

Supporting Information

Palladium-catalyzed Asymmetric Hydrogenation of Lactones under Base-Free Condition

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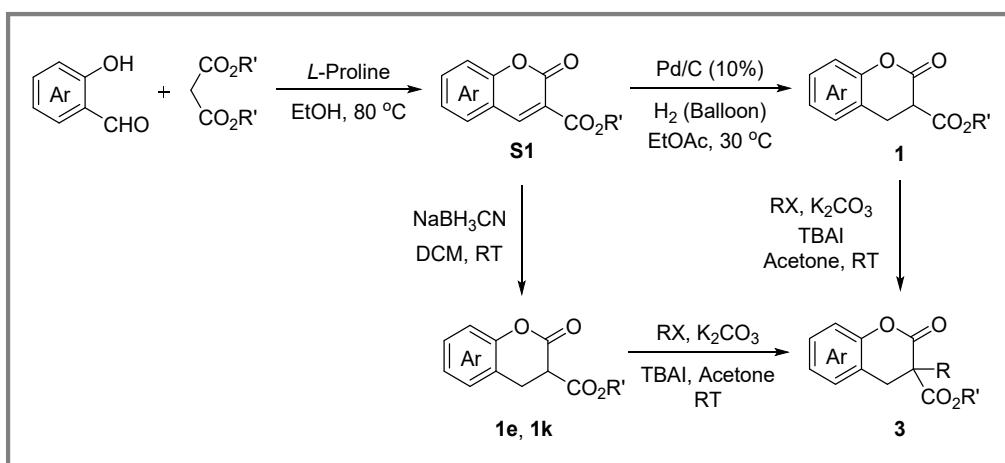
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1. General and Materials

General: All reactions were carried out under an atmosphere of nitrogen using the standard Schlenk techniques, unless otherwise noted. ^1H NMR, ^{13}C NMR and ^{19}F NMR spectra were recorded at room temperature in CDCl_3 on 400 MHz instrument with TMS as internal standard. Enantiomeric excess was determined by HPLC analysis, using the chiral column described below in detail. Optical rotations were measured by polarimeter. Flash column chromatography was performed on silica gel (200-300 mesh). The heat source for all heating reactions is the oil bath. High-resolution mass spectrometry (HRMS) was measured on an electrospray ionization (ESI) apparatus using the time-of-flight (TOF) mass spectrometry. All reactions were monitored by TLC analysis.

Materials: Commercially available reagents and anhydrous solvents were used throughout without the further purification.

2. Synthesis of Substrate Lactones

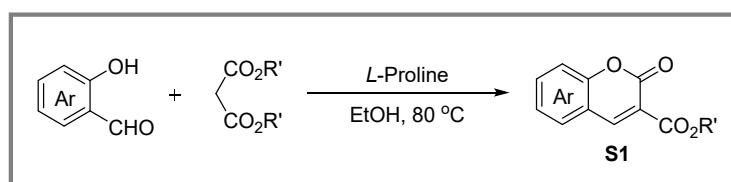


The substrate lactones **1** were prepared from the readily available 2-hydroxyarylaldehydes and malonates over two steps according to the known methods.^[1,2] The first step is the synthesis of the intermediate coumarin-3-carboxylates **S1**, which are the known compounds; the second step is Pd/C-catalyzed hydrogenation to afford the substrate lactones. For **1e** and **1k** which is sensitive to Pd/C-catalyzed hydrogenation system, sodium cyanoborohydride was used for reduction. Among them, lactones **1a**, **1b**, **1h**, **1j**, **1m** and **1o** were the known compounds.^[1,3]

The substrate lactones **3** with quaternary stereocenter were prepared through the alkylation of above lactones. Among them, lactones **3ae** and **3af** were the known compounds.^[4]

2.1. The Synthesis of Alkoxy carbonyl Substituted Lactones

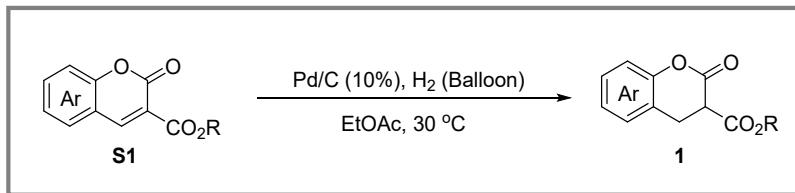
General Procedure for the Synthesis of Intermediate Coumarin-3-carboxylates **S1**:



To a mixture of 2-hydroxyarylaldehyde (7.0 mmol), malonate (8.4 mmol) and ethanol (5 mL), L-proline (0.350 g, 3 mmol) was added and heated to 80 °C overnight. After the completion of the

reaction (monitored by TLC), the mixture was cooled to room temperature and poured in water (50 mL). After stirred for 10 min, the mixture was filtered and the residue was washed with cool ethanol to give a crude product **S1** for next step without the further purification.

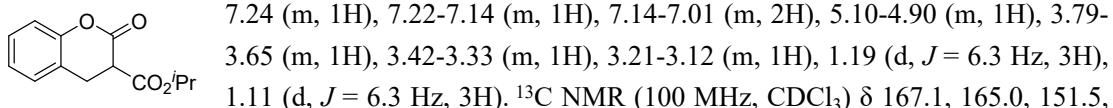
General Procedure for the Synthesis of Lactones **1 with Pd/C:**



To a solution of the above coumarin-3-carboxylates **S1** (7.0 mmol) in ethyl acetate (5 mL), 10% palladium on activated charcoal (20% wt of **S1**) was added, then the mixture was charged hydrogen gas (balloon). After the reaction completion (monitored by TLC), the solid was filtered off and the volatiles were removed under the reduced pressure. Then, the residue was purified by silica gel column using hexanes/ethyl acetate (5:1) as eluent to afford the desired alkoxy carbonyl lactones **1**.

Iso-Propyl 2-oxochromane-3-carboxylate (1c):

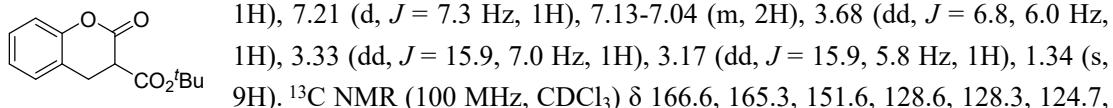
Reaction conduct under 20 mmol scale. 2.098 g, 45% yield over two steps, white solid, mp 61-62 °C, known compound, $R_f = 0.60$ (hexanes/ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.28-



128.7, 128.3, 124.8, 120.8, 116.8, 70.0, 46.6, 27.4, 21.5, 21.4. The HRMS Calculated for $\text{C}_{13}\text{H}_{15}\text{O}_4$ [$\text{M}+\text{H}]^+$ 235.0965, found: 235.0969.

tert-Butyl 2-oxochromane-3-carboxylate (1d):

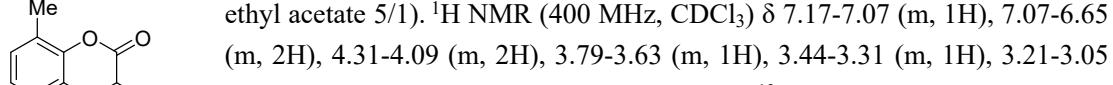
Reaction conduct under 20 mmol scale. 1.809 g, 36% yield over two steps, white solid, mp 45-46 °C, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.23 (m,



121.0, 116.7, 83.2, 47.3, 27.68, 27.65. HRMS Calculated for $\text{C}_{14}\text{H}_{17}\text{O}_4$ [$\text{M}+\text{H}]^+$ 249.1121, found: 249.1120.

Ethyl 8-methyl-2-oxochromane-3-carboxylate (1f):

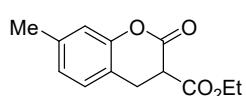
0.745 g, 45% yield over two steps, white solid, mp 65-66 °C, new compound, $R_f = 0.70$ (hexanes/



167.7, 165.1, 149.7, 130.2, 126.2, 125.7, 124.3, 120.6, 62.1, 46.4, 27.5, 15.6, 14.0. The HRMS Calculated for $\text{C}_{13}\text{H}_{15}\text{O}_4$ [$\text{M}+\text{H}]^+$ 235.0965, found: 235.0963.

Ethyl 7-methyl-2-oxochromane-3-carboxylate (1g):

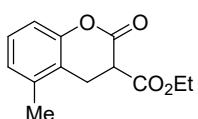
0.401 g, 24% yield over two steps, white solid, mp 58-59 °C, new compound, $R_f = 0.50$ (hexanes/



ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.08-6.93 (m, 2H), 6.93-6.82 (m, 1H), 4.25-4.06 (m, 2H), 3.73-3.59 (m, 1H), 3.35-3.23 (m, 1H), 3.13-3.00 (m, 1H), 2.26 (s, 3H), 1.31-1.08 (m, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.6, 165.0, 149.3, 134.4, 129.1, 128.7, 120.4, 116.4, 62.1, 46.4, 27.2, 20.7, 13.9. The HRMS Calculated for $\text{C}_{13}\text{H}_{15}\text{O}_4$ $[\text{M}+\text{H}]^+$ 235.0965, found: 235.0967.

Ethyl 5-methyl-2-oxochromane-3-carboxylate (1i):

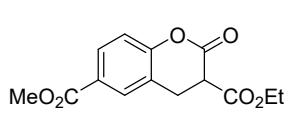
0.820 g, 50% yield over two steps, white solid, mp 61-62 °C, new compound, $R_f = 0.55$ (hexanes/



ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.19-7.07 (m, 1H), 7.03-6.85 (m, 2H), 4.27-4.11 (m, 2H), 3.79-3.66 (m, 1H), 3.39-3.29 (m, 1H), 3.15-3.04 (m, 1H), 2.31 (s, 3H), 1.21 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.7, 164.9, 151.6, 136.6, 128.1, 126.3, 119.5, 114.6, 62.2, 46.1, 24.3, 19.1, 14.0. The HRMS Calculated for $\text{C}_{13}\text{H}_{15}\text{O}_4$ $[\text{M}+\text{H}]^+$ 235.0965, found: 235.0975.

3-Ethyl 6-methyl 2-oxochromane-3,6-dicarboxylate (1l):

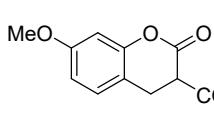
0.905 g, 46% yield over two steps, white solid, new compound, mp 72-73 °C, $R_f = 0.50$ (hexanes/



ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 8.06 -7.81 (m, 2H), 7.19-7.04 (m, 1H), 4.29-4.12 (m, 2H), 3.92 (s, 3H), 3.86-3.75 (m, 1H), 3.46 (dd, $J = 16.1, 8.0$ Hz, 1H), 3.25 (dd, $J = 16.2, 6.1$ Hz, 1H), 1.21 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.1, 166.0, 164.0, 154.6, 130.5, 130.1, 126.8, 120.8, 116.9, 62.4, 52.3, 46.0, 27.2, 14.0. The HRMS Calculated for $\text{C}_{14}\text{H}_{15}\text{O}_6$ $[\text{M}+\text{H}]^+$ 279.0863, found: 279.0866.

Ethyl 7-methoxy-2-oxochromane-3-carboxylate (1n):

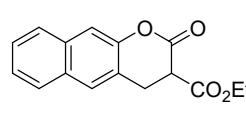
0.642 g, 37% yield over two steps, white solid, new compound, mp 75-76 °C, $R_f = 0.70$ (hexanes/



ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.18-6.98 (m, 1H), 6.82-6.34 (m, 2H), 4.30-4.09 (m, 2H), 3.94-3.60 (m, 4H), 3.39-3.23 (m, 1H), 3.19-2.99 (m, 1H), 1.20 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.6, 164.8, 160.0, 152.1, 128.7, 112.4, 110.9, 102.4, 62.1, 55.6, 46.6, 26.7, 14.0. The HRMS Calculated for $\text{C}_{13}\text{H}_{15}\text{O}_5$ $[\text{M}+\text{H}]^+$ 251.0914, found: 251.0932.

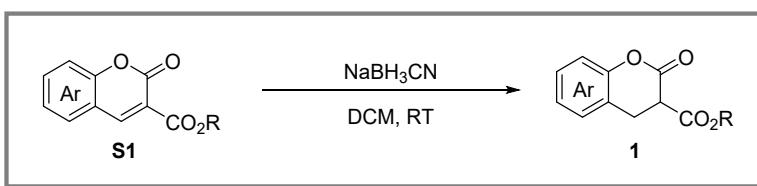
Ethyl 2-oxo-3,4-dihydro-2H-benzo[g]chromene-3-carboxylate (1p):

Reaction conducted at 1.05 mmol scale. 0.151 g, 31% yield over two steps, white solid, new com-



pound, mp 99-100 °C, $R_f = 0.60$ (hexanes/ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.79 (d, $J = 8.0$ Hz, 2H), 7.70 (s, 1H), 7.57-7.39 (m, 3H), 4.26-4.11 (m, 2H), 3.84 (dd, $J = 8.0, 5.8$ Hz, 1H), 3.58 (dd, $J = 15.7, 8.1$ Hz, 1H), 3.36 (dd, $J = 15.9, 5.6$ Hz, 1H), 1.19 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.4, 164.9, 149.4, 133.2, 130.7, 127.5, 127.44, 127.38, 126.8, 125.8, 121.0, 113.2, 62.3, 46.7, 27.9, 14.0. HRMS Calculated for $\text{C}_{16}\text{H}_{15}\text{O}_4$ $[\text{M}+\text{H}]^+$ 271.0965, found: 271.0970.

General Procedure for the Synthesis of Lactones **1 with Sodium Cyanoborohydride:**



To a solution of the coumarin-3-carboxylates **S1** (7 mmol) in dichloromethane (5 mL), sodium cyanoborohydride (0.660 g, 10.5 mmol) was added slowly at room temperature. Then the mixture was stirred at room temperature until the completion of reduction (monitored by TLC). The volatiles were removed under the reduced pressure. Then, the crude residue was purified by silica gel column using hexanes/ethyl acetate (5:1) as eluent to afford the desired substrate lactones **1**.

Benzyl 2-oxochromane-3-carboxylate (1e**):**

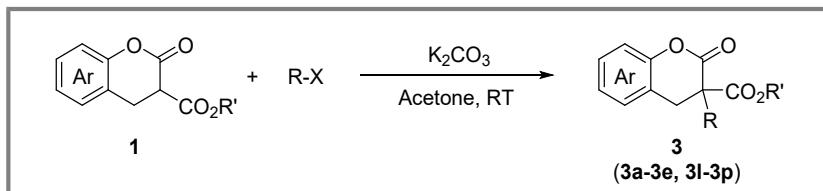
0.416 g, 18% yield over two steps, white solid, new compound, mp 61-62 °C, $R_f = 0.40$ (hexanes/ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.33-7.28 (m, 3H), 7.27-7.20 (m, 3H), 7.19-7.15 (m, 1H), 7.12-7.07 (m, 1H), 7.06-7.02 (m, 1H), 5.17 (q, $J = 12.3$ Hz, 2H), 3.80 (dd, $J = 8.5, 6.1$ Hz, 1H), 3.41 (dd, $J = 16.0, 8.5$ Hz, 1H), 3.17 (dd, $J = 16.0, 6.0$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 167.5, 164.7, 151.4, 134.9, 128.8, 128.6, 128.5, 128.3, 128.1, 124.9, 120.6, 116.9, 67.8, 46.4, 27.3. The HRMS Calculated for $\text{C}_{17}\text{H}_{15}\text{O}_4$ $[\text{M}+\text{H}]^+$ 283.0965, found: 283.0976.

Ethyl 7-chloro-2-oxochromane-3-carboxylate (1k**):**

0.366 g, 21% yield over two steps, white solid, new compound, mp 81-82 °C, $R_f = 0.60$ (hexanes/ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.22-6.96 (m, 3H), 4.26-4.15 (m, 2H), 3.76 (dd, $J = 8.0, 6.1$ Hz), 3.38 (dd, $J = 16.1, 8.0$ Hz), 3.15 (dd, $J = 16.1, 6.1$ Hz), 1.21 (t, $J = 7.1$ Hz). ^{13}C NMR (100 MHz, CDCl_3) δ 167.2, 164.0, 151.7, 134.0, 129.2, 125.0, 119.2, 117.3, 62.4, 46.1, 26.9, 14.0. HRMS Calculated for $\text{C}_{12}\text{H}_{12}\text{ClO}_4$ $[\text{M}+\text{H}]^+$ 255.0419 (^{35}Cl), found: 255.0419 (^{35}Cl). $[\text{M}+\text{H}]^+$ 257.0353 (^{37}Cl), found: 257.0393 (^{37}Cl).

2.2. Synthesis of (+/-)-Benzyl Substituted Lactones with Quaternary Stereocenter

The substrate lactones **3** with the quaternary stereocenter could be synthesized through direct alkylation of the lactones **1** with halogenated hydrocarbon according to the known methods.^[4]

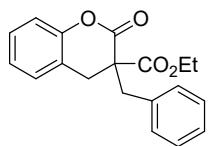


To a mixture of lactone **1** (1.5 mmol), potassium carbonate (0.622 g, 4.5 mmol) and acetone (4 mL), the halogenated hydrocarbon (1.9 mmol) was added. Then, the mixture was stirred under room temperature overnight. After the completion of the reaction (monitored by TLC), the solid was filtered, and the volatiles were removed under the reduced pressure. Then the crude residue was purified by silica gel column using hexanes/ethyl acetate (10:1-5:1) as eluent to afford the

desired products **3**.

(+/-)-Ethyl 3-benzyl-2-oxochromane-3-carboxylate (3a):

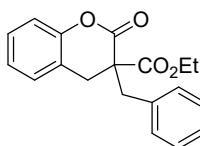
Reaction conduct under 6 mmol scale. 1.282 g, 47% yield, white solid, the known compound,^[5] R_f



= 0.80 (hexanes/ethyl acetate 5/1), ^1H NMR (400 MHz, CDCl_3) δ 7.31-7.19 (m, 6H), 7.14-7.00 (m, 3H), 4.09-3.97 (m, 2H), 3.50 (d, $J = 13.8$ Hz, 1H), 3.32 (d, $J = 13.8$ Hz, 1H), 3.22 (d, $J = 15.8$ Hz, 1H), 2.87 (d, $J = 15.8$ Hz, 1H), 0.99 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.5, 167.1, 151.0, 135.0, 130.8, 128.5, 128.42, 128.38, 127.4, 124.7, 121.5, 116.4, 62.2, 54.6, 39.3, 32.1, 13.8.

(+/-)-Ethyl 3-(4-methylbenzyl)-2-oxochromane-3-carboxylate (3b):

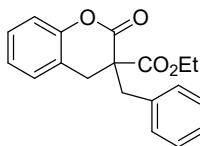
0.431 g, 89% yield, pale pink solid, mp 110-111 °C, new compound, $R_f = 0.80$ (hexanes/ethyl



acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.24-7.00 (m, 8H), 4.11-3.96 (m, 2H), 3.46 (d, $J = 13.8$ Hz, 1H), 3.31-3.18 (m, 2H), 2.87 (d, $J = 15.8$ Hz, 1H), 2.32 (s, 3H), 1.01 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 167.1, 151.0, 137.0, 131.9, 130.6, 129.1, 128.5, 128.4, 124.6, 121.5, 116.4, 62.2, 54.6, 38.9, 32.0, 21.1, 13.8. HRMS Calculated for $\text{C}_{20}\text{H}_{21}\text{O}_4$ [M+H]⁺ 325.1434, found: 325.1437.

(+/-)-Ethyl 3-(4-fluorobenzyl)-2-oxochromane-3-carboxylate (3c):

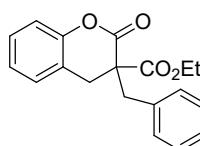
0.370 g, 75% yield, colorless oil, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 5/1). ^1H NMR



(400 MHz, CDCl_3) δ 7.29-7.20 (m, 3H), 7.18-6.90 (m, 5H), 4.14-3.91 (m, 2H), 3.45 (d, $J = 13.9$ Hz, 1H), 3.33-3.17 (m, 2H), 2.87 (d, $J = 15.7$ Hz, 1H), 0.99 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.4, 167.0, 162.2 (d, $^1J_{\text{F-C}} = 245.8$ Hz), 150.9, 132.3 (d, $^3J_{\text{F-C}} = 8.0$ Hz), 130.7 (d, $^4J_{\text{F-C}} = 3.5$ Hz), 128.5 (d, $^3J_{\text{F-C}} = 21.9$ Hz), 124.7, 121.2, 116.4, 115.2 (d, $^2J_{\text{F-C}} = 21.4$ Hz), 62.3, 54.6, 38.5, 32.2, 13.7. ^{19}F NMR (376 MHz, CDCl_3) δ -115.21. HRMS Calculated for $\text{C}_{19}\text{H}_{18}\text{FO}_4$ [M+H]⁺ 329.1184, found: 329.1183.

(+/-)-Ethyl 3-(naphthalen-2-ylmethyl)-2-oxochromane-3-carboxylate (3d):

0.476 g, 88% yield, pale yellow solid, mp 123-124 °C, new compound, $R_f = 0.70$ (hexanes/ethyl

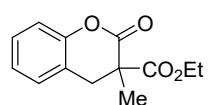


acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.84-7.68 (m, 4H), 7.51-7.37 (m, 3H), 7.24-7.17 (m, 1H), 7.13-6.96 (m, 3H), 4.13-3.96 (m, 2H), 3.58 (dd, $J = 76.6, 13.8$ Hz, 2H), 3.26 (d, $J = 15.8$ Hz, 1H), 2.89 (d, $J = 15.8$ Hz, 1H), 0.99 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 167.2, 151.0, 133.3, 132.62, 132.60, 129.7, 128.7, 128.5, 128.4, 127.9, 127.8, 127.6, 126.2, 126.0, 124.7, 121.4, 116.4, 62.3, 54.7, 39.4, 32.1, 13.8. HRMS Calculated for $\text{C}_{23}\text{H}_{21}\text{O}_4$ [M+H]⁺ 361.1434, found: 361.1431.

(+/-)-Ethyl 3-methyl-2-oxochromane-3-carboxylate (3e):

0.346 g, 99% yield, white solid, mp 92-93 °C, known compound,^[4] $R_f = 0.60$

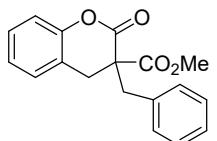
(hexanes/ethyl acetate 10/1). ^1H NMR (400 MHz, CDCl_3) δ 7.29-7.23 (m, 1H), 7.18 (d, $J = 7.3$ Hz, 1H), 7.14-6.98 (m, 2H), 4.14-3.94 (m, 2H), 3.36 (d, $J =$



15.8 Hz, 1H), 2.99 (d, J = 15.8 Hz, 1H), 1.62 (s, 3H), 1.00 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 170.6, 168.2, 151.5, 128.7, 128.2, 124.6, 121.3, 116.5, 62.1, 49.5, 35.4, 20.9, 13.7.

(+/-)-Methyl 3-benzyl-2-oxochromane-3-carboxylate (3l):

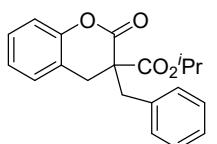
0.275 g, 59% yield, white solid, mp 87-88 °C, new compound, R_f = 0.30 (hexanes/ethyl acetate 30/1).



^1H NMR (400 MHz, CDCl_3) δ 7.34-7.20 (m, 6H), 7.15-6.98 (m, 3H), 3.60 (s, 3H), 3.40 (dd, J = 41.2, 13.8 Hz, 2H), 3.07 (dd, J = 154.4, 15.9 Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 166.9, 150.8, 134.9, 130.7, 128.6, 128.5, 128.4, 127.5, 124.8, 121.3, 116.4, 54.5, 53.1, 39.5, 31.8. HRMS Calculated for $\text{C}_{18}\text{H}_{17}\text{O}_4$ [M+H]⁺ 297.1121, found: 297.1131.

(+/-)-Isopropyl 3-benzyl-2-oxochromane-3-carboxylate (3m):

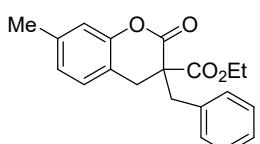
0.396 g, 81% yield, pale yellow solid, mp 101-102 °C, new compound, R_f = 0.75 (hexanes/ethyl acetate 10/1).



^1H NMR (400 MHz, CDCl_3) δ 7.34-7.26 (m, 5H), 7.24-7.18 (m, 1H), 7.12 (d, J = 7.1 Hz, 1H), 7.09-6.98 (m, 2H), 4.91-4.76 (m, 1H), 3.51 (d, J = 13.8 Hz, 1H), 3.30 (d, J = 13.8 Hz, 1H), 3.19 (d, J = 15.7 Hz, 1H), 2.87 (d, J = 15.7 Hz, 1H), 1.04 (d, J = 6.3 Hz, 3H), 0.87 (d, J = 6.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.1, 167.3, 151.1, 135.2, 130.8, 128.5, 128.4, 128.3, 127.3, 124.6, 121.6, 116.3, 70.1, 54.7, 39.1, 32.3, 21.2, 21.1. HRMS Calculated for $\text{C}_{20}\text{H}_{21}\text{O}_4$ [M+H]⁺ 325.1434, found: 325.1436.

(+/-)-Ethyl 3-benzyl-7-methyl-2-oxochromane-3-carboxylate (3n):

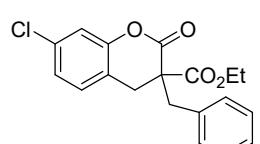
0.311 g, 64% yield, white solid, mp 104-105 °C, new compound, R_f = 0.75 (hexanes/ethyl acetate 5/1).



^1H NMR (400 MHz, CDCl_3) δ 7.30-7.25 (m, 5H), 7.03-6.95 (m, 1H), 6.93-6.78 (m, 2H), 4.12-3.97 (m, 2H), 3.39 (dd, J = 57.7, 13.8 Hz, 2H), 3.19 (d, J = 15.8 Hz, 1H), 2.82 (d, J = 15.8 Hz, 1H), 2.30 (s, 3H), 1.02 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 167.3, 150.8, 138.7, 135.1, 130.8, 128.3, 128.1, 127.3, 125.4, 118.2, 116.8, 62.2, 54.7, 39.3, 31.7, 21.2, 13.8. HRMS Calculated for $\text{C}_{20}\text{H}_{21}\text{O}_4$ [M+H]⁺ 325.1434, found: 325.1440.

(+/-)-Ethyl 3-benzyl-7-chloro-2-oxochromane-3-carboxylate (3o):

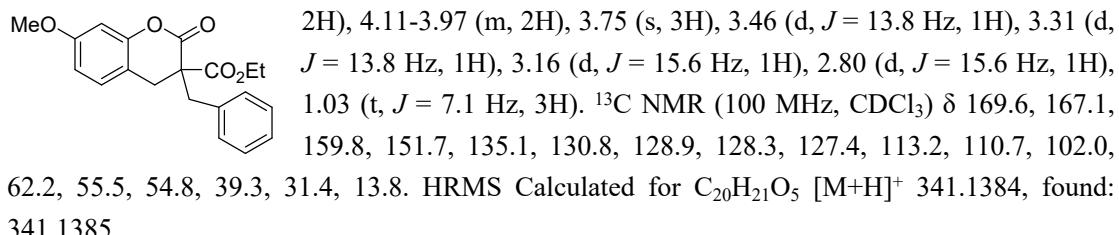
0.378 g, 73% yield, white solid, mp 125-126 °C, new compound, R_f = 0.65 (hexanes/ethyl acetate 10/1).



^1H NMR (400 MHz, CDCl_3) δ 7.32-7.24 (m, 5H), 7.13-6.94 (m, 3H), 4.18-3.90 (m, 2H), 3.48 (d, J = 13.8 Hz, 1H), 3.31 (d, J = 13.8 Hz, 1H), 3.21 (d, J = 15.9 Hz, 1H), 2.83 (d, J = 15.9 Hz, 1H), 1.05 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.2, 166.3, 151.3, 134.7, 133.7, 130.7, 129.3, 128.5, 127.5, 124.8, 120.1, 116.8, 62.5, 54.4, 39.4, 31.6, 13.8. HRMS Calculated for $\text{C}_{19}\text{H}_{17}\text{ClO}_4\text{Na}$ [M+Na]⁺ 367.0708 (³⁵Cl), found: 367.0705 (³⁵Cl). [M+H]⁺ 369.0691 (³⁷Cl), found: 369.0680 (³⁷Cl).

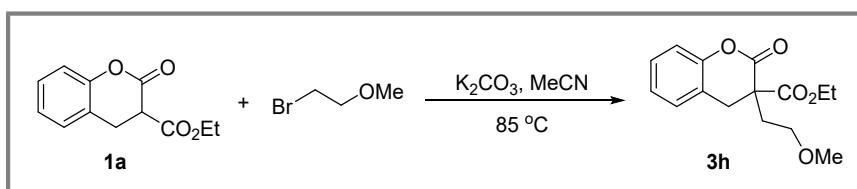
(+/-)-Ethyl 3-benzyl-7-methoxy-2-oxochromane-3-carboxylate (3p):

0.433 g, 85% yield, white solid, mp 127-128 °C, new compound, R_f = 0.70 (hexanes/ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.31-7.23 (m, 5H), 7.00 (d, J = 8.3 Hz, 1H), 6.68-6.50 (m,



2.3. Synthesis of (+/-)-Ethyl 3-(2-Methoxyethyl)-2-oxochromane-3-carboxylate (3h)

The lactone **3h** with quaternary stereogenic center could be conveniently synthesized through alkylation of lactone **1a** with 1-bromo-2-methoxyethane in the presence of potassium carbonate.

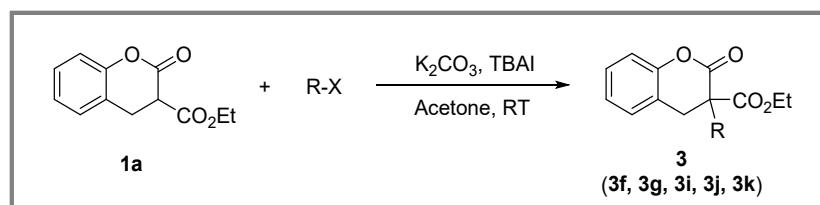


To a mixture of lactone **1a** (0.327 g, 1.5 mmol), potassium carbonate (0.622 g, 4.5 mmol) and acetonitrile (5 mL), 1-bromo-2-methoxyethane (0.18 mL, 1.9 mmol) was added. Then the mixture was refluxed under 85 °C overnight. After the completion of the alkylation (monitored by TLC), the solid was filtered and the volatiles were removed under the reduced pressure. Then the crude residue was purified by silica gel column using hexanes/ethyl acetate (10:1-5:1) as eluent to afford the desired product **3h**.

0.245 g, 58% yield, colorless oil, new compound, R_f = 0.50 (hexanes/ethyl acetate 5/1). ^1H NMR (400 MHz, CDCl_3) δ 7.27-7.16 (m, 2H), 7.12-7.01 (m, 2H), 4.12-3.96 (m, 2H), 3.64 (t, J = 6.1 Hz, 2H), 3.37 (d, J = 15.9 Hz, 1H), 3.29 (s, 3H), 3.11 (d, J = 15.8 Hz, 1H), 2.28 (t, J = 6.1 Hz, 2H), 1.02 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.6, 167.6, 151.1, 128.6, 128.4, 124.6, 121.5, 116.4, 68.8, 62.1, 58.6, 52.0, 34.1, 33.4, 13.7. HRMS Calculated for $\text{C}_{15}\text{H}_{19}\text{O}_5$ [M+H]⁺ 279.1227, found: 279.1227.

2.4. Synthesis of (+/-)-Alkyl Substituted Lactones with Quaternary Center

The substrate lactones with quaternary carbon center **3** were synthesized through alkylation of the above lactone **1a** with alkyl halides.



To a mixture of lactone **1a** (0.327 g, 1.5 mmol), potassium carbonate (0.622 g, 4.5 mmol), *tetra*-butylammonium iodide (0.554 g, 1.5 mmol) and acetone (4.0 mL), halogenated hydrocarbons (1.9 mmol) was added. Then the mixture was stirred under room temperature overnight. After the completion of the reaction (monitored by TLC), the solid was filtered, and the volatiles were

removed under the reduced pressure. Then, the crude residue was purified by silica gel column using hexanes/ethyl acetate (10:1-5:1) as eluent to afford the desired products **3**.

(+/-)-Ethyl 3-ethyl-2-oxochromane-3-carboxylate (3f):

0.200 g, 54% yield, colorless oil, known compound,^[4] $R_f = 0.70$ (hexanes/ethyl acetate 10/1). ^1H NMR (400 MHz, CDCl_3) δ 7.26-7.16 (m, 2H), 7.12-6.96 (m, 2H), 4.14-3.94 (m, 2H), 3.29 (d, $J = 15.7$ Hz, 1H), 3.01 (d, $J = 15.7$ Hz, 1H), 2.13-1.97 (m, 2H), 1.10-0.96 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.8, 167.5, 151.2, 128.6, 128.4, 124.6, 121.5, 116.4, 61.9, 53.6, 32.3, 27.4, 13.8, 9.1.

(+/-)-Ethyl 3-methyl-2-oxochromane-3-carboxylate (3g):

0.366 g, 88% yield, colorless oil, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 10/1). ^1H NMR (400 MHz, CDCl_3) δ 7.25-7.12 (m, 2H), 7.11-6.96 (m, 2H), 4.12-3.93 (m, 2H), 3.29 (d, $J = 15.7$ Hz, 1H), 3.01 (d, $J = 15.7$ Hz, 1H), 2.05-1.89 (m, 2H), 1.54-1.44 (m, 1H), 1.40-1.28 (m, 3H), 1.00 (t, $J = 7.1$ Hz, 3H), 0.91 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.9, 167.6, 151.2, 128.5, 128.3, 124.6, 121.6, 116.4, 61.9, 53.3, 34.0, 32.7, 26.7, 23.0, 13.9, 13.8. The HRMS Calculated for $\text{C}_{16}\text{H}_{21}\text{O}_4$ $[\text{M}+\text{H}]^+$ 277.1434, found: 277.1437.

(+/-)-Ethyl 3-(cyclopropylmethyl)-2-oxochromane-3-carboxylate (3i):

0.171 g, 42% yield, colorless oil, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 10/1). ^1H NMR (400 MHz, CDCl_3) δ 7.26-7.15 (m, 2H), 7.11-6.97 (m, 2H), 4.12-3.96 (m, 2H), 3.38 (d, $J = 15.8$ Hz, 1H), 3.19 (d, $J = 15.8$ Hz, 1H), 2.00-1.87 (m, 2H), 1.03 (t, $J = 7.1$ Hz, 3H), 0.99-0.85 (m, 1H), 0.55-0.44 (m, 2H), 0.16-0.04 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.8, 167.7, 151.1, 128.5, 128.4, 124.6, 121.8, 116.4, 62.0, 53.9, 39.0, 32.7, 13.8, 6.6, 4.9, 4.4. HRMS Calculated for $\text{C}_{16}\text{H}_{18}\text{O}_4\text{K}$ $[\text{M}+\text{K}]^+$ 313.0837, found: 313.0840.

(+/-)-Ethyl 3-(cyclobutylmethyl)-2-oxochromane-3-carboxylate (3j):

0.143 g, 33% yield, colorless oil, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 10/1). ^1H NMR (400 MHz, CDCl_3) δ 7.26-7.14 (m, 2H), 7.11-6.96 (m, 2H), 4.10-3.92 (m, 2H), 3.31 (d, $J = 15.8$ Hz, 1H), 2.94 (d, $J = 15.7$ Hz, 1H), 2.67-2.47 (m, 1H), 2.19-1.99 (m, 4H), 1.90-1.66 (m, 4H), 1.02 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 169.7, 167.6, 151.0, 128.5, 128.4, 124.6, 121.7, 116.4, 62.0, 53.1, 41.0, 32.6, 32.4, 29.9, 29.6, 18.9, 13.8. HRMS Calculated for $\text{C}_{17}\text{H}_{21}\text{O}_4$ $[\text{M}+\text{H}]^+$ 289.1434, found: 289.1437.

(+/-)-Ethyl 3-(cyclohexylmethyl)-2-oxochromane-3-carboxylate (3k):

0.288 g, 61% yield, colorless oil, new compound, $R_f = 0.70$ (hexanes/ethyl acetate 10/1). ^1H NMR (400 MHz, CDCl_3) δ 7.25-7.14 (m, 2H), 7.11-6.98 (m, 2H), 4.10-3.96 (m, 2H), 3.33 (d, $J = 15.7$ Hz, 1H), 3.02 (d, $J = 15.7$ Hz, 1H), 1.96-1.87 (m, 2H), 1.74-1.58 (m, 5H), 1.55-1.43 (m, 1H), 1.26-1.08 (m, 3H), 1.07-0.95 (m, 5H). ^{13}C NMR (100 MHz, CDCl_3) δ 170.2, 167.7, 151.1, 128.5, 128.4, 124.6,

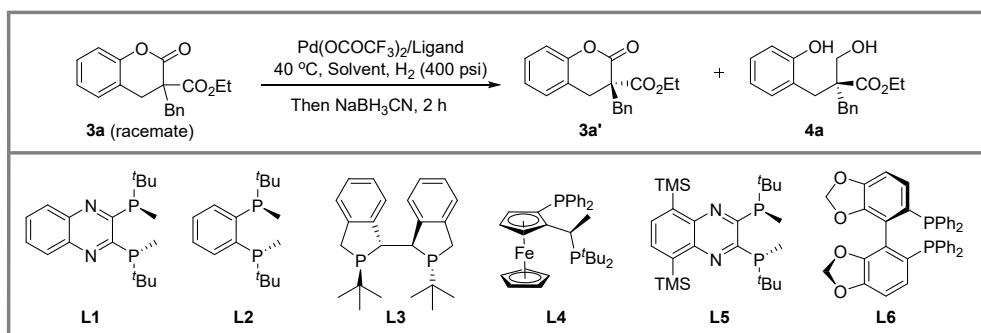
121.6, 116.4, 62.0, 53.1, 40.8, 34.7, 34.4, 34.3, 32.7, 26.3, 26.1, 13.7. HRMS Calculated for C₁₉H₂₅O₄ [M+H]⁺ 317.1747, found: 317.1758.

3. Condition Optimization for Kinetic Resolution

General Procedure: The $\text{Pd}(\text{OCOCF}_3)_2$ (0.015 mmol, 4.8 mg, 5 mol%), ligand (0.018 mmol, 6 mol%) and degassed anhydrous acetone (1.0 mL) were placed in a dried Schlenk tube under nitrogen atmosphere. The mixture was stirred under room temperature for 1 hour, the solvent was removed under vacuum to give the catalyst. Then, this catalyst was taken into a glove box filled with nitrogen gas and dissolved in solvent (1.0 mL). To the lactones **3a** (0.3 mmol, 93.0 mg) added this catalyst solution, and then the mixture was transferred to an autoclave, which was charged hydrogen gas (400 psi). The reaction mixture was stirred at 40 °C for 48 hours. After cooling to room temperature and the release of the hydrogen gas, the autoclave was opened. To the mixture added sodium cyanoborohydride (7.5 mg, 0.12 mmol). The mixture was stirred under room temperature for 2 hours, then the volatiles were removed under the reduced pressure. The crude residue was purified by column chromatography on silica gel using hexanes/ethyl acetate (10:1-5:1) as eluent to give the chiral reductive products **3a'** and **4a**.

The optimal conditions were identified as follow: Lactone **3a** (0.3 mmol), $\text{Pd}(\text{OCOCF}_3)_2$ (5 mol%), ligand **L5** (6 mol%), H_2 (400 psi), HFIP (1.0 mL), 40 °C, 48 h (entry 8).

Table S1. Optimization of the KR Reaction Conditions

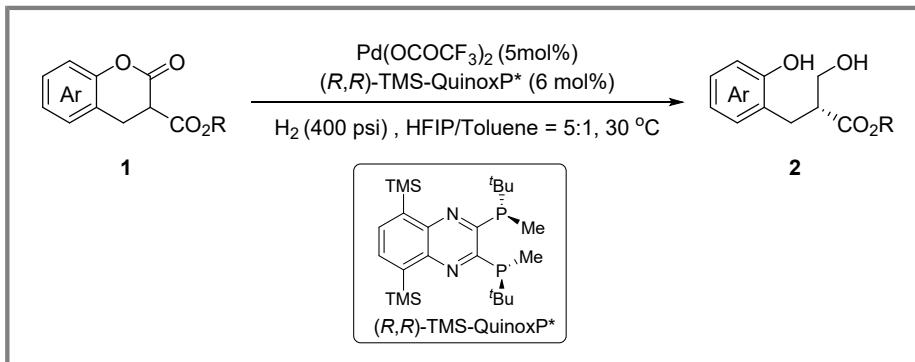


Entry ^a	Ligand	Solvent	Conv. [%] ^b	Yield of 4a ^b	Ee [%] ^c		<i>S</i> ^d
					3a'	4a	
1	L1	TFE	13	12	31	77	10.6
2	L1	DCM	<5	<5	-	-	-
3	L1	HFIP	54	52	89	72	17.6
4	L1	Toluene	<5	<5	-	-	-
5	L2	HFIP	62	61	96	50	11.3
6	L3	HFIP	54	54	95	56	12.3
7	L4	HFIP	>95	>95	-	-	-
8	L5	HFIP	53	54 (52) ^e	93	71	19.3
9	L6	HFIP	<5	<5	-	-	-
10	L5	HFIP/Toluene = 5:1	42	42	59	82	18.4
11	L5	HFIP/DCM = 5:1	53	52	77	75	16.2
12	L5	HFIP/TFE = 5:1	55	54	84	76	18.9

^a Lactone **3a** (0.3 mmol), $\text{Pd}(\text{OCOCF}_3)_2$ (5 mol%), chiral ligand (6 mol%), H_2 (400 psi), solvent (1 mL), 40 °C, 48 h. ^bDetermined by NMR, using CH_2Br_2 as internal standard. ^cDetermined by HPLC. ^dCalculated selectivity factors: *C* = ee of SM/(ee of SM + ee of TM), *s* = $\ln[(1 - \text{C})(1 - \text{ee of SM})]/\ln[(1 - \text{C})(1 + \text{ee of SM})]$. ^eIsolated yield based on the lactone **3a**.

4. Pd-catalyzed Hydrogenative DKR and KR of Lactones

4.1 Pd-catalyzed Hydrogenative Dynamic Kinetic Resolution of Lactones



General Procedure: $\text{Pd}(\text{OCOCF}_3)_2$ (0.01 mmol, 3.2 mg, 5 mol%), chiral ligand (R,R) -TMS-QuinoxP* (0.012 mmol, 5.8 mg, 6 mol%) and degassed anhydrous acetone (1.0 mL) were placed in a dried Schlenk tube under nitrogen atmosphere. The mixture was stirred under room temperature for 1 hour, the solvent was removed under vacuum to give the catalyst. Then, this catalyst was taken into a glove box filled with nitrogen and dissolved in solvent (HFIP/toluene = 5:1, 1.0 mL). To the lactones **1** (0.2 mmol) was added the catalyst solution, and the mixture was transferred to an autoclave, which was charged hydrogen gas (400 psi). The reaction mixture was stirred at 30 °C for 48 hours. After release of the hydrogen gas, the autoclave was opened, and the volatiles were removed under the reduced pressure. Then the residue was purified by column chromatography on silica gel using dichloromethane/methanol (40:1) as eluent to give the reductive products **2**.

The racemates were prepared by reduction of lactones **1** with sodium borohydride in methanol.

(+)-Ethyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (2a):

44 mg, 98% yield, colorless oil, new compound, R_f = 0.30 (dichloromethane/methanol 40/1), 91%

ee, $[\alpha]^{20}_D$ = +11.28 (c 0.86, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.54 (brs, 1H), 7.19-6.99 (m, 2H), 6.97-6.70 (m, 2H), 4.31-4.09 (m, 2H), 3.95-3.80 (m, 1H), 3.69-3.58 (m, 1H), 3.40 (brs, 1H), 3.09-2.91 (m, 2H), 2.88-2.74 (m, 1H), 1.26 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.2, 154.9, 131.1, 128.3, 124.6, 120.8, 116.7, 61.5, 61.2, 47.7, 28.1, 14.2. HPLC: Chiraldak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 16.6 min and 19.3 min (major). HRMS Calculated for $\text{C}_{12}\text{H}_{17}\text{O}_4$ [M+H]⁺ 225.1121, found: 225.1120.

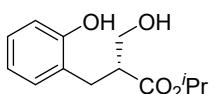
(+)-Methyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (2b):

33 mg, 79% yield, colorless oil, new compound, R_f = 0.30 (dichloromethane/methanol 40/1), 83%

ee, $[\alpha]^{20}_D$ = +9.85 (c 0.66, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.46 (brs, 1H), 7.15-7.06 (m, 2H), 6.90-6.80 (m, 2H), 3.91-3.85 (m, 1H), 3.73 (s, 3H), 3.67-3.60 (m, 1H), 3.36 (brs, 1H), 3.06-2.94 (m, 2H), 2.88-2.80 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.6, 154.8, 131.1, 128.3, 124.5, 120.8, 116.7, 61.4, 52.2, 47.6, 28.1. The HPLC: Chiraldak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 17.2 min (major) and 18.7 min. HRMS Calculated for $\text{C}_{11}\text{H}_{15}\text{O}_4$ [M+H]⁺ 211.0965, found: 211.0970.

(+)-Isopropyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (2c):

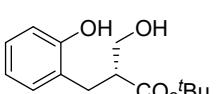
46 mg, 97% yield, colorless oil, new compound, $R_f = 0.35$ (dichloromethane/methanol 40/1), 89%



ee, $[\alpha]^{20}_D = +13.59$ (c 0.92, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.58 (brs, 1H), 7.16-7.02 (m, 2H), 6.94-6.74 (m, 2H), 5.15-4.96 (m, 1H), 3.88 (dd, $J = 11.5, 3.8$ Hz, 1H), 3.62 (dd, $J = 11.4, 4.5$ Hz, 1H), 3.39 (brs, 1H), 3.08-2.91 (m, 2H), 2.84-2.69 (m, 1H), 1.32-1.16 (m, 6H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.8, 154.9, 131.1, 128.3, 124.6, 120.7, 116.8, 68.8, 61.5, 47.8, 28.2, 21.8, 21.7. The HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 12.8 min and 15.2 min (major). HRMS Calculated for $\text{C}_{13}\text{H}_{19}\text{O}_4$ $[\text{M}+\text{H}]^+$ 239.1278, found: 239.1280.

(+)-*tert*-Butyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (2d):

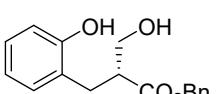
44 mg, 87% yield, colorless oil, new compound, $R_f = 0.37$ (dichloromethane/methanol 40/1), 81%



ee, $[\alpha]^{20}_D = +13.41$ (c 0.88, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.51 (brs, 1H), 7.20-7.05 (m, 2H), 6.98-6.74 (m, 2H), 3.89 (dd, $J = 11.5, 3.4$ Hz, 1H), 3.55 (dd, $J = 11.2, 3.2$ Hz, 1H), 3.19 (brs, 1H), 3.08-2.88 (m, 2H), 2.78-2.61 (m, 1H), 1.47 (s, 9H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.9, 155.2, 131.1, 128.3, 124.7, 120.7, 117.2, 82.1, 61.5, 48.3, 28.3, 28.1. HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 14.8 min and 16.8 min (major). HRMS Calculated for $\text{C}_{14}\text{H}_{20}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 275.1259, found: 275.1249.

(+)-Benzyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (2e):

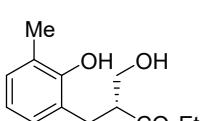
56 mg, 99% yield, white solid, new compound, mp 78-79 °C, $R_f = 0.20$ (dichloromethane/me-



thanol 40/1), 88% ee, $[\alpha]^{20}_D = +7.82$ (c 1.12, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.71-7.27 (m, 6H), 7.15-7.04 (m, 2H), 6.91-6.77 (m, 2H), 5.24-5.09 (m, 2H), 3.91 (dd, $J = 11.4, 3.7$ Hz, 1H), 3.66 (dd, $J = 11.5, 4.5$ Hz, 1H), 3.31 (brs, 1H), 3.10-2.98 (m, 2H), 2.93-2.85 (m, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.9, 154.8, 135.5, 131.1, 128.7, 128.5, 128.33, 128.30, 124.5, 120.9, 116.7, 66.9, 61.4, 47.7, 28.1. The HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 22.6 min (minor) and 24.4 min (major). HRMS Calculated for $\text{C}_{17}\text{H}_{19}\text{O}_4$ $[\text{M}+\text{H}]^+$ 287.1279, found: 287.1279.

(+)-Ethyl 3-hydroxy-2-(2-hydroxy-3-methylbenzyl)propanoate (2f):

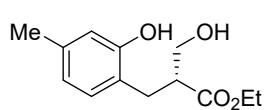
47 mg, 99% yield, pale yellow oil, new compound, $R_f = 0.40$ (dichloromethane/methanol 40/1),



72% ee, $[\alpha]^{20}_D = +8.08$ (c 0.94, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.24 (brs, 1H), 7.00 (dd, $J = 21.8, 7.4$ Hz, 2H), 6.83-6.71 (m, 1H), 4.22 (q, $J = 7.1$ Hz, 2H), 3.93 (dd, $J = 11.5, 3.3$ Hz, 1H), 3.58 (d, $J = 8.9$ Hz, 1H), 3.11 (brs, 1H), 3.07-2.96 (m, 2H), 2.79-2.72 (m, 1H), 2.26 (s, 3H), 1.30 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.5, 153.4, 129.6, 128.5, 125.9, 124.0, 120.3, 61.31, 61.26, 47.7, 28.3, 16.5, 14.2. HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 10.0 min and 10.6 min (major). The HRMS Calculated for $\text{C}_{13}\text{H}_{19}\text{O}_4$ $[\text{M}+\text{H}]^+$ 239.1278, found: 239.1274.

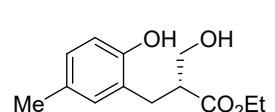
(+)-Ethyl 3-hydroxy-2-(2-hydroxy-4-methylbenzyl)propanoate (2g):

43 mg, 90% yield, colorless oil, new compound, $R_f = 0.30$ (DCM/methanol 40/1), 89% ee, $[\alpha]^{20}_D$

 $= +9.77$ (c 0.86, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.45 (brs, 1H), 6.97 (d, $J = 7.4$ Hz, 1H), 6.77-6.57 (m, 2H), 4.19 (q, $J = 7.1$ Hz, 2H), 3.88 (dd, $J = 11.4, 3.6$ Hz, 1H), 3.64 (d, $J = 7.9$ Hz, 1H), 3.40 (brs, 1H), 3.05-2.89 (m, 2H), 2.85-2.73 (m, 1H), 2.26 (s, 3H), 1.27 (t, $J = 7.1$ Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.3, 154.7, 138.3, 130.8, 121.6, 121.4, 117.3, 61.4, 61.2, 47.8, 27.8, 21.1, 14.2. HPLC: Chiralpak IB, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 21.2 min (major) and 22.8 min. HRMS Calculated for C₁₃H₁₉O₄ [M+H]⁺ 239.1278, found: 239.1282.

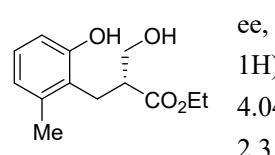
(+)-Ethyl 3-hydroxy-2-(2-hydroxy-5-methylbenzyl)propanoate (2h):

42 mg, 88% yield, colorless oil, new compound, $R_f = 0.40$ (dichloromethane/methanol 40/1), 90%

 $ee, [\alpha]^{20}_D = +8.33$ (c 0.84, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 6.99-6.86 (m, 2H), 6.76 (d, $J = 8.8$ Hz, 1H), 4.25-4.13 (m, 2H), 3.89 (dd, $J = 11.5, 3.7$ Hz, 1H), 3.62 (dd, $J = 11.6, 4.5$ Hz, 1H), 3.05-2.92 (m, 2H), 2.83-2.74 (m, 1H), 2.24 (s, 3H), 1.27 (t, $J = 7.1$ Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.3, 152.6, 131.5, 129.9, 128.8, 124.3, 116.6, 61.4, 61.2, 47.7, 28.1, 20.5, 14.2. HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 25.2 min and 27.6 min (major). HRMS Calculated for C₁₃H₁₉O₄ [M+H]⁺ 239.1278, found: 239.1278.

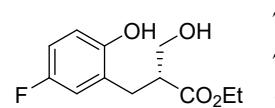
(+)-Ethyl 3-hydroxy-2-(2-hydroxy-6-methylbenzyl)propanoate (2i):

41 mg, 86% yield, colorless oil, new compound, $R_f = 0.40$ (dichloromethane/methanol 40/1), 80%

 $ee, [\alpha]^{20}_D = +17.19$ (c 0.82, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.58 (brs, 1H), 7.08-6.95 (m, 1H), 6.75 (d, $J = 7.8$ Hz, 2H), 4.22 (q, $J = 7.1$ Hz, 2H), 4.04-3.87 (m, 1H), 3.68-3.38 (m, 2H), 3.14-2.96 (m, 2H), 2.81-2.65 (m, 1H), 2.31 (s, 3H), 1.29 (t, $J = 7.1$ Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.5, 155.2, 138.1, 127.6, 123.4, 122.8, 114.5, 61.6, 61.3, 45.9, 24.4, 19.4, 14.2. HPLC: Chiral pak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 13.2 min and 16.6 min (major). HRMS Calculated for C₁₃H₁₉O₄ [M+H]⁺ 239.1278, found: 239.1272.

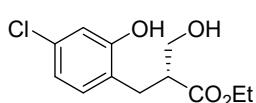
(+)-Ethyl 2-(5-fluoro-2-hydroxybenzyl)-3-hydroxypropanoate (2j):

47 mg, 97% yield, pale yellow oil, new compound, $R_f = 0.30$ (dichloromethane/methanol 40/1),

 $79\% ee, [\alpha]^{20}_D = +12.73$ (c 0.94, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.38 (brs, 1H), 6.96-6.73 (m, 3H), 4.20 (q, $J = 7.1$ Hz, 2H), 3.91 (dd, $J = 11.5, 3.6$ Hz, 1H), 3.60 (dd, $J = 11.5, 4.1$ Hz, 1H), 3.21 (brs, 1H), 3.05-2.91 (m, 2H), 2.85-2.73 (m, 1H), 1.28 (t, $J = 7.1$ Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.0, 157.0 (d, ¹J_{F-C} = 238.1 Hz), 151.0 (d, ⁴J_{F-C} = 2.0 Hz), 126.1 (d, ³J_{F-C} = 7.2 Hz), 118.0 (d, ³J_{F-C} = 8.1 Hz), 116.9 (d, ²J_{F-C} = 22.6 Hz), 114.7 (d, ²J_{F-C} = 22.7 Hz), 61.4, 61.3, 47.4, 28.2, 14.1. ¹⁹F NMR (376 MHz, CDCl₃) δ -124.01. HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 10.2 min and 11.5 min (major). HRMS Calculated for C₁₂H₁₆FO₄ [M+H]⁺ 243.1027, found: 243.1027.

(+)-Ethyl 2-(4-chloro-2-hydroxybenzyl)-3-hydroxypropanoate (2k):

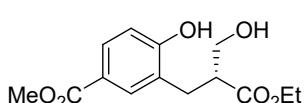
50 mg, 97% yield, colorless oil, new compound, $R_f = 0.40$ (dichloromethane/methanol 40/1), 81%



ee, $[\alpha]^{20}_D = +9.97$ (c 1.00, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.85 (brs, 1H), 7.01 (d, J = 8.1 Hz, 1H), 6.91-6.72 (m, 2H), 4.19 (q, J = 7.1 Hz, 2H), 3.89 (dd, J = 11.5, 3.7 Hz, 1H), 3.63 (dd, J = 11.4, 4.4 Hz, 1H), 3.33 (brs, 1H), 3.04-2.89 (m, 2H), 2.84-2.73 (m, 1H), 1.27 (t, J = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.1, 155.7, 133.2, 131.8, 123.2, 120.8, 117.1, 61.44, 61.41, 47.5, 27.8, 14.2. HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 14.4 min and 16.2 min (major). HRMS Calculated for C₁₂H₁₆ClO₄ [M+H]⁺ Cl³⁵ 259.0732, found: 259.0746. [M+H]⁺ Cl³⁷ 261.0706, found: 261.0716.

(+)-Methyl 3-(3-ethoxy-2-(hydroxymethyl)-3-oxopropyl)-4-hydroxybenzoate (2l):

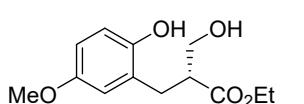
56 mg, 99% yield, colorless oil, new compound, $R_f = 0.35$ (dichloromethane/methanol 40/1), 76%



ee, $[\alpha]^{20}_D = +31.16$ (c 1.12, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.91-7.77 (m, 2H), 6.90 (d, J = 8.4 Hz, 1H), 4.26-4.14 (m, 2H), 3.92 (dd, J = 11.6, 3.8 Hz, 1H), 3.88 (s, 3H), 3.63 (dd, J = 11.6, 4.6 Hz, 1H), 3.07-2.99 (m, 2H), 2.87-2.81 (m, 1H), 1.29 (t, J = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.1, 167.1, 159.7, 133.2, 130.4, 124.5, 122.4, 116.8, 61.4, 52.0, 47.4, 28.1, 14.1. HPLC: Chiralpak AS-H column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 22.5 min and 26.0 min (major). The HRMS Calculated for C₁₄H₁₈O₆K [M+K]⁺ 321.0735, found: 321.0728.

(+)-Ethyl 3-hydroxy-2-(2-hydroxy-5-methoxybenzyl)propanoate (2m):

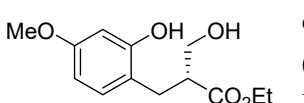
30 mg, 59% yield, colorless oil, new compound, $R_f = 0.40$ (dichloromethane/methanol 40/1), 89%



ee, $[\alpha]^{20}_D = +4.83$ (c 0.60, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.05 (brs, 1H), 6.81 (d, J = 8.6 Hz, 1H), 6.78-6.54 (m, 2H), 4.20 (q, J = 7.1 Hz, 2H), 3.91 (dd, J = 11.4, 3.2 Hz, 1H), 3.74 (s, 3H), 3.58 (d, J = 10.9 Hz, 1H), 3.16 (brs, 1H), 3.08-2.91 (m, 2H), 2.85-2.72 (m, 1H), 1.28 (t, J = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.2, 153.7, 148.9, 125.6, 117.8, 116.0, 113.5, 61.33, 61.29, 55.7, 47.6, 28.3, 14.2. HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 19.5 min and 21.1 min (major). The HRMS Calculated for C₁₃H₁₉O₅ [M+H]⁺ 255.1227, found: 255.1237.

(+)-Ethyl 3-hydroxy-2-(2-hydroxy-4-methoxybenzyl)propanoate (2n):

46 mg, 91% yield, colorless oil, new compound, $R_f = 0.40$ (dichloromethane/methanol 40/1), 88%

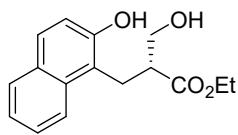


ee, $[\alpha]^{20}_D = +12.17$ (c 0.92, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.64 (brs, 1H), 6.98 (d, J = 8.2 Hz, 1H), 6.57-6.25 (m, 2H), 4.19 (q, J = 7.1 Hz, 2H), 3.88 (dd, J = 11.5, 3.8 Hz, 1H), 3.74 (s, 3H), 3.62 (dd, J = 11.5, 4.5 Hz, 1H), 3.33 (brs, 1H), 3.02-2.86 (m, 2H), 2.82-2.68 (m, 1H), 1.27 (t, J = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 175.3, 159.8, 155.9, 131.5, 116.7, 106.6, 102.5, 61.4, 61.2, 55.3, 47.8, 27.6, 14.2. HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 32.7 min (major) and 35.2 min. The HRMS Calculated for C₁₃H₁₉O₅

$[M+H]^+$ 255.1227, found: 255.1234.

(+)-Ethyl (*R*)-3-hydroxy-2-((2-hydroxynaphthalen-1-yl)methyl)propanoate (2o):

51 mg, 93% yield, white solid, new compound, mp 82-83 °C, R_f = 0.60 (dichloromethane/methanol 40/1), 87% ee, $[\alpha]^{20}_D$ = +10.11 (c 1.00, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.08 (brs, 1H), 7.90 (d, J = 8.5 Hz, 1H), 7.79 (d, J = 8.1 Hz, 1H), 7.69 (d, J = 8.8 Hz, 1H), 7.52 (t, J = 7.7 Hz, 1H), 7.42-7.30 (m, 1H), 7.21 (d, J = 8.8 Hz, 1H), 4.28 (q, J = 7.1 Hz, 2H), 4.09-3.95 (m, 1H), 3.72-3.27 (m, 4H), 3.00-2.93 (m, 1H), 1.34 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.8, 153.2, 133.2, 129.5, 128.92, 128.85, 126.7, 123.2, 122.2, 119.6, 115.6, 61.5, 61.4, 45.9, 23.6, 14.2. HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 13.1 min (minor) and 15.6 min (major). HRMS Calculated for $\text{C}_{16}\text{H}_{19}\text{O}_4$ $[M+H]^+$ 275.1278, found: 275.1285.

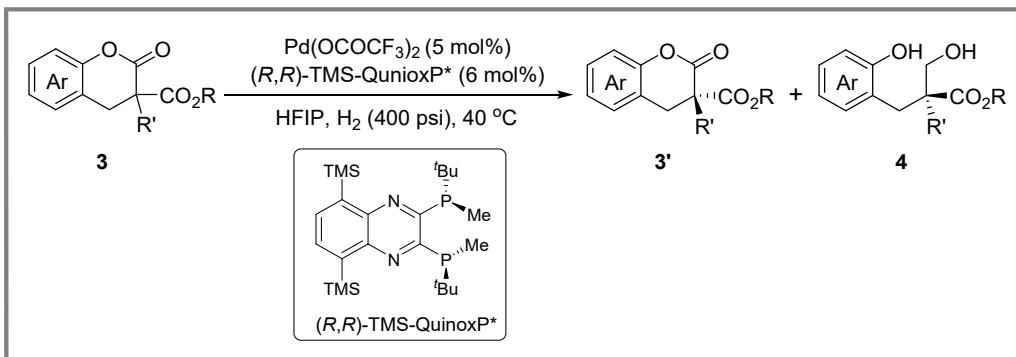


nol 40/1), 87% ee, $[\alpha]^{20}_D$ = +10.11 (c 1.00, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.08 (brs, 1H), 7.90 (d, J = 8.5 Hz, 1H), 7.79 (d, J = 8.1 Hz, 1H), 7.69 (d, J = 8.8 Hz, 1H), 7.52 (t, J = 7.7 Hz, 1H), 7.42-7.30 (m, 1H), 7.21 (d, J = 8.8 Hz, 1H), 4.28 (q, J = 7.1 Hz, 2H), 4.09-3.95 (m, 1H), 3.72-3.27 (m, 4H), 3.00-2.93 (m, 1H), 1.34 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.8, 153.2, 133.2, 129.5, 128.92, 128.85, 126.7, 123.2, 122.2, 119.6, 115.6, 61.5, 61.4, 45.9, 23.6, 14.2. HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 13.1 min (minor) and 15.6 min (major). HRMS Calculated for $\text{C}_{16}\text{H}_{19}\text{O}_4$ $[M+H]^+$ 275.1278, found: 275.1285.

(+)-Ethyl 3-hydroxy-2-((3-hydroxynaphthalen-2-yl)methyl)propanoate (2p):

38 mg, 70% yield, colorless oil, new compound, R_f = 0.40 (dichloromethane/methanol 40/1), 69% ee, $[\alpha]^{20}_D$ = +12.55 (c 0.76, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.82-7.52 (m, 3H), 7.41-7.35 (m, 1H), 7.33-7.28 (m, 1H), 7.23 (s, 1H), 4.21 (q, J = 7.1 Hz, 2H), 3.93 (dd, J = 11.5, 3.6 Hz, 1H), 3.66 (dd, J = 11.5, 4.7 Hz, 1H), 3.24-3.14 (m, 2H), 2.97-2.90 (m, 1H), 1.28 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.2, 153.2, 134.0, 130.1, 129.0, 127.3, 127.2, 126.1, 126.0, 123.7, 111.3, 61.6, 61.3, 47.9, 28.7, 14.2. HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 14.9 min (major) and 17.8 min. The HRMS Calculated for $\text{C}_{16}\text{H}_{19}\text{O}_4$ $[M+H]^+$ 275.1278, found: 275.1279.

4.2 Pd-catalyzed Hydrogenative Kinetic Resolution of Lactones with Quaternary Centers



General Procedure: The $\text{Pd}(\text{OCOCF}_3)_2$ (0.015 mmol, 4.8 mg, 5 mol%), ligand (R,R) -TMS-QuinoxP* (0.018 mmol, 8.6 mg, 6 mol%) and degassed anhydrous acetone (1.0 mL) were placed in a dried Schlenk tube under nitrogen atmosphere. The mixture was stirred under room temperature for 1 hour, the solvent was removed under vacuum to give the catalyst. Then, this catalyst was taken into a glove box filled with nitrogen gas and dissolved in 1,1,1,3,3,3-hexafluoro-2-propanol (HFIP, 1.0 mL). To the lactones **3** (0.3 mmol) added this catalyst solution, and then the mixture was transferred to an autoclave, which was charged hydrogen gas (400 psi). The reaction mixture was stirred at 40 °C for 55-120 hours. After cooling to room temperature and the release of the hydrogen gas, the autoclave was opened. To the mixture added sodium cyanoborohydride (7.5 mg, 0.12 mmol). The mixture was stirred under room temperature for 2 hours, then the volatiles were removed under the reduced pressure. The crude residue was purified by column chromatography on silica gel using hexanes/ethyl acetate (10:1-5:1) as eluent to give the chiral reductive products **3'** and **4**.

The racemates were prepared by reduction of lactones **3** with sodium borohydride in methanol.

(+)-Ethyl 2-benzyl-3-hydroxy-2-(2-hydroxybenzyl)propanoate (**4a**):

React for 55 h. 48 mg, 52% yield, colorless oil, new compound, $R_f = 0.40$ (hexanes/ethyl acetate 5/1), 70.8% ee, $[\alpha]^{20}_D = +4.90$ (c 0.96, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.26-7.02 (m, 7H), 7.01-6.80 (m, 2H), 4.27-4.06 (m, 2H), 3.71 (d, $J = 11.7$ Hz, 1H), 3.39 (d, $J = 11.7$ Hz, 1H), 3.26-3.17 (m, 2H), 3.01 (d, $J = 14.1$ Hz, 1H), 2.86 (d, $J = 13.5$ Hz, 1H), 1.27 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.2, 155.6, 136.6, 132.5, 129.8, 128.7, 128.4, 126.9, 122.5, 120.5, 117.2, 61.7, 61.1, 53.6, 40.7, 34.7, 14.1. HPLC: Chiral-pak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 9.6 min and 10.3 min (major). HRMS Calculated for $\text{C}_{19}\text{H}_{22}\text{O}_4\text{Na}$ [$\text{M}+\text{H}$]⁺ 337.1410, found: 337.1416.

(+)-Ethyl (*S*)-3-benzyl-2-oxochromane-3-carboxylate (**3a'**):

Kinetic resolution from hydrogenation of **3a**. 44 mg, 47% yield, 93.0% ee, $[\alpha]^{20}_D = +34.18$ (c 0.88, CHCl_3). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 9.5 min (major) and 10.7 min.

(+)-Ethyl 3-hydroxy-2-(2-hydroxybenzyl)-2-(4-methylbenzyl)propanoate (**4b**):

React for 55 h. 52 mg, 54% yield, colorless oil, new compound, $R_f = 0.37$ (hexanes/ethyl acetate

5/1), 61.6% ee, $[\alpha]^{20}_D = +7.31$ (c 1.04, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.20 (brs, 1H), 7.19-7.11 (m, 2H), 7.05 (d, $J = 7.8$ Hz, 2H), 6.97 (d, $J = 7.9$ Hz, 2H), 6.93-6.82 (m, 2H), 4.25-4.09 (m, 2H), 3.69 (d, $J = 11.3$ Hz, 1H), 3.40 (d, $J = 11.7$ Hz, 2H), 3.25-3.14 (m, 2H), 3.02 (d, $J = 14.1$ Hz, 1H), 2.85 (d, $J = 13.5$ Hz, 1H), 2.30 (s, 3H), 1.28 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.4, 155.7, 136.4, 133.4, 132.5, 129.6, 129.1, 128.7, 122.5, 120.4, 117.3, 61.9, 61.1, 53.5, 40.2, 34.7, 21.1, 14.2. HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 9.1 min and 10.3 min (major). HRMS Calculated for $\text{C}_{20}\text{H}_{25}\text{O}_4$ [$\text{M}+\text{H}]^+$ 329.1747, found: 329.1755.

(+)-Ethyl 3-(4-methylbenzyl)-2-oxochromane-3-carboxylate (3b):

Kinetic resolution from hydrogenation of **3b**. 43 mg, 44% yield, 84.1% ee, $[\alpha]^{20}_D = +35.11$ (c 0.86, CHCl_3). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 8.0 min (major) and 9.1 min.

(+)-Ethyl 2-(4-fluorobenzyl)-3-hydroxy-2-(2-hydroxybenzyl)propanoate (4c):

React for 72 h. 61 mg, 61% yield, colorless oil, new compound, $R_f = 0.30$ (hexanes/ethyl acetate 5/1), 60.1% ee, $[\alpha]^{20}_D = +5.66$ (c 1.22, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.41 (brs, 1H), 7.19-7.05 (m, 4H), 6.97-6.77 (m, 4H), 4.27-3.82 (m, 3H), 3.63 (d, $J = 11.8$ Hz, 1H), 3.44 (d, $J = 11.7$ Hz, 1H), 3.21-2.91 (m, 4H), 1.23 (t, $J = 7.2$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.8, 161.8 (d, ${}^1J_{\text{F-C}} = 245.0$ Hz), 155.2, 132.4 (d, ${}^4J_{\text{F-C}} = 3.0$ Hz), 132.3, 131.3 (d, ${}^3J_{\text{F-C}} = 7.8$ Hz), 128.7, 122.8, 120.7, 116.7, 115.2 (d, ${}^2J_{\text{F-C}} = 21.1$ Hz), 61.0, 60.9, 53.8, 39.9, 34.6, 14.1. ^{19}F NMR (376 MHz, CDCl_3) δ -115.88. HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 10.2 min and 10.7 min (major). HRMS Calculated for $\text{C}_{19}\text{H}_{22}\text{FO}_4$ [$\text{M}+\text{H}]^+$ 333.1497, found: 333.1498.

(+)-Ethyl 3-(4-fluorobenzyl)-2-oxochromane-3-carboxylate (3c'):

Kinetic resolution from hydrogenation of **3c**. 37 mg, 38% yield, 98.3% ee, $[\alpha]^{20}_D = +33.17$ (c 0.74, CHCl_3). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 9.3 min (major) and 11.2 min.

(+)-Ethyl 3-hydroxy-2-(2-hydroxybenzyl)-2-(naphthalen-2-ylmethyl)propanoate (4d):

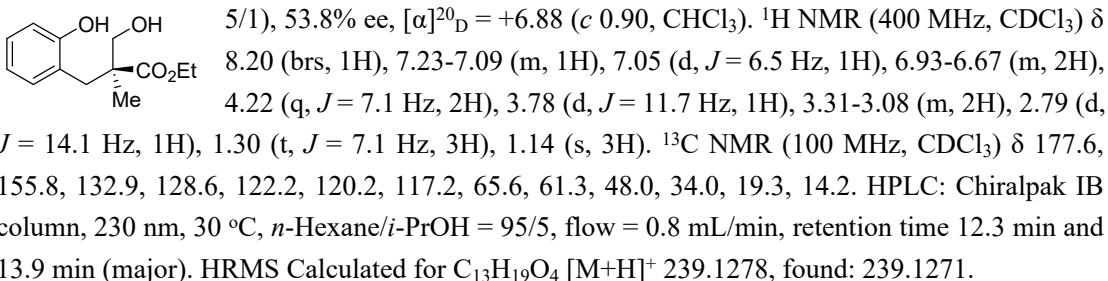
React for 120 h. 49 mg, 45% yield, colorless oil, new compound, $R_f = 0.30$ (hexanes/ethyl acetate 5/1), 67.1% ee, $[\alpha]^{20}_D = +3.74$ (c 0.98, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.33 (brs, 1H), 7.79-7.66 (m, 3H), 7.56 (s, 1H), 7.46-7.37 (m, 2H), 7.22 (d, $J = 7.7$ Hz, 1H), 7.17-7.08 (m, 2H), 6.95-6.80 (m, 2H), 4.14 (q, $J = 7.1$ Hz, 2H), 3.68 (d, $J = 11.7$ Hz, 2H), 3.48 (d, $J = 11.7$ Hz, 1H), 3.34 (d, $J = 13.4$ Hz, 1H), 3.24 (d, $J = 14.1$ Hz, 1H), 3.16-3.02 (m, 2H), 1.21 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.2, 155.5, 134.3, 133.4, 132.5, 132.4, 128.7, 128.6, 128.1, 127.9, 127.62, 127.59, 126.1, 125.7, 122.7, 120.6, 117.0, 61.5, 61.1, 53.9, 40.8, 34.8, 14.2. HPLC: Chiralpak IA column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 18.7 min (minor) and 26.5 min (major). HRMS Calculated for $\text{C}_{23}\text{H}_{25}\text{O}_4$ [$\text{M}+\text{H}]^+$ 365.1747, found: 365.1750.

(+)-Ethyl 3-(naphthalen-2-ylmethyl)-2-oxochromane-3-carboxylate (3d'):

Kinetic resolution from hydrogenation of **3d**. 60 mg, 55% yield, 59.7% ee, $[\alpha]^{20}_D = +14.32$ (*c* 1.20, CHCl₃). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 11.4 min (major) and 13.5 min.

(+)-Ethyl 3-hydroxy-2-(2-hydroxybenzyl)-2-methylpropanoate (4e):

React for 72 h. 45 mg, 64% yield, colorless oil, new compound, R_f = 0.34 (hexanes/ethyl acetate

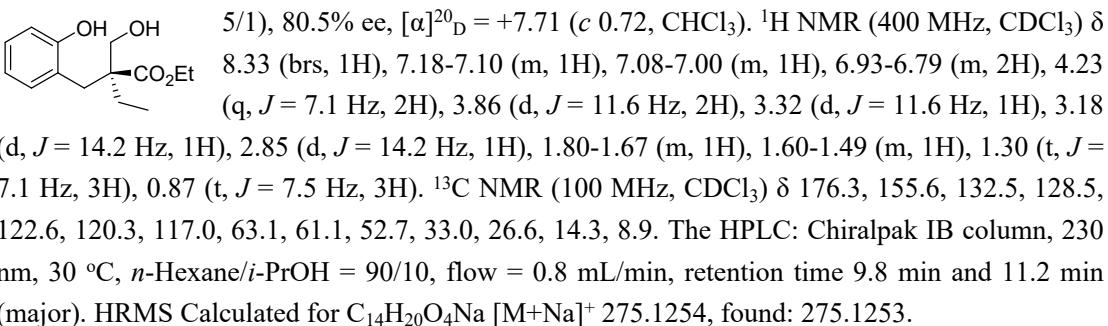


(+)-Ethyl 3-methyl-2-oxochromane-3-carboxylate (3e'):

Kinetic resolution from hydrogenation of **3e**. 25 mg, 35% yield, 98.4% ee, $[\alpha]^{20}_D = +11.34$ (*c* 0.50, CHCl₃). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 9.8 min (major) and 11.0 min.

(+)-Ethyl 2-(2-hydroxy-5-methylbenzyl)-2-(hydroxymethyl)butanoate (4f):

React for 60 h. 36 mg, 49% yield, colorless oil, new compound, R_f = 0.38 (hexanes/ethyl acetate

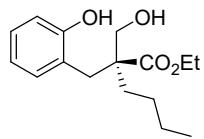


(+)-Ethyl 3-ethyl-6-methyl-2-oxochromane-3-carboxylate (3f'):

Kinetic resolution from hydrogenation of substrate lactone **3f**. 35 mg, 47% yield, 88.6% ee, $[\alpha]^{20}_D = +14.91$ (*c* 0.70, CHCl₃). The HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 7.9 min (major) and 9.7 min (minor).

(+)-Ethyl 2-(2-hydroxybenzyl)-2-(hydroxymethyl)pentanoate (4g):

React for 72 h. 47 mg, 57% yield, colorless oil, new compound, R_f = 0.40 (hexanes/ethyl acetate 5/1), 75.0% ee, $[\alpha]^{20}_D = +10.32$ (*c* 0.94, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.30 (brs, 1H), 7.19-7.09 (m, 1H), 7.08-6.99 (m, 1H), 6.94-6.74 (m, 2H), 4.31-4.15 (m, 2H), 3.82 (t, *J* = 29.3 Hz, 2H), 3.30 (d, *J* = 11.7 Hz, 1H), 3.18 (d, *J* = 14.2 Hz, 1H), 2.84 (d, *J* = 14.2 Hz, 1H), 1.74-1.64 (m, 1H),



1.53-1.43 (m, 1H), 1.33-1.18 (m, 7H), 0.89 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.5, 155.7, 132.6, 128.5, 122.6, 120.2, 117.1, 63.5, 61.0, 52.2, 33.5, 33.3, 26.7, 23.2, 14.3, 13.9. HPLC: Chi- ralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 8.9 min and 9.4 min (major). HRMS Calculated for $\text{C}_{16}\text{H}_{24}\text{O}_4\text{Na} [\text{M}+\text{Na}]^+$ 303.1567, found: 303.1572.

(+)-Ethyl 2-oxo-3-propylchromane-3-carboxylate (3g'):

Kinetic resolution from hydrogenation of **3g**. 36 mg, 43% yield, 99.0% ee, $[\alpha]^{20}_D$ = +15.97 (c 0.72, CHCl_3). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 7.1 min (major) and 8.6 min.

(+)-Ethyl 2-(2-hydroxybenzyl)-2-(hydroxymethyl)-4-methoxybutanoate (4h):

React for 66 h. 39 mg, 46% yield, colorless oil, new compound, R_f = 0.20 (hexanes/ethyl acetate 5/1), 84.4% ee, $[\alpha]^{20}_D$ = +20.99 (c 0.78, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.16-7.09 (m, 1H), 7.05-6.97 (m, 1H), 6.89 (d, J = 7.9 Hz, 1H), 6.84-6.74 (m, 1H), 4.16 (q, J = 7.1 Hz, 2H), 3.77 (d, J = 12.1 Hz, 1H), 3.59-3.37 (m, 3H), 3.33 (s, 3H), 2.99 (dd, J = 35.6, 14.1 Hz, 2H), 2.19-2.07 (m, 1H), 1.94-1.85 (m, 1H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.6, 155.9, 132.2, 128.6, 122.5, 120.2, 117.3, 69.0, 62.8, 61.1, 58.8, 51.4, 34.6, 33.9, 14.1. HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 14.1 min and 17.5 min (major). HRMS Calculated for $\text{C}_{15}\text{H}_{22}\text{O}_5\text{Na} [\text{M}+\text{Na}]^+$ 303.1359, found: 303.1364.

(+)-Ethyl 3-(2-methoxyethyl)-2-oxochromane-3-carboxylate (3h'):

Kinetic resolution from hydrogenation of **3h**. 44 mg, 53% yield, 66.4% ee, $[\alpha]^{20}_D$ = +17.33 (c 0.88, CHCl_3). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 9.2 min (major) and 10.1 min.

(+)-Ethyl 3-cyclopropyl-2-(2-hydroxybenzyl)-2-(hydroxymethyl)propanoate (4i):

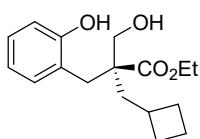
React for 72 h. 56 mg, 68% yield, colorless oil, new compound, R_f = 0.30 (hexanes/ethyl acetate 5/1), 39.8% ee, $[\alpha]^{20}_D$ = +9.10 (c 1.12, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.30 (brs, 1H), 7.20-7.11 (m, 1H), 7.07-6.95 (m, 1H), 6.95-6.74 (m, 2H), 4.33-4.16 (m, 2H), 4.09 (d, J = 11.7 Hz, 1H), 3.39 (d, J = 11.7 Hz, 2H), 3.18 (d, J = 14.2 Hz, 1H), 2.83 (d, J = 14.2 Hz, 1H), 1.90-1.80 (m, 1H), 1.33 (t, J = 7.1 Hz, 3H), 0.65-0.51 (m, 1H), 0.49-0.36 (m, 2H), 0.08-0.03 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.7, 155.9, 132.6, 128.6, 122.3, 120.2, 117.4, 63.4, 61.1, 52.8, 39.0, 34.0, 14.2, 6.5, 5.3, 4.1. HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 9.9 min and 11.4 min (major). HRMS Calculated for $\text{C}_{16}\text{H}_{23}\text{O}_4 [\text{M}+\text{H}]^+$ 279.1591, found: 279.1597.

(+)-Ethyl 3-(cyclopropylmethyl)-2-oxochromane-3-carboxylate (3i'):

Kinetic resolution from hydrogenation of **3i**. 26 mg, 32% yield, 98.8% ee, $[\alpha]^{20}_D$ = +32.66 (c 0.52, CHCl_3). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 7.6 min (major) and 9.6 min.

(+)-Ethyl 3-cyclobutyl-2-(2-hydroxybenzyl)-2-(hydroxymethyl)propanoate (4j):

React for 55 h under 50 °C. 58 mg, 67% yield, colorless oil, new compound, $R_f = 0.25$ (hexanes/



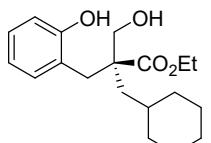
ethyl acetate 5/1), 48.0% ee, $[\alpha]^{20}_D = +2.33$ (c 1.16, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.44 (brs, 1H), 7.17-7.10 (m, 1H), 7.08-6.98 (m, 1H), 6.91-6.73 (m, 2H), 4.24-4.12 (m, 2H), 3.82 (d, $J = 11.7$ Hz, 2H), 3.29 (d, $J = 11.7$ Hz, 1H), 3.13 (d, $J = 14.1$ Hz, 1H), 2.83 (d, $J = 14.2$ Hz, 1H), 2.37-2.26 (m, 1H), 2.02-1.92 (m, 2H), 1.89-1.76 (m, 2H), 1.74-1.54 (m, 4H), 1.30 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 176.4, 155.7, 132.6, 128.5, 122.6, 120.3, 117.0, 63.1, 61.0, 51.9, 41.2, 33.7, 32.5, 30.1, 29.2, 18.8, 14.2. HPLC: Chiraldak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 9.1 min and 9.9 min (major). HRMS Calculated for $\text{C}_{17}\text{H}_{24}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 315.1567, found: 315.1567.

(+)-Ethyl 3-(cyclobutylmethyl)-2-oxochromane-3-carboxylate (3j'):

Kinetic resolution from hydrogenation of **3j**. 28 mg, 33% yield, 95.6% ee, $[\alpha]^{20}_D = +24.69$ (c 0.56, CHCl_3). HPLC: Chiraldak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 7.6 min (major) and 8.9 min.

(+)-Ethyl 3-cyclohexyl-2-(2-hydroxybenzyl)-2-(hydroxymethyl)propanoate (4k):

React for 72 h. 62 mg, 64% yield, colorless oil, new compound, $R_f = 0.30$ (hexanes/ethyl acetate



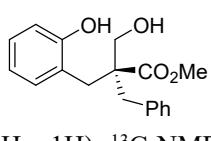
5/1), 55.0% ee, $[\alpha]^{20}_D = +6.51$ (c 1.04, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.27 (brs, 1H), 7.20-7.10 (m, 1H), 7.07-7.00 (m, 1H), 6.98-6.87 (m, 1H), 6.86-6.76 (m, 1H), 4.22 (q, $J = 7.1$ Hz, 2H), 3.96 (d, $J = 11.7$ Hz, 1H), 3.44 (brs, 1H), 3.26 (d, $J = 11.9$ Hz, 1H), 3.16 (d, $J = 14.1$ Hz, 1H), 2.75 (d, $J = 14.1$ Hz, 1H), 1.79-1.70 (m, 1H), 1.67-1.59 (m, 4H), 1.52 (d, $J = 13.2$ Hz, 1H), 1.47-1.40 (m, 1H), 1.32 (t, $J = 7.1$ Hz, 3H), 1.25-1.08 (m, 4H), 0.98-0.85 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3) δ 177.2, 155.8, 132.7, 128.5, 122.5, 120.2, 117.2, 63.2, 61.0, 51.4, 41.3, 34.9, 34.7, 34.1, 33.7, 26.4, 26.3, 26.1, 14.1. HPLC: Chiraldak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 6.2 min (major) and 7.2 min. HRMS Calculated for $\text{C}_{19}\text{H}_{28}\text{O}_4\text{Na}$ $[\text{M}+\text{Na}]^+$ 339.1567, found: 339.1534.

(+)-Ethyl 3-(cyclohexylmethyl)-2-oxochromane-3-carboxylate (3k'):

Kinetic resolution from hydrogenation of **3k**. 34 mg, 36% yield, 97.0% ee, $[\alpha]^{20}_D = +14.87$ (c 0.68, CHCl_3). HPLC: Chiraldak OD-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 5.6 min (major) and 6.2 min.

(+)-Methyl 2-benzyl-3-hydroxy-2-(2-hydroxybenzyl)propanoate (4l):

React for 50 h. 46 mg, 51% yield, colorless oil, new compound, $R_f = 0.35$ (hexanes/ethyl acetate



5/1), 77.4% ee, $[\alpha]^{20}_D = +13.75$ (c 0.64, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.08 (brs, 1H), 7.27-7.04 (m, 7H), 6.93-6.81 (m, 2H), 3.69 (s, 3H), 3.68-3.28 (m, 3H), 3.27-3.14 (m, 2H), 3.04 (d, $J = 14.1$ Hz, 1H), 2.90 (d, $J = 13.5$ Hz, 1H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.6, 155.6, 136.6, 132.4, 129.7, 128.7, 128.5, 126.9, 122.4, 120.5, 117.2, 61.6, 53.8, 51.8, 40.7, 34.7. HPLC: Chiraldak IB column, 230 nm, 30 °C, *n*-

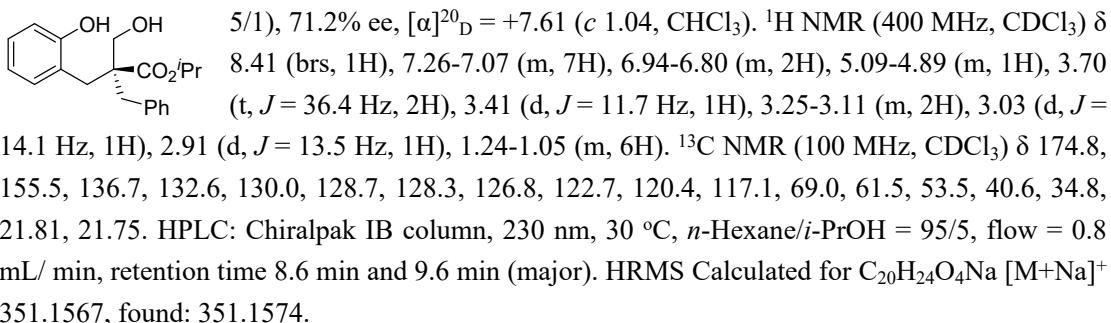
Hexane/*i*-PrOH = 97/3, flow = 1.0 mL/ min, retention time 13.8 min and 15.0 min (major). HRMS Calculated for C₁₈H₂₀O₄Na [M+Na]⁺ 323.1254, found: 323.1268.

(+)-Methyl 3-benzyl-2-oxochromane-3-carboxylate (3l'):

Kinetic resolution from hydrogenation of **3l**. 45 mg, 48% yield, 72.0% ee, $[\alpha]^{20}_D = +32.00$ (*c* 0.60, CHCl₃). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 12.0 min and 23.5 min (major).

(+)-Isopropyl 2-benzyl-3-hydroxy-2-(2-hydroxybenzyl)propanoate (4m):

React for 72 h. 52 mg, 53% yield, colorless oil, new compound, R_f = 0.35 (hexanes/ethyl acetate

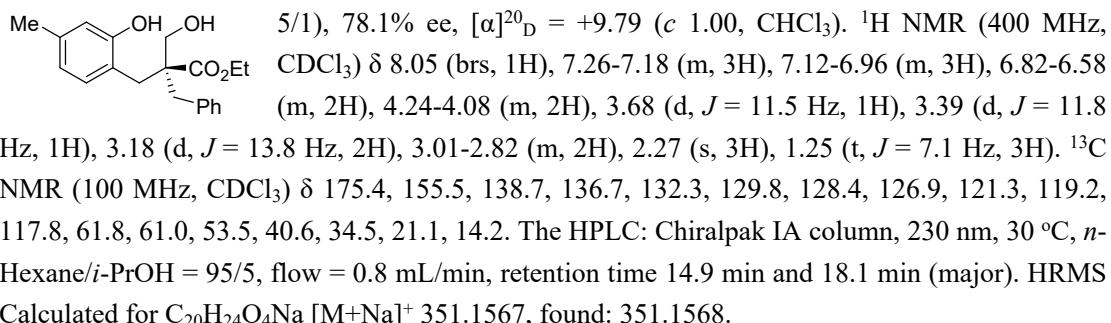


(+)-Isopropyl 3-benzyl-2-oxochromane-3-carboxylate (3m'):

Kinetic resolution from hydrogenation of **3l**. 45 mg, 46% yield, 80.8% ee, $[\alpha]^{20}_D = +7.62$ (*c* 0.90, CHCl₃). HPLC: Chiralpak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 7.6 min (major) and 8.1 min.

(+)-Ethyl 2-benzyl-3-hydroxy-2-(2-hydroxy-4-methylbenzyl)propanoate (4n):

React for 56 h. 50 mg, 50% yield, colorless oil, new compound, R_f = 0.40 (hexanes/ethyl acetate



(+)-Ethyl 3-benzyl-7-methyl-2-oxochromane-3-carboxylate (3n'):

Kinetic resolution from hydrogenation of **3m**. 49 mg, 50% yield, 88.1% ee, $[\alpha]^{20}_D = +29.00$ (*c* 0.98, CHCl₃). HPLC: Chiralpak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 97/3, flow = 0.8 mL/min, retention time 9.0 min (major) and 10.6 min.

(+)-Ethyl 2-benzyl-2-(4-chloro-2-hydroxybenzyl)-3-hydroxypropanoate (4o):

React for 72 h. 51 mg, 50% yield, colorless oil, new compound, R_f = 0.40 (hexanes/ethyl acetate 5/1), 59.3% ee, $[\alpha]^{20}_D = +10.33$ (*c* 1.02, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 8.52 (brs, 1H),

7.26-7.18 (m, 3H), 7.13-7.00 (m, 3H), 6.91 (d, J = 2.1 Hz, 1H), 6.86-6.77 (m, 1H), 4.18-4.09 (m, 2H), 3.64 (d, J = 11.7 Hz, 1H), 3.41 (d, J = 11.7 Hz, 1H), 3.16 (d, J = 13.5 Hz, 2H), 2.98 (d, J = 14.2 Hz, 1H), 2.87 (d, J = 13.5 Hz, 1H), 1.23 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.0, 156.5, 136.4, 133.7, 133.2, 129.8, 128.4, 127.0, 121.2, 120.6, 117.5, 61.6, 61.2, 53.5, 40.7, 34.2, 14.1. HPLC: Chiraldak IB column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 14.2 min (major) and 15.5 min. HRMS Calculated for $\text{C}_{19}\text{H}_{21}\text{ClO}_4\text{Na}$ [M+Na]⁺ 371.1021 (³⁵Cl), found: 371.1027 (³⁵Cl). [M+H]⁺ 373.1004 (³⁷Cl), found: 373.1003 (³⁷Cl).

(+)-Ethyl 3-benzyl-7-chloro-2-oxochromane-3-carboxylate (3o'):

Kinetic resolution from hydrogenation of **3n**. 51 mg, 49% yield, 82.1% ee, $[\alpha]^{20}_D$ = +32.32 (*c* 1.02, CHCl_3). HPLC: Chiraldak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 97/3, flow = 0.8 mL/min, retention time 9.3 min (major) and 10.0 min.

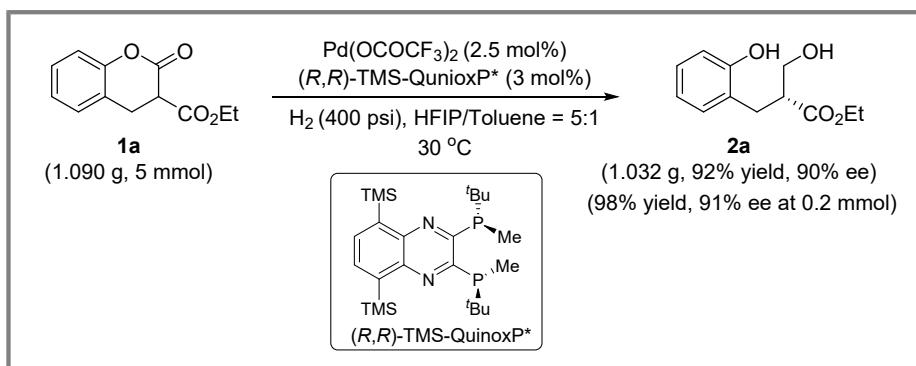
(+)-Ethyl 2-benzyl-3-hydroxy-2-(2-hydroxy-4-methoxybenzyl)propanoate (4p):

React for 72 h. 67 mg, 65% yield, colorless oil, new compound, R_f = 0.25 (hexanes/ethyl acetate 5/1), 49.5% ee, $[\alpha]^{20}_D$ = +14.74 (*c* 1.34, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 8.47 (brs, 1H), 7.24-7.15 (m, 3H), 7.12-7.05 (m, 2H), 7.00 (d, J = 8.4 Hz, 1H), 6.53-6.36 (m, 2H), 4.18-4.06 (m, 2H), 3.72 (d, J = 2.2 Hz, 3H), 3.65 (d, J = 11.6 Hz, 1H), 3.42 (d, J = 11.6 Hz, 1H), 3.20-3.09 (m, 2H), 3.01-2.84 (m, 2H), 1.22 (t, J = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 175.3, 160.0, 156.5, 136.8, 132.9, 129.8, 128.4, 126.8, 114.8, 106.7, 102.4, 61.5, 61.0, 55.3, 53.7, 40.7, 34.1, 14.1. HPLC: Chiraldak IC column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 80/20, flow = 0.8 mL/min, retention time 9.0 min (major) and 11.3 min. HRMS Calculated for $\text{C}_{20}\text{H}_{25}\text{O}_5$ [M+H]⁺ 345.1697, found: 345.1697.

(+)-Ethyl 3-benzyl-7-methoxy-2-oxochromane-3-carboxylate (3p'):

Kinetic resolution from hydrogenation of **3o**. 36 mg, 35% yield, 97.9% ee, $[\alpha]^{20}_D$ = +42.97 (*c* 0.72, CHCl_3). HPLC: Chiraldak OD-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 9.6 min (major) and 10.8 min.

5. Experiment at Gram Scale

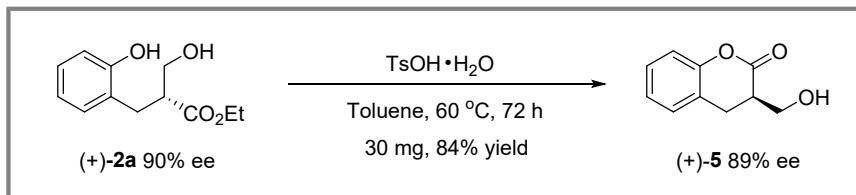


The metal precursor $\text{Pd}(\text{OCOCF}_3)_2$ (0.125 mmol, 40.3 mg, 2.5 mol%), chiral ligand $(\text{R},\text{R})\text{-TMS-QuinoxP}^*$ (0.15 mmol, 71.9 mg, 3 mol%) and degassed anhydrous acetone (10 mL) were placed in a dried Schlenk tube under nitrogen atmosphere. The mixture was stirred under room temperature for 2 hour. The solvent was removed under vacuum to give the chiral catalyst. This catalyst was taken into a glove box filled with nitrogen and dissolved in solvent (HFIP/toluene = 5:1, 6.0 mL). To the mixture of lactone **1a** (5 mmol, 1.090 g) and solvent (HFIP/toluene = 5:1, 6.0 mL) was added the above catalyst solution, and the mixture was transferred to an autoclave, which was charged hydrogen gas (400 psi). The reaction mixture was stirred at 30 °C for 120 hours. After the release of hydrogen gas, the autoclave was opened and the volatiles were removed under the reduced pressure. Then, the residue was purified by column chromatography on silica gel using dicholormethane/methanol (40:1) as eluent to give the pure reductive product **2a** (1.032 g, 92% yield, 90% ee).

The enantiomeric excess of the reductive product **2a** was determined by the chiral HPLC with the Chiralpak AS-H column.

6. Product Elaboration

6.1 Transesterification

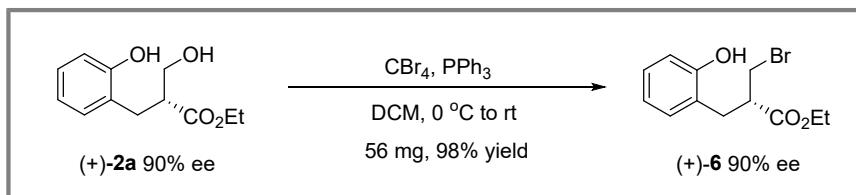


To a Schlenk tube, reductive product ethyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (+)-2a (90% ee, 44.8 mg, 0.2 mmol), toluene (4.0 mL) and *p*-toluenesulfonic acid monohydrate (3.8 mg, 0.02 mmol) were added under nitrogen gas. Then the mixture was stirred for 72 hours under 60 °C. After completion of reaction (monitored by TLC), water (10 mL) and ethyl acetate (5 mL) were added, and the water layer was extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated under the reduced pressure. The crude residue was purified by flash column chromatography on silica gel using dichloromethane/methanol (40:1) as eluent to give the chiral transesterification product (-)-5 without any loss of optical purity.

(-)-3-(Hydroxymethyl)chroman-2-one (5)

30 mg, 84% yield, viscous oil, new compound, $R_f = 0.60$ (dichloromethane/methanol 40/1), 89% ee, $[\alpha]^{20}_D = -14.09$ (*c* 0.44, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.28-7.20 (m, 2H), 7.17-7.02 (m, 2H), 4.17-3.84 (m, 2H), 3.04 (t, *J* = 14.3 Hz, 1H), 2.97-2.79 (m, 2H), 2.63 (t, *J* = 6.5 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃) δ 171.0, 151.2, 128.4, 128.2, 124.7, 122.5, 116.8, 61.8, 41.6, 26.3. HPLC: Chiraldak AS-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 90/10, flow = 0.8 mL/min, retention time 31.1 min (major) and 32.8. HRMS Calculated for C₁₀H₁₁O₃ [M+H]⁺ 179.0703, found: 179.0697.

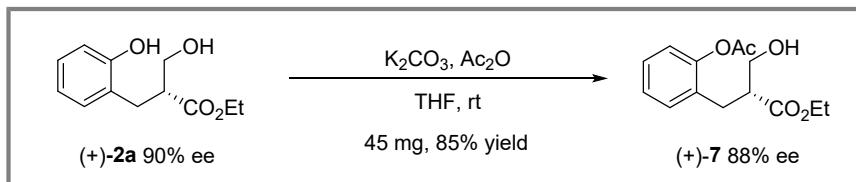
6.2 Appel Reaction



To a Schlenk tube, reductive product ethyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (+)-2a (90% ee, 44.8 mg, 0.2 mmol), dichloromethane (2.0 mL) and carbon tetrabromide (73.0 mg, 0.22 mmol) were added under nitrogen gas. Then triphenylphosphine (78.7 mg, 0.3 mmol) in dichloromethane (2.0 mL) was added dropwise at 0 °C. The mixture was allowed to reach room temperature and stirred for 72 hours. After completion of reaction (monitored by TLC), water (10 mL) and ethyl acetate (5 mL) were added, and the water layer was extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated under the reduced pressure. The crude residue was purified by flash column chromatography on silica gel using hexanes/ethyl acetate (10/1) as eluent to give the chiral brominated product (-)-6 without any loss of optical purity.

(-)Ethyl 3-bromo-2-(2-hydroxybenzyl)propanoate (6)

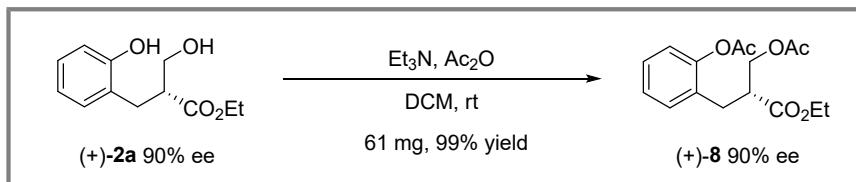
56 mg, 98% yield, viscous oil, new compound, $R_f = 0.40$ (hexanes/ethyl acetate 10/1), 90% ee, $[\alpha]^{20}_D = -1.52$ (c 1.12, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.17-7.07 (m, 2H), 6.93-6.71 (m, 2H), 6.21 (brs, 1H), 4.27-4.12 (m, 2H), 3.60 (d, $J = 5.7$ Hz, 2H), 3.29-3.18 (m, 1H), 3.13 (dd, $J = 13.8, 7.9$ Hz, 1H), 2.88 (dd, $J = 13.8, 6.0$ Hz, 1H), 1.23 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 173.7, 154.1, 131.3, 128.4, 124.4, 120.9, 116.4, 61.6, 48.1, 33.0, 31.2, 14.1. The HPLC: Chiralcel OD-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 75/25, flow = 0.8 mL/min, retention time 9.9 min and 12.2 (major). HRMS Calculated for $\text{C}_{12}\text{H}_{16}\text{BrO}_3$ [M+H]⁺ 287.0283 (⁷⁹Br), found: 287.0278 (⁷⁹Br). [M+H]⁺ 289.0263 (⁸¹Br), found: 289.0257 (⁸¹Br).

6.3 Single Acyl Protection

To a Schlenk tube, reductive product ethyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (+)-2a (90% ee, 44.8 mg, 0.2 mmol), tetrahydrofuran (3.0 mL) and potassium carbonate (56.0 mg, 0.4 mmol) were added under nitrogen gas. Then acetic anhydride (23 μ L, 0.25 mmol) was slowly added and stirred for 9 hours. After completion of reaction (monitored by TLC), water (10 mL) and ethyl acetate (5 mL) were added, and the water layer was extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated under the reduced pressure. The crude residue was purified by flash column chromatography on silica gel using dicholormethane/methanol (40:1) as eluent to give the single protected product (+)-7 without any loss of optical purity.

(+)Ethyl 2-(2-acetoxybenzyl)-3-hydroxypropanoate (7)

45 mg, 85% yield, viscous oil, new compound, $R_f = 0.60$ (dicholormethane/methanol 40/1), 88% ee, $[\alpha]^{20}_D = +7.83$ (c 0.90, CHCl_3). ^1H NMR (400 MHz, CDCl_3) δ 7.27 (s, 2H), 7.22-7.13 (m, 1H), 7.05 (d, $J = 7.9$ Hz, 1H), 4.16 (q, $J = 7.0$ Hz, 2H), 3.85-3.71 (m, 1H), 3.69-3.61 (m, 1H), 3.02-2.89 (m, 1H), 2.89-2.71 (m, 2H), 2.34 (s, 4H), 1.23 (t, $J = 7.1$ Hz, 3H). ^{13}C NMR (100 MHz, CDCl_3) δ 174.6, 169.6, 149.1, 131.1, 130.7, 128.0, 126.2, 122.6, 61.9, 60.9, 47.6, 28.9, 21.0, 14.2. HPLC: Chiralcel OJ-H column, 230 nm, 30 °C, *n*-Hexane/*i*-PrOH = 70/30, flow = 0.8 mL/min, retention time 6.9 min (major) and 8.0. HRMS Calculated for $\text{C}_{14}\text{H}_{19}\text{O}_5$ [M+H]⁺ 267.1227, found: 267.1242.

6.4 Double Acyl Protection

To a Schlenk tube, reductive product ethyl 3-hydroxy-2-(2-hydroxybenzyl)propanoate (+)-2a

(90% ee, 44.8 mg, 0.2 mmol), dichloromethane (2.0 mL) and triethylamine (69 μ L, 0.5 mmol) were added under nitrogen gas. Then acetic anhydride (47 μ L, 0.5 mmol) in was slowly added and stirred overnight. After completion of reaction (monitored by TLC), water (10 mL) and ethyl acetate (5 mL) were added, and the water layer was extracted with ethyl acetate three times. The combined organic layers were dried over anhydrous sodium sulfate, filtered and concentrated under the reduced pressure. The crude residue was purified by flash column chromatography on silica gel using hexanes/ethyl acetate (5/1) as eluent to give the double acyl protected product (-)-**8** without any loss of optical purity.

(-)-Ethyl 3-acetoxy-2-(2-acetoxybenzyl)propanoate (8)

61 mg, 99% yield, viscous oil, new compound, R_f = 0.50 (hexanes/ethyl acetate 5/1), 90% ee, $[\alpha]^{20}_D$ = -2.75 (c 1.20, CHCl₃). ¹H NMR (400 MHz, CDCl₃) δ 7.29-7.03 (m, 4H), 4.28-4.16 (m, 2H), 4.16-4.04 (m, 2H), 3.04-2.87 (m, 2H), 2.81-2.68 (m, 1H), 2.34 (s, 3H), 2.02 (s, 3H), 1.17 (t, J = 7.1 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃) δ 172.7, 170.6, 169.3, 149.2, 130.9, 130.1, 128.1, 126.2, 122.6, 64.0, 60.9, 45.1, 29.7, 20.9, 20.8, 14.1. HPLC: Chiralcel OD-H column, 254 nm, 30 °C, *n*-Hexane/*i*-PrOH = 95/5, flow = 0.8 mL/min, retention time 13.3 min and 14.0 (major). The HRMS Calculated for C₁₆H₂₁O₆ [M+H]⁺ 309.1333, found: 309.1337.

7. Determination of Absolute Configuration

7.1 Absolute Configuration of Product (+)-2o

To determine the absolute configuration of (+)-ethyl 3-hydroxy-2-((2-hydroxynaphthalen-1-yl)methyl)propanoate (**2o**, >99% ee): (+)-**2o** was completely dissolved in ethyl acetate (1.0 mL), *n*-hexane (5.0 mL) was added slowly at room temperature. The solvent diffused slowly, and the single crystal was obtained after 14 days. The structure in **Figure S1** showed the absolute configuration is (*R*). The CCDC number is 2247354. These details can be obtained free of charge via www.ccdc.com.ac.uk/data_request/cif from the Cambridge Crystallographic Data Centre.

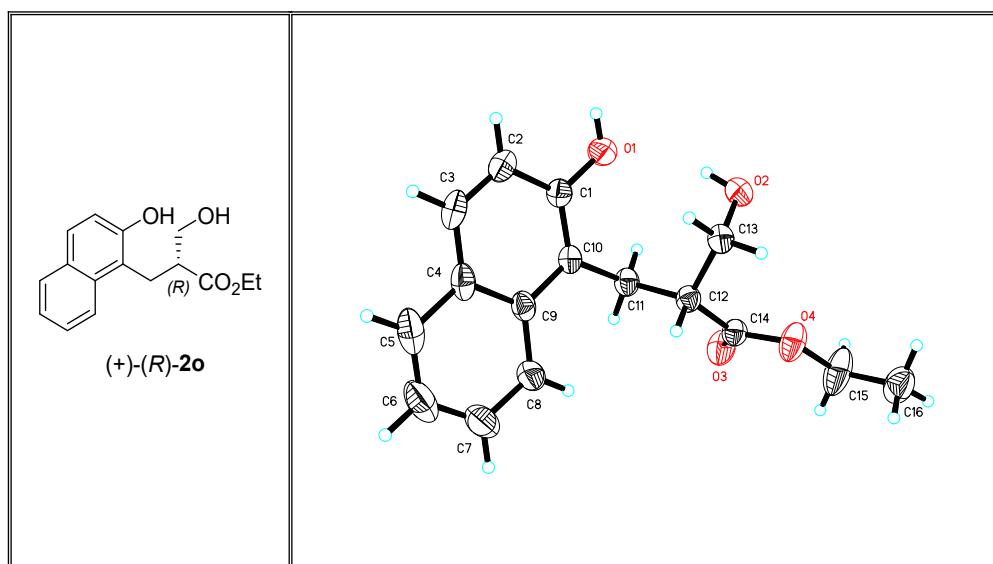
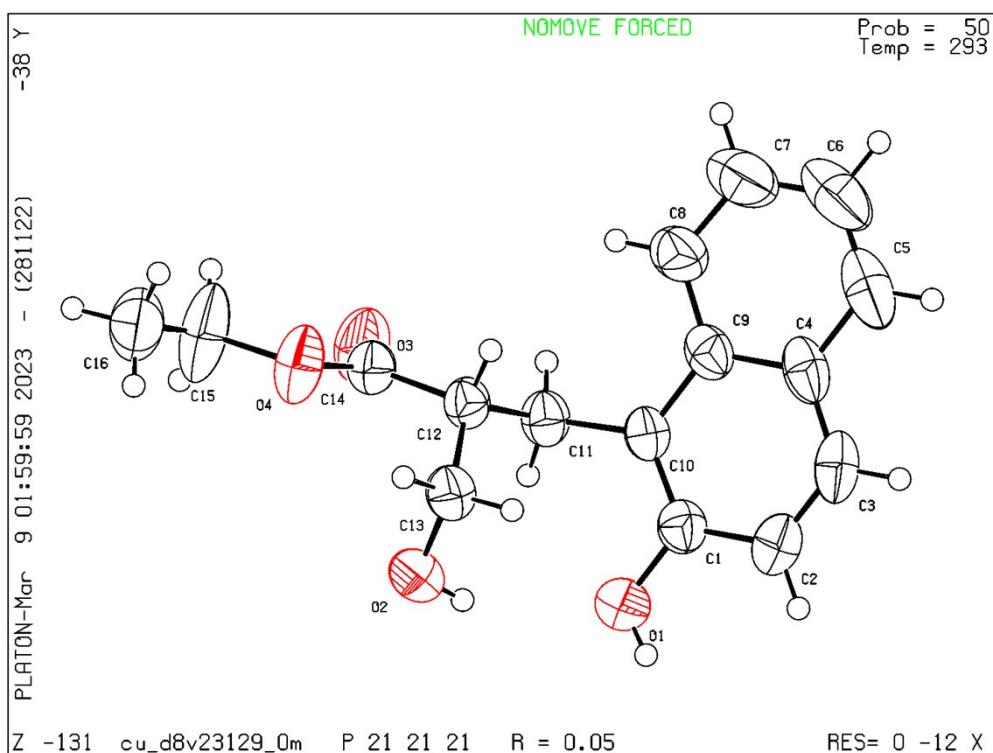


Figure S1. X-Ray Structure of Chiral Reductive Product (+)-(R)-**2o**



Crystal Data and Structure Refinement for cu_d8v21614_0m for (+)-(R)-2n

Identification code	cu_d8v23129_0m		
Empirical formula	C16 H18 O4		
Formula weight	274.30		
Temperature	293(2) K		
Wavelength	1.54178 Å		
Crystal system	Orthorhombic		
Space group	P 21 21 21		
Unit cell dimensions	$a = 7.0614(3)$ Å	$\alpha = 90^\circ$	
	$b = 10.6993(4)$ Å	$\beta = 90^\circ$	
	$c = 18.6749(8)$ Å	$\gamma = 90^\circ$	
Volume	1410.93(10) Å ³		
Z	4		
Density (calculated)	1.291 Mg/m ³		
Absorption coefficient	0.756 mm ⁻¹		
F(000)	584		
Crystal size	0.180 x 0.140 x 0.100 mm ³		
Theta range for data collection	4.763 to 67.464°.		
Index ranges	-8≤h≤8, -12≤k≤12, -22≤l≤20		
Reflections collected	9206		
Independent reflections	2510 [R(int) = 0.0598]		
Completeness to theta = 67.679°	98.1 %		
Absorption correction	Semi-empirical from equivalents		
Max. and min. transmission	0.7533 and 0.4628		
Refinement method	Full-matrix least-squares on F ²		
Data / restraints / parameters	2510 / 0 / 185		
Goodness-of-fit on F ²	1.073		
Final R indices [I>2sigma(I)]	R1 = 0.0464, wR2 = 0.1241		
R indices (all data)	R1 = 0.0519, wR2 = 0.1296		
Absolute structure parameter	0.05(15)		
Extinction coefficient	0.029(5)		
Largest diff. peak and hole	0.158 and -0.140 e.Å ⁻³		

7.2 Absolute Configuration of Product (+)-3a'

To determine the absolute configuration of (+)-ethyl 3-benzyl-2-oxochromane-3-carboxylate (**3a'**, >99% ee): (+)-**3a'** was completely dissolved in dichloromethane (0.1 mL), *n*-hexane (0.5 mL) was added slowly at room temperature. The solvent diffused slowly, and the single crystal was obtained after two days. The structure in **Figure S2** showed the absolute configuration is (*S*). The CCDC number is 2247355. These details can be obtained free of charge *via* www.ccdc.com.ac.uk/data_request/cif from the Cambridge Crystallographic Data Centre.

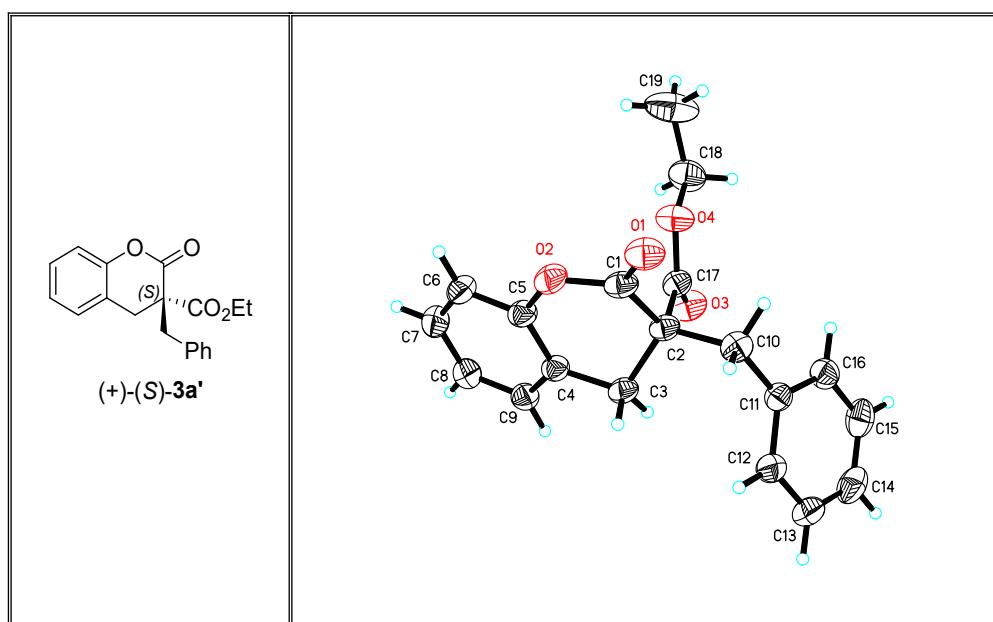
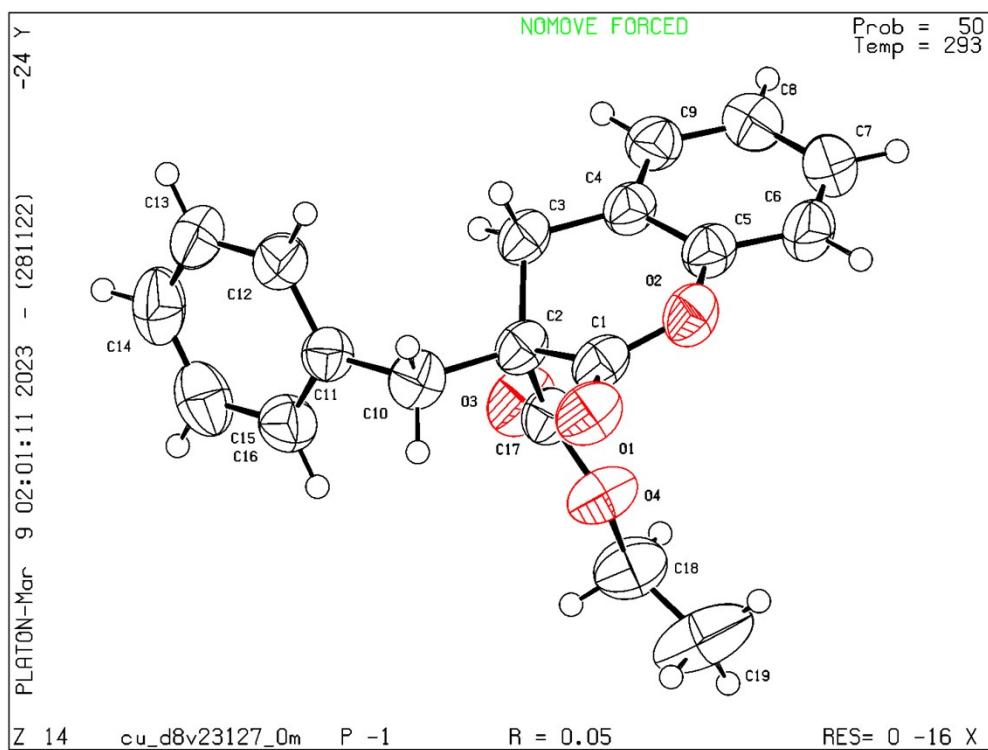


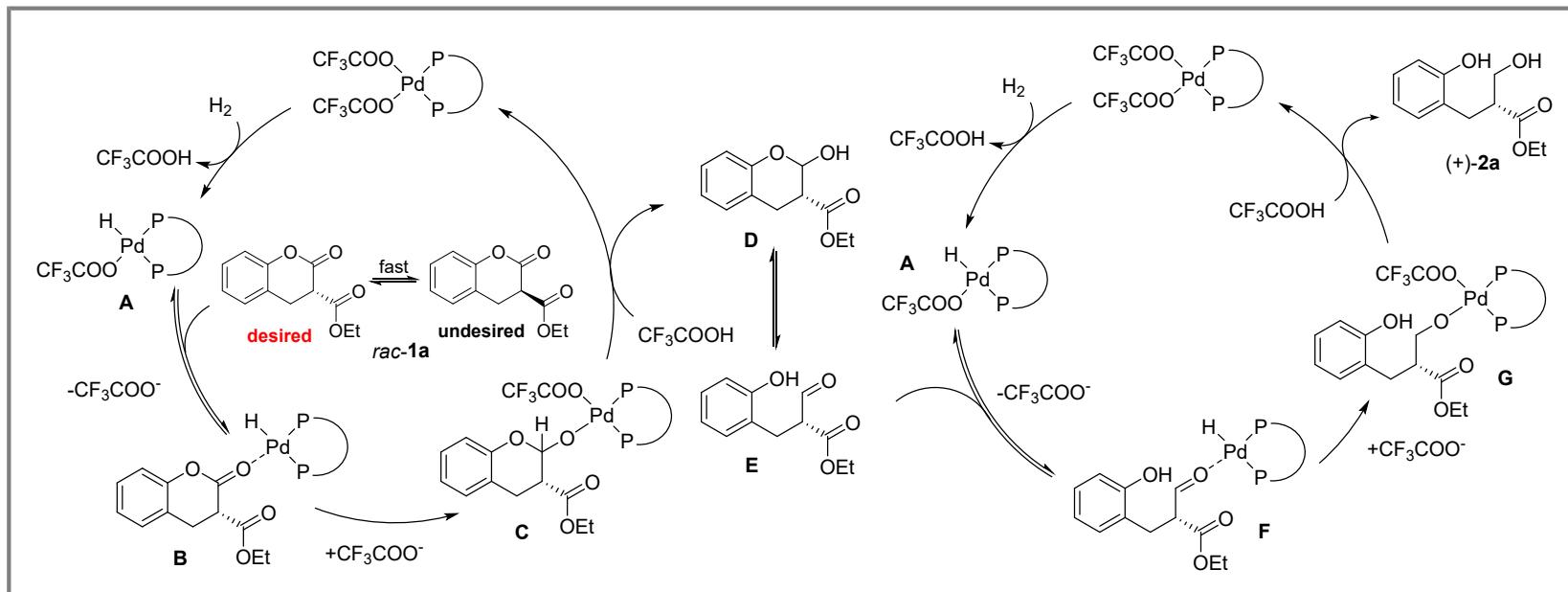
Figure S2.X-Ray Structure of Chiral Compound (+)-(S)-3a'



Crystal Data and Structure Refinement for cu_d8v21614_0m for (+)-(S)-3a'

Identification code	cu_d8v23127_0m	
Empirical formula	C19 H18 O4	
Formula weight	310.33	
Temperature	293(2) K	
Wavelength	1.54178 Å	
Crystal system	Triclinic	
Space group	P -1	
Unit cell dimensions	a = 8.2353(3) Å	α = 82.2410(10)°
	b = 9.8484(4) Å	β = 70.296(2)°
	c = 11.7427(5) Å	γ = 66.5120(10)°
Volume	822.33(6) Å³	
Z	2	
Density (calculated)	1.253 Mg/m³	
Absorption coefficient	0.714 mm⁻¹	
F(000)	328	
Crystal size	0.200 x 0.150 x 0.120 mm³	
Theta range for data collection	3.999 to 67.492°.	
Index ranges	-9<=h<=9, -11<=k<=11, -14<=l<=14	
Reflections collected	28232	
Independent reflections	2856 [R(int) = 0.0623]	
Completeness to theta = 67.679°	96.4 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7456 and 0.5300	
Refinement method	Full-matrix least-squares on F²	
Data / restraints / parameters	2856 / 0 / 210	
Goodness-of-fit on F²	1.046	
Final R indices [I>2sigma(I)]	R1 = 0.0489, wR2 = 0.1336	
R indices (all data)	R1 = 0.0505, wR2 = 0.1352	
Extinction coefficient	0.059(11)	
Largest diff. peak and hole	0.209 and -0.127 e.Å⁻³	

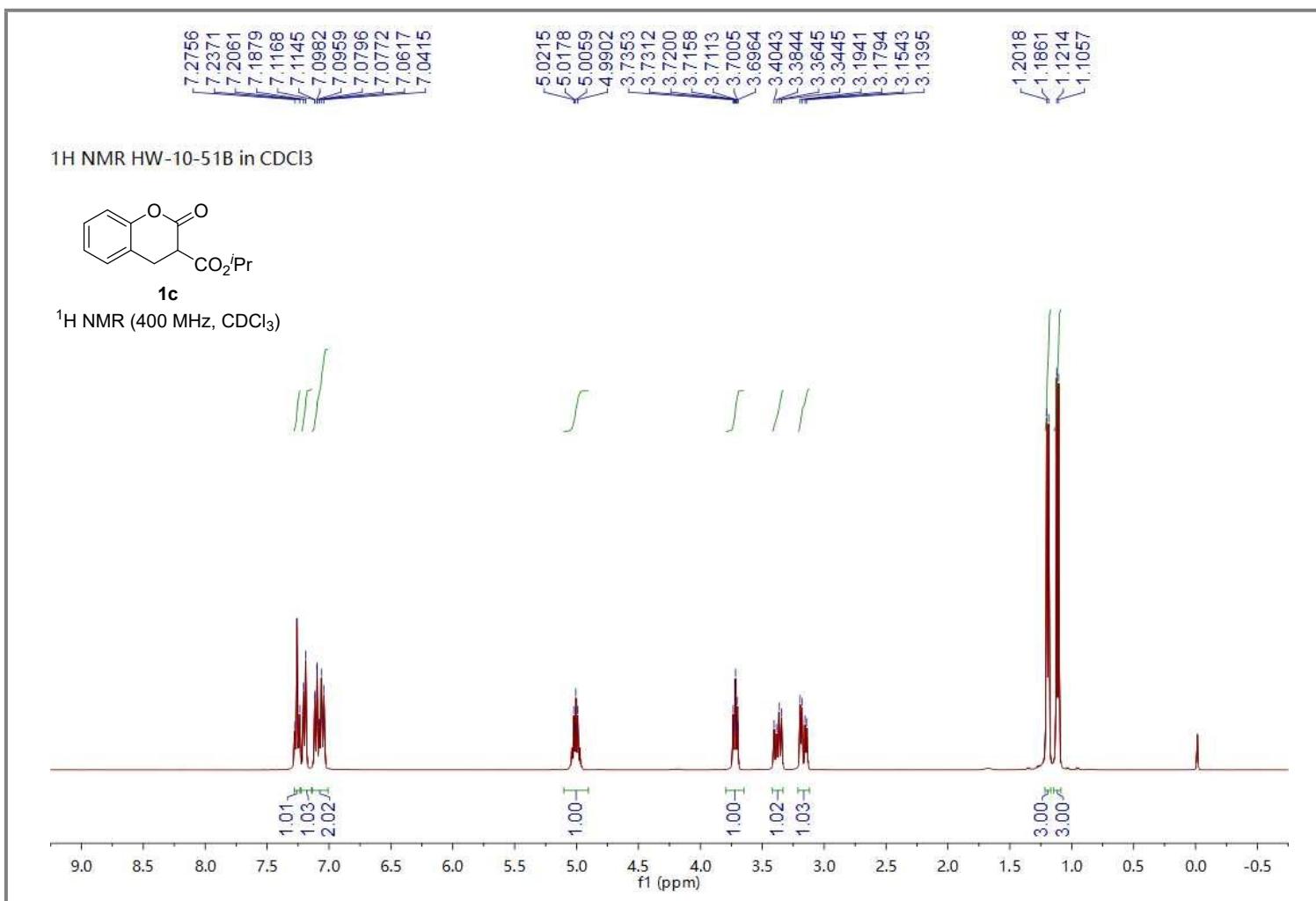
8. Proposed Mechanism of DKR



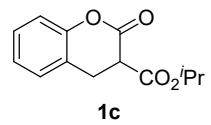
9. References

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5. Liu, Z.; Liu, Q.; Zhang, W.; Mu, R.; Yang, L.; Liu, Z.-L.; Yu, W. Selective Reduction of the Endocyclic Double Bond of 3-Substituted Coumarins by Hantzsch 1,4-Dihydropyridine. *Synthesis* **2006**, *5*, 771-774.

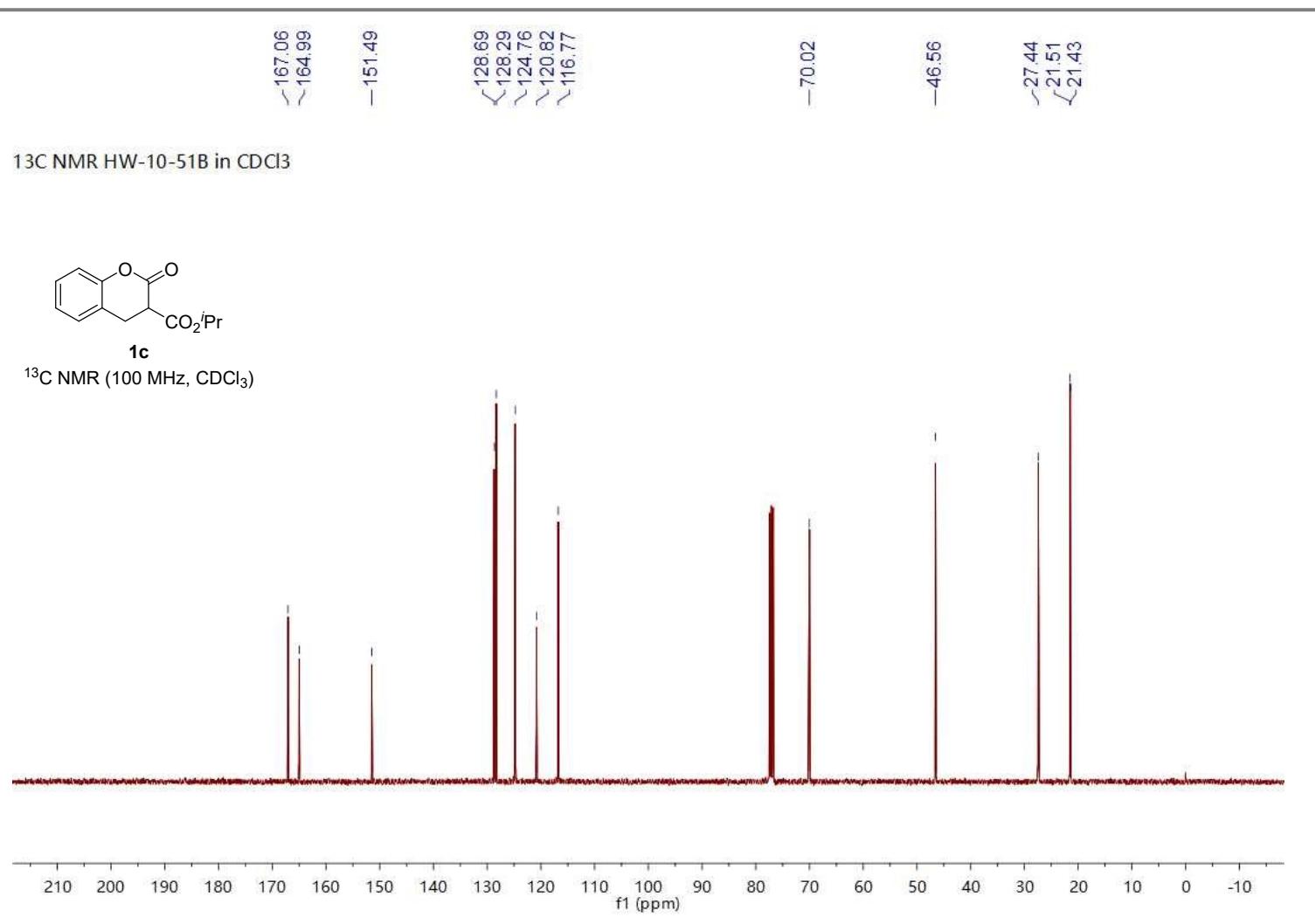
10. Copy of NMR and HPLC Spectra

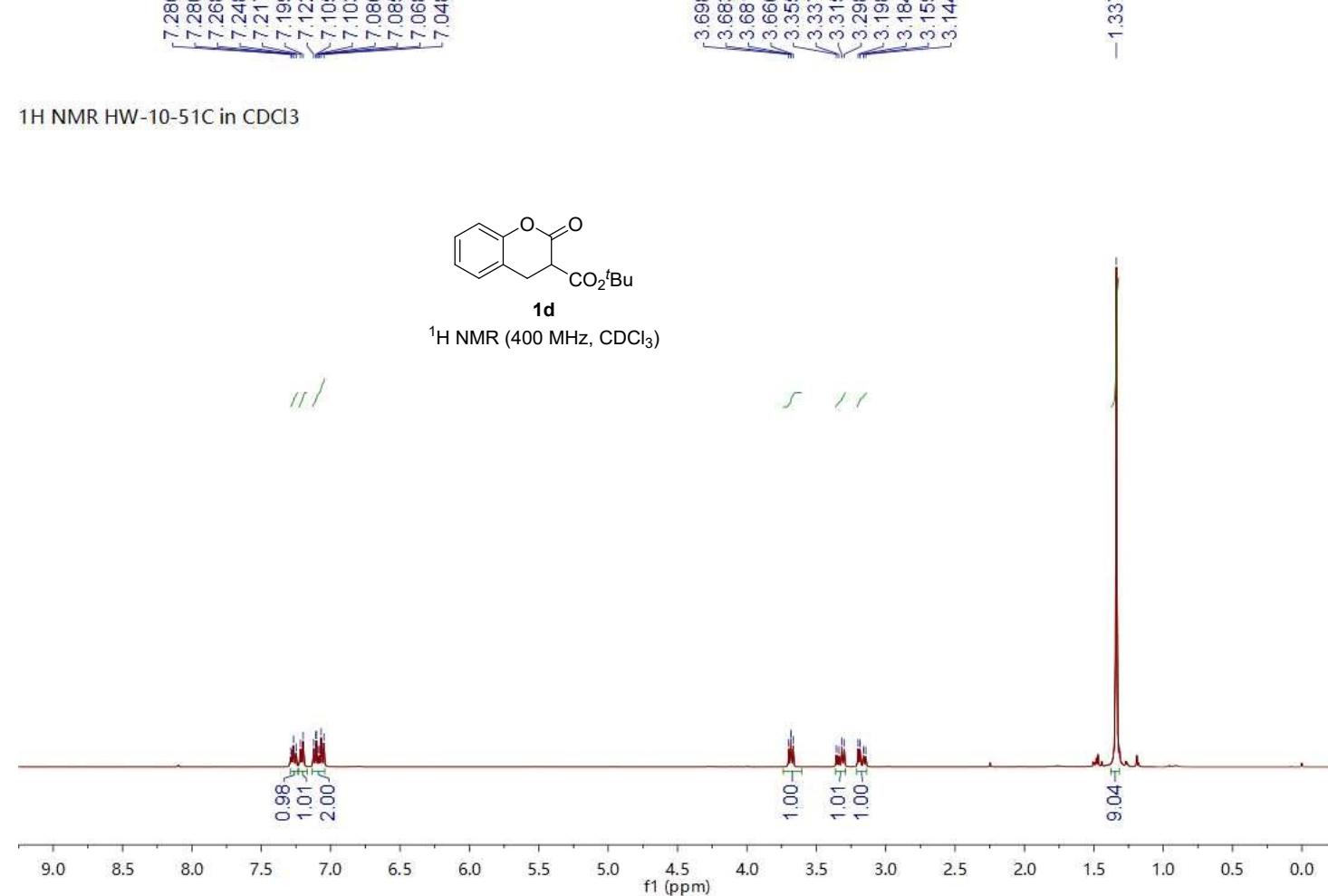


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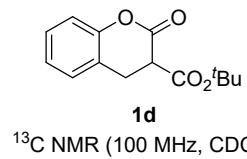


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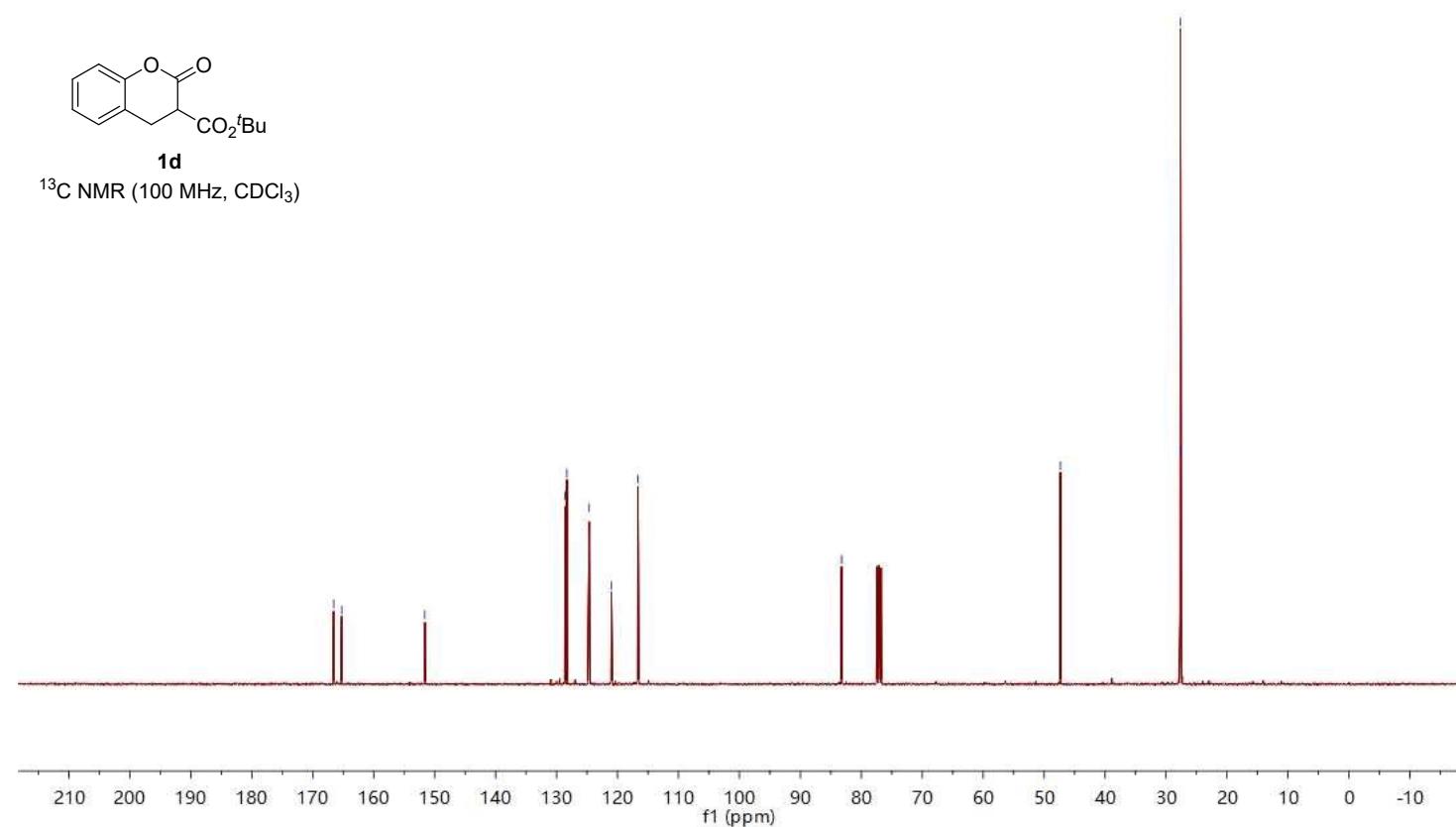


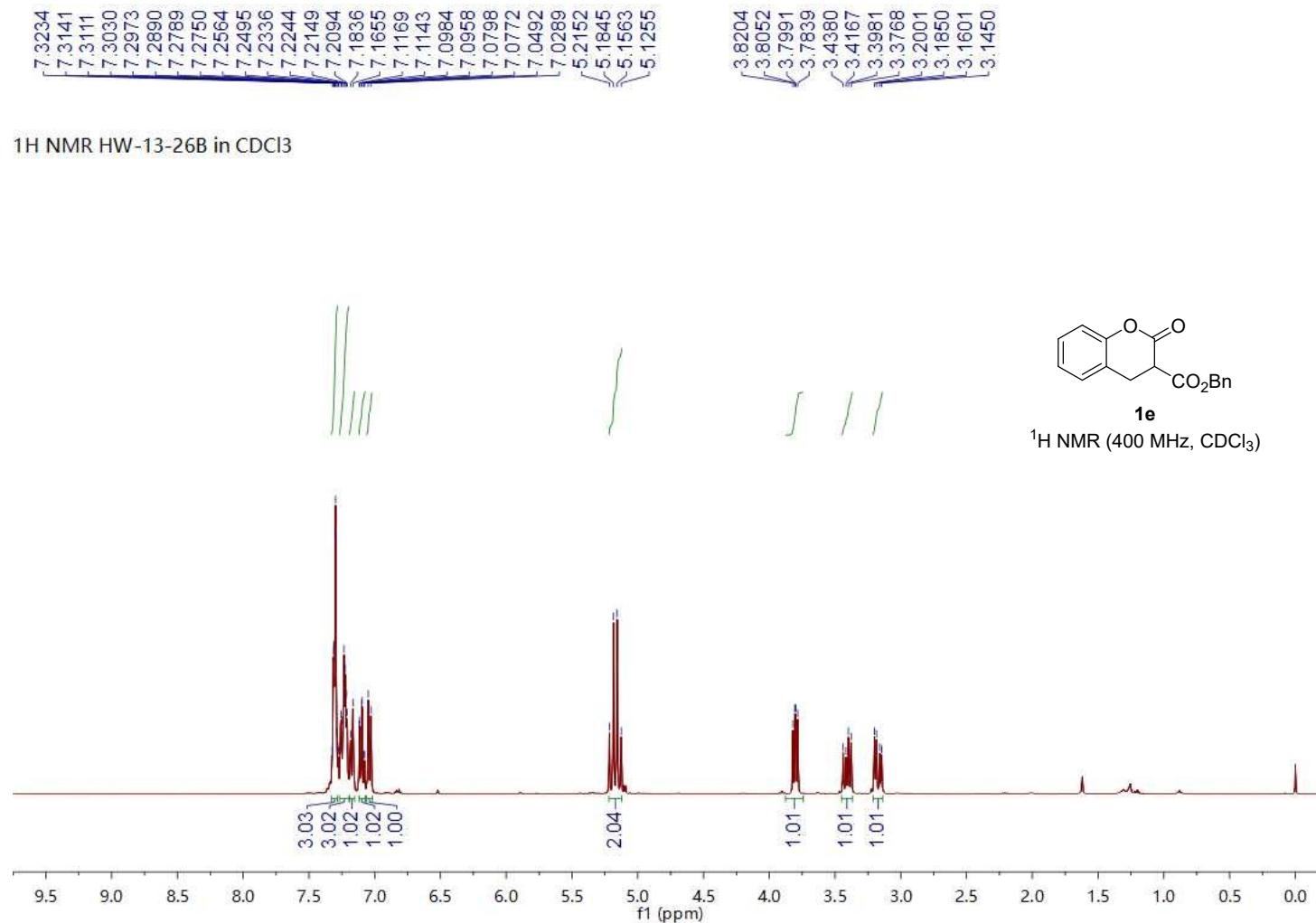


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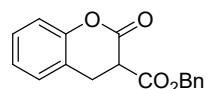


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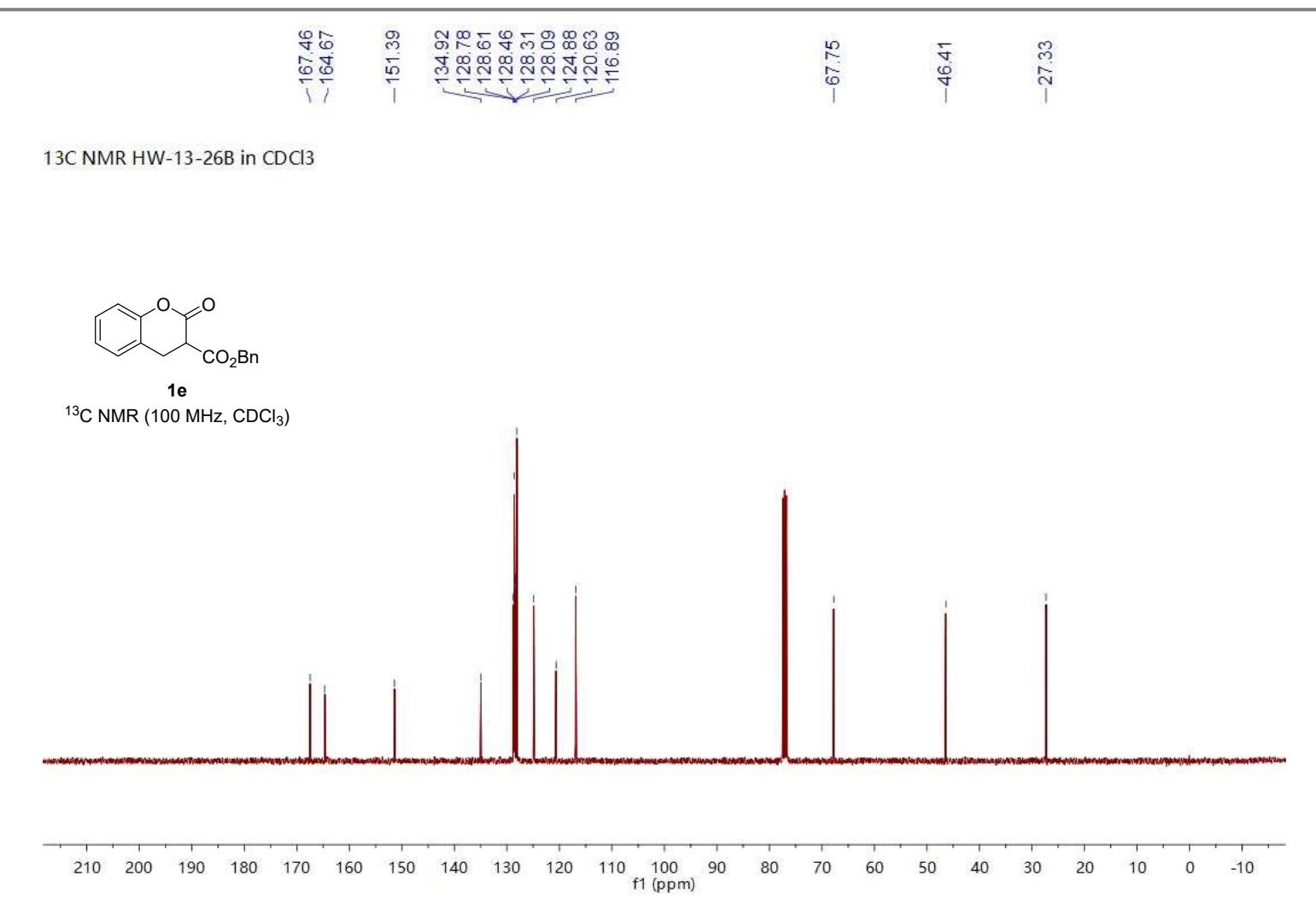


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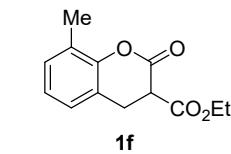


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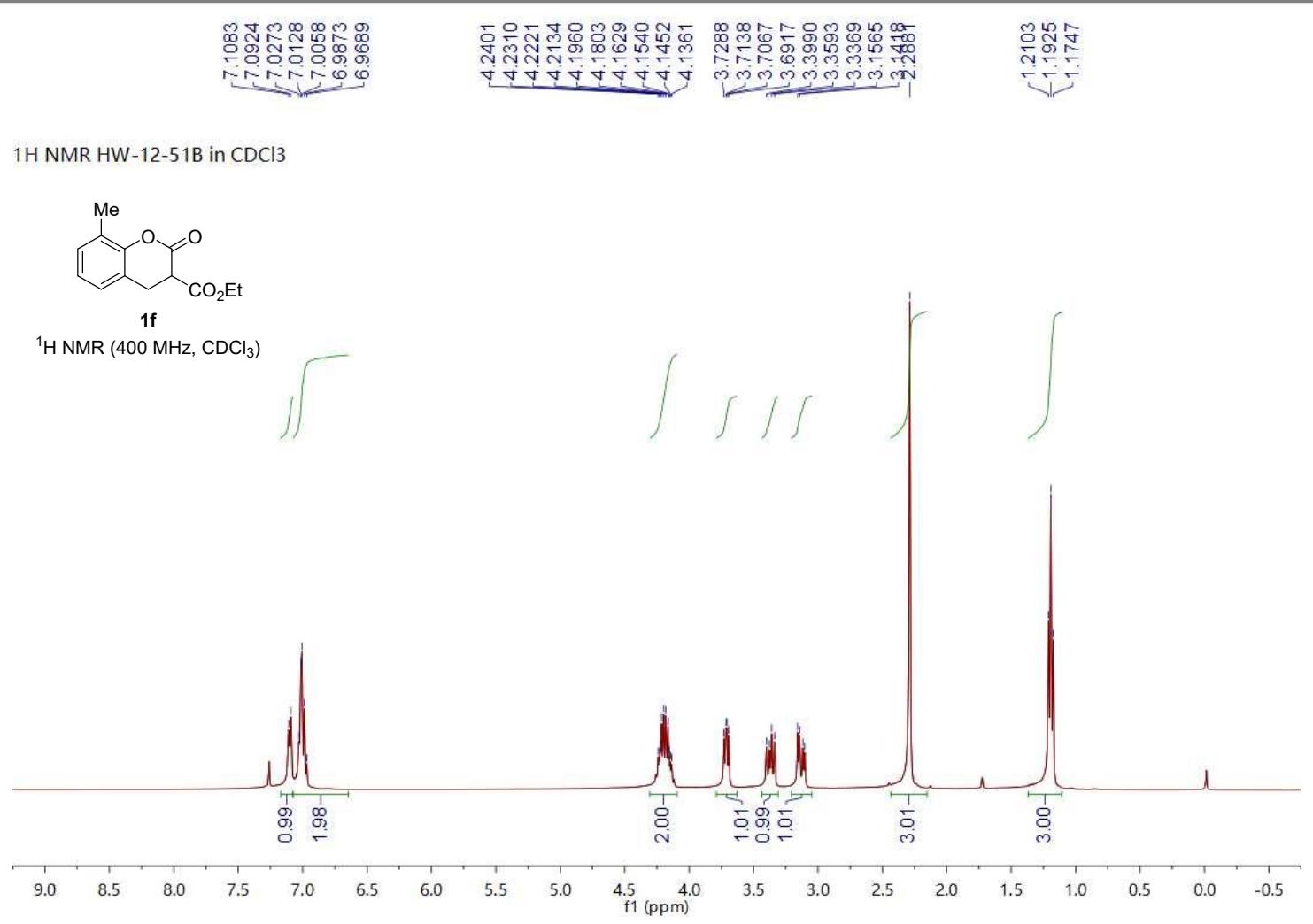
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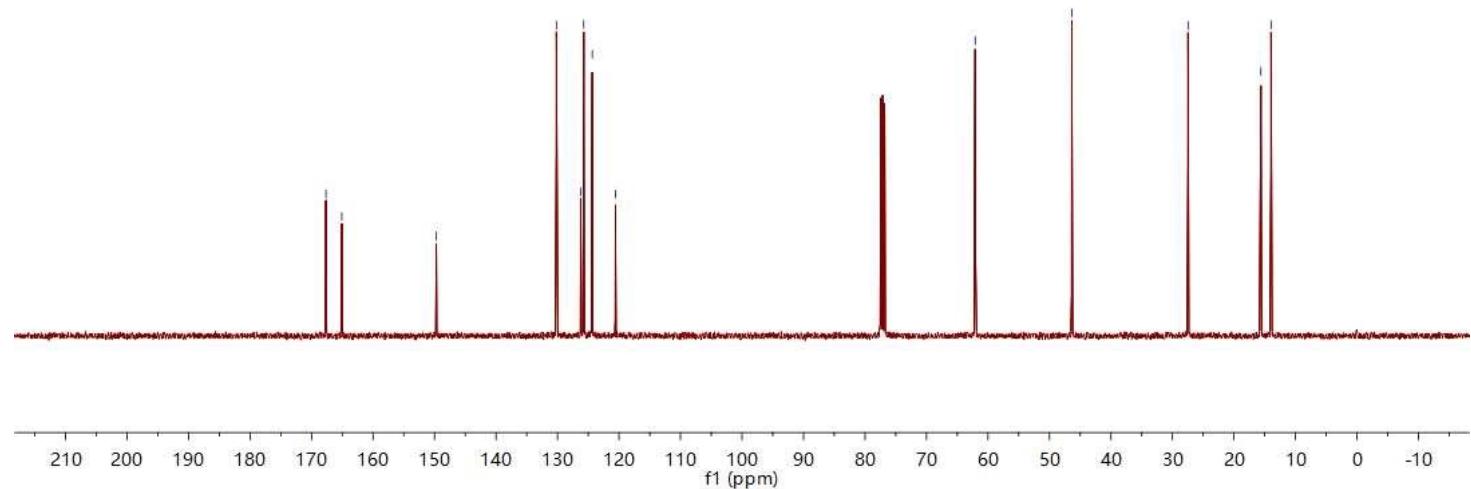
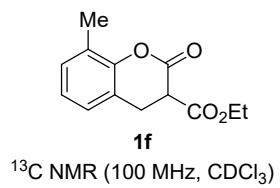
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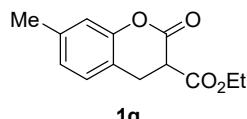
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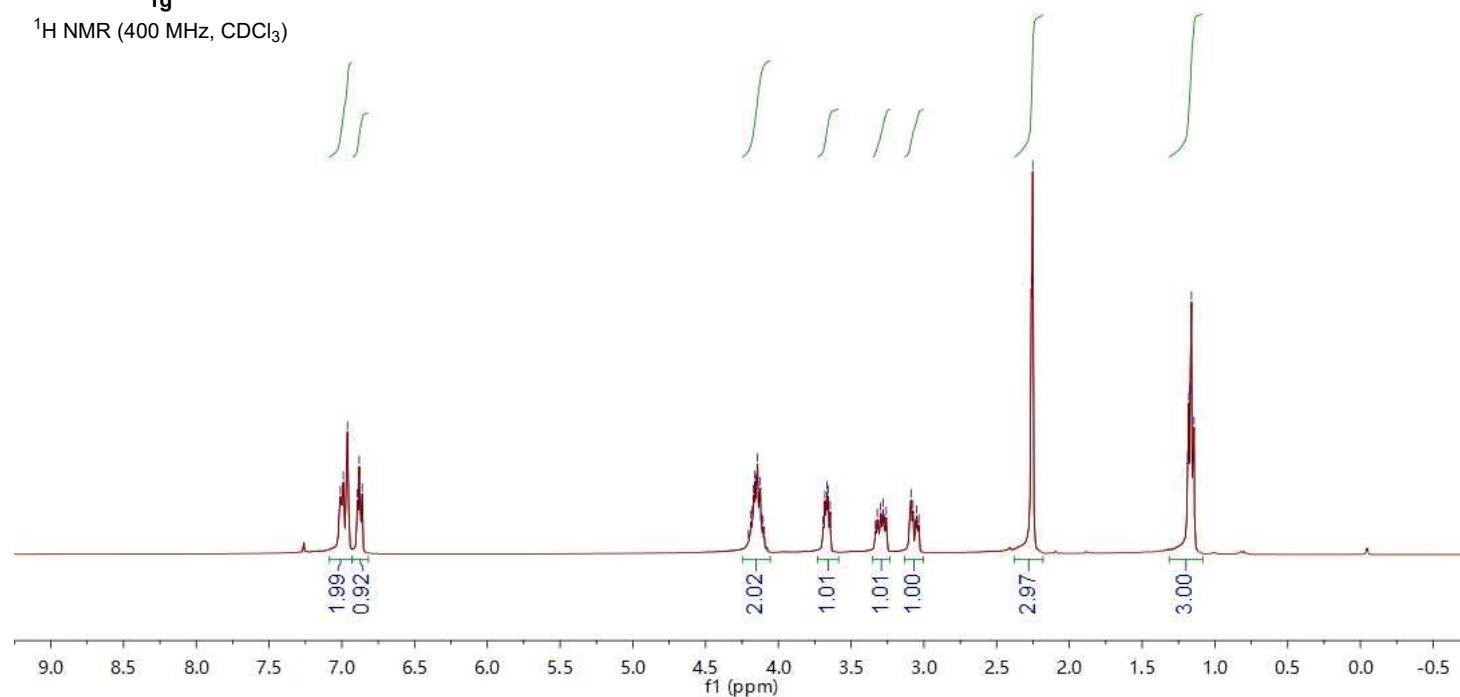
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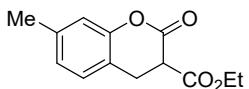
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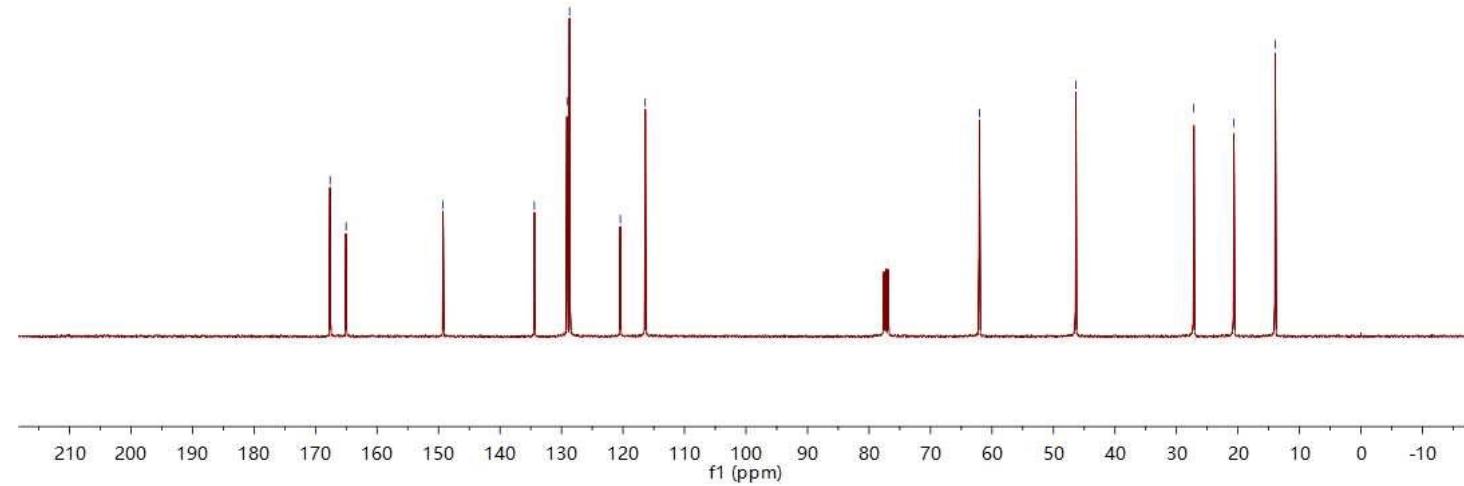
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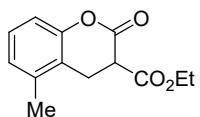
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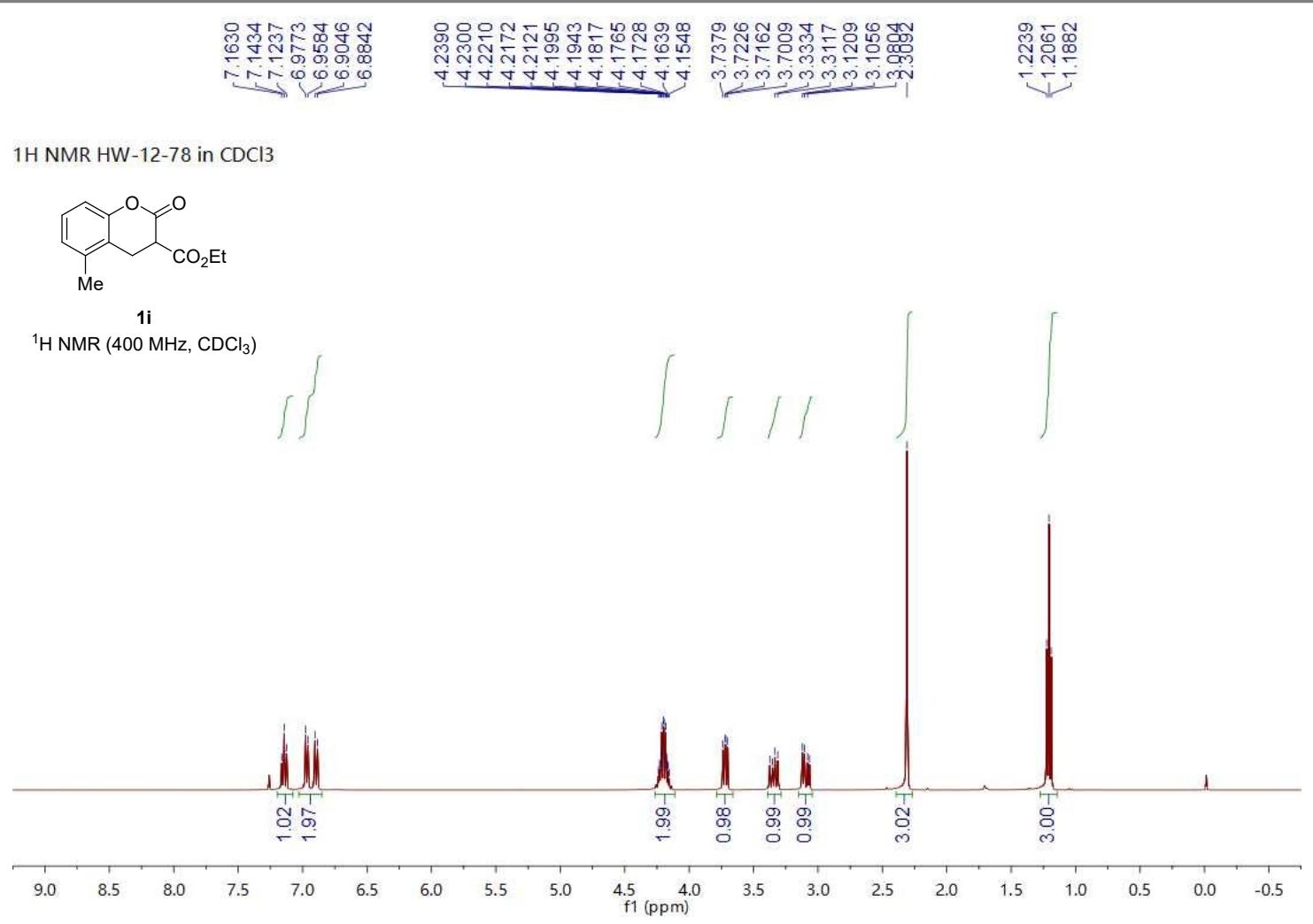
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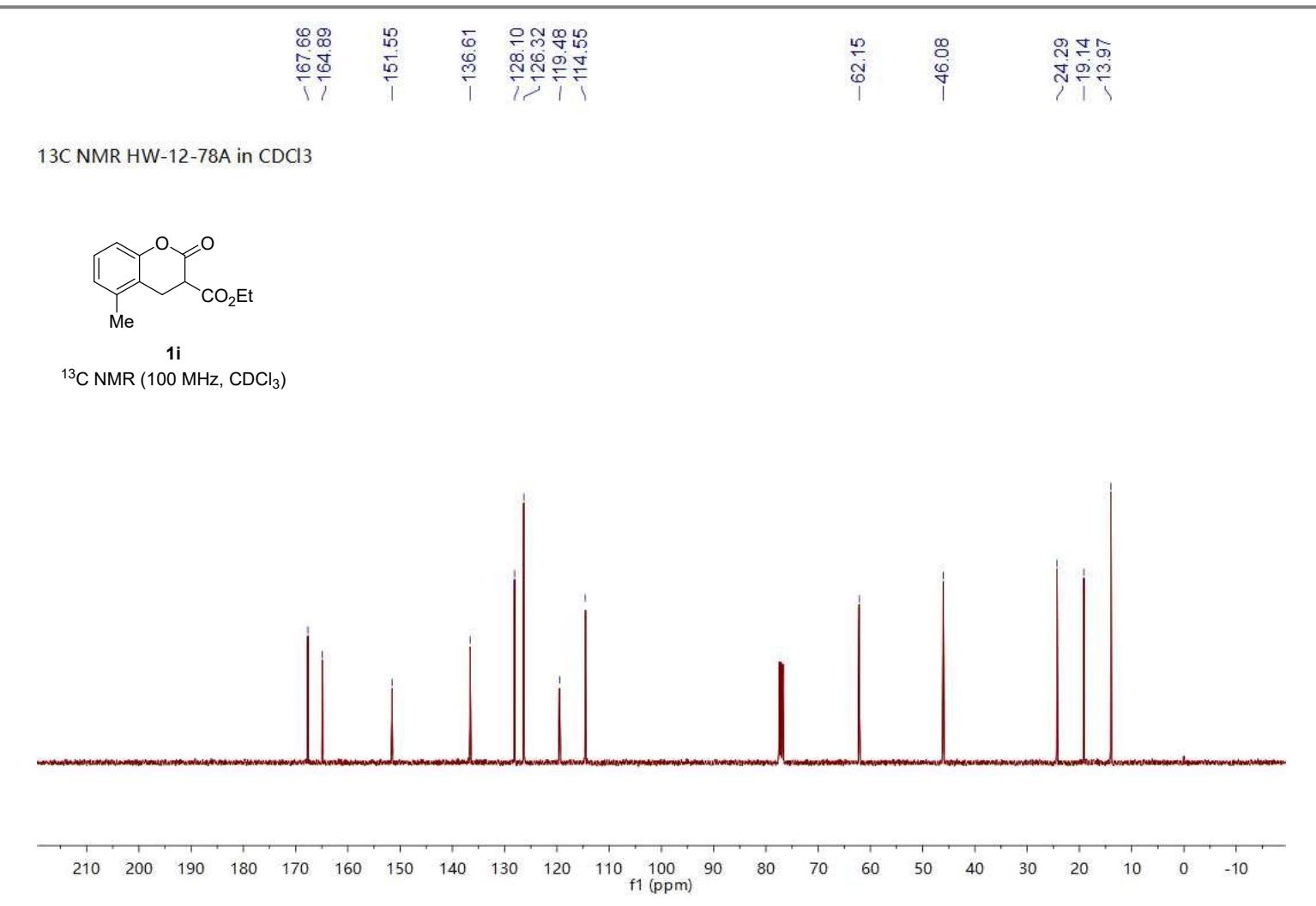


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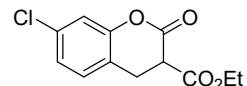


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¹H NMR (400 MHz, CDCl₃)



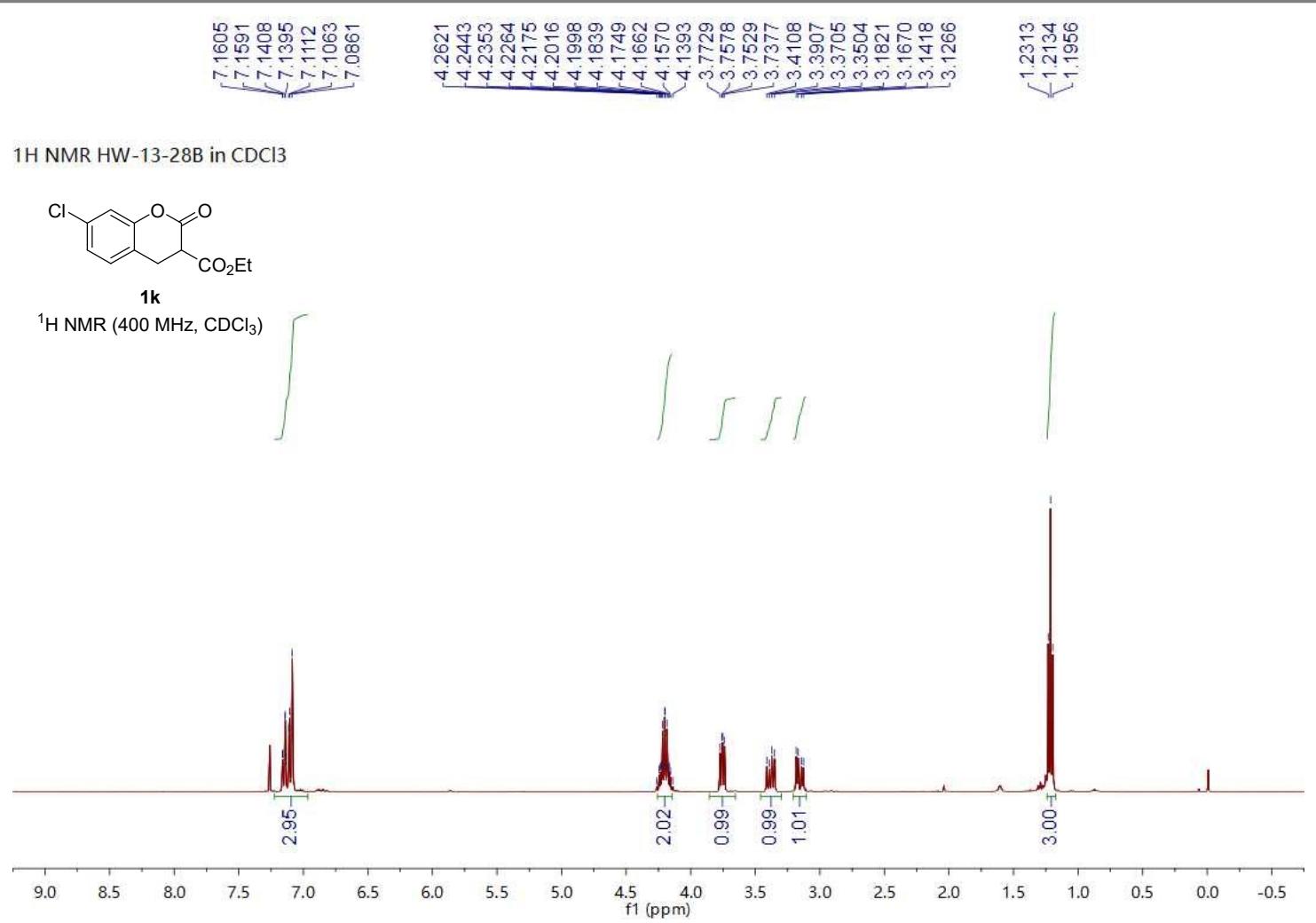


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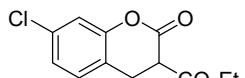


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¹H NMR (400 MHz, CDCl₃)

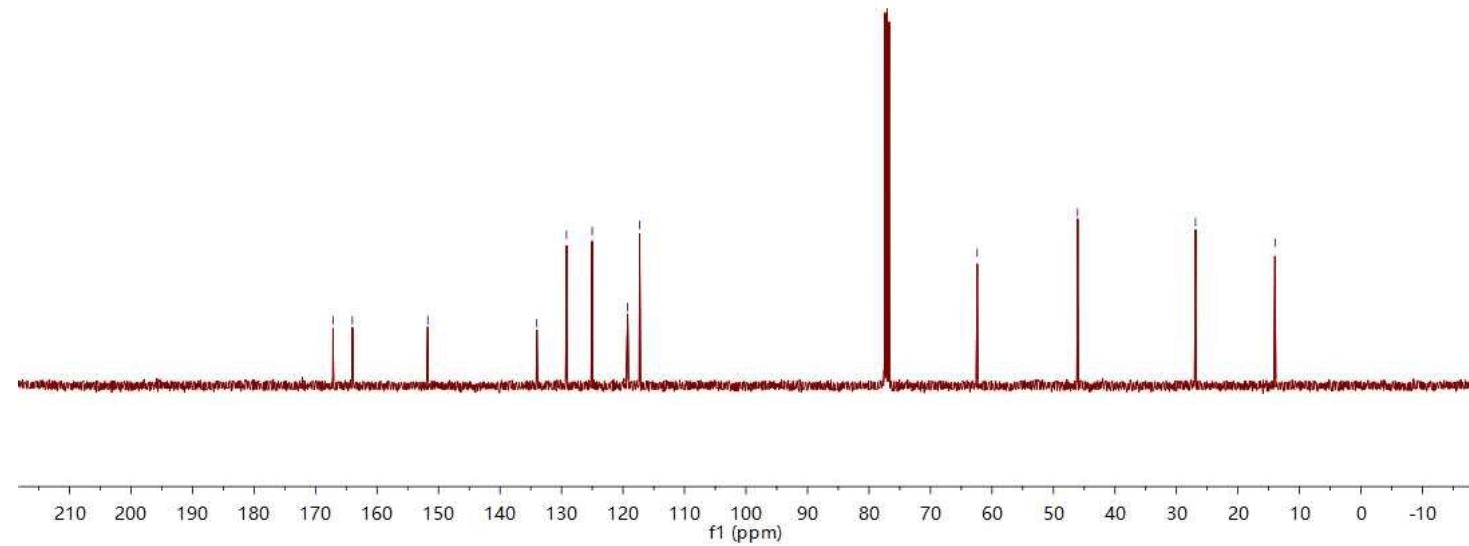


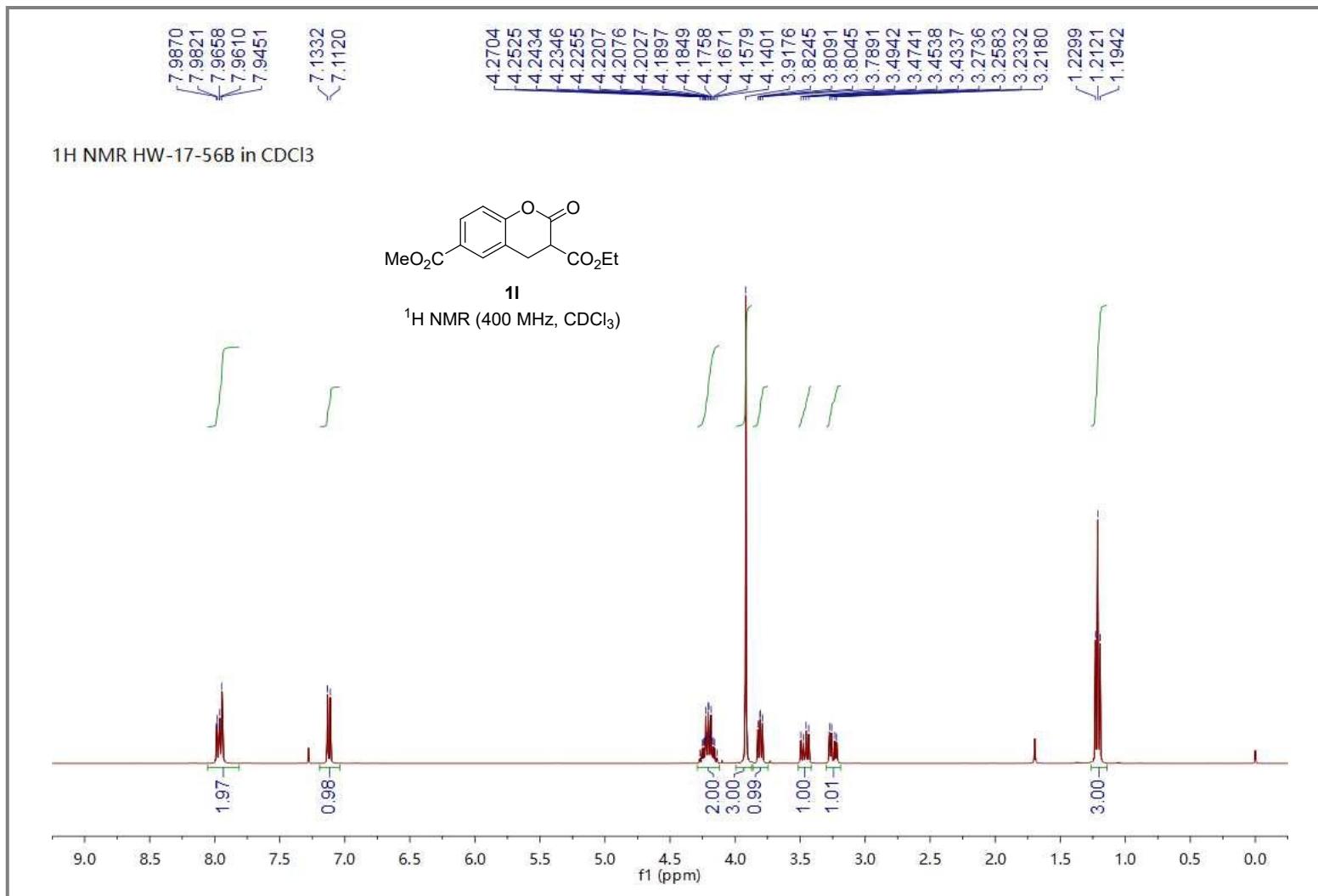
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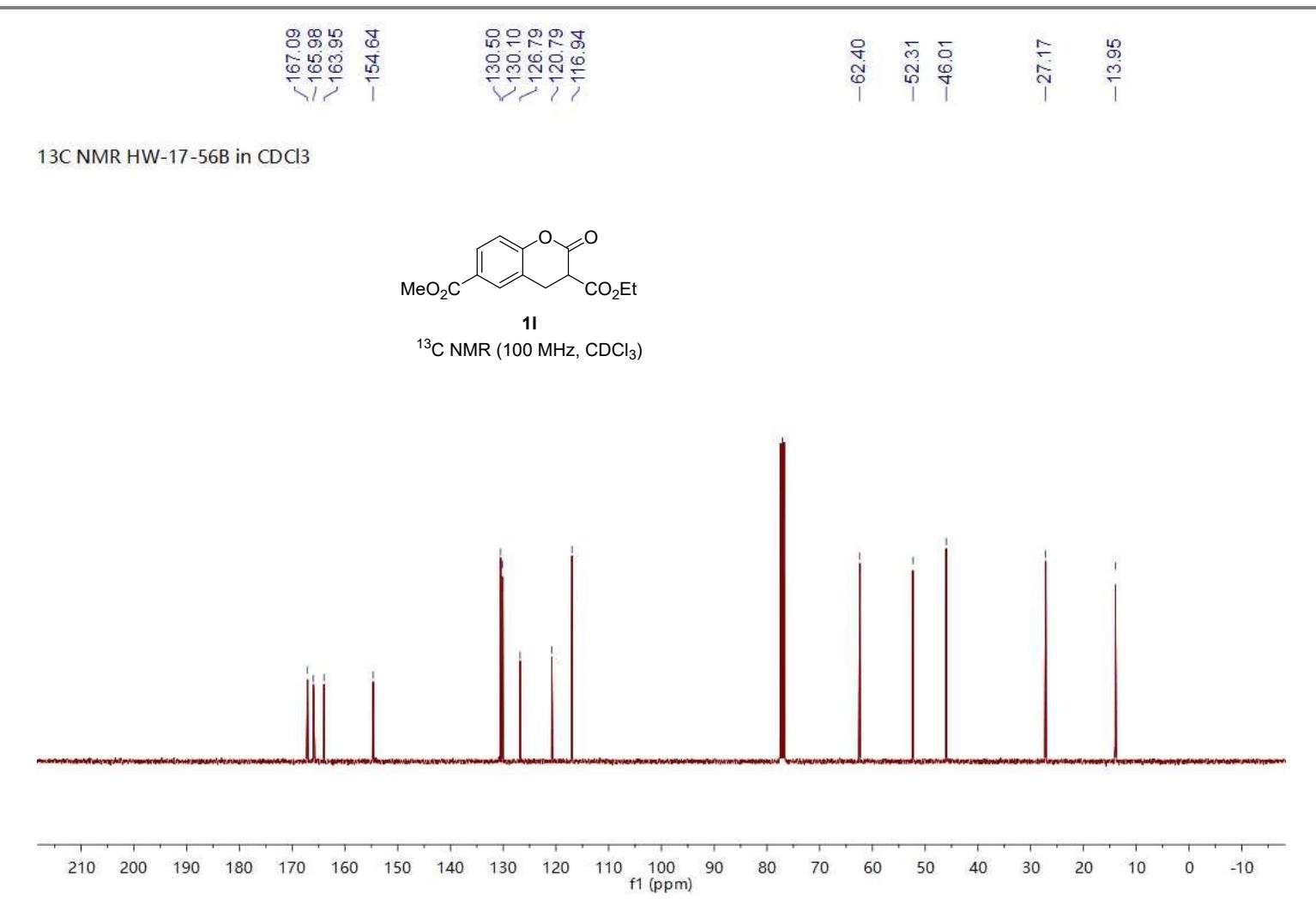


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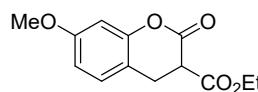
¹³C NMR (100 MHz, CDCl₃)





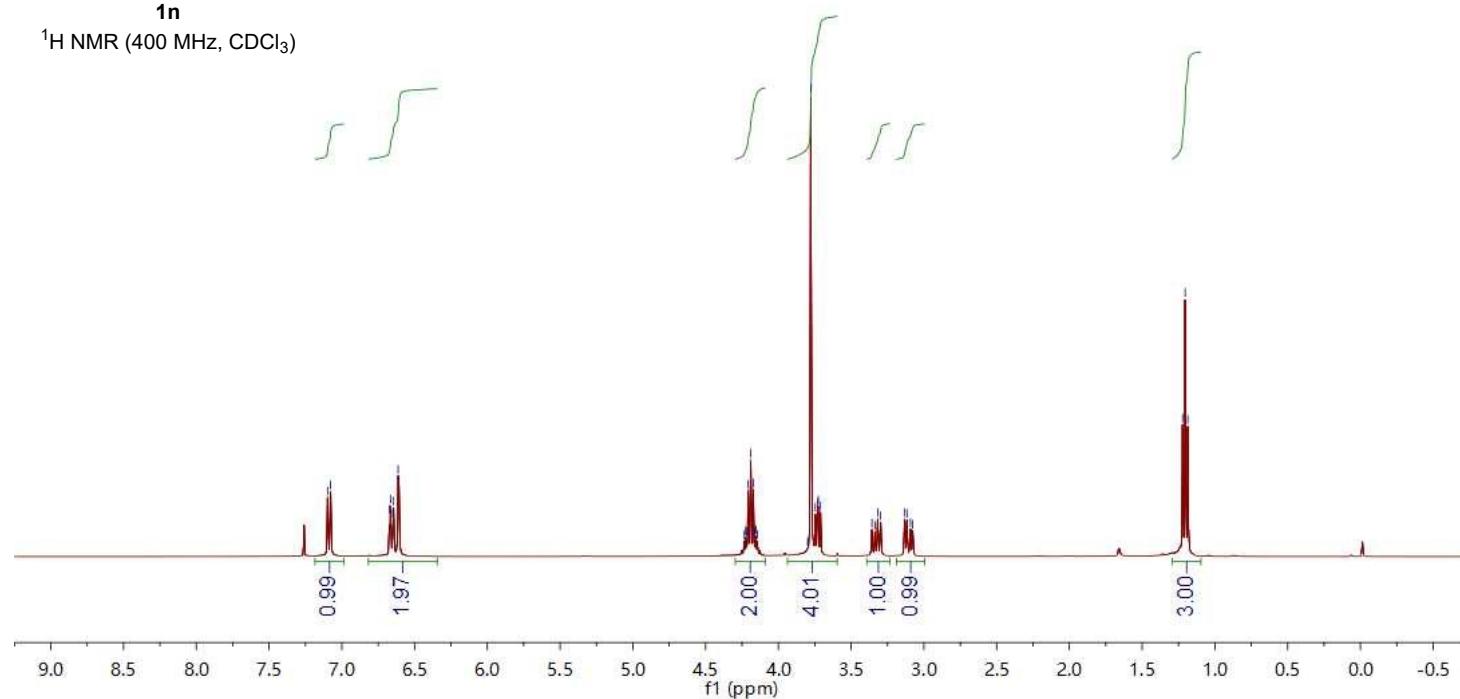


¹H NMR HW-12-82A in CDCl₃

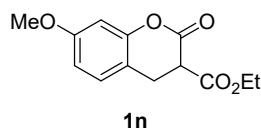


1n

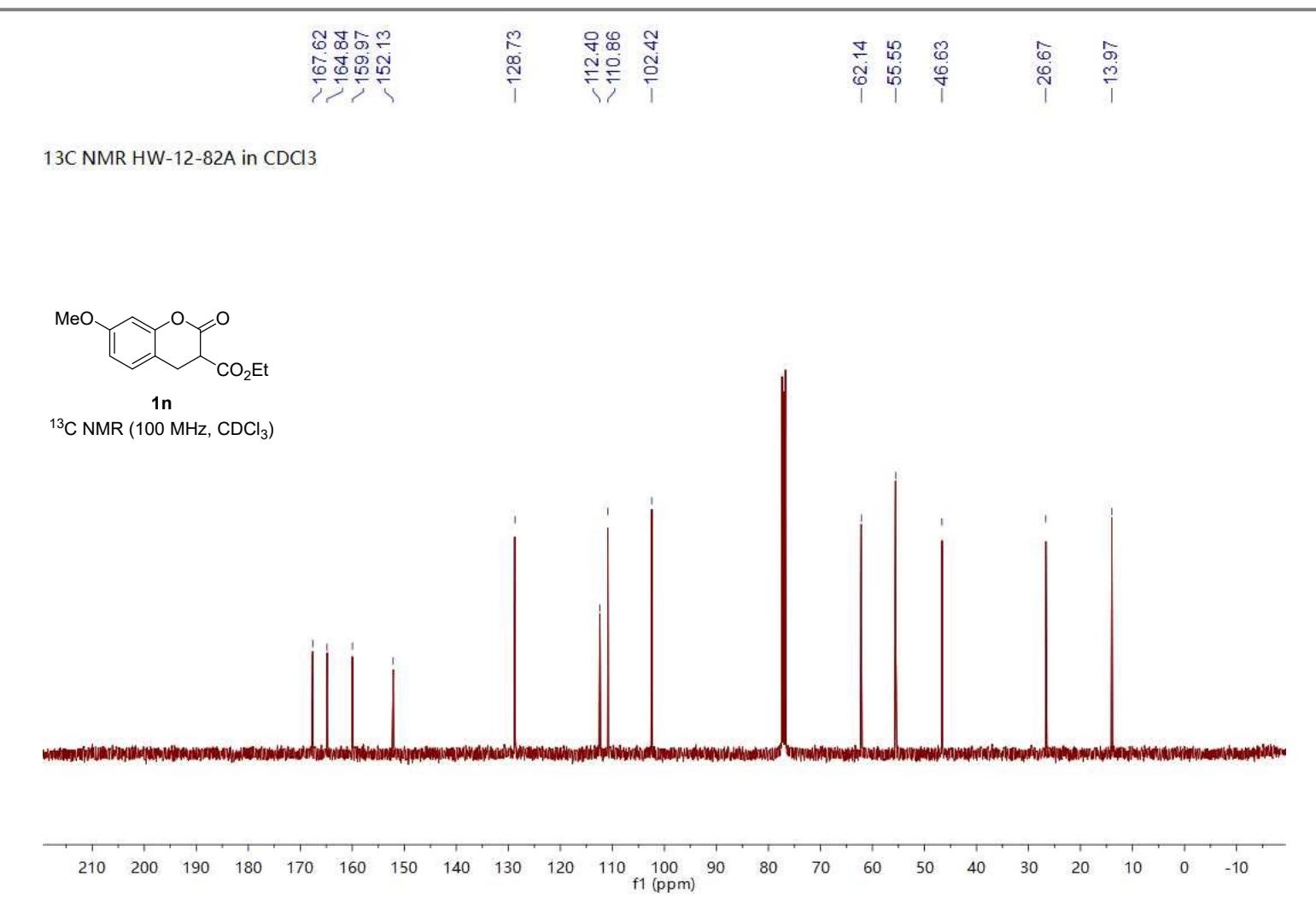
¹H NMR (400 MHz, CDCl₃)



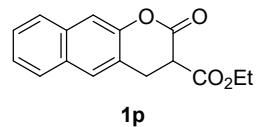
¹³C NMR HW-12-82A in CDCl₃



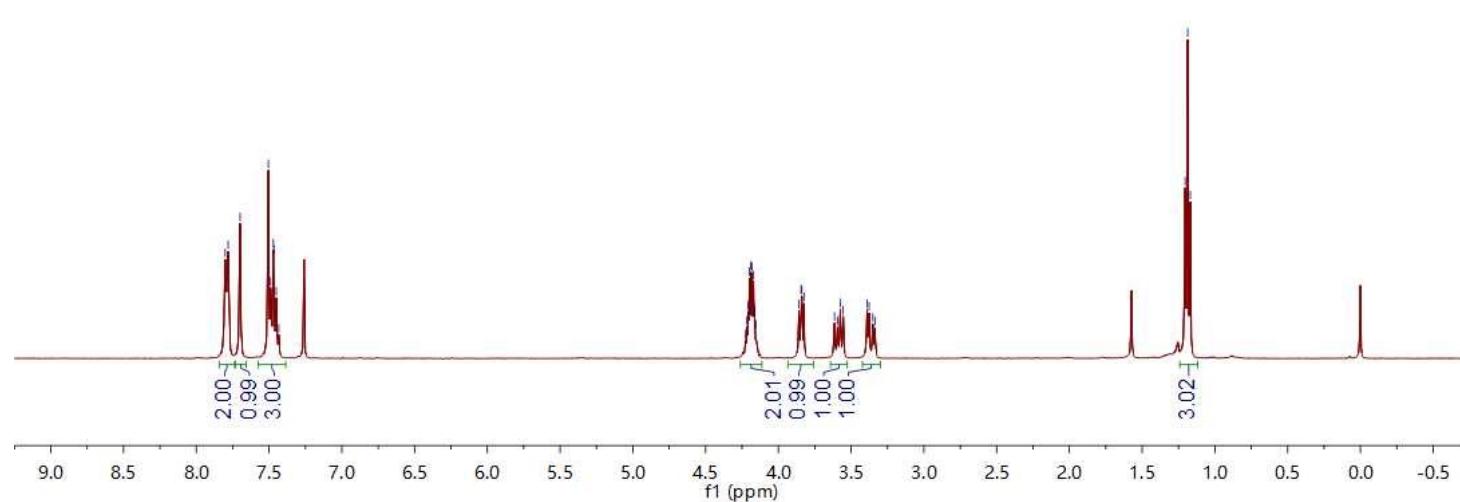
¹³C NMR (100 MHz, CDCl₃)

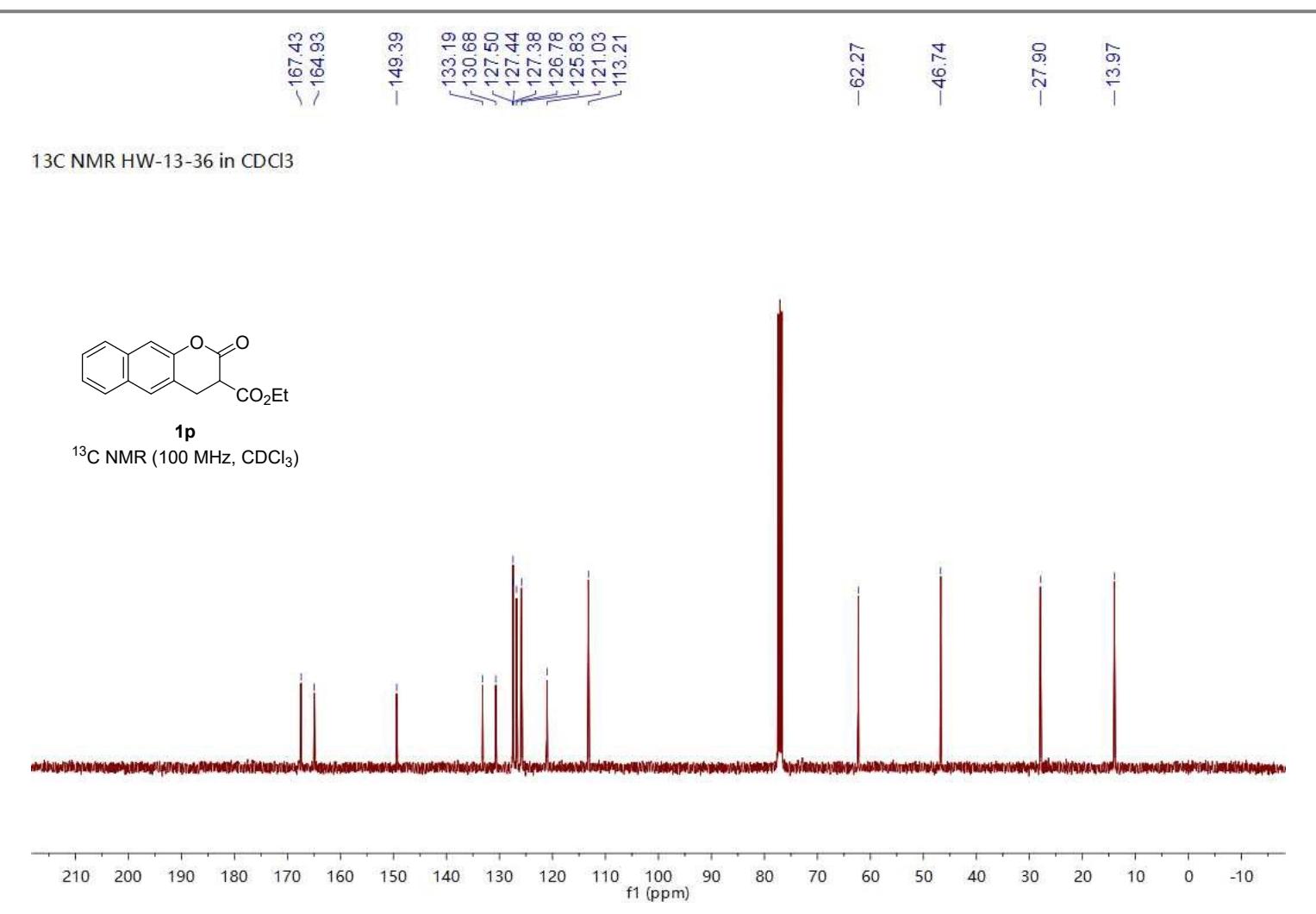


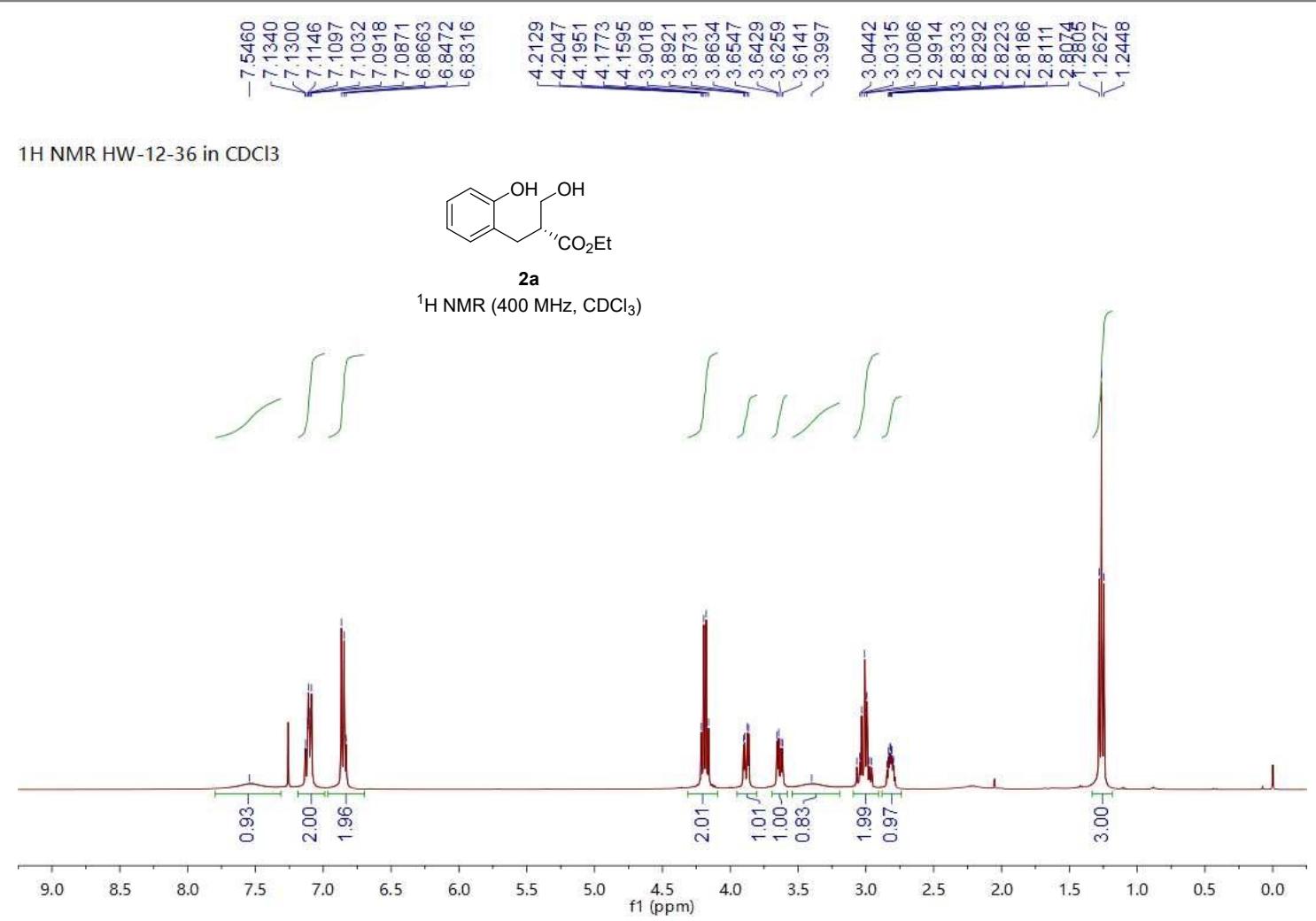
¹H NMR HW-13-36 in CDCl₃



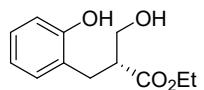
¹H NMR (400 MHz, CDCl₃)







¹³C NMR HW-12-36 in CDCl₃



2a

¹³C NMR (100 MHz, CDCl₃)

-175.23

-154.89

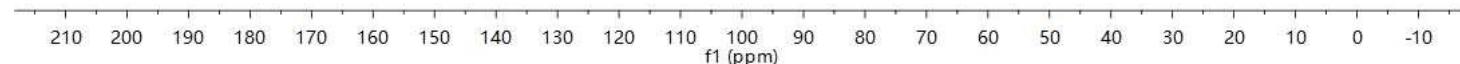
-131.07
-128.28
-124.61
-120.78
-116.71

61.45
61.24

-47.67

-28.13

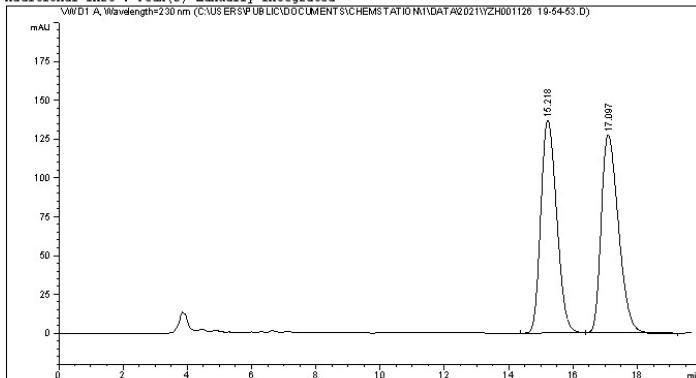
-14.16



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2021\YZH001126 19-54-53.D
Sample Name: HW-10-274/-

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Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 12/23/2021 7:54:53 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 12/23/2021 7:19:09 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:24:47 AM by SYSTEM
                                                (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

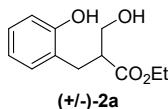


=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.218	BB	0.5413	4693.18555	137.10939	49.8009
2	17.097	BB	0.5803	4730.71143	127.37619	50.1991



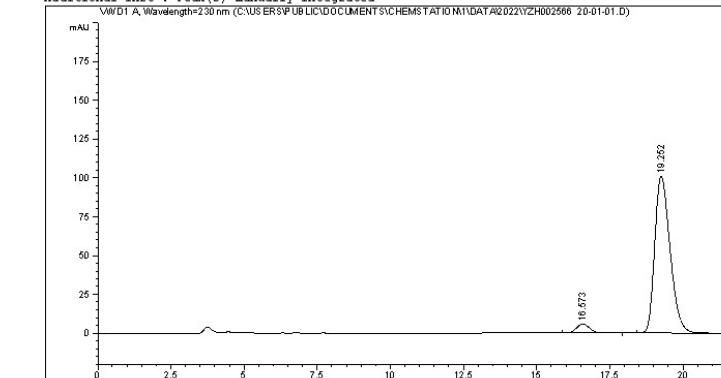
HPLC1260 II 10/17/2023 10:24:52 AM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002566 20-01-01.D
Sample Name: HW-12-36

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Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 10/21/2022 8:01:02 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/21/2022 7:49:42 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:26:34 AM by SYSTEM
                                                (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

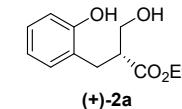


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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

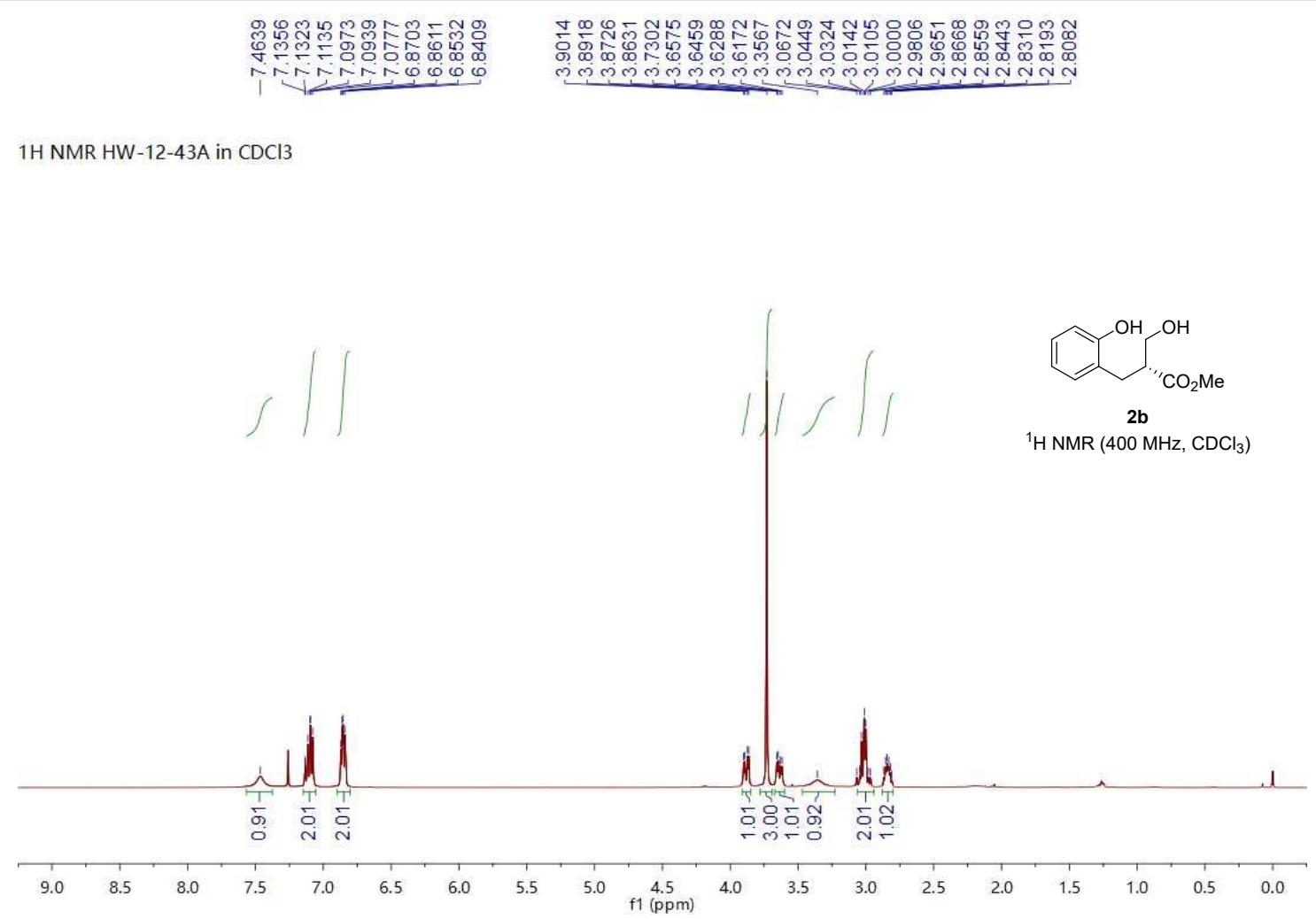
Signal 1: VWD1 A, Wavelength=230 nm

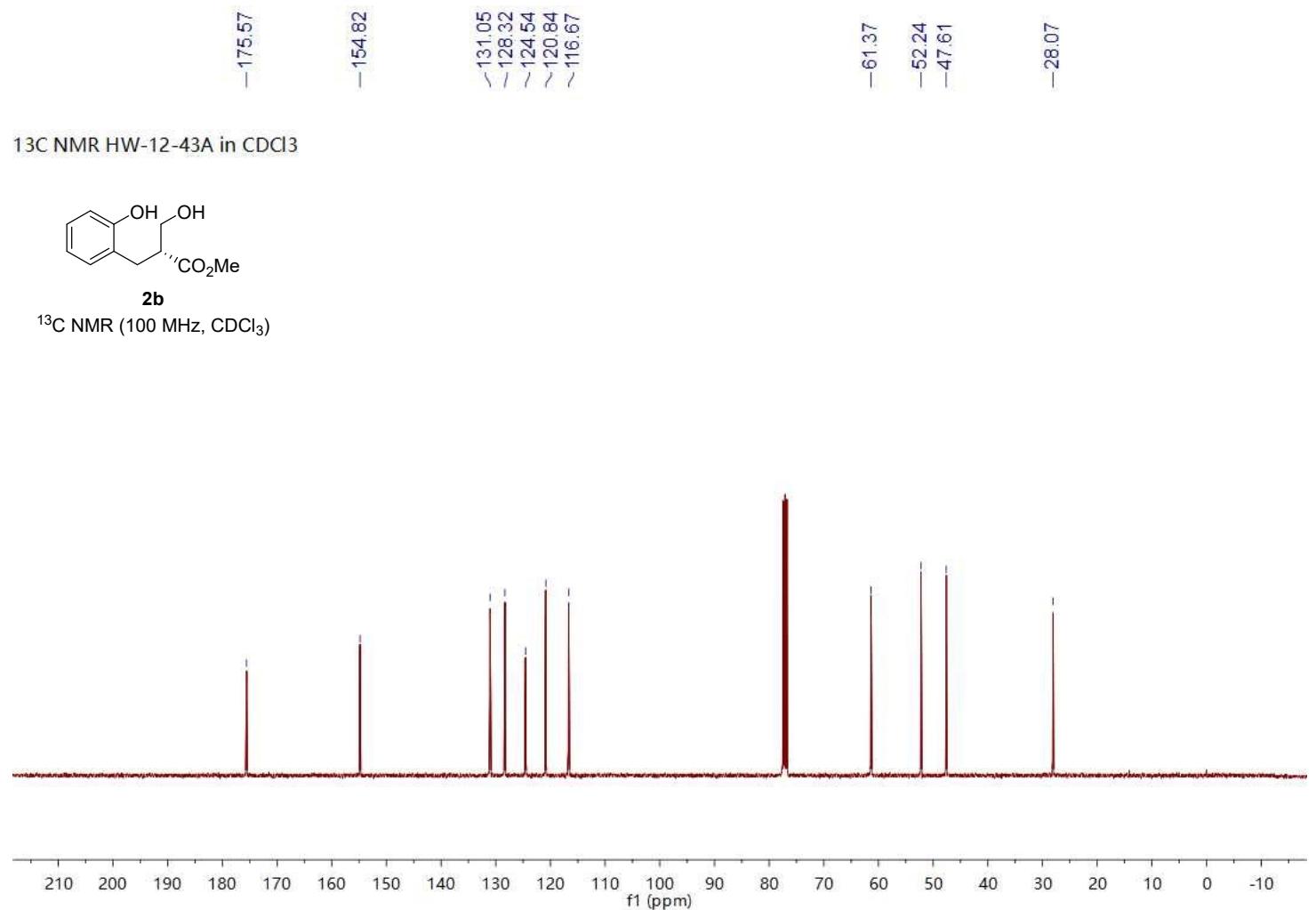
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.573	BB	0.4750	181.65741	5.90364	4.7155
2	19.252	BB	0.5613	3670.72803	101.16671	95.2845



HPLC1260 II 10/17/2023 10:26:41 AM SYSTEM

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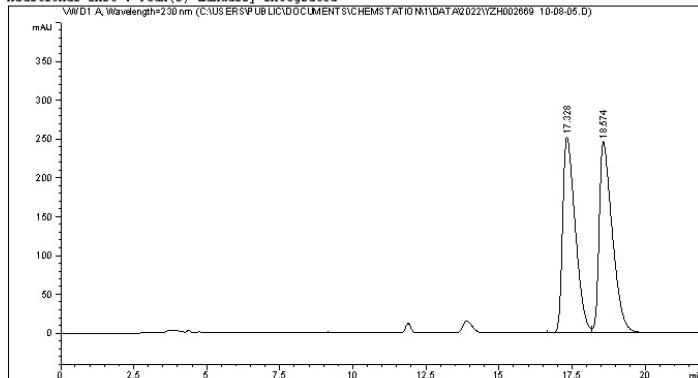




Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002669 10-08-05.D
Sample Name: HW-12-43A+/-

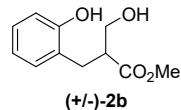
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Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 10/31/2022 10:08:06 AM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/31/2022 9:54:39 AM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:28:02 AM by SYSTEM
                           (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs



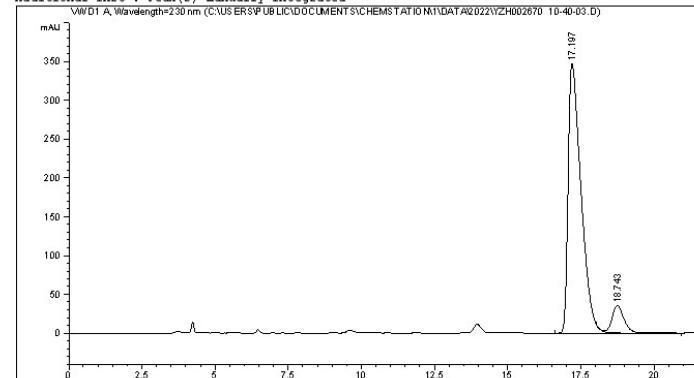
HPLC1260 II 10/17/2023 10:28:06 AM SYSTEM

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Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002670 10-40-03.D
Sample Name: HW-12-43A

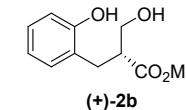
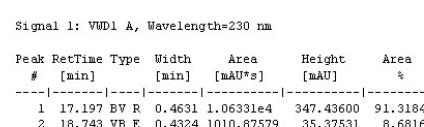
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=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 10/31/2022 10:40:03 AM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/31/2022 9:54:39 AM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:28:02 AM by SYSTEM
                           (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



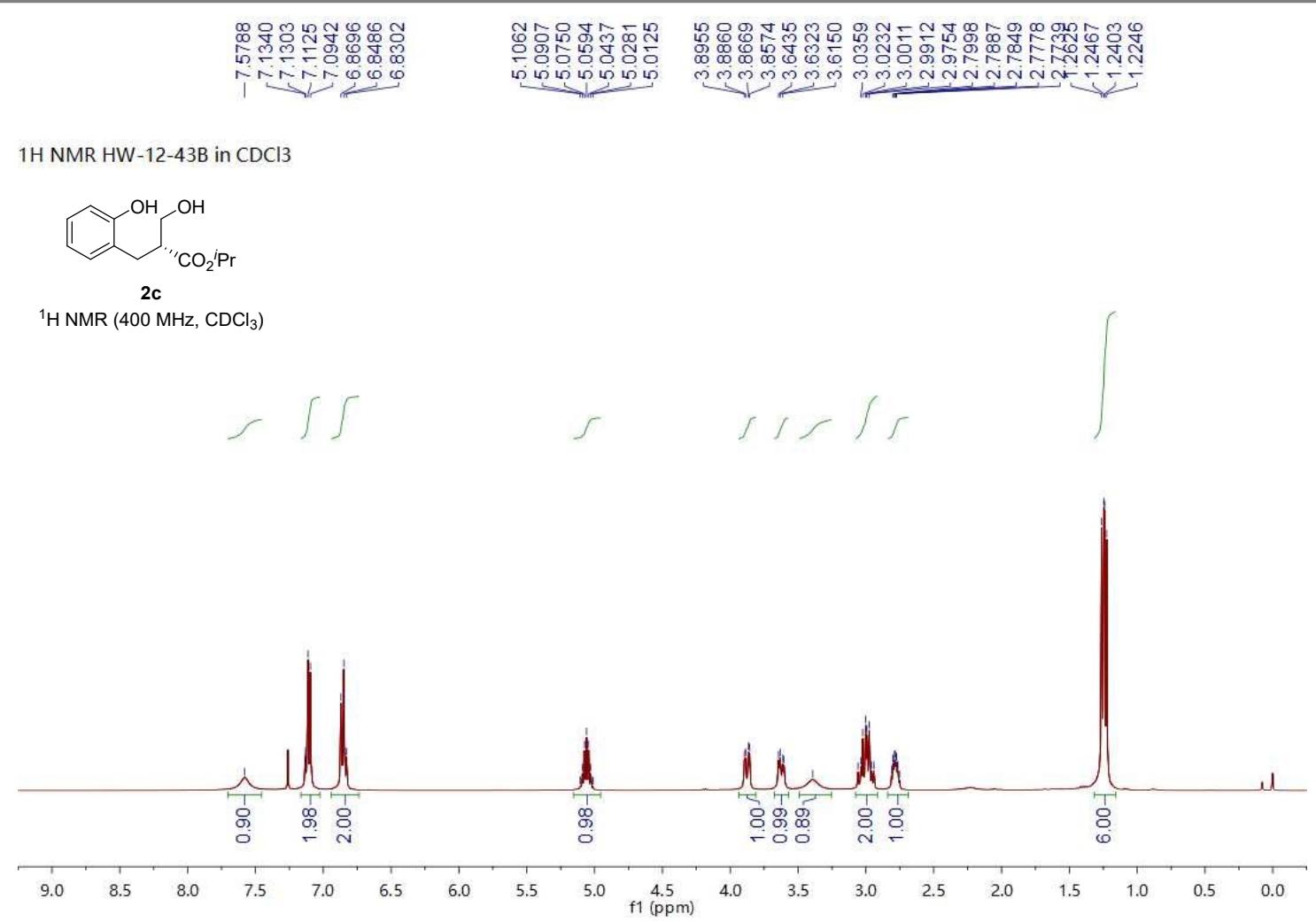
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Area Percent Report
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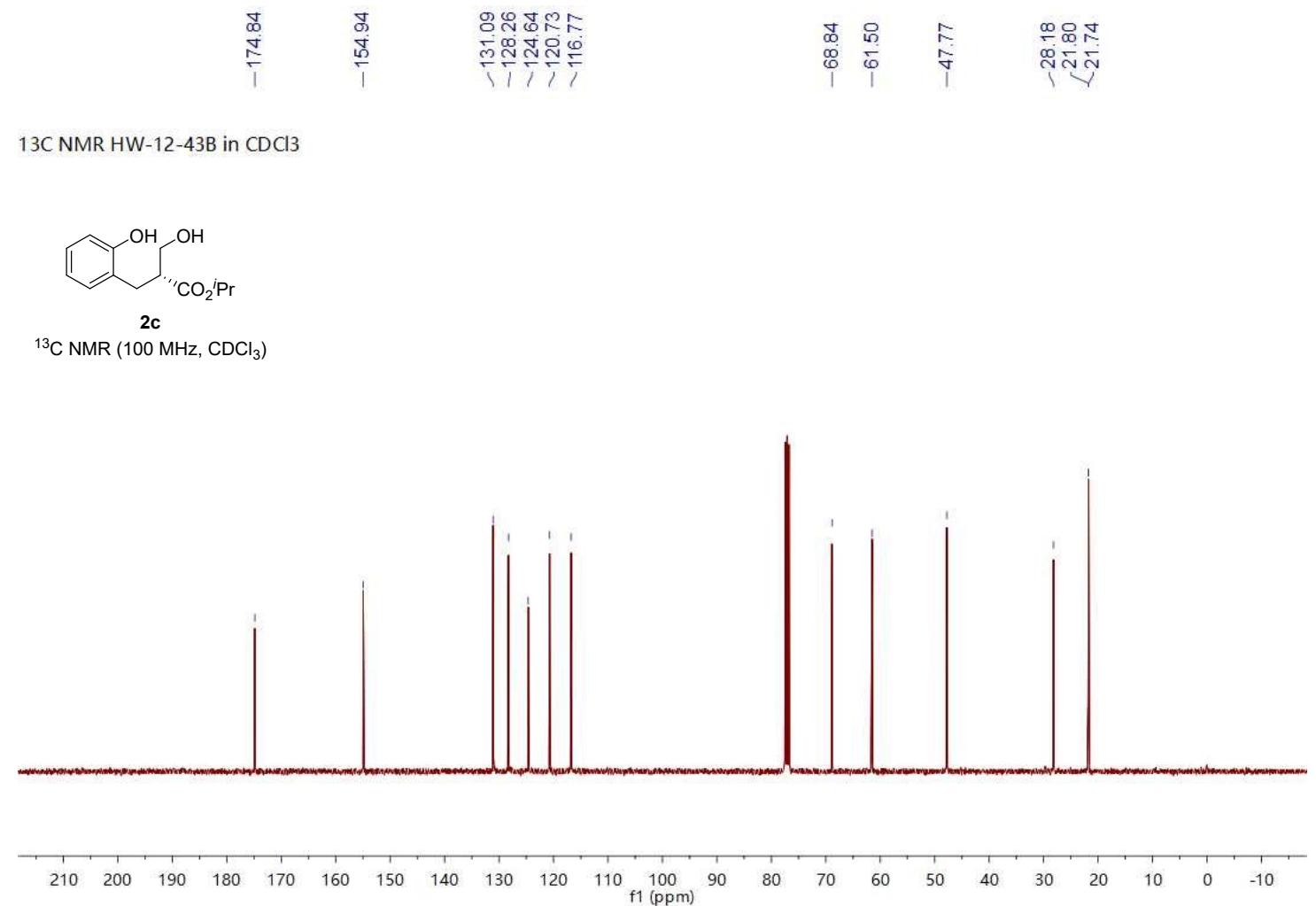
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs



HPLC1260 II 10/17/2023 10:28:49 AM SYSTEM

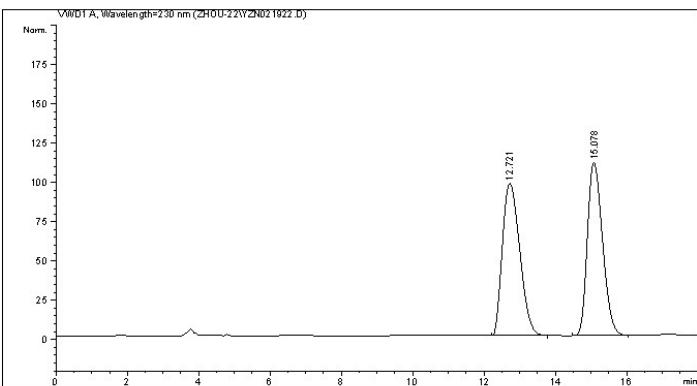
Page 1 of 2





Data File C:\CHEM32\1\DATA\ZHOU-22\YZN021922.D
Sample Name: HW-12-43B+/-

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Acq. Operator : Instrument 1 Location : -
Injection Date : 10/30/2022 10:20:46 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/30/2022 10:20:15 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:18:18 PM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

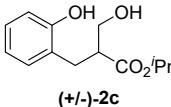


```
=====
Area Percent Report
=====
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	12.721	BB	0.5415	3272.87427	96.79517	50.1478	
2	15.078	BB	0.4621	3253.57910	110.28246	49.8522	
Totals :				6526.45337	207.07764		

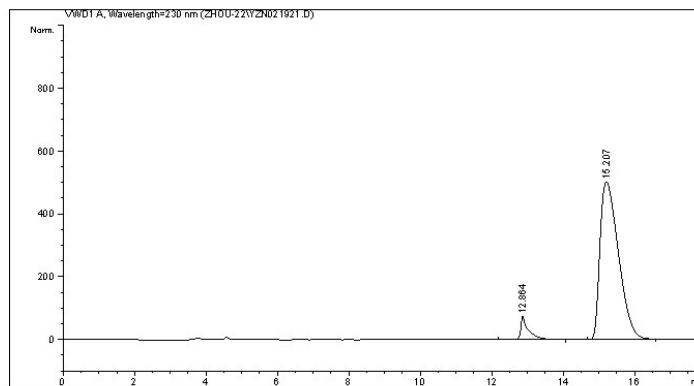


Instrument 1 10/17/2023 10:18:28 PM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-22\YZN021921.D
Sample Name: HW-12-43B

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 10/30/2022 9:58:49 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/30/2022 9:47:11 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:20:10 PM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

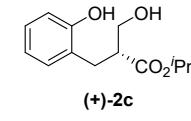


```
=====
Area Percent Report
=====
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

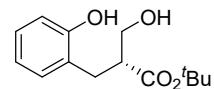
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	12.864	BB	0.1941	1091.21497	73.91610	5.7253	
2	15.207	BB	0.5657	1.79683e4	502.47910	94.2747	
Totals :				1.90595e4	576.39520		



Instrument 1 10/17/2023 10:20:13 PM

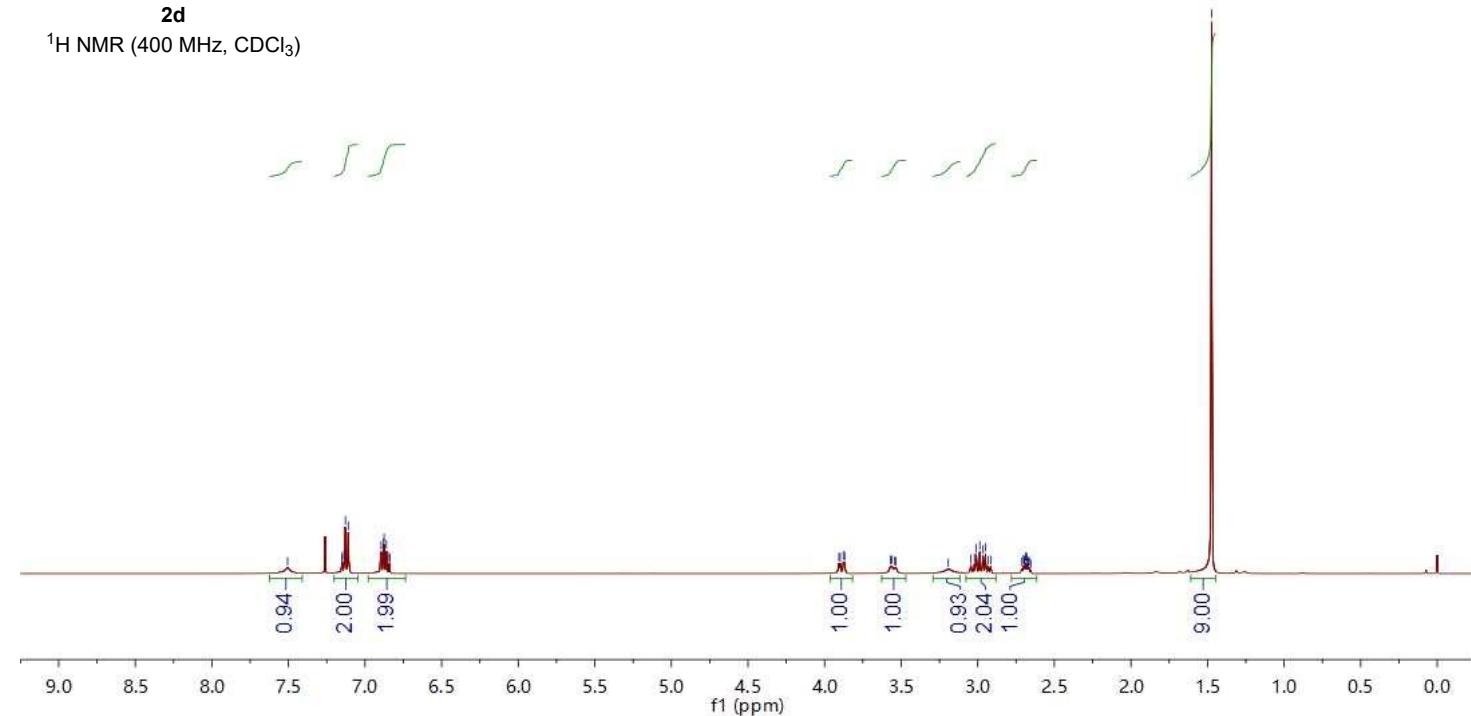
Page 1 of 1

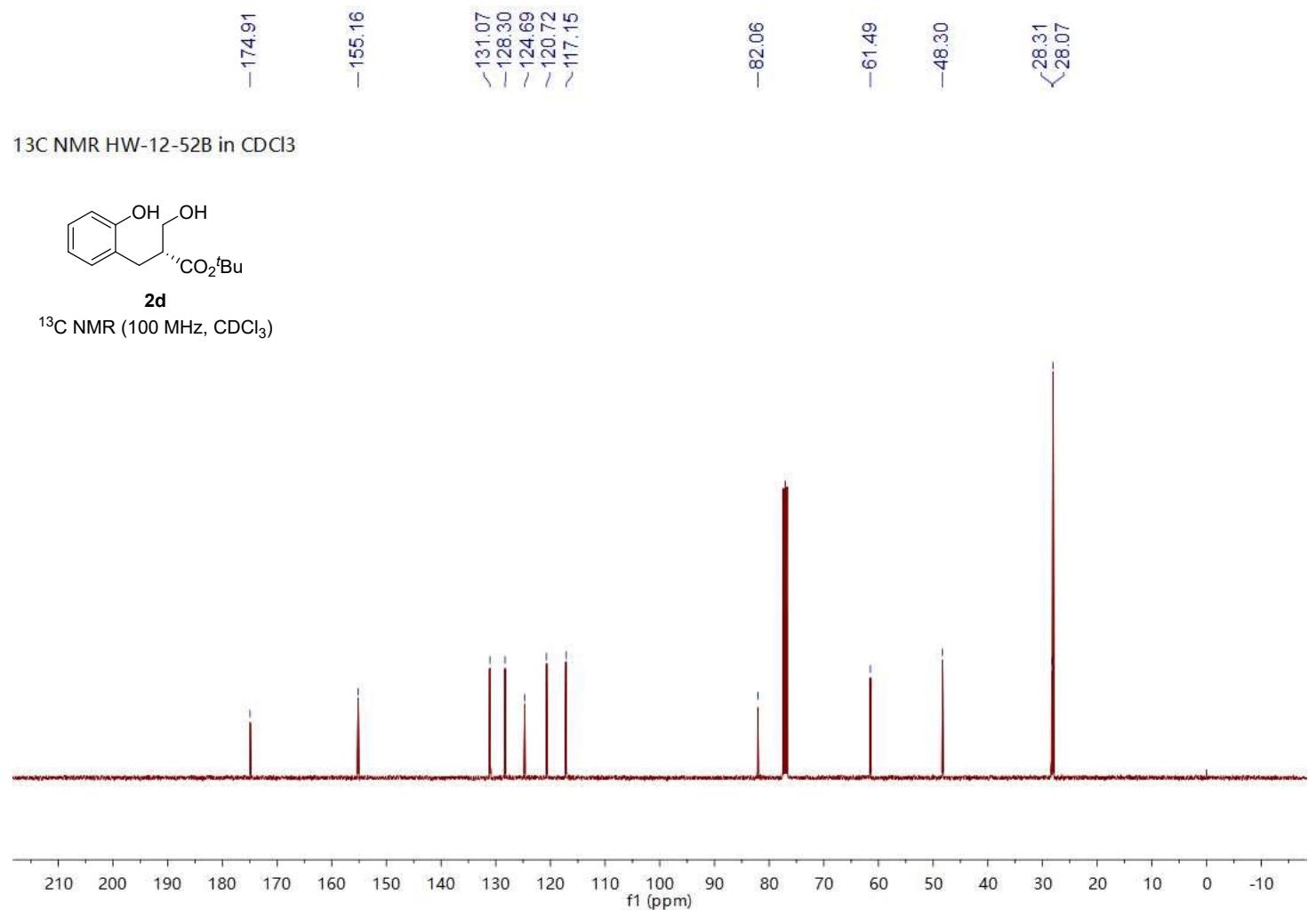
¹H NMR HW-12-52B in CDCl₃



2d

¹H NMR (400 MHz, CDCl₃)

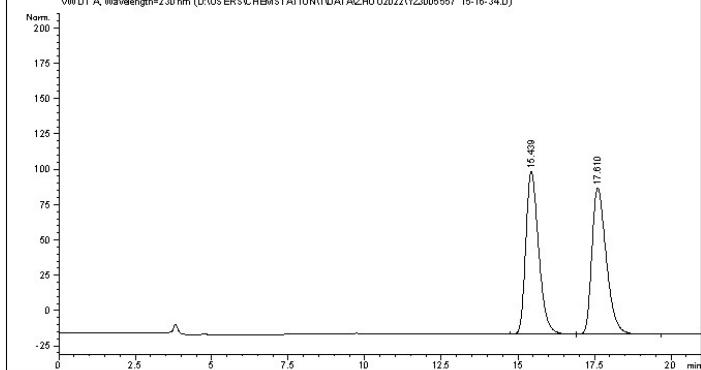




Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\Y23005557 15-16-34.D
Sample Name: HW-12-52B+/-

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Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 11/3/2022 3:16:34 PM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\lsm-1-58.M
Last changed   : 11/3/2022 3:15:39 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 10/17/2023 10:47:36 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, n-Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated
VWD1 A, Wavelength=230 nm (D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\Y23005557 15-16-34.D)

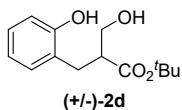


=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.439	BB	0.4512	2422.44336	82.83044	49.9052
2	17.610	BB	0.5026	2431.65088	74.54190	50.0948



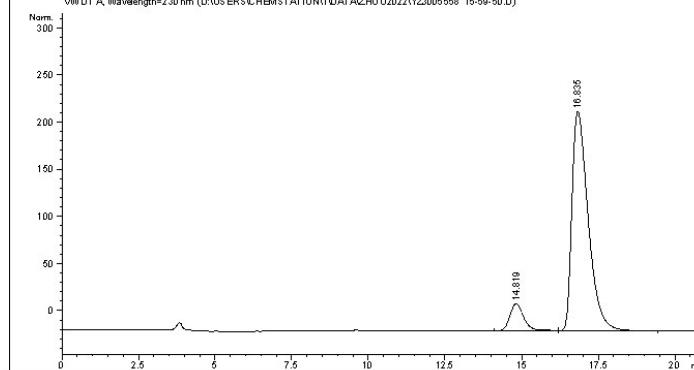
1260II 10/17/2023 10:47:38 AM SYSTEM

Page 1 of 2

Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\Y23005558 15-59-50.D
Sample Name: HW-12-52B

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 11/3/2022 3:59:50 PM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\lsm-1-58.M
Last changed   : 11/3/2022 3:59:23 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 10/17/2023 10:48:28 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, n-Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated
VWD1 A, Wavelength=230 nm (D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\Y23005558 15-59-50.D)

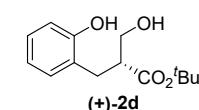


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Area Percent Report
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Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

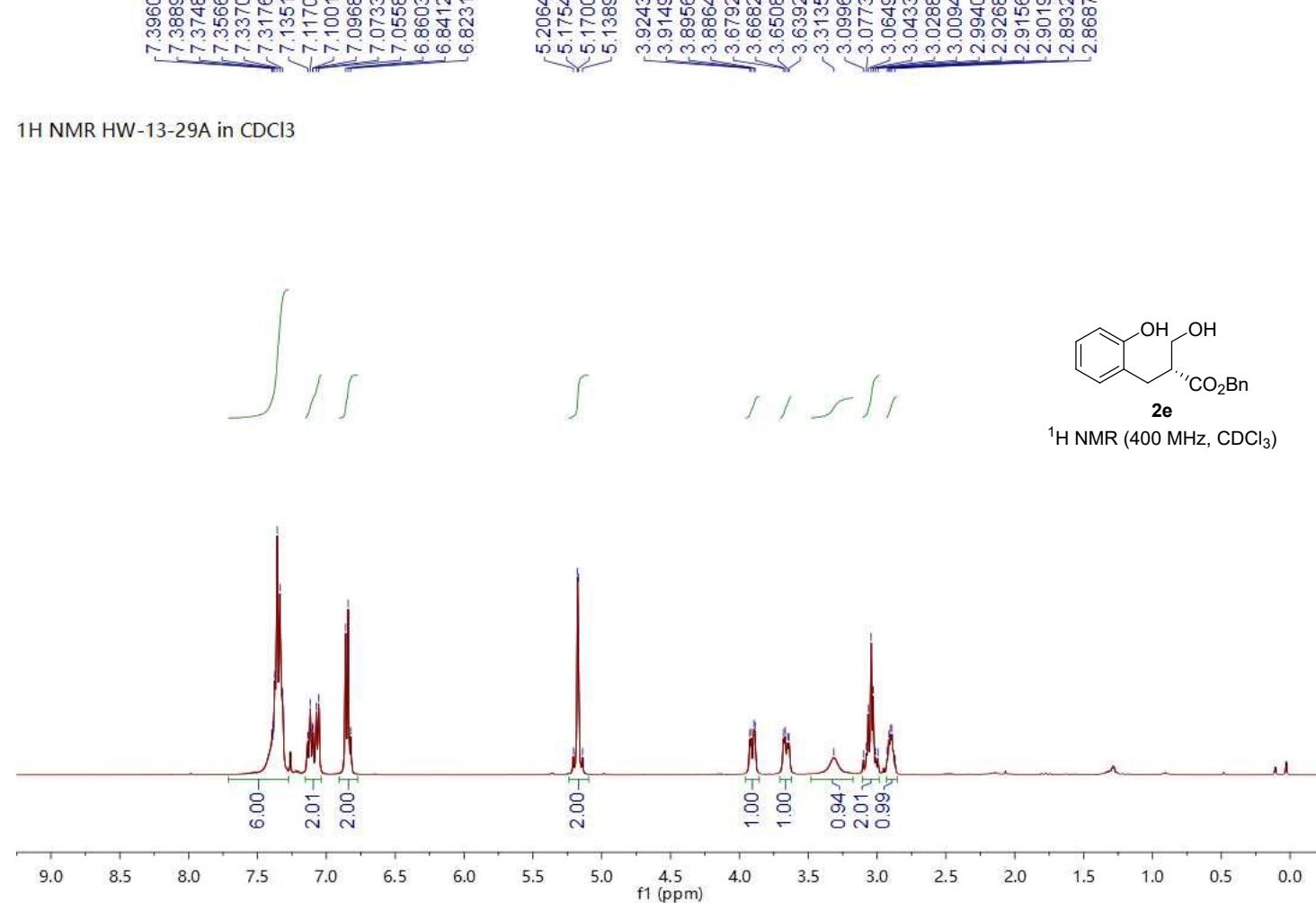
Signal 1: VWD1 A, Wavelength=230 nm

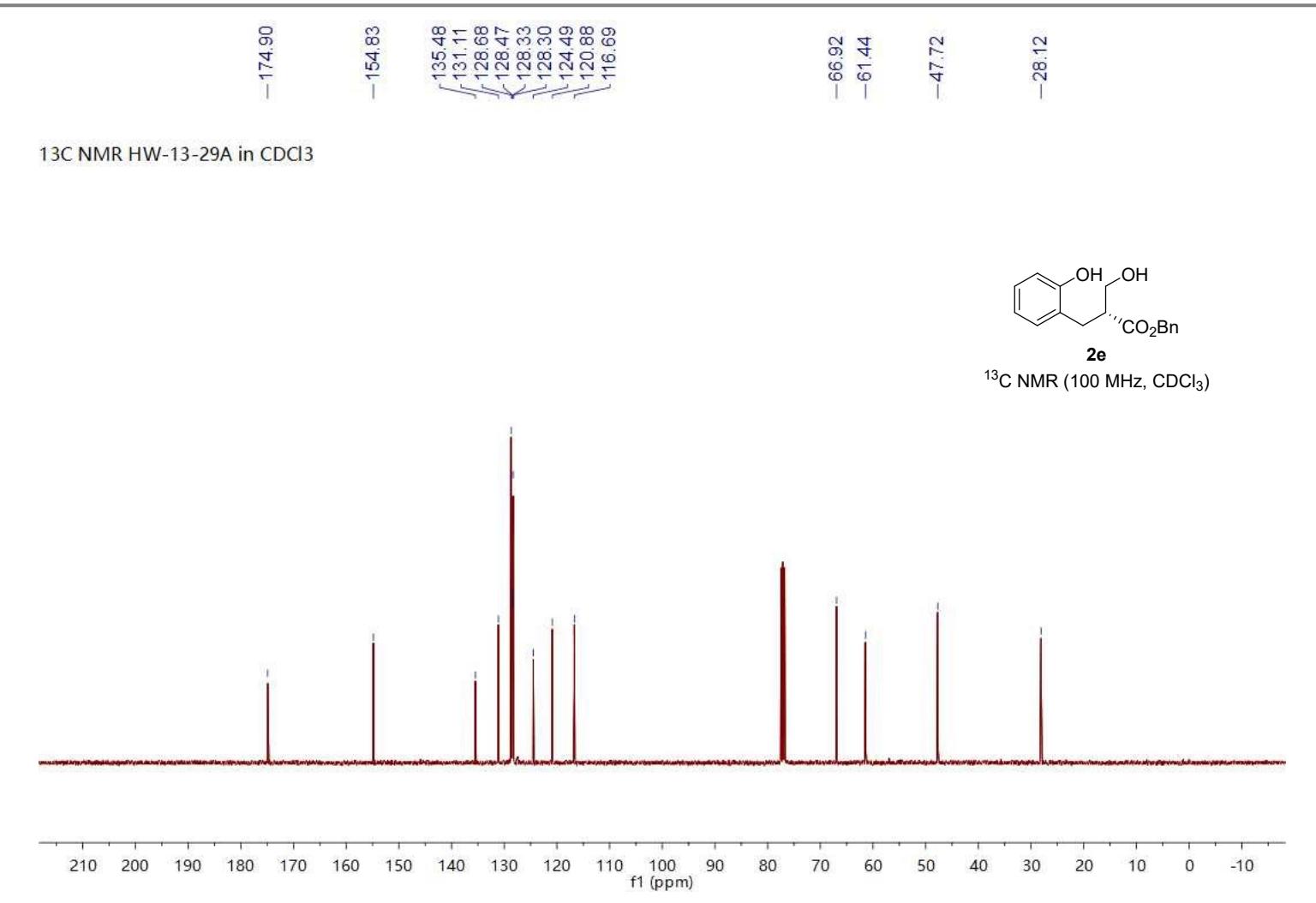
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.819	BB	0.4734	732.06317	23.63449	9.4616
2	16.835	BB	0.5639	7005.12598	190.55113	90.5384



1260II 10/17/2023 10:48:30 AM SYSTEM

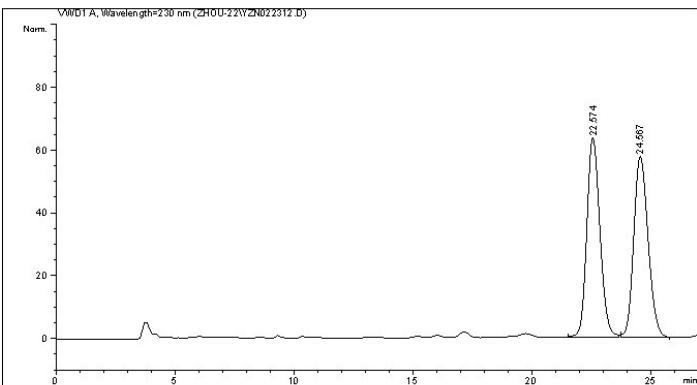
Page 1 of 2





Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022312.D
Sample Name: HW-13-29A+/-

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 1/4/2023 2:43:50 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 1/4/2023 2:41:14 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:22:19 PM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

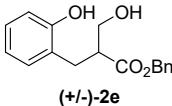


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=====
Area Percent Report
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```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

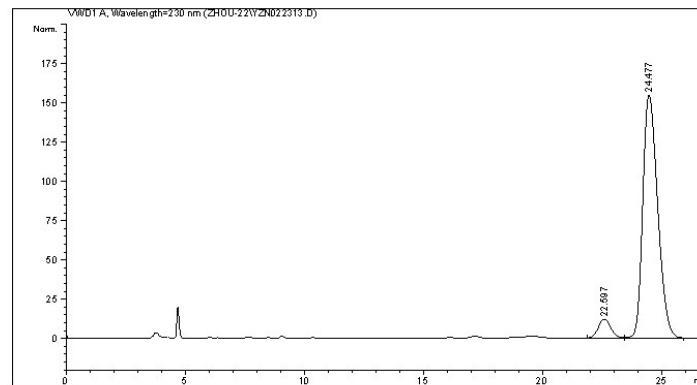
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	22.597	BB	0.5937	2422.07251	63.27313	50.1355	
2	24.567	BB	0.6518	2408.98291	57.50860	49.8645	
Totals :				4831.05542	120.78173		



Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022313.D
Sample Name: HW-13-29A

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 1/4/2023 3:12:47 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 1/4/2023 3:12:07 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:23:20 PM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

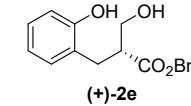


```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	22.597	BB	0.5685	436.08621	11.94907	6.1070	
2	24.477	VB	0.6742	6704.71338	154.81198	93.8930	
Totals :				7140.79959	166.76106		

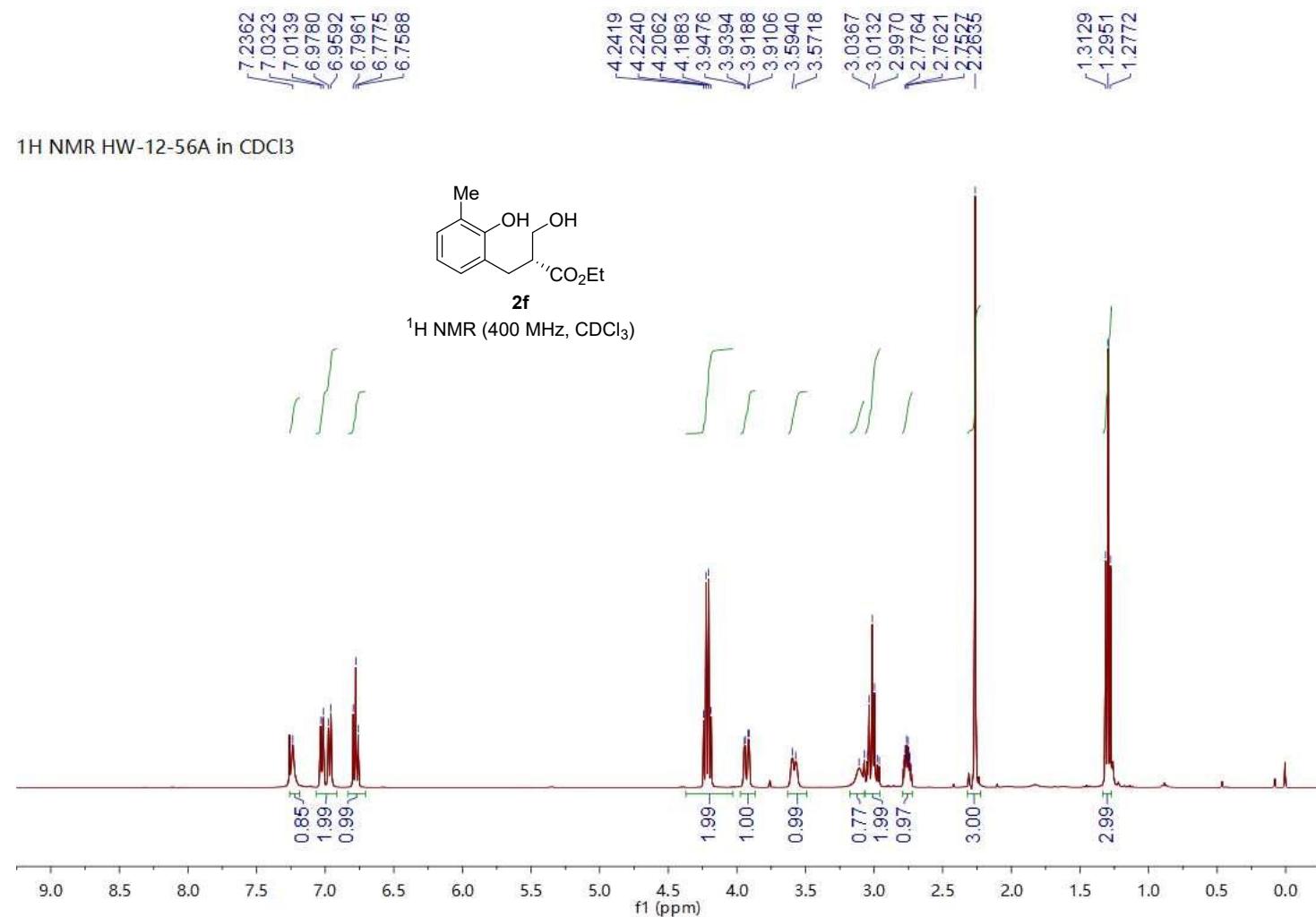


Instrument 1 10/17/2023 10:22:22 PM

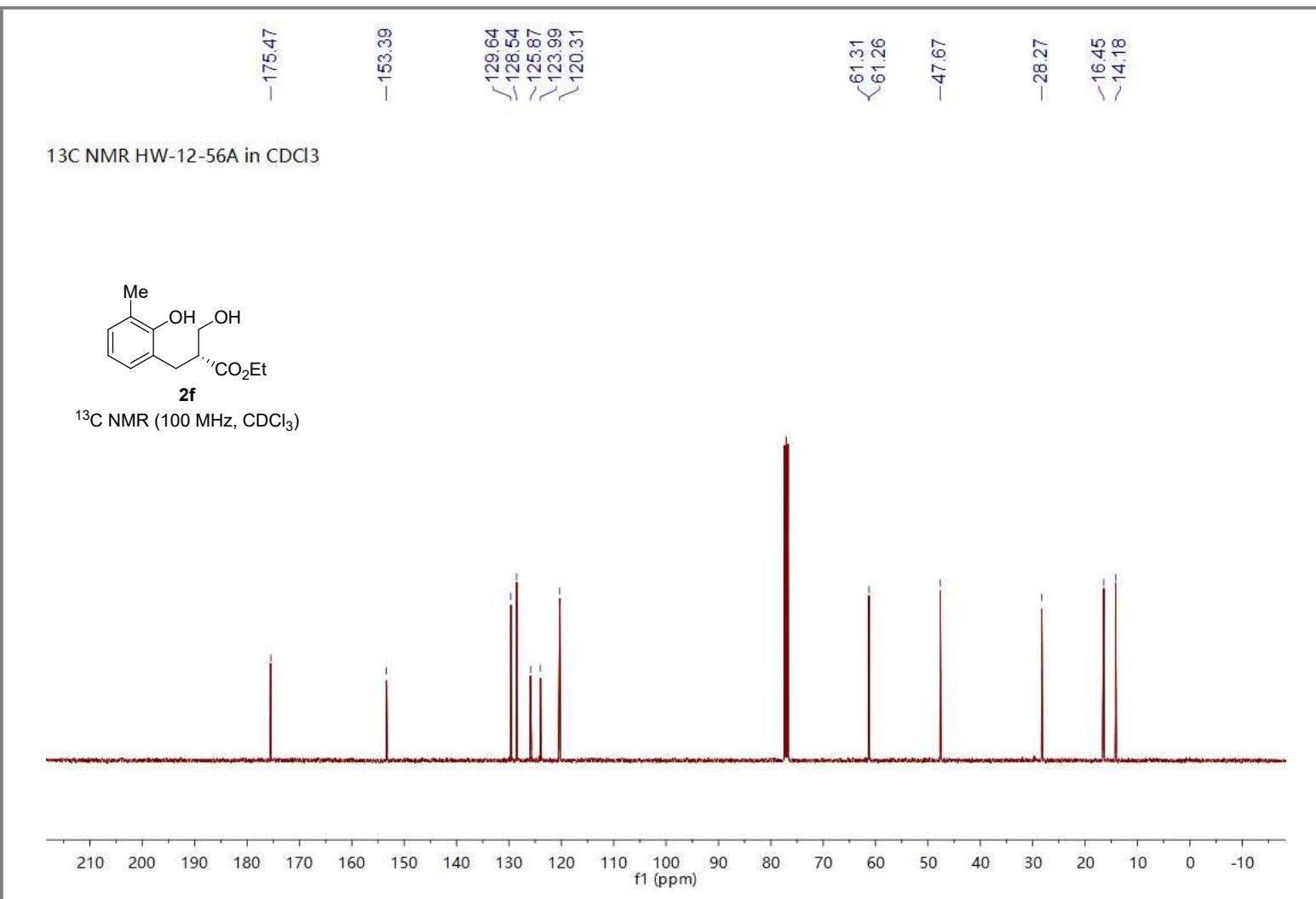
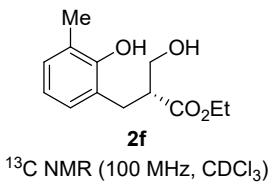
Page 1 of 1

Instrument 1 10/17/2023 10:23:25 PM

Page 1 of 1



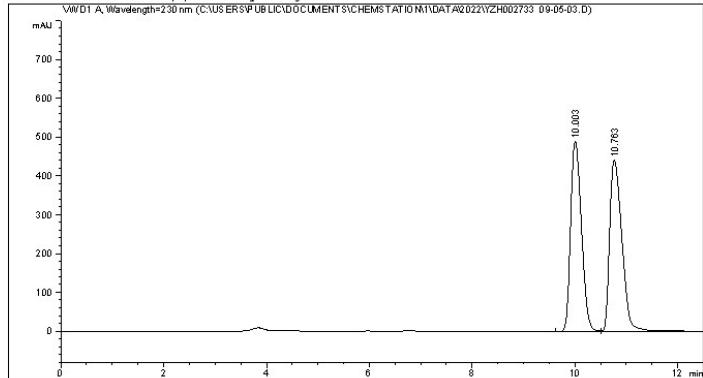
¹³C NMR HW-12-56A in CDCl₃



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002733 09-05-03.D
Sample Name: HW-12-56A+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/7/2022 9:05:03 AM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/7/2022 8:39:09 AM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:39:05 AM by SYSTEM
                                                (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

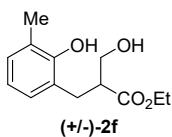


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.003	BV	0.2300	7136.70947	489.74524	49.3328
2	10.763	VB	0.2606	7329.75830	441.75406	50.6672



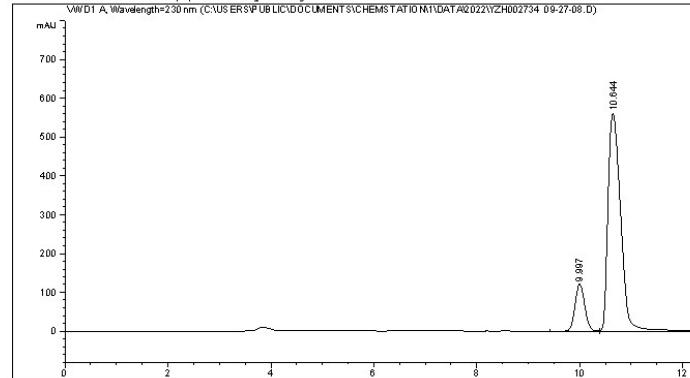
HPLC1260 II 10/17/2023 10:39:09 AM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002734 09-27-08.D
Sample Name: HW-12-56A

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/7/2022 9:27:09 AM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/7/2022 8:39:09 AM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:39:05 AM by SYSTEM
                                                (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

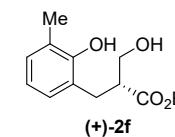


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

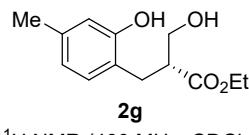
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.997	BV	0.2035	1571.79883	121.00585	14.2329
2	10.644	VB	0.2770	9471.63477	560.87994	85.7671



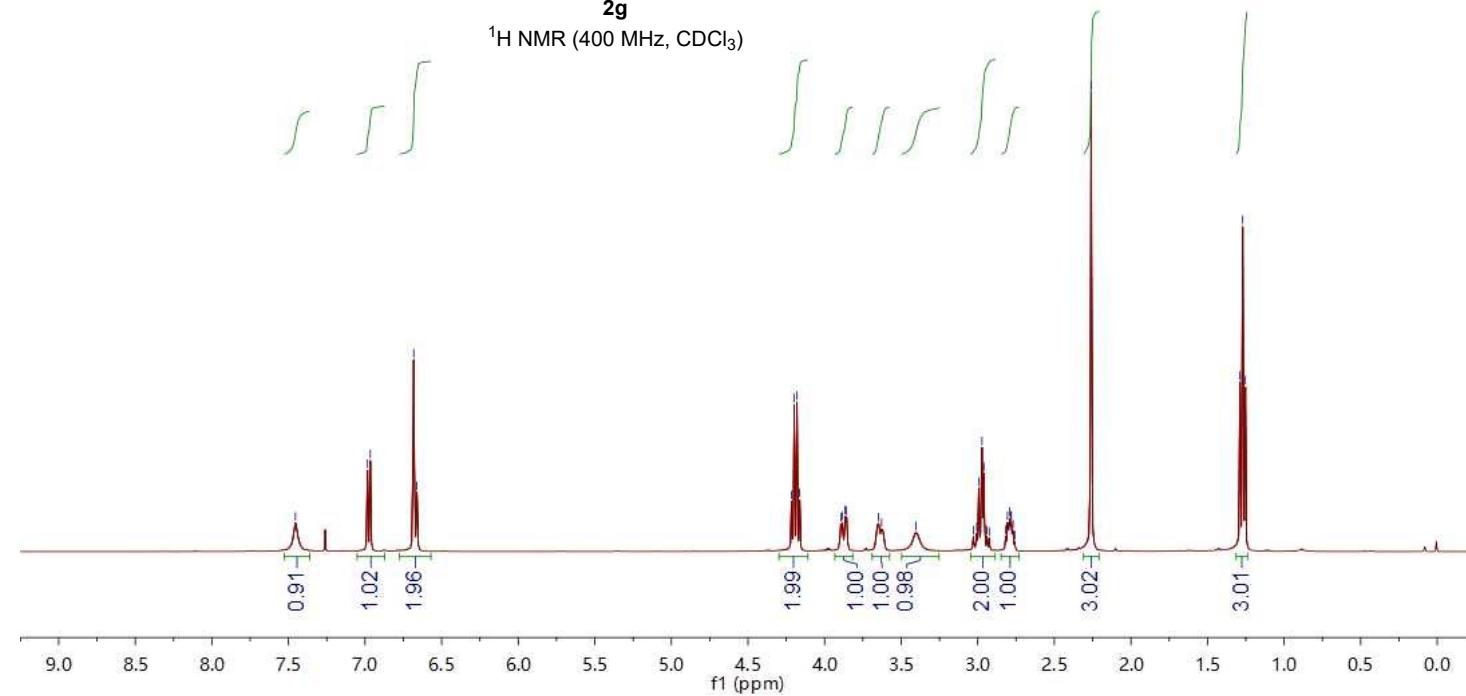
HPLC1260 II 10/17/2023 10:39:44 AM SYSTEM

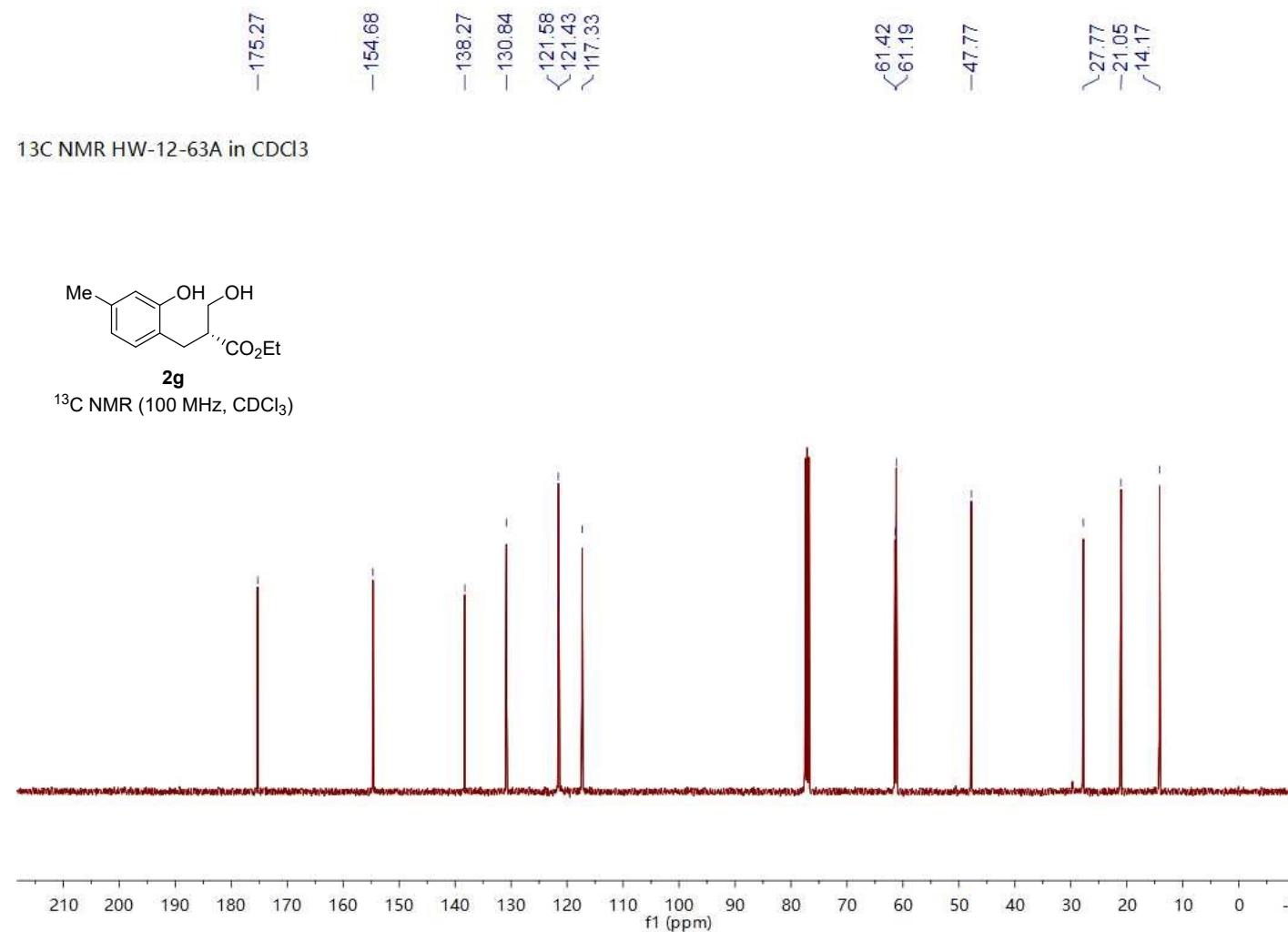
Page 1 of 2

¹H NMR HW-12-63A in CDCl₃



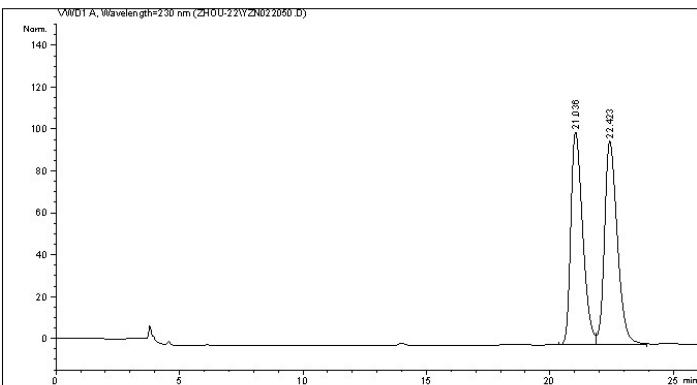
¹H NMR (400 MHz, CDCl₃)





Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022050.D
Sample Name: HW-12-63A+/-

=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 11/15/2022 10:36:57 PM
Acq. Method : C:\CHEM32\1\METHODS\SDDEF_LC.M
Last changed : 11/15/2022 10:36:31 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SDDEF_LC.M
Last changed : 10/17/2023 10:32:22 PM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm

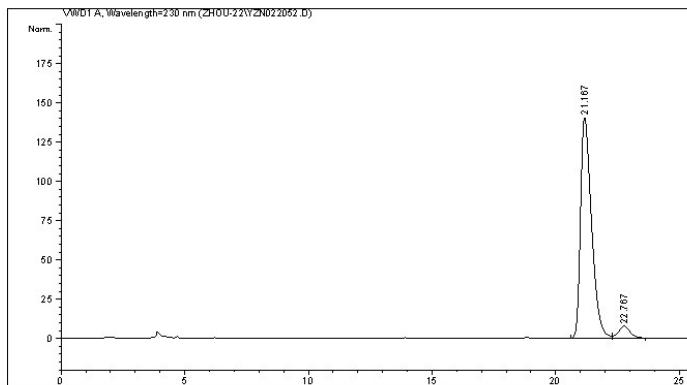


Instrument 1 10/17/2023 10:32:25 PM

Page 1 of 1

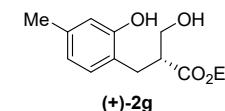
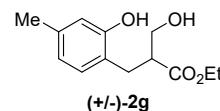
Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022052.D
Sample Name: HW-12-63A

=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 11/15/2022 11:36:05 PM
Acq. Method : C:\CHEM32\1\METHODS\SDDEF_LC.M
Last changed : 11/15/2022 11:34:22 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\SDDEF_LC.M
Last changed : 10/17/2023 10:33:11 PM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm



Instrument 1 10/17/2023 10:33:14 PM

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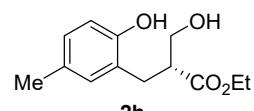


¹H NMR HW-12-52A in CDCl₃

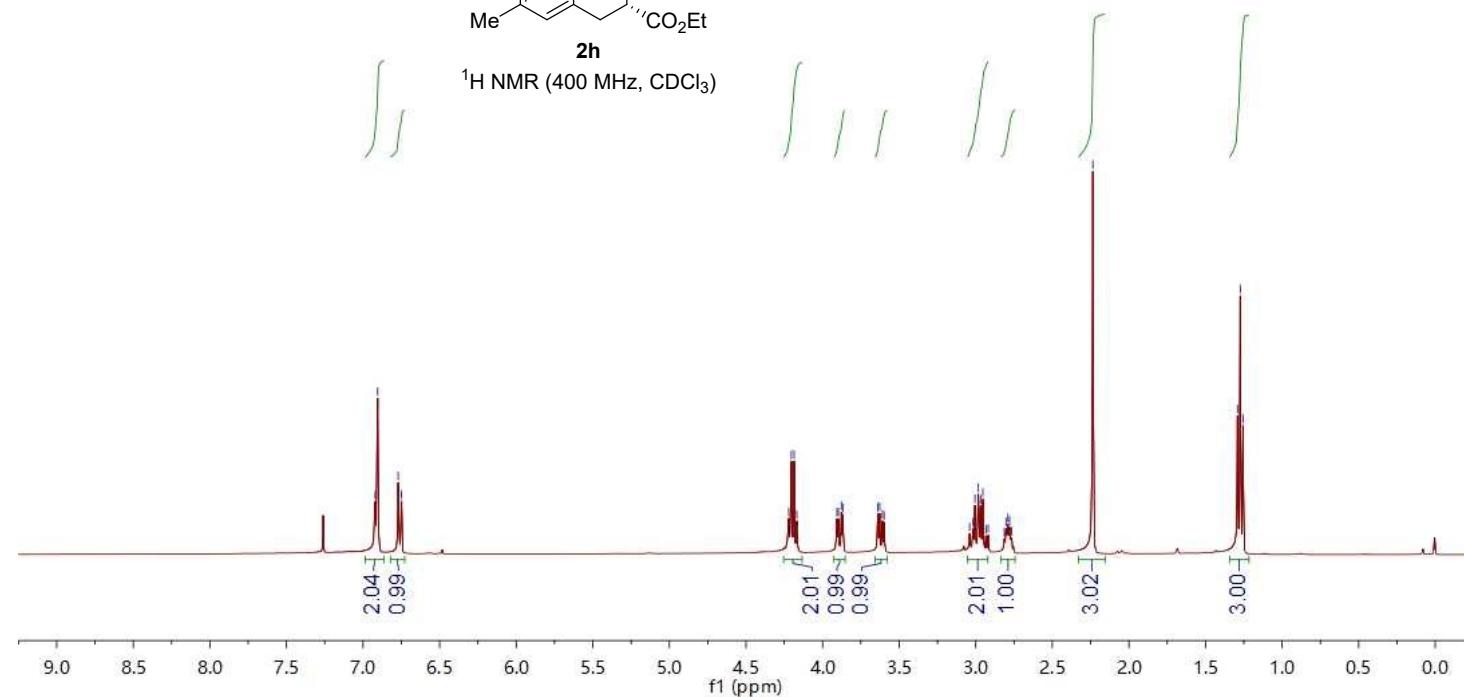
6.9209
6.9045
6.7707
6.7487

4.2207
4.2135
4.2035
4.1857
4.1678
3.9062
3.8968
3.8773
3.8680
3.6385
3.6271
3.6096
3.5983
3.0053
2.9825
2.9685
2.9534
2.7894
2.2774

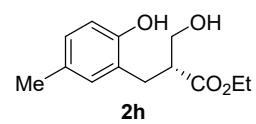
1.2910
1.2731
1.2553



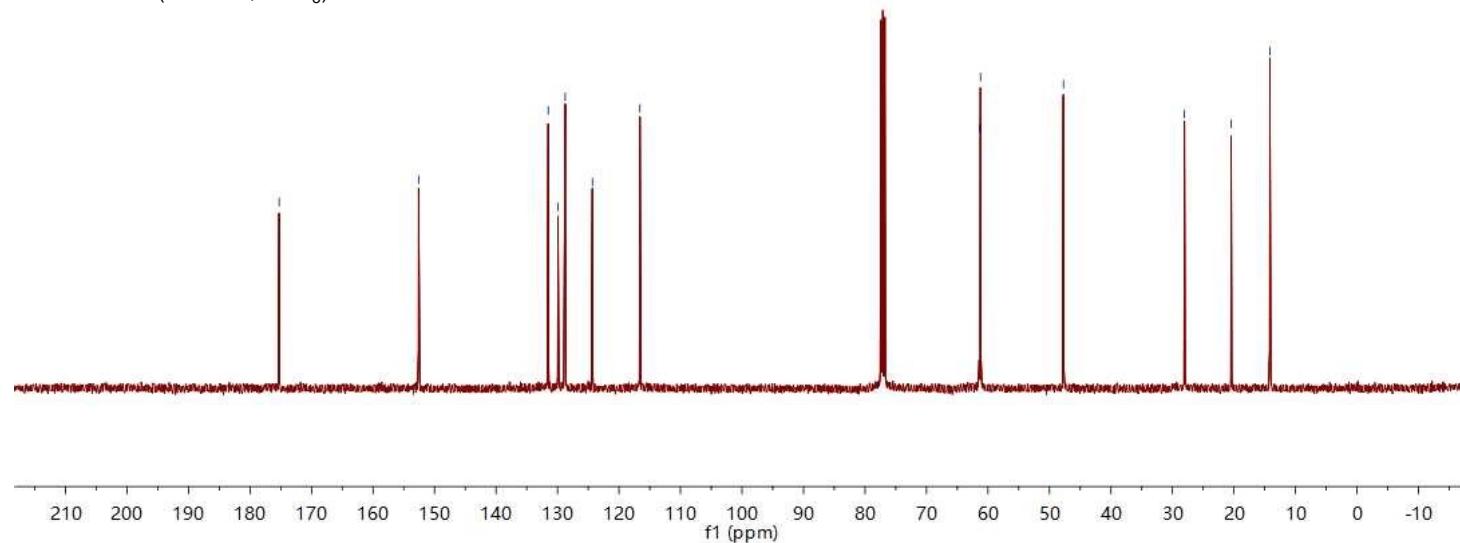
¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-12-52A in CDCl₃



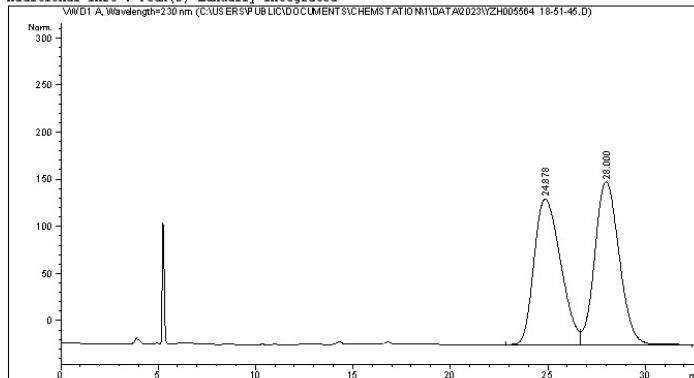
¹³C NMR (100 MHz, CDCl₃)



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005564 18-51-45.D
Sample Name: HW-12-52A+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/28/2023 6:51:45 PM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/28/2023 6:48:19 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 11:05:26 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

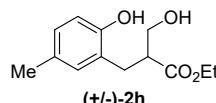


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.878	BV	1.4839	1.05224e4	111.00722	49.4465
2	28.000	VB	1.3391	1.07580e4	123.88742	50.5535



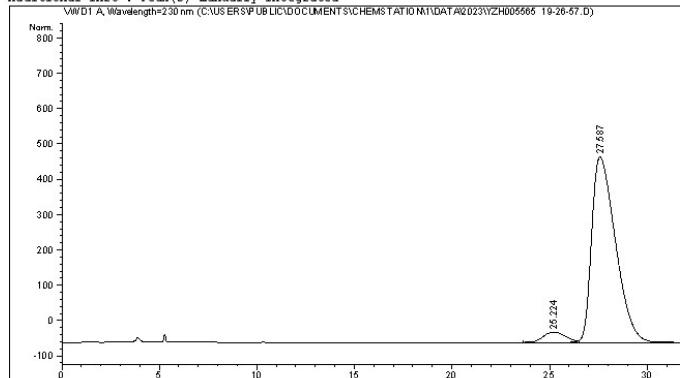
HPLC1260 II 11/29/2023 11:05:30 AM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005565 19-26-57.D
Sample Name: HW-12-52A

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/28/2023 7:26:57 PM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/28/2023 6:48:19 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 11:06:40 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

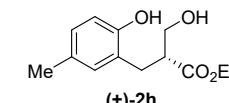


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

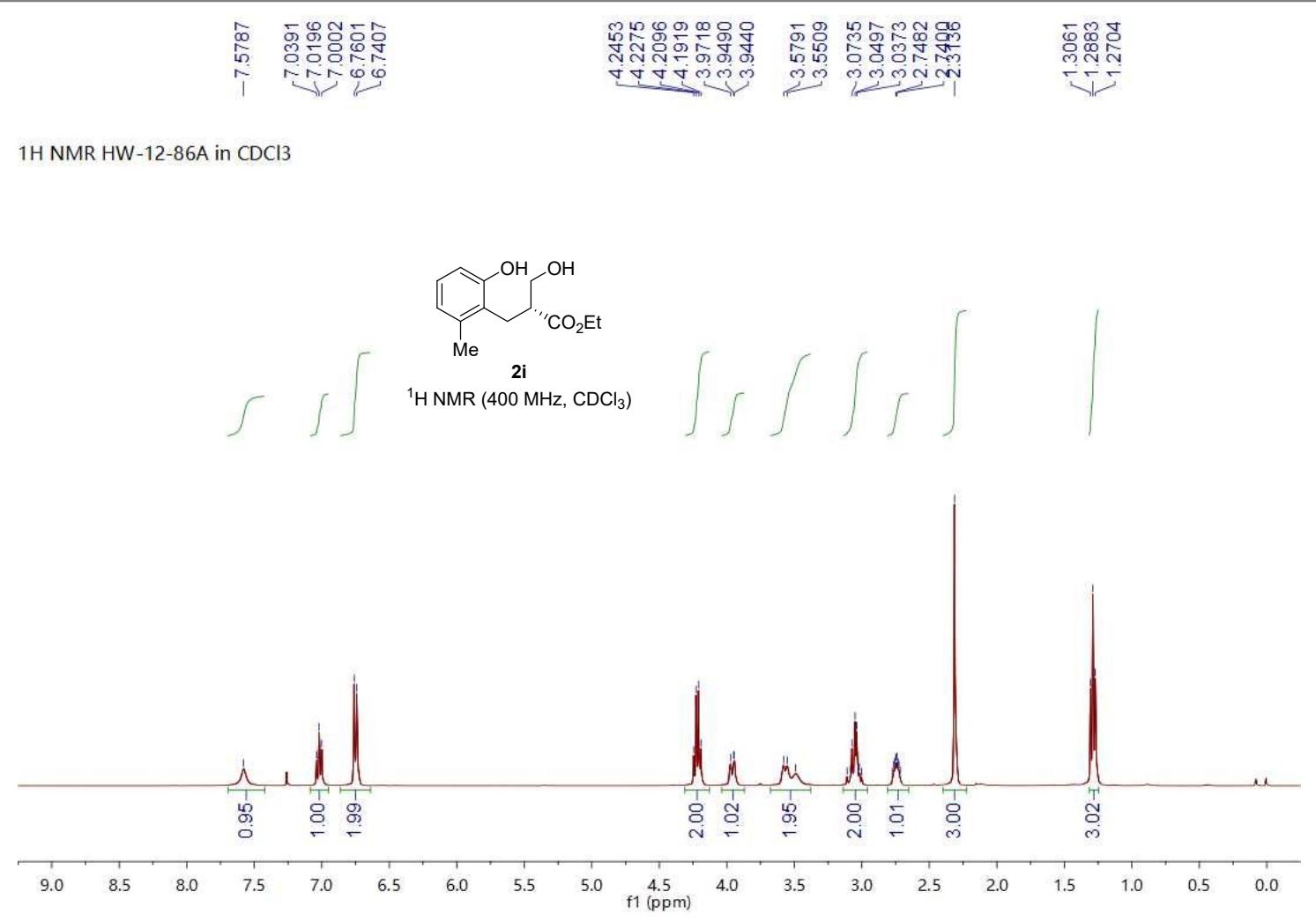
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.224	BV	1.2266	1771.88013	22.62739	4.9326
2	27.587	VB	1.3056	3.41502e4	402.61203	95.0674

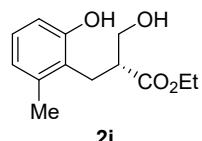


HPLC1260 II 11/29/2023 11:06:46 AM SYSTEM

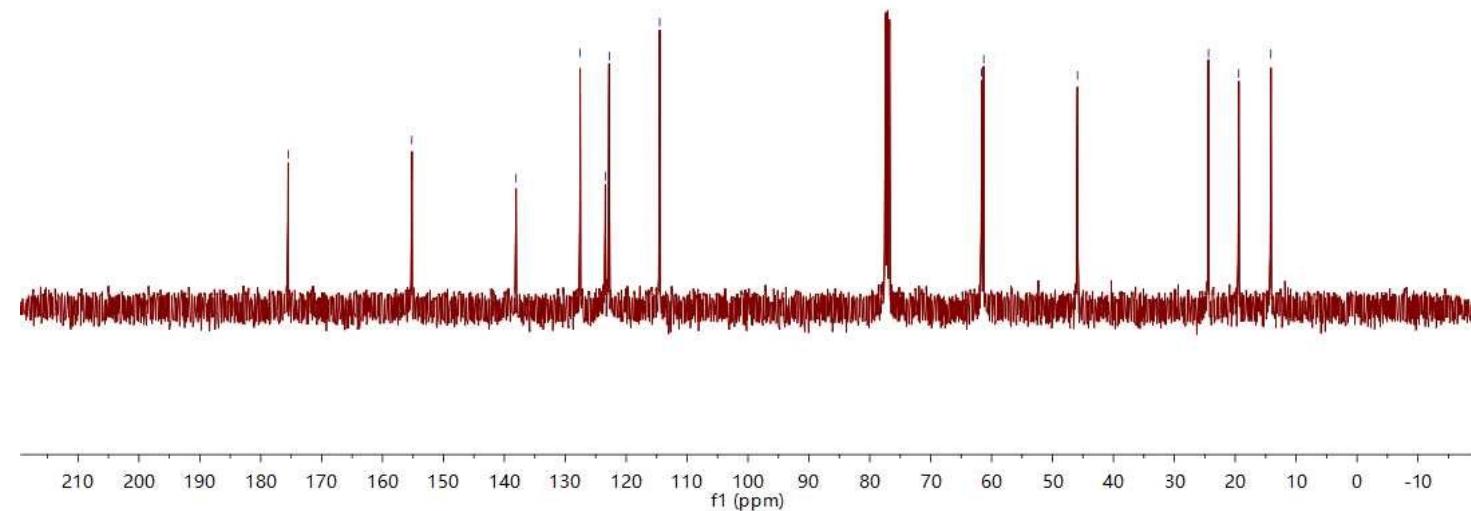
Page 1 of 2



¹³C NMR HW-12-86A in CDCl₃



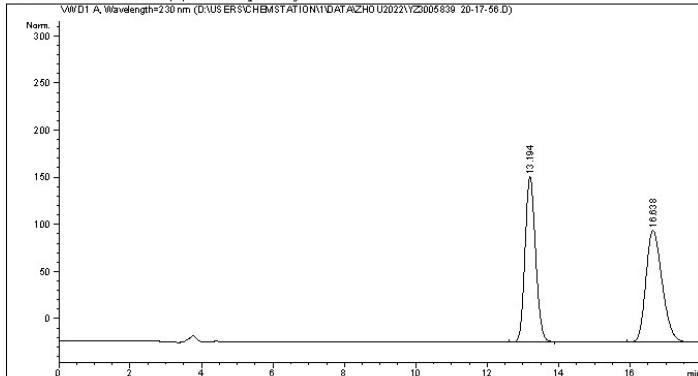
¹³C NMR (100 MHz, CDCl₃)



Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\YZ3005839 20-17-56.D
Sample Name: HW-12-86A+/-

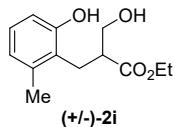
```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 12/6/2022 8:17:56 PM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\lsm-1-58.M
Last changed   : 12/6/2022 8:16:54 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 10/17/2023 10:49:33 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, n-Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



```
=====
Area Percent Report
=====

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```



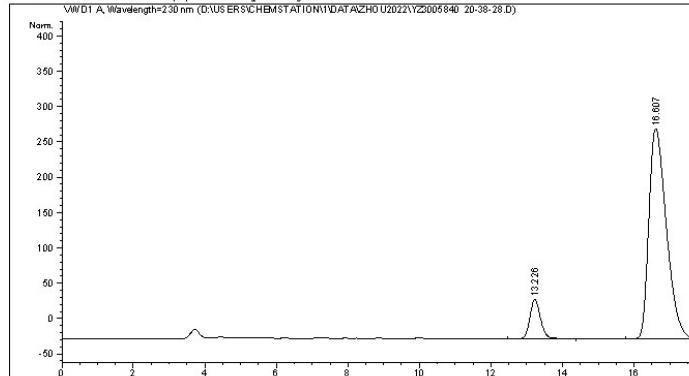
1260II 10/17/2023 10:49:36 AM SYSTEM

Page 1 of 2

Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\YZ3005840 20-38-28.D
Sample Name: HW-12-86A

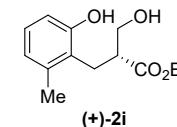
```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 12/6/2022 8:38:28 PM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\lsm-1-58.M
Last changed   : 12/6/2022 8:16:54 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 10/17/2023 10:50:22 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, n-Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



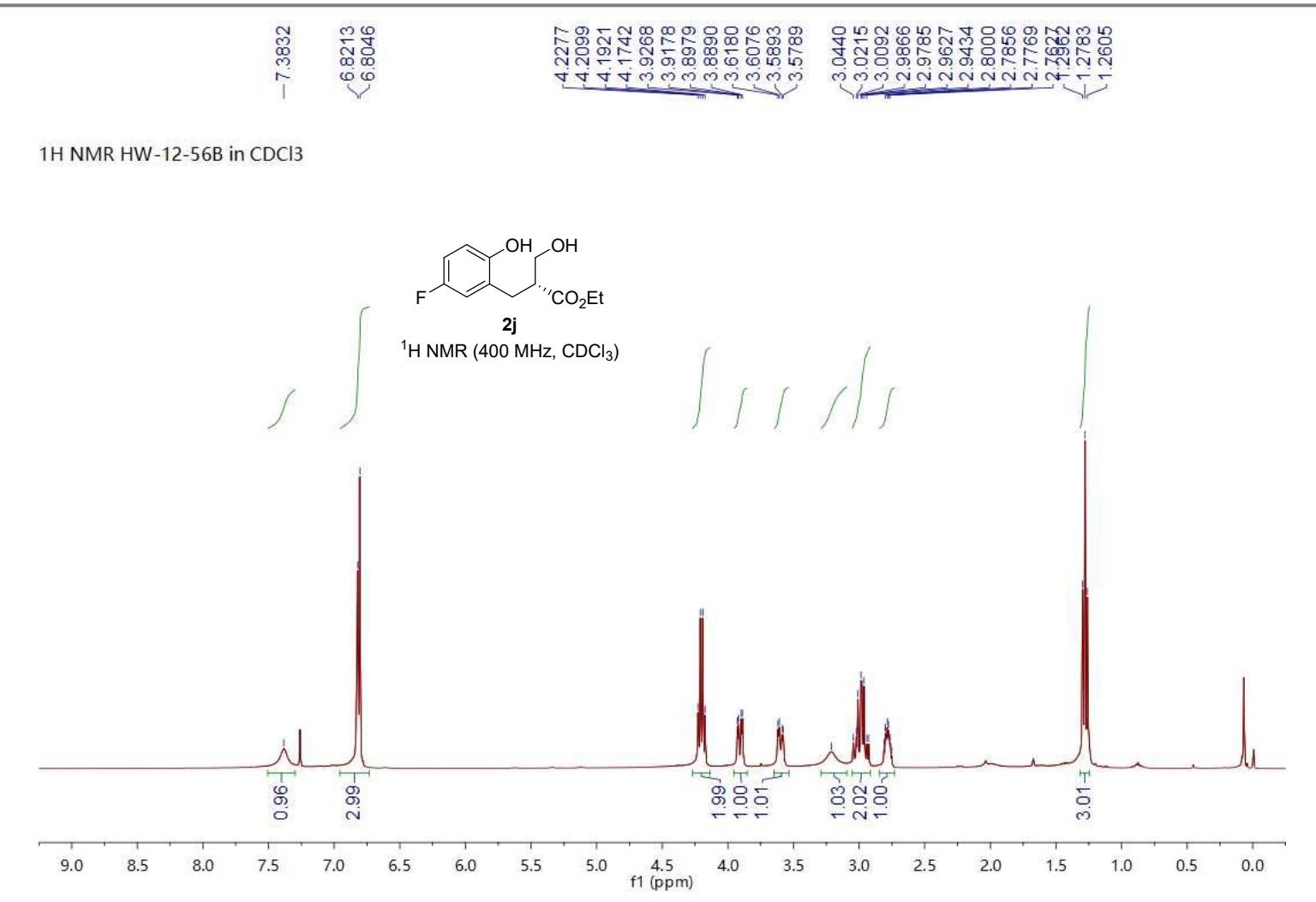
```
=====
Area Percent Report
=====

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

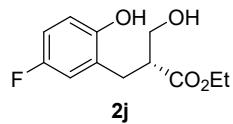


1260II 10/17/2023 10:50:25 AM SYSTEM

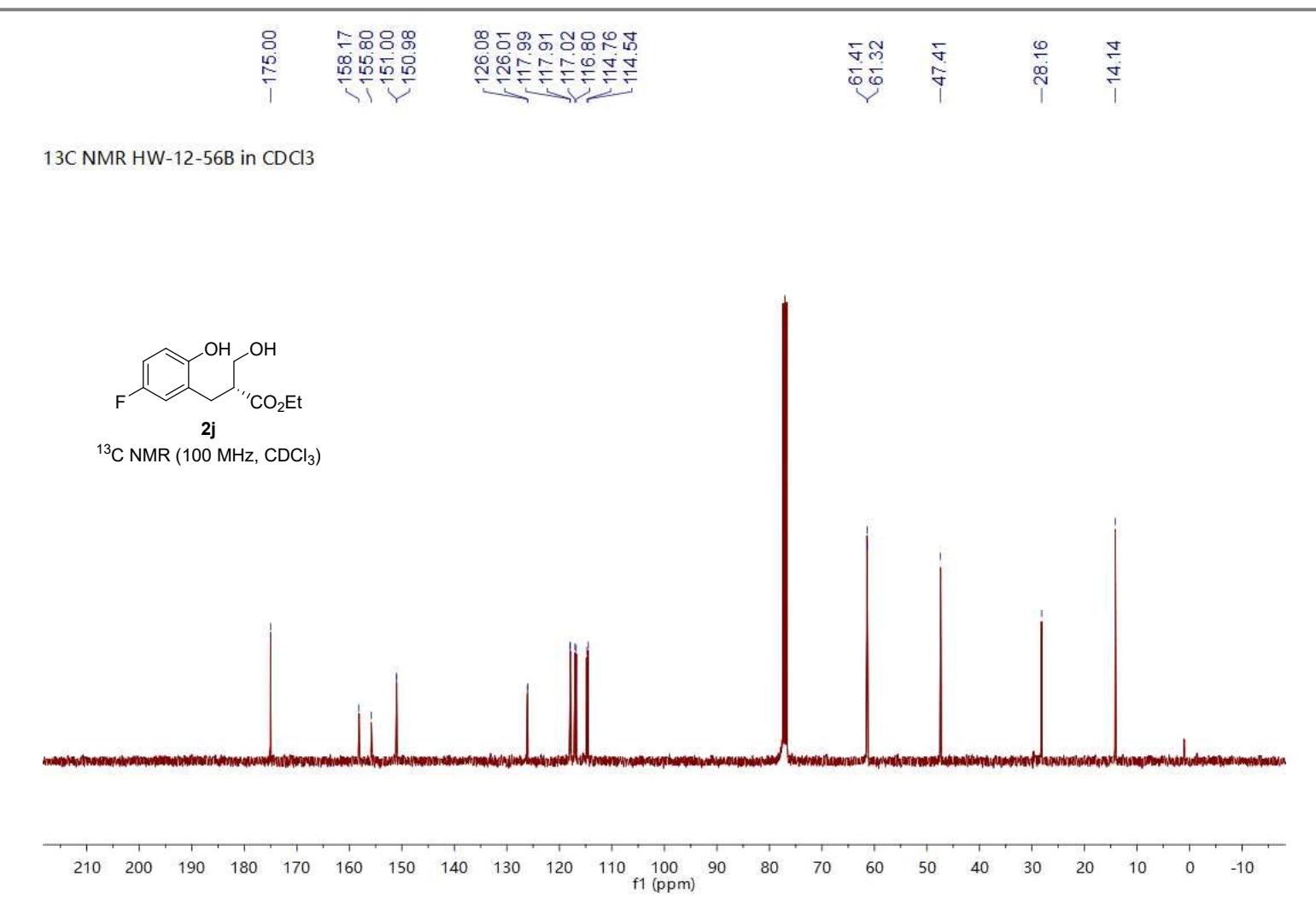
Page 1 of 2



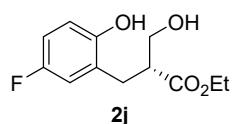
¹³C NMR HW-12-56B in CDCl₃



¹³C NMR (100 MHz, CDCl₃)

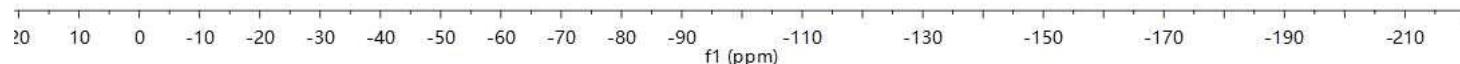


¹⁹F NMR HW-12-56B in CDCl₃



¹⁹F NMR (376 MHz, CDCl₃)

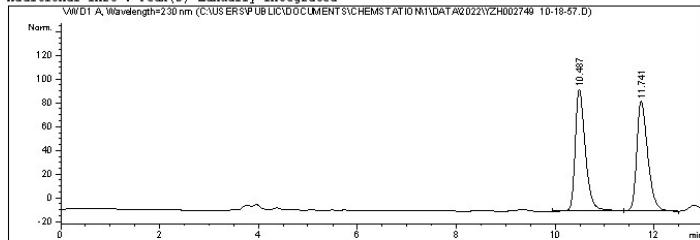
—124.0142



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002749 10-18-57.D
Sample Name: HW-12-56B+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/8/2022 10:18:58 AM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/8/2022 10:03:25 AM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 9:59:58 PM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 90/10, 0.8mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



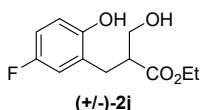
=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime	Type	Width	Area	Height	Area %
#		[min]	[mAU*s]	[mAU]	
1	BB	0.2155	1166.01672	82.20879	50.6514
2	BB	0.2330	1136.02637	74.04032	49.3486

Totals : 2302.04309 156.24911



=====
*** End of Report ***

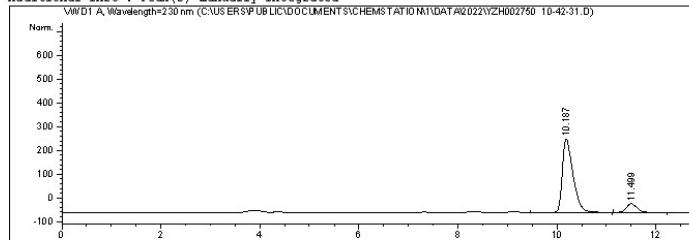
HPLC1260 II 11/29/2023 10:00:02 PM SYSTEM

Page 1 of 1

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002750 10-42-31.D
Sample Name: HW-12-56B

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/8/2022 10:42:32 AM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/8/2022 10:03:25 AM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 9:50:01 PM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 90/10, 0.8mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



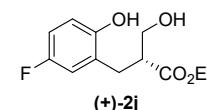
=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime	Type	Width	Area	Height	Area %
#		[min]	[mAU*s]	[mAU]	
1	VB R	0.2271	2805.53760	186.94838	89.4030
2	BB	0.2315	332.54401	21.98408	10.5970

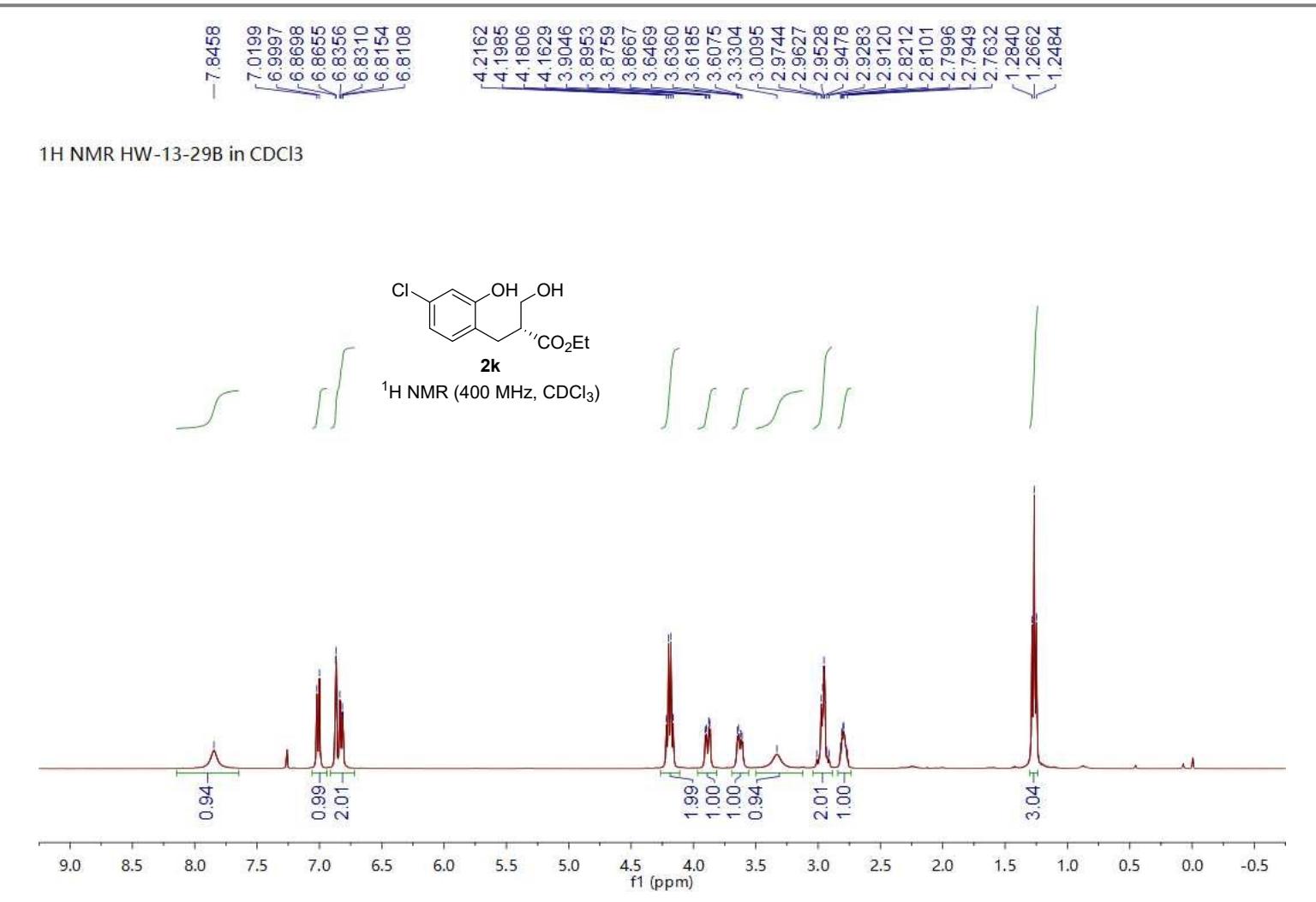
Totals : 3138.08160 208.93246

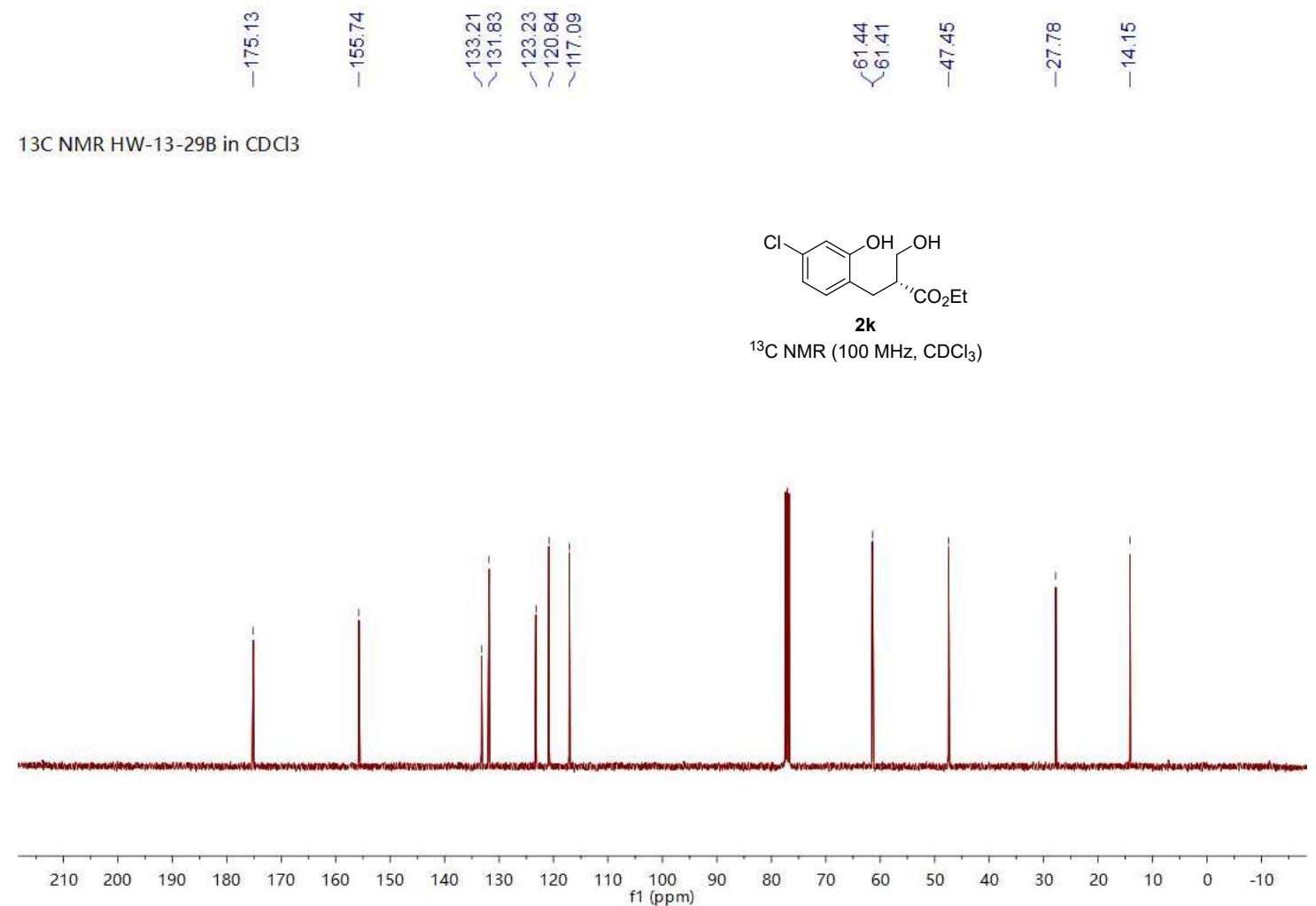


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*** End of Report ***

HPLC1260 II 11/29/2023 9:50:05 PM SYSTEM

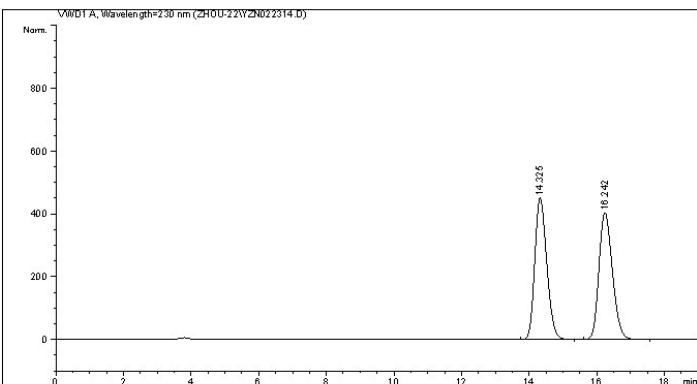
Page 1 of 1





Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022314.D
Sample Name: HW-13-29B+/-

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 1/4/2023 3:46:49 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 1/4/2023 3:44:39 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 11/30/2023 8:56:32 AM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



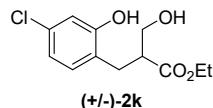
=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	14.325	BB	0.3834	1.1051e4	448.83185	49.8610	
2	16.242	BB	0.4294	1.1113e4	403.14838	50.1390	

Totals : 2.21655e4 851.98022



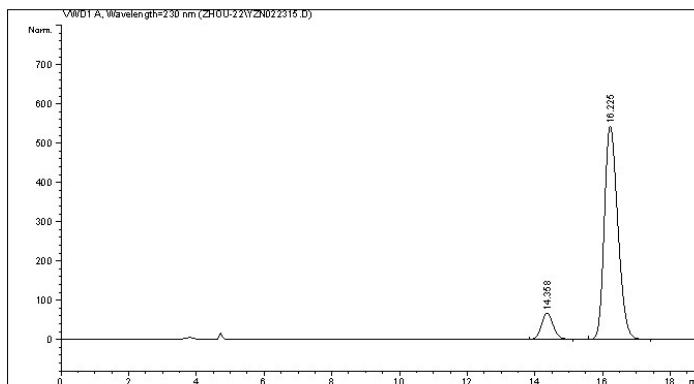
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*** End of Report ***
=====

Instrument 1 11/30/2023 8:56:36 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022315.D
Sample Name: HW-13-29B

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 1/4/2023 4:16:05 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 1/4/2023 4:15:23 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:25:01 PM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

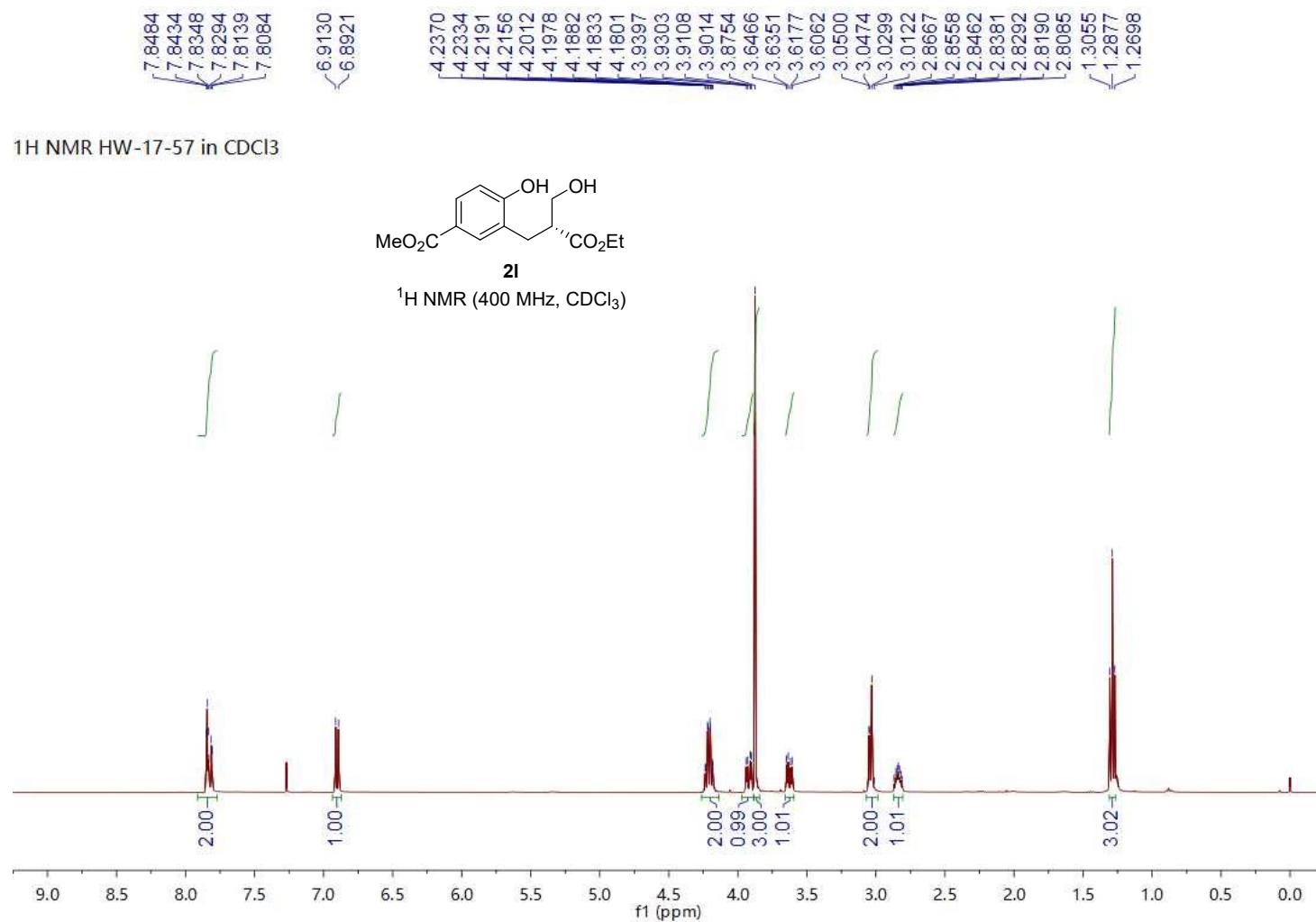
Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	14.358	BB	0.3654	1571.00635	66.62236	9.5166	
2	16.225	BB	0.4290	1.49370e4	542.43024	90.4834	

Totals : 1.65081e4 609.05260

=====
*** End of Report ***
=====

Instrument 1 10/17/2023 10:25:04 PM

Page 1 of 1



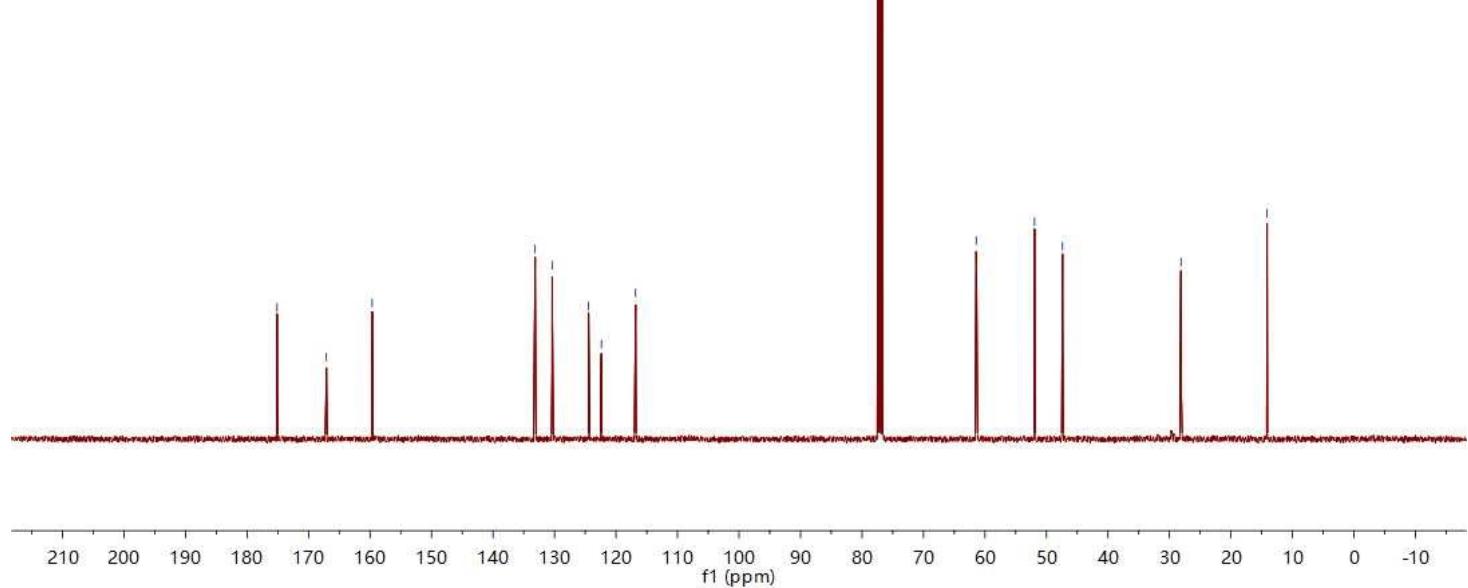
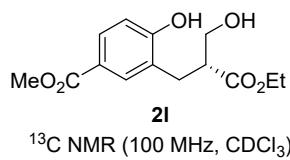
\ 175.12
- 167.09
/ 159.66

- 133.15
- 130.39
- 124.45
- 122.38
- 116.81

< 61.43
< 61.35
- 51.95
- 47.39

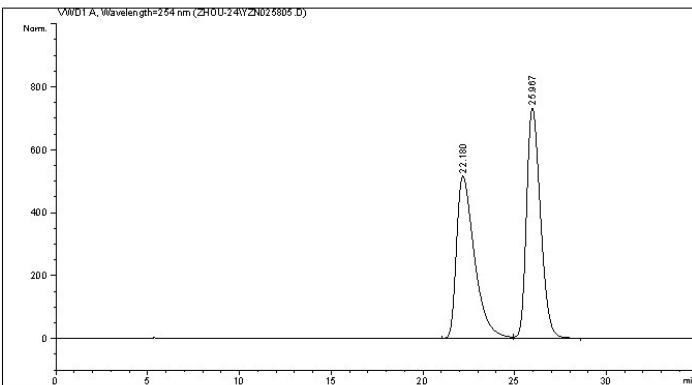
- 28.13
- 14.12

¹³C NMR HW-17-57 in CDCl₃



Data File C:\CHEM32\1\DATA\ZHOU-24\YZN025805.D
Sample Name: HW-17-57+/-

=====
Acq. Operator :
Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/9/2024 8:51:28 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/9/2024 8:50:44 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/9/2024 9:31:41 AM
(modified after loading)
Sample Info : AS-H, Hexane/iPrOH = 90/10, 0.8 mL/ min, 30 oC, 254 nm.

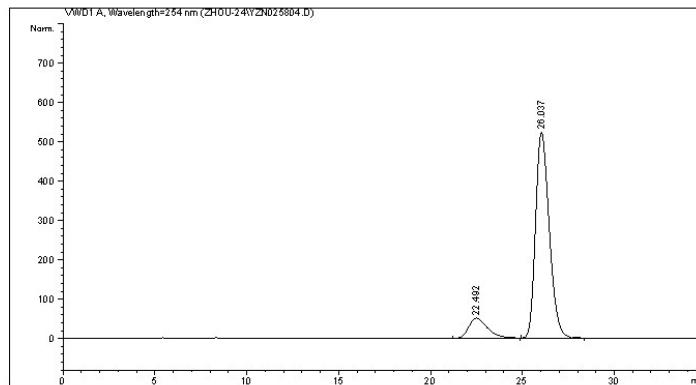


Instrument 1 5/9/2024 9:31:44 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-24\YZN025804.D
Sample Name: HW-17-57

=====
Acq. Operator :
Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/9/2024 8:42:55 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/9/2024 8:06:52 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/9/2024 9:31:16 AM
(modified after loading)
Sample Info : AS-H, Hexane/iPrOH = 90/10, 0.8 mL/ min, 30 oC, 254 nm.



Instrument 1 5/9/2024 9:31:20 AM

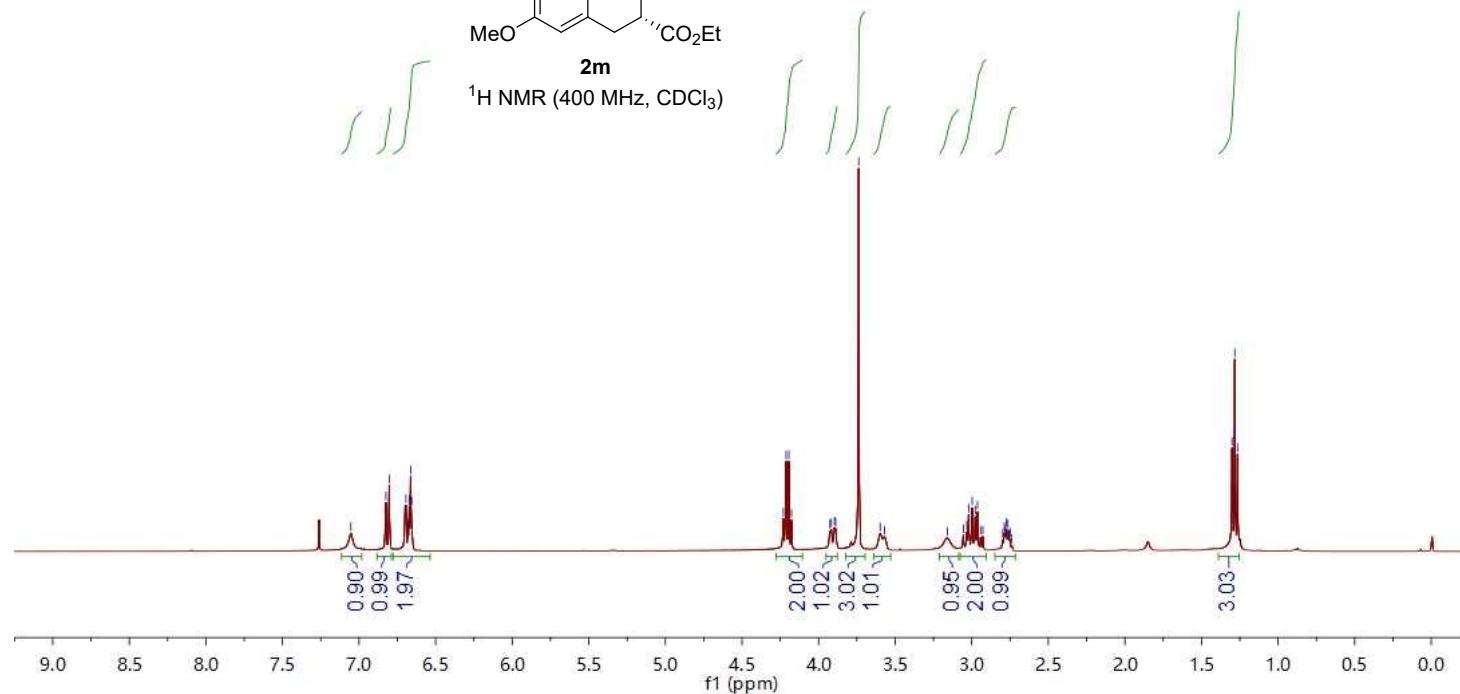
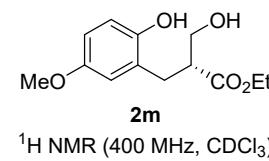
Page 1 of 1

¹H NMR HW-12-63B in CDCl₃

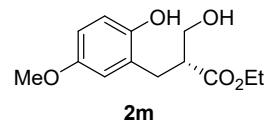
7.0533
6.8247
6.8033
6.7013
6.6938
6.6797
6.6722
6.6633
6.6560

4.2298
4.2119
4.1941
4.1763
3.9252
3.9169
3.8965
3.8886
3.7380
3.5978
3.5705

3.0551
3.0318
3.0204
2.9972
2.9774
2.9623
2.9276
2.7872
2.7820
2.7730
2.4664
1.2828
1.2650



¹³C NMR HW-12-63B in CDCl₃



¹³C NMR (100 MHz, CDCl₃)

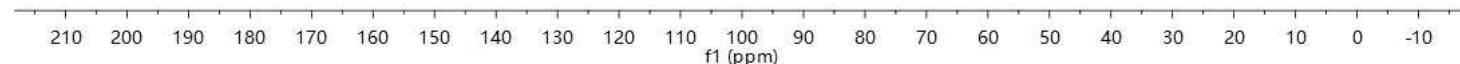
-175.19

-153.67
-148.85

-125.59
-117.84
-115.99
-113.47

\~61.33
\~61.29
\~55.71
-47.60

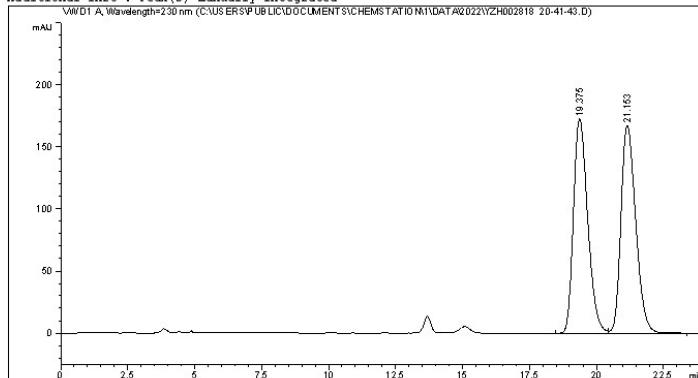
-28.30
-14.18



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002818 20-41-43.D
Sample Name: HW-12-63B+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/14/2022 8:41:43 PM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/14/2022 8:05:07 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:42:41 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

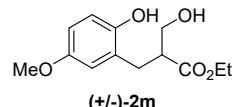


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.375	BV	0.5713	6360.90137	172.46037	49.8071
2	21.153	VB	0.5951	6410.17627	167.28079	50.1929



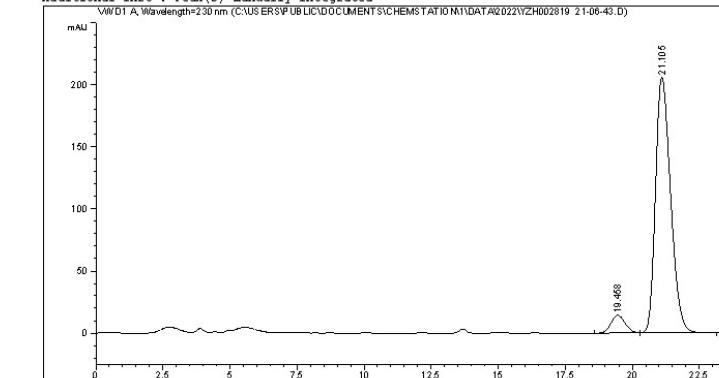
HPLC1260 II 10/17/2023 10:42:46 AM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH002819 21-06-43.D
Sample Name: HW-12-63B

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/14/2022 9:06:43 PM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/14/2022 8:05:07 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:42:41 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

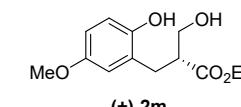


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.458	BB	0.5670	513.02692	14.18275	6.0063
2	21.105	BB	0.6060	8028.38330	205.41820	93.9937

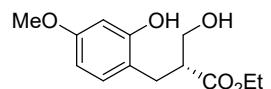


HPLC1260 II 10/17/2023 10:43:23 AM SYSTEM

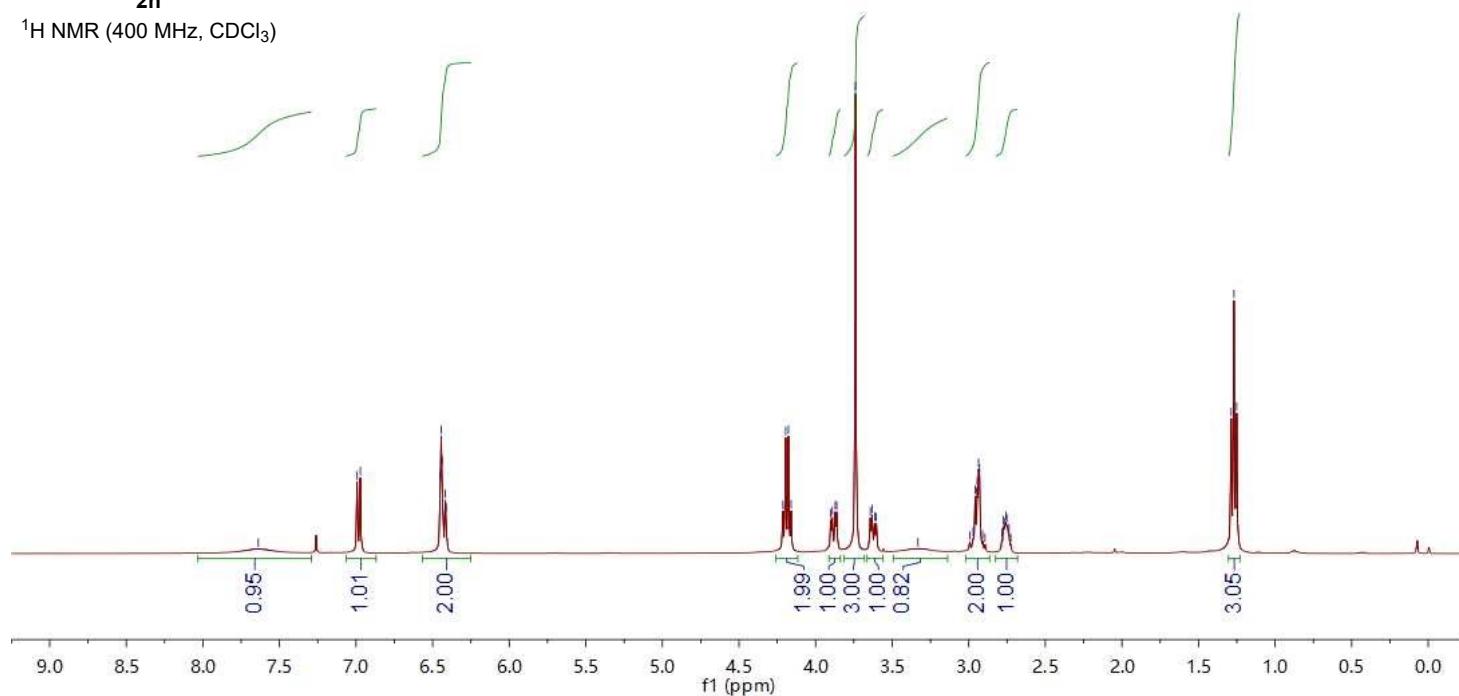
Page 1 of 2

6.9928
 6.9724
 6.4493
 6.4440
 6.4384
 6.4175
 6.4114
 -7.6382

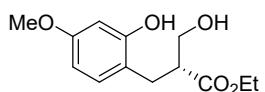
¹H NMR HW-12-86B in CDCl₃



¹H NMR (400 MHz, CDCl₃)

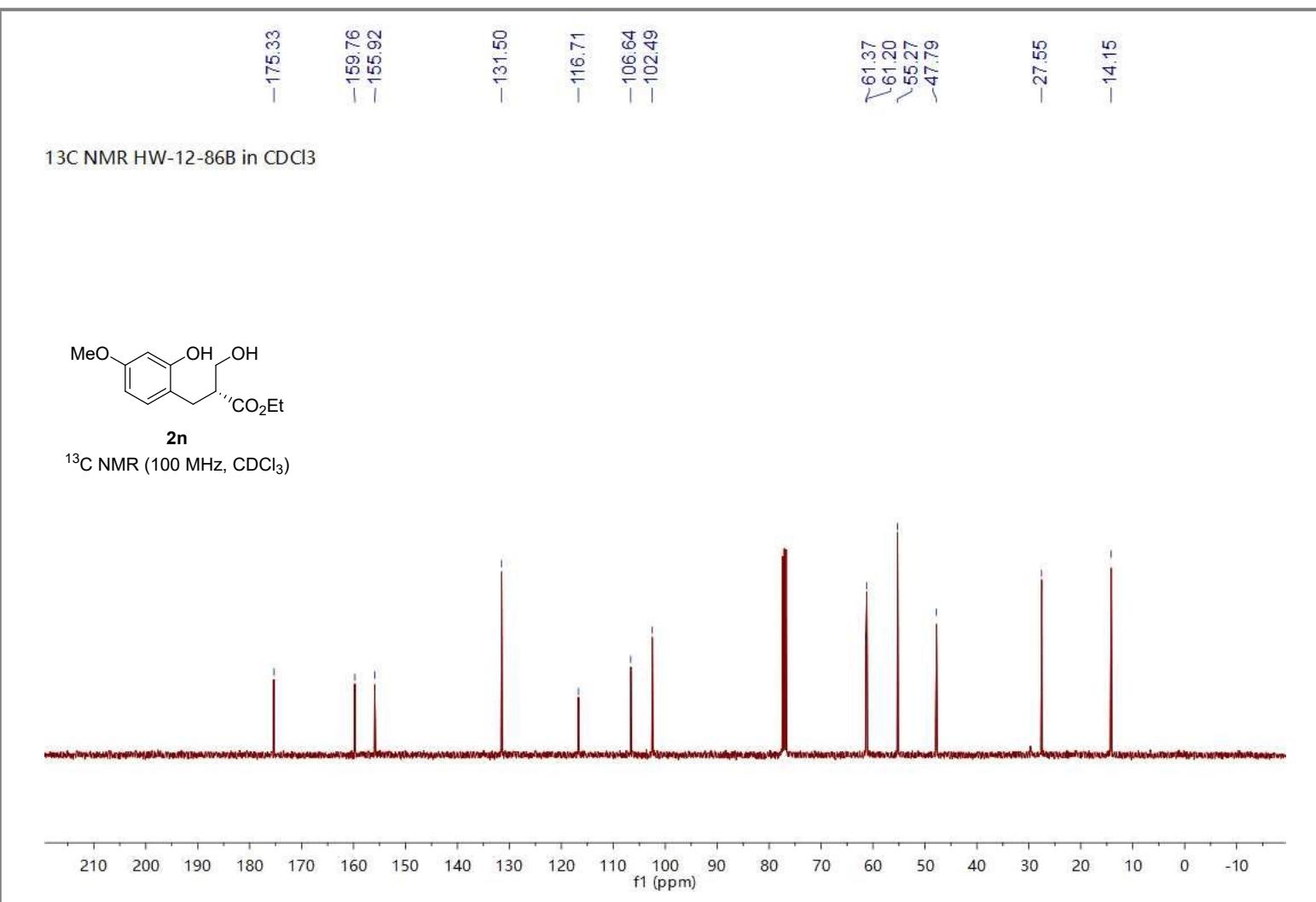


¹³C NMR HW-12-86B in CDCl₃



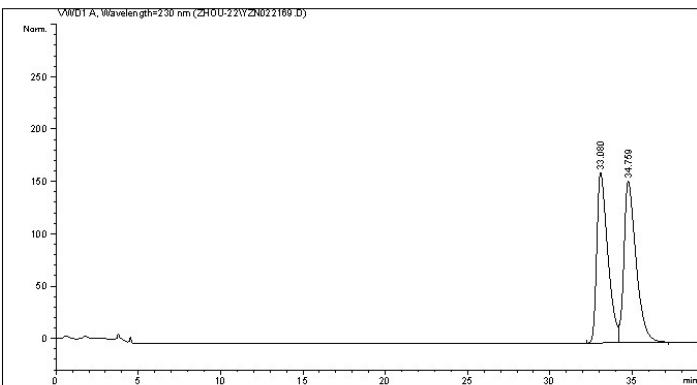
2n

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022169.D
Sample Name: HW-12-86B+/-

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 12/5/2022 9:44:16 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/5/2022 9:43:45 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:35:13 PM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



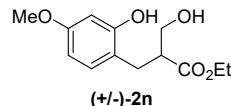
```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height *s	[mAU]	Area %
1	33.080	BV	0.6963	7529.39307	162.53297	48.7565	
2	34.759	BV	0.7740	7913.44580	154.42337	51.2435	

Totals : 1.54428e4 316.95634



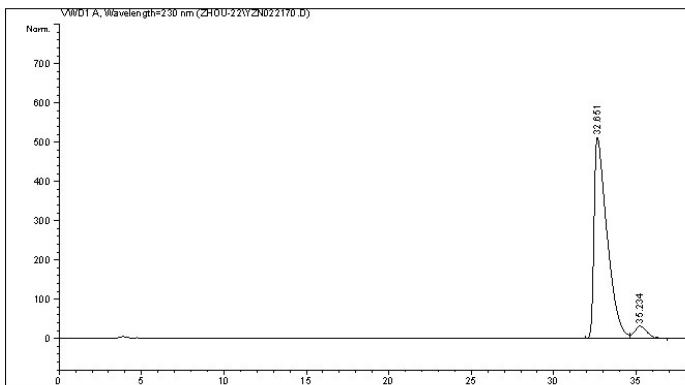
*** End of Report ***

Instrument 1 10/17/2023 10:36:31 PM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022170.D
Sample Name: HW-12-86

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 12/5/2022 10:24:58 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/5/2022 10:24:08 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:39:39 PM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

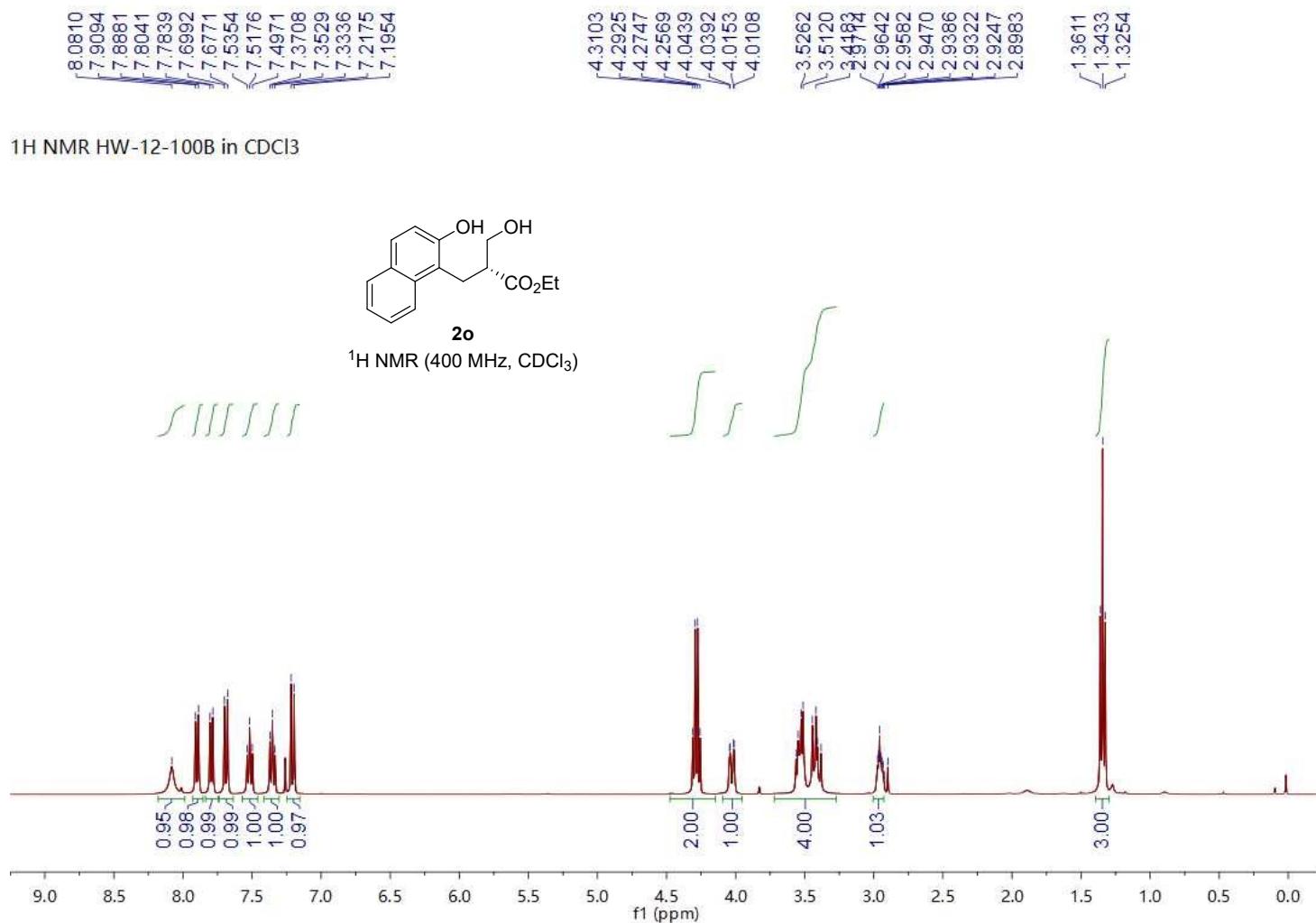
Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height *s	[mAU]	Area %
1	32.651	BV	0.7691	2.80348e4	512.62573	94.5243	
2	35.234	BV	0.7861	1624.01208	31.21306	5.4757	

Totals : 2.96588e4 543.83879

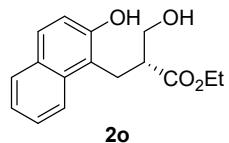
*** End of Report ***

Instrument 1 10/17/2023 10:39:42 PM

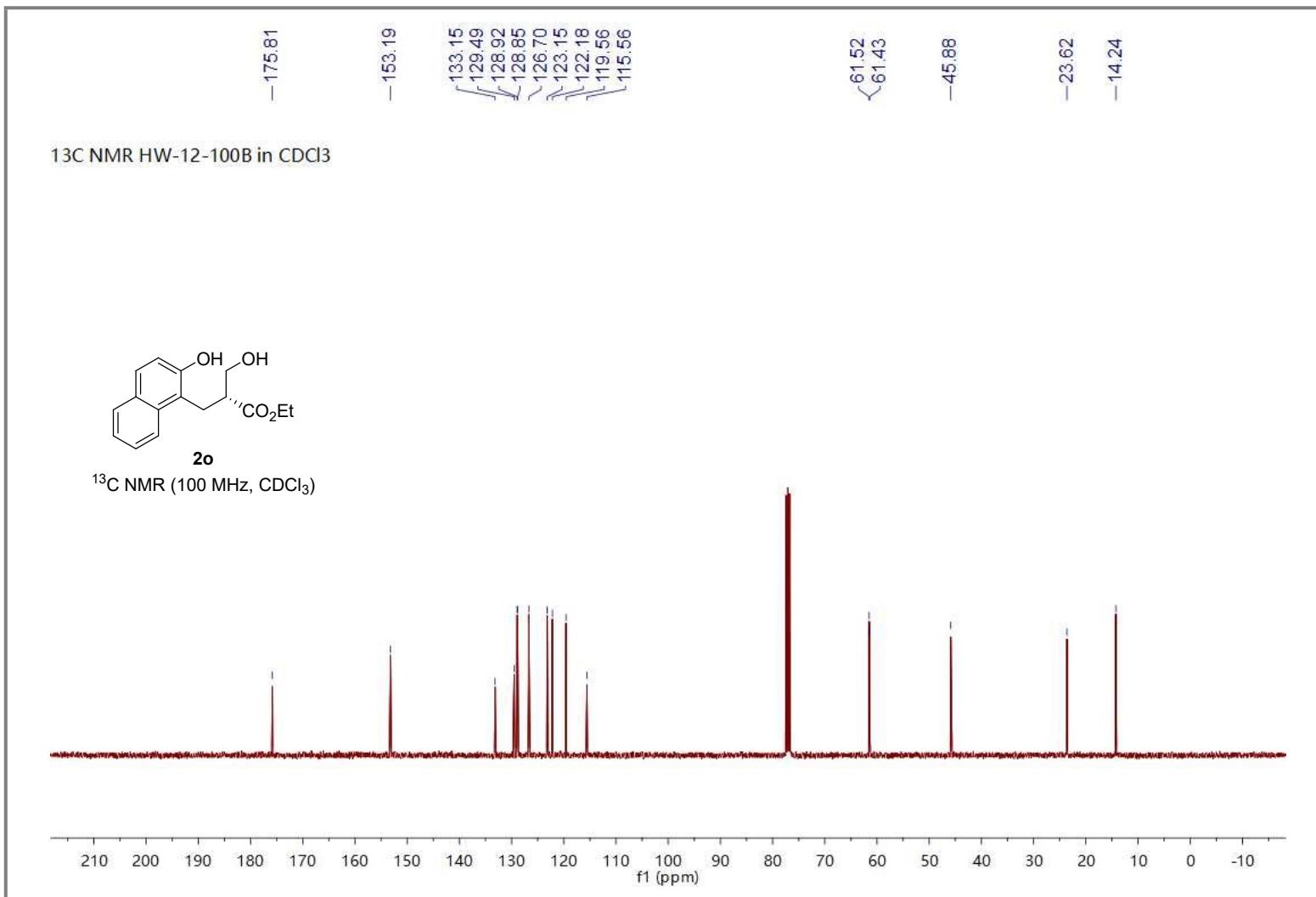
Page 1 of 1



¹³C NMR HW-12-100B in CDCl₃



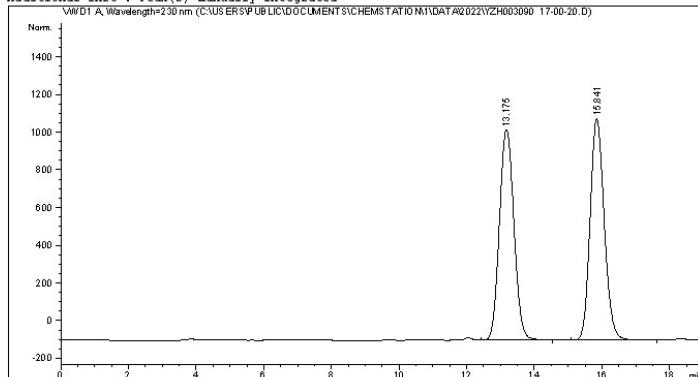
¹³C NMR (100 MHz, CDCl₃)



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH003090 17-00-20.D
Sample Name: HW-12-100B+-

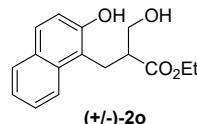
```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 12/20/2022 5:00:20 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 12/20/2022 3:36:44 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:56:22 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```



Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.175	BB	0.4682	2.73494e4	918.88074	49.3684
2	15.841	BB	0.4508	2.80491e4	965.84302	50.6316

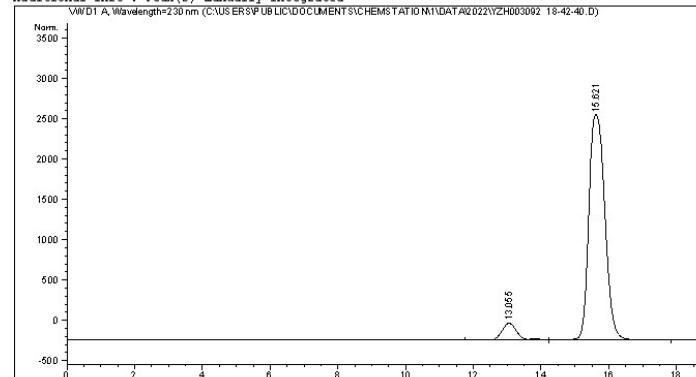
HPLC1260 II 10/17/2023 10:56:26 AM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YZH003092 18-42-40.D
Sample Name: HW-12-100B

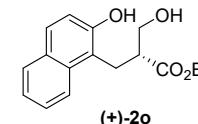
```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 12/20/2022 6:42:40 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 12/20/2022 6:41:56 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/17/2023 10:57:18 AM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```



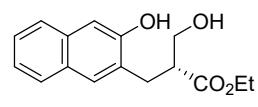
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.055	VV R	0.4646	5408.42383	172.75548	6.6842
2	15.621	VB	0.5206	7.55050e4	2302.19604	93.3158

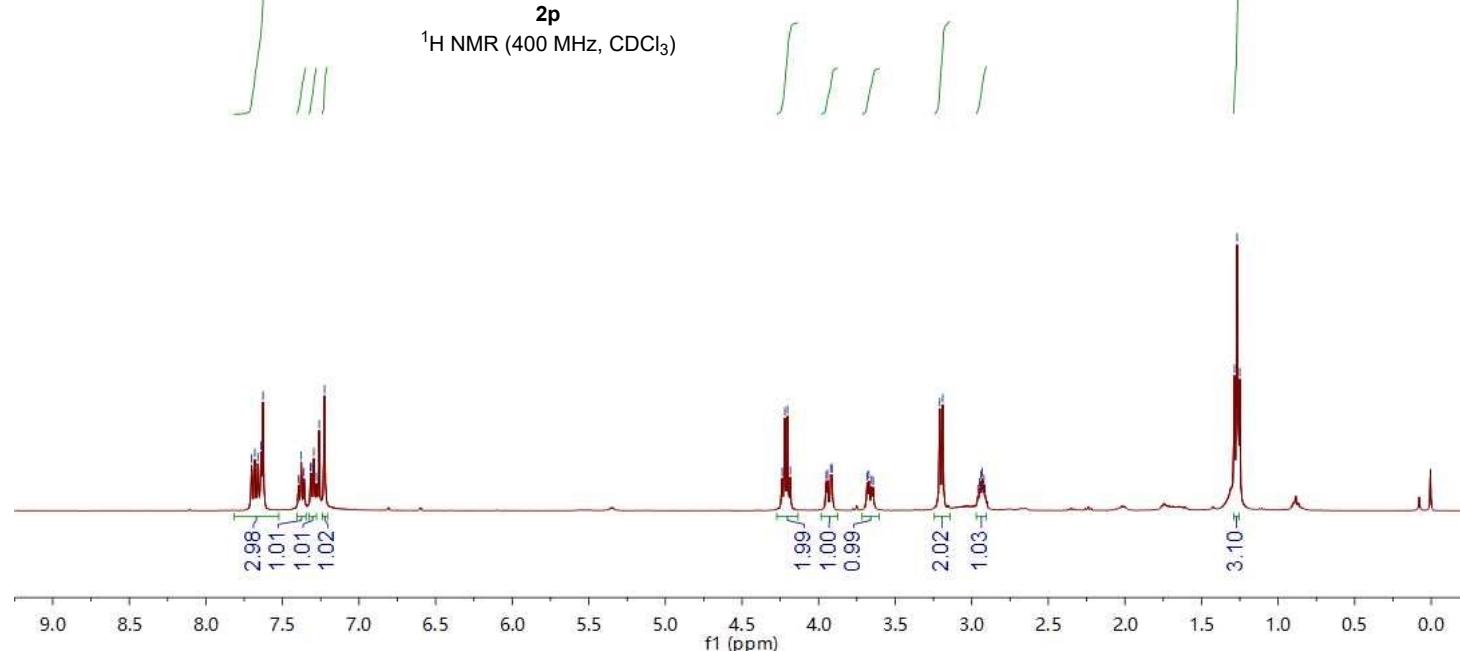
HPLC1260 II 10/17/2023 10:57:29 AM SYSTEM

Page 1 of 2

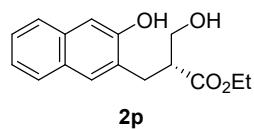
¹H NMR HW-13-37 in CDCl₃



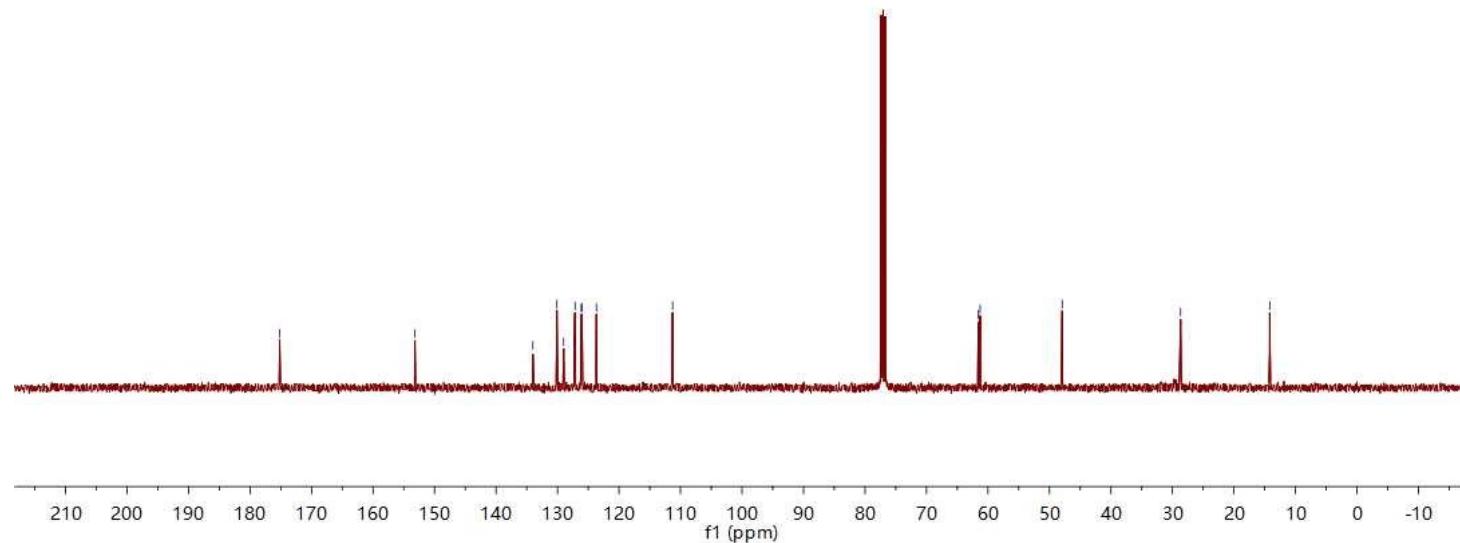
2p
¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-13-37 in CDCl₃



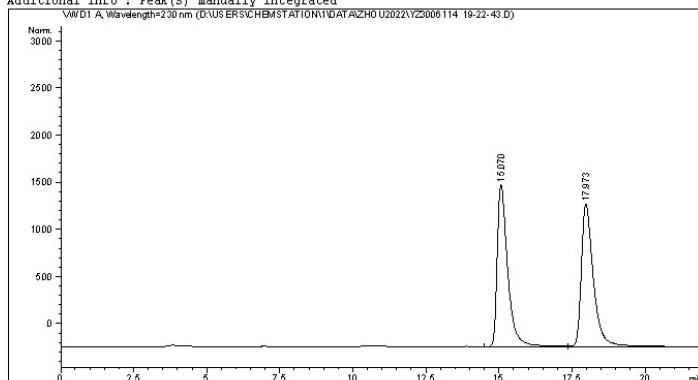
¹³C NMR (100 MHz, CDCl₃)



Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\Y23006114 19-22-43.D
Sample Name: HW-13-37+-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 1/9/2023 7:22:43 PM    Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 1/9/2023 7:07:57 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 10/17/2023 10:51:55 AM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, n-Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



=====

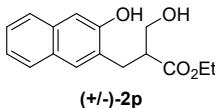
Area Percent Report

=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.070	BV	0.3706	3.05775e4	1231.83813	49.7197
2	17.973	VB	0.4277	3.09223e4	1084.37146	50.2803



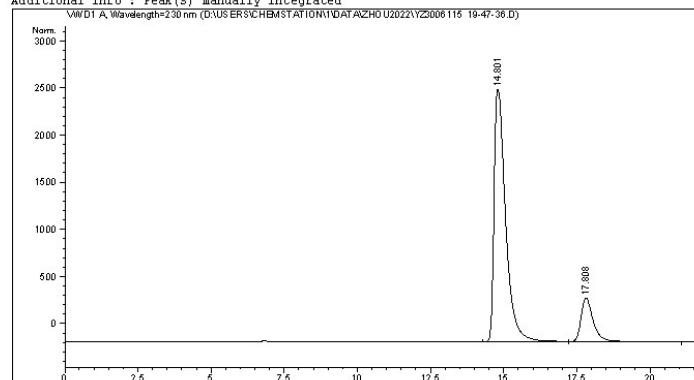
1260II 10/17/2023 10:52:25 AM SYSTEM

Page 1 of 2

Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2022\Y23006115 19-47-36.D
Sample Name: HW-13-37+-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 1/9/2023 7:47:36 PM    Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 1/9/2023 7:07:57 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 10/17/2023 10:51:55 AM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, n-Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



=====

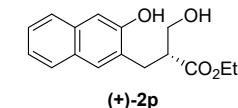
Area Percent Report

=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs

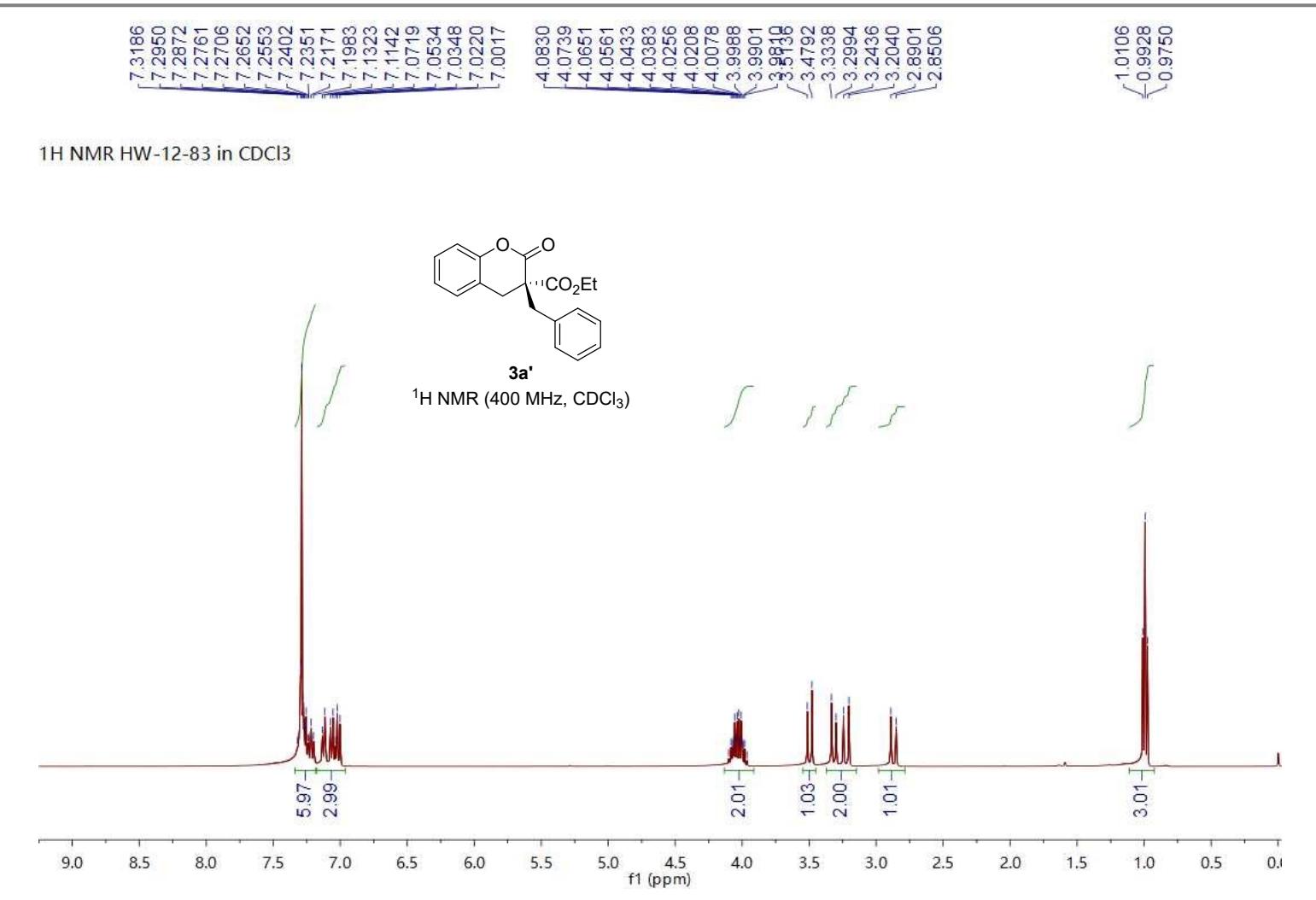
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.801	BV	0.4165	6.33097e4	2297.26587	84.3860
2	17.808	VB	0.4372	1.17143e4	400.56366	15.6140

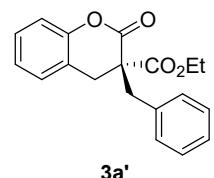


1260II 10/17/2023 10:52:00 AM SYSTEM

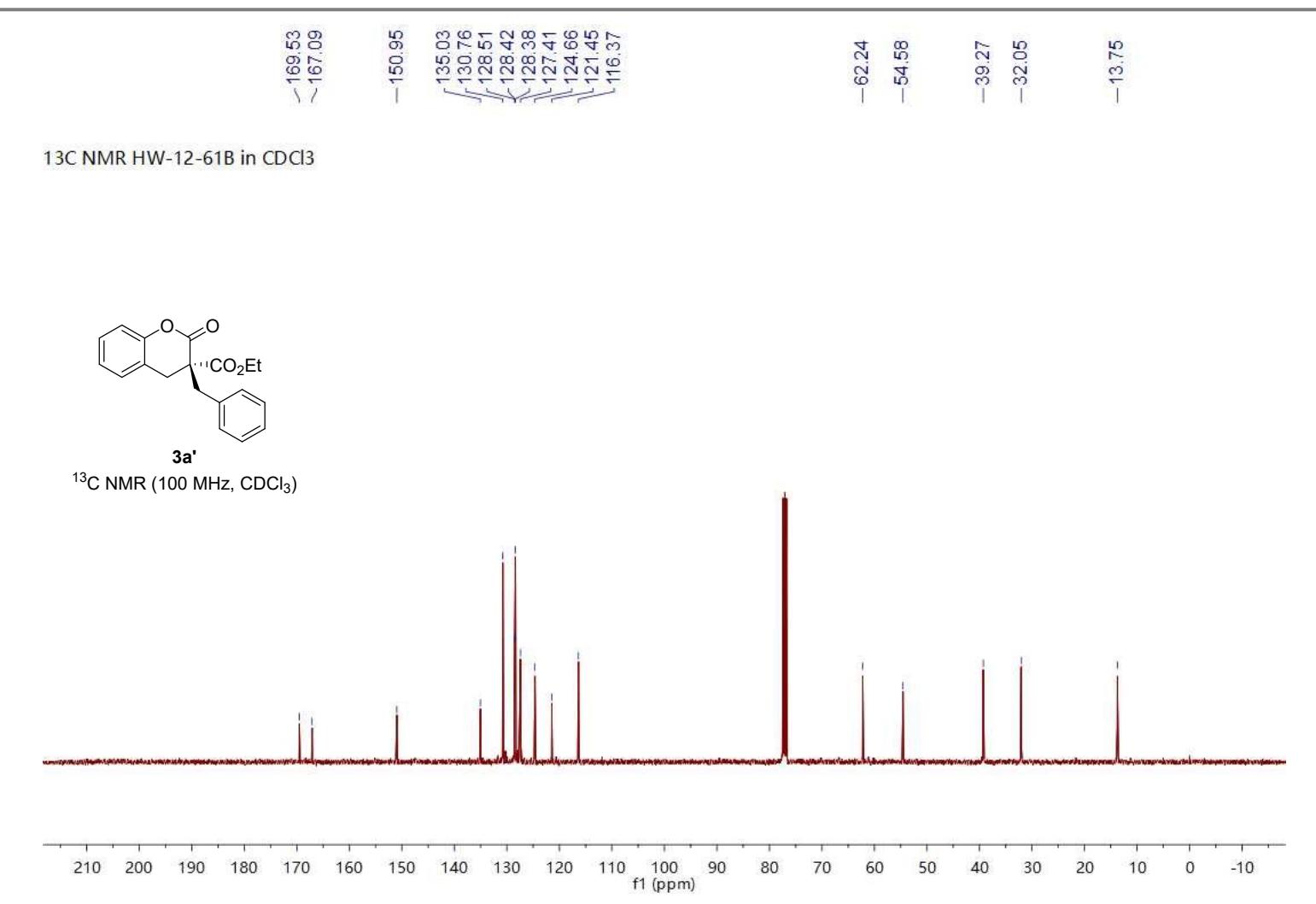
Page 1 of 2



¹³C NMR HW-12-61B in CDCl₃



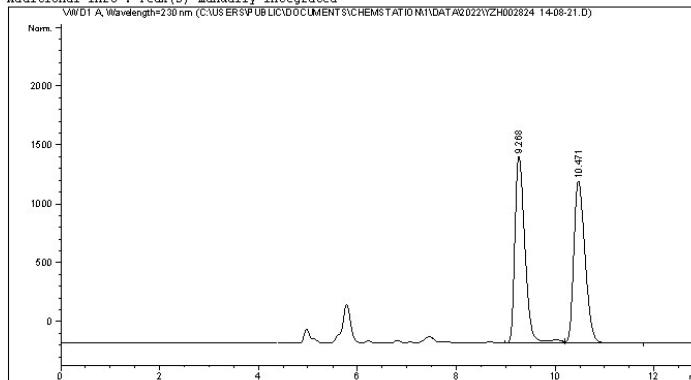
¹³C NMR (100 MHz, CDCl₃)



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2022\YHZ002824 14-08-21.D
Sample Name: HW-12-64C SM+/-

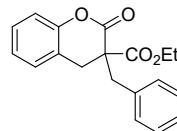
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Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/15/2022 2:08:21 PM      Inj : 1
                                                Inj Volume: No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/15/2022 1:49:03 PM by SYSTEM
                                                (modified after loading)
Analysis Method: C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/16/2023 8:53:54 PM by SYSTEM
                                                (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



```
=====
Area Percent Report
=====

Sorted By       : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```



Signal 1: VWD1 A, Wavelength=230 nm

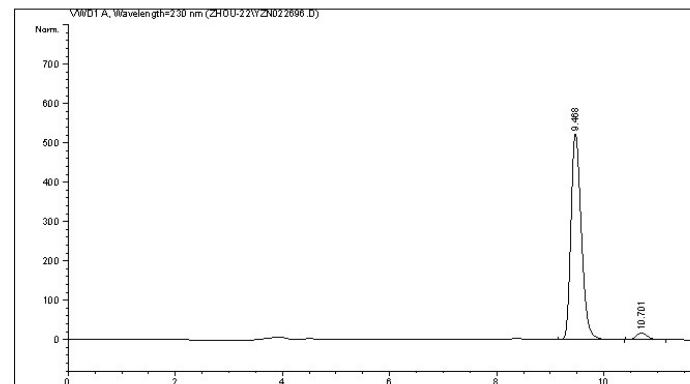
Peak RetTime	Type	Width	Area	Height	Area
#	[min]	[min]	[mAU*s]	[mAU]	%
1	9.268	0.2125	1.7476e4	1216.52478	50.7952
2	10.471	0.2456	1.66107e4	1055.36694	49.2048

HPLC1260 II 10/16/2023 8:54:04 PM SYSTEM

Page 1 of 2

Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022696.D
Sample Name: HW-14-8A SM'

```
=====
Acq. Operator : 
Acq. Instrument : Instrument 1          Location : -
Injection Date : 3/22/2023 8:46:31 AM
Acq. Method   : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 3/22/2023 8:34:55 AM
                                                (modified after loading)
Analysis Method: C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/18/2023 4:00:41 AM
                                                (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



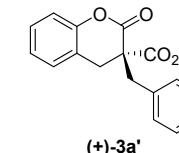
=====
Area Percent Report
=====

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime	Type	Width	Area	Height	Area	
#	[min]	[min]	[mAU*s]	*s	[mAU]	%
1	9.468	VB	0.2098	7061.64648	523.32916	96.4830
2	10.701	BB	0.2273	257.40636	17.46237	3.5170

Totals : 7319.05484 540.79154

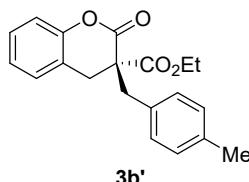


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*** End of Report ***
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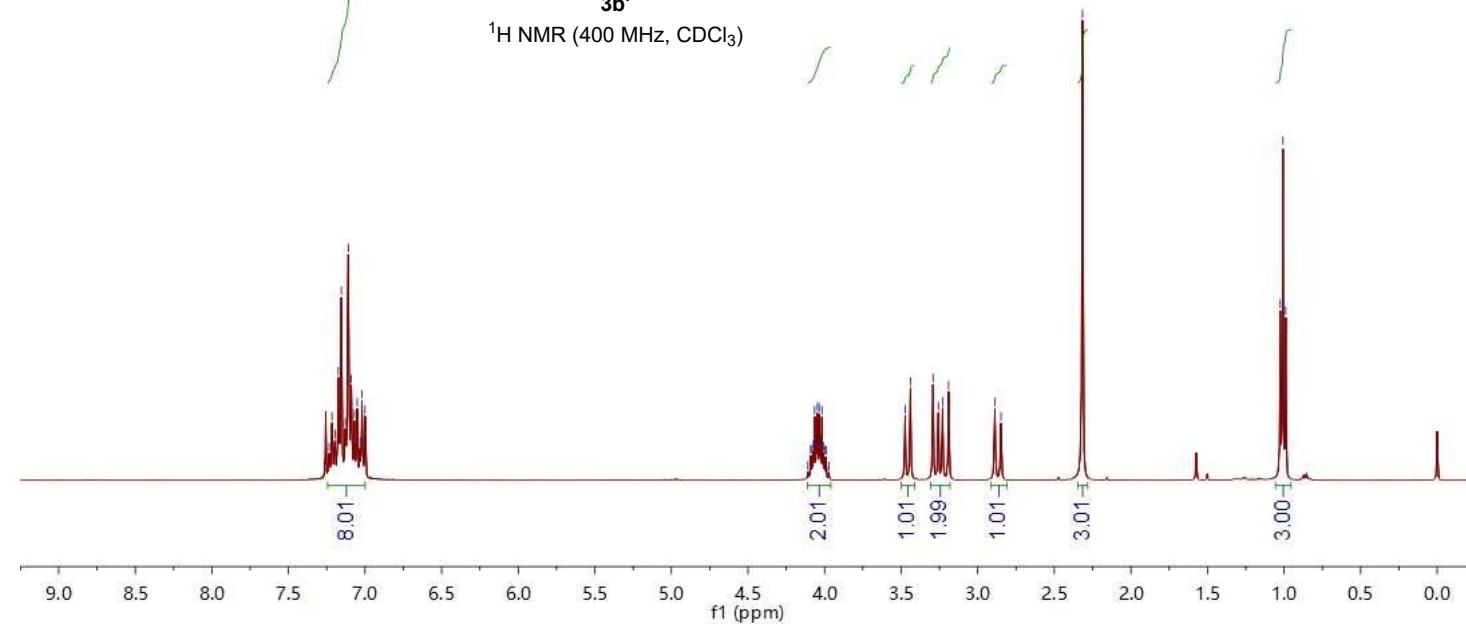
Instrument 1 10/18/2023 4:00:52 AM

Page 1 of 1

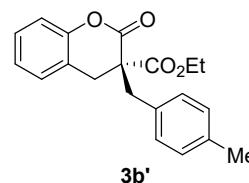
¹H NMR HW-13-98A in CDCl₃



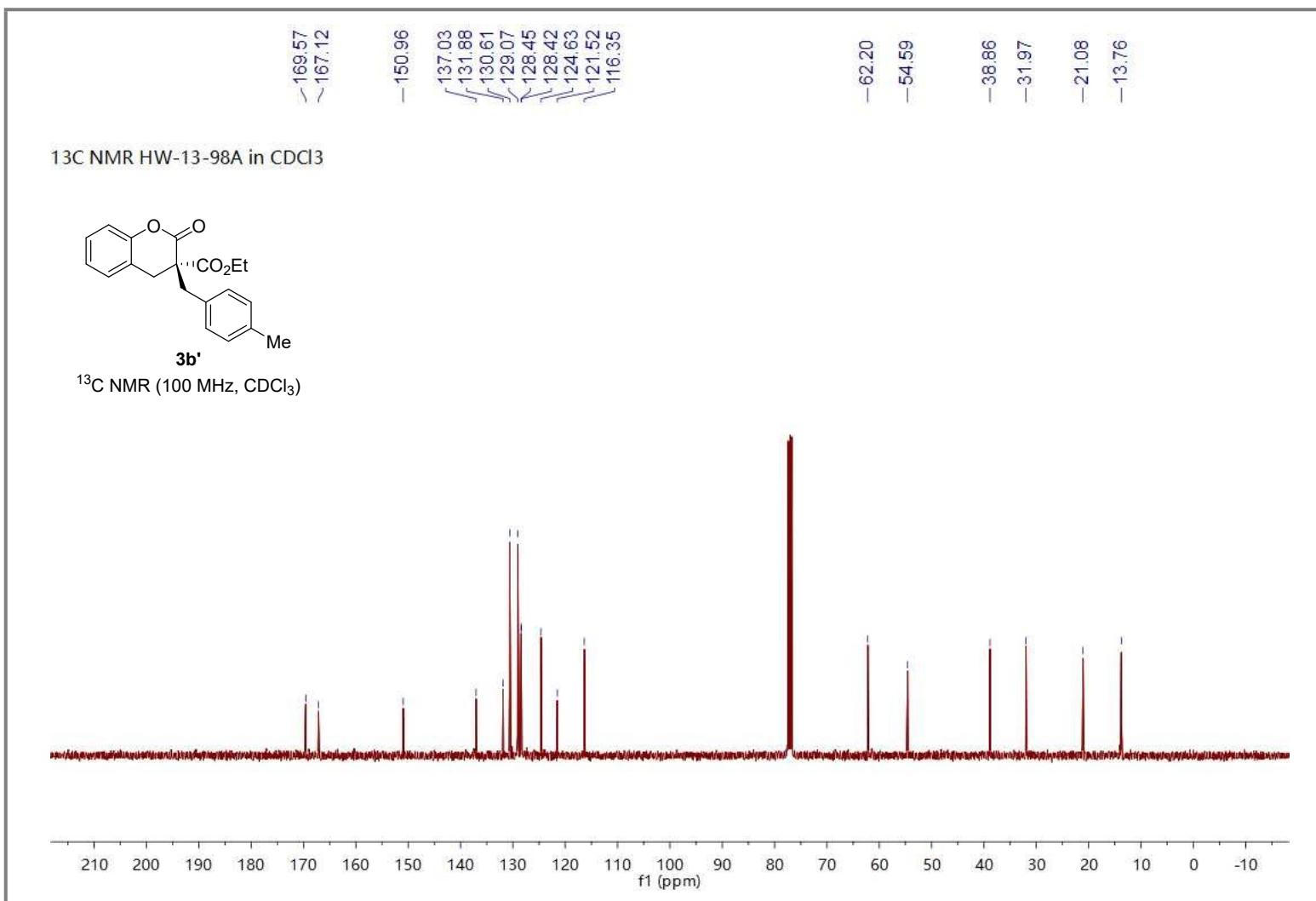
¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-13-98A in CDCl₃

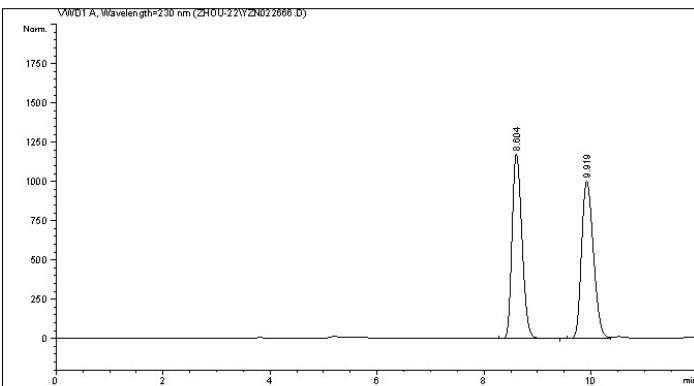


¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022666.D
Sample Name: HW-13-98A+/-

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Acq. Operator : Instrument 1 Location : -
Injection Date : 3/18/2023 4:50:21 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/18/2023 4:49:11 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 8:47:42 AM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230nm
```



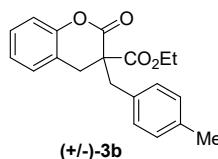
=====
Area Percent Report
=====

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Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	8.604	BB	0.2026	1.5103e4	1173.28320	49.4957	
2	9.919	BV	0.2419	1.5411e4	1002.69934	50.5043	

Totals : 3.05154e4 2175.98254

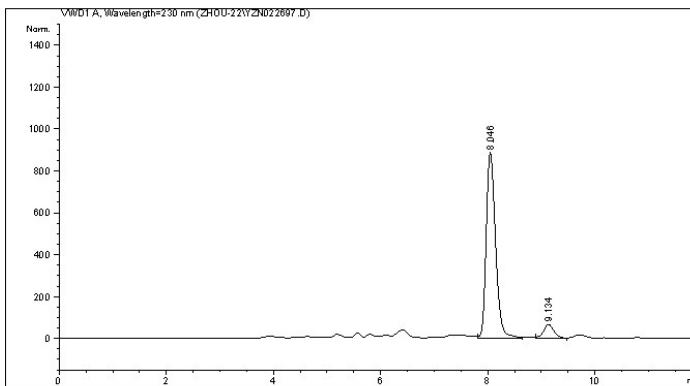


Instrument 1 10/17/2023 8:47:46 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022697.D
Sample Name: HW-14-8B SM'

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 3/22/2023 9:09:22 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/22/2023 9:06:42 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 8:49:46 AM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



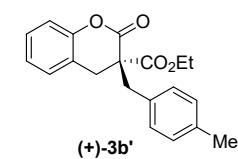
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Area Percent Report
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Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

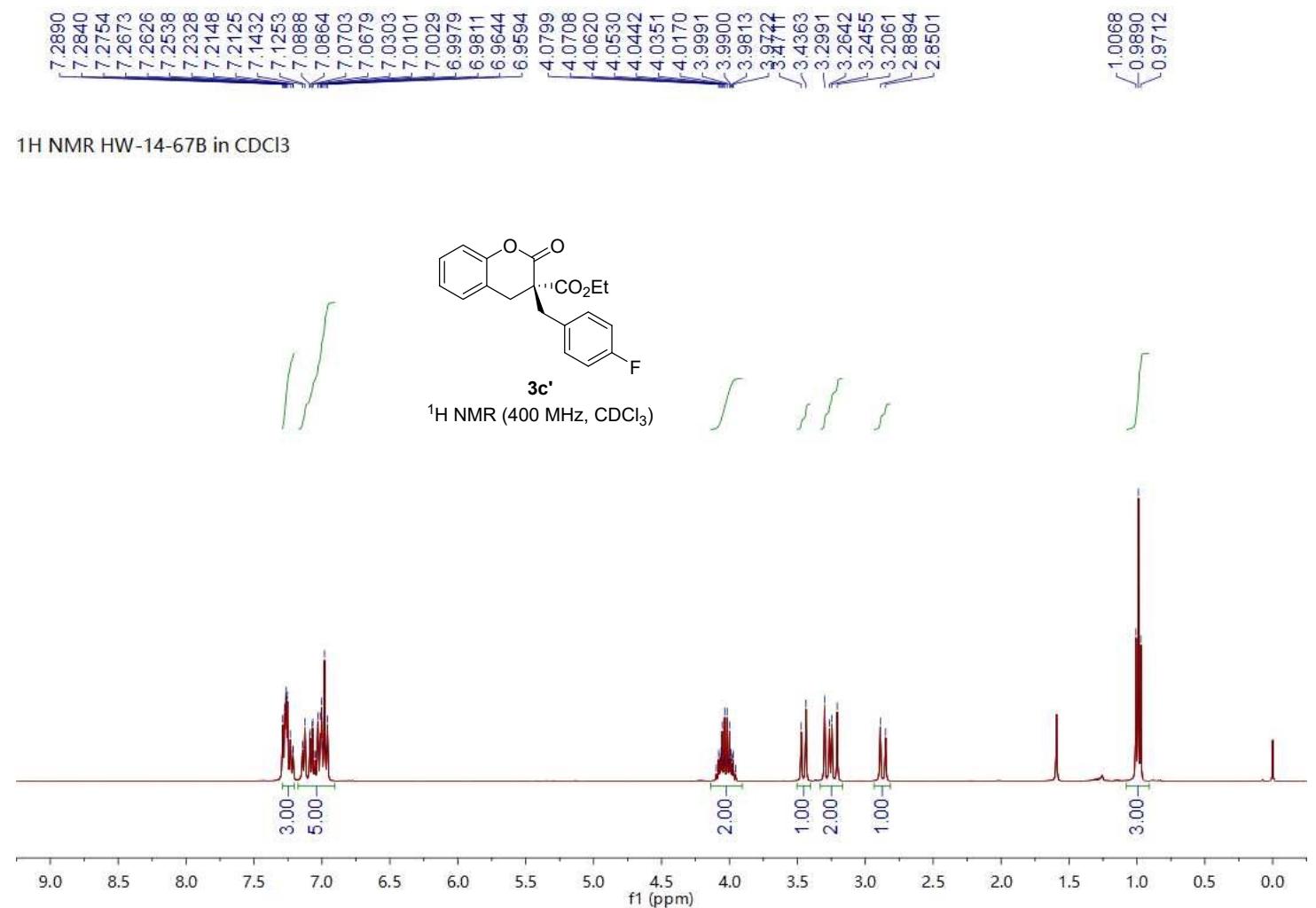
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	8.046	VV	0.1888	1.0811e4	886.86145	92.0598	
2	9.134	VV	0.2106	932.47943	66.90748	7.9402	

Totals : 1.17438e4 953.76893

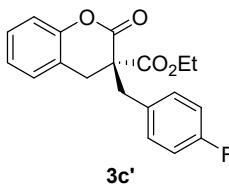


Instrument 1 10/17/2023 8:49:50 AM

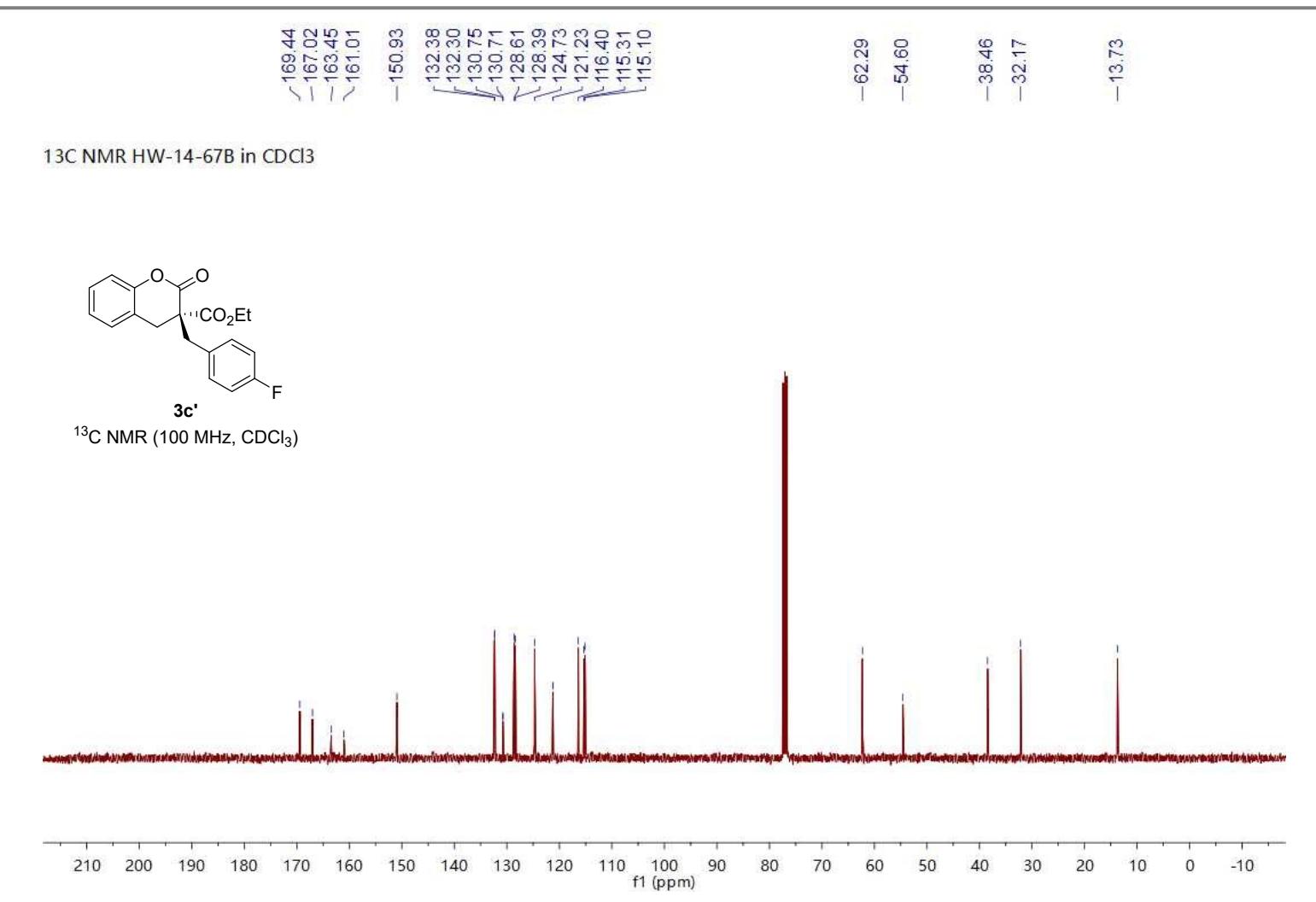
Page 1 of 1



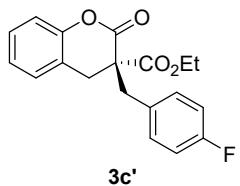
¹³C NMR HW-14-67B in CDCl₃



¹³C NMR (100 MHz, CDCl₃)

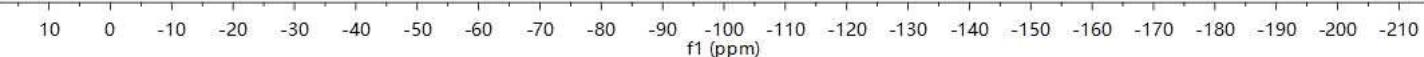


¹⁹F NMR HW-14-67B in CDCl₃



¹⁹F NMR (376 MHz, CDCl₃)

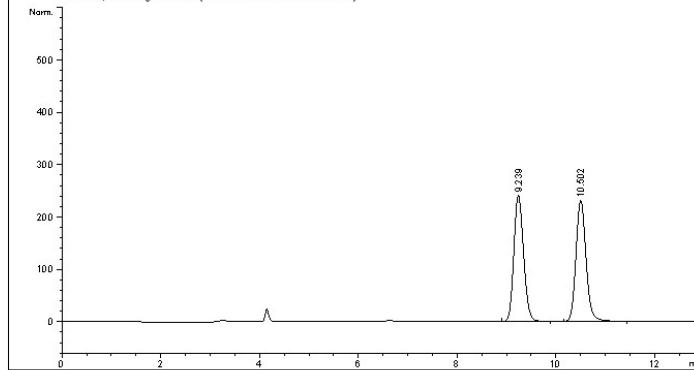
-115.2128



Data File C:\CHEM32\1\DATA\ZHOU-22\YZH005562 17-30-03.D
Sample Name: HW-14-67B+/-

```
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Acq. Operator : SYSTEM
Acq. Instrument : HPLC1260 II
Injection Date : 11/28/2023 5:30:03 PM
Inj Volume : No inj
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Acq. Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed : 11/28/2023 5:27:19 PM by SYSTEM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\def_LC.M
Last changed : 11/30/2023 8:58:34 AM
(modified after loading)
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VWD1A, Wavelength=230 nm (ZHOU-22\YZH005562 17-30-03.D)

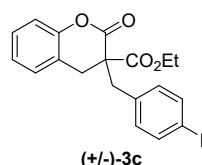


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Area Percent Report

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=====
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.239	BB	0.2119	3275.86230	240.48276	50.0812
2	10.502	BB	0.2191	3265.23950	230.76292	49.9188
Totals :				6541.10181	471.24568	



=====
*** End of Report ***

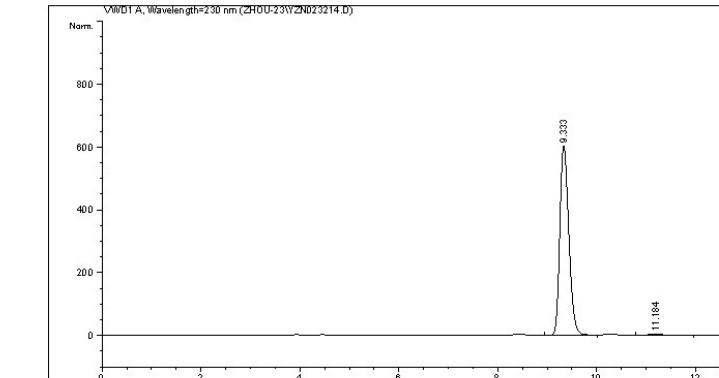
Instrument 1 11/30/2023 8:58:44 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023214.D
Sample Name: HW-14-73B SM

```
=====
Acq. Operator : 
Acq. Instrument : Instrument 1
Injection Date : 5/30/2023 4:58:20 AM
Location : -
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Acq. Method : C:\CHEM32\1\METHODS\def_LC.M
Last changed : 5/30/2023 4:57:54 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\def_LC.M
Last changed : 10/17/2023 8:58:26 AM
(modified after loading)
Sample Info : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

VWD1A, Wavelength=230 nm (ZHOU-23\YZN023214.D)

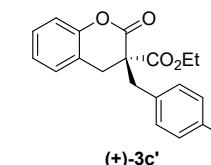


=====
Area Percent Report

```
=====
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

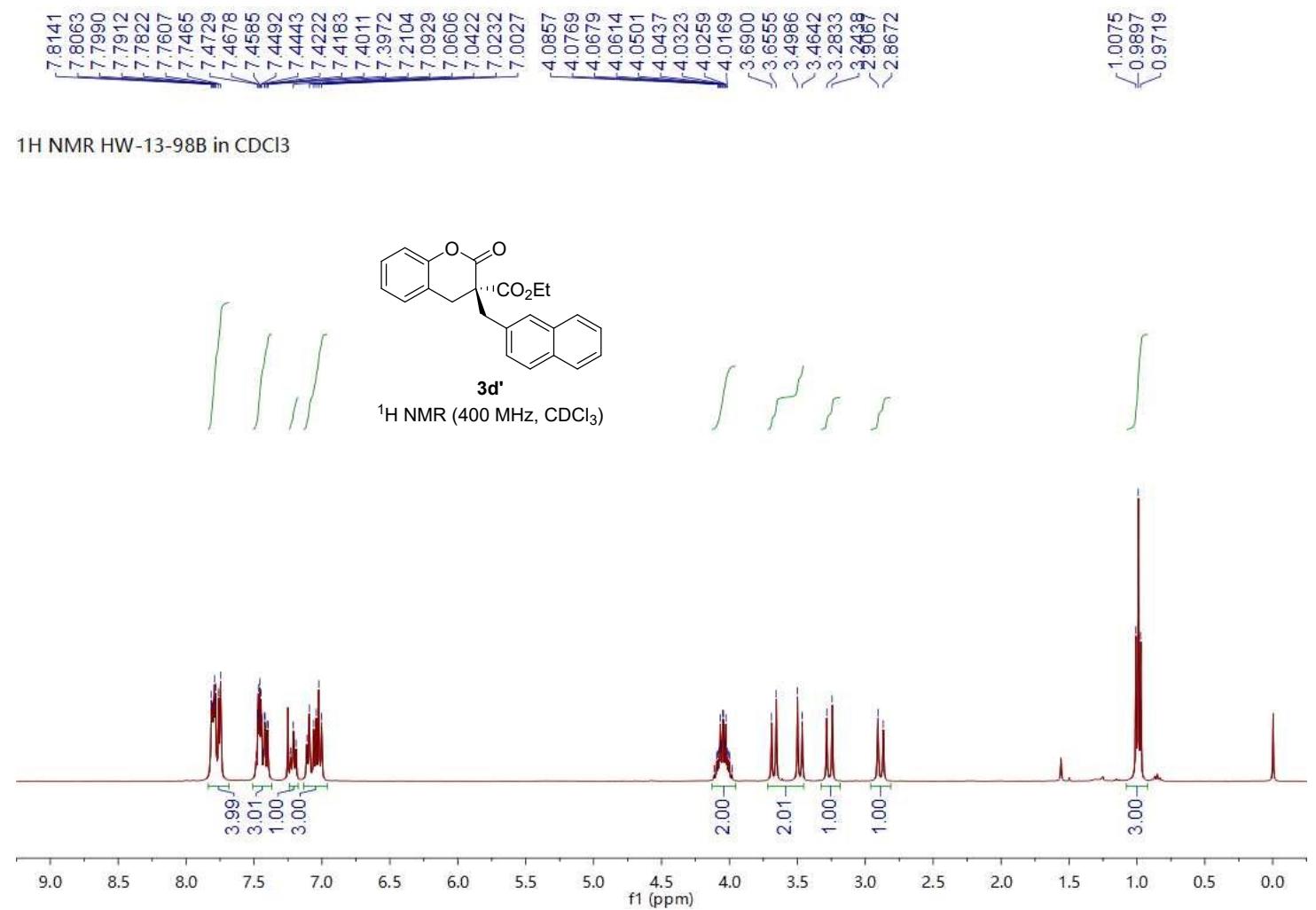
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.333	BV	0.1972	7633.78906	603.07947	99.1347
2	11.184	BV	0.3151	66.63427	3.07884	0.8653
Totals :				7700.42333	606.15831	

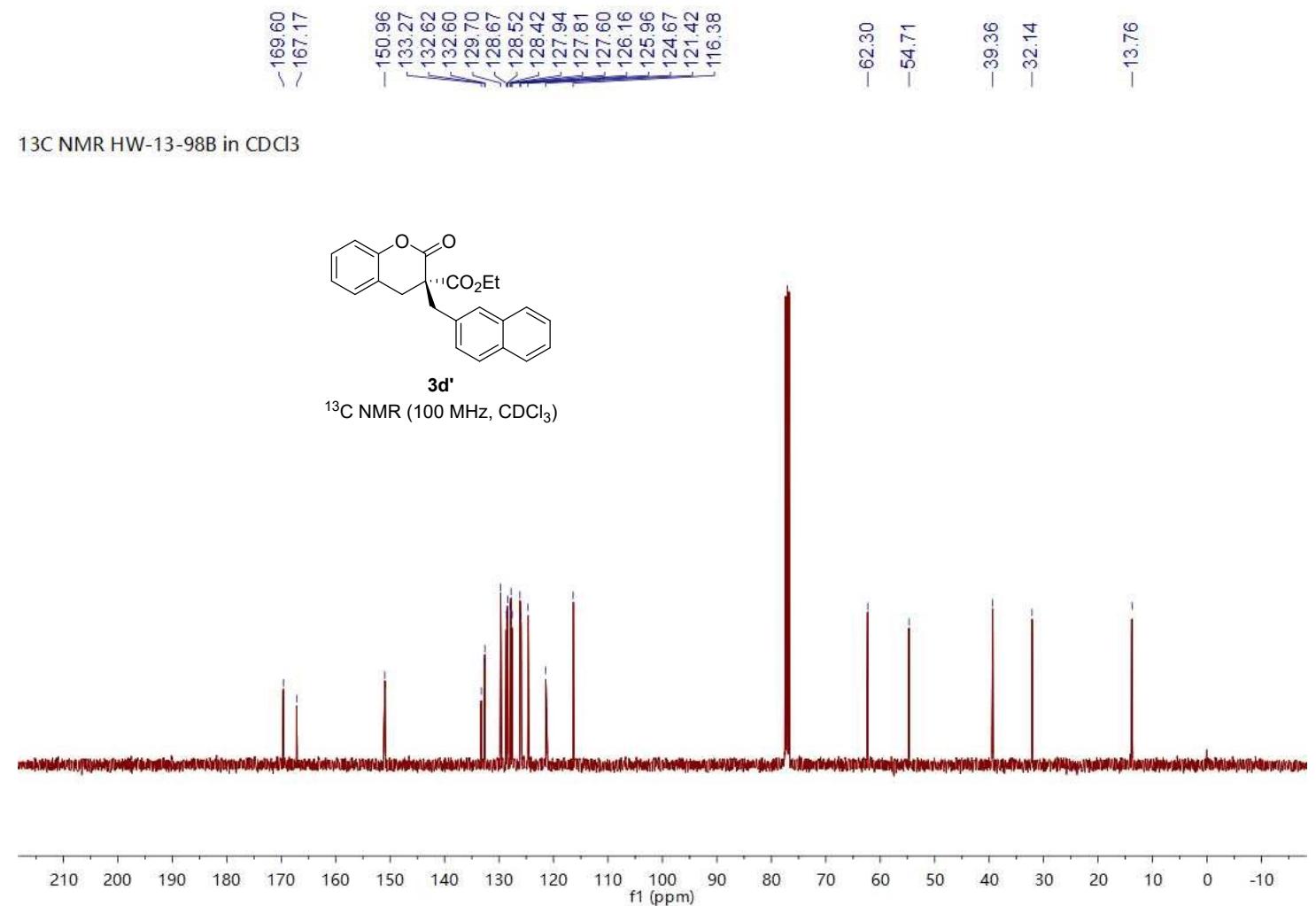


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*** End of Report ***

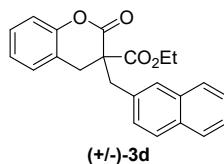
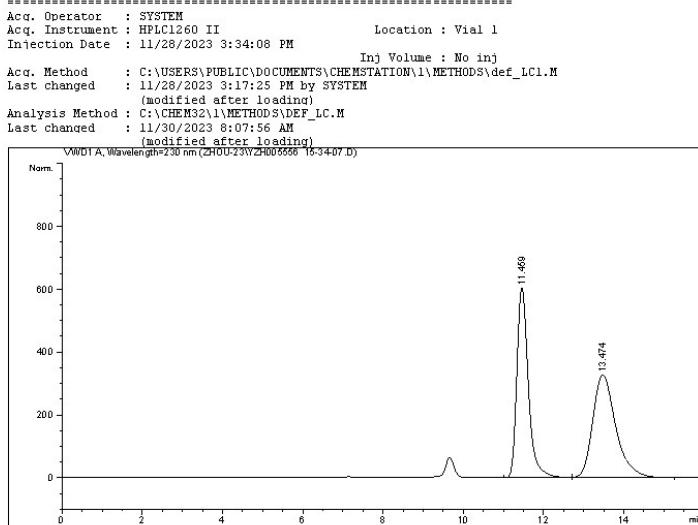
Instrument 1 10/17/2023 8:58:36 AM

Page 1 of 1





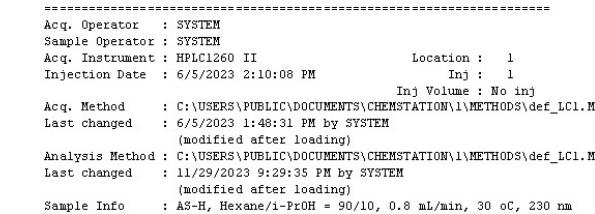
Data File C:\CHEM32\1\DATA\ZHOU-23\YZH005556 15-34-07.D
Sample Name: HW-13-98B +/-



Instrument 1 11/30/2023 8:08:17 AM

Page 1 of 1

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH004326 14-10-08.D
Sample Name: HW-14-76SM

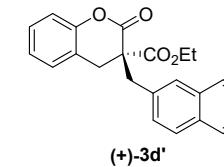


Area Percent Report

```
=====
Sorted By       : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Sample Amount  : 1.00000 [ng/μl] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: VWD1 A, Wavelength=230 nm

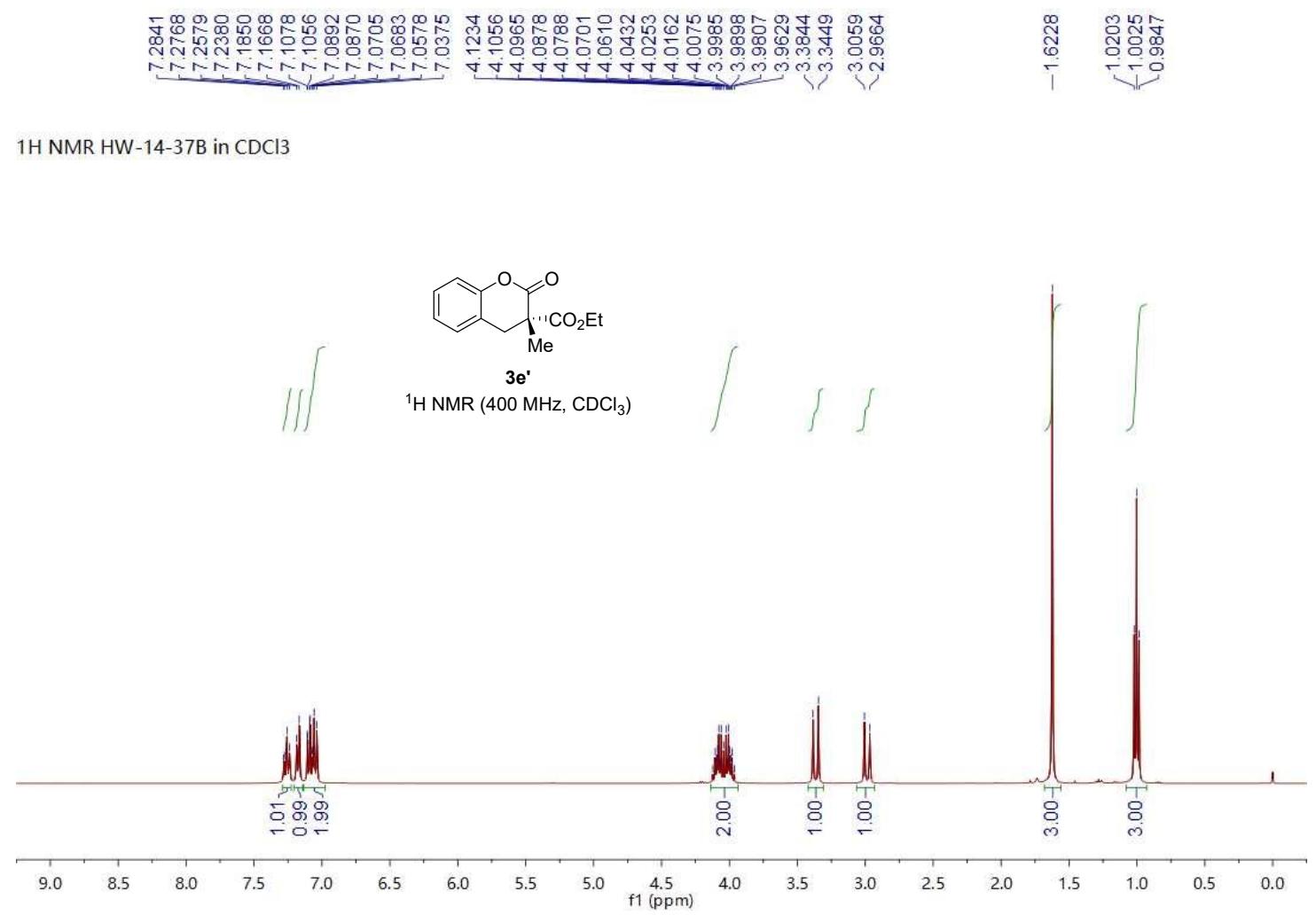
Peak #	RetTime	Type	Width	Area	Height	Area %
	[min]		[min]	[mAU*s]	[mAU]	%
1	11.389	VV R	0.3307	4.77353e4	2337.02051	79.8645
2	13.496	BB	0.5983	1.20351e4	316.75845	20.1355



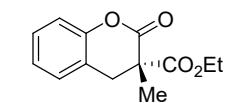
HPLC1260 II 11/29/2023 9:29:43 PM SYSTEM

Page 1 of 1

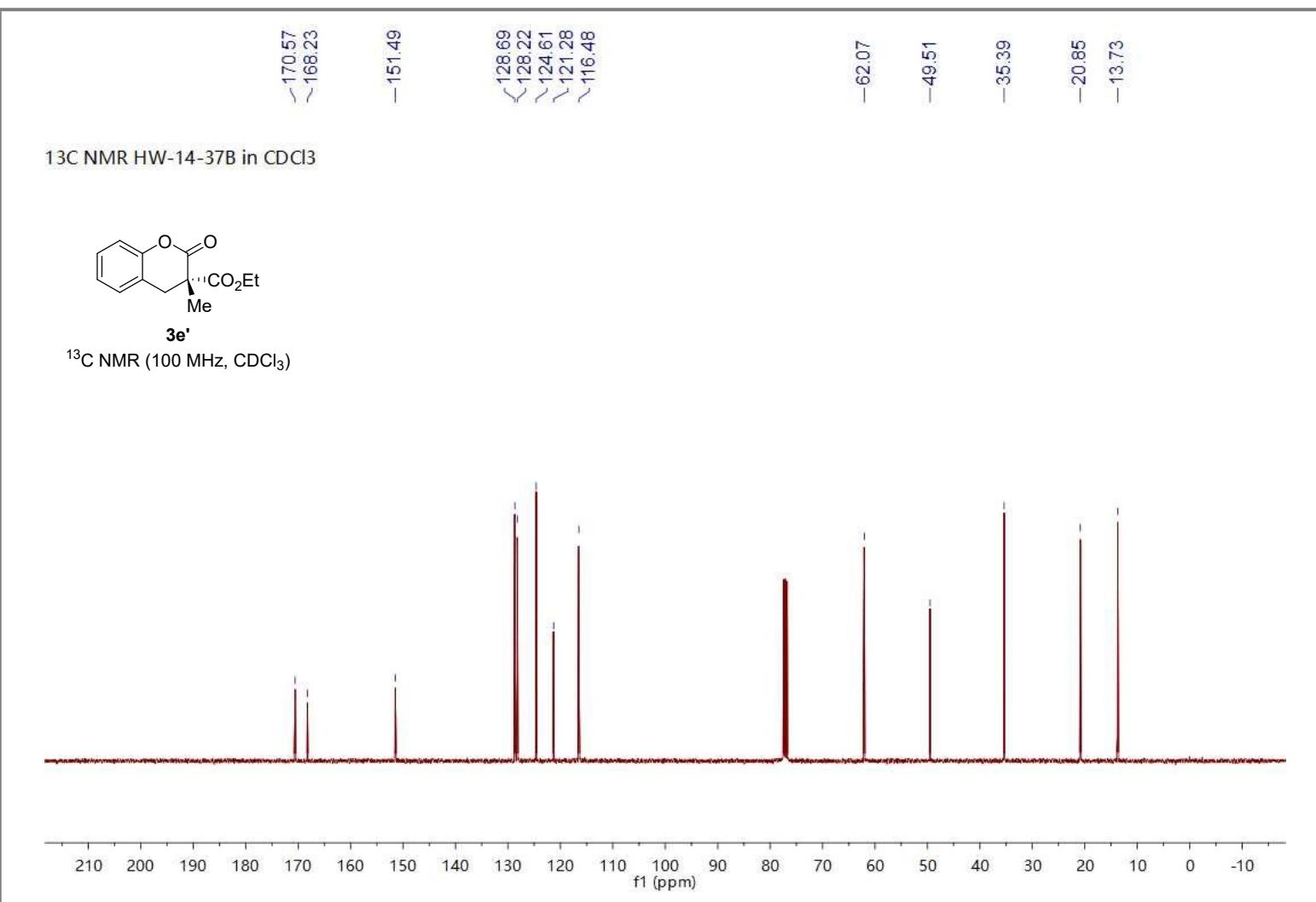
¹H NMR HW-14-37B in CDCl₃



¹³C NMR HW-14-37B in CDCl₃

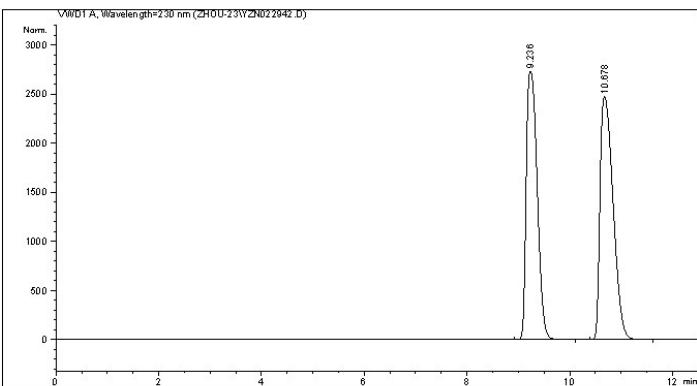


3e'
¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022942.D
Sample Name: HW-14-41B SM+/-

```
=====
Acq. Operator :                               Location : -
Injection Date : 4/22/2023 9:08:35 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/22/2023 9:08:11 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:15:12 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8mL/min, 30 oC, 230 nm
```



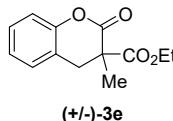
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Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	Area [mAU*s]	Area %
1	9.236	BB	0.2377	4.03326e4	2733.45776	~48.1167	
2	10.678	BB	0.2808	4.34900e4	2474.20898	~51.8833	

Totals : 8.38226e4 5207.66675

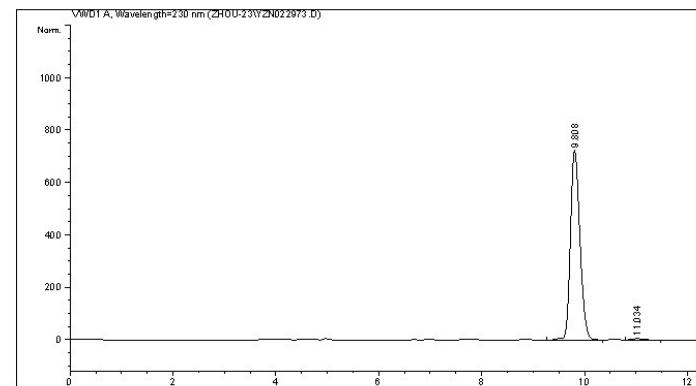


Instrument 1 10/17/2023 9:15:16 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022973.D
Sample Name: HW-14-41B SM'

```
=====
Acq. Operator :                               Location : -
Injection Date : 4/26/2023 3:50:09 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/26/2023 3:46:00 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:16:21 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



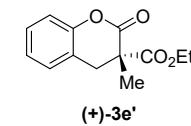
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	Area [mAU*s]	Area %
1	9.808	BV	0.1985	9239.00488	723.36456	~99.1812	
2	11.034	BV	0.2306	76.27379	5.07842	~0.8188	

Totals : 9315.27867 728.44298

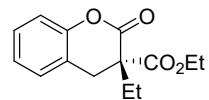


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*** End of Report ***
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Instrument 1 10/17/2023 9:16:29 AM

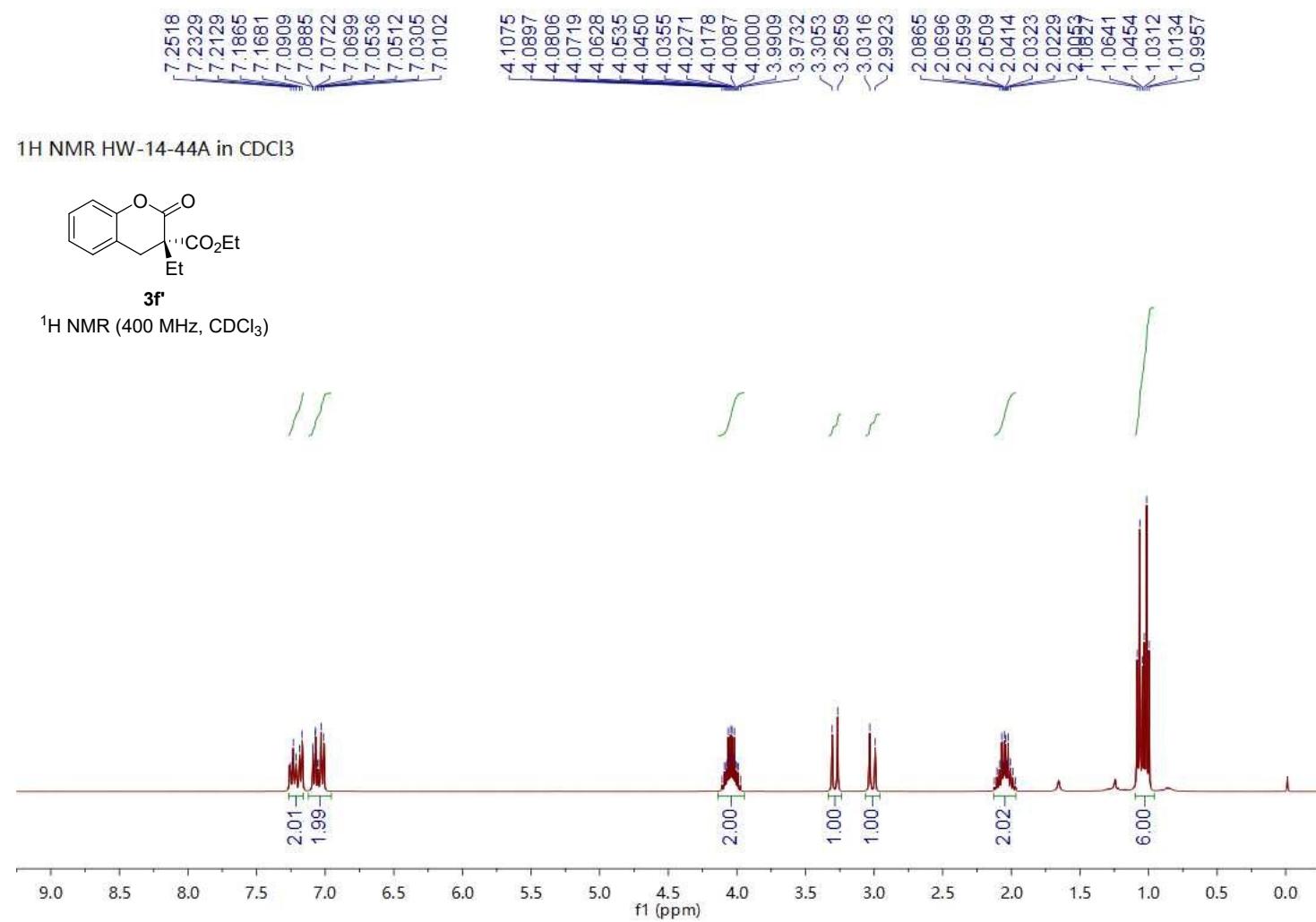
Page 1 of 1

¹H NMR HW-14-44A in CDCl₃

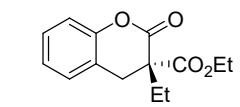


3f

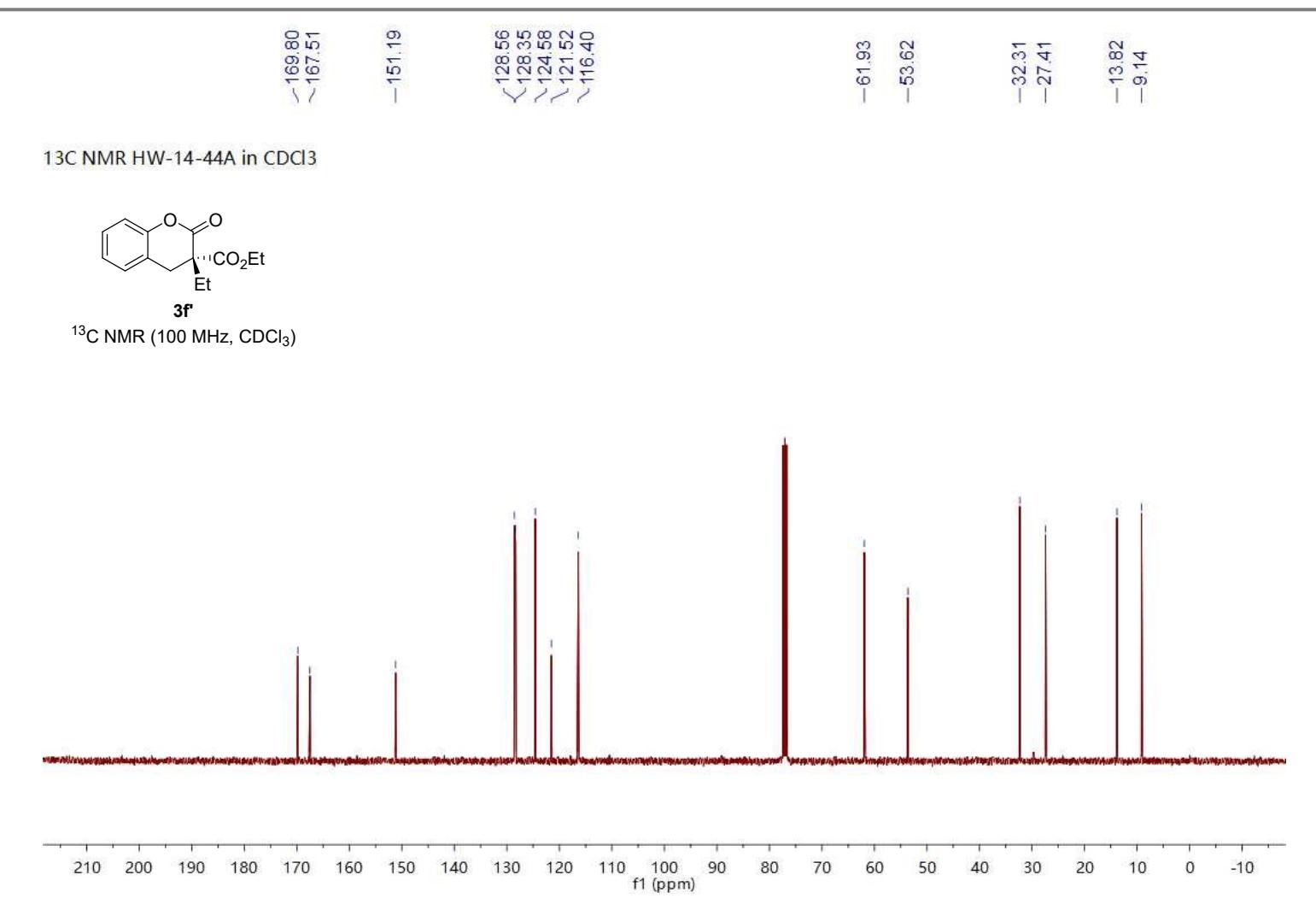
¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-14-44A in CDCl₃

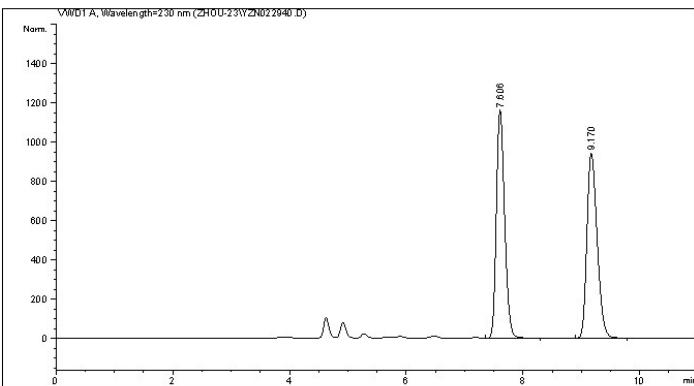


3f
¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022940.D
Sample Name: HW-14-34A SM+/-

```
=====
Acq. Operator :                               Location : -
Injection Date : 4/22/2023 8:14:01 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/22/2023 8:35:28 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:24:39 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8mL/min, 30 oC, 230 nm
```



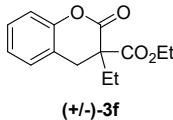
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	7.606	VB	0.1526	1.1410	3e-04	1160.53186	49.8726
2	9.170	VV	0.1903	1.1468	7e-04	940.75964	50.1274

Totals : 2.28790e4 2101.29150

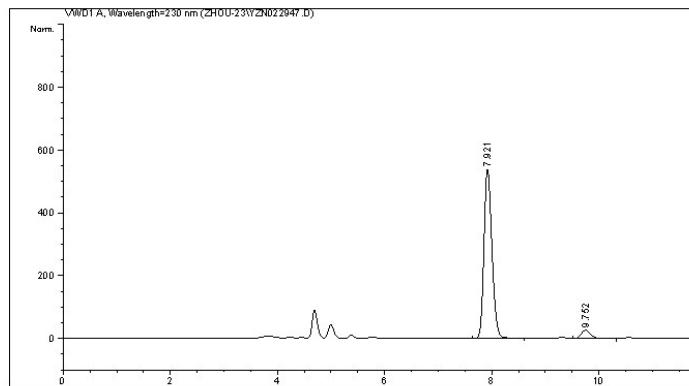


Instrument 1 10/17/2023 9:24:42 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022947.D
Sample Name: HW-14-32A SM

```
=====
Acq. Operator :                               Location : -
Injection Date : 4/23/2023 1:27:16 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/23/2023 1:16:53 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 1/12/2024 3:53:05 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



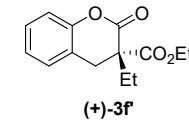
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	7.921	VV	0.1605	5609.98291	540.15234	94.2767	
2	9.752	VV	0.1929	340.56915	27.16190	5.7233	

Totals : 5950.55206 567.31424

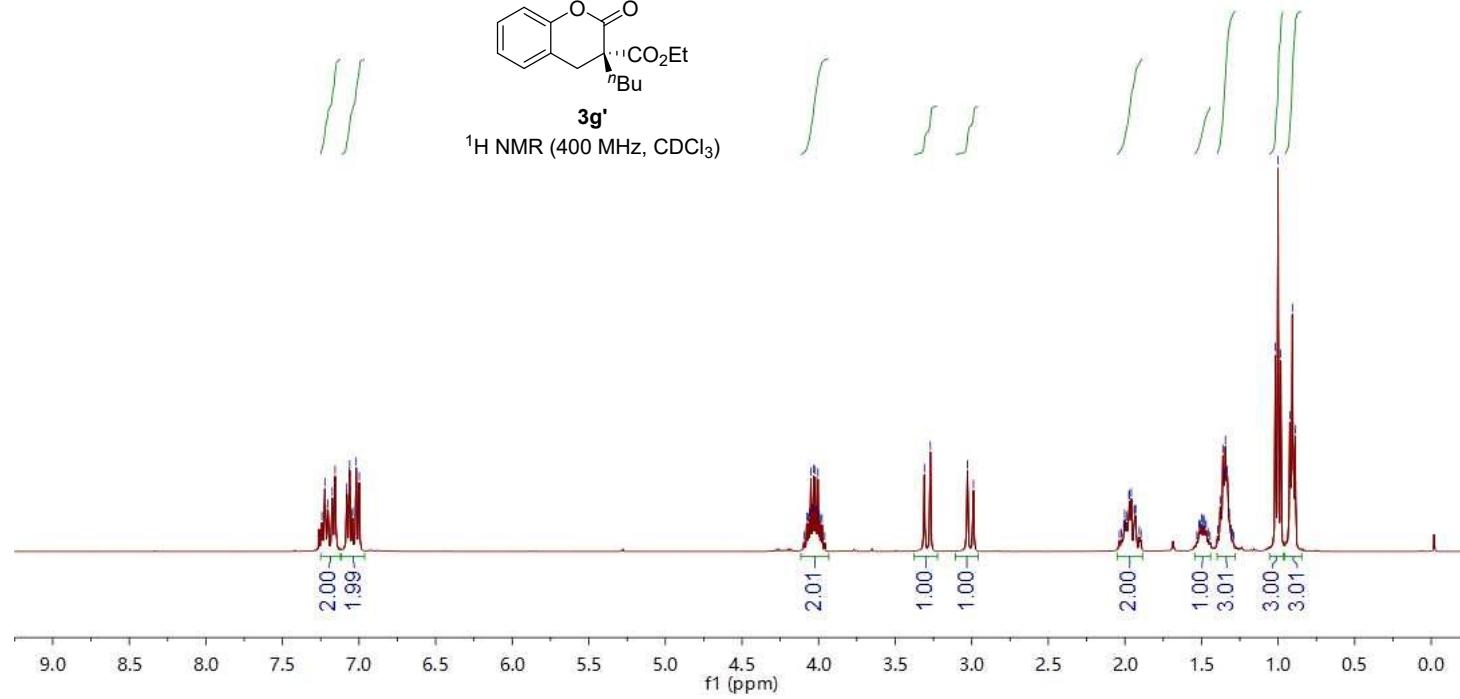
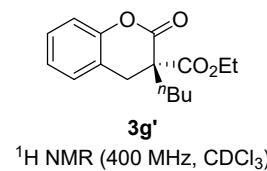


Instrument 1 1/12/2024 3:53:20 AM

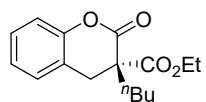
Page 1 of 1

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4.0506
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¹H NMR HW-14-37D in CDCl₃

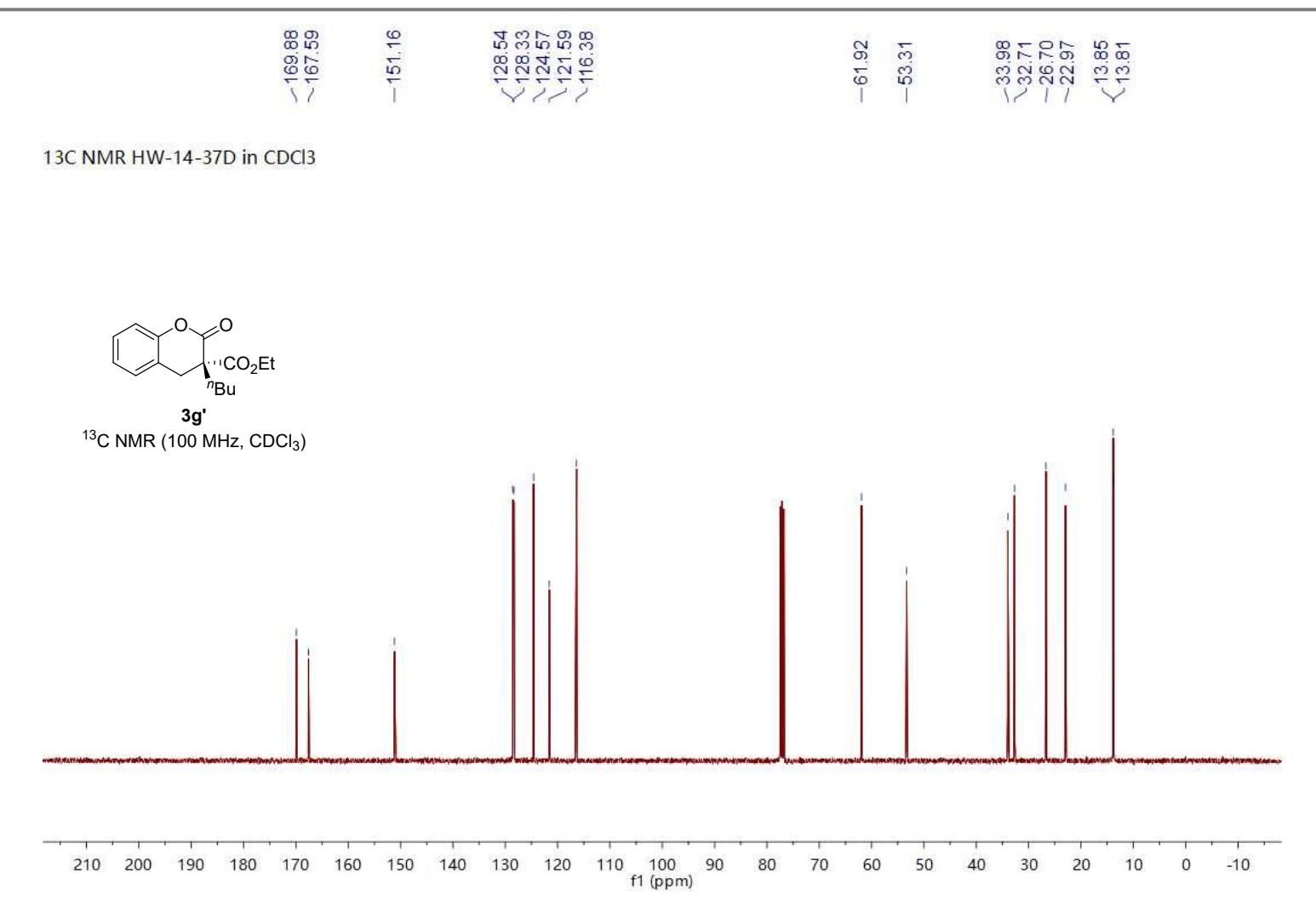


¹³C NMR HW-14-37D in CDCl₃



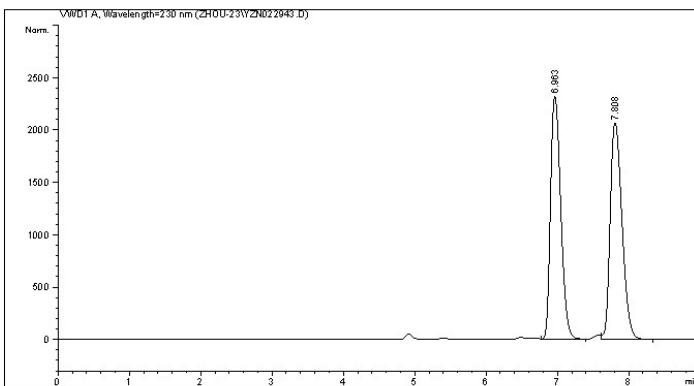
3g'

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022943.D
Sample Name: HW-14-41C SM+/-

```
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Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/22/2023 9:32:44 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:19:47 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8mL/min, 30 oC, 230 nm
```



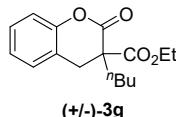
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	6.963	VV	0.1572	2.32125e4	2325.45605	49.3297	
2	7.808	VV	0.1810	2.38433e4	2069.13452	50.6703	

Totals : 4.70558e4 4394.59058

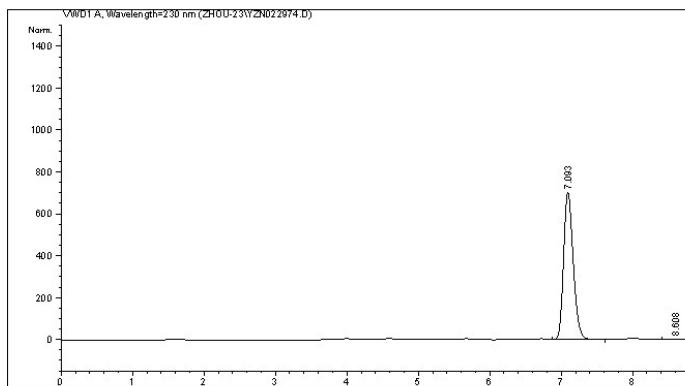


Instrument 1 10/17/2023 9:19:50 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022974.D
Sample Name: HW-14-41C SM'

```
=====
Acq. Operator :                               Location : -
Injection Date : 4/26/2023 4:05:12 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/26/2023 4:04:48 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:20:58 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



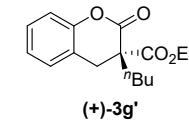
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	7.093	VV	0.1444	6612.80273	704.59741	99.4928	
2	8.608	VB	0.1508	33.71329	3.43738	0.5072	

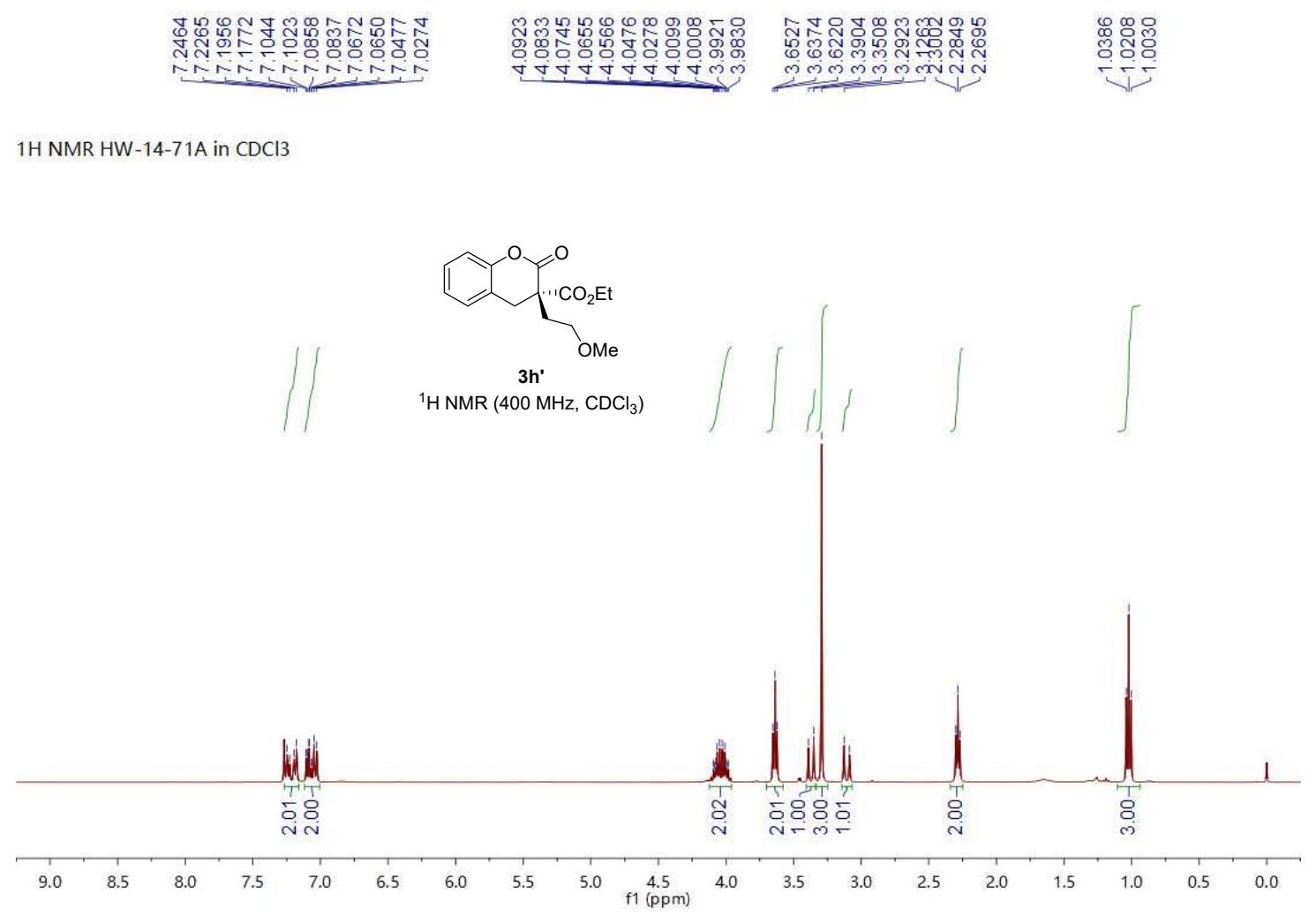
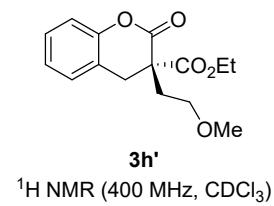
Totals : 6646.51603 708.03480



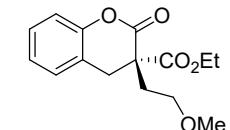
Instrument 1 10/17/2023 9:21:04 AM

Page 1 of 1

¹H NMR HW-14-71A in CDCl₃



¹³C NMR HW-14-71A in CDCl₃



3h'
¹³C NMR (100 MHz, CDCl₃)

> 169.55
> 167.55

-151.12

< 128.59
< 128.35
> 124.63
> 121.50
> 116.41

> 68.77
- 62.07
- 58.61
> 51.99

< 34.08
< 33.42

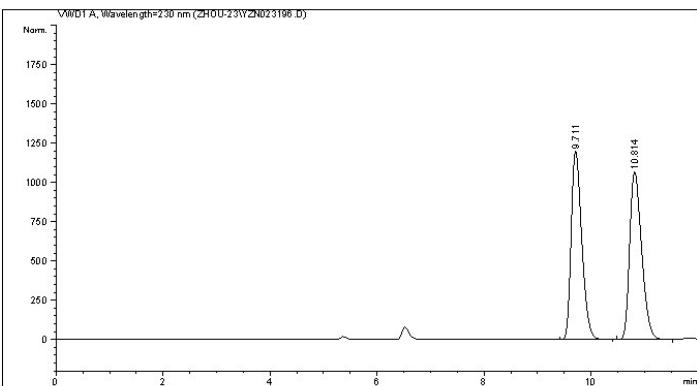
- 13.74

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10

f1 (ppm)

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023196.D
Sample Name: HW-14-70A +/-

```
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Acq. Operator :                               Location : -
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Last changed   : 5/26/2023 4:31:26 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:28:03 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



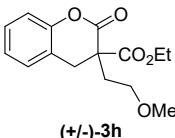
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Area Percent Report
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```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height *s	[mAU]	Area %
1	9.711	BB	0.2129	1.64655e4	1196.65088	49.8354	
2	10.814	BV	0.2412	1.65743e4	1065.27185	50.1646	

Totals : 3.30399e4 2261.92273

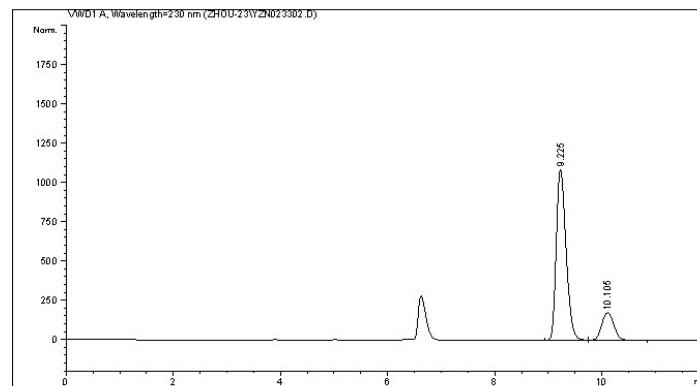


Instrument 1 10/17/2023 9:28:06 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023302.D
Sample Name: HW-14-80 SM

```
=====
Acq. Operator :                               Location : -
Injection Date : 6/13/2023 7:49:12 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 6/13/2023 7:44:25 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 11/30/2023 9:02:08 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



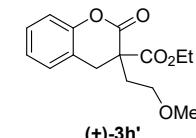
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

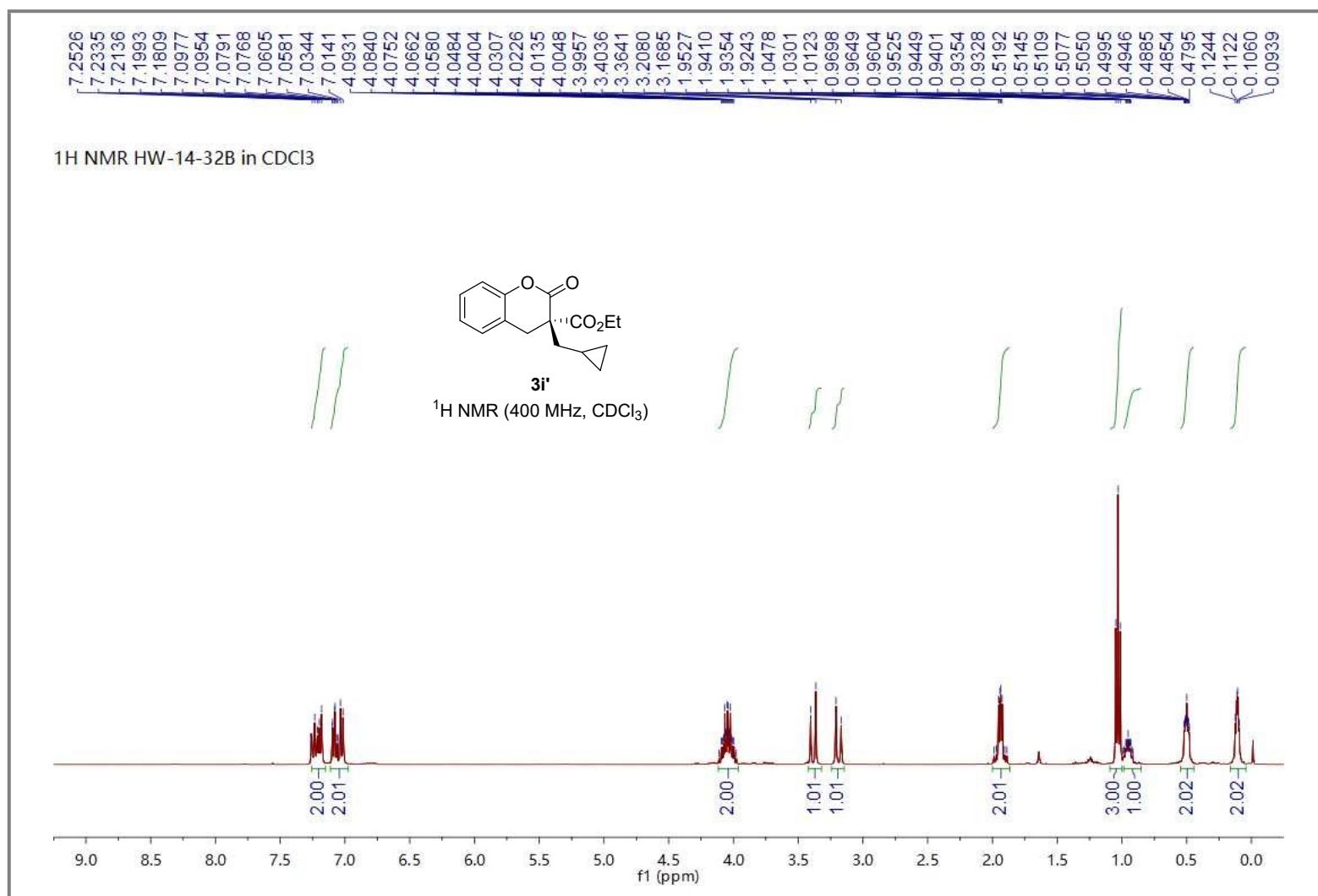
Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	Height *s	[mAU]	Area %
1	9.225	VV	0.1976	1.38067e4	1087.73401	83.2211	
2	10.105	VB	0.2539	2783.67700	175.13556	16.7789	

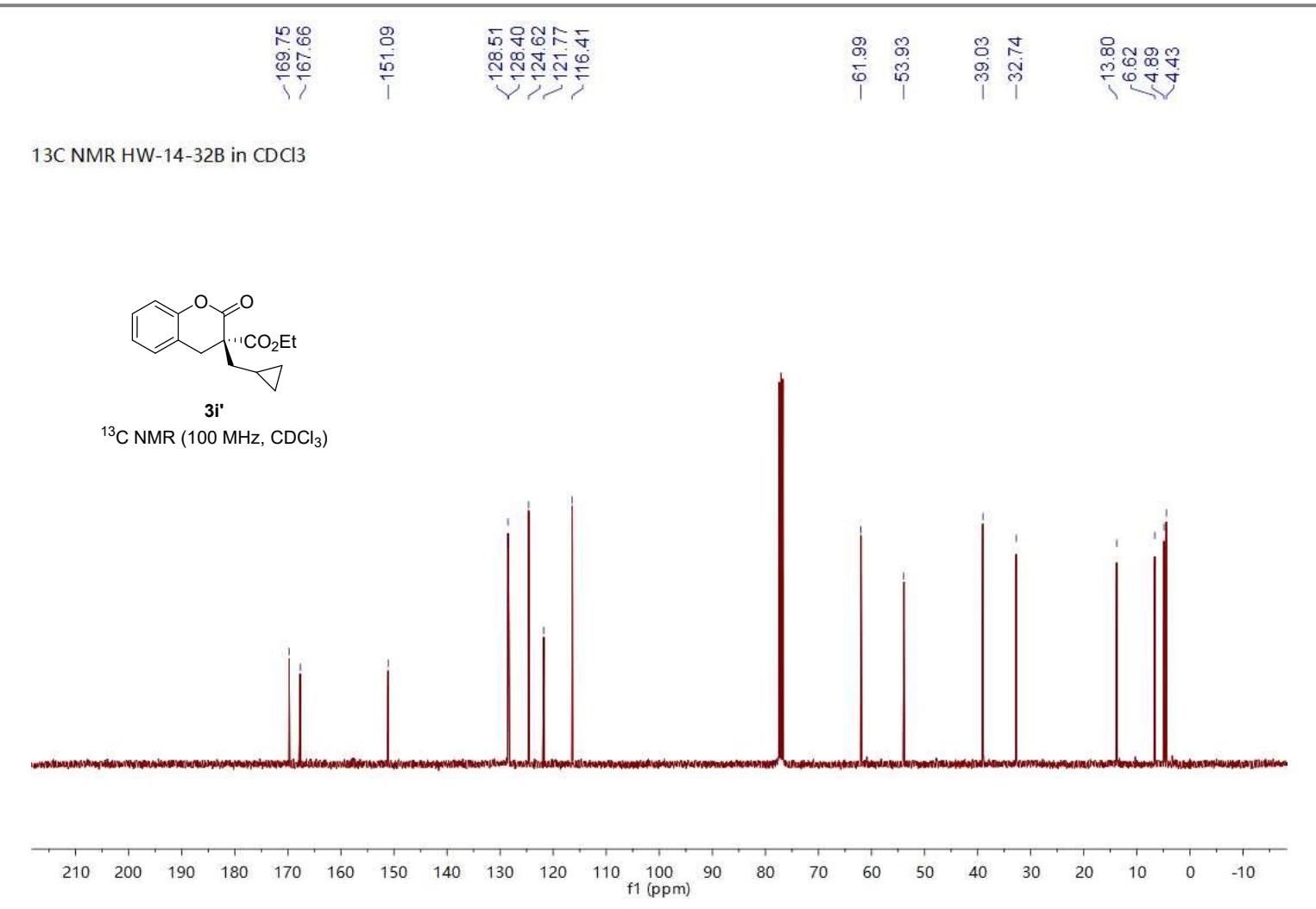
Totals : 1.65904e4 1262.86957



Instrument 1 11/30/2023 9:02:11 AM

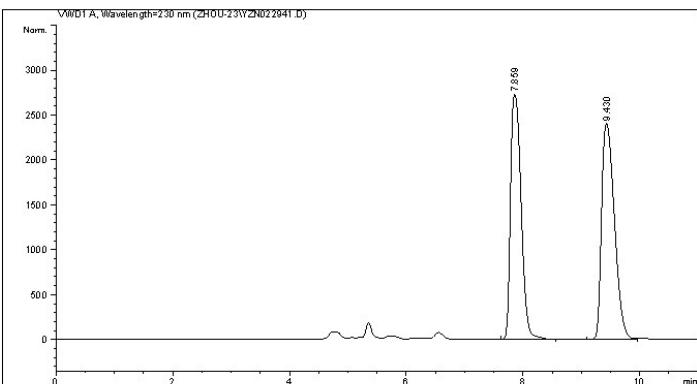
Page 1 of 1





Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022941.D
Sample Name: HW-14-34E SM+/-

```
=====
Acq. Operator :                               Location : -
Injection Date : 4/22/2023 8:53:22 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/22/2023 8:52:29 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:32:09 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8mL/min, 30 oC, 230 nm
```



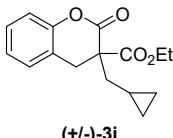
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	s	Height [mAU]	Area %
1	7.859	VV	0.1994	3.43748e4	2727.70850	48.4090	
2	9.430	VV	0.2419	3.66344e4	2403.72900	51.5910	

Totals : 7.10092e4 5131.43750

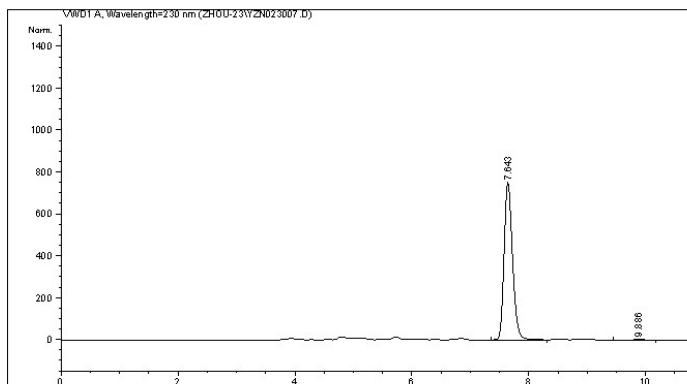


Instrument 1 10/17/2023 9:32:12 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023007.D
Sample Name: HW-14-47A SM

```
=====
Acq. Operator :                               Location : -
Injection Date : 5/5/2023 3:58:08 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/5/2023 3:57:05 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:34:49 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230nm
```



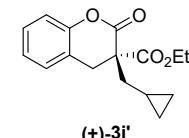
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	s	Height [mAU]	Area %
1	7.643	VV	0.1588	7590.19287	750.01428	99.3829	
2	9.886	VV	0.2269	47.12951	2.95362	0.6171	

Totals : 7637.32238 752.96790

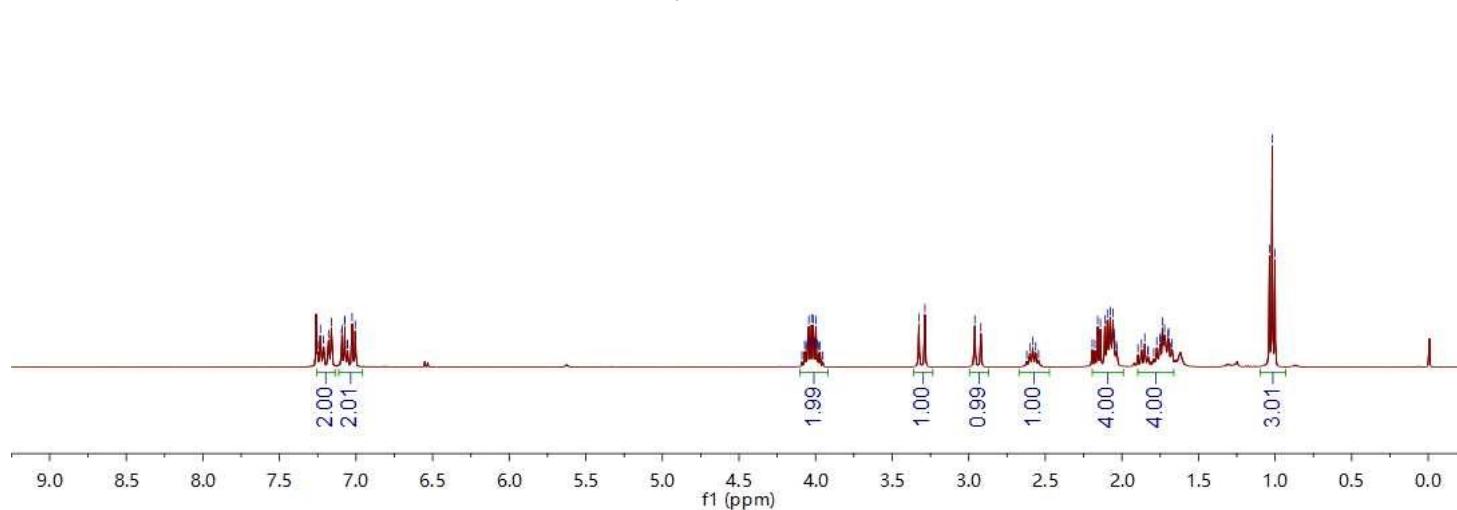
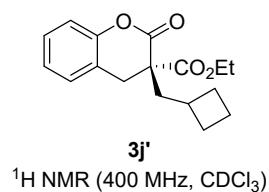


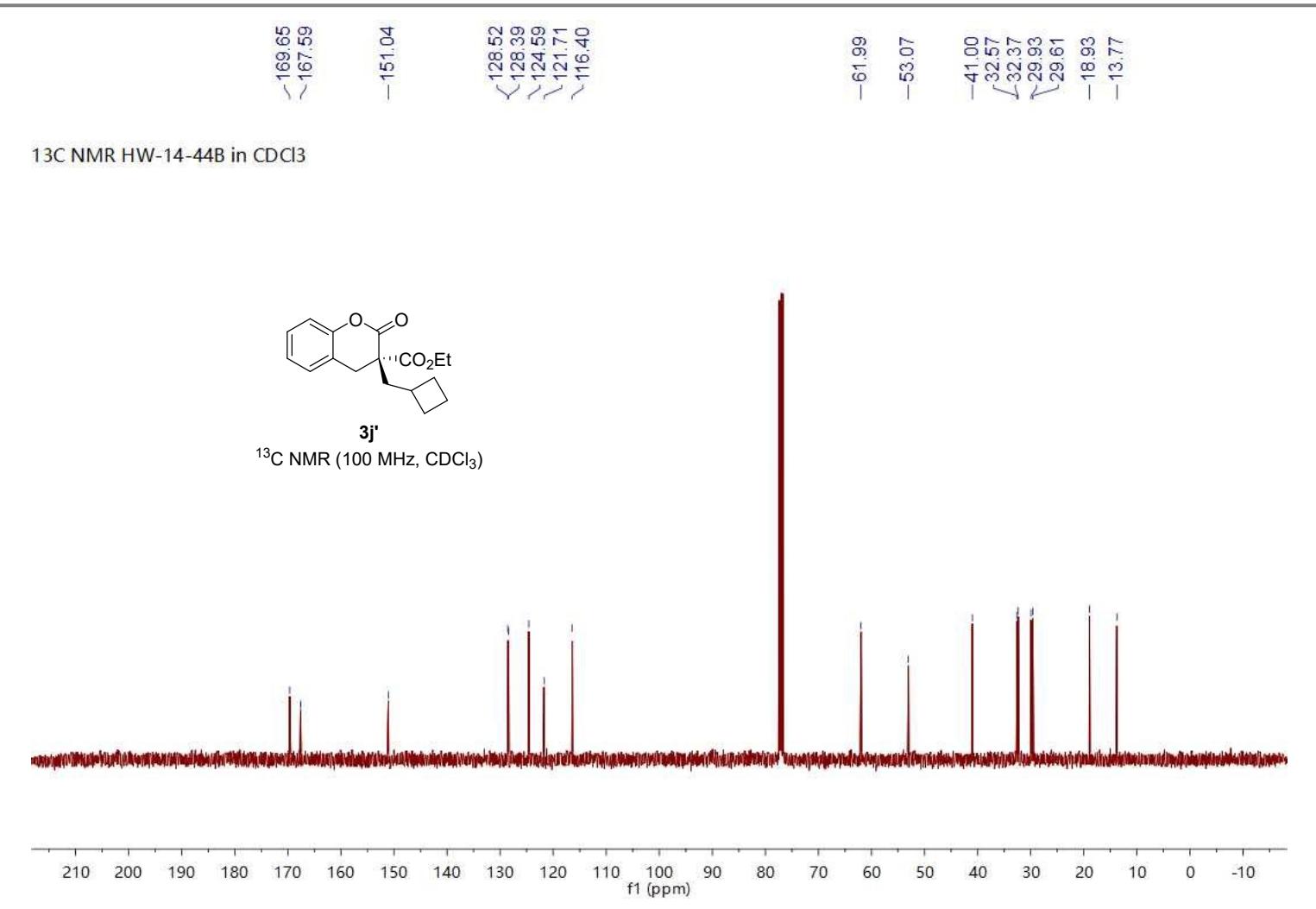
Instrument 1 10/17/2023 9:34:51 AM

Page 1 of 1

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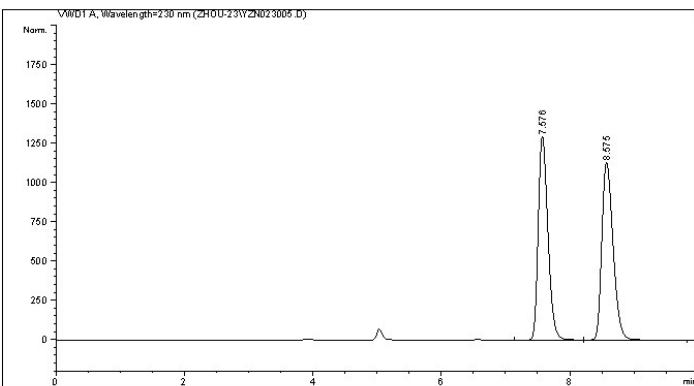
¹H NMR HW-14-44B in CDCl₃





Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023005.D
Sample Name: HW-14-47B SM+/-

```
=====
Acq. Operator :                               Location : -
Injection Date : 5/5/2023 3:26:16 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/5/2023 3:17:54 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:39:16 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230nm
```

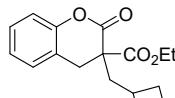


```
=====
Area Percent Report
```

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	7.576	VV	0.1639	1.36363e4	1291.87915	49.7430	
2	8.575	VV	0.1891	1.37773e4	1127.55725	50.2570	
Totals :				2.74136e4	2419.43640		



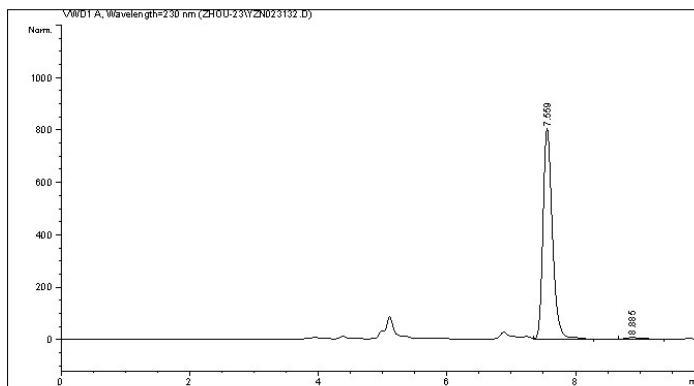
(±)-3j

Instrument 1 10/17/2023 9:39:19 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023132.D
Sample Name: HW-14-56A SM

```
=====
Acq. Operator :                               Location : -
Injection Date : 5/20/2023 8:16:59 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/20/2023 7:53:15 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:40:58 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

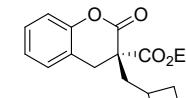


```
=====
Area Percent Report
```

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	7.559	VV	0.1608	8269.88477	803.69092	97.7994	
2	8.885	VV	0.3327	186.08423	7.74540	2.2006	
Totals :				8455.66899	811.43632		



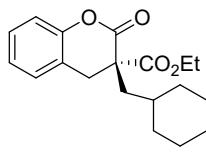
(+)-3j'

Instrument 1 10/17/2023 9:41:01 AM

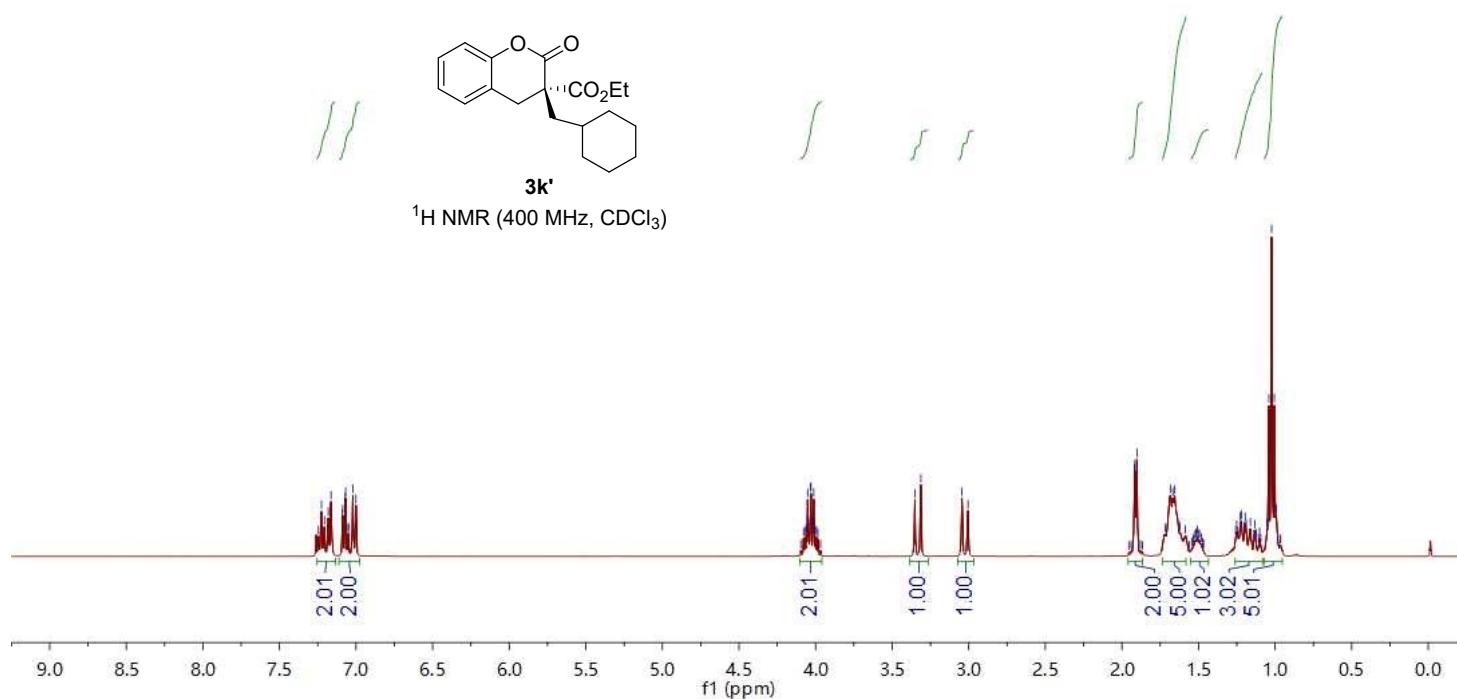
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0.9920

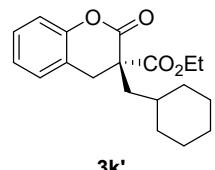
¹H NMR HW-14-51C in CDCl₃



¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-14-51C in CDCl₃



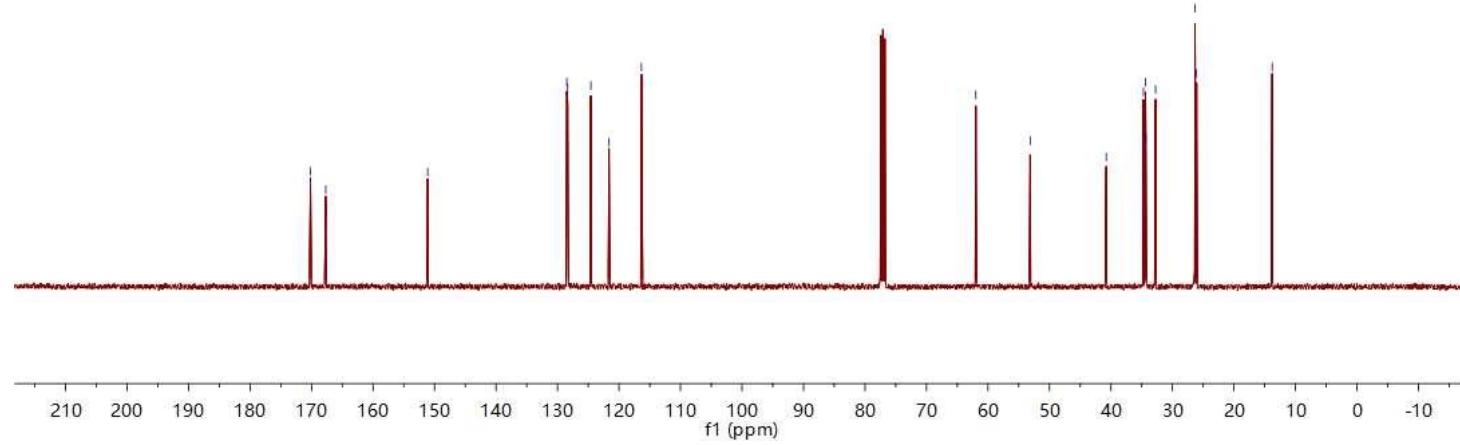
¹³C NMR (100 MHz, CDCl₃)

> 170.17
> 167.70

-151.11

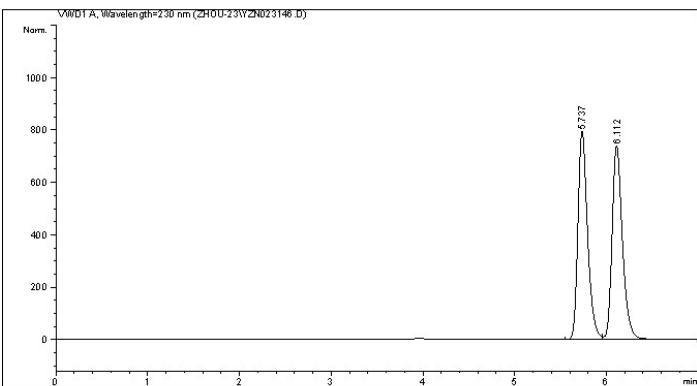
< 128.50
< 128.35
> 124.58
> 121.62
> 116.36

-61.98
-53.14
40.77
34.72
34.37
34.31
32.74
26.32
26.08
-13.78



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023146.D
Sample Name: HW-14-56B SM+/-

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Injection Date : 5/21/2023 1:46:25 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/21/2023 1:37:19 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:44:31 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



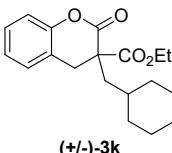
=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	*s	Height [mAU]	1	Area %
1	5.737	VV	0.1148	5801.85840	789.97021	49.8961		
2	6.112	VV	0.1271	5826.03174	737.68420	50.1039		

Totals : 1.16279e4 1527.65442



(+/-)-3k

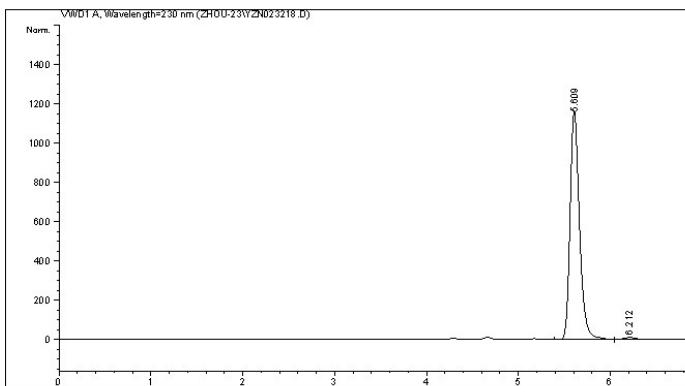
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*** End of Report ***
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Instrument 1 10/17/2023 9:44:35 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023218.D
Sample Name: HW-14-73C SM

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 5/30/2023 9:35:19 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/30/2023 9:35:01 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:45:28 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



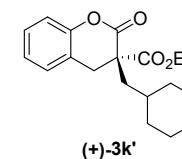
=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU]	*s	Height [mAU]	1	Area %
1	5.609	VV	0.1078	8286.52246	1143.56421	98.4766		
2	6.212	VV	0.1696	128.19243	11.10791	1.5234		

Totals : 8414.71489 1154.67212



(+)-3k'

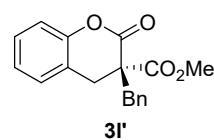
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*** End of Report ***
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Instrument 1 10/17/2023 9:45:31 AM

Page 1 of 1

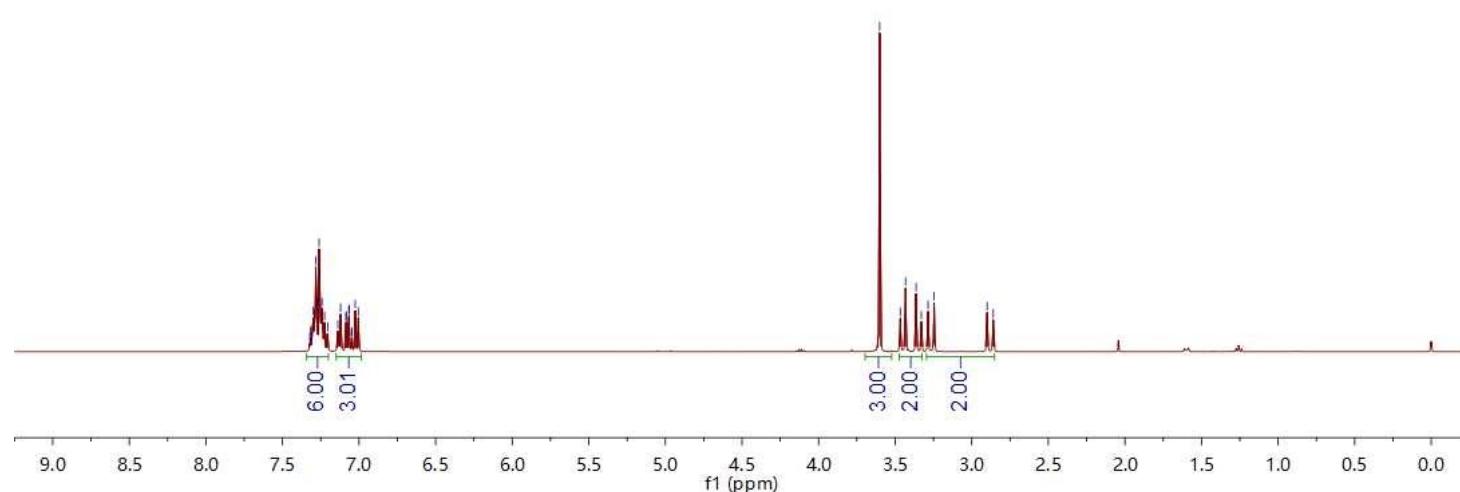
7.3216
7.3157
7.3117
7.2982
7.2925
7.2821
7.2722
7.2607
7.2557
7.2418
7.2252
7.2047
7.1382
7.1206
7.0859
7.0836
7.0674
7.0652
7.0489
7.0465
7.0251
7.0048

¹H NMR HW-17-46 in CDCl₃

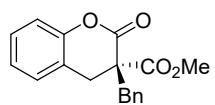


¹H NMR (400 MHz, CDCl₃)

3.6009
3.4664
3.4320
3.3635
3.3291
3.2852
3.2454
2.8994
2.8596

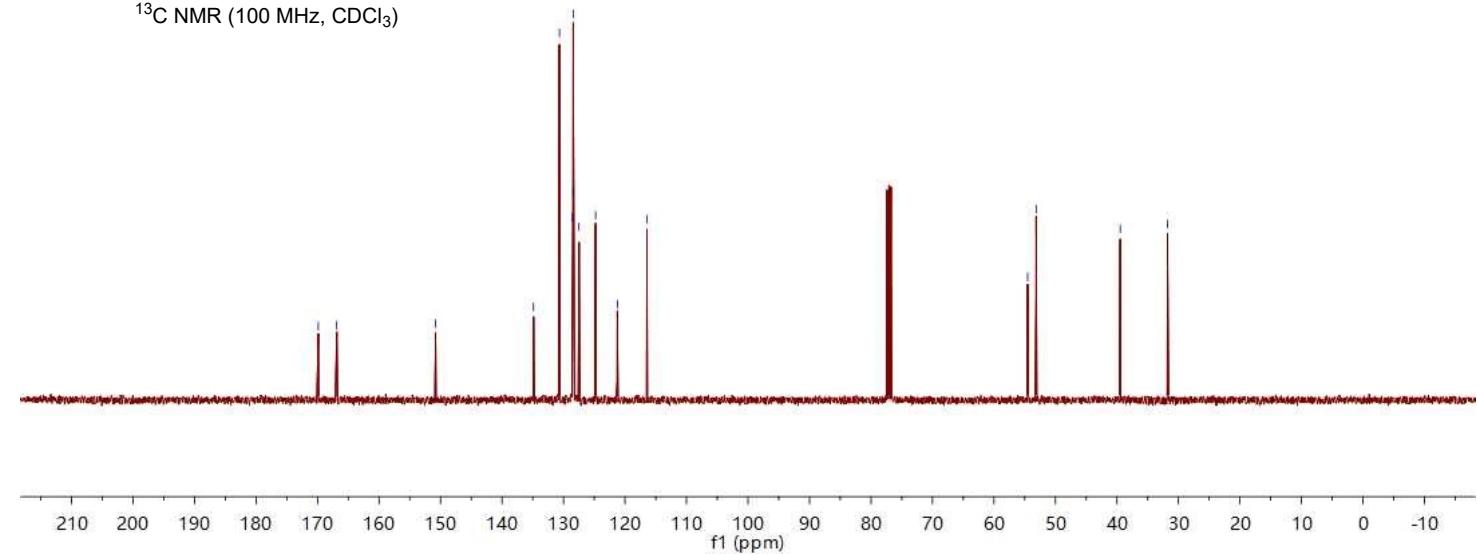


¹³C NMR HW-17-46 in CDCl₃

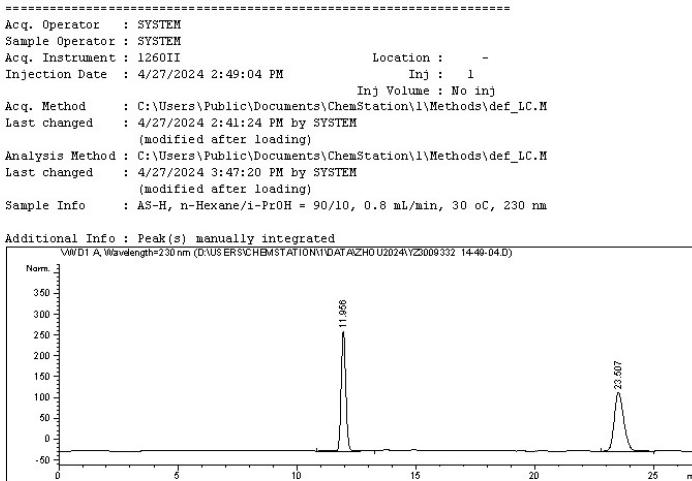


3I'

¹³C NMR (100 MHz, CDCl₃)



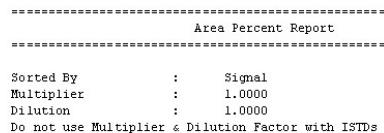
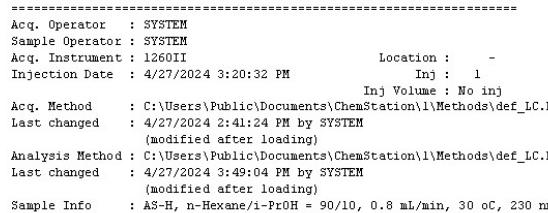
Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2024\YZ3009332 14-49-04.D
Sample Name: HW-17-48SM+/-



1260II 4/27/2024 3:48:26 PM SYSTEM

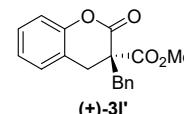
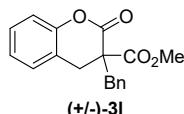
Page 1 of 1

Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2024\YZ3009333 15-20-32.D
Sample Name: HW-17-48SM

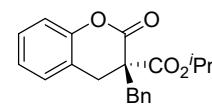


1260II 4/27/2024 3:49:09 PM SYSTEM

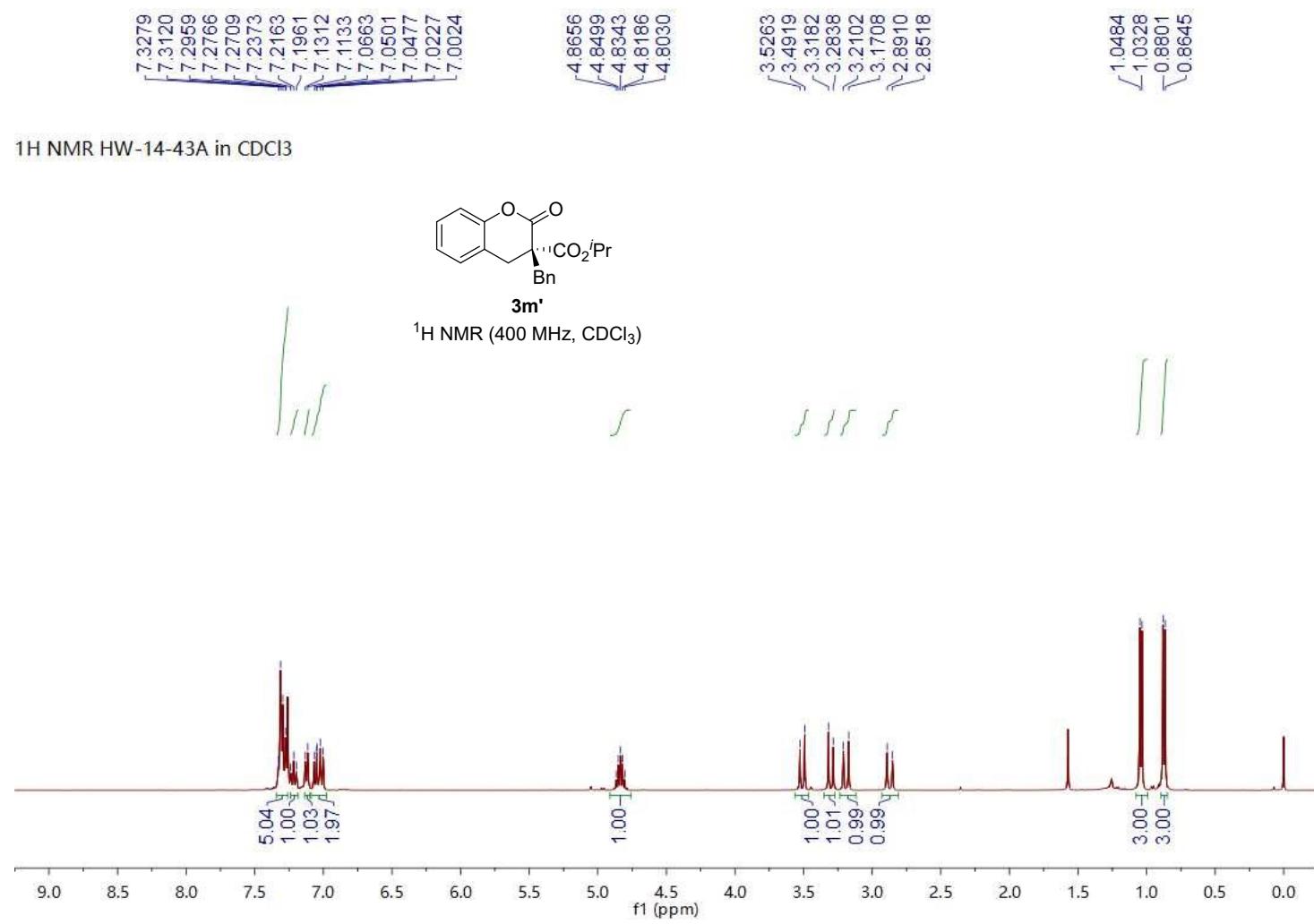
Page 1 of 1



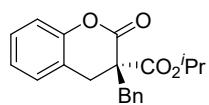
¹H NMR HW-14-43A in CDCl₃



¹H NMR (400 MHz, CDCl₃)

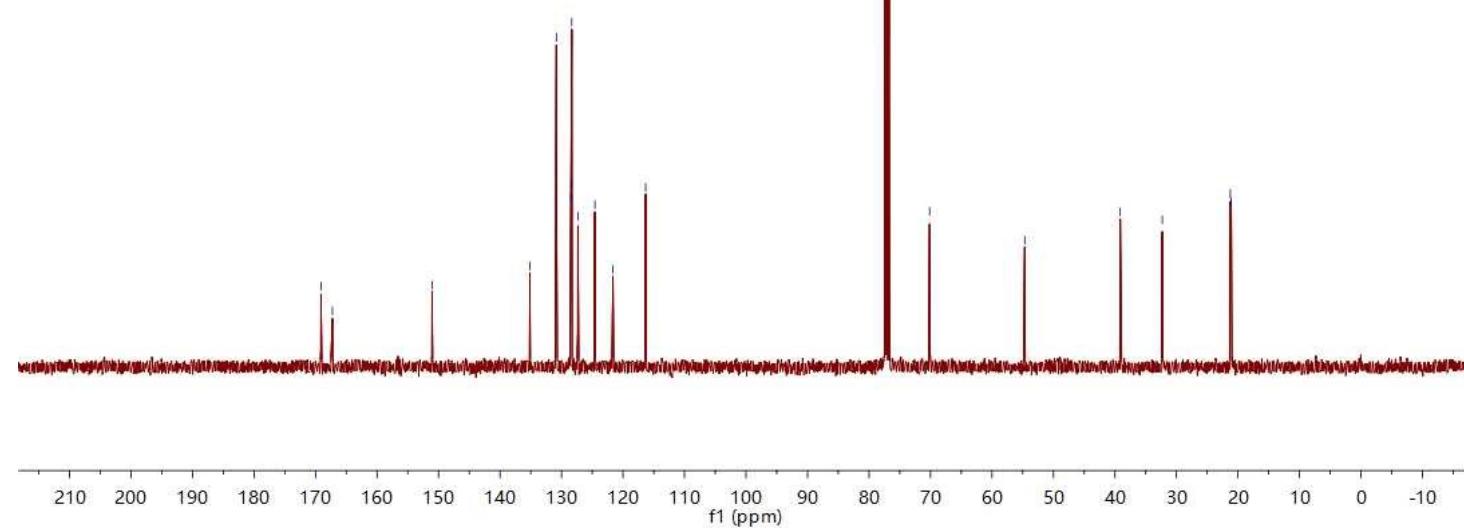


¹³C NMR HW-14-43A TM in CDCl₃



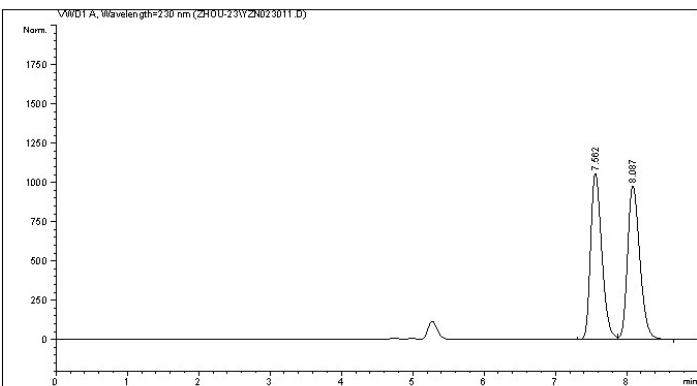
3m'

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023011.D
Sample Name: HW-14-48A SM+/-

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Acq. Operator :                               Location : -
Injection Date : 5/5/2023 4:56:07 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/5/2023 4:55:47 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:49:24 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230nm
```



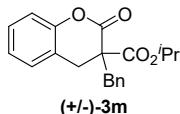
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	7.562	VV	0.1686	1.15752e4	1056.39905	49.6820	
2	8.087	VV	0.1863	1.17234e4	978.81940	50.3180	

Totals : 2.3296e4 2035.21844

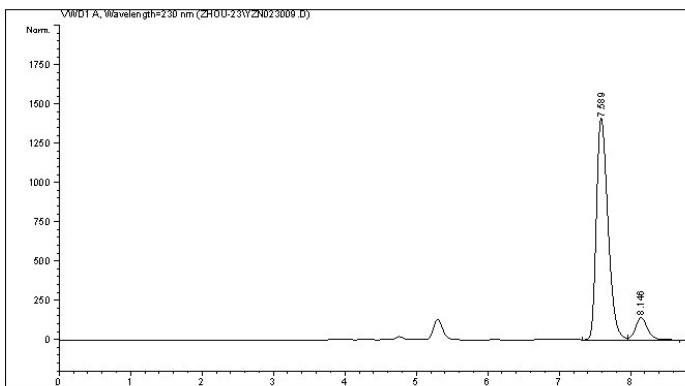


Instrument 1 10/17/2023 9:49:26 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023009.D
Sample Name: HW-14-48A SM

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=====
Acq. Operator :                               Location : -
Injection Date : 5/5/2023 4:23:58 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/5/2023 4:23:20 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:48:45 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230nm
```



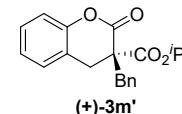
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	7.589	VV	0.1784	1.58565e4	1403.31604	90.3405	
2	8.146	VV	0.1865	1695.42773	141.34738	9.6595	

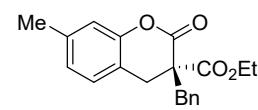
Totals : 1.75519e4 1544.66342



Instrument 1 10/17/2023 9:48:48 AM

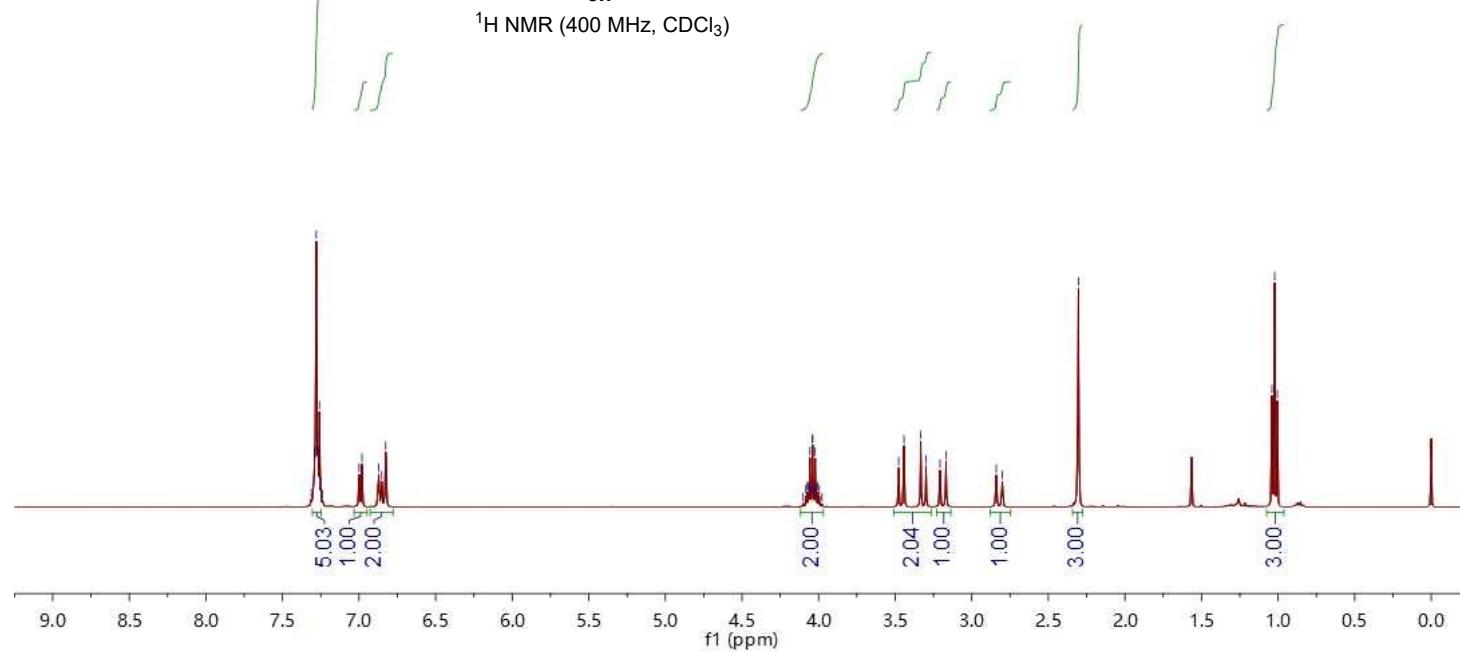
Page 1 of 1

¹H NMR HW-13-94B in CDCl₃

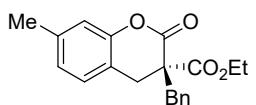


3n'

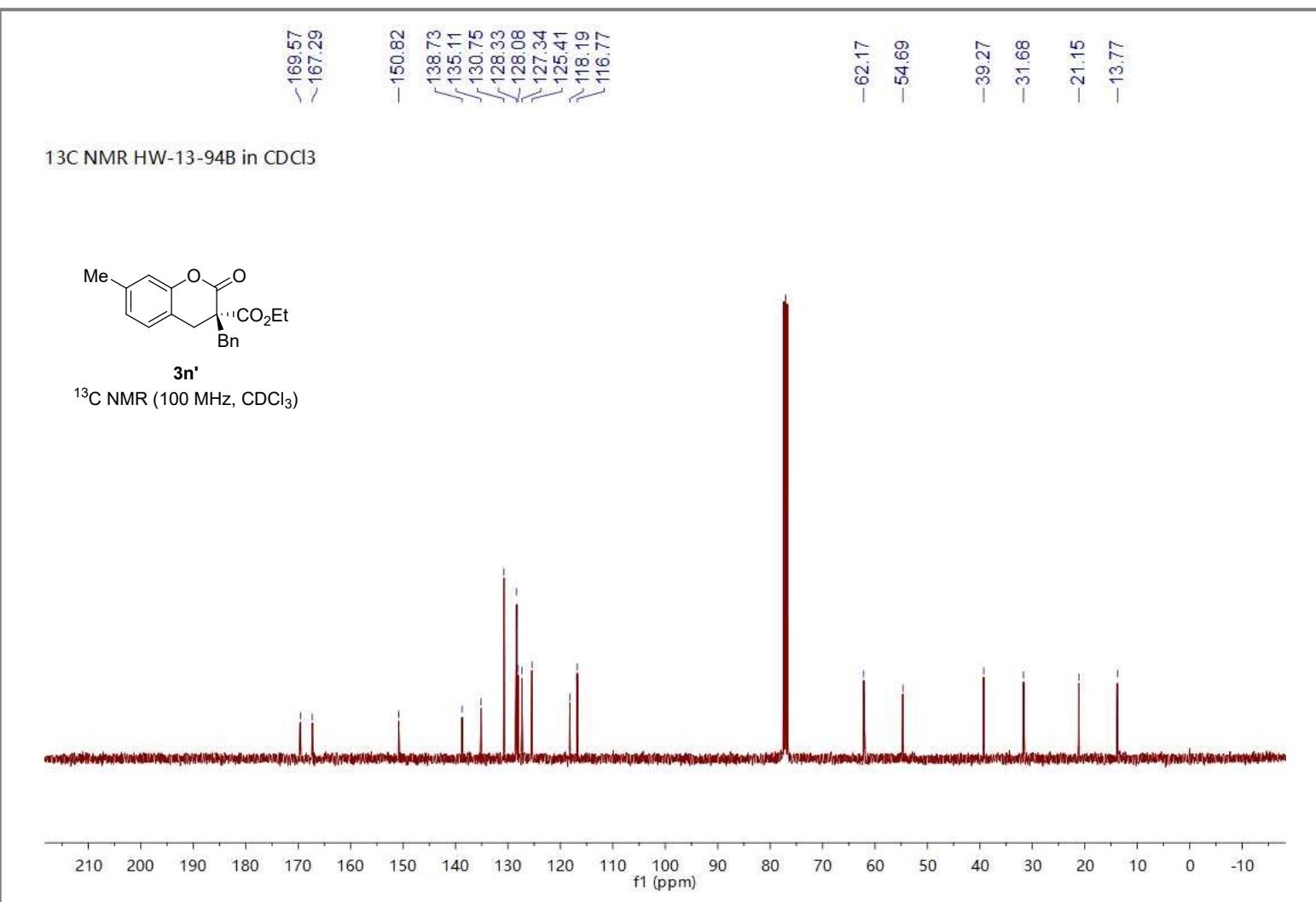
¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-13-94B in CDCl₃



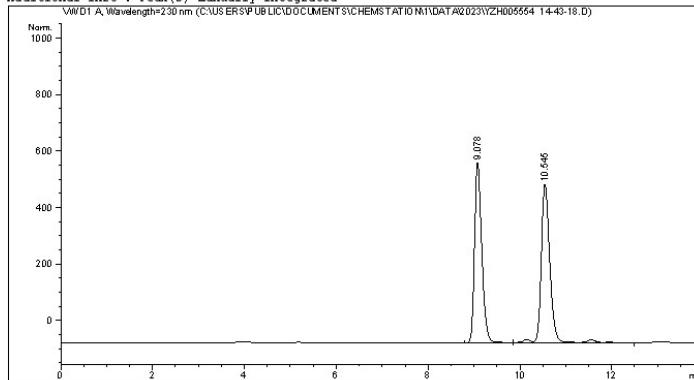
3n'
¹³C NMR (100 MHz, CDCl₃)



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005554 14-43-18.D
Sample Name: HW-13-94B+/-

```
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Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/28/2023 2:43:18 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/28/2023 2:29:47 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 11:00:00 AM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 97/3, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

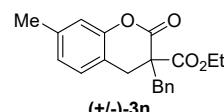


```
=====
Area Percent Report
=====

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU*s]	[mAU]	%
1	BB	0.1677	5274.76465	481.92596	48.8818
2	VV R	0.1919	5516.08203	423.66296	51.1182



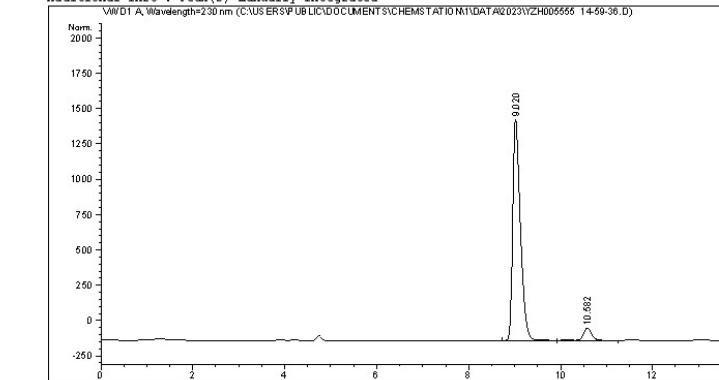
HPLC1260 II 11/29/2023 11:00:04 AM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005555 14-59-36.D
Sample Name: HW-14-12B SM'

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/28/2023 2:59:36 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/28/2023 2:29:47 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 11:01:10 AM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 97/3, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

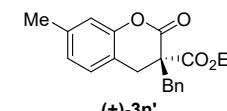


```
=====
Area Percent Report
=====

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

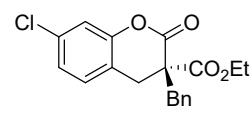
Peak RetTime	Type	Width	Area	Height	Area
# [min]		[min]	[mAU*s]	[mAU]	%
1	BB	0.1740	1.48492e4	1282.79663	94.0383
2	VV R	0.1873	941.38684	72.44398	5.9617



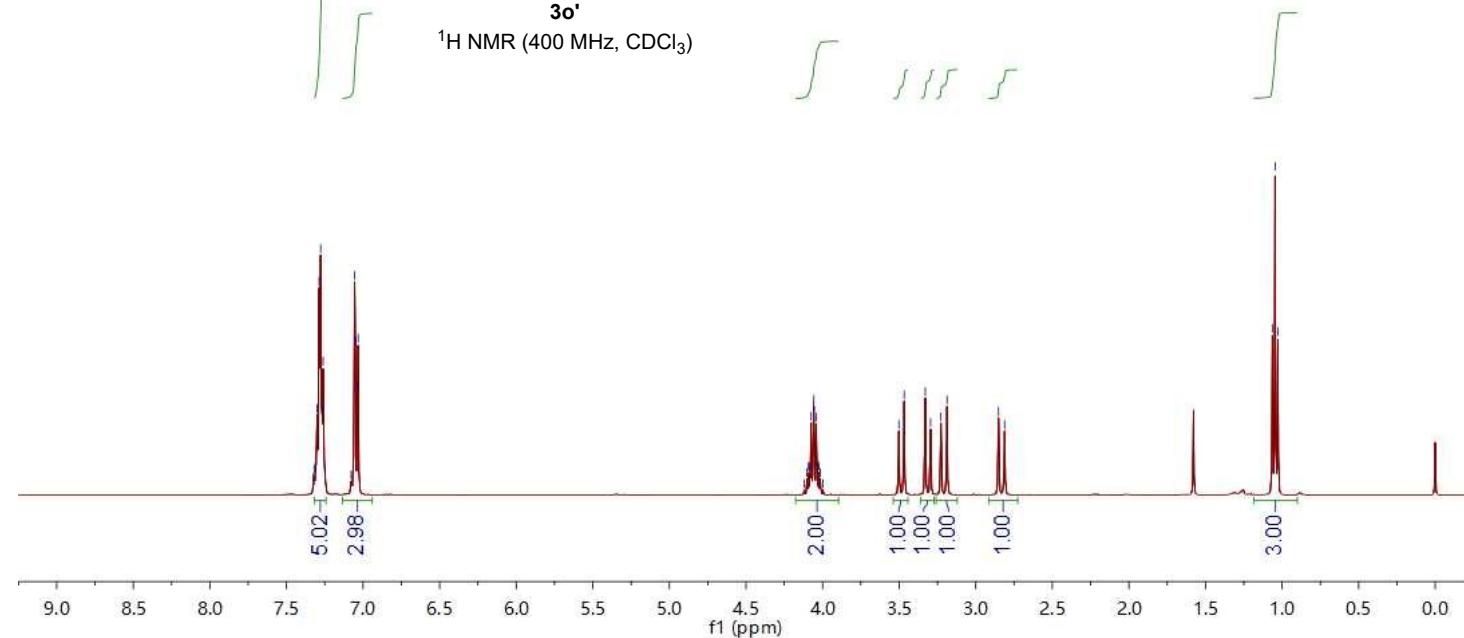
HPLC1260 II 11/29/2023 11:01:17 AM SYSTEM

Page 1 of 2

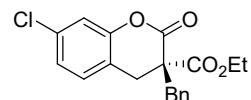
¹H NMR HW-14-46B in CDCl₃



¹H NMR (400 MHz, CDCl₃)

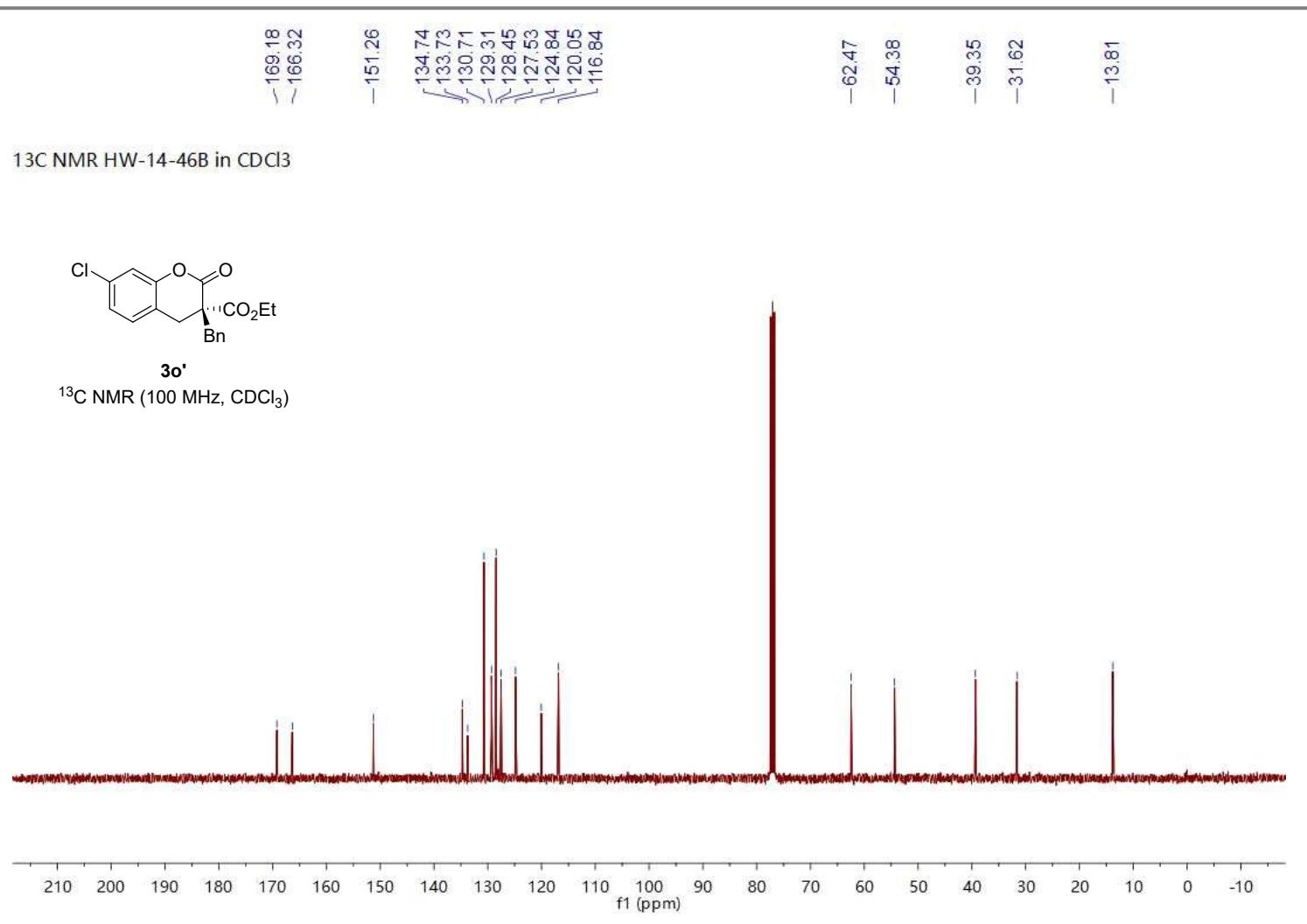


¹³C NMR HW-14-46B in CDCl₃



3o'

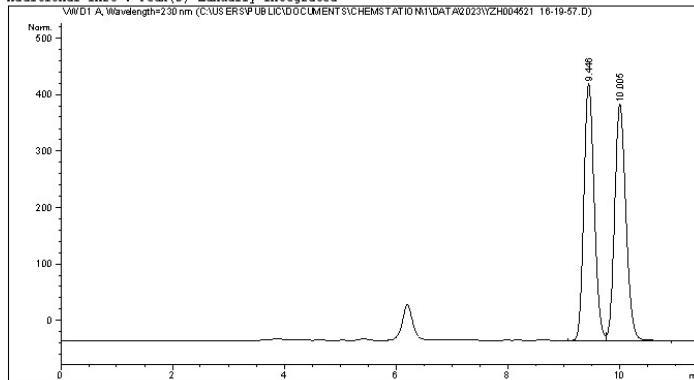
¹³C NMR (100 MHz, CDCl₃)



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH004521 16-19-57.D
Sample Name: HW-14-92SM +/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 7/10/2023 4:19:57 PM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 7/10/2023 4:05:56 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/16/2023 10:14:17 PM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 93/7, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

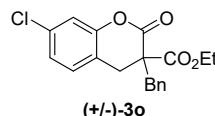


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Area Percent Report

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Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.446	BV	0.1987	4970.99463	390.02411	49.7473
2	10.005	VB	0.2174	5021.50635	358.63422	50.2527



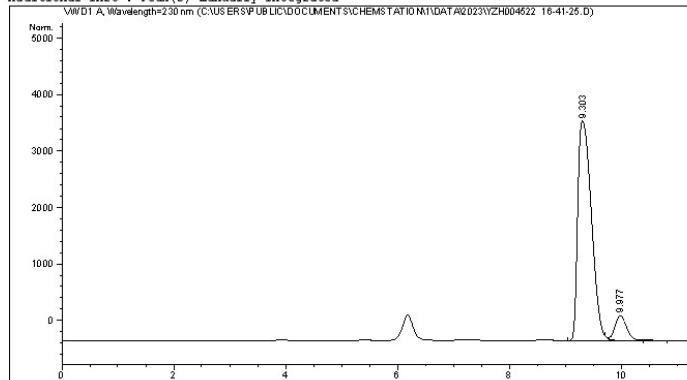
HPLC1260 II 10/16/2023 10:14:21 PM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH004522 16-41-25.D
Sample Name: HW-14-92SM

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 7/10/2023 4:41:25 PM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 7/10/2023 4:05:56 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/16/2023 10:15:20 PM by SYSTEM
                           (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 93/7, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

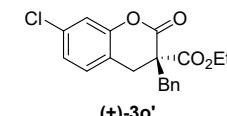


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Area Percent Report

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Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

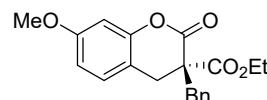
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.303	BV R	0.2700	5.37358e4	3178.20581	91.0710
2	9.977	VV E	0.2255	5268.52686	362.52792	8.9290



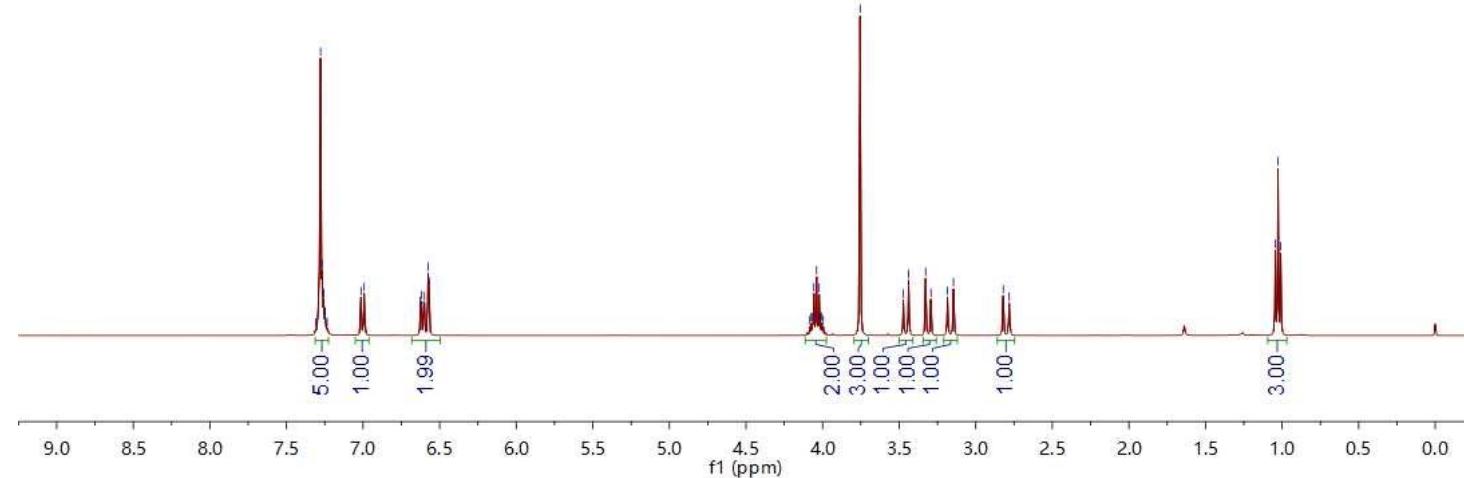
HPLC1260 II 10/16/2023 10:15:24 PM SYSTEM

Page 1 of 2

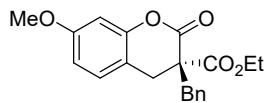
¹H NMR HW-14-64 in CDCl₃



3p'
¹H NMR (400 MHz, CDCl₃)

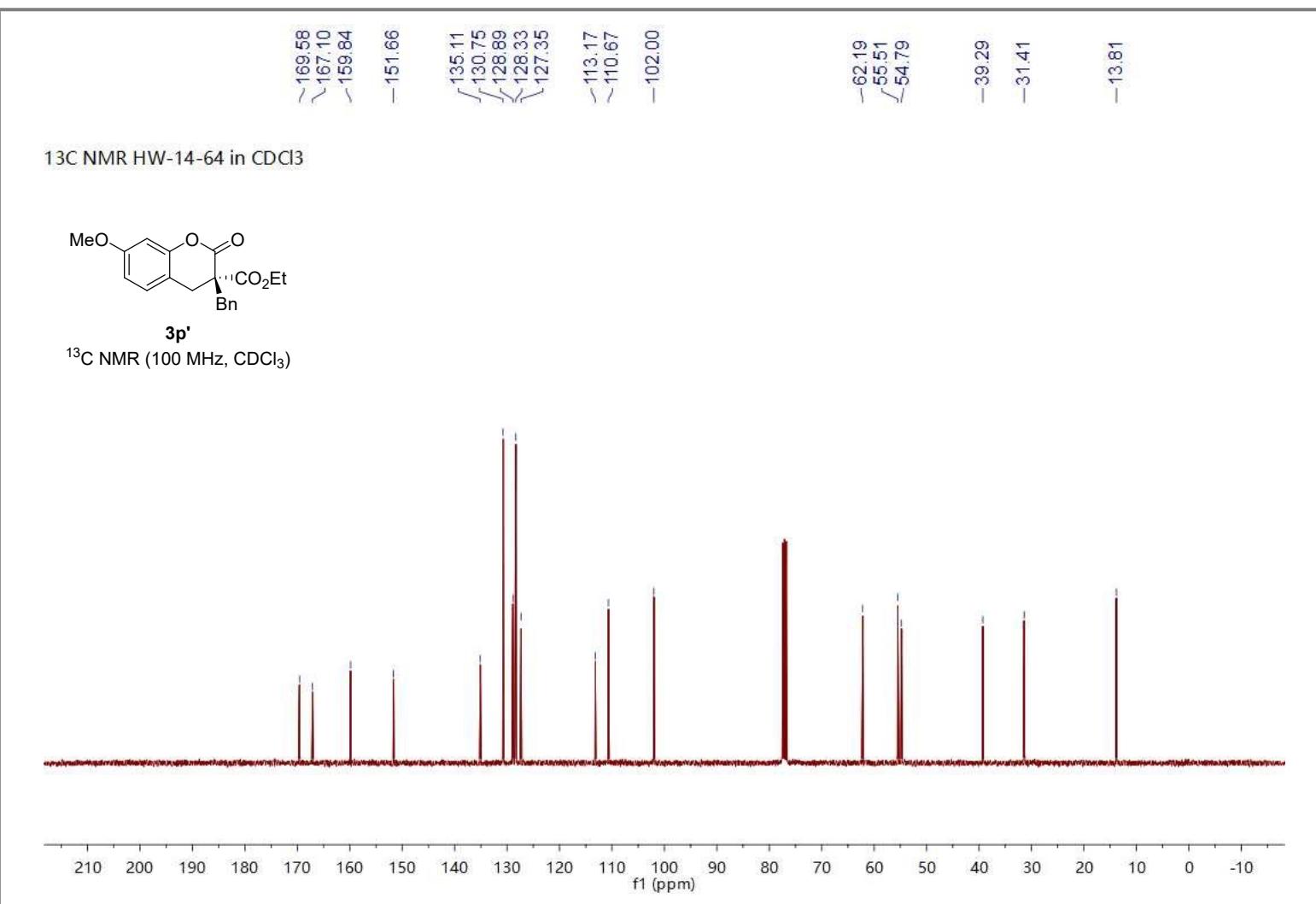


¹³C NMR HW-14-64 in CDCl₃



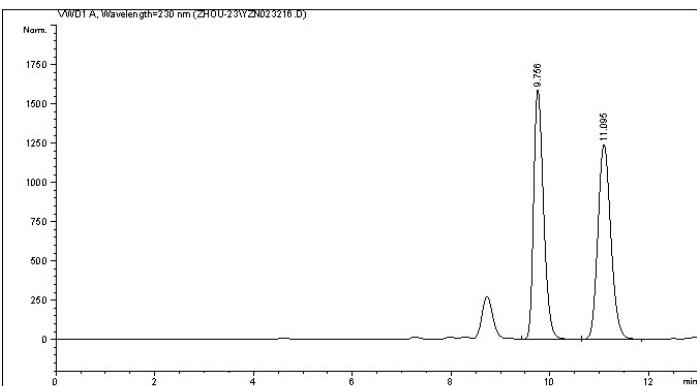
3p'

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023216.D
Sample Name: HW-14-73A SM+/-

```
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Acq. Operator : Instrument 1 Location : -
Injection Date : 5/30/2023 8:44:18 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/30/2023 8:35:12 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:08:16 AM
(modified after loading)
Sample Info : od-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

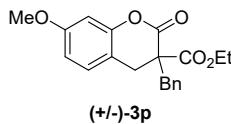


=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	9.756	VV	0.2160	2.2283e4	1589.47205	49.4825	
2	11.095	VV	0.2899	2.2749e4	1238.88599	50.5175	
Totals :				4.50324e4	2828.35803		



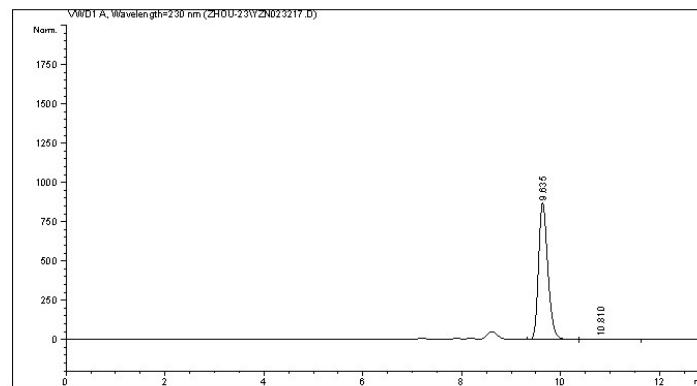
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*** End of Report ***
=====
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Instrument 1 10/17/2023 10:08:19 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023217.D
Sample Name: HW-14-73A SM

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 5/30/2023 9:17:42 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/30/2023 9:17:10 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 10:09:09 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

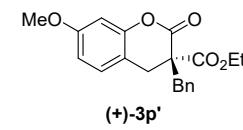


=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	9.635	VV	0.2048	1.13250e4	867.21967	98.9115	
2	10.810	VV	0.4614	124.63101	4.14512	1.0885	
Totals :				1.14497e4	871.36478		



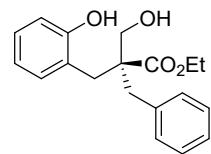
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*** End of Report ***
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Instrument 1 10/17/2023 10:09:12 AM

Page 1 of 1

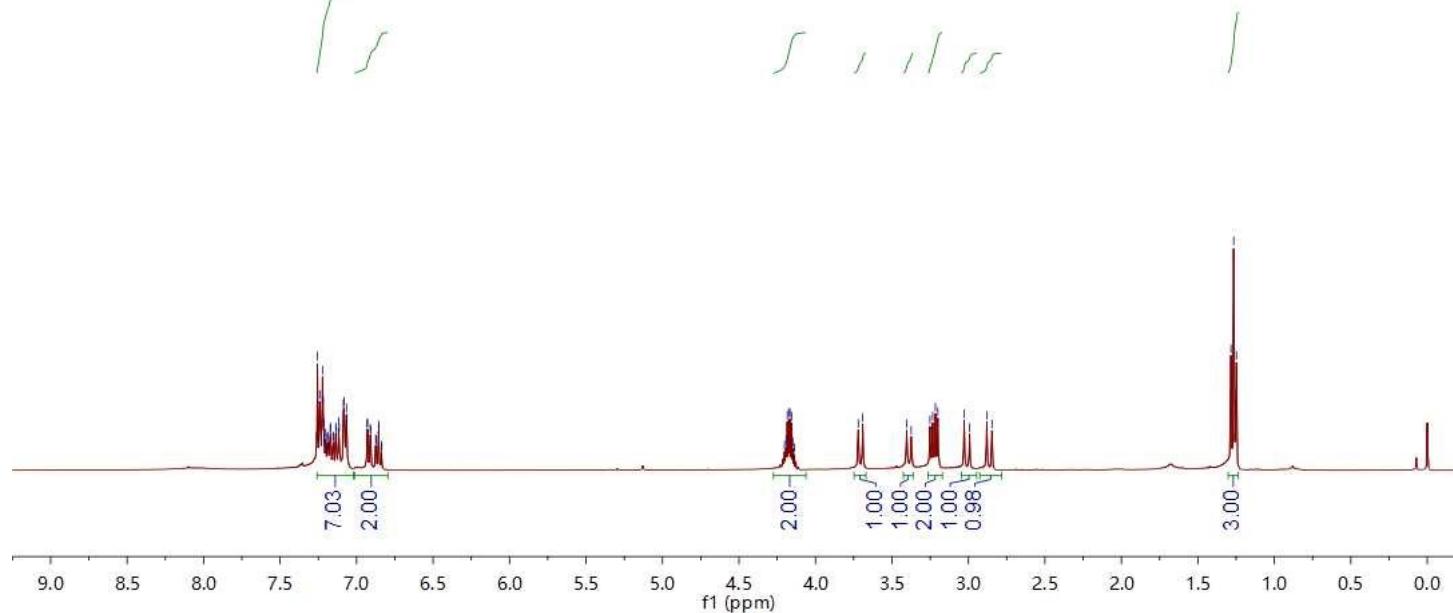
7.2561
7.2408
7.2227
7.2183
7.2141
7.2027
7.1884
7.1722
7.1703
7.1501
7.1362
7.1323
7.1172
7.1133
7.0859
7.0814
7.0661
7.0628
6.9311
6.9284
6.9109
6.9082
6.8565
6.8537
4.9821
4.1921
4.1841
4.1740
4.1662
4.1562
4.1480
4.1384
3.7201
3.6909
3.4035
3.3742
3.2512
3.2336
3.2159
3.1999
3.0277
2.9924
2.8798
2.8461

¹H NMR HW-14-8A TM in CDCl₃

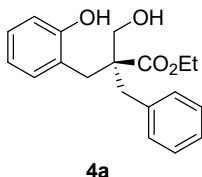


4a

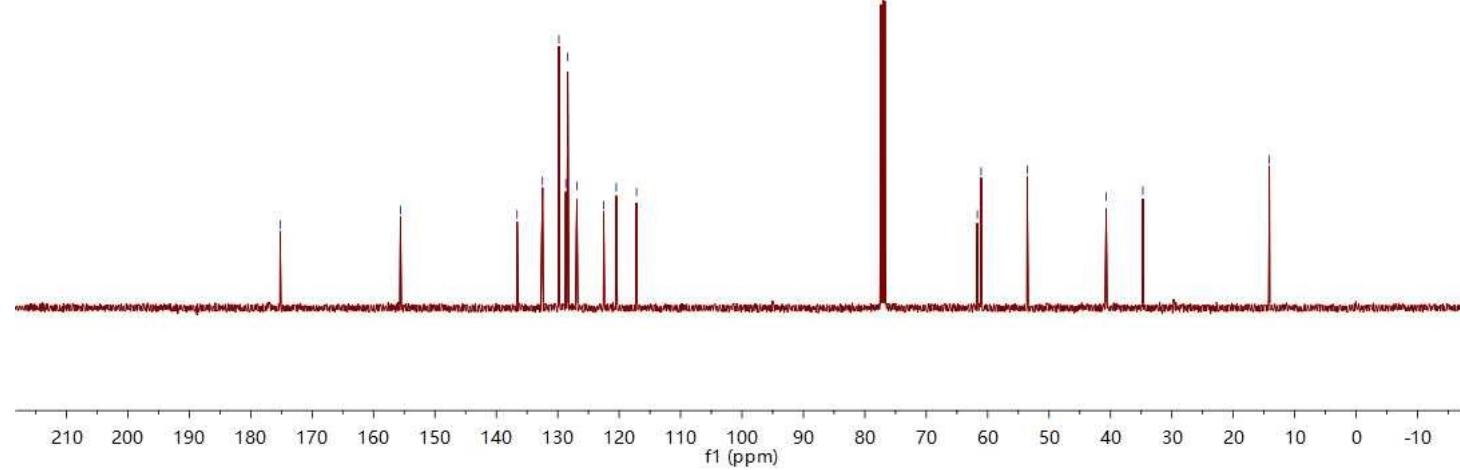
¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-14-8A TM in CDCl₃

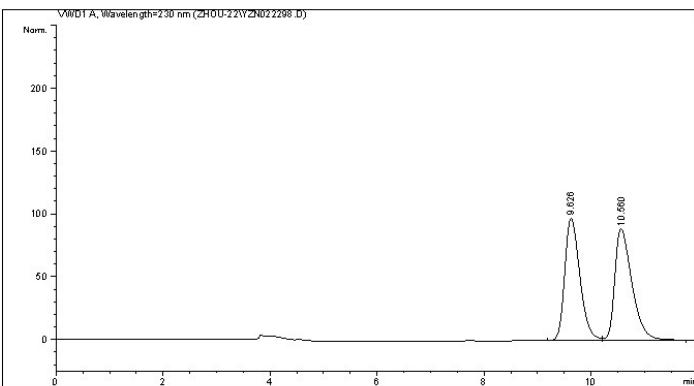


¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022298.D
Sample Name: HW-12-84+-

```
=====
Acq. Operator : Instrument 1 Location :
Injection Date : 12/30/2022 10:22:40 PM
Acq. Method : C:\CHEM32\1\METHODS\def_LC.M
Last changed : 12/30/2022 10:22:16 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\def_LC.M
Last changed : 10/17/2023 8:35:45 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



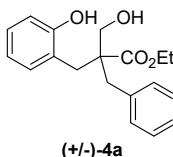
```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

#	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.626	BV	0.3044	1919.77893	97.30987	49.1682
2	10.560	VB	0.3395	1984.73413	88.77587	50.8318

Totals : 3904.51306 186.08574



(±)-4a

```
=====
*** End of Report ***
```

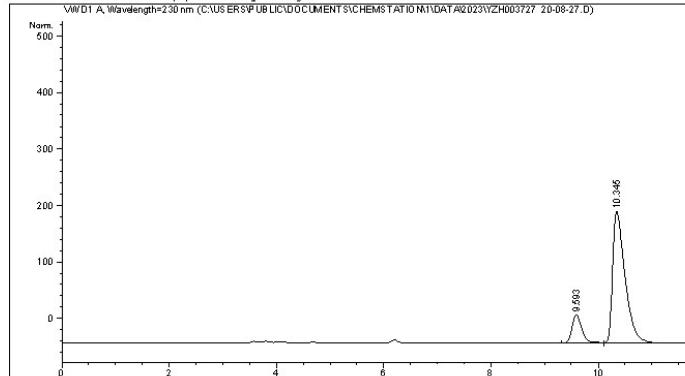
Instrument 1 10/17/2023 8:35:50 AM

Page 1 of 1

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YHZ003727 20-08-27.D
Sample Name: HW-14-8A TM

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II Location : 1
Injection Date : 3/22/2023 8:08:27 PM Inj : 1
Inj Volume : No inj
Acq. Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed : 3/22/2023 7:43:25 PM by SYSTEM
(modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed : 10/16/2023 8:57:56 PM by SYSTEM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

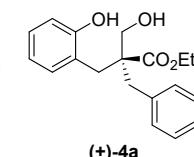


```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Sample Amount: : 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

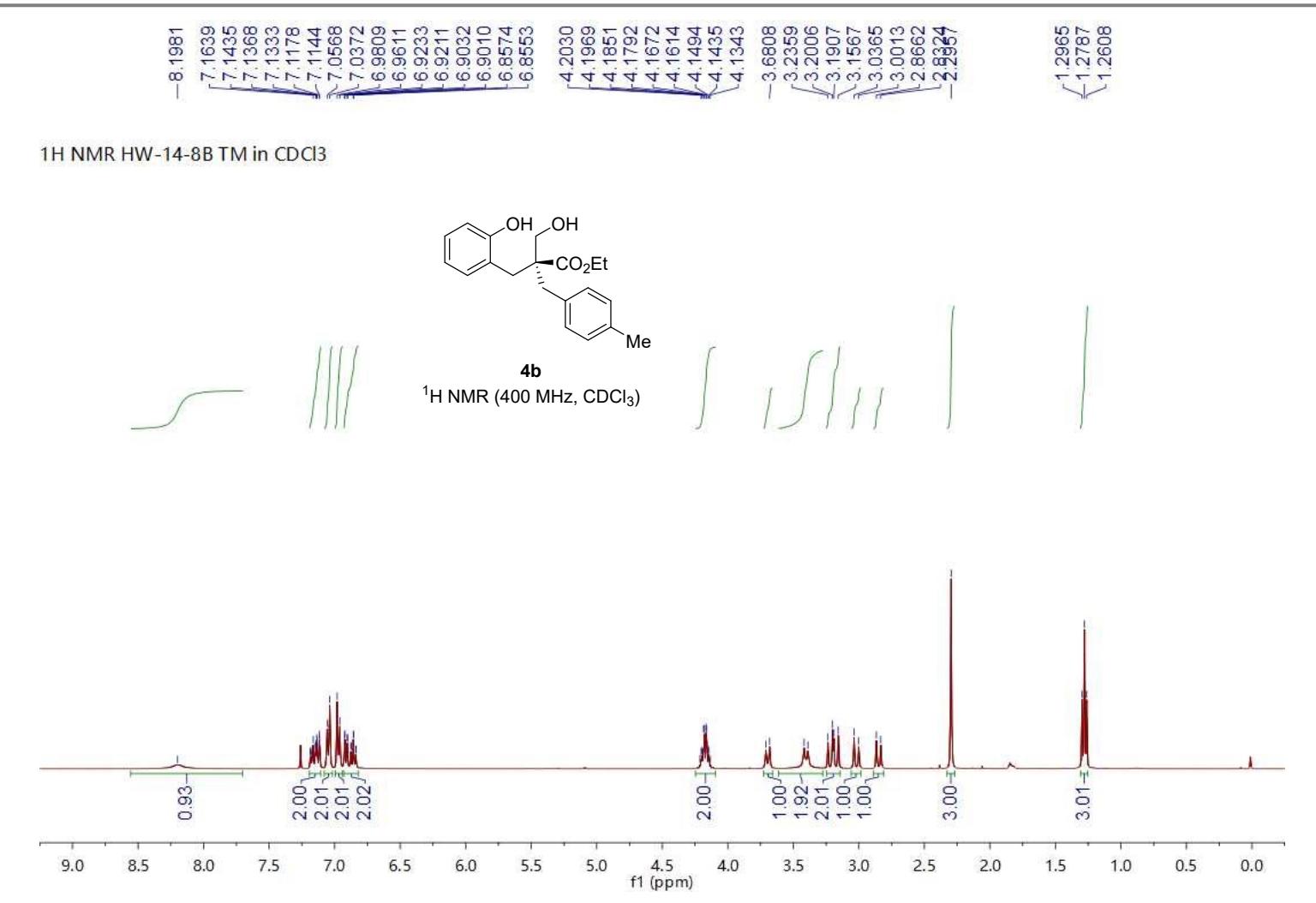
#	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.593	BV	0.1944	404.16336	31.55659	14.6134
2	10.345	VB	0.2394	2361.54614	146.99408	85.3866

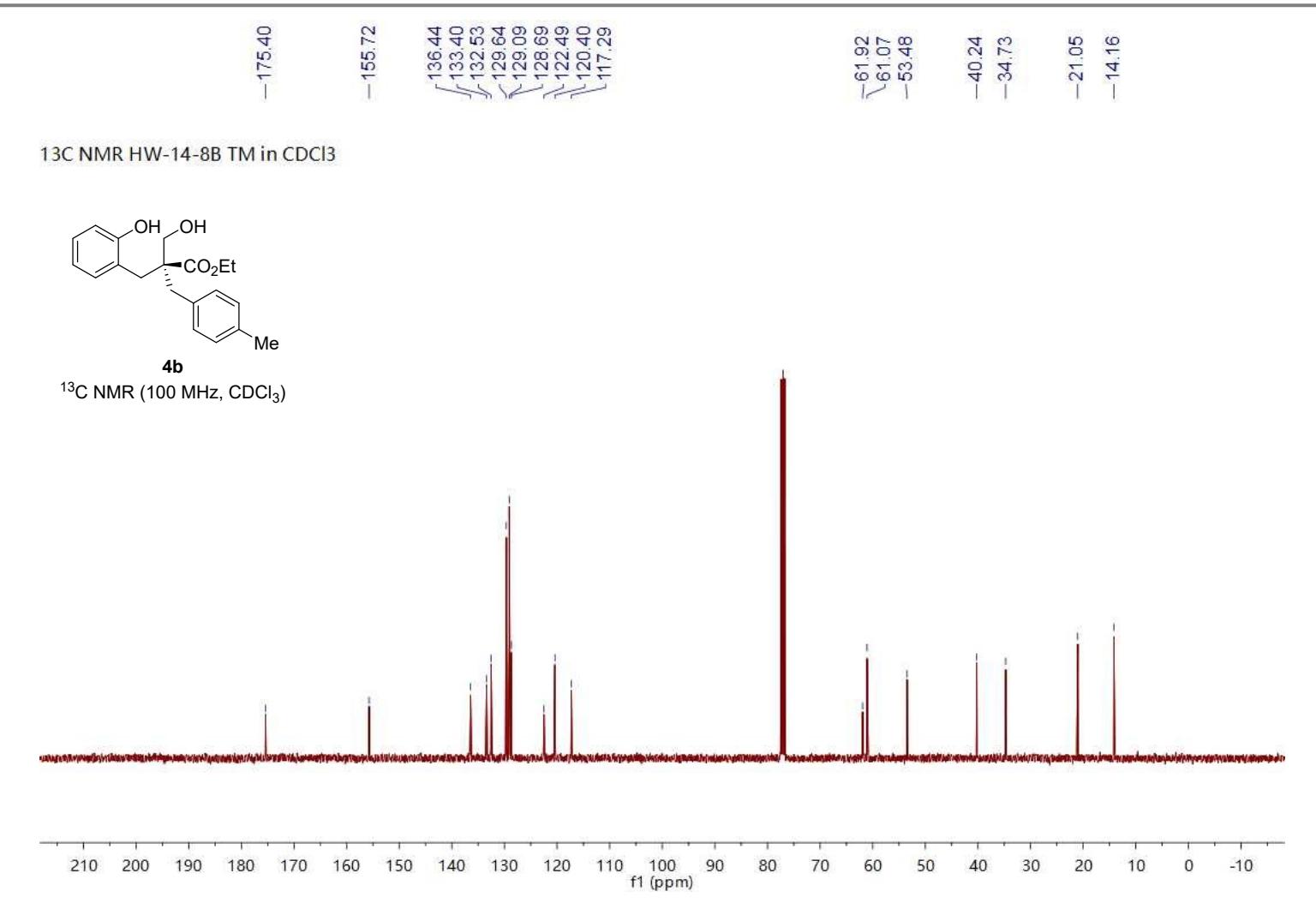


(+)-4a

HPLC1260 II 10/16/2023 8:58:00 PM SYSTEM

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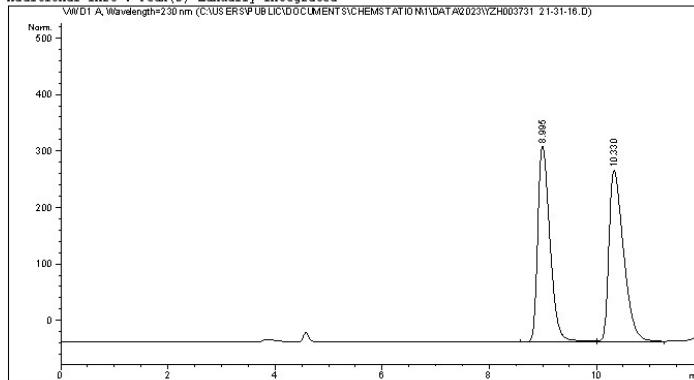




Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH003731 21-31-16.D
Sample Name: HW-14-8C TM+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 3/22/2023 9:31:16 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 3/22/2023 7:43:25 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/16/2023 9:05:43 PM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

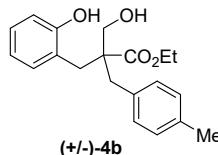


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: : 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.995	BV R	0.2435	4275.72021	271.88998	48.8896
2	10.330	VB	0.2896	4469.95020	237.51076	51.1104



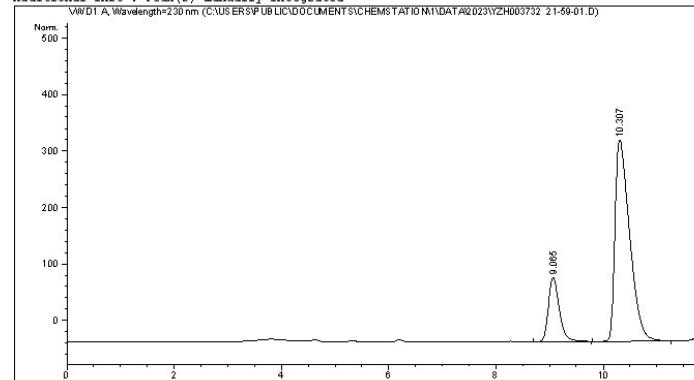
HPLC1260 II 10/16/2023 9:05:48 PM SYSTEM

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Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH003732 21-59-01.D
Sample Name: HW-14-8C TM

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 3/22/2023 9:59:02 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 3/22/2023 7:43:25 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/16/2023 9:04:43 PM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

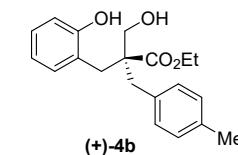


=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: : 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

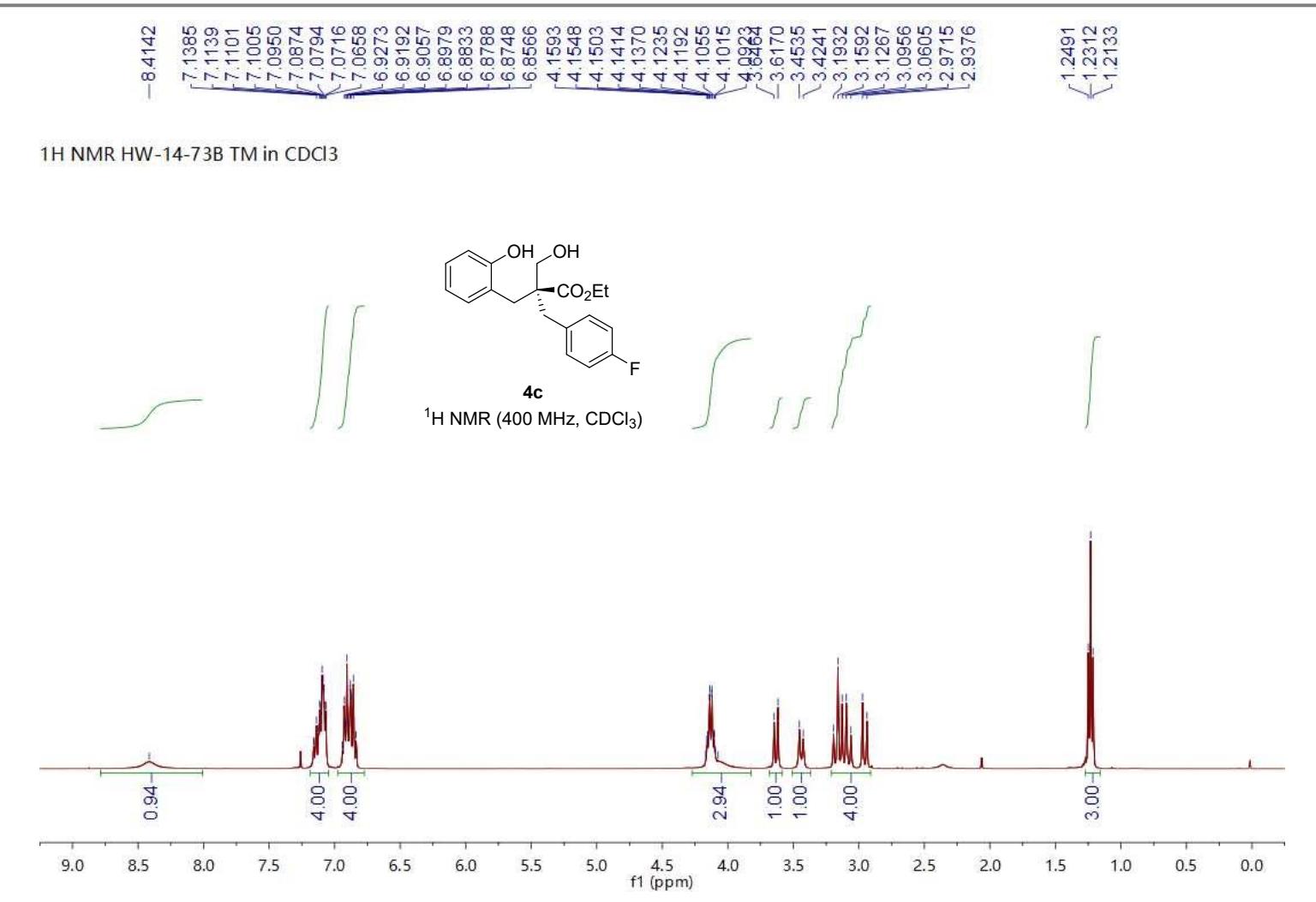
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.065	BB	0.2145	1265.45081	90.30990	19.1985
2	10.307	BB	0.2875	5325.93701	283.06808	80.8015



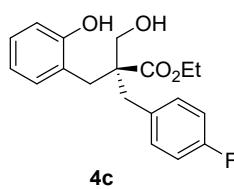
HPLC1260 II 10/16/2023 9:04:46 PM SYSTEM

Page 1 of 2

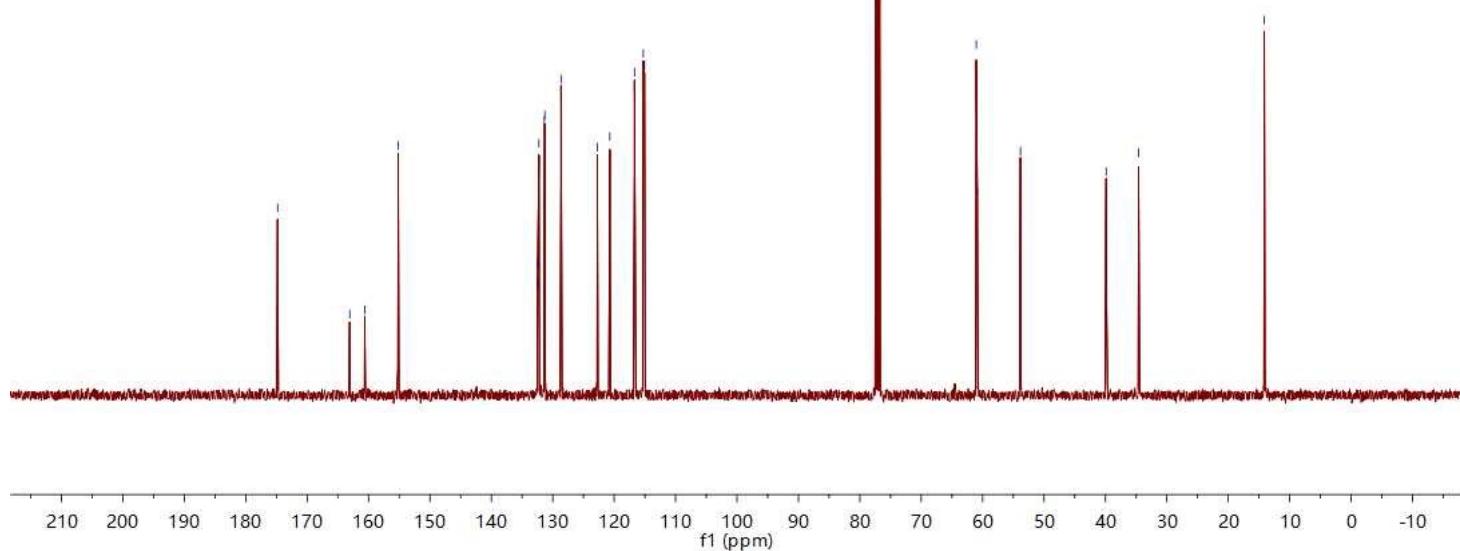


-174.80
-163.06
~160.63
-155.17
132.45
132.42
132.25
131.37
131.30
128.66
122.75
120.71
116.69
115.27
115.06
61.03
60.86
-53.83
-39.88
-34.60
-14.13

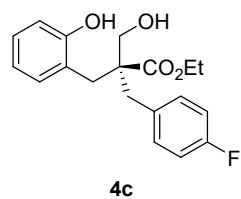
¹³C NMR HW-14-73B TM in CDCl₃



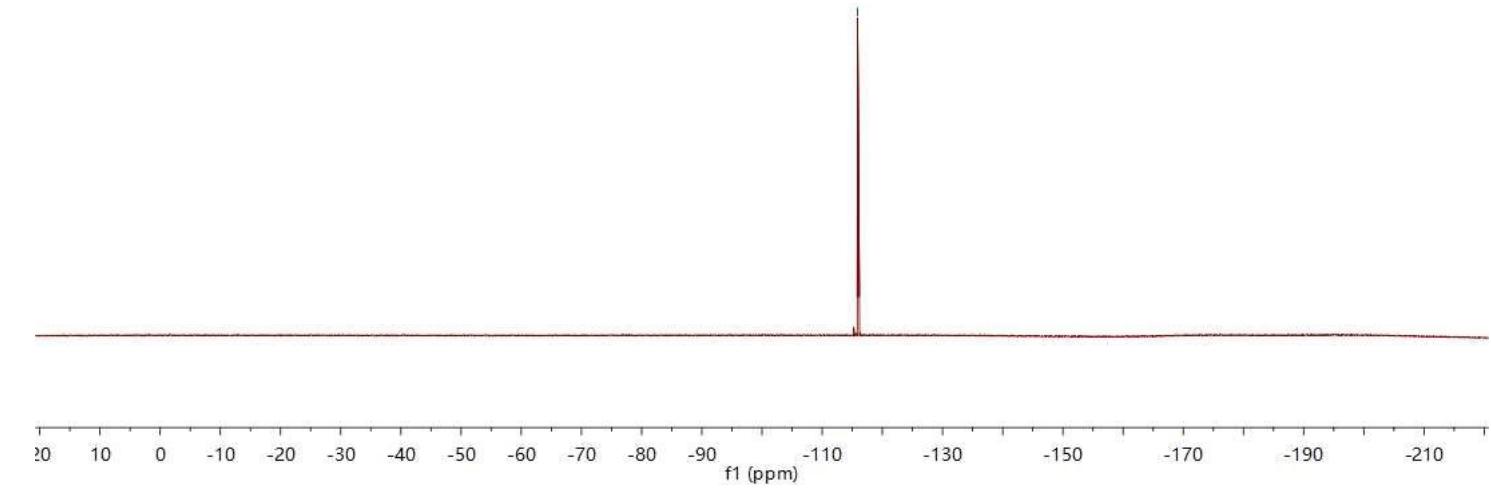
¹³C NMR (100 MHz, CDCl₃)



¹⁹F NMR HW-14-73B in CDCl₃

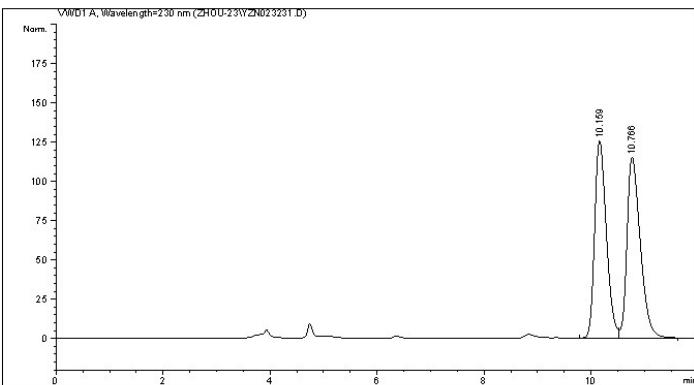


4c
¹⁹F NMR (376 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023231.D
Sample Name: HW-14-73CTH+/-

=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/31/2023 5:07:35 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/31/2023 5:07:01 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:00:02 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm

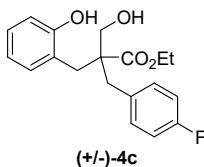


=====
Area Percent Report

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

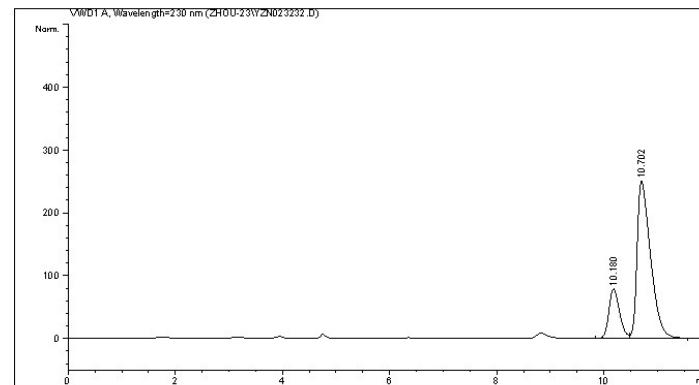
Signal 1: VWD1 A, Wavelength=230 nm

#	Peak RetTime	Type	Width	Area	Height	Area %
	[min]		[min]	[mAU]	*s	[mAU]
1	10.159	BV	0.2354	1904.47192	125.44061	48.2206
2	10.766	BV	0.2642	1988.51709	115.10017	51.0794
Totals :				3892.98901	240.54079	



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023232.D
Sample Name: HW-14-73CTH

=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/31/2023 5:22:40 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/31/2023 5:22:17 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:01:10 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm

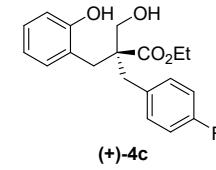


=====
Area Percent Report

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=230 nm

#	Peak RetTime	Type	Width	Area	Height	Area %
	[min]		[min]	[mAU]	*s	[mAU]
1	10.159	BV	0.2112	1080.59412	78.68358	19.9336
2	10.702	BV	0.2638	4340.38330	249.85710	80.0664
Totals :				5420.97742	328.54068	

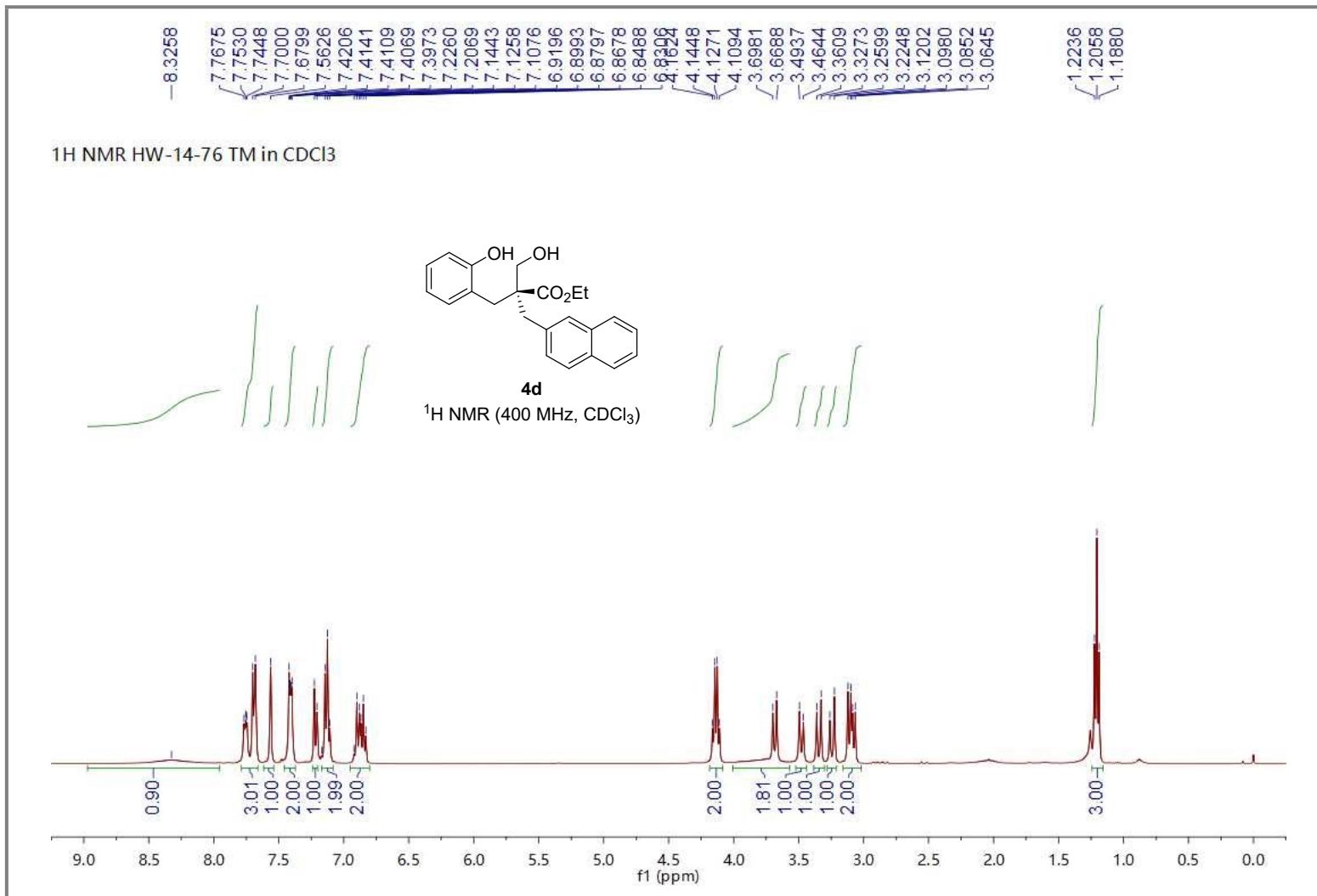


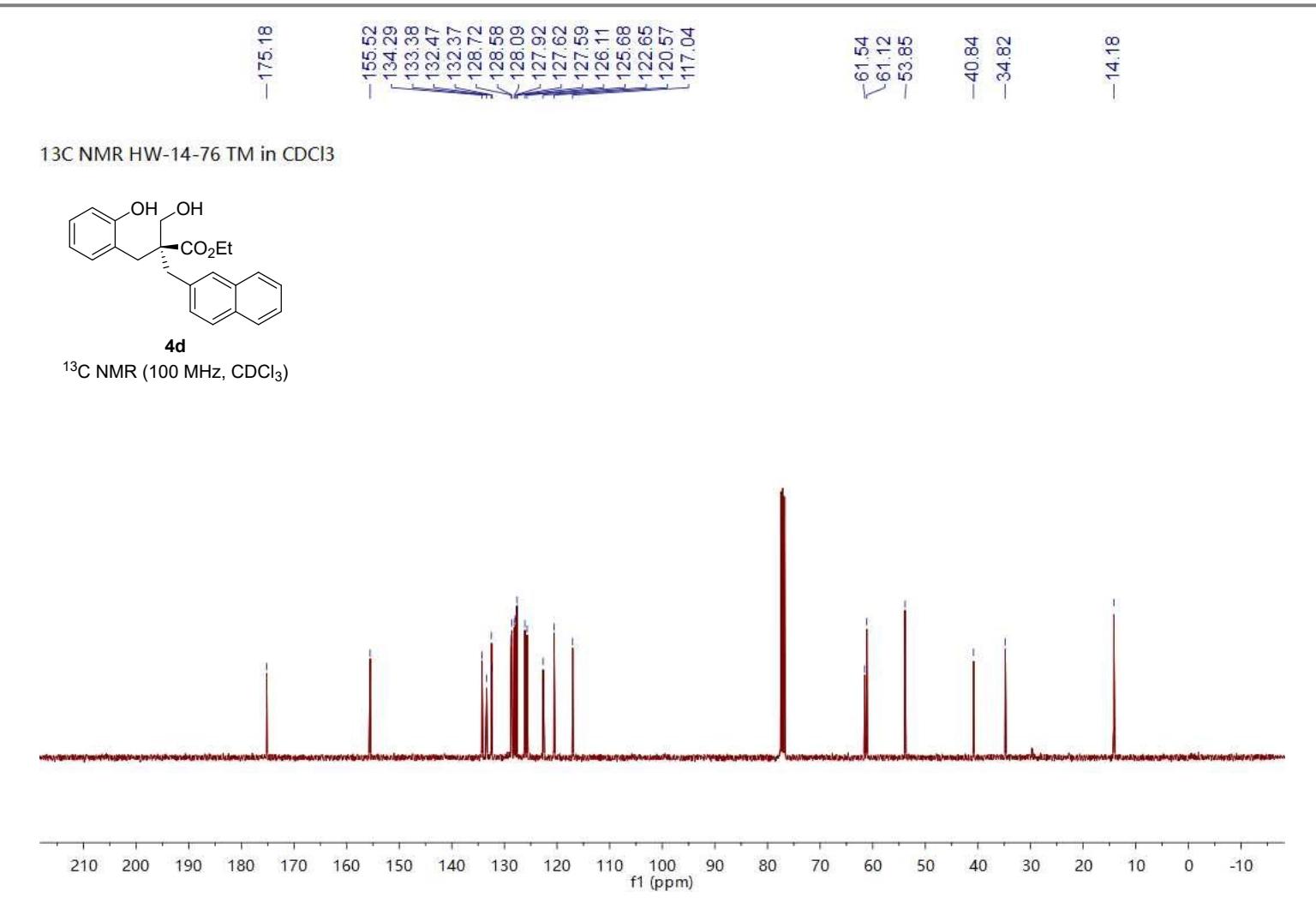
Instrument 1 10/17/2023 9:00:10 AM

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Instrument 1 10/17/2023 9:01:14 AM

Page 1 of 1

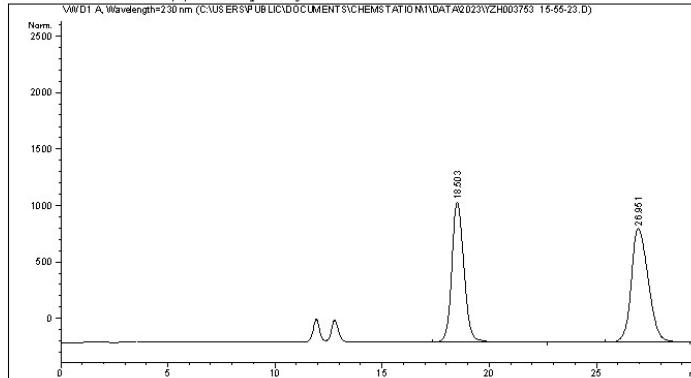




Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH003753 15-55-23.D
Sample Name: HW-14-8C TM+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 3/24/2023 3:55:23 PM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed  : 3/24/2023 2:09:49 PM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed  : 10/16/2023 9:21:23 PM by SYSTEM
                           (modified after loading)
Sample Info    : IA, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



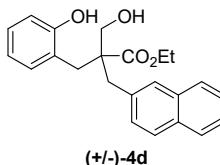
=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.503	BB	0.6219	3.2091e4	812.29633	46.9001
2	26.951	BB	0.8646	3.63340e4	662.75378	53.0999

HPLC1260 II 10/16/2023 9:21:27 PM SYSTEM



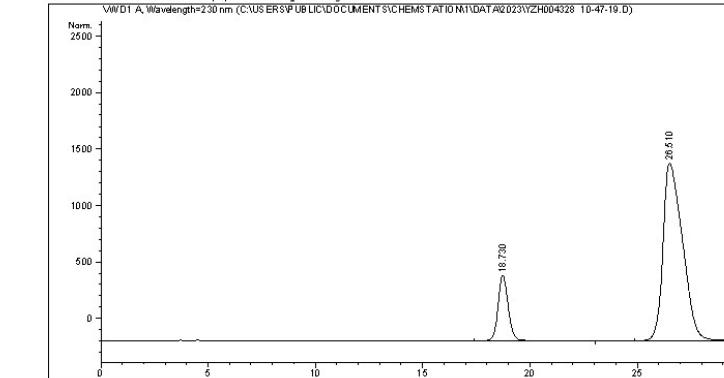
(+/-)-4d

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH004328 10-47-19.D
Sample Name: HW-14-76TM

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 6/6/2023 10:47:19 AM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed  : 6/6/2023 10:41:04 AM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed  : 10/16/2023 9:20:30 PM by SYSTEM
                           (modified after loading)
Sample Info    : IA, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



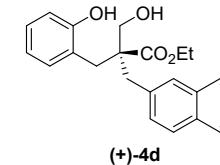
=====
Area Percent Report

```
Sorted By       : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

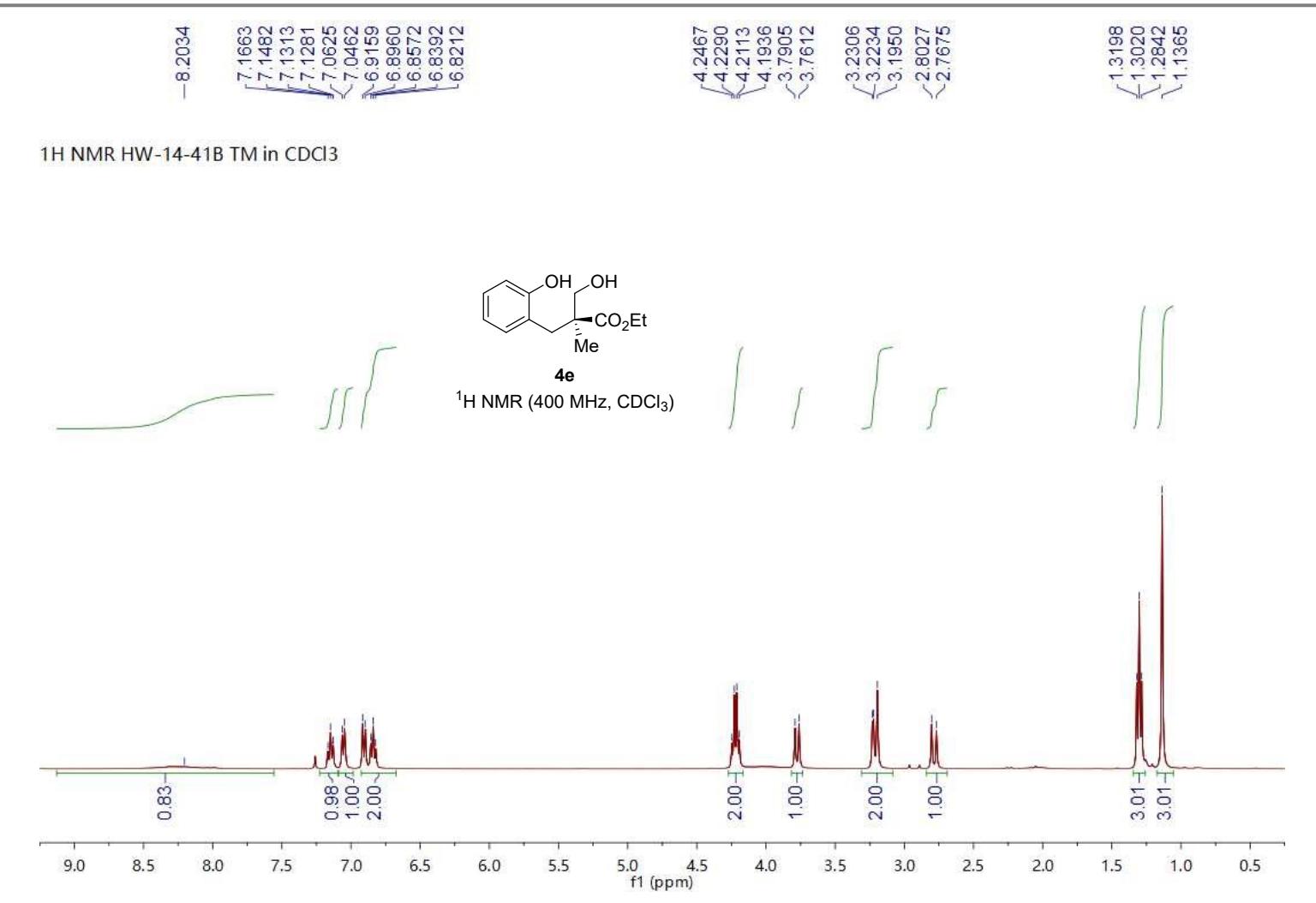
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.730	BB	0.5175	1.46792e4	437.40421	16.4315
2	26.510	BB	1.0464	7.46569e4	1182.81482	83.5685

HPLC1260 II 10/16/2023 9:20:35 PM SYSTEM



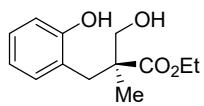
(+)-4d

Page 1 of 2



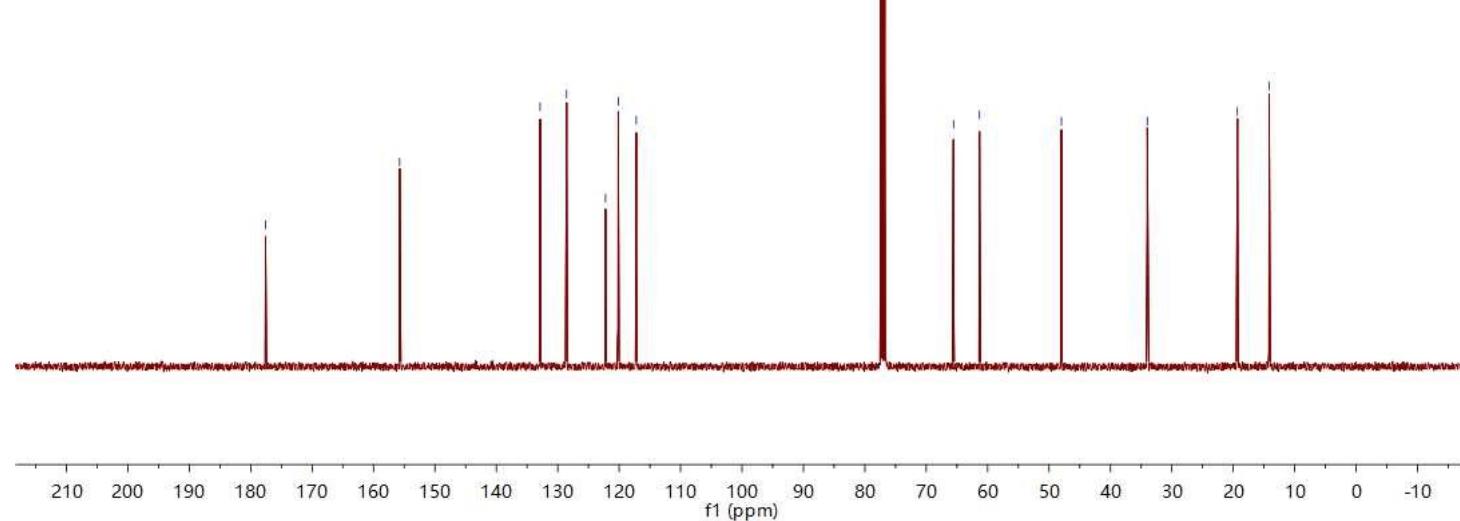
—177.58 —155.75
—132.87 —128.56
—122.23 —120.16
—117.23

¹³C NMR HW-14-41B TM in CDCl₃



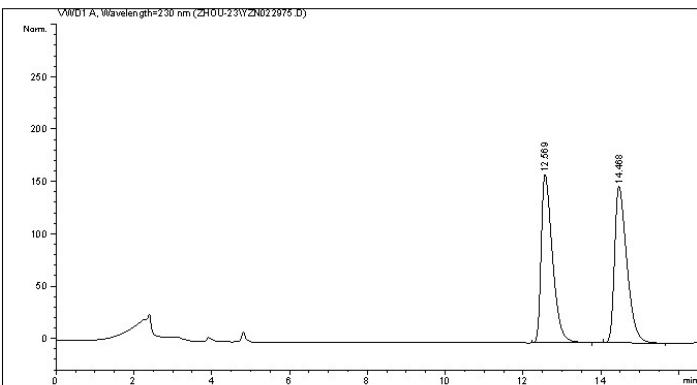
4e

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022975.D
Sample Name: HW-14-41B TM+/-

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Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/26/2023 4:19:55 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:17:42 AM
                                         (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



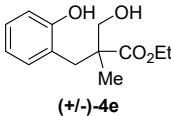
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=====
Area Percent Report
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```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak	RetTime	Type	Width	Area	Height	Area
#	[min]		[min]	[mAU]	*s	[mAU]
1	12.569	BB	0.3056	3226.27905	160.67441	49.6838
2	14.468	BB	0.3342	3267.35059	149.16887	50.3162

Totals : 6493.62964 309.84328

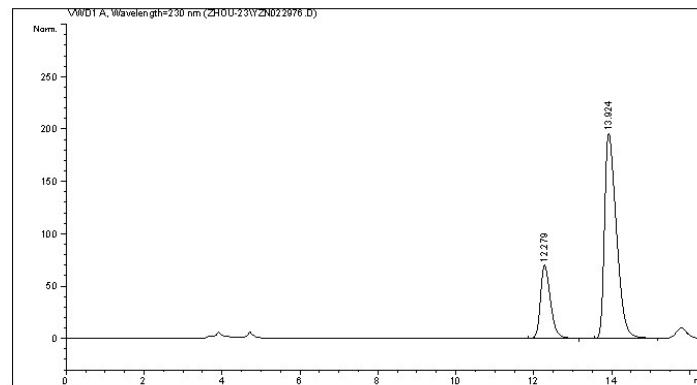


Instrument 1 10/17/2023 9:17:46 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022976.D
Sample Name: HW-14-41B TM

```
=====
Acq. Operator :                               Location : -
Injection Date : 4/26/2023 4:42:32 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 4/26/2023 4:41:54 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:17:42 AM
                                         (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



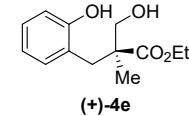
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=====
Area Percent Report
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```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

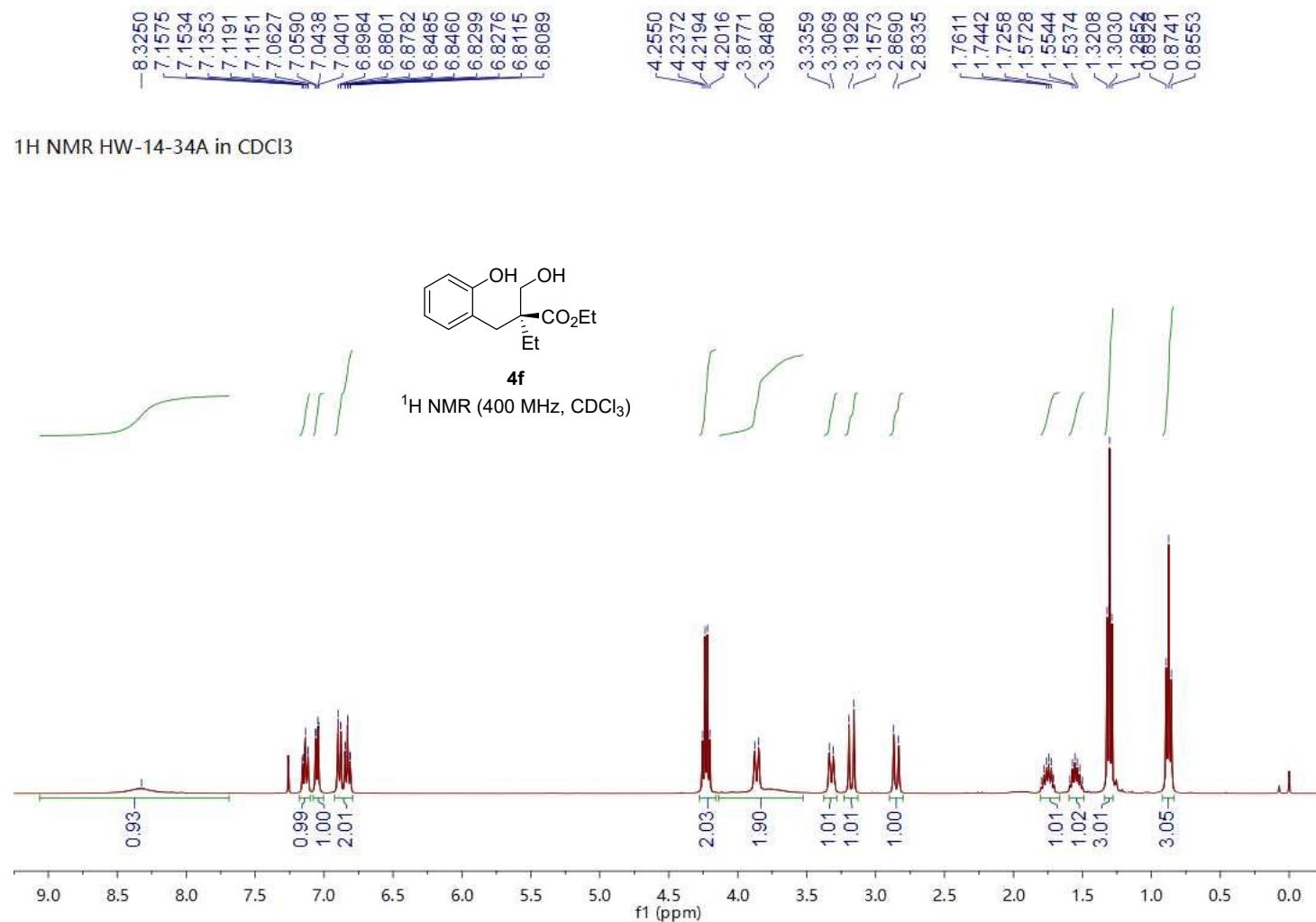
Peak	RetTime	Type	Width	Area	Height	Area
#	[min]		[min]	[mAU]	*s	[mAU]
1	12.279	BB	0.2725	1238.63635	69.80949	23.0816
2	13.924	BB	0.3279	4127.69531	195.49022	76.9184

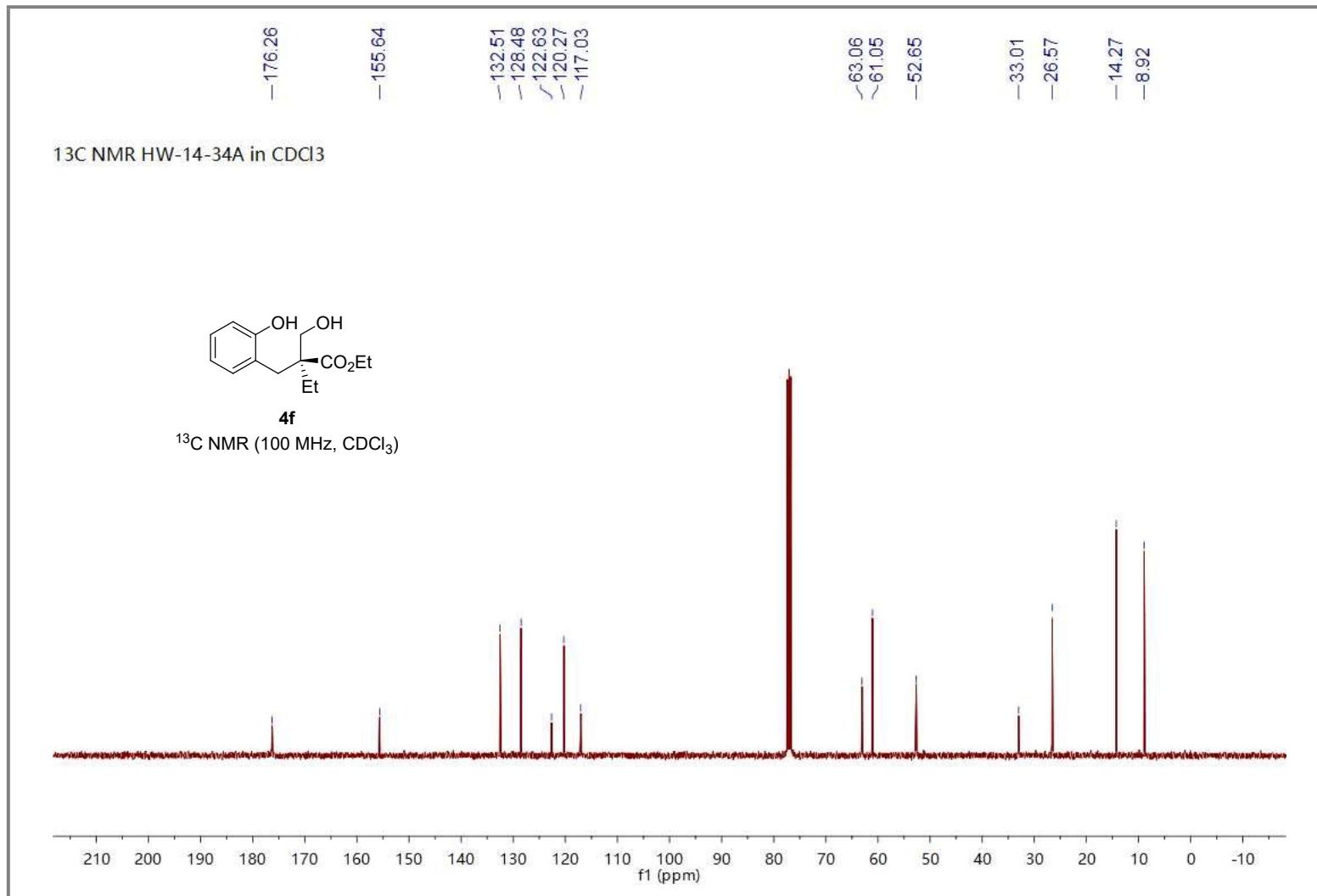
Totals : 5366.33167 265.29971



Instrument 1 10/17/2023 9:18:41 AM

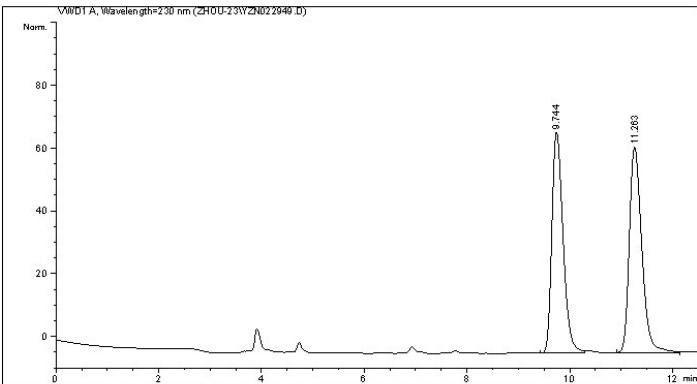
Page 1 of 1





Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022949.D
Sample Name: HW-14-32A TM+/-

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Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 4/23/2023 2:11:37 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 4/23/2023 1:59:02 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/18/2023 8:07:03 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm



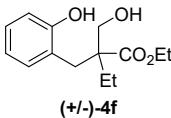
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Area Percent Report

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	9.744	BV	0.2347	1063.41235	70.32623	49.1143	
2	11.263	BB	0.2591	1101.76733	65.43362	50.8857	

Totals : 2165.17969 135.75985

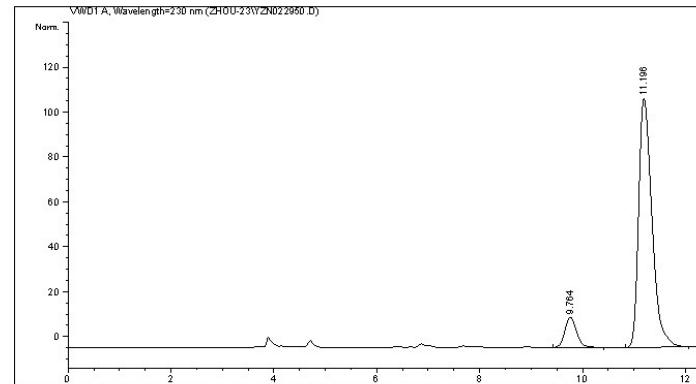


Instrument 1 10/18/2023 8:07:08 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022950.D
Sample Name: HW-14-32A TM+

=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 4/23/2023 2:26:56 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 4/23/2023 2:25:56 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/18/2023 8:08:15 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm



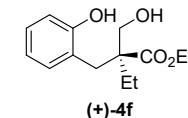
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Area Percent Report

Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	9.764	BB	0.2472	215.87962	13.53471	9.7641	
2	11.196	BB	0.2783	1995.07495	110.88701	90.2359	

Totals : 2210.95457 124.42172

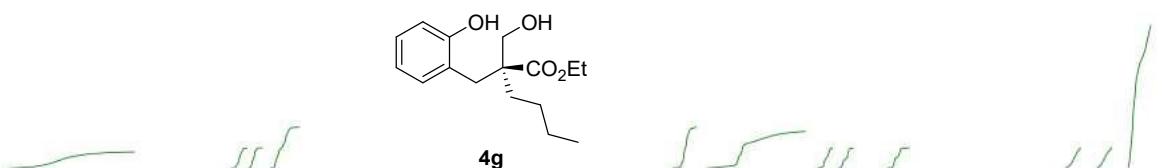


Instrument 1 10/18/2023 8:08:18 AM

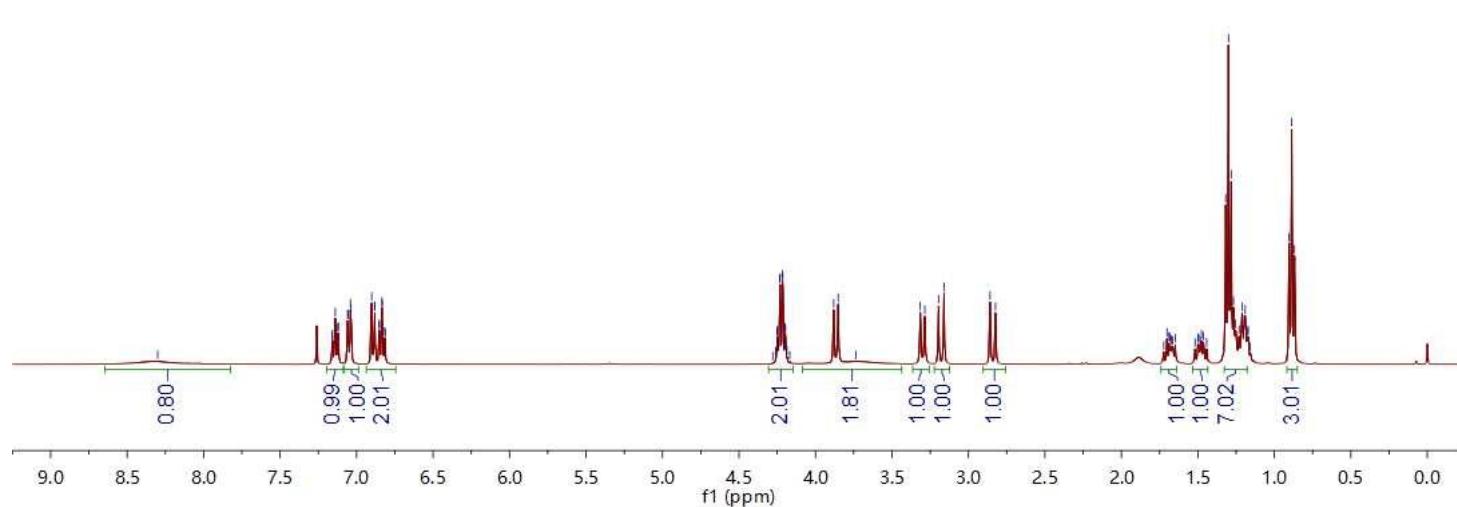
Page 1 of 1

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4.2341
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4.1956
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¹H NMR HW-14-41C TM in CDCl₃

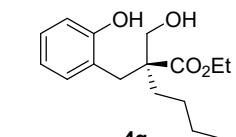


¹H NMR (400 MHz, CDCl₃)

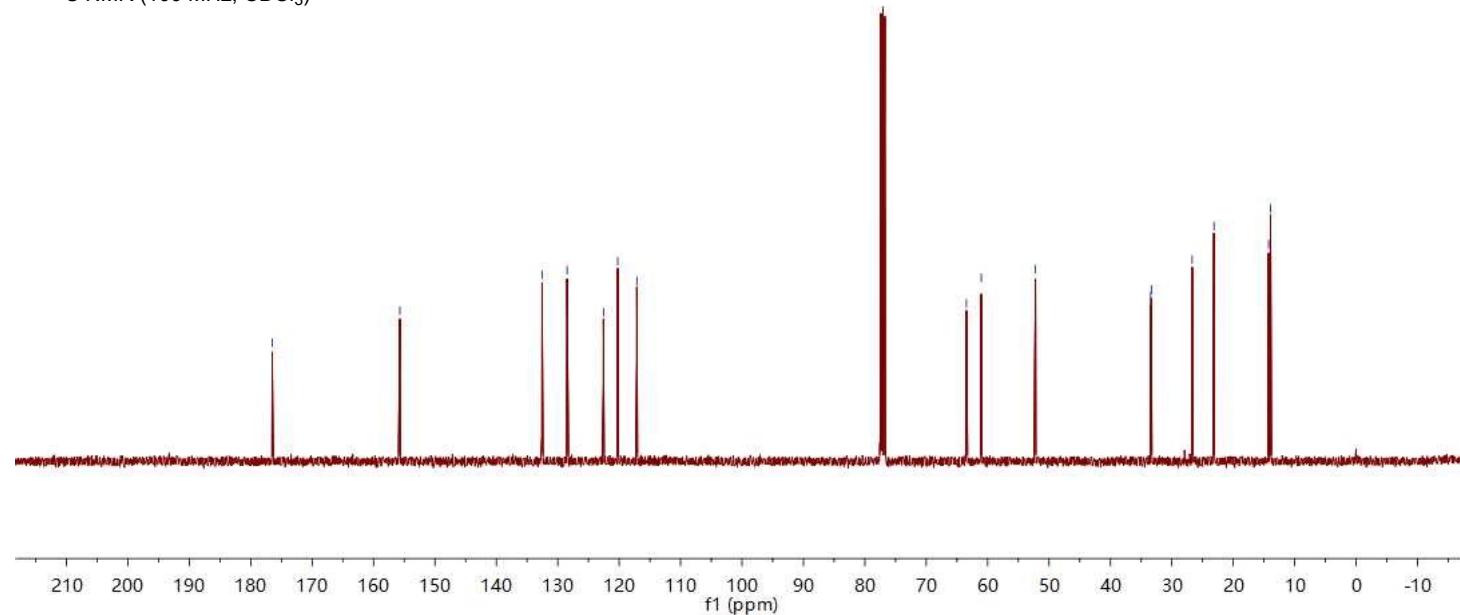


-176.50
-155.72
-132.56
-128.48
-122.56
-120.24
-117.12

¹³C NMR HW-14-41C TM in CDCl₃

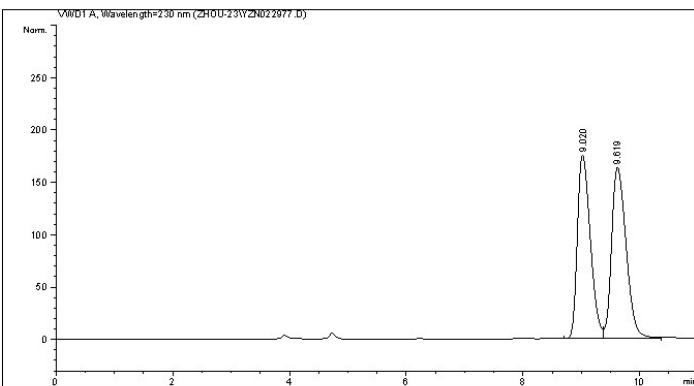


¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022977.D
Sample Name: HW-14-41C TM+/-

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Acq. Instrument : Instrument 1
Injection Date : 4/26/2023 7:45:21 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 4/26/2023 7:29:44 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:22:15 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

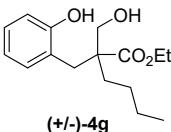


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Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

#	Peak RetTime	Type	Width	Area	Height	Area
	[min]		[min]	[mAU]	*s	[mAU]
1	9.020	BV	0.2407	2696.60937	175.18466	48.8336
2	9.619	BV	0.2705	2825.43018	163.10492	51.1664
Totals :				5522.03955	338.28958	

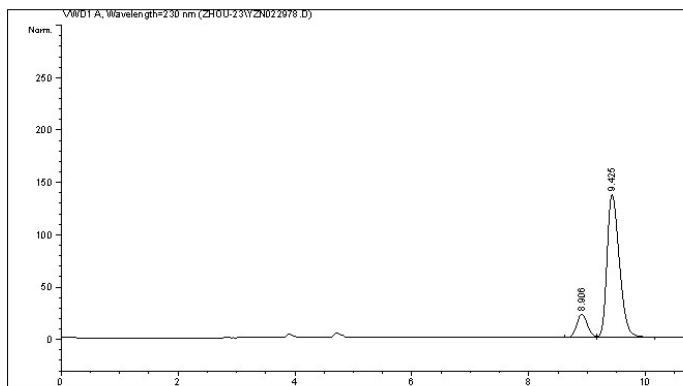


Instrument 1 10/17/2023 9:22:18 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022978.D
Sample Name: HW-14-41C TM

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1
Injection Date : 4/26/2023 8:05:34 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 4/26/2023 8:05:08 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:22:15 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

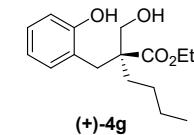


=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

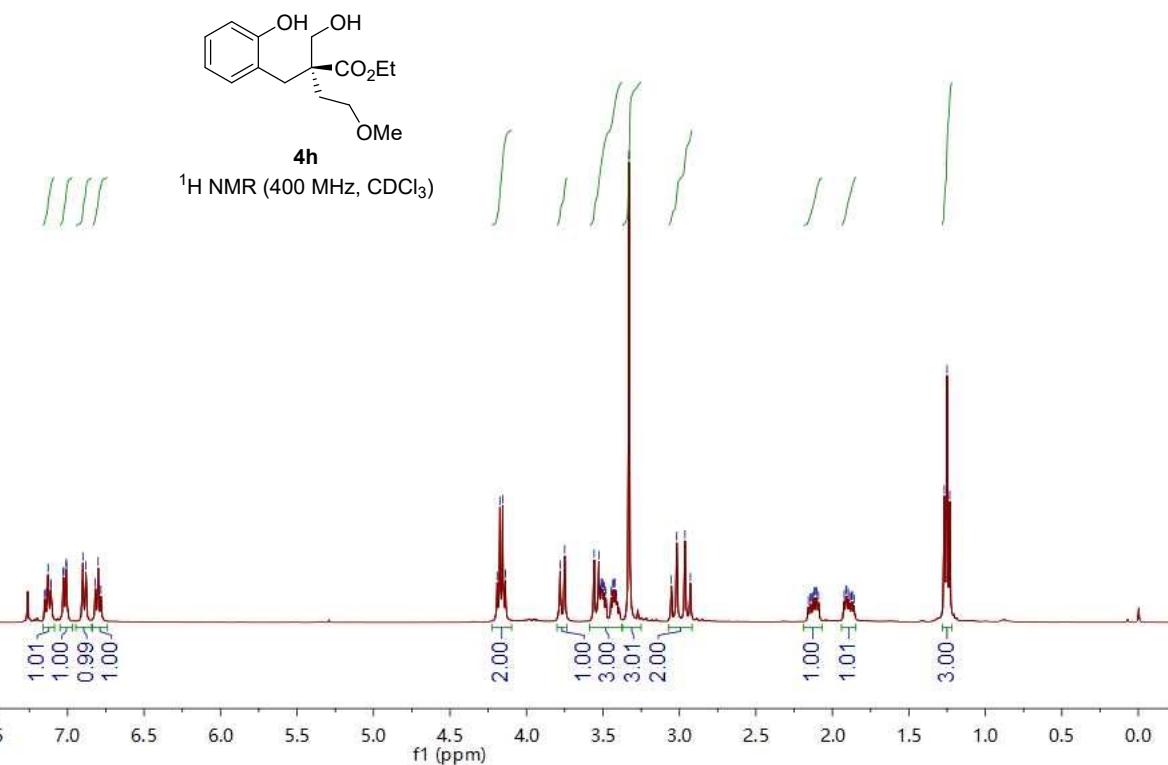
#	Peak RetTime	Type	Width	Area	Height	Area
	[min]		[min]	[mAU]	*s	[mAU]
1	8.906	BV	0.2014	284.40286	22.05935	12.5159
2	9.425	BV	0.2268	1987.93518	136.37880	87.4841
Totals :				2272.33804	158.43815	



Instrument 1 10/17/2023 9:22:54 AM

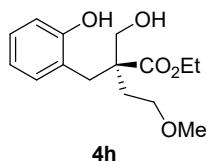
Page 1 of 1

¹H NMR HW-14-80TM in CDCl₃

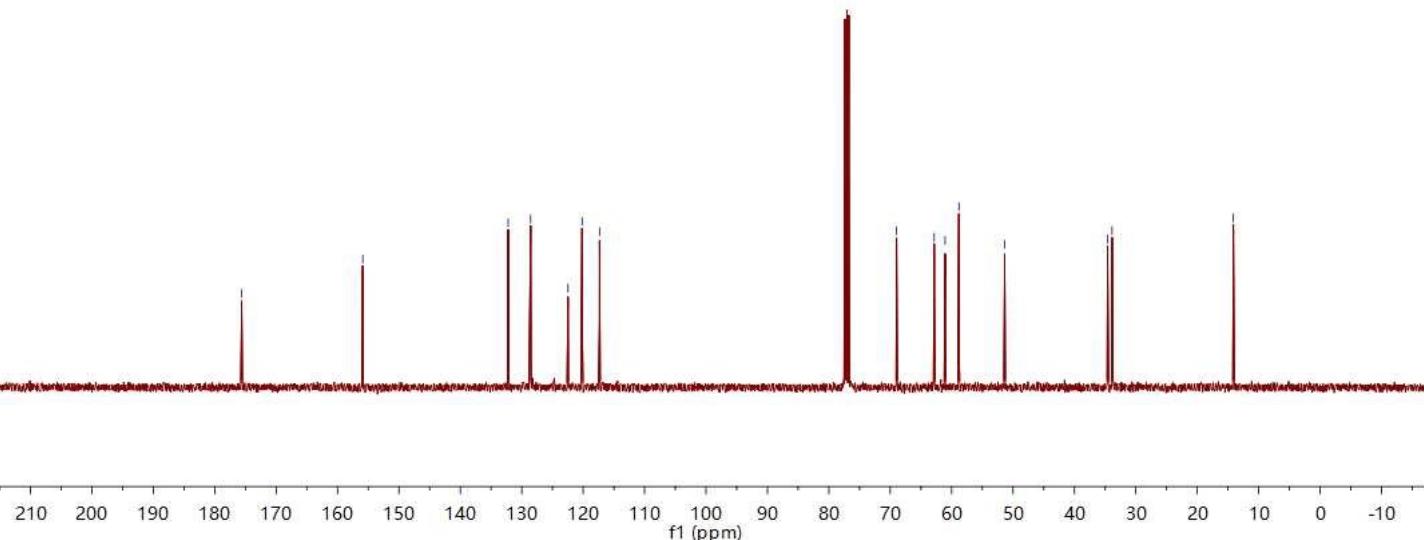


-175.61 -155.90
-132.22 -128.55
-122.47 -120.18
-117.34

¹³C NMR HW-14-80TM in CDCl₃

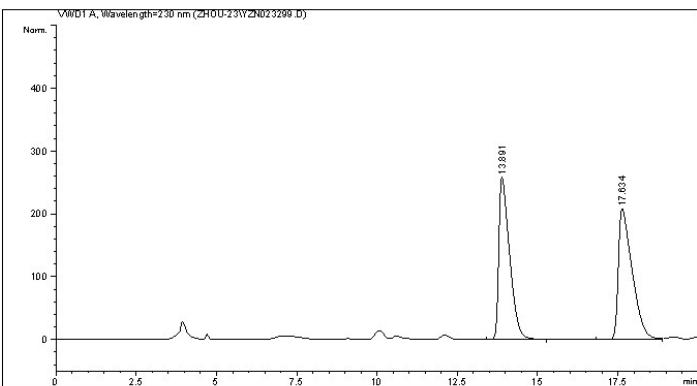


¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023299.D
Sample Name: HW-14-80 TH+/-

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Injection Date : 6/13/2023 3:10:26 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 6/13/2023 3:09:51 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:29:58 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



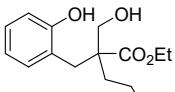
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=====
Area Percent Report
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```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	13.891	BB	0.3526	6027.66406	258.00723	49.1641	
2	17.634	BV	0.4410	6232.62939	208.18640	50.8359	

Totals : 1.22603e4 466.19363



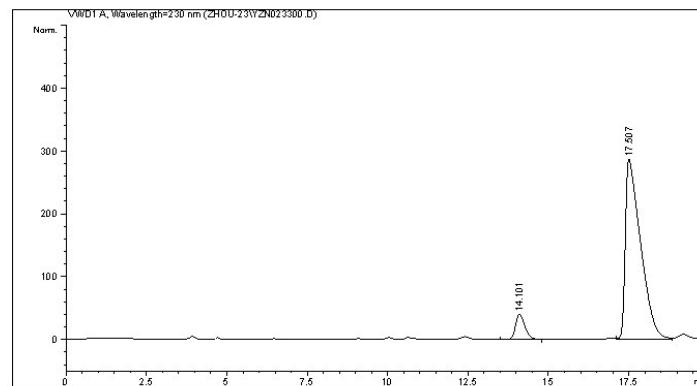
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*** End of Report ***

Instrument 1 10/17/2023 9:30:01 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023300.D
Sample Name: HW-14-80 TH

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 6/13/2023 3:38:30 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 6/13/2023 3:37:15 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:30:47 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

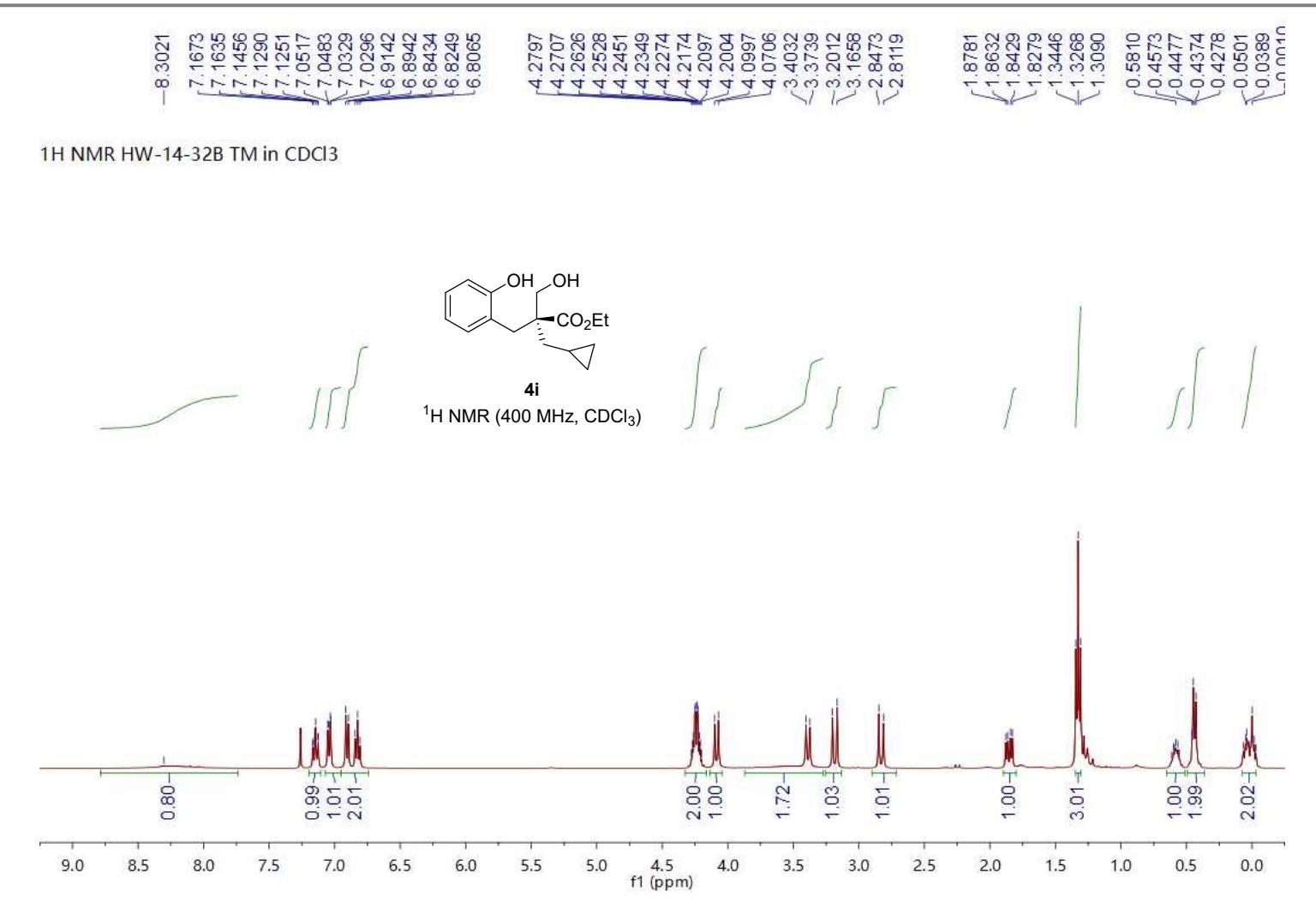
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	14.101	BB	0.3105	798.60327	39.68063	7.7841	
2	17.507	VV	0.4791	9460.86816	287.09534	92.2159	

Totals : 1.02595e4 326.77596

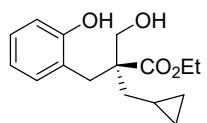
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*** End of Report ***

Instrument 1 10/17/2023 9:30:49 AM

Page 1 of 1

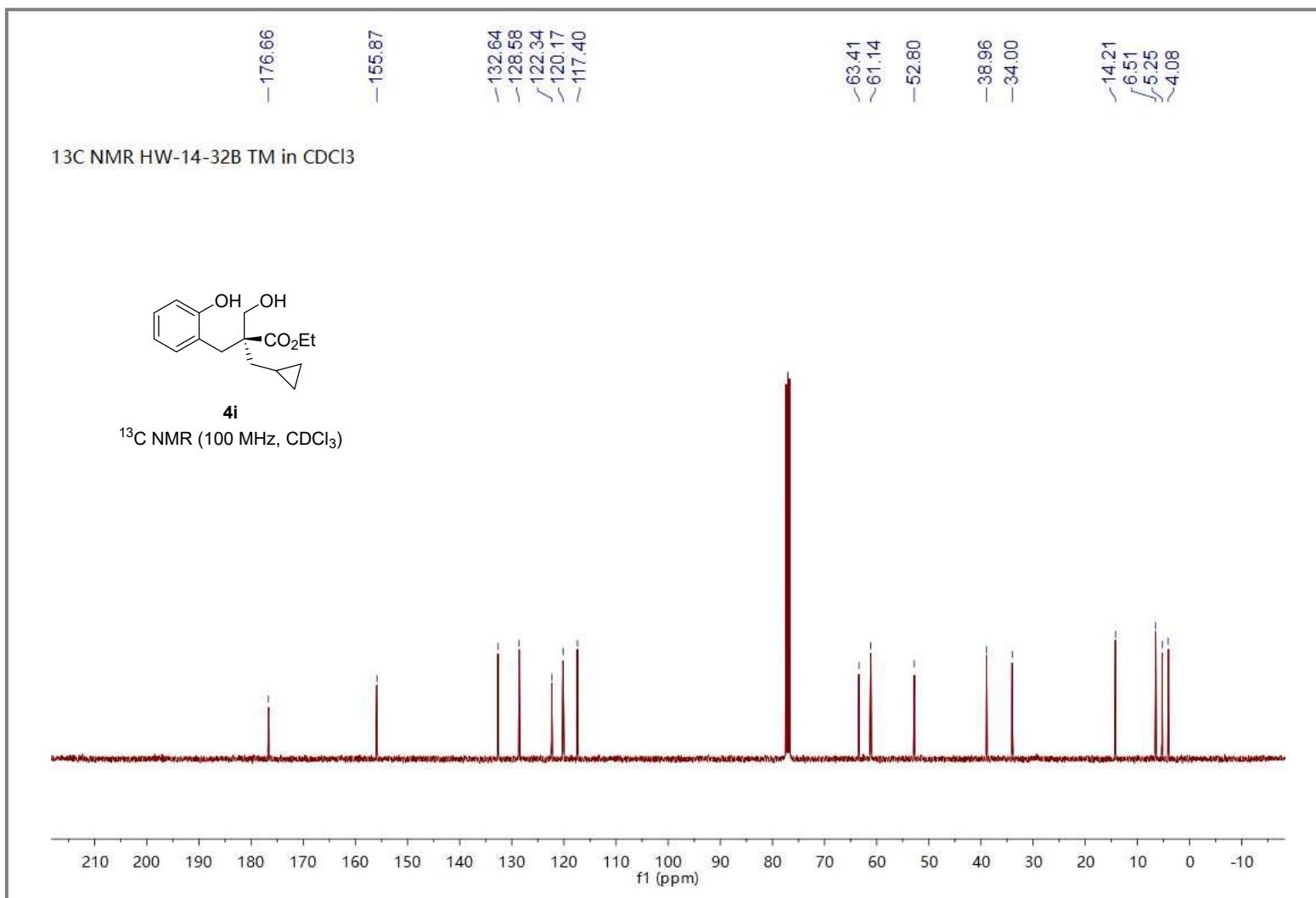


¹³C NMR HW-14-32B TM in CDCl₃



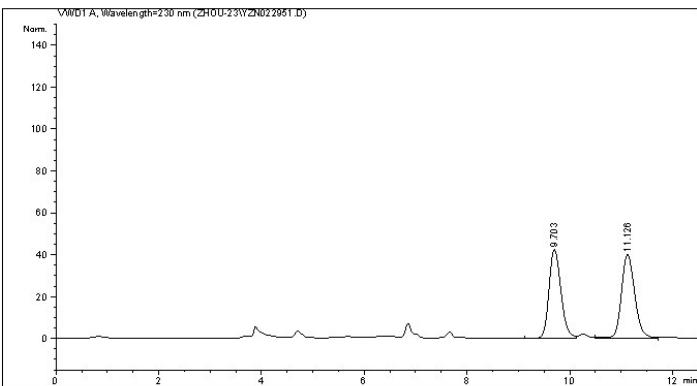
4i

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022951.D
Sample Name: HW-14-32B TM+/-

=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 4/23/2023 2:44:05 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 4/23/2023 2:42:40 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:36:03 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm

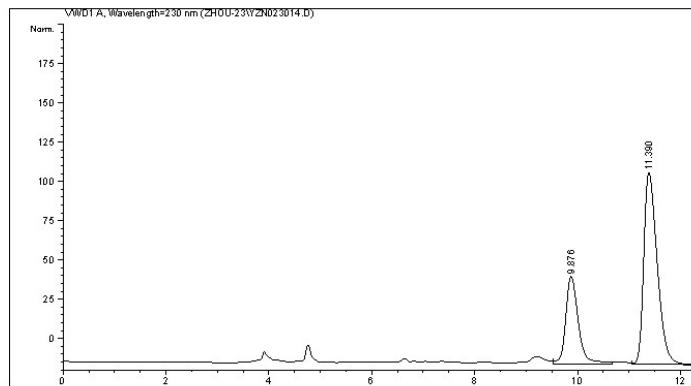


Instrument 1 10/17/2023 9:36:06 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023014.D
Sample Name: HW-14-47A TM

=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/5/2023 5:59:42 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/5/2023 5:54:33 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:37:14 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230nm

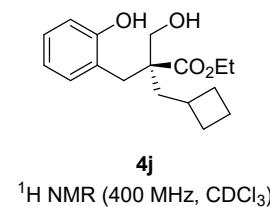


Instrument 1 10/17/2023 9:37:21 AM

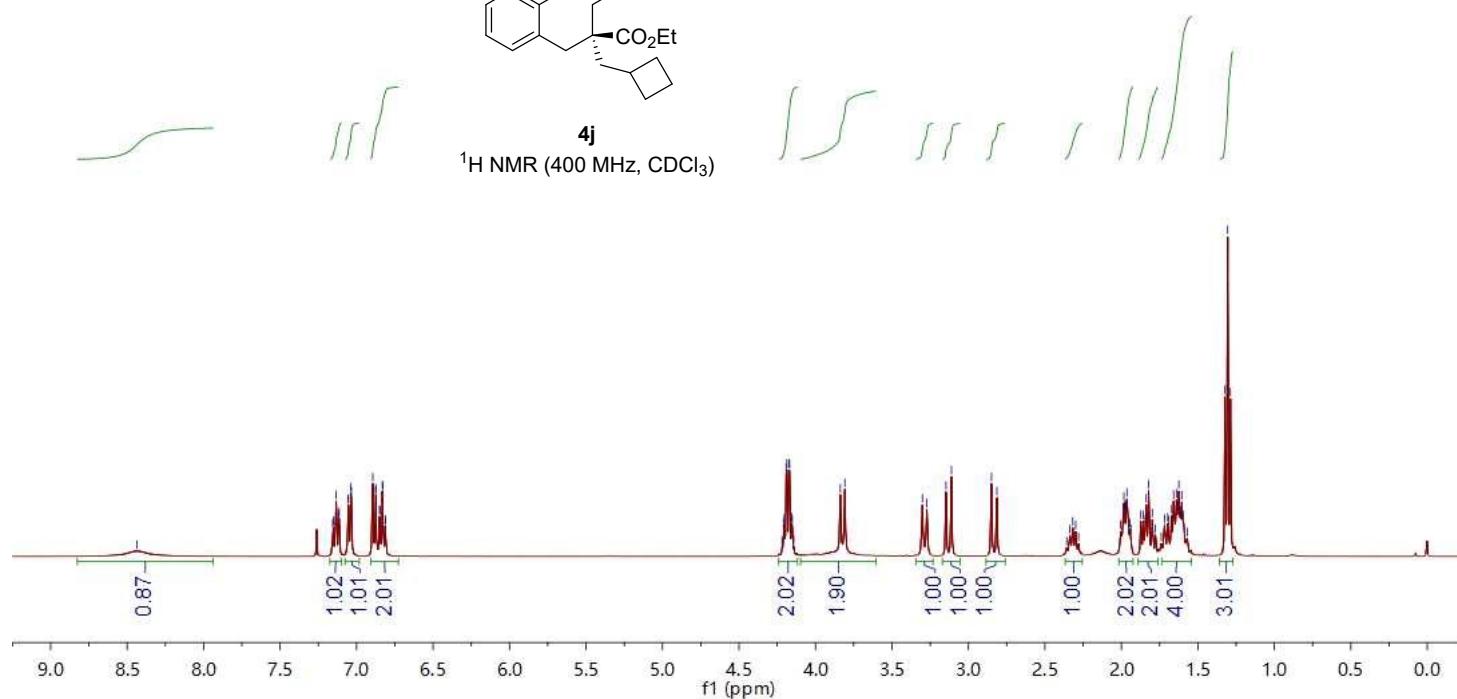
Page 1 of 1

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¹H NMR HW-14-56A in CDCl₃

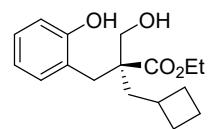


¹H NMR (400 MHz, CDCl₃)



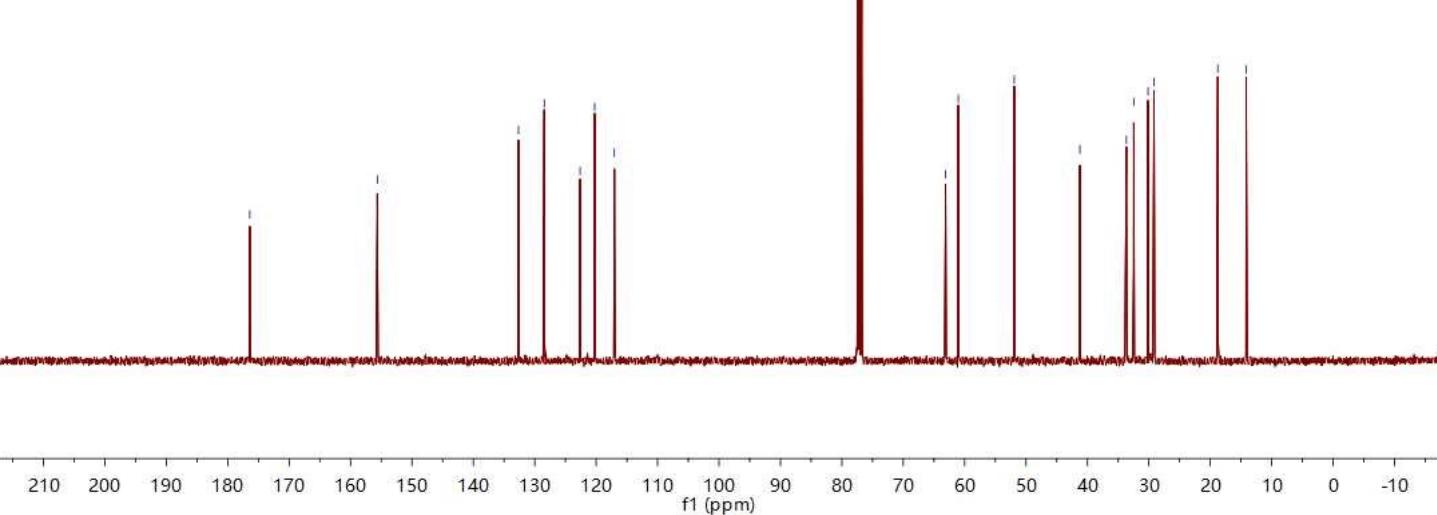
-176.40
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-132.64
-128.46
-122.62
-120.25
-117.04

¹³C NMR HW-14-56A in CDCl₃



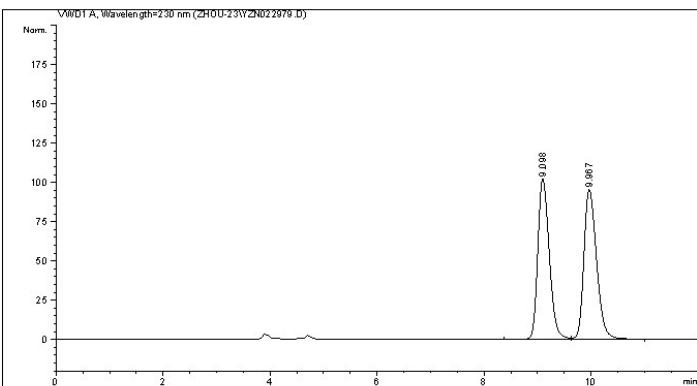
4j

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN022979.D
Sample Name: HW-14-41A TM+/-

```
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Acq. Operator :                               Location : -
Injection Date : 4/26/2023 8:23:05 AM
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Last changed   : 4/26/2023 8:22:04 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:42:07 AM
                                         (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



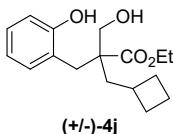
```
=====
Area Percent Report
```

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

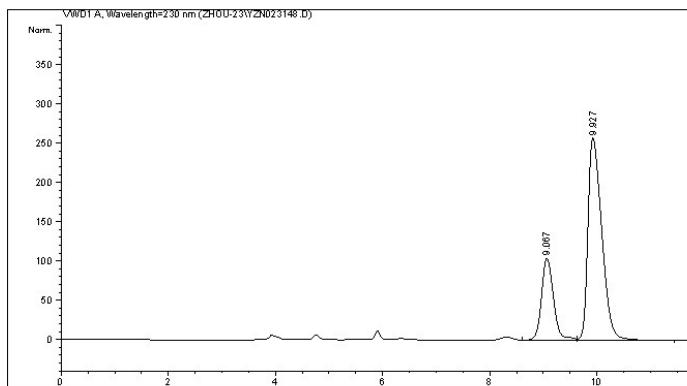
Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	9.098	VV	0.2332	1528.92163	101.94090	49.2315	
2	9.967	VV	0.2573	1576.65430	95.21244	50.7685	

Totals : 3105.57593 197.15334



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023148.D
Sample Name: HW-14-56A TM

```
=====
Acq. Operator :                               Location : -
Injection Date : 5/21/2023 2:23:55 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/21/2023 2:15:33 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:43:13 AM
                                         (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



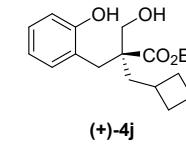
```
=====
Area Percent Report
```

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

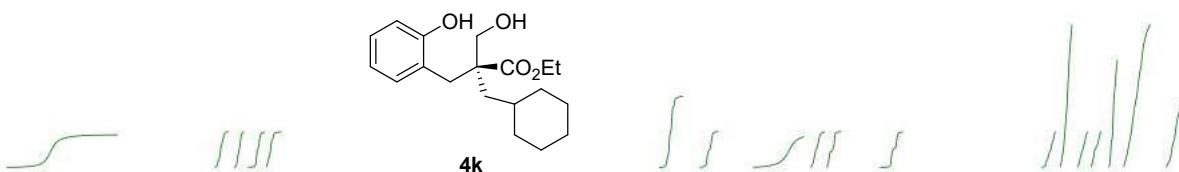
Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	9.067	VV	0.2464	1627.96777	104.17851	26.0059	
2	9.927	VV	0.2798	4632.03223	257.48196	73.9941	

Totals : 6260.00000 361.66048

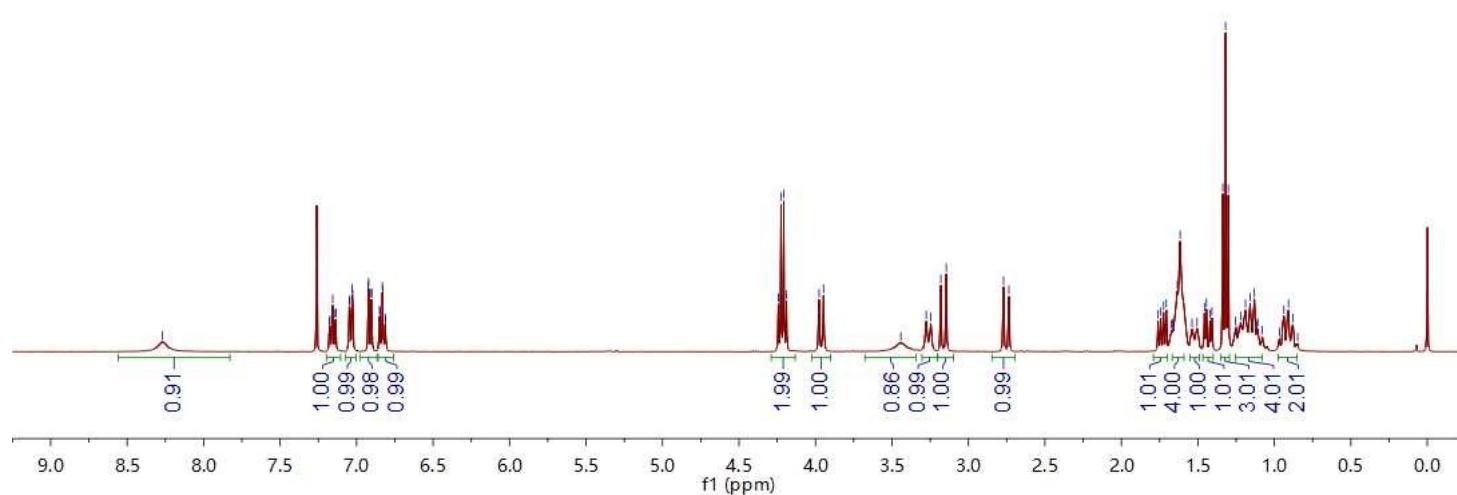


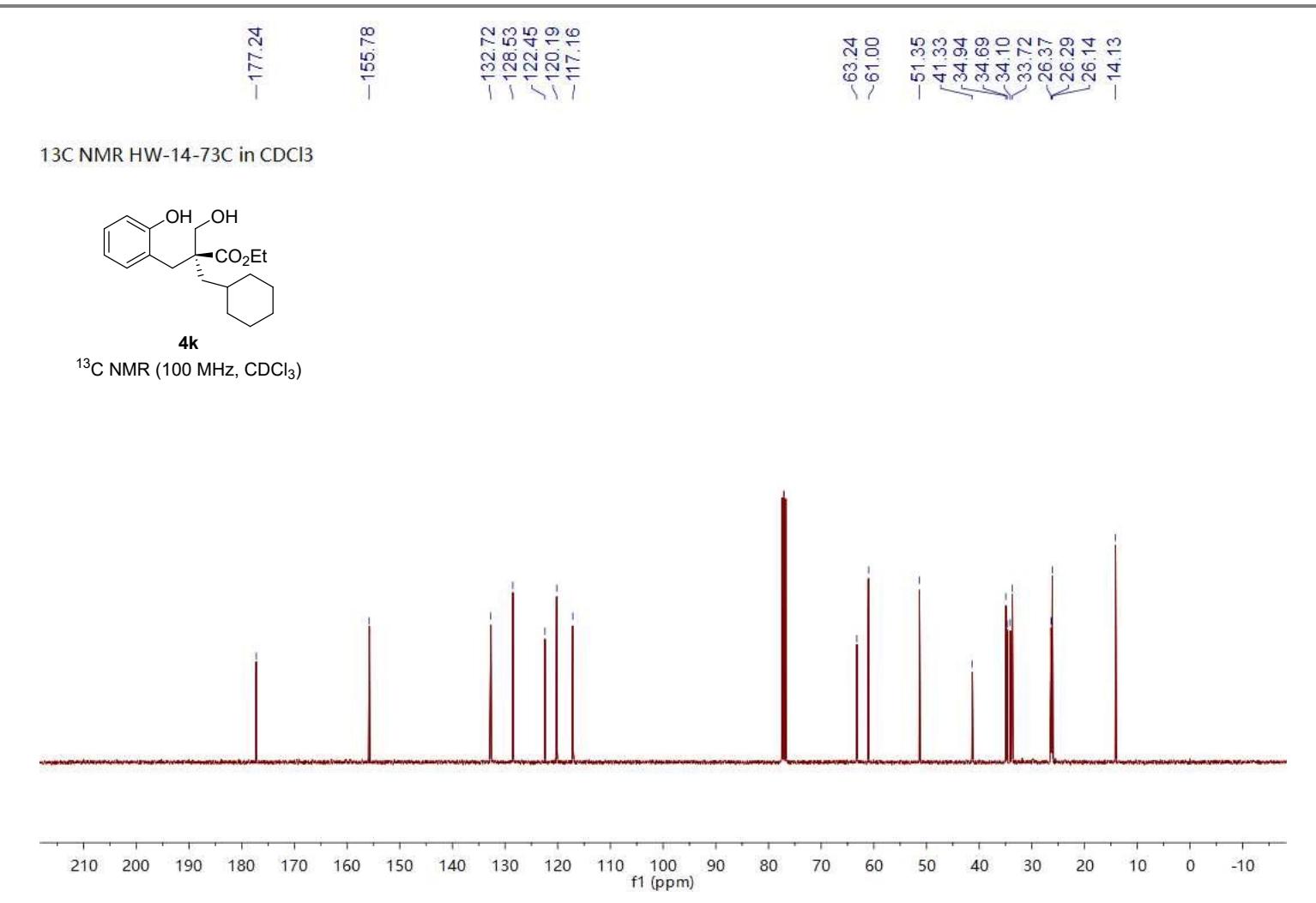
7.1781
7.1739
7.1558
7.1397
7.1355
7.0480
7.0442
7.0292
7.0253
6.9234
6.9209
6.8313
6.8287
6.9007
6.8499
6.8470
6.8129
6.8100
4.2429
4.2250
4.2072
4.1893
3.9763
3.9471
3.2759
3.2460
3.1802
3.1449
2.7704
2.7351
1.7597
1.7421
1.7239
1.7063
1.6722
1.6640
1.6340
1.6158
1.5379
1.5049
1.4570
1.4440
1.4213
1.4082
1.3368
1.3189
1.3010
1.2529
1.2199
1.1887
1.1583
1.1306
1.1153
1.1086
0.9374
0.9073
0.8789

¹H NMR HW-14-73C in CDCl₃



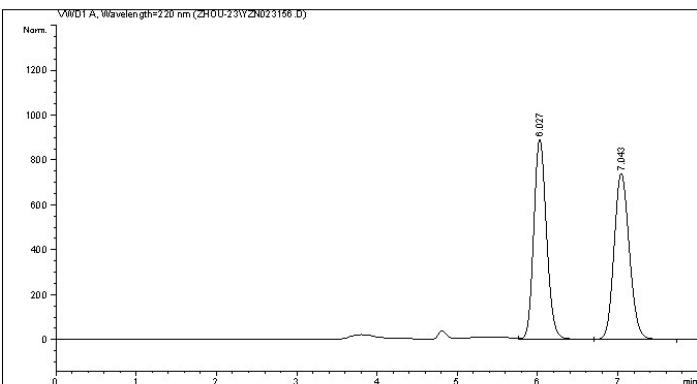
¹H NMR (400 MHz, CDCl₃)





Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023156.D
Sample Name: HW-14-56B TM+/-

```
=====
Acq. Operator :                               Location : -
Injection Date : 5/22/2023 10:18:38 PM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/22/2023 10:17:24 PM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:46:16 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 220 nm
```



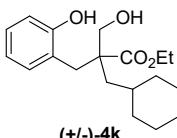
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	s	*s	[mAU]	Height	Area %
1	6.027	VV	0.1705	9900.32520	890.14624	50.2350			
2	7.043	VV	0.2074	9807.71289	738.23425	49.7650			

Totals : 1.97080e4 1628.38049

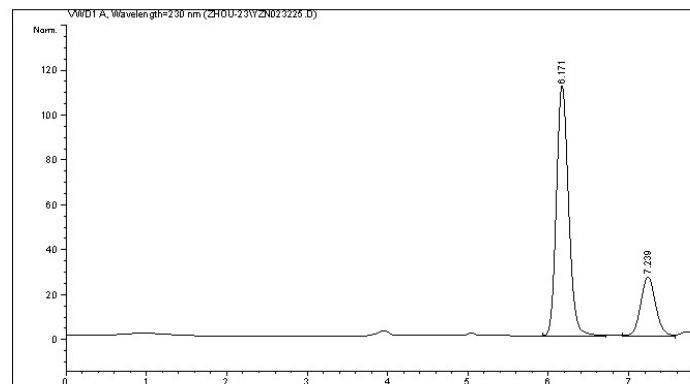


Instrument 1 10/17/2023 9:46:18 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023225.D
Sample Name: HW-14-73C TM

```
=====
Acq. Operator :                               Location : -
Injection Date : 5/31/2023 2:12:13 AM
Acq. Method  : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 5/31/2023 2:11:14 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 10/17/2023 9:47:08 AM
                                         (modified after loading)
Sample Info    : AS-H, Hexane/i-PrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```



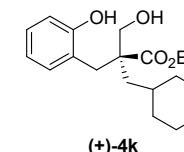
=====
Area Percent Report
=====

```
Sorted By      : Signal
Multiplier:   : 1.0000
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	s	*s	[mAU]	Height	Area %
1	6.171	BV	0.1701	1174.89648	110.97256	77.5276			
2	7.239	VV	0.1976	340.55948	26.31386	22.4724			

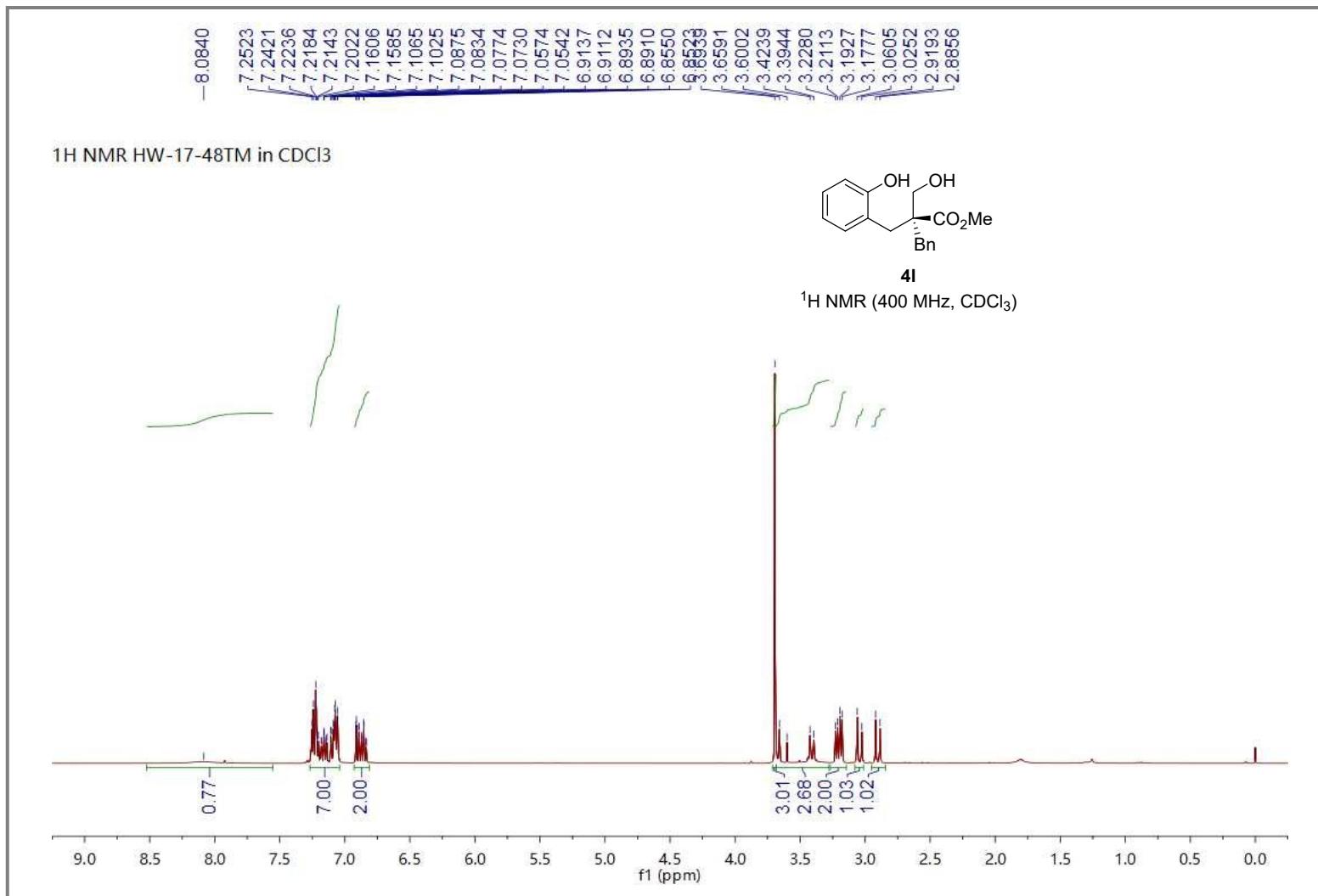
Totals : 1515.45596 137.28643



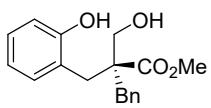
=====
*** End of Report ***
=====

Instrument 1 10/17/2023 9:47:11 AM

Page 1 of 1

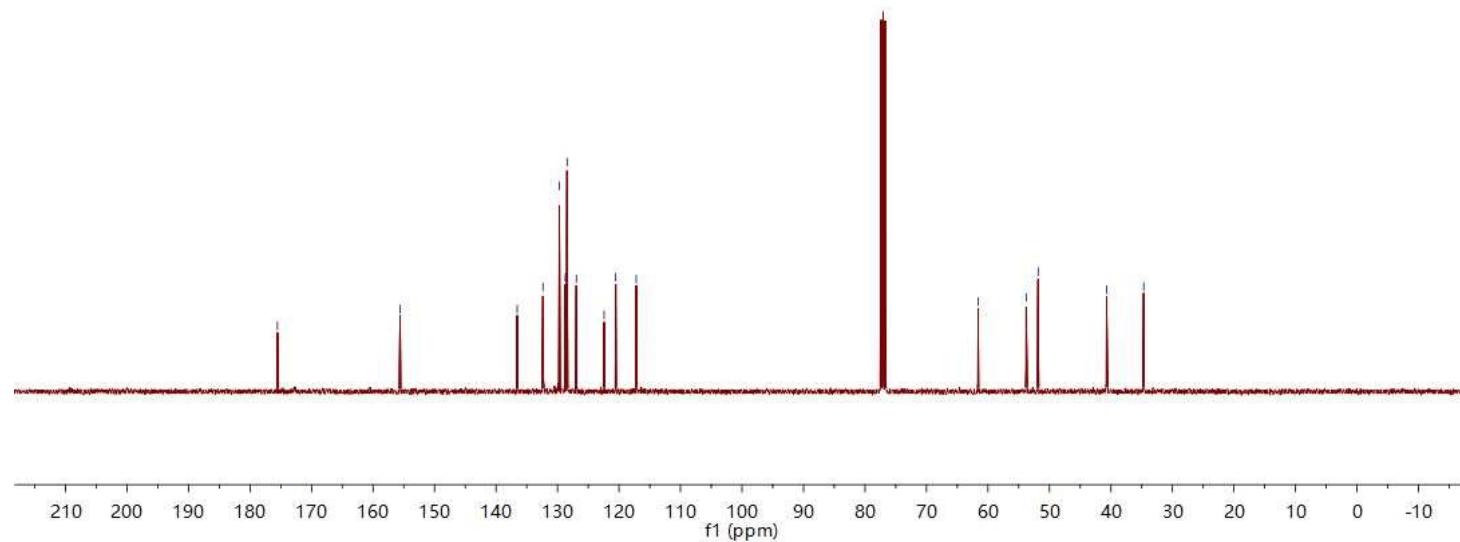


¹³C NMR HW-17-48TM in CDCl₃



4l

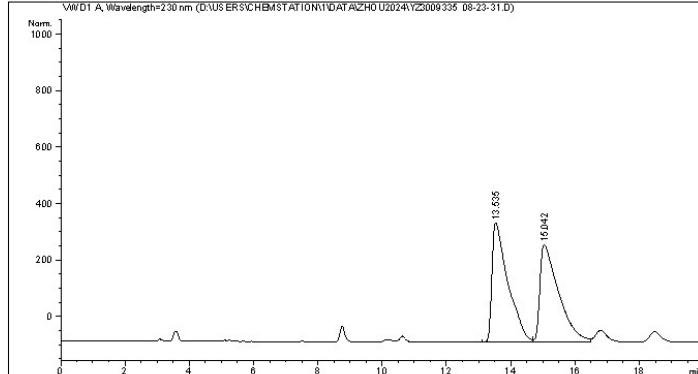
¹³C NMR (100 MHz, CDCl₃)



Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2024\YZ3009335 08-23-31.D
Sample Name: HW-17-48TM+-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 4/28/2024 8:23:31 AM    Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 4/28/2024 8:08:31 AM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 4/28/2024 10:00:18 AM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, n-Hexane/i-PrOH = 97/3, 1.0 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated
VWD1 A, Wavelength=230 nm (D:\USERS\CHEMSTATION\1\DATA\ZHOU2024\YZ3009335 08-23-31.D)

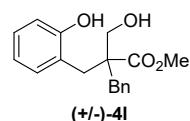


=====
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.535	BV	0.5013	8433.72168	245.18311	51.0353
2	15.042	VV	0.5842	8091.54639	199.93265	48.9647



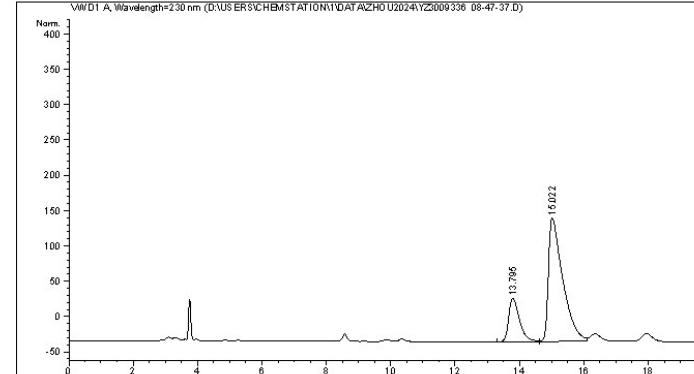
1260II 4/28/2024 10:00:20 AM SYSTEM

Page 1 of 2

Data File D:\USERS\CHEMSTATION\1\DATA\ZHOU2024\YZ3009336 08-47-37.D
Sample Name: HW-17-48TM

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : 1260II          Location : -
Injection Date : 4/28/2024 8:47:37 AM    Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 4/28/2024 8:08:31 AM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\Users\Public\Documents\ChemStation\1\Methods\def_LC.M
Last changed   : 4/28/2024 9:57:35 AM by SYSTEM
                                                (modified after loading)
Sample Info    : IB, n-Hexane/i-PrOH = 97/3, 1.0 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated
VWD1 A, Wavelength=230 nm (D:\USERS\CHEMSTATION\1\DATA\ZHOU2024\YZ3009336 08-47-37.D)

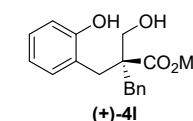


=====
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Do not use Multiplier & Dilution Factor with ISTDs
```

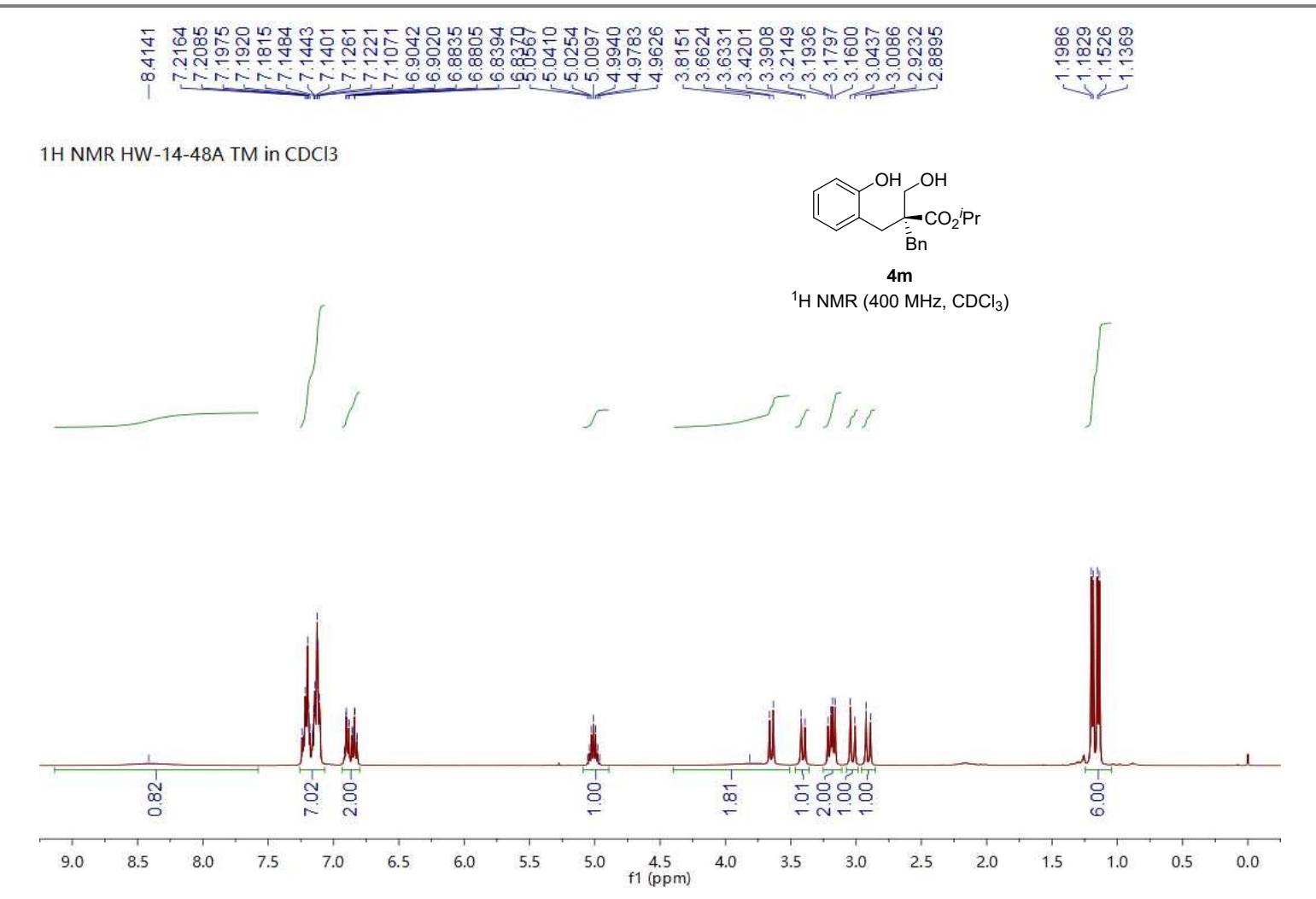
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.795	BV	0.3533	844.73486	36.59961	20.7135
2	15.022	VV	0.4490	3233.44556	104.50420	79.2865

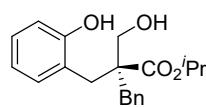


1260II 4/28/2024 9:58:13 AM SYSTEM

Page 1 of 2

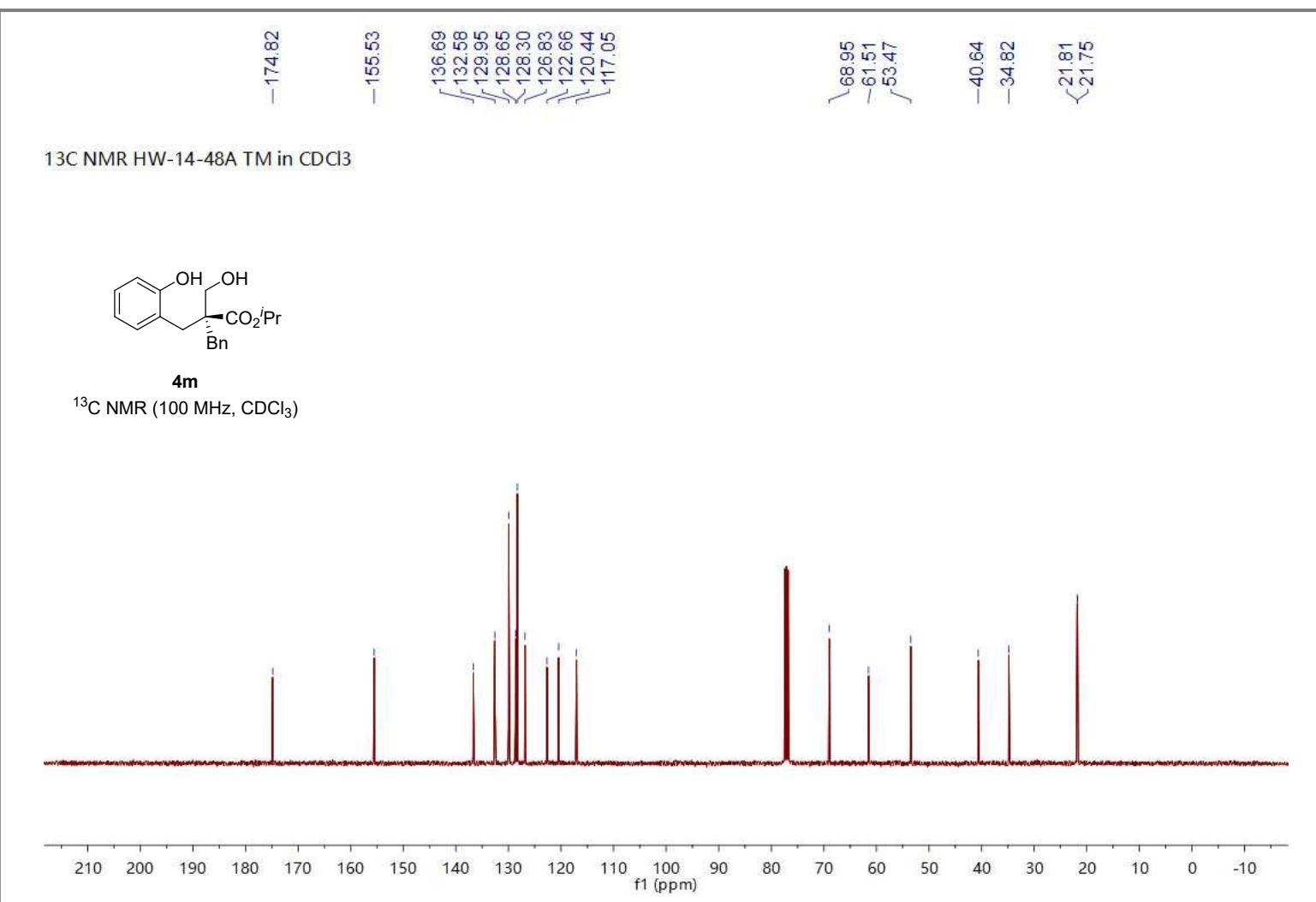


¹³C NMR HW-14-48A TM in CDCl₃



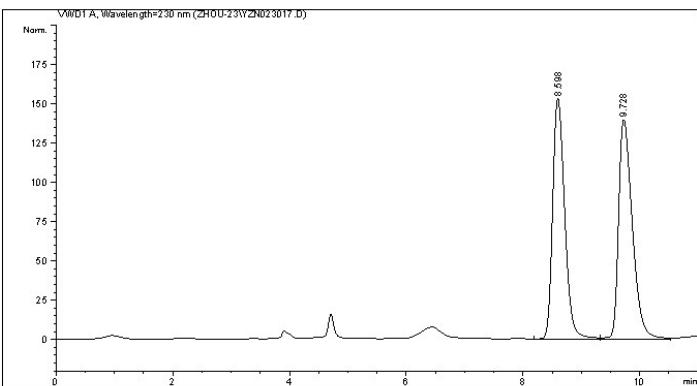
4m

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023017.D
Sample Name: HW-14-48A TM+/-

```
=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/5/2023 6:35:45 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/5/2023 6:35:18 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:50:10 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230nm
```

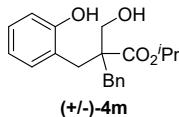


```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	8.598	VV	0.2257	2197.08643	153.03523	49.0022	
2	9.728	VV	0.2555	2286.56616	139.36580	50.9978	
Totals :				4483.65259	292.40103		

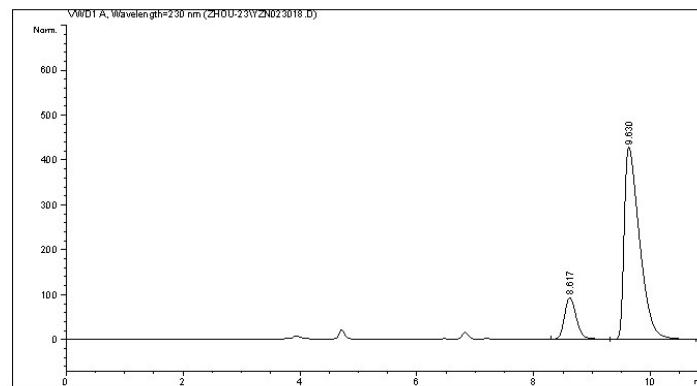


Instrument 1 10/17/2023 9:50:13 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN023018.D
Sample Name: HW-14-48A TM

```
=====
Acq. Operator : Acq. Instrument : Instrument 1 Location : -
Injection Date : 5/5/2023 6:50:22 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 5/5/2023 6:49:58 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 10/17/2023 9:51:19 AM
(modified after loading)
Sample Info : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230nm
```

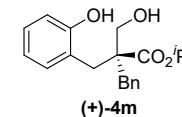


```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

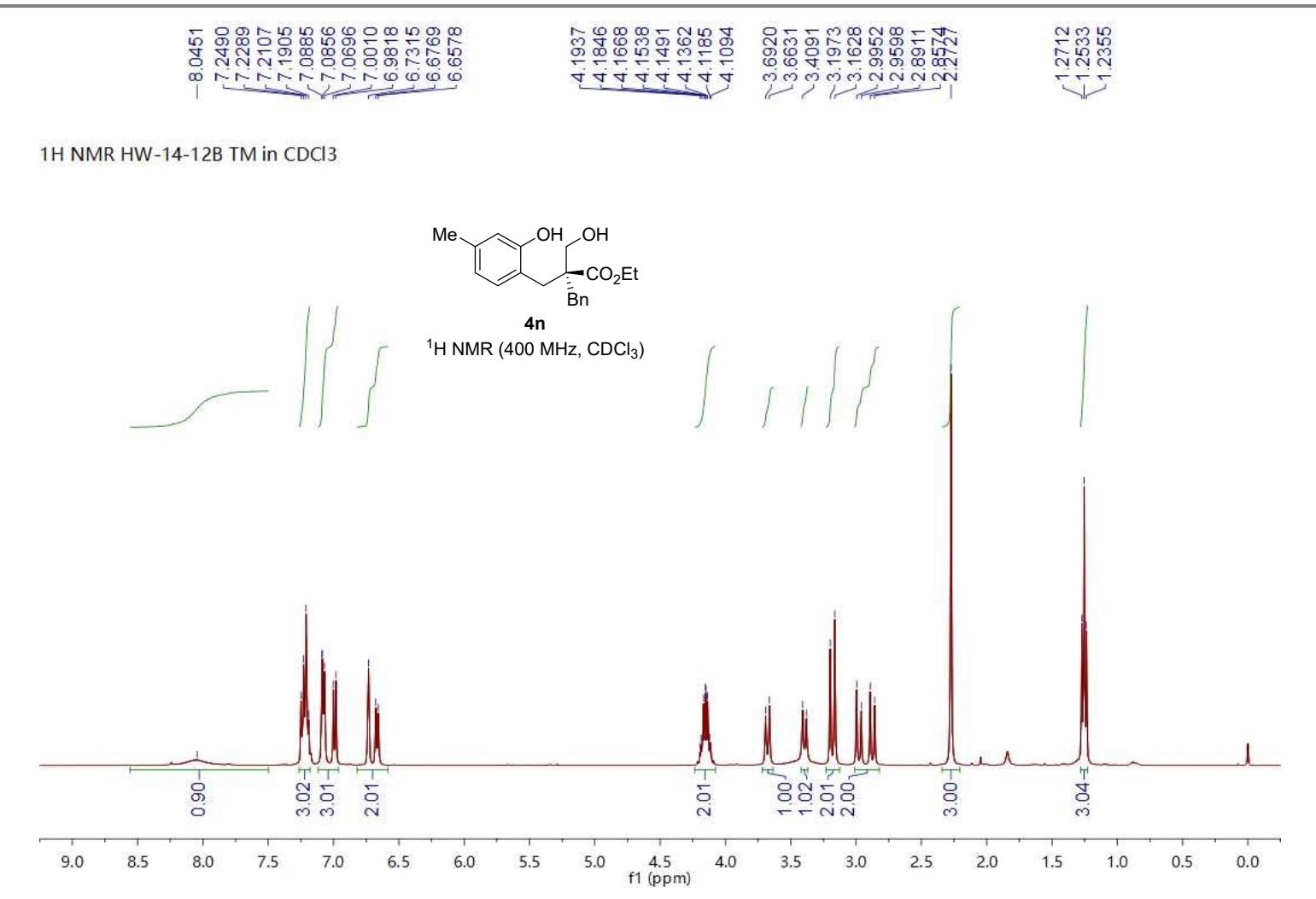
Signal 1: VWD1 A, Wavelength=230 nm

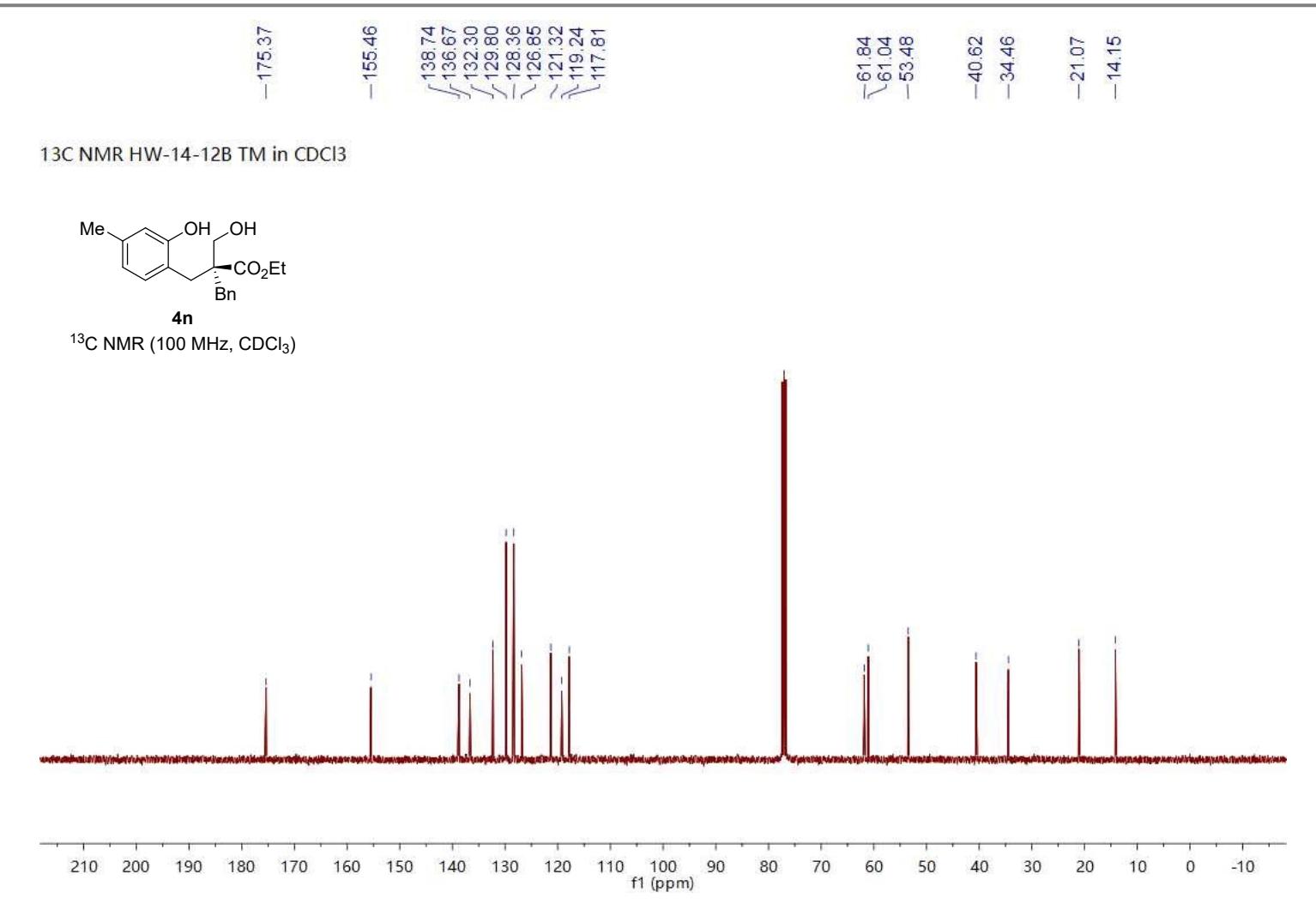
Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	8.617	VV	0.2231	1309.10754	92.63066	14.4020	
2	9.630	VV	0.2769	7780.67188	426.52310	85.5980	
Totals :				9089.77942	519.15376		



Instrument 1 10/17/2023 9:51:21 AM

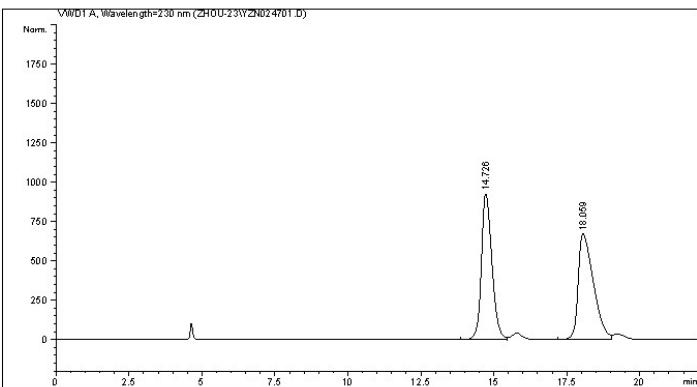
Page 1 of 1





Data File C:\CHEM32\1\DATA\ZHOU-23\YZN024701.D
Sample Name: HW-14-12BTM+-

```
=====
Acq. Operator :                               Location : -
Injection Date : 12/1/2023 2:40:00 AM
Acq. Method   : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 12/1/2023 2:31:04 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 12/1/2023 3:30:06 AM
                                         (modified after loading)
Sample Info    : IA, Hexane/iPrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



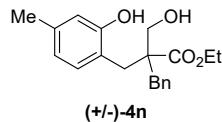
```
=====
Area Percent Report
```

```
Sorted By      : Signal
Multiplier:    :
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	14.726	BV	0.3848	2.32406e4	929.63464	48.8079	
2	18.059	BV	0.5630	2.43758e4	671.90833	51.1921	

Totals : 4.76164e4 1601.54297



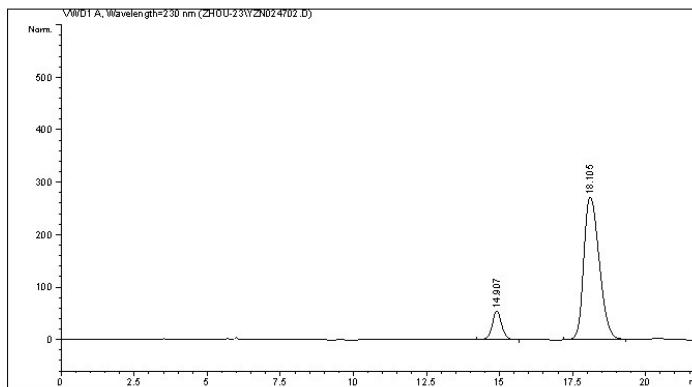
(-)-4n

Instrument 1 12/1/2023 3:30:15 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-23\YZN024702.D
Sample Name: HW-14-12BTM+

```
=====
Acq. Operator :                               Location : -
Injection Date : 12/1/2023 3:04:06 AM
Acq. Method   : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 12/1/2023 3:03:19 AM
                                         (modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed   : 12/1/2023 3:29:08 AM
                                         (modified after loading)
Sample Info    : IA, Hexane/iPrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```



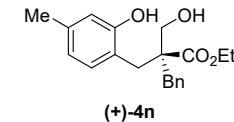
```
=====
Area Percent Report
```

```
Sorted By      : Signal
Multiplier:    :
Dilution:     : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	14.907	BB	0.3381	1226.70667	55.15179	10.9720	
2	18.105	BB	0.5808	9953.66699	271.37015	89.0280	

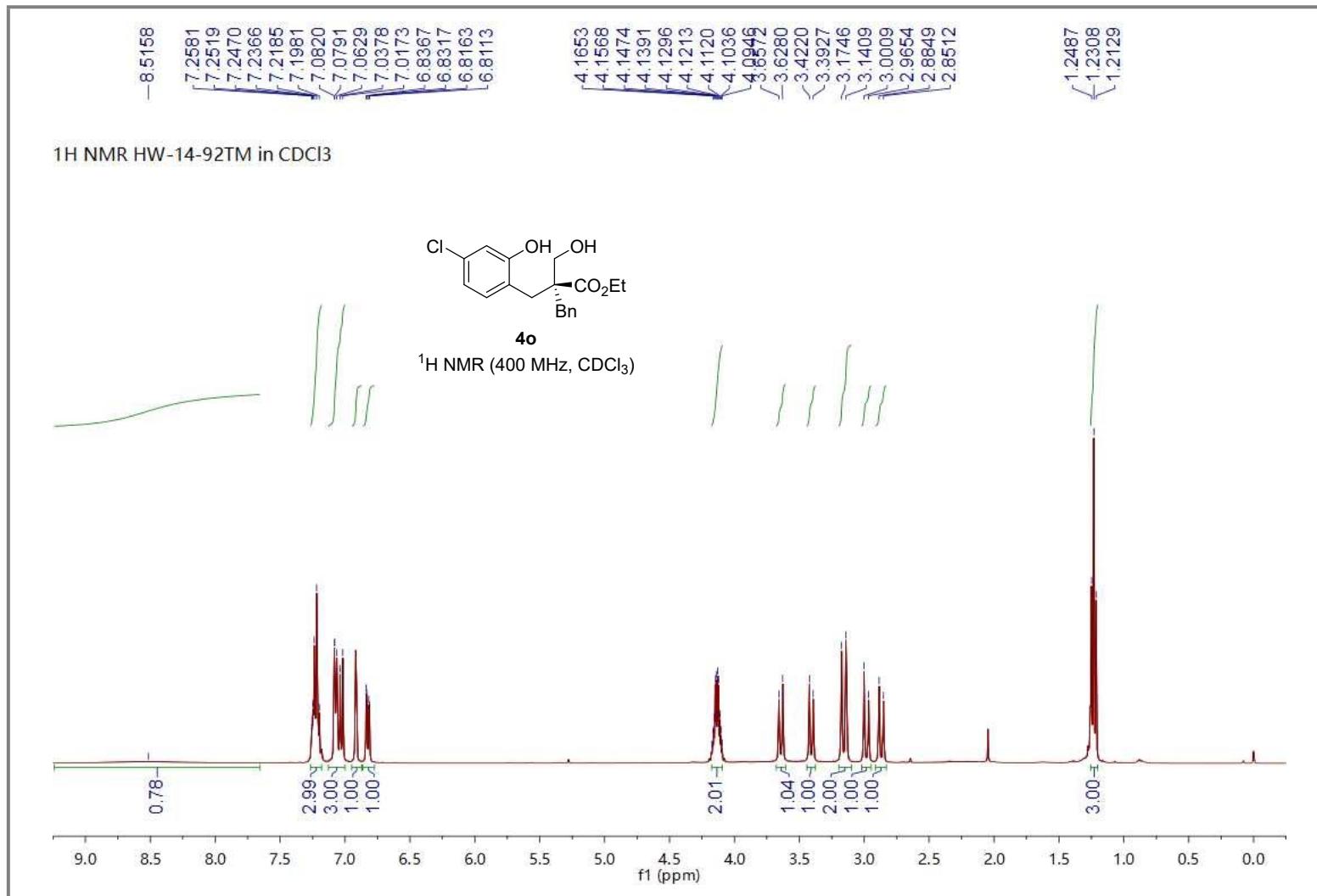
Totals : 1.11804e4 326.52193



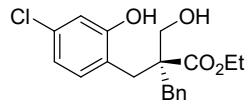
(+)-4n

Instrument 1 12/1/2023 3:29:12 AM

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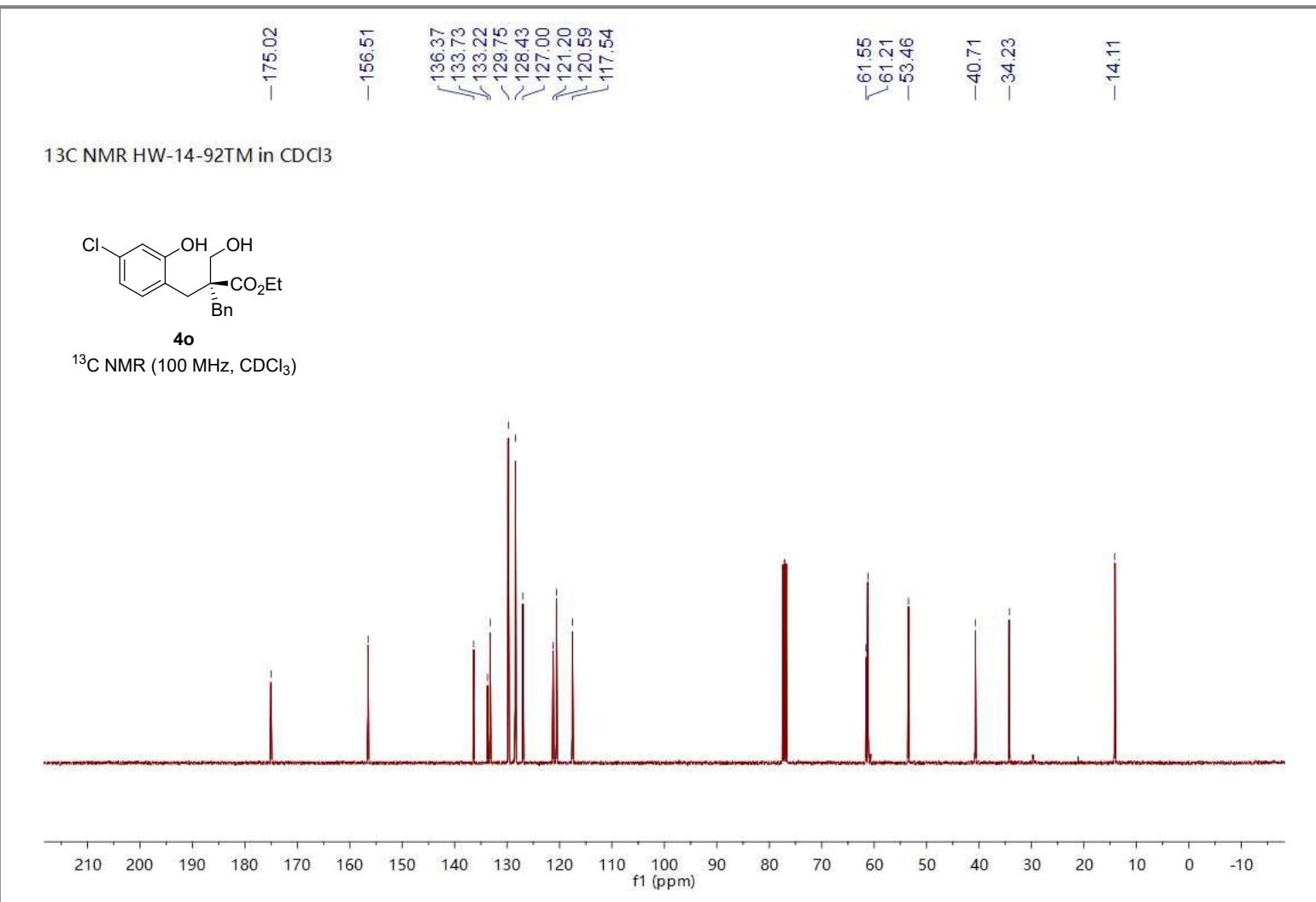


¹³C NMR HW-14-92TM in CDCl₃



4o

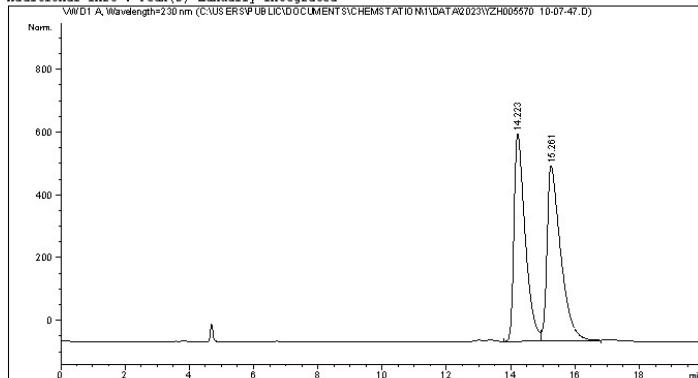
¹³C NMR (100 MHz, CDCl₃)



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005570 10-07-47.D
Sample Name: HW-14-92TM+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/29/2023 10:07:48 AM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 10:07:11 AM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 11:07:54 AM by SYSTEM
                           (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

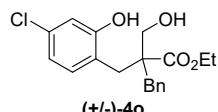


=====
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1	BV	0.3595	1.27398e4	529.96002	49.1258
2	VB	0.4330	1.31932e4	447.39484	50.8742



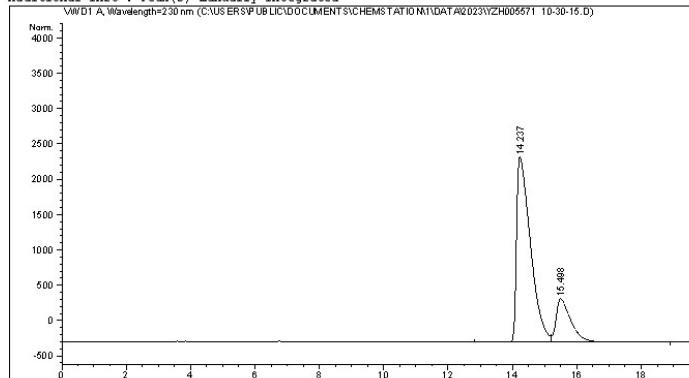
HPLC1260 II 11/29/2023 11:07:58 AM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005571 10-30-15.D
Sample Name: HW-14-92TM

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 11/29/2023 10:30:15 AM    Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 10:07:11 AM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 11/29/2023 11:08:45 AM by SYSTEM
                           (modified after loading)
Sample Info    : IB, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

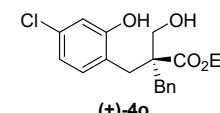


=====
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

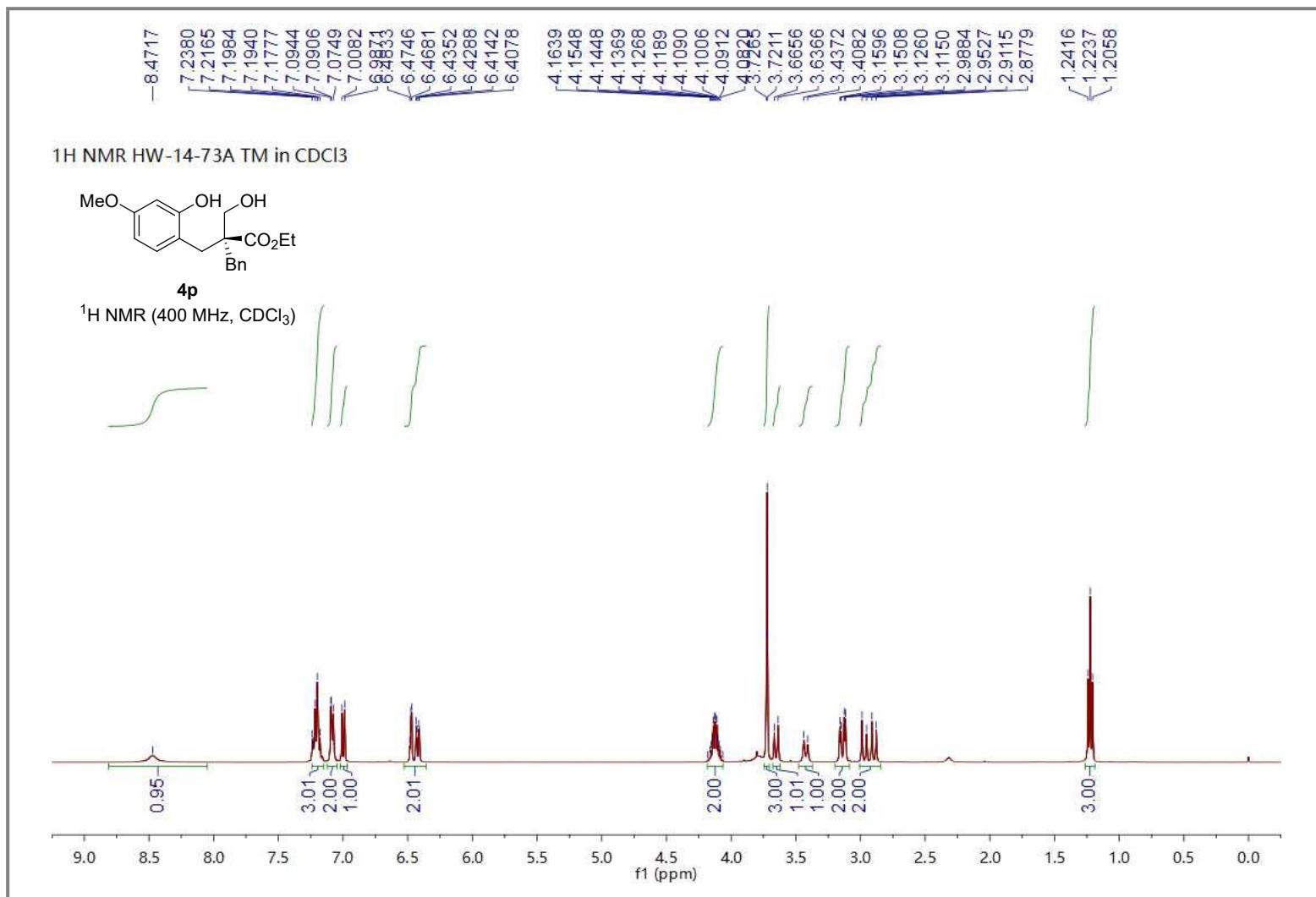
Signal 1: VWD1 A, Wavelength=230 nm

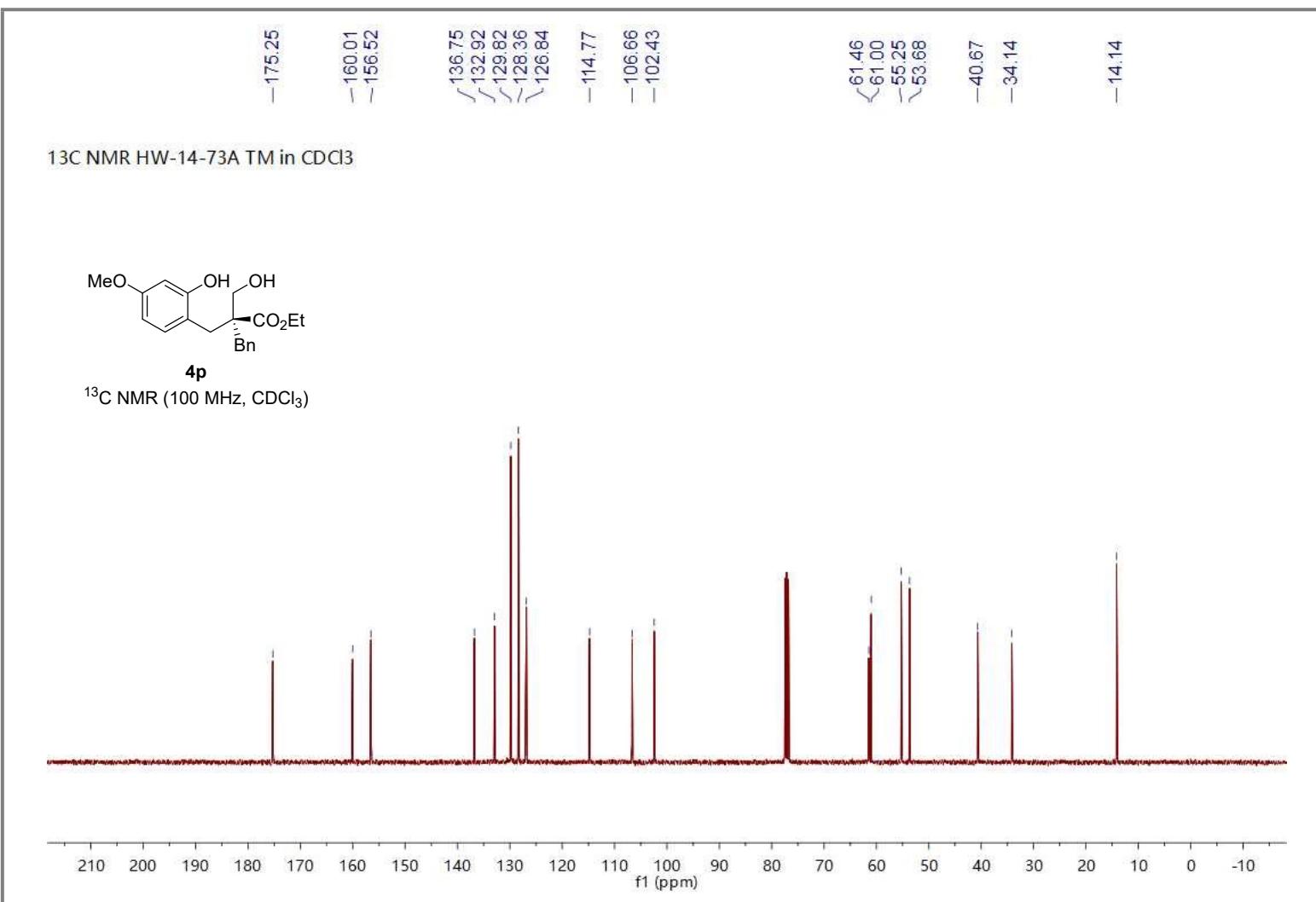
Peak RetTime	Type	Width	Area	Height	Area %
# [min]		[min]	[mAU*s]	[mAU]	%
1	VV R	0.4570	6.01549e4	2007.48206	79.6717
2	VV R	0.4721	1.53486e4	472.10394	20.3283



HPLC1260 II 11/29/2023 11:08:49 AM SYSTEM

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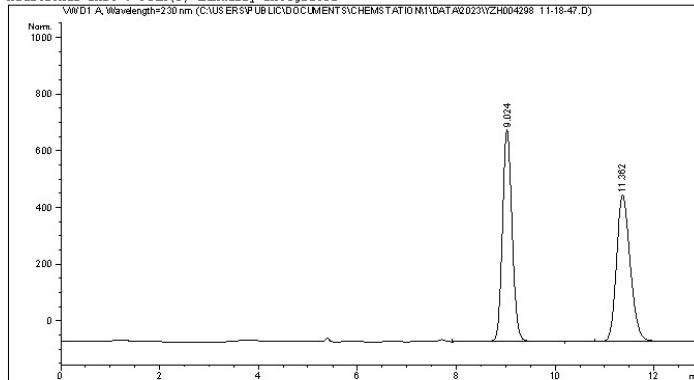




Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH004298 11-18-47.D
Sample Name: HW-14-73A TM+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 5/31/2023 11:18:47 AM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 5/31/2023 11:04:21 AM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/16/2023 10:16:44 PM by SYSTEM
                           (modified after loading)
Sample Info    : IC, Hexane/i-PrOH = 80/20, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated



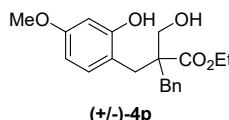
```
=====
Area Percent Report
=====

Sorted By       : Signal
Multiplier      : 1.0000
Dilution       : 1.0000
Sample Amount: : 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak	RetTime	Type	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	9.024	VB R	0.2111	8211.14258	602.08978	50.5086
2	11.362	BB	0.2986	8045.78271	416.01419	49.4914

HPLC1260 II 10/16/2023 10:16:49 PM SYSTEM

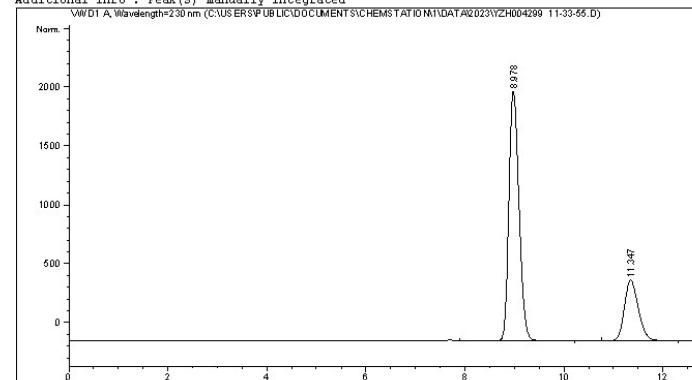


Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH004299 11-33-55.D
Sample Name: HW-14-73A TM

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 5/31/2023 11:33:56 AM      Inj : 1
Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 5/31/2023 11:04:21 AM by SYSTEM
                           (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 10/16/2023 10:18:10 PM by SYSTEM
                           (modified after loading)
Sample Info    : IC, Hexane/i-PrOH = 80/20, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

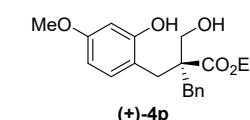


```
=====
Area Percent Report
=====
```

```
Sorted By       : Signal
Multiplier      : 1.0000
Dilution       : 1.0000
Sample Amount: : 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

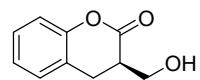
Peak	RetTime	Type	Width	Area	Height	Area
#	[min]		[min]	[mAU*s]	[mAU]	%
1	8.978	VB R	0.2214	2.56802e4	1806.03650	74.7429
2	11.347	BB	0.3036	8677.82031	440.92514	25.2571



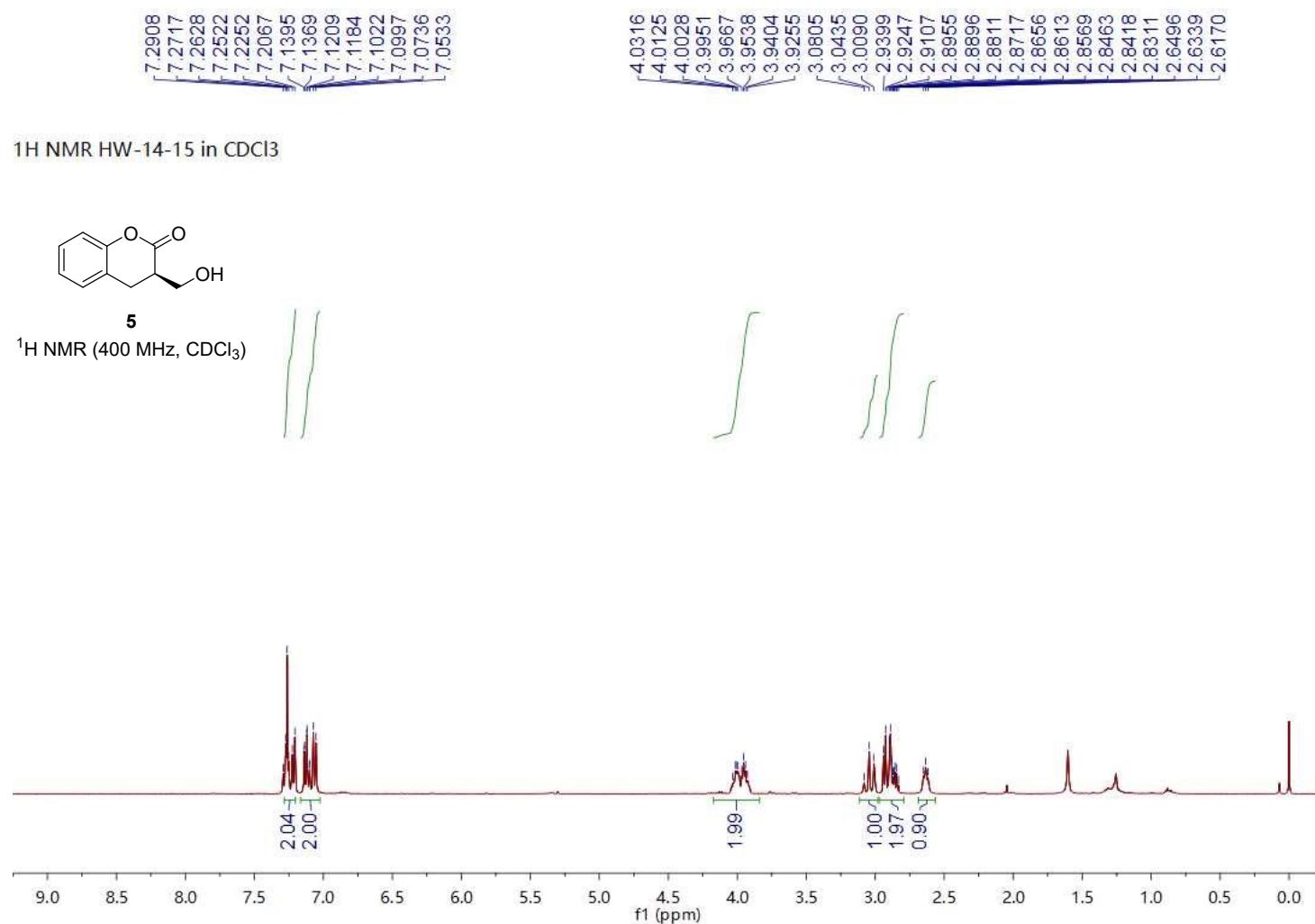
HPLC1260 II 10/16/2023 10:18:14 PM SYSTEM

Page 1 of 2

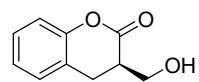
¹H NMR HW-14-15 in CDCl₃



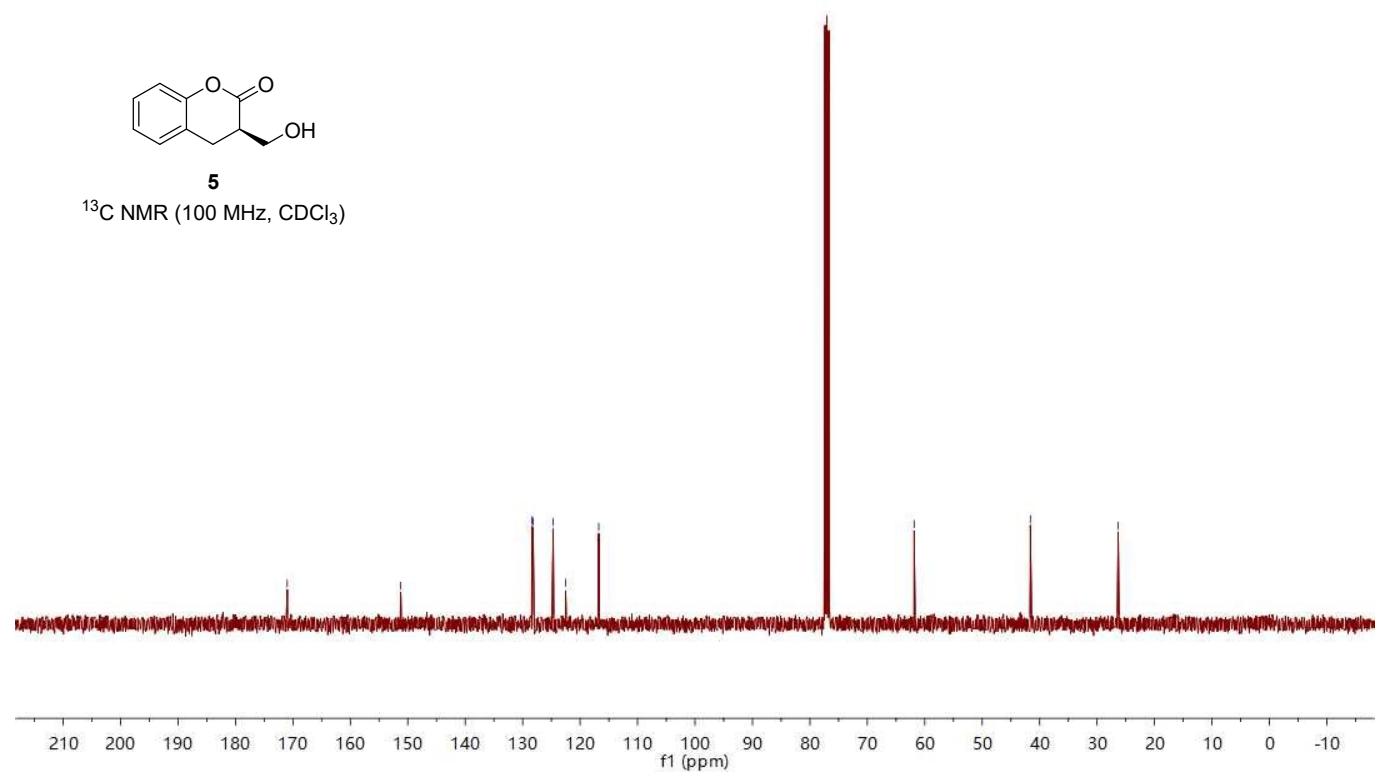
5
¹H NMR (400 MHz, CDCl₃)



¹³C NMR HW-14-15 in CDCl₃

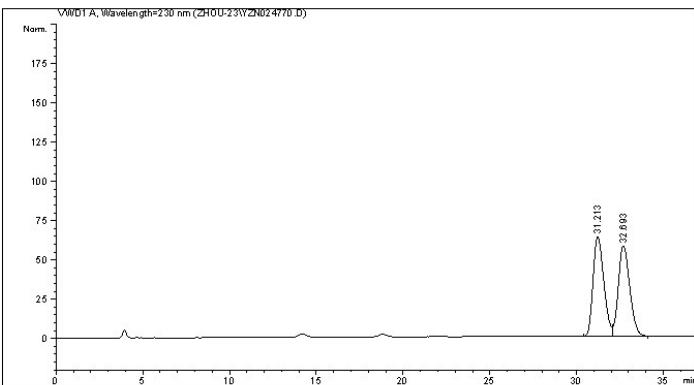


5
¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN024770.D
Sample Name: HW-16-24+/-

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 12/9/2023 2:58:54 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/9/2023 2:41:50 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/23/2023 3:47:04 AM
(modified after loading)
Sample Info : AS-H, Hexane/iPrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

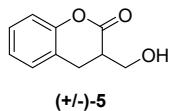


=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

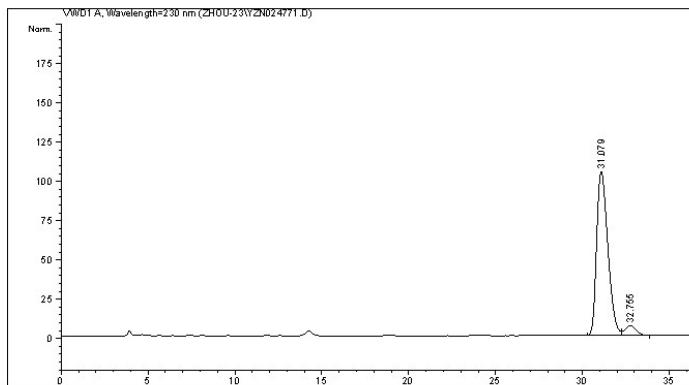
Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	31.213	BV	0.6762	2733.57471	63.41490	50.8524	
2	32.693	VB	0.7076	2641.93481	57.67999	49.1476	
Totals :				5375.50952	121.09489		



Data File C:\CHEM32\1\DATA\ZHOU-23\YZN024771.D
Sample Name: HW-16-24

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 12/9/2023 3:41:38 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/9/2023 3:41:03 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/23/2023 3:47:04 AM
(modified after loading)
Sample Info : AS-H, Hexane/iPrOH = 90/10, 0.8 mL/min, 30 oC, 230 nm
```

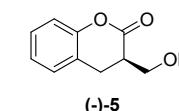


=====
Area Percent Report
=====

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height *s	[mAU]	Area %
1	31.079	BV	0.6996	4687.87695	104.49860	94.3382	
2	32.755	VB	0.6432	281.34866	6.35522	5.6618	
Totals :				4969.22562	110.85381		



Instrument 1 12/23/2023 3:47:17 AM

Page 1 of 1

Instrument 1 12/23/2023 3:48:13 AM

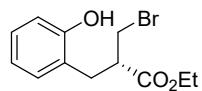
Page 1 of 1

7.1394
7.1332
7.1295
7.1220
7.1149
7.1114
7.1046
6.8805
6.8620
6.8434
6.8231
6.8103
6.8029
6.2113

4.2433
4.2255
4.2163
4.2077
4.1985
4.1894
4.1807
4.1715
4.1627
4.1536
4.1446
4.1358
4.1267
4.1089
3.6060
3.5919

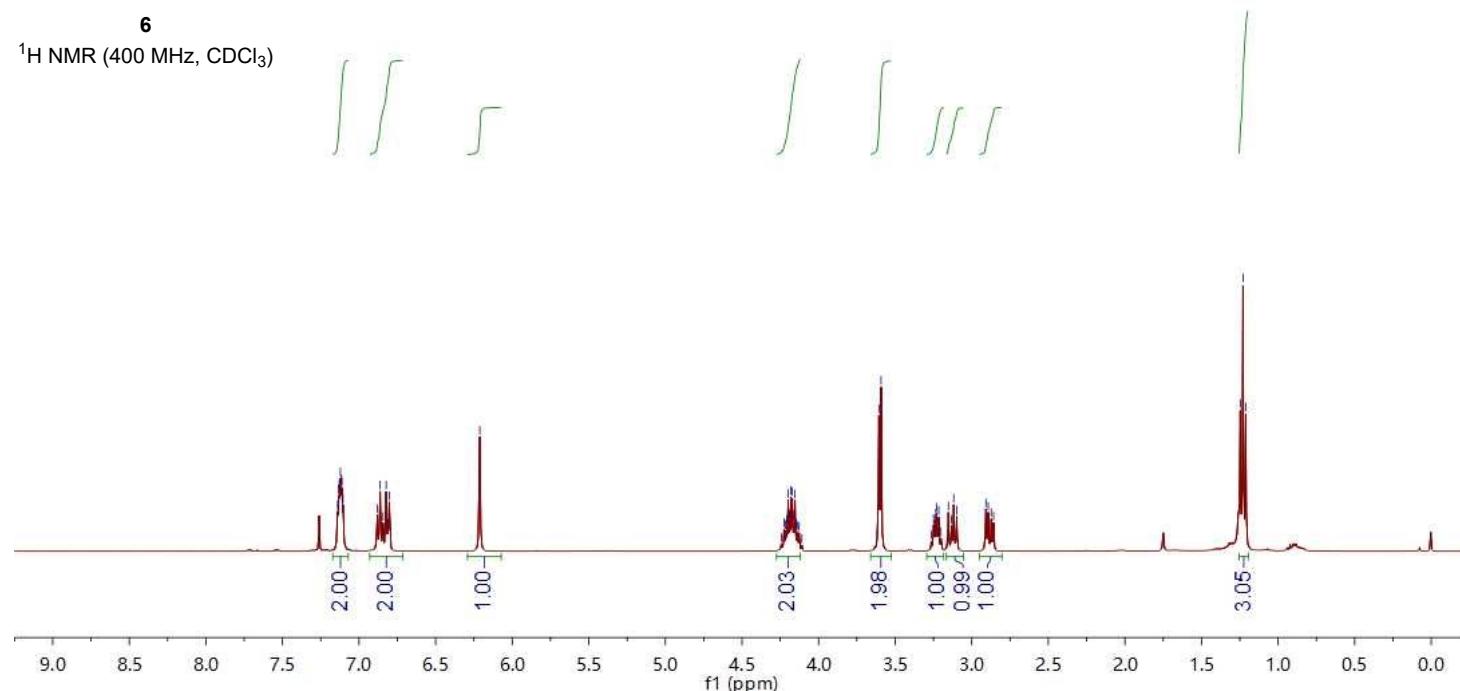
3.2491
3.2344
3.2298
3.2152
3.1523
3.1325
3.1178
3.0980
2.9074
2.8923
2.8729
2.8578
1.2290
1.2112

¹H NMR HW-13-85 in CDCl₃

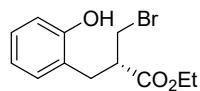


6

¹H NMR (400 MHz, CDCl₃)

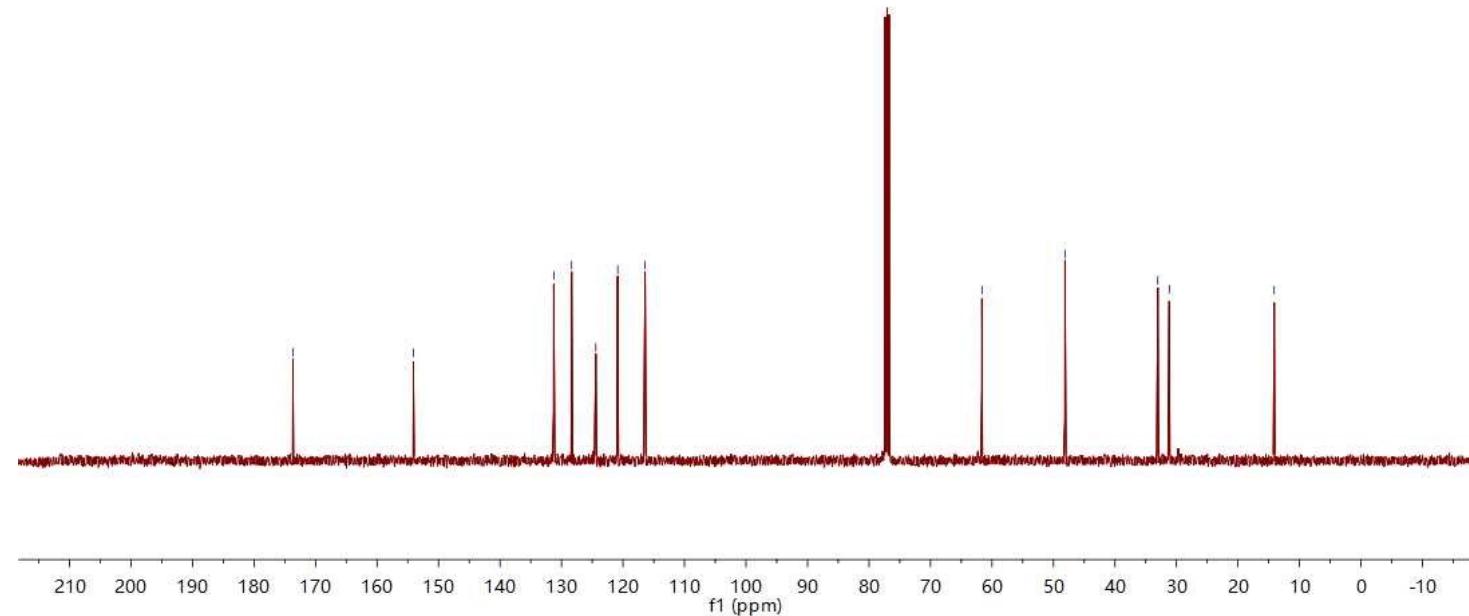


¹³C NMR HW-13-85 in CDCl₃



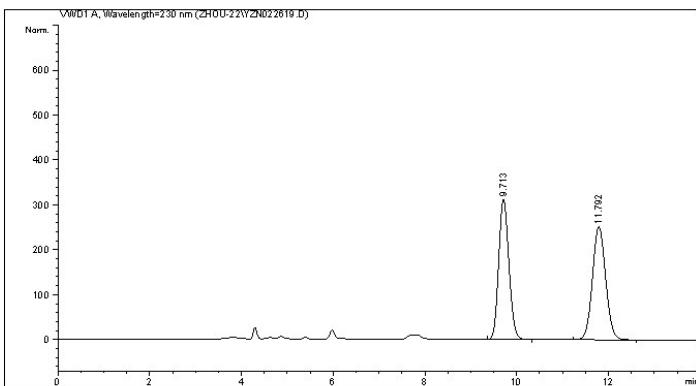
6

¹³C NMR (100 MHz, CDCl₃)



Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022619.D
Sample Name: HW-13-90+-

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1
Injection Date : 3/13/2023 8:44:04 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/13/2023 8:28:37 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/23/2023 3:38:34 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 75/25, 0.8 mL/min, 30 oC, 230nm
```



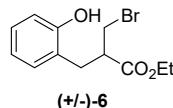
=====
Area Percent Report

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak	RetTime	Type	Width	Area	Height	Area	
#	[min]		[min]	[mAU]	*s	[mAU]	%
1	9.713	VB	0.2424	4844.71729	311.80060	48.9922	
2	11.792	BB	0.3125	5044.02588	251.65918	51.0078	

Totals : 9888.74316 563.45978

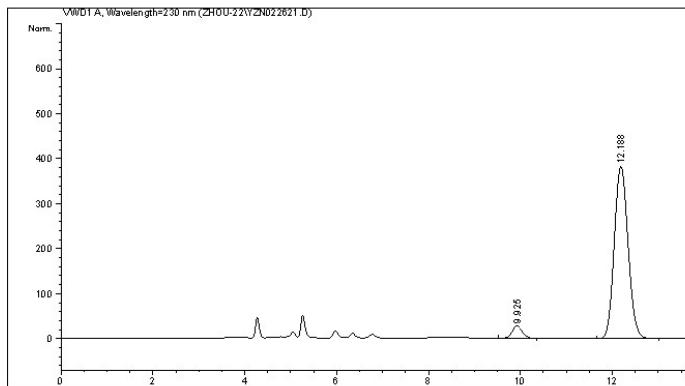


Instrument 1 12/23/2023 3:38:38 AM

Page 1 of 1

Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022621.D
Sample Name: HW-13-96

```
=====
Acq. Operator :                               Location : -
Acq. Instrument : Instrument 1
Injection Date : 3/13/2023 9:20:49 PM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 3/13/2023 9:20:02 PM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/23/2023 3:38:34 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 75/25, 0.8 mL/min, 30 oC, 230nm
```



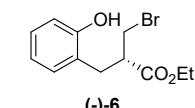
=====
Area Percent Report

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak	RetTime	Type	Width	Area	Height	Area	
#	[min]		[min]	[mAU]	*s	[mAU]	%
1	9.713	VB	0.2418	426.03473	27.28893	5.0749	
2	12.188	BB	0.3261	7968.93896	382.44785	94.9251	

Totals : 8394.97369 409.73677



Instrument 1 12/23/2023 3:39:19 AM

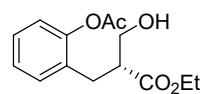
Page 1 of 1

7.2651
7.1981
7.1797
7.1619
7.0626
7.0428

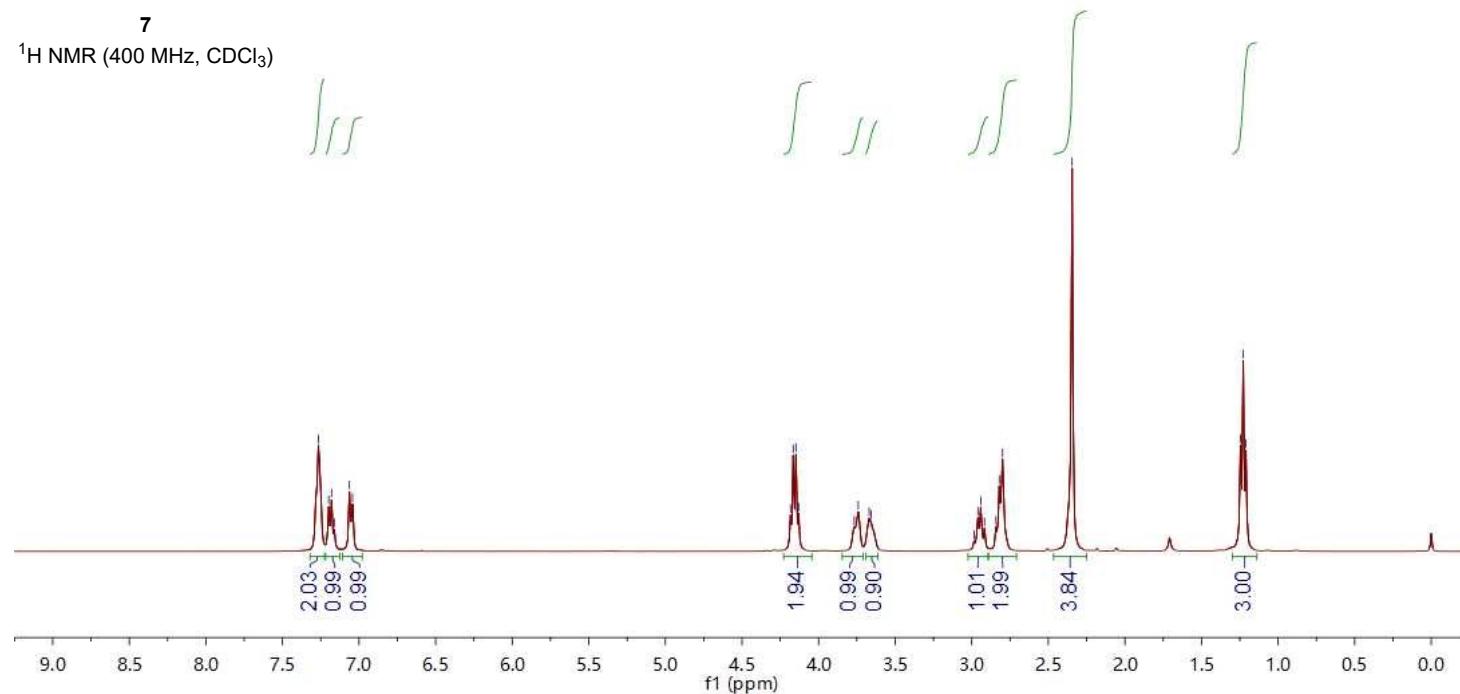
4.1833
4.1656
4.1479
4.1304
3.7409
3.6698
3.6576
2.9599
2.9413
2.8416
2.8204
2.7987

1.2450
1.2273
1.2097

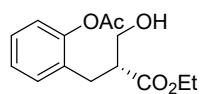
¹H NMR HW-16-31 in CDCl₃



¹H NMR (400 MHz, CDCl₃)

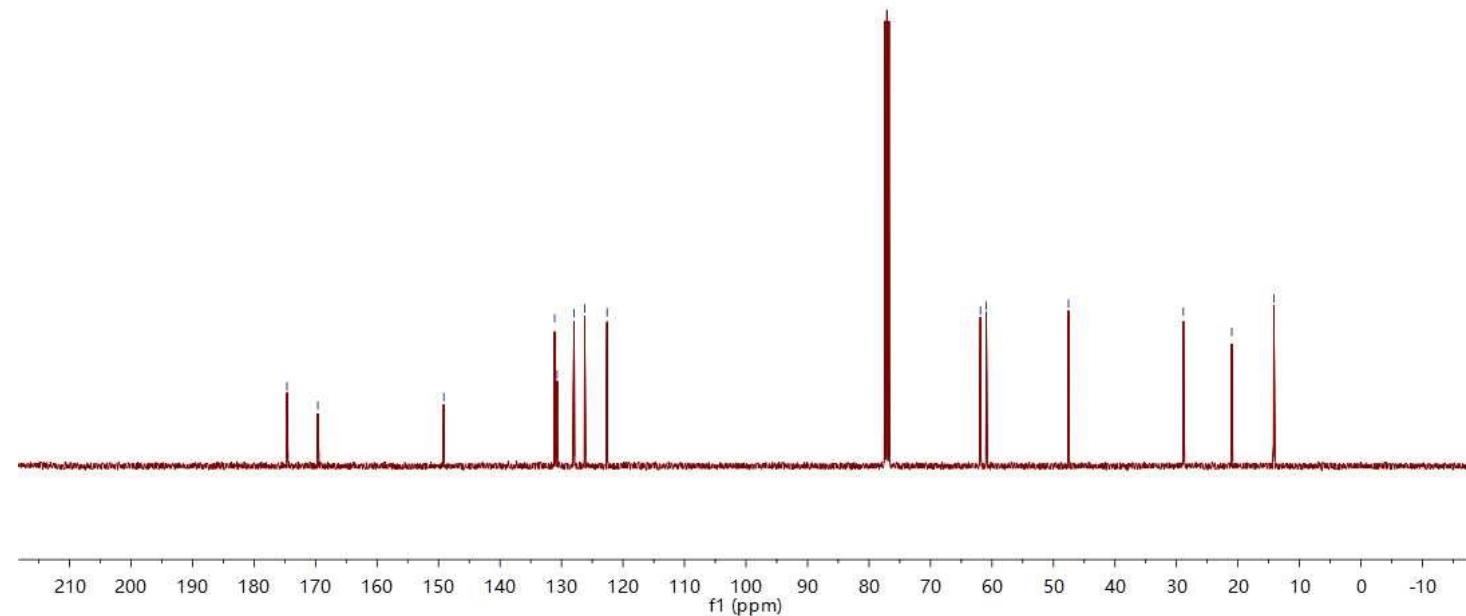


¹³C NMR HW-16-31 in CDCl₃



7

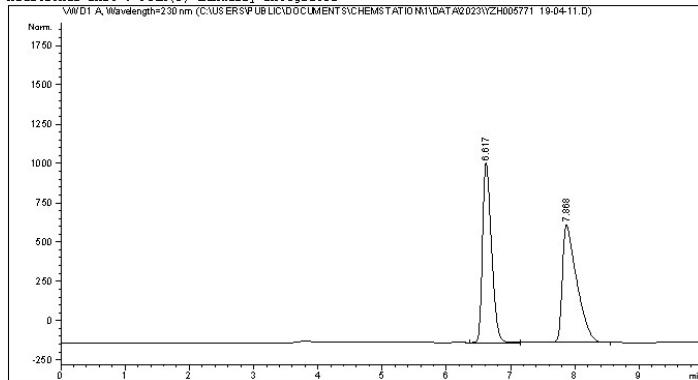
¹³C NMR (100 MHz, CDCl₃)



Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005771 19-04-11.D
Sample Name: HW-16-31+/-

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 12/15/2023 7:04:11 PM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 12/15/2023 7:01:30 PM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 12/22/2023 3:55:20 PM by SYSTEM
                                                (modified after loading)
Sample Info    : 03-H, Hexane/i-PrOH = 70/30, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

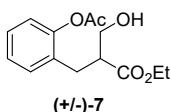


```
=====
Area Percent Report
=====

Sorted By       : Signal
Multiplier      : 1.0000
Dilution       : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=230 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.617	BB	0.1490	8325.12598	859.45837	48.6079
2	7.868	VB R	0.2215	8801.97461	565.45422	51.3921



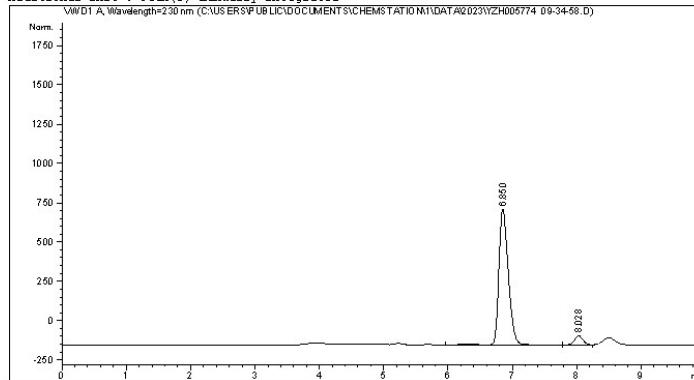
HPLC1260 II 12/22/2023 3:55:26 PM SYSTEM

Page 1 of 2

Data File C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\DATA\2023\YZH005774 09-34-58.D
Sample Name: HW-16-31

```
=====
Acq. Operator : SYSTEM
Sample Operator : SYSTEM
Acq. Instrument : HPLC1260 II          Location : 1
Injection Date : 12/16/2023 9:34:58 AM      Inj : 1
                                                Inj Volume : No inj
Acq. Method   : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 12/16/2023 9:18:01 AM by SYSTEM
                                                (modified after loading)
Analysis Method : C:\USERS\PUBLIC\DOCUMENTS\CHEMSTATION\1\METHODS\def_LC1.M
Last changed   : 12/22/2023 3:55:20 PM by SYSTEM
                                                (modified after loading)
Sample Info    : 03-H, Hexane/i-PrOH = 70/30, 0.8 mL/min, 30 oC, 230 nm
```

Additional Info : Peak(s) manually integrated

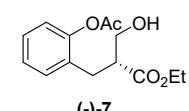


```
=====
Area Percent Report
=====

Sorted By       : Signal
Multiplier      : 1.0000
Dilution       : 1.0000
Sample Amount: 1.00000 [ng/uL] (not used in calc.)
Do not use Multiplier & Dilution Factor with ISTDs
```

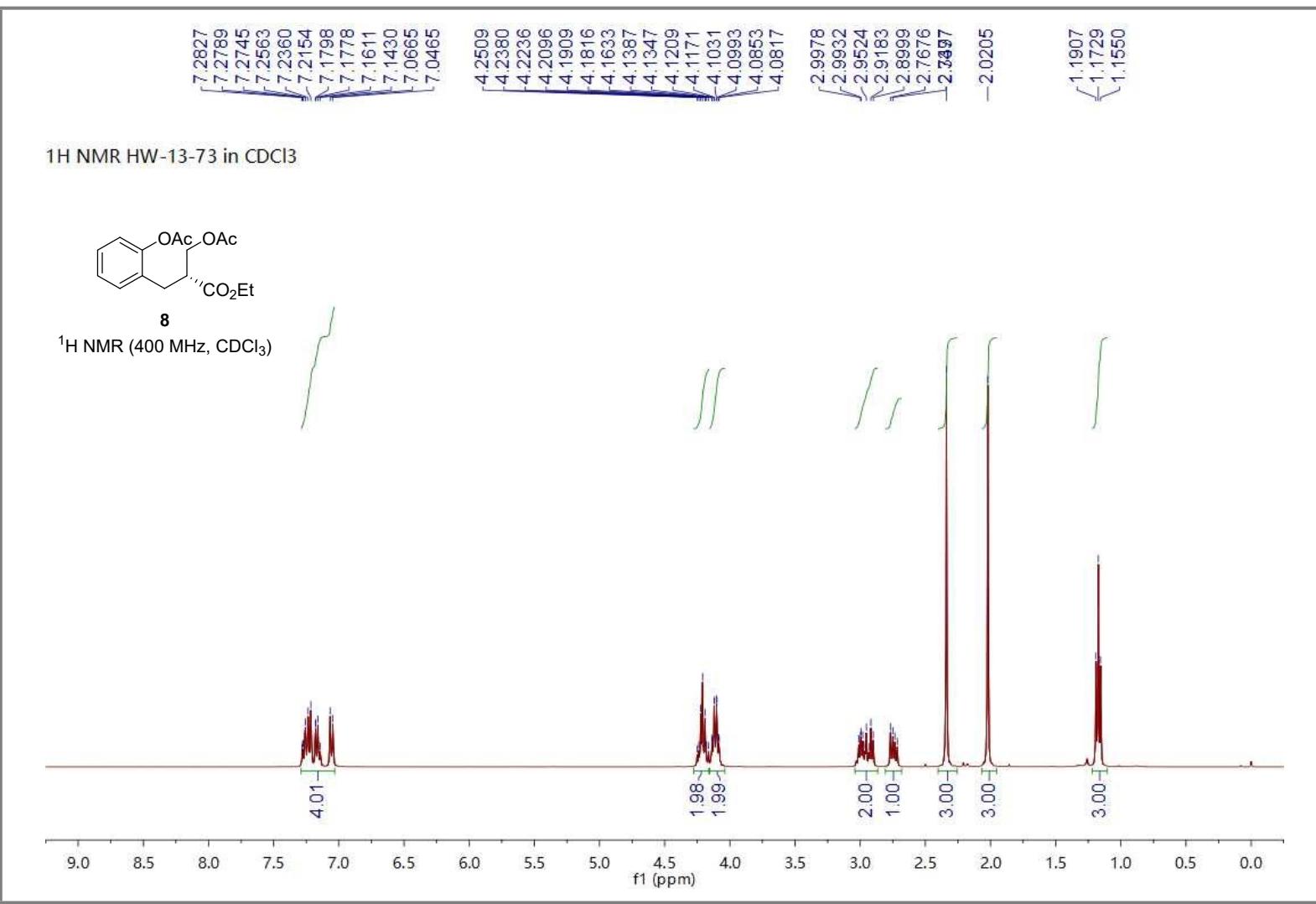
Signal 1: VWD1 A, Wavelength=230 nm

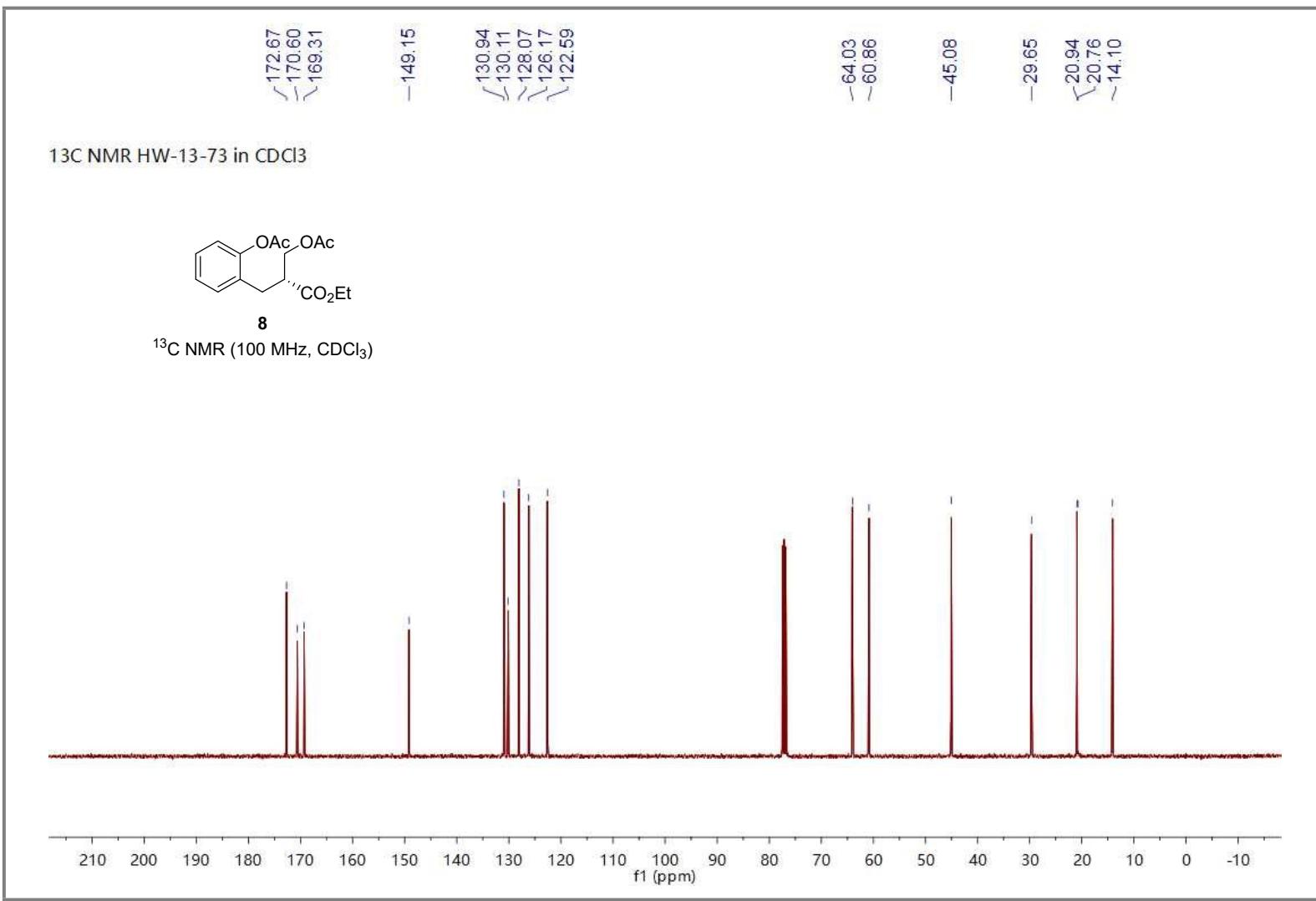
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.850	VB R	0.1558	5621.48438	553.58759	94.0795
2	8.028	BV	0.1451	353.76694	37.84434	5.9205



HPLC1260 II 12/22/2023 3:57:05 PM SYSTEM

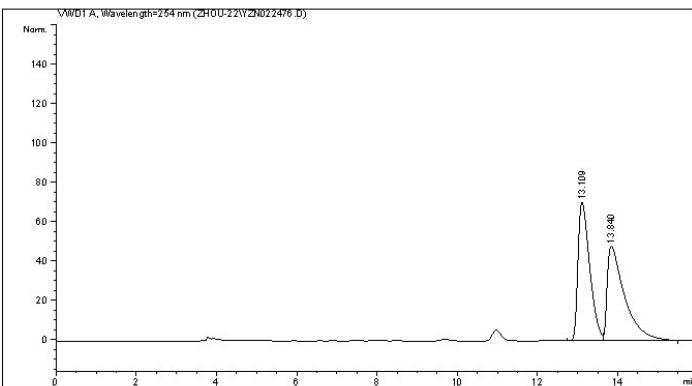
Page 1 of 2





Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022476.D
Sample Name: HW-13-73+-

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 2/23/2023 3:08:54 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 2/23/2023 2:29:51 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/23/2023 3:34:58 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 254 nm
```



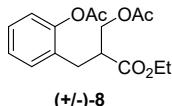
```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	13.109	VV	0.3114	1430.85986	70.39180	49.5265	
2	13.840	VB	0.4342	1458.21667	48.03914	50.4735	

Totals : 2889.07654 118.43094



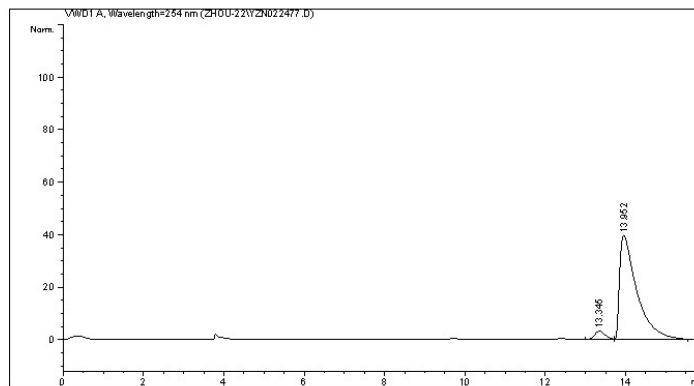
*** End of Report ***

Instrument 1 12/23/2023 3:35:17 AM

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Data File C:\CHEM32\1\DATA\ZHOU-22\YZN022477.D
Sample Name: HW-13-73+

```
=====
Acq. Operator : Instrument 1 Location : -
Injection Date : 2/23/2023 3:27:49 AM
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 2/23/2023 3:26:32 AM
(modified after loading)
Analysis Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 12/23/2023 3:37:20 AM
(modified after loading)
Sample Info : OD-H, Hexane/i-PrOH = 95/5, 0.8 mL/min, 30 oC, 254 nm
```



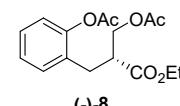
```
=====
Area Percent Report
```

```
Sorted By : Signal
Multiplier: : 1.0000
Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	*s	Height [mAU]	Area %
1	13.345	BV	0.2852	59.21564	3.20821	4.7798	
2	13.952	VB	0.4298	1179.64685	39.68708	95.2202	

Totals : 1238.86249 42.89529



*** End of Report ***

Instrument 1 12/23/2023 3:37:23 AM

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