Supporting Information

Organocatalytic [4 + 2] cyclocondensation of α,β-unsaturated acyl chlorides with imines: Highly enantioselective synthesis of dihydropyridinone and piperidine derivatives

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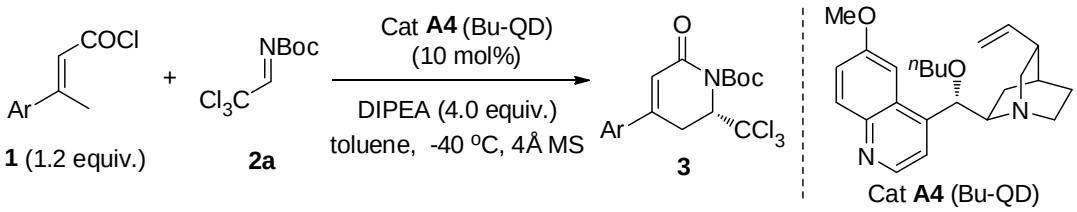
General Information

Unless otherwise indicated, all reactions were carried out under anhydrous, air-free conditions. All solvents were dried and distilled by standard procedures. α, β-Unsaturated acyl chlorides[1] and aldimines[2] was prepared according to the literatures.

Column chromatography was performed with silica gel 200 ~ 300 mesh. All ^{1}H NMR (300 MHz), ^{13}C NMR (75 MHz) spectra were recorded on a Bruker-DMX 300 spectrometer in $CDCl_3$, with tetramethylsilane as an internal standard and reported in ppm (δ). Infrared (IR) spectra were recorded on a Nicolet 6700 spectrophotometer and reported as wavenumber (cm^{-1}). Optical rotations were measured on AA-10R/Optical activity LTD operating at the sodium D line with a 100 mm path length cell, and reported as follows: $[\alpha]_D^T$ (concentration (g/100ml), solvent).

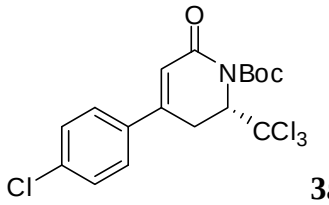
Part I Experimental part

1. Reaction with chloral-derived imine 2a (Table 2)



General Procedure A. To an oven-dried 25 mL Schlenk tube equipped with a stir bar was charged with *n*-Bu-quinidine (cat **A4**) hydrochloride salt (9.1 mg, 0.02 mmol) and 4Å MS (150 mg). This tube was closed with a septum, evacuated, and back-filled with argon. To this mixture was added freshly distilled toluene (1.0 mL) and DIPEA (0.138 mL, 0.8 mmol), then aldimines **2a** (49.3 mg, 0.2 mmol) in toluene (0.5 mL) was successively added. The mixture was cooled to -40 °C, and the solution

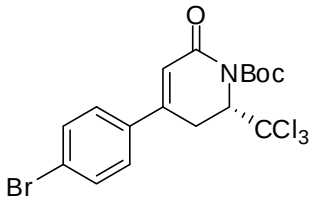
of acyl chloride **1** (0.24 mmol) in toluene (0.5 mL) was added. The reaction was stirred at -40 ^{o}C until the full consumption of **2a**. The mixture was diluted with ethyl acetate and passed through a short pad of silica gel. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (petroleum ether/ethyl acetate, 15:1) to give the desired annulation product.



3a

(*S*)-*tert*-butyl-4-(4-chlorophenyl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3a)

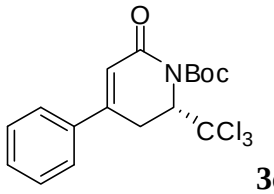
Yield: 69.3 mg (82%), yellow solid, mp: 104-106 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -40 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 12.6 min (major), - (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 9.0, 9.0 Hz, 4H), 6.27 (s, 1H), 5.59 (d, *J* = 8.0 Hz, 1H), 3.45 (d, *J* = 19.3 Hz, 1H), 3.20 (dd, *J* = 19.2, 7.9 Hz, 1H), 1.51 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.1, 152.2, 147.9, 136.8, 134.9, 129.5, 127.5, 120.9, 102.4, 84.6, 65.3, 28.1, 27.6. IR (KBr) ν 2980, 1721, 1406, 1150, 913, 787. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{17}H_{17}NO_3Cl_4Na$, 445.98548, Found 445.98489.



3b

(*S*)-*tert*-butyl-4-(4-bromophenyl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3b)

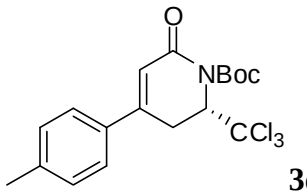
Yield: 77.5 mg (82%), orange solid, mp: 123-125 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -38 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 13.7 min (major), - (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.58 (d, *J* = 8.6 Hz, 2H), 7.39 (d, *J* = 8.6 Hz, 2H), 6.34 (d, *J* = 2.5 Hz, 1H), 5.66 (d, *J* = 7.5 Hz, 1H), 3.51 (d, *J* = 19.3 Hz, 1H), 3.27 (ddd, *J* = 19.3, 8.0, 2.6 Hz, 1H), 1.58 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.0, 152.2, 148.0, 135.3, 132.4, 127.7, 125.1, 120.9, 102.4, 84.6, 65.3, 28.1, 27.5. IR (KBr) ν 2979, 1721, 1489, 1293, 1150, 1008, 812. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{17}H_{17}BrNO_3Cl_3Na$, 489.93496, Found 489.93489.



3c

(*S*)-*tert*-butyl-2-oxo-4-phenyl-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3c)

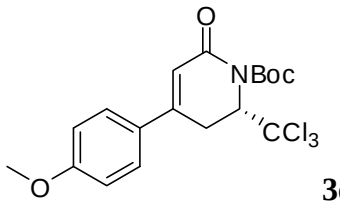
Yield: 74.1 mg (95%), yellow solid, mp: 150 - 152 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -46.7 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 9.5 min (major), - (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46 - 7.44 (m, 2H), 7.38 -7.35 (m, 3H), 6.27 (d, *J* = 2.4 Hz, 1H), 5.59 (d, *J* = 7.9 Hz, 1H), 3.49 (d, *J* = 19.4 Hz, 1H), 3.21 (ddd, *J* = 19.4, 8.0, 2.5 Hz, 1H), 1.50 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.3, 152.2, 149.2, 136.4, 130.5, 129.1, 126.2, 120.5, 102.4, 84.4, 65.4, 28.1, 27.6. IR (KBr) ν 2979, 1698, 1294, 1150, 763. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{17}H_{18}NO_3Cl_3Na$, 412.02445, Found 412.02441.



3d

(*S*)-*tert*-butyl-2-oxo-4-p-tolyl-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3d)

Yield: 53.2 mg (66%), yellow solid, mp: 132-134 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -45 (*c* 1.2, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 9.9 min (major), - (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, *J* = 7.8 Hz, 2H), 7.17 (d, *J* = 7.7 Hz, 2H), 6.26 (s, 1H), 5.58 (d, *J* = 8.0 Hz, 1H), 3.48 (d, *J* = 19.3 Hz, 1H), 3.19 (dd, *J* = 19.3, 8.0 Hz, 1H), 2.31 (s, 3H), 1.50 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.4, 152.2, 149.1, 141.1, 133.5, 129.8, 126.1, 119.6, 102.5, 84.3, 65.4, 28.1, 27.5, 21.4. IR (KBr) ν 2979, 1720, 1368, 1249, 1151, 813, 752. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{18}H_{20}NO_3Cl_3Na$, 426.04010, Found 426.03995.

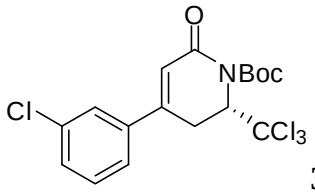


3e

(*S*)-*tert*-butyl-(4-methoxyphenyl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3e)

Yield: 73.1 mg (87%), orange solid, mp: 106-118 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -43 (*c* 1.3, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 9.9 min (major), - (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.42 (d, *J* = 8.7 Hz, 2H), 6.88 (d, *J* = 8.7 Hz, 2H), 6.22 (d, *J* = 2.1 Hz, 1H), 5.57 (d, *J* = 7.9 Hz, 1H), 3.77 (s, 3H), 3.48 (d, *J* = 19.3 Hz, 1H), 3.17 (ddd, *J* = 19.2, 8.0, 2.2 Hz, 1H), 1.50 (s, 9H). ^{13}C

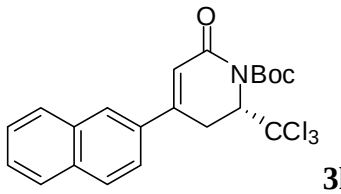
NMR (75 MHz, $CDCl_3$) δ 162.5, 161.7, 152.2, 148.6, 128.5, 127.7, 118.5, 114.5, 102.5, 84.2, 65.4, 55.5, 28.1, 27.4. IR (KBr) ν 2980, 1723, 1369, 1224, 848, 741. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{18}H_{20}NO_4Cl_3Na$, 442.03501, Found 442.03485.



3f

***(S)-tert*-butyl-4-(3-chlorophenyl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3f)**

Yield: 70.2 mg (83%), yellow solid, mp: 104-106 °C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -42 (*c* 1.0, CH_2Cl_2), HPLC analysis: 95% ee [Daicel CHIRALPAK AD-H column, 20 °C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 8.0 min (major), 9.5 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.20 (m, 4H), 6.26 (s, 1H), 5.59 (d, *J* = 7.9 Hz, 1H), 3.43 (d, *J* = 19.4 Hz, 1H), 3.21 (ddd, *J* = 19.4, 7.9, 2.5 Hz, 1H), 1.51 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.9, 152.1, 147.7, 138.3, 135.3, 130.5, 130.4, 126.3, 124.3, 121.5, 102.3, 84.6, 65.3, 28.1, 27.6. IR (KBr) ν 2980, 1721, 1251, 1094, 787. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{17}H_{17}NO_3Cl_4Na$, 445.98548, Found 445.98540.

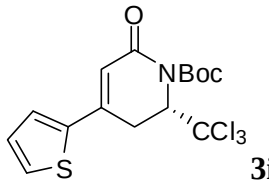


3h

***(S)-tert*-butyl-4-(naphthalen-2-yl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3h)**

Yield: 76.6 mg (87%), yellow solid, mp: 164-166 °C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -42 (*c* 1.2, CH_2Cl_2), HPLC analysis: >99% ee [Daicel

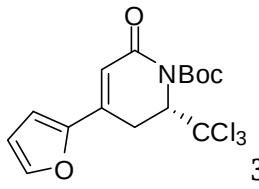
CHIRALPAK IA-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.2 mL /min, 9.5 min (major), 10.8 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.90 (s, 1H), 7.82-7.76 (m, 3H), 7.55 (d, *J* = 8.6 Hz, 1H), 7.48-7.45 (m, 2H), 6.42 (d, *J* = 2.2 Hz, 1H), 5.64 (d, *J* = 7.9 Hz, 1H), 3.63 (d, *J* = 19.2 Hz, 1H), 3.31 (ddd, *J* = 19.2, 7.8, 2.4 Hz, 1H), 1.52 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.3, 152.3, 148.9, 134.2, 133.6, 133.1, 129.0, 128.8, 127.8, 127.6, 127.1, 126.2, 123.1, 120.8, 102.5, 84.5, 65.5, 28.1, 27.6. IR (KBr) ν 3480, 1700, 1307, 879, 746, 472. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{21}H_{20}NO_3Cl_3Na$, 462.04010, Found 462.04006.



3i

***(S)-tert*-butyl-2-oxo-4-(thiophen-2-yl)-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (3i)**

Yield: 71.3 mg (90%), yellow solid, mp: 114-116 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -27 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 11.5 min (major), 13.1 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.39 (d, *J* = 4.4 Hz, 1H), 7.30 (d, *J* = 2.8 Hz, 1H), 7.05-7.03 (m, 1H), 6.25 (d, *J* = 2.1 Hz, 1H), 5.57 (d, *J* = 7.8 Hz, 1H), 3.48 (d, *J* = 19.2 Hz, 1H), 3.21 (ddd, *J* = 19.2, 8.1, 2.4 Hz 1H), 1.49 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.1, 152.0, 142.5, 140.1, 129.2, 128.5, 127.6, 117.8, 102.2, 84.3, 65.1, 28.0, 27.6. IR (KBr) ν 2979, 1720, 1622, 1294, 1224, 1150, 851, 776, 714. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{15}H_{16}NO_3Cl_3NaS$, 417.98087, Found 417.98072.

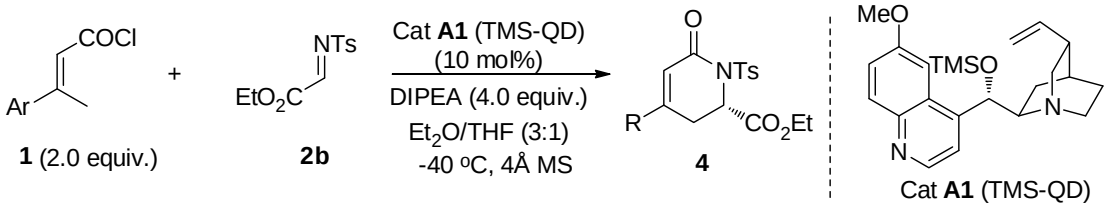


3j

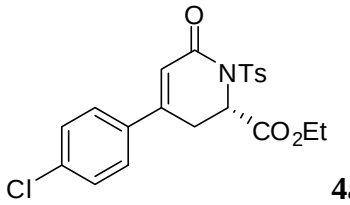
***(S)-tert*-butyl-4-(furan-2-yl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H) -carboxylate (3j)**

Yield: 56.4 mg (74%), red oil. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -16.4 (*c* 0.42, CH_2Cl_2), HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.5 mL /min, 5.0 min (minor), 6.2 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48 (s, 1H), 6.70 (d, *J* = 2.9 Hz, 1H), 6.46 (d, *J* = 1.6 Hz, 1H), 6.31 (s, 1H), 5.57 (d, *J* = 7.4 Hz, 1H), 3.37 (d, *J* = 19.1 Hz, 1H), 3.11 (dd, *J* = 19.1, 8.1 Hz, 1H), 1.49 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 162.3, 152.2, 150.3, 145.6, 137.5, 116.3, 112.7, 112.5, 102.2, 84.3, 65.1, 28.0, 25.2. IR (KBr) ν 3444, 2982, 1724, 1369, 1292, 1148, 813, 751. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{15}H_{16}NO_4Cl_3Na$, 402.00371, Found 402.00363.

2. Reaction with iminoester 2b (Table 3)



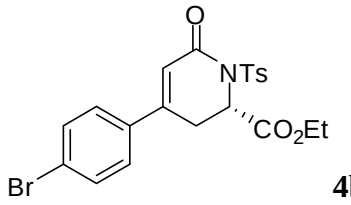
General Procedure B. The same as general procedure A, except using Cat **A1** (*O*-trimethylsilyl quinidine) as the catalyst and ether/THF (3:1) as the solvent.



4a

(*S*)-ethyl-4-(4-chlorophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxyl ate (4a)

Yield: 65.8 mg (76%), white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 169.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 97% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane /*i*-PrOH = 60:40, 0.7 mL /min, 19.8 min (minor), 26.1 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.2 Hz, 2H), 7.39 (s, 4H), 7.33 (d, *J* = 8.1 Hz, 2H), 6.14 (d, *J* = 2.3 Hz, 1H), 5.53 (d, *J* = 4.7 Hz, 1H), 4.12 (dd, *J* = 14.2, 7.0 Hz, 2H), 3.45 (d, *J* = 16.8 Hz, 1H), 3.19 (dd, *J* = 17.8, 3.7 Hz, 1H), 2.44 (s, 3H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.7, 162.3, 150.6, 145.1, 136.9, 135.7, 134.5, 129.8, 129.4, 129.1, 127.6, 119.7, 62.6, 56.3, 30.7, 21.8, 14.0. IR (KBr) ν 2980, 1743, 1493, 1205, 1088, 887, 709, 655. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{21}H_{21}O_5NClS$, 434.08235, Found 434.08203.

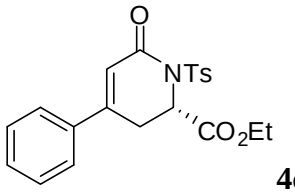


4b

(*S*)-ethyl-4-(4-bromophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxyl ate (4b)

Yield: 71.3 mg (75%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 126.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 96% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 70:30, 0.7 mL /min, 20.3 min (minor), 34.4 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.94 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 7.24 (d, *J* = 8.3 Hz, 4H), 6.06 (d, *J* = 2.3 Hz, 1H), 5.45 (m,

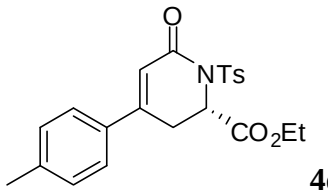
1H), 4.03 (m, 2H), 3.36 (dd, *J* = 17.6, 1.6 Hz, 1H), 3.11 (ddd, *J* = 17.7, 6.1, 2.4 Hz, 1H), 2.31 (d, *J* = 24.6 Hz, 3H), 1.01 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.6, 162.2, 150.7, 145.0, 135.6, 135.0, 132.3, 129.8, 129.1, 127.8, 125.2, 119.7, 62.5, 56.3, 30.7, 21.7, 14.0. IR (KBr) ν 2979, 1742, 1585, 1402, 1205, 1076, 886, 710, 654. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{21}H_{21}O_5NBrS$, 478.03183, Found 478.03171.



4c

(*S*)-ethyl-6-oxo-4-phenyl-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4c)

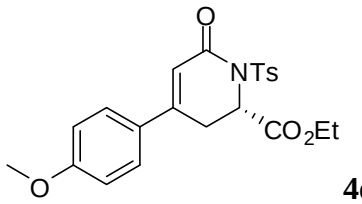
Yield: 53.0 mg (66%), white solid, mp: 108-110 °C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 167.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 95% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 70:30, 0.7 mL /min, 19.7 min (minor), 23.9 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.35 (m, 5H), 7.23 (d, *J* = 8.3 Hz, 2H), 6.07 (d, *J* = 2.4 Hz, 1H), 5.45 (dd, *J* = 6.2, 1.9 Hz, 1H), 4.22-3.83 (m, 2H), 3.41 (dd, *J* = 17.7, 1.5 Hz, 1H), 3.11 (ddd, *J* = 17.7, 6.2, 2.4 Hz, 1H), 2.33 (s, 3H), 1.01 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.7, 162.4, 151.9, 144.9, 136.1, 135.8, 130.7, 129.7, 129.0, 126.3, 119.4, 62.4, 56.3, 30.8, 21.7, 14.0. IR (KBr) ν 2981, 1744, 1596, 1354, 1167, 1015, 814, 706, 555. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{21}H_{22}O_5NS$, 400.12187, Found 400.12158.



4d

(*S*)-ethyl-6-oxo-4-p-tolyl-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4d)

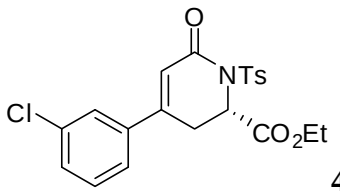
Yield: 53.0 mg (64%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 154.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 94% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm hexane/*i*-PrOH = 60:40, 0.7 mL /min, 13.8 min (minor), 16.0 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.28 (d, *J* = 8.2 Hz, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.12 (d, *J* = 8.1 Hz, 2H), 6.05 (d, *J* = 2.4 Hz, 1H), 5.44 (dd, *J* = 6.2, 1.8 Hz, 1H), 4.11-3.90 (m, 2H), 3.41 (dd, *J* = 17.7, 1.8 Hz, 1H), 3.08 (ddd, *J* = 17.7, 6.2, 2.5 Hz, 1H), 2.34 (s, 3H), 2.28 (s, 3H), 1.00 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.8, 162.6, 151.8, 144.9, 141.2, 135.8, 133.1, 129.7, 129.0, 126.2, 118.4, 62.4, 56.3, 30.7, 21.7, 21.4, 14.0. IR (KBr) ν 2979, 1744, 1607, 1204, 1025, 816, 713, 659, 544. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{22}H_{24}O_5NS$, 414.13697, Found 414.13692.



4e

(*S*)-ethyl-4-(4-methoxyphenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4e)

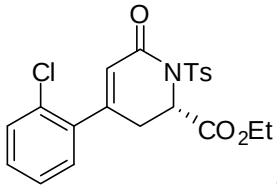
Yield: 48.9 mg (57%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 136.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 94% ee [Daicel CHIRALPAK OD-H column, 20 ° C, 254 nm hexane /*i*-PrOH = 60:40, 0.7 mL /min, 20.1 min (minor), 24.3 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (d, J = 8.4 Hz, 2H), 7.44 (d, J = 8.9 Hz, 2H), 7.32 (d, J = 8.3 Hz, 2H), 6.92 (d, J = 8.9 Hz, 2H), 6.10 (d, J = 2.4 Hz, 1H), 5.51 (dd, J = 6.2, 1.9 Hz, 1H), 4.19-3.99 (m, 2H), 3.83 (s, 3H), 3.50 (dd, J = 17.6, 2.0 Hz, 1H), 3.14 (ddd, J = 17.6, 6.2, 2.5 Hz, 1H), 2.43 (s, 3H), 1.09 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 162.7, 161.8, 151.3, 144.9, 135.9, 129.8, 129.0, 128.2, 128.0, 117.2, 114.5, 62.4, 56.3, 55.5, 30.6, 21.8, 14.0. IR (KBr) ν 2977, 1743, 1514, 1353, 1166, 1030, 832, 658. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{22}H_{24}O_6NS$, 430.13188, Found 430.13189.



4f

(*S*)-ethyl-4-(3-chlorophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4f)

Yield: 61.3 mg (71%), yellow solid, mp: 123-125 ^{o} C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 104.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 96% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/*i*-PrOH = 80:20, 0.8 mL /min, 27.7 min (minor), 33.4 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.3 Hz, 2H), 7.30 (m, 6H), 6.06 (d, *J* = 2.5 Hz, 1H), 5.45 (dd, *J* = 6.1, 1.9 Hz, 1H), 4.04 (m, 2H), 3.35 (dd, *J* = 17.7, 1.8 Hz, 1H), 3.11 (ddd, *J* = 17.7, 6.1, 2.6 Hz, 1H), 2.35 (s, 3H), 1.03 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.6, 162.1, 150.5, 145.1, 138.0, 135.6, 135.2, 130.6, 130.4, 129.8, 129.1, 126.4, 124.5, 120.5, 62.6, 56.3, 30.8, 21.8, 14.0. IR (KBr) ν 2962, 1743, 1355, 1028, 706, 557. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{21}H_{21}O_5NClS$, 434.08235, Found 434.08200.

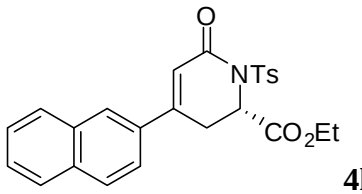


4g

(*S*)-ethyl-4-(2-chlorophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4g)

Yield: 26.6 mg (31%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 56.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 89% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 80:20, 0.5 mL /min, 30.9 min (major), 34.2 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.03 (d, *J* = 8.4 Hz, 1H 2H), 7.38 - 7.26 (m, 5H), 7.13 - 7.11 (m, 1H), 5.93 (d, *J* = 2.2 Hz, 1H), 5.47 (dd, *J* = 8.0 Hz, 2.6 Hz, 1H), 4.24 - 4.18 (m, 1H), 4.10 -4.04 (m, 1H), 3.35-3.29 (m, 2H), 2.44 (s, 3H),

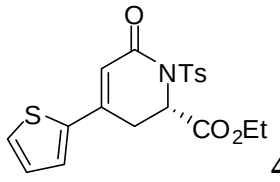
1.17 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.7, 162.0, 151.8, 145.1, 137.0, 135.6, 131.7, 130.6, 130.4, 129.9, 129.3, 129.1, 127.4, 124.3, 62.5, 56.5, 32.8, 21.8, 14.1. IR (KBr) ν 2982, 1745, 1596, 1311, 1088, 1101, 814, 704, 560. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: for: $C_{21}H_{21}O_5NClS$, 434.08235, Found 434.08217.



4h

(*S*)-ethyl-4-(naphthalen-2-yl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4h)

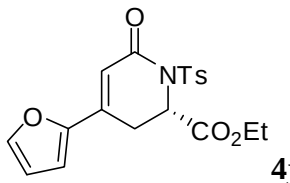
Yield: 76.7 mg (85%), yellow solid, mp: 121-123 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 183.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 96% ee [Daicel CHIRALPAK AS-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 70:30, 1.0 mL /min, 22.2 min (minor), 32.2 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.05 (d, J = 8.4 Hz, 2H), 7.93 (s, 1H), 7.89-7.82 (m, 3H), 7.57-7.54 (m, 3H), 7.34 (d, J = 8.2 Hz, 2H), 6.31 (d, J = 2.5 Hz, 1H), 5.58 (dd, J = 6.2, 1.9 Hz, 1H), 4.17-4.05 (m, 2H), 3.66 (dd, J = 17.6, 1.9 Hz, 1H), 3.30 (ddd, J = 17.6, 6.3, 2.5 Hz, 1H), 2.44 (s, 3H), 1.10 (t, J = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.9, 162.6, 151.6, 145.0, 135.9, 134.3, 133.3, 133.1, 129.9, 129.1, 129.0, 128.9, 127.8, 127.8, 127.1, 126.7, 123.1, 119.7, 62.6, 56.5, 30.9, 21.8, 14.1. IR (KBr) ν 2923, 1744, 1596, 1308, 1087, 1015, 815, 706, 554. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{25}H_{24}O_5NS$, 450.13697, Found 450.13672.



4i

(*S*)-ethyl-6-oxo-4-(thiophen-2-yl)-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4i)

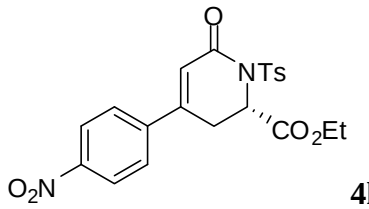
Yield: 43.7 mg (54%), white solid, mp: 143-145 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 76.0 (*c* 0.25, CH_2Cl_2), HPLC analysis: 92% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 70:30, 0.7 mL /min, 20.1 min (minor), 23.1 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.3 Hz, 2H), 7.46 (d, *J* = 5.0 Hz, 1H), 7.33 (t, *J* = 5.3 Hz, 3H), 7.09 (m, 1H), 6.12 (d, *J* = 2.3 Hz, 1H), 5.51 (dd, *J* = 6.2, 1.9 Hz, 1H), 4.10 (m, 2H), 3.49 (dd, *J* = 17.4, 1.9 Hz, 1H), 3.20 (ddd, *J* = 17.5, 6.2, 2.4 Hz, 1H), 2.43 (s, 3H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.6, 162.4, 145.0, 144.9, 140.0, 135.8, 129.9, 129.8, 129.1, 128.6, 128.3, 116.5, 62.6, 56.2, 31.1, 21.8, 14.0. IR (KBr) ν 2924, 1744, 1598, 1259, 1166, 1019, 801, 663. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{19}H_{20}O_5NS_2$, 406.07774, Found 406.07772.



4j

(*S*)-ethyl-4-(furan-2-yl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4j)

Yield: 46.6 mg (60%), red oil. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 149.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 82% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 70:30, 0.7 mL /min, 20.2 min (minor), 24.7 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.3 Hz, 2H), 7.53 (d, *J* = 1.1 Hz, 1H), 7.32 (d, *J* = 8.3 Hz, 2H), 6.73 (t, *J* = 5.6 Hz, 1H), 6.50 (dd, *J* = 3.4, 1.7 Hz, 1H), 6.19 (d, *J* = 2.3 Hz, 1H), 5.50 (dd, *J* = 6.2, 1.8 Hz, 1H), 4.15-4.02 (m, 2H), 3.35 (dd, *J* = 17.4, 1.9 Hz, 1H), 3.11 (ddd, *J* = 17.4, 6.3, 2.4 Hz, 1H), 2.41 (s, 3H), 1.09 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.6, 162.6, 150.1, 145.9, 144.9, 139.4, 135.9, 129.8, 129.1, 114.9, 113.6, 112.7, 62.5, 56.1, 28.7, 21.8, 14.0. IR (KBr) ν 2980, 1743, 1620, 1405, 1289, 1039, 922, 755, 661, 555. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{19}H_{20}O_6NS$, 390.10058, Found 390.10032.

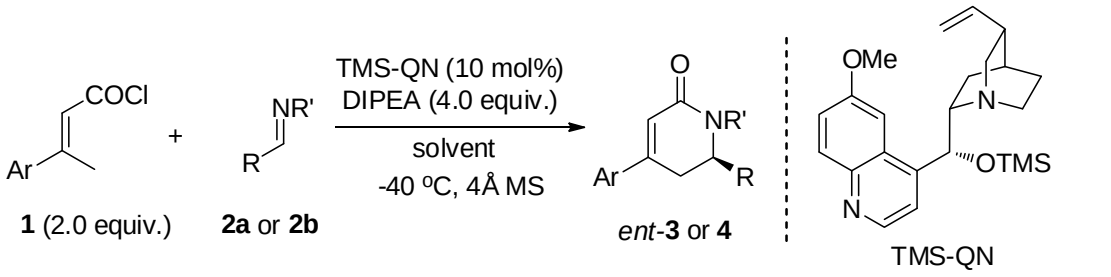


4k

(*S*)-ethyl-4-(4-nitrophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (4k)

Yield: 77.2 mg (87%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 96.2 (*c* 4.5, CH_2Cl_2), HPLC analysis: ee was not determined because of the failure of its separation on CHIRALPAK OD, AD, OJ, OB, AS, IA columns. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.17 (d, *J* = 8.8 Hz, 2H), 7.94 (d, *J* = 8.3 Hz, 2H), 7.54 (d, *J* = 8.8 Hz, 2H), 7.26 (d, *J* = 8.2 Hz, 2H), 6.16 (d, *J* = 2.5 Hz, 1H), 5.49 (dd, *J* = 6.0, 1.9 Hz, 1H), 4.04 (m, 2H), 3.39 (dd, *J* = 17.7, 1.9 Hz, 1H), 3.20 (ddd, *J* = 17.7, 6.1, 2.6 Hz, 1H), 2.33 (d, *J* = 17.2 Hz, 3H), 1.03 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.5, 161.7, 149.5, 148.8, 145.3, 142.3, 135.4, 129.8, 129.1, 127.3, 124.2, 122.4, 62.7, 56.2, 30.8, 21.8, 14.0. IR (KBr) ν 2981, 1689, 1521, 1206, 1087, 851, 556. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{21}H_{20}O_7N_2NaS$, 467.08834, Found 467.08762.

3. Reaction catalyzed by TMS-quinine (Table 4)

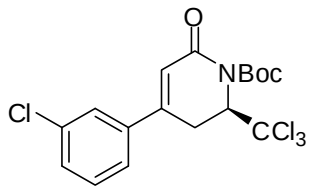


1 (2.0 equiv.) **2a** or **2b** *ent*-**3** or **4**

solvent = toluene (for the reation with **2a**)
= Et_2O/THF (3:1) (for the reaction with **2b**)

General Procedure: The same as procedure **A** for the reaction with imine **2a** or procedure **B** for the reaction with **2b** except using *O*-trimethylsilyl quinine as the

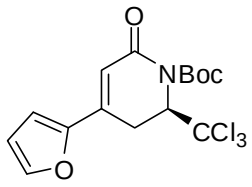
catalyst.



ent-**3f**

***(R)-tert*-butyl-4-(3-chlorophenyl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (*ent*-3f)**

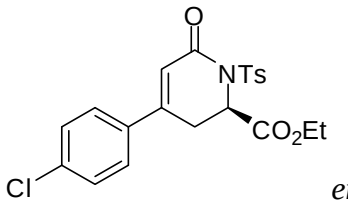
Yield: 55.2 mg (65%), yellow solid, mp: 102-104 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ 42 (*c* 1.0, CH_2Cl_2), HPLC analysis: 99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, 8.0 min (minor), 9.5 min (major)].



ent-**3j**

***(R)-tert*-butyl-4-(furan-2-yl)-2-oxo-6-(trichloromethyl)-5,6-dihydropyridine-1(2H)-carboxylate (*ent*-3j)**

Yield: 54.0 mg (71%), red oil. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ 21.6 (*c* 0.37, CH_2Cl_2), HPLC analysis: 98% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.5 mL /min, 5.0 min (major), 6.3 min (minor)].

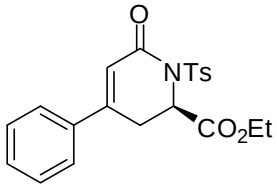


ent-**4a**

(*R*)-ethyl-4-(4-chlorophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxyl

ate (*ent*-4a)

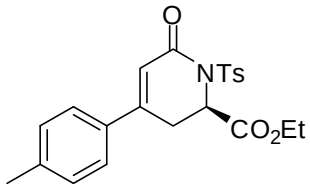
Yield: 46.2 mg (52%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -90.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 94% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane /*i*-PrOH = 60:40, 0.7 mL /min, 18.6 min (major), 25.7 min (minor)].



ent-**4c**

(*R*)-ethyl 6-oxo-4-phenyl-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (*ent*-**4c**)

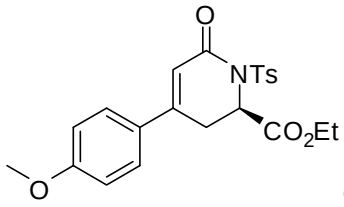
Yield: 38.2 mg (48%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -142 (*c* 1.0, CH_2Cl_2), HPLC analysis: 94% ee [Daicel CHIRALPAK OD-H column, 20 ^{o}C, 254 nm hexane/*i*-PrOH = 70:30, 0.7 mL /min, 17.4 min (major), 21.9 min (minor)].



ent-**4d**

(*R*)-ethyl 6-oxo-4-p-tolyl-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (*ent*-**4d**)

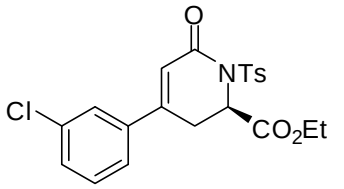
Yield: 41.5 mg (50%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -141.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 93% ee [Daicel CHIRALPAK OD-H column, 20 ^{o}C, 254 nm hexane/*i*-PrOH = 60:40, 0.7 mL /min, 13.4 min (major), 16.2 min (minor)].



ent-**4e**

(*R*)-ethyl-4-(4-methoxyphenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (*ent*-4e)

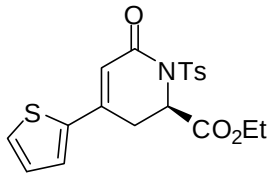
Yield: 37.7 mg (44%), yellow solid, mp: 120-122 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -162.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 92% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane /*i*-PrOH = 60:40, 0.7 mL /min, 19.6 min (major), 25.2min (minor)].



ent-**4f**

(*R*)-ethyl-4-(3-chlorophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (*ent*-4f)

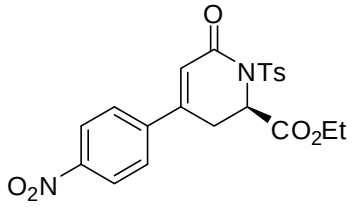
Yield: 57.0 mg (67%), yellow solid, mp: 130-132 ^{o}C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -136.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 96% ee [Daicel CHIRALPAK OD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 80:20, 0.8 mL /min, 26.3 min (major), 33.2min (minor)].



ent-**4i**

(*R*)-ethyl-6-oxo-4-(thiophen-2-yl)-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (*ent*-4i)

Yield: 38.2 mg (47%), white solid, mp: 131-133 °C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -107.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 84% ee [Daicel CHIRALPAK OD-H column, 20 °C, 254 nm hexane/i-PrOH = 70:30, 0.7 mL /min, 19.7 min (major), 23.4 min (minor)].



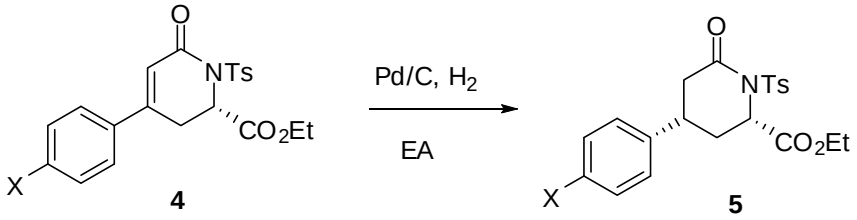
ent-**4k**

(*R*)-ethyl-4-(4-nitrophenyl)-6-oxo-1-tosyl-1,2,3,6-tetrahydropyridine-2-carboxylate (*ent*-4k)

Yield: 53.8 mg (61%), yellow waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -55.9 (*c* 4.3, CH_2Cl_2), HPLC analysis: ee was not determined because of the failure of its separation on CHIRALPAK OD, AD, OJ, OB, AS, IA columns.

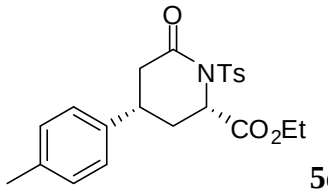
4. Chemical transformations of the dihydropyridinones (Scheme 4)

(1) Synthesis of tetrahydropyridinones (Sheme 4a)



To the solution of **4d** (25.4 mg, 0.061 mmol) or **4e** (17.7 mg, 0.041 mmol) or **4k** (41.3 mg, 0.099 mmol) in EtOAc (3 mL) was added 10% Pd/C (5.0 mg) at room temperature, then the mixture was stirred at room temperature under 1 atm of hydrogen. The reaction mixture was diluted with ethyl acetate, and passed through a short silica pad. The solvent was removed under reduced pressure to give

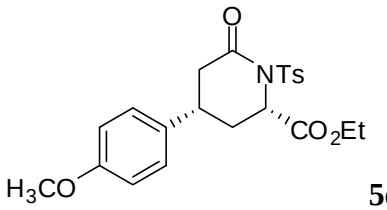
teterhydropyridinones **5.**



5d

(*2S,4S*)-ethyl 4-(4-methoxyphenyl)-6-oxo-1-tosylpiperidine-2-carboxylate (5d)

Yield: 24.3 mg (98%), dr = 10:1, white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -10.7 (*c* 1.22, $CHCl_3$), HPLC analysis: 93% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 70:30, 1.0 mL /min, 15.4 min (major), 18.4 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.9 Hz, 2H), 7.32 (d, *J* = 8.3 Hz, 2H), 7.13 (d, *J* = 8.0 Hz, 2H), 7.02 (d, *J* = 8.1 Hz, 2H), 5.04-4.99 (m, 1H), 4.32-4.15 (m, 2H), 3.09 (m,3.14 - 3.03, 1H), 2.70-2.61 (m, 2H), 2.57-2.52 (m, 1H), 2.43 (s, 3H), 2.31 (s, 3H), 2.15-2.04 (m, 1H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 170.0, 145.1, 138.4, 137.3, 136.0, 129.8, 129.7, 129.2, 126.5, 62.2, 58.4, 41.3, 36.3, 34.5, 21.8, 21.1, 14.1. IR (KBr) ν 2923, 2854, 1744, 1698, 1353, 1190, 1168, 1086, 814, 662, 565, 543. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{22}H_{25}NO_5NaS$, 438.13456, Found 438.13426.

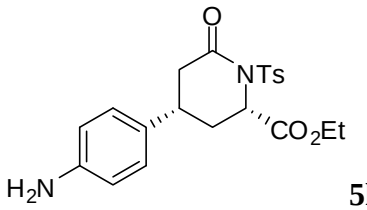


5e

(*2S,4S*)-ethyl 6-oxo-4-p-tolyl-1-tosylpiperidine-2-carboxylate (5e)

Yield: 17.0 mg (97%), dr = 10:1, white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -2.4 (*c* 0.85, $CHCl_3$), HPLC analysis: 94% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 70:30, 1.0 mL /min, 70:30, 1.0 mL /min, 21.0 min (major), 27.1 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.0 Hz, 2H), 7.32 (d, *J* = 8.2 Hz, 2H), 7.06 (d, *J* = 8.6 Hz, 2H), 6.85 (d,

J = 8.6 Hz, 2H), 5.04-4.99 (m, 1H), 4.32-4.18 (m, 2H), 3.78 (s, 3H), 3.13 - 3.03 (m, 1H), 2.70-2.60 (m, 2H), 2.56-2.50 (m, 1H), 2.43 (s, 3H), 2.13-2.02 (m, 1H), 1.29 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 170.0, 159.0, 145.1, 136.1, 133.4, 129.8, 129.2, 127.6, 114.5, 62.3, 58.4, 55.4, 41.4, 35.9, 34.6, 21.8, 14.2. IR (KBr) ν 2961, 2924, 2853, 1747, 1704, 1515, 1456, 1260, 1088, 1019, 799, 661. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{22}H_{25}NO_6NaS$, 454.12948, Found 454.12946.

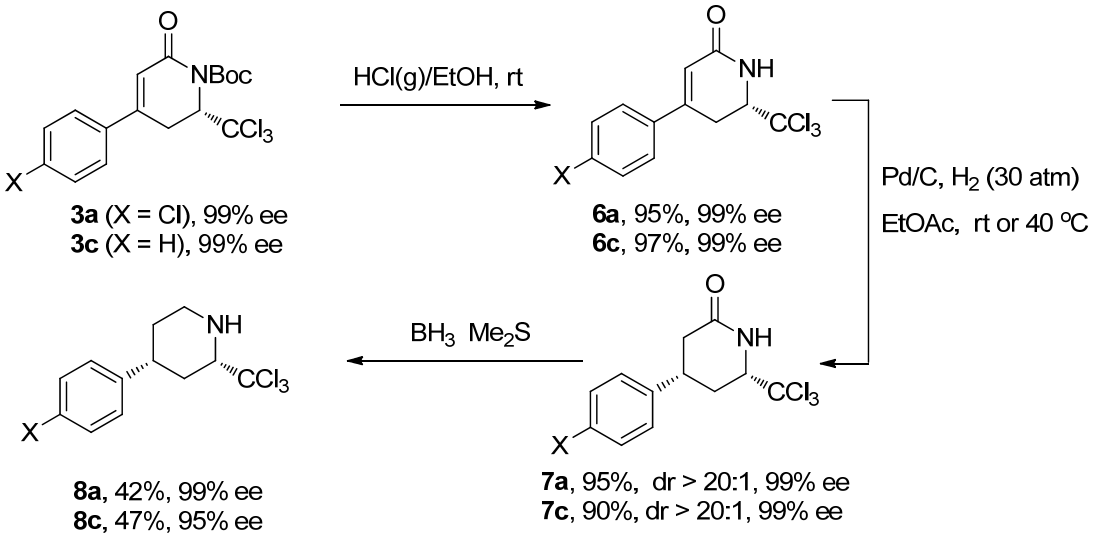


5k

(*2S,4S*)-ethyl 4-(4-aminophenyl)-6-oxo-1-tosylpiperidine-2-carboxylate (5k)

Yield: 37.0 mg (90%), dr = 10:1, white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 1:1); $[\alpha]_D^{25}$ -21.0 (*c* 0.93, $CHCl_3$), HPLC analysis: 99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 60:40, 1.0 mL /min, 70:30, 1.0 mL /min, 17.8 min (major), 37.8 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.3 Hz, 2H), 7.31 (d, *J* = 8.2 Hz, 2H), 6.90 (d, *J* = 8.3 Hz, 2H), 6.62 (d, *J* = 8.4 Hz, 2H), 5.01-4.96 (m, 1H), 4.30-4.18 (m, 2H), 3.65 (bs, 2H), 3.05-2.95 (m, 1H), 2.65-2.59 (m, 2H), 2.53-2.47 (m, 1H), 2.43 (s, 3H)2.09-1.98 (m, 1H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.5, 170.3, 145.8, 145.1, 136.1, 131.1, 129.7, 129.2, 127.5, 115.5, 62.2, 58.4, 41.4, 35.9, 34.7, 21.8, 14.1. IR (KBr) ν 3446, 2924, 1737, 1627, 1594, 1519, 1350, 1209, 1165, 1086, 1025, 659, 553. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{21}H_{25}N_2O_5S$, 417.14787, Found 417.14716.

(2) Synthesis of piperidine (Scheme 4b)



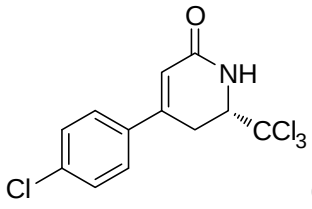
3a (X = Cl), 99% ee
3c (X = H), 99% ee

6a, 95%, 99% ee
6c, 97%, 99% ee

7a, 95%, dr > 20:1, 99% ee
7c, 90%, dr > 20:1, 99% ee

8a, 42%, 99% ee
8c, 47%, 95% ee

Deprotection: Dihydropridinone **3a** (438.6 mg, 1.04 mmol) or **3c** (419.0 mg, 1.08 mmol) was dissolved in 6 N HCl(g)/EtOH (6 mL) and the mixture was stirred at room temperature. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (PE/EA, 2:1) to give deprotective dihydropridinone **6a** or **6c**.

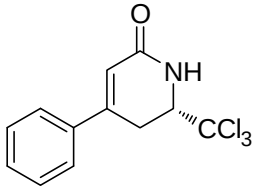


6a

***(S)*-4-(4-chlorophenyl)-6-(trichloromethyl)-5,6-dihydropyridin-2(1H)-one (6a)**

Yield: 318.2 mg (95%), white solid, mp: 155-157 ^{o} C. R_f = 0.2 (petroleum ether/ethyl acetate, 1:1); $[\alpha]_D^{25}$ 32.7 (*c* 0.52, CH_2Cl_2), HPLC analysis: 98% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 70:30, 1.0 mL /min, 14.2 min (minor), 18.6 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.20 (m, 4H), 6.66 (s, 1H), 6.21 (d, *J* = 1.4 Hz, 1H), 4.39-4.34 (m, 1H), 3.17 (d, *J* = 7.0 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 165.7, 147.4, 136.4, 135.4, 129.4, 127.4, 118.7, 102.1, 65.7,

28.0. IR (KBr) ν 3207, 2961, 1674, 1490, 1410, 1260, 1093, 1012, 808, 733. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{12}H_{10}NOCl_4$, 323.95110, Found 323.95105.

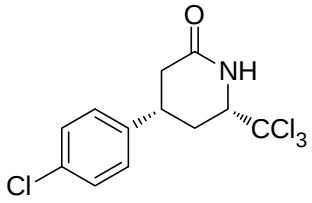


6c

***(S)*-4-phenyl-6-(trichloromethyl)-5,6-dihydropyridin-2(1H)-one (6c)**

Yield: 302.0 mg (97%), white solid, mp: 164-166 ^{o} C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ 33.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, - (minor), 24.9 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.47-7.45 (m, 2H), 7.39-7.36 (m, 3H), 6.24 (d, *J* = 1.3 Hz, 1H), 6.12 (s, 1H), 4.37 (dd, *J* = 7.0, 5.1 Hz, 1H), 3.26-3.17 (dd, *J* = 8.4, 1.2 Hz, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 166.1, 148.8, 137.0, 130.3, 129.1, 126.1, 118.2, 102.2, 65.7, 28.0. IR (KBr) ν 3481, 3227, 1675, 1620, 1446, 1293, 868, 810, 761, 692. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{12}H_{10}NO_3NaCl_3$, 311.97202, Found 311.97198.

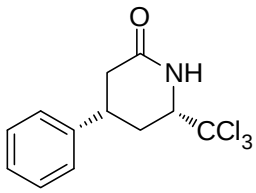
Hydrogenation: To the solution of **6a** (98.5 mg, 0.301 mmol) or **6c** (75.3 mg, 0.257 mmol) in EtOAc (3 mL) was added 10% Pd/C (10.0 mg) at 40 ^{o}C (for **S1a**) or room temperature (for **6c**), then the mixture was stirred at 40 ^{o}C or room temperature under 30 atm of hydrogen. The reaction mixture was diluted with ethyl acetate, and passed through a short silica pad. The solvent was removed under reduced pressure to give tetrahydropyridinone **7a** or **7c**.



7a

***(4S,6S)*-4-(4-chlorophenyl)-6-(trichloromethyl)piperidin-2-one (7a)**

Yield: 88.1 mg (90%), white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 1:1); $[\alpha]_D^{25}$ -21.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 90:10, 1.0 mL /min, - min (minor), 19.4 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.28-7.26 (m, 2H), 7.12-7.09 (m, 2H), 6.50 (s, 1H), 4.22 (dd, *J* = 10.2, 5.6 Hz, 1H), 3.10-2.99 (m, 1H), 2.65-2.50 (m, 2H), 2.44-2.34 (m, 1H), 1.94-1.82 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.7, 140.5, 133.4, 129.3, 128.0, 101.9, 67.3, 38.8, 36.7, 32.4. IR (KBr) ν 3217, 2925, 1674, 1493, 1396, 1313, 1092, 1013, 825, 799, 779. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{12}H_{12}NOCl_4$, 325.96675, Found 325.96618.

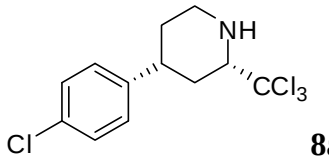


7c

***(4S,6S)*-4-phenyl-6-(trichloromethyl)piperidin-2-one (7c)**

Yield: 67.7 mg (90%), white solid, mp: 104-106 ^{o} C. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ -21.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 254 nm hexane/i-PrOH = 90:10, 1.0 mL /min, - min (minor), 13.5 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.33-7.28 (m, 2H), 7.25-7.16 (m, 3H), 6.43 (s, 1H), 4.22 (dd, *J* = 10.5, 5.4 Hz, 1H), 3.12-3.00 (m, 1H), 2.69-2.57 (m, 2H), 2.56-2.39 (m, 1H), 2.00-1.86 (m, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.1, 142.0, 129.2, 127.6, 126.6, 102.0, 67.5, 38.8, 37.3, 32.6. IR (KBr) ν 3217, 3029, 1671, 1455, 1394, 1307, 1029, 882, 780, 699, 611. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{12}H_{13}NO_3Cl_3$, 292.00572, Found 292.00570.

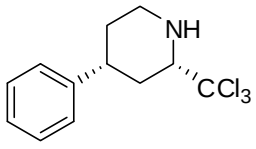
Reduction: To a solution of **7a** (21.9 mg, 0.067 mmol) or **7c** (40.0 mg, 0.137 mmol) in THF (1.0 mL) under nitrogen 10 N BH_3/Me_2S (20 μL or 41 μL, 3.0 eq) was added, then the mixture was heated to reflux for 17 h. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (PE/EA, 10:1) to give piperidine **8a** or **8c**.



8a

(2*S*,4*R*)-4-(4-chlorophenyl)-2-(trichloromethyl)piperidine (8a)

Yield: 8.8 mg (42%), white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -5.8 (*c* 1.2, CH_2Cl_2), HPLC analysis: >99% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 98:2, 1.0 mL /min, - min (minor), 20.5 min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29 (d, *J* = 8.7 Hz, 2H), 7.18 (d, *J* = 8.4 Hz, 2H), 3.44-3.35 (m, 2H), 2.91 (td, *J* = 12.1, 2.8 Hz, 1H), 2.69 (tt, *J* = 12.3, 3.5 Hz, 1H), 2.44-2.37 (m, 1H), 2.34 (br, 1H), 1.84 (d, *J* = 11.8 Hz, 1H), 1.73-1.61 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 143.8, 132.5, 128.9, 128.3, 103.2, 72.0, 46.7, 41.8, 35.6, 33.0. IR (KBr) ν 2923, 1493, 1432, 1308, 1091, 1013, 796, 763, 697. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{12}H_{14}NCl_4$, 311.98749, Found 311.98726.

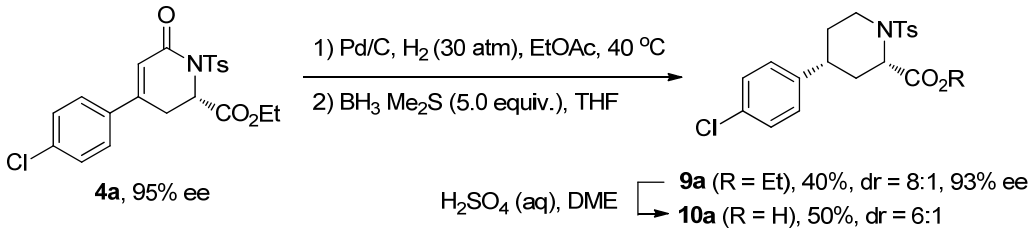


8c

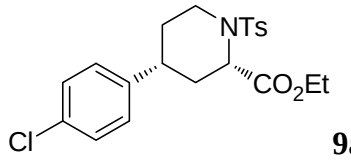
***(2S,4R)*-4-phenyl-2-(trichloromethyl)piperidine (8c)**

Yield: 18.8 mg (47%), white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 10:1); $[\alpha]_D^{25}$ -5.3 (*c* 1.5, CH_2Cl_2), HPLC analysis: 95% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 98:2, 1.0 mL /min, 10.7 min (major), 14.3 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.29-7.24 (m, 2H), 7.20 - 7.17 (m, 3H), 3.38-3.30 (m, 2H), 2.86 (td, *J* = 12.0, 2.9 Hz, 1H), 2.65 (tt, *J* = 12.3, 3.6 Hz, 1H), 2.40-2.36 (m, 1H), 1.81 (d, *J* = 12.5 Hz, 1H), 1.73-1.58 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 145.3, 128.8, 127.0, 126.8, 72.1, 46.9, 42.3, 35.6, 33.0. IR (KBr) ν 2929, 1494, 1453, 1147, 766, 754, 698, 656. HRMS (ESI) *m/z*: $[M+H]^+$ Calc. for: $C_{12}H_{15}NCl_3$, 278.02646, Found 278.02713.

(3) Synthesis of pipecolic acid (Scheme 4c)



Hydrogenation and reduction: To the solution of **4a** (211 mg, 0.487 mmol) in EtOAc (3 mL) was added 10% Pd/C (20 mg) at room temperature, then the mixture was stirred at 40 ^{o} C under 30 atm of hydrogen. The reaction mixture was diluted with ethyl acetate, and passed through a short silica pad. The solvent was removed under reduced pressure to give the corresponding tetrahydropyridinone. To the solution of the tetrahydropyridinone (56 mg, 0.128 mmol) in THF (2.0 mL) under nitrogen 10 N BH_3/Me_2S (65 μL, 5.0 eq) was added, then the mixture was stirred at room temperature for 17 h. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (PE/EA, 3:1) to give **9a.**



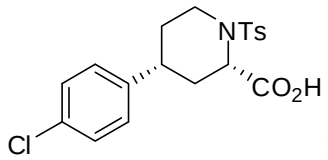
9a

(2S,4R)-ethyl 4-(4-chlorophenyl)-1-tosylpiperidine-2-carboxylate (9a)

Total Yield: 22.0 mg (40%), dr =8:1, white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 1:1); $[\alpha]_D^{25}$ -21.0 (*c* 1.0, CH_2Cl_2), HPLC analysis: 93% ee [Daicel CHIRALPAK AS-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 85:15, 1.0 mL /min, 22.2 min (minor), 27.2min (major)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.72-7.69 (m, 2H), 7.29-7.27 (m, 2H), 7.24-7.12 (m, 2H), 7.08-7.06 (m, 1H), 7.02-6.99 (d, *J* = 8.4 Hz, 1H), 4.14-4.00 (m, 2H), 3.74-3.63 (m, 2H), 2.72-2.62 (m, 1H), 2.48-2.43 (m, 1H), 2.38 (s, 3H), 2.09-2.03 (m, 2H), 1.87-1.83 (m, 2H), 1.19 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.3 (171.2), 144.10 (144.05), 143.6 (142.1), 133.5 (133.4), 132.6 (129.7), 128.9 (128.8), 128.52 (128.48), 128.3 (126.9), 61.7 (61.6), 59.4 (59.1),

45.9 (45.6), 39.2 (38.5), 36.4 (36.1), 30.70 (30.65), 21.7, 14.1. (The data in parentheses was for the minor *trans*-isomer). IR (KBr) 2926, 1737, 1494, 1347, 1166, 1092, 1057, 954, 817, 756, 651, 588, 551. HRMS (ESI) *m/z*: $[M+Na]^+$ Calc. for: $C_{21}H_{24}NO_4NaClS$, 444.10068, Found 444.10101.

Hydrolysis: To the solution of **9a** (22.0 mg, 0.052 mmol) in DME (1 mL) was charged with 8N H_2SO_4 (1.0 mL) and then the mixture was heated to reflux. The mixture was extracted with ethyl acetate, and dried with anhydrous Na_2SO_4. The solvent was removed under reduced pressure and the residue was purified by chromatography on silica gel (dichloromethane/methanol, 20:1) to give **10a** (ee% was not determined).

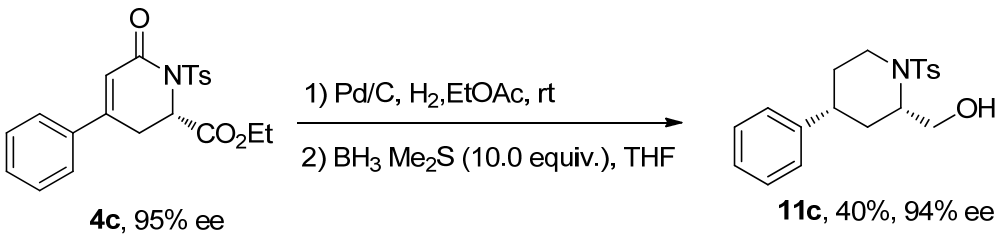


10a

(2S,4R)-4-(4-chlorophenyl)-1-tosylpiperidine-2-carboxylic acid (10a)

Yield: 10.0 mg (60%), dr =6:1, pale wax, R_f = 0.3 (dichloromethane/methanol, 10:1); $[\alpha]_D^{25}$ -56.3 (*c* 0.27, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.74-7.61 (m, 2H), 7.30-7.27 (m, 2H), 7.22-7.11 (m, 2H), 7.05 (d, *J* = 7.2 Hz, 1H), 6.99 (d, *J* = 8.3 Hz, 1H), 4.44 (bs, 1H), 3.72-3.65 (m, 2H), 2.74-2.67 (m, 1H), 2.45-2.34 (m, 4H), 2.16-2.09 (m, 1H), 2.06-1.95 (m, 1H), 1.86-1.75 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 175.7, 144.3 (143.6), 142.1, 133.34 (133.27), 132.7 (129.9), 128.9 (128.8), 128.6 (128.5), 128.3, 127.0 (126.9), 59.0 (58.8), 45.7 (45.4), 39.2 (38.5), 36.0 (35.8), 30.9 (30.8), 21.7. (The data in parentheses was for the minor *trans*-isomer). IR (KBr) ν 3386, 2923, 2853, 1719, 1494, 1340, 1165, 1092, 817, 752, 654, 580, 456. HRMS (ESI) *m/z*: $[M-H]^-$ Calc. for: $C_{19}H_{19}NO_4ClS$, 392.07178, Found 392.07182.

(4) Synthesis of piperidine methanol (Scheme 4d)



4c, 95% ee

11c, 40%, 94% ee

The procedure was carried as in Scheme 1c, except that room temperature was applied for hydrogenation, and 10 equiv. of BH_3 was used for reduction.

((*2S,4R*)-4-phenyl-1-tosylpiperidin-2-yl)methanol (11c)

Overall yield for two steps: 17.3 mg (40%). white waxy solid. R_f = 0.3 (petroleum ether/ethyl acetate, 3:1); $[\alpha]_D^{25}$ –34.7 (*c* 0.75, $CHCl_3$), HPLC analysis: 94% ee [Daicel CHIRALPAK AD-H column, 20 ^{o} C, 220 nm hexane/i-PrOH = 60:40, 1.0 mL /min, 8.9 min (major), 10.9 min (minor)]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.2 Hz, 2H), 7.30 (d, *J* = 8.1 Hz, 2H), 7.22-7.18 (m, 2H), 7.14-7.09 (m, 1H), 7.01-6.99 (m, 2H), 4.04-4.00 (m, 1H), 3.91 (dt, *J* = 8.4, 4.9 Hz, 1H), 3.02-2.85 (m, 1H),3.59-3.51 (m, 1H) 3.09-2.83 (m, 3H), 2.40 (s, 3H), 2.36-2.29 (m, 1H), 1.97-1.81 (m, 2H), 1.72-1.59 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 144.7, 144.0, 130.0, 128.7, 127.6, 126.8, 126.7, 64.9, 60.9, 47.3, 40.8, 35.4, 31.9, 21.7. IR (KBr) ν 3502, 2954, 2923, 2853, 1456, 1324, 1288, 1158, 1090, 799, 716, 700, 573. $[M+Na]^+$ Calc. for: $C_{19}H_{23}NO_3NaS$, 368.12909, Found 368.12897.

5. X-ray structures of 7c (Figure S1)

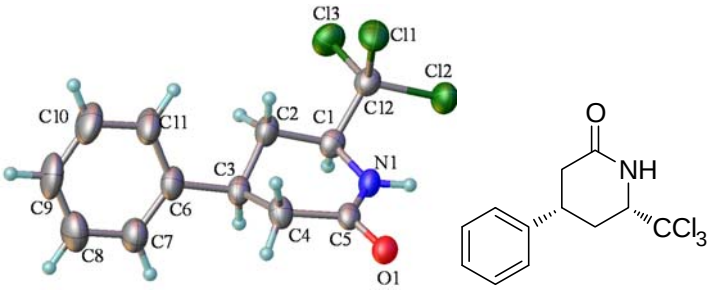


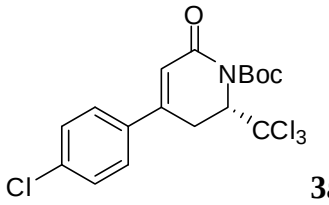
Figure S1. X-ray crystal structure of **7c**

Reference

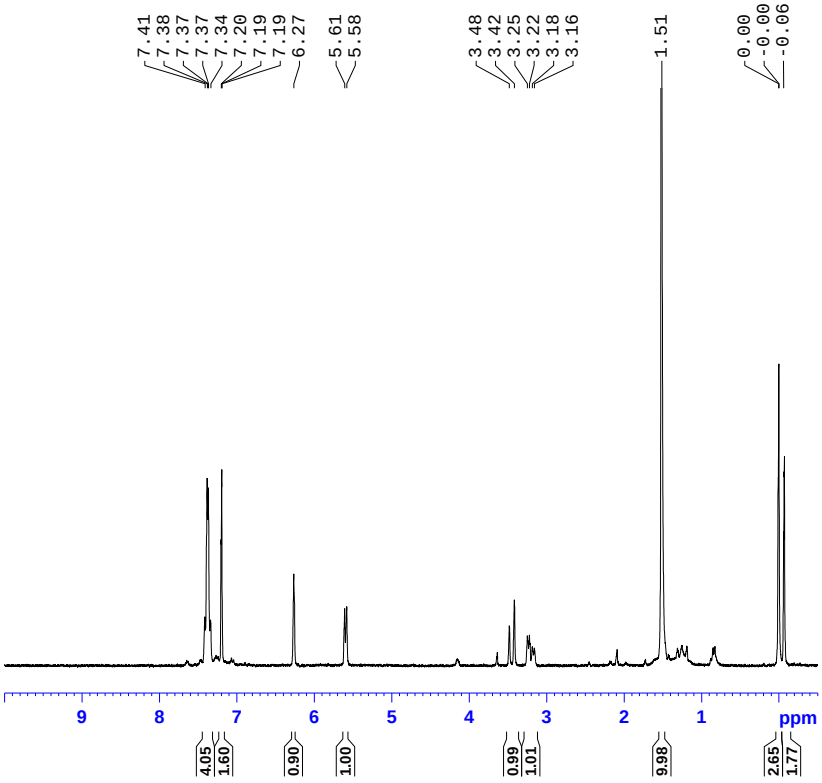
(1) Tiseni, P. S.; Peters, R. *Angew. Chem. Int. Ed.* **2007**, *46*, 5325.

(2) (a) Oliver, L. H.; Puls, L. A.; Tobey, S. L. *Tetrahedron Letters*, **2008**, *49*, 4636; (b)Vidal, J.; Hannachi, J.-C.; Hourdin, G.; Mulatier, J.-C.; Collet, A. *Tetrahedron Letters*, **1998**, 39, 8845.

Part II NMR Spectra



3a

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PROCNO               1
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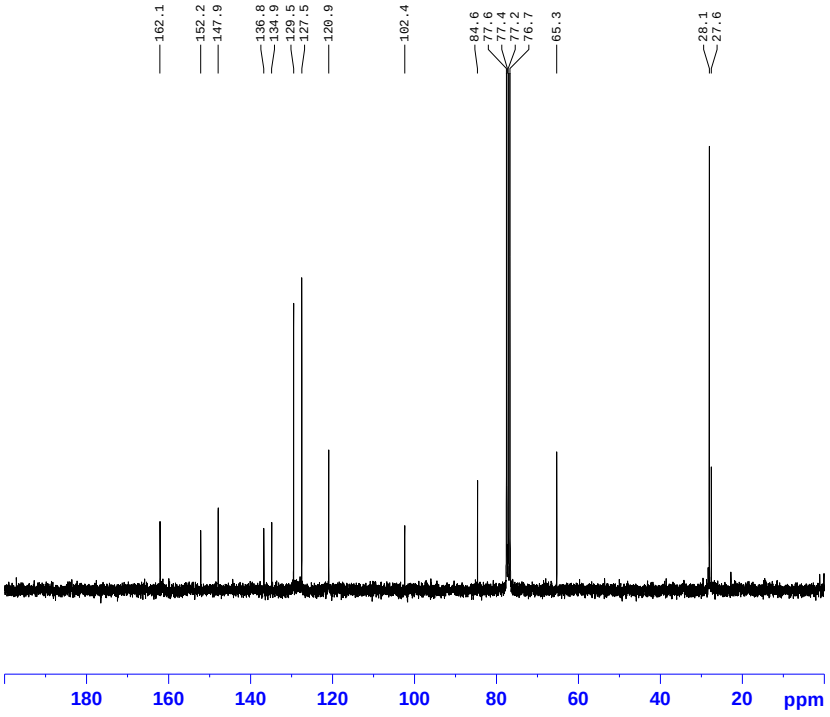
TD               32768
SOLVENT          CDCl3
NS                   6
DS                   0
SWH           6172.839 Hz
FIDRES         0.188380 Hz
AQ            2.6542580 sec
RG                 362
DW              81.000 usec
DE               6.50 usec
TE              295.6 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1               10.30 usec
PL1               3.00 dB
SFO1        300.1318534 MHz
SI                32768
SF          300.1300273 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```

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PROCNO               1
Date_         20130126
Time             20.55
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PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 968
DS                   4
SWH          17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG                 512
DW              27.800 usec
DE               6.50 usec
TE              295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1              12.50 usec
PL1              2.00 dB
SFO1        75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           100.00 usec
PL2               3.00 dB
PL12             22.74 dB
PL13             23.00 dB
SFO2        300.1312005 MHz
SI                32768
SF          75.4677389 MHz
WDW                EM
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LB               1.00 Hz
GB                  0
PC               1.40
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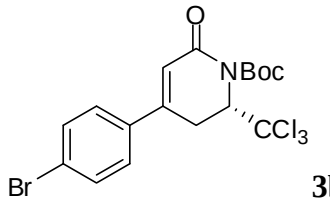
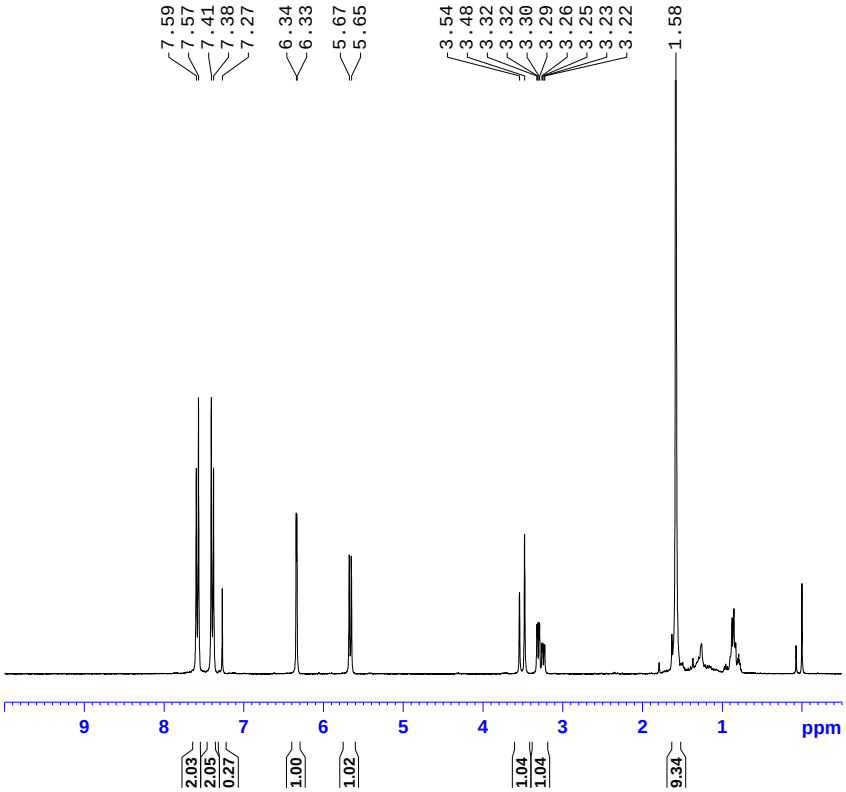


3b

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PROCNO                1
Date_          20130304
Time              18.45
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PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    3
DS                    0
SWH            8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG                  181
DW               55.600 usec
DE                8.00 usec
TE               297.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                 32768
SF          300.1300039 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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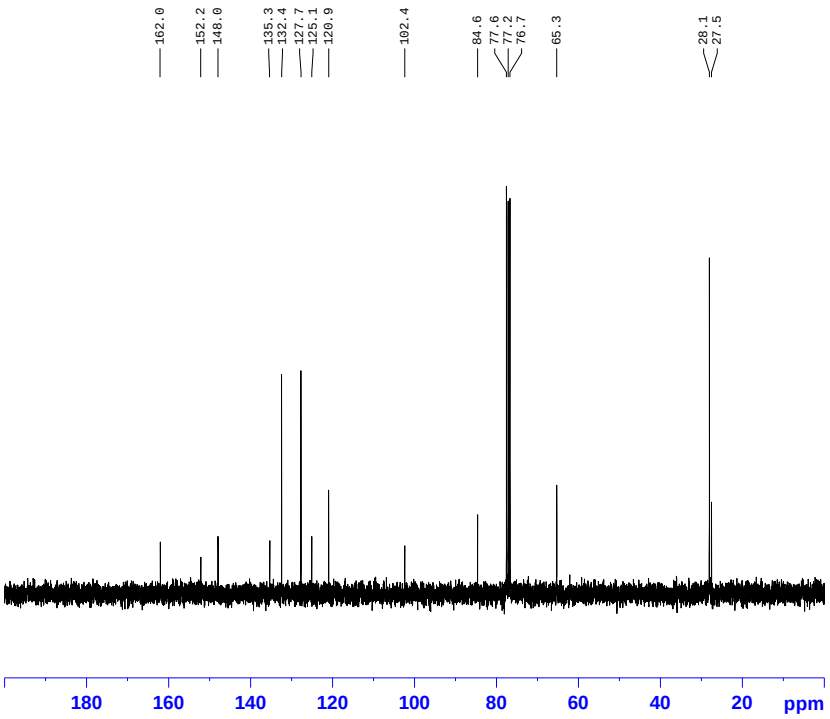


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Time              18.47
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PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   41
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG               1149.4
DW               27.800 usec
DE                6.50 usec
TE               297.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677394 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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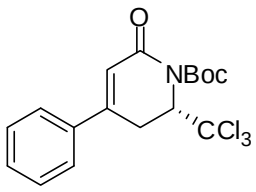
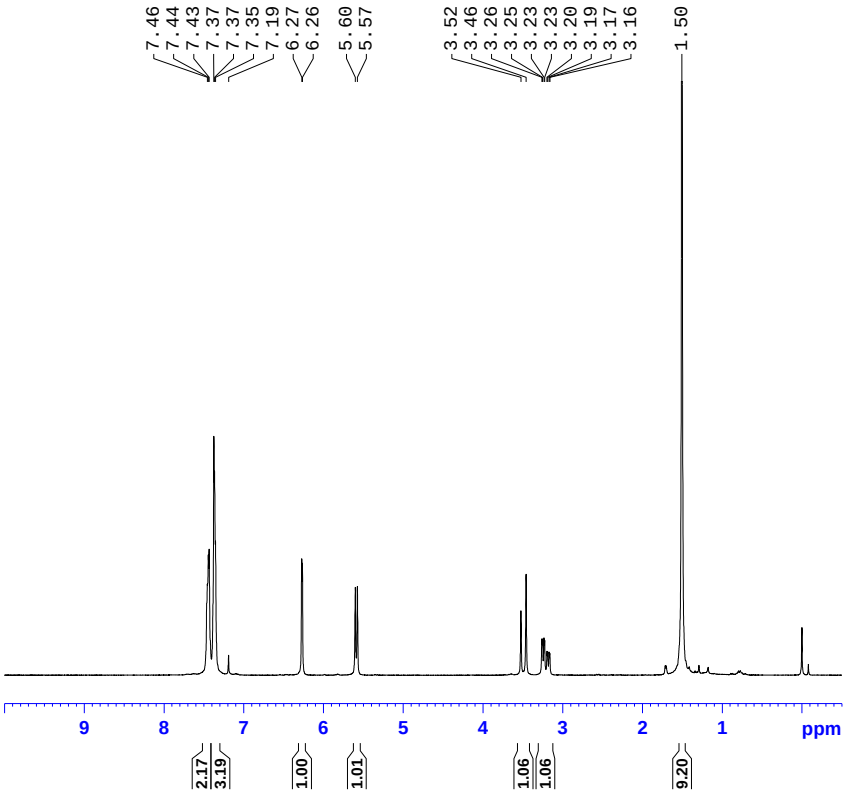


3c

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PROCNO                1
Date_          20130328
Time               0.27
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PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    3
DS                    0
SWH            6172.839 Hz
FIDRES        0.188380 Hz
AQ            2.6542580 sec
RG                   64
DW               81.000 usec
DE                6.50 usec
TE                295.4 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1318534 MHz
SI                 32768
SF          300.1300282 MHz
WDW                   EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
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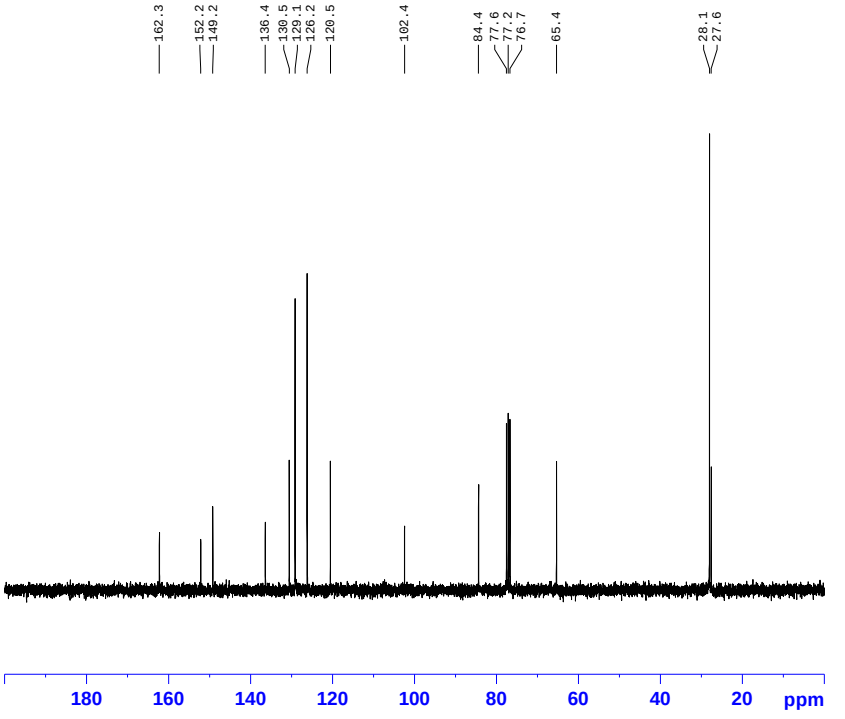


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PROCNO                1
Date_          20130328
Time               0.32
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PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   47
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                456.1
DW               27.800 usec
DE                6.50 usec
TE                295.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677441 MHz
WDW                   EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
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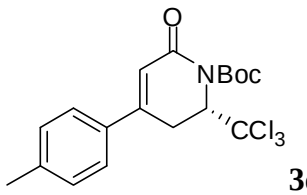
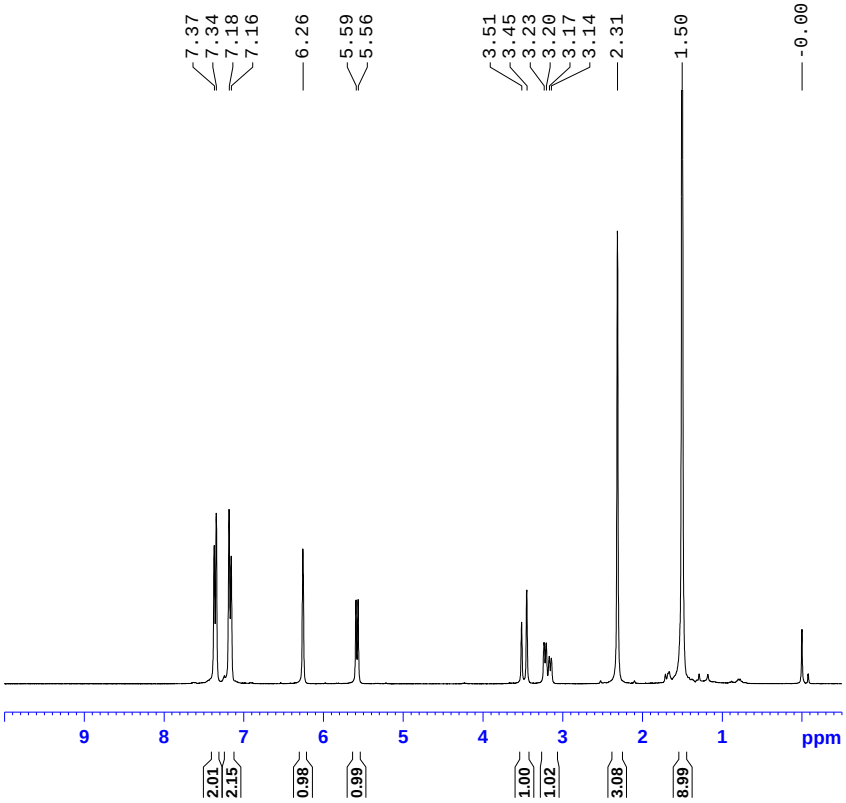


3d

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EXPNO               10
PROCNO               1
Date_         20130308
Time             13.55
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PROBHD   5 mm DUL 13C-1
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                   4
DS                   0
SWH           8992.806 Hz
FIDRES        0.137219 Hz
AQ           3.6438515 sec
RG                  64
DW             55.600 usec
DE              8.00 usec
TE              296.8 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1              10.30 usec
PL1              3.00 dB
SFO1       300.1324010 MHz
SI                32768
SF         300.1300271 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC              1.00
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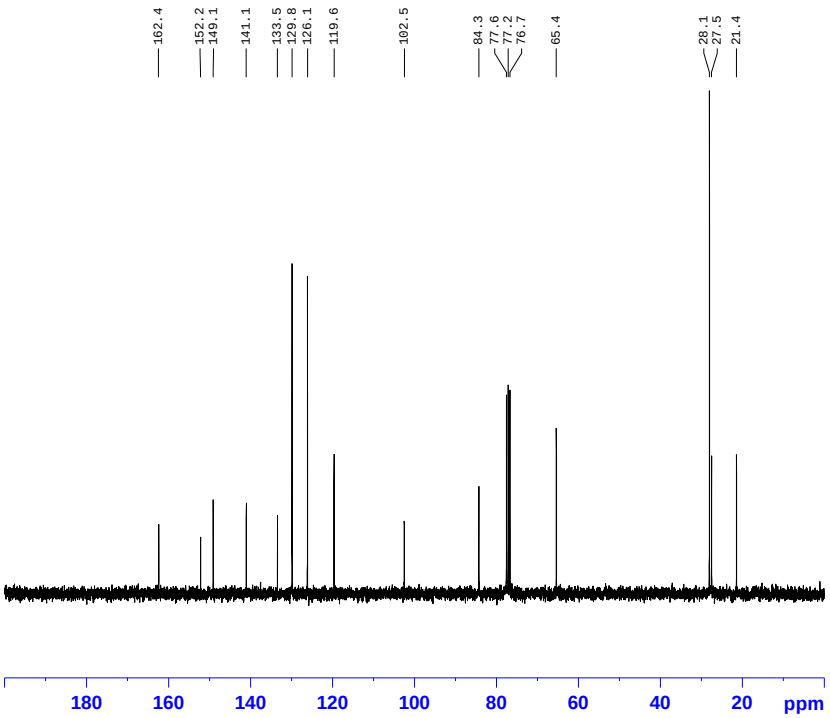


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PROCNO               1
Date_         20130308
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INSTRUM          spect
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PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                  61
DS                   4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ           1.8219508 sec
RG               812.7
DW             27.800 usec
DE              6.50 usec
TE              297.2 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1              12.50 usec
PL1              2.00 dB
SFO1       75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2          100.00 usec
PL2              3.00 dB
PL12            22.74 dB
PL13            23.00 dB
SFO2       300.1312005 MHz
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SF         75.4677425 MHz
WDW                EM
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GB                  0
PC              1.40
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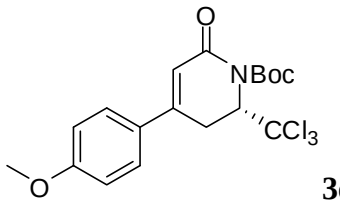

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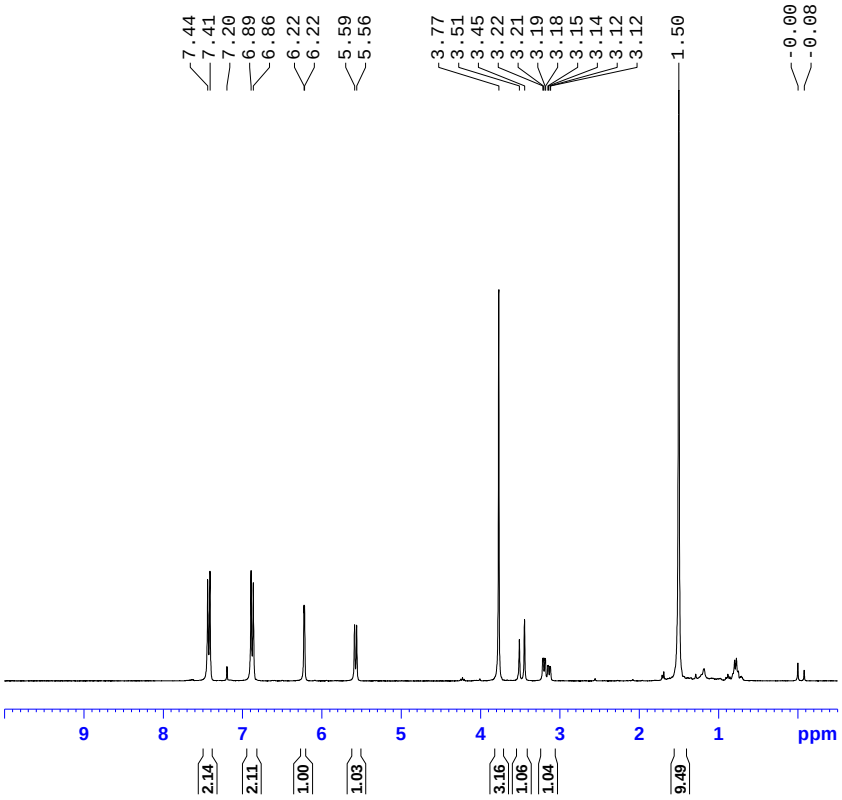


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PROCNO               1
Date_         20130311
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INSTRUM          spect
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   3
DS                   0
SWH           8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG                71.8
DW               55.600 usec
DE                8.00 usec
TE                297.3 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300251 MHz
WDW                EM
SSB                 0
LB                0.30 Hz
GB                  0
PC                1.00
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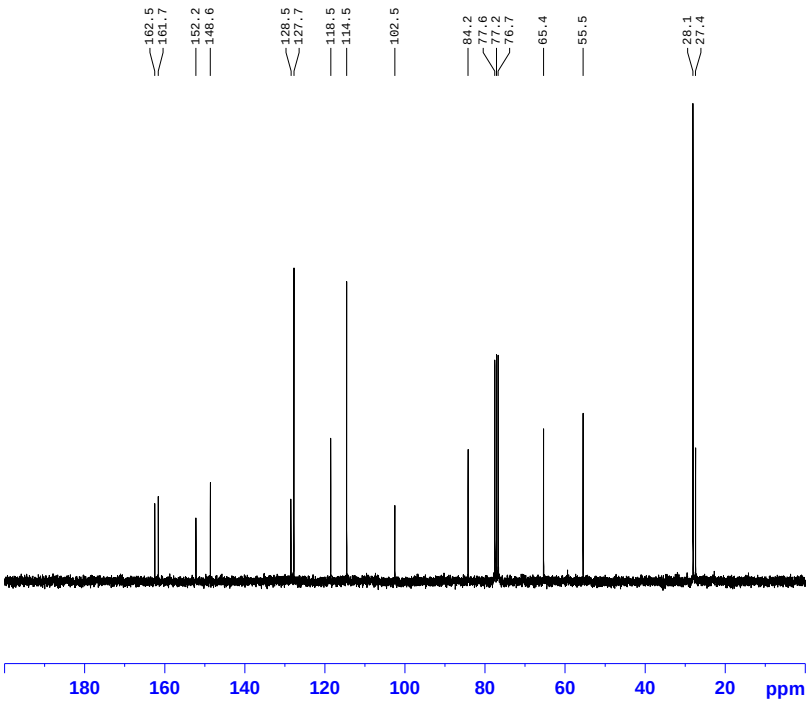


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PROCNO               1
Date_         20130311
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PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  90
DS                   4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                574.7
DW               27.800 usec
DE                6.50 usec
TE                297.8 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                3.00 dB
PL12               22.74 dB
PL13               23.00 dB
SFO2        300.1312005 MHz
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SF           75.4677427 MHz
WDW                EM
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PC                1.40
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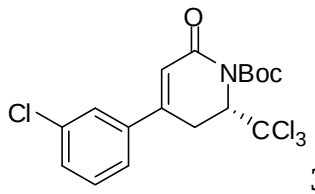
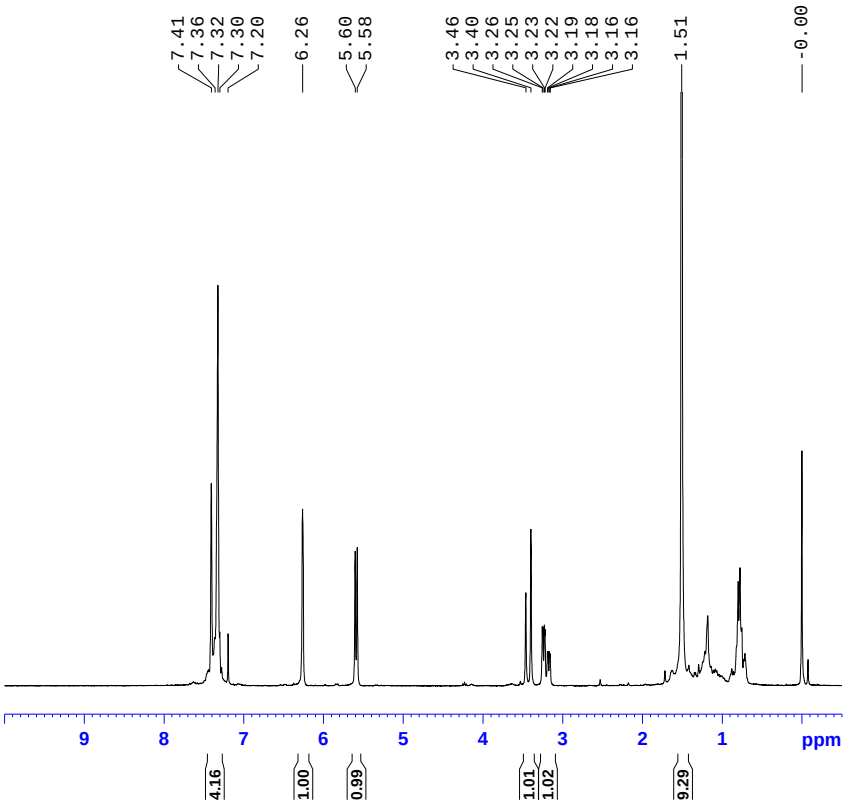


3f

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PROCNO                     1
Date_               20130308
Time                  21.30
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG                zg30
TD                     65536
SOLVENT                CDCl3
NS                         4
DS                         0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                 3.6438515 sec
RG                       114
DW                    55.600 usec
DE                     8.00 usec
TE                    296.3 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.30 usec
PL1                     3.00 dB
SFO1             300.1324010 MHz
SI                       32768
SF               300.1300252 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```
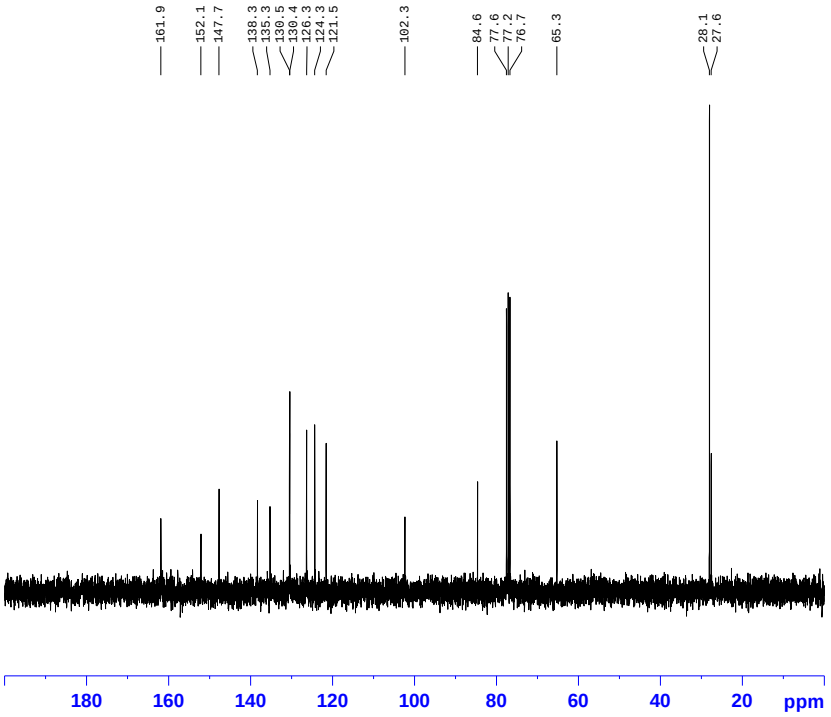
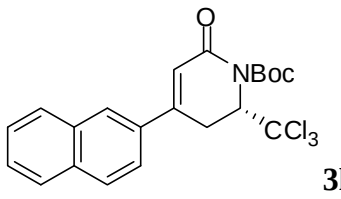
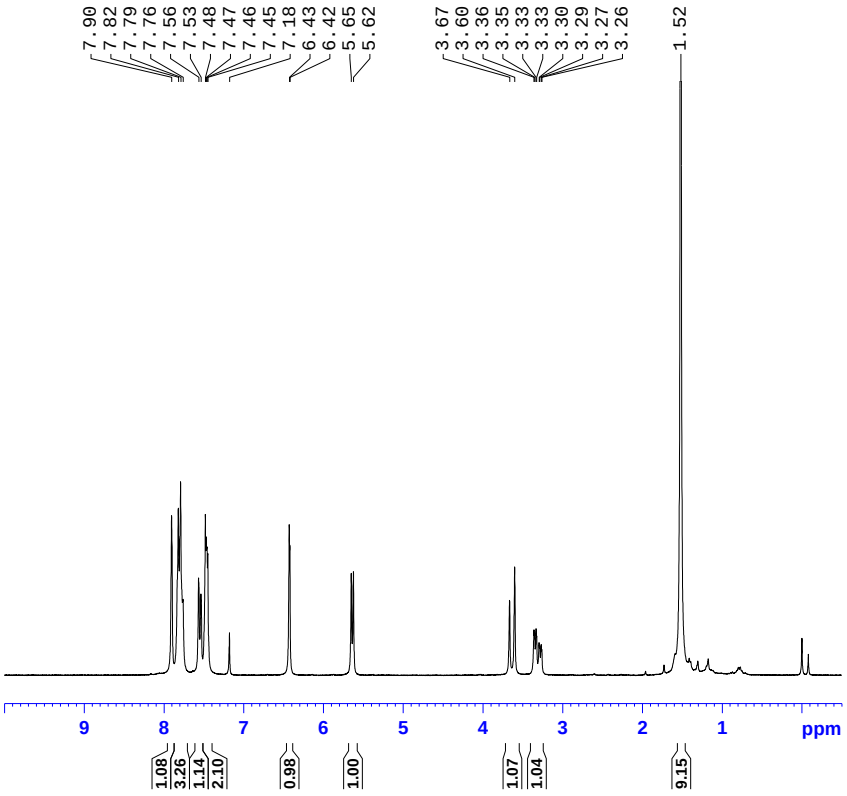



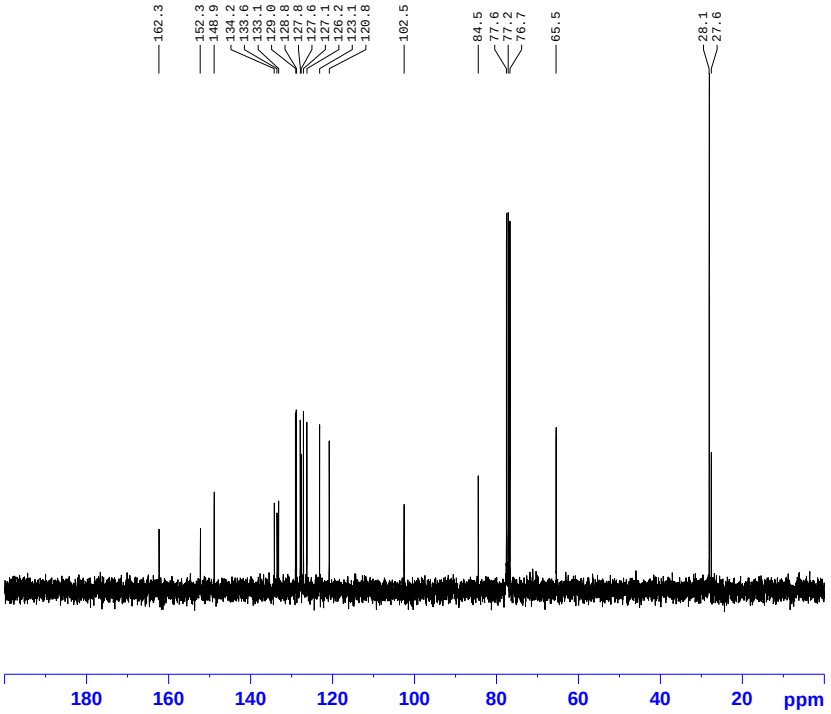

```
NAME          jwq-451d-065g1
EXPNO                     11
PROCNO                     1
Date_               20130308
Time                  21.32
INSTRUM               spect
PROBHD   5 mm DUL 13C-1
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                        26
DS                         4
SWH               17985.611 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                      1024
DW                    27.800 usec
DE                     6.50 usec
TE                    296.5 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     12.50 usec
PL1                     2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                     3.00 dB
PL12                   22.74 dB
PL13                   23.00 dB
SFO2             300.1312005 MHz
SI                       32768
SF               75.4677410 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



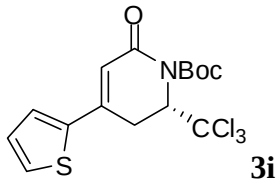
3h



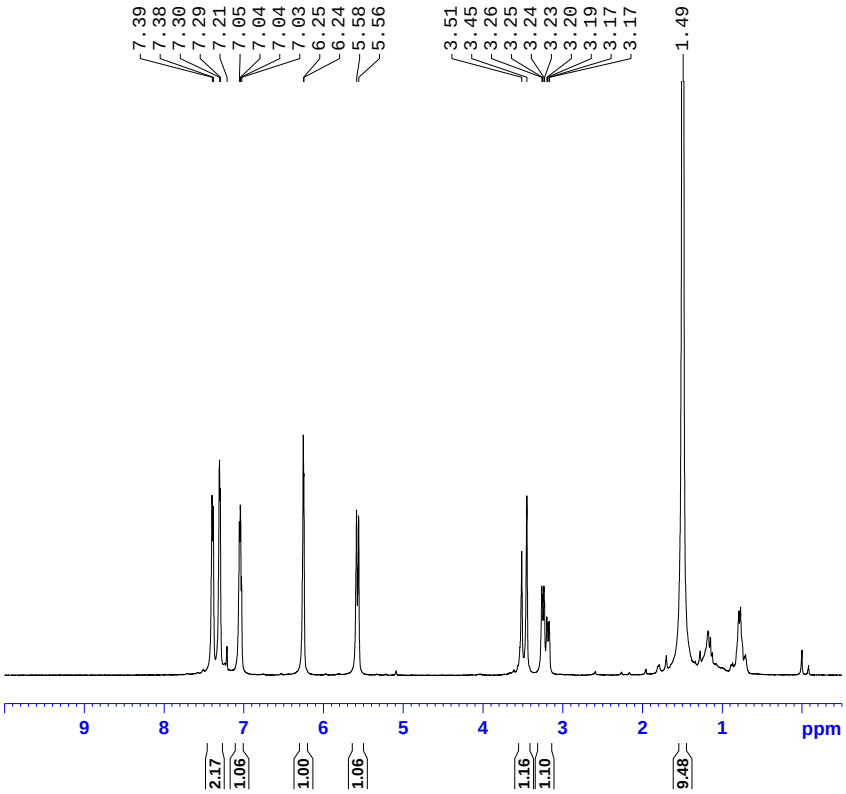

```
NAME     jwq-457e-073g1
EXPNO                11
PROCNO                1
Date_          20130328
Time               0.08
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            6172.839 Hz
FIDRES        0.188380 Hz
AQ            2.6542580 sec
RG                  181
DW               81.000 usec
DE                6.50 usec
TE                295.2 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1318534 MHz
SI                32768
SF           300.1300322 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



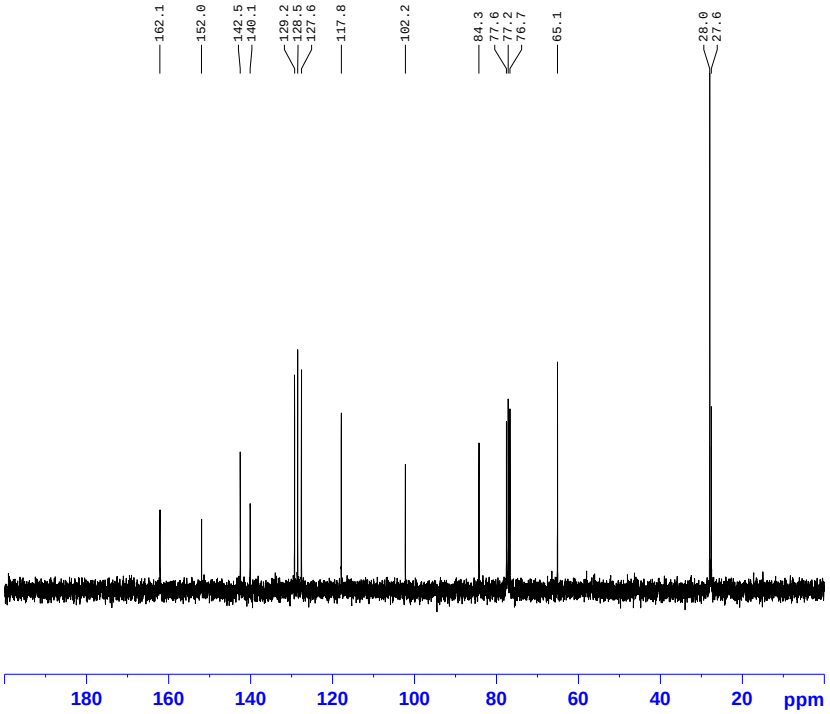

```
NAME     jwq-457e-073g1
EXPNO                10
PROCNO                1
Date_          20130328
Time               0.16
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   87
DS                    4
SWH           17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                  512
DW               27.800 usec
DE                6.50 usec
TE                295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                32768
SF           75.4677418 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



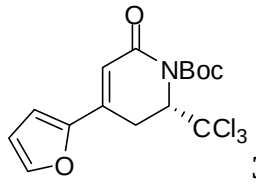
3i



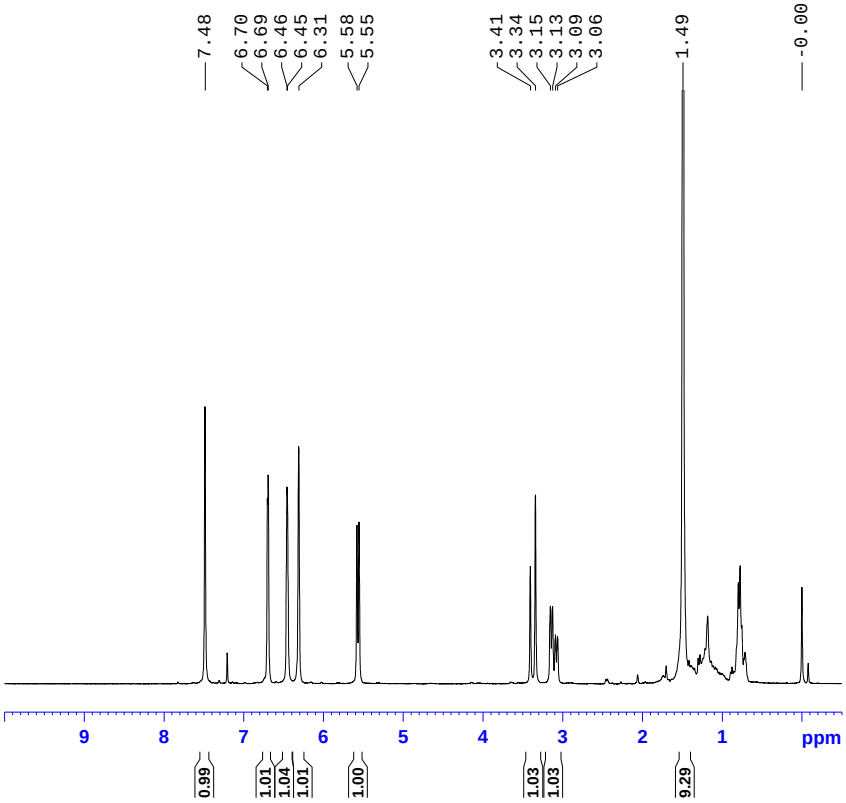

```
NAME     jwq-458a-073g1
EXPNO                 20
PROCNO                 1
Date_           20130328
Time                0.39
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                  45.3
DW                 81.000 usec
DE                  6.50 usec
TE                 295.4 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.30 usec
PL1                 3.00 dB
SFO1          300.1318534 MHz
SI                 32768
SF            300.1300227 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



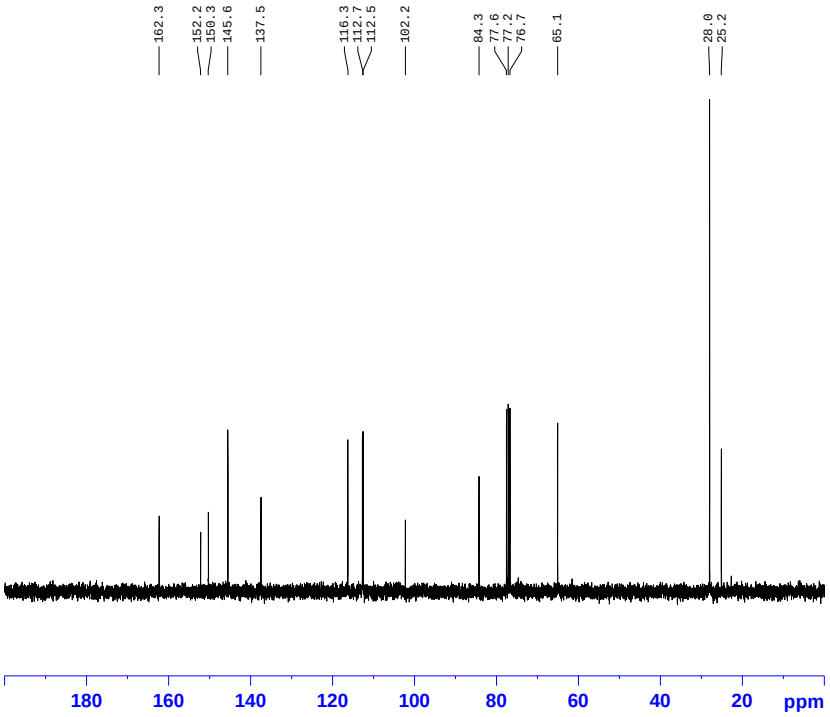

```
NAME     jwq-458a-073g1
EXPNO                 21
PROCNO                 1
Date_           20130328
Time                0.41
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    26
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                   362
DW                27.800 usec
DE                 6.50 usec
TE                295.7 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12                22.74 dB
PL13                23.00 dB
SFO2          300.1312005 MHz
SI                 32768
SF             75.4677474 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



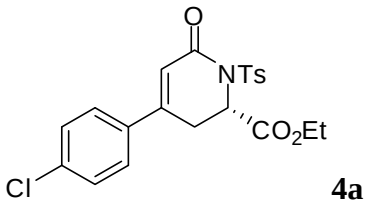
3j



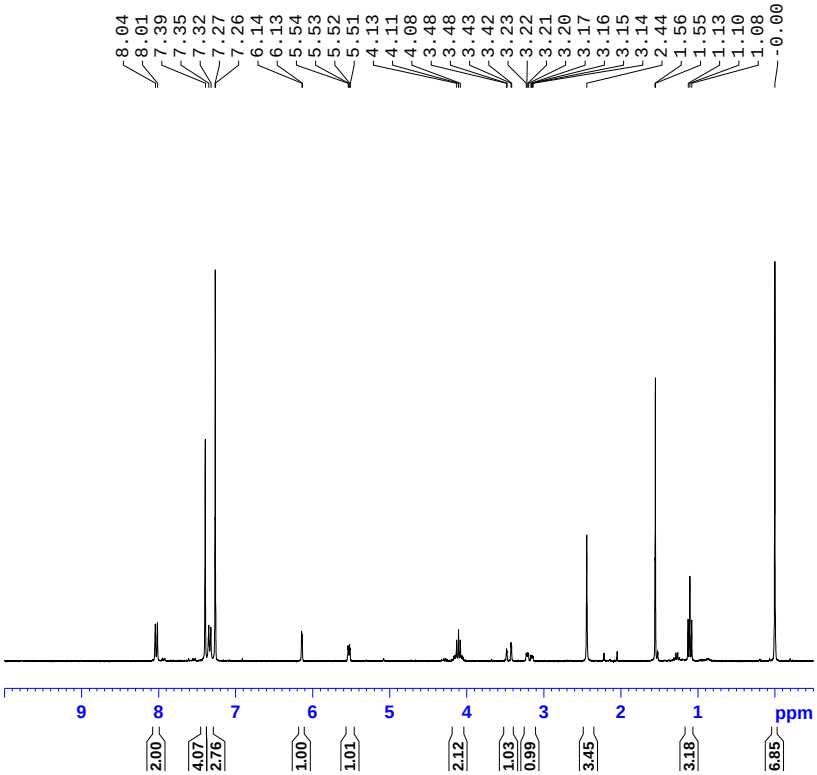

```
NAME          jwq-452d-067g1
EXPNO                     10
PROCNO                     1
Date_               20130308
Time                  21.23
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        4
DS                        0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                3.6438515 sec
RG                       64
DW                   55.600 usec
DE                    8.00 usec
TE                   296.3 K
D1               1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   10.30 usec
PL1                   3.00 dB
SFO1            300.1324010 MHz
SI                    32768
SF              300.1300215 MHz
WDW                      EM
SSB                       0
LB                   0.30 Hz
GB                        0
PC                    1.00
```



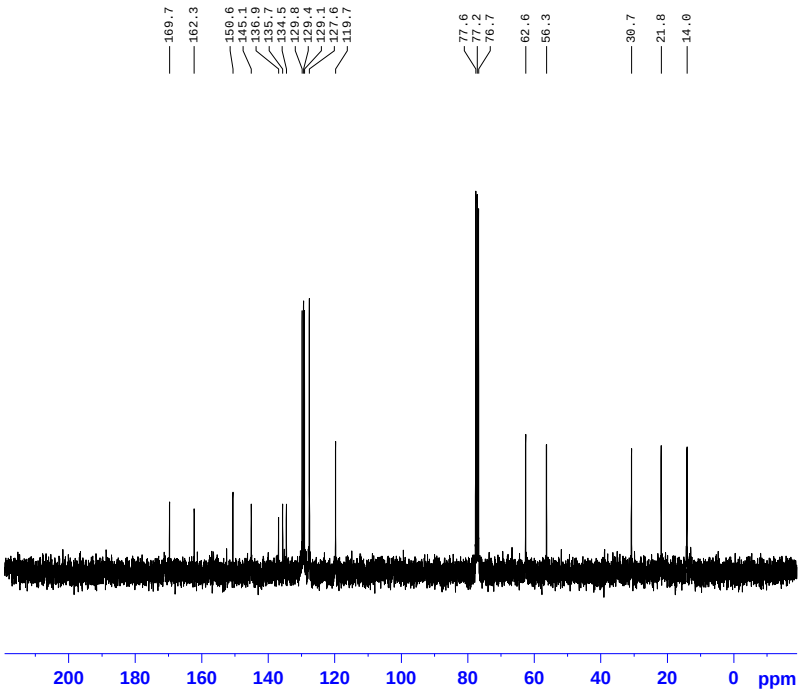

```
NAME          jwq-452d-067g1
EXPNO                     11
PROCNO                     1
Date_               20130308
Time                  21.25
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       30
DS                        4
SWH               17985.611 Hz
FIDRES             0.274439 Hz
AQ                1.8219508 sec
RG                    812.7
DW                   27.800 usec
DE                    6.50 usec
TE                   296.5 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                   3.00 dB
PL12                 22.74 dB
PL13                 23.00 dB
SFO2            300.1312005 MHz
SI                    32768
SF              75.4677430 MHz
WDW                      EM
SSB                       0
LB                   1.00 Hz
GB                        0
PC                    1.40
```



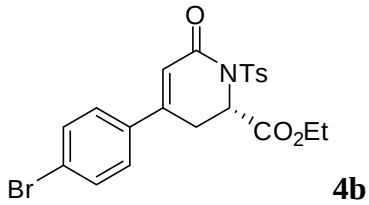
4a



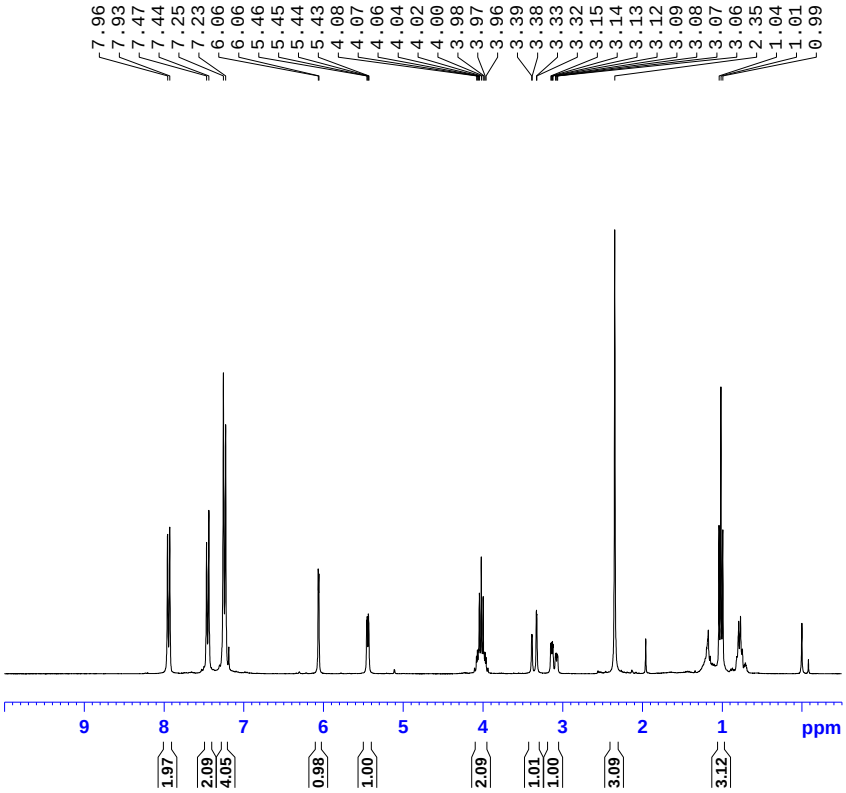

```
NAME      jwq-471b-086g1
EXPNO                 10
PROCNO                 1
Date_           20130413
Time               16.53
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    15
DS                     0
SWH             6172.839 Hz
FIDRES          0.188380 Hz
AQ              2.6542580 sec
RG                574.7
DW                81.000 usec
DE                 6.50 usec
TE                294.8 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.30 usec
PL1                 3.00 dB
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300064 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



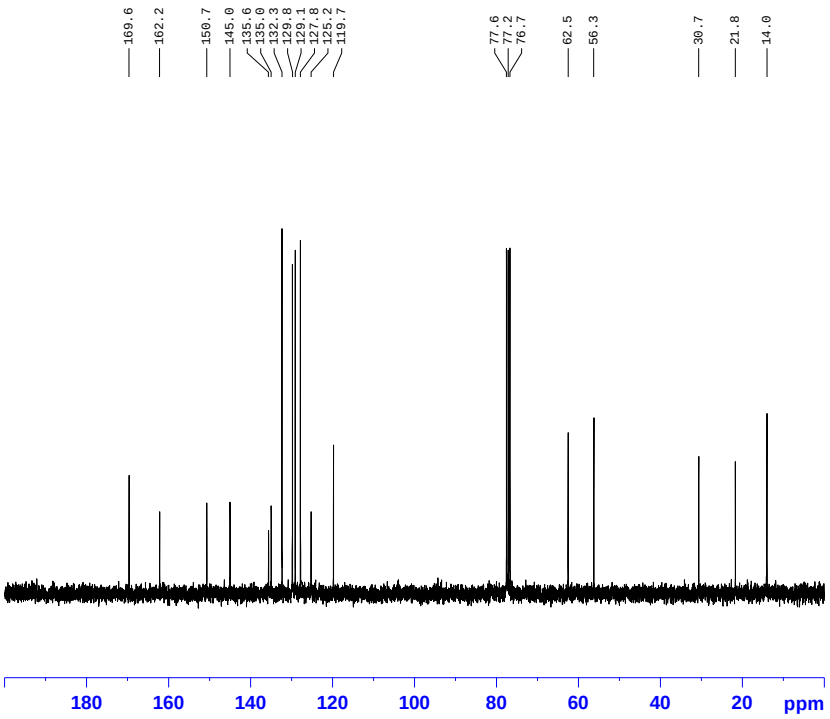

```
NAME      jwq-337a-037g1
EXPNO                 20
PROCNO                 1
Date_           20121015
Time               18.55
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   167
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 1024
DW                27.800 usec
DE                 6.50 usec
TE                296.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12               22.74 dB
PL13               23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677406 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



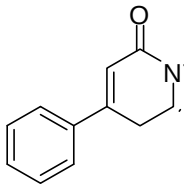
4b



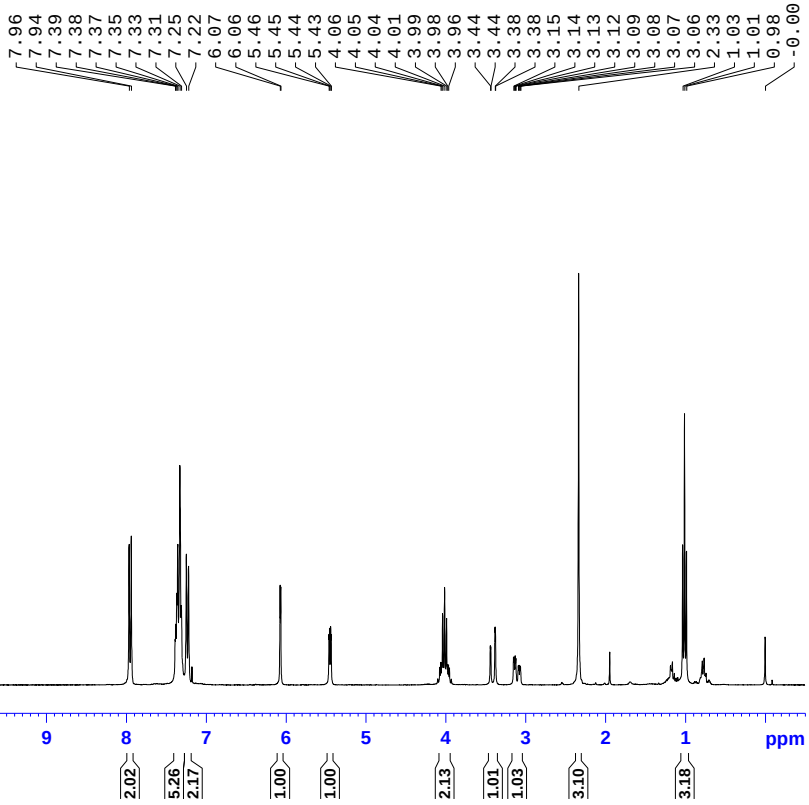

```
NAME     jwq-390c-xxxg1
EXPNO               10
PROCNO               1
Date_         20121128
Time             23.34
INSTRUM          spect
PROBHD   5 mm DUL 13C-1
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   6
DS                   0
SWH           8992.806 Hz
FIDRES        0.137219 Hz
AQ            3.6438515 sec
RG                71.8
DW               55.600 usec
DE                8.00 usec
TE               296.6 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1                1H
P1                10.30 usec
PL1                3.00 dB
SFO1       300.1324010 MHz
SI               32768
SF         300.1300276 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



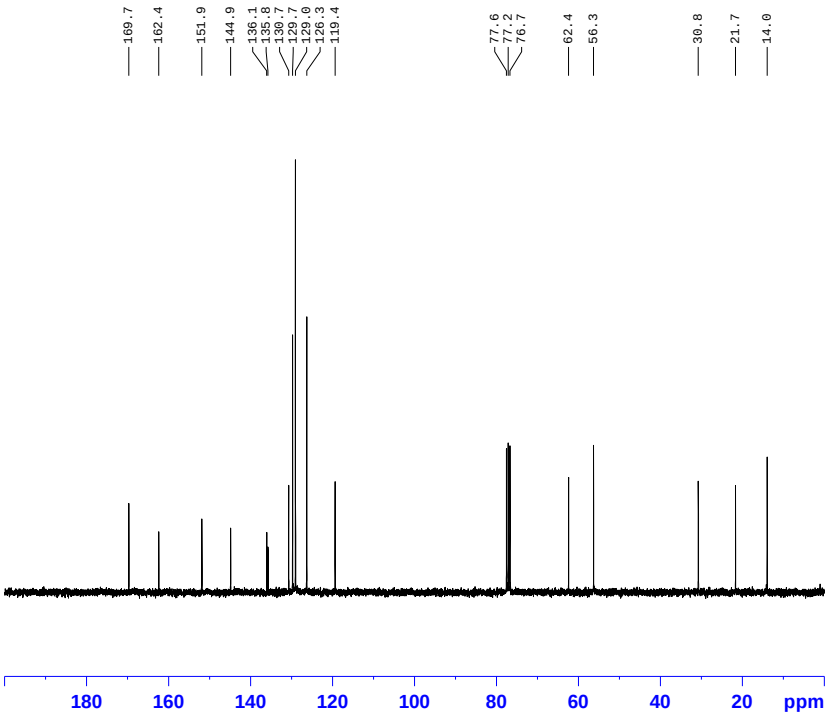

```
NAME     jwq-390c-xxxg1
EXPNO               11
PROCNO               1
Date_         20121128
Time             23.37
INSTRUM          spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  48
DS                   4
SWH          17985.611 Hz
FIDRES        0.274439 Hz
AQ            1.8219508 sec
RG                 512
DW               27.800 usec
DE                6.50 usec
TE               297.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
NUC1               13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI               32768
SF          75.4677448 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```



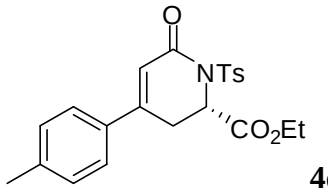
4c



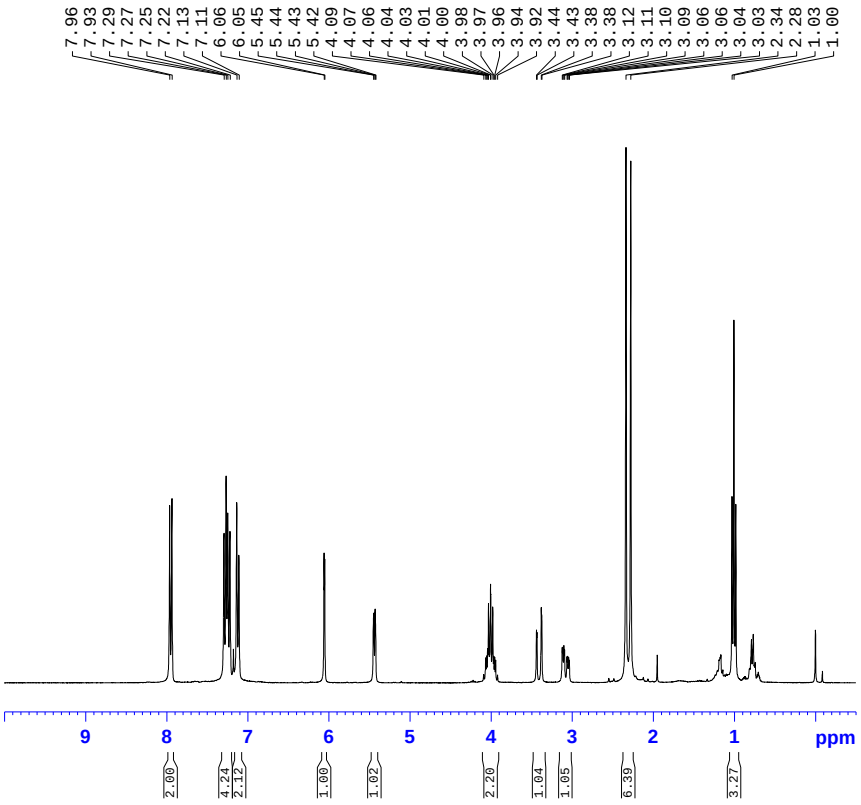

```
NAME     jwq-388a-088g1
EXPNO                10
PROCNO                1
Date_          20121127
Time              12.02
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    3
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ             3.6438515 sec
RG                   64
DW                55.600 usec
DE                 8.00 usec
TE                297.3 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300310 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



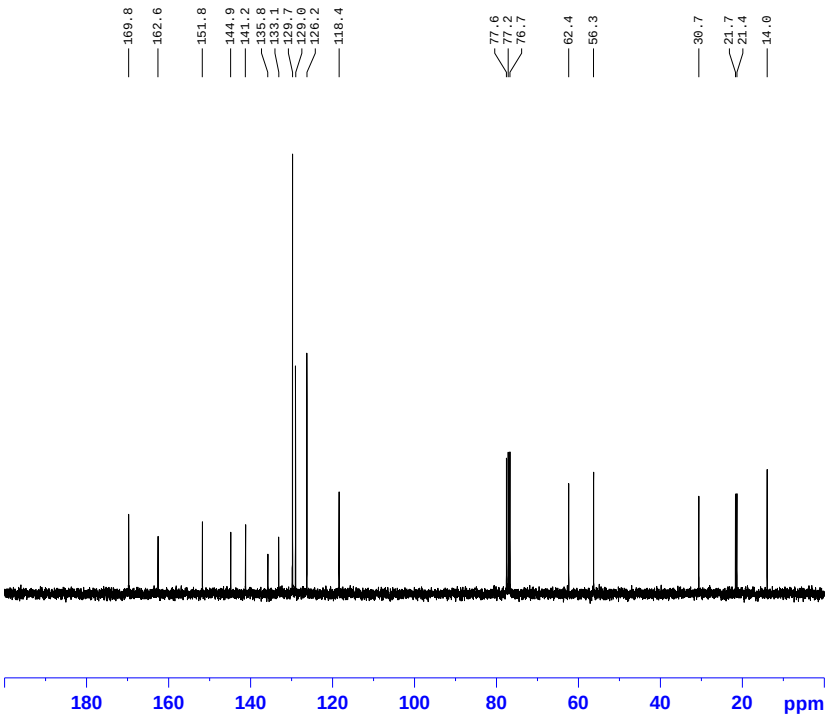

```
NAME     jwq-388a-088g1
EXPNO                11
PROCNO                1
Date_          20121127
Time              12.07
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   96
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                  512
DW                27.800 usec
DE                 6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2        300.1312005 MHz
SI                32768
SF          75.4677474 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



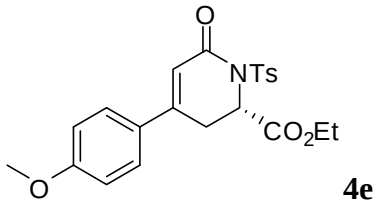
4d



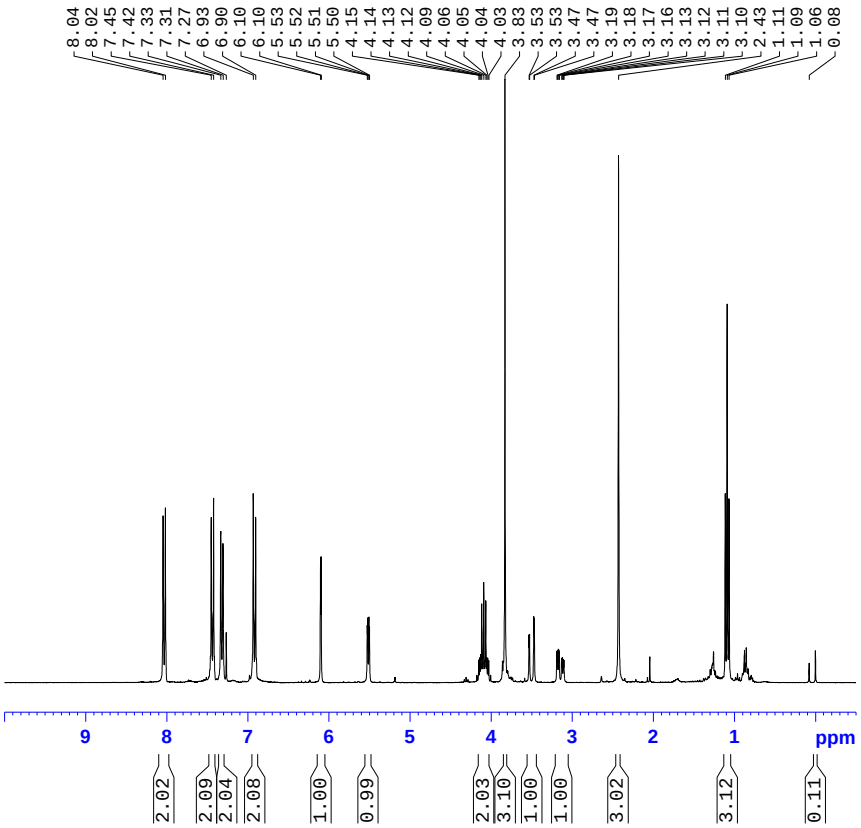

```
NAME      jwq-391c-092g1
EXPNO                 10
PROCNO                 1
Date_           20121203
Time               13.05
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     5
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                    64
DW                55.600 usec
DE                 8.00 usec
TE                295.5 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300310 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```



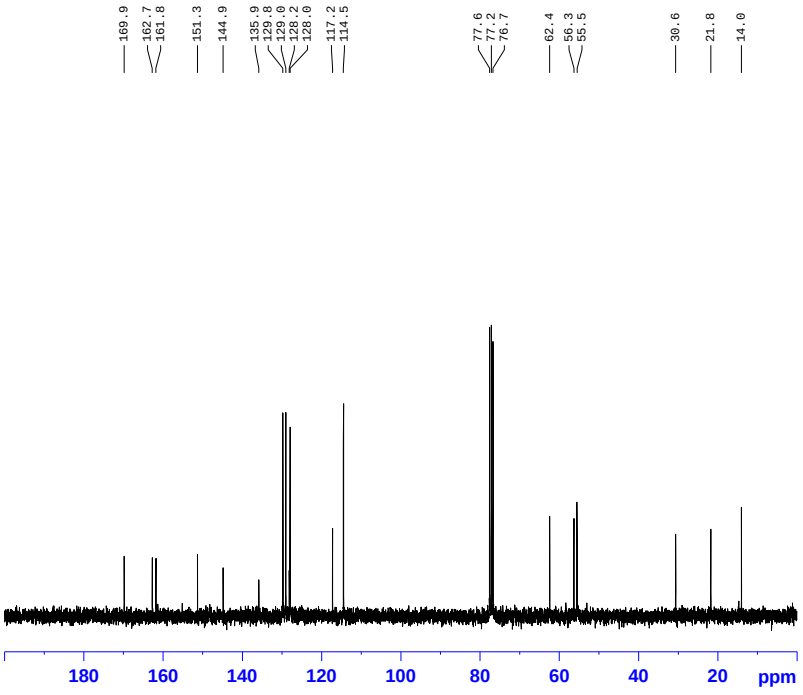

```
NAME      jwq-391c-092g1
EXPNO                 11
PROCNO                 1
Date_           20121203
Time               13.06
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    44
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                1149.4
DW                27.800 usec
DE                 6.50 usec
TE                295.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677470 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```



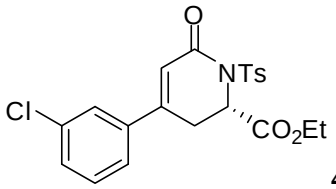
4e



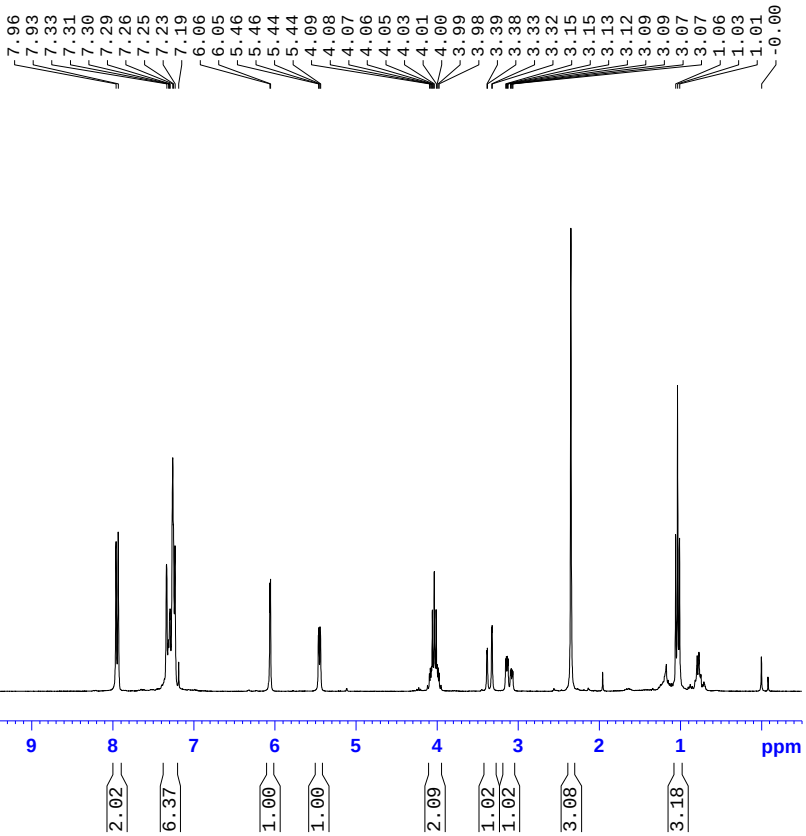

```
NAME      jwq-395a-001g1
EXPNO                 10
PROCNO                 1
Date_           20121206
Time               20.59
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     3
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   128
DW                55.600 usec
DE                 8.00 usec
TE                295.3 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300043 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



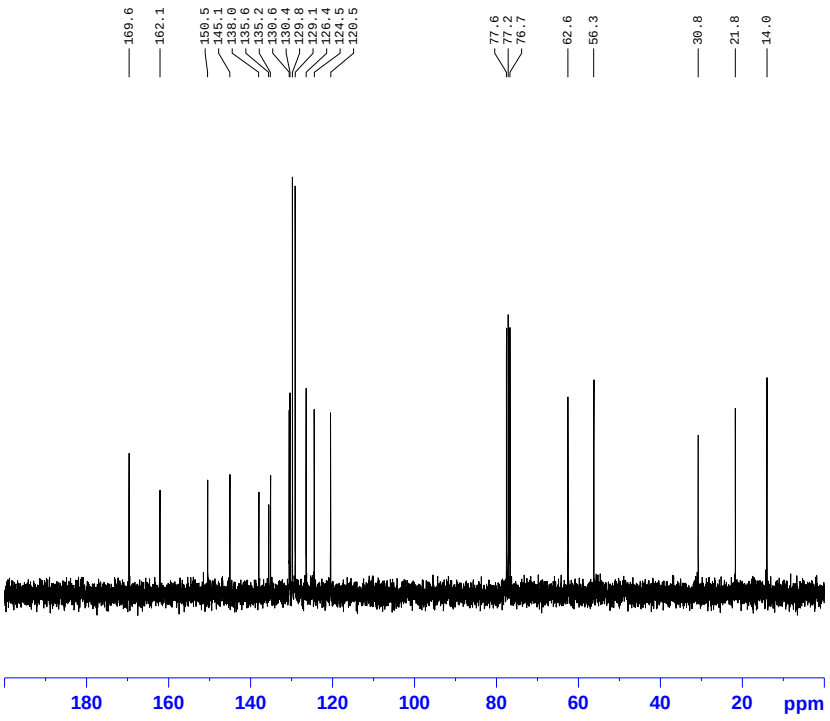

```
NAME      jwq-395a-001g1
EXPNO                 11
PROCNO                 1
Date_           20121206
Time               21.02
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    42
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                  2298.8
DW                 27.800 usec
DE                  6.50 usec
TE                295.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12               22.74 dB
PL13               23.00 dB
SFO2        300.1312005 MHz
SI                 32768
SF           75.4677428 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



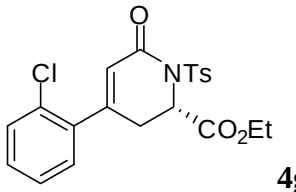
4f



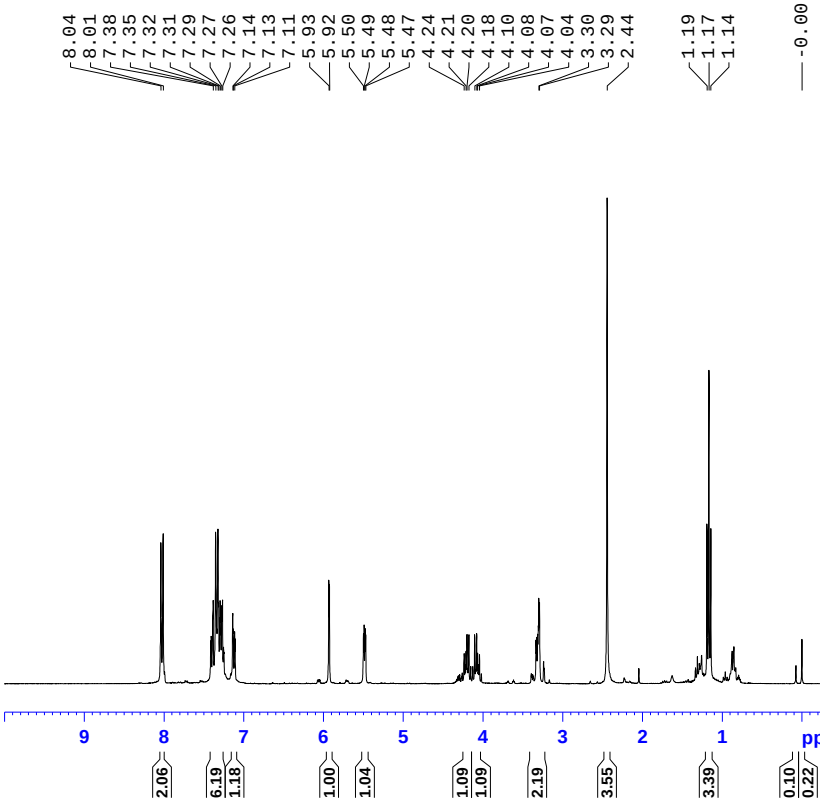

```
NAME      jwq-391a-091g1
EXPNO                 10
PROCNO                 1
Date_           20121203
Time               18.54
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     3
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                  71.8
DW                55.600 usec
DE                 8.00 usec
TE                296.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                 32768
SF          300.1300279 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



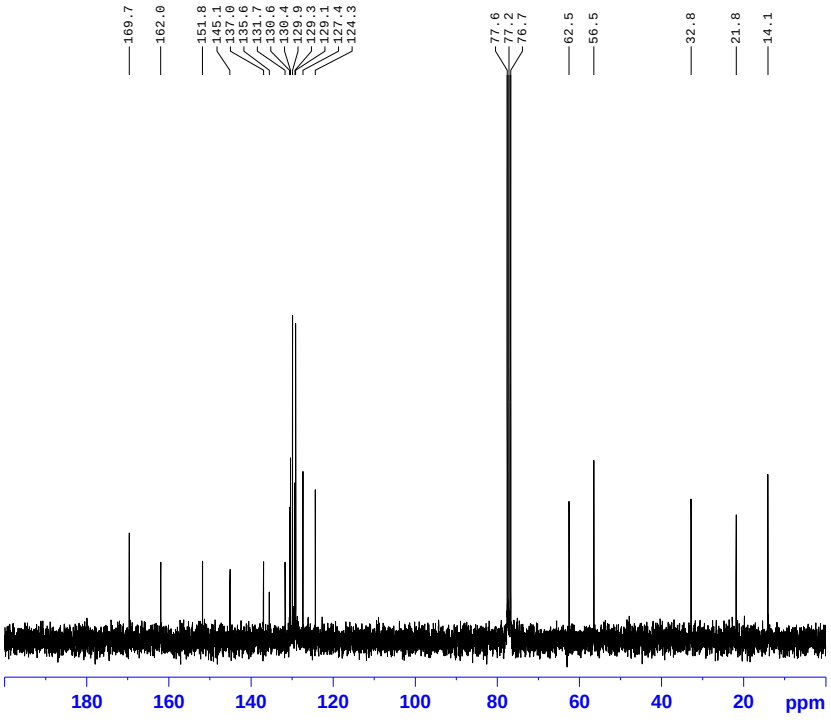

```
NAME      jwq-391a-091g1
EXPNO                 11
PROCNO                 1
Date_           20121203
Time               18.57
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    31
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                2580.3
DW                27.800 usec
DE                 6.50 usec
TE                296.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2        300.1312005 MHz
SI                 32768
SF           75.4677445 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



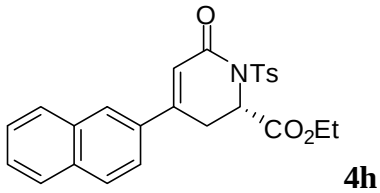
4g



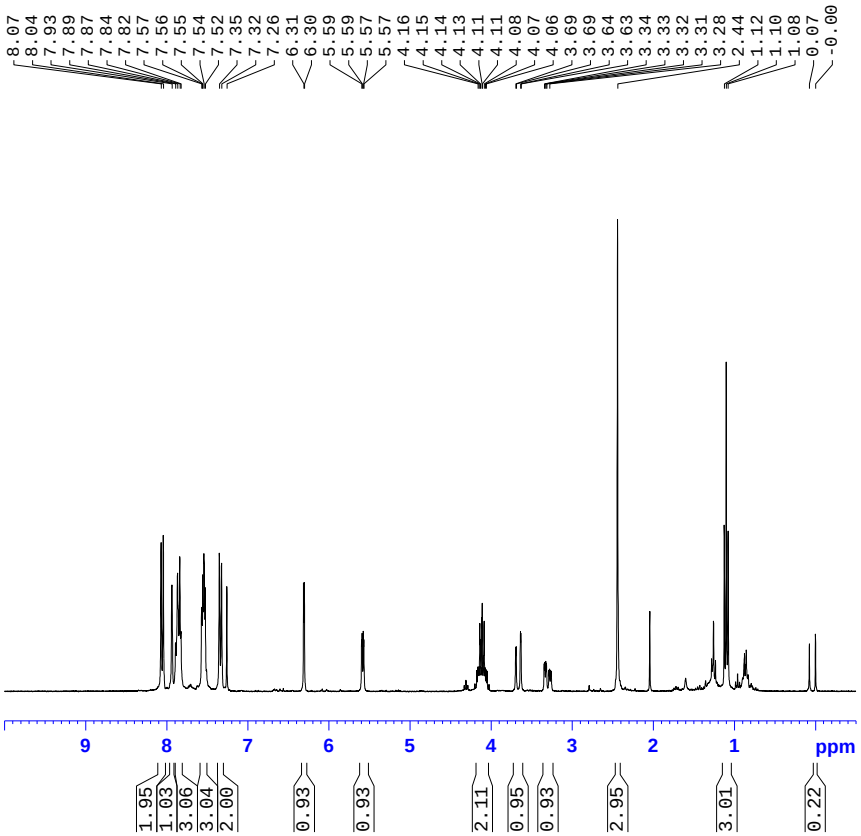

```
NAME          jwq-392c-093g1
EXPNO                     10
PROCNO                     1
Date_               20121203
Time                   18.42
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8992.806 Hz
FIDRES             0.137219 Hz
AQ                 3.6438515 sec
RG                       181
DW                    55.600 usec
DE                     8.00 usec
TE                    296.1 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    10.30 usec
PL1                    3.00 dB
SFO1             300.1324010 MHz
SI                       32768
SF               300.1300047 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



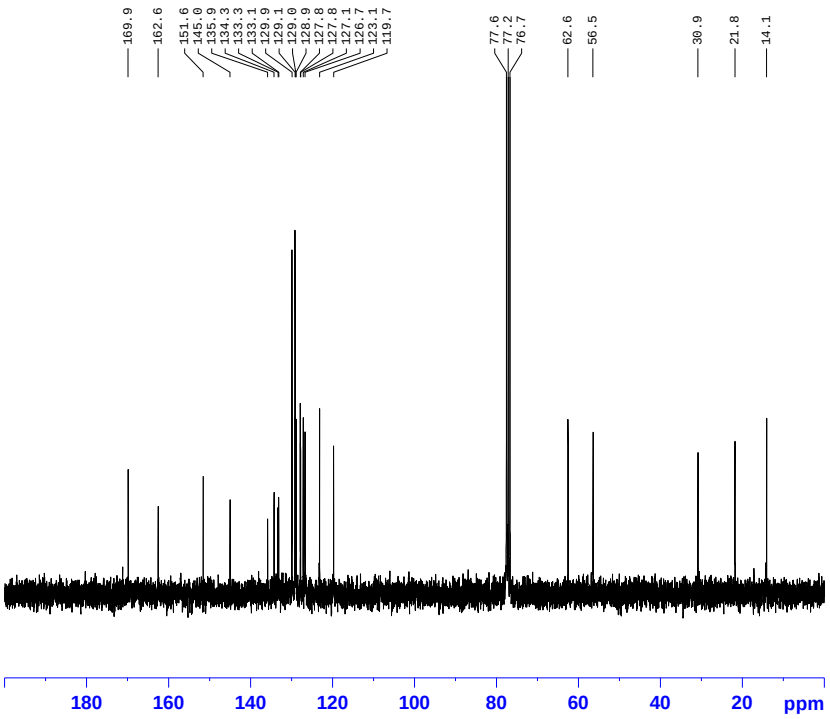

```
NAME          jwq-392c-093g1
EXPNO                     11
PROCNO                     1
Date_               20121203
Time                   18.46
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       88
DS                        4
SWH               17985.611 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                     3251
DW                   27.800 usec
DE                    6.50 usec
TE                    296.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                    12.50 usec
PL1                    2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    3.00 dB
PL12                  22.74 dB
PL13                  23.00 dB
SFO2             300.1312005 MHz
SI                       32768
SF               75.4677403 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```



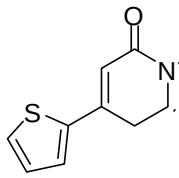
4h



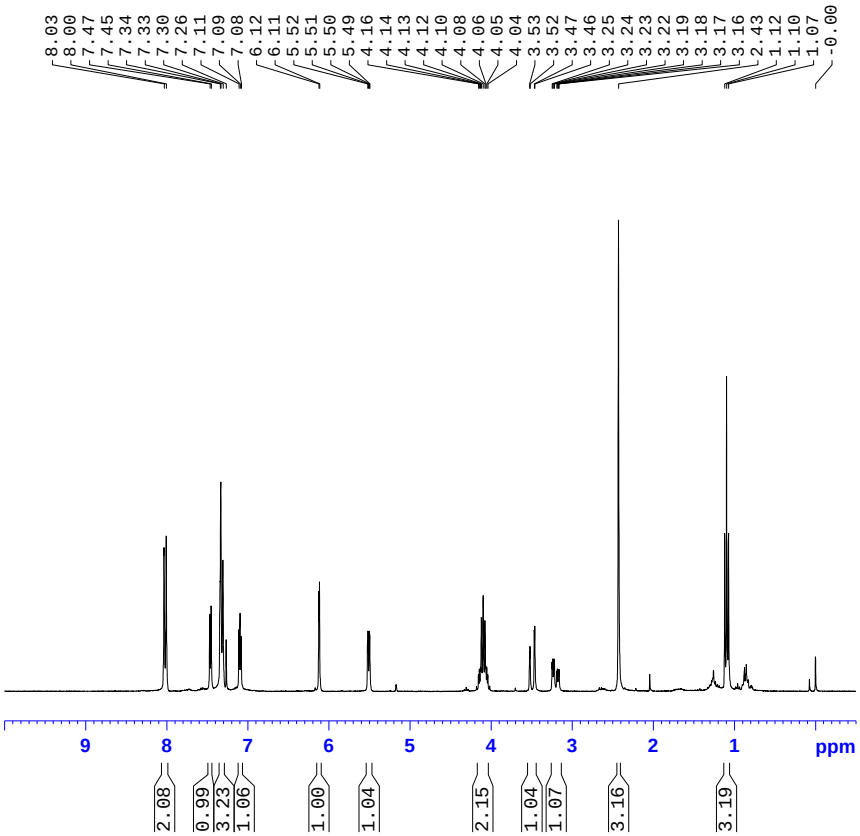

```
NAME      jwq-393d-094g1
EXPNO                 10
PROCNO                 1
Date_           20121204
Time               13.35
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     3
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                 228.1
DW                55.600 usec
DE                 8.00 usec
TE                296.6 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300069 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



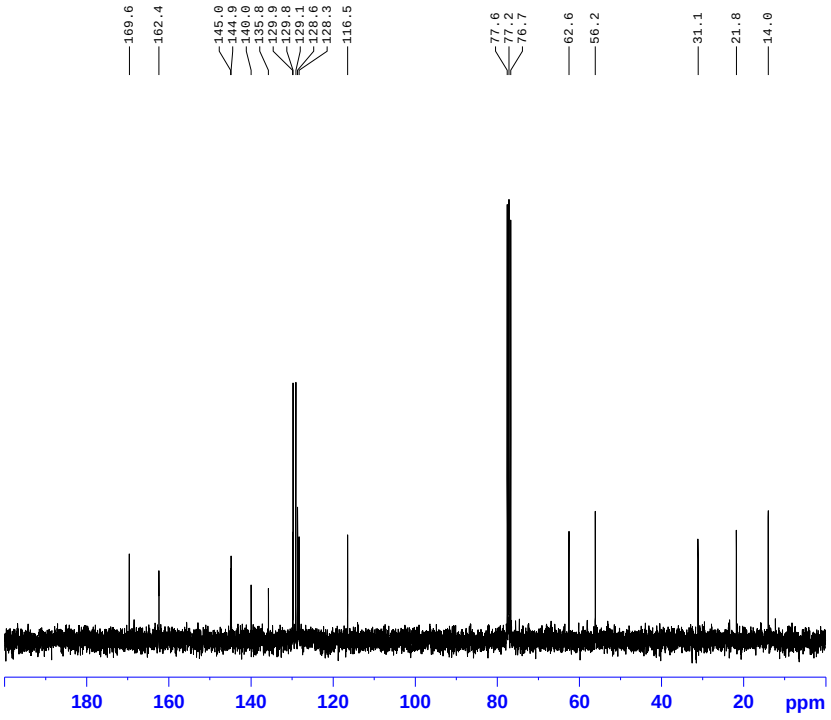

```
NAME      jwq-393d-094g1
EXPNO                 11
PROCNO                 1
Date_           20121204
Time               13.39
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   247
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 645.1
DW                27.800 usec
DE                 6.50 usec
TE                297.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677391 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```



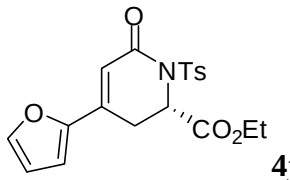
4i



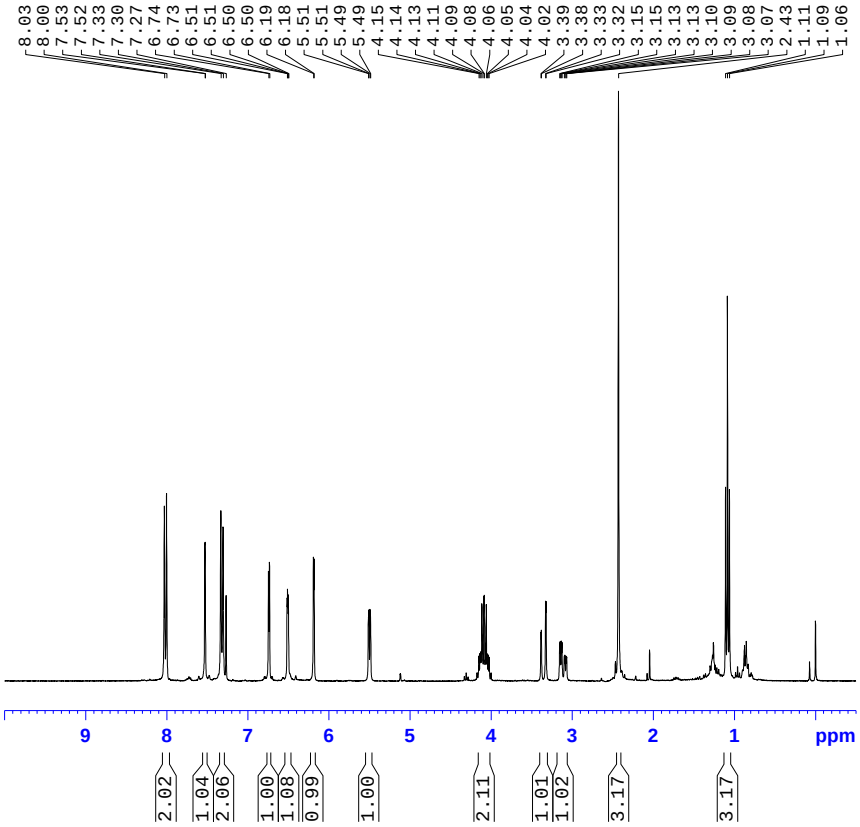

```
NAME      jwq-396a-002g1
EXPNO                 10
PROCNO                 1
Date_           20121206
Time                21.17
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      3
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ               3.6438515 sec
RG                  161.3
DW                 55.600 usec
DE                  8.00 usec
TE                 295.1 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                  10.30 usec
PL1                  3.00 dB
SFO1         300.1324010 MHz
SI                  32768
SF           300.1300047 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



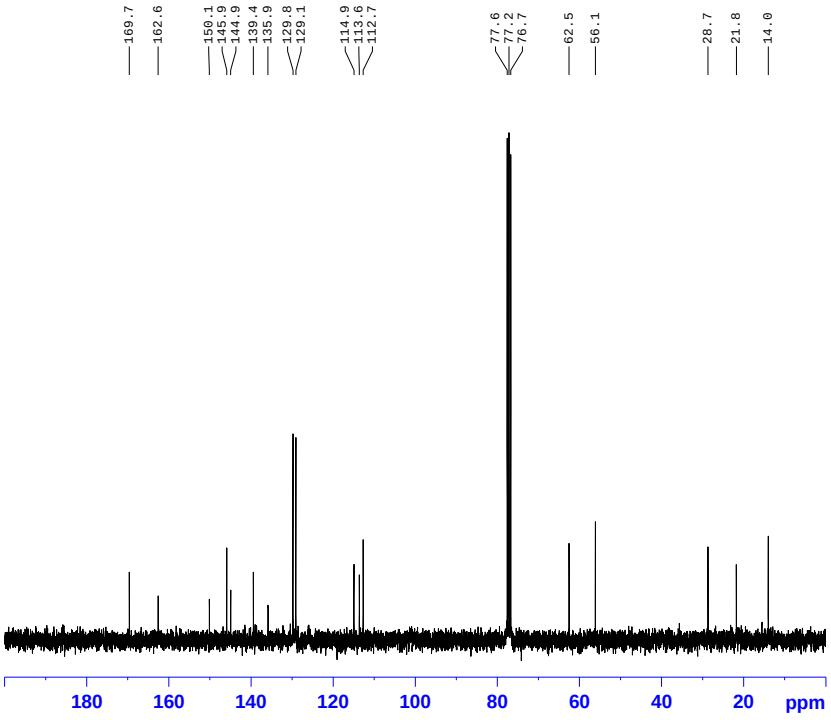

```
NAME      jwq-396a-002g1
EXPNO                 11
PROCNO                 1
Date_           20121206
Time                21.20
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     42
DS                      4
SWH             17985.611 Hz
FIDRES           0.274439 Hz
AQ               1.8219508 sec
RG                    512
DW                 27.800 usec
DE                  6.50 usec
TE                 295.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.50 usec
PL1                  2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                  3.00 dB
PL12                22.74 dB
PL13                23.00 dB
SFO2         300.1312005 MHz
SI                  32768
SF           75.4677417 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



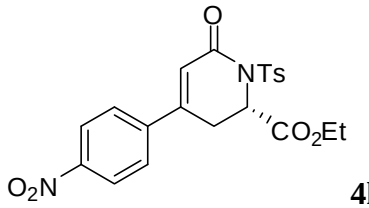
4j



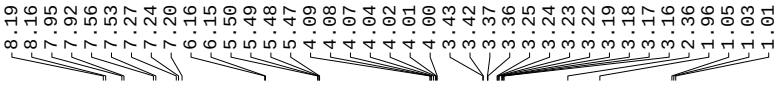

```
NAME      jwq-395c-001g1
EXPNO                 10
PROCNO                 1
Date_           20121206
Time               21.07
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ              3.6438515 sec
RG                   181
DW                55.600 usec
DE                 8.00 usec
TE                295.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300043 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```

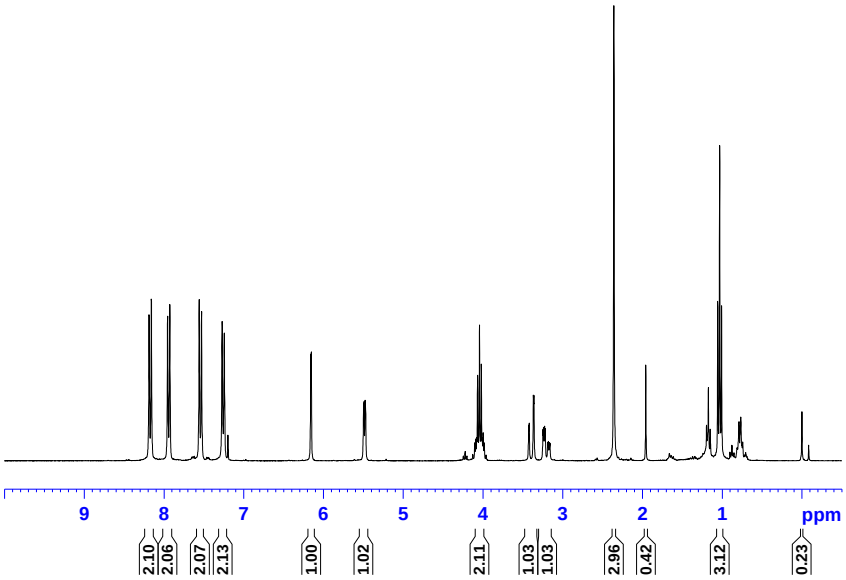



```
NAME      jwq-395c-001g1
EXPNO                 11
PROCNO                 1
Date_           20121206
Time               21.11
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    67
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 645.1
DW                27.800 usec
DE                 6.50 usec
TE                295.7 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677401 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```

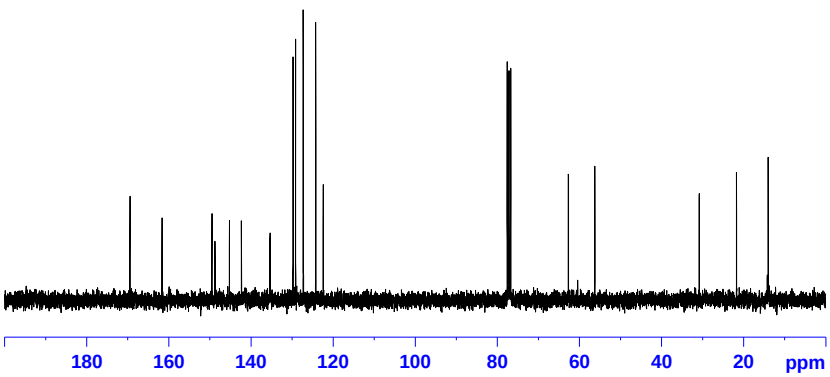


4k

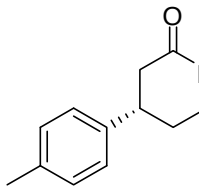




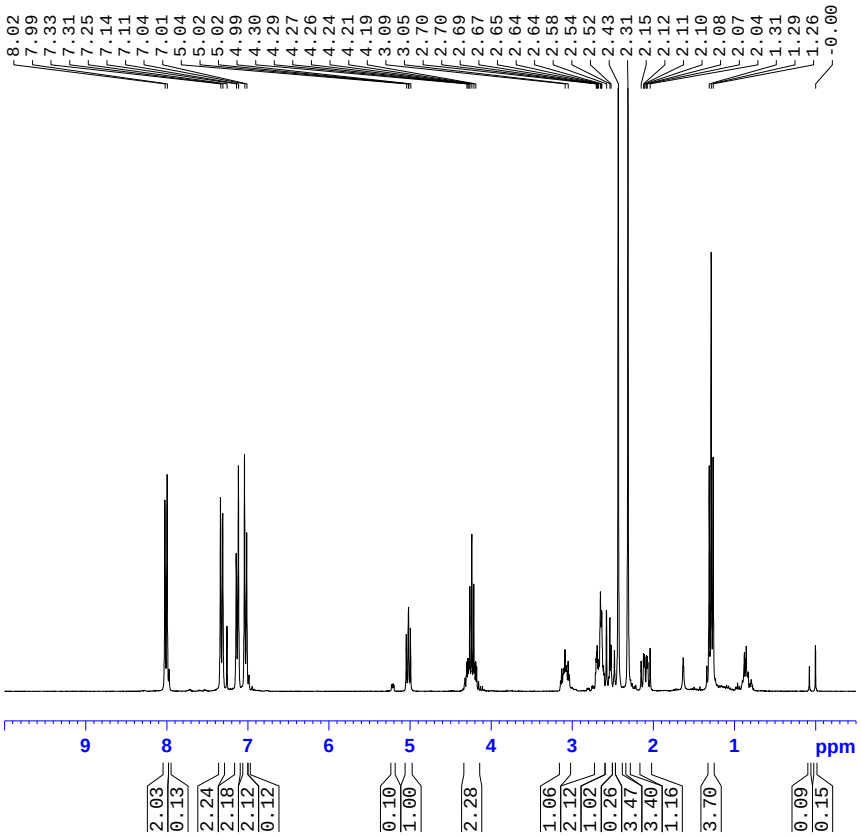

```
NAME     jwq-392a-092g1
EXPNO                10
PROCNO                1
Date_          20121203
Time              19.01
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    3
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ             3.6438515 sec
RG                 71.8
DW                55.600 usec
DE                 8.00 usec
TE                296.2 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300251 MHz
WDW                EM
SSB                 0
LB              0.30 Hz
GB                  0
PC                1.00
```



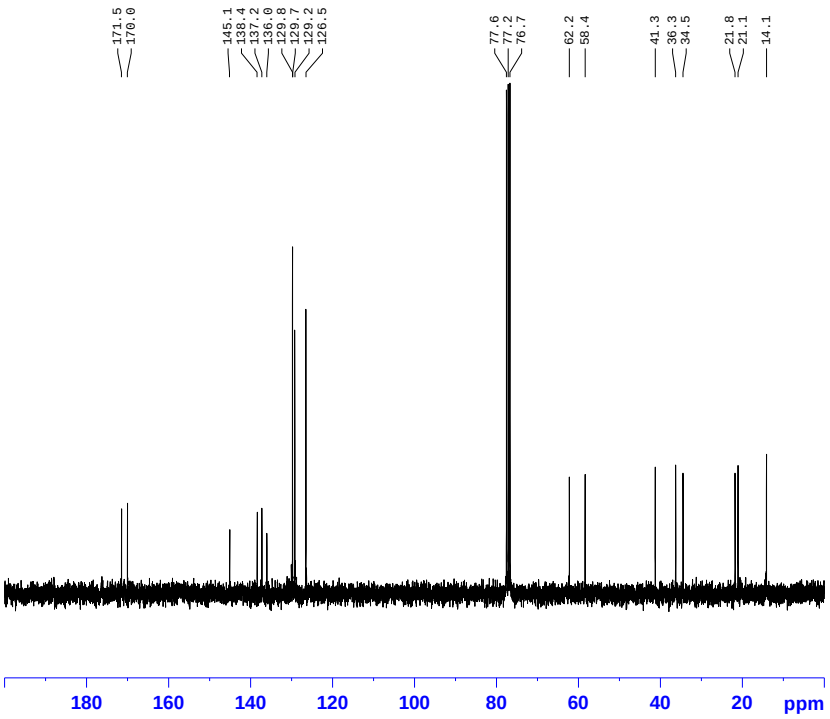

```
NAME     jwq-392a-092g1
EXPNO                11
PROCNO                1
Date_          20121203
Time              19.04
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   28
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                 3251
DW               27.800 usec
DE                 6.50 usec
TE                296.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2        300.1312005 MHz
SI                32768
SF           75.4677450 MHz
WDW                EM
SSB                 0
LB              1.00 Hz
GB                  0
PC                1.40
```



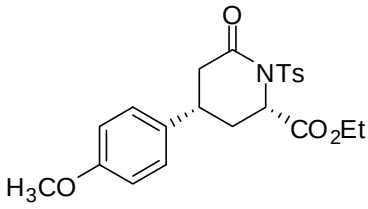
5d



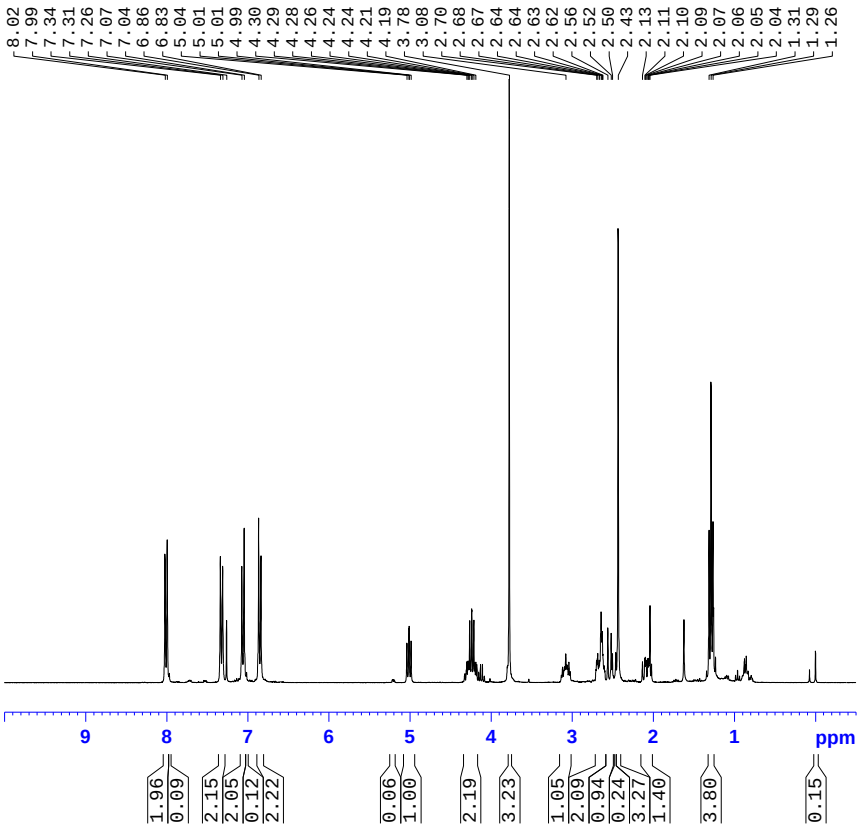

```
NAME      jwq-538b-066g1
EXPNO                 10
PROCNO                 1
Date_           20130724
Time               13.30
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    15
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 161.3
DW                55.600 usec
DE                 6.50 usec
TE                299.6 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1318534 MHz
SI                 32768
SF          300.1300087 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                 1.00
```



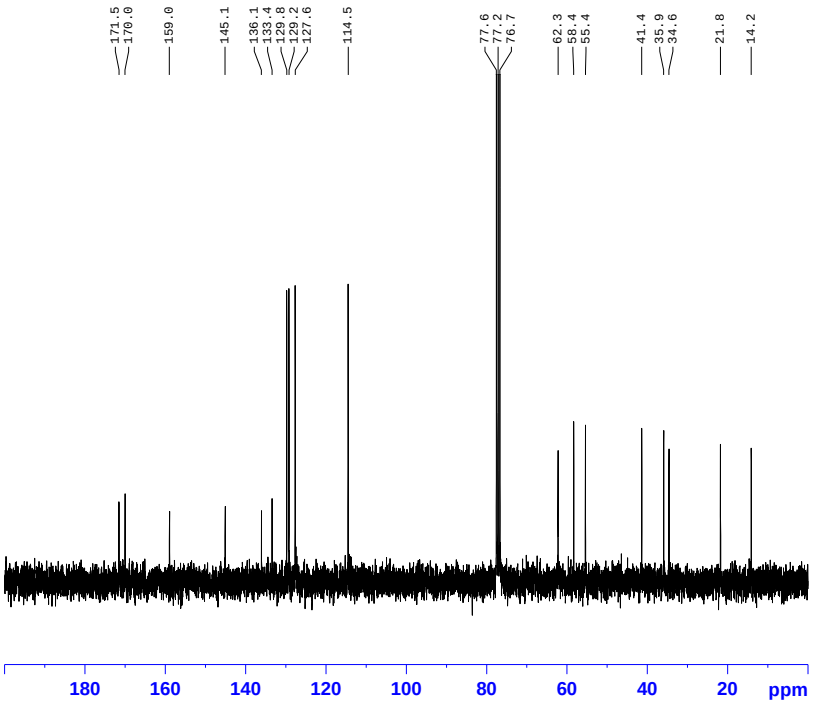

```
NAME      jwq-538b-066g1
EXPNO                 11
PROCNO                 1
Date_           20130724
Time               13.36
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    82
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 362
DW                27.800 usec
DE                 6.50 usec
TE                300.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2        300.1312005 MHz
SI                 32768
SF           75.4677405 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                 1.40
```



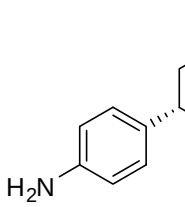
5e



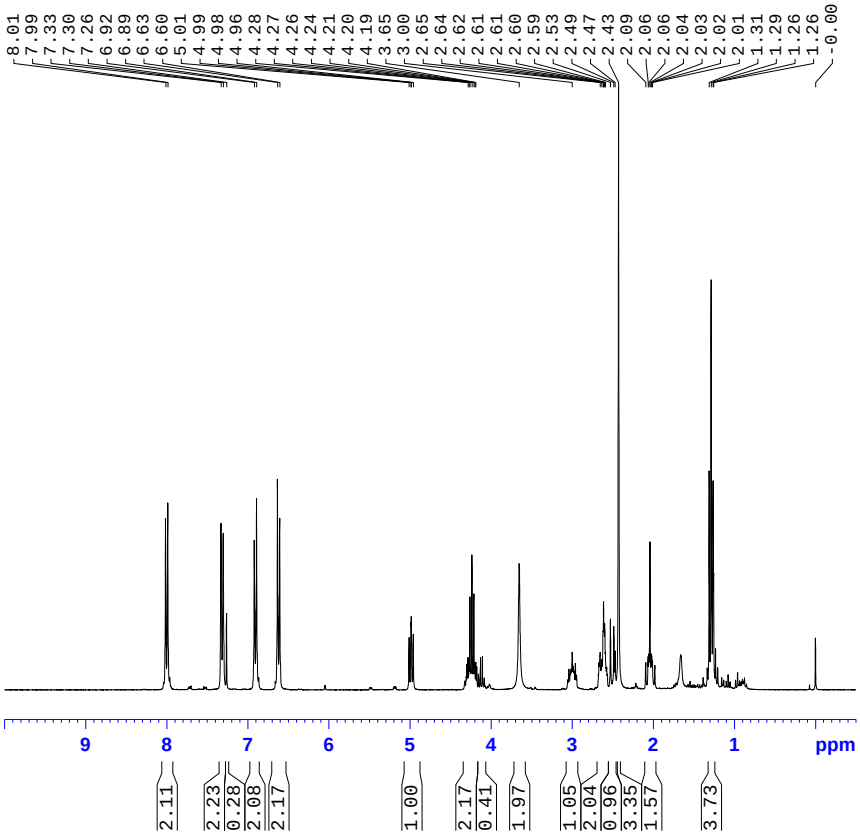

```
NAME      jwq-538c-066g1
EXPNO                 14
PROCNO                 1
Date_           20130724
Time               14.08
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    11
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                   256
DW                55.600 usec
DE                 6.50 usec
TE                299.9 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300072 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



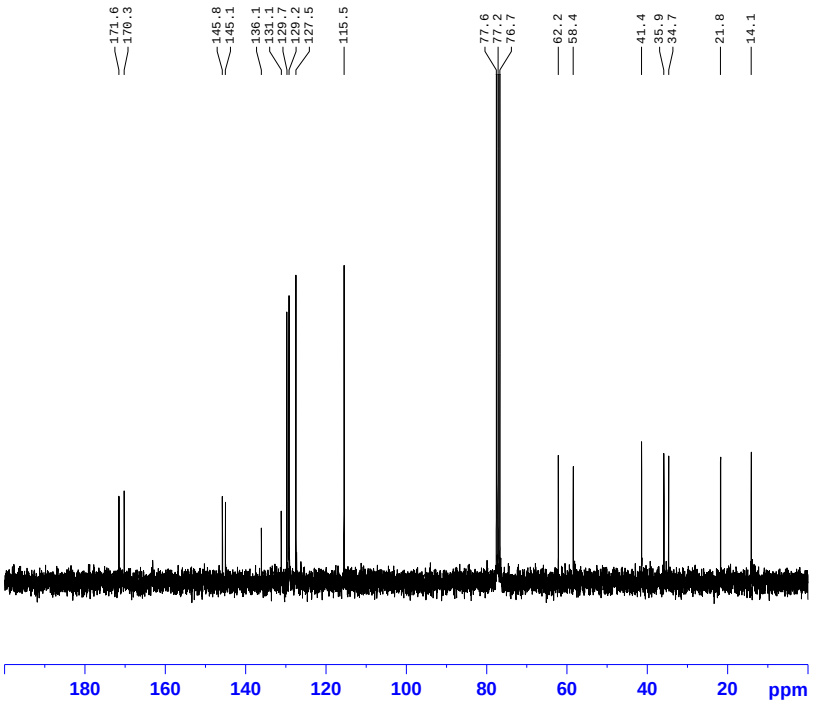

```
NAME      jwq-538c-066g1
EXPNO                 11
PROCNO                 1
Date_           20130724
Time               13.43
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   196
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                 812.7
DW                27.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677397 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



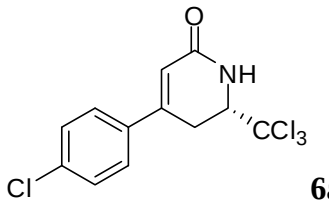
5k



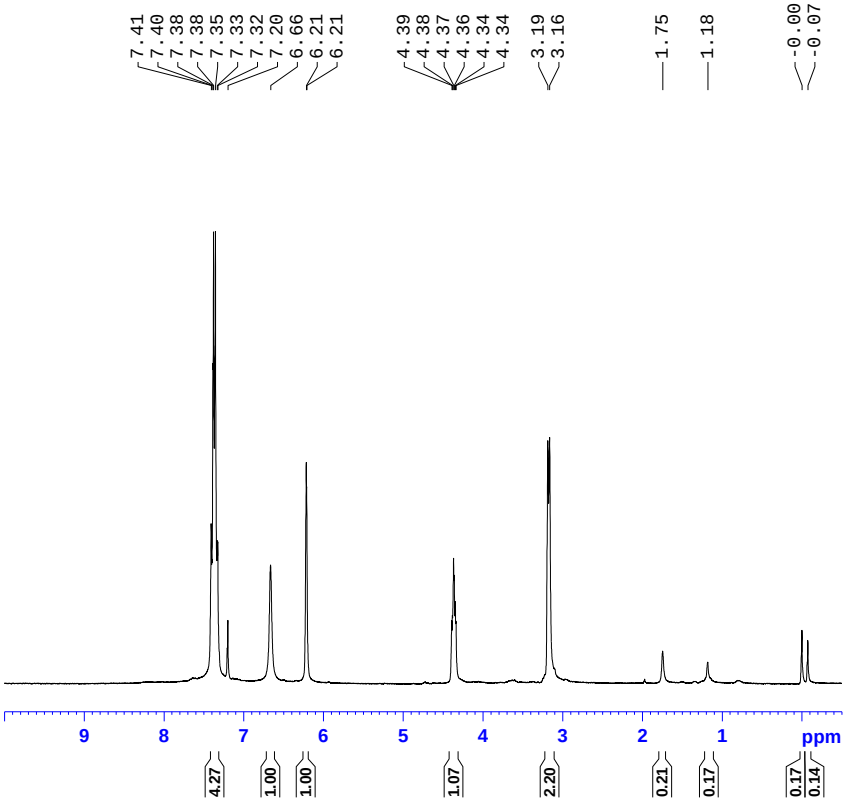

```
NAME          jwq-531b-059g1
EXPNO                 10
PROCNO                 1
Date_           20130718
Time              16.05
INSTRUM           spect
PROBHD    5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   11
DS                    0
SWH            8992.806 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                  128
DW               55.600 usec
DE                6.50 usec
TE                300.4 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1318534 MHz
SI                32768
SF           300.1300071 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



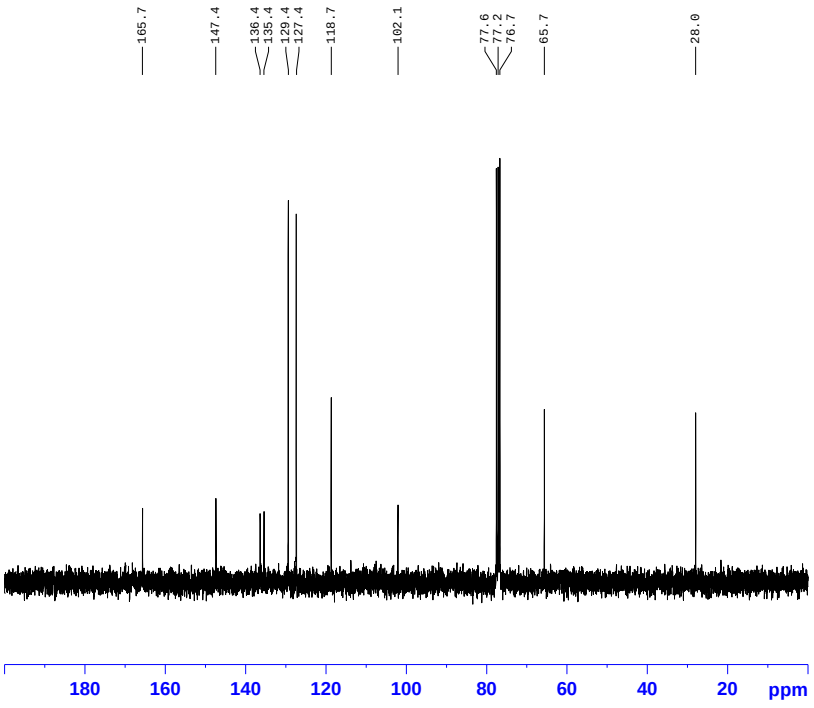

```
NAME          jwq-531b-059g1
EXPNO                 11
PROCNO                 1
Date_           20130718
Time              16.13
INSTRUM           spect
PROBHD    5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  114
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                574.7
DW               27.800 usec
DE                6.50 usec
TE                301.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                32768
SF           75.4677403 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



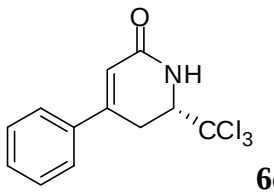
6a



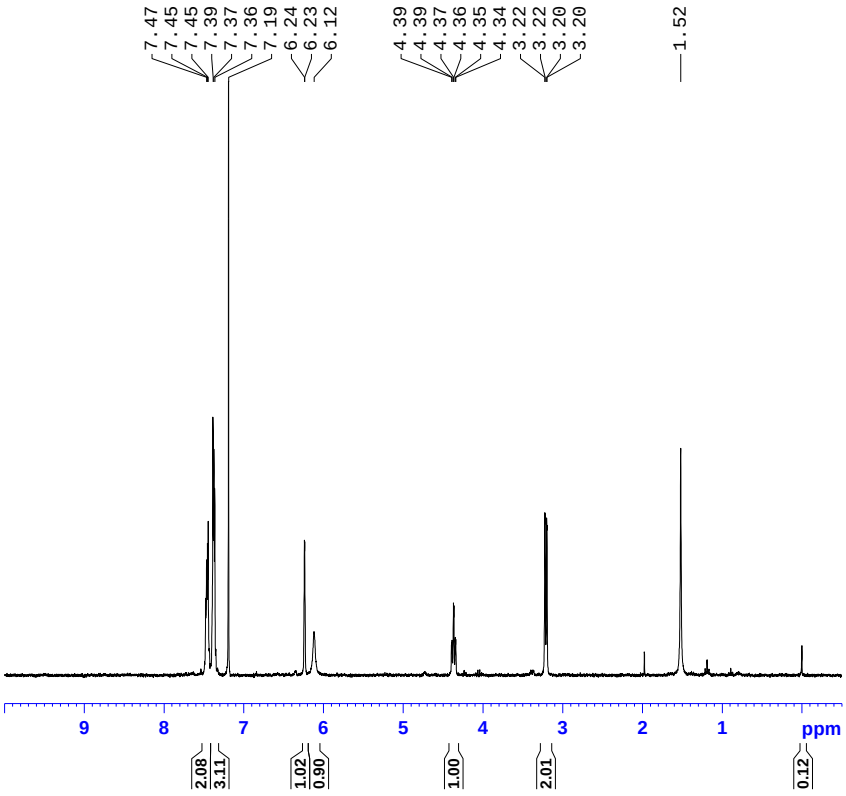

```
NAME     jwq-459c-076rg2
EXPNO                 40
PROCNO                 1
Date_           20130403
Time               19.39
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     7
DS                     0
SWH             6172.839 Hz
FIDRES             0.188380 Hz
AQ              2.6542580 sec
RG                   181
DW                81.000 usec
DE                 6.50 usec
TE                295.8 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300259 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                 1.00
```



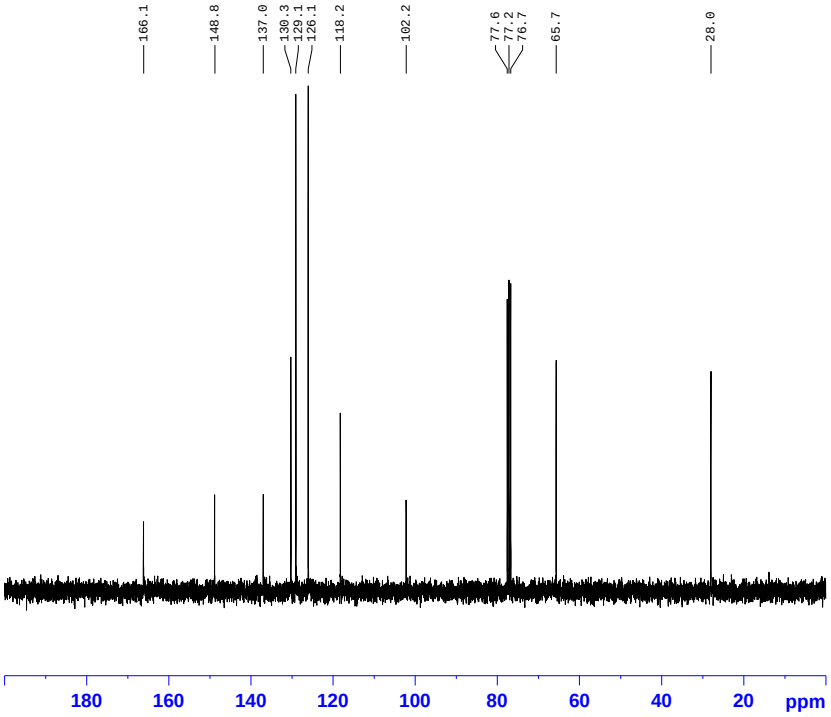

```
NAME     jwq-459c-076rg2
EXPNO                 22
PROCNO                 1
Date_           20130402
Time               11.43
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   101
DS                     4
SWH            17985.611 Hz
FIDRES             0.274439 Hz
AQ              1.8219508 sec
RG                   512
DW                27.800 usec
DE                 6.50 usec
TE                296.2 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677405 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                 1.40
```



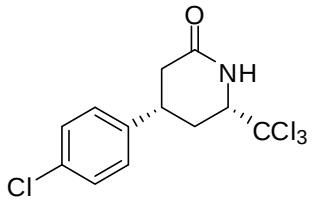
6c



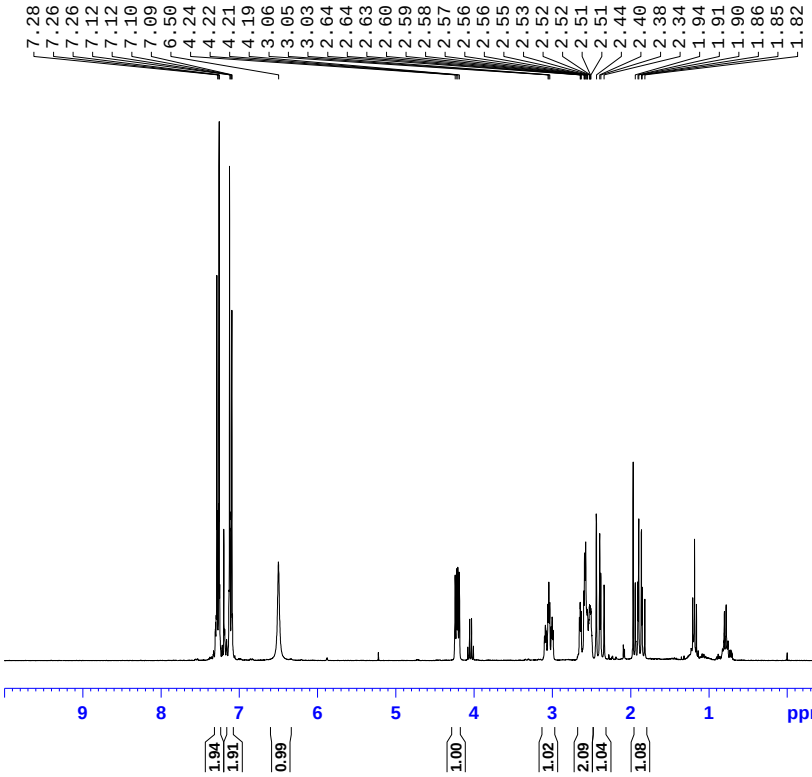

```
NAME     jwq-484a-004g1
EXPNO                10
PROCNO                1
Date_          20130507
Time               7.22
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                    6
DS                    0
SWH            8992.806 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                574.7
DW               55.600 usec
DE                6.50 usec
TE                298.2 K
D1          1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.30 usec
PL1                3.00 dB
SFO1         300.1318534 MHz
SI                32768
SF           300.1300278 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



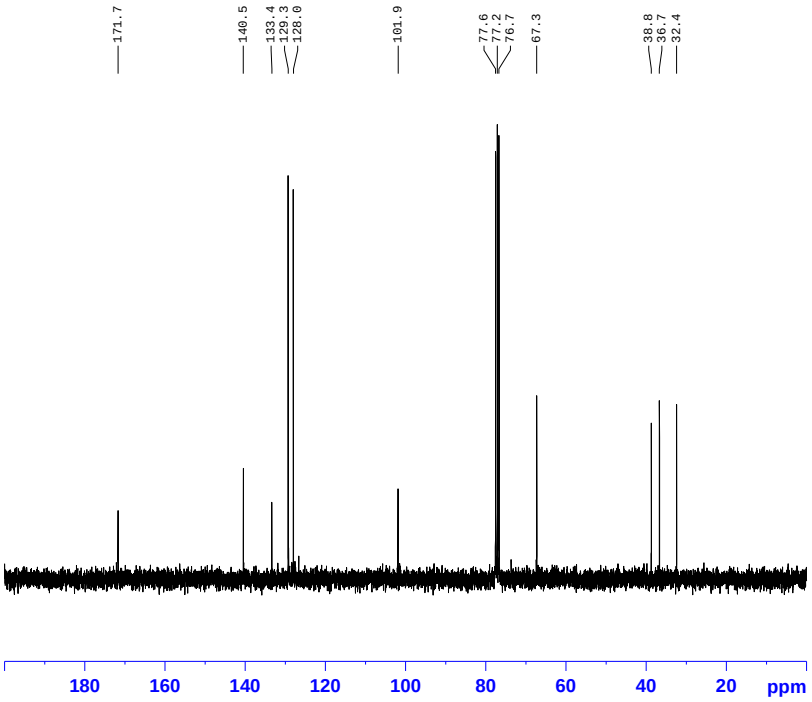

```
NAME     jwq-484b-005g1
EXPNO                11
PROCNO                1
Date_          20130506
Time              12.38
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   34
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ             1.8219508 sec
RG                812.7
DW               27.800 usec
DE                6.50 usec
TE                299.1 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2           100.00 usec
PL2                3.00 dB
PL12               22.74 dB
PL13               23.00 dB
SFO2         300.1312005 MHz
SI                32768
SF           75.4677431 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



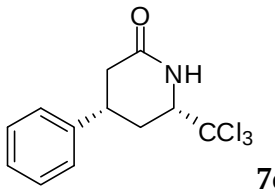
7a



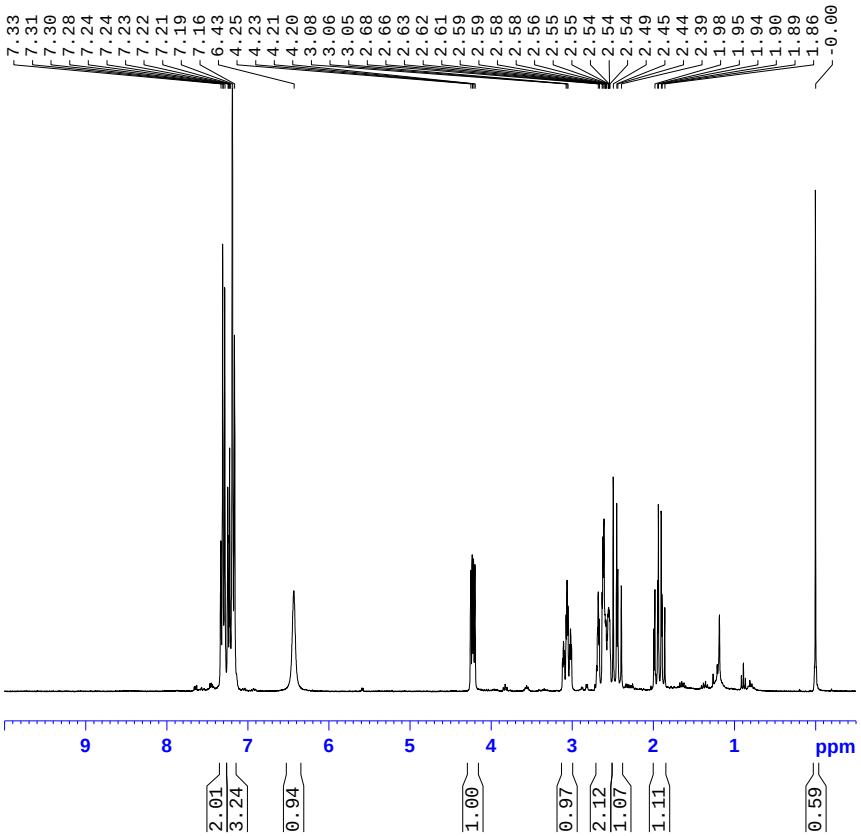

```
NAME     jwq-496a-031rg1
EXPNO                 10
PROCNO                 1
Date_           20130522
Time               18.28
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                     6
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                   128
DW                55.600 usec
DE                 6.50 usec
TE                299.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.30 usec
PL1                 3.00 dB
SFO1         300.1318534 MHz
SI                 32768
SF           300.1300263 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



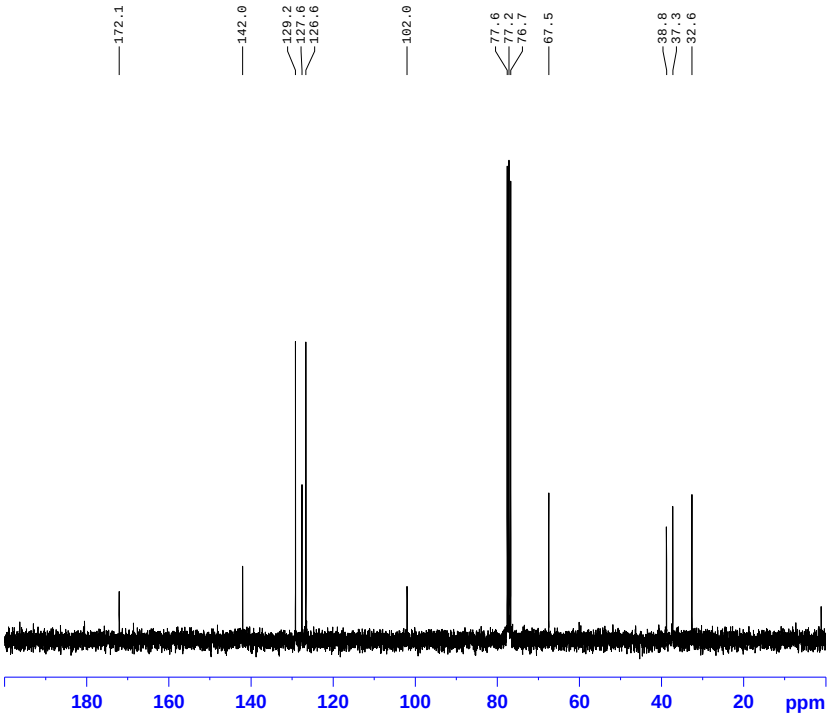

```
NAME     jwq-496a-031rg1
EXPNO                 11
PROCNO                 1
Date_           20130522
Time               18.32
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    47
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 645.1
DW                27.800 usec
DE                 6.50 usec
TE                300.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677407 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



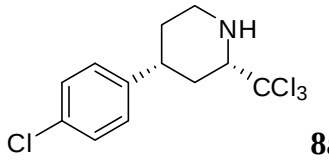
7c



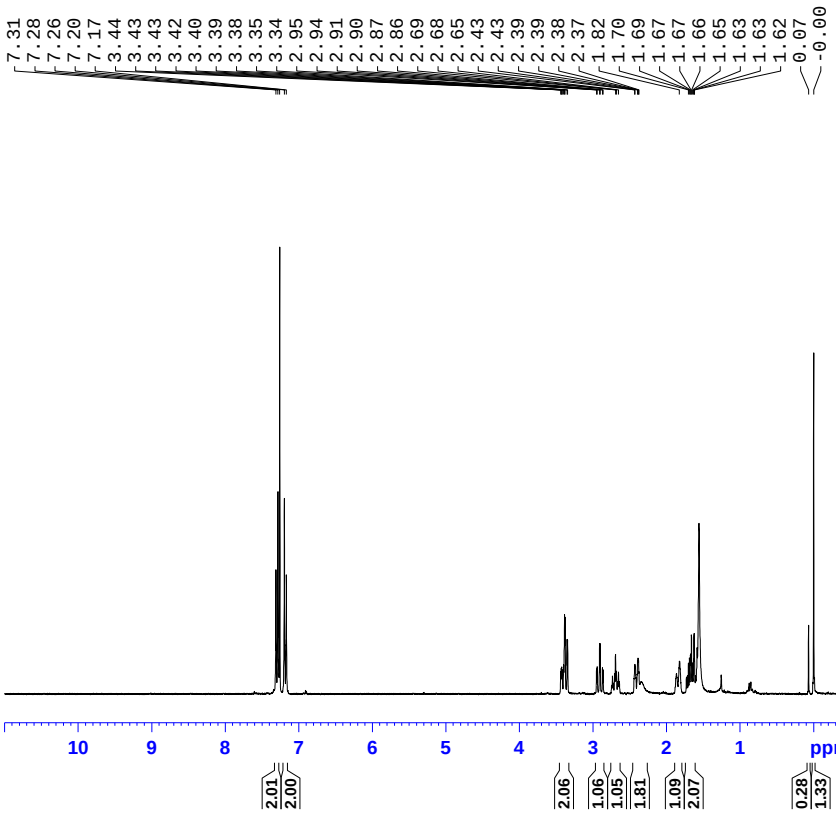

```
NAME          jwq-491c-014w1
EXPNO                     10
PROCNO                     1
Date_               20130513
Time                   14.56
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                        7
DS                        0
SWH                8992.806 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                     228.1
DW                    55.600 usec
DE                     6.50 usec
TE                    298.7 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.30 usec
PL1                     3.00 dB
SFO1            300.1318534 MHz
SI                      32768
SF              300.1300285 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```



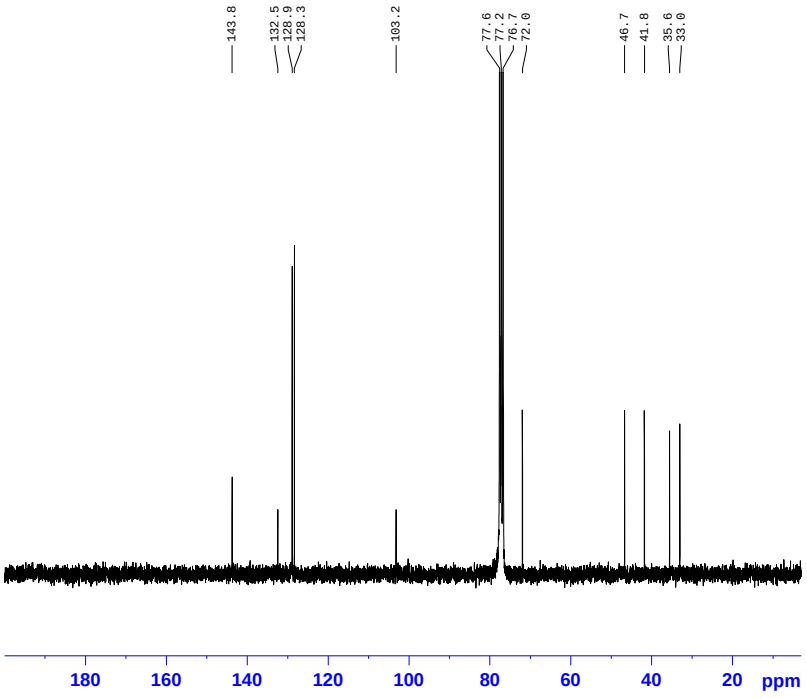

```
NAME          jwq-491c-014w1
EXPNO                     11
PROCNO                     1
Date_               20130513
Time                   14.58
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       76
DS                        4
SWH               17985.611 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                      256
DW                    27.800 usec
DE                     6.50 usec
TE                    299.0 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     13C
P1                     12.50 usec
PL1                     2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                     3.00 dB
PL12                   22.74 dB
PL13                   23.00 dB
SFO2            300.1312005 MHz
SI                      32768
SF              75.4677400 MHz
WDW                        EM
SSB                         0
LB                      1.00 Hz
GB                          0
PC                      1.40
```



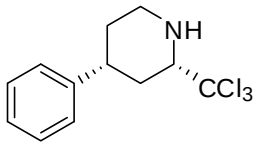
8a



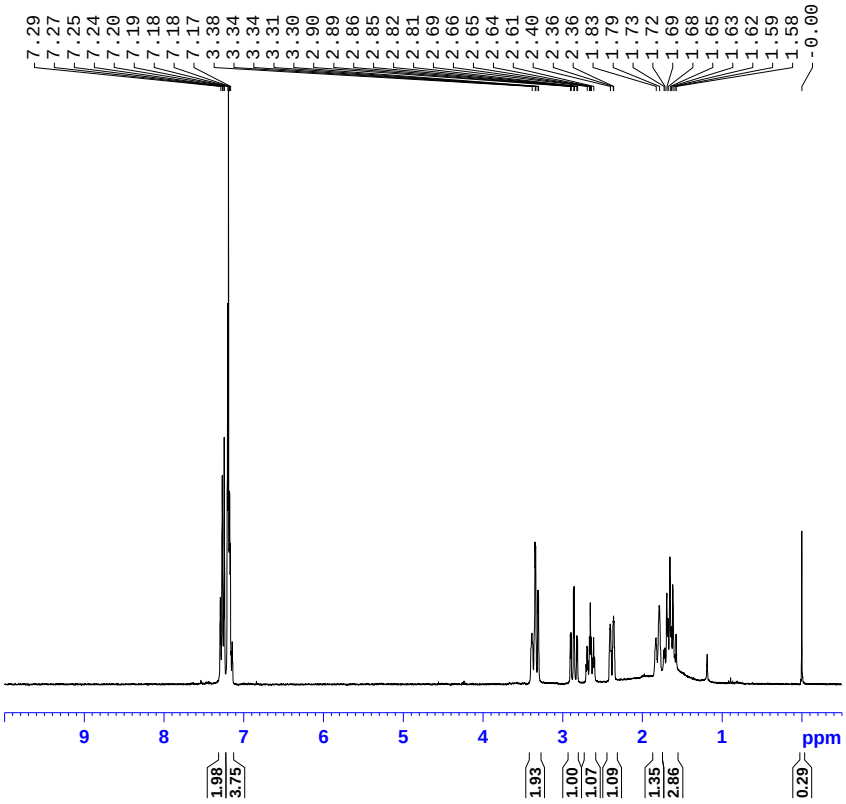

```
NAME      jwq-505b-045g1
EXPNO                 10
PROCNO                 1
Date_           20130605
Time               18.09
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                 456.1
DW                55.600 usec
DE                 6.50 usec
TE                299.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1318534 MHz
SI                 32768
SF          300.1300069 MHz
WDW                   EM
SSB                    0
LB                 0.30 Hz
GB                     0
PC                  1.00
```



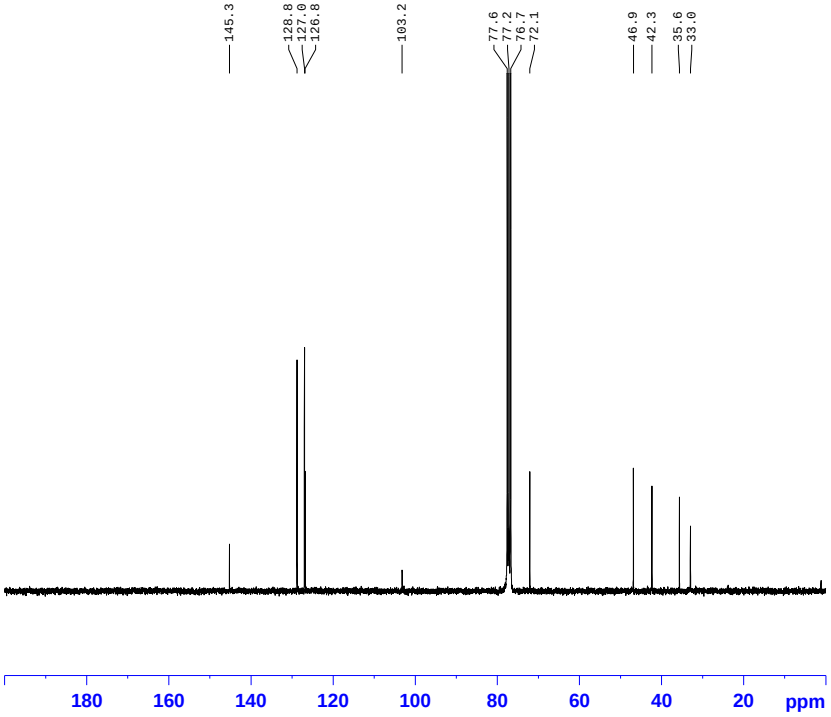

```
NAME      jwq-505b-045g1
EXPNO                 40
PROCNO                 1
Date_           20130605
Time               23.32
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  7822
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                 645.1
DW                27.800 usec
DE                 6.50 usec
TE                299.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2        300.1312005 MHz
SI                 32768
SF          75.4677364 MHz
WDW                   EM
SSB                    0
LB                 1.00 Hz
GB                     0
PC                  1.40
```



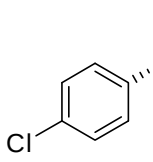
8c



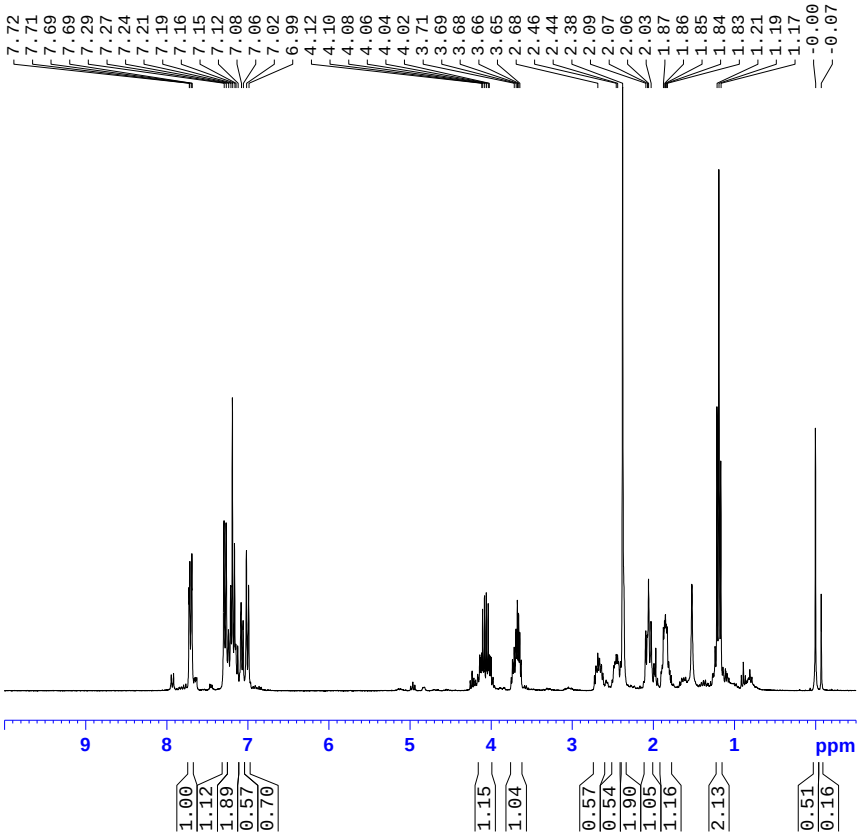

```
NAME     jwq-502b-029g1
EXPNO                10
PROCNO                1
Date_          20130521
Time              12.10
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    12
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                 456.1
DW                55.600 usec
DE                 6.50 usec
TE                299.1 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1318534 MHz
SI                 32768
SF          300.1300282 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



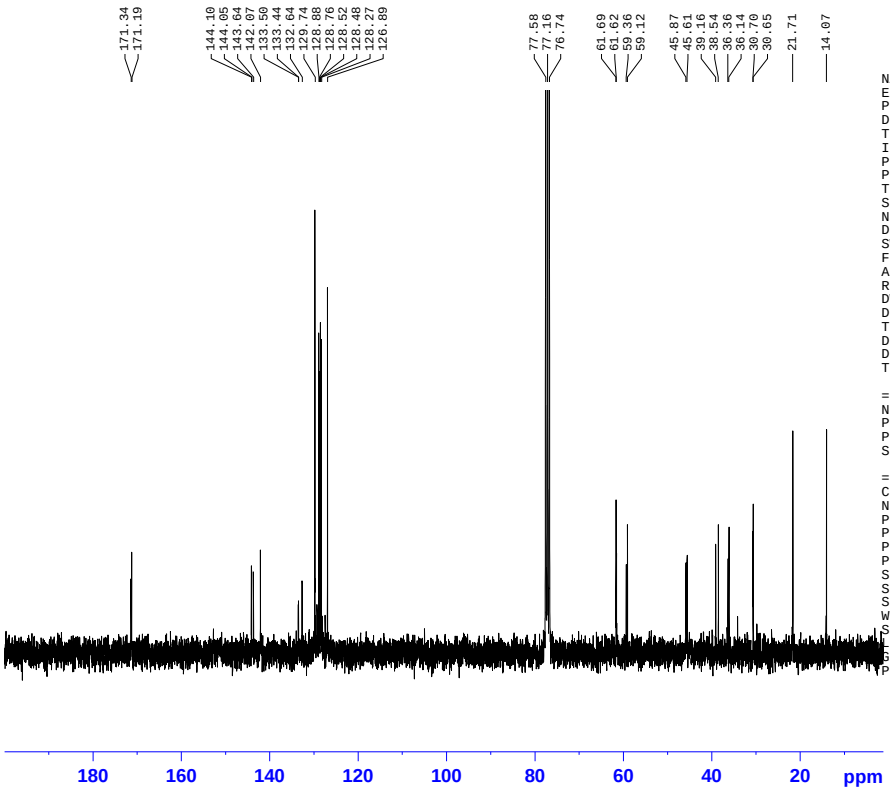

```
NAME     jwq-502b-029g1
EXPNO                20
PROCNO                1
Date_          20130523
Time               7.22
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  7556
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                 812.7
DW                27.800 usec
DE                 6.50 usec
TE                301.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677365 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```



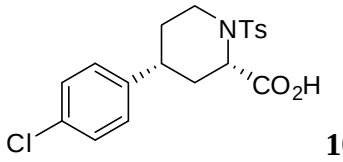
9a



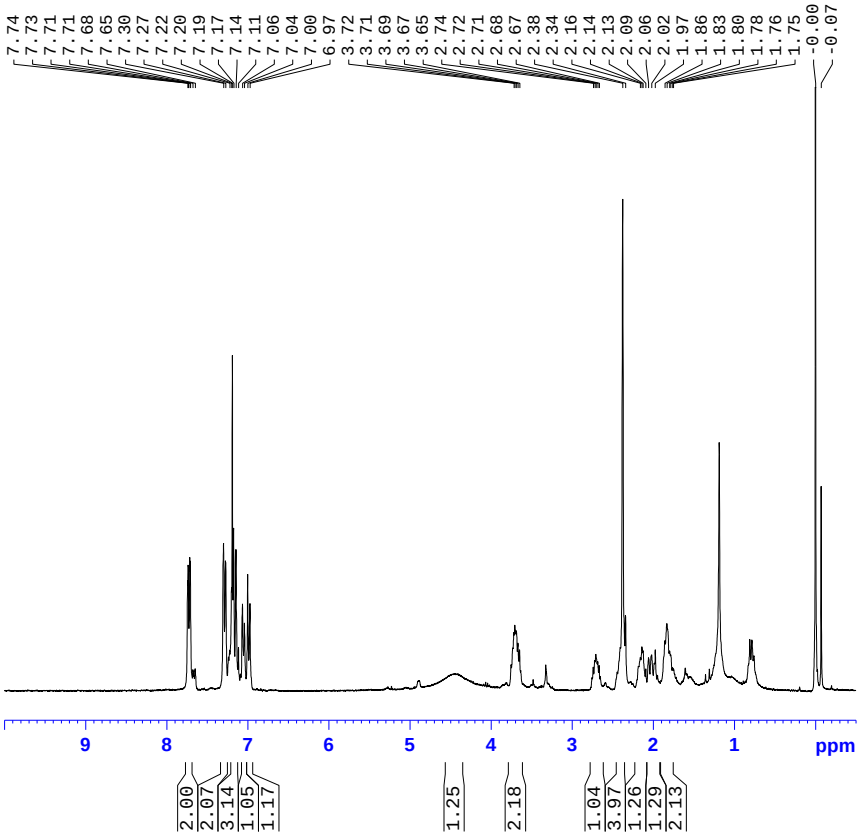

```
NAME          jwq-547b-074g1
EXPNO                     10
PROCNO                     1
Date_               20130730
Time                  17.04
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8992.806 Hz
FIDRES             0.274439 Hz
AQ                1.8219508 sec
RG                      256
DW                   55.600 usec
DE                    6.50 usec
TE                   301.3 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    10.30 usec
PL1                    3.00 dB
SFO1             300.1318534 MHz
SI                    32768
SF               300.1300288 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



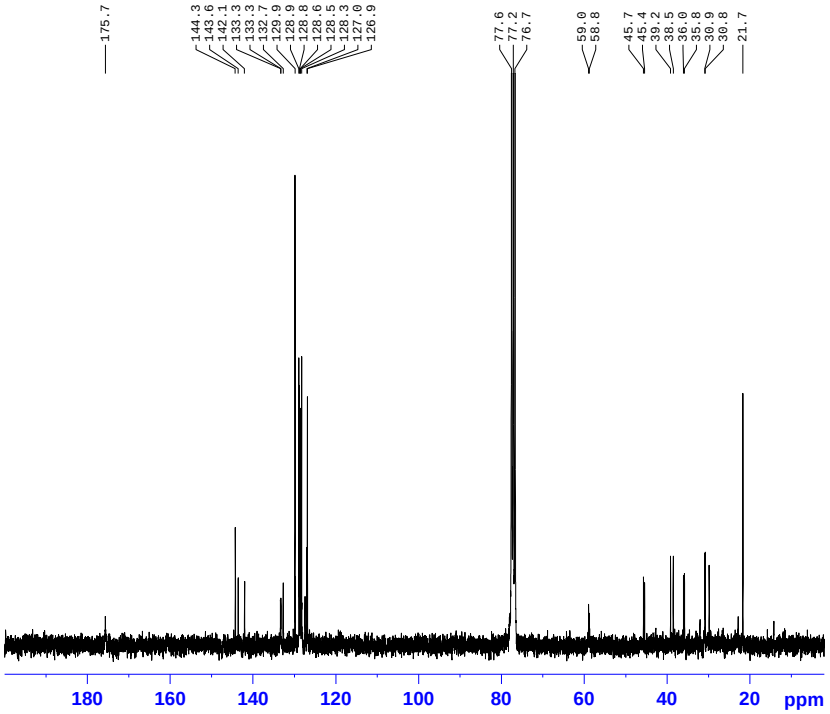

```
NAME          jwq-547b-074g1
EXPNO                     20
PROCNO                     1
Date_               20130801
Time                  12.17
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      688
DS                        4
SWH               17985.611 Hz
FIDRES             0.274439 Hz
AQ                1.8219508 sec
RG                      256
DW                   27.800 usec
DE                    6.50 usec
TE                   299.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    12.50 usec
PL1                    2.00 dB
SFO1             75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                    3.00 dB
PL12                  22.74 dB
PL13                  23.00 dB
SFO2             300.1312005 MHz
SI                    32768
SF               75.4677380 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```



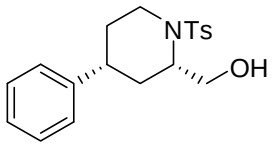
10a



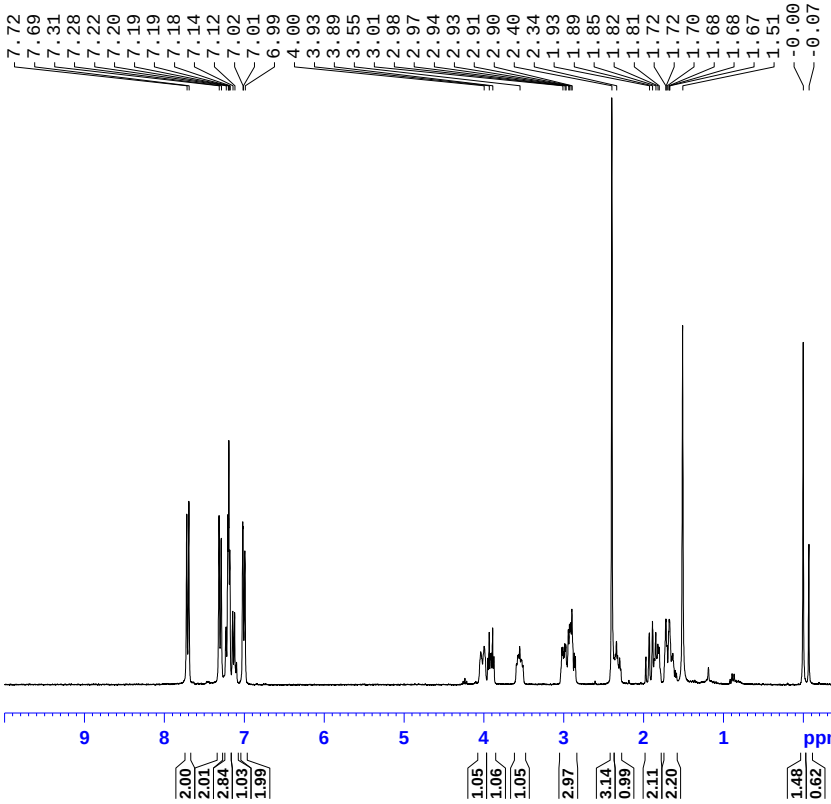

```
NAME      jwq-549a-075cg1
EXPNO                  10
PROCNO                  1
Date_            20130803
Time                12.10
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG              zg30
TD                  32768
SOLVENT             CDCl3
NS                     16
DS                      0
SWH              8992.806 Hz
FIDRES           0.274439 Hz
AQ             1.8219508 sec
RG                 456.1
DW                55.600 usec
DE                 6.50 usec
TE                 300.7 K
D1            1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                 10.30 usec
PL1                 3.00 dB
SFO1          300.1318534 MHz
SI                  32768
SF            300.1300287 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



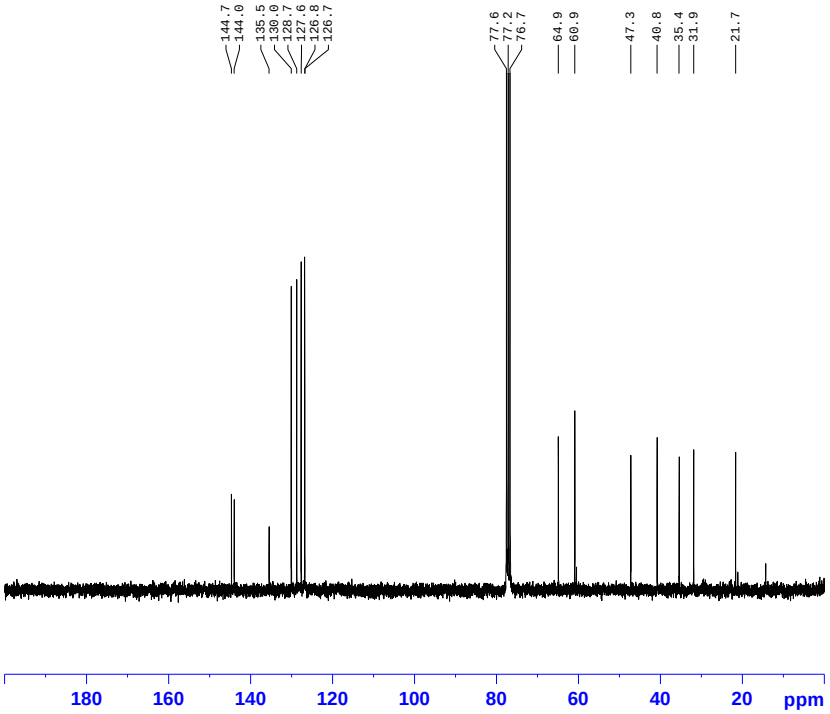

```
NAME      jwq-549a-075cg1
EXPNO                  21
PROCNO                  1
Date_            20130815
Time                8.02
INSTRUM             spect
PROBHD    5 mm DUL 13C-1
PULPROG          zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   8378
DS                      4
SWH             17985.611 Hz
FIDRES           0.274439 Hz
AQ             1.8219508 sec
RG                    512
DW                27.800 usec
DE                 6.50 usec
TE                 303.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2             100.00 usec
PL2                 3.00 dB
PL12                22.74 dB
PL13                23.00 dB
SFO2          300.1312005 MHz
SI                  32768
SF            75.4677363 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```



11c

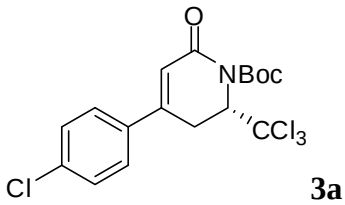



```
NAME      jwq-546b-072g1
EXPNO                 30
PROCNO                 1
Date_           20130730
Time               18.31
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    13
DS                     0
SWH             8992.806 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 456.1
DW                55.600 usec
DE                 6.50 usec
TE                301.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.30 usec
PL1                3.00 dB
SFO1        300.1318534 MHz
SI                 32768
SF          300.1300285 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```

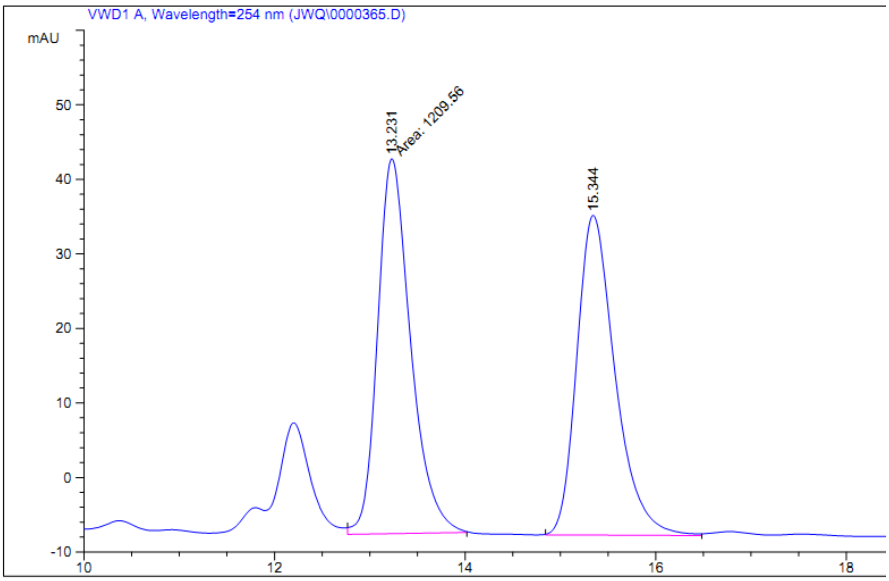


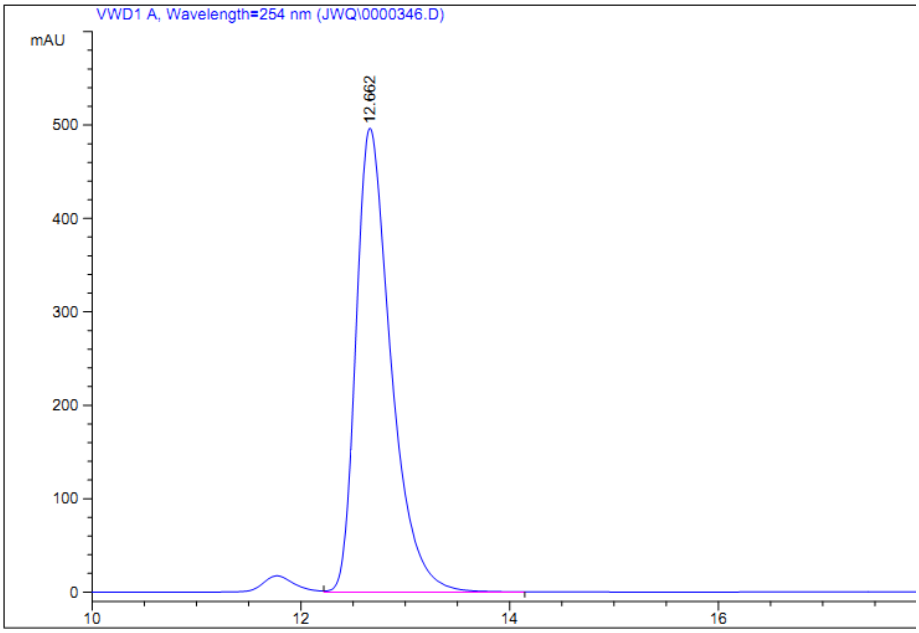

```
NAME      jwq-546b-072g1
EXPNO                 11
PROCNO                 1
Date_           20130729
Time               12.43
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   699
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ              1.8219508 sec
RG                 456.1
DW                27.800 usec
DE                 6.50 usec
TE                302.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2            100.00 usec
PL2                3.00 dB
PL12              22.74 dB
PL13              23.00 dB
SFO2         300.1312005 MHz
SI                 32768
SF           75.4677375 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

Part III HPLC Spectra

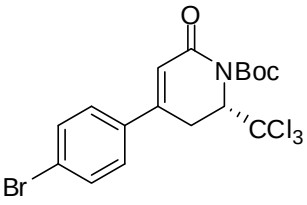


3a

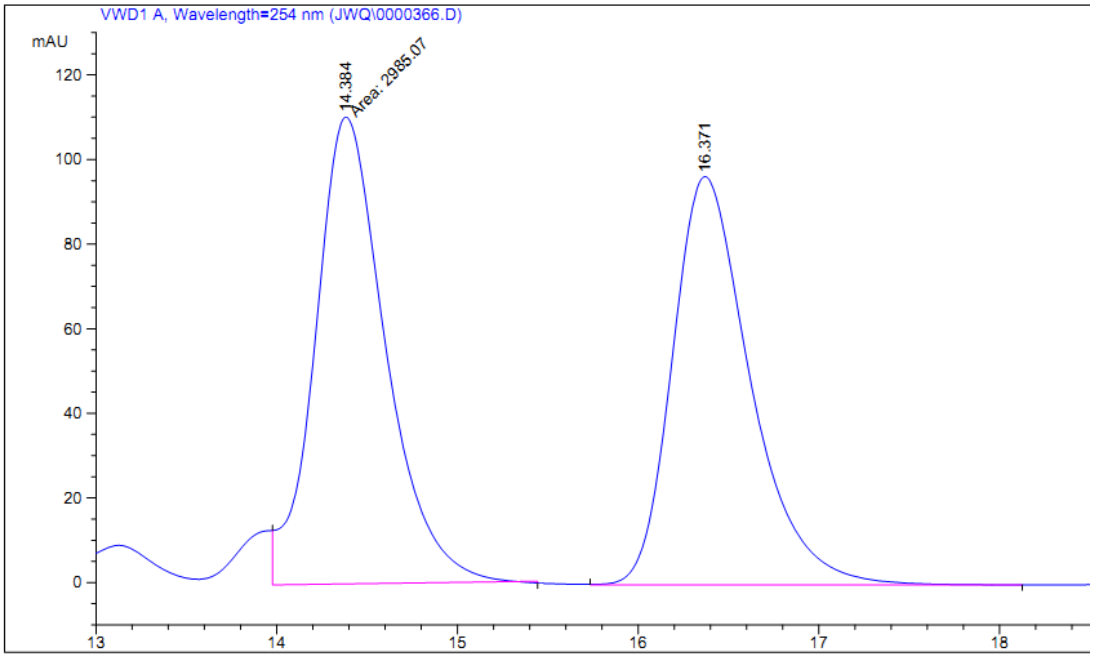


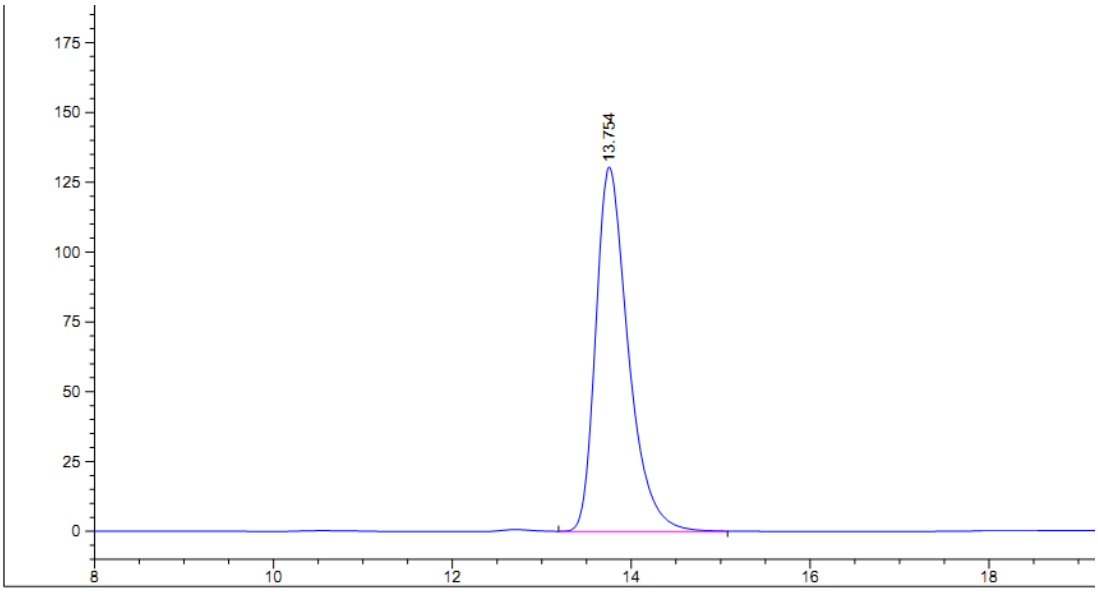


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.662	VB	0.3521	1.15151e4	496.60318	100.0000

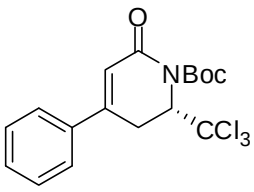


3b

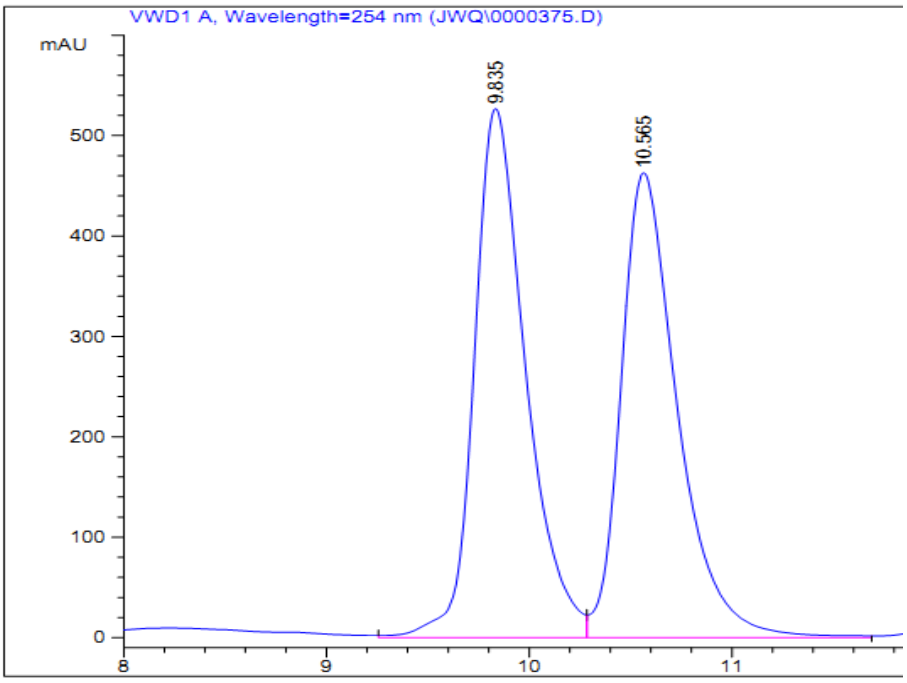


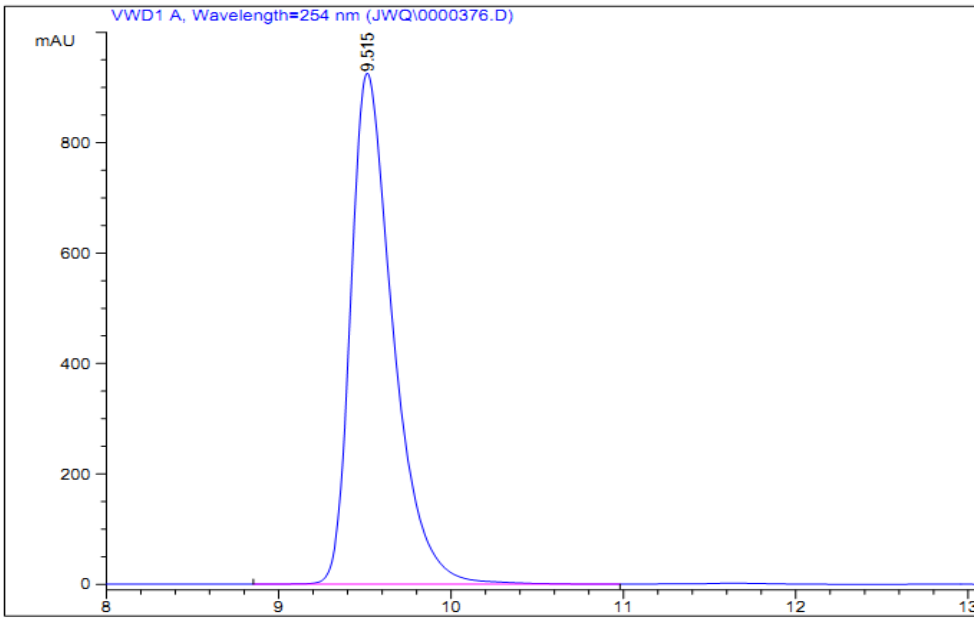


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.754	VB	0.3818	3293.33813	130.49788	100.0000

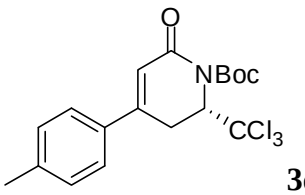


3c

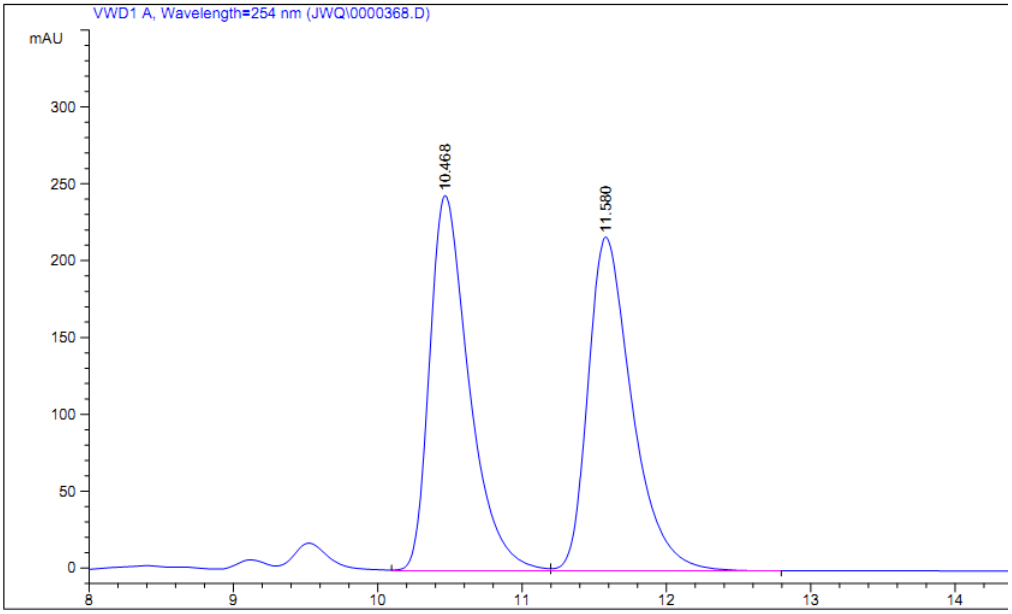


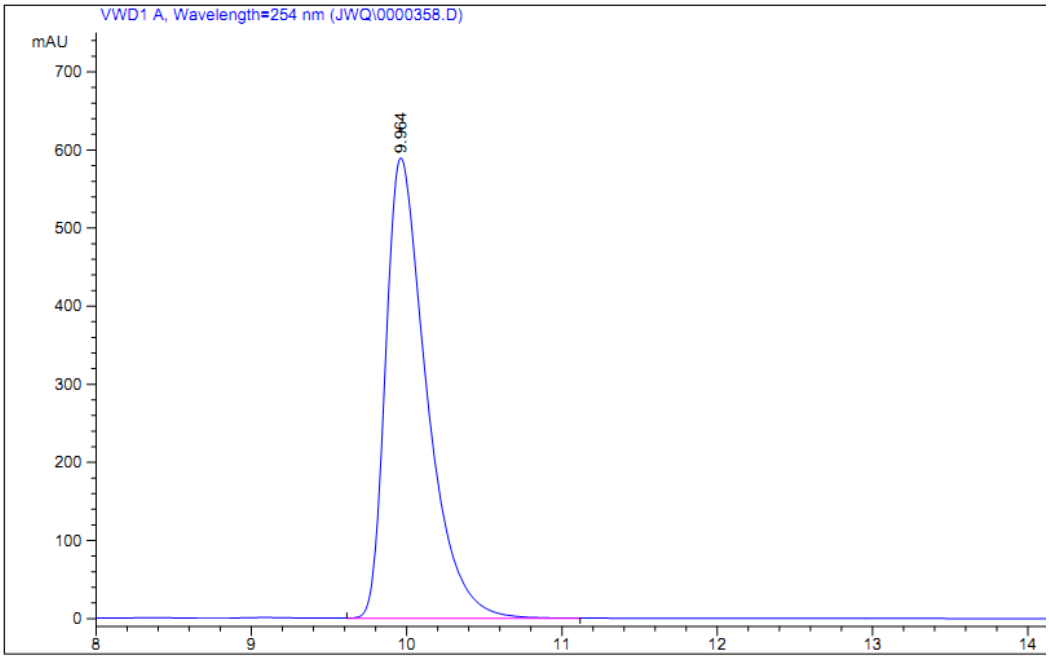


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.515	VB	0.2576	1.58108e4	925.36346	100.0000

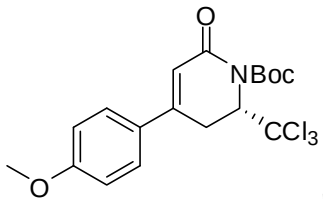


3d

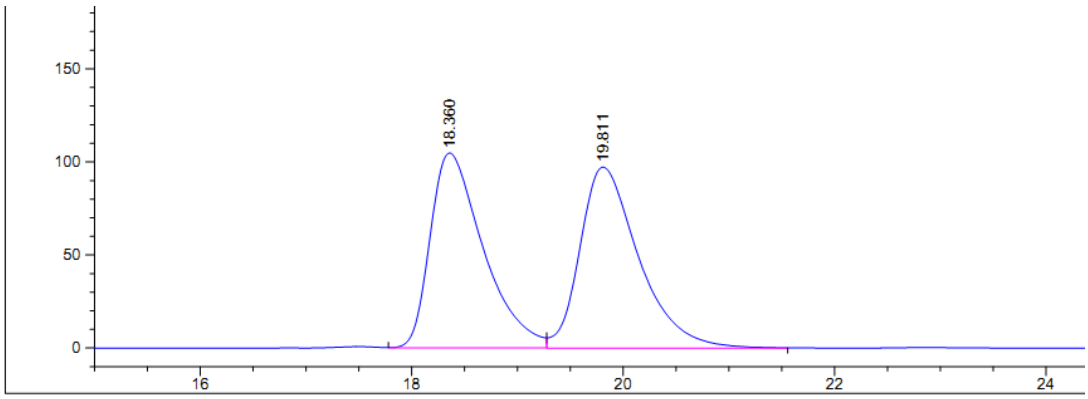


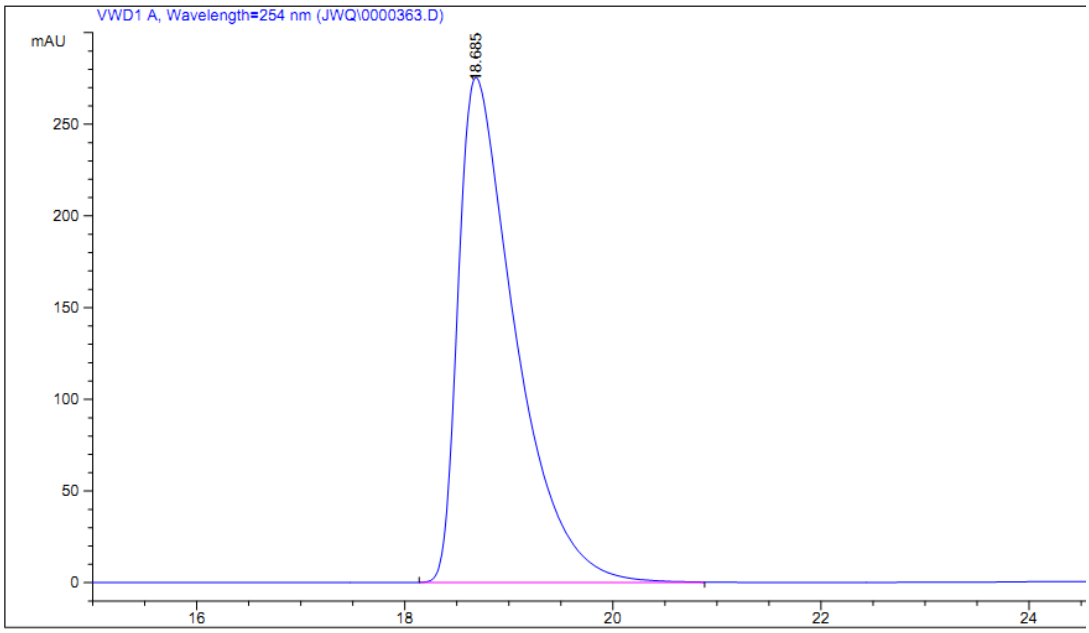


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.964	VB	0.2775	1.07845e4	589.50562	100.0000

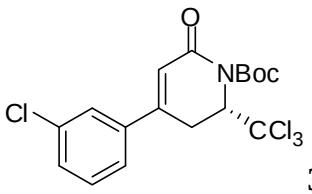


3e

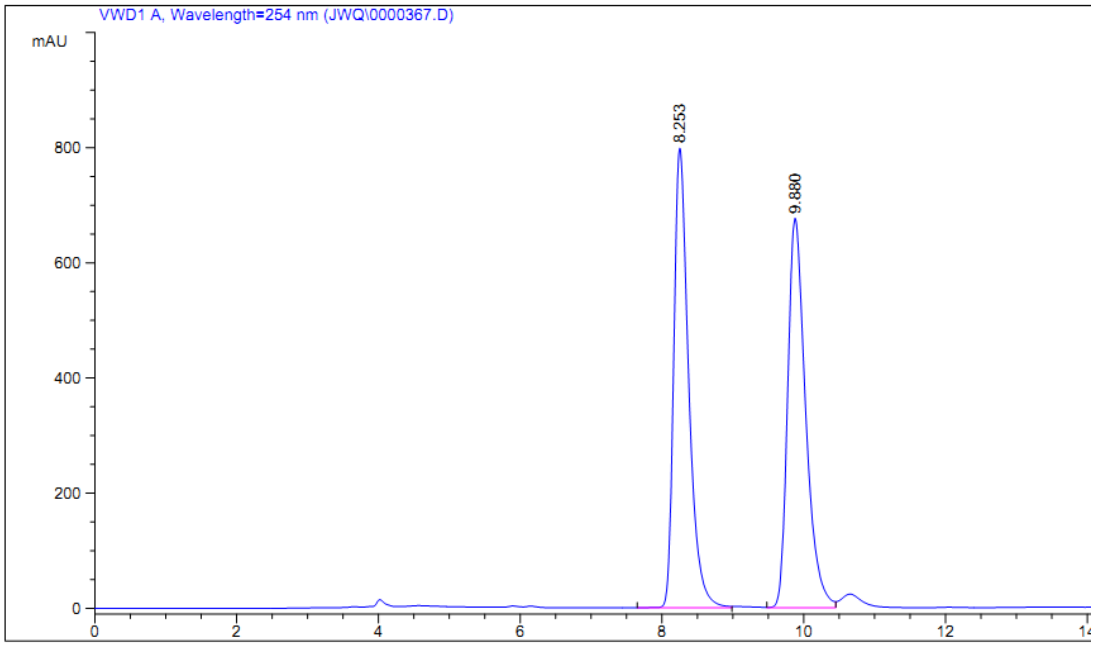


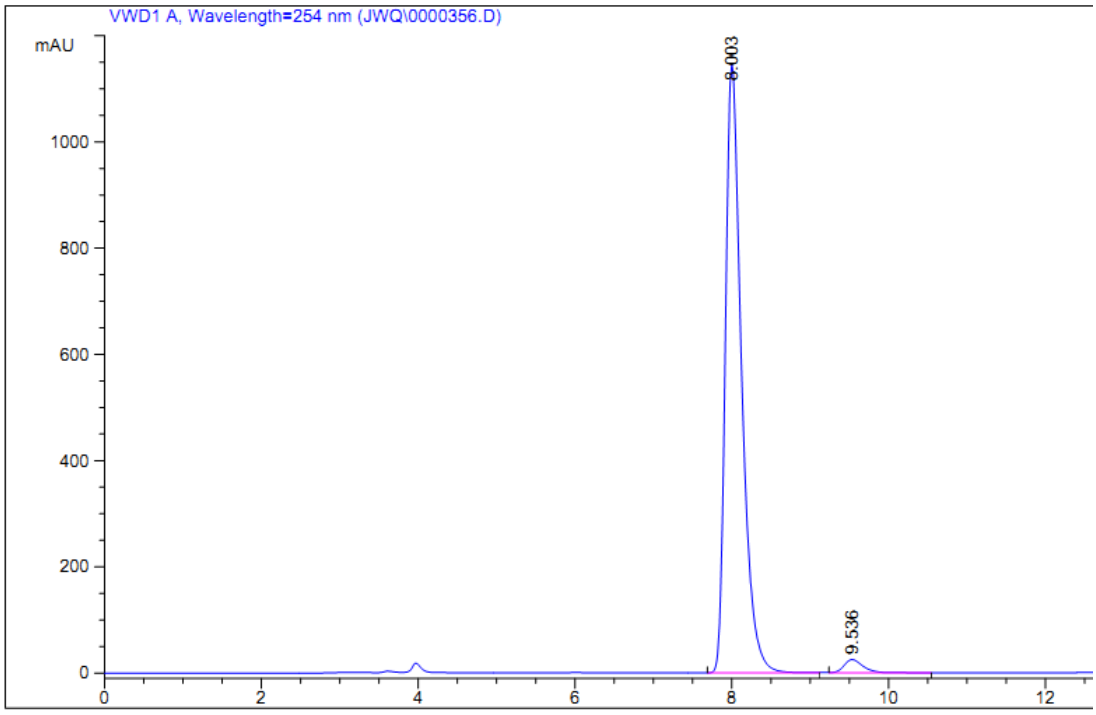


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.685	BB	0.5643	1.04982e4	275.30875	100.0000

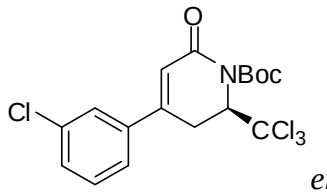


3f

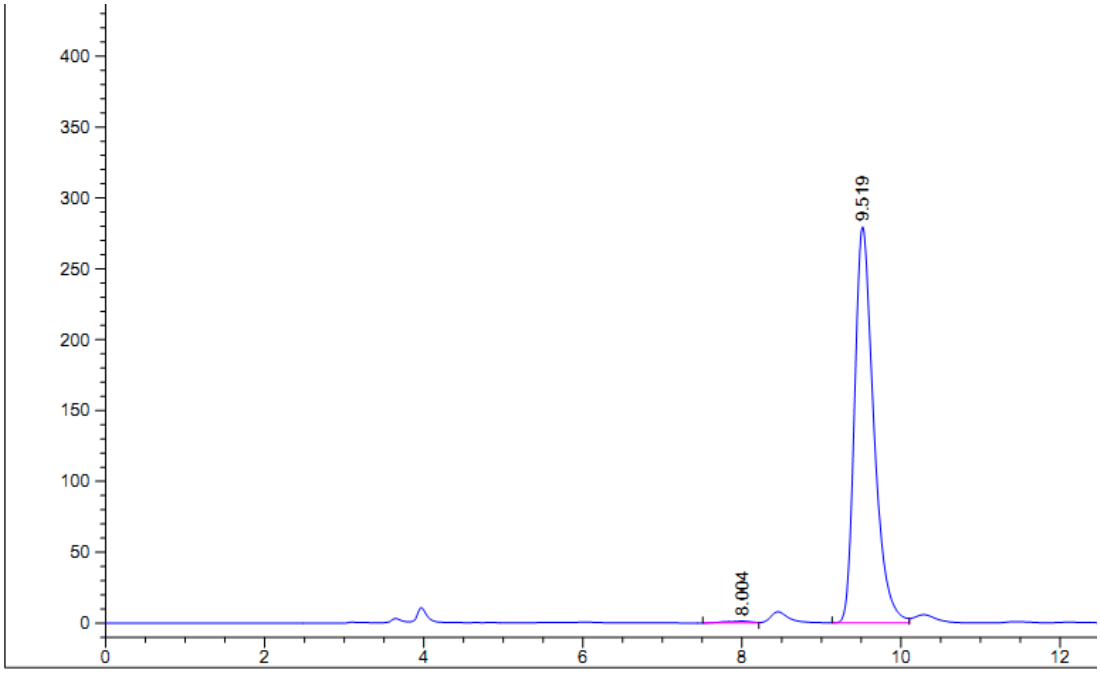




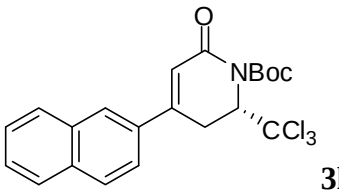
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.003	BB	0.2162	1.63734e4	1145.80591	97.3796
2	9.536	BB	0.2642	440.59320	25.31360	2.6204



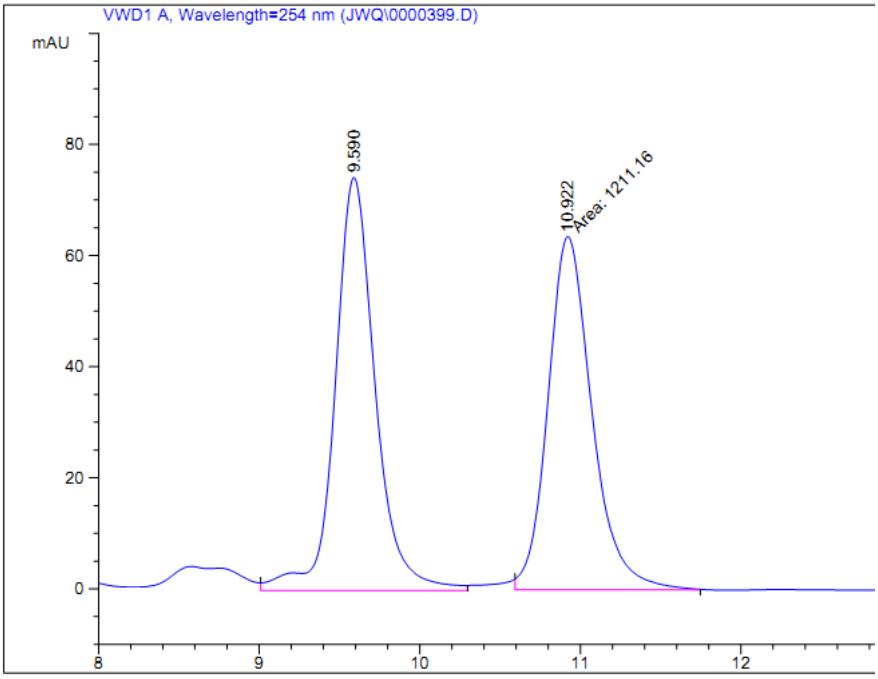
ent-**3f**

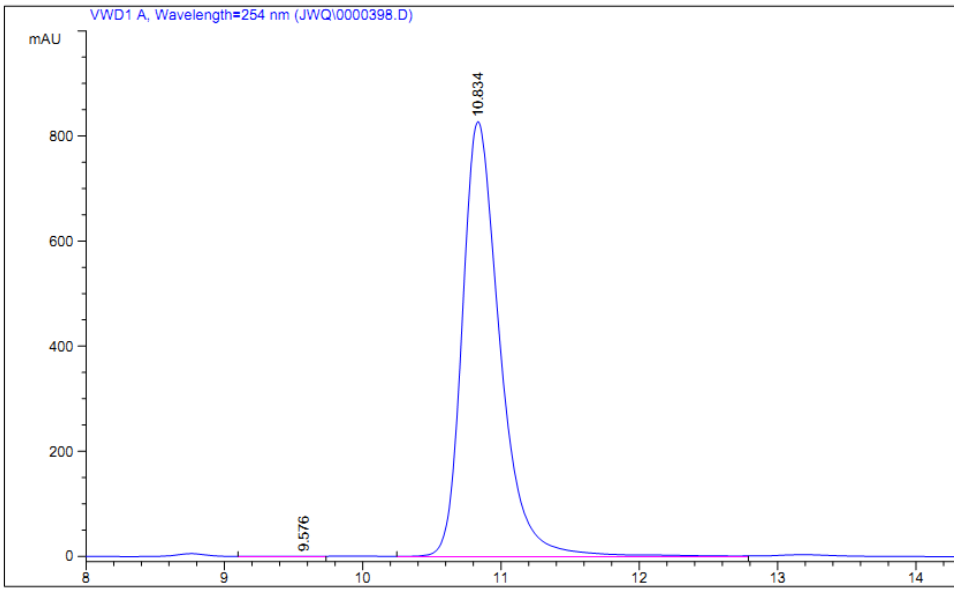


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.004	BV	0.2950	29.84758	1.40183	0.6342
2	9.519	VV	0.2534	4676.70117	279.50513	99.3658

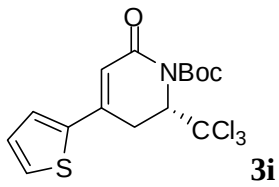


3h

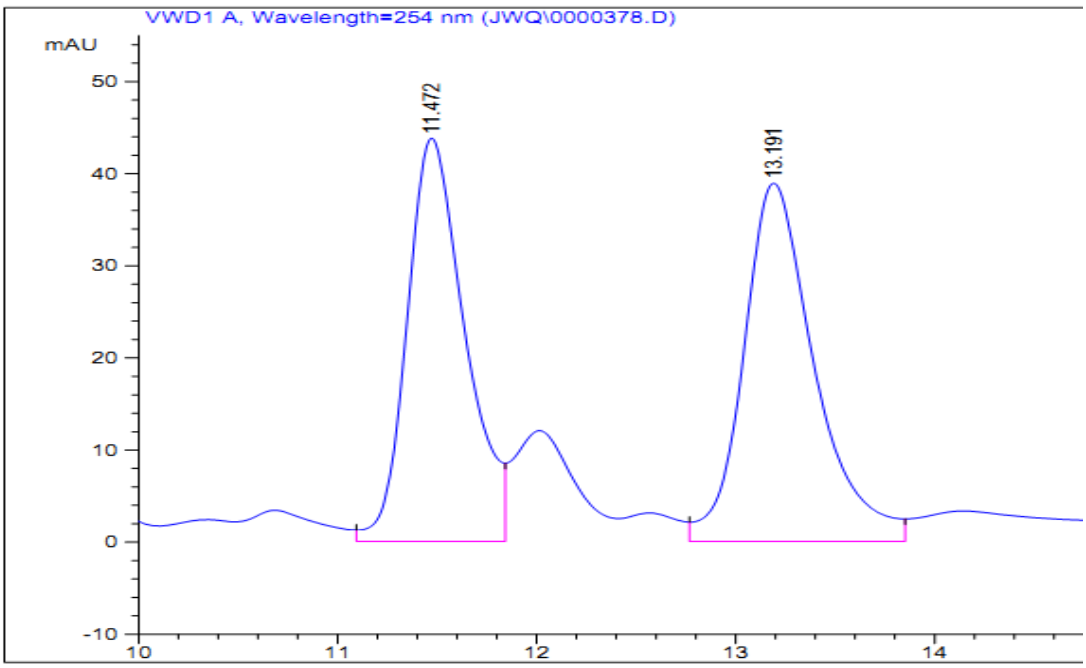


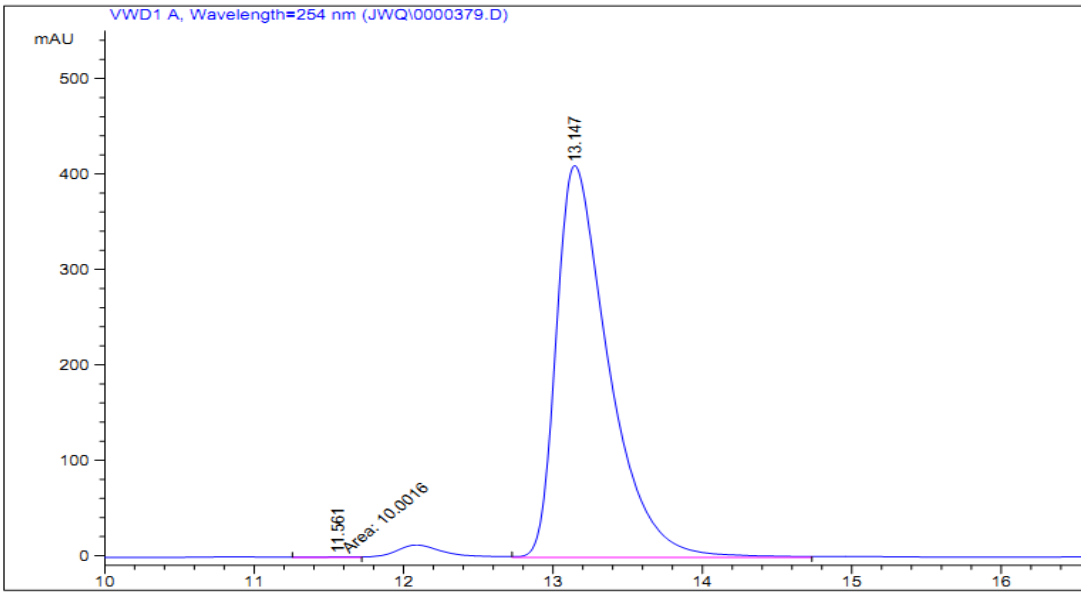


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.576	VV	0.3473	18.88475	7.32639e-1	0.1204
2	10.834	VV	0.2879	1.56705e4	827.58899	99.8796

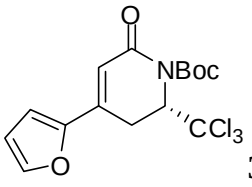


3i

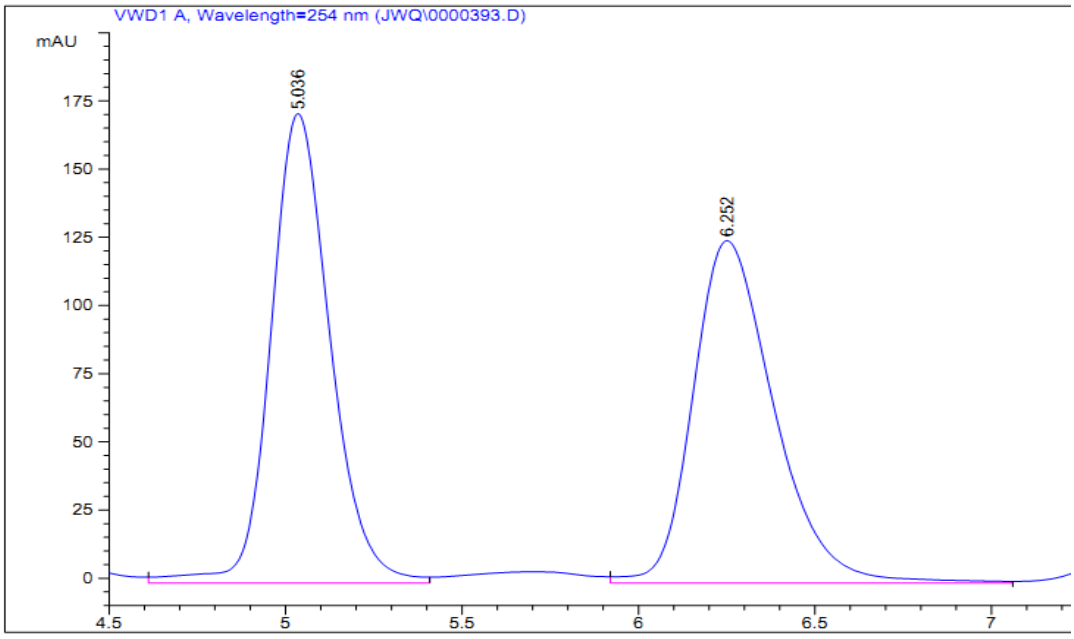


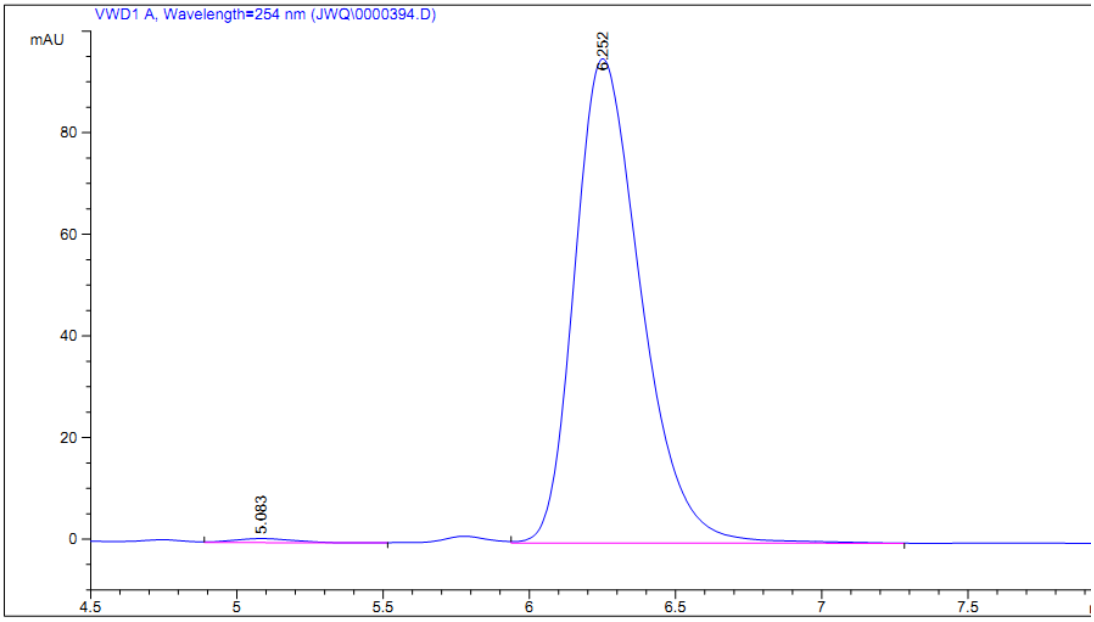


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.561	MM	0.3284	10.00163	5.07655e-1	0.1000
2	13.147	VV	0.3655	9994.08008	410.54727	99.9000

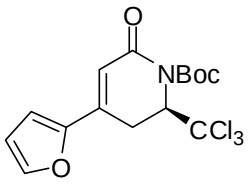


3j

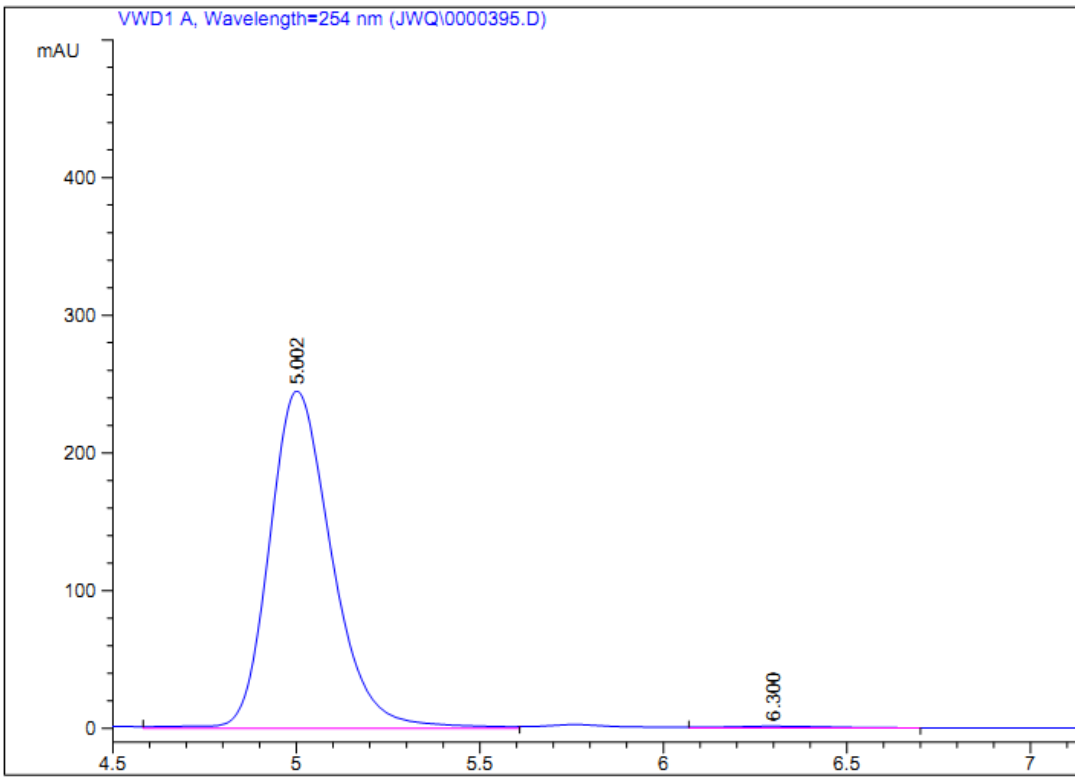




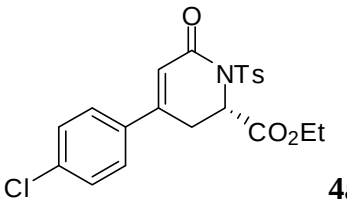
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.083	VB	0.2318	11.75584	8.17676e-1	0.7779
2	6.252	VB	0.2448	1499.52490	95.26770	99.2221



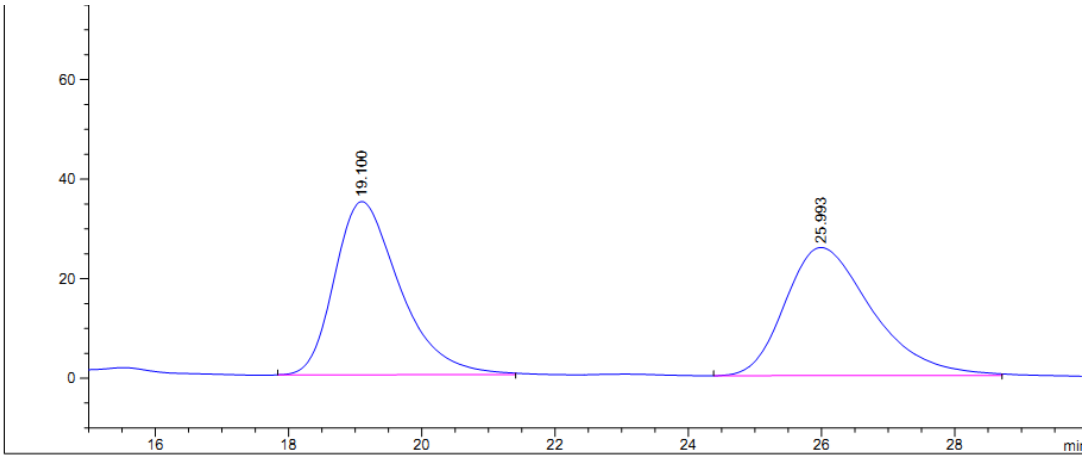
ent-**3j**

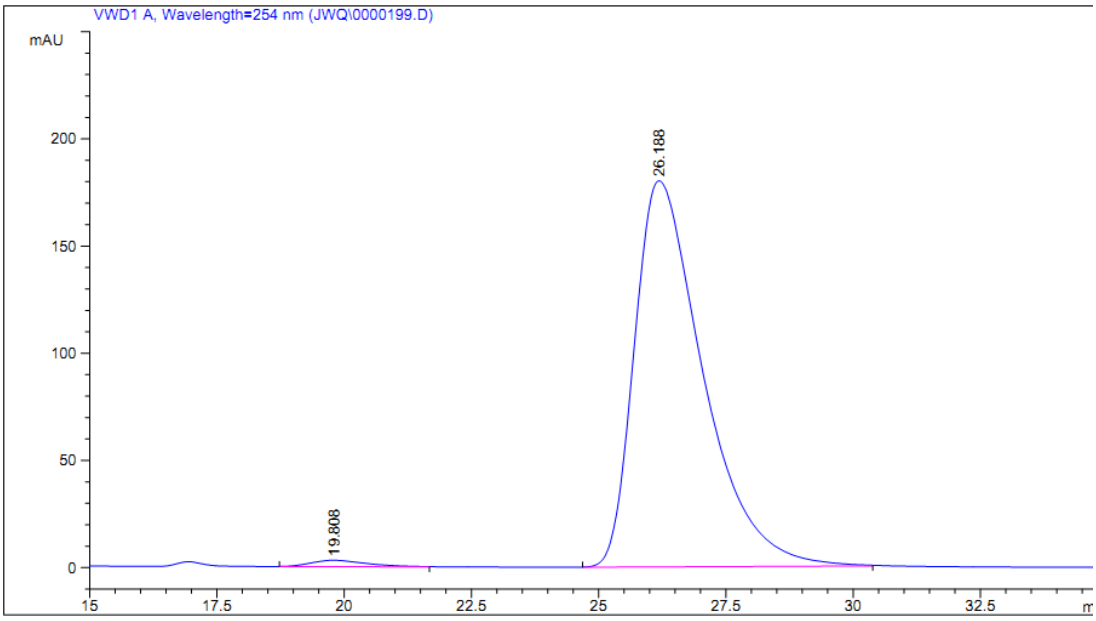


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	5.002	VV	0.1816	2895.33154	244.83955	99.1139
2	6.300	VB	0.2739	25.88619	1.40070	0.8861

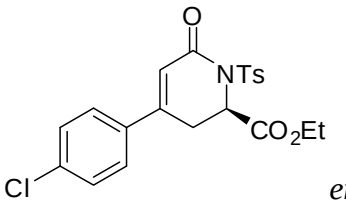


4a

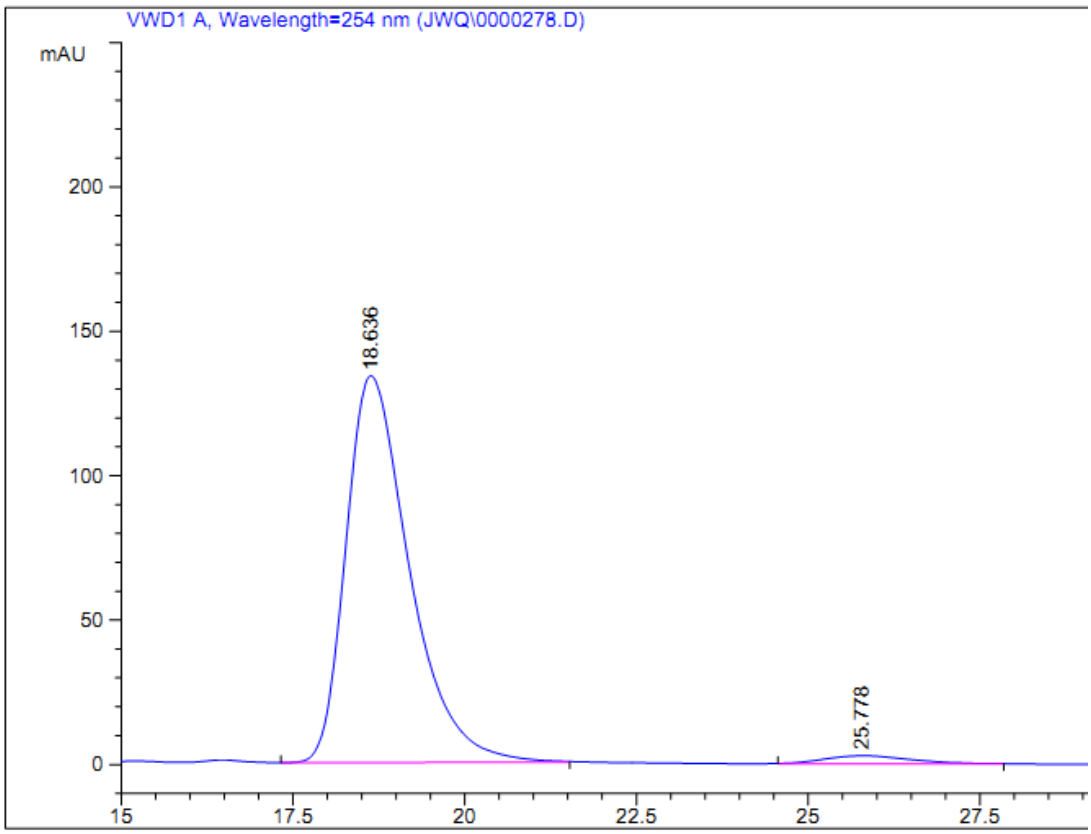




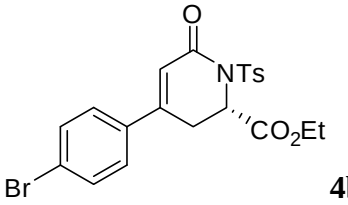
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.808	BB	1.1094	225.61183	2.97342	1.3217
2	26.188	BB	1.4175	1.68448e4	180.06673	98.6783



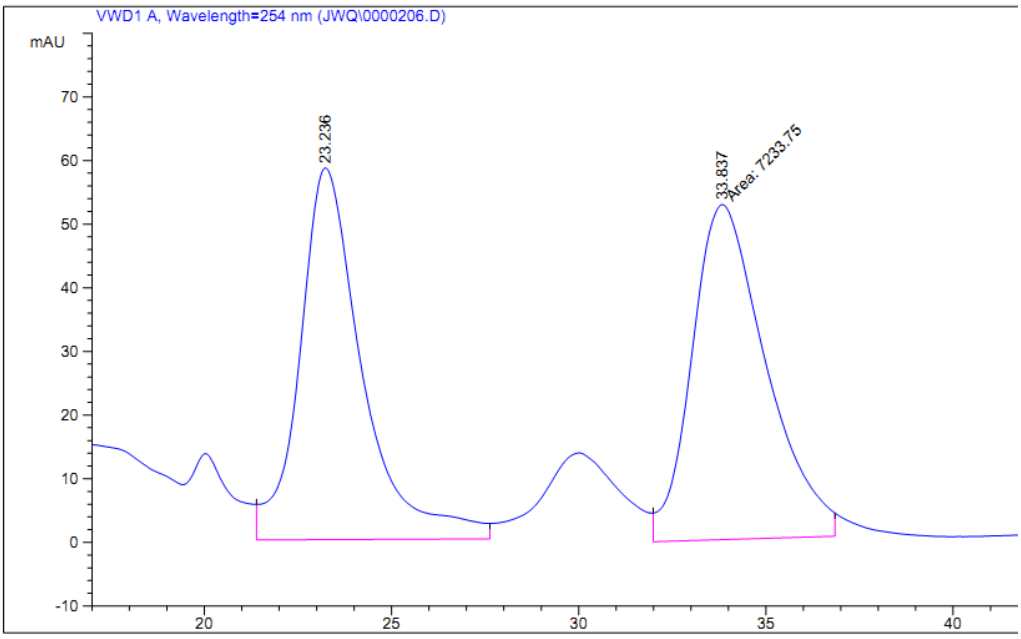
ent-**4a**

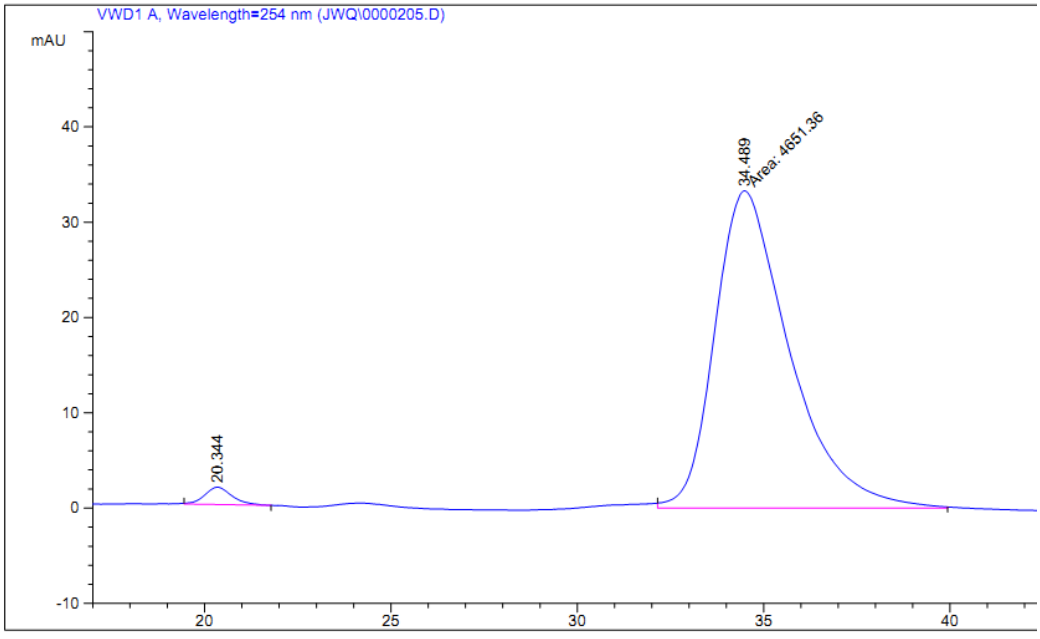


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.636	VB	0.9764	8599.90234	133.91527	97.1988
2	25.778	BB	1.2642	247.84453	2.79601	2.8012

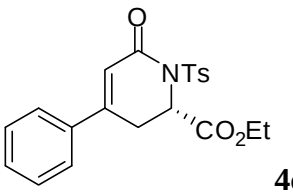


4b





Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.344	BB	0.7550	92.84692	1.82095	1.9571
2	34.489	MM	2.3274	4651.36084	33.30821	98.0429



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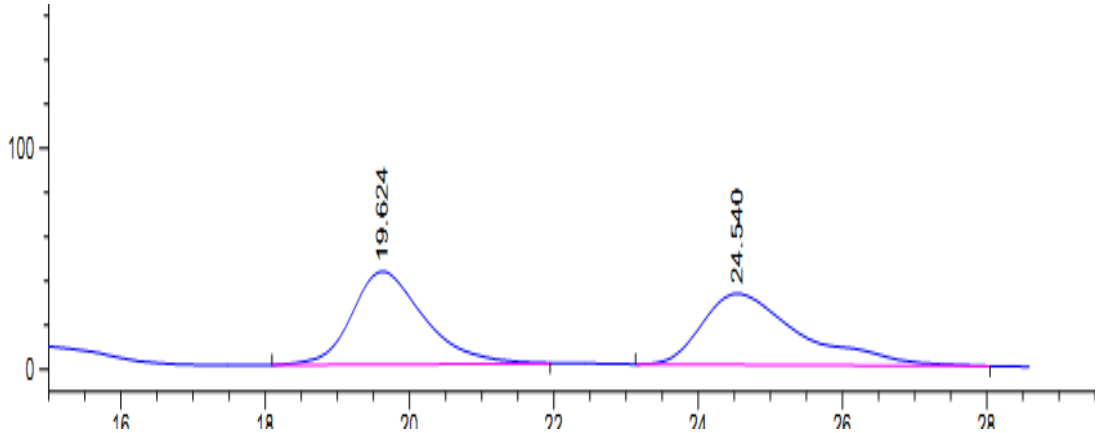
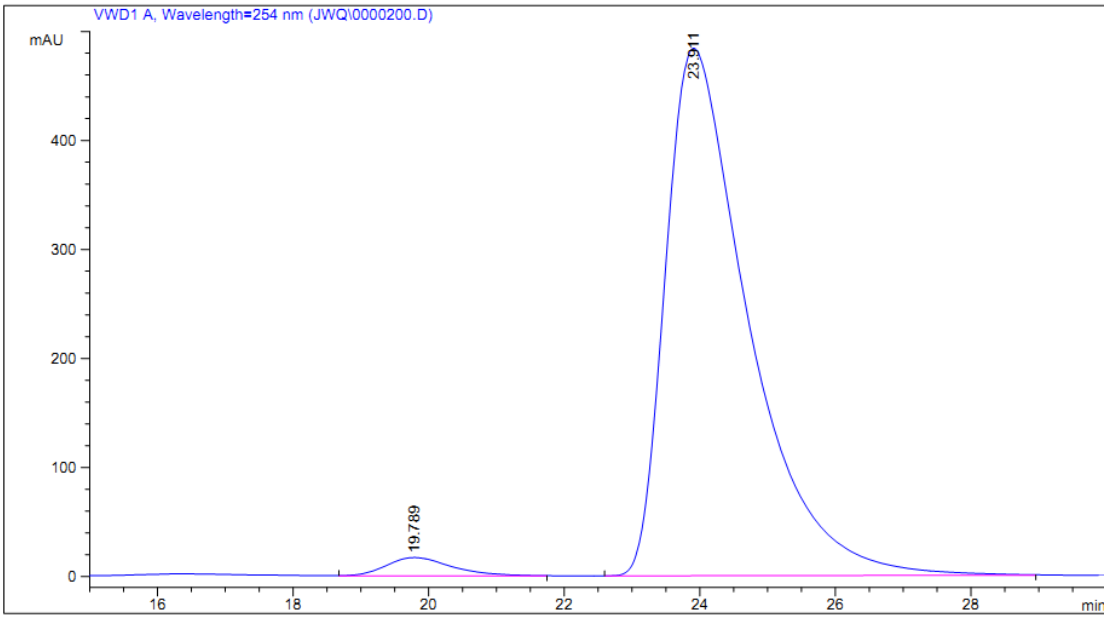
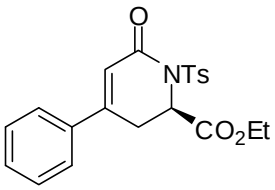
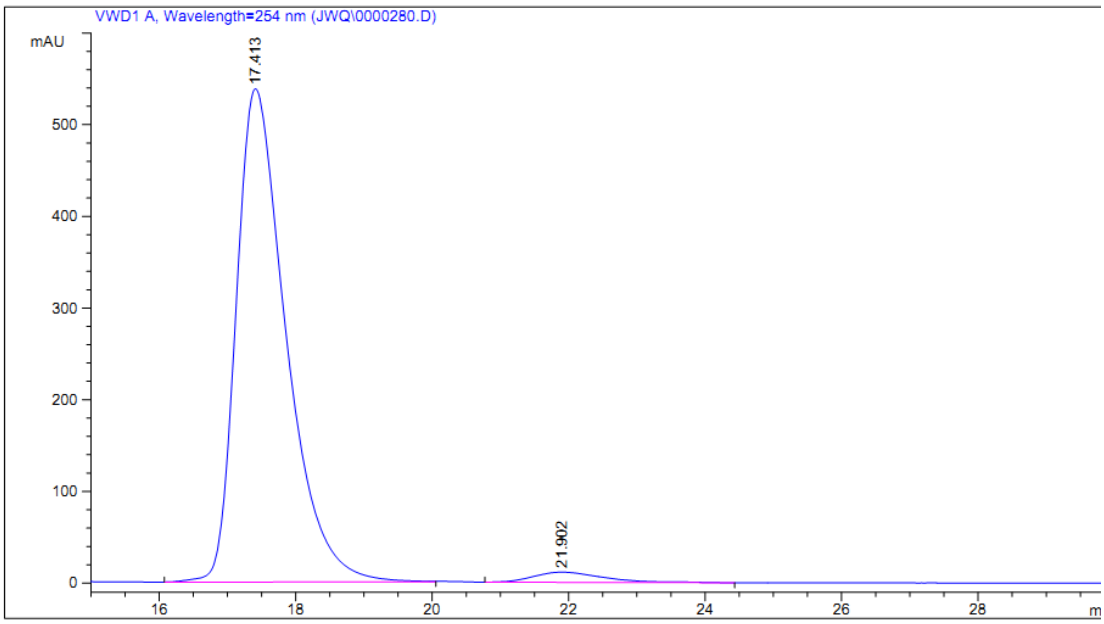




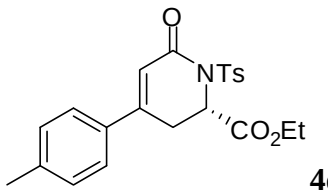
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.789	BB	1.0187	1122.24451	16.78656	2.6605
2	23.911	BB	1.2635	4.10593e4	483.98441	97.3395



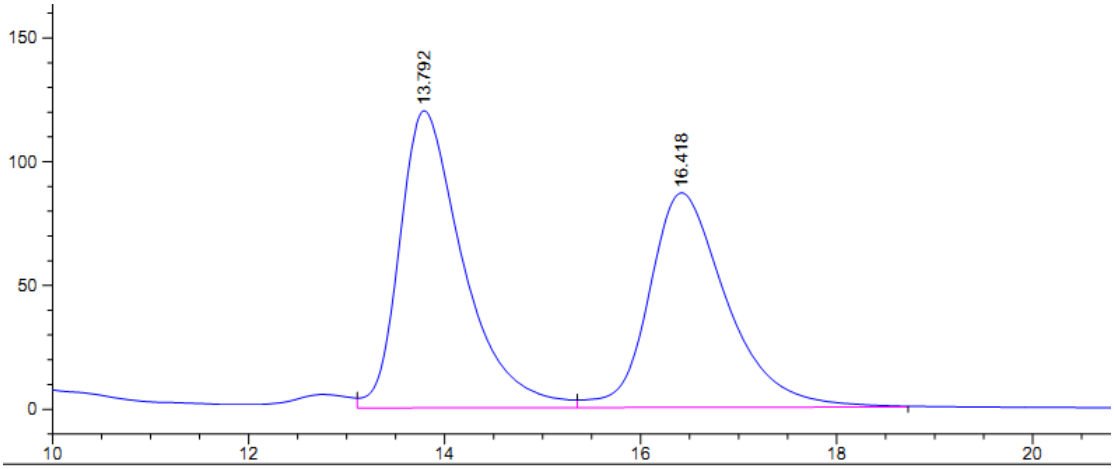
ent-**4c**

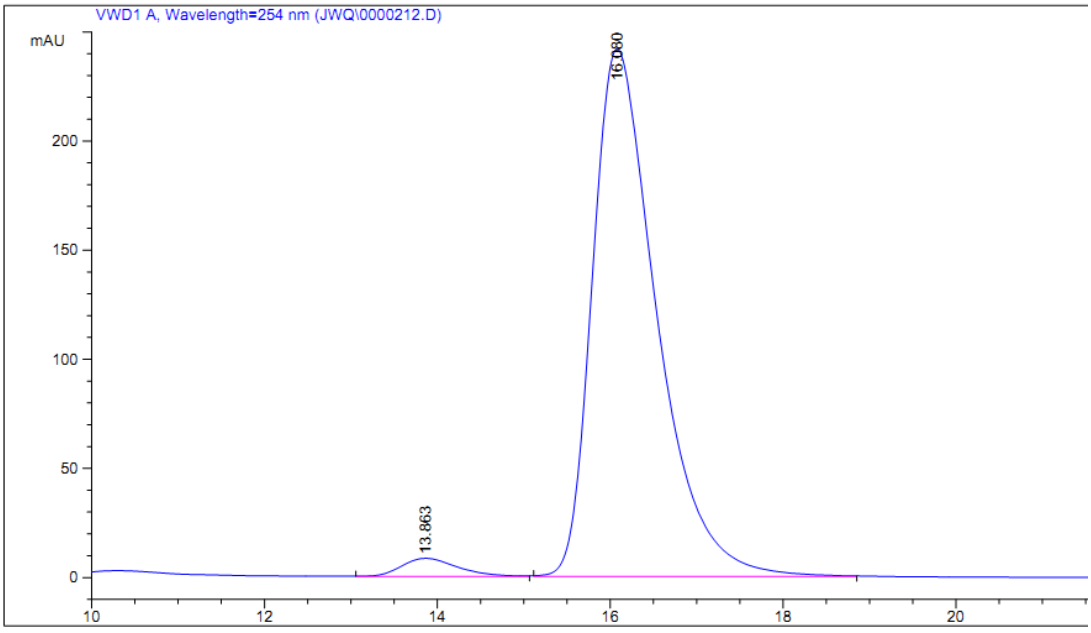


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.413	BB	0.7678	2.72192e4	538.04852	97.2043
2	21.902	BB	1.0509	782.85608	11.12638	2.7957

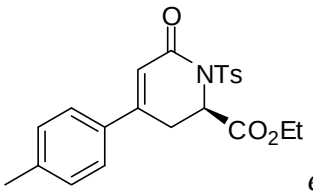


4d

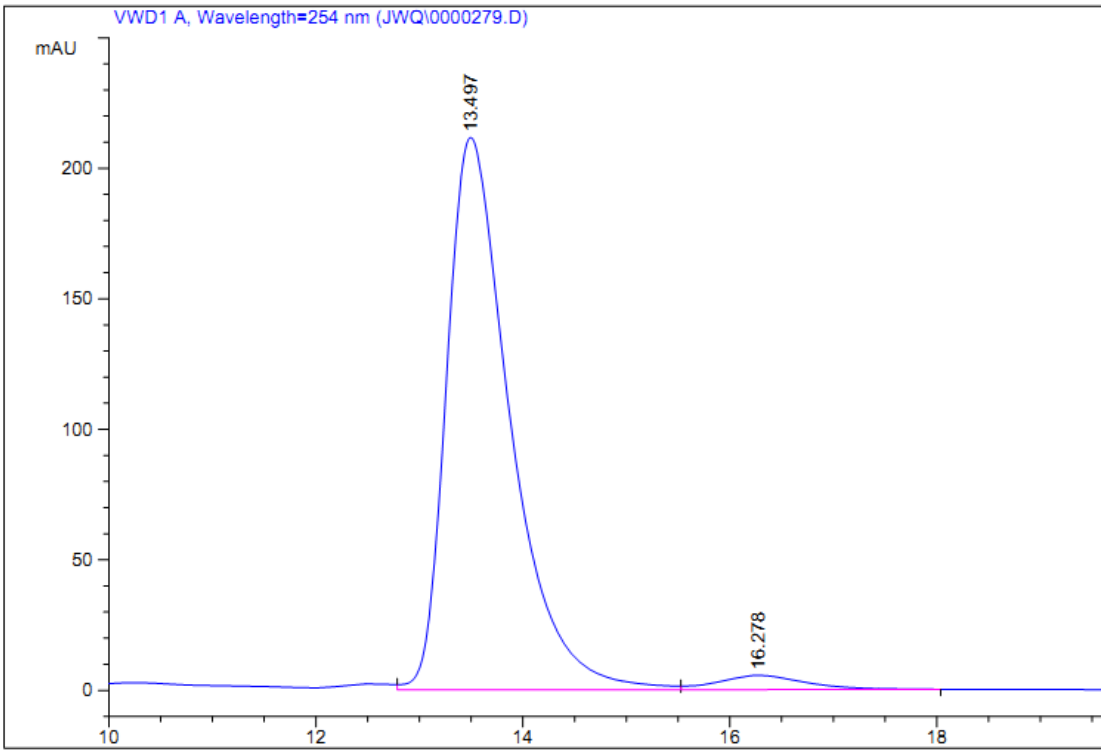




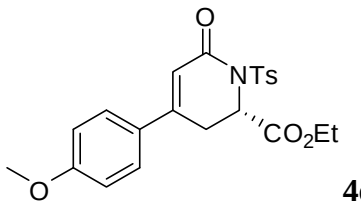
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.863	BB	0.6876	366.44202	8.28829	2.8566
2	16.080	BB	0.7819	1.24614e4	241.74977	97.1434



ent-**4d**



Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.497	VV	0.6436	9033.10645	211.62572	96.5996
2	16.278	VB	0.8552	317.97220	5.47500	3.4004



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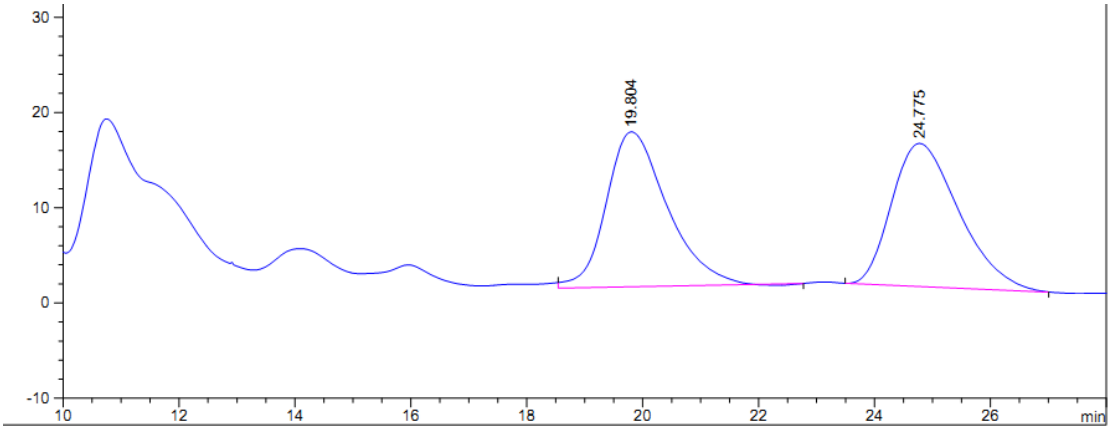
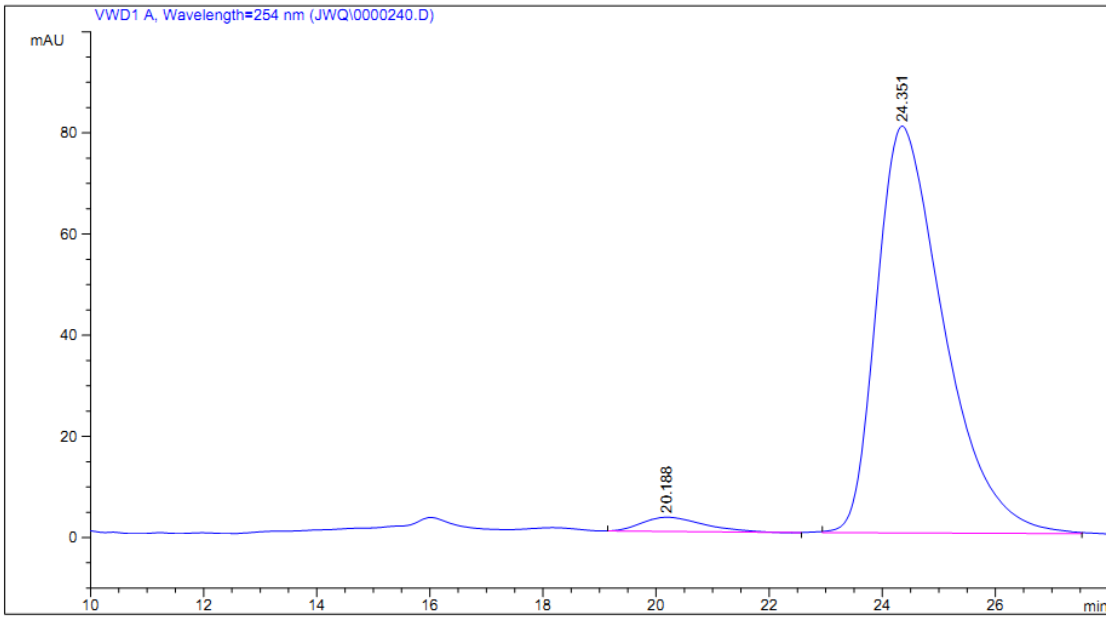
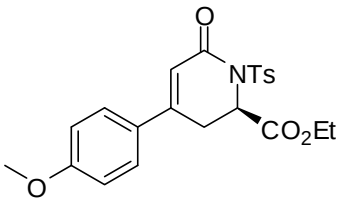




Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.188	BB	1.0734	211.19847	2.80526	3.0852
2	24.351	BB	1.2536	6634.38184	80.42169	96.9148



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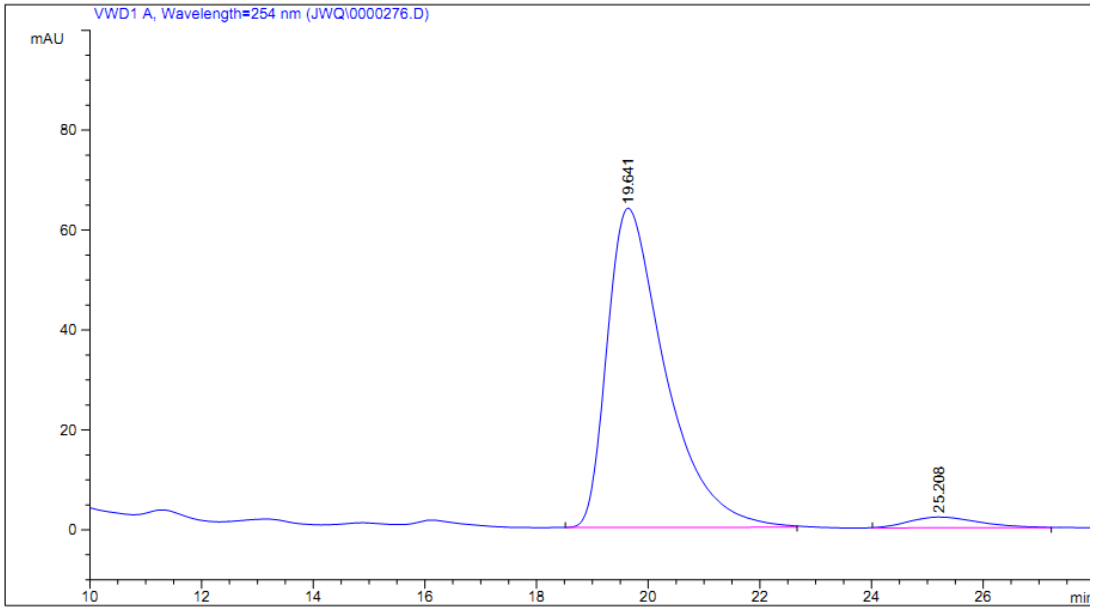
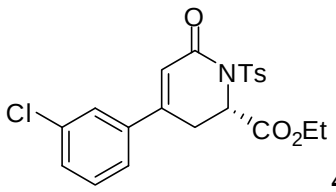
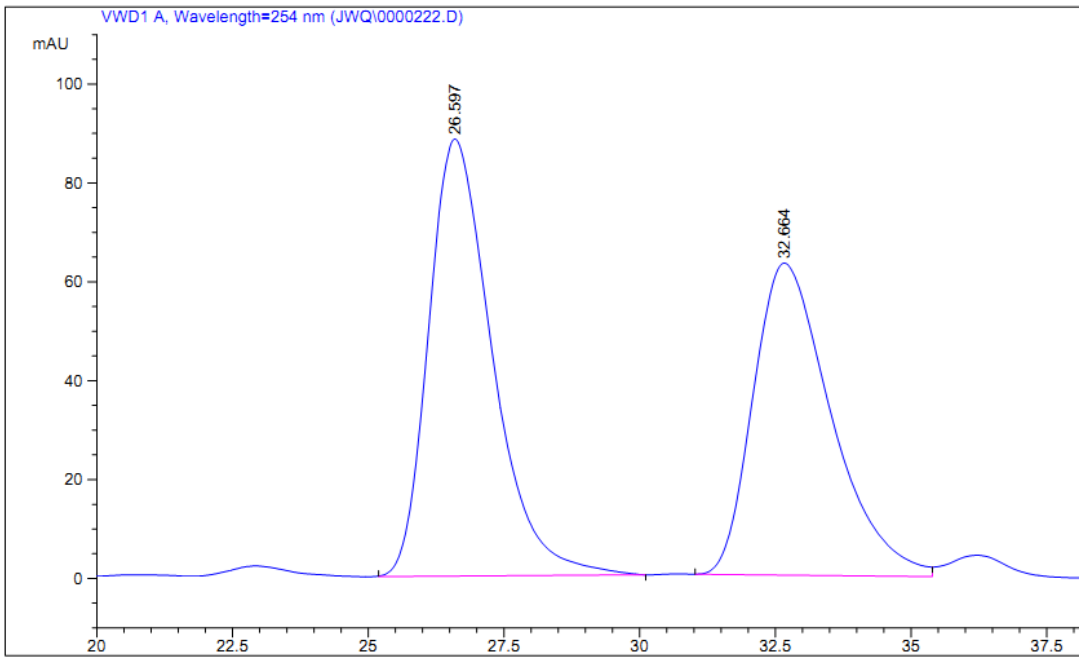
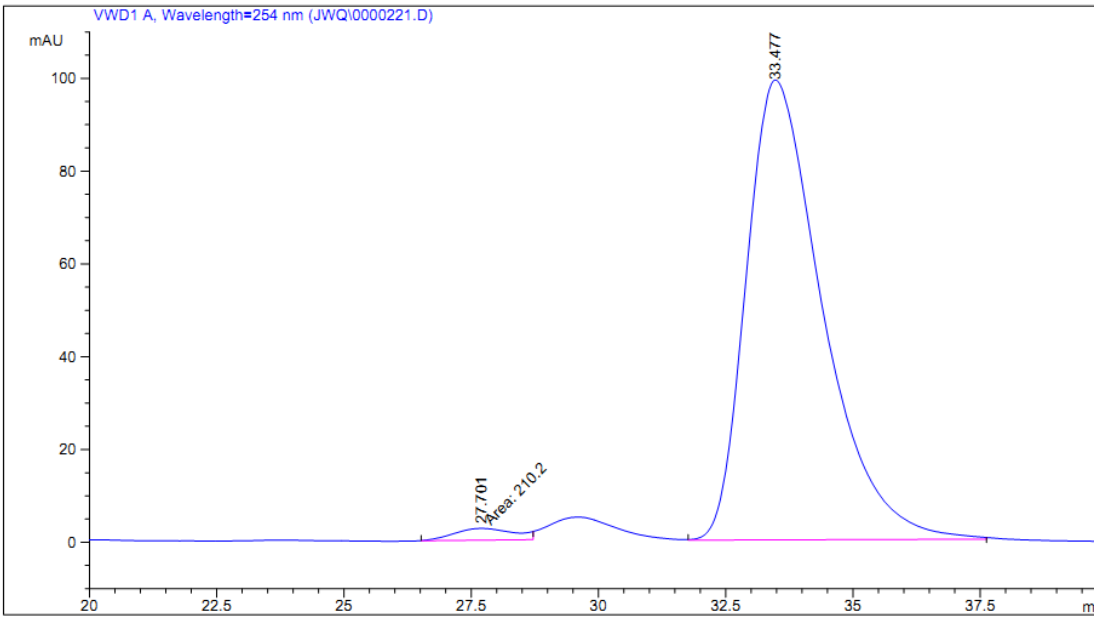


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.641	BB	1.0915	4622.03711	63.89582	96.1170
2	25.208	BB	1.1561	186.72440	2.15582	3.8830

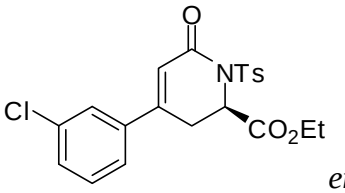


4f

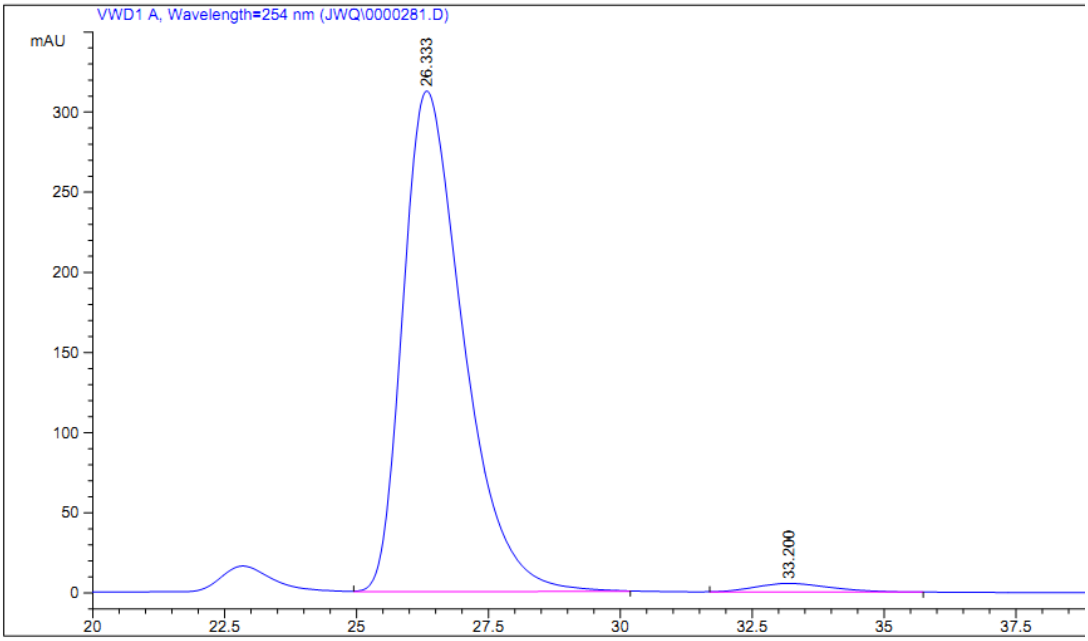




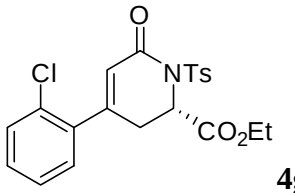
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	27.701	MM	1.3816	210.20045	2.53563	1.9983
2	33.477	BB	1.6123	1.03086e4	99.12168	98.0017



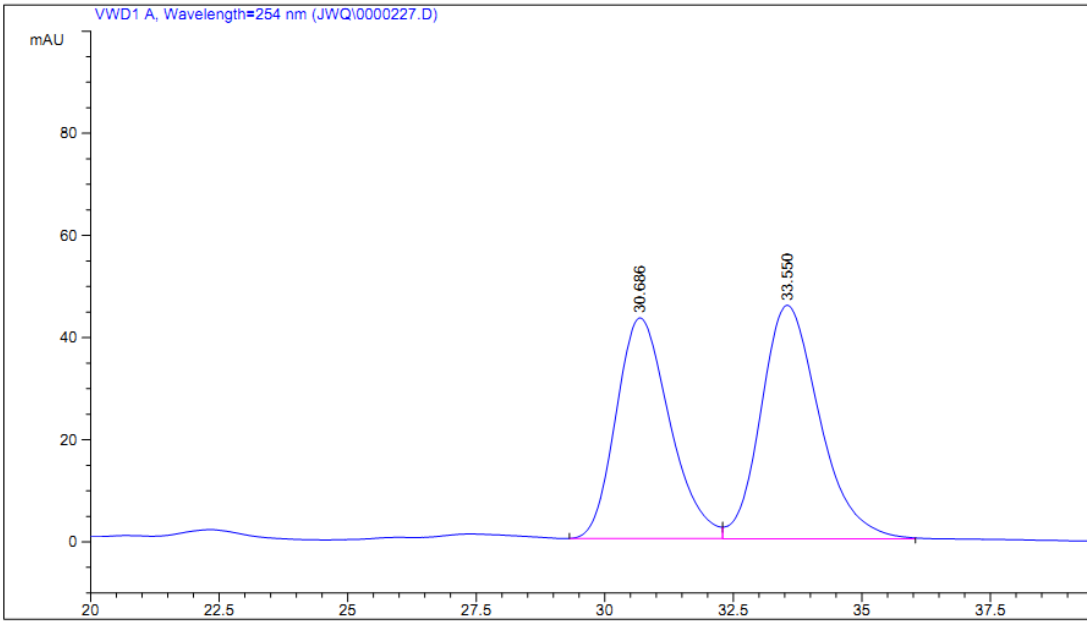
ent-**4f**

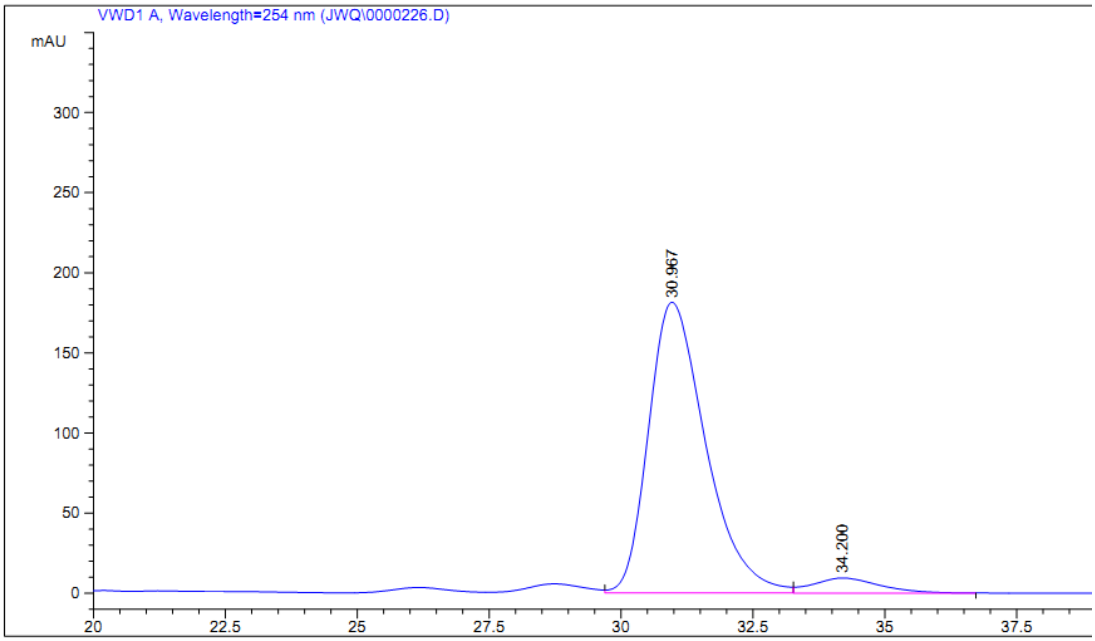


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	26.333	BB	1.2114	2.48652e4	312.28937	97.9222
2	33.200	BB	1.4650	527.61591	5.32345	2.0778

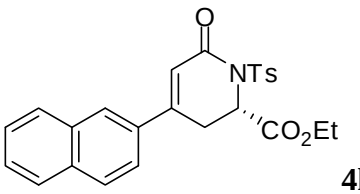


4g

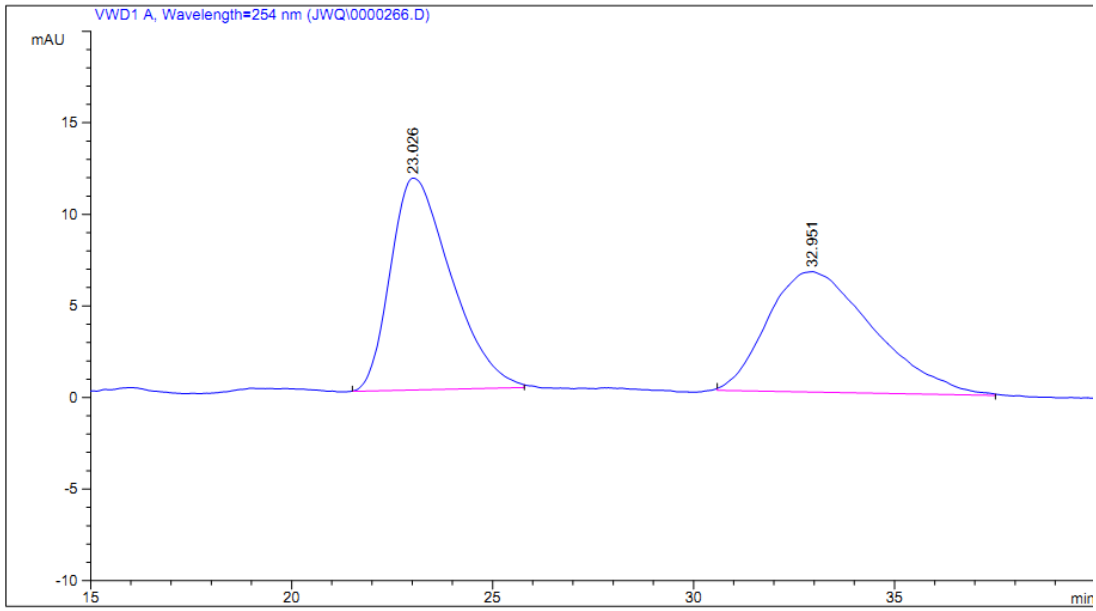


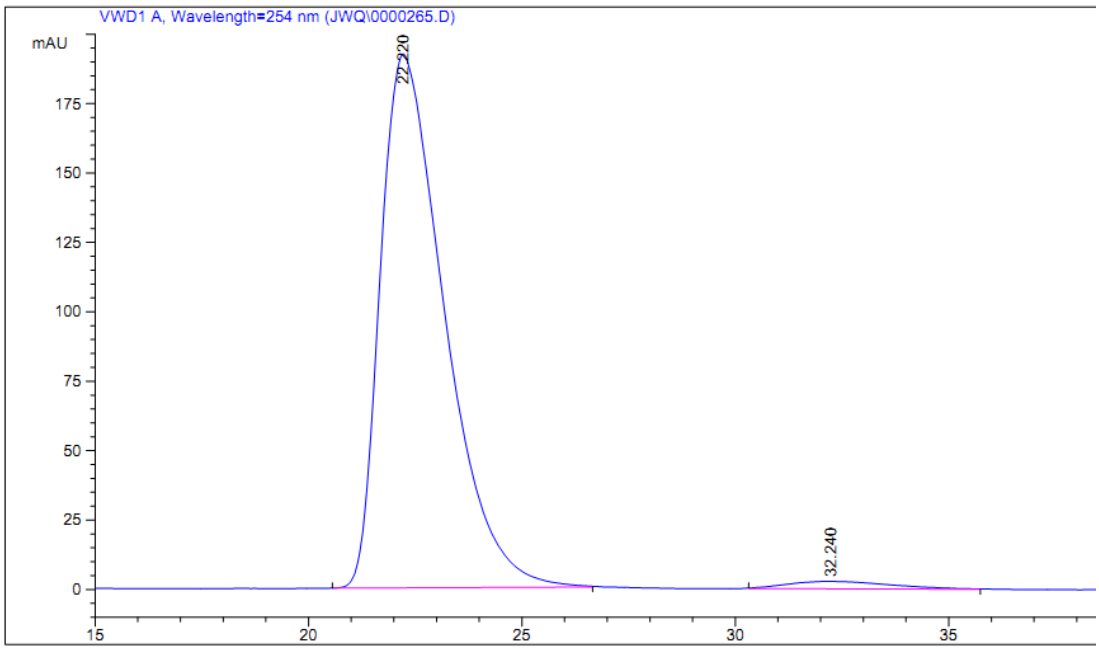


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	30.967	VV	1.1479	1.36866e4	181.45776	94.3134
2	34.200	VB	1.3078	825.22620	9.36277	5.6866

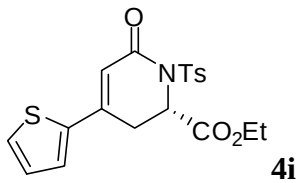


4h

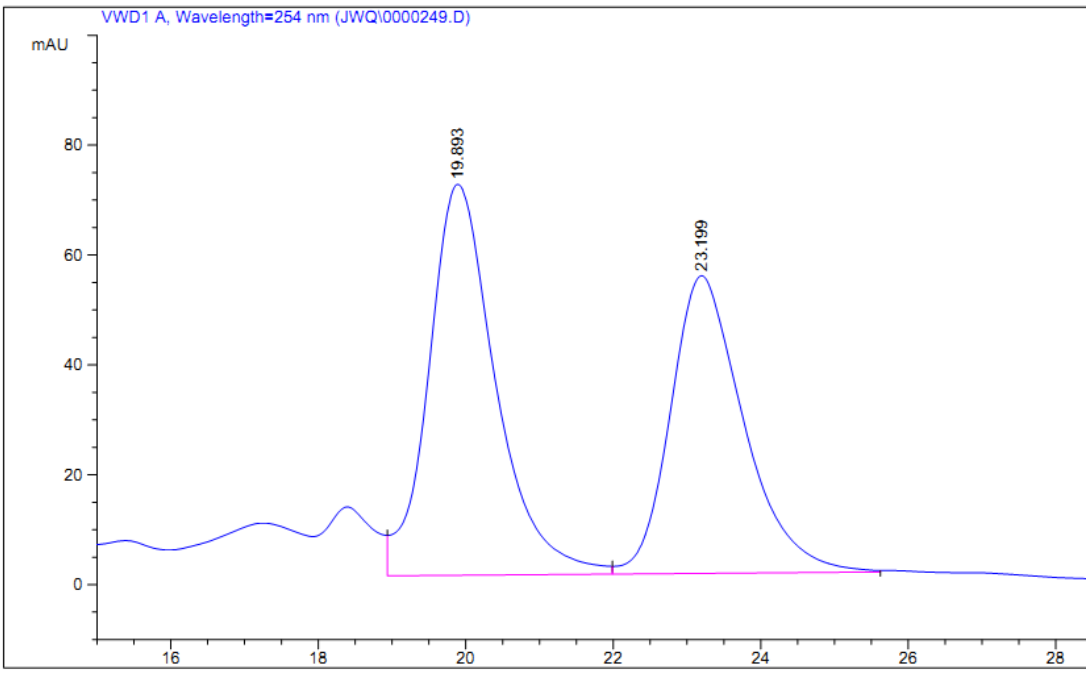


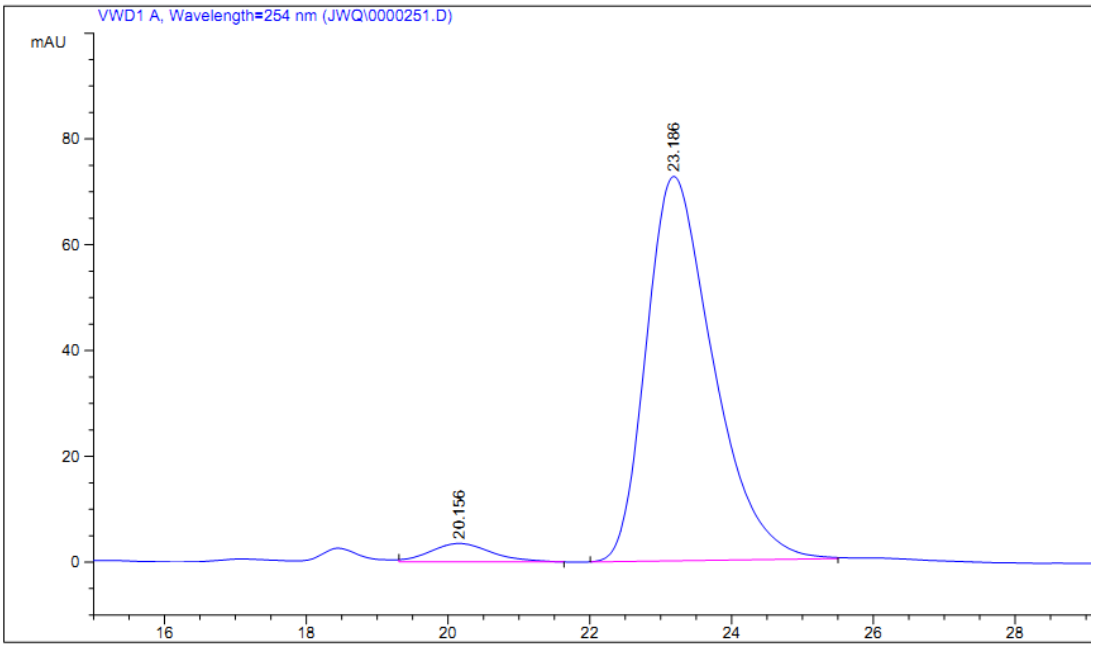


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	22.220	BB	1.5186	2.00410e4	192.31256	97.8396
2	32.240	BB	1.9864	442.53561	2.66158	2.1604

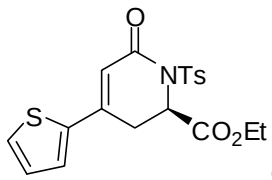


4i

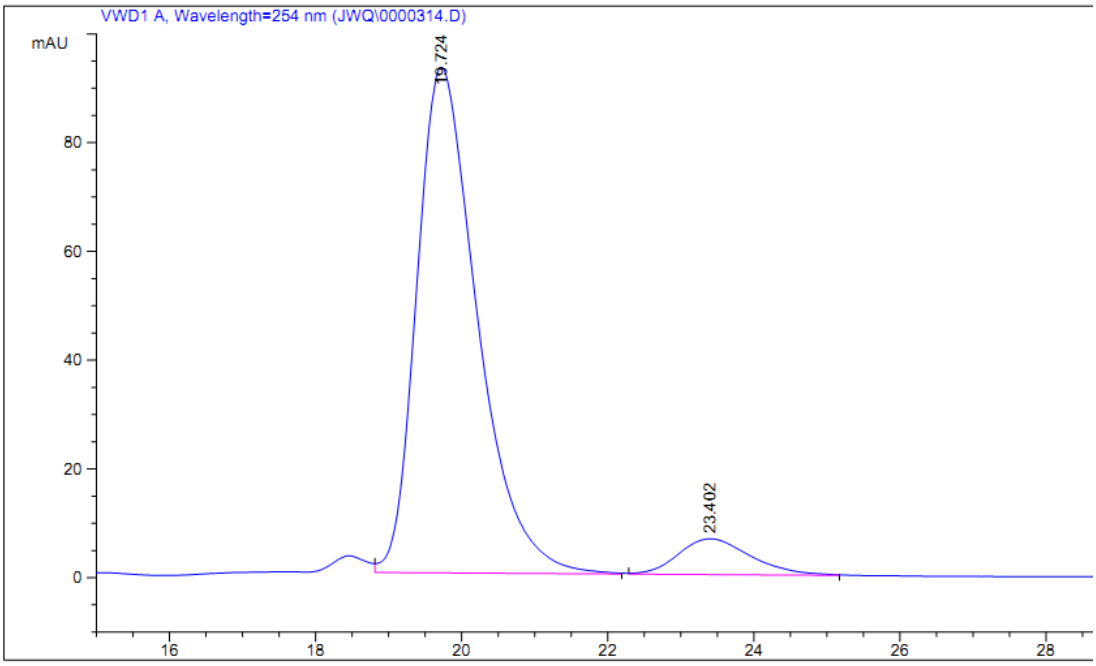




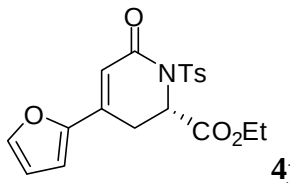
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.156	BB	0.9230	210.29291	3.49620	4.2457
2	23.186	BB	0.9859	4742.82471	72.64774	95.7543



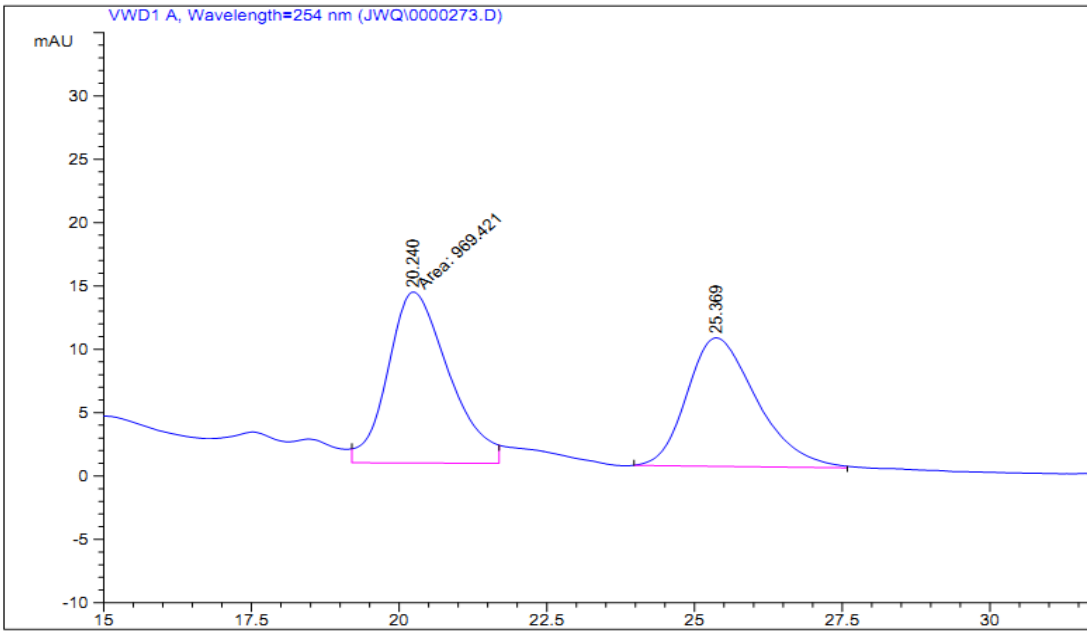
ent-**4i**

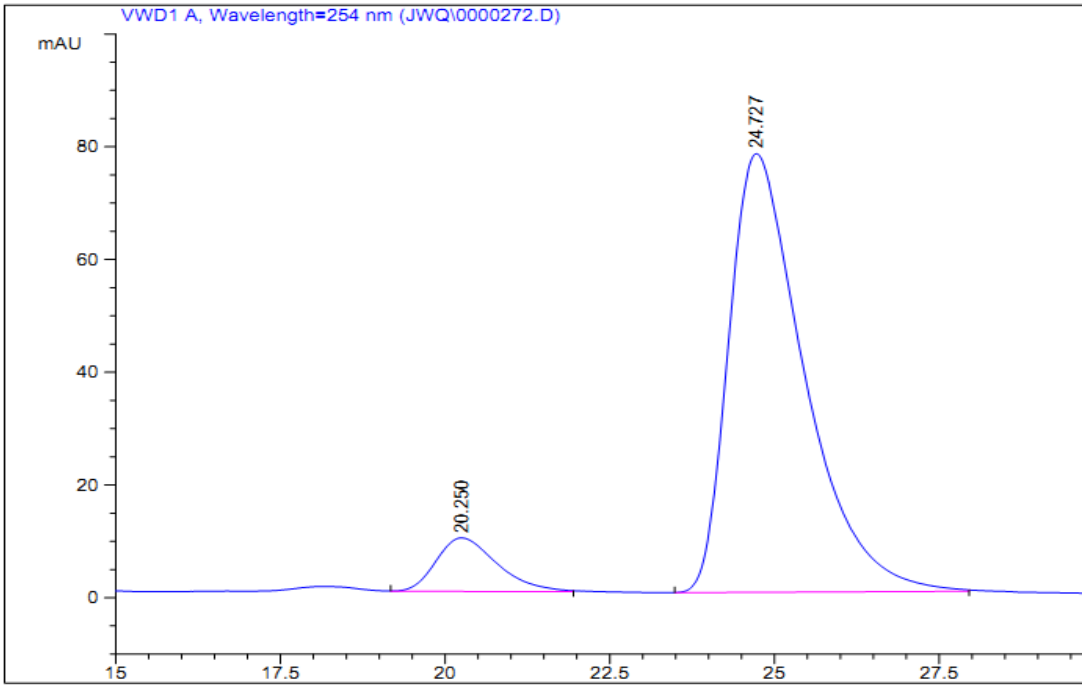


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.724	VB	0.8872	5354.57813	92.99741	92.2095
2	23.402	BB	1.0653	452.39389	6.59505	7.7905

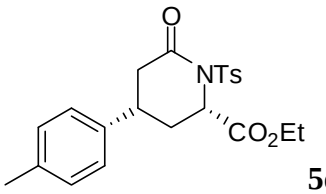


4j

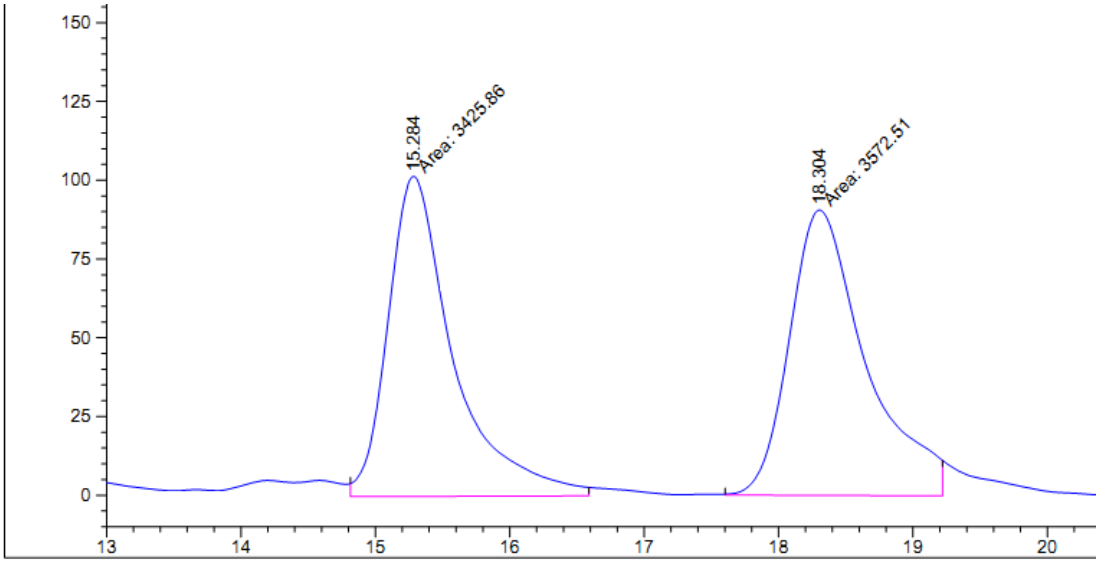


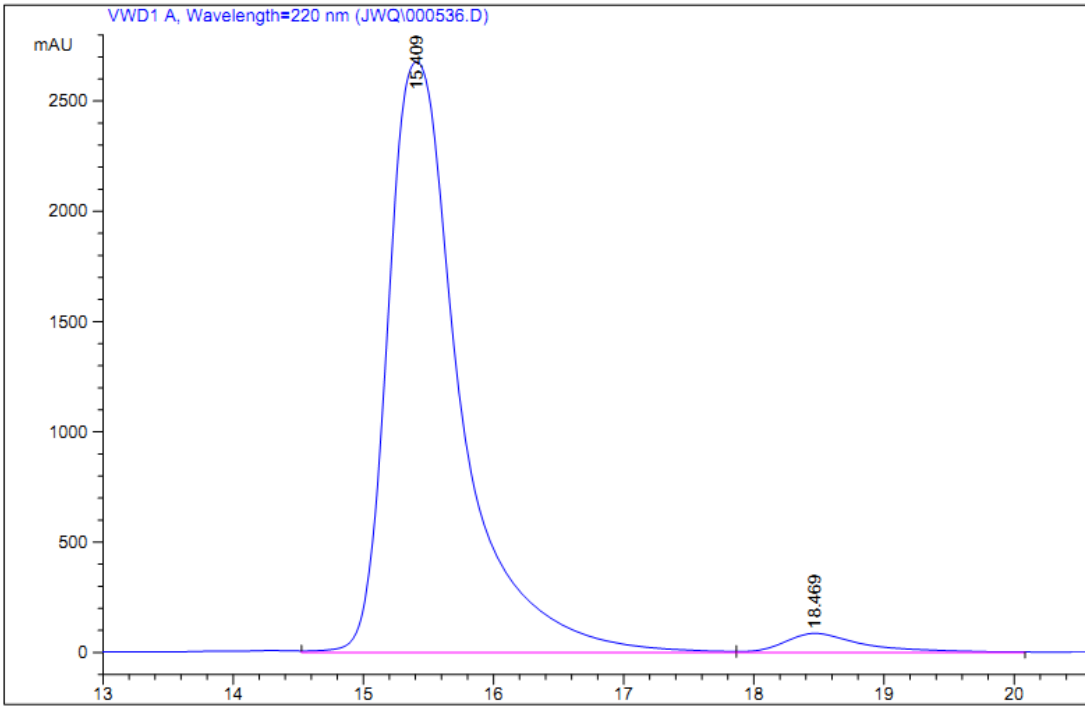


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	20.250	VB	0.9818	609.67859	9.49857	8.9681
2	24.727	BB	1.2045	6188.63574	77.81319	91.0319

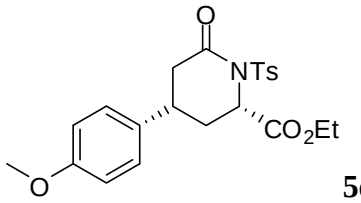


5d

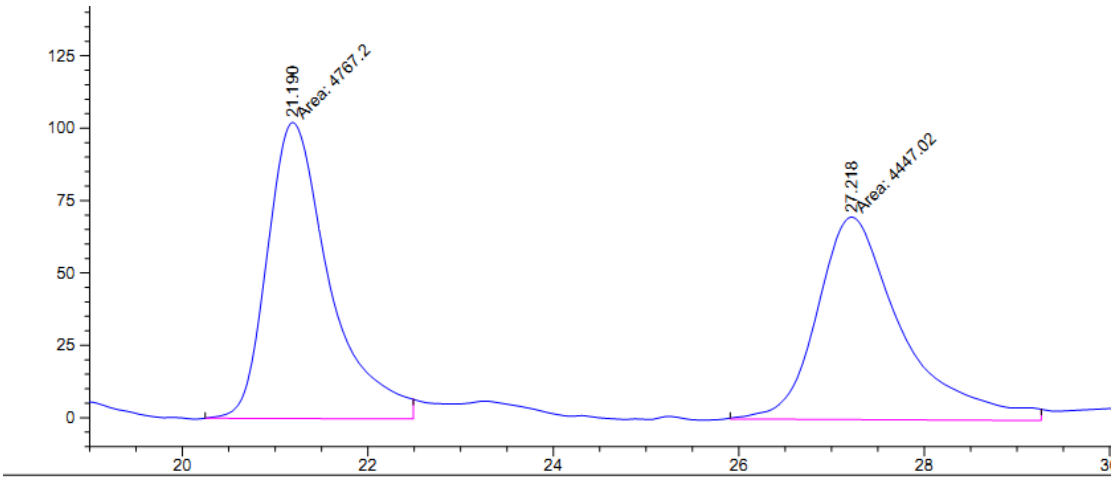


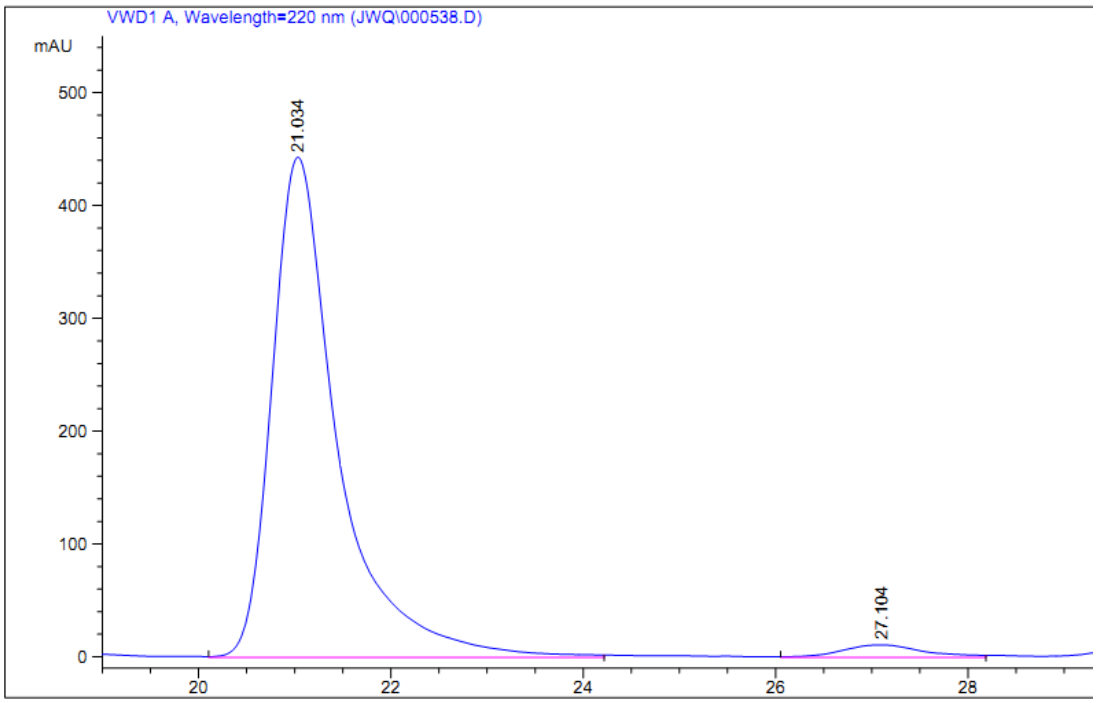


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.409	VV	0.5797	1.03695e5	2679.12549	96.4562
2	18.469	VV	0.6316	3809.75464	87.78214	3.5438

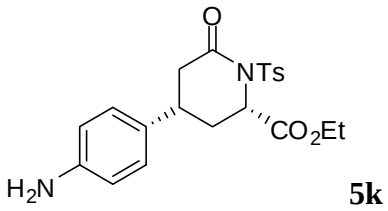


5e

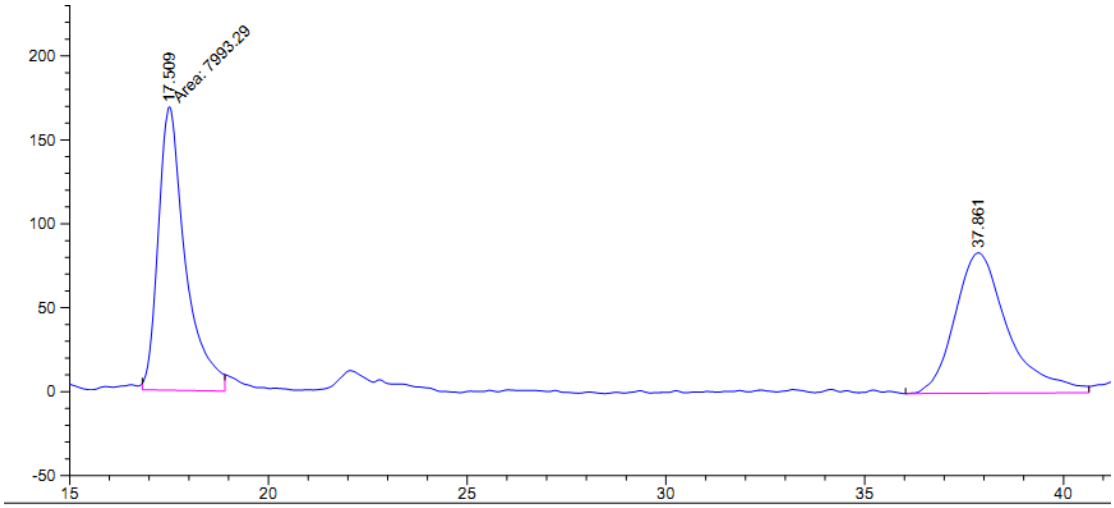


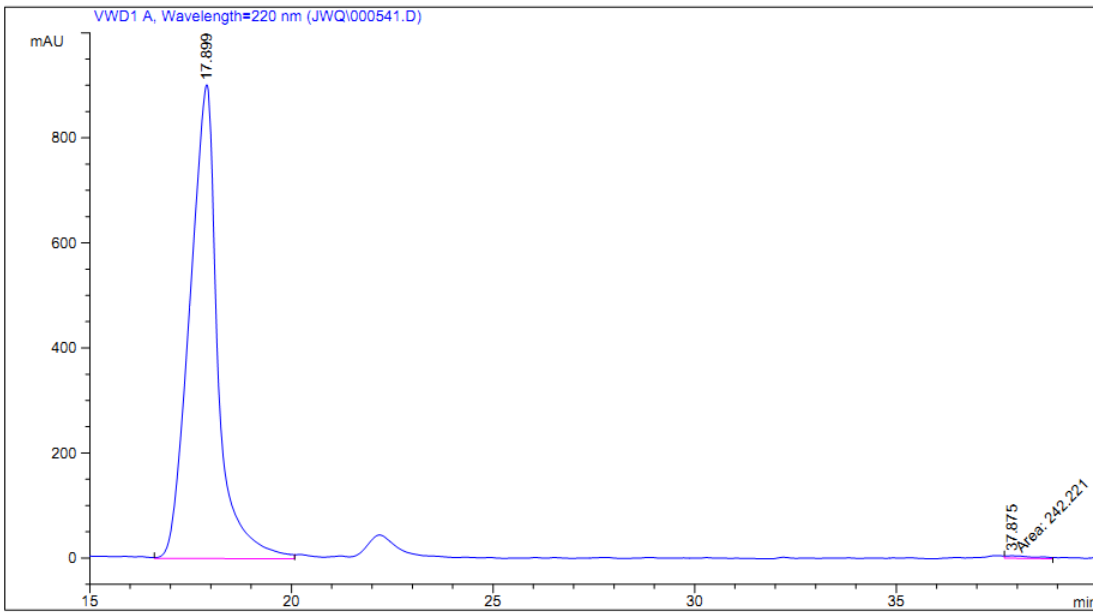


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	21.034	VV	0.7028	2.11791e4	443.43176	96.8659
2	27.104	VV	0.9050	685.24738	11.16944	3.1341

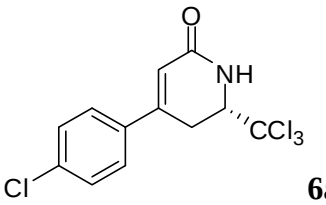


5k

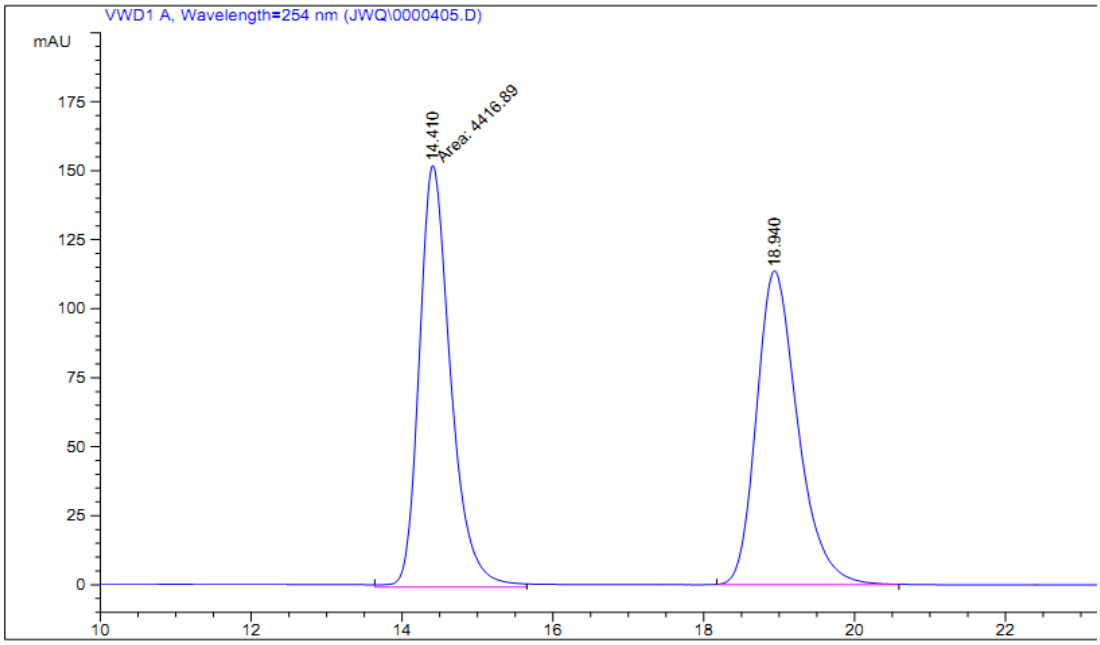


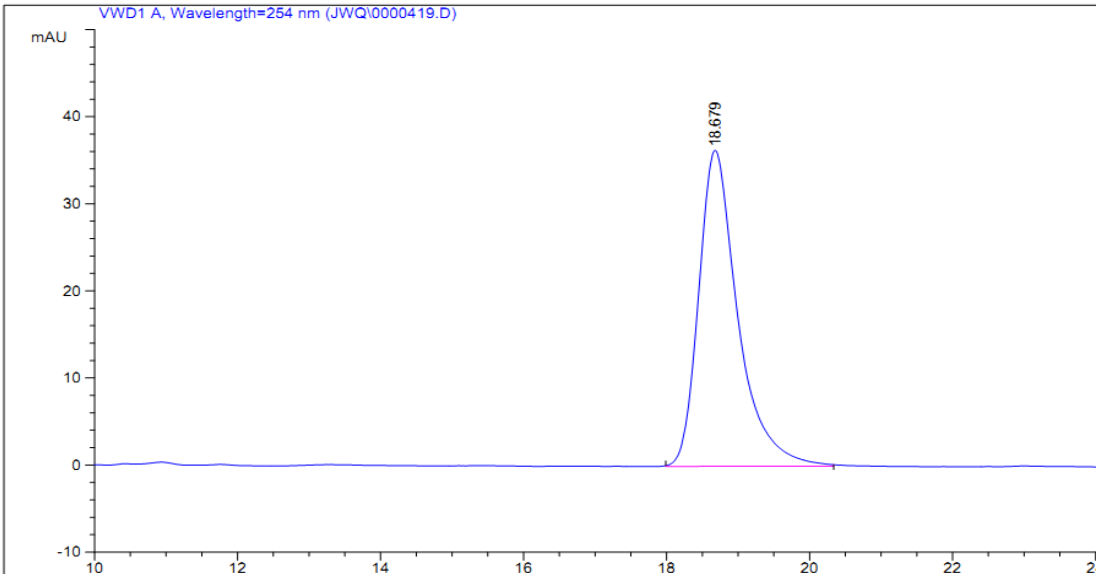


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	17.899	VV	0.7344	4.35027e4	902.05725	99.4463
2	37.875	MM	0.8844	242.22084	4.56452	0.5537

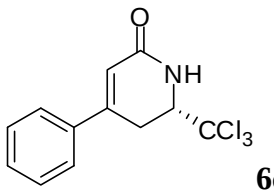


6a





Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.679	BB	0.5680	1376.66724	36.26997	100.0000



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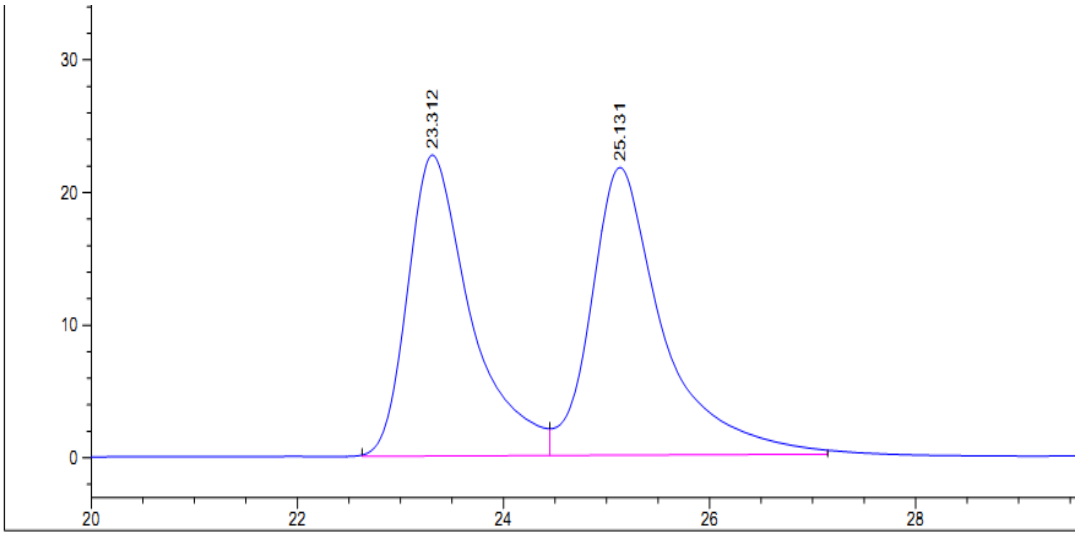
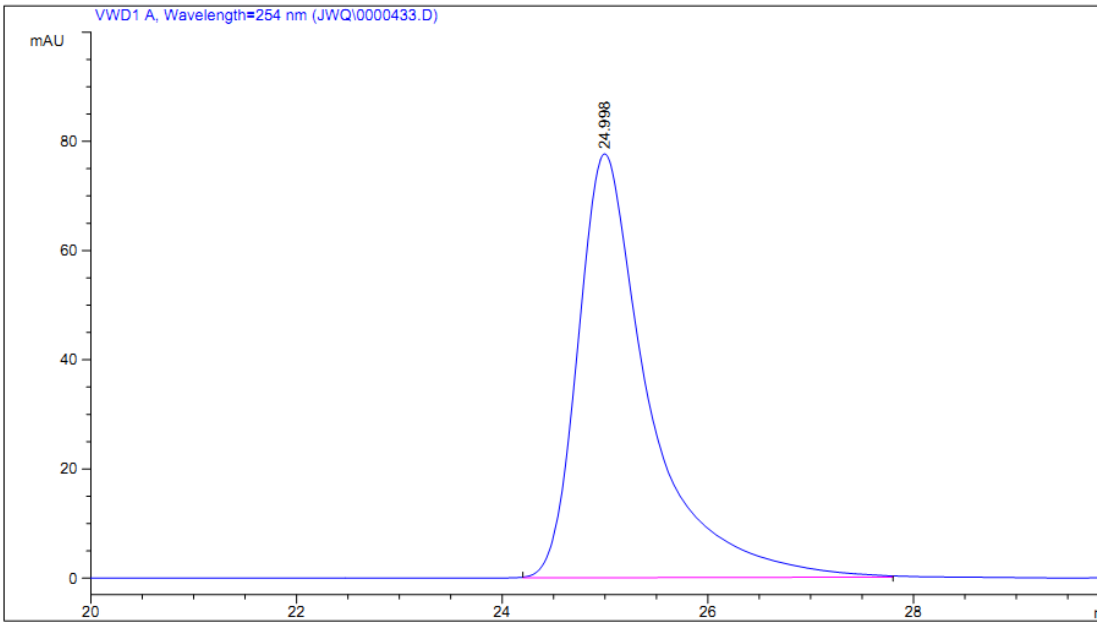
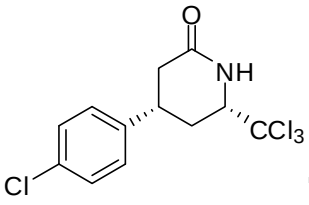




Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.998	BB	0.7017	3718.08691	77.59993	100.0000



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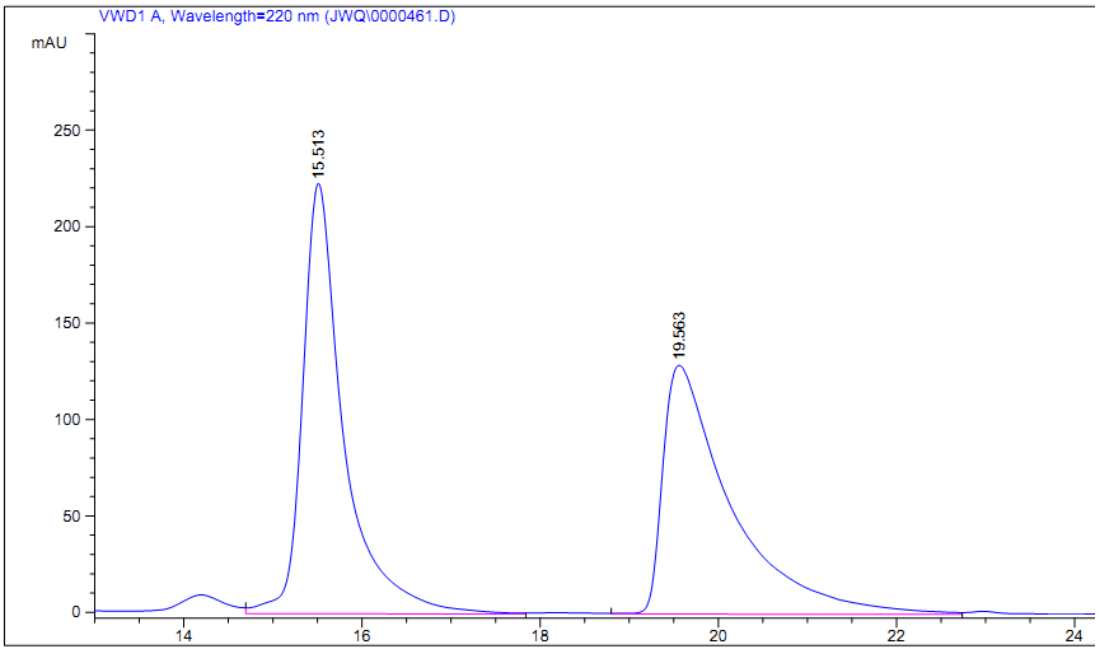
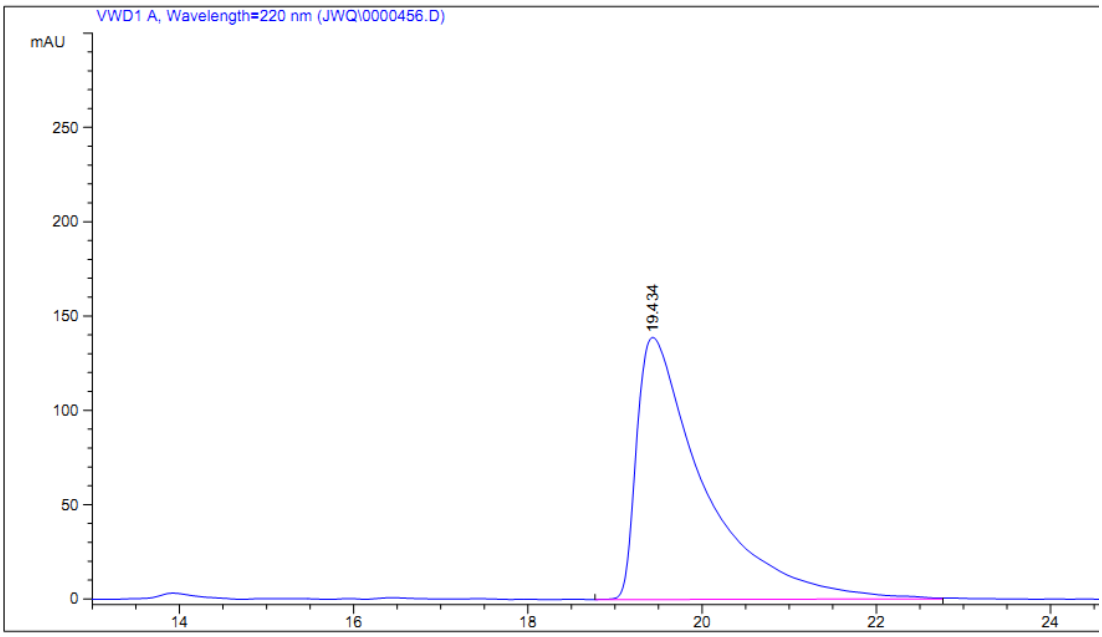
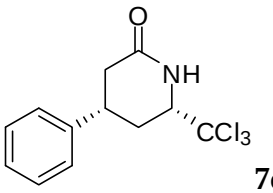




Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.434	VB	0.7575	7462.21240	138.96172	100.0000



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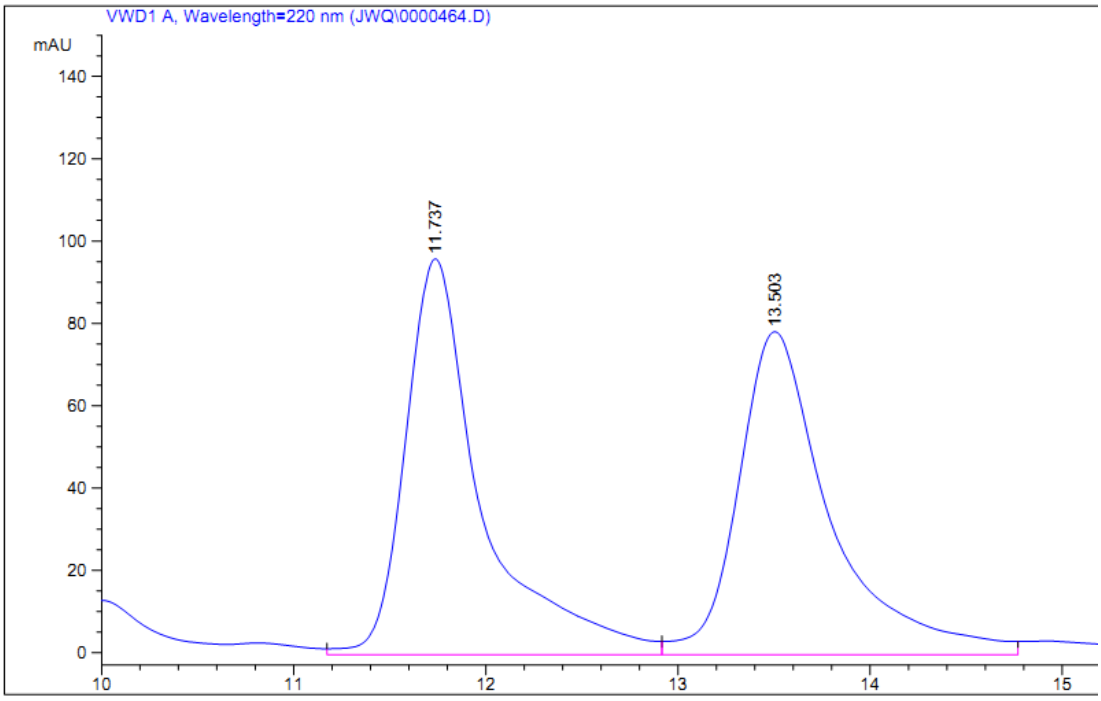
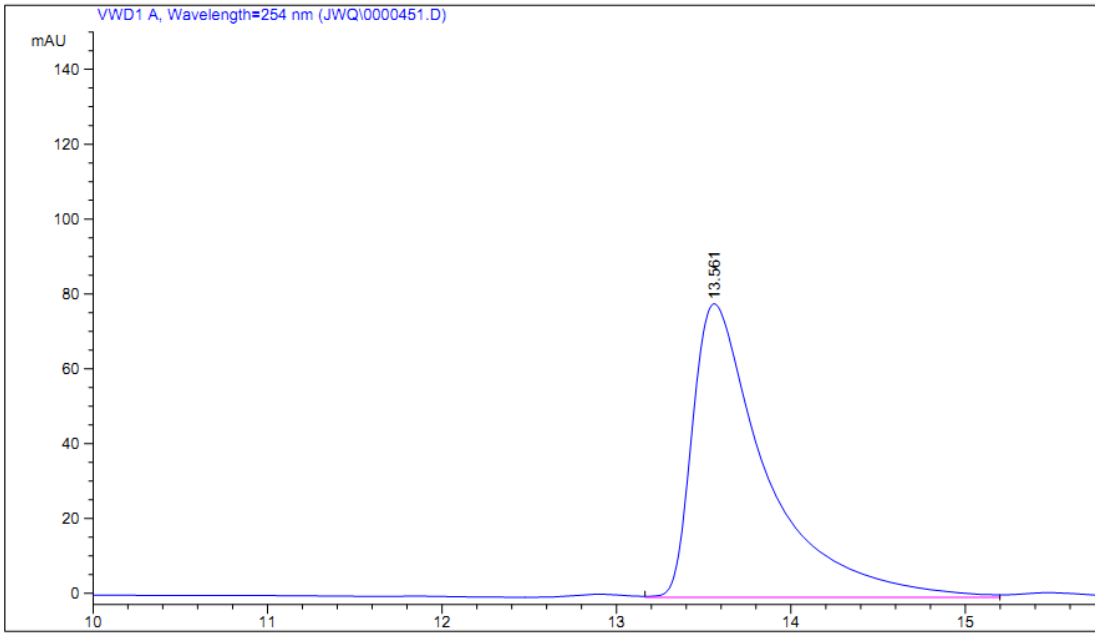
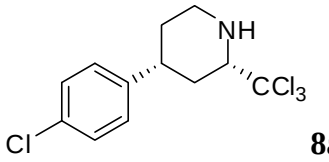
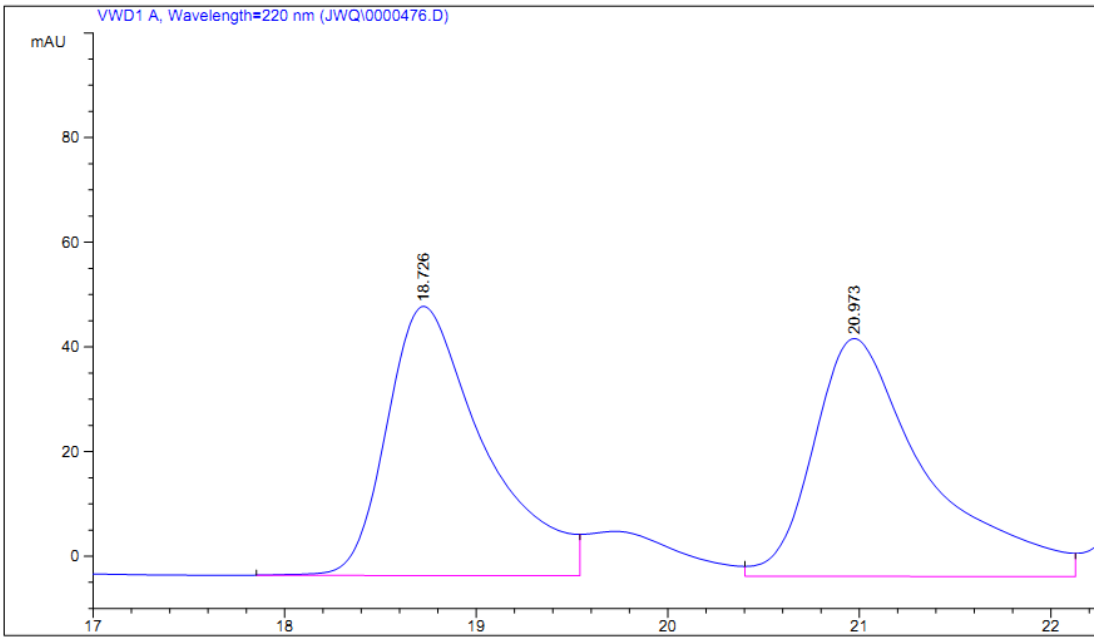


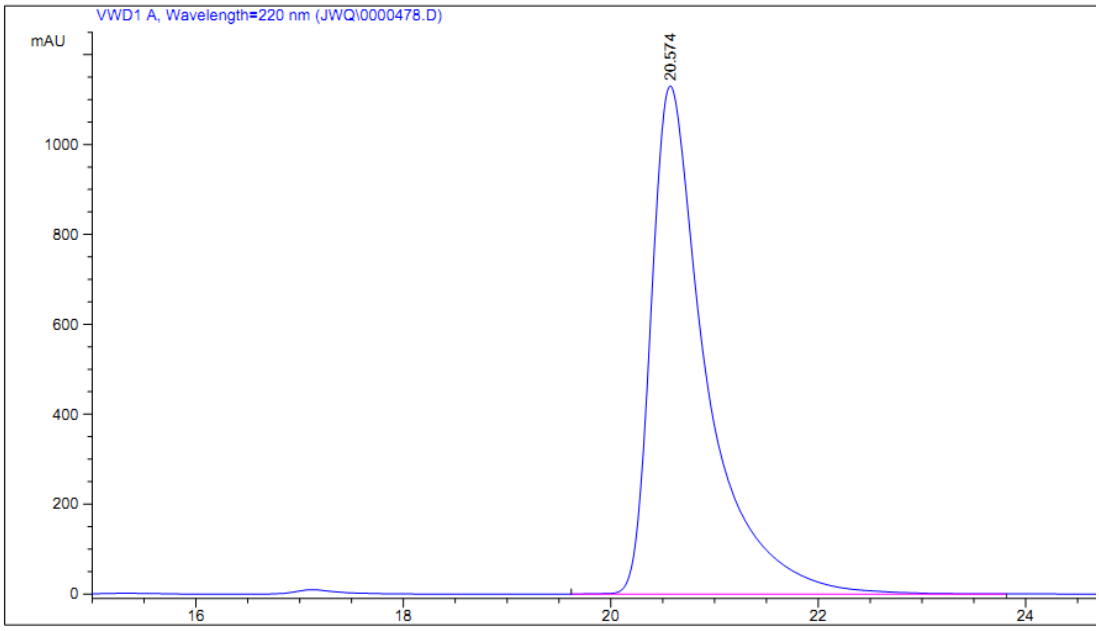


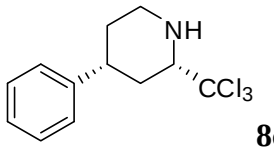
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.561	VV	0.4244	2303.67847	78.38393	100.0000


NH
CCl3
Cl

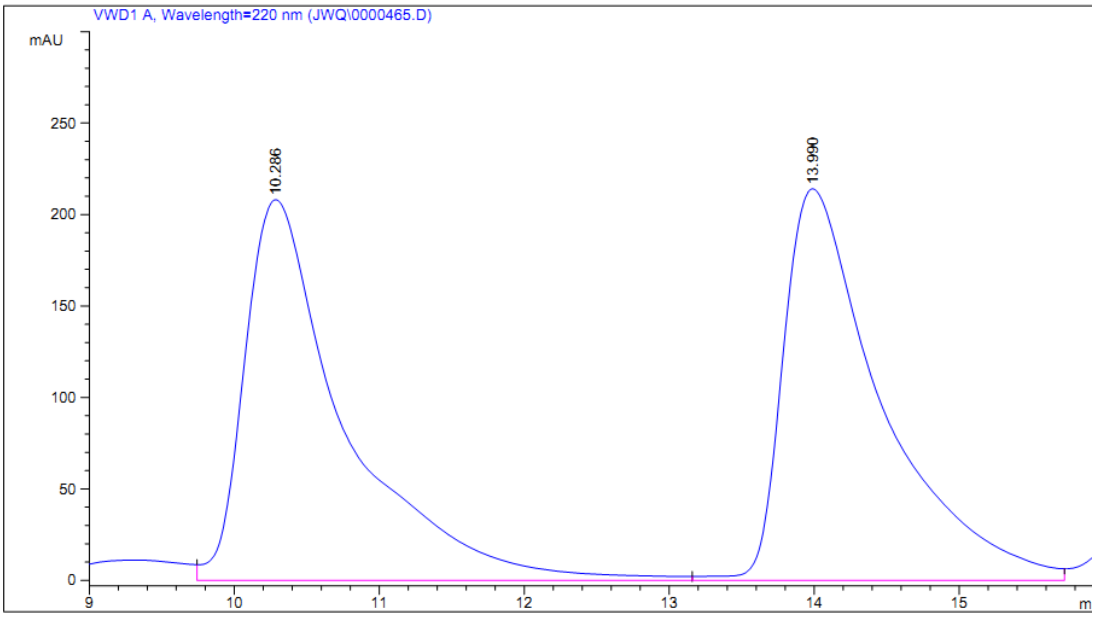
8a

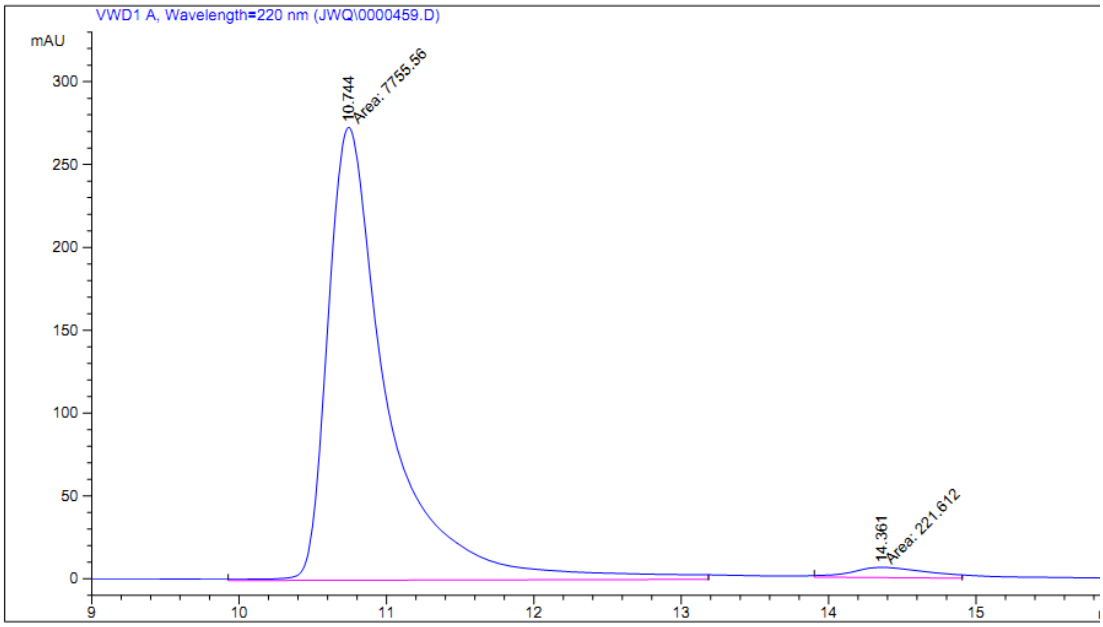

VWD1 A, Wavelength=220 nm (JWQ\0000476.D)
mAU
80
60
40
20
0
18.726
20.973
17
18
19
20
21
22


VWD1 A, Wavelength=220 nm (JWQ\0000478.D)
mAU
1000
800
600
400
200
0
20.574
16
18
20
22
24

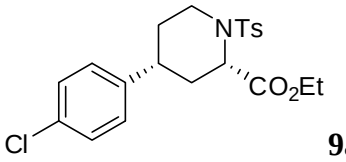


8c

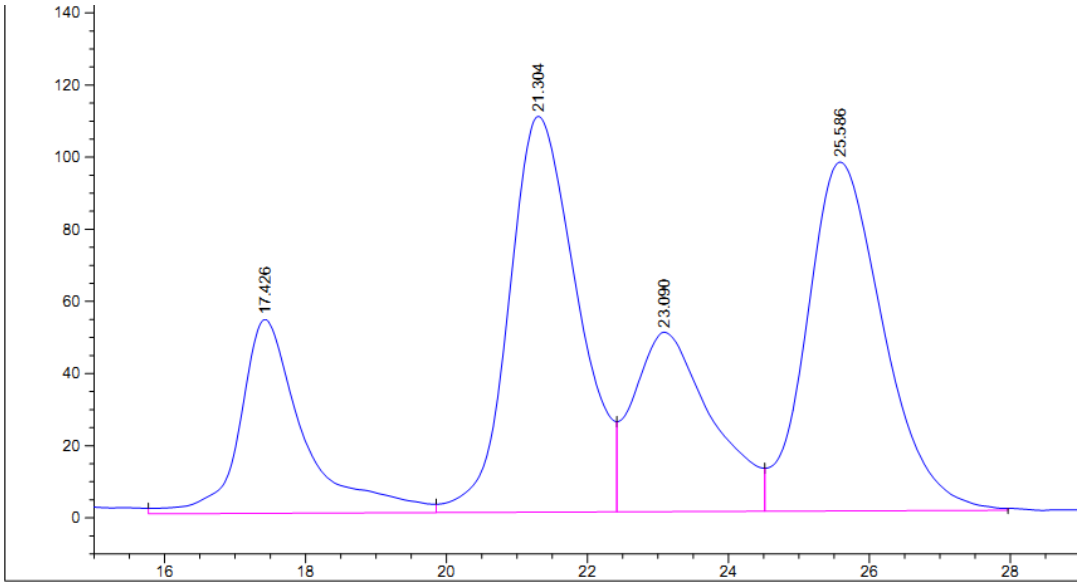


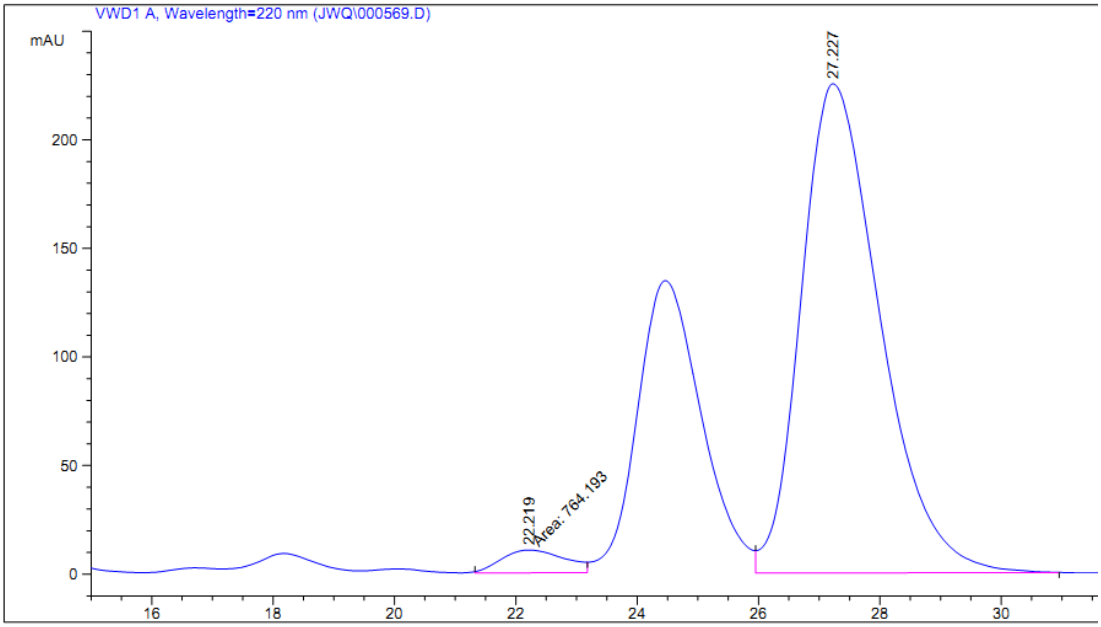


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	10.744	MM	0.4727	7755.56494	273.44739	97.2219
2	14.361	MM	0.5927	221.61246	6.23153	2.7781

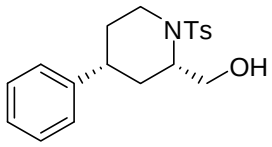


9a

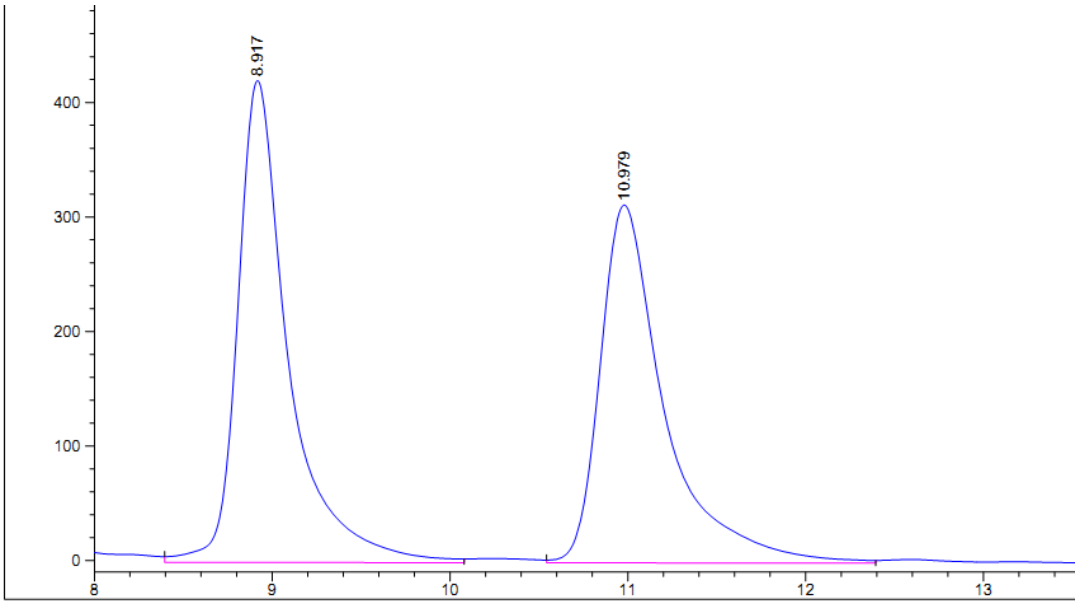


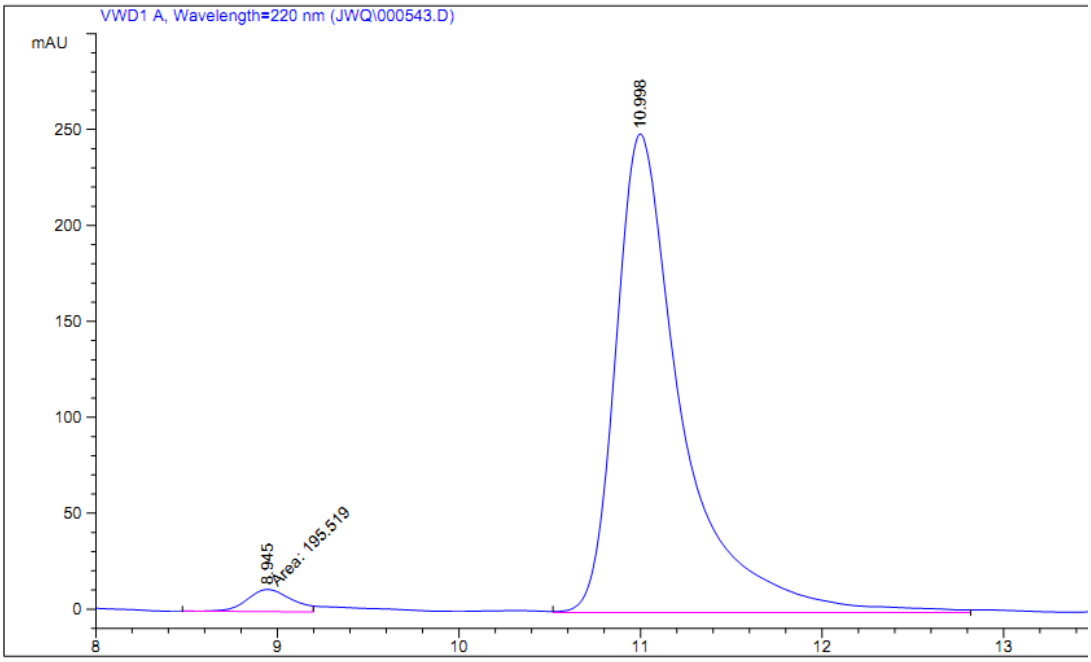


Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	22.219	MM	1.2175	764.19293	10.46087	3.6253
2	27.227	VB	1.3739	2.03151e4	225.27350	96.3747



11c





Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.945	MM	0.2818	195.51938	11.56259	3.0057
2	10.998	VV	0.3680	6309.34473	249.31384	96.9943