Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Enantioselective Synthesis of Chiral Multicyclic γ-Lactones *via* Dynamic Kinetic Resolution of Racemic γ-Keto Carboxylic Acids

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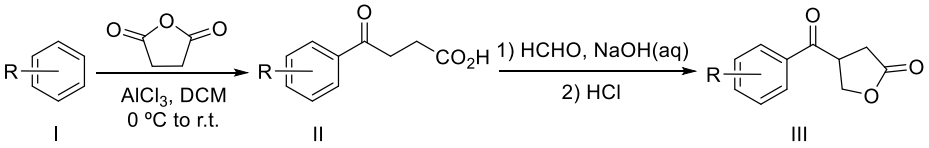
1. General Information

Unless otherwise noted, all reagents and solvents were purchased from commercial suppliers and used without further purification. NMR spectra were recorded on Bruker ADVANCE III (400 MHz) spectrometers for ^{1}H NMR and ^{13}C NMR. CD_3OD and $CDCl_3$ were the solvents used for the NMR analysis, with tetramethylsilane as the internal standard. Chemical shifts were reported upfield to TMS (0.00 ppm) for ^{1}H NMR and relative to $CDCl_3$ (77.3 ppm) for ^{13}C NMR. Optical rotation was determined using a Perkin Elmer 343 polarimeter. HPLC analysis was conducted on an Agilent 1260 Series instrument. Column Chromatography was performed with silica gel Merck 60 (300-400 mesh). All new products were further characterized by HRMS. A positive ion mass spectrum of sample was acquired on a Thermo LTQ-FT mass spectrometer with an electrospray ionization source.

2. General procedure for the synthesis of β-keto carboxylic acids



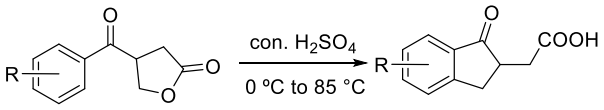
Preparation of III according to the literature [1][2]:

To a solution of substituted benzene (I, 75 mmol) and succinic anhydride (50 mmol) dissolved in 100 mL of anhydrous CH_2Cl_2 was slowly added aluminum chloride (100 mmol) at 0 ℃. After completion, the reaction mixture was warmed to room temperature and stirred overnight. Then the reaction mixture was cooled to 0 ℃ and 6N HCl was added carefully. When all aluminum chloride was quenched, the solution was extracted with CH_2Cl_2 three times and the combined organic phase was washed with H_2O and brine and dried over Na_2SO_4, filtered and concentrated under vacuum. Compound II can be used directly without further purification.

β-benzoyl propionic acid (compound II, 50 mmol) was added to a solution of 0.5M aqueous NaOH (110 mL). Formalin (37%, 4.1 mL) was added to the above solution and the reaction mixture was stirred at room temperature for 1h. Concentrated HCl (32%, 7.2 mL) was added and the solution was stirred overnight. When the reaction

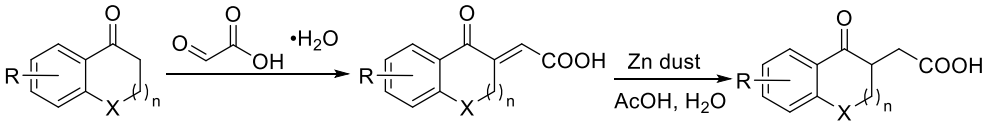
was completed, the mixture was extracted with ethyl acetate three times and washed with brine and dried over Na_2SO_4. The solvent was removed under vacuum and the residue was purified by chromatography on silica gel using hexanes-ethyl acetate (5:1) as eluent to give the target compound III.

*Preparation of V (**1a-1o**) [3]:*



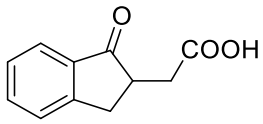
III IV

Compound III (10 mmol) was added slowly to 5 mL concentrated H_2SO_4 at 0 ℃ and then heated to 85 ℃ for 3h. The solution was poured into a beaker containing ice-water mixture. The mixture was extracted with DCM for three times, washed with brine and dried over Na_2SO_4. The organic layer was concentrated under vacuum and the residue was purified by chromatography on silica gel using hexanes-ethyl acetate (3:1) and 0.5 % acetic acid as eluent to give the target compound.



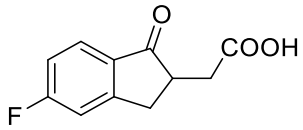
V VI VII

To a round bottom flask was added ketone V (10 mmol) and glyoxylic acid (15 mmol). The mixture was stirred at 95 ℃ for 3h. The reaction mixture was dissolved in acetic acid (15 mL) and water (5 mL). Zinc dust (15 mL) was added over 1h to the solution and the mixture was stirred for additional 3h. The mixture was diluted with ethyl acetate and then filtered through celite. The filtration was extracted with ethyl acetate, washed with brine, dried over Na_2SO_4 and concentrated under vacuum. The residue was purified by chromatography on silica gel using hexanes-ethyl acetate (3:1) and 0.5 % acetic acid as eluent to give the target compound.



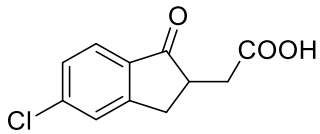
2-(1-oxo-2,3-dihydro-1*H*-inden-2-yl) acetic acid (1a)

White solid, 5 g, 41 % yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.75 (d, *J* = 7.6 Hz, 1H), 7.69 (t, *J* = 7.2 Hz, 1H), 7.58 (d, *J* = 7.6 Hz, 1H), 7.45 (t, *J* = 7.4 Hz, 1H), 3.51-3.47 (m, 1H), 3.05 - 2.88 (m, 3H), 2.76 - 2.72 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 193.34, 173.57, 162.03, 135.82, 126.60, 121.05, 120.37, 117.56, 70.03, 42.45, 29.44. ESI-HRMS Calculated for $C_{11}H_{10}O_3^+$ ($[M+H]^+$): 190.0630, found: 190.0632.



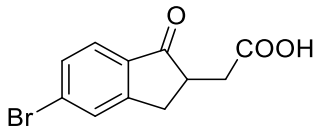
2-(5-fluoro-1-oxo-2,3-dihydro-1*H*-inden-2-yl) acetic acid (1b)

Light yellow solid, 0.48g, 70% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.74 (dd, *J* = 8.5, 5.4 Hz, 1H), 7.26 (dd, *J* = 8.8, 2.1 Hz, 1H), 7.15 (td, *J* = 8.9, 2.3 Hz, 1H), 3.52 - 3.35 (m, 1H), 3.05 - 2.80 (m, 3H), 2.77- 2.65 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 206.25, 173.86, 167.39 (d, J_{C-F} = 254.5 Hz), 157.13 (d, J_{C-F} = 10.1 Hz), 132.79, 125.64 (d, J_{C-F} = 11.1 Hz), 115.30, 115.06, 112.85(d, J_{C-F} = 23.23 Hz), 43.78, 33.98, 32.36, 32.34. ESI-HRMS Calculated for $C_{11}H_9FO_3^+$ ($[M+H]^+$): 208.0536, found: 208.0533.



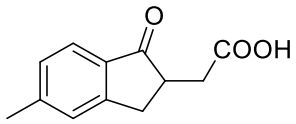
2-(5-chloro-1-oxo-2,3-dihydro-1*H*-inden-2-yl) acetic acid (1c)

Light yellow solid, 0.41g, 71% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.66 (d, *J* = 8.2 Hz, 1H), 7.57 (s, 1H), 7.41 (dd, *J* = 8.2, 1.6 Hz, 1H), 3.46 - 3.40 (m, 1H), 3.03 - 2.81 (m, 3H), 2.76 - 2.70 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 207.23, 172.76, 167.45, 166.23, 157.18, 157.08, 132.79, 125.69, 125.58, 115.30, 115.06, 112.96, 112.73, 43.78, 33.98, 32.36, 32.34. ESI-HRMS Calculated for $C_{11}H_9FO_3^+$ ($[M+H]^+$): 224.0240, found: 224.0238.



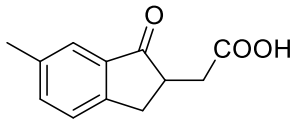
2-(5-bromo-1-oxo-2,3-dihydro-1*H*-inden-2-yl) acetic acid (1d)

Light yellow solid, 0.45g, 68% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.76 (s, 1H), 7.64 - 7.51 (m, 2H), 3.52 -3.36 (m, 1H), 3.03 - 2.80 (m, 3H), 2.80 - 2.65 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 208.13, 172.76, 167.45, 166.23, 157.18, 157.08, 132.79, 125.69, 125.58, 115.30, 115.06, 112.96, 112.73, 43.78, 33.98, 32.36, 32.34. ESI-HRMS Calculated for $C_{11}H_9BrO_3^+$ ([M+H]$^+$): 267.9735, found: 267.9738.



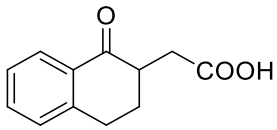
2-(5-methyl-1-oxo-2,3-dihydro-1*H*-inden-2-yl) acetic acid (1e)

White solid, 0.4g, 75% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.58 (d, *J* = 7.9 Hz, 1H), 7.34 (s, 1H), 7.26-7.17 (m, 1H), 3.41 - 3.35 (m, 1H), 2.97 - 2.91 (m, 1H), 2.89 - 2.83 (m, 2H), 2.67 - 2.61 (m, 1H), 2.43 (s, 3H). ^{13}C NMR (101 MHz, CD_3OD) δ 207.78, 174.04, 154.52, 146.44, 133.80, 128.36, 126.66, 123.05, 43.71, 34.17, 32.31, 20.68. ESI-HRMS Calculated for $C_{12}H_{12}O_3^+$ ([M+H]$^+$):204.0786, found: 204.0784.



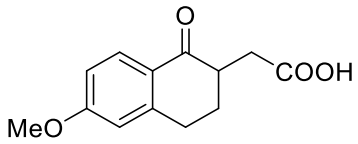
2-(6-methyl-1-oxo-2,3-dihydro-1*H*-inden-2-yl) acetic acid (1f)

White solid, 0.43g, 74% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.66 - 7.14 (m, 3H), 3.41 - 3.34 (m, 1H), 2.98 - 2.81 (m, 3H), 2.71 - 2.64 (m, 1H), 2.39 (s, 3H). ^{13}C NMR (101 MHz, CD_3OD) δ 208.62, 208.27, 174.03, 151.34, 137.32, 136.28, 136.04, 135.15, 127.35, 126.09, 123.01, 120.56, 43.91, 43.54, 34.19, 34.15, 32.08, 31.28, 19.67, 16.36. ESI-HRMS Calculated for $C_{12}H_{12}O_3^+$ ([M+H]$^+$):204.0786, found: 204.0785.



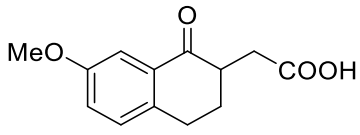
2-(1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl) acetic acid (1g)

White solid, 0.38g, 40% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.99 - 7.84 (m, 1H), 7.52 - 7.48 (m, 1H), 7.42 - 7.23 (m, 2H), 3.18 - 3.10 (m, 1H), 3.06 - 2.93 (m, 2H), 2.86 (dd, *J* = 16.6, 5.9 Hz, 1H), 2.47 (dd, *J* = 16.6, 6.4 Hz, 1H), 2.27 - 2.21 (m, 1H), 2.06 - 1.90 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 199.12, 174.76, 144.51, 133.30, 131.96, 128.67, 126.65, 126.24, 44.68, 34.37, 28.97, 28.80. ESI-HRMS Calculated for $C_{12}H_{12}O_3^+$ ($[M+H]^+$): 204.0786, found: 204.0788.



2-(6-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl) acetic acid (1h)

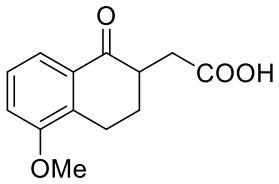
White solid, 0.51g, 50% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 8.12 - 7.68 (m, 1H), 6.95 - 6.64 (m, 2H), 3.86 (s, 3H), 3.19 - 3.02 (m, 1H), 3.02 - 2.77 (m, 3H), 2.50 - 2.35 (m, 1H), 2.33 - 2.13 (m, 1H), 2.10 - 1.85 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 202.03, 178.81, 167.98, 151.14, 133.08, 129.32, 116.98, 116.09, 58.56, 48.32, 38.39, 33.09, 32.97. ESI-HRMS Calculated for $C_{13}H_{14}O_4^+$ ($[M+H]^+$): 234.0892, found: 234.0891.



2-(7-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl) acetic acid (1i)

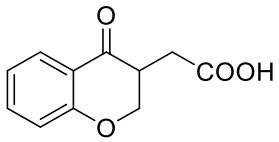
White solid, 0.48g, 49% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.43 (d, *J* = 2.8 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.09 (dd, *J* = 8.4, 2.8 Hz, 1H), 3.80 (s, 3H), 3.09 - 2.81 (m, 4H), 2.47 (dd, *J* = 16.5, 6.4 Hz, 1H), 2.28 - 2.16 (m, 1H), 2.03 - 1.87 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 199.31, 174.82, 156.94, 133.01, 132.93, 126.68, 118.14,

114.23, 54.82, 44.13, 34.26, 28.21, 22.33. ESI-HRMS Calculated for $C_{13}H_{14}O_4^+$ ($[M+H]^+$): 234.0892, found: 234.0889.



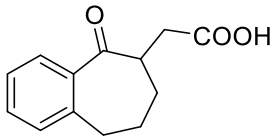
2-(5-methoxy-1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl) acetic acid (1j)

White solid, 0.48g, 48%yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.43 (d, *J* = 2.8 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.09 (dd, *J* = 8.4, 2.8 Hz, 1H), 3.80 (s, 3H), 3.10 - 2.78 (m, 4H), 2.47 (dd, *J* = 16.5, 6.4 Hz, 1H), 2.27 - 2.15 (m, 1H), 2.02 - 1.87 (m, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 199.00, 174.76, 158.42, 136.91, 132.72, 129.87, 120.96, 109.10, 54.42, 44.58, 34.42, 29.21, 27.98. ESI-HRMS Calculated for $C_{13}H_{14}O_4^+$ ($[M+H]^+$): 234.0892, found: 234.0890.



2-(4-oxochroman-3-yl) acetic acid (1k)

White solid, 0.45g, 40% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.82 (dd, *J* = 7.9, 1.7 Hz, 1H), 7.54 - 7.50 (m, 1H), 7.12 - 6.92 (m, 2H), 4.61 (dd, *J* = 11.2, 5.4 Hz, 1H), 4.33 (dd, *J* = 12.3, 11.3 Hz, 1H), 3.30 - 3.21 (m, 1H), 2.81 (dd, *J* = 17.2, 5.1 Hz, 1H), 2.48 (dd, *J* = 17.2, 7.3 Hz, 1H). ^{13}C NMR (101 MHz, CD_3OD) δ 193.34, 173.57, 162.03, 135.82, 126.60, 121.05, 120.37, 117.56, 70.03, 42.45, 29.44. ESI-HRMS Calculated for $C_{11}H_{10}O_4^+$ ($[M+H]^+$): 206.0579, found: 206.0576.

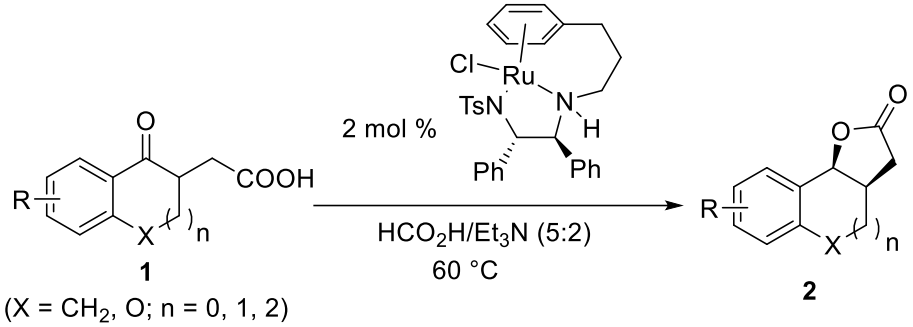


2-(5-oxo-6,7,8,9-tetrahydro-5H-benzo[7]annulen-6-yl) acetic acid (1l)

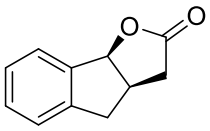
White solid, 0.39g, 38% yield; ^{1}H NMR (400 MHz, CD_3OD) δ 7.60 (dd, *J* = 8.0,

1.4 Hz, 1H), 7.43 - 7.39 (m, 1H), 7.31 - 7.23 (m, 2H), 3.41 - 3.32 (m, 1H), 3.18 - 3.05 (m, 1H), 3.02 - 2.84 (m, 2H), 2.52 - 2.47 (m, 1H), 2.15 - 2.12 (m, 1H), 1.99 - 1.94 (m, 1H), 1.73 - 1.50 (m, 2H). ^{13}C NMR (101 MHz, CD_3OD) δ 206.39, 174.56, 142.37, 139.29, 131.33, 129.69, 128.01, 125.97, 45.69, 35.61, 32.98, 29.42, 25.47. ESI-HRMS Calculated for $C_{13}H_{14}O_3^+$ ($[M+H]^+$): 218.0943, found: 218.0941.

3. General procedure for asymmetric hydrogenation of γ-keto carboxylic acids



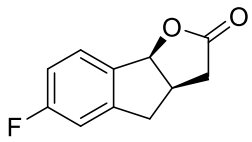
A suspension of γ-keto carboxylic acid (0.16 mmol), **Cat** (0.0032 mmol), 5:2 HCO_2H/Et_3N (1 mL) were stirred under N_2 at 60 ℃ for 12 h until completion according to TLC detection. 5.0 mL 2N HCl was added to the reaction, the mixture was then extracted with ethyl acetate (3 ×5mL) three times, washed with brine, dried over Na_2SO_4 and concentrated. The desired product was purified by silica gel chromatography (hexanes: ethyl acetate = 1:1). The enantioselectivity of the products was determined by HPLC analysis. The racemic samples of diastereomeric mixtures of **2a-2l** for HPLC analysis were prepared following literature procedures.



(3a*R*,8b*S*)-3,3a,4,8b-tetrahydro-2*H*-indeno[1,2-b]furan-2-one (2a)

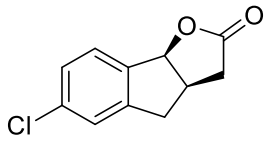
White solid, 25 mg, 90% yield, 98% ee, dr = 20:1; $[\alpha]_D^{20}$ = +100 (c = 0.985, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 85:15; flow rate = 0.8 mL/min; UV detection at 210 nm; t_R = 10.9 min (major), 12.0 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.4 Hz, 1H), 7.39 - 7.26 (m, 3H), 5.90 (d, *J* = 7.1 Hz, 1H), 3.48 - 3.21 (m, 2H), 3.04 - 2.73

(m, 2H), 2.47 - 2.27 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.97, 142.51, 138.76, 130.01, 127.60, 126.39, 125.37, 87.70, 37.93, 37.35, 35.70. ESI-HRMS Calculated for $C_{11}H_{10}O_2^+$ ($[M+H]^+$): 174.0681, found: 174.0680.



(3a*R*,8b*S*)-6-fluoro-3,3a,4,8b-tetrahydro-2*H*-indeno[1,2-b]furan-2-one (2b)

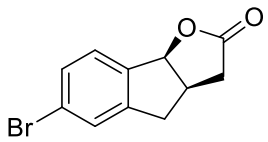
Light yellow solid, 27.5 mg, 90% yield, 98% ee, dr = 20:1; $[\alpha]_D^{20}$ = +84.8 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 17.8 min (major), 19.2 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 - 7.37 (m, 1H), 7.09 - 6.85 (m, 2H), 5.84 (d, *J* = 7.1 Hz, 1H), 3.43 - 3.39 (m, 1H), 3.32 (dd, *J* = 16.6, 8.5 Hz, 1H), 2.91 - 2.85 (m, 2H), 2.41 (dd, *J* = 4.0, 8.0 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.67, 164.17 (d, J_{C-F} = 249.47), 145.17(d, J_{C-F} = 8.1 Hz), 134.62, 127.83 (d, J_{C-F} = 9.1 Hz), 115.09 (d, J_{C-F} = 23.2 Hz), 112.15 (d, J_{C-F} = 22.2 Hz), 86.77, 38.02, 38.00, 37.92, 35.68. ESI-HRMS Calculated for $C_{11}H_9FO_2^+$ ($[M+H]^+$): 192.0587, found: 192.0585.



(3a*R*,8b*S*)-6-chloro-3,3a,4,8b-tetrahydro-2*H*-indeno[1,2-b]furan-2-one (2c)

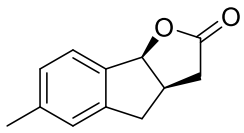
Light yellow solid, 30.6 mg, 92% yield, 99% ee, dr = 20:1; $[\alpha]_D^{20}$ = +73 (c = 0.6, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak IB column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 28.0 min (major), 30.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (d, *J* = 8.0 Hz, 1H), 7.29 (d, *J* = 1.8 Hz, 2H), 5.84 (d, *J* = 7.1 Hz, 1H), 3.46 - 3.35 (m, 1H), 3.31 (dd, *J* = 16.6, 8.4 Hz, 1H), 2.95 - 2.85 (m, 2H), 2.39 (dd, *J* = 18.2, 5.6 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 144.49, 137.31, 128.05, 127.47, 125.56, 86.72, 37.78, 37.65, 35.54.

ESI-HRMS Calculated for $C_{11}H_9ClO_2^+$ ([M+H]$^+$): 208.0291, found: 208.0292.



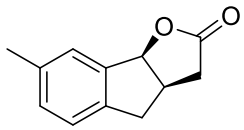
(3a*R*,8b*S*)-6-bromo-3,3a,4,8b-tetrahydro-2*H*-indeno[1,2-b]furan-2-one (2d)

Light yellow solid, 35.8 mg, 89% yield, 99% ee, dr=20:1; $[\alpha]_D^{20}$ = +62 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak IB column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 29.6 min (major), 32.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (d, *J* = 4.9 Hz, 2H), 7.35 (d, *J* = 8.5 Hz, 1H), 5.83 (d, *J* = 7.1 Hz, 1H), 3.43 - 3.36 (m, 1H), 3.31 (dd, *J* = 10.7, 5.8 Hz, 1H), 2.96 - 2.87 (m, 2H), 2.40 (dd, *J* = 13.4, 4.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.51, 144.78, 137.81, 130.92, 128.59, 127.80, 124.26, 86.76, 37.72, 37.57, 35.49. ESI-HRMS Calculated for $C_{11}H_9BrO_2^+$ ([M+H]$^+$): 251.9786, found: 251.9783.



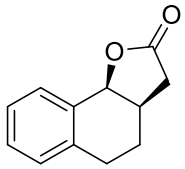
(3a*R*,8b*S*)-6-methyl-3,3a,4,8b-tetrahydro-2*H*-indeno[1,2-b]furan-2-one (2e)

White solid, 27.7 mg, 92% yield, 98% ee, dr = 20:1; $[\alpha]_D^{20}$ = +90.3 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralcel OD column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 23.3 min (minor), 24.3 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.7 Hz, 1H), 7.16 - 6.97 (m, 2H), 5.86 (d, *J* = 7.0 Hz, 1H), 3.42 - 3.31 (m, 1H), 3.32 - 3.21 (m, 1H), 2.93 - 2.86 (m, 2H), 2.43 - 2.37 (m, 1H), 2.36 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 177.06, 142.80, 140.14, 135.96, 128.53, 126.08, 125.84, 87.62, 37.85, 37.57, 35.81, 21.50. ESI-HRMS Calculated for $C_{12}H_{12}O_2^+$ ([M+H]$^+$): 188.0837, found: 188.0838.



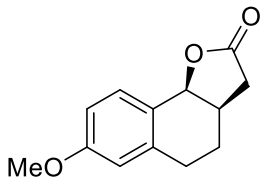
(3a*R*,8b*S*)-7-methyl-3,3a,4,8b-tetrahydro-2*H*-indeno[1,2-b]furan-2-one (2f)

White solid, 27.3 mg, 91% yield, 99% ee, dr = 20:1; $[\alpha]_D^{20}$ = +105.7 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 14.9 min (major), 20.3 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (s, 1H), 7.16 (s, 2H), 5.86 (d, *J* = 7.2 Hz, 1H), 3.38 - 3.34 (m, 1H), 3.27 (dd, *J* = 16.1, 8.4 Hz, 1H), 2.87 (m, 2H), 2.40 (dd, *J* = 15.4, 9.7 Hz, 1H), 2.36 (s, 3H).^{13}C NMR (101 MHz, $CDCl_3$) δ 177.03, 139.39, 138.90, 137.44, 130.96, 126.77, 125.06, 87.72, 37.60, 37.52, 35.73, 21.21. ESI-HRMS Calculated for $C_{12}H_{12}O_2^+$ ($[M+H]^+$): 188.0837, found: 188.0835.



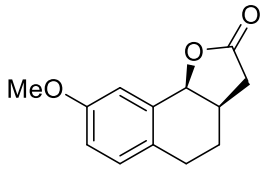
(3a*R*,9b*S*)-3a,4,5,9b-tetrahydronaphtho[1,2-b]furan-2(3*H*)-one (2g)

White solid, 26.1 mg, 87% yield, 99% ee, dr = 20:1; $[\alpha]_D^{20}$ = +37.4 (c = 0.5, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 19.6 min (minor), 23.0 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50 - 7.42 (m, 1H), 7.27 - 7.29 (m, 2H), 7.22 - 7.13 (m, 1H), 5.44 (d, *J* = 5.9 Hz, 1H), 2.93 (dd, *J* = 17.4, 8.1 Hz, 1H), 2.87 - 2.81 (m, 1H), 2.80 - 2.69 (m, 2H), 2.43 (dd, *J* = 17.4, 2.2 Hz, 1H), 1.93 - 1.90 (m, 1H), 1.70 - 1.56 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.37, 137.51, 131.11, 130.93, 128.92, 128.77, 126.73, 78.52, 36.43, 34.74, 27.77, 25.15. ESI-HRMS Calculated for $C_{12}H_{12}O_2^+$ ($[M+H]^+$): 188.0837, found: 188.0839.



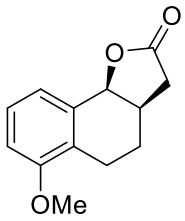
(3a*R*,9b*S*)-7-methoxy-3a,4,5,9b-tetrahydronaphtho[1,2-b]furan-2(3*H*)-one (2h)

White solid, 31.4 mg, 91% yield, 99% ee, dr = 20:1; $[\alpha]_D^{20}$ = +20.3 (c = 0.3, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD column, hexane: isopropanol = 90: 10; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 14.8 min (minor), 16.6 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.5 Hz, 1H), 6.83 (dd, *J* = 8.5, 2.5 Hz, 1H), 6.67 (d, *J* = 2.2 Hz, 1H), 5.40 (d, *J* = 5.7 Hz, 1H), 3.81 (s, 3H), 2.92 (dd, *J* = 17.4, 8.0 Hz, 1H), 2.89 - 2.78 (m, 1H), 2.76 - 2.66 (m, 2H), 2.44 - 2.39 (m, 1H), 1.90 - 1.87 (m, 1H), 1.67 - 1.57 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.48, 159.81, 139.14, 132.29, 123.41, 113.16, 113.03, 78.59, 55.31, 36.67, 34.81, 28.15, 25.07. ESI-HRMS Calculated for $C_{13}H_{14}O_3^+$ ($[M+H]^+$): 218.0943, found: 218.0944.



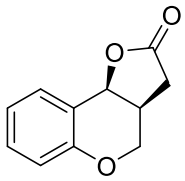
(3a*R*,9b*S*)-8-methoxy-3a,4,5,9b-tetrahydronaphtho[1,2-b]furan-2(3*H*)-one (2i)

White solid, 31.4 mg, 90% yield, 99% ee, dr = 20:1; $[\alpha]_D^{20}$ = +47.2 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 28.4 min (major), 29.9 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.08 (d, *J* = 8.5 Hz, 1H), 6.97 (d, *J* = 2.6 Hz, 1H), 6.86 (dd, *J* = 8.4, 2.7 Hz, 1H), 5.40 (d, *J* = 6.0 Hz, 1H), 3.80 (s, 3H), 2.92 (dd, *J* = 17.4, 8.1 Hz, 1H), 2.83 - 2.72 (m, 2H), 2.72 - 2.60 (m, 1H), 2.42 (dd, *J* = 17.4, 2.5 Hz, 1H), 1.92 - 1.89 (m, 1H), 1.69 - 1.54 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.38, 158.19, 132.00, 129.76, 129.52, 116.15, 114.37, 78.69, 55.42, 36.21, 34.67, 26.89, 25.43. ESI-HRMS Calculated for $C_{13}H_{14}O_3^+$ ($[M+H]^+$): 218.0943, found: 218.0941.



(3a*R*,9b*S*)-6-methoxy-3a,4,5,9b-tetrahydronaphtho[1,2-b]furan-2(3*H*)-one (2j)

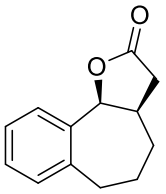
White solid, 32 mg, 92% yield, 99% ee, dr = 20:1; $[\alpha]_D^{20}$ = +25.3 (c = 0.8, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 25.3 min (major), 37.8 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 7.7 Hz, 1H), 6.83 (d, *J* = 8.1 Hz, 1H), 5.42 (d, *J* = 5.8 Hz, 1H), 3.84 (s, 3H), 2.99 (dt, *J* = 17.4, 4.4 Hz, 1H), 2.90 (dd, *J* = 17.3, 7.9 Hz, 1H), 2.78 - 2.67 (m, 1H), 2.50 - 2.38 (m, 2H), 1.96 - 1.90 (m, 1H), 1.61 - 1.50 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.48, 156.64, 132.08, 127.04, 126.71, 122.79, 109.85, 78.53, 55.49, 36.27, 34.35, 24.32, 20.88. ESI-HRMS Calculated for $C_{13}H_{14}O_3^+$ ($[M+H]^+$): 218.0943, found: 218.0942.



(3a*S*,9b*S*)-3a,9b-dihydro-4*H*-furo[3,2-c]chromen-2(3*H*)-one (2k)

White solid, 25.9 mg, 85% yield, 96% ee, dr = 20:1; $[\alpha]_D^{20}$ = +62.5 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95: 5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 27.5 min (major), 38.3 min (minor). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.33 - 7.28 (m, 1H), 7.04 - 7.00 (m, 1H), 6.92 (d, *J* = 8.3 Hz, 1H), 5.49 (d, *J* = 6.3 Hz, 1H), 4.21 (dd, *J* = 11.5, 4.3 Hz, 1H), 3.85 - 3.80 (m, 1H), 3.05 - 2.98 (m, 1H), 2.87 (dd, *J* = 17.7, 8.3 Hz, 1H), 2.46 (dd, *J* = 17.7, 4.2 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.43, 155.10, 131.44, 130.75, 121.97, 118.49, 117.45, 74.29, 64.85,

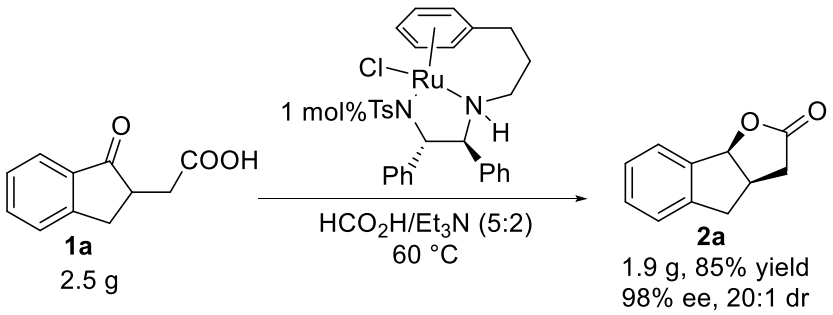
33.61, 31.17. ESI-HRMS Calculated for $C_{11}H_{10}O_3^+$ ($[M+H]^+$): 190.0630, found: 190.0631.



(3a*R*,10b*S*)-3,3a,4,5,6,10b-hexahydro-2*H*-benzo[6,7]cyclohepta[1,2-b]furan-2-one (2l)

White solid, 22.6 mg, 70% yield, 98% ee, dr = 20:1; $[\alpha]_D^{20}$ = -14.4 (c = 1.00, $CHCl_3$); The enantiomeric excess was determined by HPLC on Chiralpak AD-H column, hexane: isopropanol = 95:5; flow rate = 1.0 mL/min; UV detection at 210 nm; t_R = 12.1 min (minor), 13.2 min (major). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 - 7.27 (m, 3H), 7.18 - 7.10 (m, 1H), 5.86 (d, *J* = 9.2 Hz, 1H), 2.87 - 2.73 (m, 2H), 2.70 - 2.63 (m, 2H), 2.10 (dd, *J* = 17.8, 10.7 Hz, 1H), 1.72 - 1.68 (m, 3H), 1.03 - 0.87 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.67, 135.67, 135.46, 128.65, 128.20, 126.90, 123.24, 81.18, 36.37, 34.06, 30.22, 27.99, 22.85. ESI-HRMS Calculated for $C_{13}H_{14}O_2^+$ ($[M+H]^+$): 202.0994, found: 202.0995.

4. Gram scale reaction

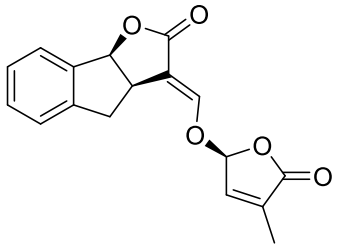


A suspension of **1a** (2.5 g, 13.15 mmol), **cat. C** (0.082 g, 0.1315 mmol), 5:2 HCO_2H/Et_3N (10 mL) were stirred under N_2 at 60 ℃ for until completion according to TLC detection. 10 mL 2N HCl was added to the reaction, the mixture was then extracted with ethyl acetate (3×15 mL) three times, washed with brine, dried over Na_2SO_4 and concentrated. The crude product was purified by silica gel chromatography (hexanes:

ethyl acetate = 1:1)，affording desired product in 85% yield with 98% ee.

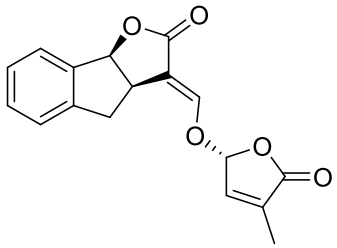
5. Synthesis of (+)-GR24 and (+)-*epi*-GR24[4]

To a solution of **2a** (1.1 g, 6.3 mmol) and methyl formate (0.82 mL, 9.45 mmol) in anhydrous THF (15 mL) was added potassium *tert*-butoxide (0.85 g, 7.56 mmol) in small portions at 0 ℃ under nitrogen. The reaction mixture was stirred at room temperature until completion. THF was removed in vacuo. The resulting solid was resolved in 20 mL anhydrous DMF under N_2. To this solution was added bromobutenolide (1.67 g, 9.45 mmol) and the reaction mixture was stirred overnight. The reaction was quenched with saturated aqueous ammonium chloride (20 mL). The reaction mixture was diluted with ethyl acetate (50 mL) and washed with water (3×30 mL). The organic extract was then washed with brine, dried over Na_2SO_4, and the solvent removed under vacuum. The residue was purified by column chromatography，affording pure (+)-GR24 and (+)-*epi*-GR24.



(+)-epi-GR24

White Solid, 0.65 g, 35% yield, $[\alpha]_D^{20}$ = +292 (c = 0.50, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.50-7.49 (m, 1H), 7.35-7.23 (m, 1H), 6.99 (s, 1H), 6.21 (s, 1H), 5.96 (d, *J* = 8.0 Hz, 1H), 3.97-3.92 (m, 1H), 3.42 (dd, *J* = 16.9, 9.3 Hz, 1H), 3.10 (dd, *J* = 16.9, 3.1 Hz, 1H), 2.03 (t, *J* = 1.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.42, 170.40, 151.29, 142.69, 141.25, 138.76, 135.76, 130.05, 127.45, 126.34, 125.28, 113.26, 100.79, 86.00, 38.79, 37.42, 29.69, 10.74.



(+)-GR24

White solid, 0.65 g, 35% yield, $[\alpha]_D^{20}$ = +451 (c = 0.50, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.51-7.49 (m, 2H), 7.35 (m, 3H), 7.00 (s, 1H), 6.21 (s, 1H), 5.95 (d, *J* = 7.9 Hz, 1H), 3.97-3.92 (m, 1H), 3.44 (dd, *J* = 16.9, 9.4 Hz, 1H), 3.11 (dd, *J* = 16.9, 3.2 Hz, 1H), 2.03 (t, *J* = 1.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.44, 170.37, 151.27, 142.66, 141.16, 138.82, 135.83, 130.04, 127.49, 126.42, 125.18, 113.14, 100.71, 85.99, 38.85, 37.31, 10.73.

6. Reference:

(1) R. Z. Guo *et al*. *Bioorg. Org. Lett*., 2016, **18**, 504–507;

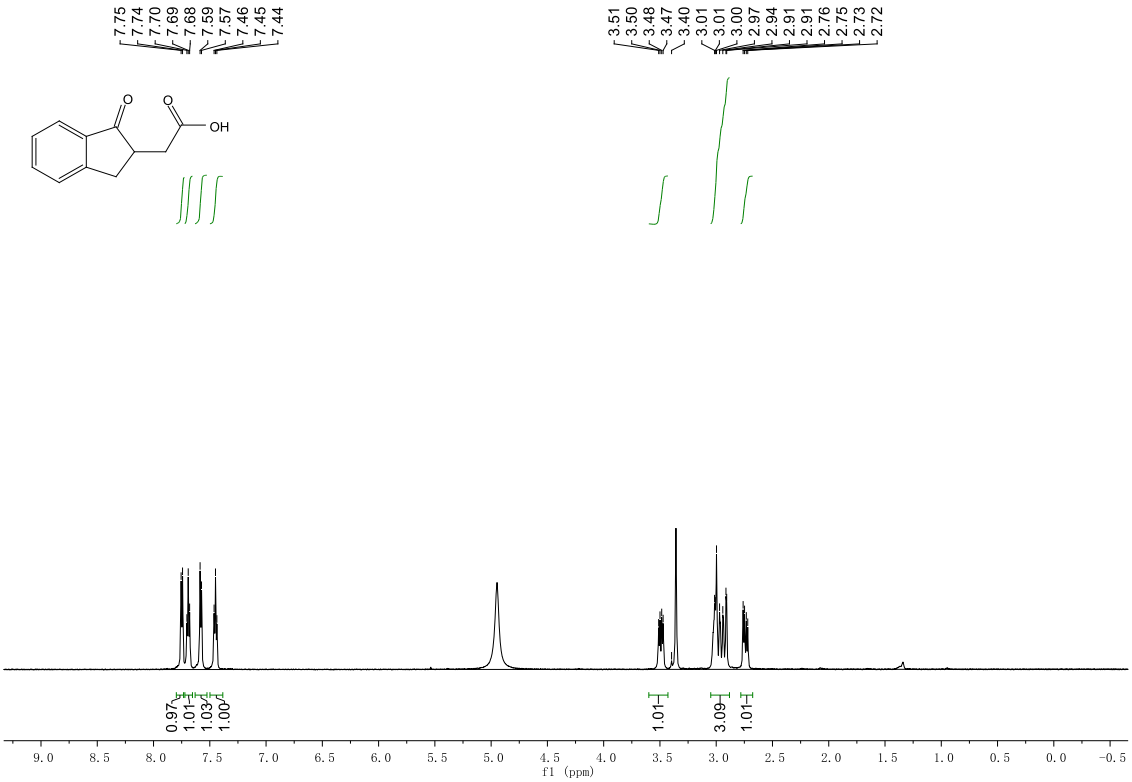
(2) F. Meneghetti *et al*. *Eur. J. Org. Chem*. 2015, 4907–4912;

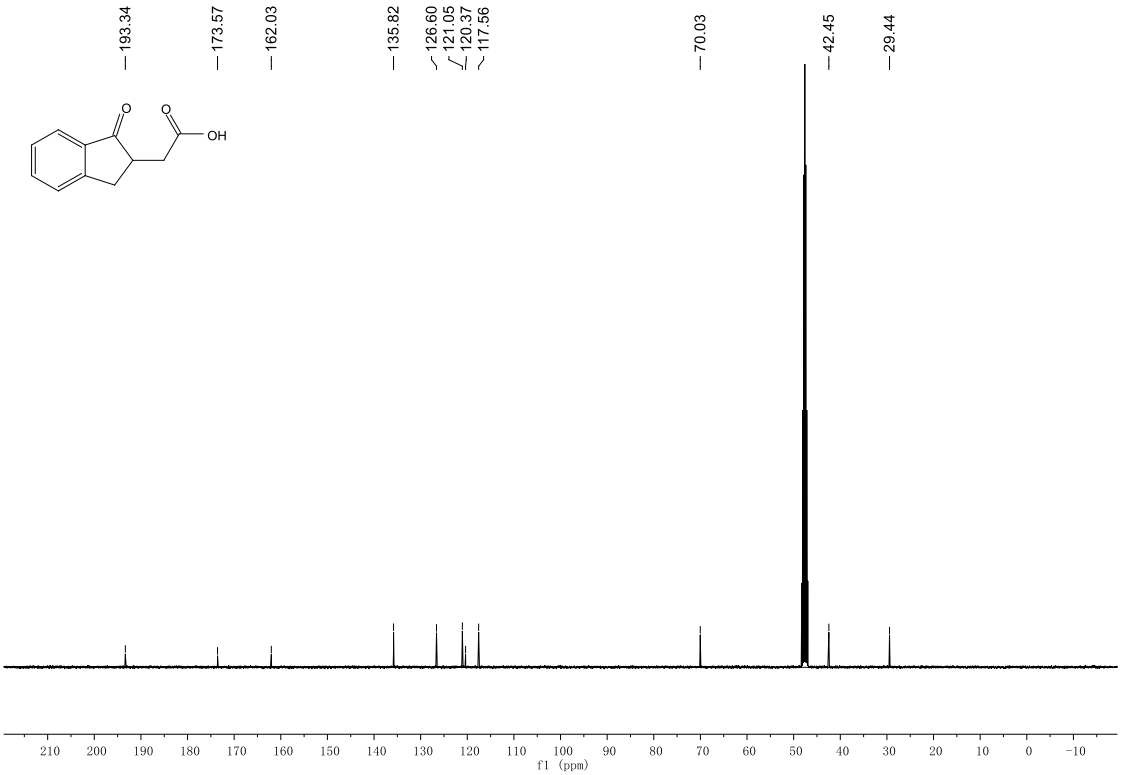
(3) L. J. Bromhead *et al., Aust. J. Chem.* 2015, **68**, 1221–1227;

(4) H. Malik *et al*. *Tetrahedron*, 2010, **66**, 7198-7203.

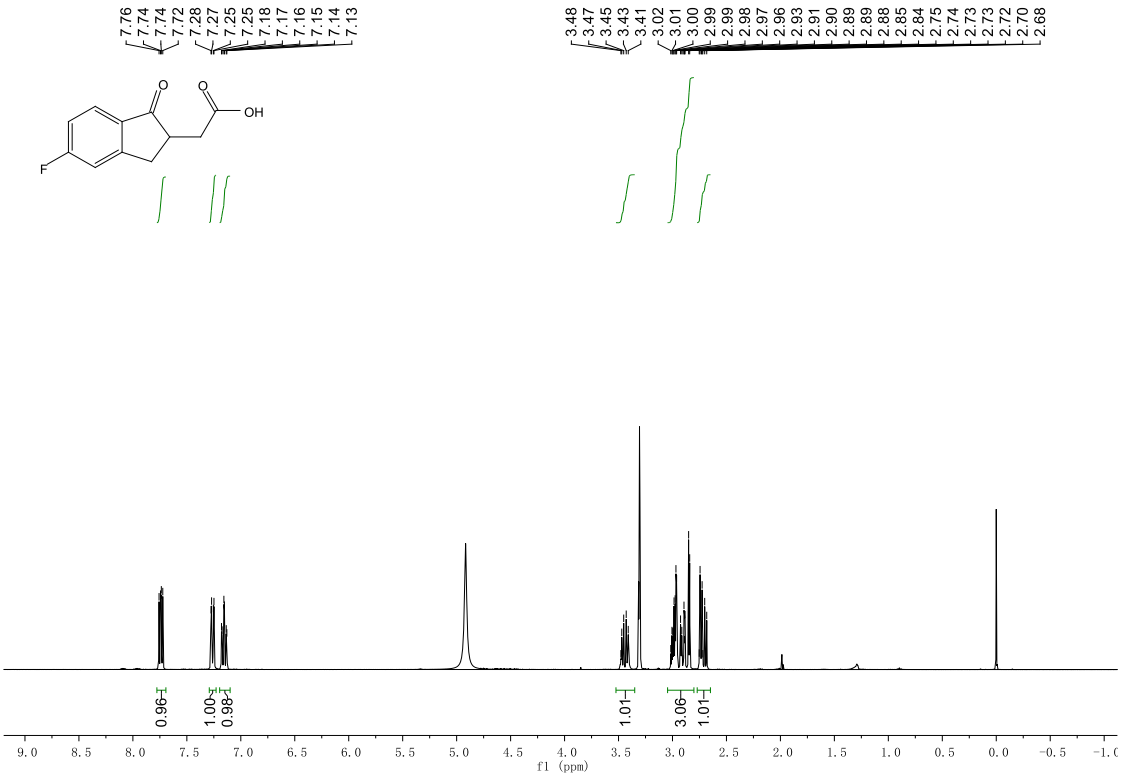
7. NMR spectra

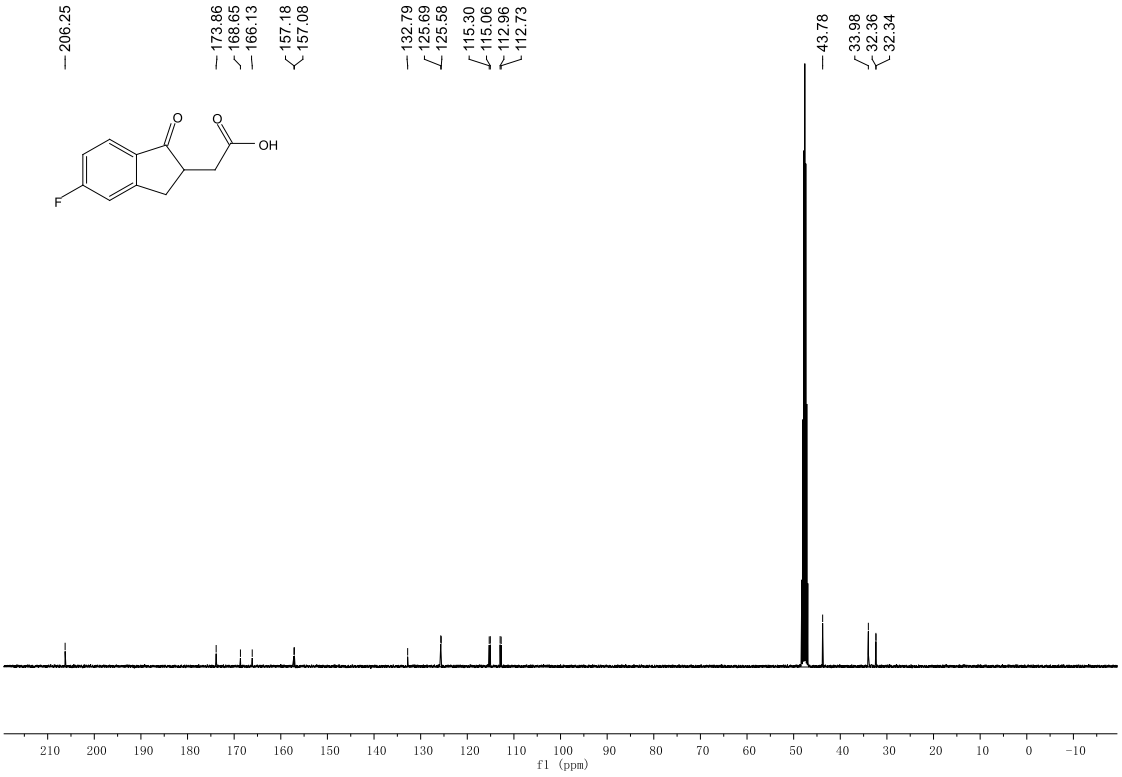
NMR spectra of **1a**



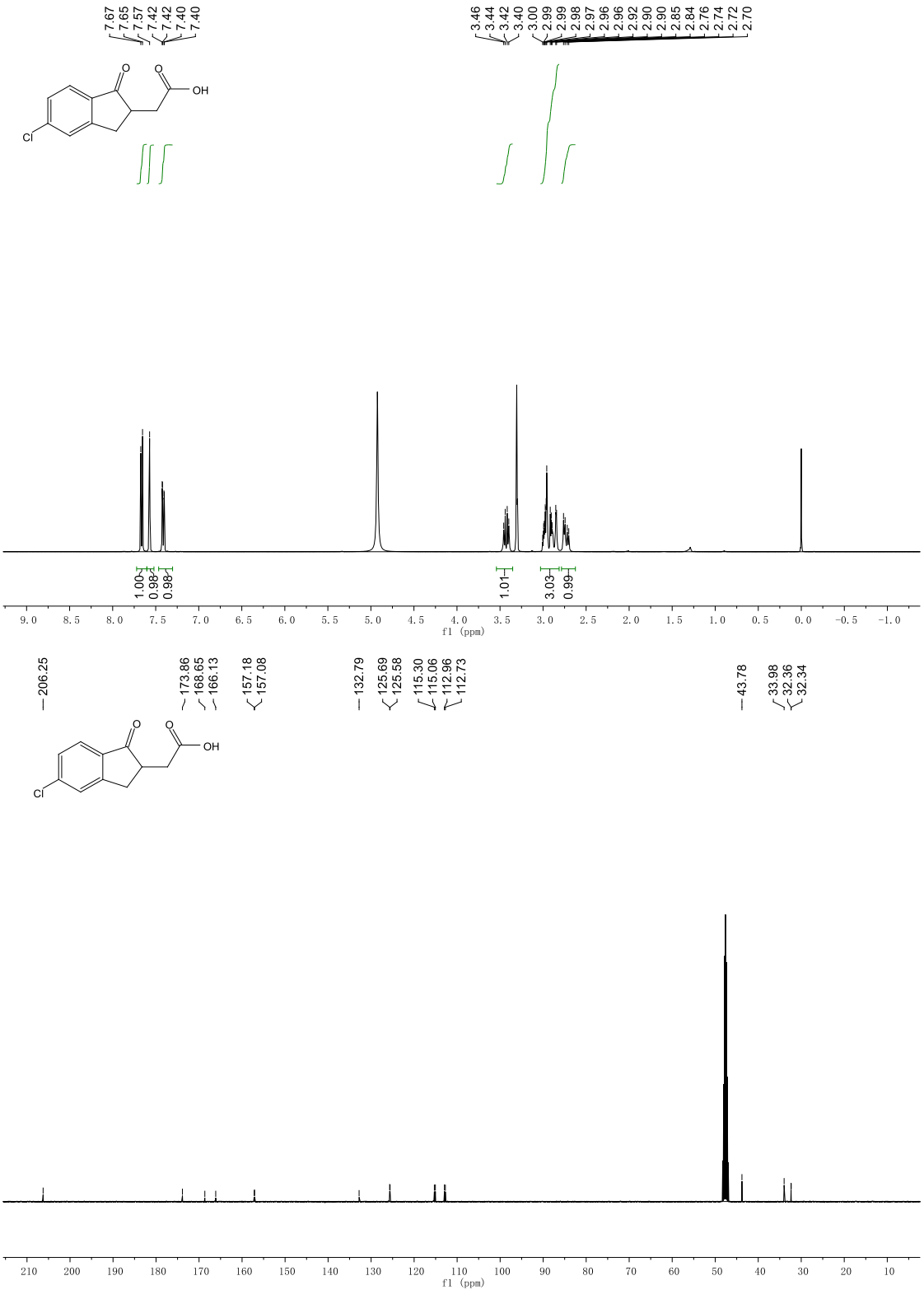


NMR spectra of **1b**

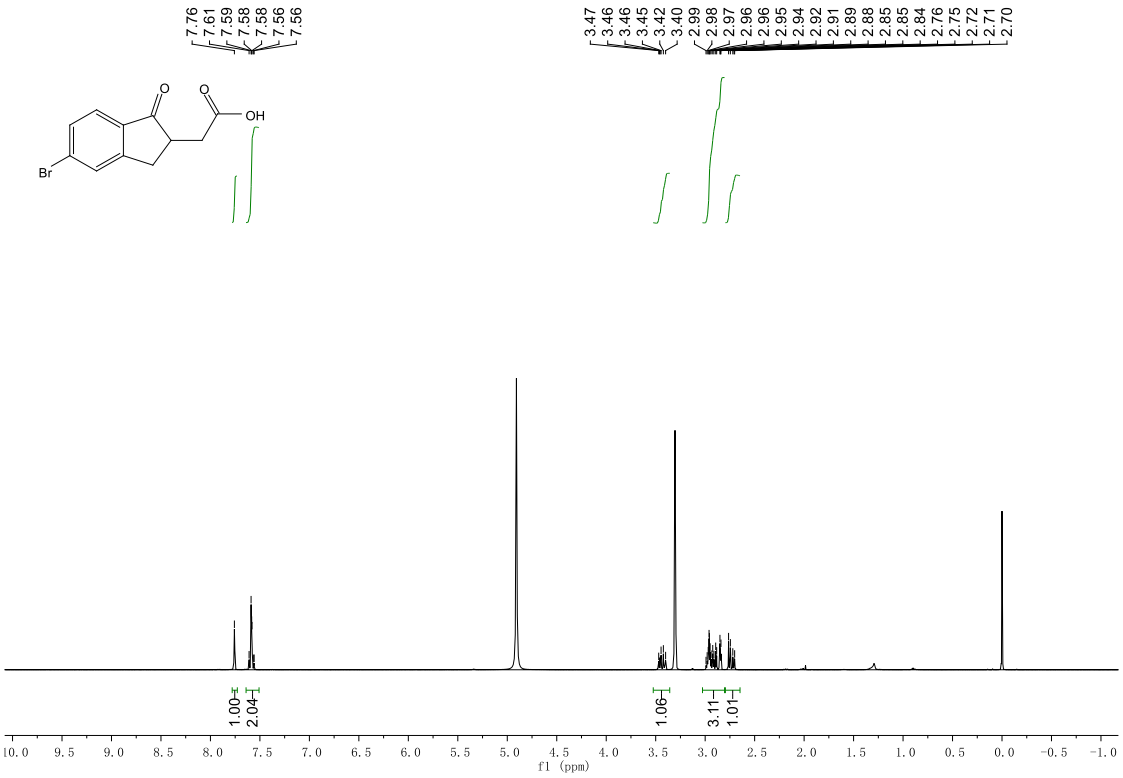


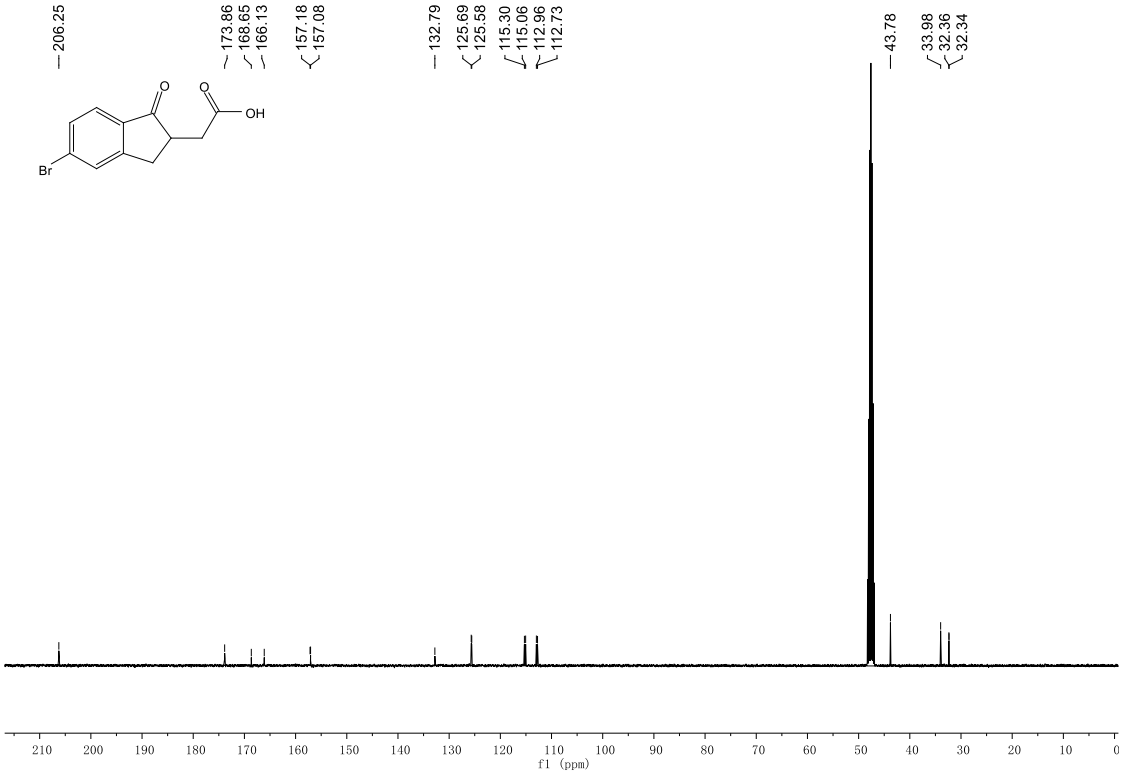


NMR spectra of **1c**

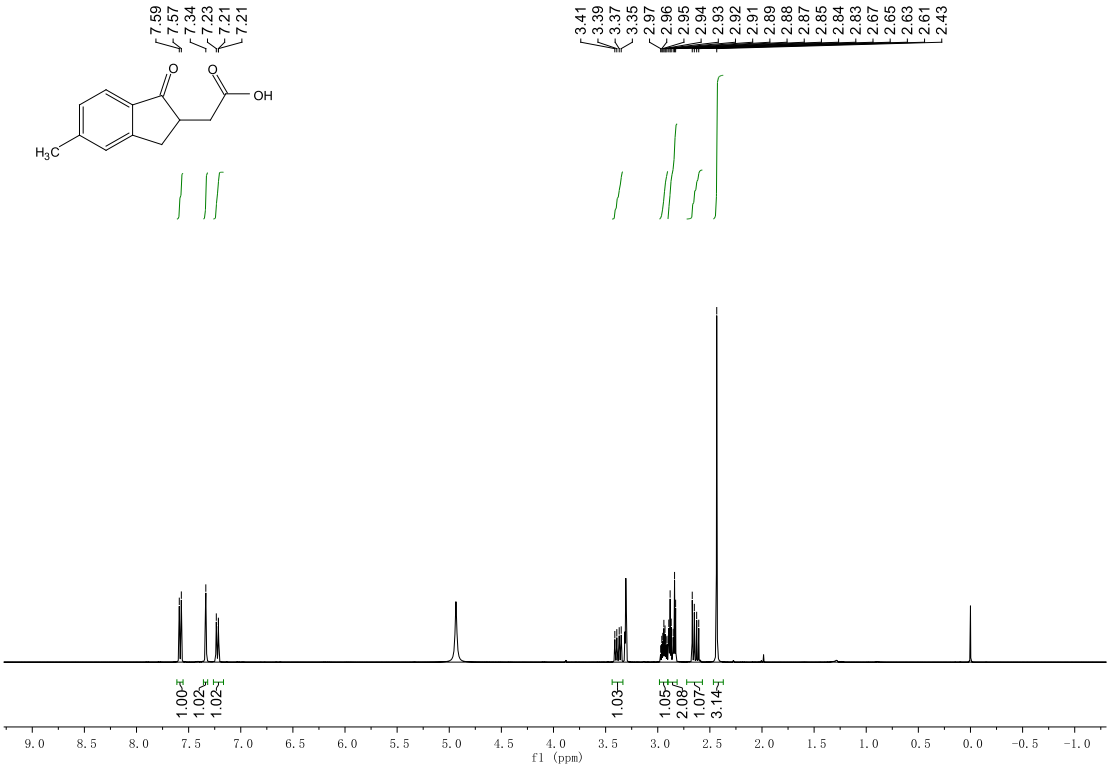


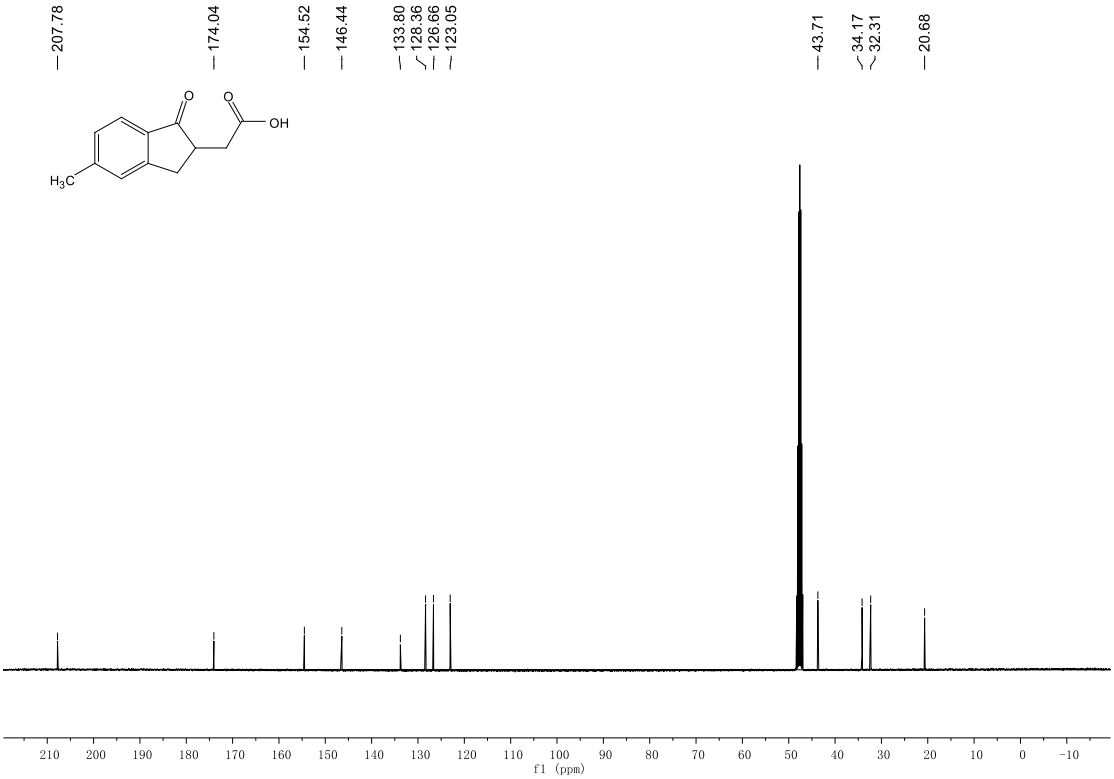
NMR spectra of **1d**



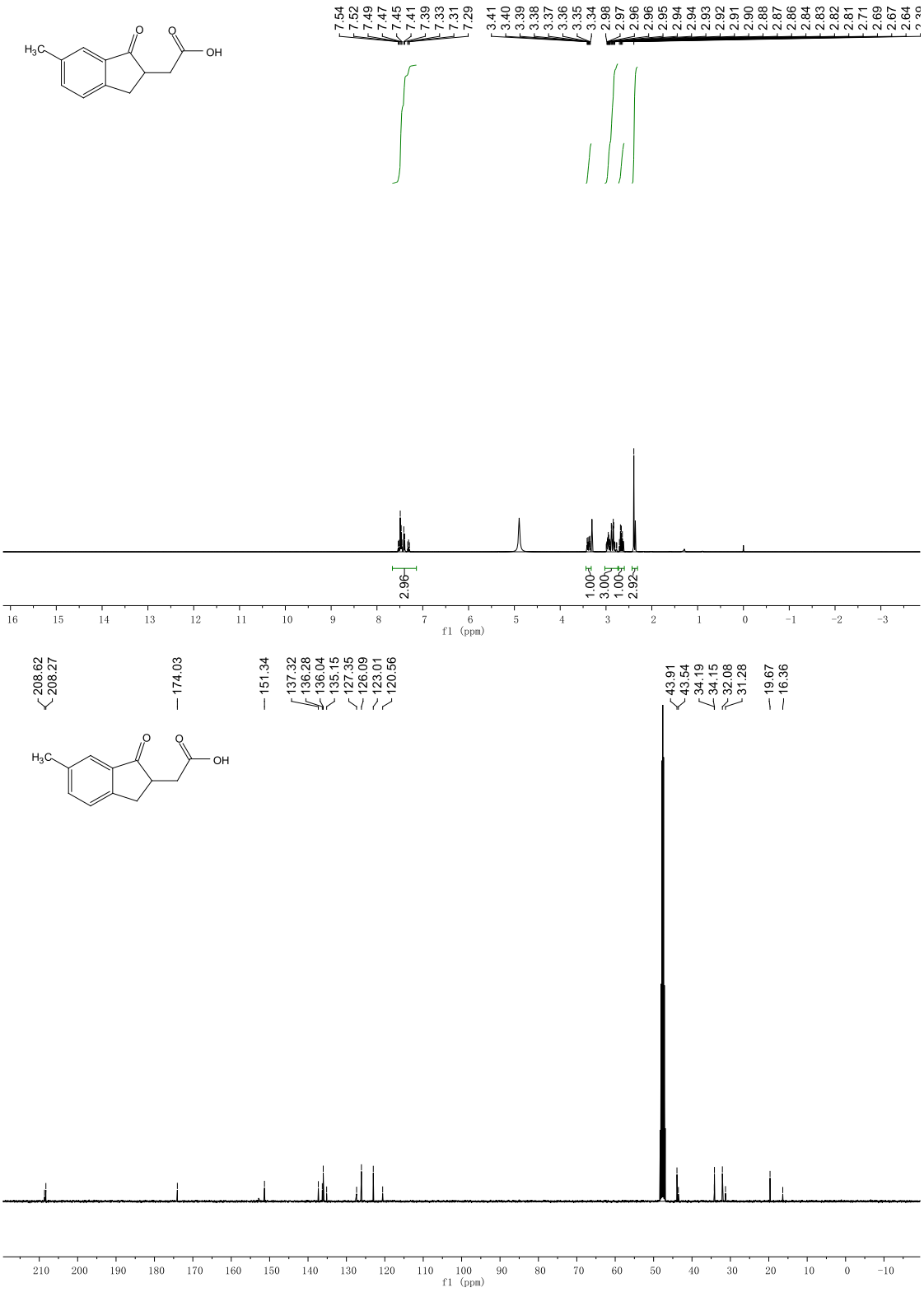


NMR spectra of **1e**

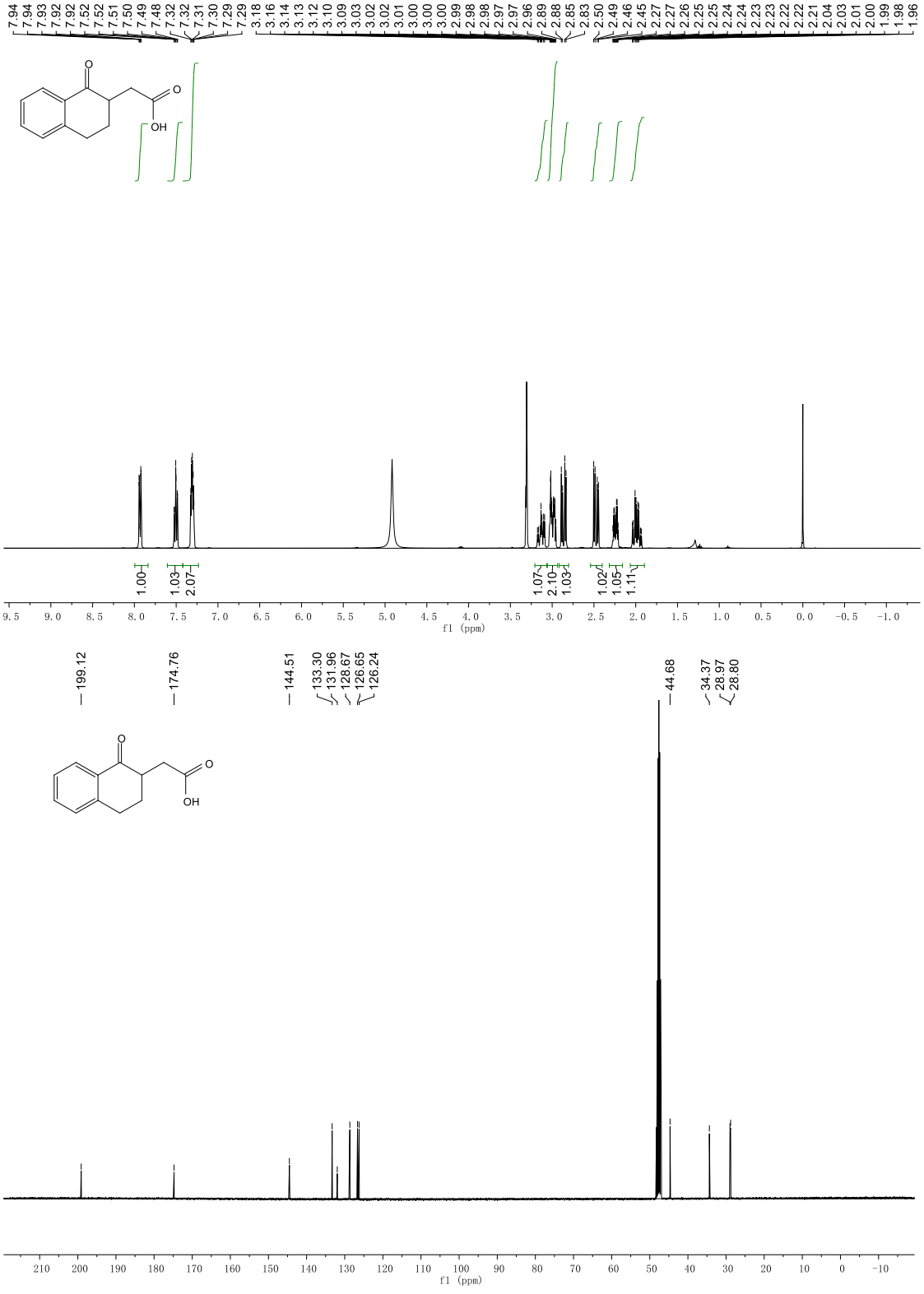




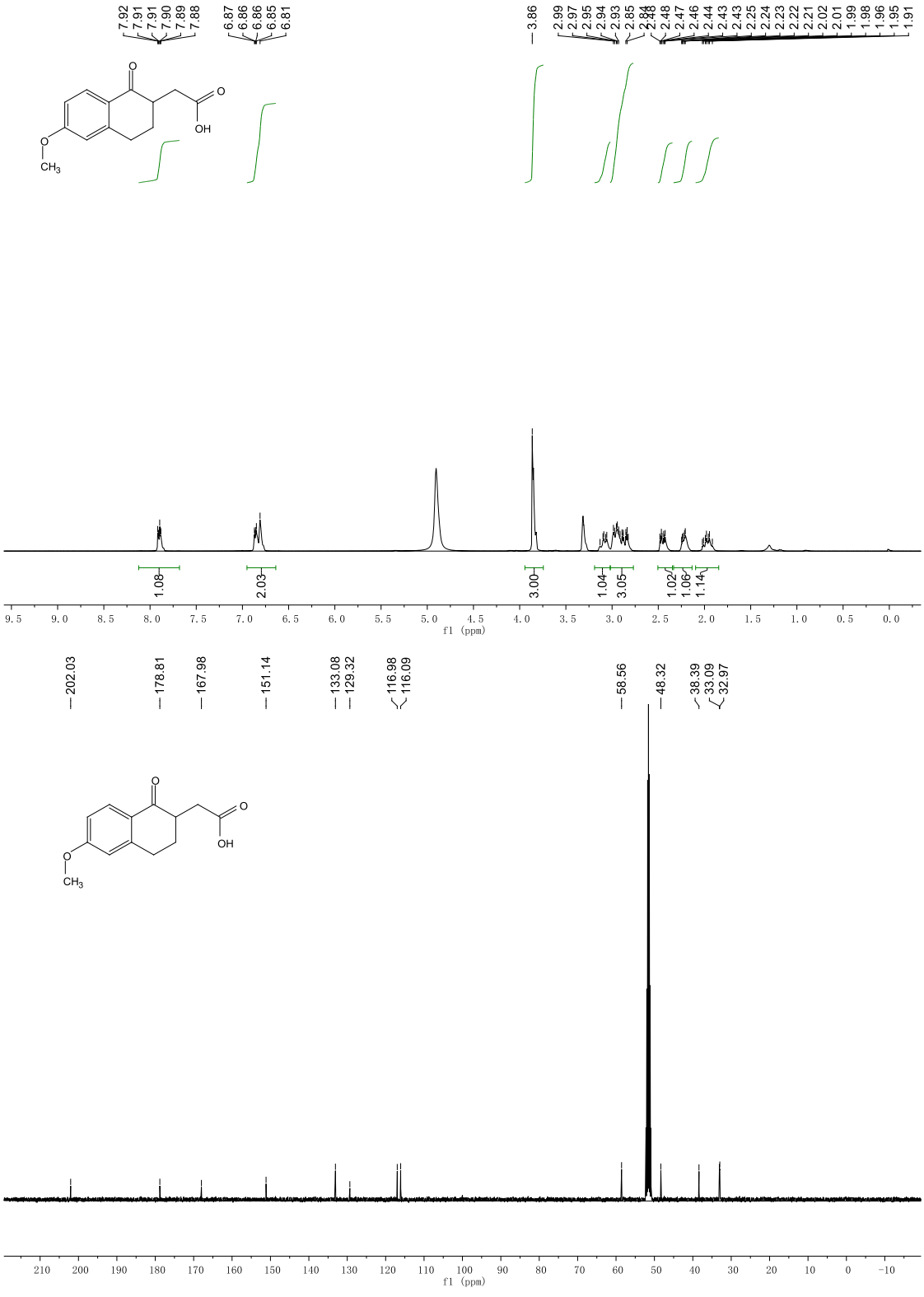
NMR spectra of **1f**



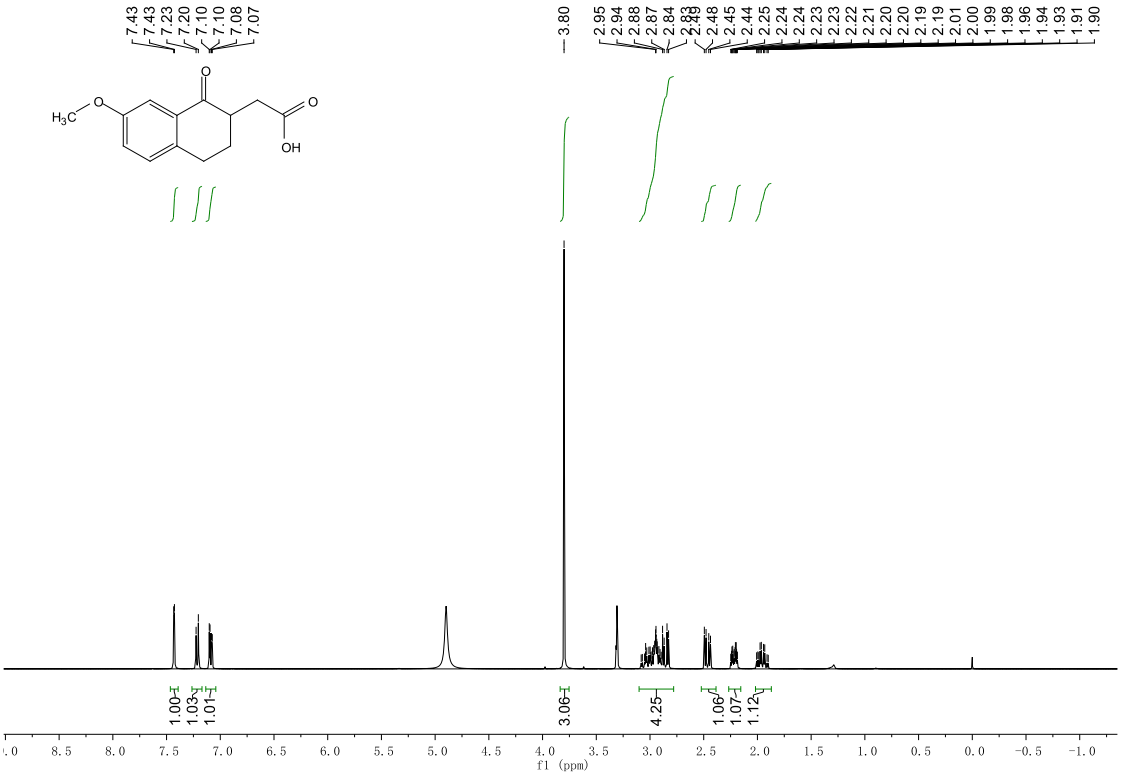
NMR spectra of **1g**

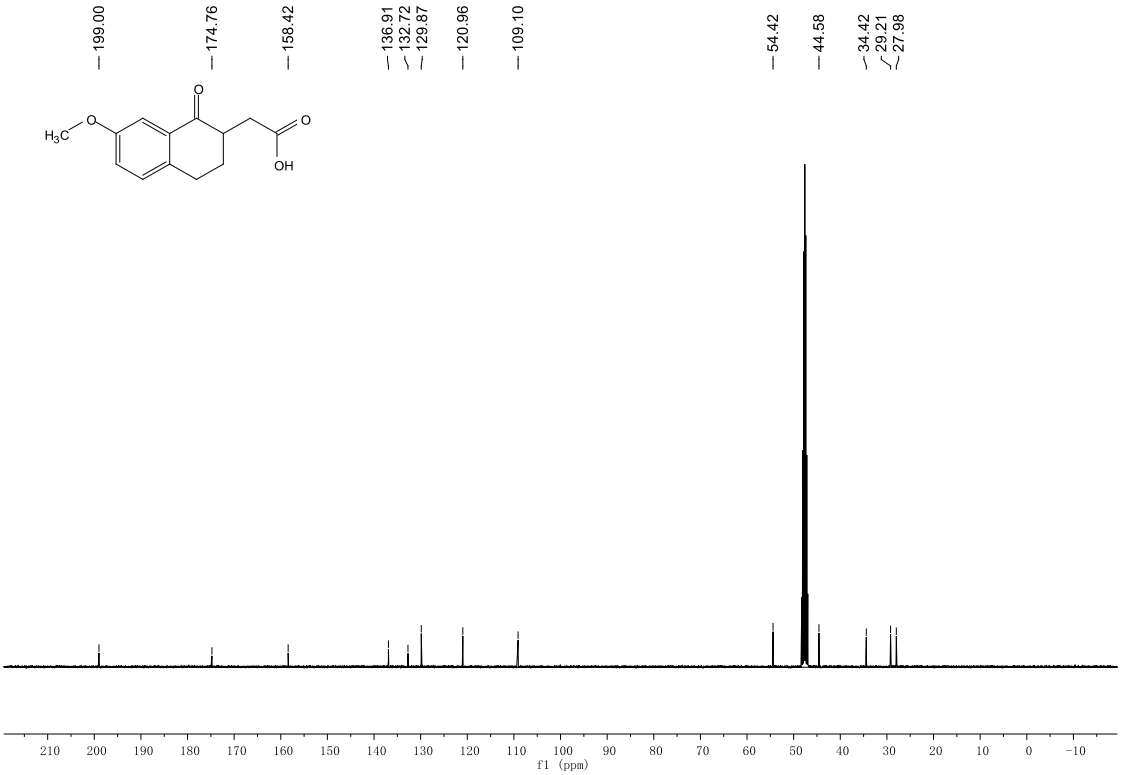


NMR spectra of **1h**

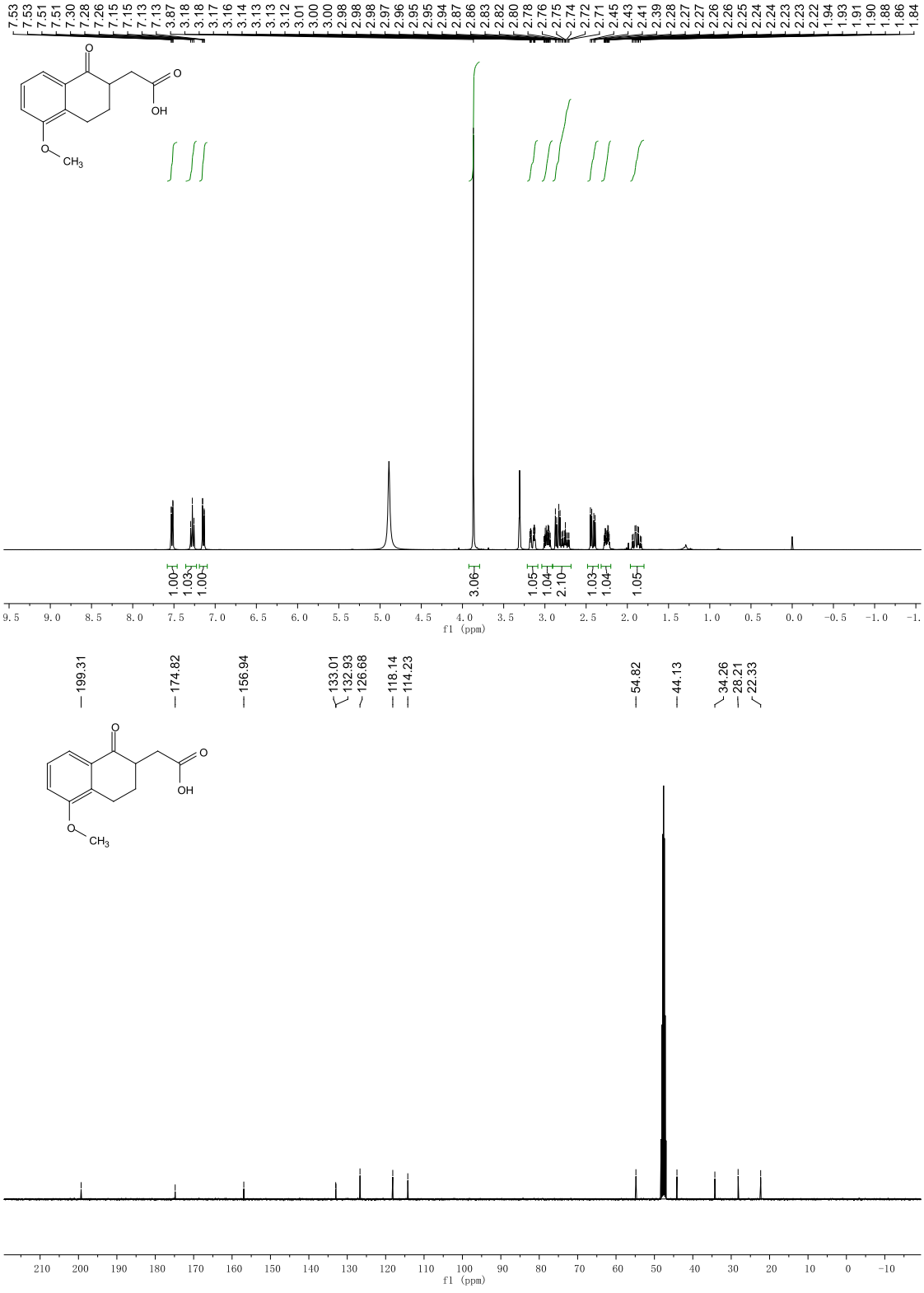


NMR spectra of **1i**

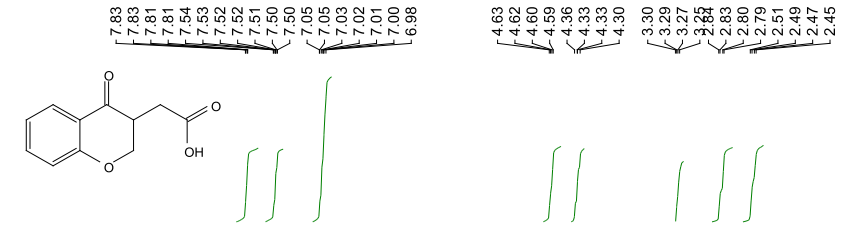


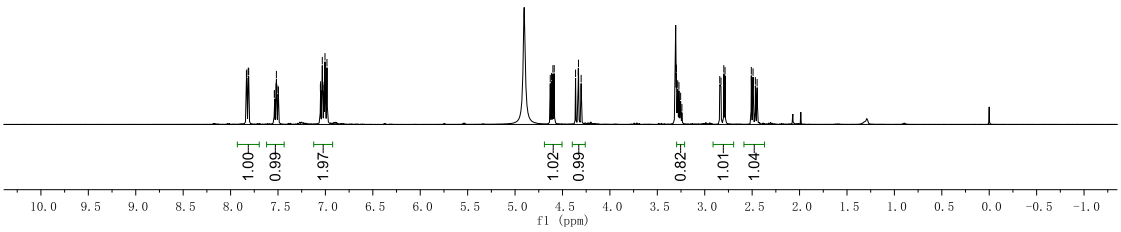


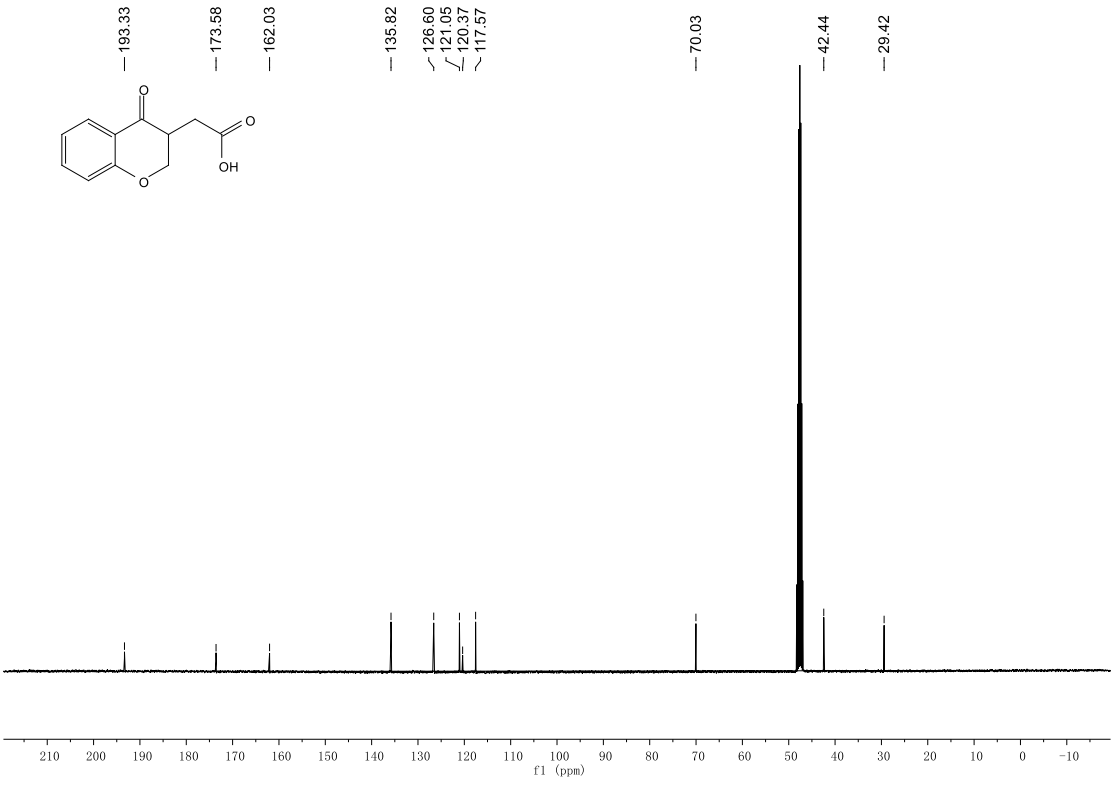
NMR spectra of **1j**



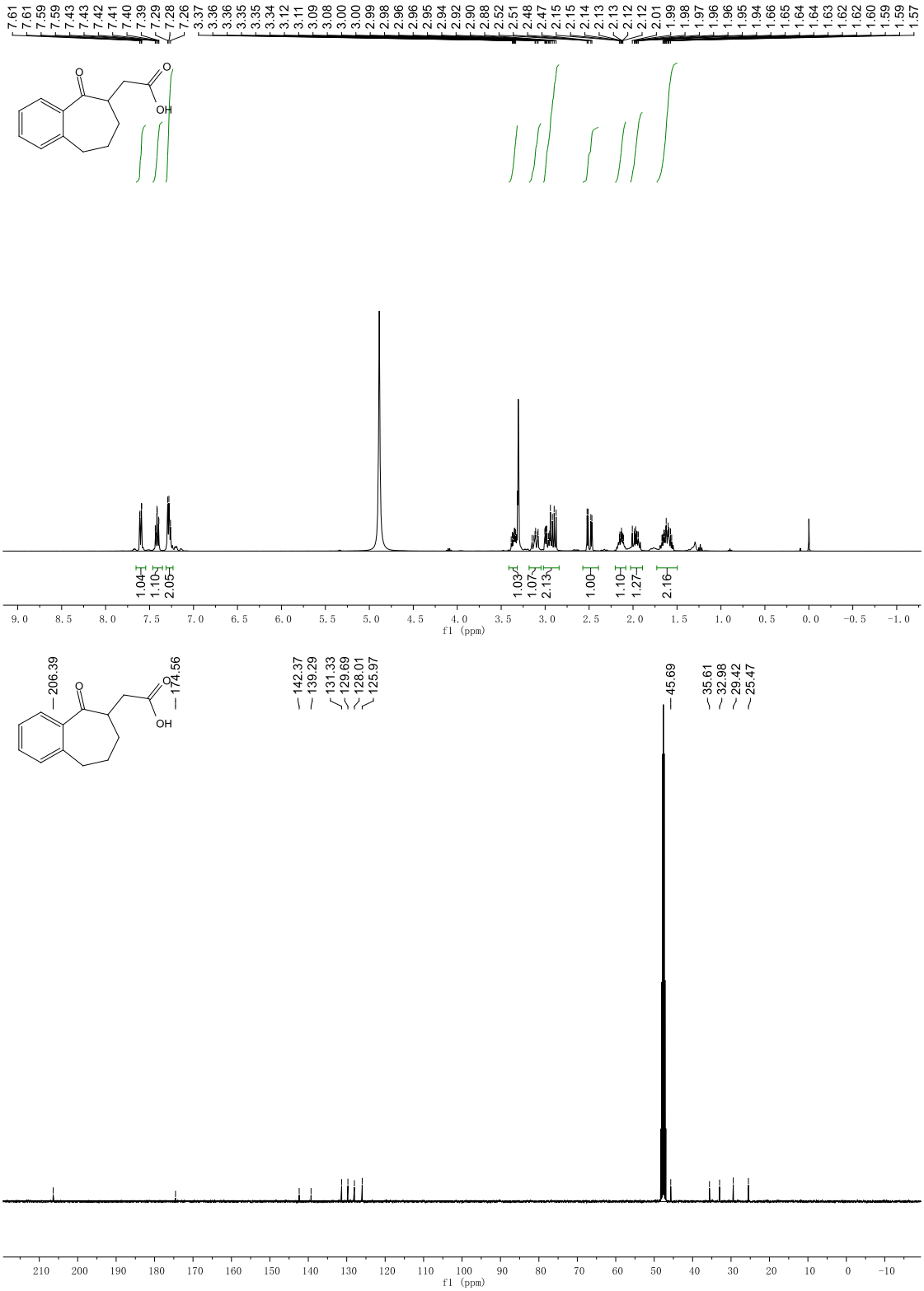
NMR spectra of **1k**



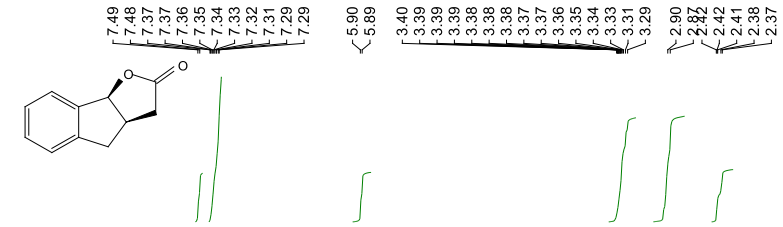


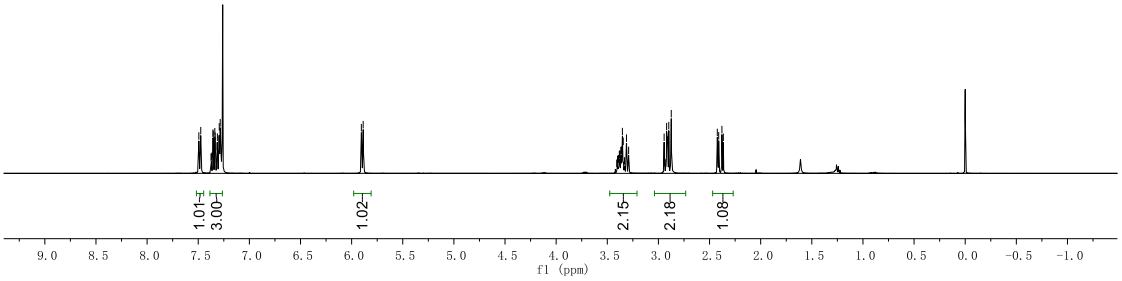


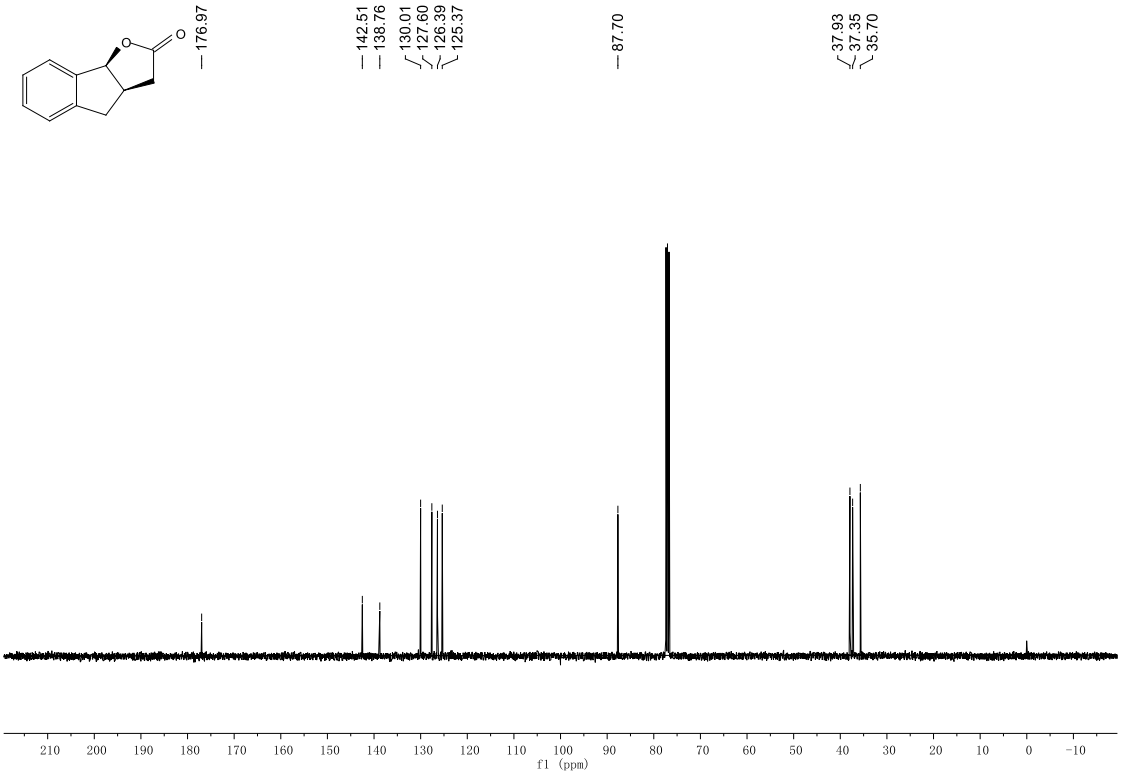
NMR spectra of **1l**



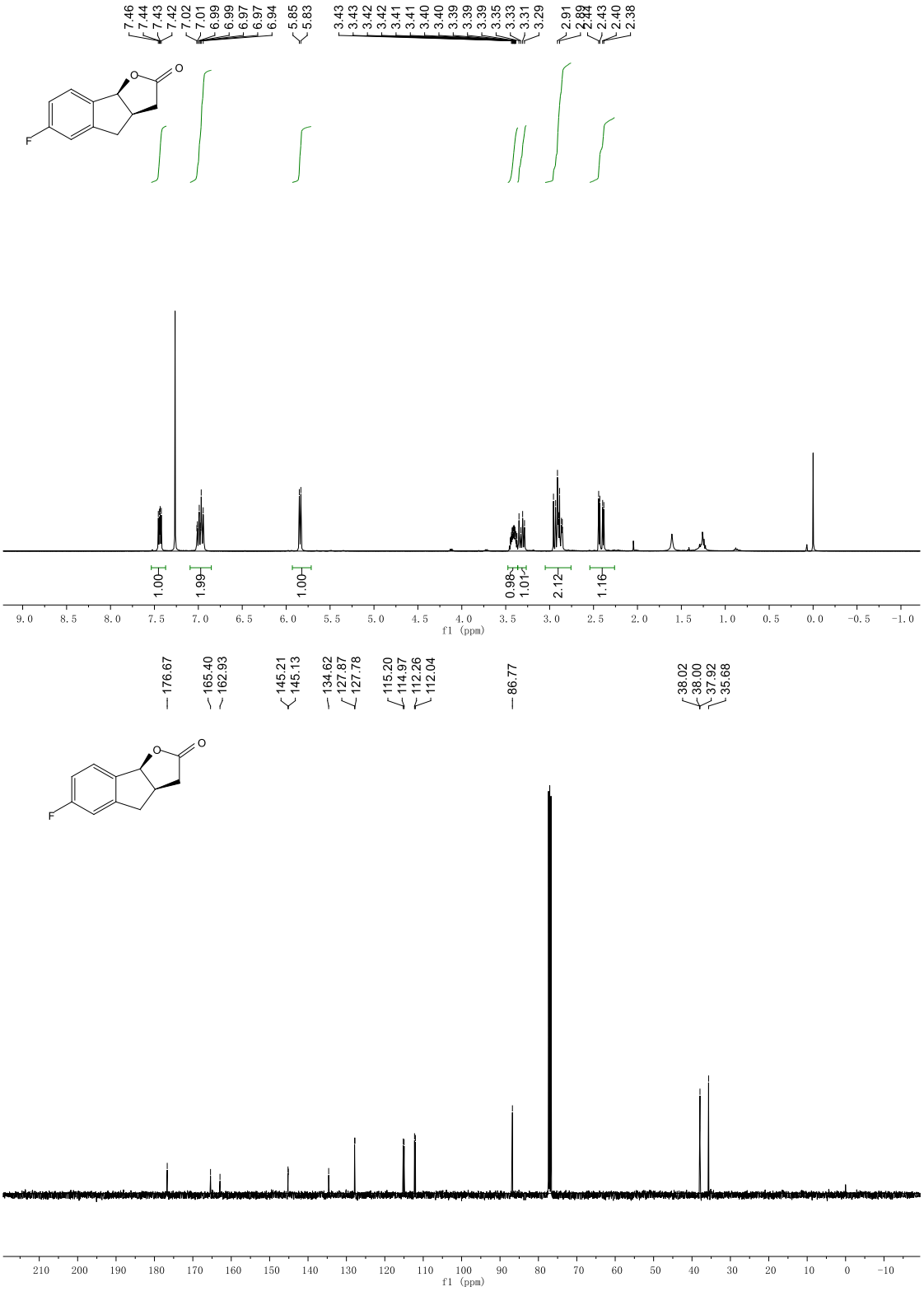
NMR spectra of **2a**



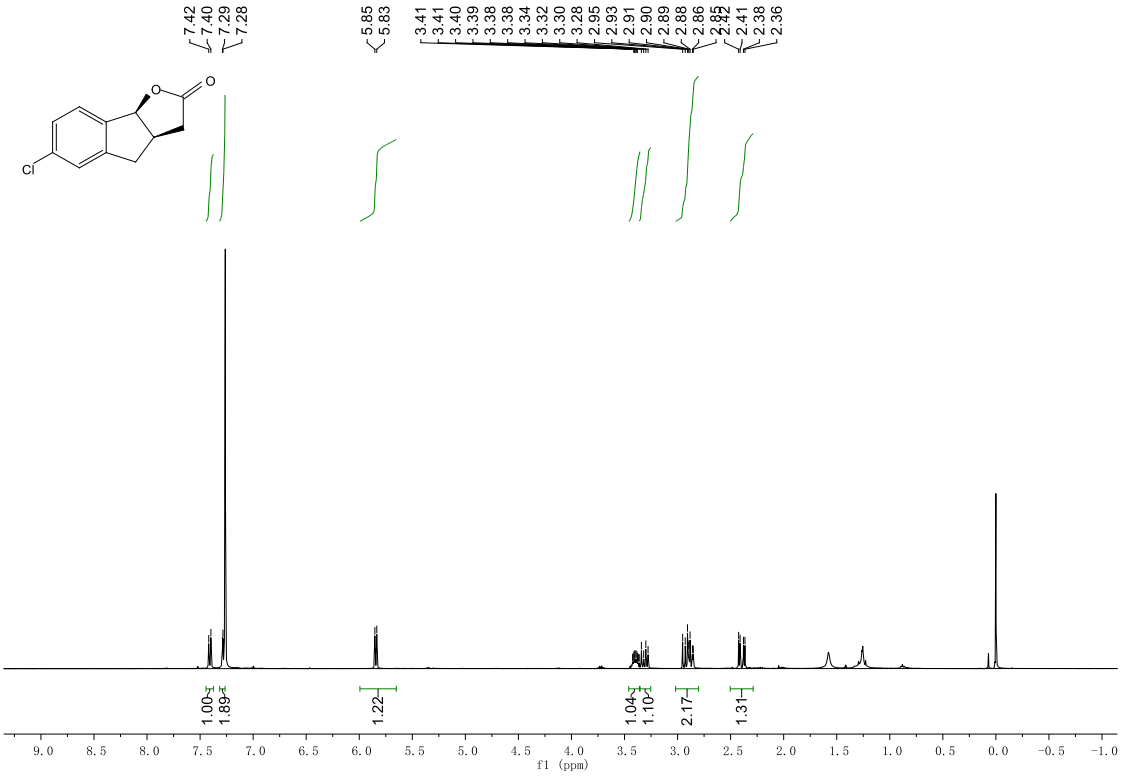


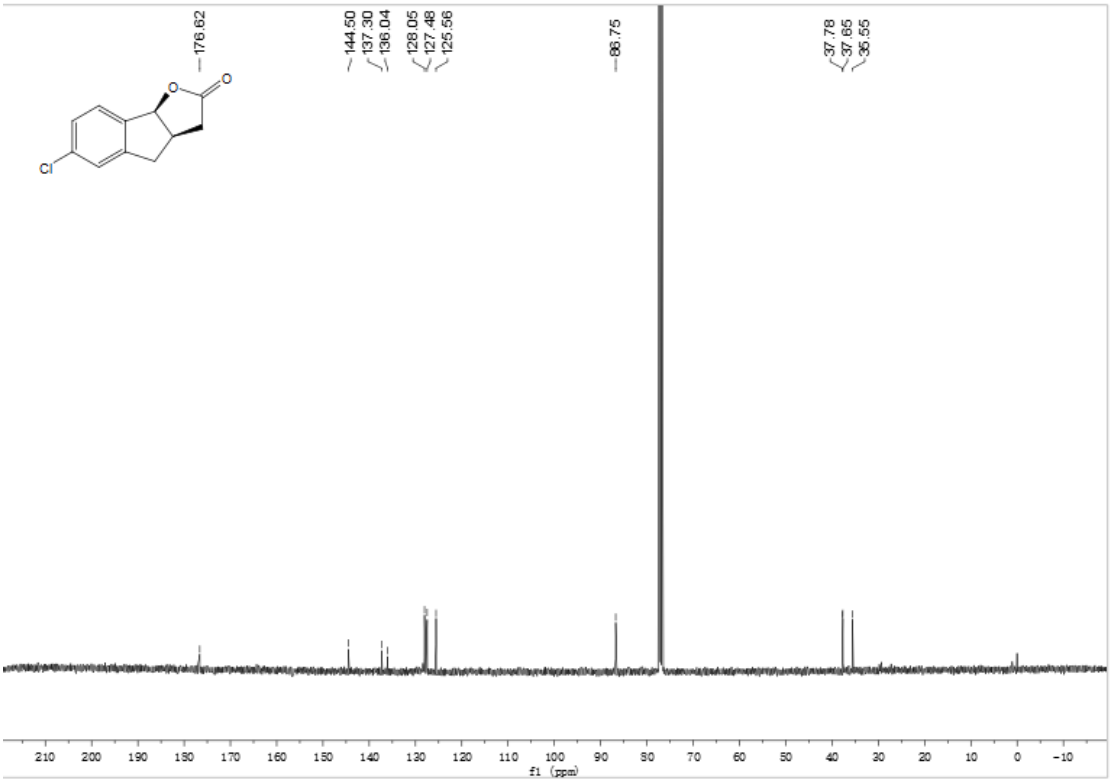


NMR spectra of **2b**

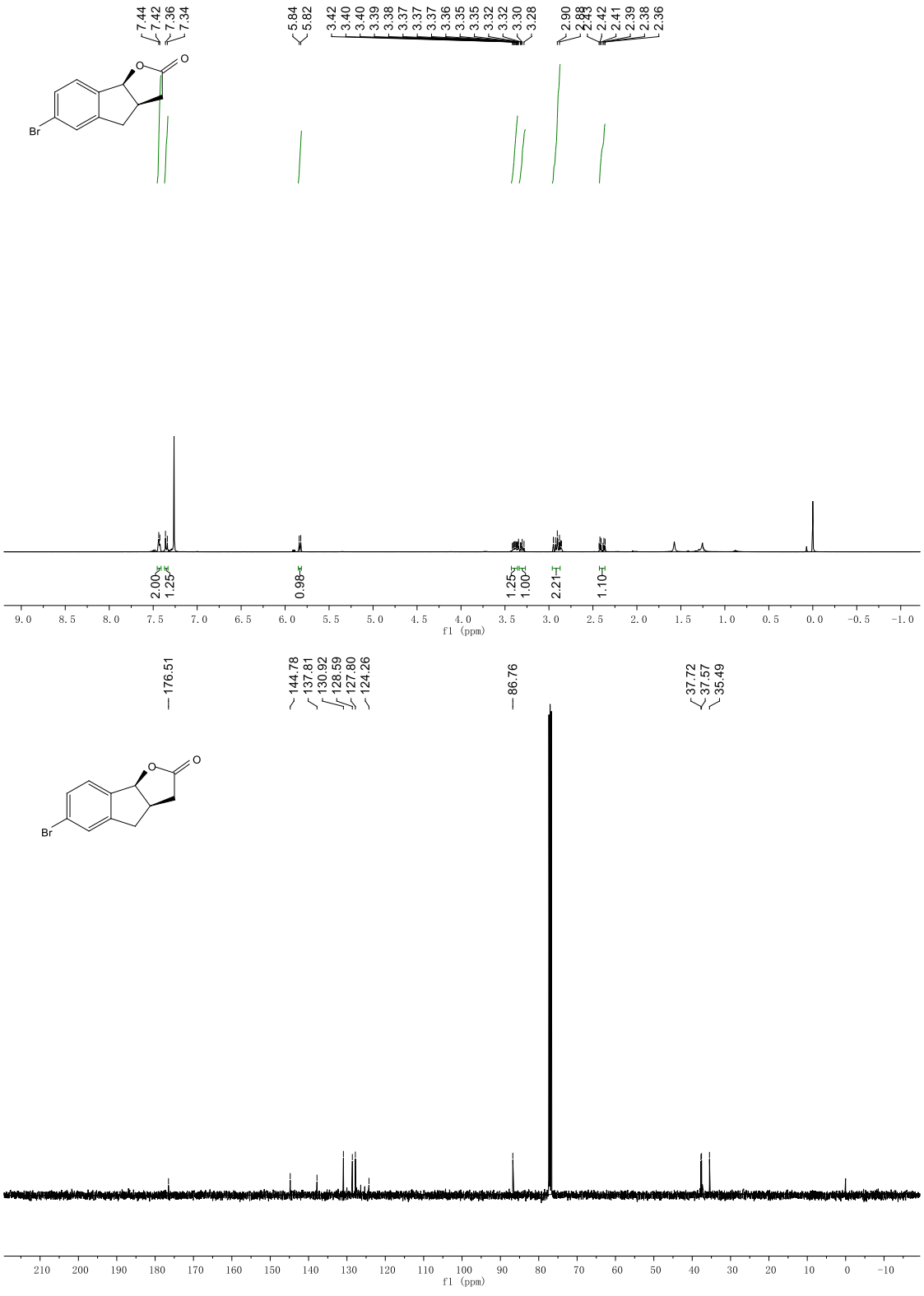


NMR spectra of **2c**

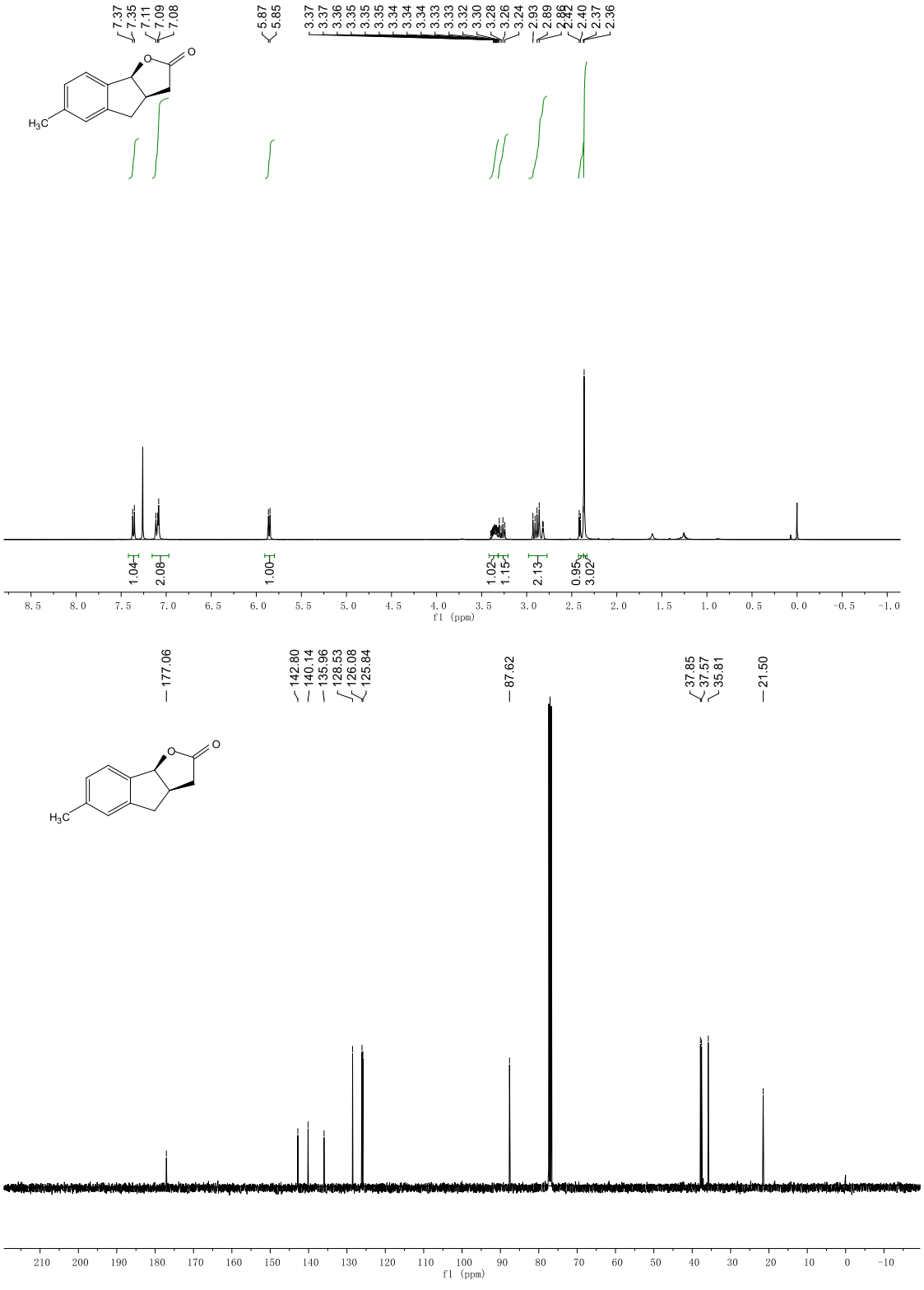




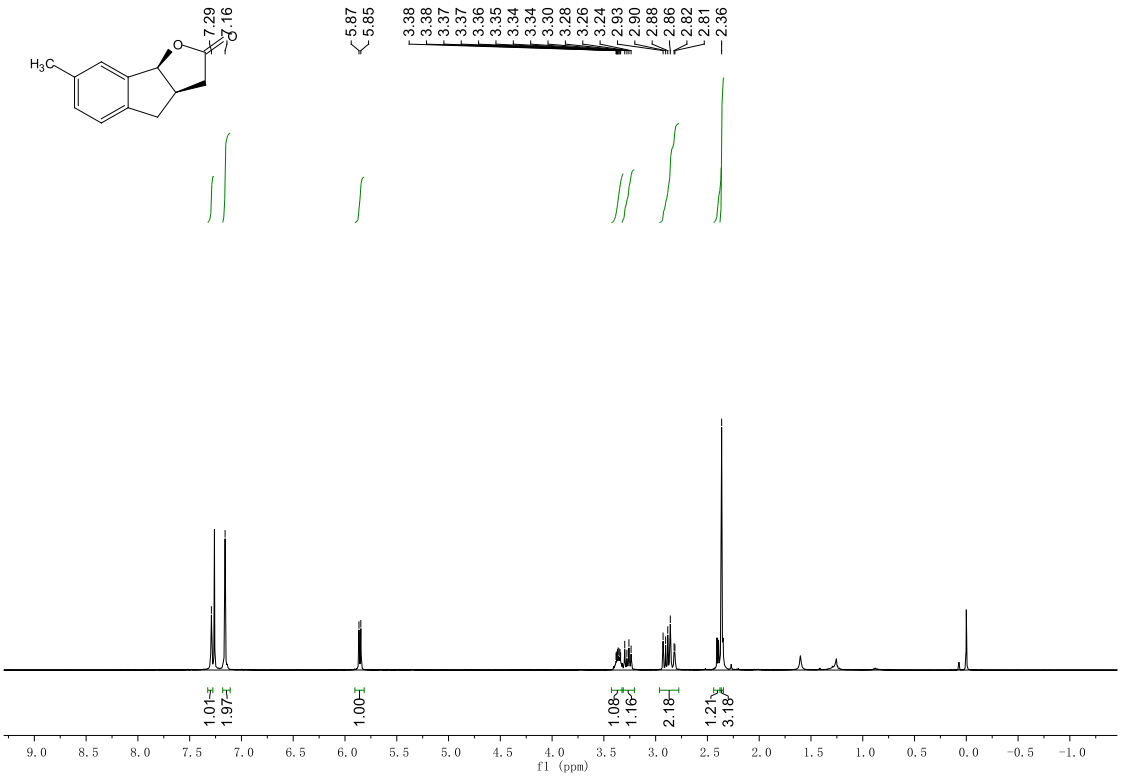
NMR spectra of **2d**

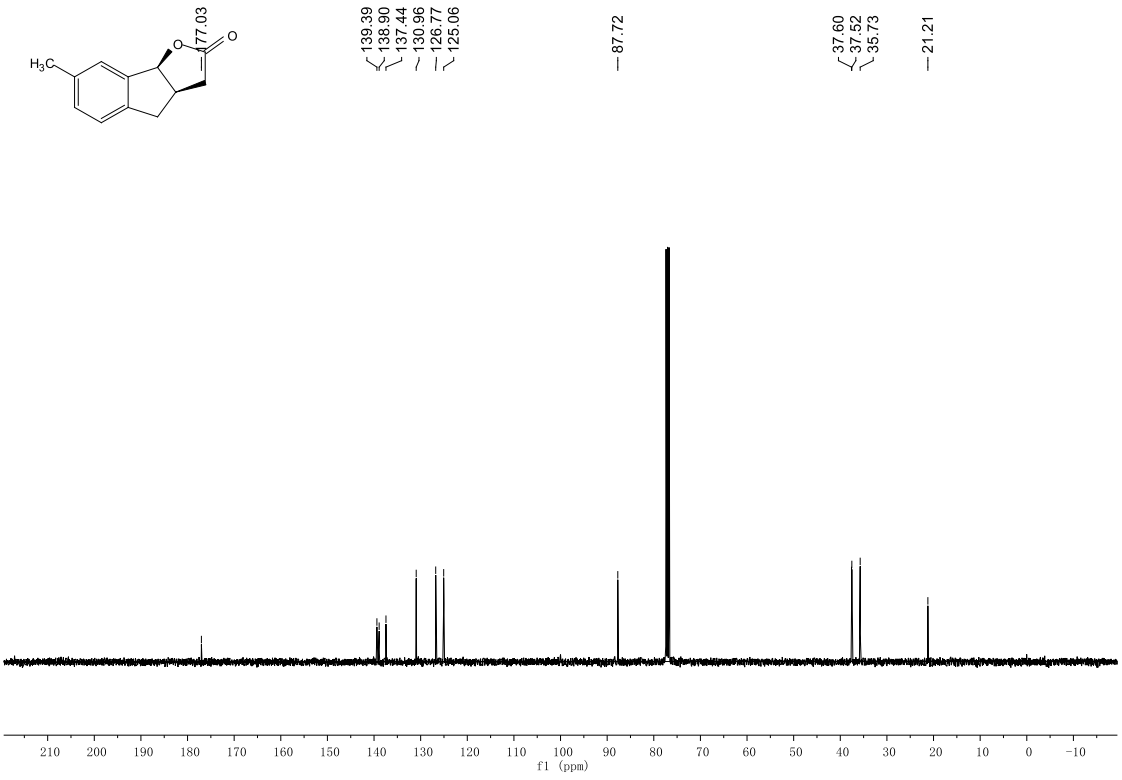


NMR spectra of **2e**

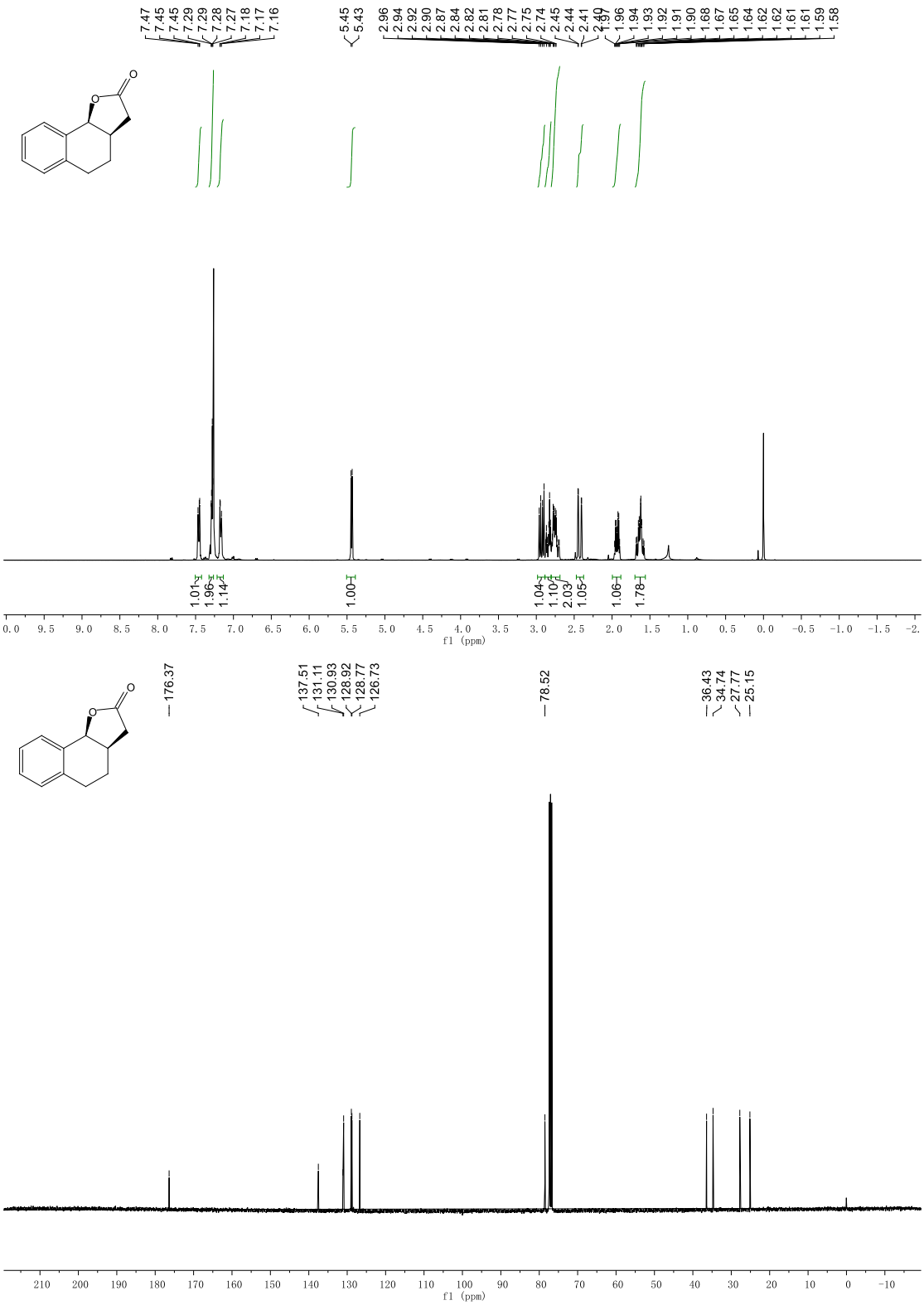


NMR spectra of **2f**

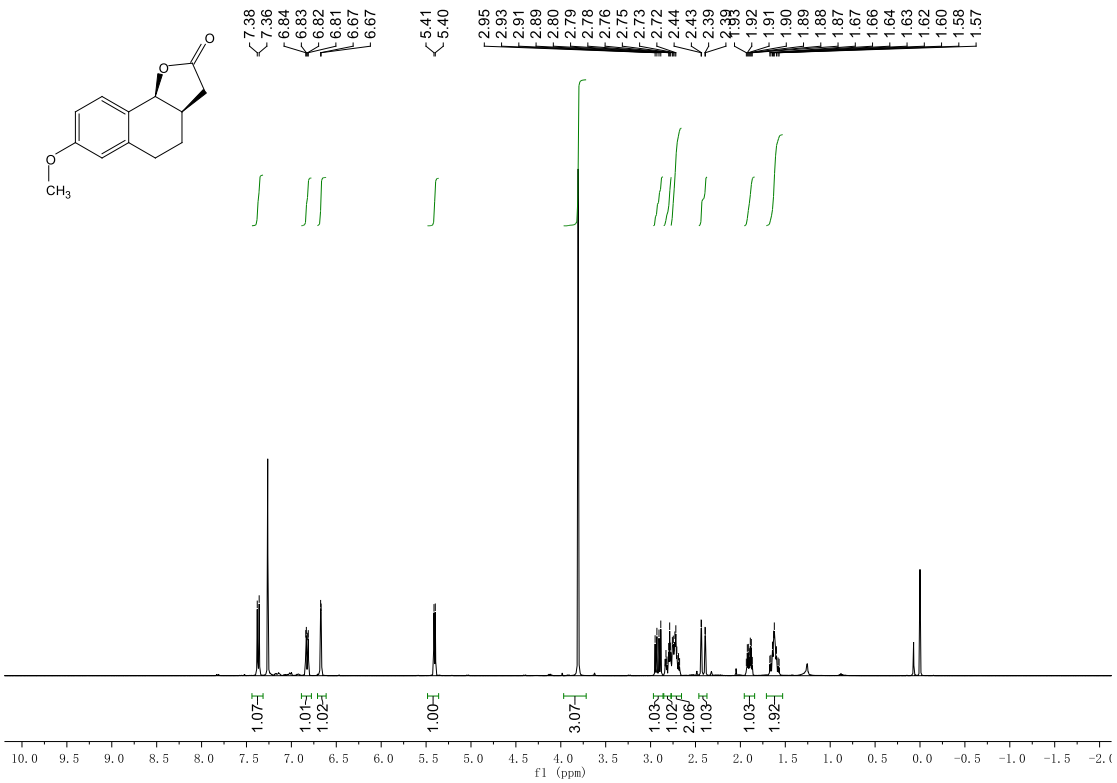


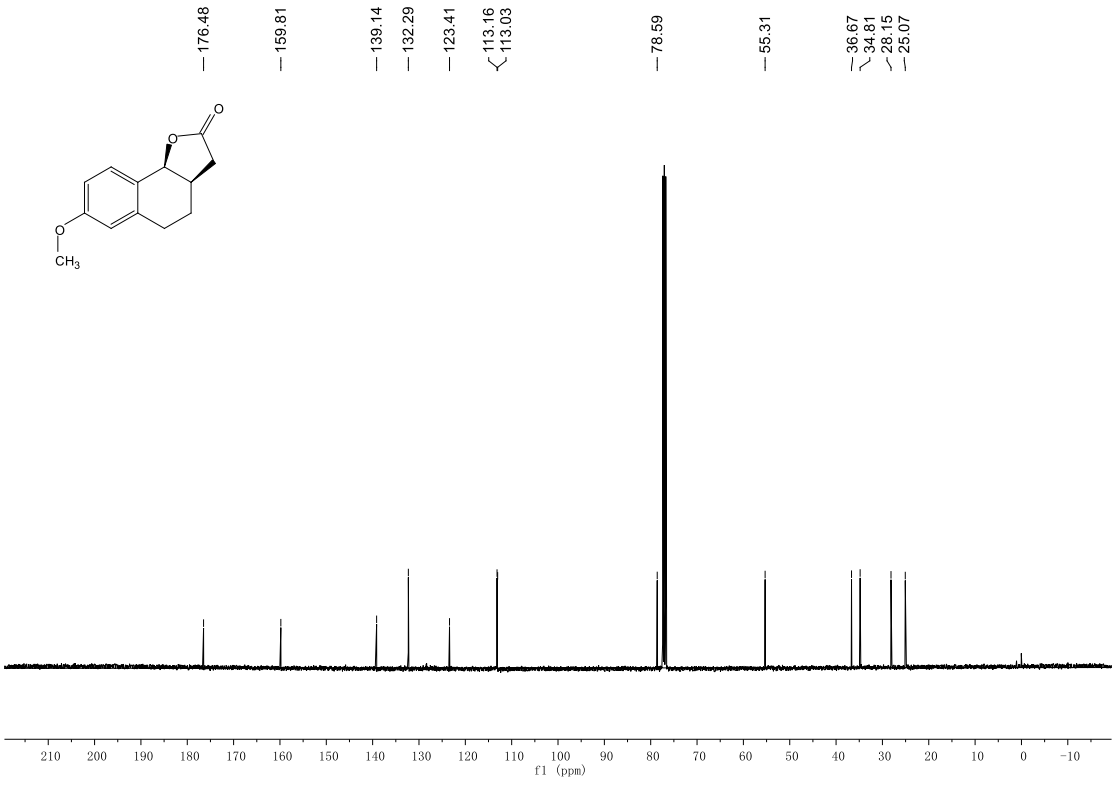


NMR spectra of **2g**

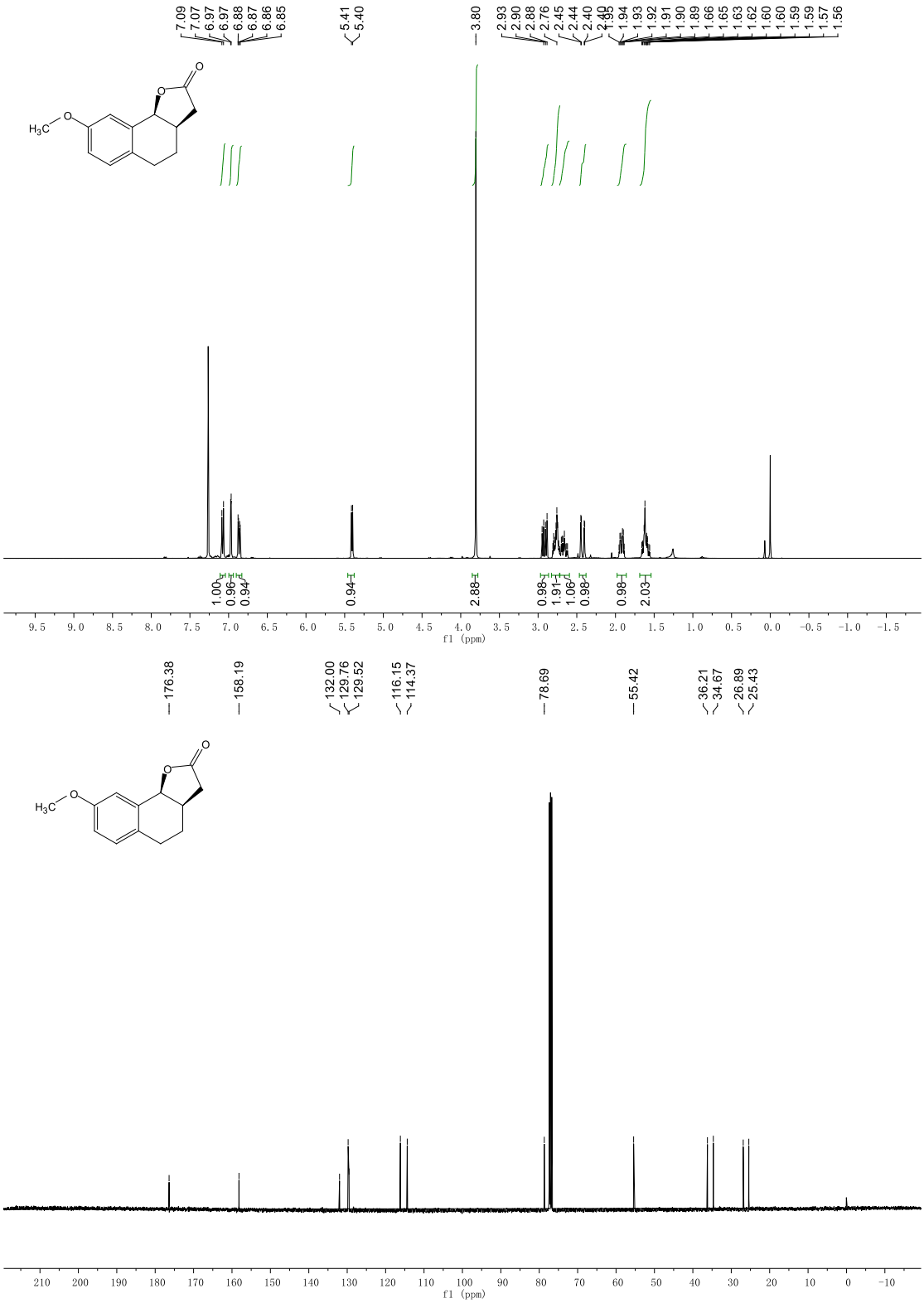


NMR spectra of **2h**

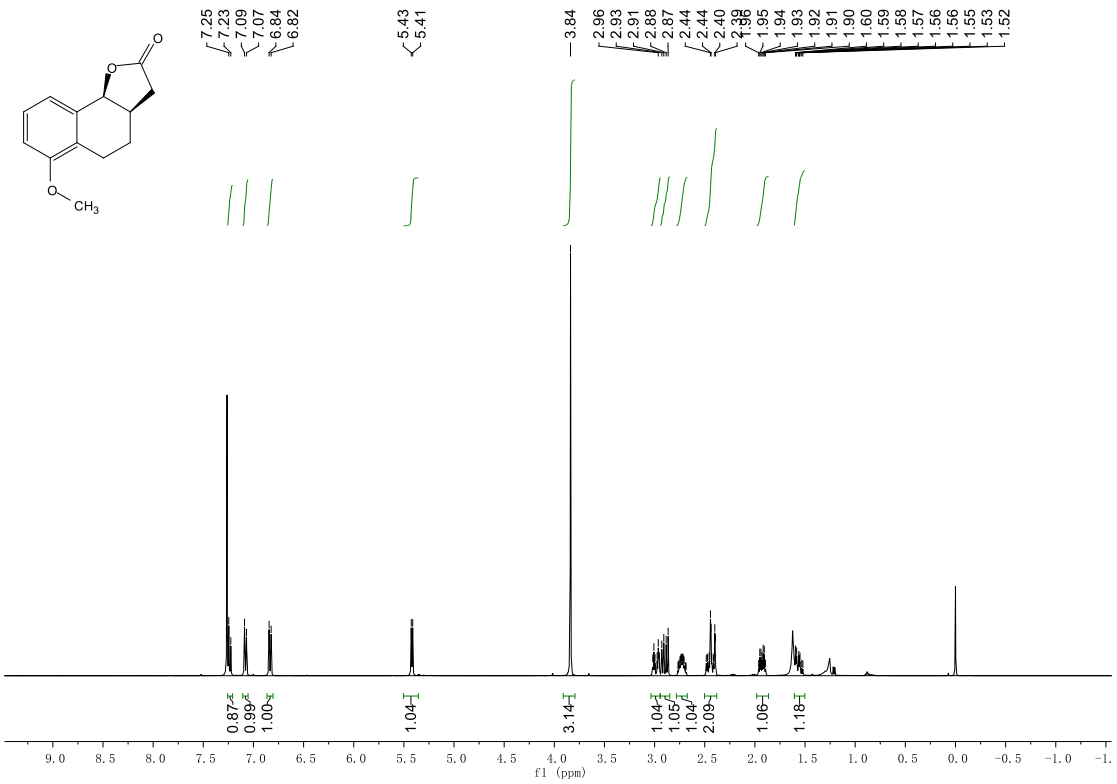


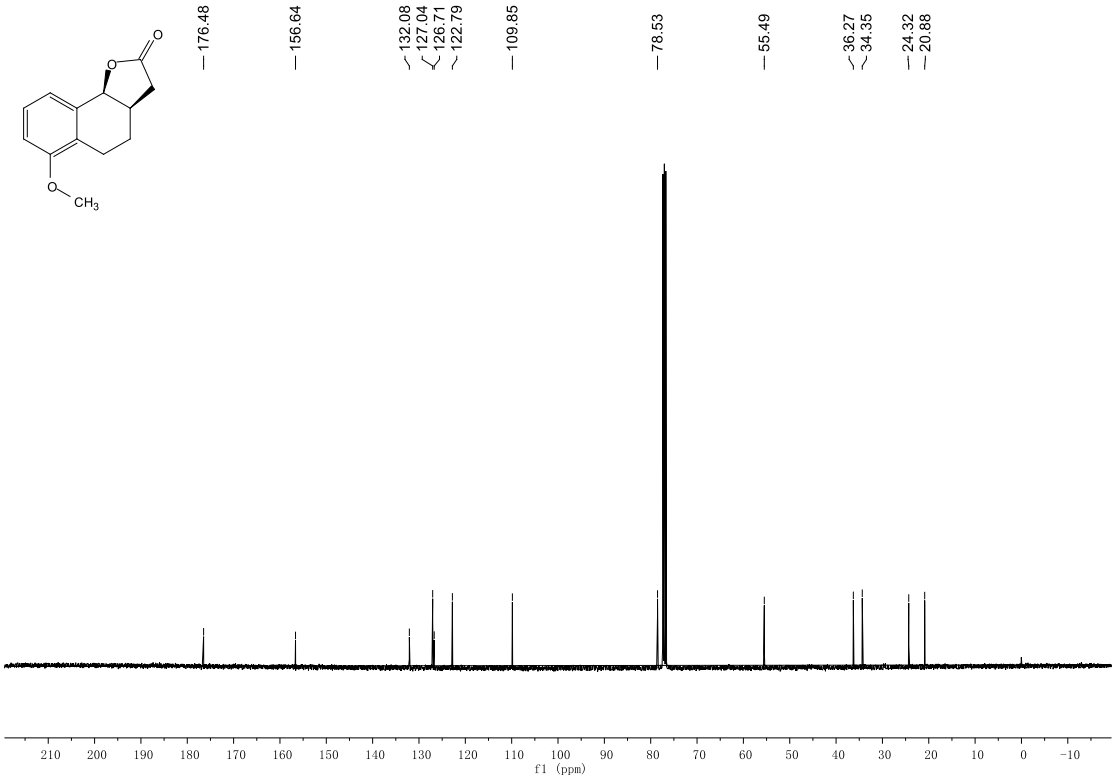


NMR spectra of **2i**

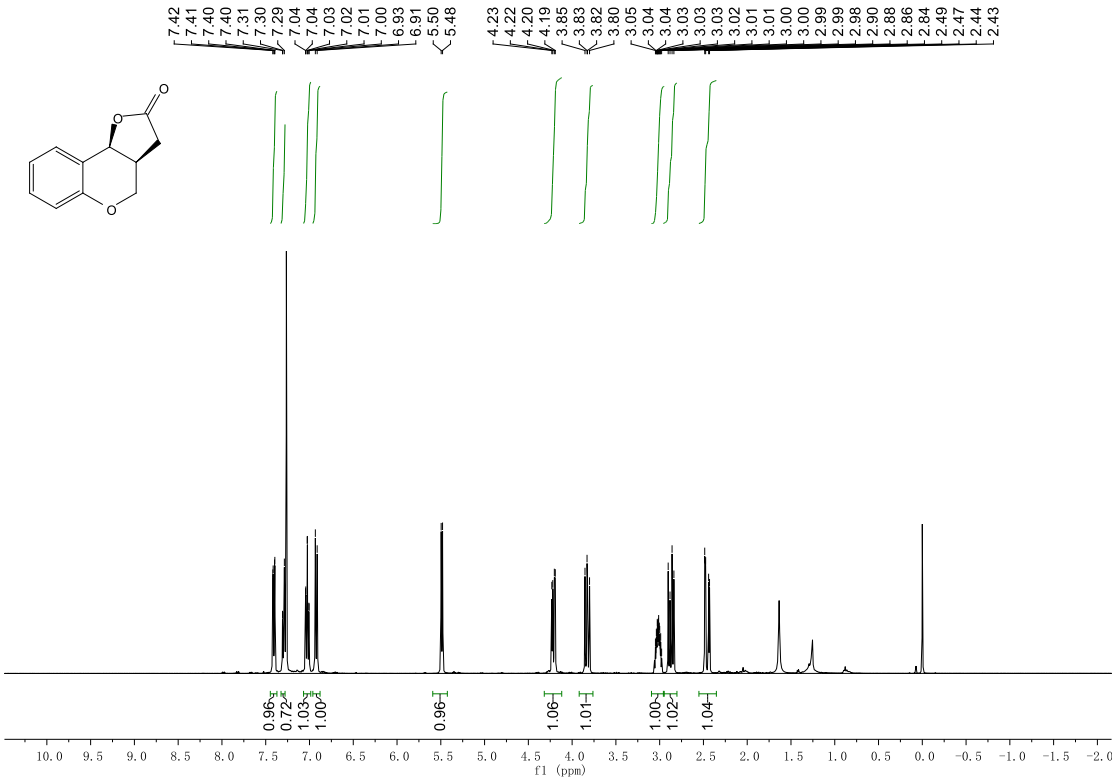


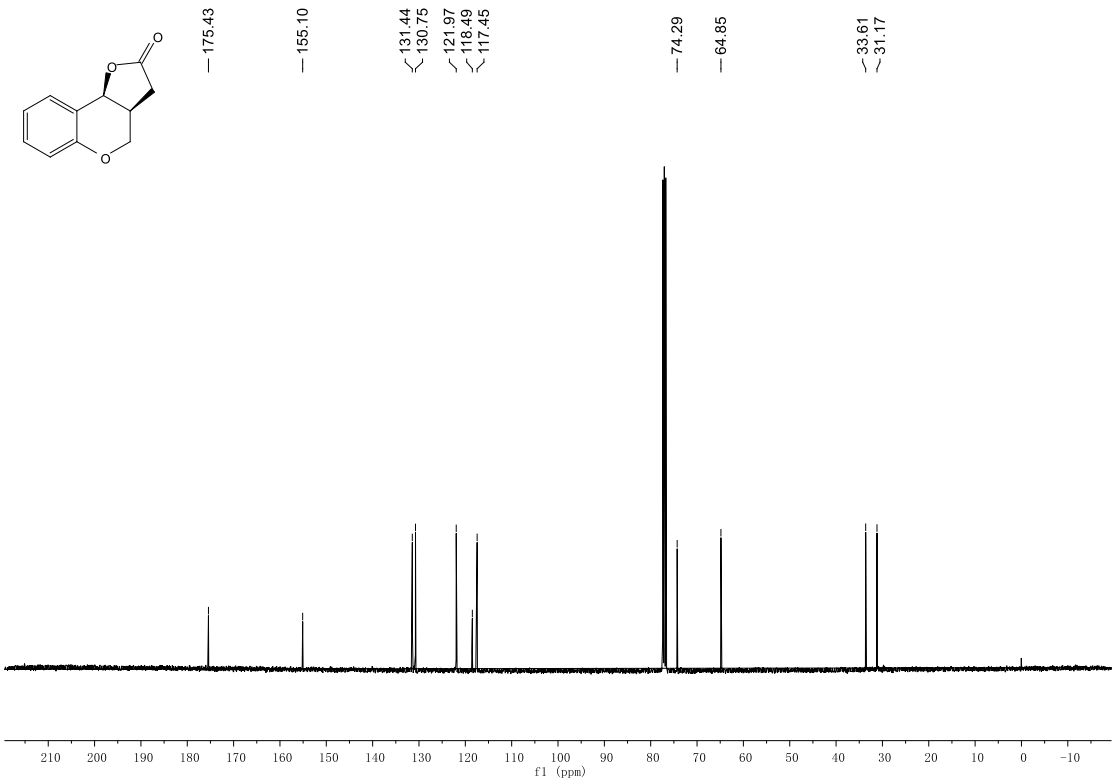
NMR spectra of **2j**



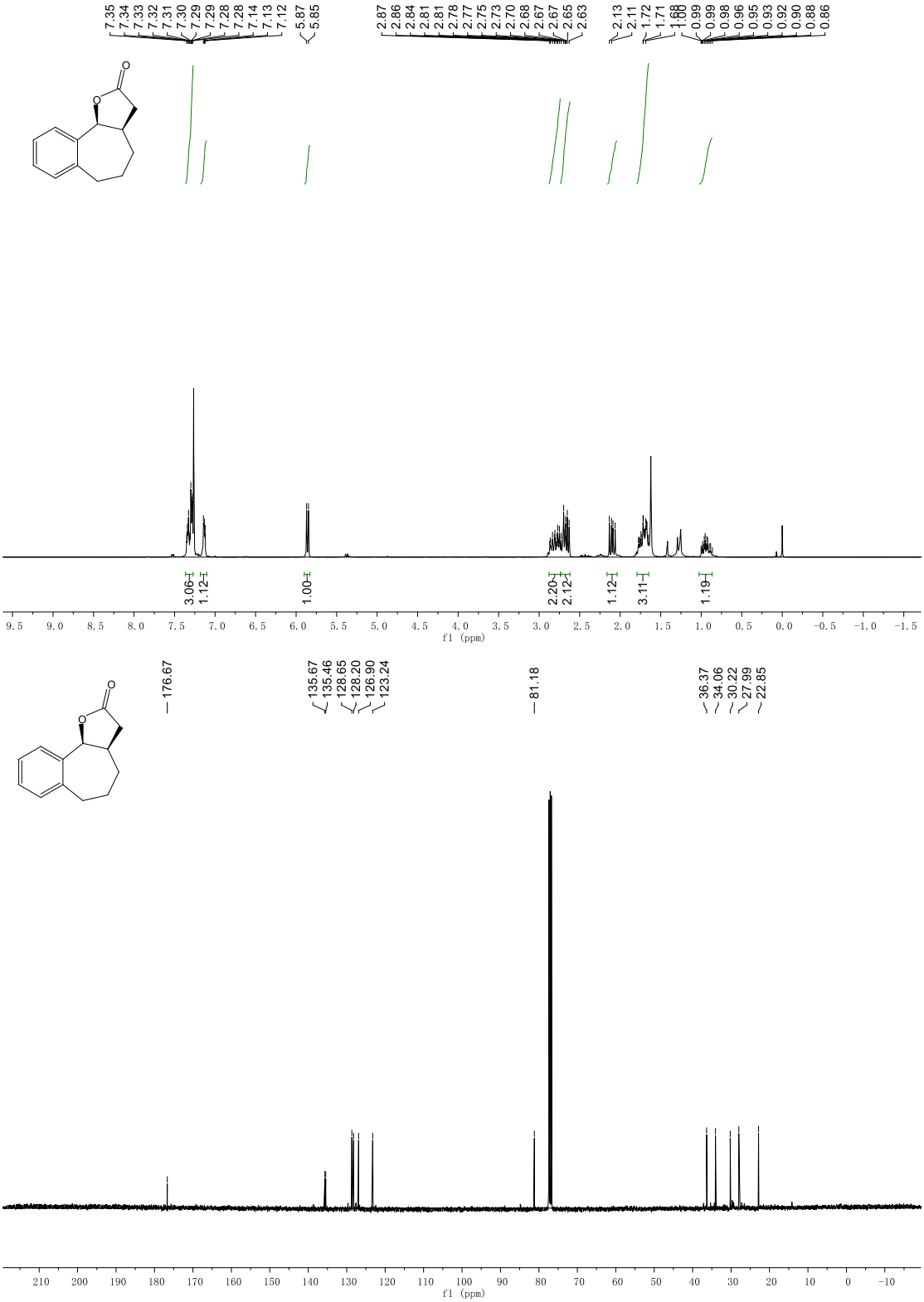


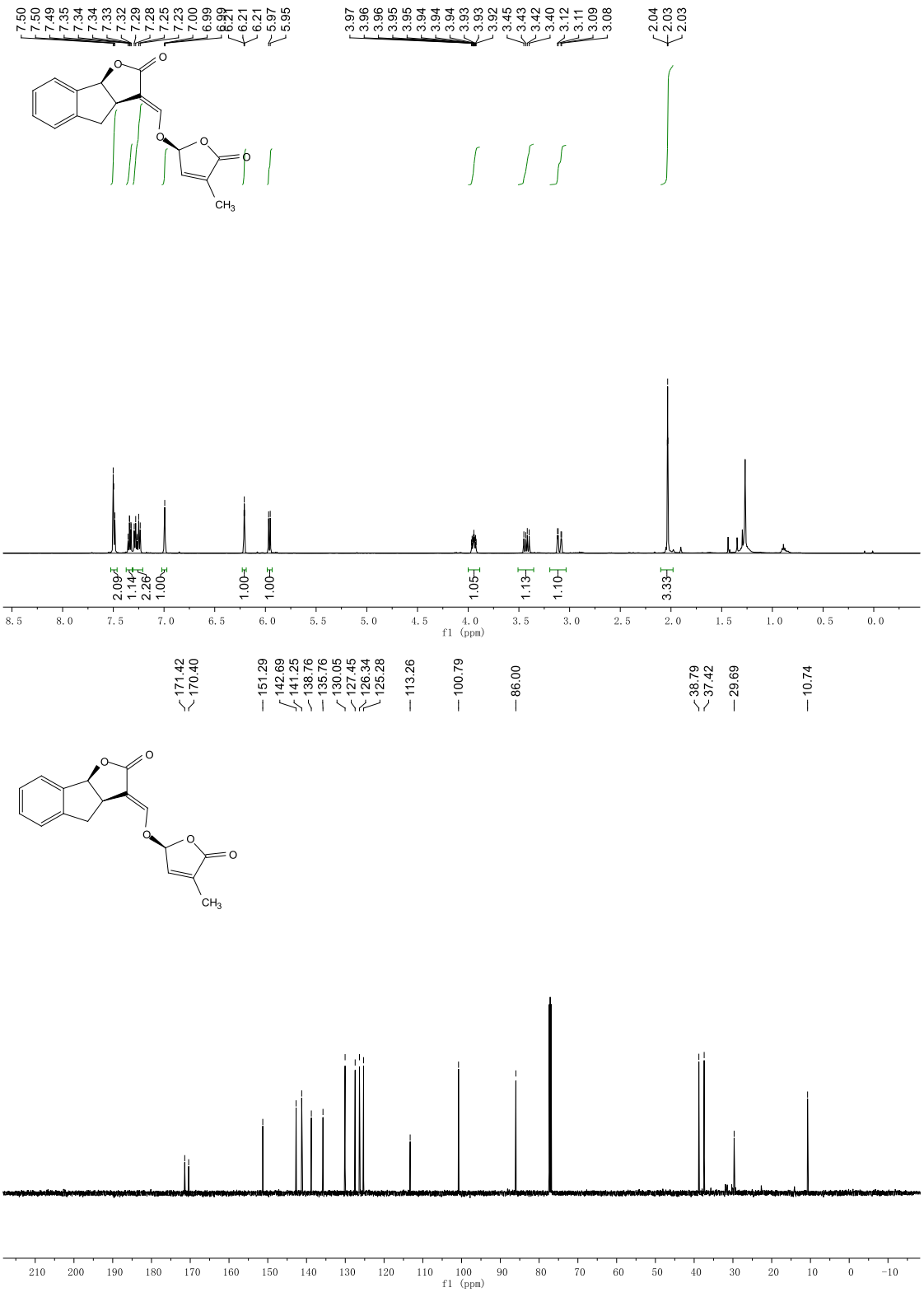
NMR spectra of **2k**





NMR spectra of **2l**

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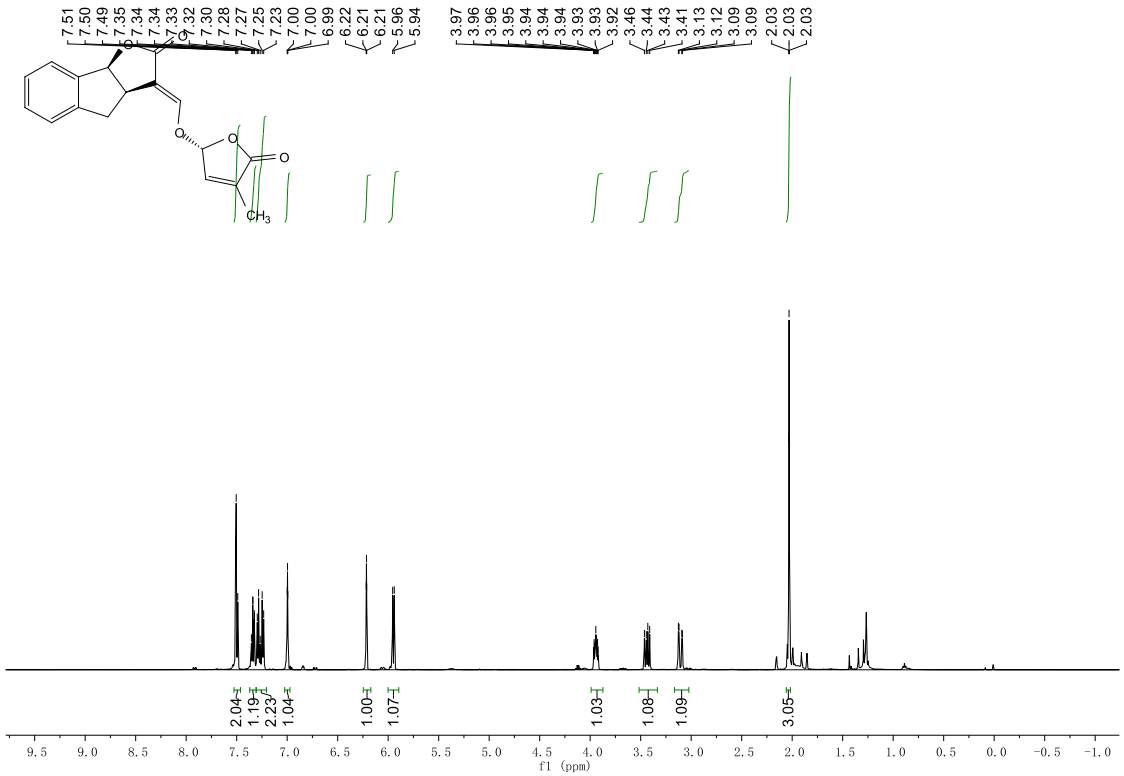
7.50
7.49
7.35
7.34
7.34
7.33
7.32
7.29
7.28
7.25
7.23
7.00
6.99
6.21
6.21
5.97
5.95
3.97
3.96
3.96
3.95
3.95
3.94
3.94
3.94
3.93
3.93
3.92
3.45
3.43
3.42
3.40
3.12
3.11
3.09
3.08
2.04
2.03
2.03
O
O
O
O
O
CH3
2.09
1.14
2.26
1.00
1.00
1.00
1.05
1.13
1.10
3.33
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
171.42
170.40
151.29
142.69
141.25
138.76
135.76
130.05
127.45
126.34
125.28
113.26
100.79
86.00
38.79
37.42
29.69
10.74
O
O
O
O
O
CH3
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)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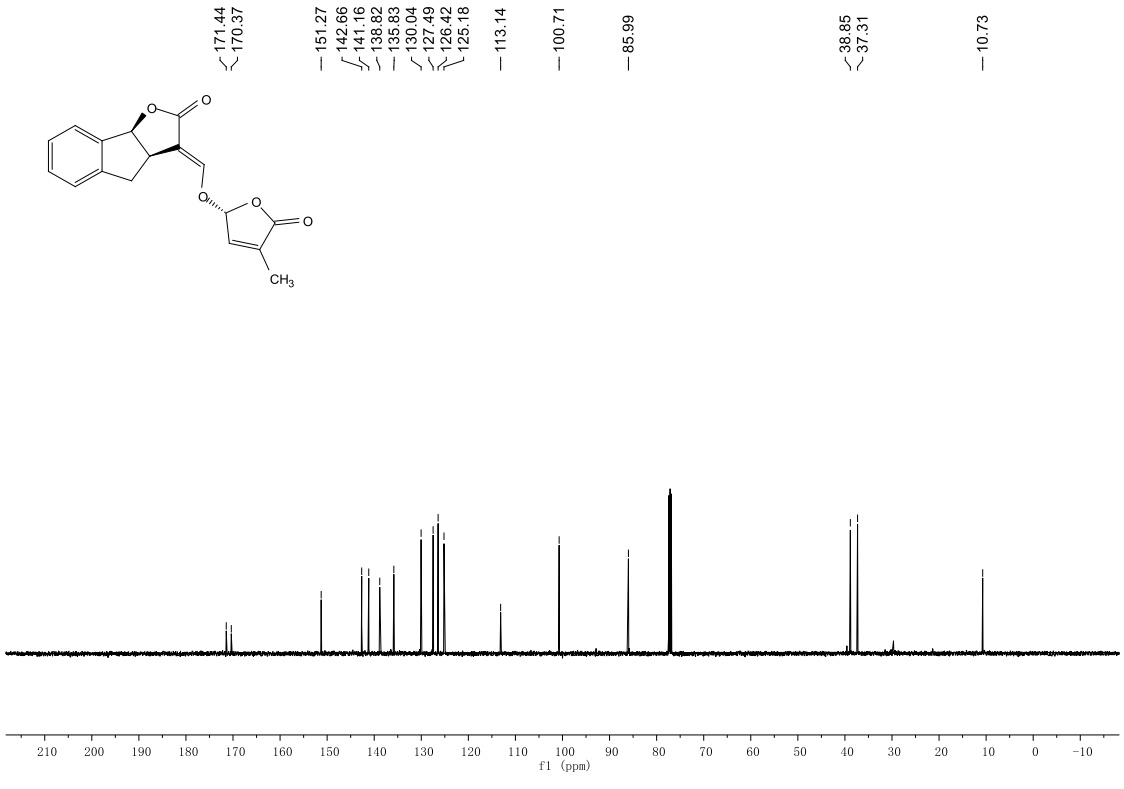
7.50
7.49
7.35
7.34
7.34
7.33
7.32
7.30
7.28
7.27
7.25
7.23
7.00
7.00
6.99
6.22
6.21
6.21
5.96
5.94
3.97
3.96
3.96
3.95
3.94
3.94
3.94
3.93
3.93
3.92
3.46
3.44
3.43
3.41
3.13
3.12
3.09
3.09
2.03
2.03
2.03
O
O
O
CH3
2.04
1.19
2.23
1.04
1.00
1.07
1.03
1.08
1.09
3.05
f1 (ppm)


171.44
170.37
151.27
142.66
141.16
138.82
135.83
130.04
127.49
126.42
125.18
113.14
100.71
85.99
38.85
37.31
10.73
O
O
O
O
O
CH3
f1 (ppm)

8. HPLC spectra of 2a-2l

Data File E:\DATA\XZC\XZC-4-159\XZC-4-159-2 2017-07-04 10-30-15\031-0201.D
Sample Name: XZC-4-159-RAC

```
    ======================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   2
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 31
    Injection Date  : 7/4/2017 10:41:48 AM               Inj :   1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\XZC\XZC-4-159\XZC-4-159-2 2017-07-04 10-30-15\VWD-AD(1-2)-85-15-
                      0.8ML-3UL-210NM-20MIN.M
    Last changed    : 7/4/2017 10:30:15 AM by SYSTEM
    Analysis Method : E:\DATA\XZC\XZC-4-159\XZC-4-159-2 2017-07-04 10-30-15\VWD-AD(1-2)-85-15-
                      0.8ML-3UL-210NM-20MIN.M (Sequence Method)
    Last changed    : 4/8/2019 11:19:47 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
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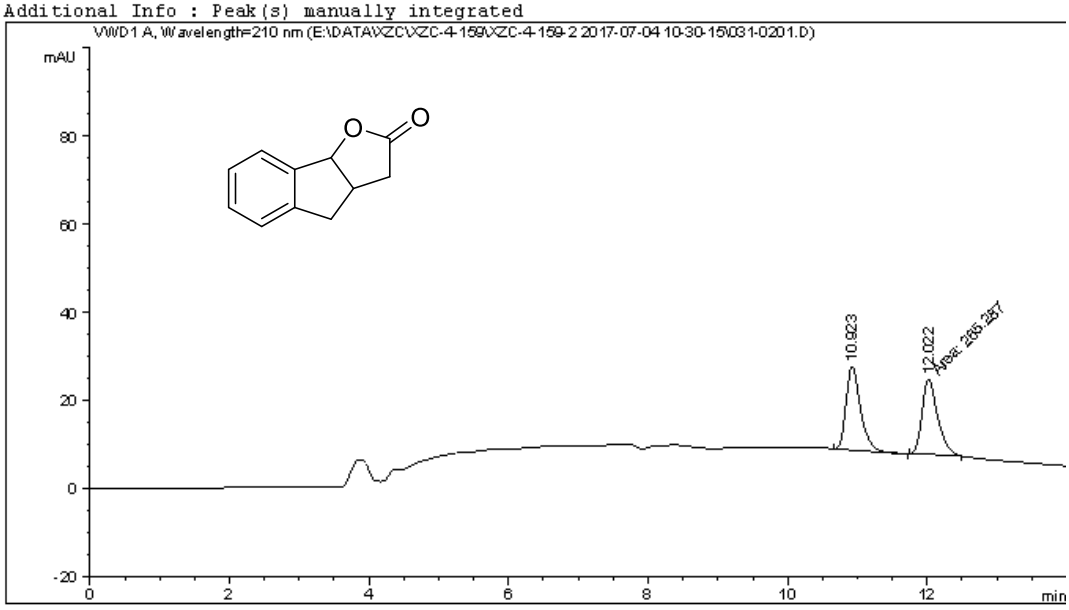


```
    ======================================================================
                              Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  10.923 BV    0.2170  275.72559   19.03780  50.9647
       2  12.022 MM    0.2582  265.28693   17.12114  49.0353

    Totals :                   541.01251   36.15894


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

1260HPLC-VWD 4/8/2019 11:19:54 AM SYSTEM                    Page 1 of 1
```

Data File E:\DATA\XZC\XZC-4-159\XZC-4-159-3 2017-07-04 11-12-30\097-1101.D
Sample Name: XZC-4-159-6

```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  11
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 97
    Injection Date  : 7/4/2017 2:30:48 PM                 Inj :  1
                                                   Inj Volume : 3.000 µl
    Acq. Method     : E:\DATA\XZC\XZC-4-159\XZC-4-159-3 2017-07-04 11-12-30\VWD-AD(1-2)-85-15-
                      0.8ML-3UL-210NM-20MIN.M
    Last changed    : 7/4/2017 11:12:30 AM by SYSTEM
    Analysis Method : E:\DATA\XZC\XZC-4-159\XZC-4-159-3 2017-07-04 11-12-30\VWD-AD(1-2)-85-15-
                      0.8ML-3UL-210NM-20MIN.M (Sequence Method)
    Last changed    : 4/8/2019 11:18:14 AM by SYSTEM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
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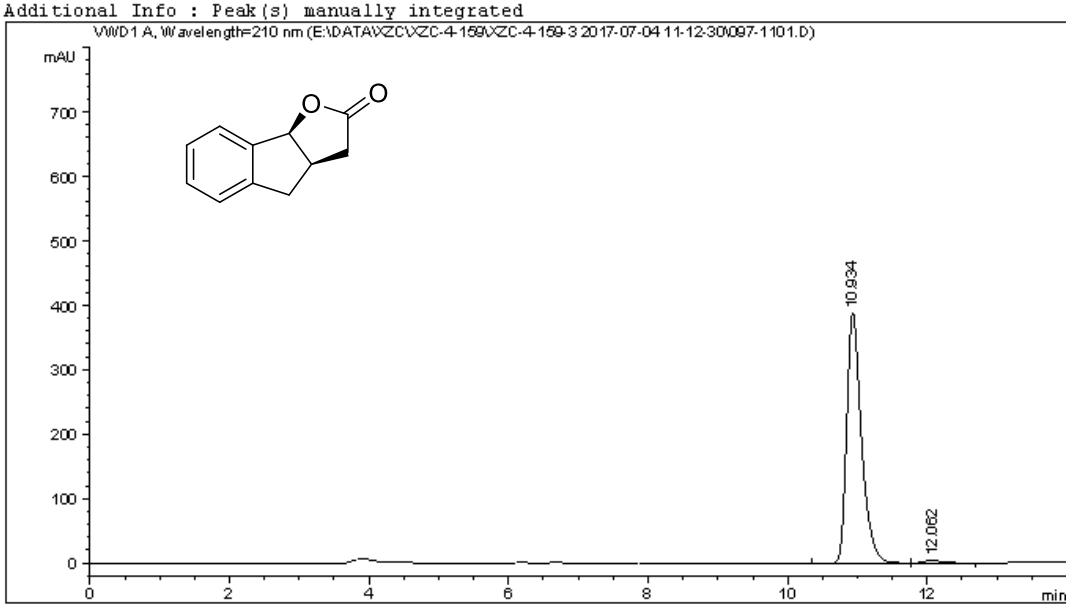
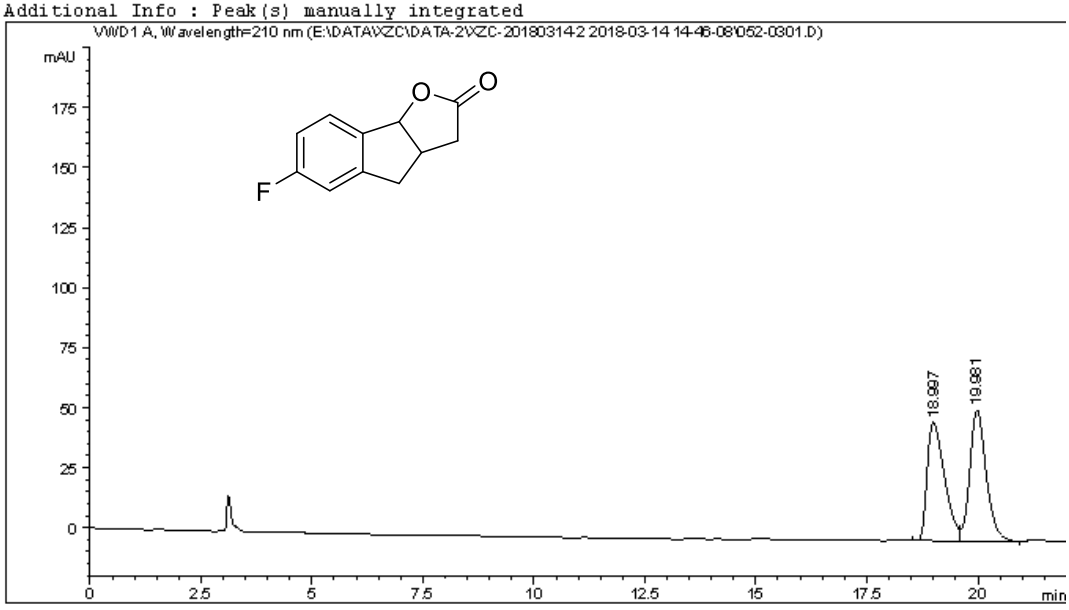



```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  10.934 BB    0.2208 5685.89355  388.29538  98.7844
       2  12.062 BB    0.2554   69.96854    4.07334   1.2156

    Totals :                  5755.86209  392.36872


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

Data File E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\052-0301.D
Sample Name: XZC-pF-RAC-1

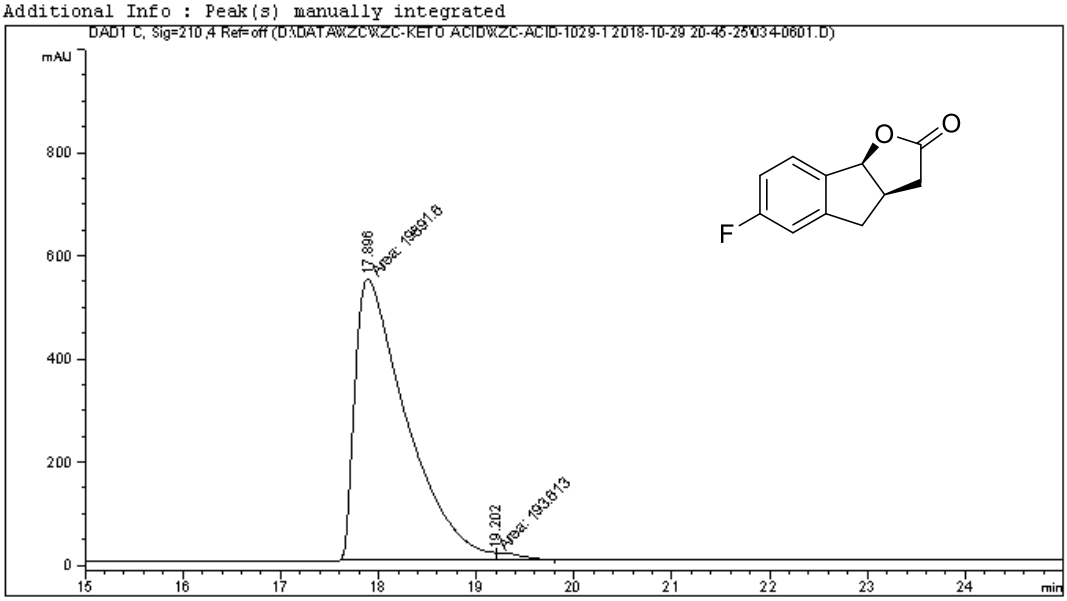
```
    ====================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :   3
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 52
    Injection Date  : 3/14/2018 3:38:36 PM               Inj :   1
                                                   Inj Volume : 2.000 µl
    Acq. Method     : E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\VWD-AD(1-2)-95-5-
                      1ML-2UL-210NM-40MIN.M
    Last changed    : 3/14/2018 2:46:08 PM by SYSTEM
    Analysis Method : E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\VWD-AD(1-2)-95-5-
                      1ML-2UL-210NM-40MIN.M (Sequence Method)
    Last changed    : 4/8/2019 10:58:20 AM by SYSTEM
                      (modified after loading)
    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=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  18.997 BV    0.4186 1336.70410   49.26241  48.8079
       2  19.981 VB    0.3899 1402.00256   54.53653  51.1921

    Totals :                  2738.70667  103.79893


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

Data File D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\034-0601.D
Sample Name: XZC-acid-pF

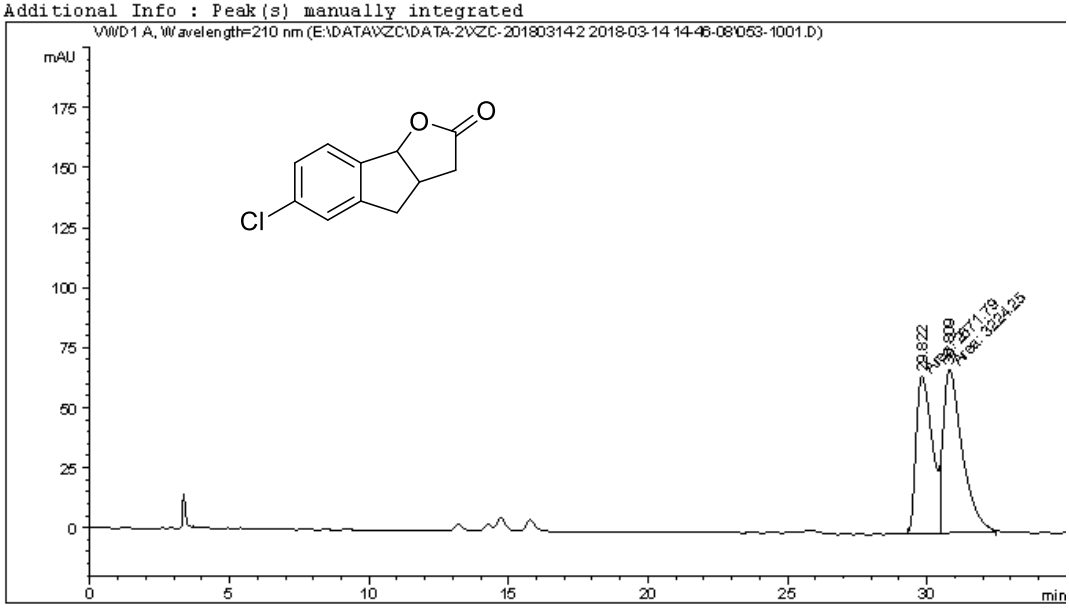
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   6
    Acq. Instrument : Instrument 2                    Location : Vial 34
    Injection Date  : 10/29/2018 10:26:43 PM              Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\DAD-AD(1-6)-
                      95-5-1ML-5UL-210-35MIN.M
    Last changed    : 10/29/2018 8:46:43 PM
    Analysis Method : D:\METHOD\XZC\VWD-OD(1-6)-90-10-1ML-5UL-210NM-40MIN.M
    Last changed    : 10/30/2018 12:00:56 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 C, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  17.896 MF    0.6084 1.98916e4   544.92200  99.0360
       2  19.202 FM    0.2470  193.61253   13.06445   0.9640

    Totals :                  2.00852e4   557.98644
```

Data File E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\053-1001.D
Sample Name: XZC-pCl-RAC-2

Acq. Operator	: SYSTEM	Seq. Line	: 10
Acq. Instrument	: 1260HPLC-VWD	Location	: Vial 53
Injection Date	: 3/14/2018 7:24:35 PM	Inj	: 1
		Inj Volume	: 2.000 µl
Acq. Method	: E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\VWD-IB(1-6)-95-5-1ML-2UL-210NM-40MIN.M		
Last changed	: 3/14/2018 3:52:29 PM by SYSTEM		
Analysis Method	: E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\VWD-IB(1-6)-95-5-1ML-2UL-210NM-40MIN.M (Sequence Method)		
Last changed	: 4/8/2019 11:00:51 AM by SYSTEM (modified after loading)		
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

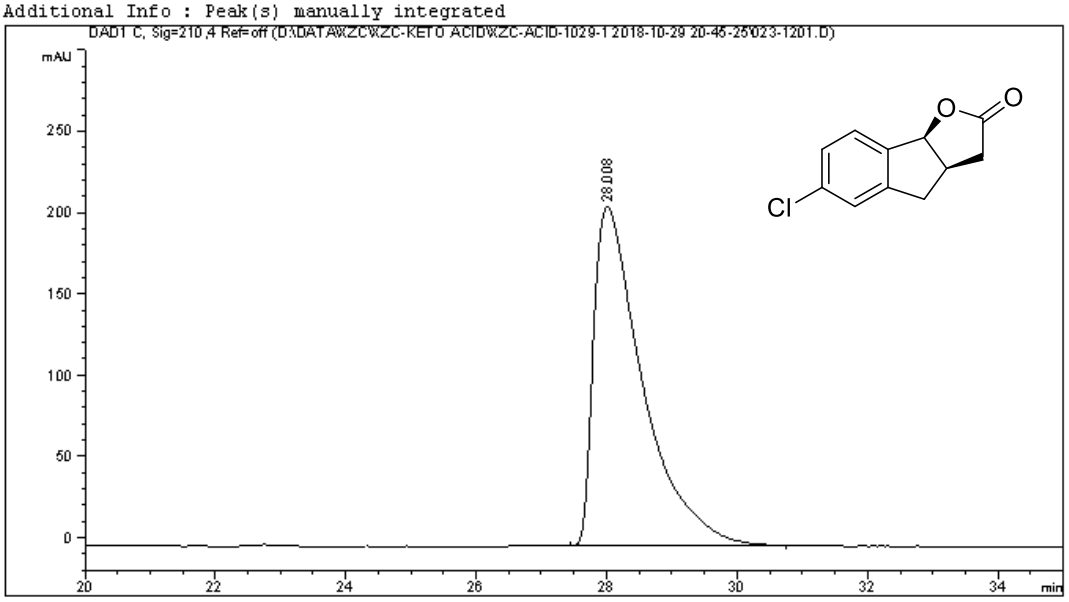
Signal 1: VWD1 A, Wavelength=210 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.822	MF	0.6802	2671.78833	65.46633	45.3150
2	30.809	FM	0.7901	3224.25220	68.01778	54.6850
Totals :				5896.04053	133.48411	

*** End of Report ***

Data File D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\023-1201.D
Sample Name: XZC-acid-pCl

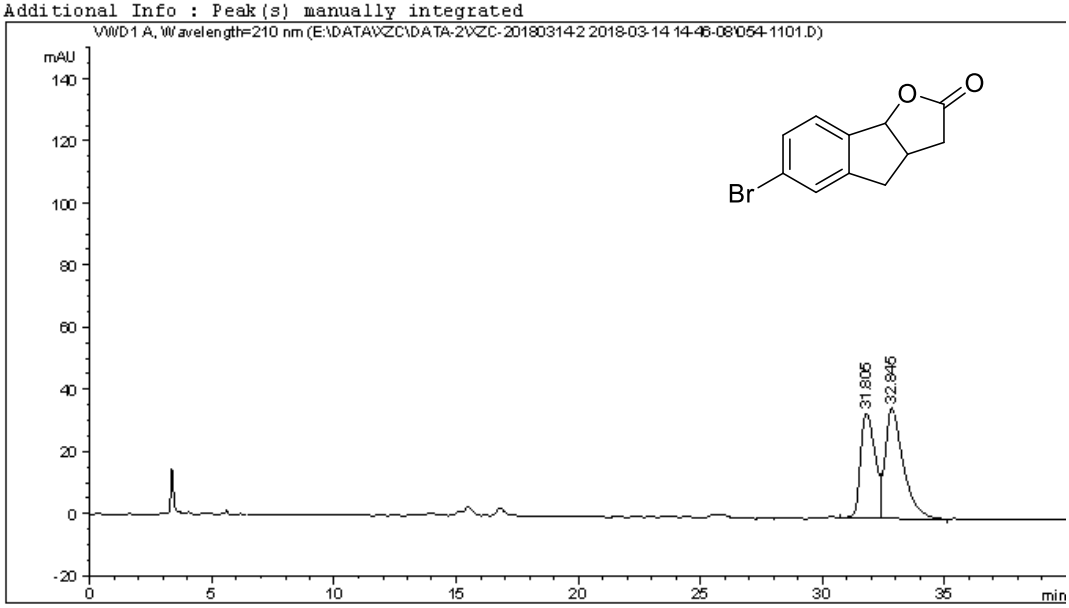
```
    ======================================================================
    Acq. Operator   :                               Seq. Line :  12
    Acq. Instrument : Instrument 2                   Location : Vial 23
    Injection Date  : 10/30/2018 2:07:48 AM               Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\DAD-IB(1-2)-
                      95-5-1ML-5UL-210-35MIN.M
    Last changed    : 10/29/2018 9:14:44 PM
    Analysis Method : D:\METHOD\LWD\DAD-AD(1-6)-95-5-1ML-3UL-ALL-10MIN.M
    Last changed    : 10/30/2018 12:04:06 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 C, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  28.008 BB    0.7630 1.08725e4   208.29680 100.0000

    Totals :                  1.08725e4   208.29680


    ======================================================================
```

Data File E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\054-1101.D
Sample Name: XZC-pBr-RAC-2

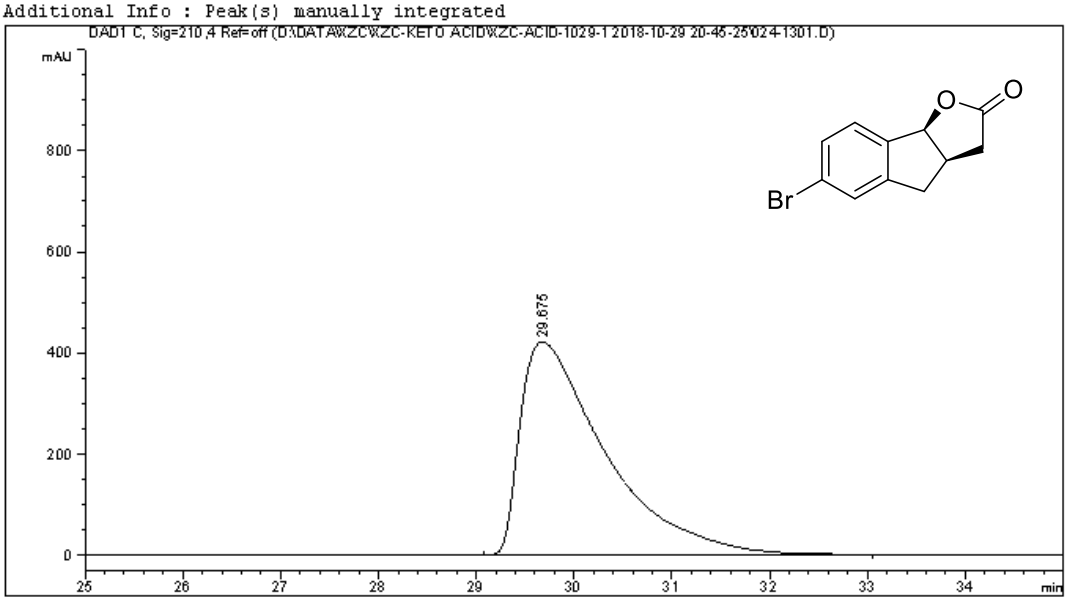
```
    ==================================================================
    Acq. Operator   : SYSTEM                        Seq. Line :  11
    Acq. Instrument : 1260HPLC-VWD                   Location : Vial 54
    Injection Date  : 3/14/2018 8:05:19 PM               Inj :  1
                                                   Inj Volume : 2.000 µl
    Acq. Method     : E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\VWD-IB(1-6)-95-5-
                      1ML-2UL-210NM-40MIN.M
    Last changed    : 3/14/2018 3:52:29 PM by SYSTEM
    Analysis Method : E:\DATA\XZC\DATA-2\XZC-20180314-2 2018-03-14 14-46-08\VWD-IB(1-6)-95-5-
                      1ML-2UL-210NM-40MIN.M (Sequence Method)
    Last changed    : 4/8/2019 11:04:40 AM by SYSTEM
                      (modified after loading)
    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=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  31.805 BV    0.6424 1402.03259   33.69883  43.3821
       2  32.845 VB    0.7539 1829.79211   35.40828  56.6179

    Totals :                  3231.82471   69.10712


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

Data File D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\024-1301.D
Sample Name: XZC-acid-pBr

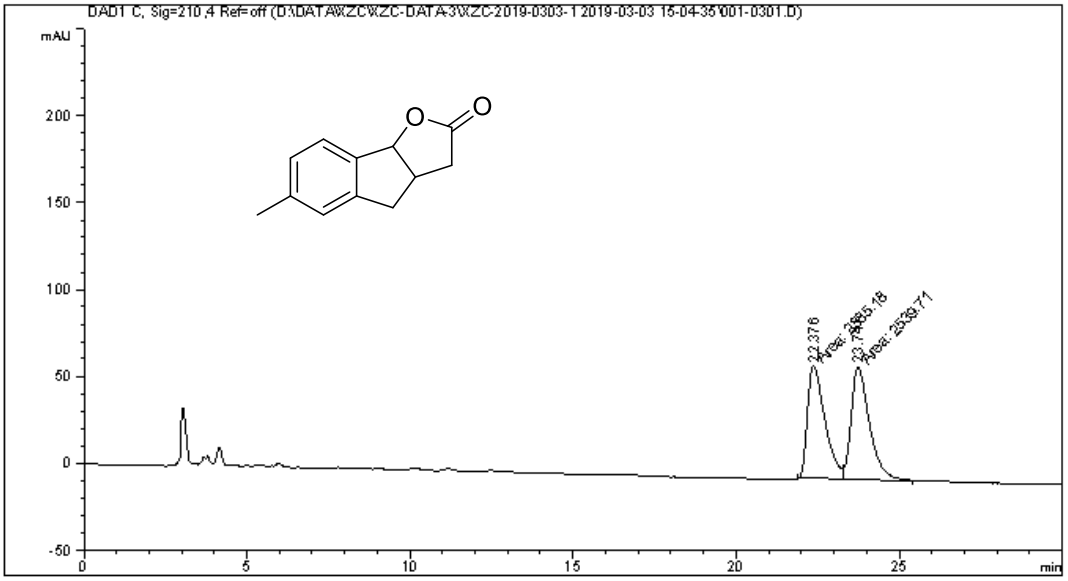
```
    ======================================================================
    Acq. Operator   :                               Seq. Line :  13
    Acq. Instrument : Instrument 2                   Location : Vial 24
    Injection Date  : 10/30/2018 2:43:47 AM               Inj :  1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\DAD-IB(1-2)-
                      95-5-1ML-5UL-210-35MIN.M
    Last changed    : 10/29/2018 9:14:44 PM
    Analysis Method : D:\METHOD\XZC\VWD-OD(1-6)-90-10-1ML-5UL-210NM-40MIN.M
    Last changed    : 10/30/2018 2:17:17 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 C, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  29.675 BB    0.8708 2.53935e4   421.53577 100.0000

    Totals :                  2.53935e4   421.53577


    ======================================================================
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0303-1 2019-03-03 15-04-35\001-0301.D
Sample Name: xzc-pMe-rac

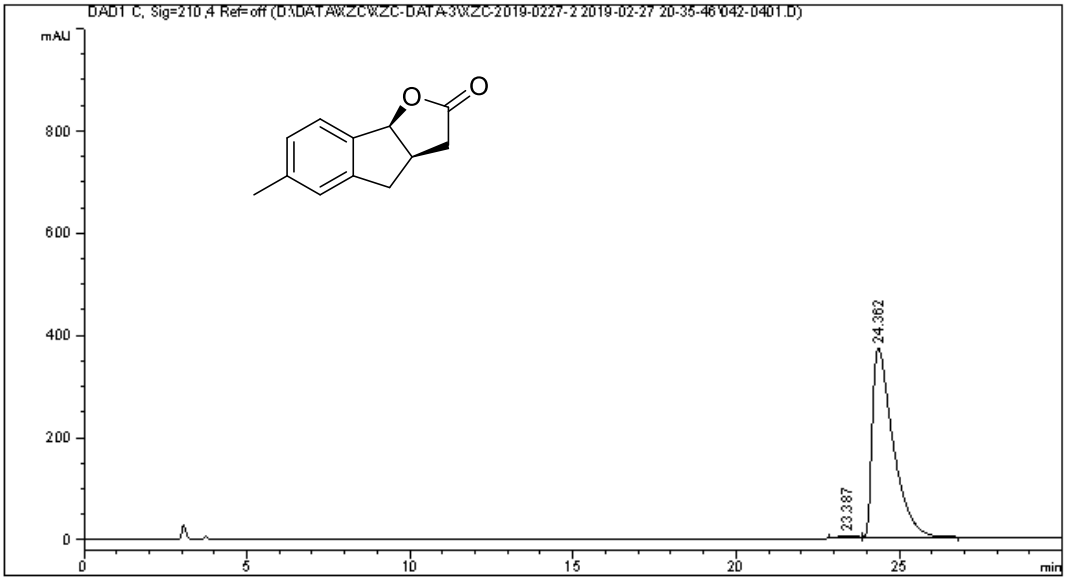
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   3
    Acq. Instrument : Instrument 2                    Location : Vial 1
    Injection Date  : 3/3/2019 3:28:25 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0303-1 2019-03-03 15-04-35\DAD-OD(1-2)-95-5
                      --1ML-5UL-210NM-40MIN.M
    Last changed    : 10/9/2018 3:51:57 PM
    Analysis Method : D:\METHOD\XZC\VWD-AD(1-2)-95-5-1ML-5UL-210NM-30MIN.M
    Last changed    : 3/3/2019 7:00:04 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 C, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  22.376 MF    0.6157 2385.17847   64.56731  48.4311
       2  23.741 FM    0.6524 2539.71045   64.87852  51.5689

    Totals :                  4924.88892  129.44583
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0227-2 2019-02-27 20-35-46\042-0401.D
Sample Name: xzc-pMe

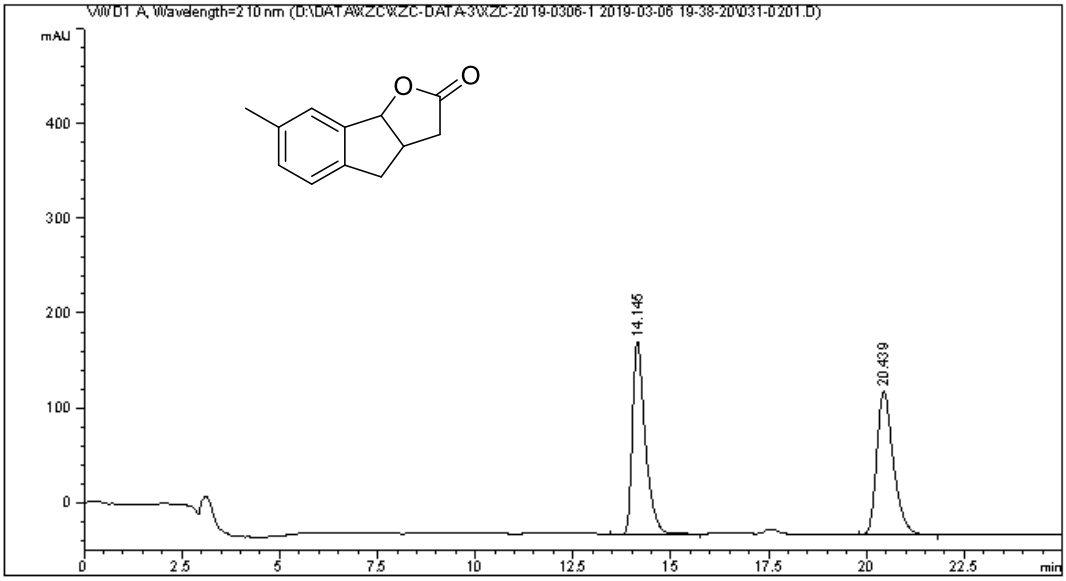
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   4
    Acq. Instrument : Instrument 2                    Location : Vial 42
    Injection Date  : 2/27/2019 9:50:52 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0227-2 2019-02-27 20-35-46\DAD-OD(1-2)-95-5
                      --1ML-5UL-210NM-40MIN.M
    Last changed    : 10/9/2018 3:51:57 PM
    Analysis Method : D:\METHOD\XZC\VWD-AD(1-2)-95-5-1ML-5UL-210NM-30MIN.M
    Last changed    : 3/3/2019 6:56:44 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 C, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  23.387 BV    0.4517  158.85115    4.15810   0.9559
       2  24.362 VB    0.6184 1.64592e4   371.88657  99.0441

    Totals :                  1.66181e4   376.04466
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0306-1 2019-03-06 19-38-20\031-0201.D
Sample Name: XZC-mMe-RAC

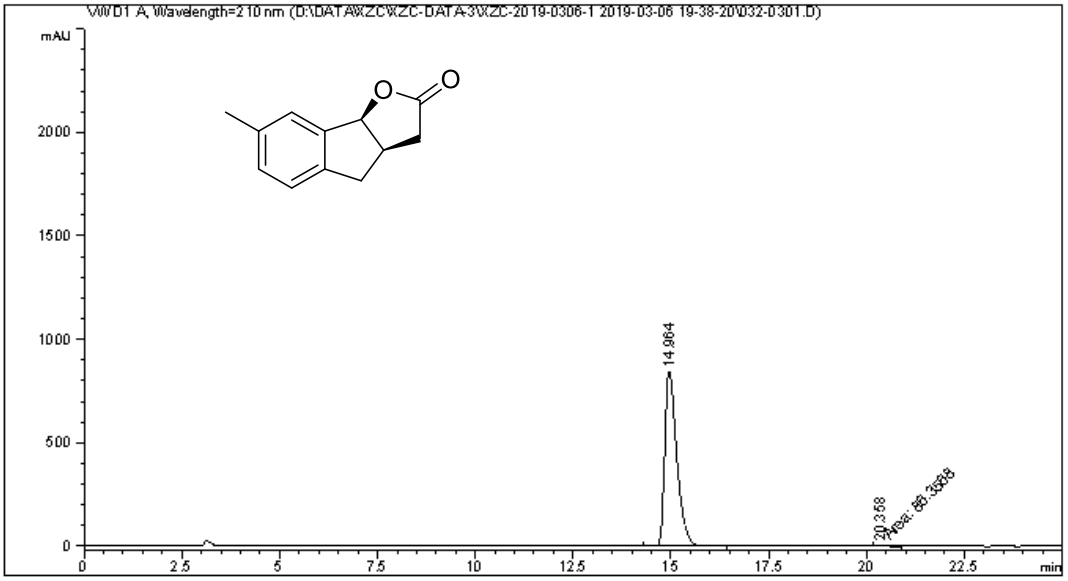
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   2
    Acq. Instrument : Instrument 1                    Location : Vial 31
    Injection Date  : 3/6/2019 7:50:08 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0306-1 2019-03-06 19-38-20\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\LWD\VWD-IA(1-6)-959-1-0.5ML-2UL-210NM-35MIN.M
    Last changed    : 3/7/2019 2:19:54 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  14.145 BB    0.3437 4643.11279  203.12628  50.9618
       2  20.439 BB    0.4463 4467.85303  151.38580  49.0382

    Totals :                  9110.96582  354.51208
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0306-1 2019-03-06 19-38-20\032-0301.D
Sample Name: XZC-mMe

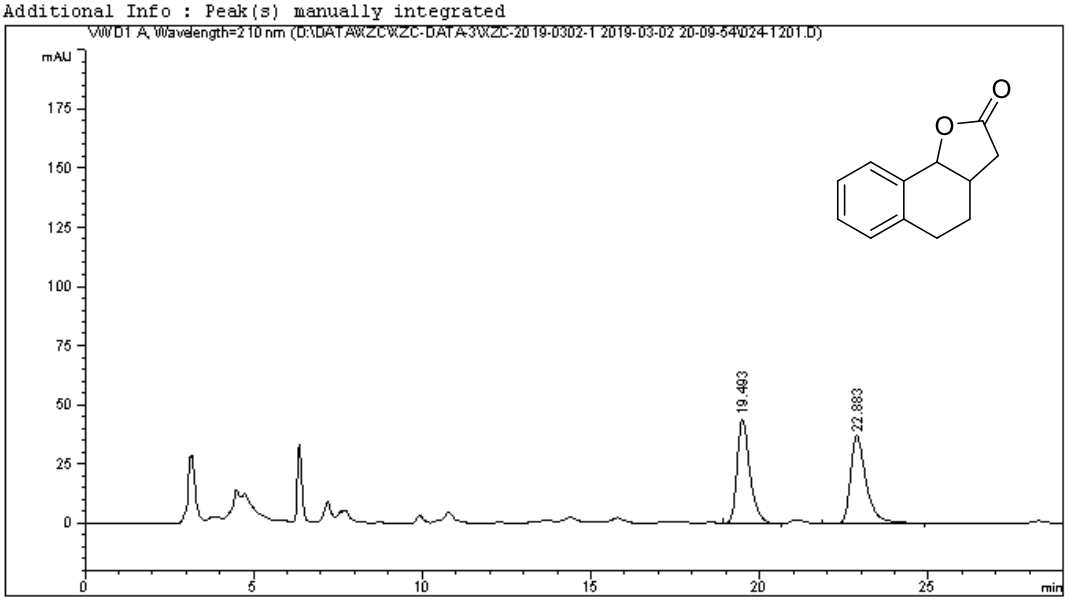
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   3
    Acq. Instrument : Instrument 1                    Location : Vial 32
    Injection Date  : 3/6/2019 8:30:58 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0306-1 2019-03-06 19-38-20\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\LWD\VWD-IA(1-6)-959-1-0.5ML-2UL-210NM-35MIN.M
    Last changed    : 3/7/2019 2:22:29 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  14.964 BB    0.3209 1.80084e4   847.57568  99.5228
       2  20.358 MM    0.5932   86.35678    2.42628   0.4772

    Totals :                  1.80947e4   850.00196
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0302-1 2019-03-02 20-09-54\024-1201.D
Sample Name: XZC-hex-RAC

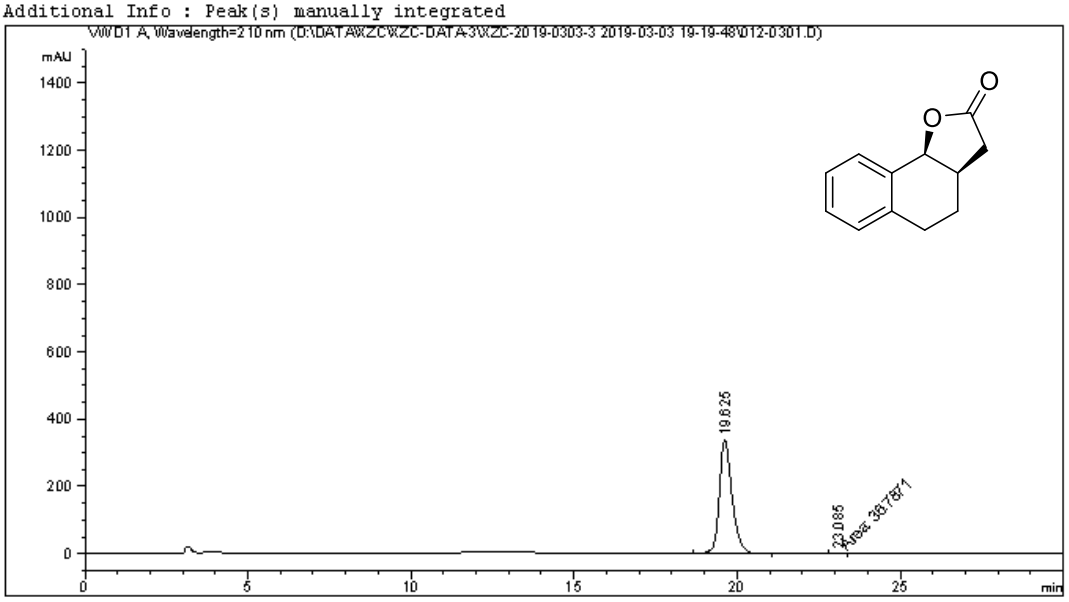
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :  12
    Acq. Instrument : Instrument 1                    Location : Vial 24
    Injection Date  : 3/3/2019 12:50:09 AM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0302-1 2019-03-02 20-09-54\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-30MIN.M
    Last changed    : 3/2/2019 8:06:09 PM
    Analysis Method : D:\METHOD\ZX\VWD-AD(1-2)-90-10-0.5ML-3UL-254NM-60MIN.M
    Last changed    : 3/13/2019 3:29:13 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  19.493 BB    0.3972 1153.15930   43.79002  48.2949
       2  22.883 BB    0.4928 1234.58728   37.33555  51.7051

    Totals :                  2387.74658   81.12557
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0303-3 2019-03-03 19-19-48\012-0301.D
Sample Name: XZC-hex

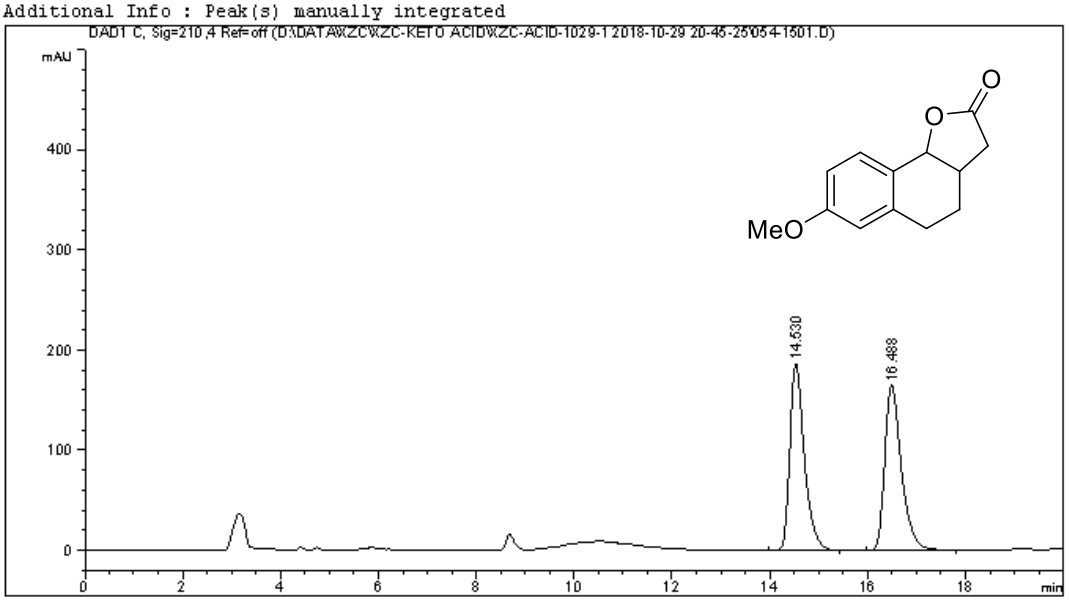
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   3
    Acq. Instrument : Instrument 1                    Location : Vial 12
    Injection Date  : 3/3/2019 8:03:25 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0303-3 2019-03-03 19-19-48\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-30MIN.M
    Last changed    : 3/2/2019 8:06:09 PM
    Analysis Method : D:\METHOD\GUAN YUQING\LONGJIAO\DAD-0J(1-6)-99-1-0.5ML-5UL-ALL-60MIN.M
    Last changed    : 3/13/2019 3:30:05 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  19.625 BB    0.4099 9240.15527  339.09265  99.6035
       2  23.085 MM    0.3791   36.78712    1.46427   0.3965

    Totals :                  9276.94239  340.55692
```

Data File D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\054-1501.D
Sample Name: XZC-acid-pMeO-rac

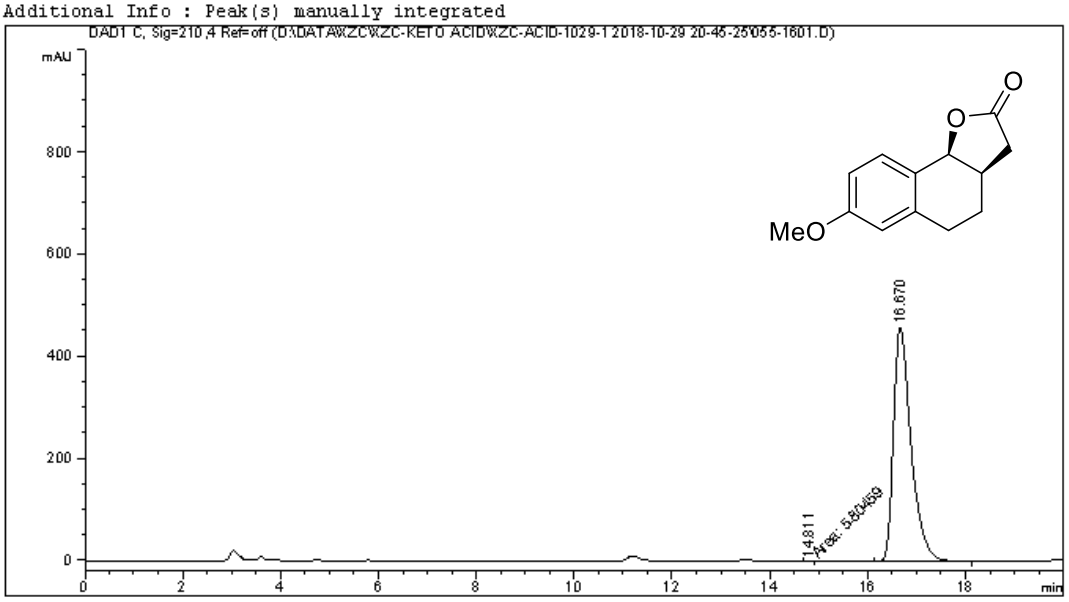
```
    ======================================================================
    Acq. Operator   :                              Seq. Line :  15
    Acq. Instrument : Instrument 2                  Location : Vial 54
    Injection Date  : 10/30/2018 3:30:58 AM              Inj :  1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\DAD-AD(1-6)-
                      90-10-1ML-5UL-210-25MIN.M
    Last changed    : 10/29/2018 8:42:51 PM
    Analysis Method : D:\METHOD\XZC\VWD-AD(1-2)-95-5-1ML-5UL-210NM-30MIN.M
    Last changed    : 3/3/2019 3:41:05 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 C, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  14.530 BB    0.3104 3838.75977  186.28035  49.6911
       2  16.488 BB    0.3527 3886.48804  165.05344  50.3089

    Totals :                  7725.24780  351.33379
```

Data File D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\055-1601.D
Sample Name: XZC-acid-pMeO

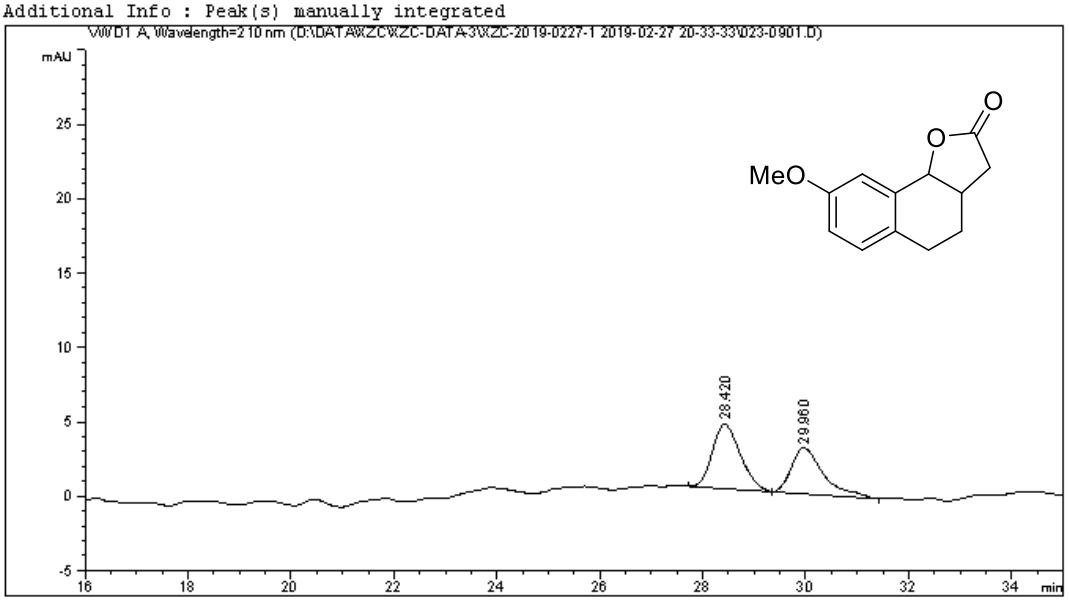
```
    ======================================================================
    Acq. Operator   :                              Seq. Line :  16
    Acq. Instrument : Instrument 2                  Location : Vial 55
    Injection Date  : 10/30/2018 3:56:59 AM              Inj :  1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-KETO ACID\XZC-ACID-1029-1 2018-10-29 20-45-25\DAD-AD(1-6)-
                      90-10-1ML-5UL-210-25MIN.M
    Last changed    : 10/29/2018 8:42:51 PM
    Analysis Method : D:\METHOD\XZC\VWD-AD(1-2)-95-5-1ML-5UL-210NM-30MIN.M
    Last changed    : 3/3/2019 3:45:43 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: DAD1 C, Sig=210,4 Ref=off

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  14.811 MM    0.2260    5.80459 3.73527e-1   0.0508
       2  16.670 BB    0.3767 1.14145e4   458.07208  99.9492

    Totals :                  1.14203e4   458.44561
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0227-1 2019-02-27 20-33-33\023-0901.D
Sample Name: xzc-2MeO-rac

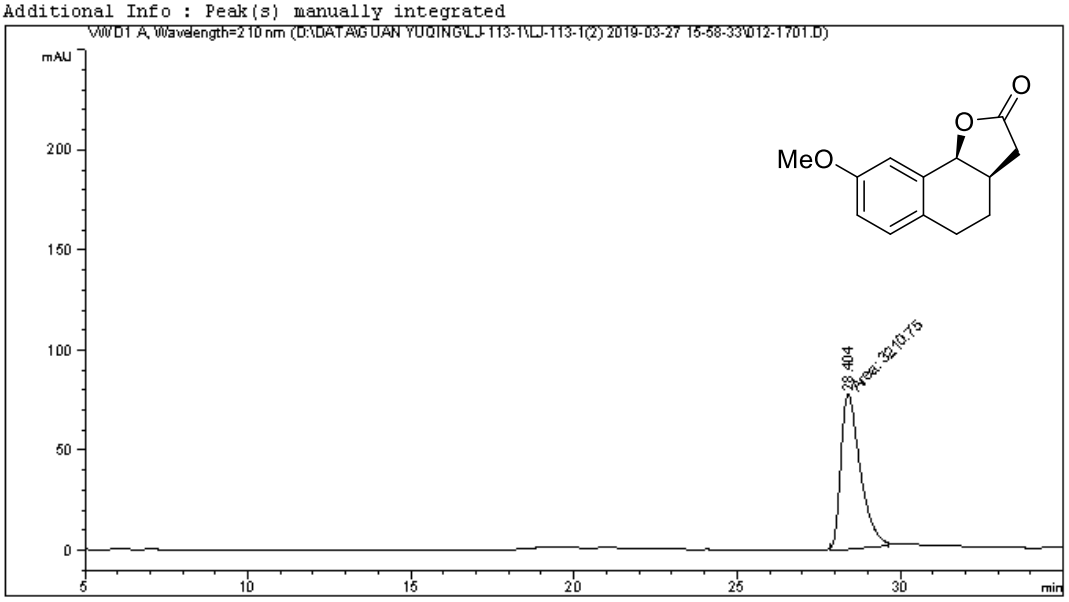
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   9
    Acq. Instrument : Instrument 1                    Location : Vial 23
    Injection Date  : 2/28/2019 1:01:17 AM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0227-1 2019-02-27 20-33-33\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\LYH\VWD-AD(1-2)-95-5-1ML-1UL-210NM-60MIN.M
    Last changed    : 4/8/2019 4:52:50 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  28.420 BB    0.5586  163.65729    4.33341  55.2386
       2  29.960 BB    0.6178  132.61629    3.09563  44.7614

    Totals :                   296.27357    7.42904
```

Data File D:\DATA\GUAN YUQING\LJ-113-1\LJ-113-1(2) 2019-03-27 15-58-33\012-1701.D
Sample Name: xzc-2MeO

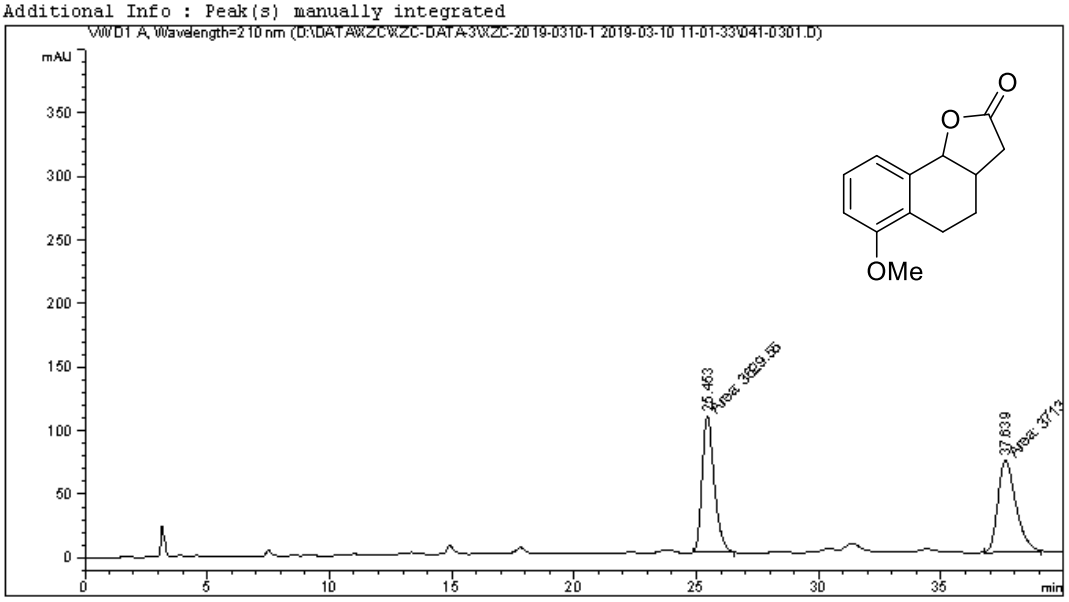
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :  17
    Acq. Instrument : Instrument 1                    Location : Vial 12
    Injection Date  : 3/27/2019 11:03:35 PM              Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\GUAN YUQING\LJ-113-1\LJ-113-1(2) 2019-03-27 15-58-33\VWD-AD(1-2)-95
                      -5-1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\GUAN YUQING\LONGJIAO\DAD-OD(1-2)-99-1-1ML-5UL-ALL-60MIN.M
    Last changed    : 3/28/2019 9:07:09 AM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  28.404 MM    0.6931 3210.74707   77.20502 100.0000

    Totals :                  3210.74707   77.20502


    ======================================================================
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 11-01-33\041-0301.D
Sample Name: XZC-4MeO-rac

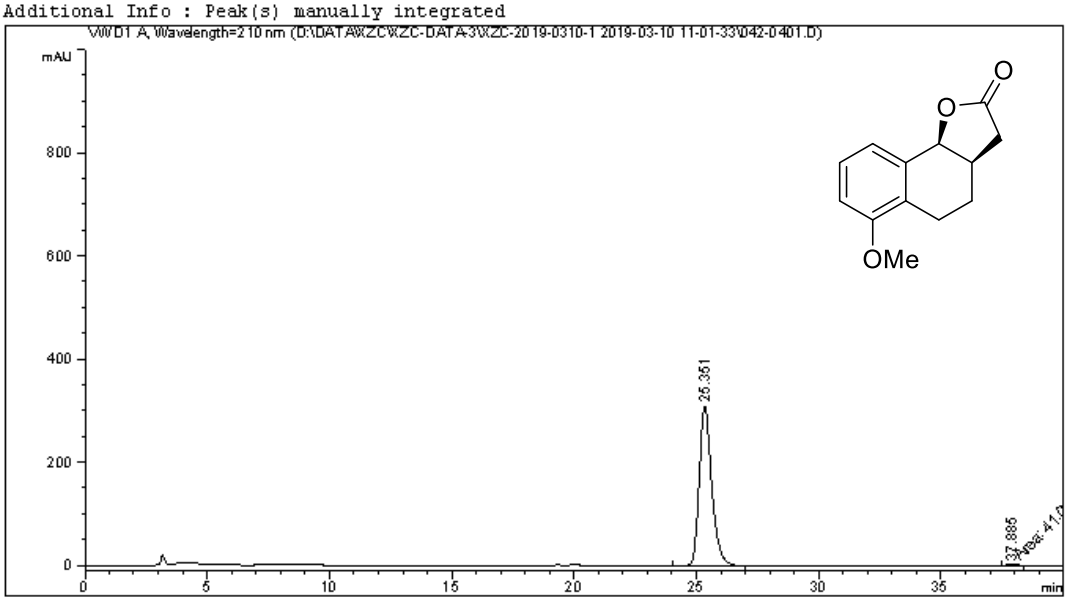
```
    ======================================================================
    Acq. Operator   :                              Seq. Line :   3
    Acq. Instrument : Instrument 1                  Location : Vial 41
    Injection Date  : 3/10/2019 11:25:39 AM              Inj :   1
                                                  Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 11-01-33\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\GUAN YUQING\LONGJIAO\VWD-OD(1-6)-99-1-0.5ML-5UL-254NM-60MIN.M
    Last changed    : 3/11/2019 3:05:02 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  25.453 MM    0.5668 3629.55347  106.73274  49.4276
       2  37.639 MM    0.8557 3713.62280   72.33047  50.5724

    Totals :                  7343.17627  179.06321
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 11-01-33\042-0401.D
Sample Name: XZC-4Me0

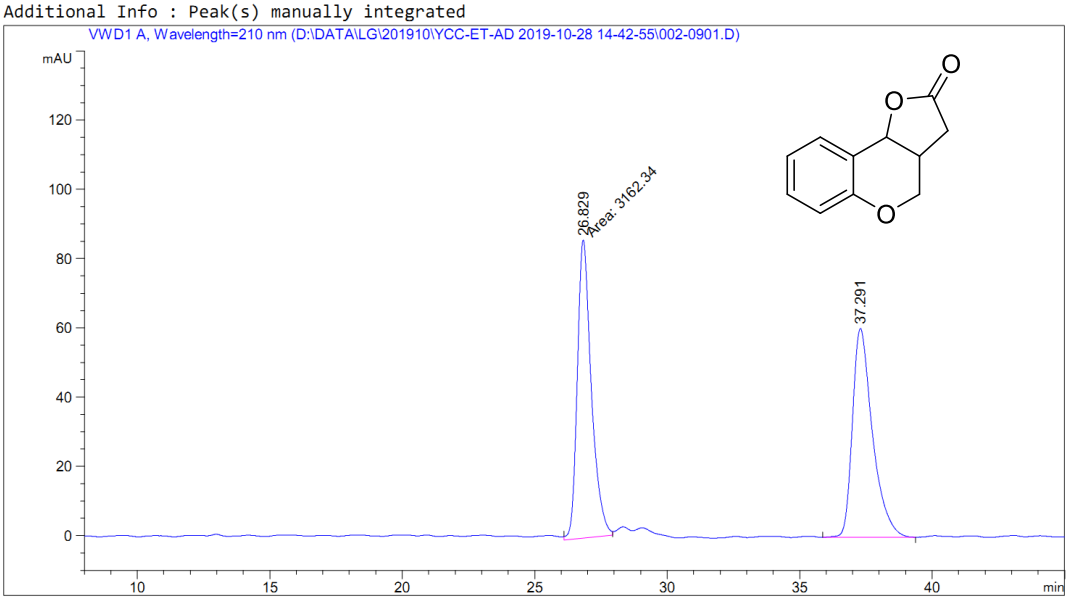
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   4
    Acq. Instrument : Instrument 1                    Location : Vial 42
    Injection Date  : 3/10/2019 12:06:31 PM              Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 11-01-33\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\GUAN YUQING\LONGJIAO\VWD-OD(1-6)-99-1-0.5ML-5UL-254NM-60MIN.M
    Last changed    : 3/11/2019 3:07:15 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  25.351 BB    0.5319 1.08740e4   308.75864  99.6236
       2  37.885 MM    0.6678   41.07934    1.02520   0.3764

    Totals :                  1.09150e4   309.78384
```

Data File D:\DATA\LG\201910\YCC-ET-AD 2019-10-28 14-42-55\002-0901.D
Sample Name: CYZ-RAC-2

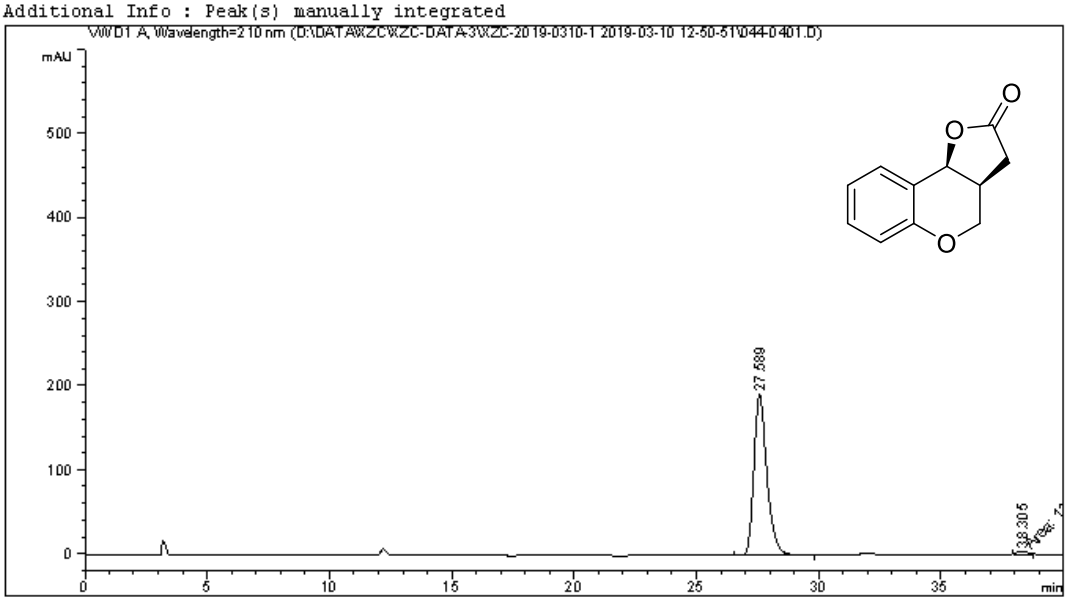
```
=====================================================================
Acq. Operator   :                              Seq. Line :   9
Acq. Instrument : Instrument 1                  Location : Vial 2
Injection Date  : 10/28/2019 7:14:30 PM              Inj :   1
                                               Inj Volume : 3.000 µl
Acq. Method     : D:\DATA\LG\201910\YCC-ET-AD 2019-10-28 14-42-55\VWD-AD(1-2)-95-5-1ML-3UL-
                  210NM-45MIN.M
Last changed    : 11/12/2018 8:14:27 PM
Analysis Method : D:\METHOD\TL\VWD-AS(1-2)-80-20-1ML-3UL-210NM-60MIN.M
Last changed    : 10/28/2019 2:22:21 PM
                  (modified after loading)
Additional Info : Peak(s) manually integrated
```




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

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


Signal 1: VWD1 A, Wavelength=210 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  26.829 MM    0.6125 3162.34351   86.04386  49.9184
   2  37.291 BB    0.7877 3172.67725   60.31116  50.0816

Totals :                  6335.02075  146.35502
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 12-50-51\044-0401.D
Sample Name: XZC-hex-0

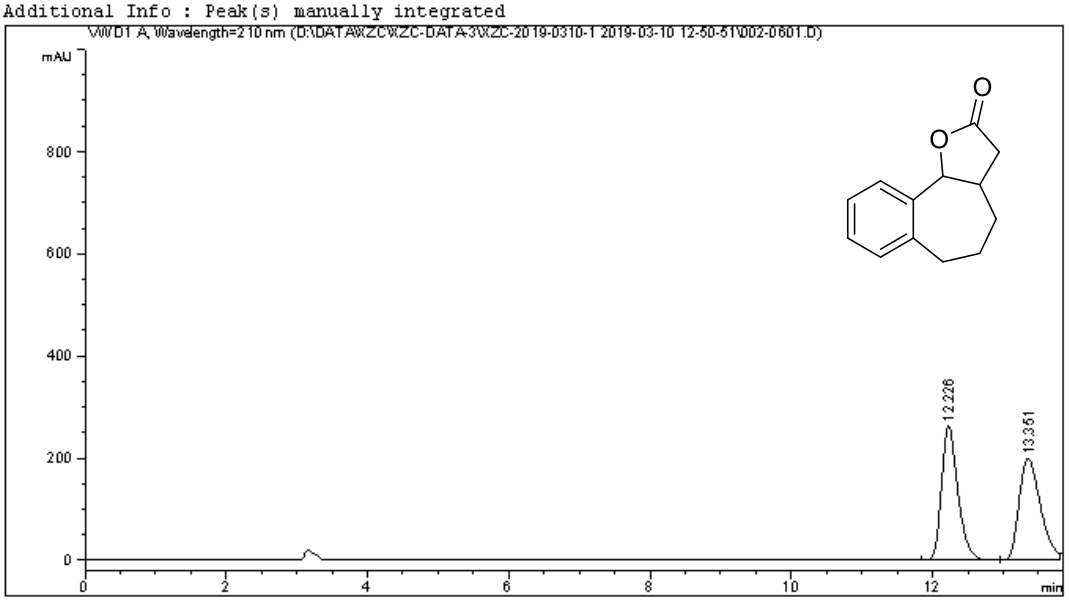
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   4
    Acq. Instrument : Instrument 1                    Location : Vial 44
    Injection Date  : 3/10/2019 1:46:21 PM                 Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 12-50-51\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\ZX\VWD-AD(1-2)-90-10-0.5ML-3UL-254NM-60MIN.M
    Last changed    : 3/13/2019 3:19:10 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  27.589 BB    0.5549 7052.86572  191.42136  98.2471
       2  38.305 MM    0.5638  125.83205    3.71980   1.7529

    Totals :                  7178.69777  195.14116
```

Data File D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 12-50-51\002-0601.D
Sample Name: XZC-hep-rac-1

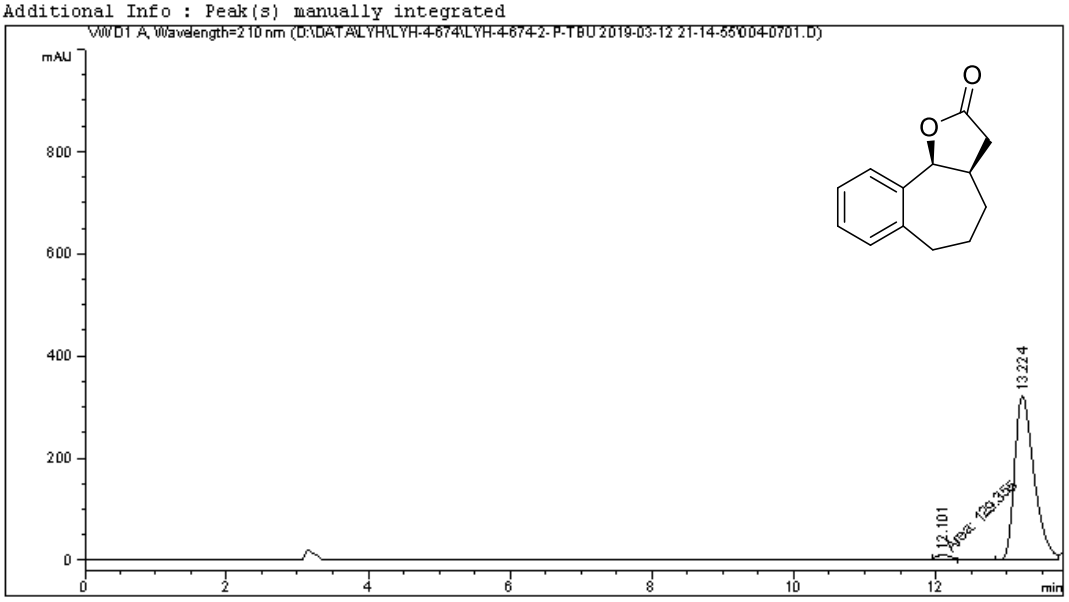
```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   6
    Acq. Instrument : Instrument 1                    Location : Vial 2
    Injection Date  : 3/10/2019 3:08:02 PM                 Inj :   1
                                                   Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\XZC\XZC-DATA-3\XZC-2019-0310-1 2019-03-10 12-50-51\VWD-AD(1-2)-95-5
                      -1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\GUAN YUQING\LONGJIAO\VWD-OD(1-6)-99-1-0.5ML-5UL-254NM-60MIN.M
    Last changed    : 3/11/2019 3:10:27 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]       %
    ----|-------|----|-------|----------|----------|--------|
       1  12.226 BB    0.2426 4202.47021  262.70895  50.1776
       2  13.351 BV    0.3258 4172.71436  198.83009  49.8224

    Totals :                  8375.18457  461.53905
```

Data File D:\DATA\LYH\LYH-4-674\LYH-4-674-2-P-TBU 2019-03-12 21-14-55\004-0701.D
Sample Name: XZC-hep

```
    ======================================================================
    Acq. Operator   :                                Seq. Line :   7
    Acq. Instrument : Instrument 1                    Location : Vial 4
    Injection Date  : 3/13/2019 12:40:44 AM              Inj :   1
                                                    Inj Volume : 5.000 µl
    Acq. Method     : D:\DATA\LYH\LYH-4-674\LYH-4-674-2-P-TBU 2019-03-12 21-14-55\VWD-AD(1-2)-95-
                      5-1ML-5UL-210NM-40MIN.M
    Last changed    : 7/10/2018 8:21:32 PM
    Analysis Method : D:\METHOD\ZX\VWD-AD(1-2)-90-10-0.5ML-3UL-254NM-60MIN.M
    Last changed    : 3/13/2019 3:10:10 PM
                      (modified after loading)
    Additional Info : Peak(s) manually integrated
```




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

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


    Signal 1: VWD1 A, Wavelength=210 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]   [mAU*s]     [mAU]        %
    ----|-------|----|-------|----------|----------|--------|
       1  12.101 MM    0.2222  129.35524    9.70152   2.0625
       2  13.224 BV    0.2909 6142.51025  321.56558  97.9375

    Totals :                  6271.86549  331.26710
```