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A New Chiral C_1-Symmetric NHC-Catalyzed Addition to α-Aryl Substituted α,β-Disubstituted Enals: Enantioselective Synthesis of Fully Functionalized Dihydropyranones

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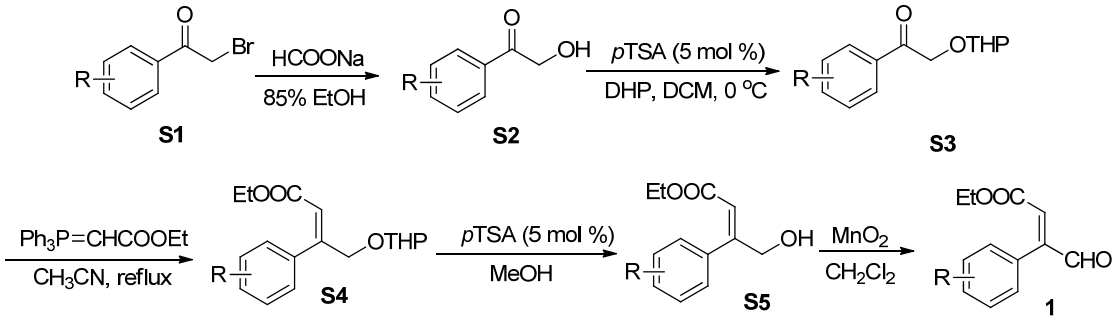
Contents

1. General Information

Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Analytical thin-layer chromatography (TLC) was performed on silicycle silica gel plates with F-254 indicator and compounds were visualized by irradiation with UV light. Flash chromatography was carried out utilizing silica gel 200-300 mesh. ^{1}H NMR, ^{13}C NMR spectra were recorded on a Bruker AM-400 spectrometer (400 MHz ^{1}H, 100 MHz ^{13}C). The spectra were recorded in $CDCl_3$ as the solvent at room temperature, ^{1}H and ^{13}CNMR chemical shifts are reported in ppm relative to either the residual solvent peak (^{13}C) (δ = 77.00 ppm) or TMS (^{1}H) (δ = 0 ppm) as an internal standard. Data for ^{1}H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet), integration, coupling constant (Hz) and assignment. Data for ^{13}C NMR are reported as chemical shift. IR spectra were recorded using Nicolet NEXUS 670 FT-IR instrument and are reported in wavenumbers (cm^{-1}). HRMS were performed on Bruker Apex II mass instrument (ESI). Enantiomeric excess values were determined by HPLC with employing a Daicel Chirapak AD-H/OD-H/OJ-H on Agilent 1100 series and Waters 600 Deltaeluting with *i*-PrOH and *n*-hexane. Optical rotation was measured on the Perkin Elmer 341 polarimeter with $[\alpha]_D$ values reported in degrees; concentration (c) is in g/100 mL.

2. Preparation and Spectral Data of α-Aryl Substituted α,β-Disubstituted Aldehydes



2.1 General Procedure for α-Aryl Substituted α,β-Disubstituted Aldehydes

A solution of **S1** (35 mmol) of *α*-bromo ketone and 15.0g (220 mmol) of sodium formate in 90 mL 85% ethanol was heated to reflux for 12h. After the reaction was completed, the mixture was cool to ambient temperature, and ethanol was removed in *vacuo*, diluted with water (20 mL), aqueous layer was extracted with ethyl acetate (3 × 40 mL), and the combined organic phases were dried over Na_2SO_4, filtered and concentrated. The ketols **S2** were recrystallized from 95% ethanol.

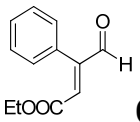
A 250 mL flame-dried round bottom flask was charged with **S2** (22 mmol) and *p*-toluenesulfonicacid 230 mg (1.1 mmol) under a dry N_2 atmosphere. After DCM (130 mL) was added to the flask, the mixture was cooled to 0 ^oC. 3,4-2H-dihydropyran (DHP) 9.26 g (110 mmol) was then added dropwise to the mixture. After a further 2 hours of stirring at 0 ^oC (determined by TLC), the reaction mixture was quenched with saturated $NaHCO_3$. The organic layer was separated and the aqueous layer was extracted into DCM (3 × 20 mL). The combined organics were washed with brine, dried over Na_2SO_4, filtered, and concentrated in *vacuo*, and purified with flash chromatography (eluent, ethyl acetate/petroleum ether 1/9) to yield crude product **S3**.

The crude product **S3** (10 mmol) was dissolved in acetonitrile (20 mL) in a 50 mL round bottom flask, ethyl 2-(triphenylphosphoranylidene)acetate 4.0 g (12 mmol) was added to the solution. The mixture was refluxed for 12 h. The mixture was evaporated to distill off one third of acetonitrile, and then continued to react for about 12 h (determined by TLC) until the reaction was complete. Solvent was distilled off, the concentrate was extracted with ether and washed with water. The combined organicswere dried over Na_2SO_4, filtered, and concentrated in *vacuo*, and purified with flash chromatography (eluent, ethyl acetate/petroleum ether 1/9) to yield crude coupling product **S4**.

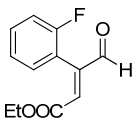
The crude product **S4** (7.5 mmol) was dissolved in MeOH (40 mL) and then cooled to 0 ^{o}C. *p*-Toluenesulfonic acid 71.3 mg (0.375 mmol) was added to the mixture. The reaction was brought to ambient temperature. After afurther 3 hours of stirring at ambient temperature (determined by TLC), the reaction mixture was quenched with saturated $NaHCO_3$ at 0 ^{o}C. MeOH was distilled off, and the concentrate was extracted with ethyl acetate (3 × 20 mL). The combined organics were washed with brine, dried over Na_2SO_4, filtered, and concentrated in *vacuo*, yielding **S5** that was used directly without further purification in the next step.

To a stirred solution of **S5** (5 mmol) in CH_2Cl_2 (50 ml) was added MnO_2 (55 mol) at room temperature. After completion of the reaction (monitored by TLC), MnO_2 was removed, and the organic layers were concentrated in *vacuo*, the crude product was purified by silica gel column chromatography using (eluent, ethyl acetate/petroleum ether 1/8) to afford pure α,β-unsaturated aldehyde **1** as light yellow liquid.

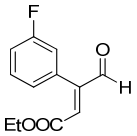
2.2 Spectral Data of Substrates



(E)-ethyl 4-oxo-3-phenylbut-2-enoate (1a) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.73 (s, 1H), 7.40–7.36 (m, 3H), 7.25–7.20 (m, 2H), 6.69 (s, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 1.10 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.7, 165.0, 149.3, 136.0, 131.2, 128.9, 128.8, 127.8, 61.1, 13.6.

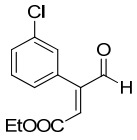


(E)-ethyl 3-(2-fluorophenyl)-4-oxobut-2-enoate (1b) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.74 (s, 1H), 742–7.36 (m, 1H), 7.19–7.14 (m, 2H), 7.10 (t, 1H), 6.84 (s, 1H), 4.17–4.12 (d, *J* = 7.2 Hz, 2H), 1.14 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.7, 164.3, 160.7, 158.3, 144.6, 137.8, 130.9, 130.89, 130.7, 130.69, 123.64, 123.61, 119.5, 119.3, 115.4, 115.2, 61.3, 13.7.

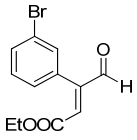


(E)-ethyl 3-(3-chlorophenyl)-4-oxobut-2-enoate (1c)[1] H NMR (400 MHz, $CDCl_3$): δ = 9.75 (s, 1H), 7.40–7.34 (m, 1H), 7.13–7.08 (td, *J* = 2.4 Hz, *J* = 3.6 Hz, 1H), 7.00–6.94 (m, 2H), 6.74 (s, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 1.15 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR

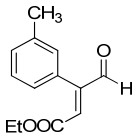
(100 MHz, $CDCl_3$): δ = 192.1, 164.6, 163.4, 160.9, 148.2, 137.0, 133.2, 133.1, 129.5, 129.46, 124.7, 116.2, 116.0, 115.8, 61.3, 13.7.



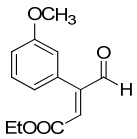
(E)-ethyl 3-(3-chlorophenyl)-4-oxobut-2-enoate (1d) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.73 (s, 1H), 7.39–7.31 (m, 2H), 7.22 (s, 1H), 7.11 (d, *J* = 7.6 Hz, 1H), 6.73 (s, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 1.15 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.1, 164.5, 148.1, 137.0, 133.7, 132.8, 129.1, 129.0, 128.9, 127.1, 61.4, 13.7.



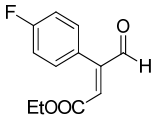
(E)-ethyl 3-(3-bromophenyl)-4-oxobut-2-enoate (1e) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.70 (s, 1H), 7.53–7.50 (m, 1H), 7.37 (t, *J* = 2.0 Hz, 1 H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.16–7.13 (m, 1H), 6.72 (s, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 1.14 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.0, 164.4, 147.8, 137.0, 133.0, 131.8, 131.7, 129.3, 127.5, 121.7, 61.3, 13.7.



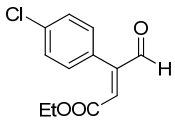
(E)-ethyl 4-oxo-3-(m-tolyl)but-2-enoate (1f) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.74 (s, 1H), 7.27 (q, *J* = 7.6 Hz, 1H), 7.20 (d, *J* = 7.6 Hz, 1H), 7.02 (d, *J* = 7.6 Hz, 2H), 6.68 (s, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 2.36 (s, 3H), 1.13 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.8, 165.1, 149.5, 137.5, 135.8, 131.1, 129.8, 129.4, 127.9, 125.9, 61.1, 21.3, 13.7.



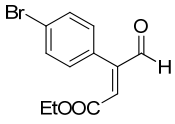
(E)-ethyl 3-(3-methoxyphenyl)-4-oxobut-2-enoate (1g) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.74 (s, 1H), 7.30 (t, *J* = 8.0 Hz, 1H), 6.94 (dd, *J* = 2.4 Hz, *J* = 8.4 Hz, 1H), 6.81–6.77 (m, 1H), 6.70 (s, 1H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.80 (s, 3H), 1.13 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.5, 165.0, 159.1, 149.1, 136.0, 132.5, 129.1, 121.2, 114.6, 114.5, 61.2, 55.2, 13.8.



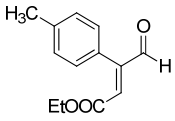
(E)-ethyl 3-(4-fluorophenyl)-4-oxobut-2-enoate (1h) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.74 (s, 1H), 7.26–7.21 (m, 2H), 7.11–7.07 (m, 2H), 6.71 (s, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 1.16 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.6, 164.8, 164.4, 161.9, 148.6, 136.6, 131.1, 131.0, 126.9, 126.8, 115.2, 115.0, 61.3, 13.8.



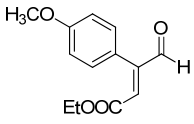
(E)-ethyl 3-(4-chlorophenyl)-4-oxobut-2-enoate (1i) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.73 (s, 1H), 7.38 (dd, *J* = 2.0 Hz, *J* = 6.8 Hz, 2H), 7.16 (dd, *J* = 1.6 Hz, *J* = 6.8 Hz, 2H), 6.71 (s, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 1.17 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.4, 164.7, 148.5, 136.7, 135.2, 130.4, 129.4, 128.2, 61.4, 13.8.



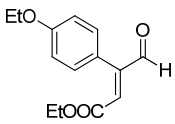
(E)-ethyl 3-(4-bromophenyl)-4-oxobut-2-enoate (1g) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.73 (s, 1H), 7.53 (d, *J* = 8.4 Hz, 2H), 7.10 (d, *J* = 8.4 Hz, 2H), 6.72 (s, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 1.17 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.3, 164.7, 148.6, 136.7, 131.2, 130.7, 130.0, 123.5, 61.4, 13.8.



(E)-ethyl 4-oxo-3-(p-tolyl)but-2-enoate (1k) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.73 (s, 1H), 7.20 (d, *J* = 7.6 Hz, 2H), 7.13 (m, 2H), 6.66 (s, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 2.37 (s, 3H), 1.16 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.9, 165.1, 149.4, 139.0, 135.5, 128.8, 128.6, 128.1, 61.1, 21.3, 13.8.

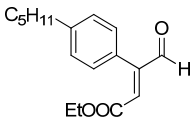


(E)-ethyl 3-(4-methoxyphenyl)-4-oxobut-2-enoate (1l) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.70 (s, 1H), 7.21 (dd, *J* = 2.0 Hz, *J* = 6.8 Hz, 2H), 6.92 (dd, *J* = 2.0 Hz, *J* = 6.8 Hz, 2H), 6.63 (s, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.82 (s, 3H), 1.18 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.3, 165.2, 160.3, 148.9, 135.2, 130.6, 123.0, 113.4, 61.1, 55.2, 13.9.

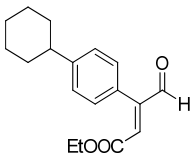


(E)-ethyl 3-(4-ethoxyphenyl)-4-oxobut-2-enoate (1m) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.70 (s, 1H), 7.21 (d, *J* = 8.4 Hz, 2H), 6.92 (d, *J* = 8.8 Hz, 2H), 6.62 (s, 1H), 4.17

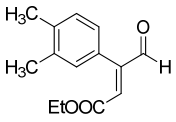
(q, *J* = 7.2 Hz, 2H), 4.06 (q, *J* = 7.2 Hz, 2H), 1.42 (t, *J* = 7.2 Hz, 3H), 1.18 (t, *J* = 7.2Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.3, 165.3, 159.8, 148.8, 135.1, 130.6, 122.9, 114.0, 63.4, 61.1, 14.7, 13.9.



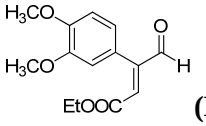
(E)-ethyl 4-oxo-3-(4-pentylphenyl)but-2-enoate (1n) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.75 (s, 1H), 7.22 (d, *J* = 8.4 Hz, 2H), 7.16 (t, *J* = 1.6 Hz, *J* = 6.4 Hz, 2H), 6.66 (s, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 2.64 (t, *J* = 7.6 Hz, *J* = 8 Hz, 2H), 1.67–1.59 (m, 2H), 1.37–1.31 (m, 4H), 1.15 (t, *J* = 7.2 Hz, 3H), 0.91 (t, *J* = 6.8 Hz, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.0, 165.2, 149.4, 144.1, 135.5, 128.9, 128.3, 128.0, 61.2, 35.8, 31.5, 30.9, 22.5, 14.0, 13.8.



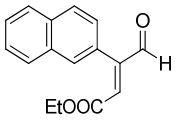
(E)-ethyl 3-(4-cyclohexylphenyl)-4-oxobut-2-enoate (1o) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.76 (s, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.17 (d, *J* = 8 Hz, 2H), 6.66 (s, 1H), 4.16–4.11 (q, *J* = 7.2 Hz, 2H), 2.54–2.49 (m,1H), 1.89–1.83 (m, 4H), 1.76 (d, *J* = 8.4 Hz, 1H), 1.47–1.34 (m, 4H), 1.29–1.24 (m, 1H), 1.13 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.1, 165.3, 149.3, 149.2, 135.5, 129.0, 128.5, 126.5, 61.2, 44.4, 34.3, 26.8, 26.1, 13.8.



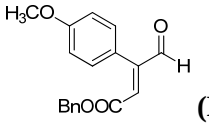
(E)-ethyl 3-(3,4-dimethylphenyl)-4-oxobut-2-enoate (1p) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.73 (s, 1H), 7.16 (d, *J* = 7.6 Hz, 2H), 7.00–6.96 (m, 2H), 6.65 (s, 1H), 4.18–4.12 (q, *J* = 7.2 Hz, 2H), 2.27 (d, 6H), 1.16 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.1, 165.2, 149.5, 137.8, 136.1, 135.3, 129.9, 129.3, 128.6, 126.4, 61.1, 19.7, 19.6, 13.8.



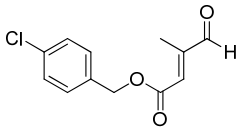
(E)-ethyl 3-(3,4-dimethoxyphenyl)-4-oxobut-2-enoate (1q) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.76 (s, 1H), 6.91–6.81 (m, 3H), 6.66 (s, 1H), 4.20–4.14 (q, *J* = 7.2 Hz, 2H), 3.91 (s, 3H), 3.87 (s, 3H), 1.19 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 193.1, 165.3, 149.8, 148.5, 148.4, 135.4, 123.5, 122.2, 112.5, 110.6, 61.1, 55.8, 13.9.



(E)-ethyl 3-(naphthalen-2-yl)-4-oxobut-2-enoate (1r) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.73 (s, 1H), 7.81–7.77 (m, 3H), 7.70 (s, 1H), 7.45–7.40 (m, 2H), 7.27 (dd, *J* = 1.2 Hz, *J* = 8.4 Hz, 1H), 6.67 (s, 1H), 4.04 (q, *J* = 7.2 Hz, 2H), 0.99 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 192.6, 164.8, 149.0, 136.1, 133.1, 132.5, 128.6, 128.5, 128.1, 127.5, 127.2, 126.5, 126.3, 126.0, 60.9 ,13.5.



(E)-benzyl 3-(4-methoxyphenyl)-4-oxobut-2-enoate (1s) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.71 (s, 1H), 7.31 (t, *J* = 3.2 Hz, 3H), 7.19–7.15 (m, 4H), 6.87–6.84 (m, 2H), 6.65 (s, 1H), 5.12 (s, 2H), 3.80 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ =193.1, 165.1, 160.3, 149.1, 134.9, 134.7, 130.6, 128.4, 128.36, 123.0, 113.5, 66.9, 55.1.



(E)-4-chlorobenzyl 3-methyl-4-oxobut-2-enoate (1x) ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.55 (s, 1H), 7.38–7.82 (m, 4H), 6.54–6.53 (m, 1H), 5.21 (s, 2H), 2.88 (d, *J* = 1.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 194.3, 165.1, 151.2, 134.7, 134.5, 133.7, 129.8, 128.9, 66.0, 10.9.

3. General Procedure for the Synthesis of Chiral N-Heterocyclic Carbene Catalysts

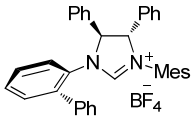
A flame-dried, round-bottom flask equipped with reflux condenser was charged with $Pd(OAc)_2$ (89 mg, 0.4 mmol), *rac*-binap (498 mg, 0.8 mmol), NaO*t*-Bu (769 mg, 8.0 mmol), **C1**(4.0 mmol), and (*S*,*S*)-1,2-diphenyl ethylenediamine (1.3 g, 6.0 mmol) under a dry N_2 atmosphere. Toluene (70 mL) was added to the mixture. After 10 hours at 100 °C, the solution was allowed to cool to room temperature, passed through a plug of Celite, concentrated, and purified by silica gel chromatography (eluent, ethyl acetate/petroleum ether 1/1) to afford productdiamine **C2** as light yellow oil.

Diamine **C2** (3.0 mmol) was added to aflame-dried,round-bottom flask charged with $Pd(OAc)_2$ (67 mg, 0.3mmol), *rac*-binap (373 mg, 0.6 mmol), NaO*t*-Bu (578 mg, 6.0mmol), mesityl bromide (598 mg, 3.0mmol) under a dry N_2 atmosphere. Toluene (70 mL) was added

to the mixture. After 12 hours at 110 °C, the solution was allowed to cool to room temperature and then passed through a plug of Celite, concentrated, and purified by silica gel column chromatography (eluent, petroleum ether/Et_2O 20/1) to afford product **C3**.

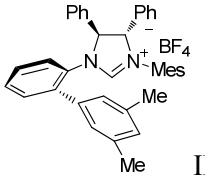
A flame-dried, round-bottom flask equipped with a short path distillation set was charged with diamine **C3** (2.07mmol), $HC(OEt)_3$ (13 mL), and NH_4BF_4 (325 mg, 3.10mmol). After 3 hours at 115 °C, the solution was allowed to cool to room temperature, concentrated, and purified by silicagel column chromatography (eluent, petroleum ether/EtOAc 1/3) to afford the corresponding imidazolinium salt, which was triturated (hexane/CH_2Cl_2) to a white solid.

(4S,5S)-1-([1,1'-biphenyl]-2-yl)-3-mesityl-4,5-diphenyl-4,5-dihydro-1H-imidazol-3-ium (5e)



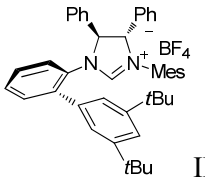
IR(KBr): 703, 759, 1065, 1220, 1261, 1456, 1959, 1618, 1710, 3062, 3432 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.74 (s, 1H), 7.79–7.77 (m, 1H), 7.69–7.67 (m, 3H), 7.49(d, *J* = 3.2 Hz, 2H), 7.37–7.25 (m, 10H), 7.15 (t, *J* = 7.6 Hz, 2H), 6.91(s, 1H),6.67(s, 1H), 6.59(d, *J* = 7.6 Hz, 2H), 5.42(d, *J* = 9.6 Hz, 1H), 5.27(d, *J* = 9.6 Hz, 1H), 2.53(s, 3H), 2.18(s, 3H), 1.81(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.5, 140.1, 138.6, 137.9, 135.2, 134.8, 134.3, 131.7, 131.4, 131.0, 130.4, 130.3, 130.1, 130.0, 129.8, 129.7, 129.6, 129.5, 129.4, 129.1, 128.9, 128.7, 128.6, 128.4, 127.8, 75.1, 72.5, 20.9, 18.7, 17.9. HRMS (ESI): $[M]^+$ calcd for $[C_{36}H_{33}N_2]$: 493.2638, found: 493.2630.

(4S,5S)-1-(3',5'-dimethyl-[1,1'-biphenyl]-2-yl)-3-mesityl-4,5-diphenyl-4,5-dihydro-1H-imidazol-3-ium (5f)



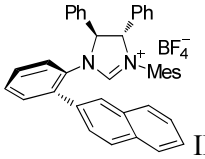
IR(KBr): 701, 1060, 1222, 1266, 1456, 1621, 1959, 2920, 3061, 3438 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.73 (s, 1H), 7.77–7.75 (m, 1H), 7.39–7.09 (m, 13H), 6.91 (s, 1H), 6.66 (s, 1H), 6.60 (d, *J* = 7.6 Hz, 2H), 5.43 (d, *J* = 9. 2 Hz, 1H), 5.36 (d, *J* = 9.2 Hz, 1H), 2.55 (s, 3H), 2.48 (s, 6H), 2.18 (s, 3H), 1.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.2, 140.1, 139.3, 138.5, 138.1, 135.0, 134.9, 134.6, 132.0, 131.3, 131.0, 130.4, 130.1, 130.0, 129.8, 129.6, 129.3, 129.2, 128.9, 128.5, 128.3, 127.7, 126.7, 74.9, 72.3, 21.3, 20.8, 18.6, 17.7. HRMS (ESI): $[M]^+$ calcd for $[C_{38}H_{37}N_2]$: 521.2951, found: 521.2952.

(4S,5S)-1-(3',5'-di-tert-butyl-[1,1'-biphenyl]-2-yl)-3-mesityl-4,5-diphenyl-4,5-dihydro-1H-imidazol-3-ium (5h)



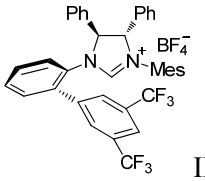
IR(KBr): 697, 1065, 1222, 1268, 1455, 1619, 1959, 2959, 3063, 3437 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.74 (s, 1H), 7.90 (d, *J* = 7.2 Hz, 1H), 7.75 (s, 1H), 7.41–7.13 (m, 16H), 6.91 (s, 1H), 6.63 (s, 1H), 6.47 (d, *J* = 6.4 Hz, 2H), 5.71 (d, *J* = 8.4 Hz, 1H), 5.31 (d, *J* = 8.4 Hz, 1H), 2.45 (s, 3H), 2.19 (s, 3H), 1.65 (s, 3H), 1.40 (s, 18H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.4, 152.0, 140.2, 139.0, 138.1, 135.3, 134.9, 132.0, 131.6, 131.5, 130.4, 130.3, 130.0, 129.7, 129.6, 129.5, 129.1, 128.8, 128.6, 128.3, 127.4, 123.1, 123.0, 75.2, 72.1, 35.1, 31.5, 20.9, 18.5, 18.2. HRMS (ESI): $[M]^+$ calcd for $[C_{44}H_{49}N_2]$: 605.3890, found: 605.3887.

(4S,5S)-3-mesityl-1-(2-(naphthalen-2-yl)phenyl)-4,5-diphenyl-4,5-dihydro-1H-imidazol-3-ium (5i)



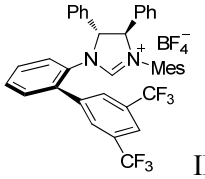
IR(KBr): 702, 784, 1061, 1220, 1266, 1455, 1620, 1959, 2922, 3057, 3437 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.82 (s, 1H), 8.16 (d, *J* = 8.4 Hz, 1H), 8.09 (d, *J* = 7.2 Hz, 1H), 8.02–7.96 (m, 2H), 7.78 (d, *J* = 7.2Hz, 1H), 7.73–7.67 (m, 2H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.37–7.23 (m, 9H), 7.07 (t, *J* = 7.2 Hz, 1H), 6.87 (s, 1H), 6.63 (s, 3H), 6.24 (d, *J* = 7.2 Hz, 2H), 5.33 (d, *J* = 9.2 Hz, 1H), 5.17 (d, *J* = 9.2 Hz, 1H), 2.51 (s, 3H), 2.15 (s, 3H), 1.74 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.4, 140.1, 138.0, 135.7, 135.3, 134.7, 134.5, 133.4, 133.0, 131.7, 131.5, 131.1, 130.3, 130.1, 130.08, 130.0, 129.7, 129.6, 129.5, 129.4, 128.6, 128.3, 128.2, 128.15, 128.08, 128.0, 127.7, 127.5, 127.3, 126.5, 74.9, 72.4, 20.8, 18.6, 17.8. HRMS (ESI): $[M]^+$ calcd for $[C_{40}H_{35}N_2]$: 543.2795, found: 543.2791.

(4S,5S)-1-(3',5'-bis(trifluoromethyl)-[1,1'-biphenyl]-2-yl)-3-mesityl-4,5-diphenyl-4,5-dihydro-1H-imidazol-3-ium (5j)



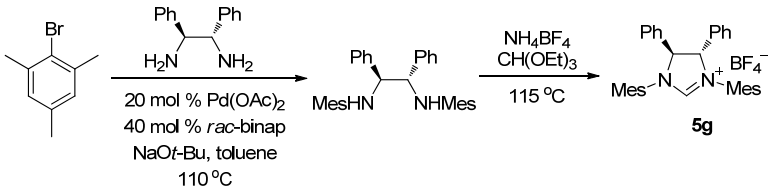
IR(KBr): 701, 1066, 1138, 1281, 1381, 1618, 1959, 2928, 3066, 3437 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.89 (s, 1H), 8.10 (s, 1H), 7.99 (d, *J* = 7.2 Hz, 3H), 7.37–7.33 (m, 4H), 7.32–7.25 (m, 2H), 7.21–7.13 (m, 4H), 7.09–7.06 (m, 2H), 6.93 (s, 1H), 6.73–6.71 (m, 3H), 5.51 (d, *J* = 10.4 Hz, 1H), 5.08 (d, *J* = 10 Hz, 1H), 2.52 (s, 3H), 2.22 (s, 3H), 1.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.4, 141.2, 140.1, 135.7, 134.6, 133.9, 133.4, 133.2, 132.9, 132.5, 132.2, 131.8, 131.7, 131.1, 130.8, 130.6, 130.3, 129.97, 129.95, 129.8, 129.6, 129.5, 129.2, 128.5, 128.2, 127.7, 127.0, 124.3, 122.3, 121.6, 75.6, 73.2, 20.9, 18.7, 17.7. HRMS (ESI): $[M]^+$ calcd for $[C_{38}H_{31}F_6N_2]$: 629.2386, found: 629.2383.

(4R,5R)-1-(3',5'-bis(trifluoromethyl)-[1,1'-biphenyl]-2-yl)-3-mesityl-4,5-diphenyl-4,5-dihydro-1H-imidazol-3-ium (5k)



IR(KBr): 701, 1066, 1138, 1281, 1381, 1618, 1959, 2928, 3066, 3437 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.89 (s, 1H), 8.11 (s, 1H), 7.99–7.97 (m, 3H), 7.36–7.24 (m, 5H), 7.21–7.13 (m, 4H), 7.12–7.06 (m, 2H), 6.93 (s, 1H), 6.74–6.71 (m, 3H), 5.50 (d, *J* = 10.4 Hz, 1H), 5.07 (d, *J* = 10.4 Hz, 1H), 2.52 (s, 3H), 2.22 (s, 3H), 1.87 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.4, 141.2, 140.1, 135.7, 134.6, 133.9, 133.4, 133.2, 132.8, 132.5, 132.2, 131.8, 131.7, 131.0, 130.8, 130.6, 130.3, 129.9, 129.8, 129.6, 129.5, 129.1, 128.5, 128.2, 127.7, 127.0, 124.3, 122.3, 121.6, 118.9, 75.6, 73.2, 20.9, 18.7, 17.7. HRMS (ESI): $[M]^+$ calcd for $[C_{38}H_{31}F_6N_2]$: 629.2386, found: 629.2383.

Representative procedure for synthesis of imidazolinium salt 5g:



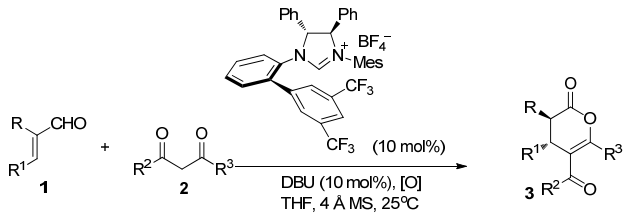
A flame-dried, round-bottom flask equipped with a reflux condenser was charged with $Pd(OAc)_2$ (106 mg, 0.472 mmol), *rac*-binap (587 mg, 0.944 mmol), NaO*t*-Bu (906 mg, 9.44 mmol), mesityl bromide (986 mg, 4.96 mmol), and (*S*,*S*)-1,2-diphenyl ethylenediamine (500 mg, 2.36 mmol) under a dry N_2 atmosphere. Toluene (60 mL) was added to the mixture. After 12 hours at 100 ^oC, the solution was allowed to cool to room temperature, passed through a plug of Celite, concentrated, and purified by silica gel chromatography (eluent, petroleum ether/Et_2O 1/20) to afford product diamine.

A flame-dried, round-bottom flask equipped with a short path distillation set was charged withdiamine (2.2 mmol), $HC(OEt)_3$ (14 mL), and NH_4BF_4 (334 mg, 3.10 mmol). After 3 hours at 115 ^oC, the solution was allowed to cool to room temperature, concentrated, and purified by silicagel column chromatography (eluent, petroleum ether/EtOAc 1/3)to afford the corresponding imidazolinium salt **2**, which was triturated (hexane/CH_2Cl_2) to a white solid.

(4S,5S)-1,3-dimesityl-4,5-diphenyl-4,5-dihydro-1H-imidazol-3-ium (5g) IR (KBr): 700, 759, 854, 1060, 1224, 1267, 1456, 1959, 1620, 2823, 3040, 3438 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 8.53 (s, 1H), 7.37 (d, *J* = 7.6 Hz, 10H), 6.96 (s, 2H), 6.70 (s, 2H), 6.01 (s, 2H), 2.66 (s, 3H), 2.19 (s, 3H), 1.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 157.9, 140.2, 136.0, 134.3, 131.1, 130.6, 130.1, 130.0, 129.4, 128.8, 128.5, 72.8, 20.8, 18.8, 18.1. HRMS (ESI): $[M]^+$ calcd for $[C_{33}H_{35}N_2]$: 459.2800, found: 459.2792.

4. General Procedure and Spectral Data of Products

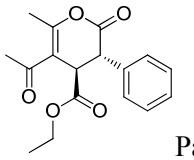
4.1 General Procedure for the synthesis of Fully FunctionalizedDihydropyranones



To a flame dried vessel were successively added NHC (0.01 mmol, 7.16 mg), 4 Å Molecular sieves (40.0 mg), DBU (0.01 mmol, 1.52 mg in 0.25 mL THF), oxidant (0.1 mmol, 40.8 mg), and **2a** (0.3 mmol, 30.3 mg) under N_2 atmosphere. After 10 min, **1a** (0.1 mmol, 20.4 mg in 0.25 mL THF) was added to the mixture. The reaction was stirred at 25^oC for a specified reaction time (monitored by TLC). When the reaction was completed, the solvent was evaporated under reduced pressure and the residue was purified by silica gel flash column chromatography (petroleumether/EtOAc) to give the corresponding dihydropyranones**3a**.

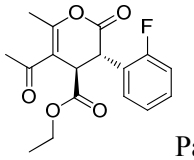
4.2 Analytical Data of Fully Substituted Dihydropyranones

(3S,4R)-ethyl 5-acetyl-6-methyl-2-oxo-3-phenyl-3,4-dihydro-2H-pyran-4-carboxylate (3a)



Pale Yellow Liquid. $[\alpha]_D^{20}$ = 41 (*c* 1.0, CH_2Cl_2, 96% ee); IR(KBr): 702, 1027, 1157, 1261, 1363, 1456, 1601, 1730, 1791, 2371, 2856, 2925, 3275 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.37–7.31 (m, 3H), 7.21–7.19 (m, 2H), 4.38 (d, *J* = 2.4 Hz, 1H), 4.22–4.16 (q, *J* = 7.2 Hz, 2H), 4.12 (d, *J* = 1.6 Hz, 1H), 2.33 (t, 6H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.6, 170.5, 165.3, 160.2, 133.9, 129.2, 128.3, 126.8, 112.6, 62.1, 45.6, 45.5, 30.2, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 23.24 min, minor enantiomer t_R = 27.88 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{17}H_{18}O_5]$: 303.1227, found: 303.1229.

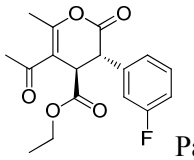
(3S,4R)-ethyl
5-acetyl-3-(2-fluorophenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3b)



Pale Yellow Liquid. $[\alpha]_D^{20}$ = -10 (*c* 1.0, CH_2Cl_2, 91% ee); IR(KBr): 763, 1038, 1114, 1159, 1367, 1457, 1590, 1728, 2375, 2923, 3390 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.36–7.31 (m, 1H), 7.14–7.10 (m, 2H), 7.03–7.00 (m, 1H), 4.55 (d, *J* = 4.4 Hz, 1H), 4.18–4.13 (q, *J* = 7.2 Hz, 2H), 4.04 (d, *J* = 3.6 Hz, 1H), 2.37 (s, 3H), 2.28 (s, 3H), 1.19 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8, 170.5, 164.7, 161.5, 159.8, 159.0, 130.4, 130.3, 128.4, 128.38, 124.8, 124.7, 121.6, 116.3, 116.0, 113.1, 62.1, 45.44, 45.42, 41.21, 41.19, 30.2, 19.2, 13.8. The enantiomeric excess was determined by HPLC with an AD-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; minor enantiomer t_R = 7.32 min, major enantiomer t_R = 8.79 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{17}H_{17}FO_5]$: 321.1133, found: 321.1137.

(3S,4R)-ethyl

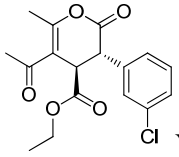
5-acetyl-3-(3-fluorophenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3c)



Pale Yellow Liquid. $[\alpha]_D^{20}$ = 48 (*c* 1.0, CH_2Cl_2,90% ee); IR(KBr): 743, 796, 1023, 1105, 1261, 1377, 1459, 1591, 1731, 2922, 2958, 3409 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.35–7.30 (m, 1H), 7.05–7.00 (m, 2H), 6.96–6.93 (dd, *J* = 2 Hz, *J* = 5.6 Hz, 1H), 4.37 (d, *J* = 2.8 Hz, 1H), 4.22–4.16(q, *J* = 7.2 Hz, 2H), 4.12–4.11 (dd, *J* = 0.8 Hz, *J* = 2.4 Hz, 1H), 2.36 (s, 3H), 2.33 (d, *J* = 0.8 Hz, 3H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.5, 170.3, 164.8, 164.2, 161.8, 160.0, 136.2, 136.1, 130.9, 130.8, 122.64, 122.61, 115.6, 115.4, 114.4, 114.2, 112.8, 62.2, 45.39, 45.38, 45.3, 30.3, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 90:10), 1 mL/min; minor enantiomer t_R = 18.80 min, major enantiomer t_R = 22.91 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{17}H_{17}FO_5]$: 321.1133, found: 321.1134.

(3S,4R)-ethyl

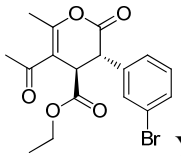
5-acetyl-3-(3-chlorophenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3d)



Yellow Liquid. $[\alpha]_D^{20}$ = 94 (*c* 1.0, CH_2Cl_2, 93% ee); IR(KBr): 796, 1025, 1117, 1260, 1381, 1599, 1731, 1786, 2368, 2963, 3370 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.32–7.29 (m, 2H), 7.22 (s, 1H), 7.09–7.07 (m, 1H), 4.35 (d, *J* = 2.8 Hz, 1H), 4.21–4.16 (q, *J* = 7.2 Hz, 2H), 4.11 (d, *J* = 2 Hz, 1H), 2.37 (s, 3H), 2.34 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.5, 170.3, 164.8, 160.0, 135.7, 135.1, 130.4, 128.7, 127.4, 125.0, 112.8, 62.2, 45.3, 45.2, 30.3, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 95:5), 1 mL/min; minor enantiomer t_R = 28.51 min, major enantiomer t_R = 33.50 min. HRMS (ESI): $[M+Na]^+$ calcd for $[C_{17}H_{17}ClO_5]$: 359.0657, found: 359.0664.

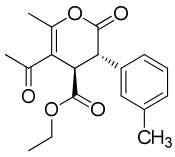
(3S,4R)-ethyl

5-acetyl-3-(3-bromophenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3e)



Yellow Liquid. $[\alpha]_D^{20}$ = 85 (*c* 1.0, CH_2Cl_2, 90% ee); IR(KBr): 691, 790, 1028, 1073, 1120, 1254, 1426, 1595, 1730, 1787, 2924, 3380 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.47 (d, *J* = 8 Hz, 1H), 7.38 (s, 1H), 7.22 (t, *J* = 8 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 4.34 (d, *J* = 2.8 Hz, 1H), 4.21–4.16 (q, *J* = 7.2 Hz, 2H), 4.10 (d, *J* = 2 Hz, 1H), 2.36 (s, 3H), 2.33 (d, *J* = 0.8 Hz, 3H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.5, 170.3, 164.7, 160.0, 136.0, 131.6, 130.7, 130.3, 125.4, 123.2, 112.9, 62.3, 45.33, 45.30, 30.3, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 90:10), 1 mL/min; minor enantiomer t_R = 20.54 min, major enantiomer t_R = 25.35 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{17}H_{17}BrO_5]$: 381.0332, found: 381.0337.

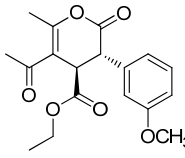
(3S,4R)-ethyl 5-acetyl-6-methyl-2-oxo-3-(m-tolyl)-3,4-dihydro-2H-pyran-4-carboxylate (3f)



Yellow Liquid. $[\alpha]_D^{20}$ = 56 (*c* 1.0, CH_2Cl_2, 94% ee); IR(KBr): 737, 1024, 1155, 1262, 1362, 1462, 1608, 1732, 1788, 2926, 3196cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.22 (t, *J* = 7.6 Hz, 1H), 7.13(d, *J* = 7.2 Hz, 1H), 7.02 (s, 1H), 6.98 (d, *J* = 7.6 Hz, 1H), 4.33 (d, *J* = 2 Hz, 1H), 4.22–4.17 (q, *J* = 7.2 Hz, 2H), 4.10 (s, 1H), 2.33 (d, 9H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.6, 170.6, 165.4, 160.2, 139.0, 133.8, 129.1, 129.0, 127.7, 123.6, 112.6, 62.1, 45.59, 45.58, 30.2, 21.4, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 13.69 min, minor enantiomer t_R = 18.97 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{18}H_{20}O_5]$: 317.1384, found: 317.1389.

(3S,4R)-ethyl

5-acetyl-3-(3-methoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3g)

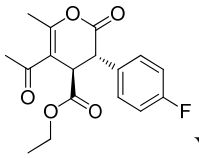


Yellow Liquid. $[\alpha]_D^{20}$ = 38 (*c* 1.0, CH_2Cl_2, 94% ee); IR(KBr): 737, 1044, 1155, 1263, 1362, 1459, 1601, 1730, 2926, 3195 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.26 (t, *J* = 8 Hz, 1H), 6.86–6.83 (dd, *J* = 2.4 Hz, *J* = 4.4 Hz, 1H), 6.78–6.75 (m, 2H), 4.35 (d, *J* =

2 Hz, 1H), 4.23–4.17 (q, *J* = 7.2 Hz, 2H), 4.12 (d, *J* = 1.2 Hz, 1H), 3.78 (s, 3H), 2.35 (d, 6H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7, 170.5, 165.3, 160.2, 160.1, 135.3, 130.2, 129.0, 118.9, 113.6, 112.9, 112.7, 62.2, 55.3, 45.5, 45.4, 30.3, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 17.54 min, minor enantiomer t_R = 21.70 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{18}H_{20}O_6]$: 333.1333, found: 333.1331.

(3S,4R)-ethyl

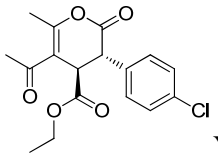
5-acetyl-3-(4-fluorophenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3h)



Yellow Liquid. $[\alpha]_D^{20}$ = 20 (*c* 1.0, CH_2Cl_2, 90% ee); IR(KBr): 778, 1043, 1117, 1158, 1261, 1456, 1594, 1731, 2372, 2924, 3366 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.21–7.17 (dd, *J* = 5.2 Hz, *J* = 8.4 Hz, 2H), 7.04(t, 2H), 4.34 (d, *J* = 2.8 Hz, 1H), 4.21–4.16(q, *J* = 7.2 Hz, 2H), 4.10 (d, *J* = 2 Hz, 1H), 2.36 (s, 3H), 2.33 (s, 3H), 1.23 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.6, 170.4, 165.2, 163.7, 161.3, 159.9, 129.59, 129.56, 128.8, 128.7, 116.3, 116.1, 112.9, 62.2, 45.4, 45.0, 30.3, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 14.31 min, minor enantiomer t_R = 16.48 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{17}H_{17}FO_5]$: 321.1133, found: 321.1138.

(3S,4R)-ethyl

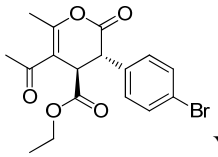
5-acetyl-3-(4-chlorophenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3i)



Yellow Liquid. $[\alpha]_D^{20}$ = 51 (*c* 1.0, CH_2Cl_2, 90% ee); IR(KBr): 741, 798, 1022, 1094, 1261, 1460, 1599, 1732, 2372, 2962, 3374 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.34–7.32 (dd, *J* = 2 Hz, *J* = 6.8 Hz, 2H), 7.04(d, *J* = 8.4 Hz, 2H), 4.34 (d, *J* = 2.8 Hz, 1H), 4.21–4.16 (q, *J* = 7.2 Hz, 2H), 4.11–4.10 (dd, *J* = 0.8 Hz, *J* = 2.8 Hz, 1H), 2.36 (s, 3H), 2.32 (s, 3H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.5, 170.3, 165.0, 159.9, 134.5, 132.3, 129.4, 128.3, 112.9, 62.2, 45.2, 45.1, 30.3, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an AD-H column. (*n*-hexane:*i*-PrOH = 90:10), 1 mL/min; major enantiomer t_R = 14.19 min, minor enantiomer t_R = 17.84 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{17}H_{17}ClO_5]$: 337.0837, found: 337.0842.

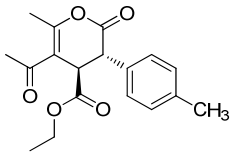
(3S,4R)-ethyl

5-acetyl-3-(4-bromophenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3j)



Yellow Liquid. $[\alpha]_D^{20}$ = 63 (*c* 1.0, CH_2Cl_2, 93% ee); IR(KBr): 739, 814, 1021, 1152, 1261, 1363, 1458, 1732, 1790, 2924, 3308 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.49 (d, *J* = 8.8 Hz, 2H), 7.10(d, *J* = 8 Hz, 2H), 4.32 (d, *J* = 2.8 Hz, 1H), 4.21–4.16 (q, *J* = 7.2 Hz, 2H), 4.11 (d, *J* = 2 Hz, 1H), 2.36 (s, 3H), 2.32 (d,*J* = 0.4 Hz, 3H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.5, 170.3, 164.9, 159.9, 132.8, 132.4, 128.6, 122.5, 112.9, 62.2, 45.2, 45.1, 30.3, 19.4, 13.9.The enantiomeric excess was determined by HPLC with an AD-H column. (*n*-hexane:*i*-PrOH = 95:5), 1 mL/min; major enantiomer t_R = 26.67 min, minor enantiomer t_R = 33.20 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{17}H_{17}BrO_5]$: 381.0332, found: 381.0337.

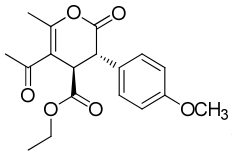
(3S,4R)-ethyl 5-acetyl-6-methyl-2-oxo-3-(p-tolyl)-3,4-dihydro-2H-pyran-4-carboxylate (3k)



Yellow Liquid. $[\alpha]_D^{20}$ = 111 (*c* 1.0, CH_2Cl_2, 95% ee); IR(KBr): 737, 809, 1025, 1117, 1259, 1378, 1459, 1611, 1733, 1787, 2925, 3444 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.15 (d, *J* = 8 Hz, 2H), 7.09 (d, *J* = 8 Hz, 2H), 4.33 (d, *J* = 2.4 Hz, 1H), 4.22–4.16 (q, *J* = 7.2 Hz, 2H), 4.09 (d, *J* = 1.6 Hz, 1H), 2.33 (d, 9H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7, 170.6, 165.5, 160.2, 138.2, 130.8, 129.9, 126.7, 112.6, 62.1, 45.6, 45.3, 30.2, 21.0, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 12.74 min, minor enantiomer t_R = 14.11 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{18}H_{20}O_5]$: 317.1384, found: 317.1381.

(3S,4R)-ethyl

5-acetyl-3-(4-methoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3l)

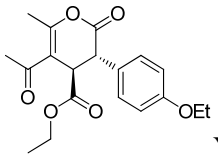


Yellow Liquid. $[\alpha]_D^{20}$ = 133 (*c* 1.0, CH_2Cl_2, 95% ee); IR(KBr): 738, 1036, 1117, 1222, 1362, 1459, 1604, 1714, 2924, 3405 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.13 (d, *J* = 8.8 Hz, 2H), 6.87–6.85 (dd, *J* = 2 Hz, *J* = 6.4 Hz, 2H), 4.31 (d, *J* = 2.4 Hz, 1H),

4.21–4.16 (q, J = 7.2 Hz, 2H), 4.07 (d, J = 1.6 Hz, 1H), 3.79 (s, 3H), 2.34–2.32 (m, 6H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8, 170.6, 165.6, 160.1, 159.5, 128.0, 125.7, 114.6, 112.7, 62.1, 55.3, 45.6, 44.9, 30.2, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 24.64 min, minor enantiomer t_R = 31.60 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{18}H_{20}O_6]$: 333.1333, found: 333.1329.

(3S,4R)-ethyl

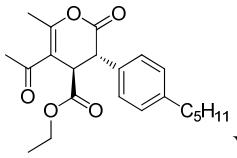
5-acetyl-3-(4-ethoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3m)



Yellow Liquid. $[\alpha]_D^{20}$ = 91 (*c* 1.0, CH_2Cl_2, 98% ee); IR(KBr): 825, 1044, 1149, 1252, 1365, 1513, 1612, 1732, 1788, 2926, 2960 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.11 (d, J = 8.8 Hz, 2H), 6.86 (d, J = 8.8 Hz, 2H), 4.30 (d, J = 2.4 Hz, 1H), 4.21–4.16 (q, J = 7.2 Hz, 2H), 4.07 (d, J = 1.6 Hz, 1H), 4.03–3.98 (q, J = 7.2 Hz, 2H), 2.34 (d, 6H), 1.40 (t, J = 7.2 Hz, 3H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8, 170.7, 165.6, 160.2, 158.8, 128.0, 125.5, 115.1, 112.7, 63.5, 62.1, 45.6, 44.9, 30.2, 19.4, 14.7, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 16.66 min, minor enantiomer t_R = 21.09 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{19}H_{22}O_6]$: 347.1489, found: 347.1493.

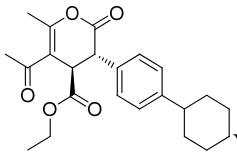
(3S,4R)-ethyl

5-acetyl-6-methyl-2-oxo-3-(4-pentylphenyl)-3,4-dihydro-2H-pyran-4-carboxylate (3n)



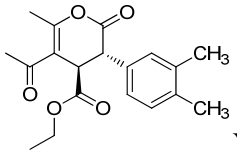
Yellow Liquid. $[\alpha]_D^{20}$ = 44 (*c* 1.0, CH_2Cl_2, 97% ee); IR(KBr): 739, 1039, 1119, 1155, 1261, 1422, 1595, 1731, 2373, 2925, 3346 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.16 (d, J = 8 Hz, 2H), 7.10 (d, J = 8 Hz, 2H), 4.33 (d, J = 2 Hz, 1H), 4.22–4.16 (q, J = 7.2 Hz, 2H), 4.08 (d, J = 1.6 Hz, 1H), 2.57 (t, J = 8 Hz, 2H), 2.32 (m, 6H),1.62–1.55 (m, 2H), 1.37–1.27 (m, 4H), 1.24 (t, J = 7.2 Hz, 3H), 0.88 (t, J = 6.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7, 170.6, 165.5, 160.2, 143.2, 131.0, 129.2, 126.7, 112.6, 62.1, 45.6, 45.3, 35.4, 31.4, 30.9, 30.2, 22.4, 19.4, 13.93, 13.91. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 92:8), 1 mL/min; minor enantiomer t_R = 9.61 min, major enantiomer t_R = 12.01 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{22}H_{28}O_5]$: 373.2010, found: 373.2007.

(3S,4R)-ethyl
5-acetyl-3-(4-cyclohexylphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3o)



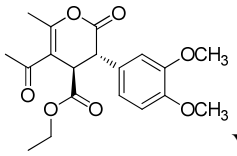
Yellow Liquid. $[\alpha]_D^{20}$ = 190 (*c* 1.0, CH_2Cl_2, 96% ee); IR(KBr): 737, 1057, 1148, 1251, 1362, 1448, 1644, 1733, 1788, 2926, 3373 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.18 (d, *J* = 8.4 Hz, 2H), 7.12(d, *J* = 8.4 Hz, 2H), 4.33 (d, *J* = 2.4 Hz, 1H), 4.21–4.16 (q, *J* = 7.2 Hz, 2H), 4.09 (d, *J* = 1.6 Hz, 1H), 2.50 (d, *J* = 10.4 Hz, 1H), 2.34 (d, 6H),1.84 (d, *J* = 8 Hz, 4H), 1.54 (d, *J* = 8.8 Hz, 1H), 1.42–1.32 (m, 4H), 1.21–1.21 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7, 170.6, 165.5, 160.2, 148.2, 131.1, 127.6, 126.7, 112.5, 62.0, 45.5, 45.3, 44.0, 34.2, 30.2, 26.7, 26.0, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 95:5), 1 mL/min; minor enantiomer t_R = 16.08 min, major enantiomer t_R = 21.30 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{23}H_{28}O_5]$: 385.2010, found: 385.2011.

(3S,4R)-ethyl
5-acetyl-3-(3,4-dimethylphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3p)



Yellow Liquid. $[\alpha]_D^{20}$ = 152 (*c* 1.0, CH_2Cl_2, 96% ee); IR(KBr): 737, 1040, 1148, 1381, 1454, 1609, 1732, 1789, 2375, 2924, 3348 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.09 (d, *J* = 8 Hz, 1H), 6.96 (s, 1H), 6.88 (d, *J* = 1.6 Hz, 1H), 4.30 (d, *J* = 2 Hz, 1H), 4.22–4.17 (q, *J* = 7.2 Hz, 2H), 4.08 (d, *J* = 1.2 Hz, 1H), 2.33 (d, 6H), 2.23 (s, 6H), 1.25 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7, 170.7, 165.6, 160.3, 137.5, 136.8, 131.1, 130.3, 128.1, 123.8, 112.5, 62.1, 45.6, 45.2, 30.2, 19.8, 19.4, 19.3, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 12.81 min, minor enantiomer t_R = 15.79 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{19}H_{22}O_5]$: 331.1540, found: 331.1538.

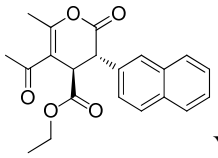
(3S,4R)-ethyl
5-acetyl-3-(3,4-dimethoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3q)



Yellow Liquid. $[\alpha]_D^{20}$ = 114 (*c* 1.0, CH_2Cl_2, 96% ee); IR(KBr): 774, 1043, 1117, 1190, 1421, 1596, 1786, 2375, 2924, 3344 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 6.28 (d, *J* = 8.4 Hz, 1H), 6.74 (t, 2H), 4.32 (d, *J* = 2 Hz, 1H), 4.22–4.17 (q, *J* = 7.2 Hz, 2H), 4.13 (s,

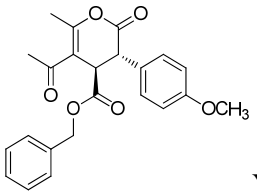
1H), 3.86 (d, 6H), 2.37 (s, 3H), 2.31 (s, 3H), 1.25 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8, 170.6, 165.6, 159.9, 149.4, 149.0, 125.9, 118.9, 113.0, 111.5, 110.3, 62.1, 55.9, 55.88, 45.1, 45.0, 30.3, 19.4, 14.0. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 29.38 min, minor enantiomer t_R = 35.93 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{19}H_{22}O_7]$: 363.1438, found: 363.1442.

(3S,4R)-ethyl
5-acetyl-6-methyl-3-(naphthalen-2-yl)-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3r)



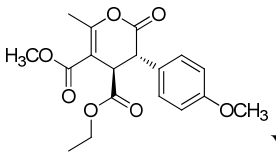
Yellow Liquid. $[\alpha]_D^{20}$ = 92 (*c* 1.0, CH_2Cl_2, 96% ee); IR(KBr): 738, 1038, 1149, 1261, 1362, 1605, 1731, 1787, 2371, 2927, 3298 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.82–7.76 (m, 3H), 7.61 (s, 1H), 7.25–7.48 (m, 2H), 7.36–7.34 (dd, J = 1.6 Hz, J = 6Hz, 1H), 4.54 (d, J = 2.4 Hz, 1H), 4.27 (d, J = 1.6 Hz, 1H), 4.23–4.18 (q, J = 7.2 Hz, 2H), 2.33 (s, 6H), 1.24 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.6, 170.6, 165.4, 160.2, 133.2, 132.8, 131.1, 129.2, 127.9, 127.6, 126.7, 126.6, 125.9, 124.4, 112.8, 62.1, 45.8, 45.4, 30.3, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 40.64 min, minor enantiomer t_R = 63.10 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{21}H_{20}O_5]$: 353.1384, found: 353.1386.

(3S,4R)-benzyl
5-acetyl-3-(4-methoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3s)



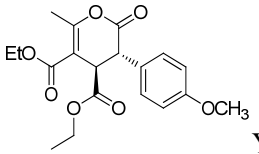
Yellow Liquid. $[\alpha]_D^{20}$ = 115 (*c* 1.0, CH_2Cl_2, 96% ee); IR(KBr): 787, 1042, 1117, 1260, 1422, 1596, 1786, 2370, 2924, 3363 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.39–7.32 (m, 3H), 7.31–7.25 (m, 2H), 7.11 (d, J = 8.4 Hz, 2H), 6.85–6.83 (m, 2H), 5.15 (s, 2H), 4.31 (d, J = 2.8 Hz, 1H), 4.14–4.13 (m, 1H), 3.77 (s, 3H), 2.29 (d, J = 0.8 Hz, 3H), 2.27 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.6, 170.5, 165.5, 160.1, 159.5, 134.9, 128.6, 128.5, 128.2, 128.0, 125.6, 114.6, 112.7, 67.7, 55.2, 45.5, 44.9, 30.2, 19.4. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; minor enantiomer t_R = 27.13 min, major enantiomer t_R = 29.99 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{23}H_{22}O_6]$: 395.1489, found: 395.1494.

(3S,4R)-4-ethyl5-methyl
3-(4-methoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4,5-dicarboxylate (3t)



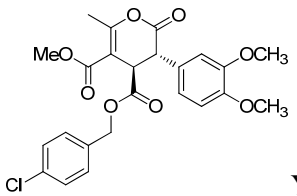
Yellow Liquid. $[\alpha]_D^{20}$ = 108 (*c* 1.0, CH_2Cl_2, 95% ee); IR(KBr): 795, 1032, 1149, 1259, 1379, 1462, 1514, 1655, 1726, 1788, 2956, 3434 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.15 (d, *J* = 8.8 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 4.26 (d, *J* = 2.8 Hz, 1H), 4.19–4.14 (m, 2H), 4.10–4.09 (m, 1H), 3.79 (s, 3H), 3.77 (s, 3H), 2.38 (d, *J* = 0.8 Hz, 3H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.8, 165.9, 165.7, 162.0, 159.4, 128.2, 125.7, 114.5, 104.1, 61.8, 55.2, 52.0, 45.2, 45.1, 18.8, 13.9. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 90:10), 1 mL/min; major enantiomer t_R = 14.51 min, minor enantiomer t_R = 19.87 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{18}H_{20}O_7]$: 349.1282, found: 349.1286.

(3S,4R)-diethyl
3-(4-methoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4,5-dicarboxylate (3u)



Yellow Liquid. $[\alpha]_D^{20}$ = 115 (*c* 1.0, CH_2Cl_2, 96% ee); IR(KBr): 799, 1072, 1260, 1376, 1461, 1611, 1655, 1721, 1787, 2923, 3371 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.16 (d, *J* = 7.6 Hz, 2H), 6.88 (d, *J* = 8.8 Hz, 2H), 4.29–4.10 (m, 6H), 3.79 (s, 3H), 2.38 (d, *J* = 0.8 Hz, 3H), 1.30 (t, *J* = 7.2 Hz, 3H), 1.21 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.9, 165.9, 165.4, 161.7, 159.4, 128.2, 125.7, 114.4, 104.4, 61.8, 61.1, 55.2, 45.3, 45.0, 18.7, 14.1, 13.9. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 90:10), 1 mL/min; major enantiomer t_R = 13.34 min, minor enantiomer t_R = 19.39 min. HRMS (ESI): $[M+H]^+$ calcd for $[C_{19}H_{22}O_7]$: 363.1438, found: 363.1444.

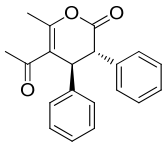
(3S,4R)-4-(4-chlorobenzyl) 5-ethyl
3-(3,4-dimethoxyphenyl)-6-methyl-2-oxo-3,4-dihydro-2H-pyran-4,5-dicarboxylate (3v)



Yellow Liquid. $[\alpha]_D^{20}$ = 69 (*c* 1.0, CH_2Cl_2, 83% ee); IR(KBr): 801, 1076, 1261, 1460, 1597, 1688, 1735, 1786, 2369, 2925, 3371 cm^{-1}; ^1H NMR (400 MHz,

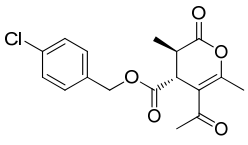
$CDCl_3$): δ = 7.33–7.29 (m, 2H), 7.20 (d, *J* = 8.4 Hz, 2H), 6.80 (d, *J* = 8.4 Hz, 1H), 6.75–6.69 (m, 2H), 5.09 (s, 2H), 4.26 (d, *J* = 3.6 Hz, 1H), 4.12–4.20 (m, 1H), 3.85 (s, 3H), 3.84 (s, 3H), 3.72 (s, 3H), 2.36 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 170.5, 165.7, 165.5, 161.9, 149.1, 148.9, 134.2, 133.5, 129.3, 128.6, 125.5, 119.0, 111.2, 110.2, 104.1, 66.4, 55.72, 55.7, 52.0, 45.2, 44.7, 18.7. The enantiomeric excess was determined by HPLC with an OD-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 34.98 min, minor enantiomer t_R = 54.46 min. HRMS (ESI): $[M+Na]^+$ calcd for $[C_{24}H_{23}Cl_1O_8]$: 497.0974, found: 497.0982.

(3S,4S)-5-acetyl-6-methyl-3,4-diphenyl-3,4-dihydro-2H-pyran-2-one (3w)



Pale Yellow Liquid. $[\alpha]_D^{20}$ = 23 (*c* 1.0, CH_2Cl_2, 71% ee); IR(KBr): 740, 1042, 1140, 1268, 1356, 1621, 1740, 1785, 2375, 2933, 3331 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.38–7.28 (m, 6H), 7.24 (d, *J* = 7.2 Hz, 2H), 7.19 (d, *J* = 7.2 Hz, 2H), 4.35 (s, 1H), 4.10 (d, *J* = 2.4 Hz, 1H), 2.42 (s, 3H), 2.09 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 197.8, 166.2, 159.3, 139.6, 135.5, 129.5, 129.2, 128.2, 128.0, 127.0, 126.9, 115.9, 52.9, 46.4, 30.0, 18.9. The enantiomeric excess was determined by HPLC with an AD-H column. (*n*-hexane:*i*-PrOH = 90:10), 1 mL/min; minor enantiomer t_R = 13.90 min, major enantiomer t_R = 19.88 min. HRMS (ESI): $[M+Na]^+$ calcd for $[C_{20}H_{18}O_3]$: 329.1172, found: 329.1159.

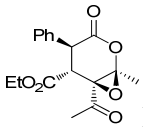
(3R,4R)-4-chlorobenzyl 5-acetyl-3,6-dimethyl-2-oxo-3,4-dihydro-2H-pyran-4-carboxylate (3x)



Pale Yellow Liquid. $[\alpha]_D^{20}$ = 42 (*c* 1.0, CH_2Cl_2, 88% ee); IR(KBr): 738, 1038, 1134, 1270, 1364, 1618, 1752, 1780, 2369, 2938, 3340 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.36–7.32 (m, 2H), 7.25 (d, *J* = 8.8 Hz, 2H), 5.10 (s, 2H), 3.64 (d, *J* = 2.4 Hz, 1H), 3.15–3.09 (qd, *J* = 3.2 Hz, *J* = 4 Hz, 1H), 2.36 (s, 3H), 2.33 (s, 3H), 1.29 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.8, 170.7, 167.7, 160.1, 134.4, 133.4, 129.6, 128.8, 111.9, 66.6, 45.8, 35.3, 30.3, 19.4, 15.3. The enantiomeric excess was determined by HPLC with an AS-H column. (*n*-hexane:*i*-PrOH = 85:15), 1 mL/min; minor enantiomer t_R = 29.03 min, major enantiomer t_R = 34.01 min. HRMS (ESI): $[M+Na]^+$ calcd for $[C_{17}H_{17}ClO_5]$: 359.0657, found: 359.0672.

(1R,4S,5R,6R)-ethyl

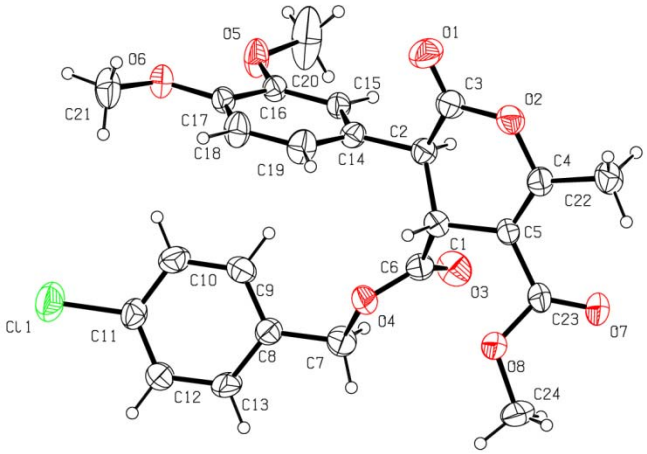
6-acetyl-1-methyl-3-oxo-4-phenyl-2,7-dioxabicyclo[4.1.0]heptane-5-carboxylate (6a)

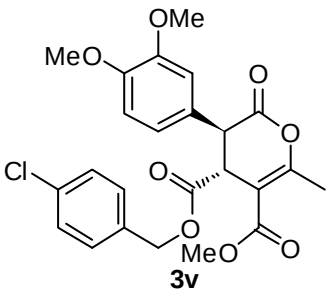


Pale Yellow Liquid. $[\alpha]_D^{20}$ = 39 (*c* 1.0, CH_2Cl_2, 95% ee); IR(KBr): 749, 1028, 1155, 1262, 1383, 1598, 1730, 2373, 2924, 3364 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.37–7.29 (m, 3H), 7.21 (d, *J* = 6.8 Hz, 2H), 4.37 (d, *J* = 2.4 Hz, 1H), 4.22–4.17 (q, *J* = 7.2 Hz, 2H), 4.11 (d, *J* = 1.2 Hz, 1H), 2.33 (d, 6H), 1.24 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ = 196.7, 170.6, 165.4, 160.2, 133.9, 129.2, 129.15, 128.4, 126.9, 112.7, 62.2, 45.7, 45.6, 30.2, 19.4, 13.9. The enantiomeric excess was determined by HPLC with an OJ-H column. (*n*-hexane:*i*-PrOH = 80:20), 1 mL/min; major enantiomer t_R = 15.36 min, minor enantiomer t_R = 17.89 min. HRMS (ESI): $[M+Na]^+$ calcd for $[C_{17}H_{18}O_6]$: 341.0996, found: 341.0995.

5. X-ray Crystallographic Data

X-ray Crystallographic Data of Compound 4d





3v

CCDC 1037041

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	alpha=113.081(12)	beta=96.350(11)	gamma=102.204(14)
Temperature:	292 K		

	Calculated	Reported
Volume	1158.1(3)	1158.1(3)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C24 H23 Cl O8	C24 H23 Cl O8
Sum formula	C24 H23 Cl O8	C24 H23 Cl O8
Mr	474.87	474.87
Dx,g cm-3	1.362	1.362
Z	2	2
Mu (mm-1)	0.212	0.212
F000	496.0	496.0
F000'	496.58	
h,k,lmax	11,14,14	11,14,14
Nref	4569	4553
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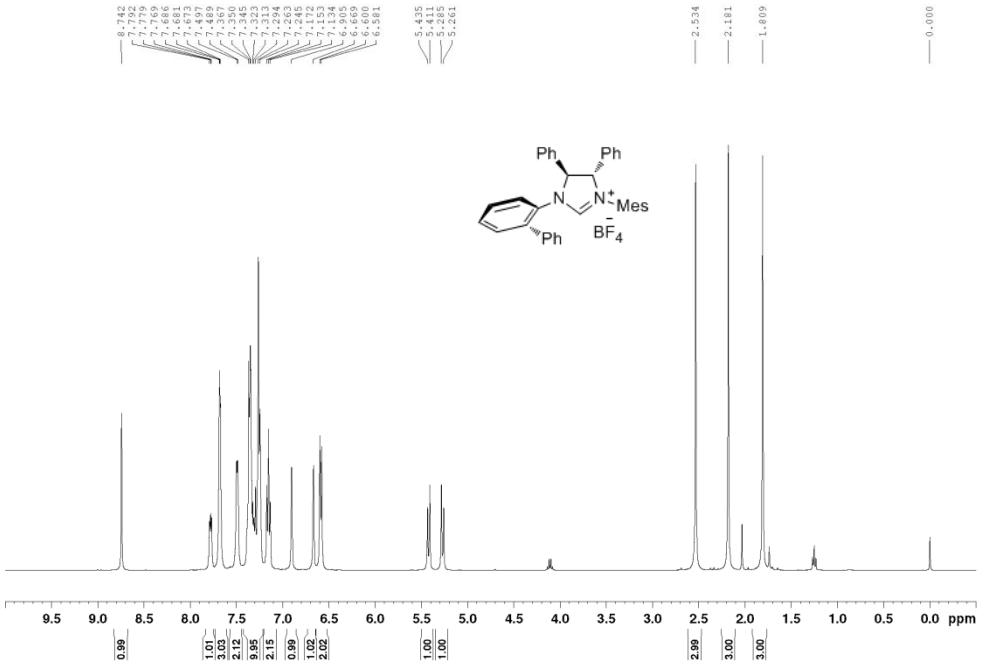
Correction method= MULTI-SCAN

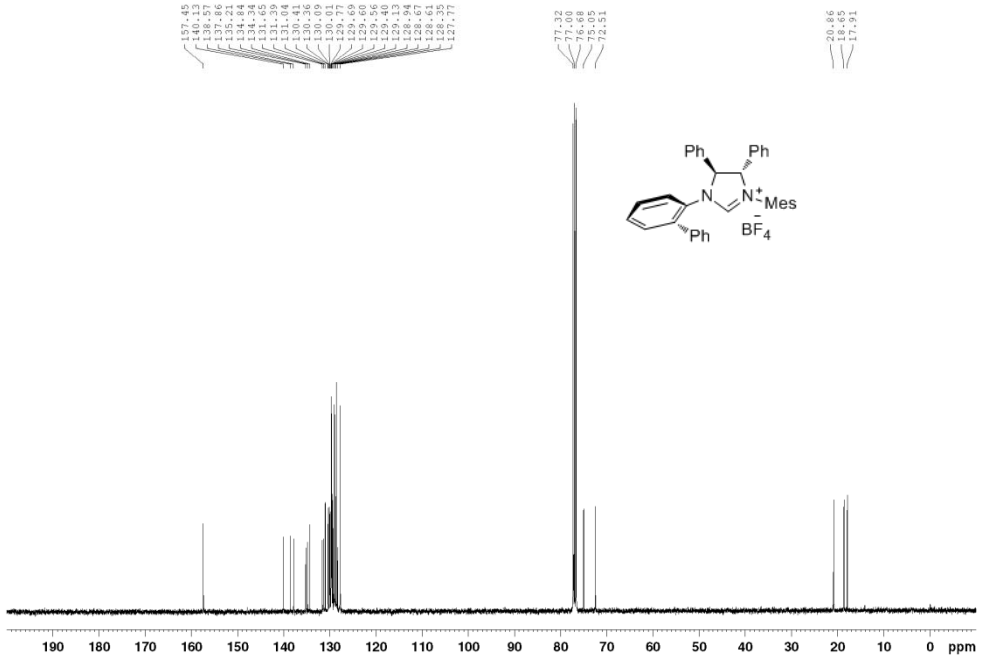
Data completeness= 0.996 Theta(max)= 26.020

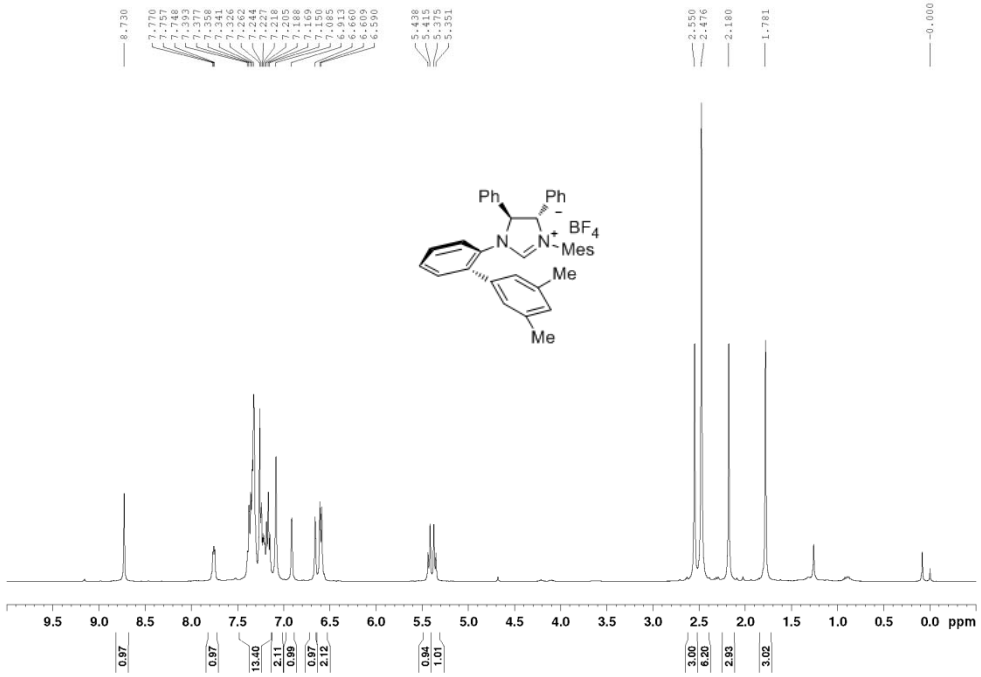
R(reflections)= 0.0652(2049) wR2(reflections)= 0.1818(4553)

S = 1.030 Npar= 302

6. NMR and HPLC spectrogram



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Ph
BF4
N
N+
Mes
Me
Me
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
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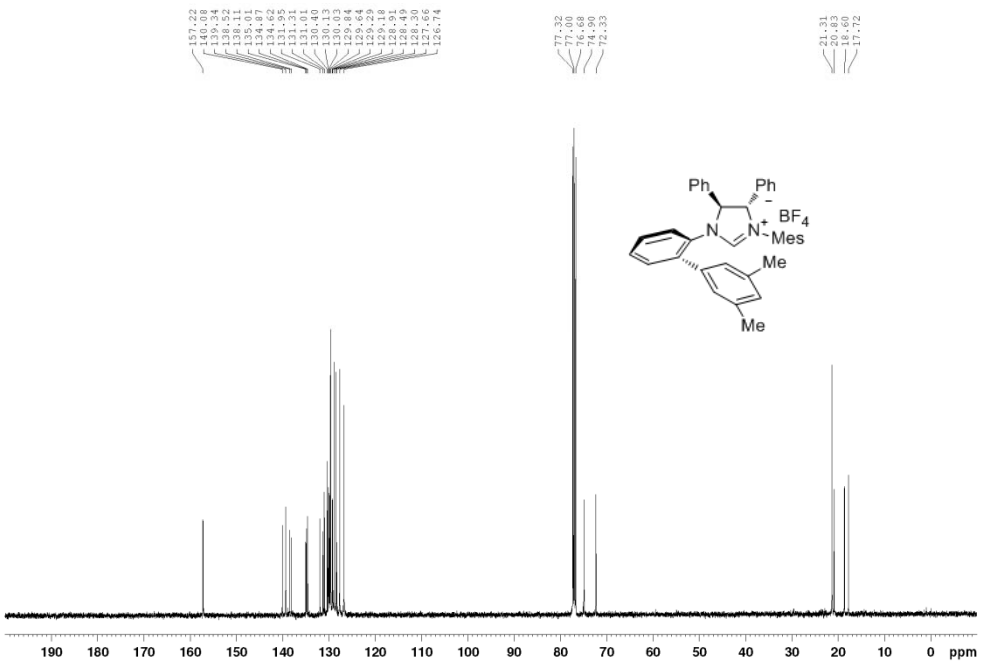
6.20
2.93
3.02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20.83
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Me
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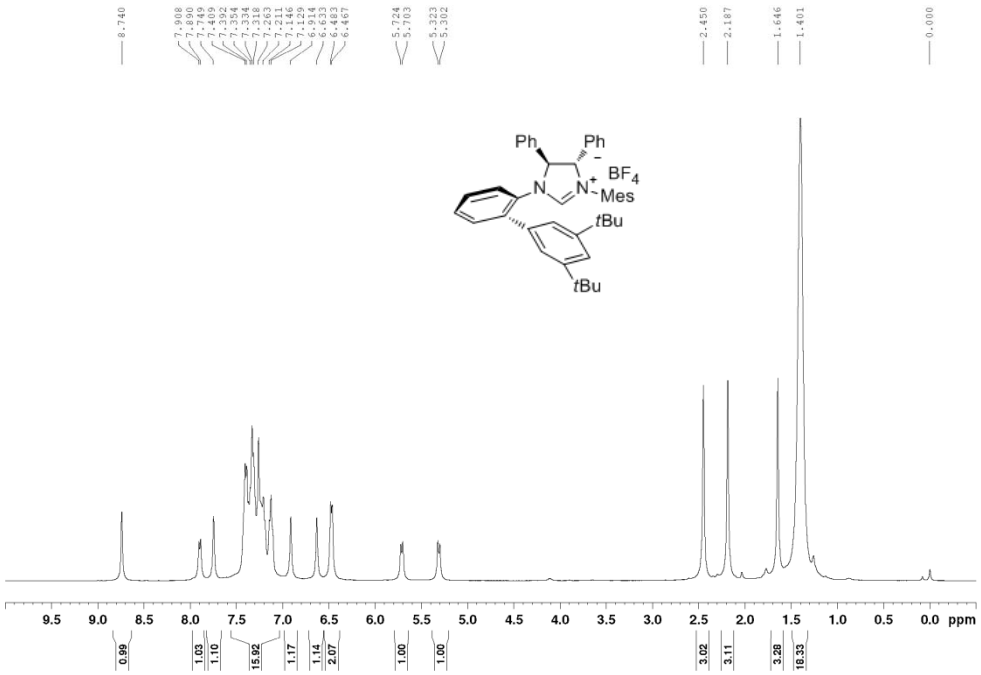
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6.467
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5.703
5.323
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Ph
BF4
Mes
tBu
tBu
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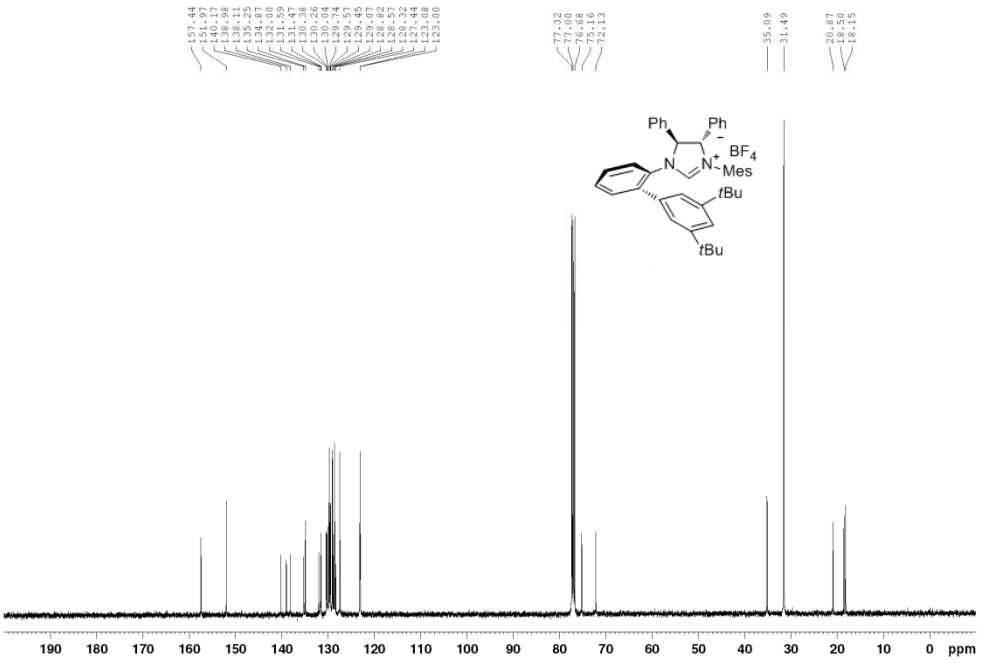
18.33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123.00
77.32
77.00
76.68
75.16
72.13
35.09
31.49
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Ph
Ph
BF4
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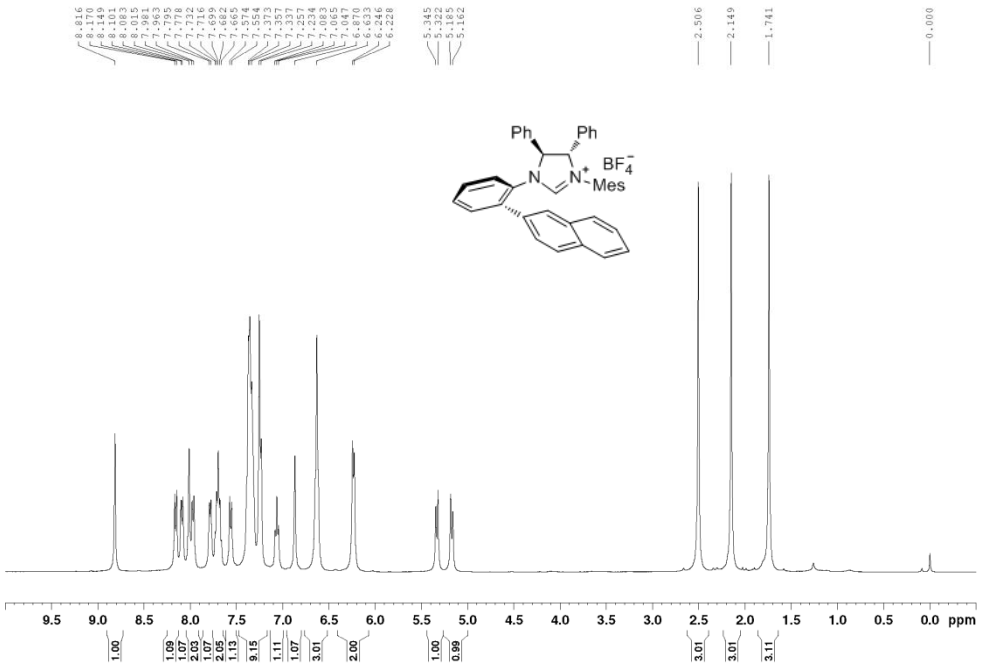
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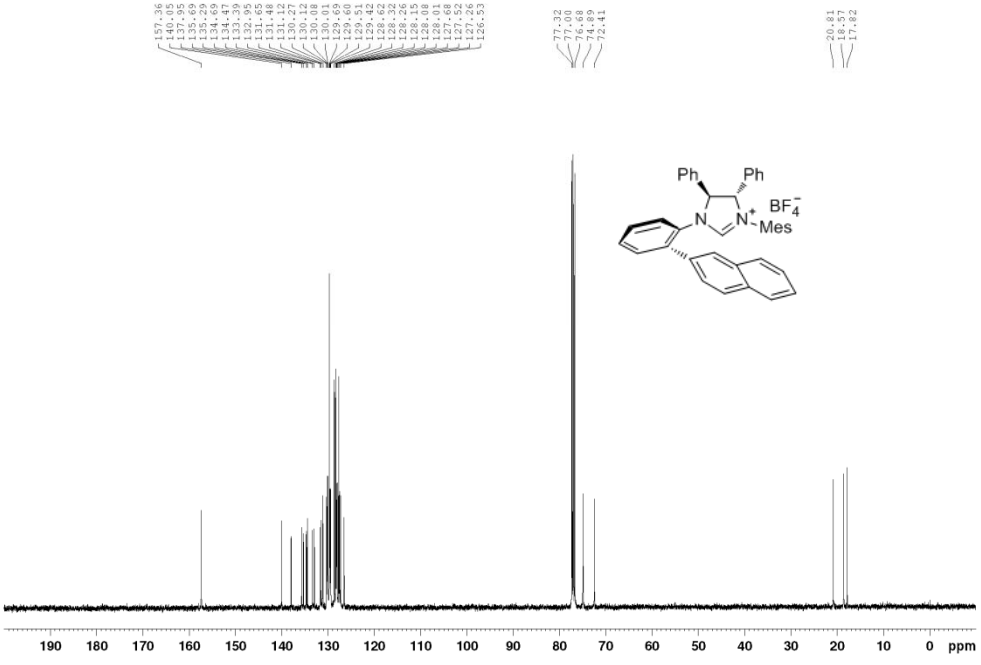
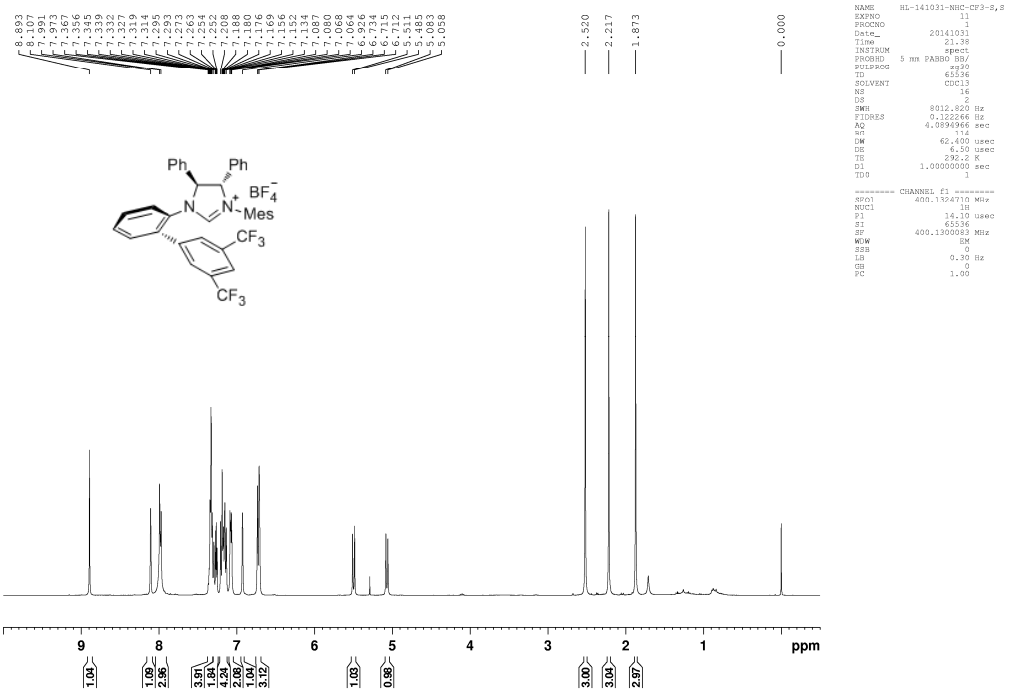
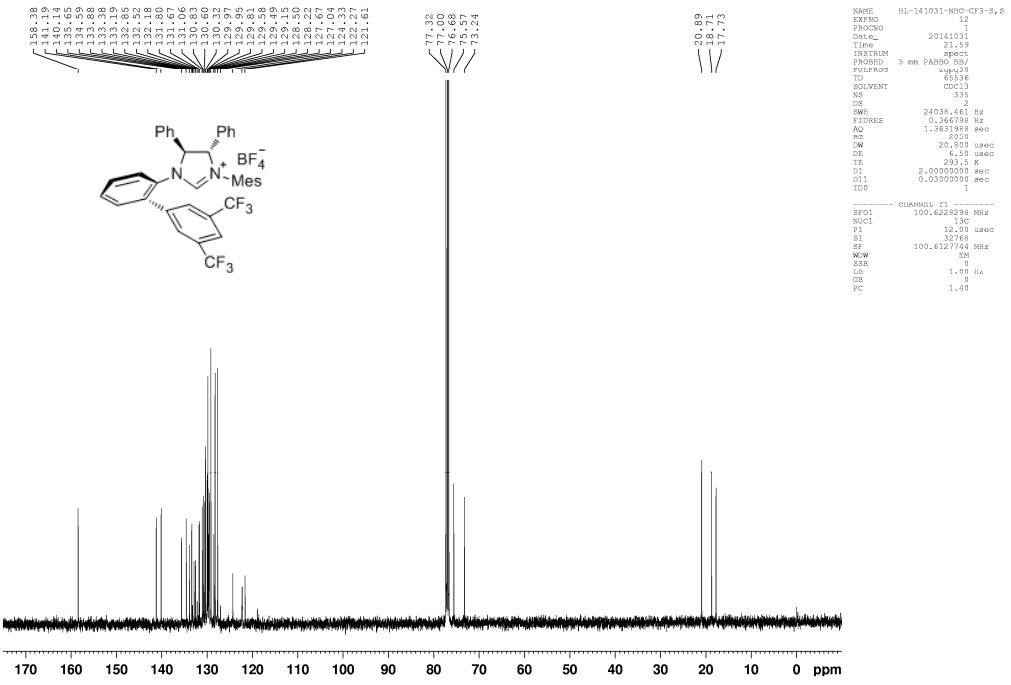
Ph
Ph
BF4
N
N+
Mes
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
1.09
1.07
2.03
1.07
2.05
1.13
9.15
1.11
1.07
3.01
2.00
1.00
0.99
3.01
3.01
3.11

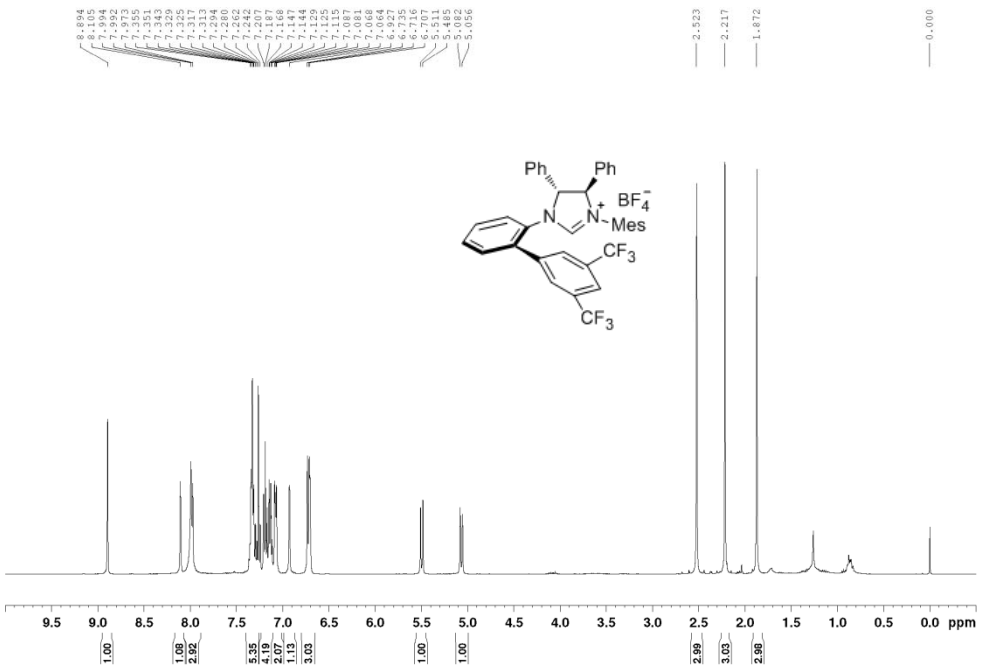

157.36
140.05
137.95
135.69
135.29
134.69
134.47
133.39
132.95
131.65
131.48
131.12
130.27
130.12
130.08
130.01
129.69
129.60
129.51
129.42
128.62
128.32
128.26
128.15
128.08
128.01
127.68
127.52
127.26
126.53
77.32
77.00
76.68
74.89
72.41
20.81
18.57
17.82
Ph
Ph
BF4
N
N+
Mes
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

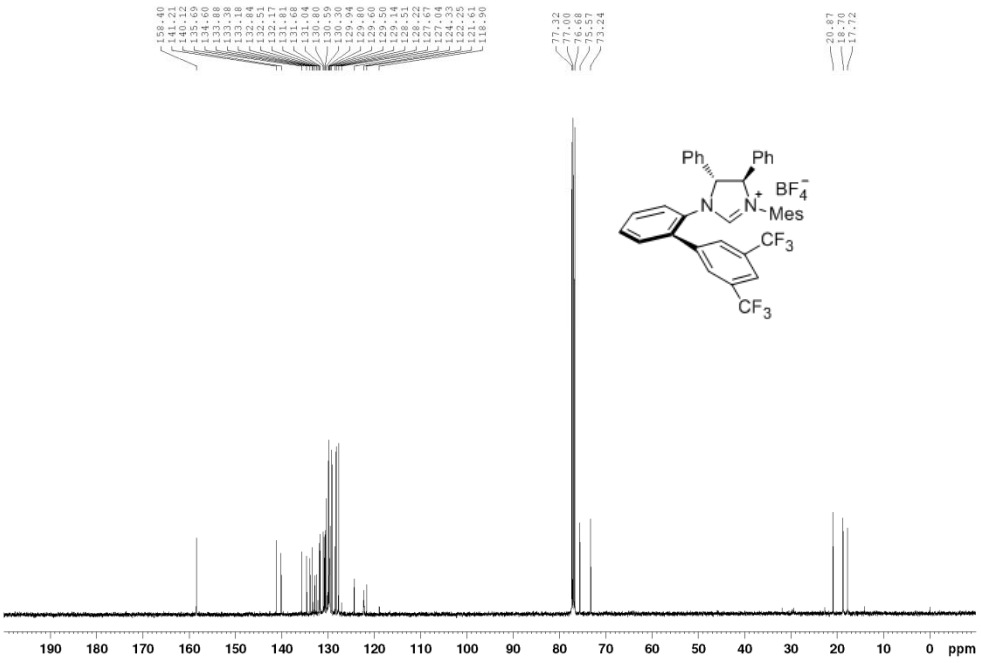


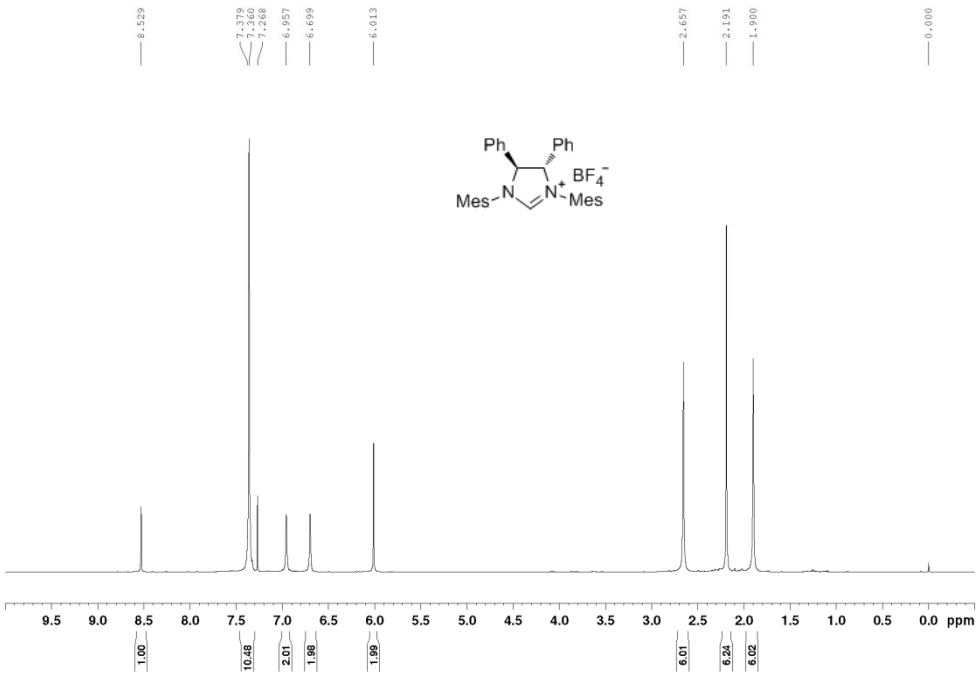

```
NAME        HL-141031-NHC-CF3-S,S
EXPNO                 11
PROCNO                 1
Date_           20141031
Time                21.38
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                   114
DW                62.400 usec
DE                 6.50 usec
TE                292.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 14.10 usec
SI                 65536
SF            400.1300083 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

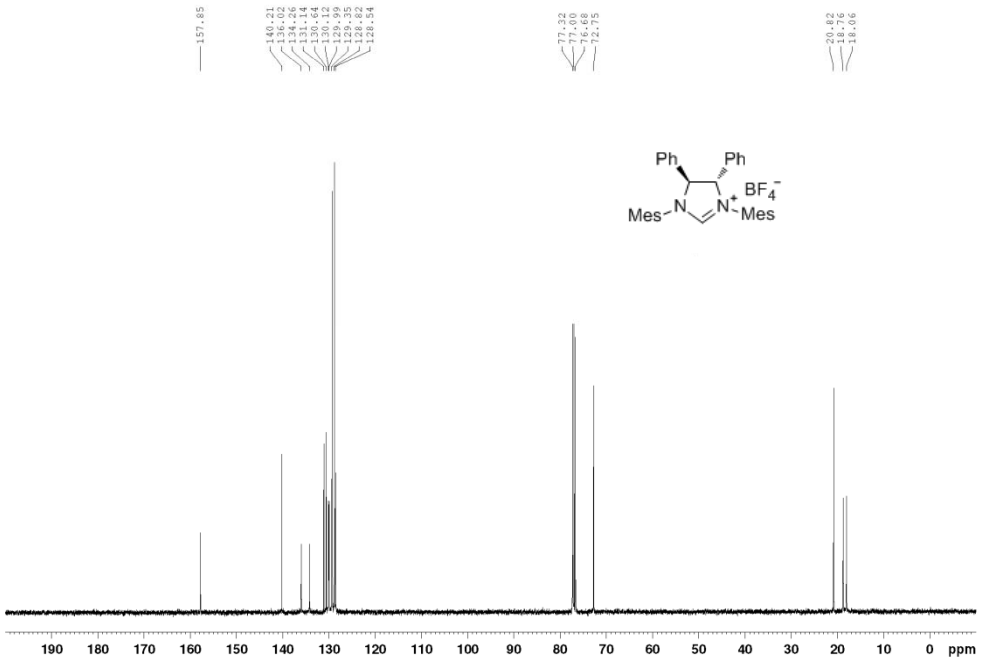


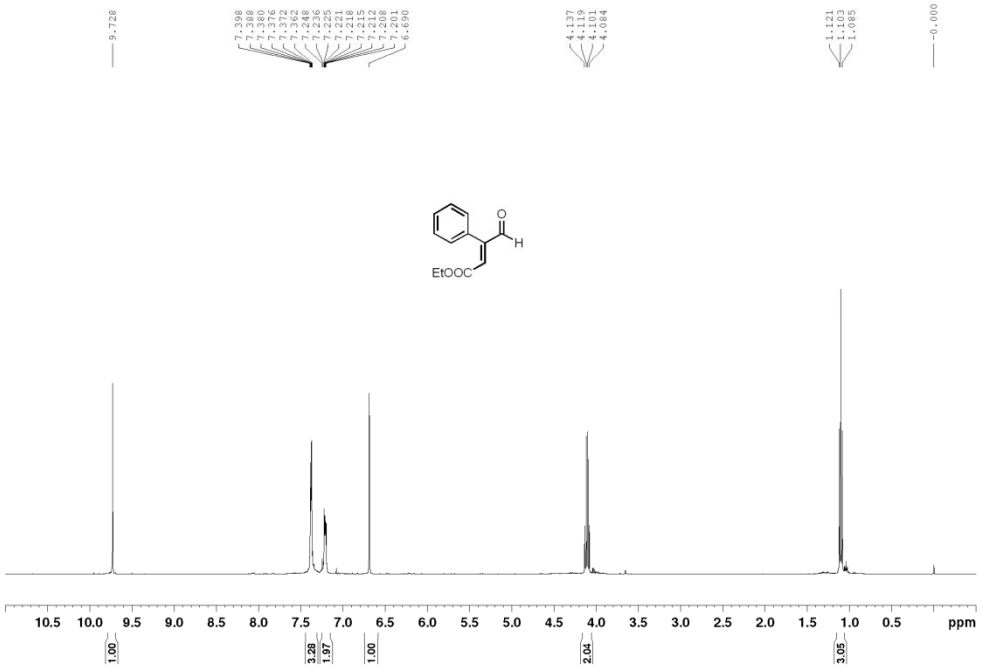

```
NAME        HL-141031-NHC-CF3-S,S
EXPNO                 12
PROCNO                 1
Date_           20141031
Time                21.59
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   335
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE                293.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

--------- CHANNEL f1 ---------
SFO1          100.6228298 MHz
NUC1                  13C
P1                 12.00 usec
SI                 32768
SF            100.6127744 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

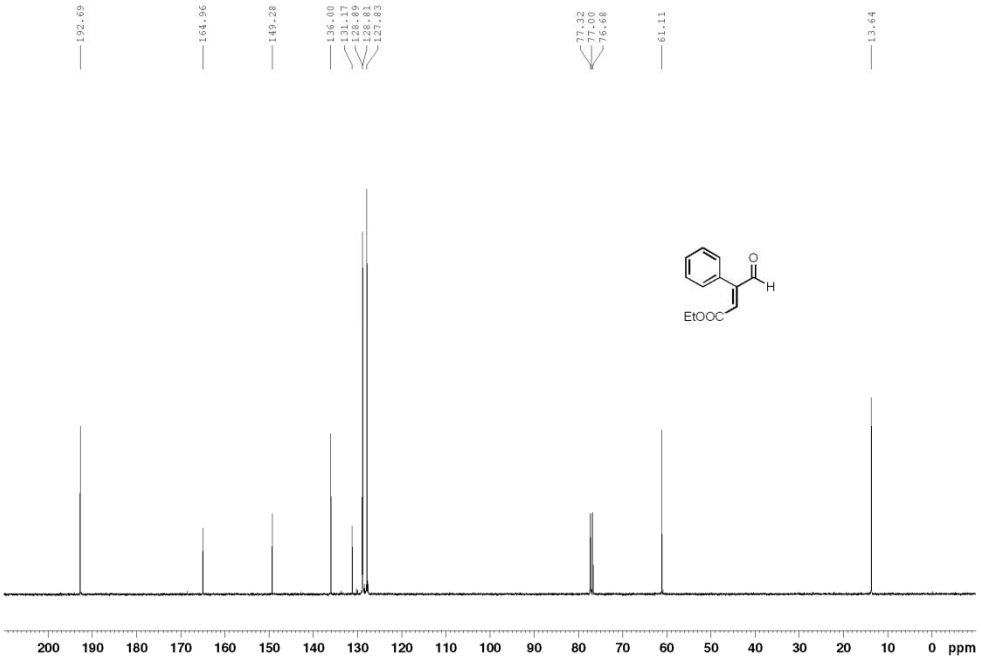

Ph
Ph
BF4−
N
N+
Mes
CF3
CF3
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

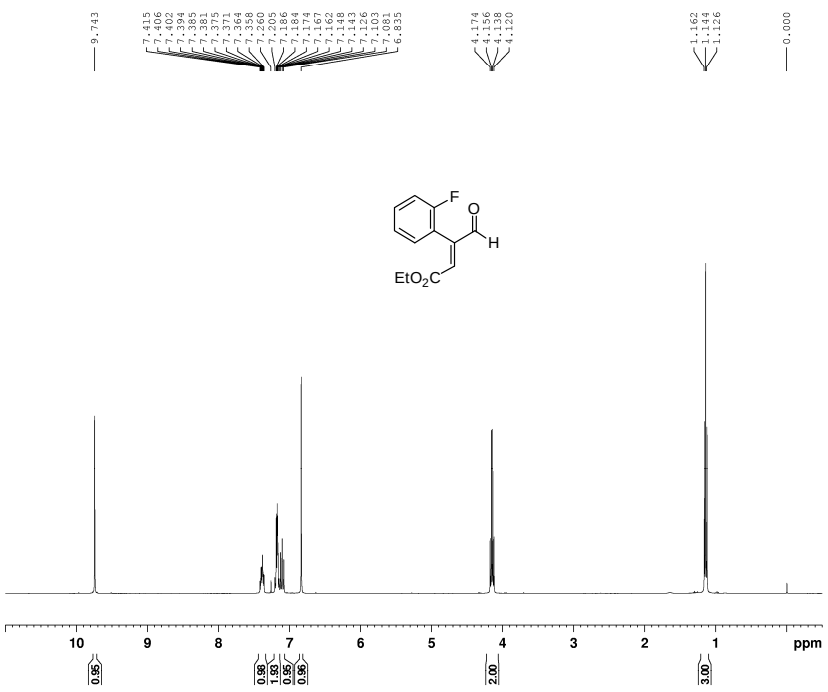

Ph
Ph
BF4−
N
N+
Mes
CF3
CF3
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.529
7.379
7.360
7.268
6.957
6.699
6.013
2.657
2.191
1.900
0.000
Ph
Ph
BF4−
Mes
N
N+
Mes
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
1.00
10.48
2.01
1.98
1.99
6.01
6.24
6.02

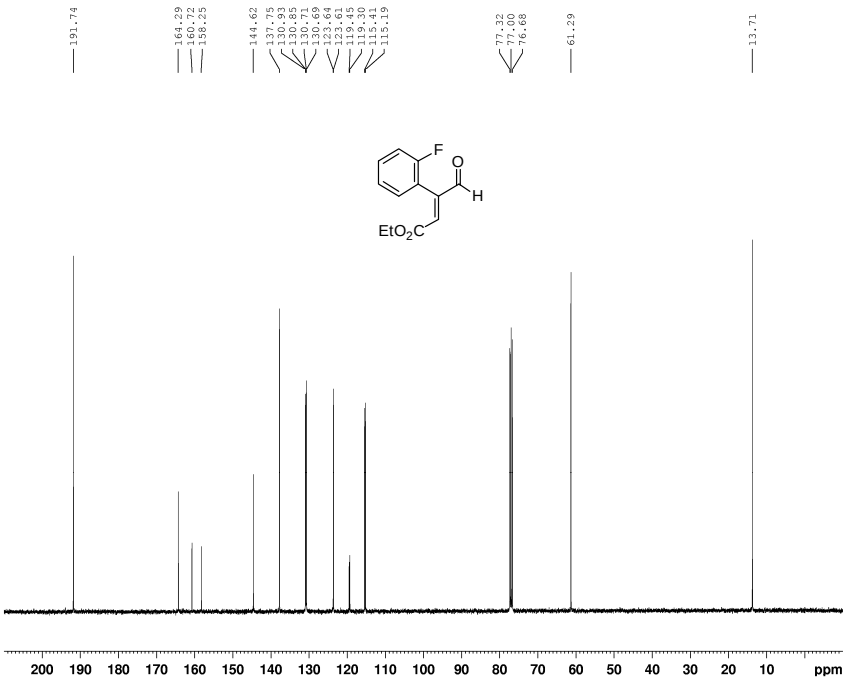

157.85
140.21
136.02
134.26
131.14
130.64
130.02
129.99
129.35
128.82
128.54
77.32
77.00
76.68
72.75
20.82
18.76
18.06
Ph
Ph
BF4−
Mes
N
N+
Mes
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


9.728
7.398
7.388
7.380
7.376
7.372
7.362
7.248
7.236
7.225
7.221
7.218
7.215
7.212
7.208
7.201
6.690
4.137
4.119
4.101
4.084
1.121
1.103
1.085
0.000
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
3.20
1.97
1.00
2.04
3.05

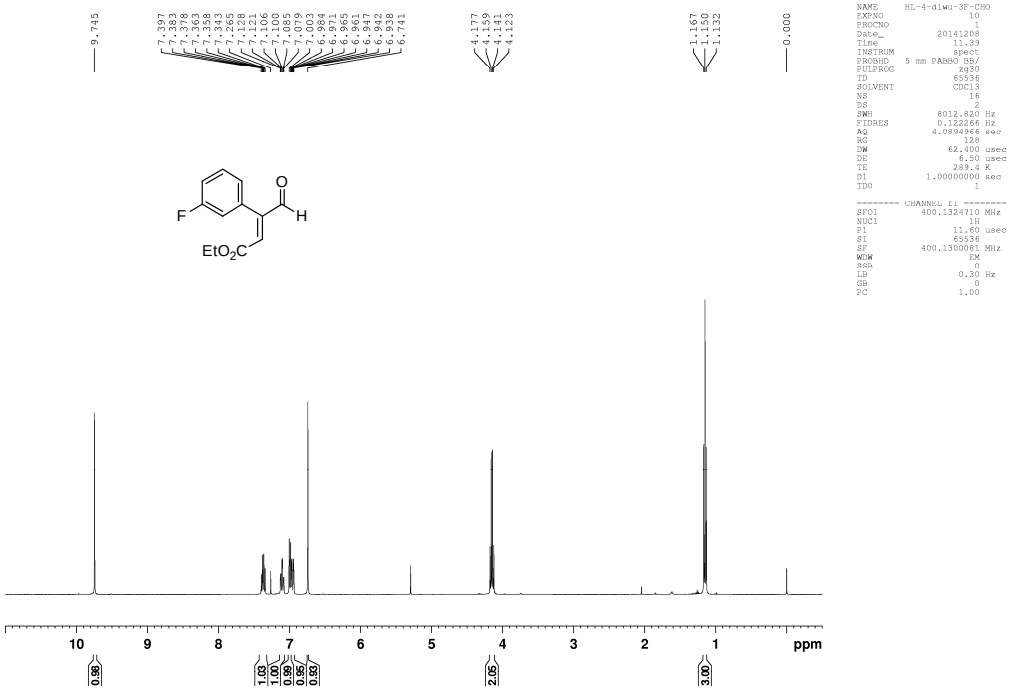

192.69
164.96
149.28
136.00
131.17
128.89
128.31
127.83
77.32
77.00
76.68
61.11
13.64
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



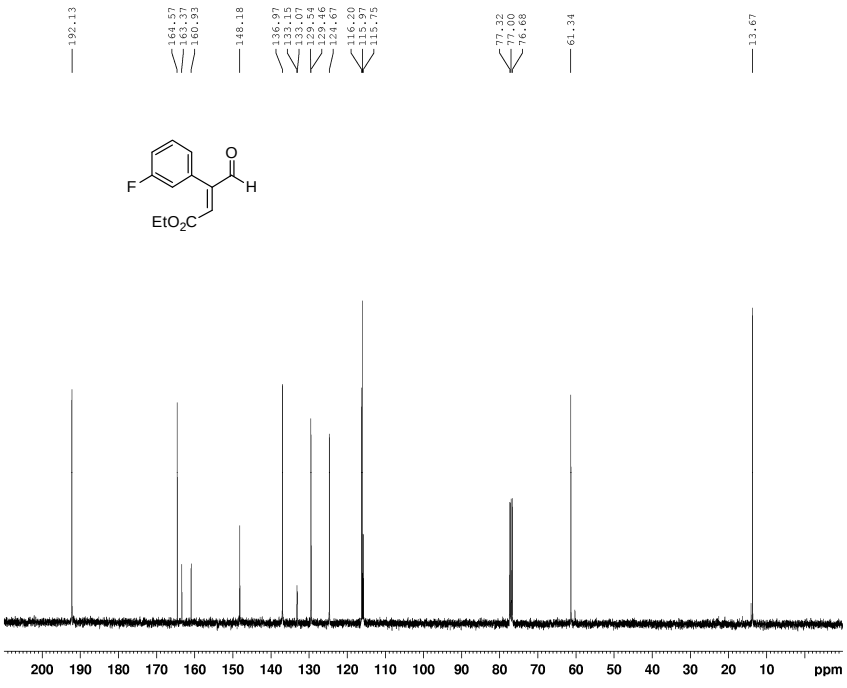

```
NAME        HL-diwu-ph-2-F
EXPNO               11
PROCNO               1
Date_         20140403
Time             12.27
INSTRUM          spect
PROBHD    5 mm PABBO BB/
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG                80.6
DW              62.400 usec
DE               6.50 usec
TE              292.8 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                11.60 usec
SI                65536
SF          400.1300094 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



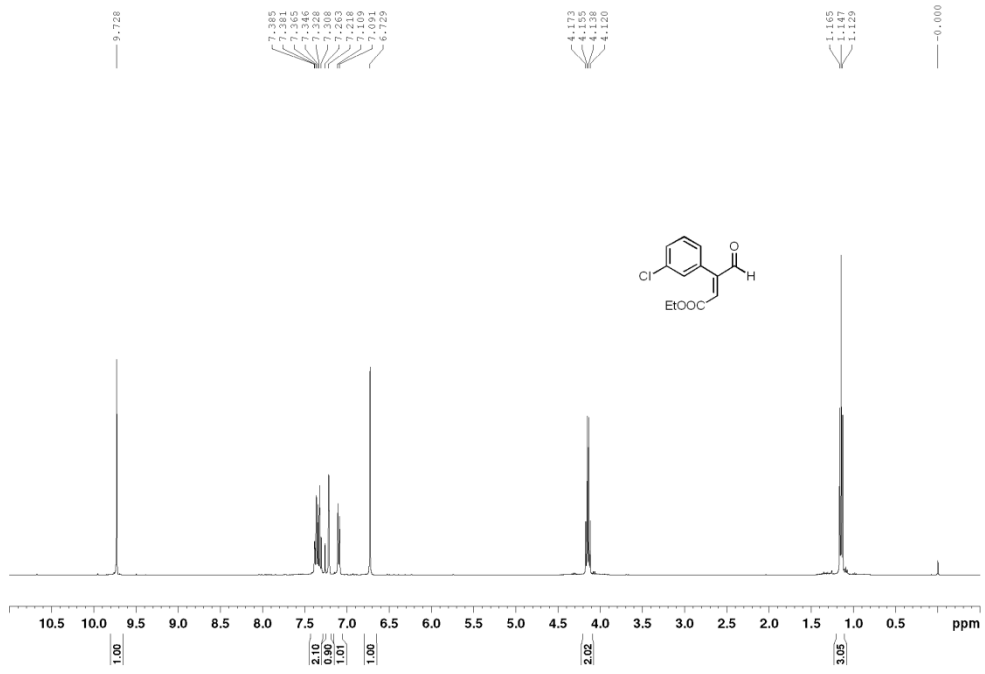

```
NAME        HL-diwu-ph-2-F
EXPNO               12
PROCNO               1
Date_         20140403
Time             12.51
INSTRUM          spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD              65536
SOLVENT          CDCl3
NS                 400
DS                   2
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.2631900 sec
RG                812
DW              20.800 usec
DE               6.50 usec
TE              294.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                14.45 usec
SI                32768
SF          100.6127780 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

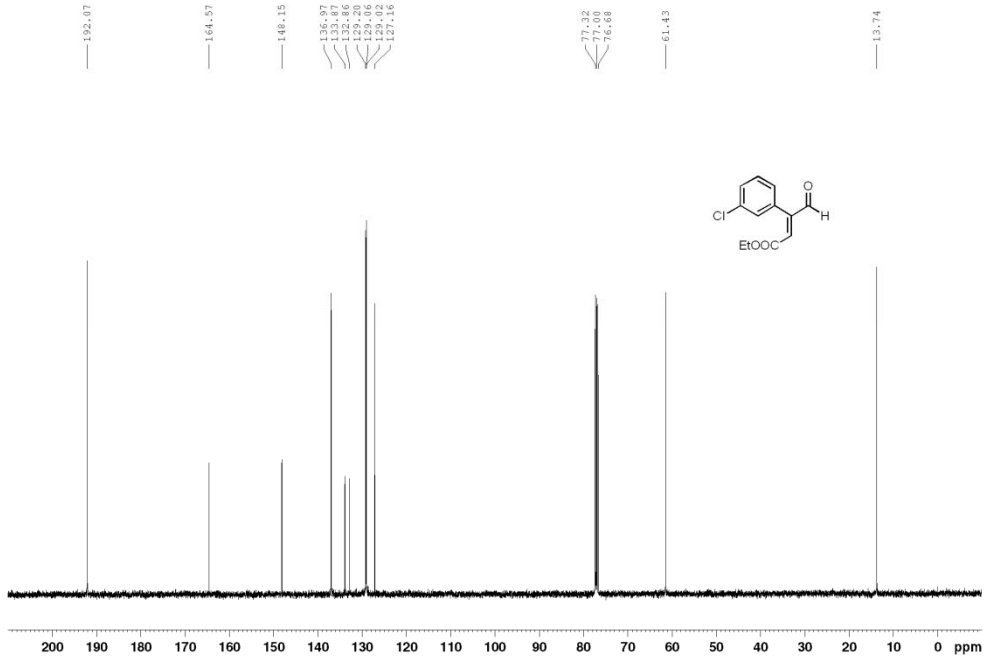


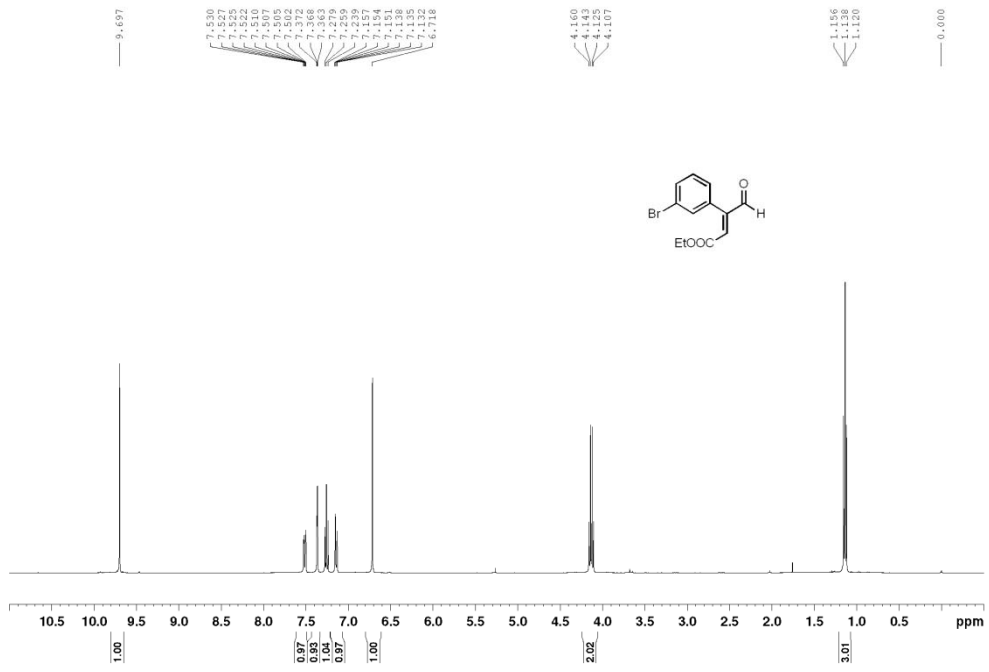

```
NAME        HL-4-diwu-3F-CHO
EXPNO                 10
PROCNO                 1
Date_          20141208
Time               11.39
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                   128
DW                62.400 usec
DE                 6.50 usec
TE                289.4 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 11.60 usec
SI                 65536
SF            400.1300081 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```

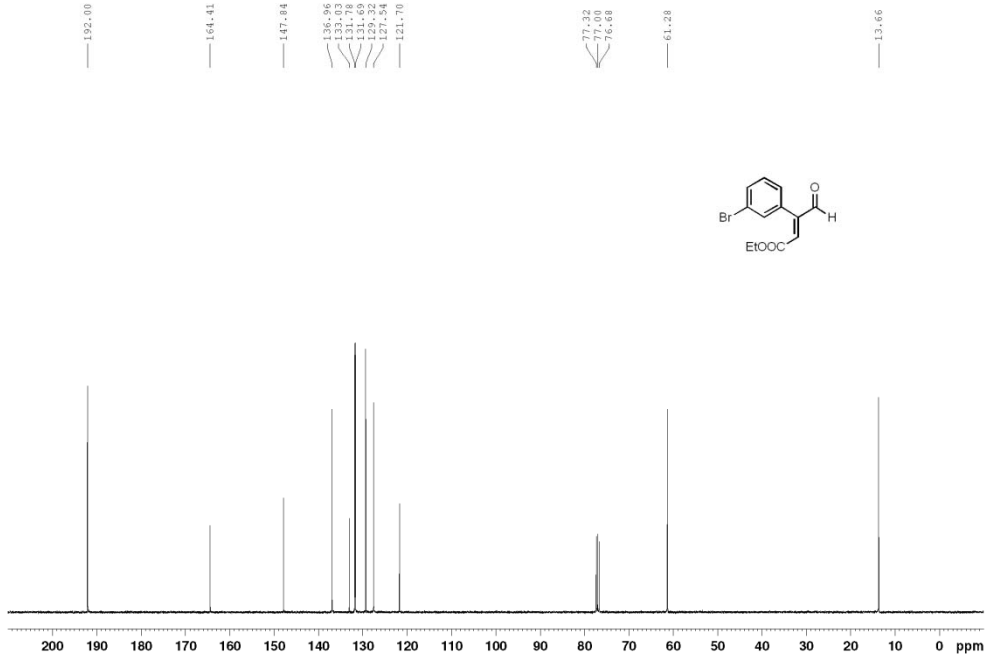


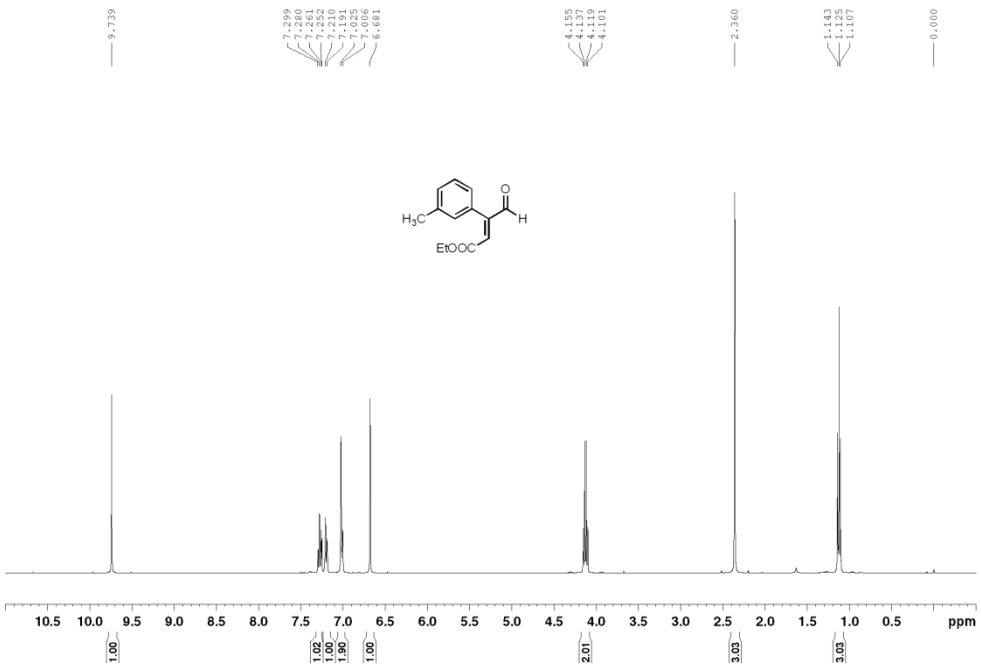

```
NAME        HL-4-diwu-3F
EXPNO                 11
PROCNO                 1
Date_          20141205
Time               21.47
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   150
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                   575
DW                20.800 usec
DE                 6.50 usec
TE                292.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 12.00 usec
SI                 32768
SF            100.6127803 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

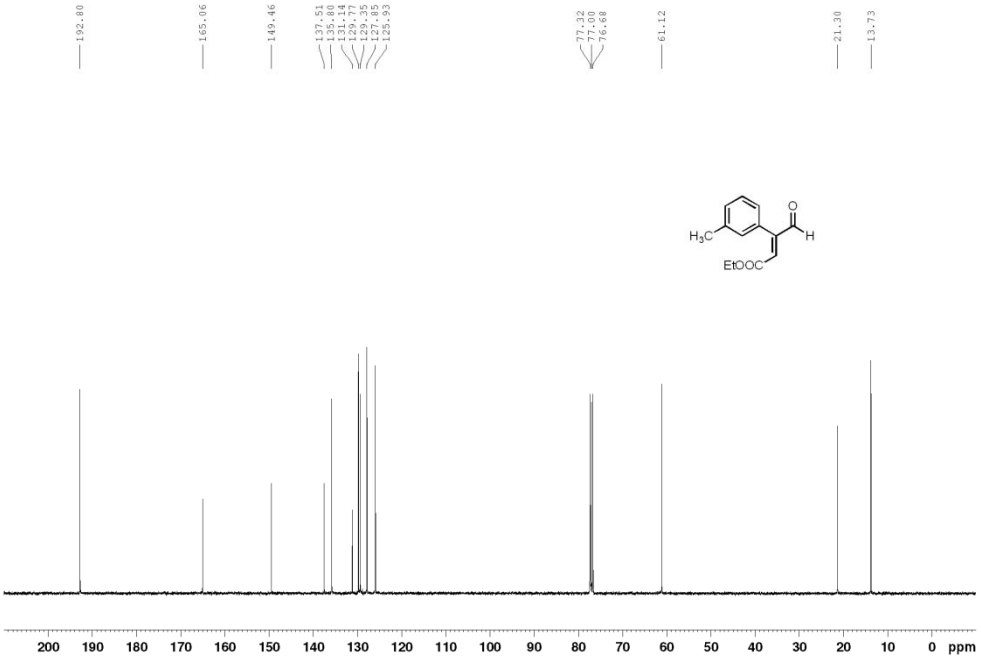

9.728
7.385
7.381
7.365
7.346
7.308
7.300
7.263
7.218
7.109
7.091
6.729
4.173
4.155
4.138
4.120
1.165
1.147
1.129
-0.000
Cl
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
2.10
0.90
1.01
1.00
2.02
3.05

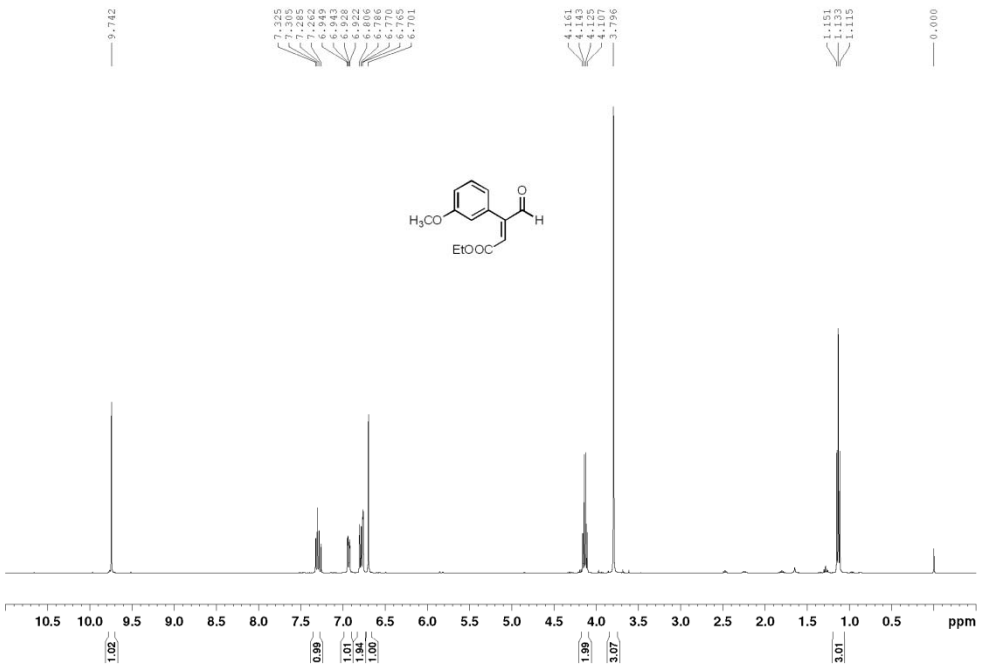

192.07
164.57
148.15
136.97
133.87
132.86
129.80
129.06
129.02
127.16
77.32
77.00
76.68
61.43
13.74
Cl
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

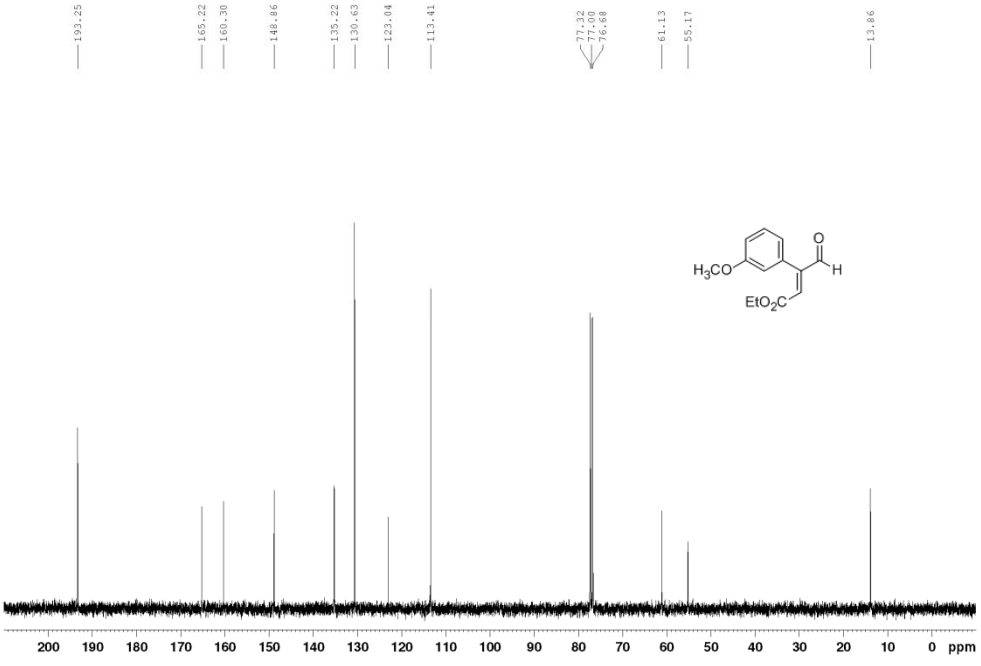

9.697
7.530
7.527
7.525
7.522
7.510
7.507
7.505
7.503
7.372
7.368
7.363
7.279
7.278
7.239
7.157
7.154
7.151
7.138
7.135
7.132
6.718
4.160
4.143
4.125
4.107
1.156
1.138
1.120
0.000
Br
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
0.97
0.93
1.04
0.97
1.00
2.02
3.01

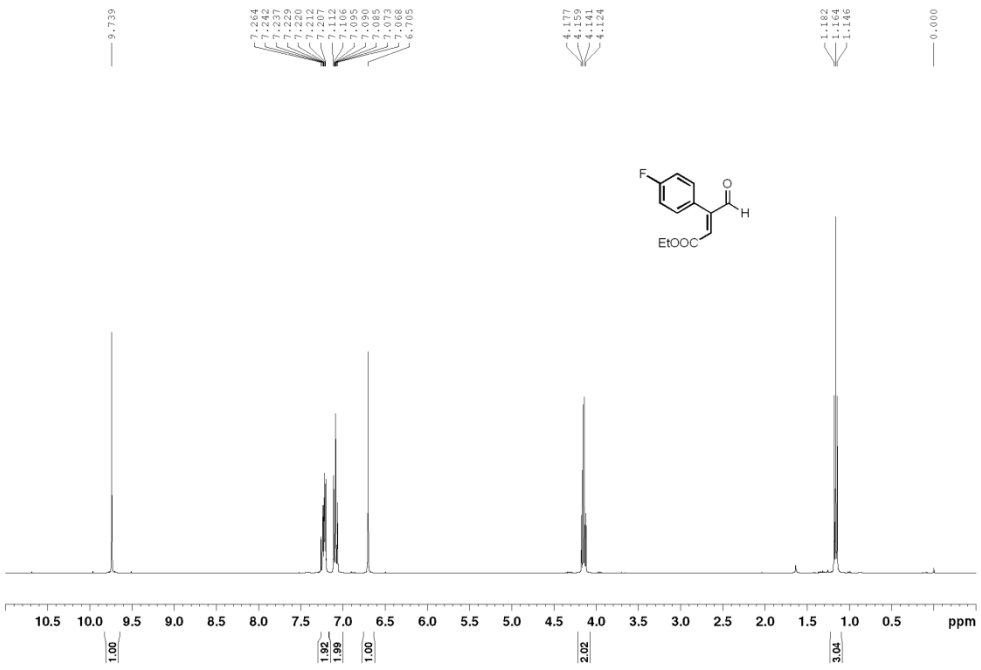

192.00
164.41
147.84
136.96
133.03
131.78
131.69
129.22
127.54
121.70
77.32
77.00
76.68
61.28
13.66
Br
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

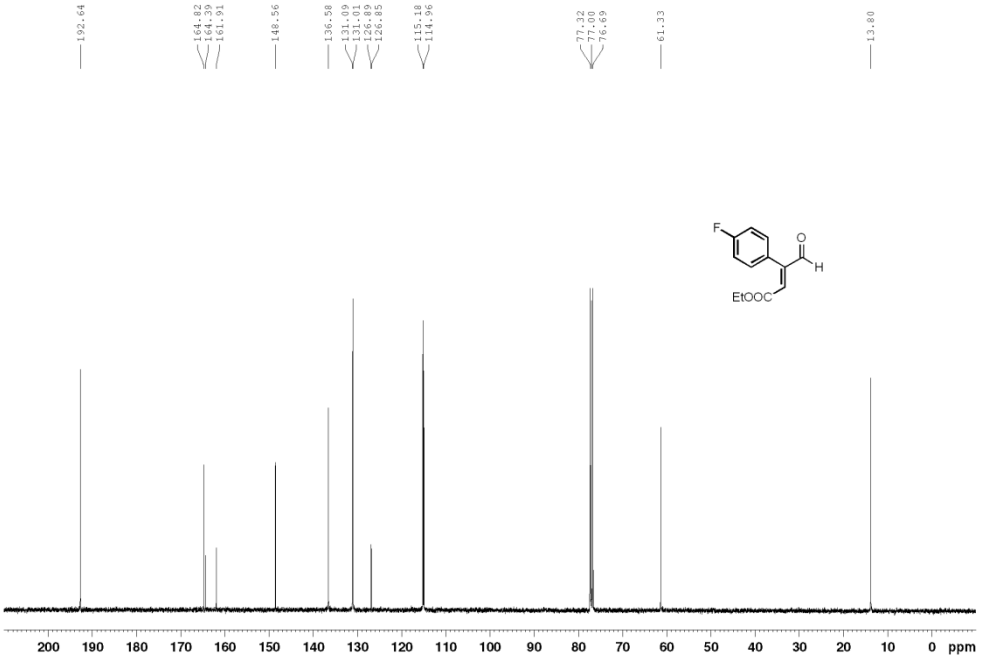

9.739
7.299
7.290
7.261
7.252
7.210
7.191
7.085
7.066
6.681
4.155
4.137
4.119
4.101
2.360
1.143
1.125
1.107
0.000
H3C
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.02
1.00
1.90
1.00
2.01
3.03
3.03

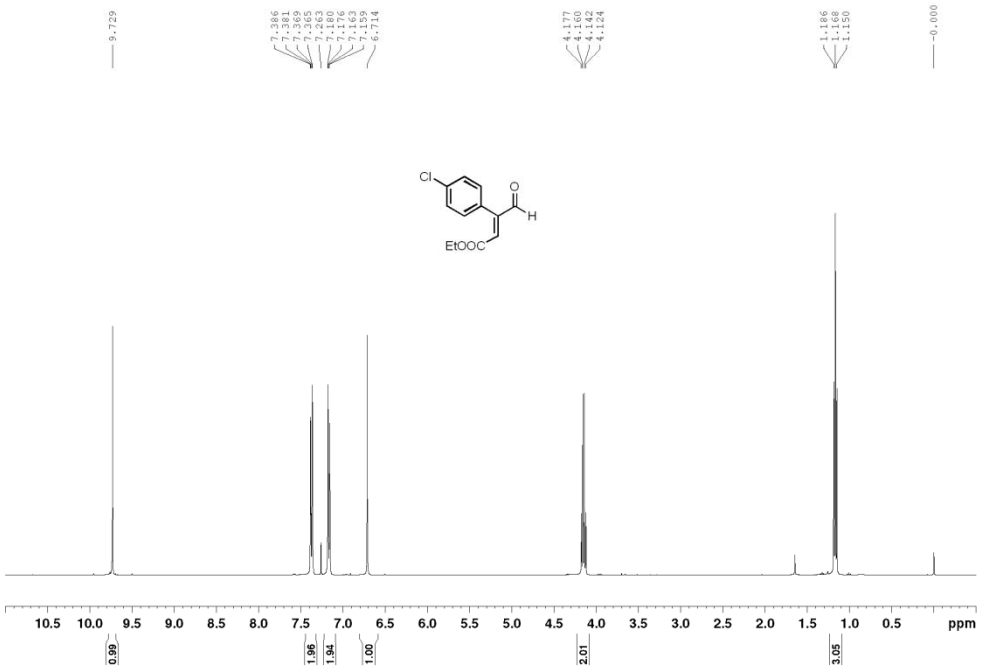

192.80
165.06
149.46
137.51
135.80
131.14
129.77
129.35
127.85
125.93
77.32
77.00
76.68
61.12
21.30
13.73
H3C
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

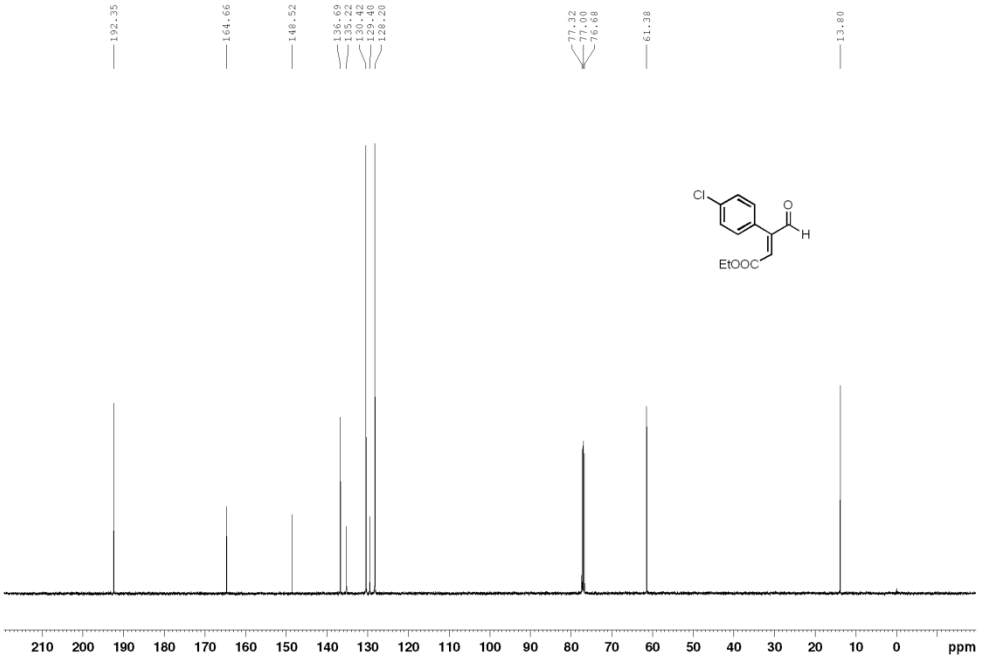

9.742
7.325
7.305
7.285
7.262
6.949
6.943
6.928
6.922
6.806
6.786
6.770
6.765
6.701
4.161
4.143
4.125
4.107
3.796
1.151
1.133
1.115
0.000
H3CO
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.02
0.99
1.01
1.94
1.00
1.99
3.07
3.01

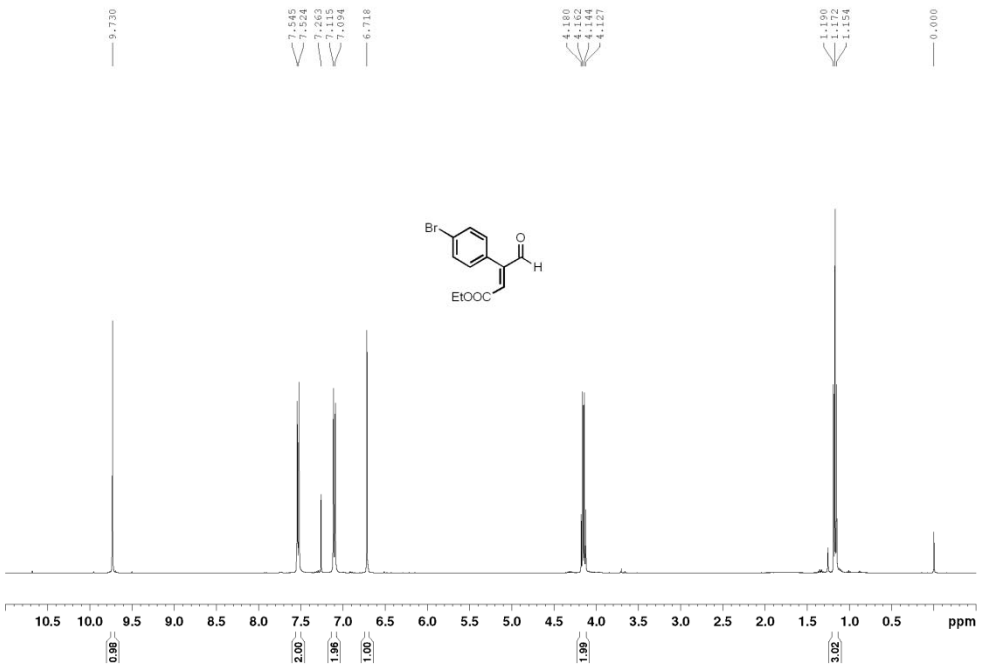

193.25
165.22
160.30
148.86
135.22
130.63
123.04
113.41
77.32
77.00
76.68
61.13
55.17
13.86
H3CO
O
H
EtO2C
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

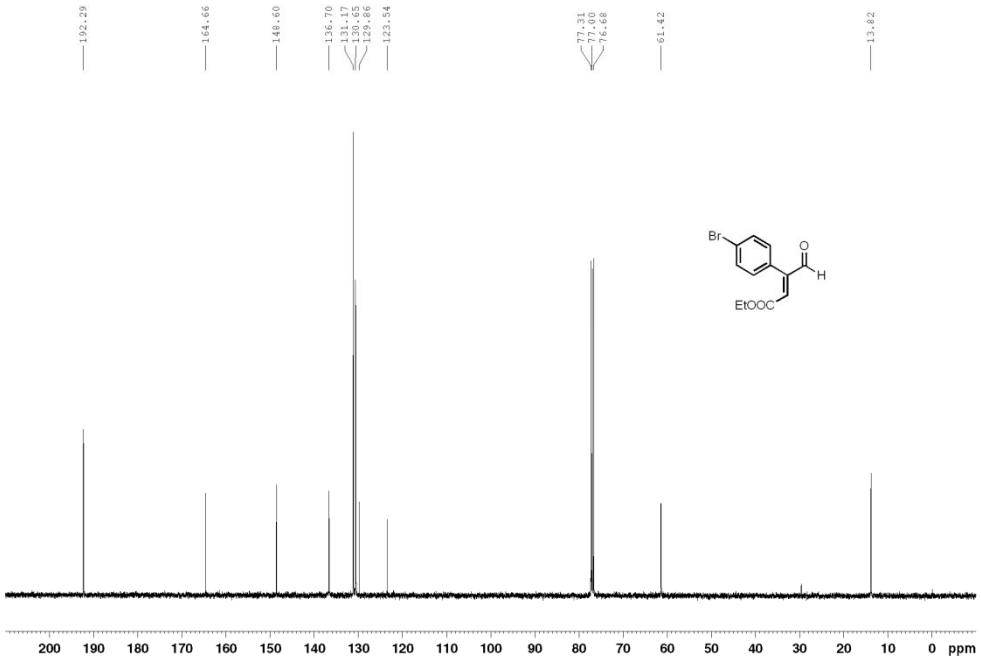

9.739
7.264
7.242
7.237
7.229
7.220
7.212
7.207
7.112
7.106
7.095
7.090
7.085
7.073
7.068
6.705
4.177
4.159
4.141
4.124
1.182
1.164
1.146
0.000
F
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.00
1.92
1.99
1.00
2.02
3.04

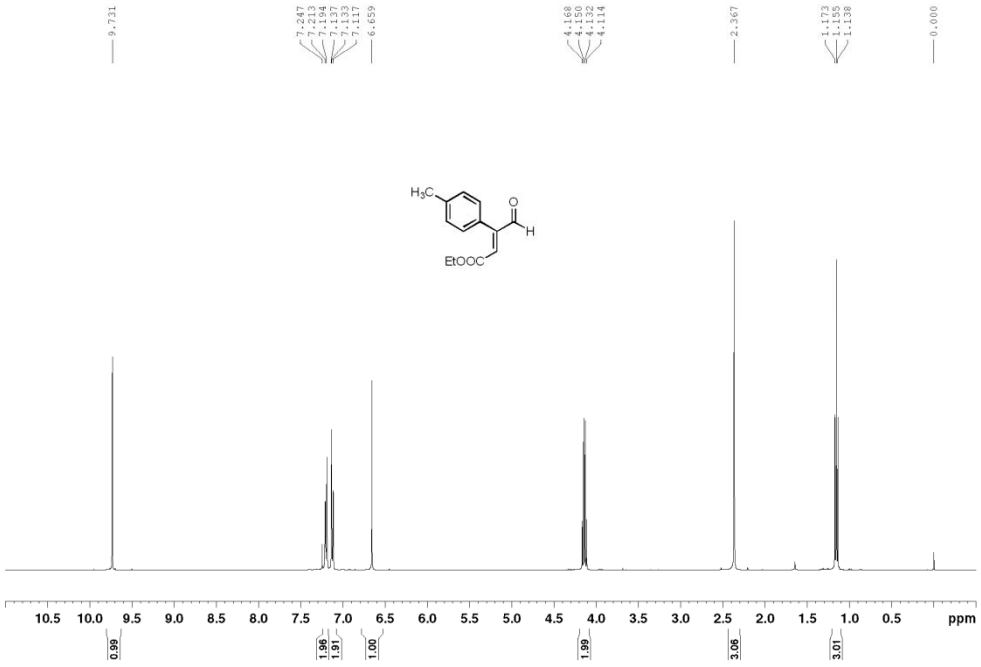

192.64
164.82
164.39
161.91
148.56
136.58
131.09
131.01
126.89
126.85
115.18
114.96
77.32
77.00
76.69
61.33
13.80
F
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

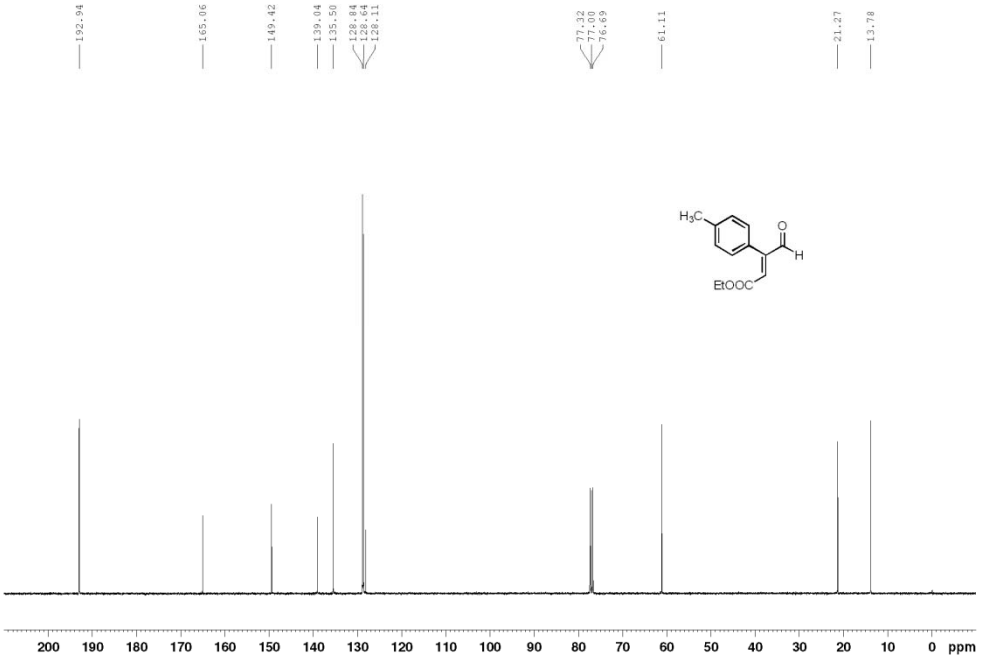

9.729
7.386
7.381
7.369
7.365
7.263
7.180
7.176
7.165
7.159
6.714
4.177
4.160
4.142
4.124
1.186
1.168
1.150
0.000
Cl
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.99
1.96
1.94
1.00
2.01
3.05

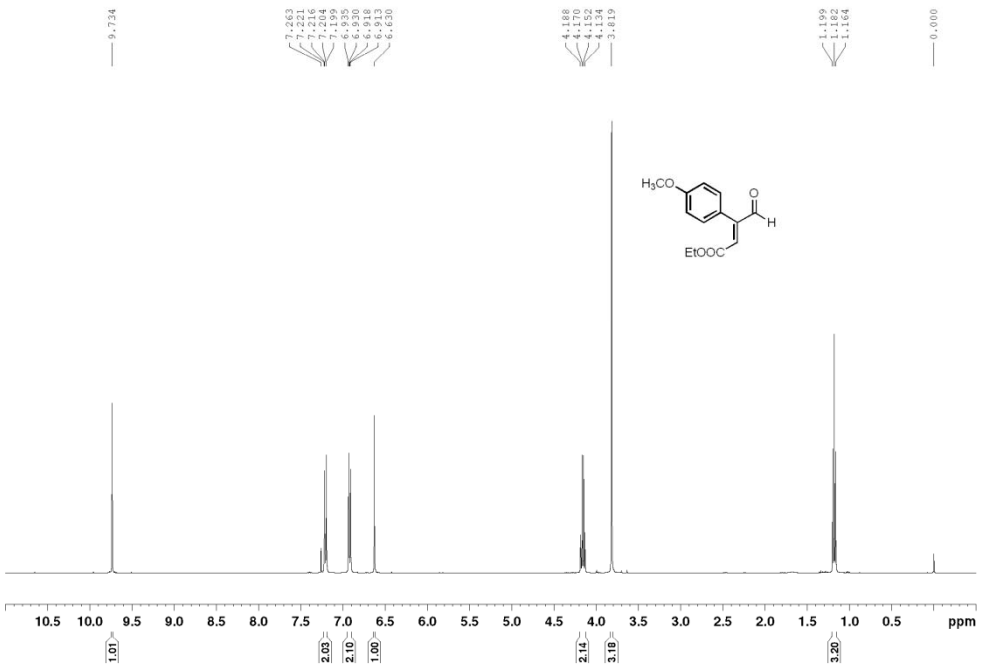

192.35
164.66
148.52
136.69
135.22
130.42
129.40
128.20
77.32
77.00
76.68
61.38
13.80
Cl
O
H
EtOOC
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

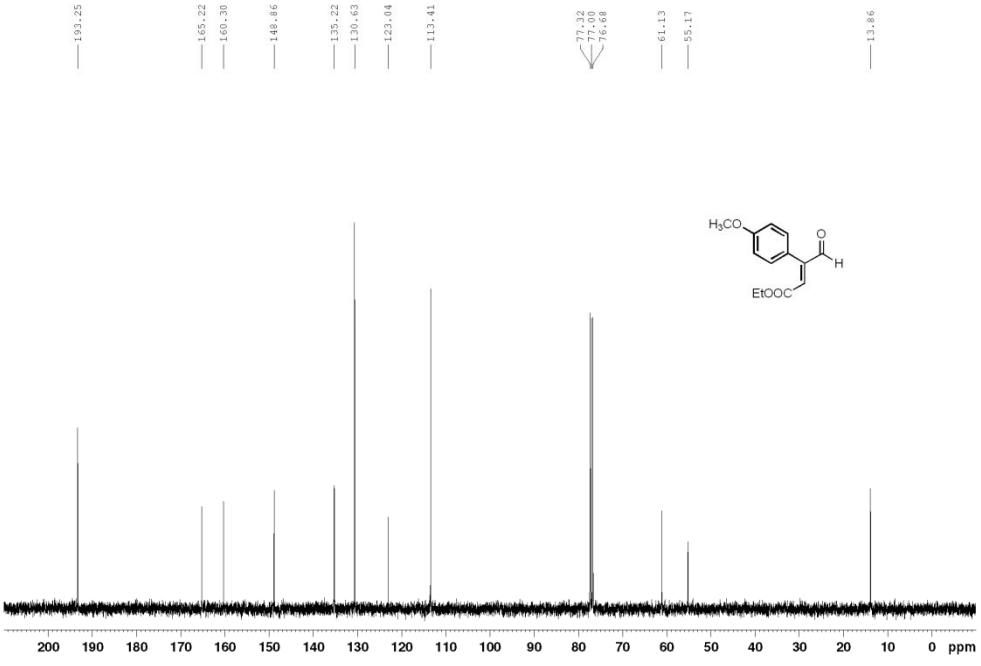

9.730
7.545
7.524
7.263
7.115
7.094
6.718
4.180
4.162
4.144
4.127
1.190
1.172
1.154
0.000
Br
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.98
2.00
1.96
1.00
1.99
3.02

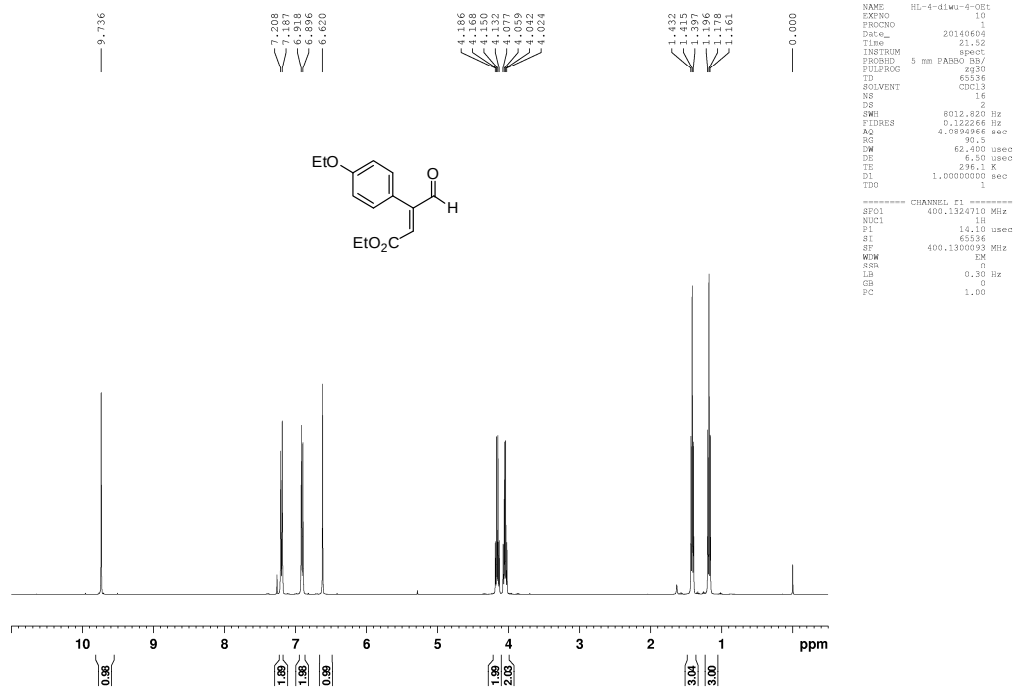

192.29
164.66
148.60
136.70
131.17
130.65
129.86
123.54
77.31
77.00
76.68
61.42
13.82
Br
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


9.731
7.247
7.233
7.194
7.137
7.133
7.117
6.659
4.168
4.156
4.132
4.114
2.367
1.173
1.155
1.138
0.000
H3C
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.99
1.96
1.91
1.00
1.99
3.06
3.01

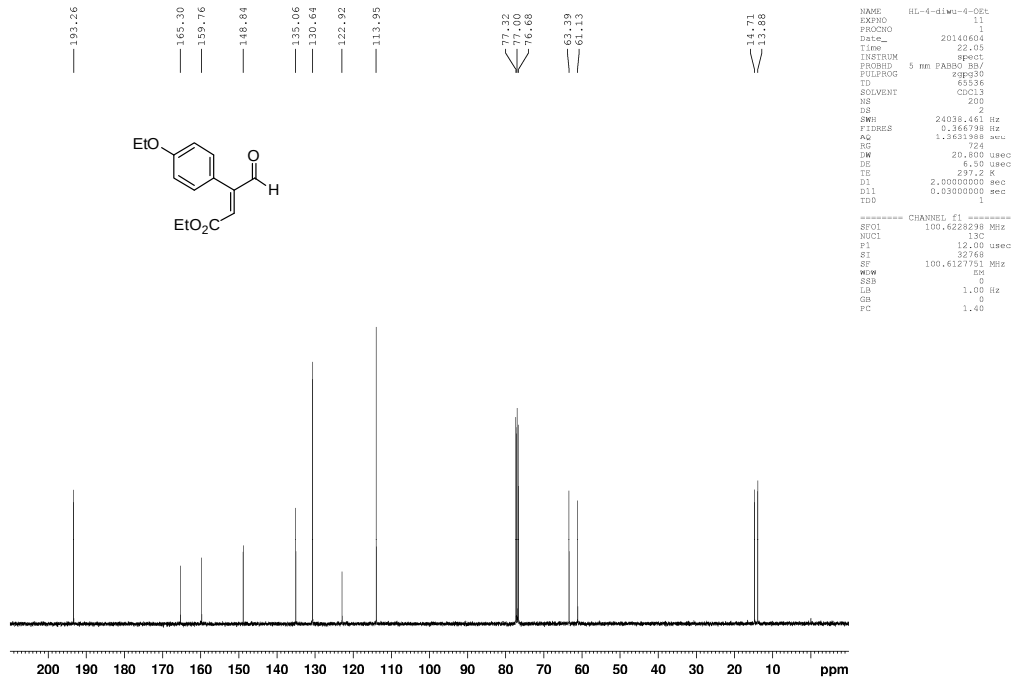

192.94
165.06
149.42
139.04
135.50
128.84
128.64
128.11
77.32
77.00
76.69
61.11
21.27
13.78
H3C
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


9.734
7.263
7.221
7.216
7.204
7.199
6.935
6.930
6.918
6.913
6.630
4.188
4.170
4.152
4.134
3.819
1.199
1.182
1.164
0.000
H3CO
O
H
EtOOC
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.01
2.03
2.10
1.00
2.14
3.16
3.20

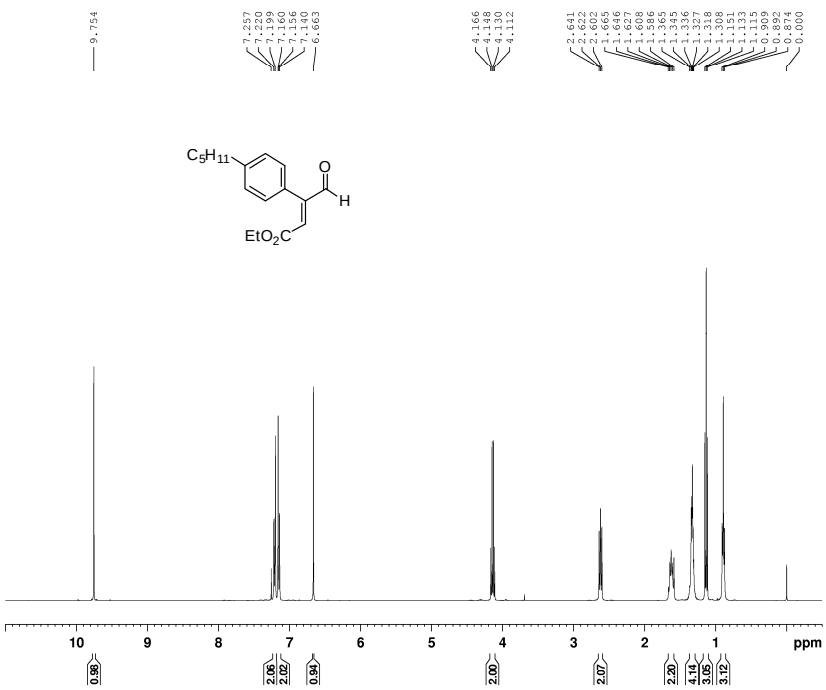

193.25
165.22
160.30
148.86
135.22
130.63
123.04
113.41
77.32
77.00
76.68
61.13
55.17
13.86
H3CO
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



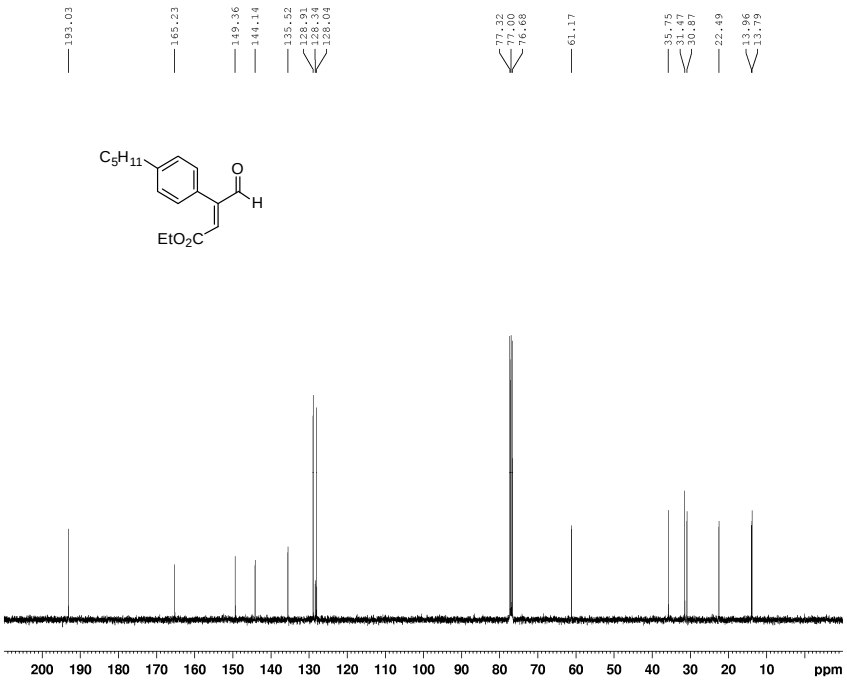

```
NAME          HL-4-diwu-4-OEt
EXPNO                      10
PROCNO                      1
Date_              20140604
Time                   21.52
INSTRUM                spect
PROBHD     5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894866 sec
RG                      90.5
DW                    62.400 usec
DE                     6.50 usec
TE                    296.1 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                      14.10 usec
SI                      65536
SF               400.1300093 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```



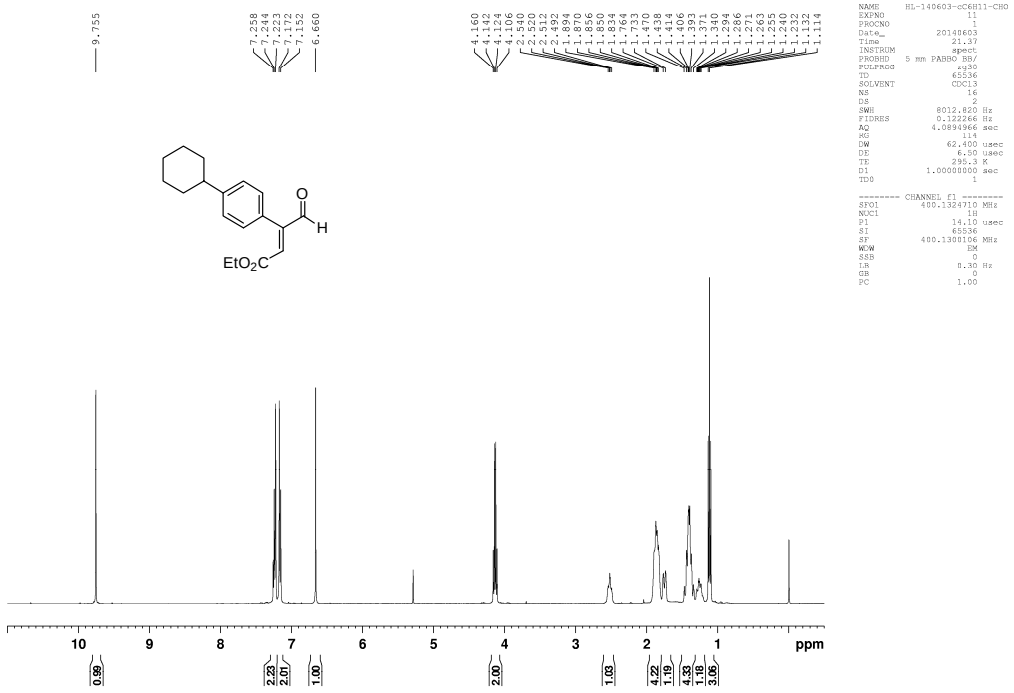

```
NAME          HL-4-diwu-4-OEt
EXPNO                      11
PROCNO                      1
Date_              20140604
Time                   22.05
INSTRUM                spect
PROBHD     5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      200
DS                        2
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      724
DW                    20.800 usec
DE                     6.50 usec
TE                    297.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             100.6228298 MHz
NUC1                       13C
P1                      12.00 usec
SI                      32768
SF               100.6127751 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



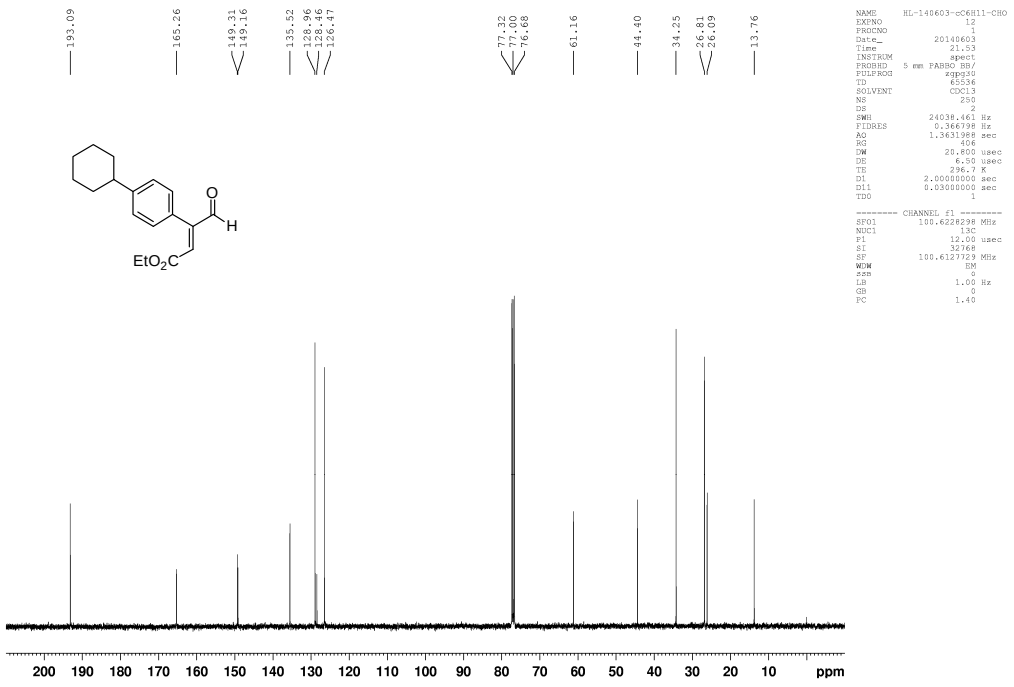

```
NAME          HL-4-diwu-4-amyl
EXPNO                       10
PROCNO                       1
Date_              20140708
Time                    21.34
INSTRUM                 spect
PROBHD     5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                          16
DS                           2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                         101
DW                  62.400 usec
DE                   6.50 usec
TE                   296.0 K
D1                1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                      1H
P1                   14.10 usec
SI                     65536
SF              400.1300107 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                   1.00
```



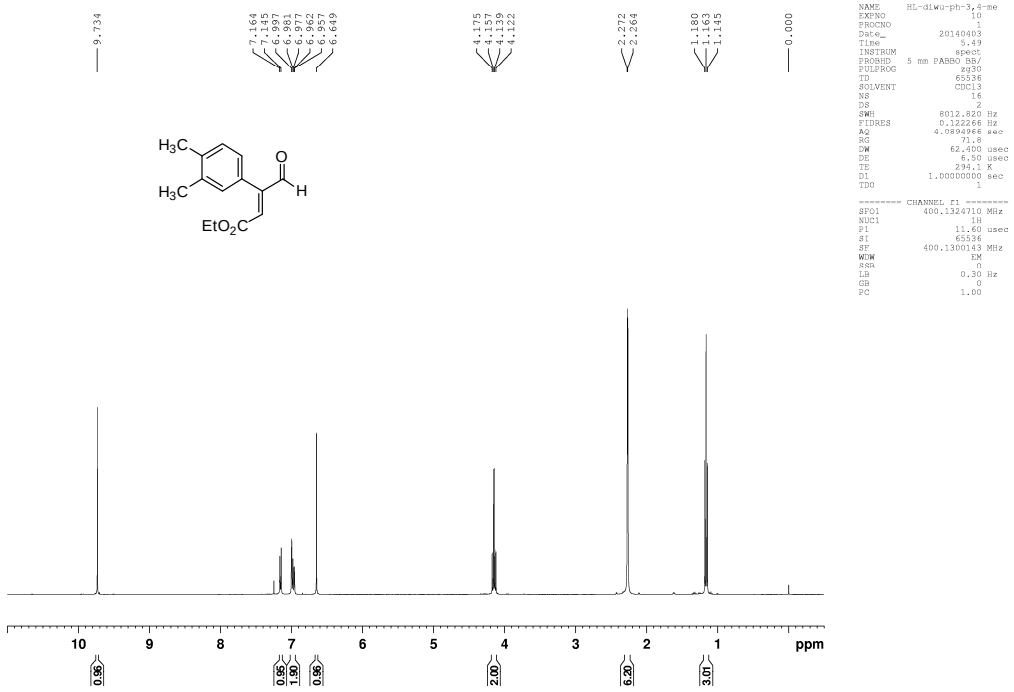

```
NAME          HL-4-diwu-4-amyl
EXPNO                       11
PROCNO                       1
Date_              20140708
Time                    21.39
INSTRUM                 spect
PROBHD     5 mm PABBO BB/
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                          85
DS                           2
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                        1030
DW                  20.800 usec
DE                   6.50 usec
TE                   297.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                      13C
P1                   12.00 usec
SI                     32768
SF              100.6127729 MHz
WDW                        EM
SSB                         0
LB                   1.00 Hz
GB                          0
PC                   1.40
```



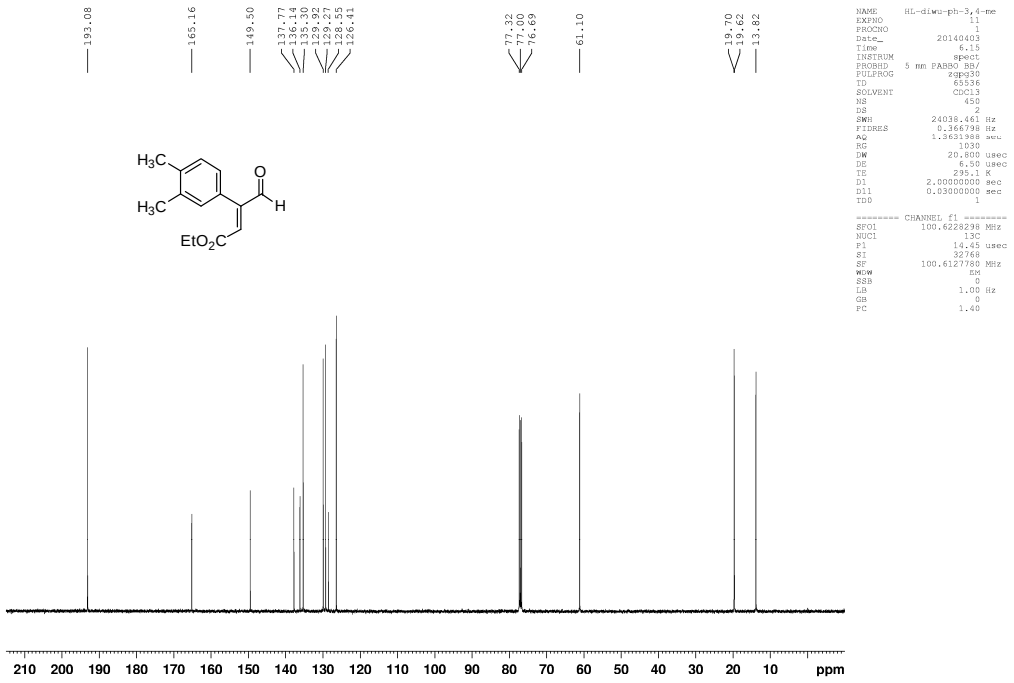

```
NAME          HL-140603-cC6H11-CHO
EXPNO                           11
PROCNO                           1
Date_                     20140603
Time                         21.37
INSTRUM                      spect
PROBHD     5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                      CDCl3
NS                              16
DS                               2
SWH                     8012.820 Hz
FIDRES                  0.122266 Hz
AQ                      4.0894966 sec
RG                             114
DW                        62.400 usec
DE                         6.50 usec
TE                        295.3 K
D1                     1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
SFO1                  400.1324710 MHz
NUC1                          1H
P1                         14.10 usec
SI                         65536
SF                    400.1300106 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



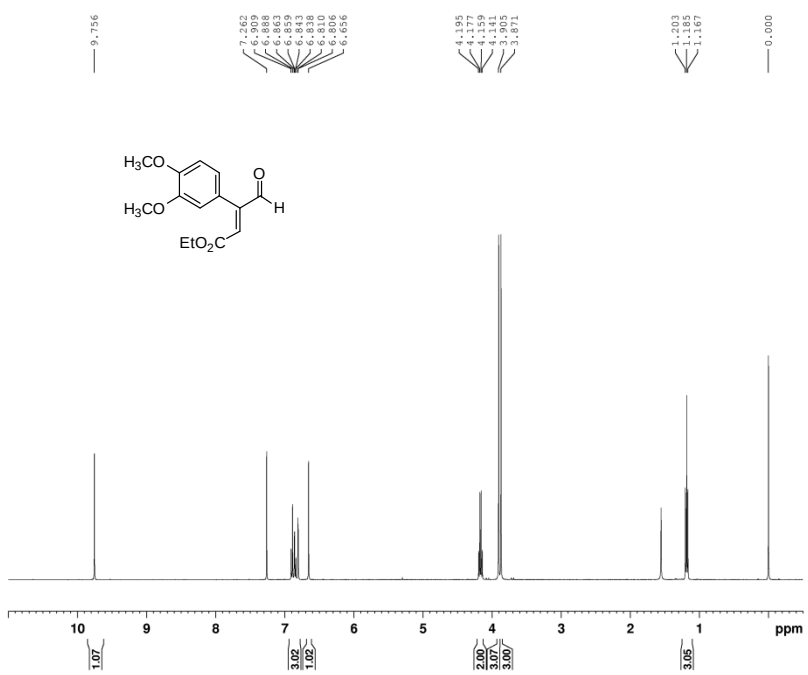

```
NAME          HL-140603-cC6H11-CHO
EXPNO                           12
PROCNO                           1
Date_                     20140603
Time                         21.53
INSTRUM                      spect
PROBHD     5 mm PABBO BB/
PULPROG                   zgpg30
TD                          65536
SOLVENT                      CDCl3
NS                             250
DS                               2
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                             406
DW                        20.800 usec
DE                         6.50 usec
TE                        296.7 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                              1

======== CHANNEL f1 ========
SFO1                  100.6228298 MHz
NUC1                          13C
P1                         12.00 usec
SI                         32768
SF                    100.6127729 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



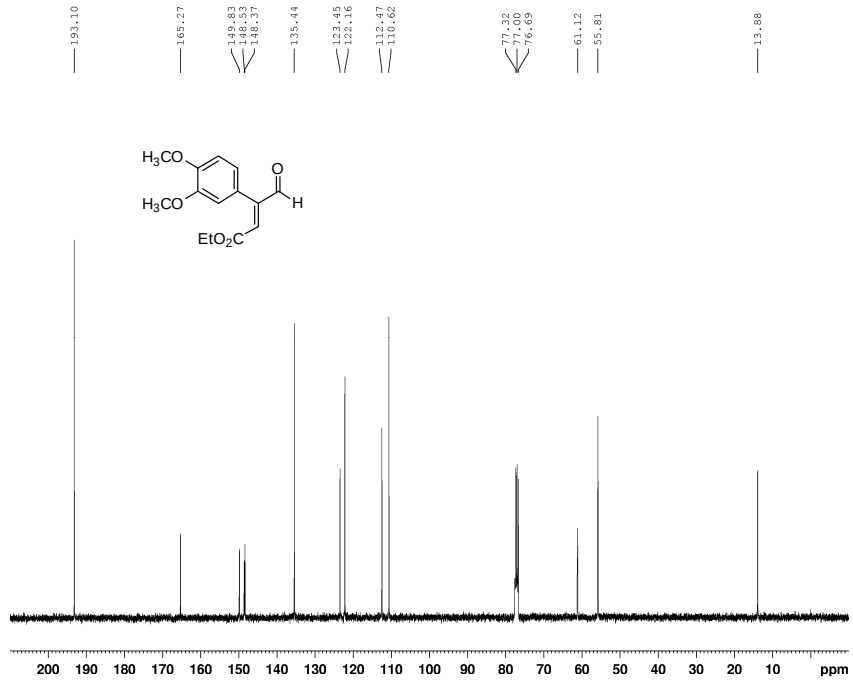

```
NAME          HL-diwu-ph-3,4-me
EXPNO                 10
PROCNO                 1
Date_           20140403
Time                5.49
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894966 sec
RG                 71.8
DW                62.400 usec
DE                 6.50 usec
TE                294.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1               1H
P1                11.60 usec
SI                65536
SF           400.1300143 MHz
WDW                EM
SSB                 0
LB                0.30 Hz
GB                  0
PC                1.00
```



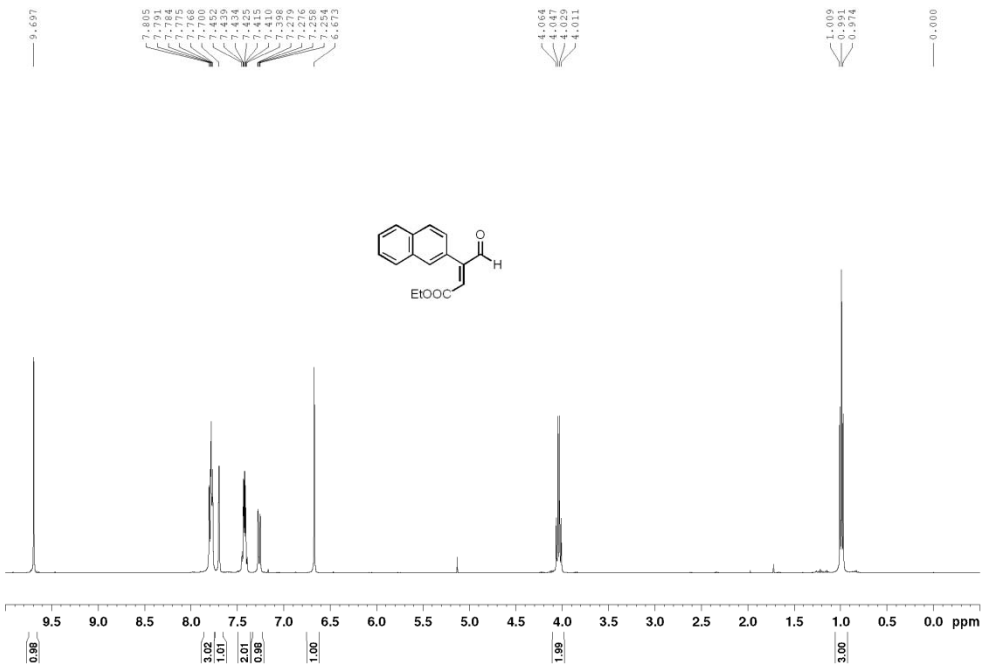

```
NAME          HL-diwu-ph-3,4-me
EXPNO                 11
PROCNO                 1
Date_           20140403
Time                6.15
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  450
DS                    2
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631988 sec
RG                 1030
DW                20.800 usec
DE                 6.50 usec
TE                295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228298 MHz
NUC1               13C
P1                14.45 usec
SI                32768
SF           100.6127780 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```

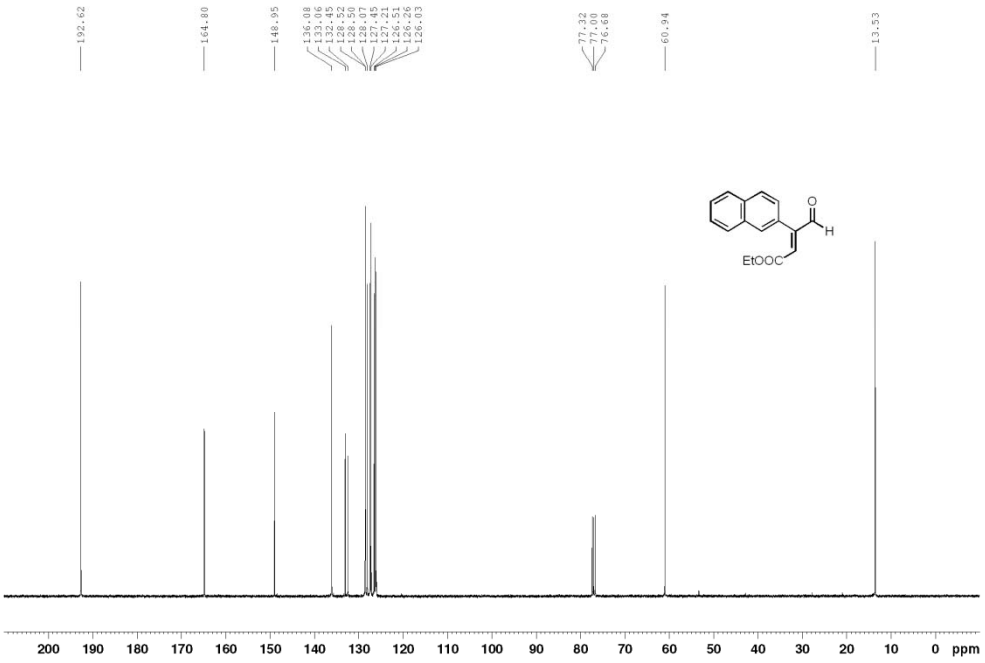


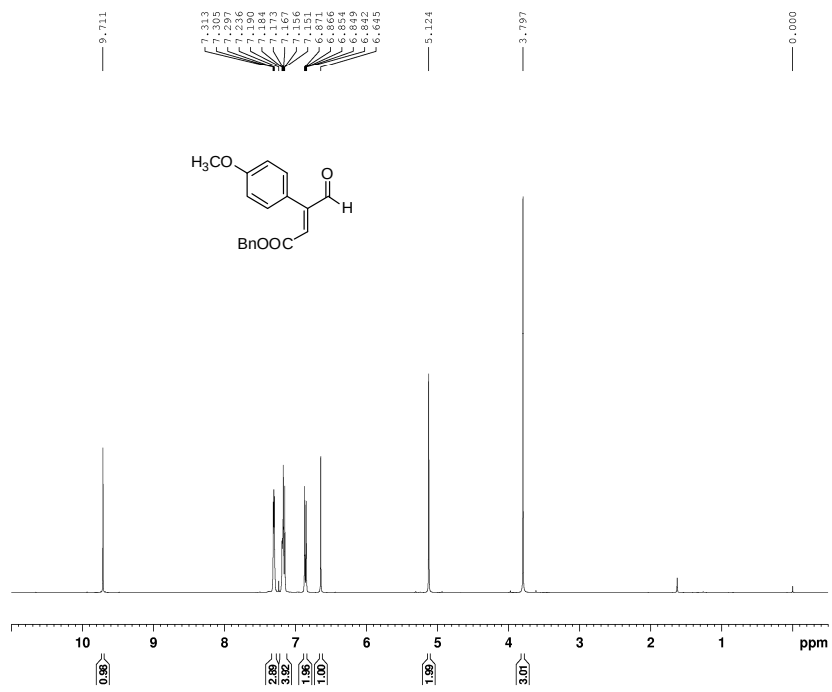

```
NAME          HL-140603-diOMe-CHO
EXPNO                          10
PROCNO                          1
Date_                    20140603
Time                        21.31
INSTRUM                     spect
PROBHD   5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                             16
DS                              2
SWH                     8012.820 Hz
FIDRES                  0.122266 Hz
AQ                      4.0894966 sec
RG                            322
DW                         62.400 usec
DE                          6.50 usec
TE                         295.4 K
D1                      1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                 400.1324710 MHz
NUC1                         1H
P1                          14.10 usec
SI                         65536
SF                   400.1300088 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



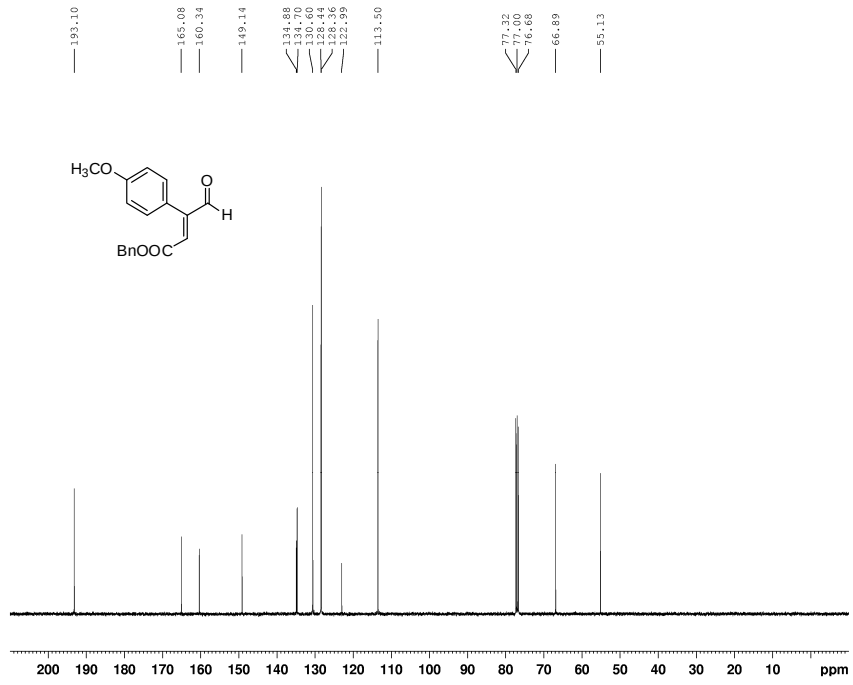

```
NAME          HL-4-diwu-3,4-diOMe
EXPNO                          11
PROCNO                          1
Date_                    20140528
Time                        21.58
INSTRUM                     spect
PROBHD   5 mm PABBO BB/
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                            250
DS                              2
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                            575
DW                         20.800 usec
DE                          6.50 usec
TE                         296.9 K
D1                      2.00000000 sec
D11                     0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                 100.6228298 MHz
NUC1                         13C
P1                          12.00 usec
SI                         32768
SF                   100.6127711 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```


9.697
7.805
7.791
7.784
7.775
7.768
7.700
7.452
7.439
7.434
7.425
7.415
7.410
7.398
7.279
7.276
7.258
7.254
6.673
4.064
4.047
4.029
4.011
1.009
0.991
0.974
0.000
O
H
EtOOC
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.98
3.02
1.01
2.01
0.98
1.00
1.99
3.00

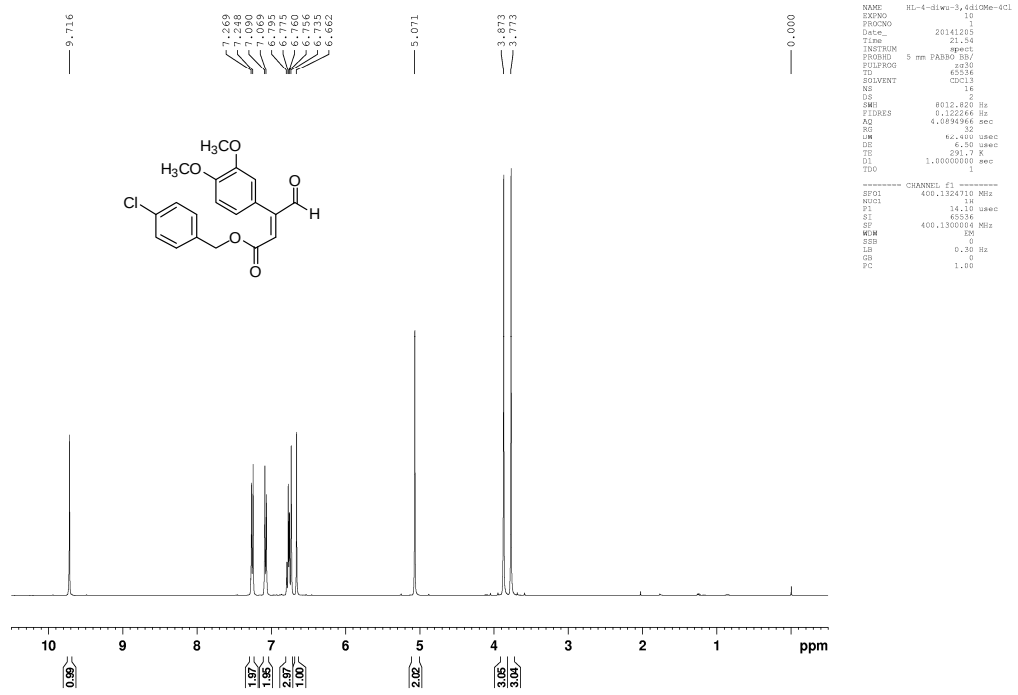

192.62
164.80
148.95
136.08
133.06
132.45
128.52
128.50
128.07
127.45
127.21
126.51
126.20
126.03
77.32
77.00
76.68
60.94
13.53
O
H
EtOOC
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



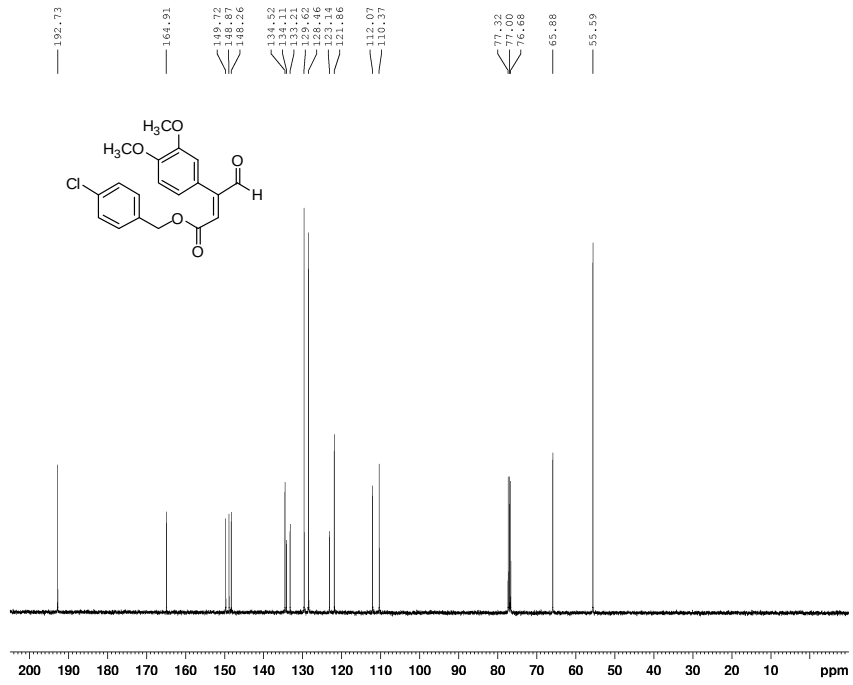

```
NAME        HL-4-diwu-4OMe-COOBn
EXPNO               10
PROCNO               1
Date_         20141028
Time             21.36
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG                 90.6
DW              62.400 usec
DE               6.50 usec
TE              294.2 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.10 usec
SI                65536
SF          400.1300192 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



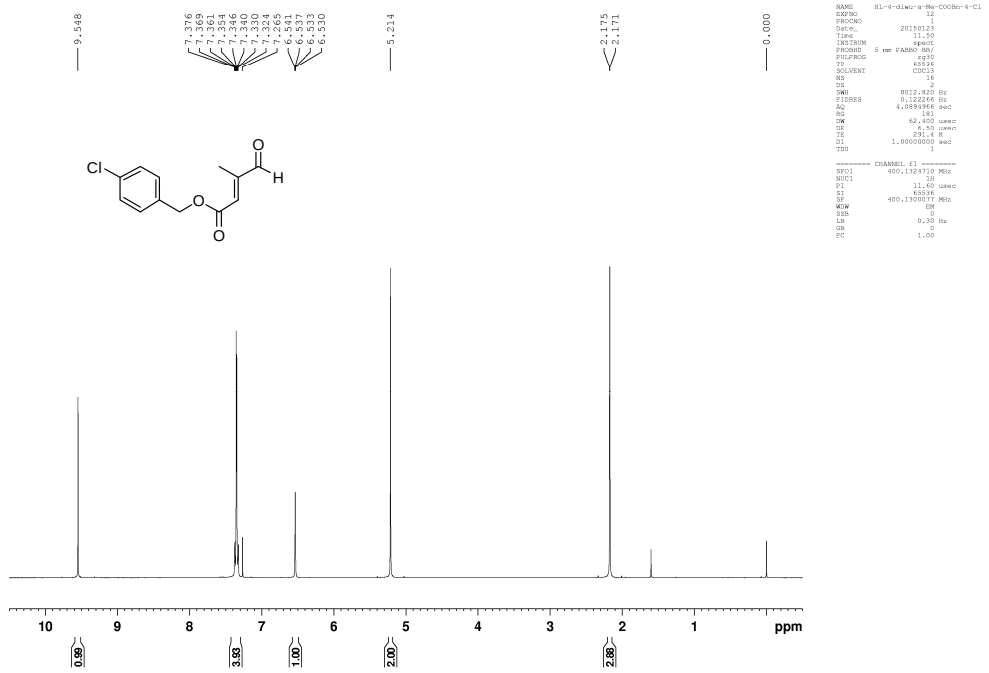

```
NAME        HL-4-diwu-4OMe-COOBn
EXPNO               11
PROCNO               1
Date_         20141028
Time             21.49
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                 200
DS                   2
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                1030
DW              20.800 usec
DE               6.50 usec
TE              295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                12.00 usec
SI                32768
SF          100.6127802 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

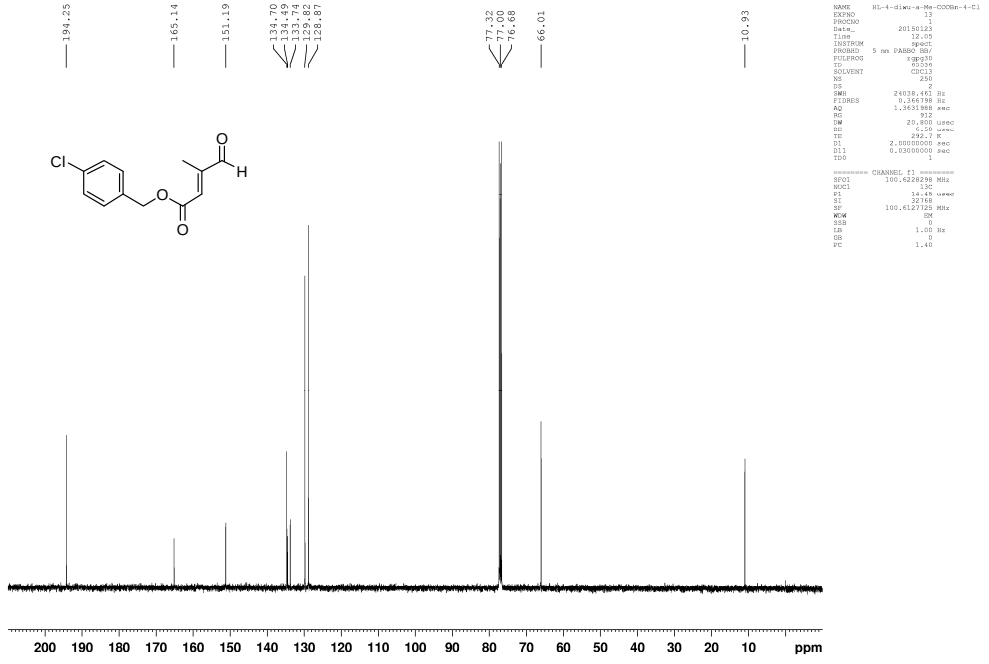


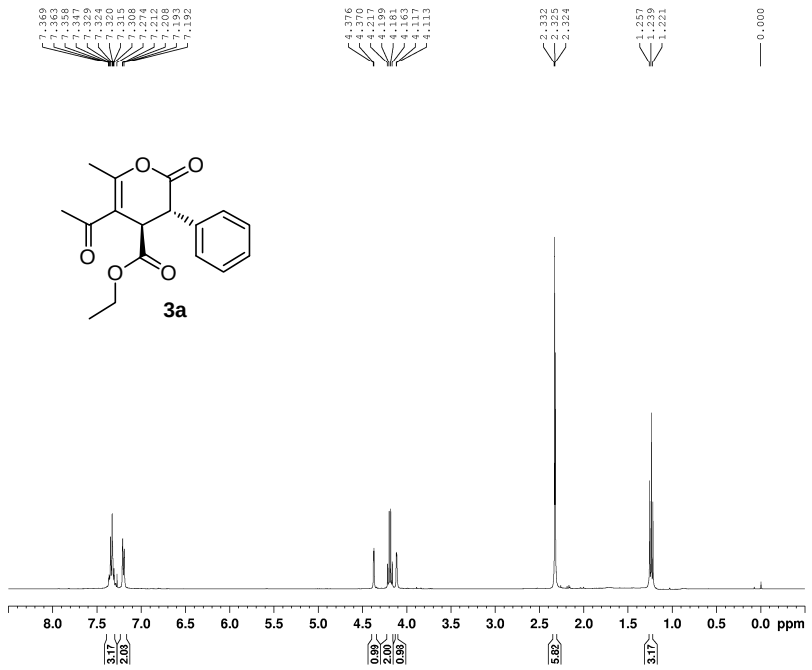

```
NAME        HL-4-diwu-3,4diOMe-4Cl
EXPNO                           10
PROCNO                           1
Date_                     20141205
Time                         21.54
INSTRUM                      spect
PROBHD     5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8012.820 Hz
FIDRES                   0.122266 Hz
AQ                       4.0894966 sec
RG                             32
DW                       62.400 usec
DE                        6.50 usec
TE                        291.7 K
D1                       1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                   400.1324710 MHz
NUC1                           1H
P1                          14.10 usec
SI                          65536
SF                     400.1300004 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



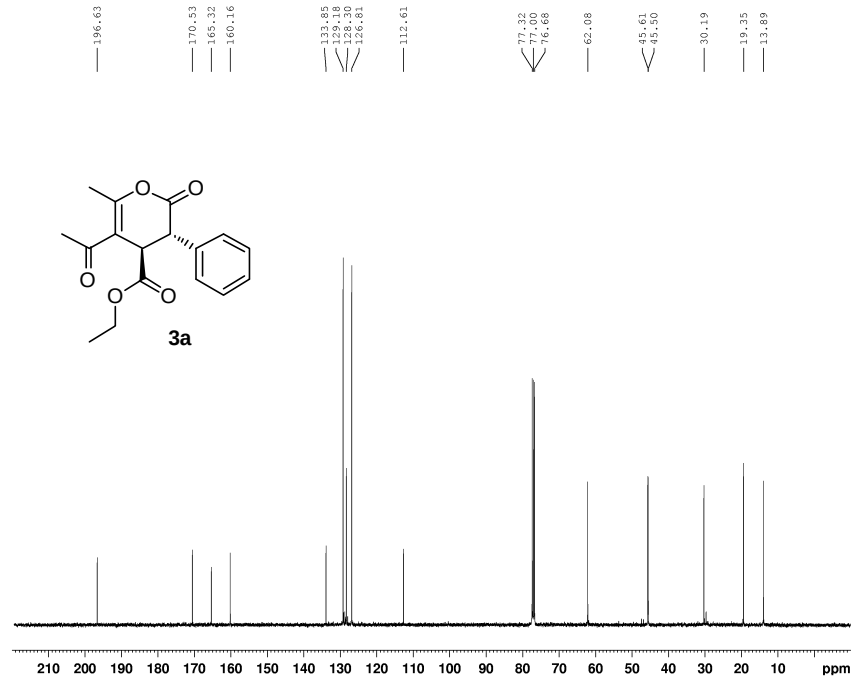

```
NAME        HL-4-diwu-3,4diOMe-4Cl
EXPNO                           11
PROCNO                           1
Date_                     20141205
Time                         22.04
INSTRUM                      spect
PROBHD     5 mm PABBO BB/
PULPROG                   zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            150
DS                              2
SWH                     24038.461 Hz
FIDRES                   0.366798 Hz
AQ                       1.3631988 sec
RG                            912
DW                       20.800 usec
DE                        6.50 usec
TE                        292.8 K
D1                       2.00000000 sec
D11                      0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                   100.6228298 MHz
NUC1                           13C
P1                          12.00 usec
SI                          32768
SF                     100.6127912 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```


9.548
7.376
7.369
7.361
7.354
7.346
7.340
7.330
7.324
7.265
6.537
6.533
6.530
5.214
2.175
2.171
0.000
Cl
O
H
O
O
10
9
8
7
6
5
4
3
2
1
ppm
0.99
3.93
1.00
2.00
2.88

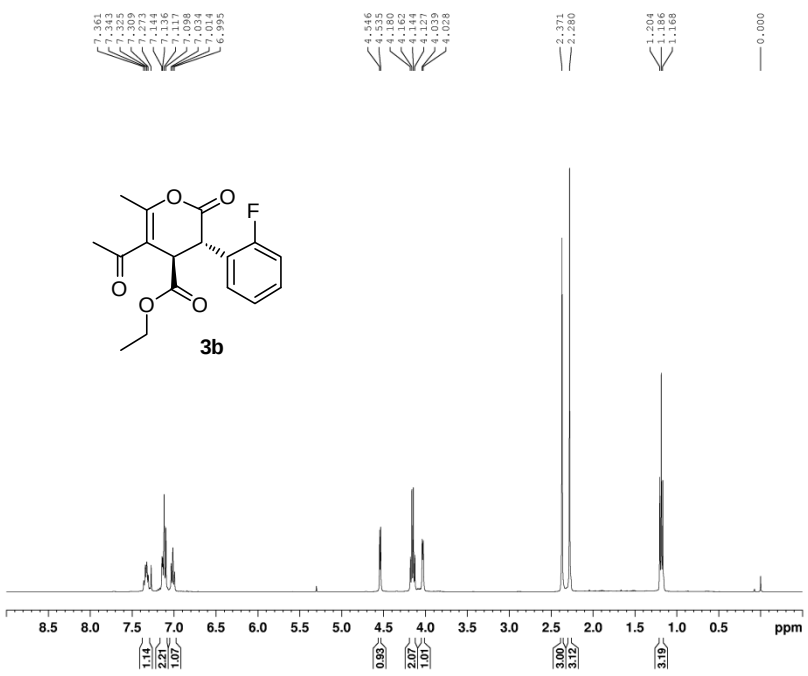

194.25
165.14
151.19
134.70
134.49
133.74
129.82
128.87
77.32
77.00
76.68
66.01
10.93
Cl
O
H
O
O
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm



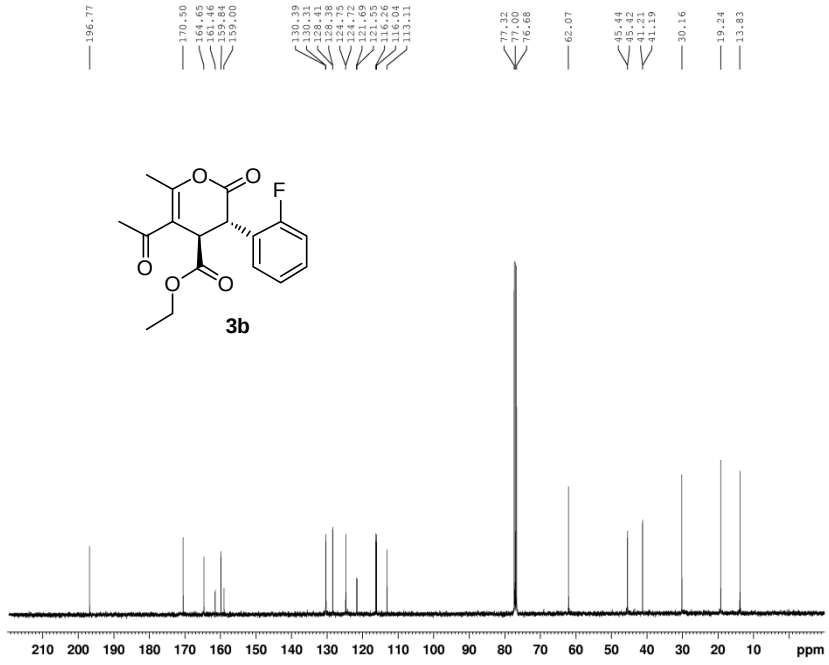

```
NAME          HL-4-chanwu-ph
EXPNO                     12
PROCNO                     1
Date_               20140409
Time                   14.15
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                      88.6
DW                    62.400 usec
DE                     6.50 usec
TE                    295.2 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                       1H
P1                     11.60 usec
SI                      65536
SF               400.1300041 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



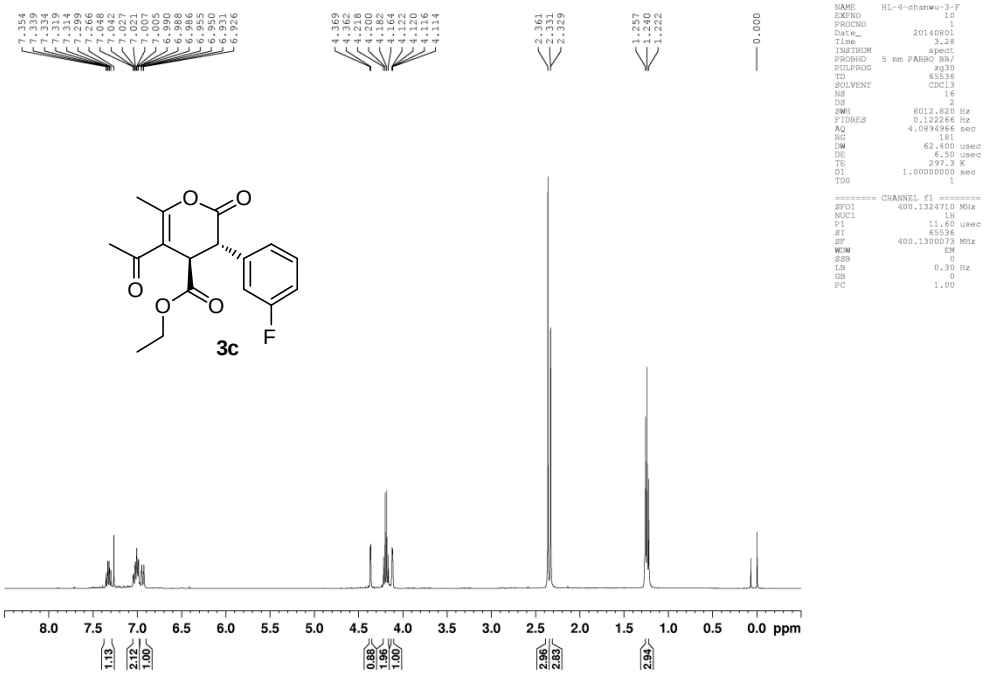

```
NAME          HL-4-chanwu-ph
EXPNO                     13
PROCNO                     1
Date_               20140409
Time                   14.39
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       400
DS                         2
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      912
DW                    20.800 usec
DE                     6.50 usec
TE                    296.5 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             100.6228298 MHz
NUC1                       13C
P1                     14.45 usec
SI                      32768
SF               100.6127751 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



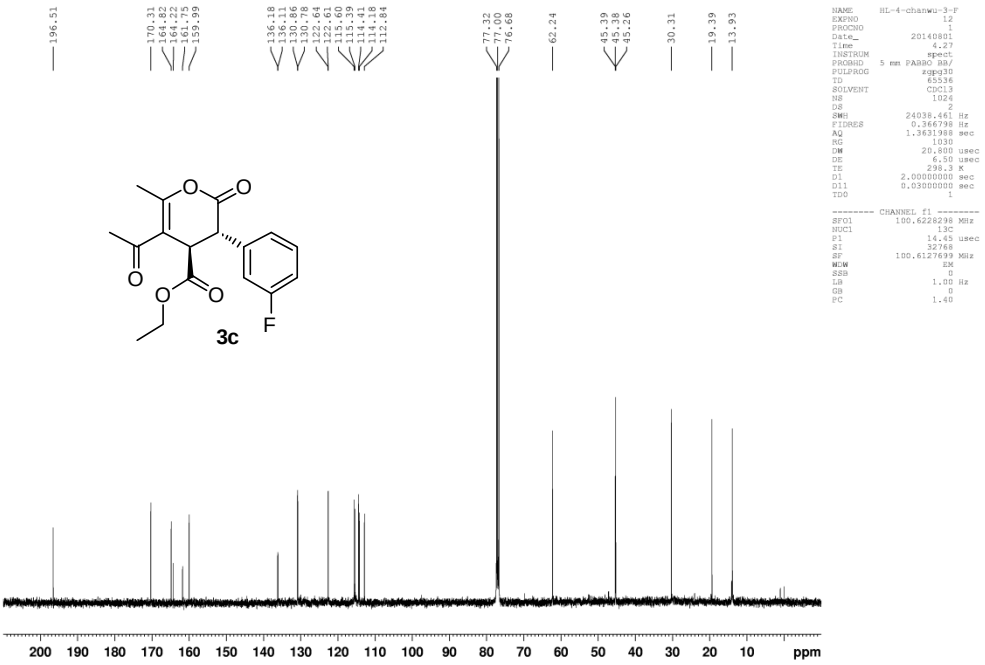

```
NAME          HL-chanwu-Ph-2F
EXPNO                      10
PROCNO                      1
Date_              20140411
Time                   12.08
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                       114
DW                  62.400 usec
DE                   6.50 usec
TE                  293.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1               400.1324710 MHz
NUC1                       1H
P1                    11.60 usec
SI                     65536
SF                 400.1300042 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



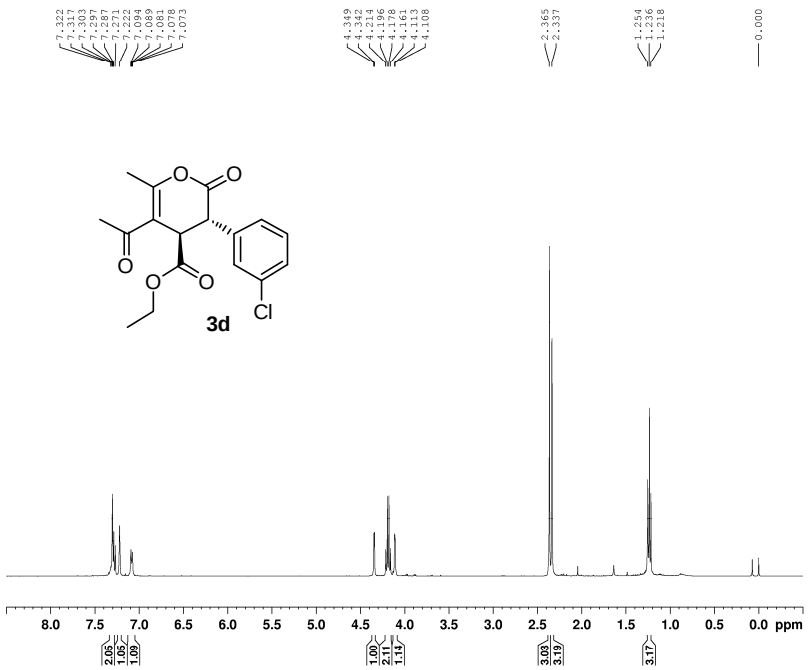

```
NAME          HL-chanwu-Ph-2F
EXPNO                      11
PROCNO                      1
Date_              20140411
Time                   12.32
INSTRUM                spect
PROBHD   5 mm PABBO BA/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       400
DS                        2
SWH               24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                      1030
DW                  20.800 usec
DE                   6.50 usec
TE                  294.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1               100.6228298 MHz
NUC1                       13C
P1                    14.45 usec
SI                     32768
SF                 100.6127736 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



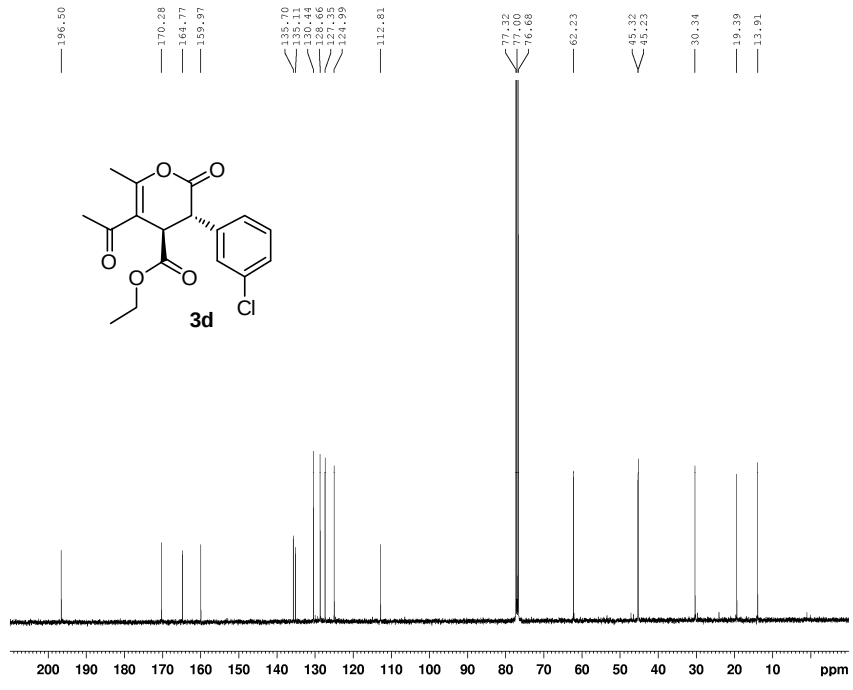

```
NAME            HL-4-chanwu-3-F
EXPNO                        10
PROCNO                        1
Date_               20140801
Time                      3.28
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                   CDCl3
NS                           16
DS                            2
SWH                    8012.820 Hz
FIDRES                 0.122266 Hz
AQ                     4.0894966 sec
RG                          181
DW                     62.400 usec
DE                      6.50 usec
TE                      297.3 K
D1                     1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 400.1324710 MHz
NUC1                         1H
P1                     11.60 usec
SI                       65536
SF                   400.1300073 MHz
WDW                          EM
SSB                           0
LB                       0.30 Hz
GB                            0
PC                         1.00
```



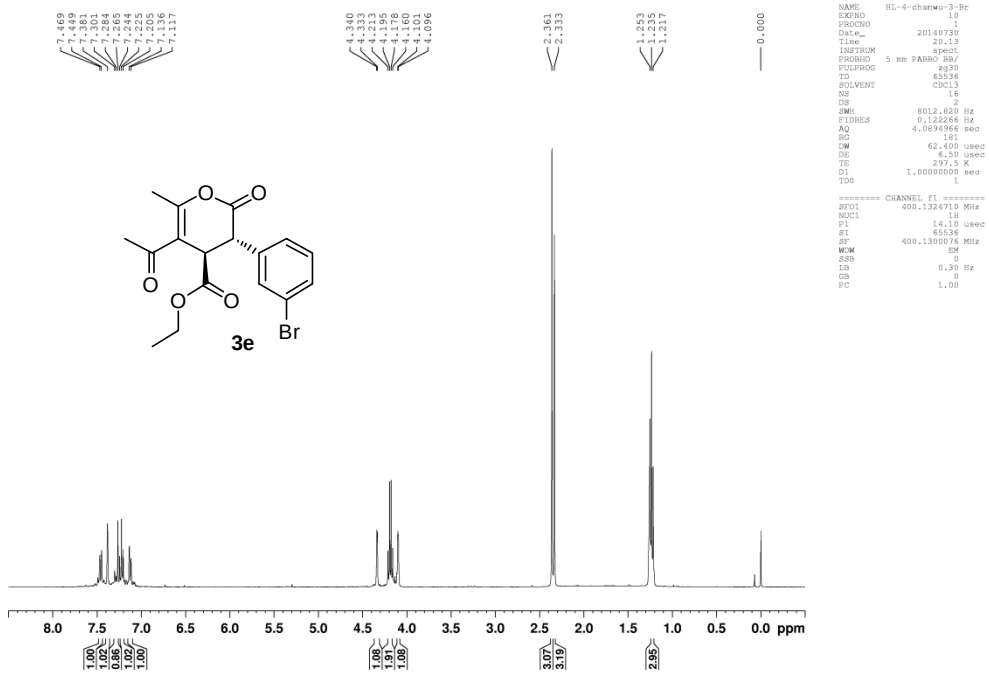

```
NAME            HL-4-chanwu-3-F
EXPNO                        12
PROCNO                        1
Date_               20140801
Time                      4.27
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                   zgpg30
TD                       65536
SOLVENT                   CDCl3
NS                         1024
DS                            2
SWH                   24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631988 sec
RG                         1030
DW                     20.800 usec
DE                      6.50 usec
TE                      298.3 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                 100.6226298 MHz
NUC1                         13C
P1                     14.45 usec
SI                       32768
SF                   100.6127699 MHz
WDW                          EM
SSB                           0
LB                     1.00 Hz
GB                            0
PC                         1.40
```



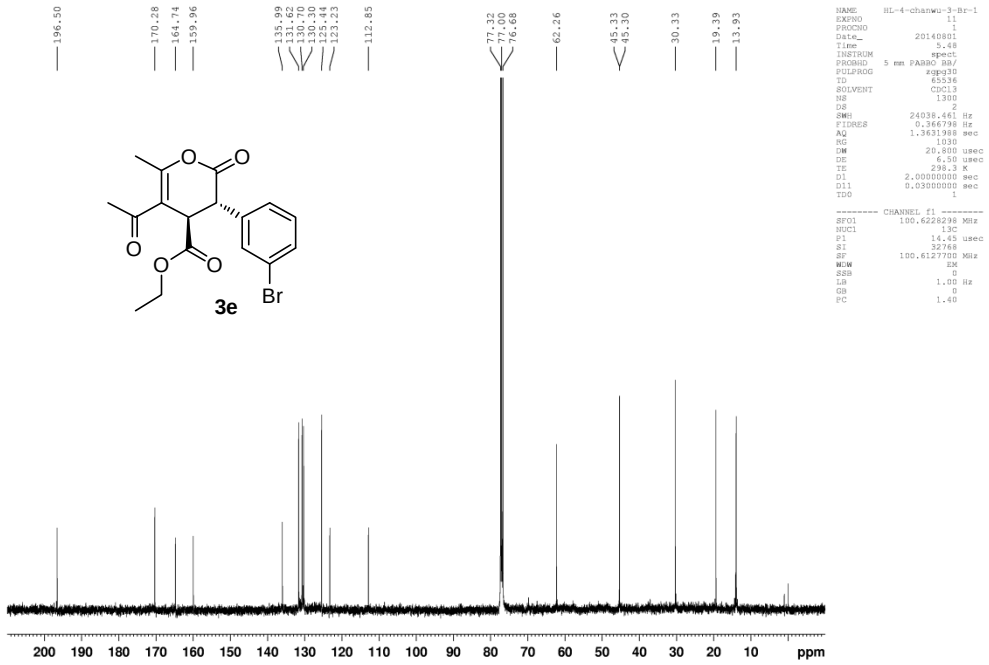

```
NAME          HL-4-141115-3Cl
EXPNO                      22
PROCNO                      1
Date_              20141115
Time                   21.04
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                      144
DW                   62.400 usec
DE                    6.50 usec
TE                   293.0 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                     1H
P1                    14.10 usec
SI                    65536
SF            400.1300054 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



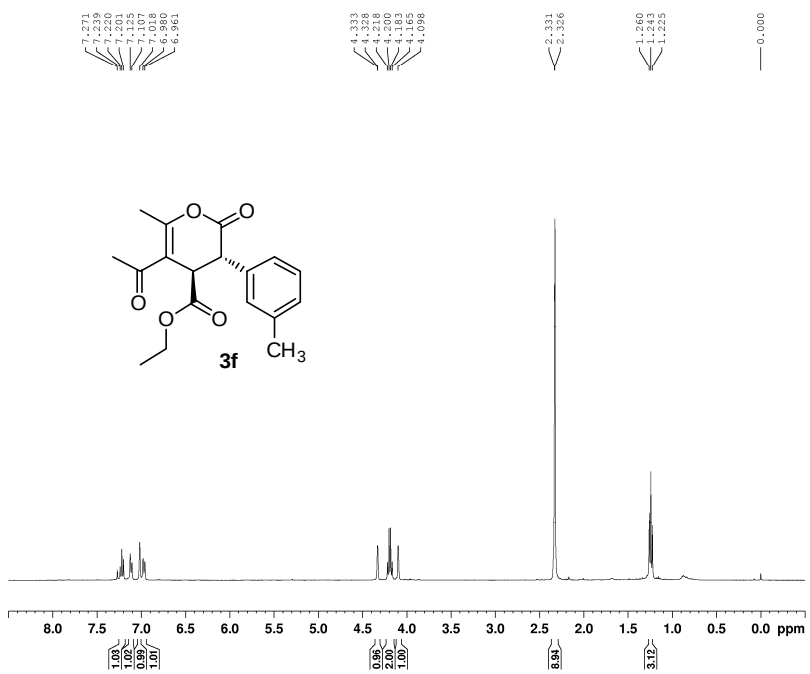

```
NAME          HL-4-141115-3Cl
EXPNO                      23
PROCNO                      1
Date_              20141115
Time                   21.54
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      850
DS                        2
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                     1030
DW                   20.800 usec
DE                    6.50 usec
TE                   294.5 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                     13C
P1                    12.00 usec
SI                    32768
SF            100.6127729 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```



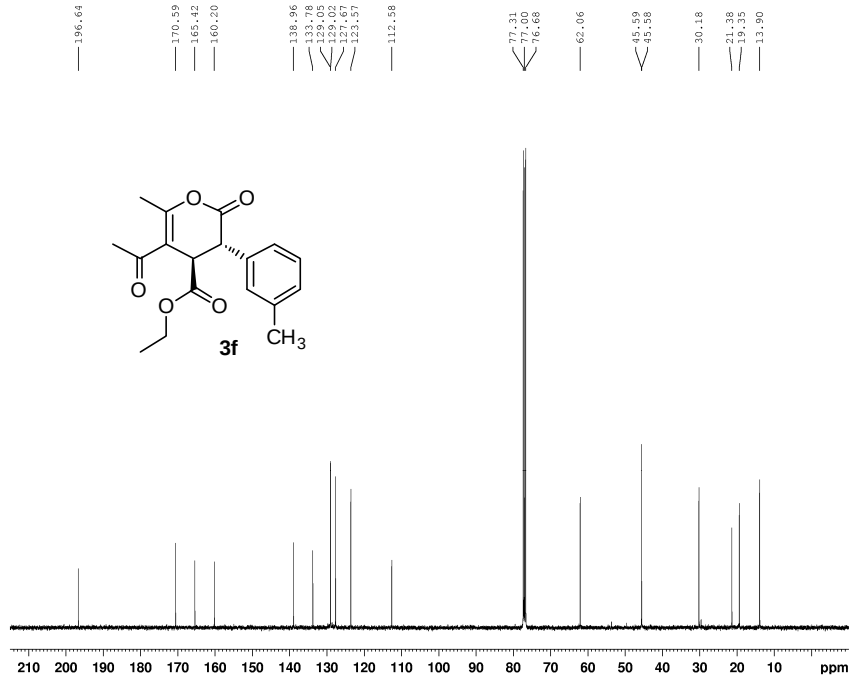

```
NAME            HL-4-chanwu-3-Br
EXPNO                 10
PROCNO                 1
Date_           20140730
Time                20.13
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                   181
DW                62.400 usec
DE                 6.50 usec
TE                297.5 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                   1H
P1                 14.10 usec
SI                 65536
SF              400.1300076 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



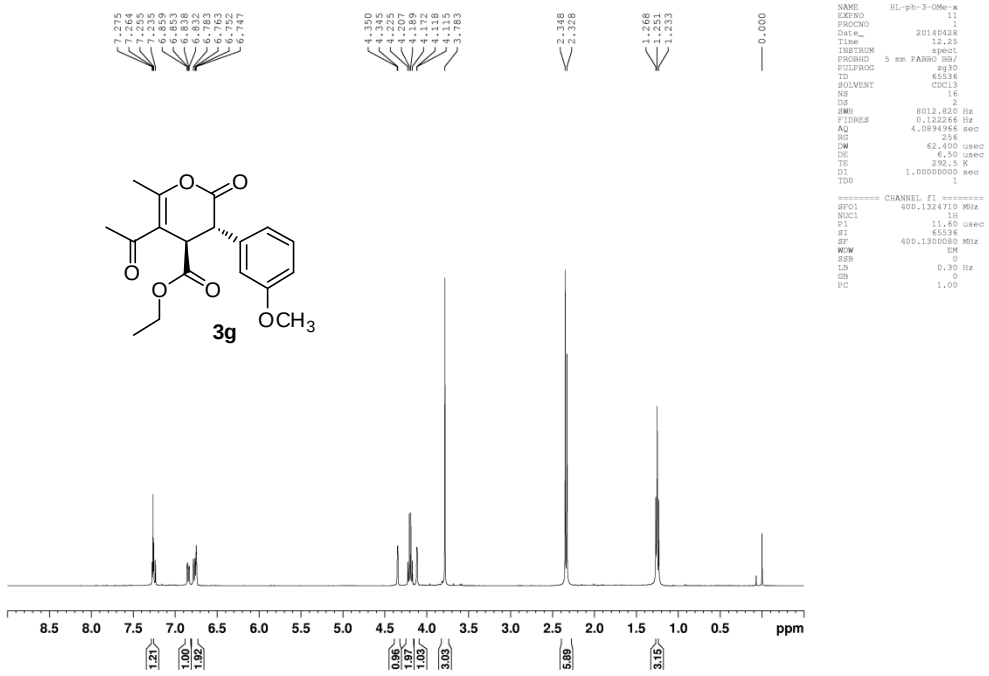

```
NAME            HL-4-chanwu-3-Br-1
EXPNO                 11
PROCNO                 1
Date_           20140801
Time                 5.48
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1300
DS                     2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                  1030
DW                20.800 usec
DE                 6.50 usec
TE                298.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

--------- CHANNEL f1 ---------
SFO1            100.6226298 MHz
NUC1                   13C
P1                 14.45 usec
SI                 32768
SF              100.6127700 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



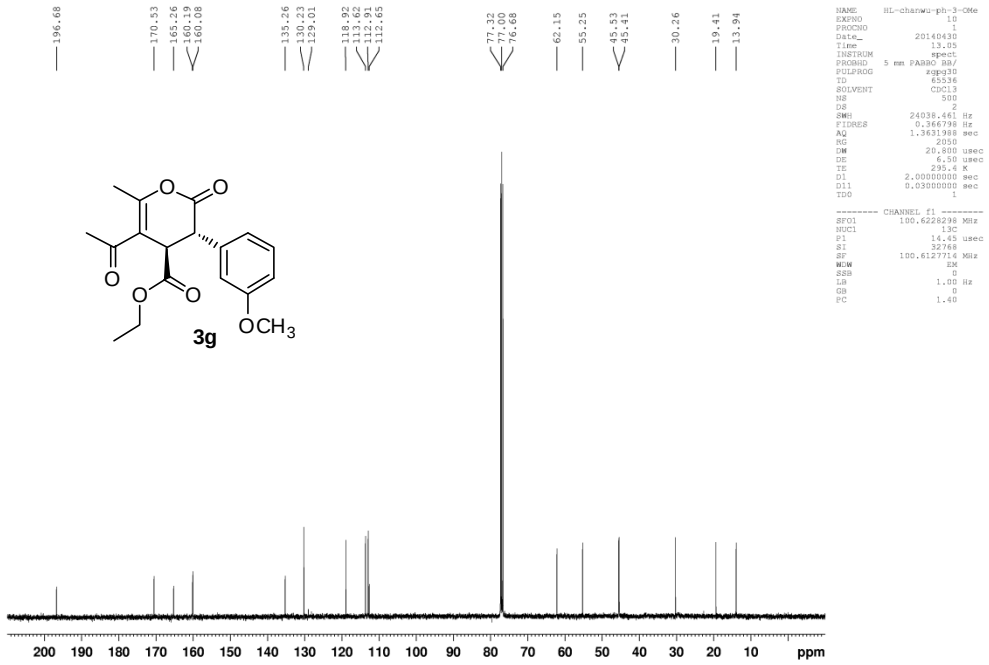

```
NAME          HL-4-chanwu-Ph-3-Me
EXPNO                          10
PROCNO                          1
Date_                    20140408
Time                        21.40
INSTRUM                     spect
PROBHD    5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8012.820 Hz
FIDRES                  0.122266 Hz
AQ                      4.0894966 sec
RG                          90.5
DW                        62.400 usec
DE                         6.50 usec
TE                        296.2 K
D1                    1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                400.1324710 MHz
NUC1                        1H
P1                        14.10 usec
SI                         65536
SF                  400.1300054 MHz
WDW                           EM
SSB                            0
LB                          0.30 Hz
GB                             0
PC                          1.00
```



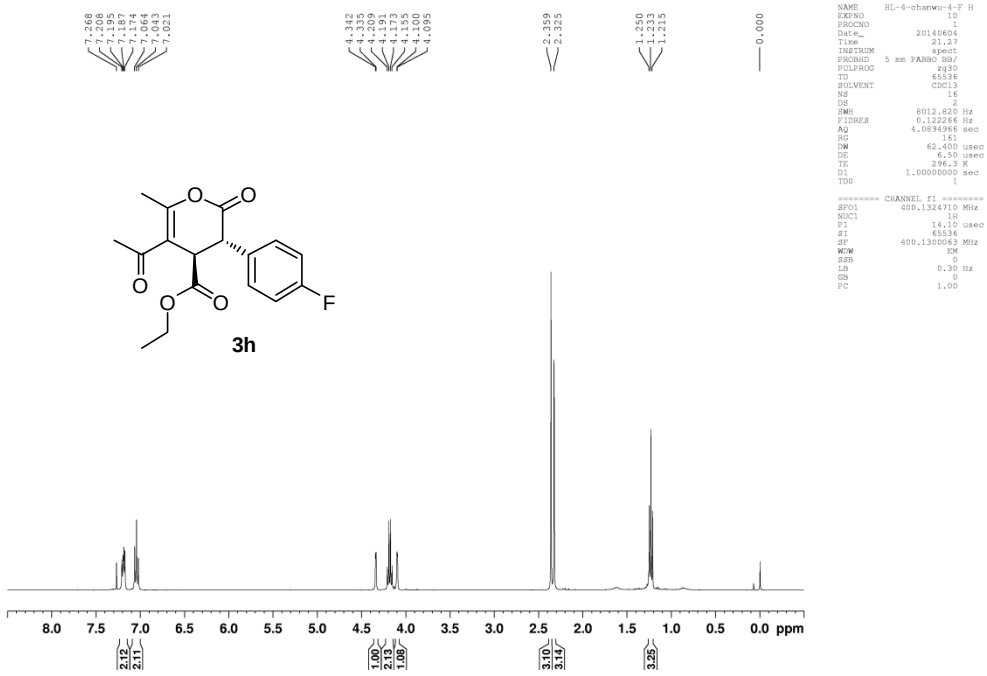

```
NAME          HL-4-chanwu-Ph-3-Me
EXPNO                          11
PROCNO                          1
Date_                    20140408
Time                        22.05
INSTRUM                     spect
PROBHD    5 mm PABBO BB/
PULPROG                  zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           400
DS                             2
SWH                    24038.461 Hz
FIDRES                  0.366798 Hz
AQ                      1.3631988 sec
RG                           575
DW                        20.800 usec
DE                         6.50 usec
TE                        297.6 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                100.6228298 MHz
NUC1                        13C
P1                        12.00 usec
SI                         32768
SF                  100.6127744 MHz
WDW                           EM
SSB                            0
LB                          1.00 Hz
GB                             0
PC                          1.40
```



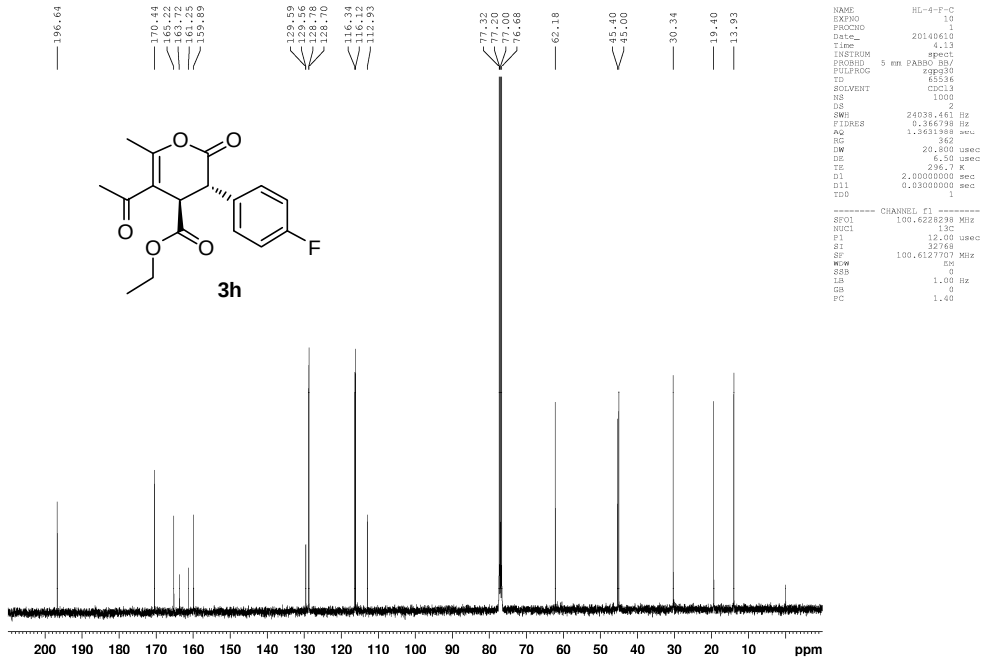

```
NAME          HL-ph-3-OMe-x
EXPNO                    11
PROCNO                    1
Date_          20140428
Time                 12.25
INSTRUM              spect
PROBHD   5 mm PABBO BA/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                    256
DW                62.400 usec
DE                 6.50 usec
TE                 292.5 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                   1H
P1                 11.60 usec
SI                  65536
SF              400.1300080 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



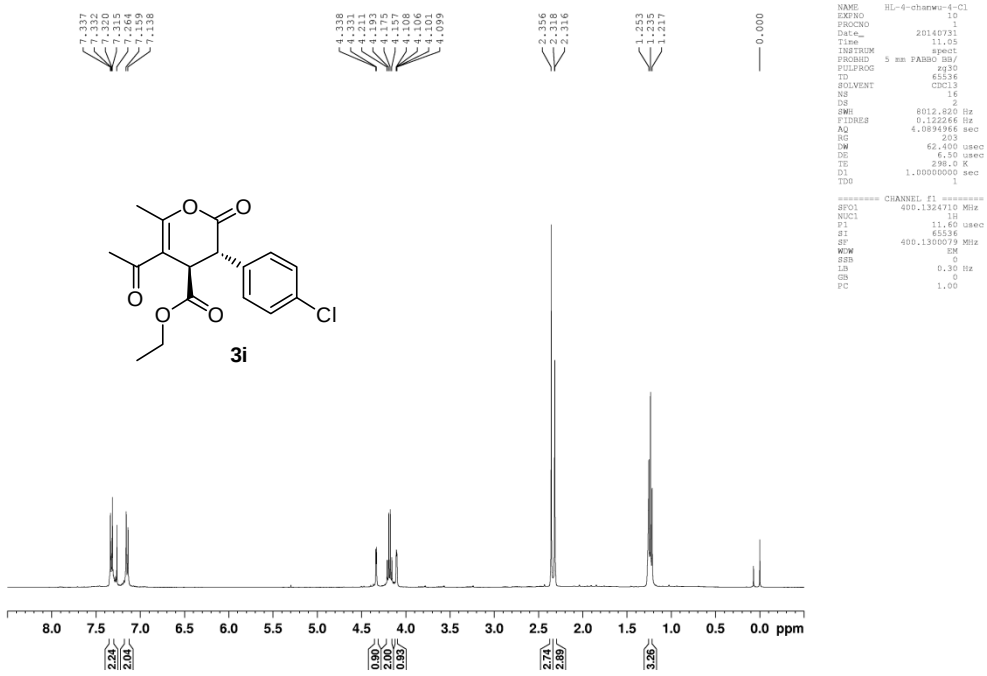

```
NAME          HL-chanwu-ph-3-OMe
EXPNO                    10
PROCNO                    1
Date_          20140430
Time                 13.05
INSTRUM              spect
PROBHD   5 mm PABBO BA/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    500
DS                      2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                   2050
DW                20.800 usec
DE                 6.50 usec
TE                 295.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

--------- CHANNEL f1 ---------
SFO1            100.6226298 MHz
NUC1                   13C
P1                 14.45 usec
SI                  32768
SF              100.6127714 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



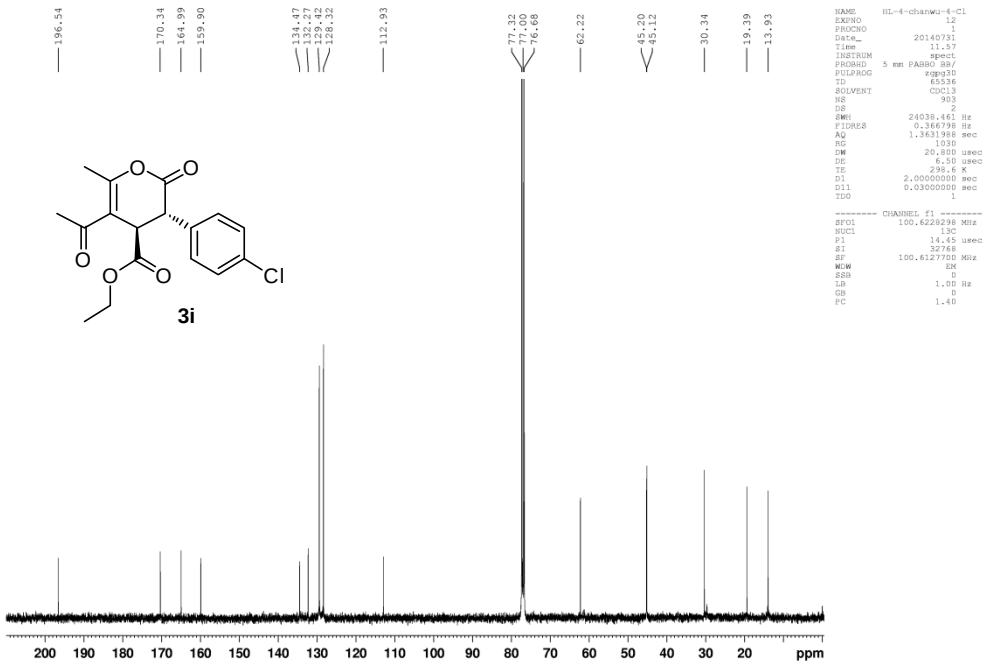

```
NAME          HL-4-chanwu-4-F H
EXPNO                       10
PROCNO                       1
Date_              20140604
Time                    21.27
INSTRUM                 spect
PROBHD     5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                          16
DS                           2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894966 sec
RG                         161
DW                  62.400 usec
DE                   6.50 usec
TE                  296.3 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1324710 MHz
NUC1                        1H
P1                  14.10 usec
SI                     65536
SF                  400.1300063 MHz
WDW                        EM
SSB                         0
LB                   0.30 Hz
GB                          0
PC                   1.00
```



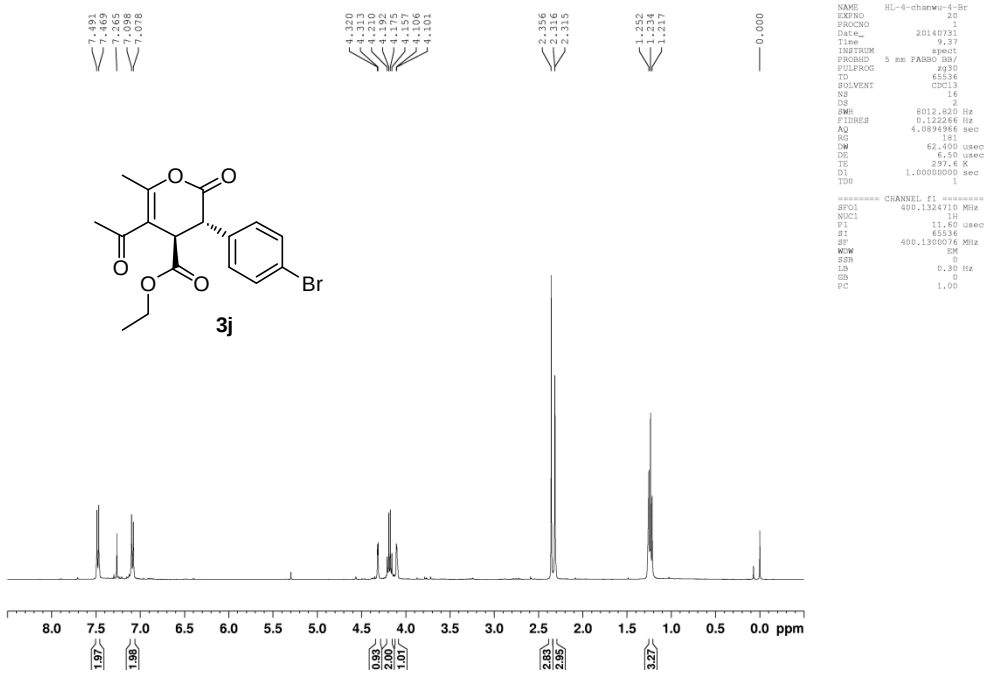

```
NAME                HL-4-F-C
EXPNO                     10
PROCNO                     1
Date_            20140610
Time                  4.13
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    1000
DS                       2
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                     362
DW                20.800 usec
DE                 6.50 usec
TE                296.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1              100.6228298 MHz
NUC1                      13C
P1                12.00 usec
SI                   32768
SF               100.6127707 MHz
WDW                      EM
SSB                       0
LB                  1.00 Hz
GB                        0
PC                  1.40
```



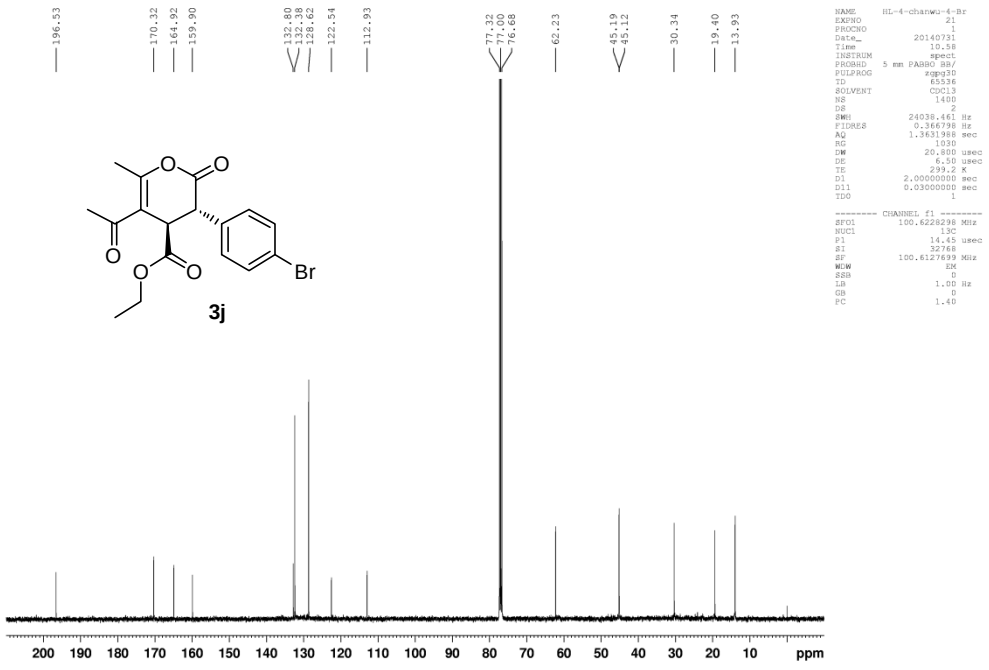

```
NAME          HL-4-chanwu-4-Cl
EXPNO                       10
PROCNO                       1
Date_              20140731
Time                    11.05
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                          16
DS                           2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894966 sec
RG                         203
DW                    62.400 usec
DE                     6.50 usec
TE                     298.0 K
D1                 1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               400.1324710 MHz
NUC1                       1H
P1                    11.60 usec
SI                       65536
SF                 400.1300079 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



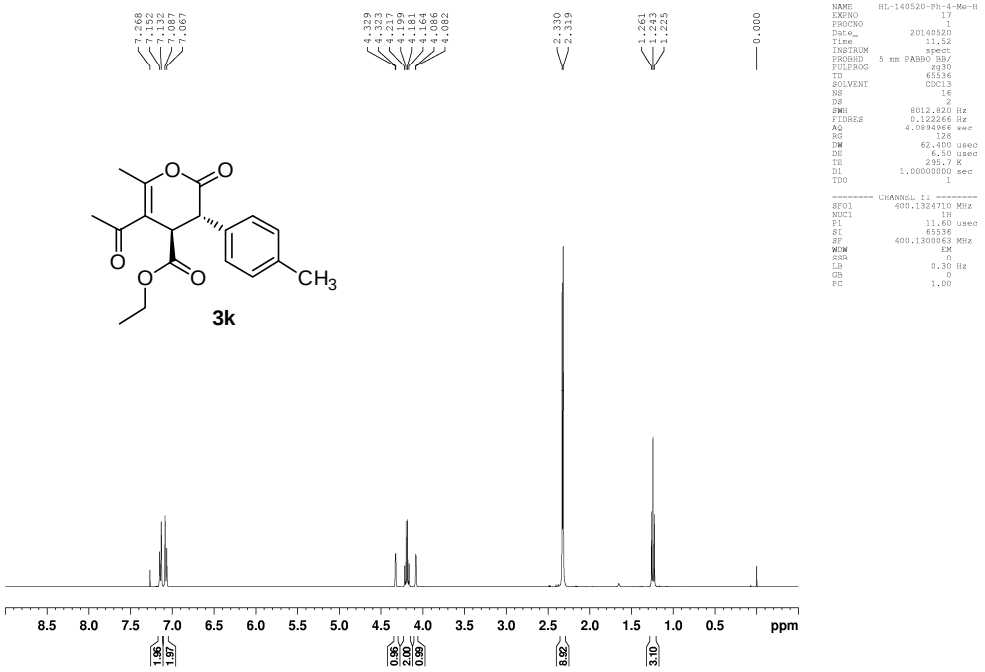

```
NAME          HL-4-chanwu-4-Cl
EXPNO                       12
PROCNO                       1
Date_              20140731
Time                    11.57
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                     65536
SOLVENT                CDCl3
NS                         903
DS                           2
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                        1030
DW                    20.800 usec
DE                     6.50 usec
TE                     298.6 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               100.6228298 MHz
NUC1                       13C
P1                    14.45 usec
SI                       32768
SF                 100.6127700 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



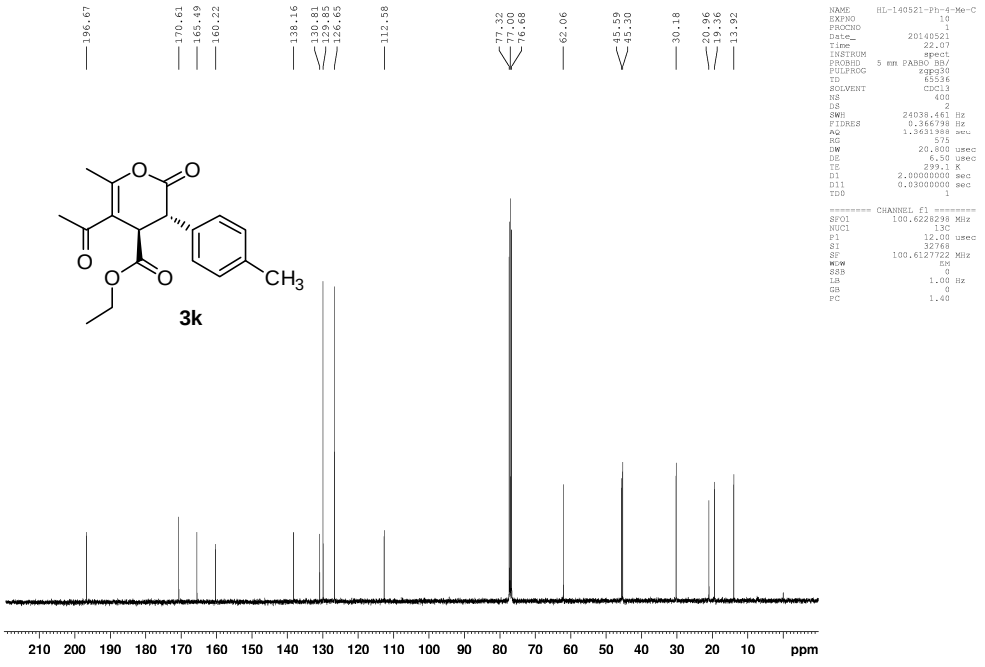

```
NAME          HL-4-chanwu-4-Br
EXPNO                   20
PROCNO                   1
Date_              20140731
Time                  9.37
INSTRUM               spect
PROBHD     5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                      181
DW                 62.400 usec
DE                  6.50 usec
TE                  297.6 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1324710 MHz
NUC1                     1H
P1                  11.60 usec
SI                    65536
SF               400.1300076 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                    1.00
```



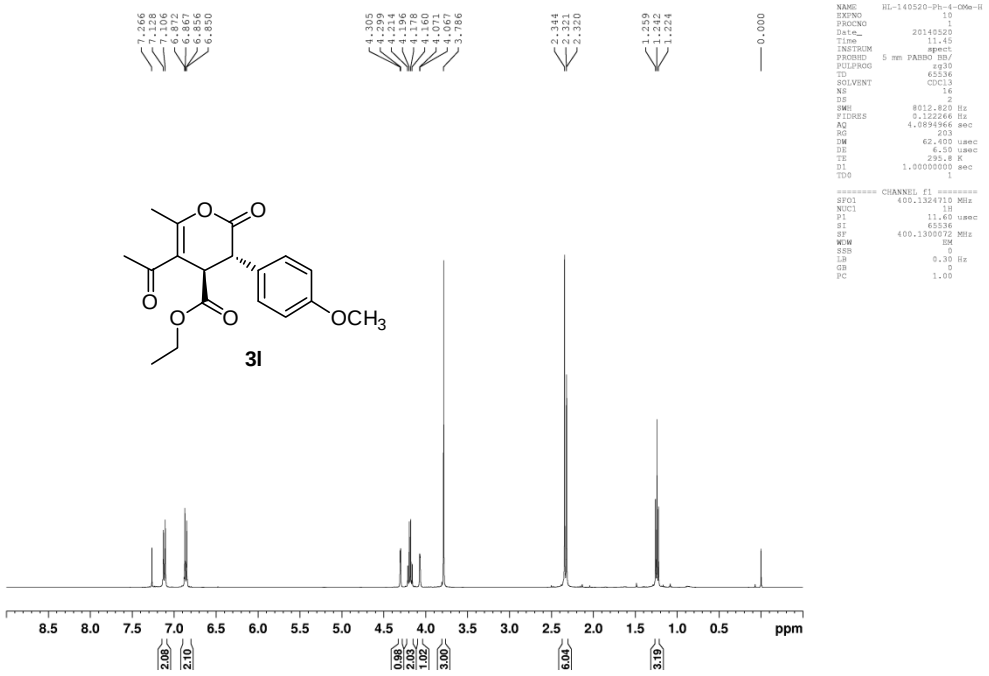

```
NAME          HL-4-chanwu-4-Br
EXPNO                   21
PROCNO                   1
Date_              20140731
Time                 10.58
INSTRUM               spect
PROBHD     5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1400
DS                        2
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      1030
DW                 20.800 usec
DE                  6.50 usec
TE                  299.2 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             100.6228298 MHz
NUC1                     13C
P1                  14.45 usec
SI                    32768
SF               100.6127699 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                    1.40
```



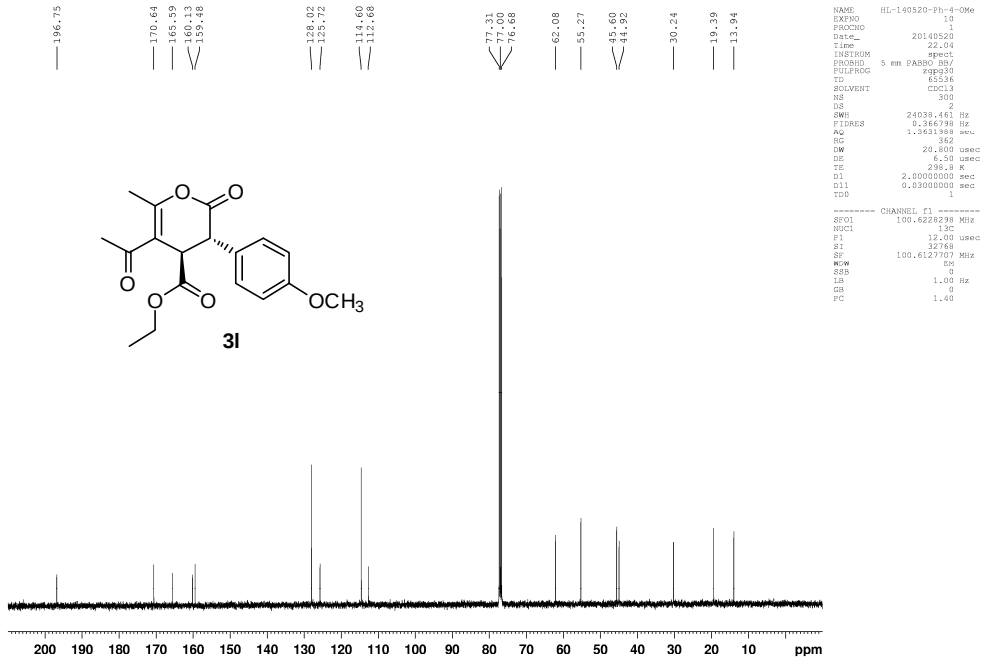

```
NAME          HL-140520-Ph-4-Me-H
EXPNO                          17
PROCNO                          1
Date_                    20140520
Time                        11.52
INSTRUM                      spect
PROBHD    5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8012.820 Hz
FIDRES                   0.122266 Hz
AQ                       4.0894966 sec
RG                            128
DW                         62.400 usec
DE                          6.50 usec
TE                         295.7 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                 400.1324710 MHz
NUC1                         1H
P1                         11.60 usec
SI                          65536
SF                   400.1300063 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



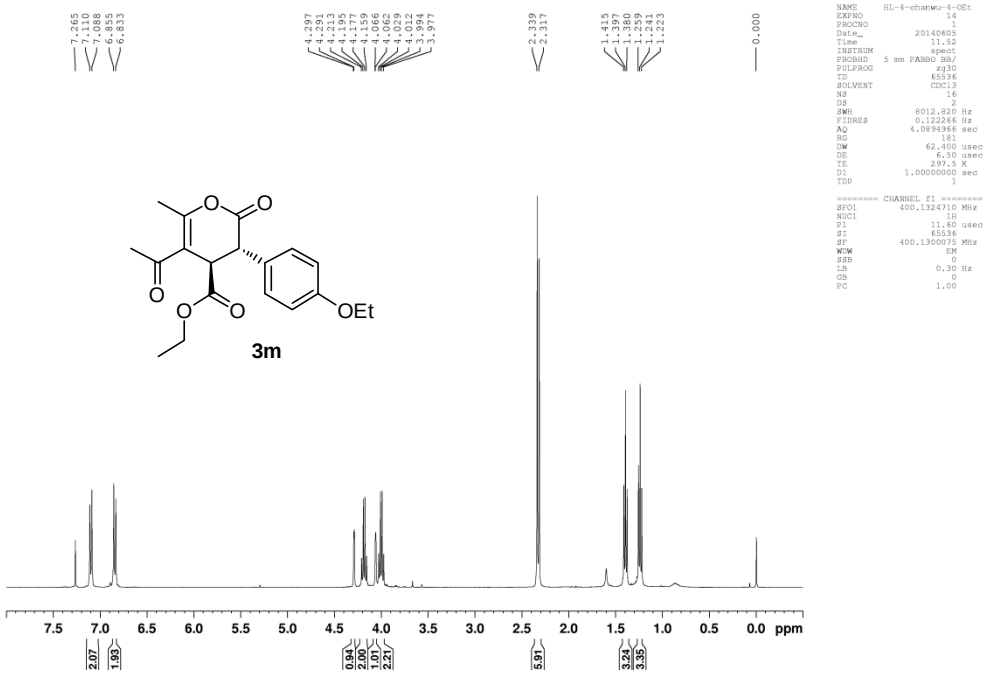

```
NAME          HL-140521-Ph-4-Me-C
EXPNO                          10
PROCNO                          1
Date_                    20140521
Time                        22.07
INSTRUM                      spect
PROBHD    5 mm PABBO BB/
PULPROG                   zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            400
DS                              2
SWH                     24038.461 Hz
FIDRES                   0.366798 Hz
AQ                       1.3631988 sec
RG                            575
DW                         20.800 usec
DE                          6.50 usec
TE                         299.1 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                 100.6228298 MHz
NUC1                         13C
P1                         12.00 usec
SI                          32768
SF                   100.6127722 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```



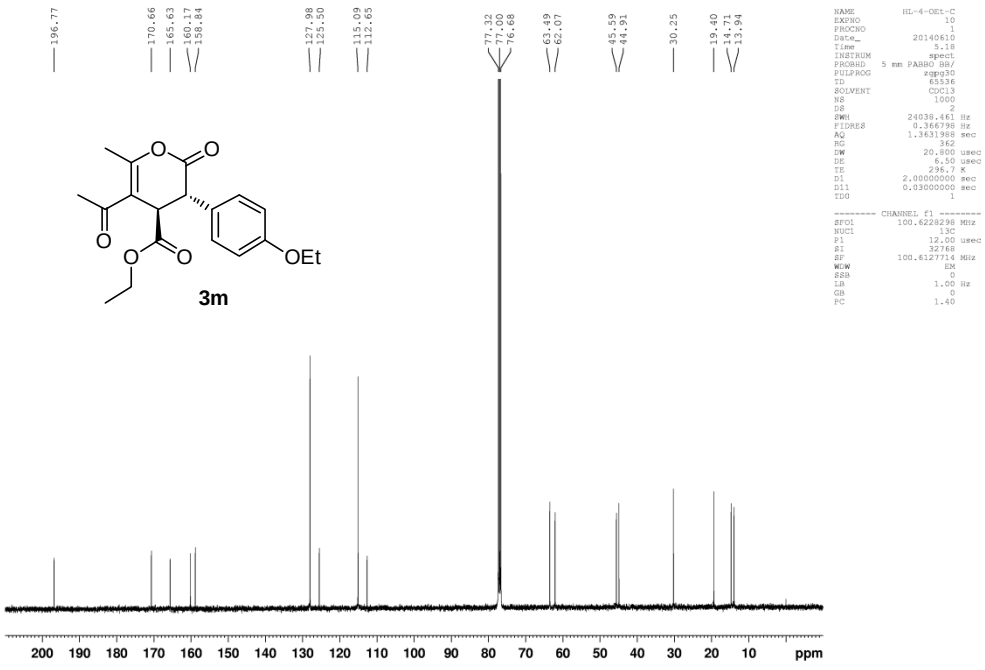

```
NAME          HL-140520-Ph-4-OMe-H
EXPNO                          10
PROCNO                          1
Date_                    20140520
Time                        11.45
INSTRUM                     spect
PROBHD       5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                    8012.820 Hz
FIDRES                 0.122266 Hz
AQ                     4.0894966 sec
RG                           203
DW                        62.400 usec
DE                         6.50 usec
TE                        295.8 K
D1                     1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                       1H
P1                      11.60 usec
SI                      65536
SF             400.1300072 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```



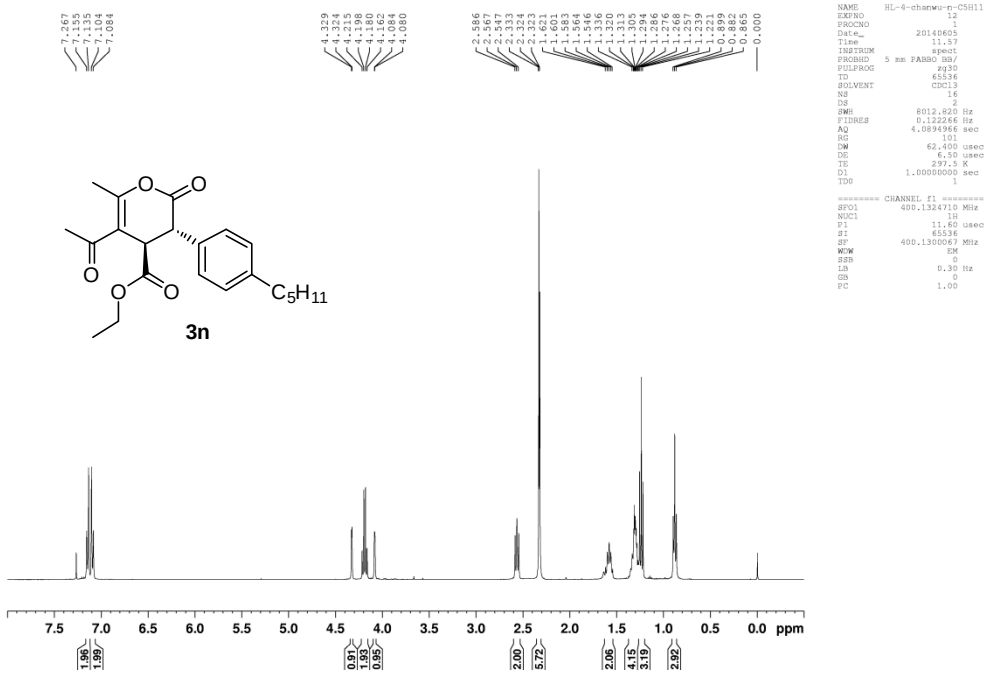

```
NAME          HL-140520-Ph-4-OMe
EXPNO                          10
PROCNO                          1
Date_                    20140520
Time                        22.04
INSTRUM                     spect
PROBHD       5 mm PABBO BB/
PULPROG                   zgpg30
TD                         65536
SOLVENT                    CDCl3
NS                           300
DS                             2
SWH                   24038.461 Hz
FIDRES                 0.366798 Hz
AQ                     1.3631988 sec
RG                           362
DW                        20.800 usec
DE                         6.50 usec
TE                        298.8 K
D1                     2.00000000 sec
D11                    0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1           100.6228298 MHz
NUC1                       13C
P1                      12.00 usec
SI                      32768
SF             100.6127707 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



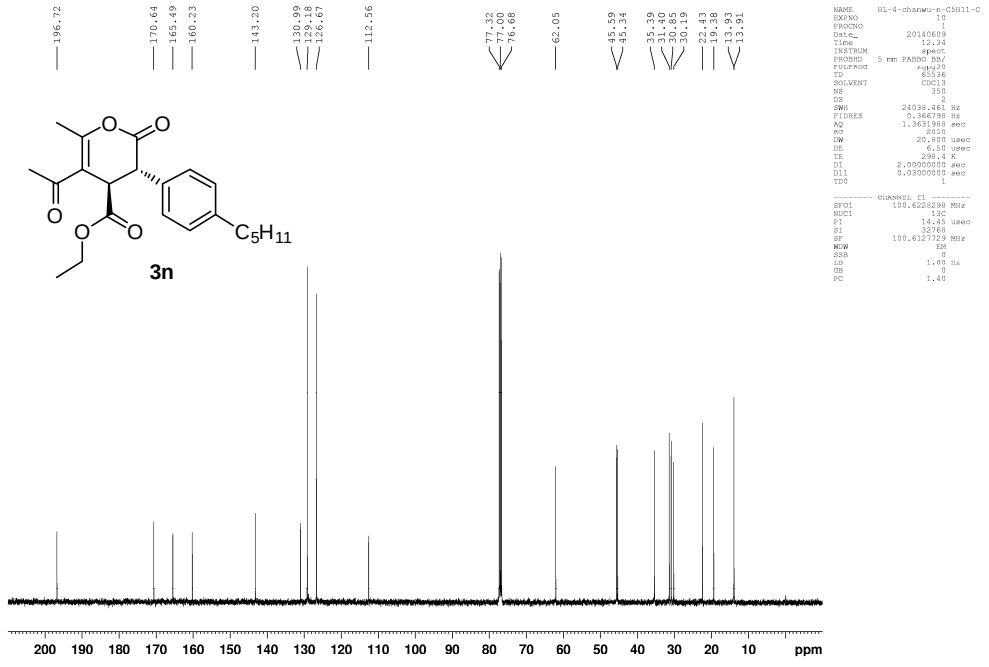

```
NAME          HL-4-chanwu-4-OEt
EXPNO                        14
PROCNO                        1
Date_               20140605
Time                     11.52
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           16
DS                            2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894966 sec
RG                          181
DW                   62.400 usec
DE                    6.50 usec
TE                    297.5 K
D1                  1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1              400.1324710 MHz
NUC1                        1H
P1                   11.60 usec
SI                       65536
SF                400.1300075 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                    1.00
```



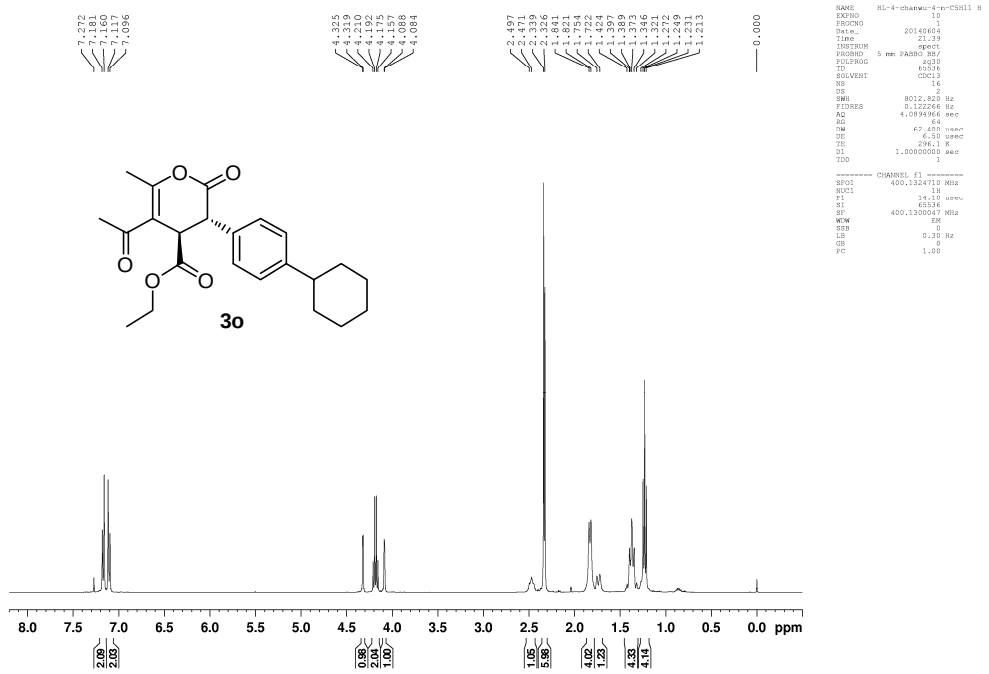

```
NAME          HL-4-OEt-C
EXPNO                        10
PROCNO                        1
Date_               20140610
Time                      5.18
INSTRUM                  spect
PROBHD     5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                        1000
DS                            2
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                          362
DW                   20.800 usec
DE                    6.50 usec
TE                    296.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1              100.6228298 MHz
NUC1                        13C
P1                   12.00 usec
SI                       32768
SF                100.6127714 MHz
WDW                         EM
SSB                          0
LB                    1.00 Hz
GB                           0
PC                    1.40
```



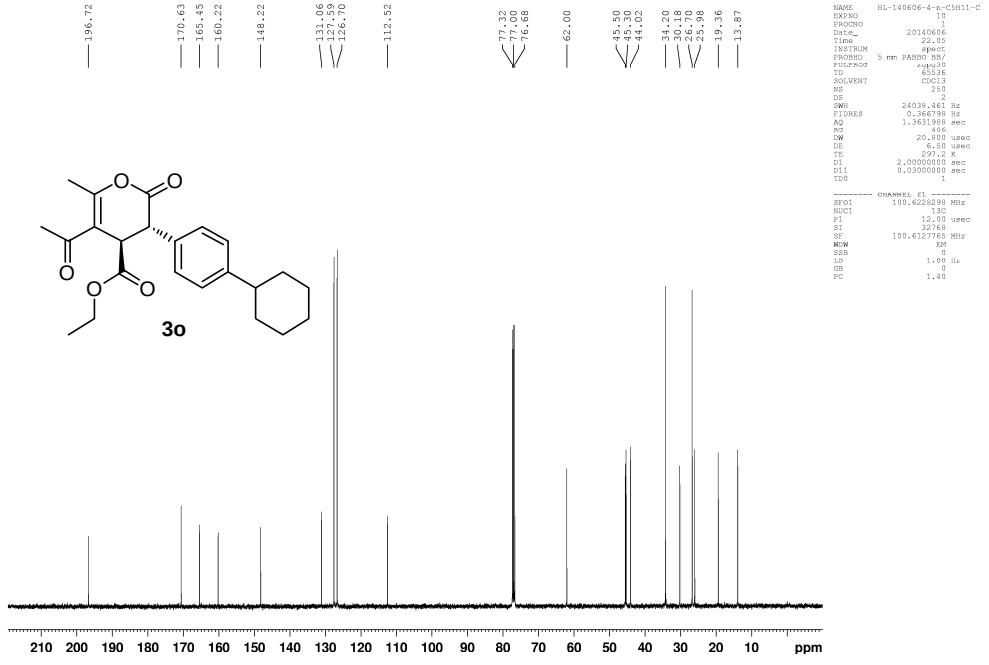

```
NAME          HL-4-chanwu-n-C5H11
EXPNO                      12
PROCNO                      1
Date_                20140605
Time                    11.57
INSTRUM                 spect
PROBHD     5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         16
DS                          2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894966 sec
RG                        101
DW                   62.400 usec
DE                    6.50 usec
TE                   297.5 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1              400.1324710 MHz
NUC1                      1H
P1                    11.60 usec
SI                     65536
SF                400.1300067 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```



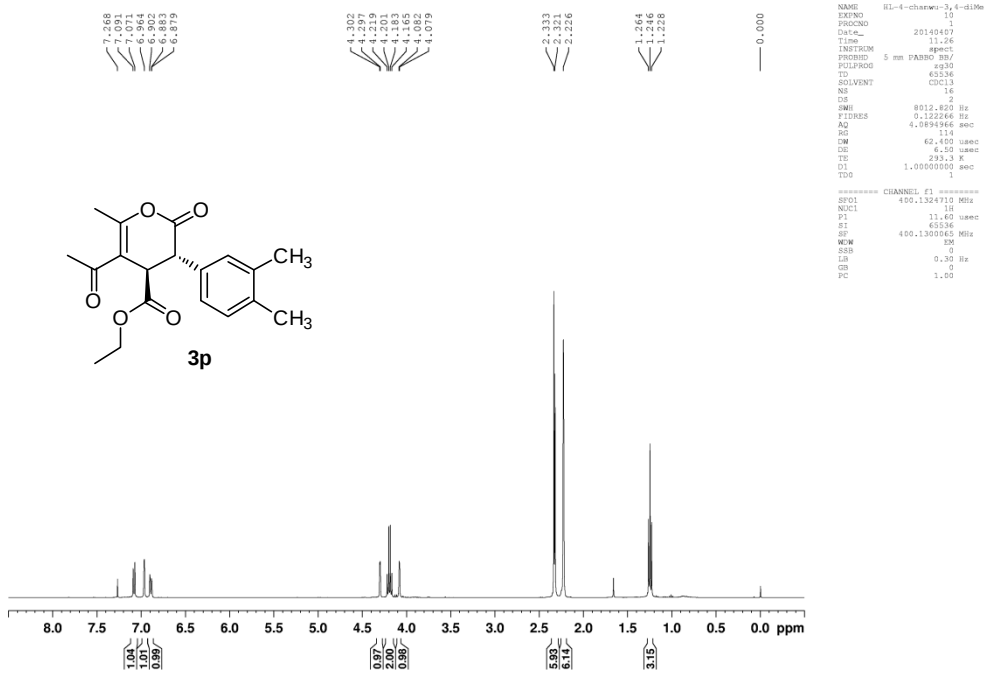

```
NAME          HL-4-chanwu-n-C5H11-C
EXPNO                      10
PROCNO                      1
Date_                20140609
Time                    12.34
INSTRUM                 spect
PROBHD     5 mm PABBO BB/
PULPROG                 cpg30
TD                     65536
SOLVENT                CDCl3
NS                        350
DS                          2
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                       2828
DW                   20.800 usec
DE                    6.50 usec
TE                   296.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1              100.6228298 MHz
NUC1                      13C
P1                    14.45 usec
SI                     32768
SF                100.6127729 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                      1.40
```



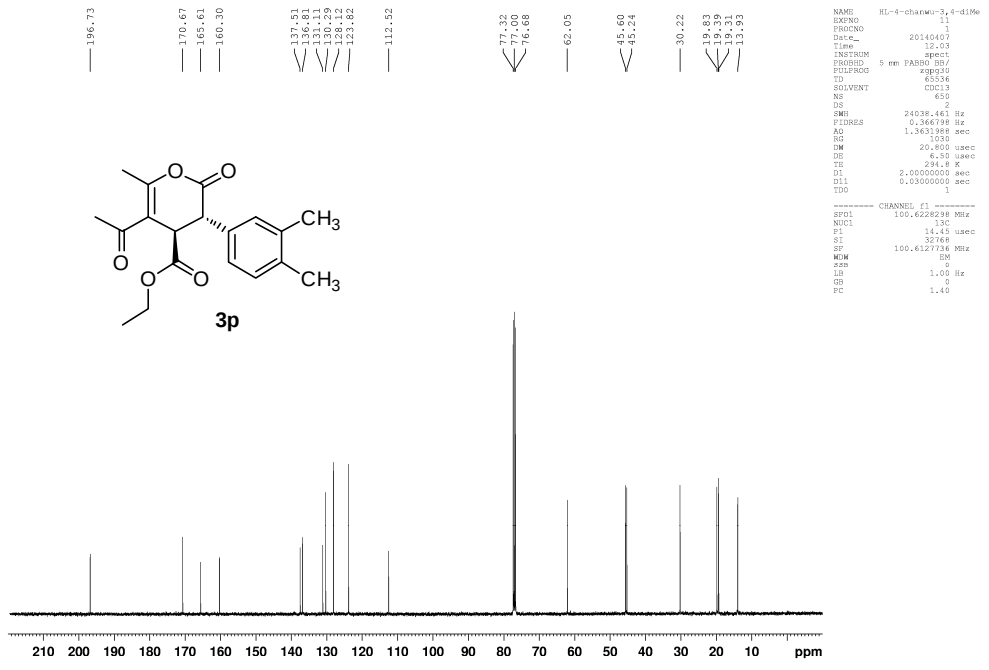

```
NAME          HL-4-chanwu-4-n-C5H11 H
EXPNO                 10
PROCNO                 1
Date_           20140604
Time                21.39
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                    64
DW                62.400 usec
DE                 6.50 usec
TE                296.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                   1H
P1                  14.10 usec
SI                 65536
SF             400.1300047 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



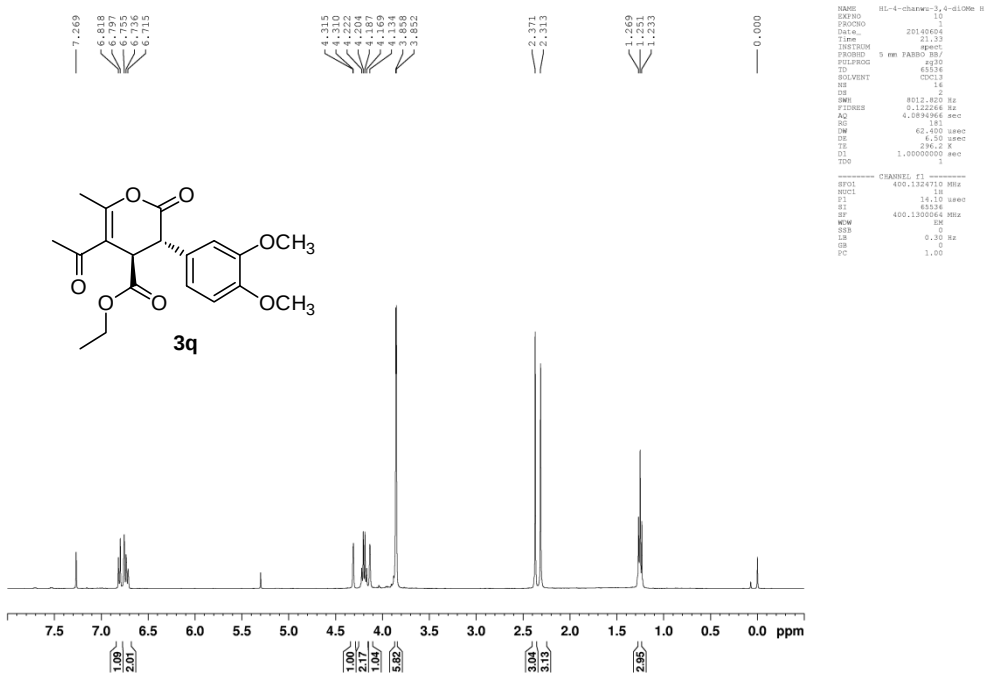

```
NAME          HL-140606-4-n-C5H11-C
EXPNO                 10
PROCNO                 1
Date_           20140606
Time                22.05
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   250
DS                     2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                   406
DW                20.800 usec
DE                 6.50 usec
TE                297.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1           100.6228298 MHz
NUC1                   13C
P1                 12.00 usec
SI                 32768
SF             100.6127765 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



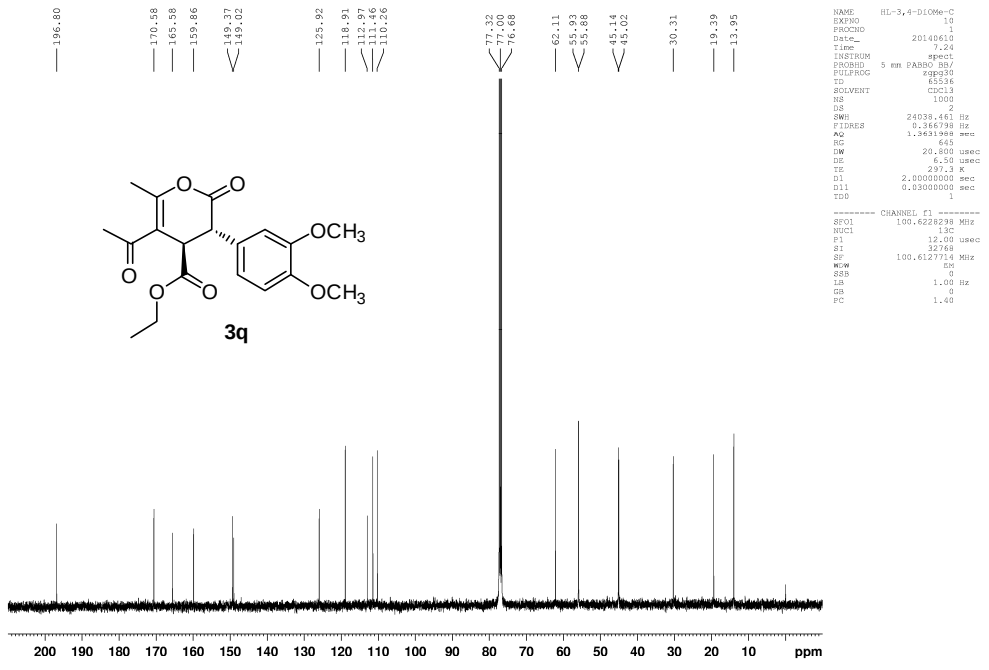

```
NAME          HL-4-chanwu-3,4-diMe
EXPNO                           10
PROCNO                           1
Date_                     20140407
Time                         11.26
INSTRUM                      spect
PROBHD     5 mm PABBO BB/
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                       8012.820 Hz
FIDRES                    0.122266 Hz
AQ                        4.0894966 sec
RG                            114
DW                         62.400 usec
DE                          6.50 usec
TE                         293.3 K
D1                    1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                  400.1324710 MHz
NUC1                           1H
P1                          11.60 usec
SI                          65536
SF                    400.1300065 MHz
WDW                            EM
SSB                             0
LB                           0.30 Hz
GB                              0
PC                           1.00
```



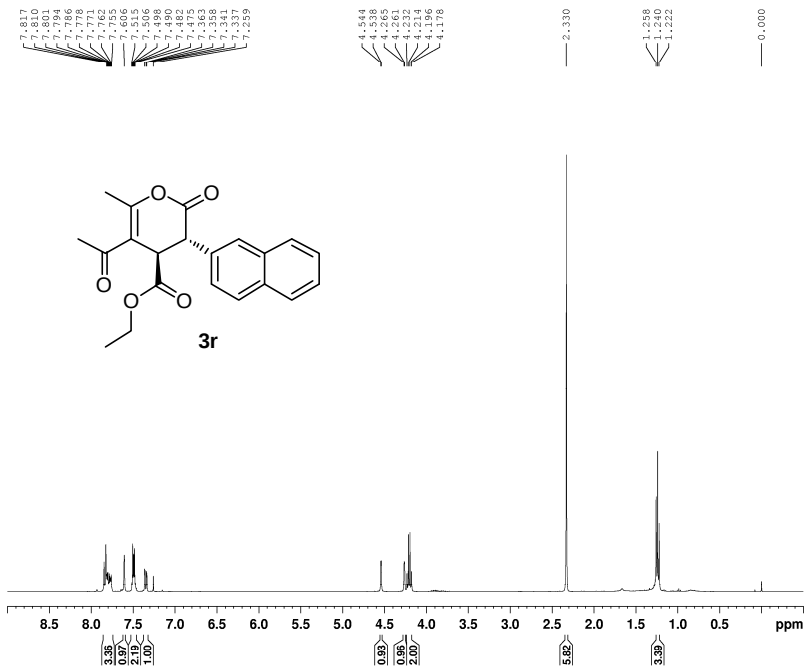

```
NAME          HL-4-chanwu-3,4-diMe
EXPNO                           11
PROCNO                           1
Date_                     20140407
Time                         12.03
INSTRUM                      spect
PROBHD     5 mm PABBO BB/
PULPROG                   zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                             650
DS                              2
SWH                       24038.461 Hz
FIDRES                    0.366798 Hz
AQ                        1.3631988 sec
RG                            1030
DW                         20.800 usec
DE                          6.50 usec
TE                         294.8 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                  100.6228298 MHz
NUC1                           13C
P1                          14.45 usec
SI                          32768
SF                    100.6127736 MHz
WDW                            EM
SSB                             0
LB                           1.00 Hz
GB                              0
PC                           1.40
```



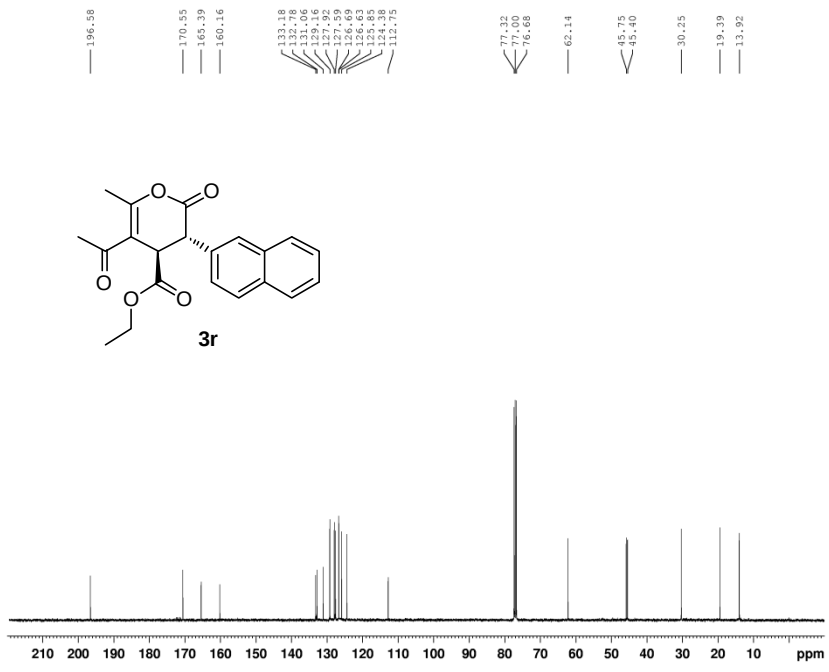

```
NAME        HL-4-chanwu-3,4-diOMe H
EXPNO                 10
PROCNO                 1
Date_           20140604
Time                21.33
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                   181
DW                62.400 usec
DE                 6.50 usec
TE                296.2 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                   1H
P1                  14.10 usec
PL1                 65536
SF              400.1300064 MHz
WDW                    EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



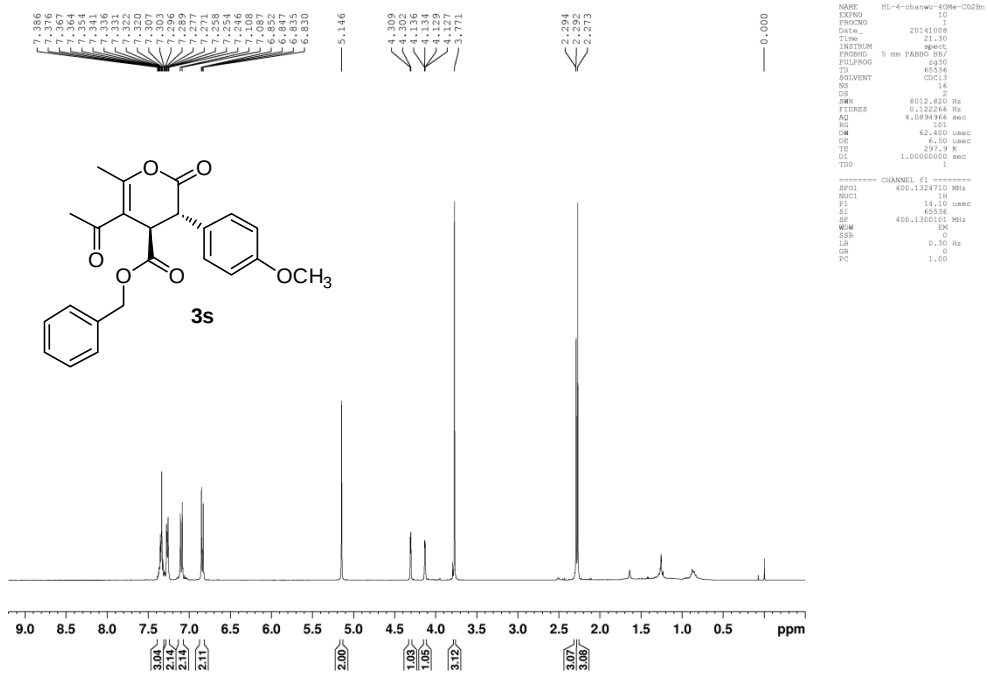

```
NAME        HL-3,4-diOMe-C
EXPNO                 10
PROCNO                 1
Date_           20140610
Time                 7.24
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  1000
DS                     2
SWH              24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                   645
DW                20.800 usec
DE                 6.50 usec
TE                297.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                   13C
P1                  12.00 usec
PL1                 32768
SF              100.6127714 MHz
WDW                    EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



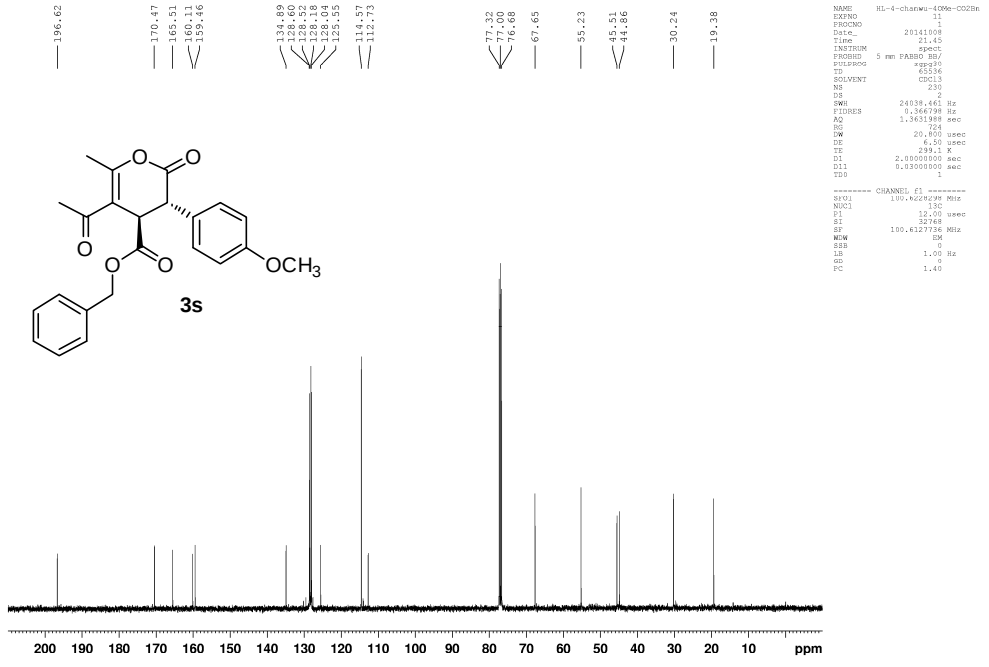

```
NAME        HL-4-chanwu-2naph
EXPNO                 10
PROCNO                 1
Date_          20140411
Time                3.11
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                  90.5
DW                62.400 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 11.60 usec
SI                 65536
SF            400.1300099 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



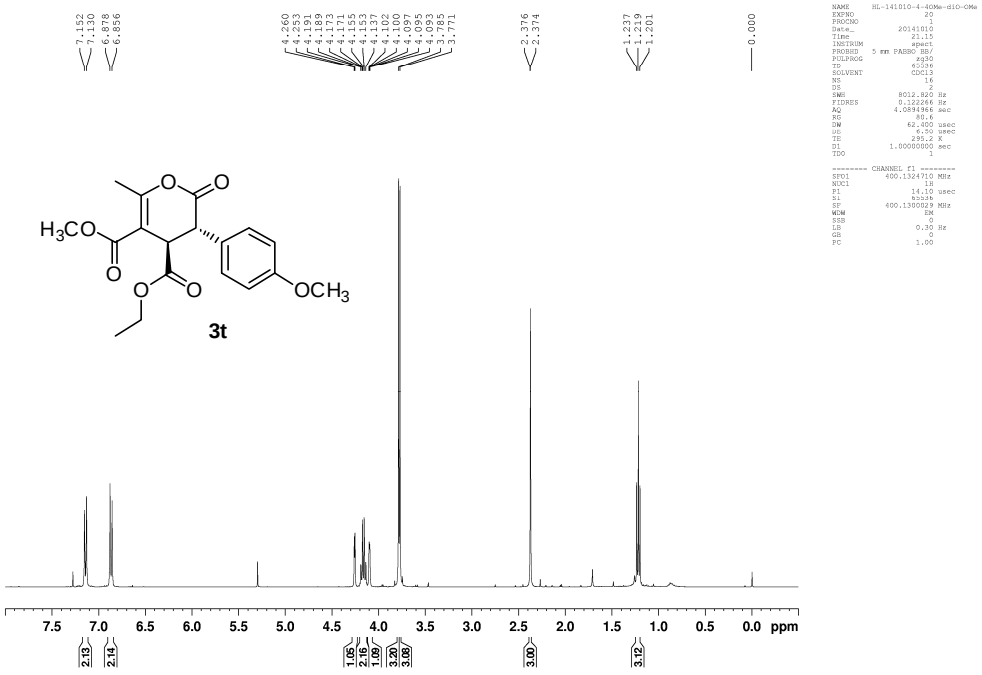

```
NAME        HL-4-chanwu-2naph
EXPNO                 11
PROCNO                 1
Date_          20140411
Time                3.38
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   450
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  1030
DW                20.800 usec
DE                 6.50 usec
TE                296.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 14.45 usec
SI                 32768
SF            100.6127751 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



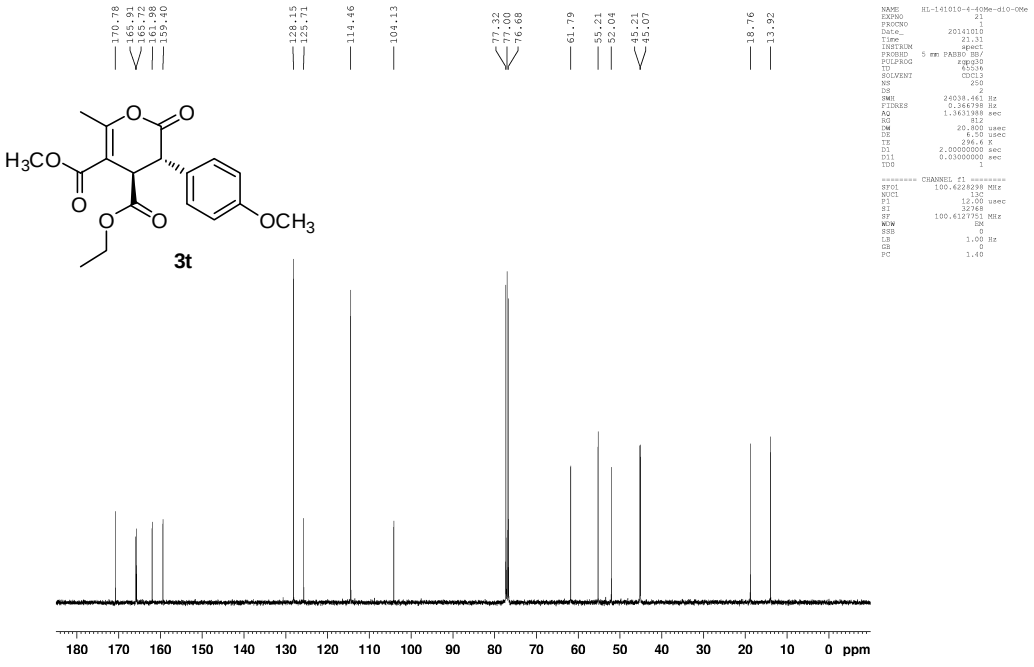

```
NAME          HL-4-chanwu-40Me-CO2Bn
EXPNO                 10
PROCNO                 1
Date_           20141008
Time                21.30
INSTRUM              spect
PROBHD     5 mm PABBO BB/
PULPROG          zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                  101
DW                62.400 usec
DE                 6.50 usec
TE                297.9 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                   1H
P1                 14.10 usec
SI                 65536
SF              400.1300101 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



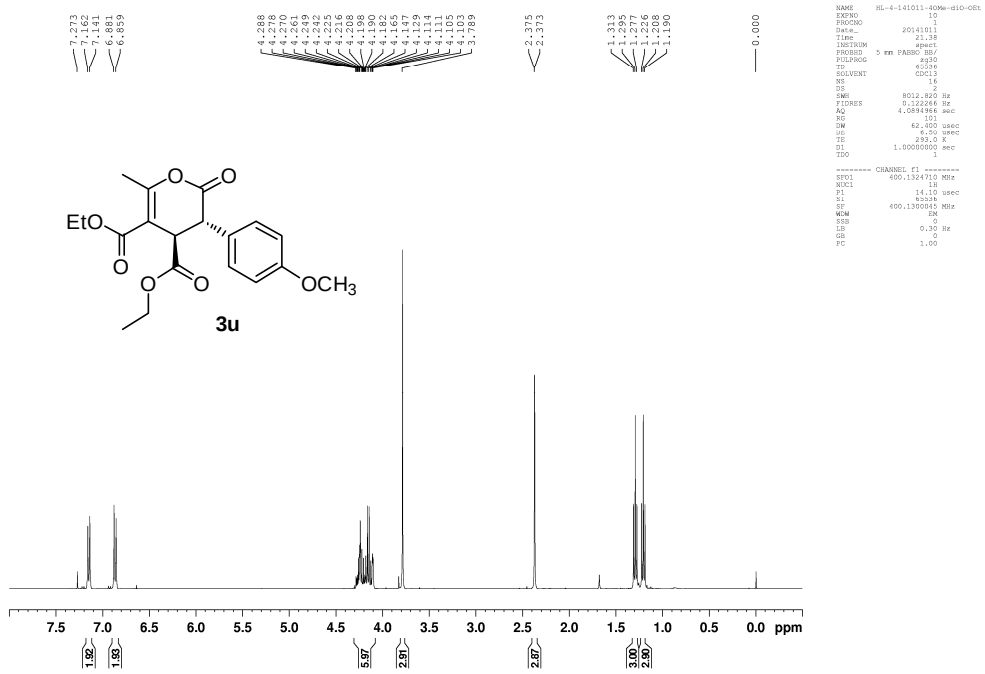

```
NAME          HL-4-chanwu-40Me-CO2Bn
EXPNO                 11
PROCNO                 1
Date_           20141008
Time                21.45
INSTRUM              spect
PROBHD     5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  230
DS                    2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                  724
DW                20.800 usec
DE                 6.50 usec
TE                299.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                   13C
P1                 12.00 usec
SI                 32768
SF              100.6127736 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



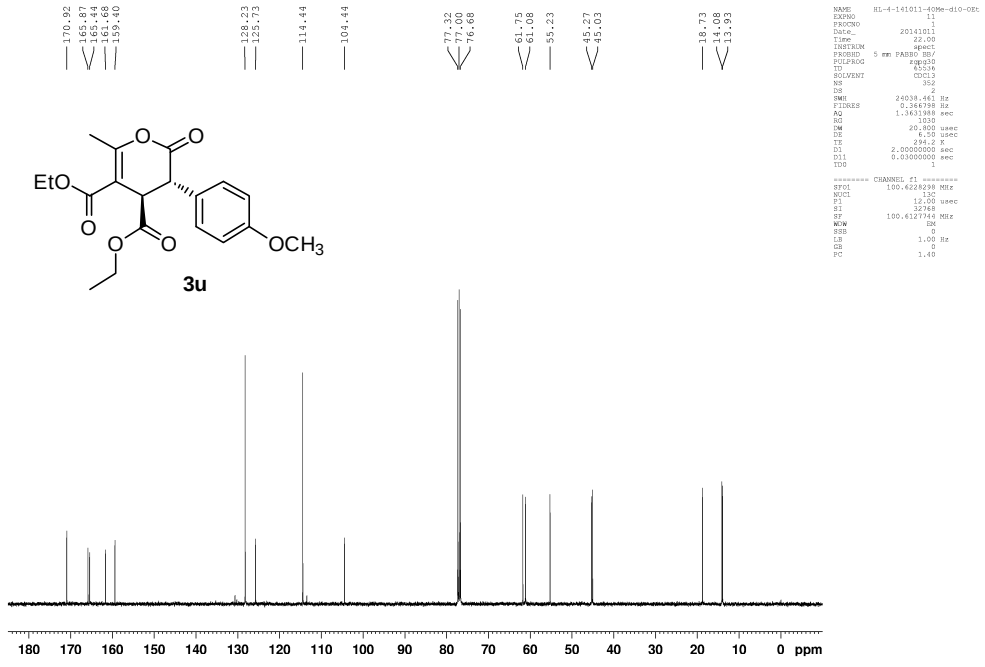

```
NAME        HL-141010-4-4OMe-di0-OMe
EXPNO               20
PROCNO               1
Date_         20141010
Time               21.15
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894966 sec
RG                80.6
DW              62.400 usec
DE               6.50 usec
TE             295.2 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                14.10 usec
PLW1             50.00 W
SF          400.1300029 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



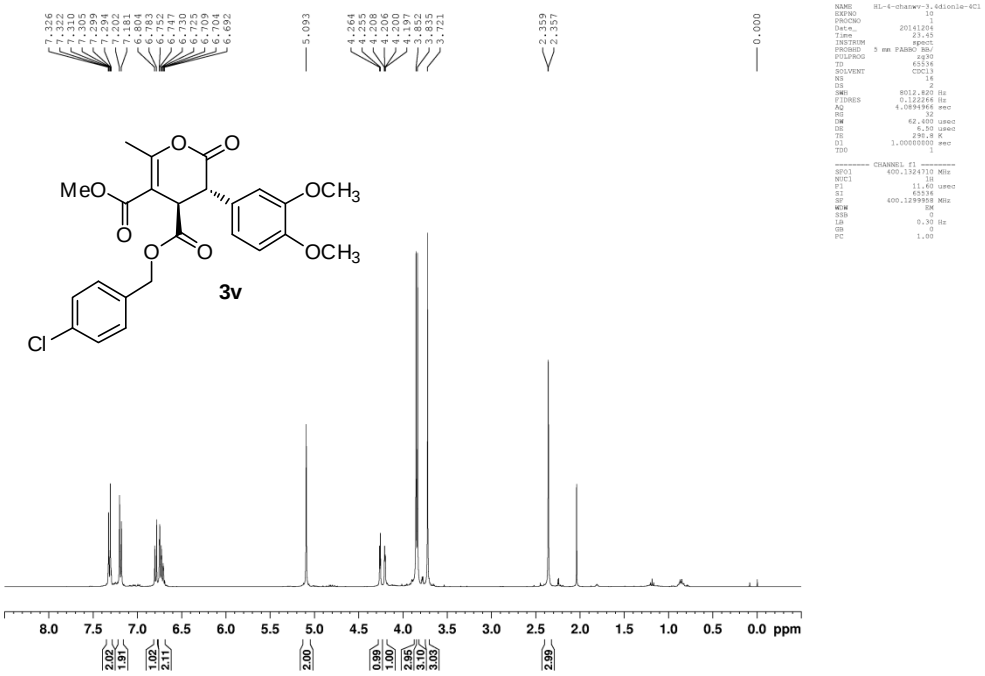

```
NAME        HL-141010-4-4OMe-di0-OMe
EXPNO               21
PROCNO               1
Date_         20141010
Time               21.31
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD              65536
SOLVENT          CDCl3
NS                 250
DS                   2
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                812
DW              20.800 usec
DE               6.50 usec
TE             296.6 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        100.6228298 MHz
NUC1                13C
P1                12.00 usec
SI                32768
SF          100.6127751 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```




```
NAME        HL-4-141011-4OMe-di0-OEt
EXPNO                 10
PROCNO                 1
Date_          20141011
Time                21.38
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                   101
DW                62.400 usec
DE                 6.50 usec
TE                293.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 14.10 usec
PL1                0.00 dB
SF            400.1300045 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




```
NAME        HL-4-141011-4OMe-di0-OEt
EXPNO                 11
PROCNO                 1
Date_          20141011
Time                22.00
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   352
DS                     2
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  1030
DW                20.800 usec
DE                 6.50 usec
TE                294.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 12.00 usec
SI                 32768
SF            100.6127744 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```


7.326
7.323
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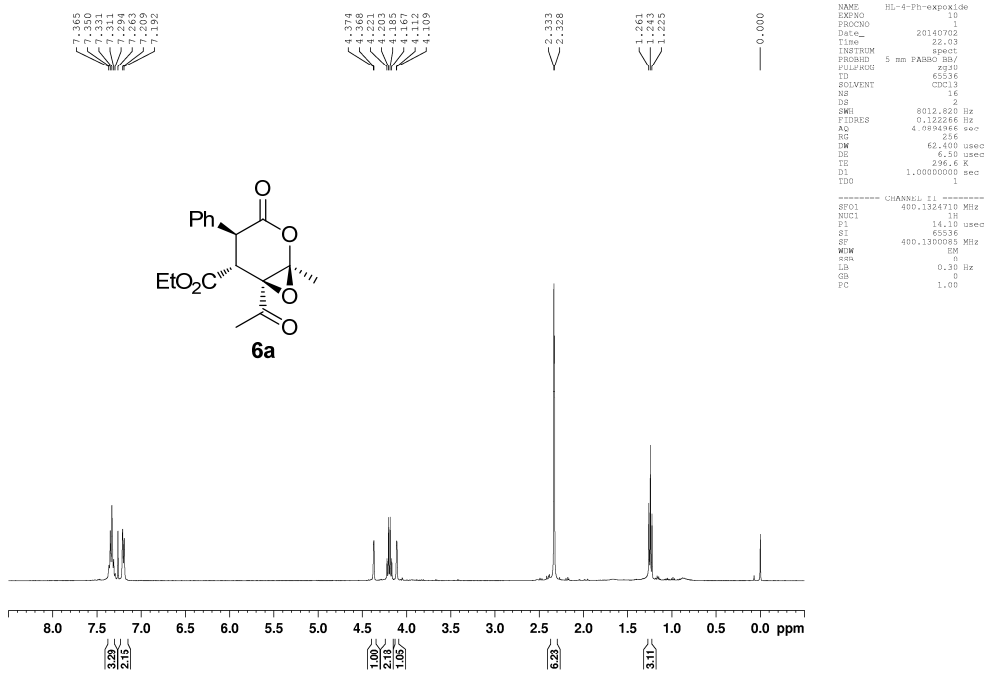
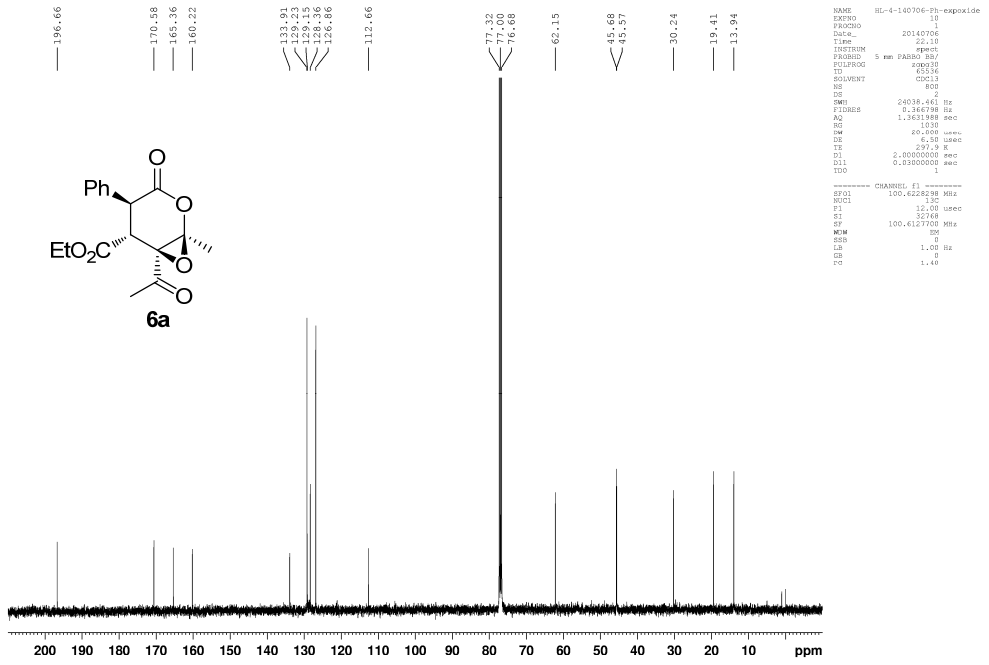
7.294
7.292
7.181
6.804
6.783
6.752
6.737
6.730
6.725
6.709
6.704
6.692
5.093
4.264
4.255
4.208
4.206
4.200
4.197
3.852
3.835
3.721
2.359
2.357
0.000
MeO
OCH3
OCH3
Cl
3v
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
2.02
1.91
1.02
2.11
2.00
0.99
1.00
2.95
3.10
3.03
2.99

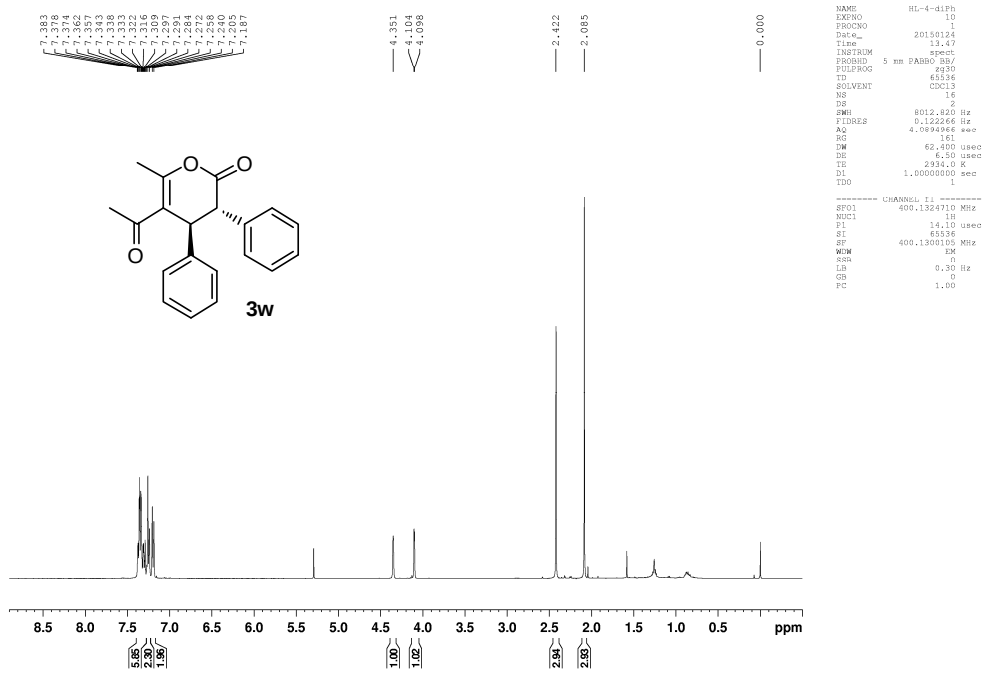

170.46
165.69
165.46
161.91
149.13
148.87
134.17
133.47
129.32
128.60
125.91
119.01
111.18
110.24
104.07
77.32
77.00
76.68
66.44
55.72
55.70
52.01
45.21
44.68
18.70
MeO
OCH3
OCH3
Cl
3v
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm



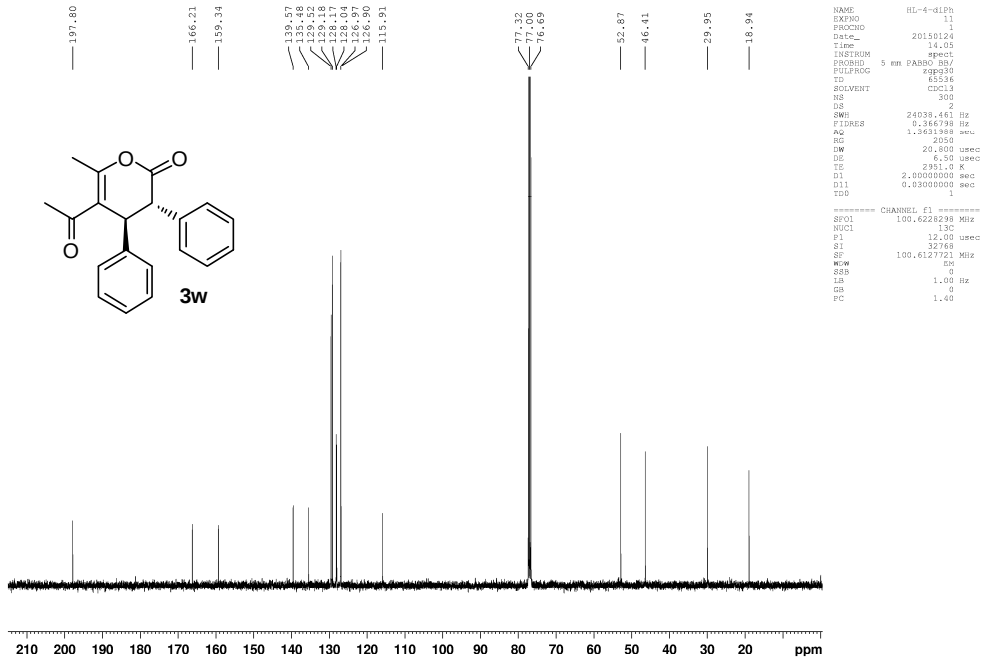

```
NAME        HL-4-Ph-expoxide
EXPNO                 10
PROCNO                 1
Date_           20140702
Time                22.03
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                   256
DW                62.400 usec
DE                 6.50 usec
TE                296.6 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                   1H
P1                  14.10 usec
SI                 65536
SF              400.1300085 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



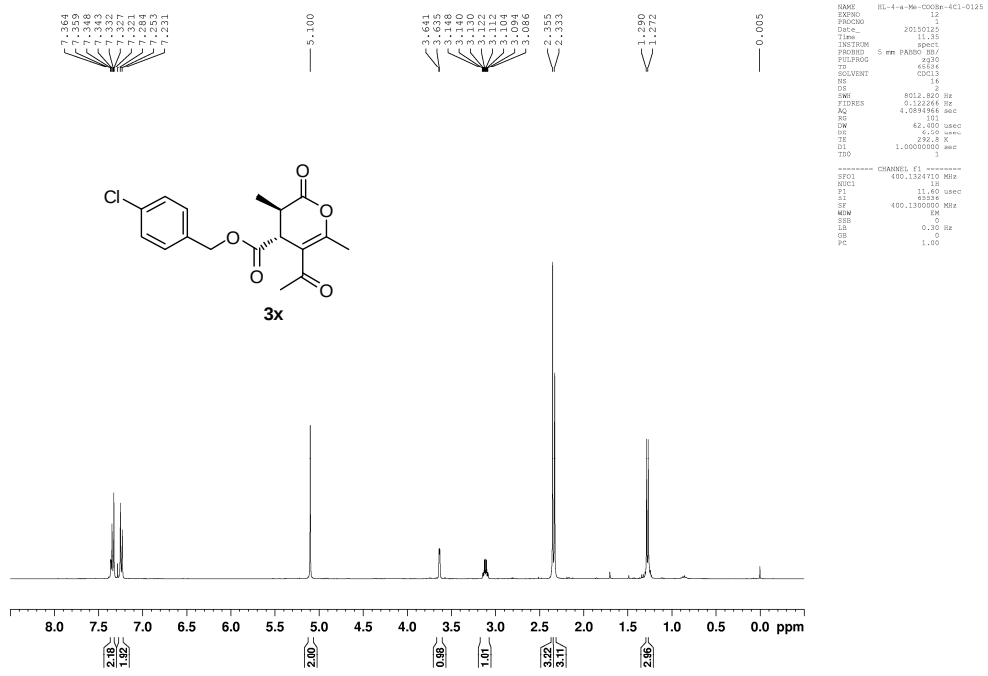

```
NAME        HL-4-140706-Ph-expoxide
EXPNO                 10
PROCNO                 1
Date_           20140706
Time                22.10
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   800
DS                     2
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                  1030
DW                20.000 usec
DE                 6.50 usec
TE                297.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1            100.6228298 MHz
NUC1                   13C
P1                  12.00 usec
SI                 32768
SF              100.6127700 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



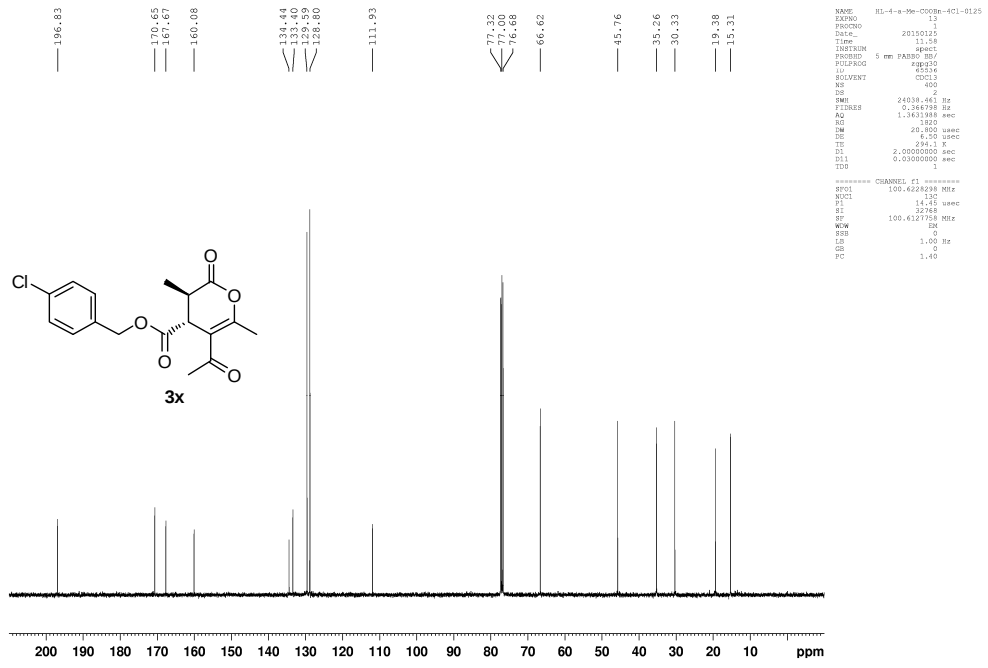

```
NAME            HL-4-diPh
EXPNO                  10
PROCNO                  1
Date_           20150124
Time                13.47
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                   161
DW                62.400 usec
DE                 6.50 usec
TE               2934.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 14.10 usec
SI                 65536
SF            400.1300105 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```




```
NAME            HL-4-diPh
EXPNO                  11
PROCNO                  1
Date_           20150124
Time                14.05
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   300
DS                     2
SWH             24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631988 sec
RG                  2050
DW                20.800 usec
DE                 6.50 usec
TE               2951.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          100.6228298 MHz
NUC1                  13C
P1                 12.00 usec
SI                 32768
SF            100.6127721 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

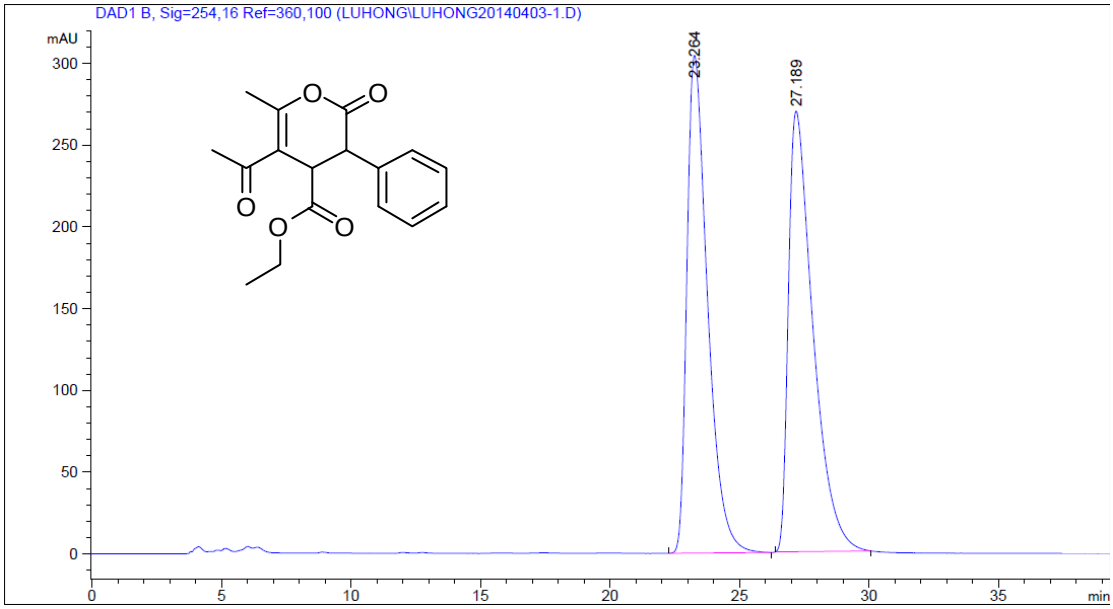



```
NAME        HL-4-a-Me-COOBn-4Cl-0125
EXPNO              12
PROCNO              1
Date_        20150125
Time            11.35
INSTRUM          spect
PROBHD  5 mm PABBO BB/
PULPROG         zg30
TD             65536
SOLVENT        CDCl3
NS                 16
DS                  2
SWH          8012.820 Hz
FIDRES       0.122266 Hz
AQ           4.0894966 sec
RG                101
DW            62.400 usec
DE             6.50 usec
TE           292.8 K
D1           1.00000000 sec
TD0                 1

======== CHANNEL f1 ========
SFO1       400.1324710 MHz
NUC1               1H
P1              11.60 usec
SI                65536
SF         400.1300000 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              1.00
```

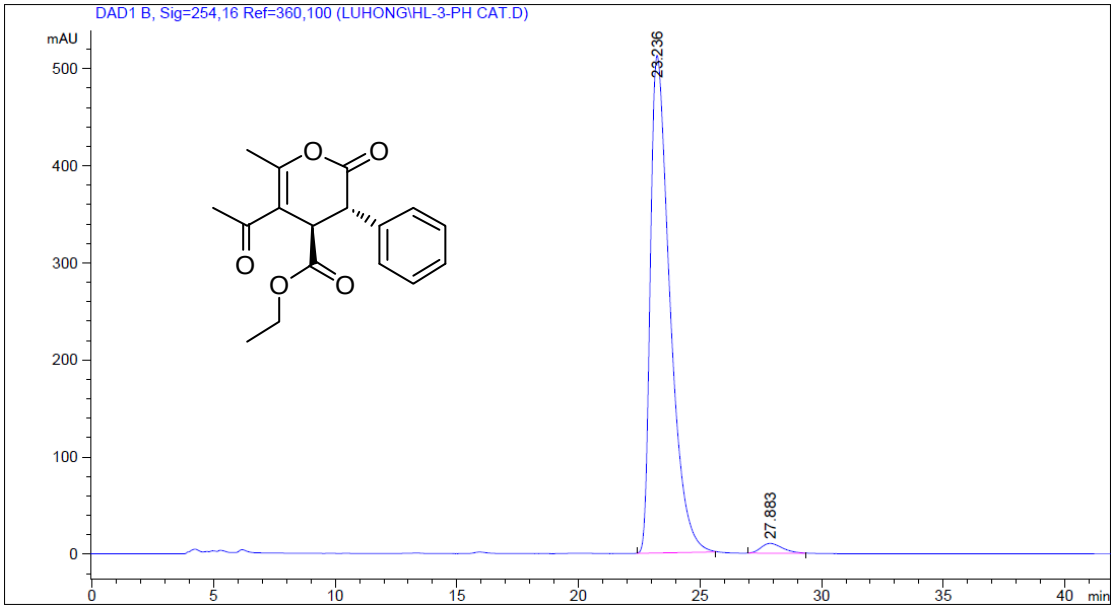



```
NAME        HL-4-a-Me-COOBn-4Cl-0125
EXPNO              13
PROCNO              1
Date_        20150125
Time            11.58
INSTRUM          spect
PROBHD  5 mm PABBO BB/
PULPROG       zgpg30
TD             65536
SOLVENT        CDCl3
NS                400
DS                  2
SWH         24038.461 Hz
FIDRES       0.366798 Hz
AQ           1.3631988 sec
RG                1820
DW            20.800 usec
DE             6.50 usec
TE           294.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
SFO1       100.6228298 MHz
NUC1              13C
P1              14.45 usec
SI                32768
SF         100.6127758 MHz
WDW               EM
SSB                0
LB              1.00 Hz
GB                 0
PC              1.40
```

3a

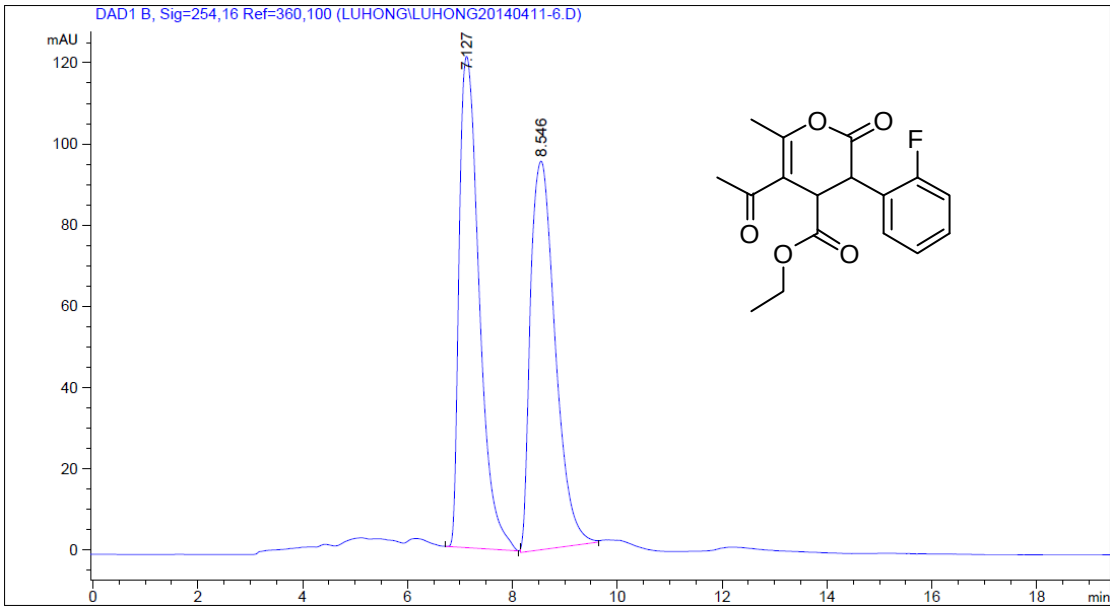


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	23.264	1.67971e4	304.28860	49.6592
2	DAD 254, 16 nm	27.189	1.77228e4	269.36551	50.3408

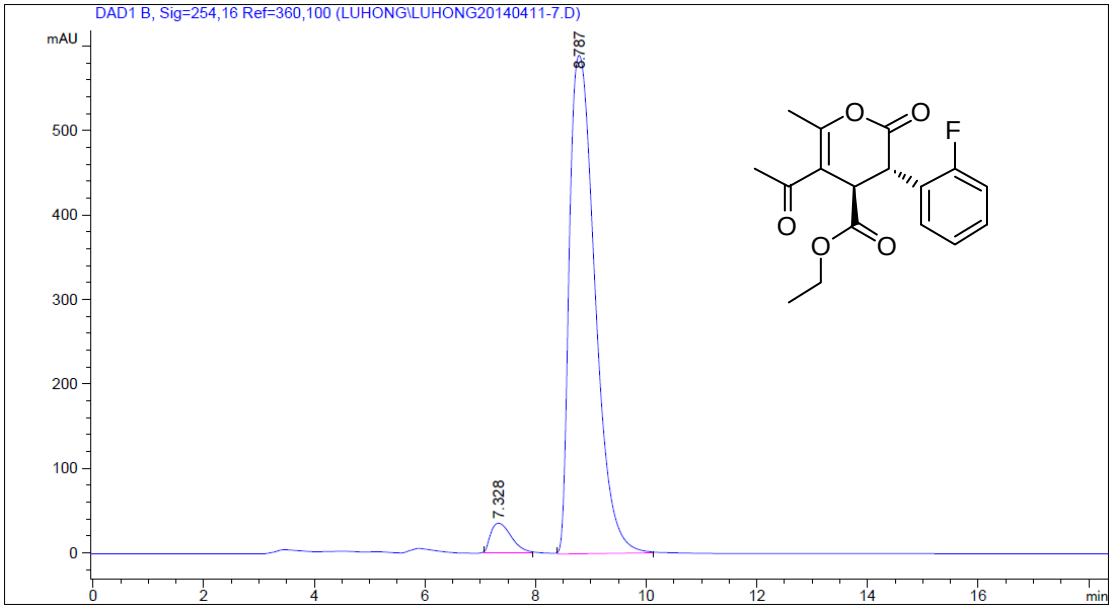


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	23.236	2.85998e4	512.54529	97.9166
2	DAD 254, 16 nm	27.883	608.53680	9.97842	2.0834

3b

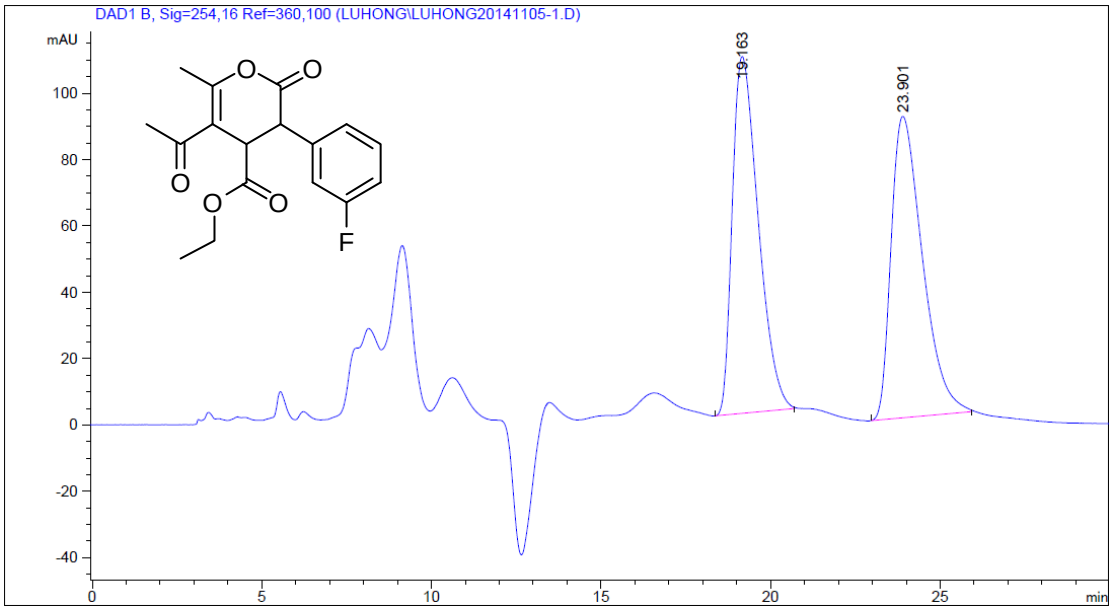


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	7.127	3217.05884	121.02797	50.5970
2	DAD 254, 16 nm	8.546	3141.13696	95.70715	49.4030

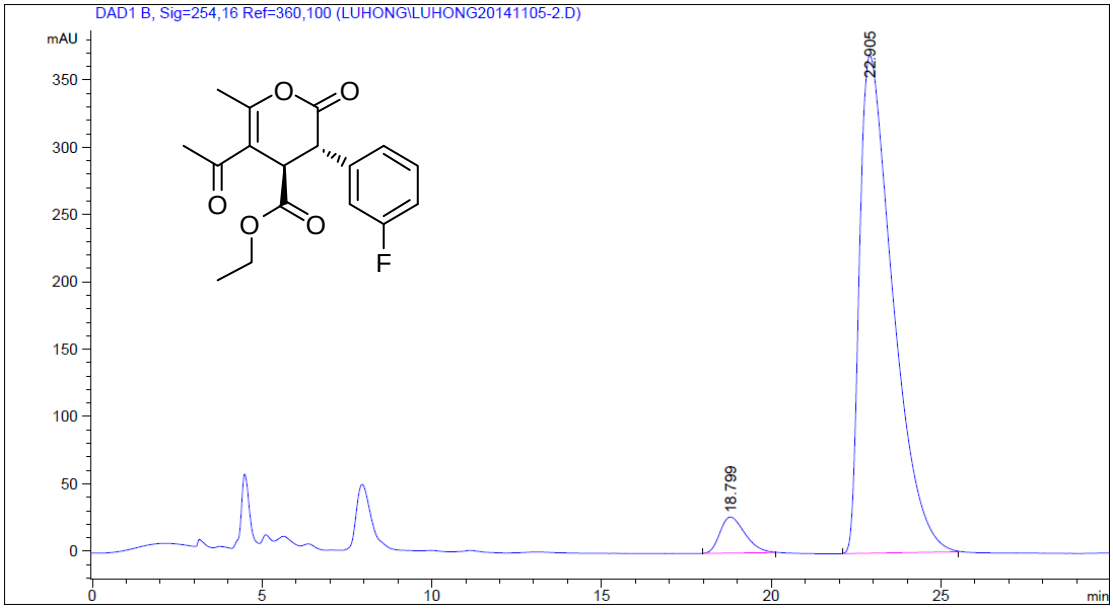


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	7.328	905.84814	34.85149	4.5650
2	DAD 254, 16 nm	8.787	1.89375e4	590.19281	95.4350

3c

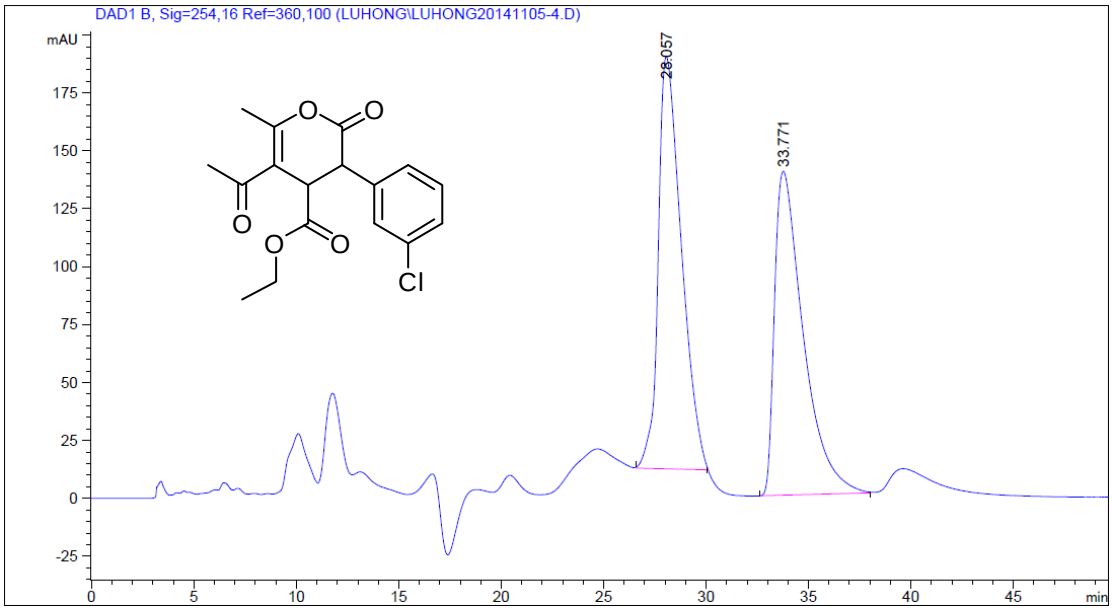


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	19.163	5708.45703	107.59546	49.5045
2	DAD 254, 16 nm	23.901	5822.72803	90.86617	50.4955

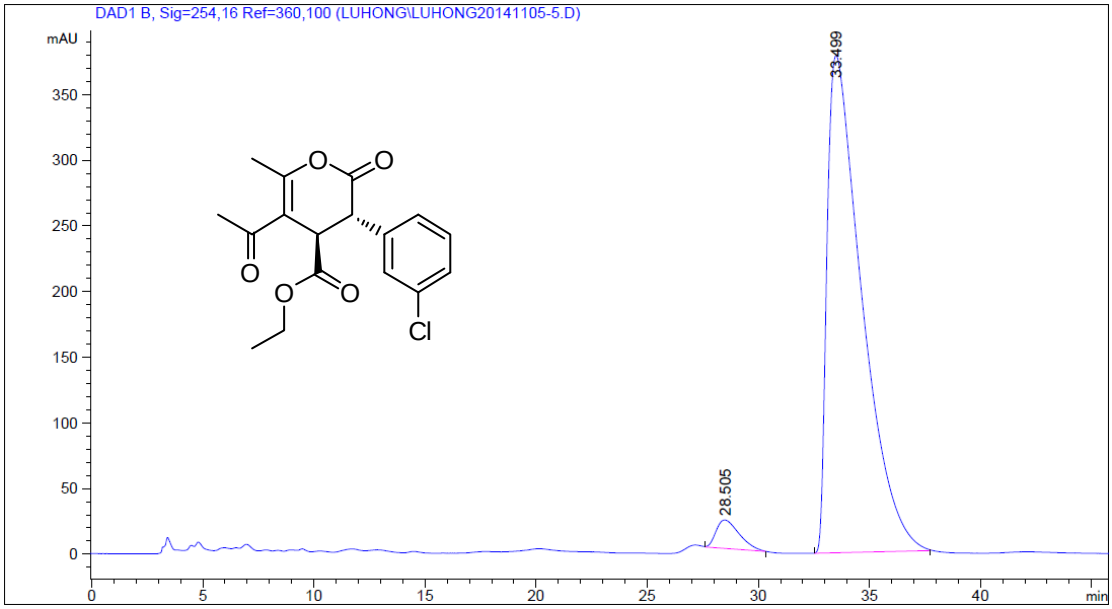


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	18.799	1369.08203	26.75384	5.1654
2	DAD 254, 16 nm	22.905	2.51358e4	369.87375	94.8346

3d

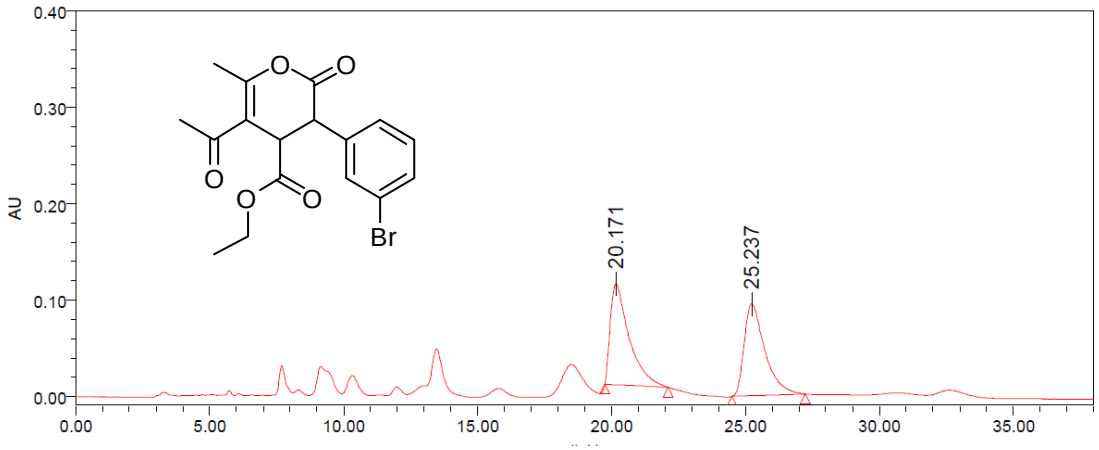


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	28.057	1.39738e4	178.06345	50.6433
2	DAD 254, 16 nm	33.771	1.36187e4	139.83339	49.3567

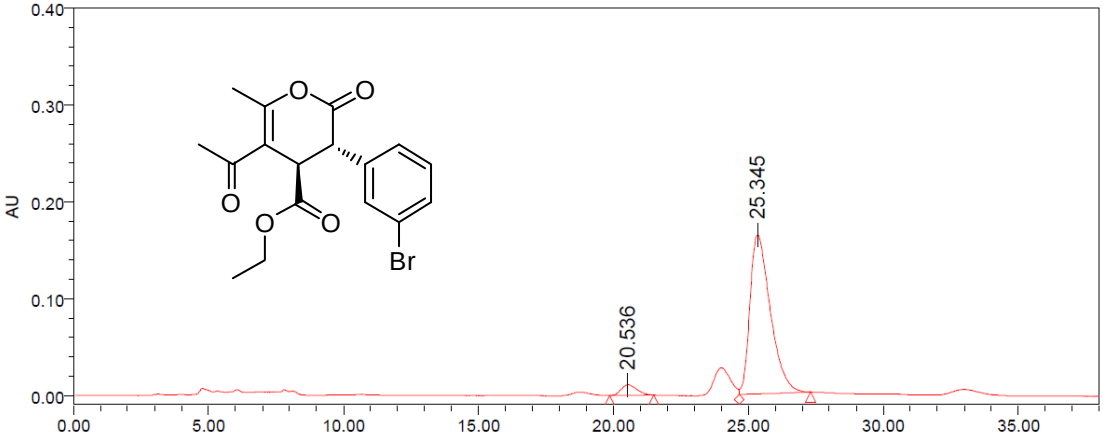


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	28.505	1565.34631	21.65634	3.6695
2	DAD 254, 16 nm	33.499	4.10933e4	378.78516	96.3305

3e

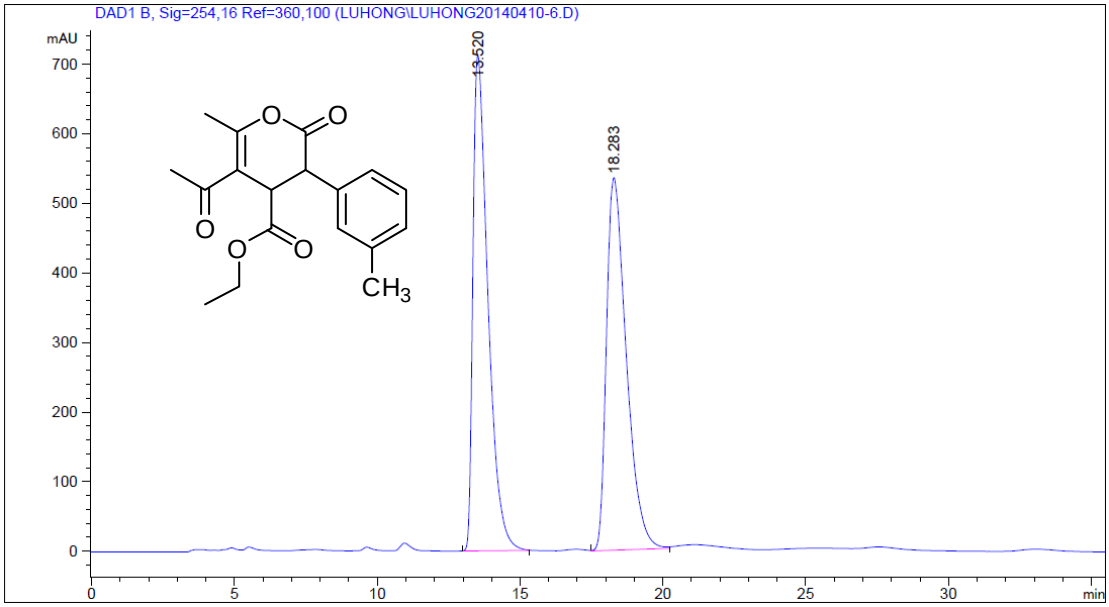


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	PDA254 nm	20.171	5129799	104702	50.13
2	PDA 254nm	25.237	5102963	95283	49.87

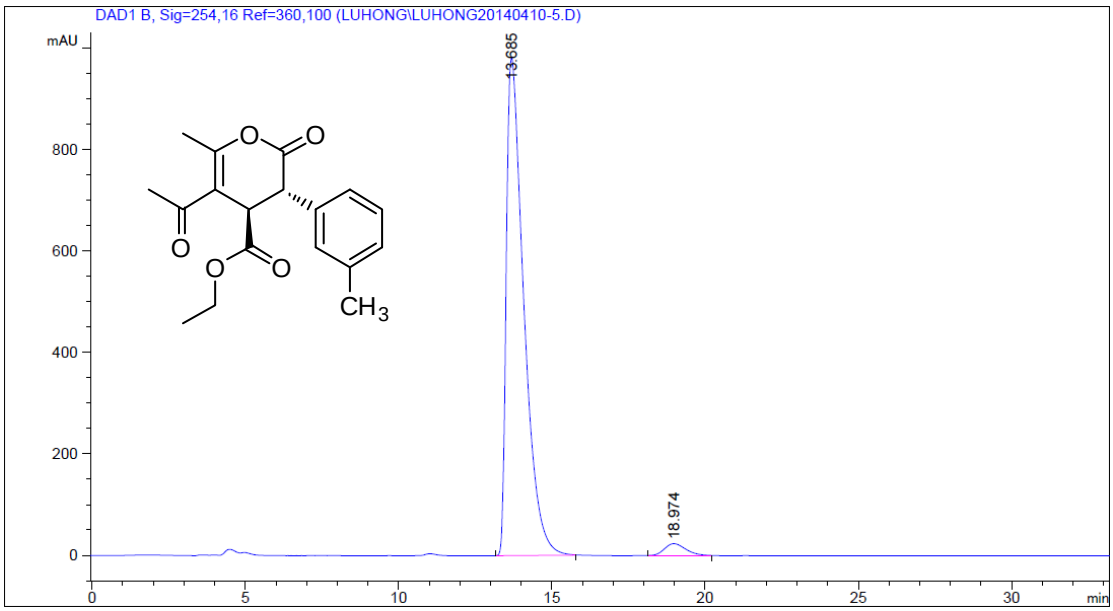


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA254 nm	20.536	453859	10968	5.00
2	PDA254 nm	25.345	8625663	164280	95.00

3f

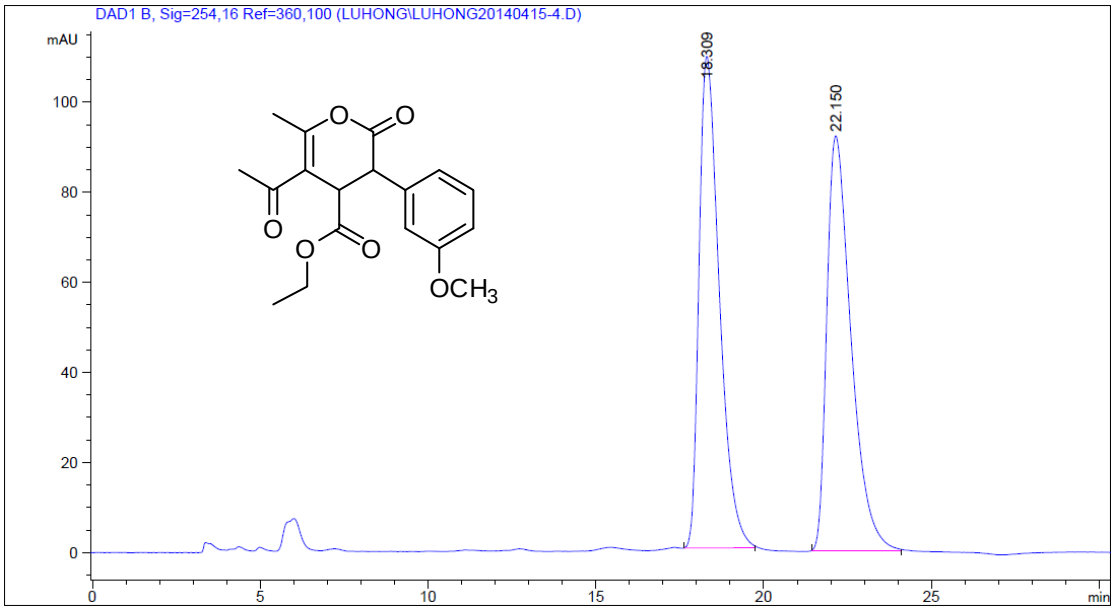


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	13.520	2.60074e4	712.30029	50.1697
2	DAD 254, 16 nm	18.283	2.58315e4	535.31207	49.8303

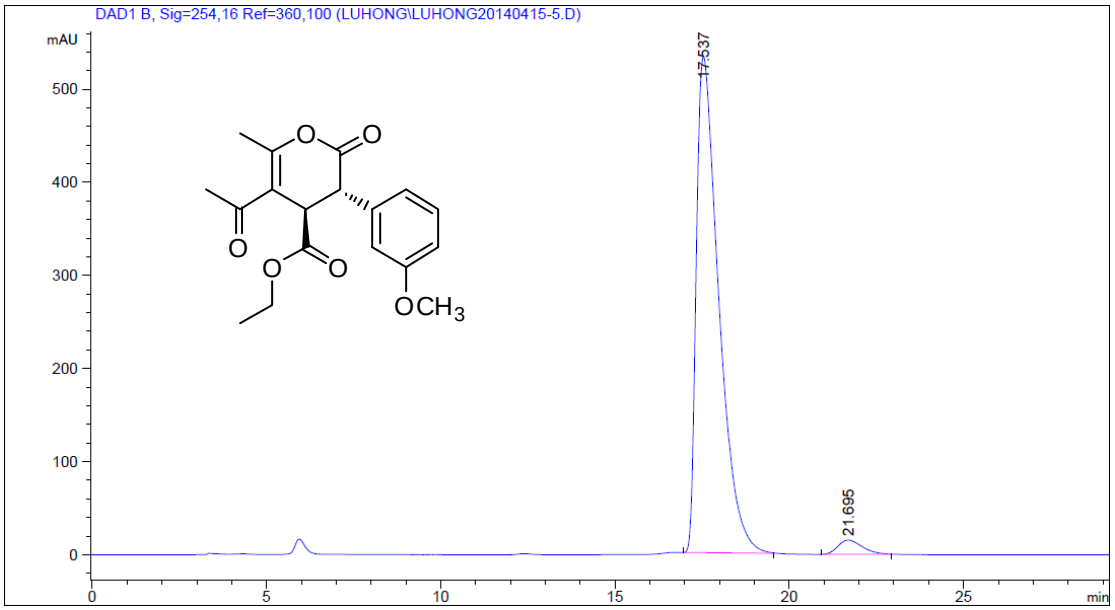


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	13.685	3.87218e4	982.26636	97.1668
2	DAD 254, 16 nm	18.974	1129.05334	23.39927	2.8332

3g

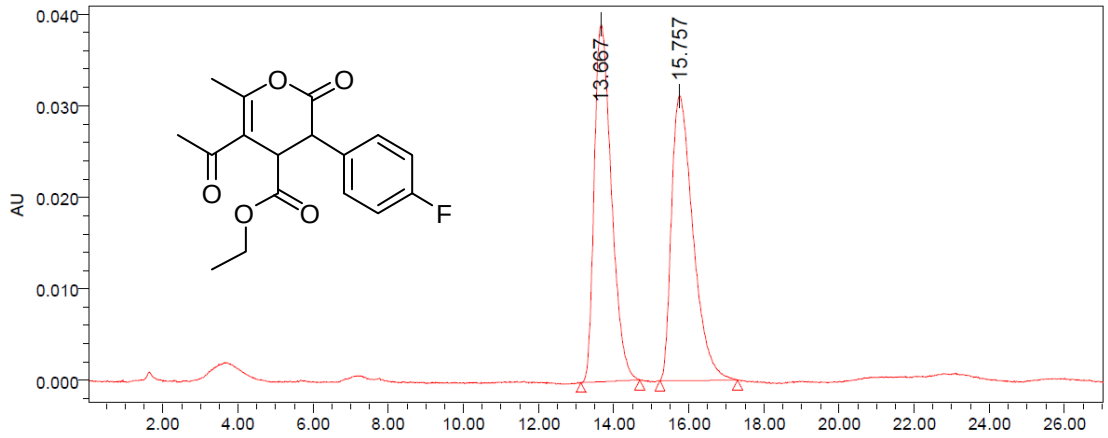


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	18.309	4591.86377	109.14375	49.7706
2	DAD 254, 16 nm	22.150	4634.18506	92.21734	50.2294

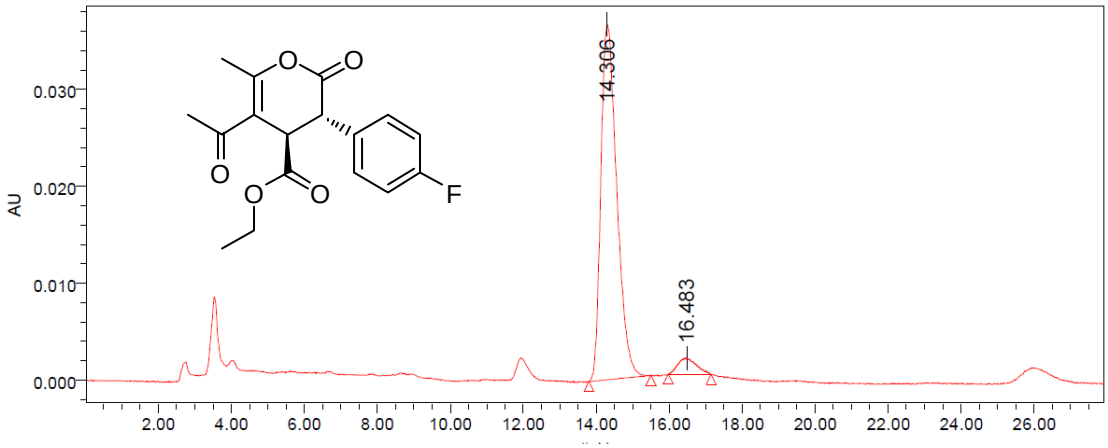


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	17.537	2.37991e4	533.15991	96.9647
2	DAD 254, 16 nm	21.695	744.98730	15.36586	3.0353

3h

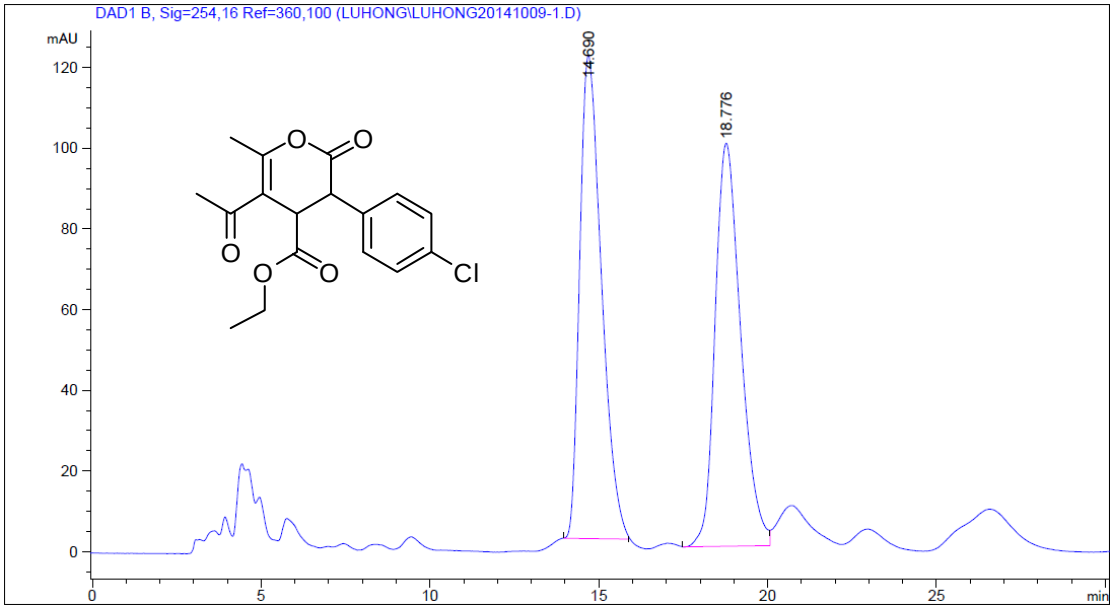


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	13.667	1240049	39046	49.90
2	PDA246.9 nm	15.757	1245039	31146	50.10

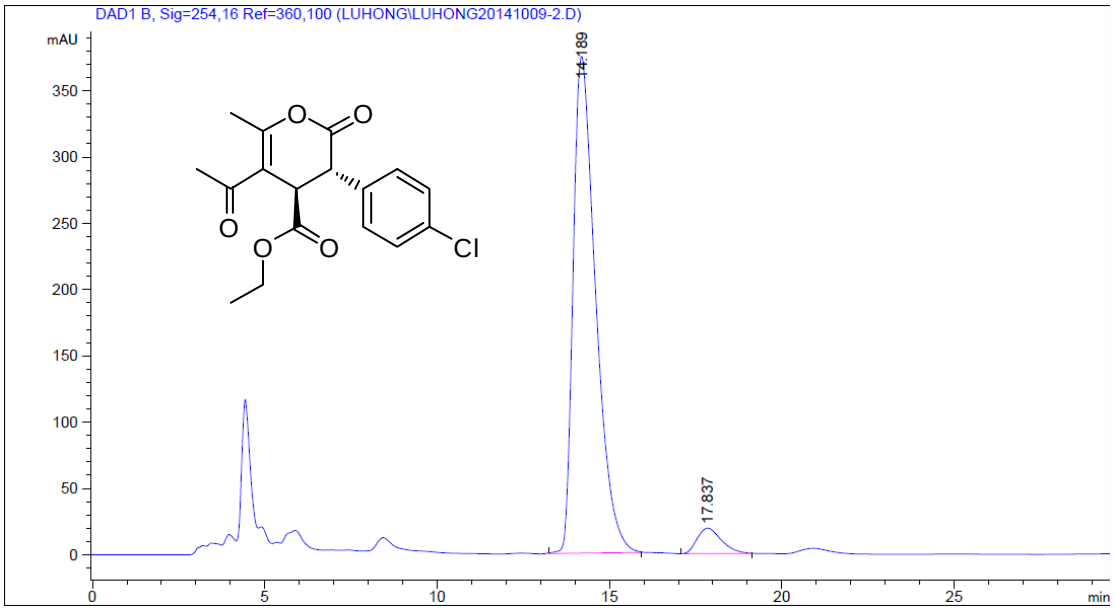


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	14.306	1109875	36656	94.95
2	PDA246.9 nm	16.483	58971	1670	5.05

3i

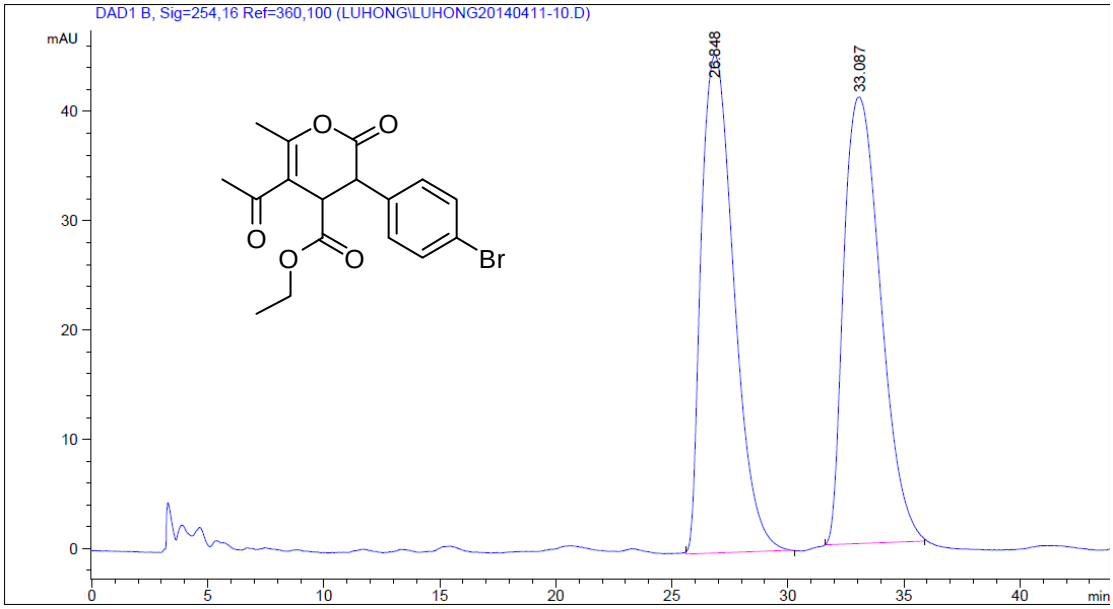


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	14.690	5360.80029	119.75430	50.4051
2	DAD 254, 16 nm	18.776	5274.63818	99.87841	49.5949

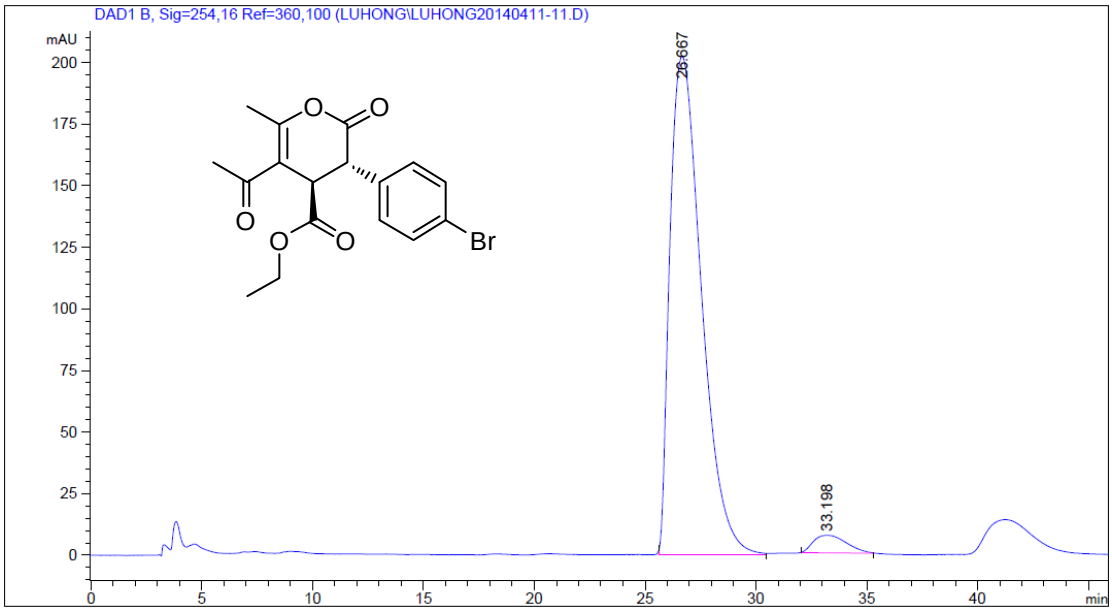


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	14.189	1.69883e4	374.87115	94.7678
2	DAD 254, 16 nm	17.837	937.93311	19.23451	5.2322

3j

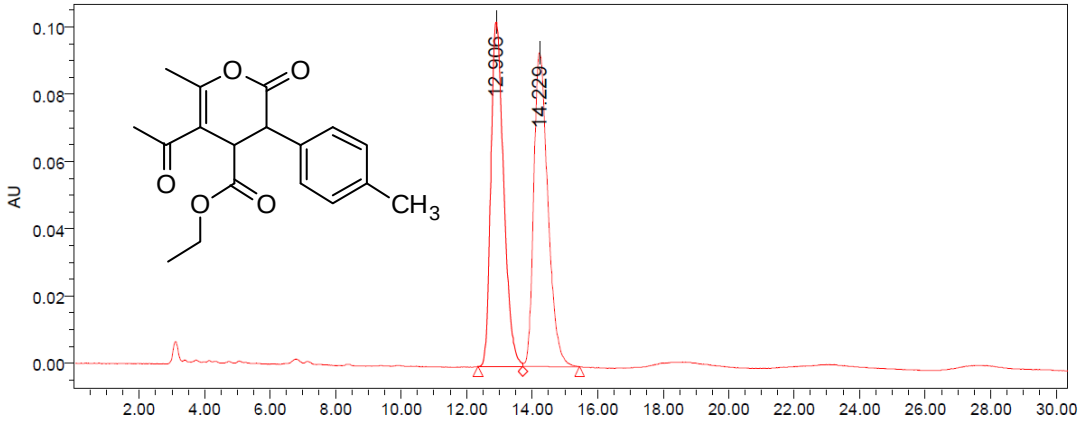


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	26.848	4462.83496	45.53188	49.7318
2	DAD 254, 16 nm	33.087	4510.97314	40.85940	50.2682

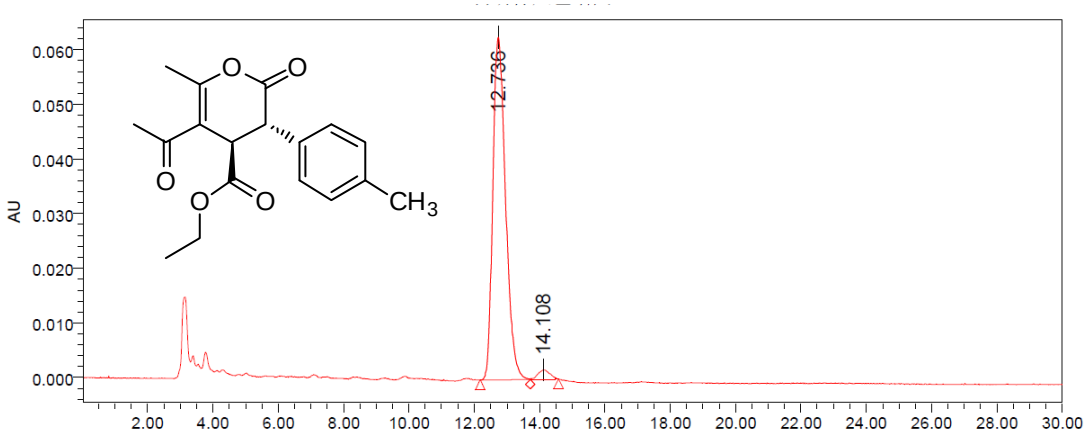


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	26.667	1.99684e4	202.37297	96.4331
2	DAD 254, 16 nm	33.198	738.58948	7.13121	3.5669

3k

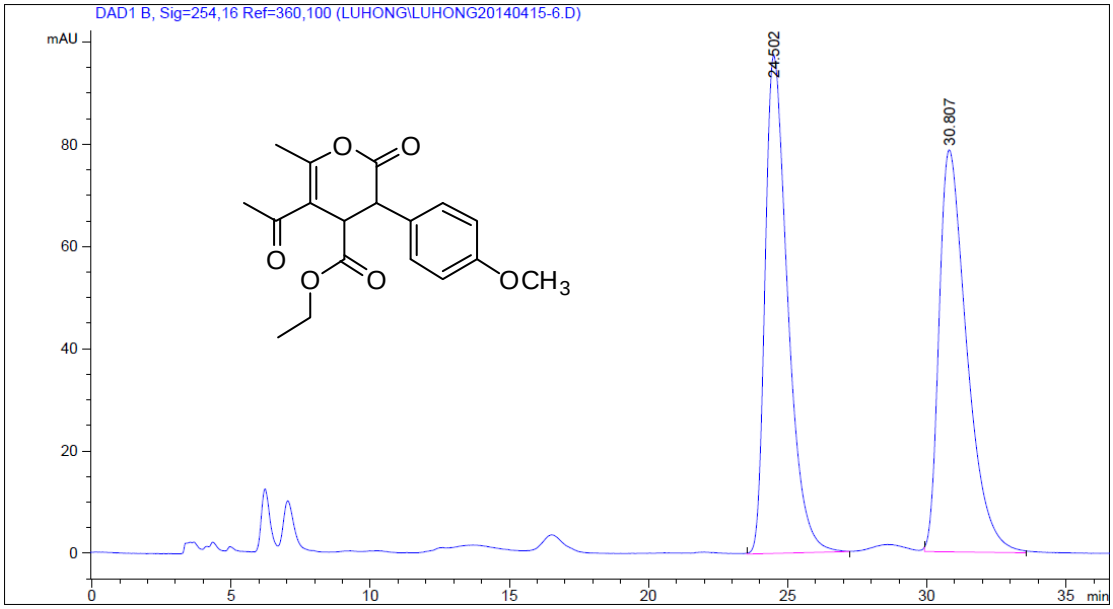


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	12.906	2790713	102444	49.88
2	PDA246.9 nm	14.229	2803883	93367	50.12

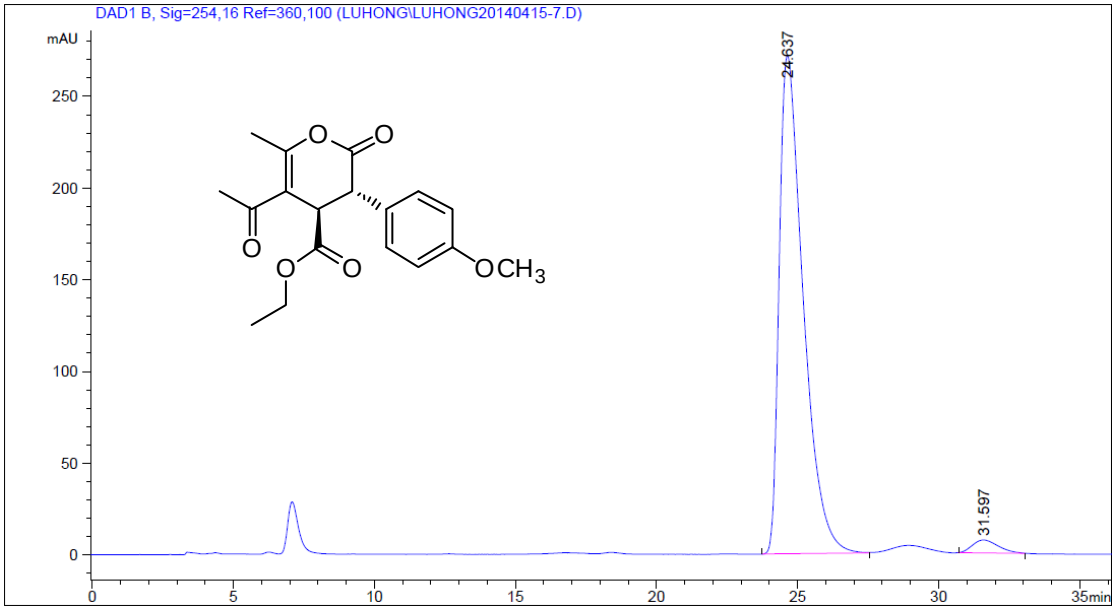


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	12.736	1690588	62738	97.49
2	PDA246.9 nm	14.108	43514	1731	2.51

3l

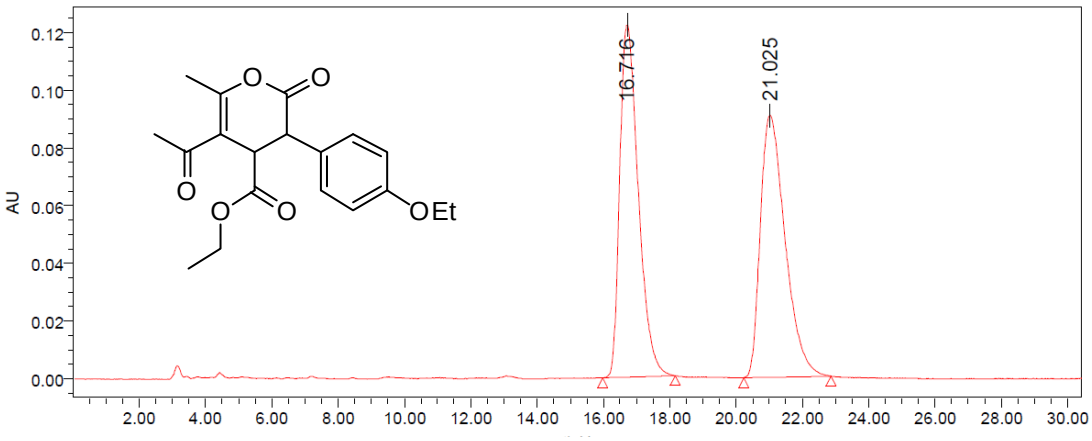


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	24.502	5495.23730	97.43780	50.1952
2	DAD 254, 16 nm	30.807	5452.49951	78.66367	49.8048

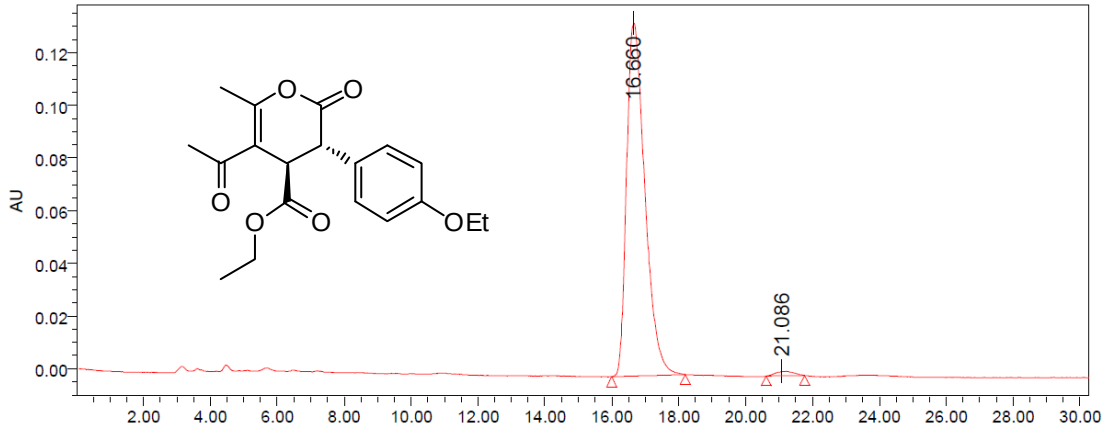


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	24.637	1.60160e4	271.59885	97.2659
2	DAD 254, 16 nm	31.597	450.20514	7.07779	2.7341

3m

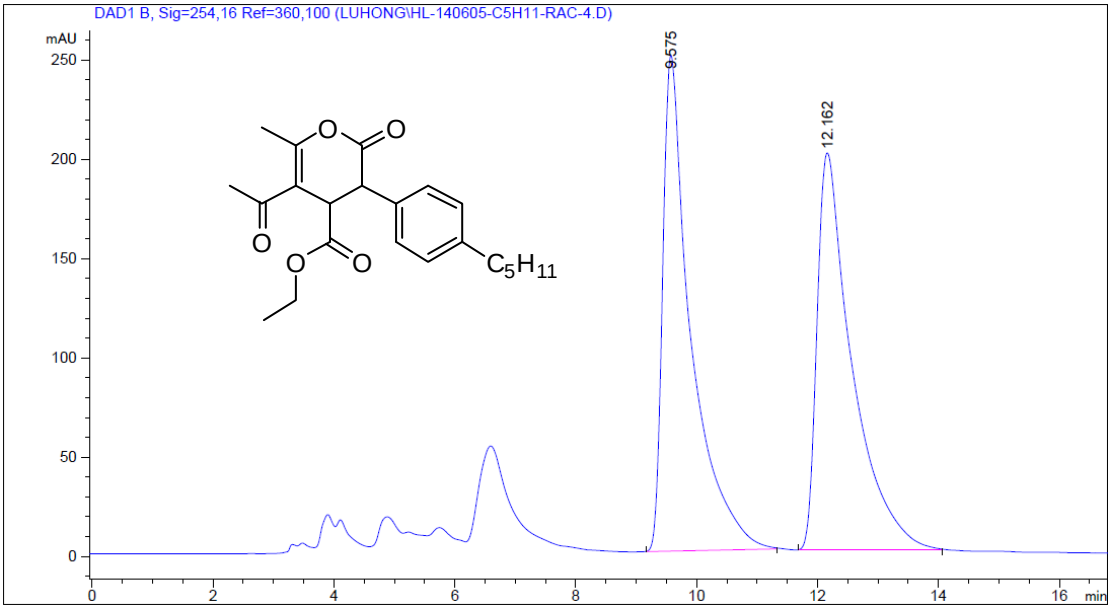


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	16.716	4743145	122087	50.19
2	PDA246.9 nm	21.025	4707496	90775	49.81

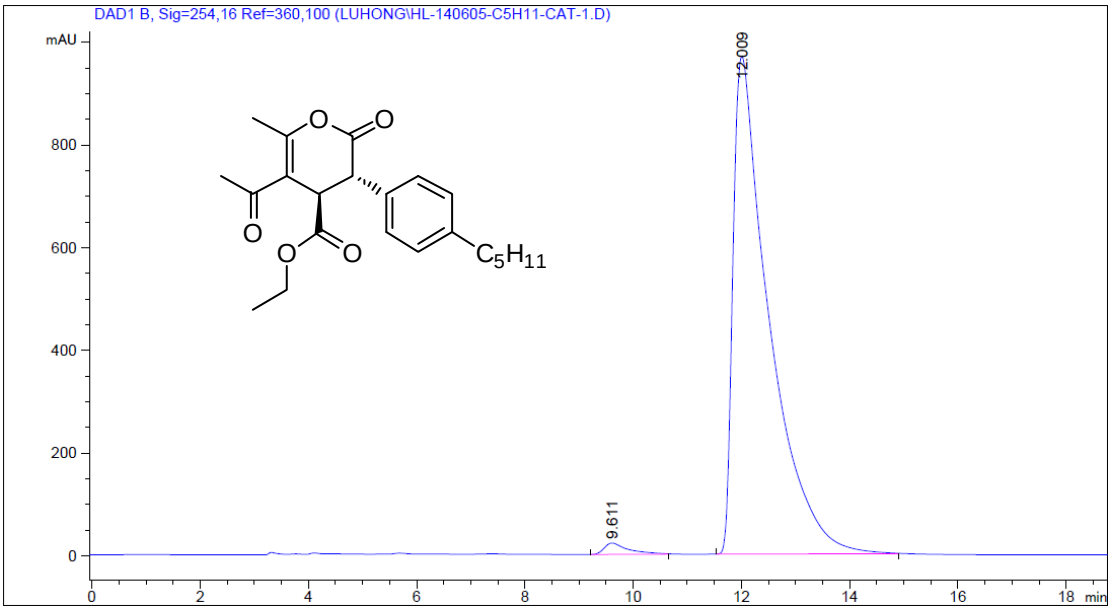


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	16.660	5088941	134227	98.77
2	PDA246.9 nm	21.086	63474	1641	1.23

3n

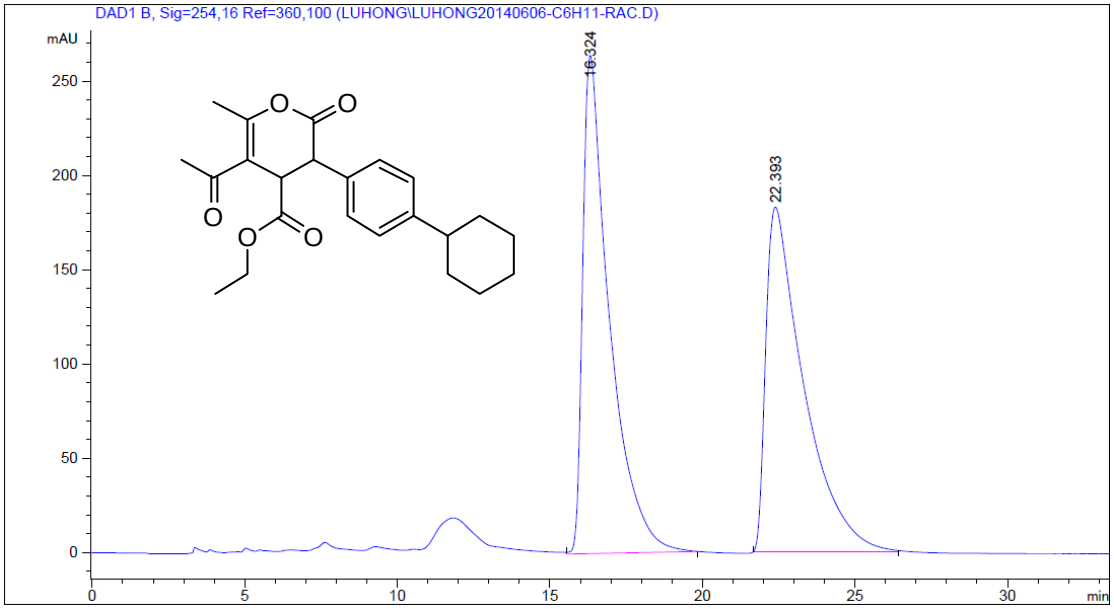


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	9.575	8085.87207	249.60936	50.1708
2	DAD 254, 16 nm	12.162	8030.80518	199.89389	49.8292

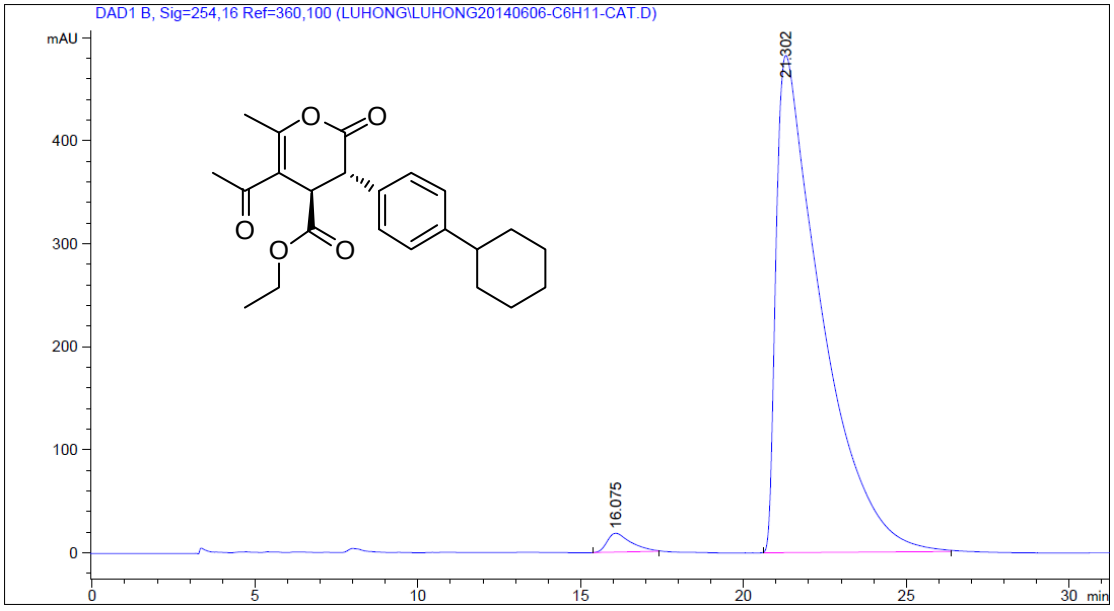


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	9.611	682.96936	22.1537	1.5015
2	DAD 254, 16 nm	12.009	4.48028e4	969.76141	98.4985

3o

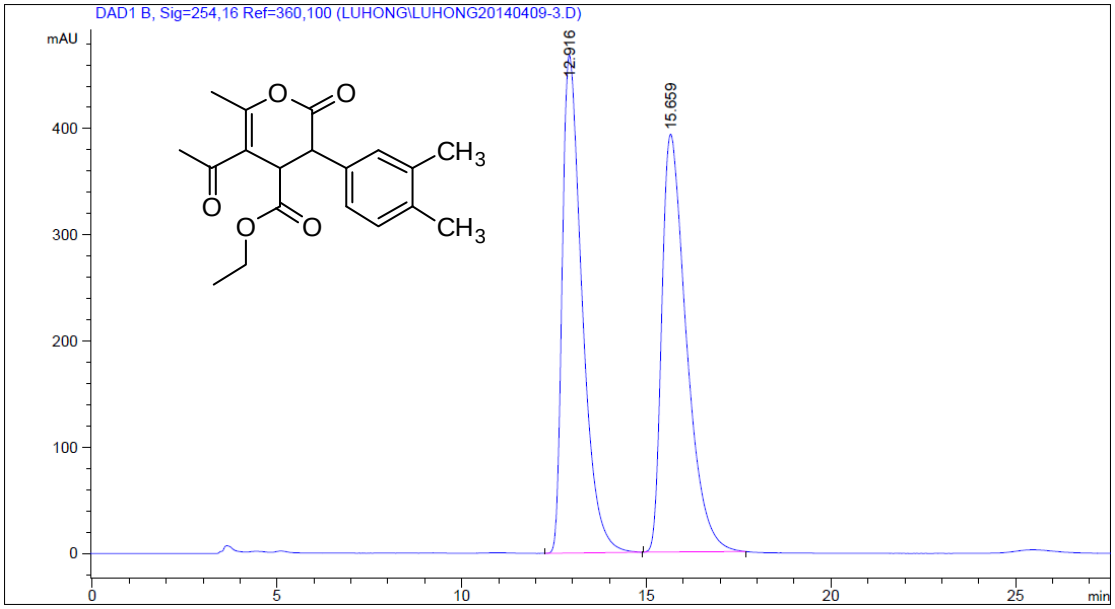


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	16.324	1.60939e4	264.19598	50.5960
2	DAD 254, 16 nm	22.393	1.57148e4	182.63591	49.4040

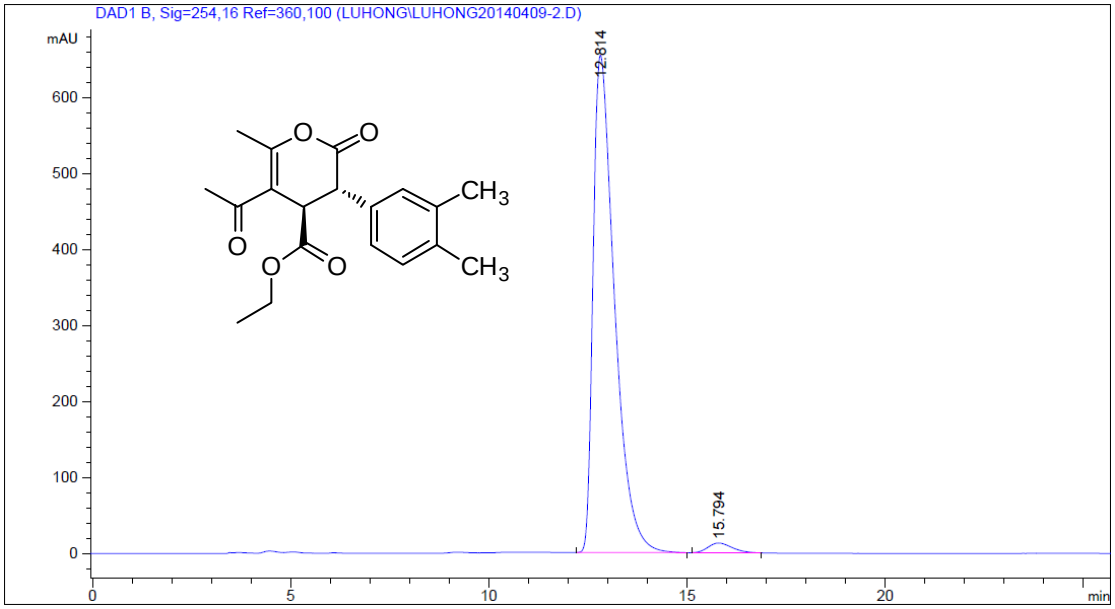


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	16.075	935.47333	18.44045	1.9669
2	DAD 254, 16 nm	21.302	4.66265e4	482.55835	98.0331

3p

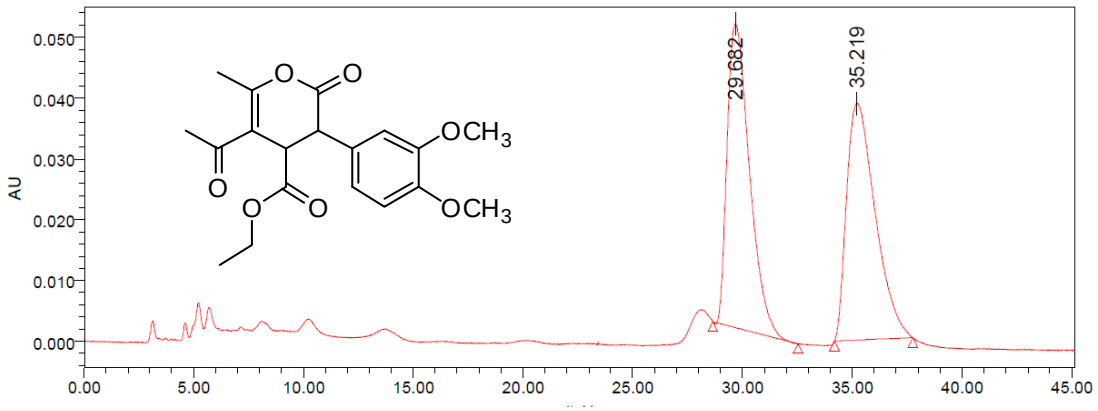


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	12.916	1.75131e4	468.87976	49.0660
2	DAD 254, 16 nm	15.659	1.81799e4	393.25751	50.9340

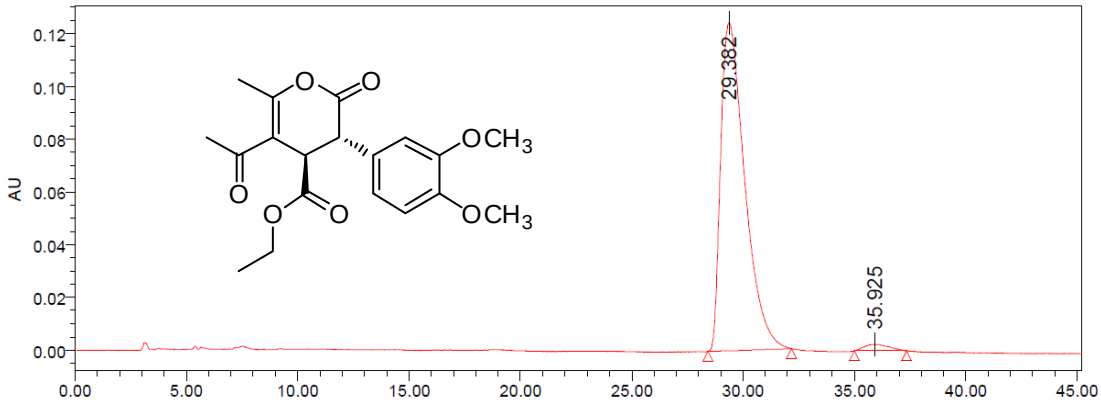


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	12.814	2.44865e4	654.27258	97.8444
2	DAD 254, 16 nm	15.794	539.47058	12.76532	2.1556

3q

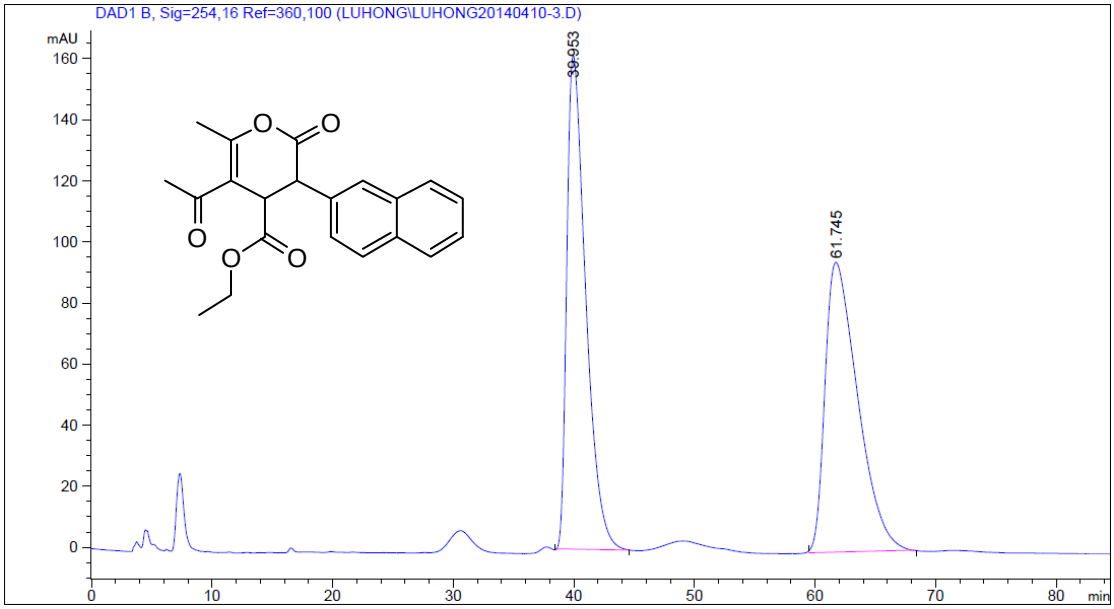


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	29.682	3540273	49972	50.03
2	PDA246.9 nm	35.219	3535808	38939	49.97

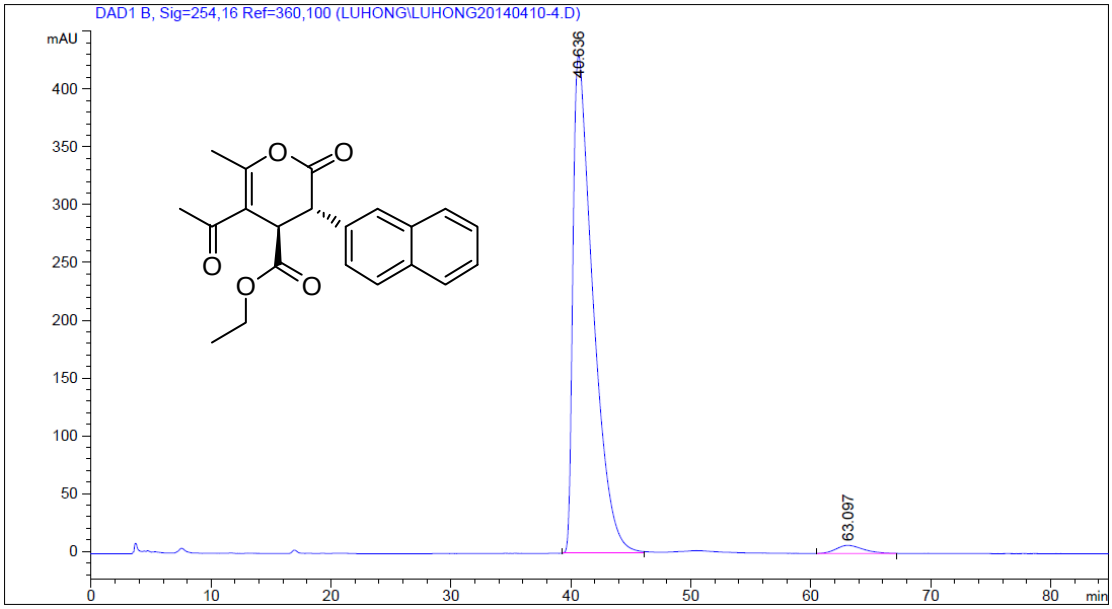


Peak	Processed Channel	Retention Time (min)	Peak Area (AU*s)	Peak Height (AU)	Peak Area (%)
1	PDA246.9 nm	29.382	9339267	124412	97.96
2	PDA246.9 nm	35.925	194163	2466	2.04

3r

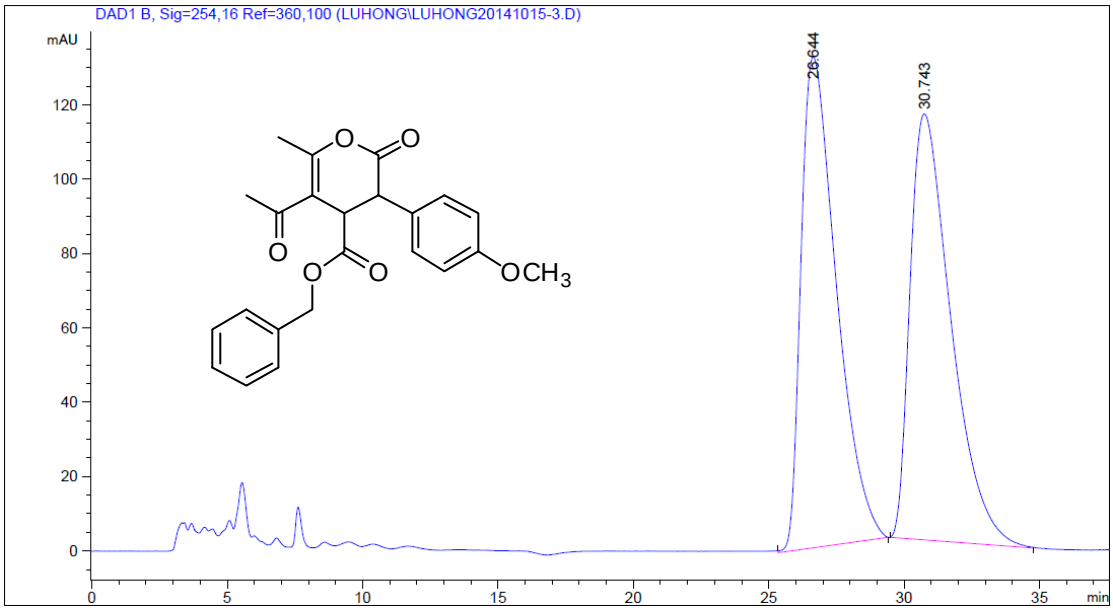


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	39.953	1.75848e4	161.70930	49.6348
2	DAD 254, 16 nm	61.745	1.78436e4	94.90437	50.3652

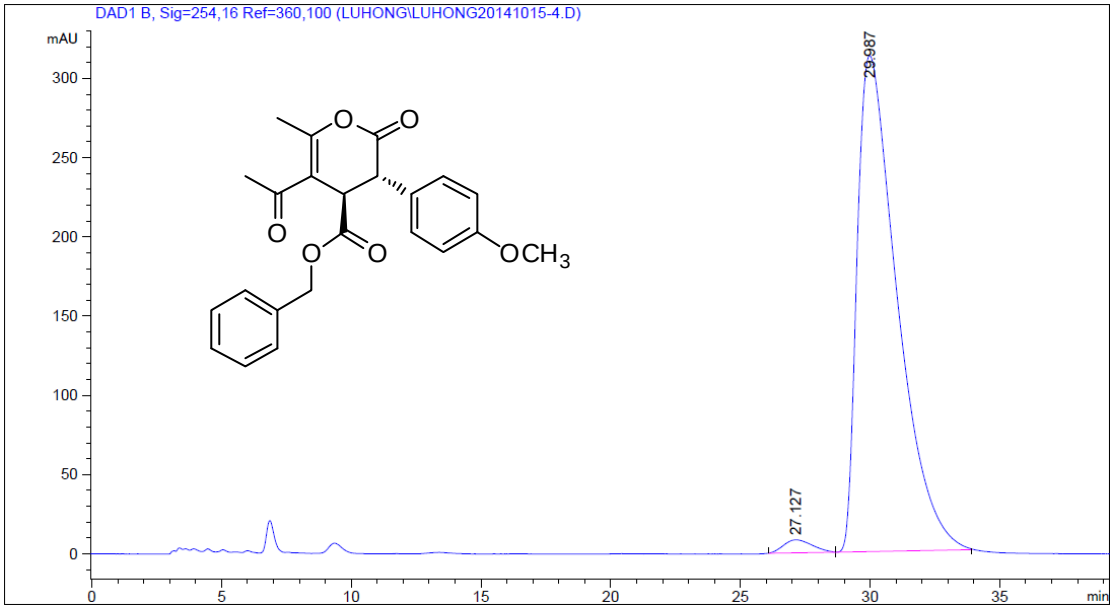


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	40.636	4.99372e4	430.04370	97.8326
2	DAD 254, 16 nm	63.097	1106.34253	7.03193	2.1674

3s

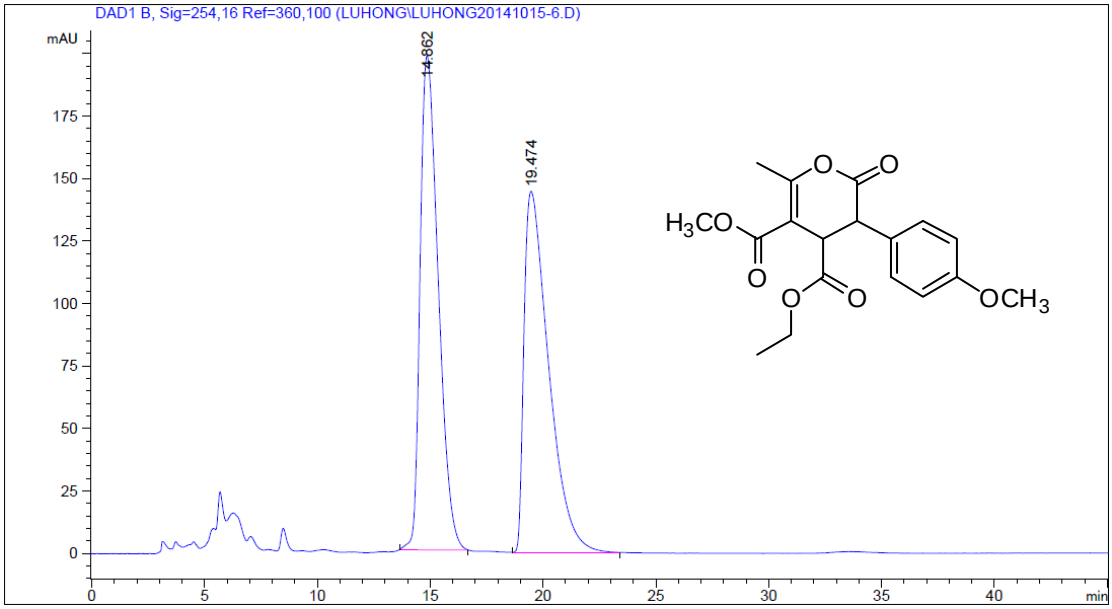


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	26.644	1.19727e4	132.14328	49.8551
2	DAD 254, 16 nm	30.743	1.20423e4	114.58701	50.1449

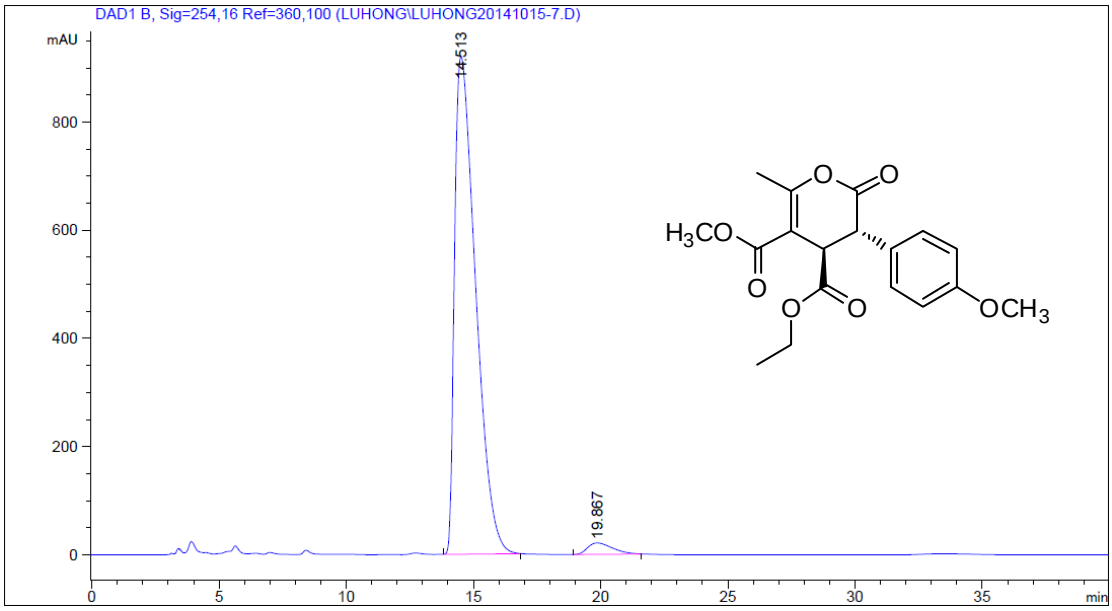


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	27.127	626.90314	8.19894	1.8409
2	DAD 254, 16 nm	29.987	3.34267e4	312.95358	98.1591

3t

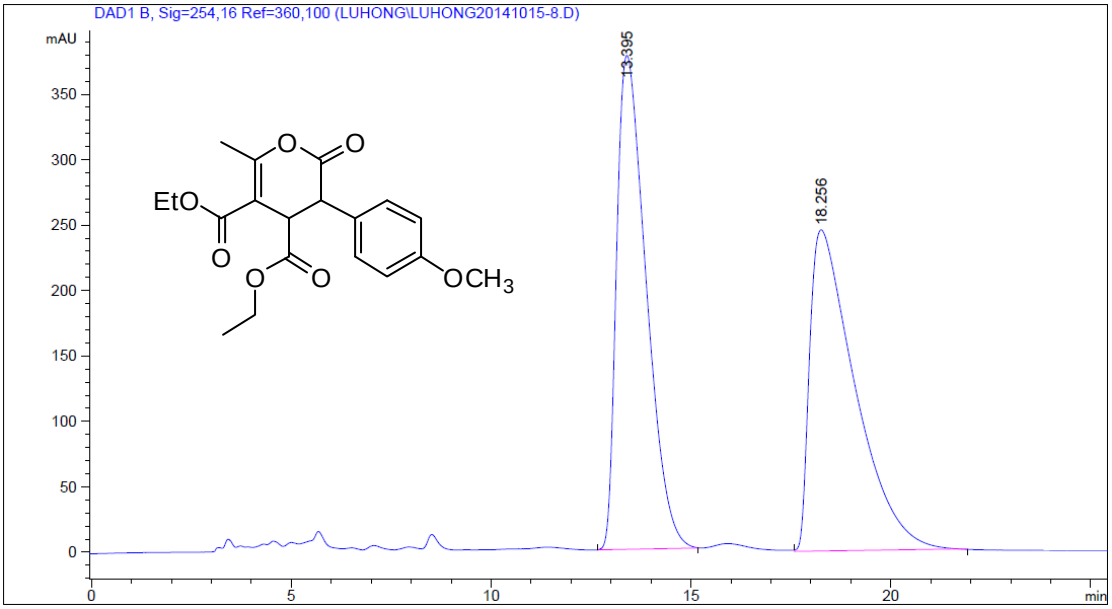


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	14.862	1.10374e4	197.91924	50.1807
2	DAD 254, 16 nm	19.474	1.09579e4	144.61237	49.8193

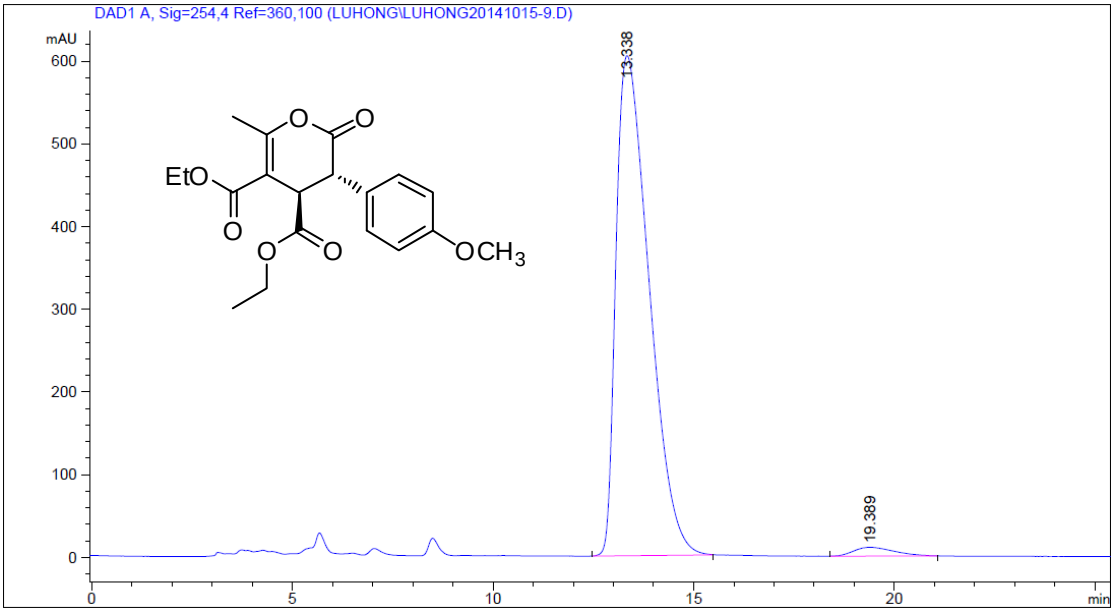


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	14.513	5.27739e4	920.35876	97.3579
2	DAD 254, 16 nm	19.867	1432.20190	20.98921	2.6421

3u

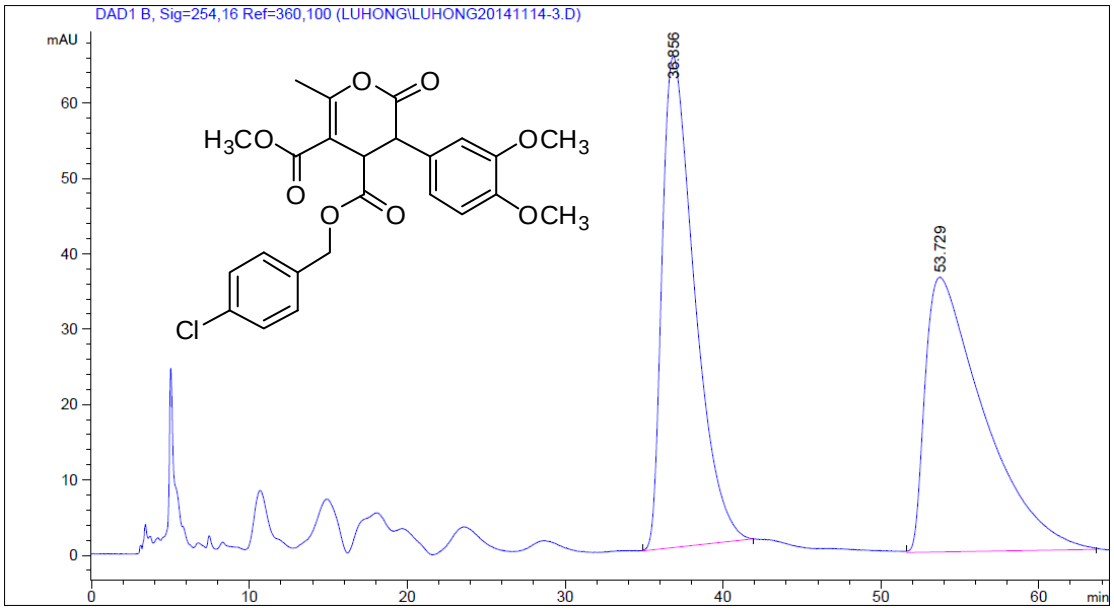


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	13.395	1.93720e4	377.46848	50.1193
2	DAD 254, 16 nm	18.256	1.92798e4	245.45526	49.8807

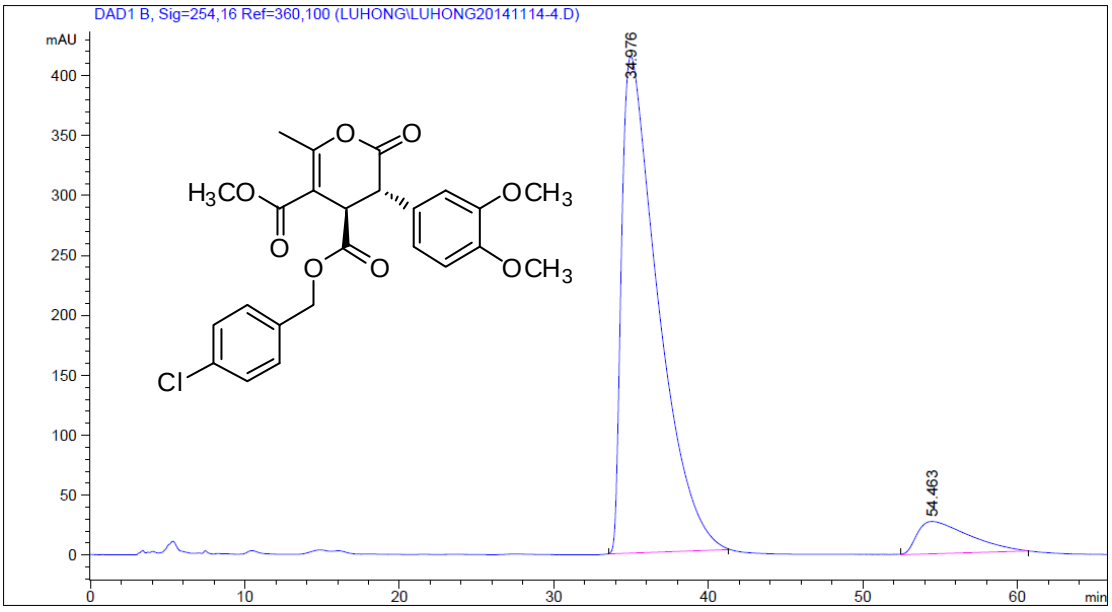


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	13.338	3.46210e4	604.72217	97.9056
2	DAD 254, 16 nm	19.389	740.59613	10.58835	2.0944

3v

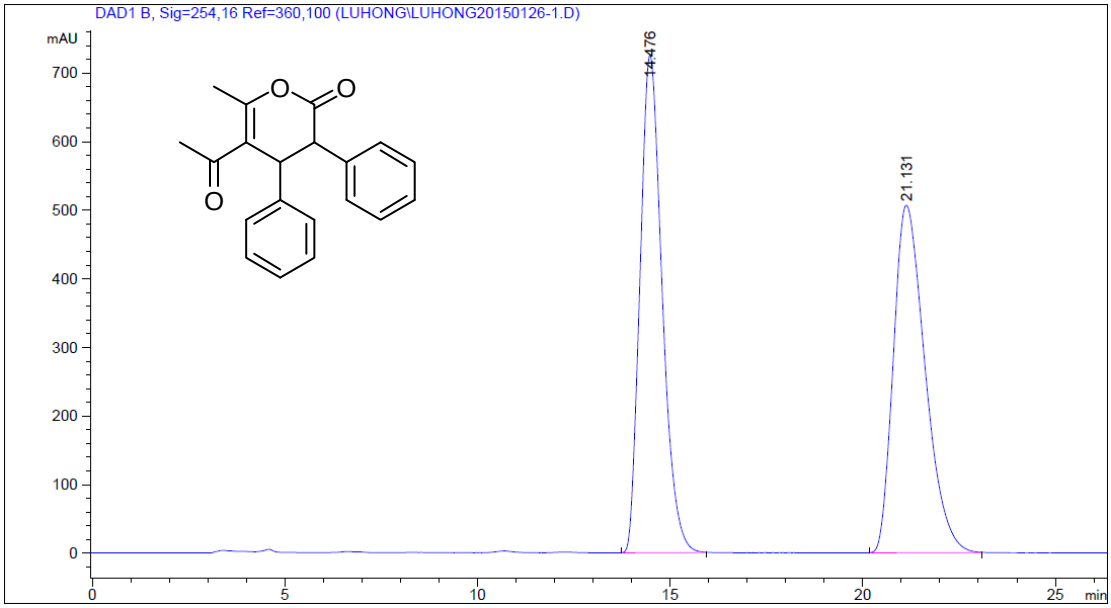


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	36.856	9330.18750	65.16678	49.7995
2	DAD 254, 16 nm	53.729	9405.30176	36.44764	50.2005

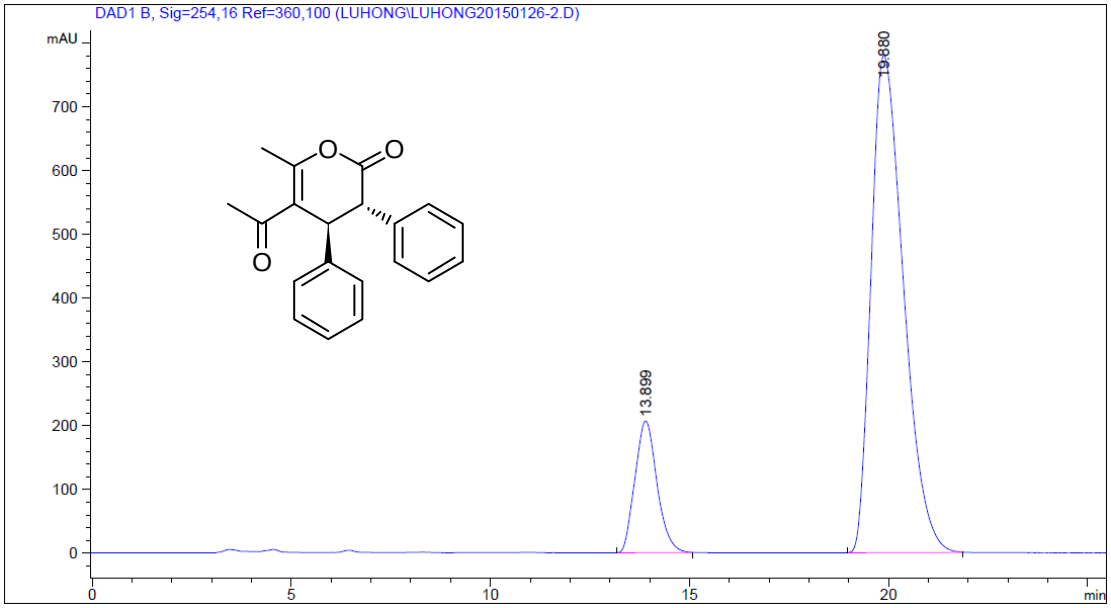


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	34.976	6.77280e4	414.29953	91.5319
2	DAD 254, 16 nm	54.463	6265.88574	27.00175	8.4681

3w

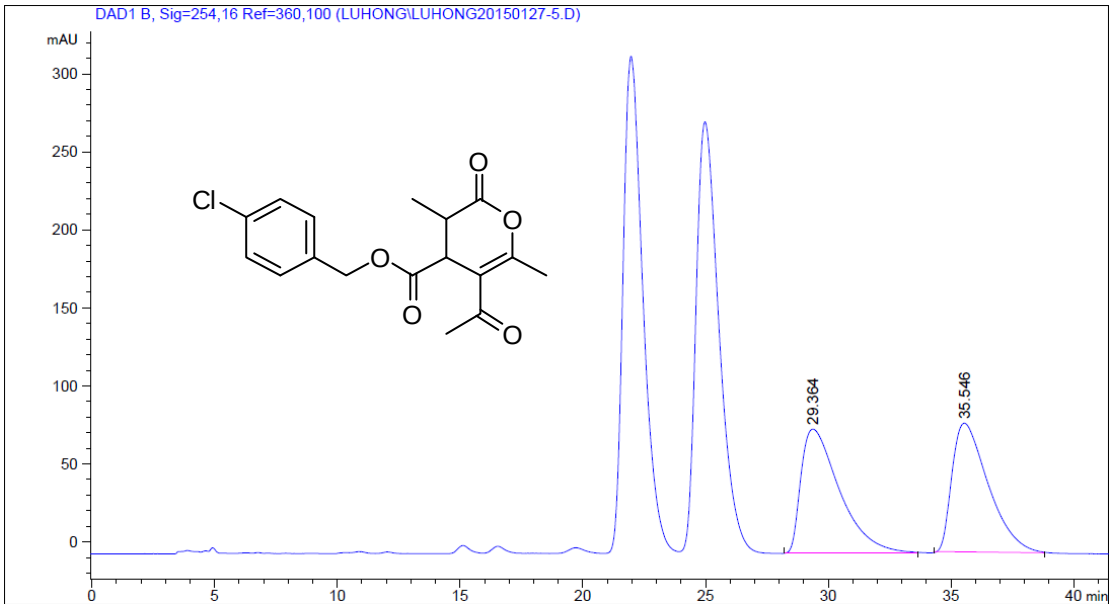


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	14.476	2.88310e4	725.20691	49.9210
2	DAD 254, 16 nm	21.131	2.89223e4	506.57614	50.0790

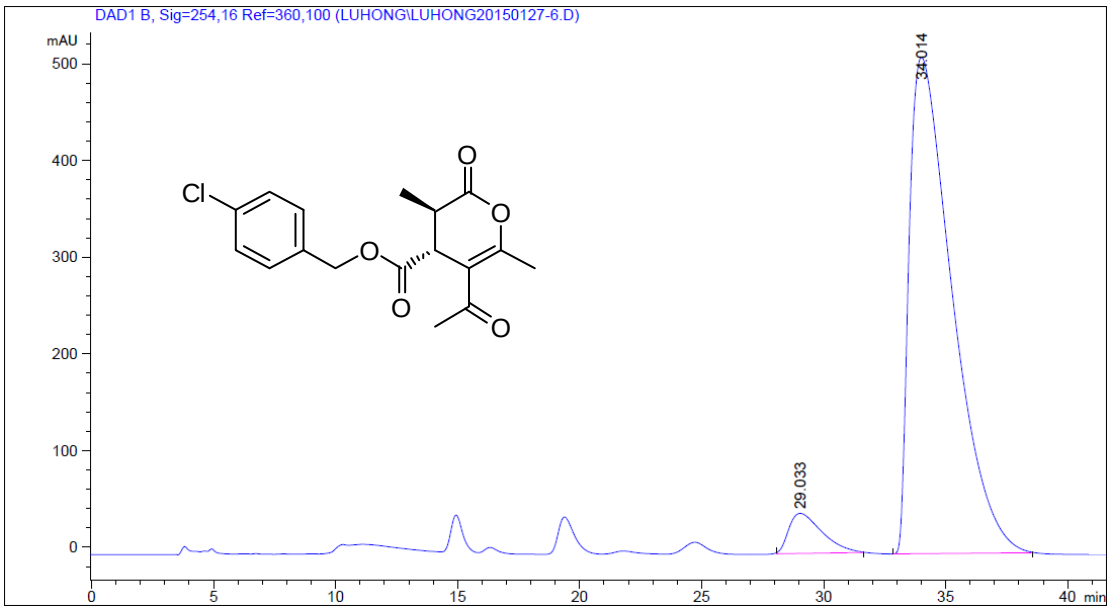


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	13.899	7955.07373	206.62357	15.2991
2	DAD 254, 16 nm	19.880	4.40420e4	779.88367	84.7009

3x

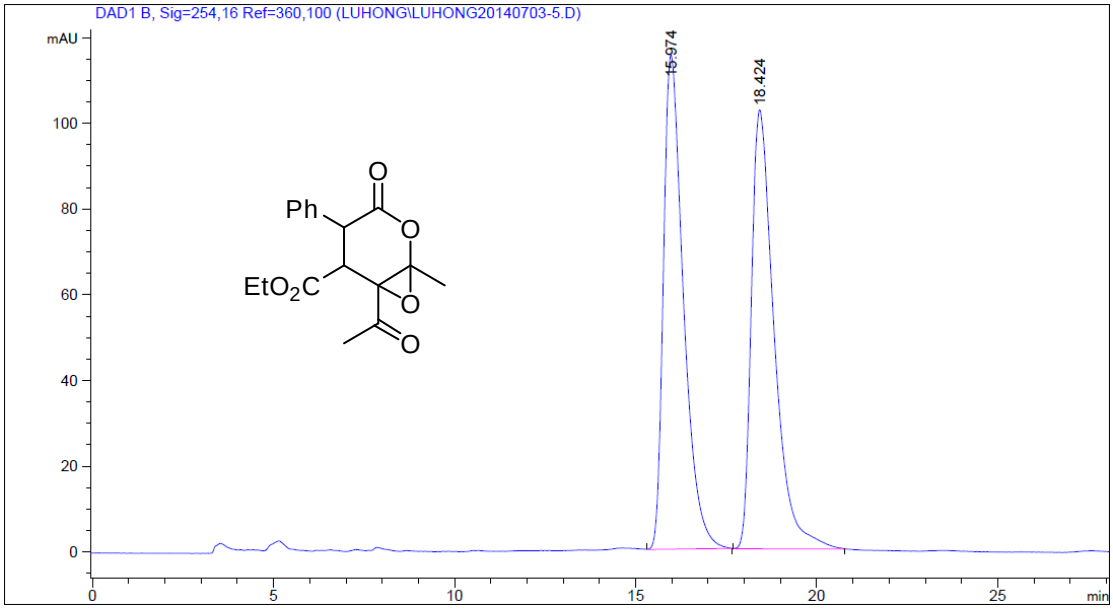


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	29.364	8643.55469	79.28326	50.5026
2	DAD 254, 16 nm	35.546	8471.50195	82.48608	49.4974

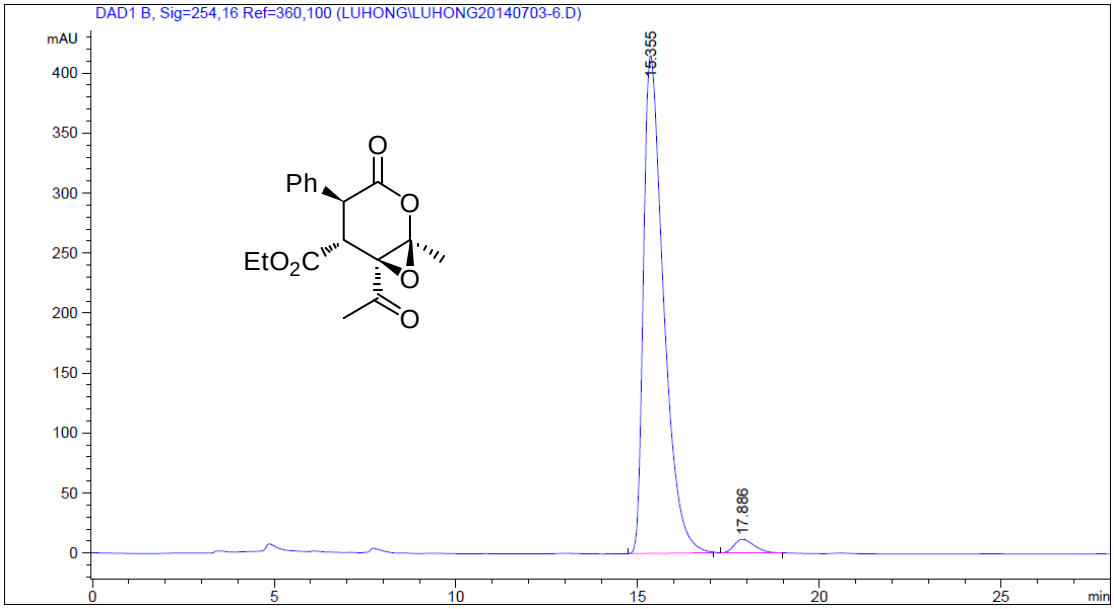


Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	29.033	3923.86377	41.47171	5.8839
2	DAD 254, 16 nm	34.014	6.27641e4	513.21808	94.1161

6a



Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	15.974	4424.85010	115.26480	49.6742
2	DAD 254, 16 nm	18.424	4482.89453	102.39242	50.3258



Peak	Processed Channel	Retention Time (min)	Peak Area (mAU*s)	Peak Height (mAU)	Peak Area (%)
1	DAD 254, 16 nm	15.355	1.59502e4	414.93729	97.3614
2	DAD 254, 16 nm	17.886	432.26294	11.25341	2.6386