



Supporting Information

for

Towards an asymmetric β -selective addition of azlactones to allenates

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Full experimental and analytical details and copies of NMR spectra and HPLC traces

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1. General information

^1H -, ^{13}C - spectra were recorded on a Bruker Avance III 300 MHz spectrometer with a broad band observe probe. All NMR spectra were referenced on the solvent residual peak (CDCl_3 : δ 7.26 ppm for ^1H NMR and δ 77.16 ppm for ^{13}C NMR). NMR data are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet), coupling constants (Hz).

High-resolution mass spectra were obtained using a Thermo Fisher Scientific LTQ Orbitrap XL with an Ion Max API Source and analyses were made in the positive ionization mode if not otherwise stated.

Infrared (IR) spectra were recorded on a Bruker Alpha II FTIR spectrometer with diamond ATR-module using OPUS software package and are reported in terms of frequency of absorption (cm^{-1}).

HPLC was performed using a Shimadzu Prominence system with a diode array detector with a CHIRALPAK AD-H, CHIRAL ART Amylose-SA, (250×4.6 mm, $5 \mu\text{m}$) chiral stationary phase. Optical rotations were recorded on a Schmidt + Haensch Polarimeter Model UniPol L1000 at 589 nm ($[\alpha]_{\text{D}}$ values are listed in $\text{deg}/(\text{dm}(\text{g}/\text{cm}^3))$; concentration c is given in $\text{g}/100$ mL).

Unless otherwise stated, all chemicals were purchased from commercial suppliers and used without further purification.

Azlactones¹ and allenates² were synthesized according to known procedures.

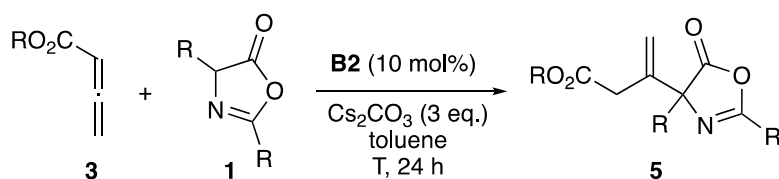
Dry solvents were obtained from an MBraun-SPS-800 solvent purification system. All reactions were carried out under argon atmosphere unless stated otherwise.

1) a) Macovei, C.; Vicennati, P.; Quinton, J.; Nevers, M.-C.; Volland, H.; Créminon, C.; Taran, F. *Chem. Commun.* **2012**, 48, 4411-4413; b) de Mello, A. C.; Momo, P. B.; Burtoloso, A. C. B.; Amarante, G. W. *J. Org. Chem.* **2018**, 83, 11399-11406; c) Žabka, M.; Kocian, A.; Bilka, S.; Andrejčák, S.; Šebesta, R. *Eur. J. Org. Chem.* **2019**, 6077-6087.

2) Zebrowski, P.; Röser, K.; Chrenko, D.; Pospíšil, J.; Waser, M. *Synthesis* **2023**, 55, 1706-1713.

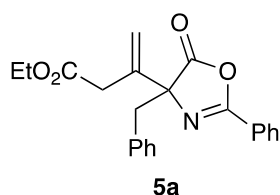
2. Asymmetric Protocol

General procedure



An oven-dried Schlenk tube equipped with a stirring bar was charged with the azlactone **1** (0.05–0.1 mmol), catalyst **B2** (10 mol % related to **1**), and Cs_2CO_3 (3 equiv). Then the respective allenolate **3** (2 equiv) and toluene (0.05 M with respect to **1**) were added and the mixture was stirred at room temperature for 24 h (Ar atmosphere). The crude product was passed through a short column of silicagel (rinsed with DCM and EtOAc), concentrated under reduced pressure, and subsequently purified by preparative TLC (silica gel, heptanes/EtOAc = 4:1) to obtain the products **2** in the given yields and enantiopurities.

Characterization of the products



Compound 5a: Obtained as a colorless oil in 61% yield with er = 81:19.

$[\alpha]_{\text{D}}^{22}$ ($c = 1.1$, CHCl_3) = -11.4° .

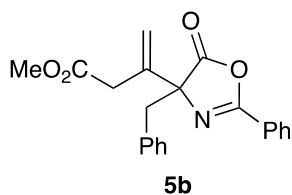
^1H NMR (300 MHz, δ , CDCl_3 , 298 K): 7.85 (2H, dd, $J = 8.6, 1.4$ Hz), 7.54 (1H, t, $J = 7.4$ Hz), 7.43 (2H, t, $J = 7.53$ Hz), 7.11–7.24 (5H, m), 5.79 (1H, s), 5.37 (1H, s), 3.90–4.14 (2H, m), 3.16–3.52 (4H, m), 1.15 (3H, t, $J = 7.1$ Hz).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 177.4, 171.0, 160.3, 139.1, 133.8, 132.6, 130.5, 128.6, 128.0, 127.8, 127.3, 125.6, 118.1, 75.9, 60.9, 44.9, 39.3, 13.9.

IR (neat): 3080, 3070, 2917, 1815, 1732, 1656, 1480, 1175, 1093, 1059, 1030, 974, 893, 694 cm^{-1} .

HRMS for $\text{C}_{22}\text{H}_{21}\text{NO}_4$ $[\text{M}+\text{H}]^+$: m/z calcd: 364.1543, found: 364.1554.

HPLC (Chiralpak SA, eluent: n -hexane:iPrOH = 100/2, 0.5 $\text{mL} \cdot \text{min}^{-1}$, 20 $^\circ\text{C}$, $\lambda = 254$ nm) retention times: $t_{\text{major}} = 16.15$ min, $t_{\text{minor}} = 17.00$ min.



Compound 5b: Obtained as a colorless oil in 67% yield with er = 80:20.

$[\alpha]_D^{22}$ (c = 0.93, CHCl₃) = -13.8°.

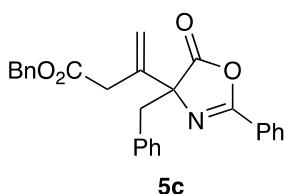
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.81-7.88 (2H, m), 7.49-7.58 (1H, m), 7.38-7.48 (2H, m), 7.13-7.23 (5H, m), 5.78 (1H, s), 5.36 (1H, s), 3.56 (3H, s), 3.20-3.48 (4H, m).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 177.4, 171.4, 160.3, 138.9, 133.7, 132.6, 130.4, 128.6, 128.0, 127.8, 127.3, 125.5, 118.2, 75.9, 51.9, 44.8, 39.1.

IR (neat): 3046, 2940, 1814, 1736, 1656, 1451, 1167, 1093, 972, 892, 779, 694 cm⁻¹.

HRMS for C₂₁H₁₉NO₄[M+H]⁺: m/z calcd: 350.1387, found: 350.1377.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/1, 0.5 mL·min⁻¹, 20 °C, λ = 254 nm) retention times: t_{major} = 52.66 min, t_{minor} = 56.59 min.



Compound 5c: Obtained as a colorless oil in 47% yield with er = 82:18.

$[\alpha]_D^{22}$ (c = 1.05, CHCl₃) = -21.4°.

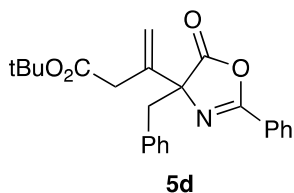
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.76-7.83 (2H, m), 7.49-7.57 (1H, m), 7.35-7.45 (2H, m), 7.23-7.35 (5H, m), 7.12-7.21 (5H, m), 5.79 (1H, s), 5.37 (1H, s), 5.01 (1H, d, J = 0.57 Hz), 3.26-3.51 (4H, m).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 177.4, 170.9, 160.3, 138.9, 135.5, 133.7, 132.6, 130.4, 128.6, 128.5, 128.5, 128.3, 128.0, 127.8, 127.3, 125.5, 118.3, 75.9, 66.6, 44.9, 39.2.

IR (neat): 3057, 2944, 1820, 1732, 1656, 1155, 1093, 1059, 971, 746, 693 cm⁻¹.

HRMS for C₂₇H₂₃NO₄ [M+H]⁺: m/z calcd: 426.1700, found: 426.1690.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 mL·min⁻¹, 20 °C, λ = 254 nm) retention times: t_{major} = 37.04 min, t_{minor} = 41.42 min.



Compound 5d: Obtained as a colorless oil in 56% yield with er = 67:33.

[α]_D²² (c = 1.1, CHCl₃) = +7.9°.

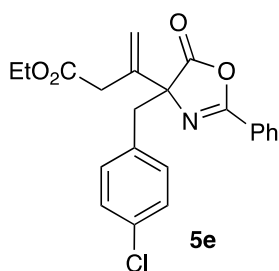
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.82-7.88 (2H, m), 7.49-7.56 (1H, m), 7.38-7.46 (2H), 7.10-7.24 (5H, m), 5.76 (1H, s), 5.34 (1H, s), 3.14-3.42 (4H, m), 1.35 (9H, s).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 177.5, 170.3, 160.1, 139.5, 132.5, 130.4, 128.5, 128.0, 127.2, 125.7, 117.8, 81.0, 76.0, 44.9, 40.3, 27.8.

IR (neat): 3040, 2932, 1814, 1718, 1655, 1148, 1096, 1058, 969, 699 cm⁻¹.

HRMS for C₂₄H₂₅NO₄ [M+H]⁺: m/z calcd: 392.1856, found: 392.1860.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 mL·min⁻¹, 20 °C, λ = 254 nm) retention times: t_{major} = 13.73 min, t_{minor} = 14.74 min.



Compound 5e: Obtained as a yellow oil in 65% yield with er = 77:23.

[α]_D²² (c = 0.97, CHCl₃) = -20.1°.

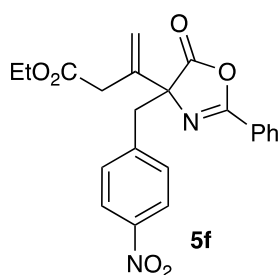
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 8.06 (2H, d, J = 8.76 Hz), 7.82-7.90 (1H, m), 7.52-7.60 (2H, m), 7.35-7.50 (4H, m), 5.79 (1H, s), 5.37 (1H, s), 5.01 (1H, d, J = 0.57 Hz), 3.98-4.22 (2H, m), 3.26-3.51 (4H, m), 1.15 (3H, t, J = 7.14 Hz).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 177.2, 170.9, 160.5, 138.9, 133.3, 132.8, 132.3, 131.7, 128.7, 128.2, 127.8, 125.4, 118.3, 75.6, 61.0, 44.1, 39.3, 13.9.

IR (neat): 3055, 2923, 1810, 1731, 1657, 1509, 1480, 1158, 1095, 1016, 803, 712 cm^{-1} .

HRMS for $\text{C}_{22}\text{H}_{20}\text{ClNO}_4$ $[\text{M}+\text{H}]^+$: m/z calcd: 398.1154, found: 398.1149.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 150/1, 0.5 $\text{mL} \cdot \text{min}^{-1}$, 20 $^\circ\text{C}$, λ = 254 nm) retention times: t_{minor} = 39.63 min, t_{major} = 41.52 min.



Compound 5f: Obtained as a yellow oil in 49% yield with er = 79:21.

$[\alpha]_{\text{D}}^{22}$ (c = 0.98, CHCl_3) = -13.7° .

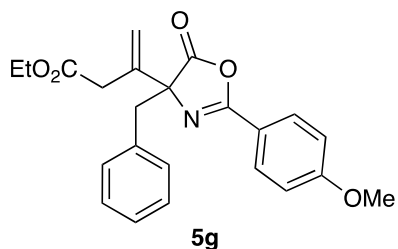
^1H NMR (300 MHz, δ , CDCl_3 , 298 K): 8.06 (2H, d, J = 8.76 Hz), 7.82-7.90 (2H, m), 7.53-7.61 (1H, m), 7.35-7.50 (4H, m), 5.78 (1H, s), 5.39 (1H, s), 3.93-4.09 (2H, m), 3.18-3.56 (4H, m), 1.15 (3H, t, J = 7.14 Hz).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 176.9, 170.8, 160.8, 147.3, 141.5, 138.6, 133.1, 131.4, 128.8, 127.8, 125.0, 123.2, 118.7, 75.2, 61.0, 44.3, 39.2, 13.9.

IR (neat): 3053, 2919, 1812, 1731, 1657, 1513, 1473, 1345, 1257, 1180, 1110, 1028, 833, 699, 523 cm^{-1} .

HRMS for $\text{C}_{22}\text{H}_{20}\text{N}_2\text{O}_6$ $[\text{M}+\text{H}]^+$: m/z calcd: 409.1394, found: 409.1398.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 $\text{mL} \cdot \text{min}^{-1}$, 20 $^\circ\text{C}$, λ = 254 nm) retention times: t_{minor} = 52.67 min, t_{major} = 55.12 min.



Compound 5g: Obtained as a colorless oil in 51% yield with er = 82:18.

$[\alpha]_{\text{D}}^{22}$ (c = 1.00, CHCl₃) = -36.9°.

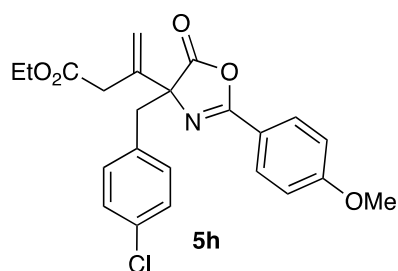
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.79 (2H, d, *J* = 8.9 Hz), 7.13-7.22 (5H, m), 6.92 (2H, d, *J* = 8.9 Hz), 5.77 (1H, s), 5.35 (1H, s), 4.01 (2H, m), 3.86 (3H, s), 3.18-3.45 (4H, m), 1.15 (3H, t, *J* = 7.1 Hz).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 177.6, 171.0, 163.0, 159.9, 139.3, 133.9, 130.4, 129.7, 128.0, 127.2, 117.9, 117.9, 114.0, 75.8, 60.8, 55.4, 44.9, 39.4, 13.9.

IR (neat): 3020, 3048, 2943, 1813, 1731, 1654, 1607, 1511, 1256, 1170, 1093, 1028, 974, 840, 700 cm⁻¹.

HRMS for C₂₃H₂₃NO₅ [M+H]⁺: m/z calcd: 394.1649, found: 394.1665.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 mL·min⁻¹, 20 °C, λ = 254 nm) retention times: *t*_{major} = 38.93 min, *t*_{minor} = 48.14 min.



Compound 5h: Obtained as a colorless oil in 80% yield with er = 81:19.

$[\alpha]_{\text{D}}^{22}$ (c = 0.90, CHCl₃) = -73°.

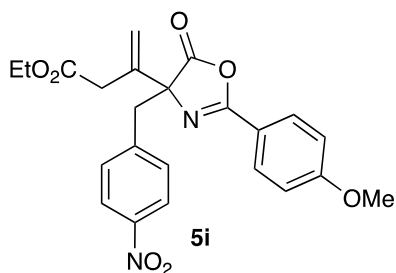
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.81 (2H, d, *J* = 8.9 Hz), 7.09-7.19 (4H, m), 6.94 (2H, d, *J* = 8.9 Hz), 5.35 (1H, s), 5.75 (1H, s), 3.91-4.10 (2H, m), 3.87 (3H, s), 3.15-3.44 (4H, m), 1.16 (3H, t, *J* = 7.1 Hz).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 177.4, 170.9, 163.2, 160.2, 139.2, 133.2, 132.5, 131.8, 129.7, 128.2, 118.1, 117.7, 114.1, 75.5, 60.9, 55.4, 44.1, 39.3, 13.9.

IR (neat): 3010, 2727, 1813, 1731, 1653, 1607, 1511, 1034, 1257, 1171, 1068, 1027, 975, 838, 742, 604, 511 cm^{-1} .

HRMS for $\text{C}_{23}\text{H}_{22}\text{ClNO}_5$ $[\text{M}+\text{H}]^+$: m/z calcd: 428.1259, found: 428.1275.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 $\text{mL} \cdot \text{min}^{-1}$, 20 $^\circ\text{C}$, λ = 254 nm) retention times: t_{major} = 40.07 min, t_{minor} = 42.30 min.



Compound 5i: obtained as a pale-yellow oil in 80% yield with er = 78:22.

$[\alpha]_{\text{D}}^{22}$ (c = 1.1, CHCl_3) = -22° .

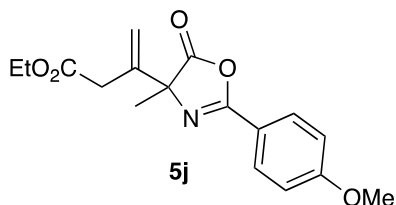
^1H NMR (300 MHz, δ , CDCl_3 , 298 K): 8.06 (2H, d, J = 8.76 Hz), 7.80 (2H, d, J = 8.97 Hz), 7.39 (2H, d, J = 8.76 Hz), 6.93 (2H, d, J = 8.97 Hz), 5.77 (1H, s), 5.38 (1H, s), 3.95-4.07 (2H, m), 3.88 (3H, s), 3.18-3.52 (4H, m), 1.16 (3H, t, J = 7.1 Hz).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 177.1, 170.8, 163.4, 160.5, 147.2, 141.7, 138.9, 131.4, 129.7, 123.1, 118.5, 117.3, 114.2, 75.1, 61.0, 55.4, 44.3, 39.3, 13.9.

IR (neat): 3040, 2946, 2765, 1814, 1727, 1656, 1606, 1511, 1341, 1299, 1254, 1171, 1084, 1033, 840, 713, 498 cm^{-1} .

HRMS for $\text{C}_{23}\text{H}_{22}\text{N}_2\text{O}_7$ $[\text{M}+\text{H}]^+$: m/z calcd: 439.1500., found: 439.1496.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.8 $\text{mL} \cdot \text{min}^{-1}$, 20 $^\circ\text{C}$, λ = 254 nm) retention times: t_{major} = 55.99 min, t_{minor} = 59.93 min.



Compound 5j: Obtained as a colorless oil in 67% yield with er = 78:22.

$[\alpha]_D^{22}$ (c = 0.97, CHCl₃) = +19.7°.

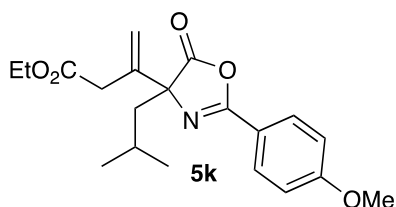
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.96 (2H, d, *J* = 8.9 Hz), 7.01 (2H, d, *J* = 9.0 Hz), 5.60 (1H, s), 5.29 (1H, s), 3.97-4.10 (2H, m), 3.90 (3H, s), 3.37 (1H, dd, *J* = 16.2, 0.9 Hz), 3.17 (1H, dd, *J* = 16.2, 0.8 Hz), 1.17 (3H, t, *J* = 7.14 Hz).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 178.7, 170.9, 163.3, 160.3, 139.9, 129.9, 118.0, 117.5, 114.2, 71.0, 60.9, 55.5, 38.8, 25.2, 13.9.

IR (neat): 3080, 2911, 1973, 1750, 1814, 1733, 1655, 1449, 1157, 1031, 928, 692 cm⁻¹.

HRMS for C₁₇H₁₉NO₅ [M+H]⁺: *m/z* calcd: 288.1230, found: 288.1238.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 mL·min⁻¹, 20 °C, λ = 254 nm) retention times: *t*_{major} = 31.90 min, *t*_{minor} = 45.37 min.



Compound 5k: Obtained as a colorless oil in 46% yield with er = 83:17.

$[\alpha]_D^{22}$ (c = 0.91 CHCl₃) = +5.4°.

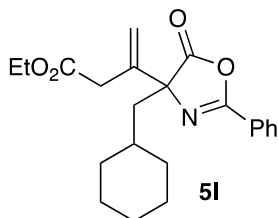
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.95 (2H, d, *J* = 8.9 Hz), 6.97 (2H, d, *J* = 8.9 Hz), 5.57 (1H, s), 5.21 (1H, s), 3.90-4.09 (2H, m), 3.87 (3H, s), 3.35 (1H, d, *J* = 16.4 Hz), 3.16 (1H, d, *J* = 16.2 Hz), 2.08 (1H, dd, *J* = 13.8, 5.1 Hz), 1.83 (1H, dd, *J* = 13.8, 7.4 Hz), 1.58-1.73 (1H, m), 1.13 (3H, t, *J* = 7.1 Hz), 0.82-0.91 (6H, m).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 178.9, 171.0, 163.2, 159.8, 140.1, 129.8, 118.2, 117.1, 114.1, 74.4, 60.8, 55.5, 47.3, 39.0, 24.9, 24.2, 23.0, 13.9.

IR (neat): 3045, 2955, 1810, 1733, 1654, 1607, 1511, 1305, 1256, 1170, 1028, 970, 885, 840, 734, 604 cm⁻¹.

HRMS for $C_{20}H_{25}NO_5$ $[M+H]^+$: m/z calcd: 360.1805, found: 360.1806.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, $0.5 \text{ mL} \cdot \text{min}^{-1}$, $20 \text{ }^\circ\text{C}$, $\lambda = 254 \text{ nm}$) retention times: $t_{\text{major}} = 23.55 \text{ min}$, $t_{\text{minor}} = 31.88 \text{ min}$.



Compound 5l: Obtained as a colorless oil 65% yield with er = 81:19.

$[\alpha]_D^{22}$ ($c = 0.94$, CHCl_3) = $+6.9^\circ$.

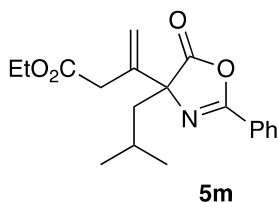
$^1\text{H NMR}$ (300 MHz, δ , CDCl_3 , 298 K): 7.89-7.98 (2H, m), 7.47-7.54 (1H, m), 7.37-7.46 (2H, m), 5.51 (1H, s), 5.16 (1H, s), 3.81-4.01 (2H, m), 2.96-3.35 (2H, m), 1.99 (1H, dd, $J = 14.2, 4.8 \text{ Hz}$), 1.76 (1H, dd, $J = 7.0, 14 \text{ Hz}$), 1.38-1.71 (13H, m), 1.05 (1H, t, $J = 7.1 \text{ Hz}$).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 178.7, 171.0, 160.1, 139.7, 132.7, 128.7, 127.9, 125.9, 117.3, 74.4, 60.8, 46.0, 39.0, 34.6, 34.1, 33.6, 26.1, 26.1, 26.0, 13.9.

IR (neat): 3040, 2922, 2852, 1816, 1735, 1656, 1449, 1320, 1156, 1031, 972, 693 cm^{-1} .

HRMS for $C_{23}H_{27}NO_4$ $[M+H]^+$: m/z calcd: 370.2013, found: 370.2014.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, $0.5 \text{ mL} \cdot \text{min}^{-1}$, $20 \text{ }^\circ\text{C}$, $\lambda = 254 \text{ nm}$) retention times: $t_{\text{minor}} = 13.73 \text{ min}$, $t_{\text{major}} = 15.60 \text{ min}$.



Compound 5m: Obtained as a colorless oil in 70% yield with er = 79:21.

$[\alpha]_D^{22}$ ($c = 0.95$, CHCl_3) = $+19.7^\circ$.

$^1\text{H NMR}$ (300 MHz, δ , CDCl_3 , 298 K): (2H, m), 7.56-7.64 (1H, m), 7.46-7.55 (2H, m), 5.62 (1H, s), 5.26 (1H, s), 3.90-4.06 (2H, m), 3.38 (1H, dd, $J = 16.4, 0.9 \text{ Hz}$), 3.20 (1H, d, $J = 16.3 \text{ Hz}$), 2.13 (1H,

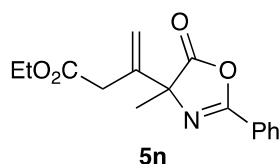
dd, $J = 13.9, 5.0$ Hz), 1.87 (1H, dd, $J = 13.9, 7.4$ Hz), 1.61-1.77 (1H, m), 1.15 (3H, t, $J = 7.14$ Hz), 0.81-0.94 (6H, m).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 178.7, 170.9, 160.1, 139.8, 132.7, 128.7, 128.4, 127.9, 117.3, 74.5, 60.8, 47.3, 38.9, 24.9, 24.2, 22.5.

IR (neat): 3049, 2827, 2923, 2712, 1814, 1734, 1656, 1451, 1321, 1258, 1155, 1030, 970, 779, 700 cm^{-1} .

HRMS for $\text{C}_{19}\text{H}_{23}\text{NO}_4$ $[\text{M}+\text{H}]^+$: m/z calcd: 330.1700, found: 330.1707.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 $\text{mL} \cdot \text{min}^{-1}$, 20 $^\circ\text{C}$, $\lambda = 254$ nm) retention times: $t_{\text{major}} = 12.38$ min, $t_{\text{minor}} = 14.14$ min.



Compound 5n: Obtained as a colorless oil in 71% yield with er = 75:25.

$[\alpha]_{\text{D}}^{22}$ ($c = 0.98$, CHCl_3) = +19.7 $^\circ$.

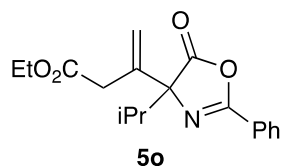
^1H NMR (300 MHz, δ , CDCl_3 , 298 K): 7.93 (2H, dd, $J = 8.6, 1.4$ Hz), 7.51 (1H, t, $J = 7.5$ Hz), 7.42 (2H, t, $J = 7.4$ Hz), 5.52 (1H, s), 5.21 (1H, s), 3.86-4.04 (2H, m), 3.09 (1H, d, $J = 16.3$ Hz), 3.29 (1H, d, $J = 16.3$ Hz), 1.61 (3H, s), 1.07 (3H, t, $J = 7.14$ Hz).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 178.55, 170.90, 160.62, 139.65, 132.86, 129.20, 128.78, 128.50, 127.99, 119.06, 117.67, 71.17, 60.94, 38.84, 25.18, 13.95.

IR (neat): 3012, 2818, 2714, 1815, 1712, 1646, 1515, 1484, 1369, 1161, 1027, 915, 711 cm^{-1} .

HRMS for $\text{C}_{16}\text{H}_{17}\text{NO}_4$ $[\text{M}+\text{H}]^+$: m/z calcd: 288.1230, found: 288.1238.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 $\text{mL} \cdot \text{min}^{-1}$, 20 $^\circ\text{C}$, $\lambda = 254$ nm) retention times: $t_{\text{major}} = 16.47$ min, $t_{\text{minor}} = 19.96$ min.



Compound 5o: Obtained as a colorless oil in 81% yield with er = 74:26.

$[\alpha]_D^{22}$ (c = 0.97 CHCl₃) = +25.7.

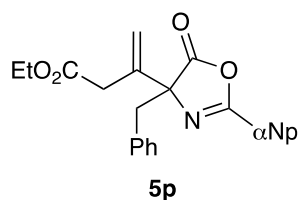
¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.99-8.08 (2H, m), 7.55-7.63 (1H, m), 7.45-7.50 (2H, m), 5.64 (1H, s), 5.31 (1H, s), 3.87-4.03 (2H, m), 3.38 (1H, dd, *J* = 16.3, 1.0 Hz), 3.22 (1H, dd, *J* = 16.3, 0.6 Hz), 2.50-2.45 (1H, m), 1.14 (3H, t, *J* = 7.1 Hz), 1.07 (3H, d, *J* = 6.7 Hz), 0.89 (3H, d, *J* = 6.7 Hz).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 178.3, 171.0, 160.5, 138.9, 132.7, 128.7, 128.0, 125.8, 117.7, 78.3, 60.8, 39.3, 36.5, 16.7, 16.7, 13.9.

IR (neat): 3042, 2945, 1840, 1734, 1657, 1322, 1156, 1059, 1026, 964, 882, 798, 692 cm⁻¹.

HRMS for C₁₈H₂₁NO₄ [M+H]⁺: m/z calcd: 316.1543, found: 316.1558.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 mL·min⁻¹, 20 °C, λ = 254 nm) retention times: *t*_{major} = 12.21 min, *t*_{minor} = 16.53 min.



Compound 5p: Obtained as a colorless oil 91% yield with er = 73:27

$[\alpha]_D^{22}$ (c = 1.00, CHCl₃) = -35.2°.

¹H NMR (300 MHz, δ, CDCl₃, 298 K): 8.19 (1H, s), 7.89 (1H, dd, *J* = 8.6, 1.6 Hz), 7.75-7.83 (3H, m), 7.40-7.51 (2H, m), 7.00-7.16 (5H, m), 5.73 (1H, s), 5.30 (1H, s), 3.81-4.00 (2H, m), 3.11-3.43 (4H, m), 1.05 (3H, t, *J* = 7.14 Hz).

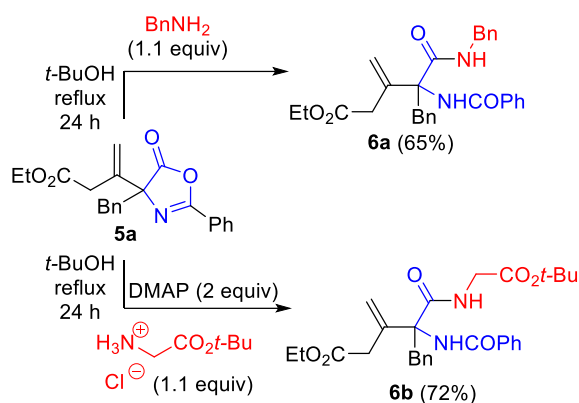
¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 177.4, 171.0, 160.4, 139.2, 135.3, 133.8, 132.4, 130.4, 129.2, 129.1, 128.6, 128.3, 128.1, 127.9, 127.3, 126.9, 123.4, 122.8, 118.1, 76.1, 60.9, 45.0, 39.4, 13.9.

IR (neat): 3060, 2958, 1817, 1731, 1653, 1233, 1157, 1093, 1030, 986, 943, 901, 864, 822, 755, 700, 475 cm⁻¹.

HRMS for C₂₆H₂₃NO₄ [M+H]⁺: m/z calcd: 414.1700, found: 414.1712.

HPLC (Chiralpak AD-H, eluent: *n*-hexane:iPrOH = 100/3, 0.5 mL·min⁻¹, 20 °C, λ = 254 nm) retention times: $t_{\text{major}} = 30.22$ min, $t_{\text{minor}} = 33.44$ min.

3. Ring opening reactions



Compound 6a:

In a flame-dried Schlenk tube under Ar atmosphere at room temperature, 0.08 mmol of compound **5a** in 0.5 mL *tert*-butyl alcohol and 0.09 mmol of benzylamine (1.1 equivalents), were added. This mixture was refluxed for 24 hours in a preheated oil bath. After cooling to room temperature and evaporation of solvent under vacuum, the crude reaction mixture was purified through PTLC silica gel chromatography, using 33% ethyl acetate in heptane as eluent, yielding 65% of the pure product (0.024 g).

^1H NMR (300 MHz, δ , CDCl_3 , 298 K): 7.96 (1H, s), 7.66-7.78 (3H, m), 7.46-7.54 (1H, m), 7.30-7.45 (7H, m), 7.06-7.22 (3H, m), 6.87-6.94 (2H, m), 5.54 (1H, s), 5.43 (1H, s), 4.42-4.60 (2H, m), 4.16 (2H, q, $J = 7.1$ Hz), 4.03 (1H, d, $J = 12.9$ Hz), 3.71 (1H, d, $J = 17.3$ Hz), 3.27 (1H, d, $J = 17.4$ Hz), 3.13 (1H, d, $J = 12.9$ Hz), 1.27 (3H, t, $J = 7.14$ Hz).

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 MHz, δ , CDCl_3 , 298 K): 173.4, 171.3, 165.9, 143.5, 137.7, 135.3, 134.6, 131.5, 130.0, 128.7, 128.5, 128.1, 128.0, 127.6, 127.0, 126.9, 119.6, 65.9, 61.3, 44.3, 39.1, 38.4, 14.1.

IR (neat): 3347, 3070, 2662, 2980, 1733, 1670, 1643, 1504, 1474, 1243, 1028, 695, 600 cm^{-1} .

HRMS for $\text{C}_{29}\text{H}_{30}\text{N}_2\text{O}_4$ $[\text{M}+\text{H}]^+$: m/z calcd. 471.2278, found: 471.2283.

Compound 6b:

In a flame-dried Schlenk tube under argon atmosphere 0.08 mmol of compound **5a** in 0.5 mL *tert*-butyl alcohol were combined with 0.16 mmol DMAP (2 equiv) and 0.09 mmol of glycine *tert*-butyl ester hydrochloride (1.1 equivalents). This mixture was refluxed for 24 hours in a preheated oil bath. After cooling to room temperature and evaporation of solvent under vacuum, the crude reaction mixture was purified through PTLC silica gel chromatography using 33% ethyl acetate in heptane as eluent, yielding 72% of the pure product (0.028 g).

¹H NMR (300 MHz, δ, CDCl₃, 298 K): 7.80 (1H, s), 7.65-7.74 (2H, m), 7.45-7.57 (2H, m), 7.36-7.45 (2H), 7.12-7.23 (3H, m), 7.03-7.11 (2H, m), 5.66 (1H, s), 5.52 (1H, s), 3.88-4.20 (5H, m), 3.60 (1H, d, *J* = 17.0 Hz), 3.20-3.31 (2H, m), 1.53 (9H, s), 1.25 (3H, t, *J* = 7.1 Hz).

¹³C{¹H} NMR (75 MHz, δ, CDCl₃, 298 K): 172.9, 171.2, 168.3, 166.0, 143.0, 135.4, 134.6, 131.5, 130.1, 128.5, 128.0, 127.0, 119.6, 82.4, 66.2, 61.2, 42.5, 39.1, 38.6, 28.0, 14.0.

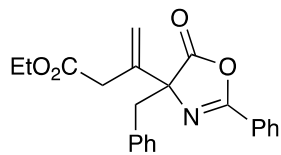
IR (neat): 3359, 1749, 1735, 1646, 1508, 1477, 1368, 1248, 1217, 1152, 1031, 745, 689, 597, 451 cm⁻¹.

HRMS for C₂₈H₃₄N₂O₆ [M+H]⁺: *m/z* calcd. 495.2489, found: 495.248.

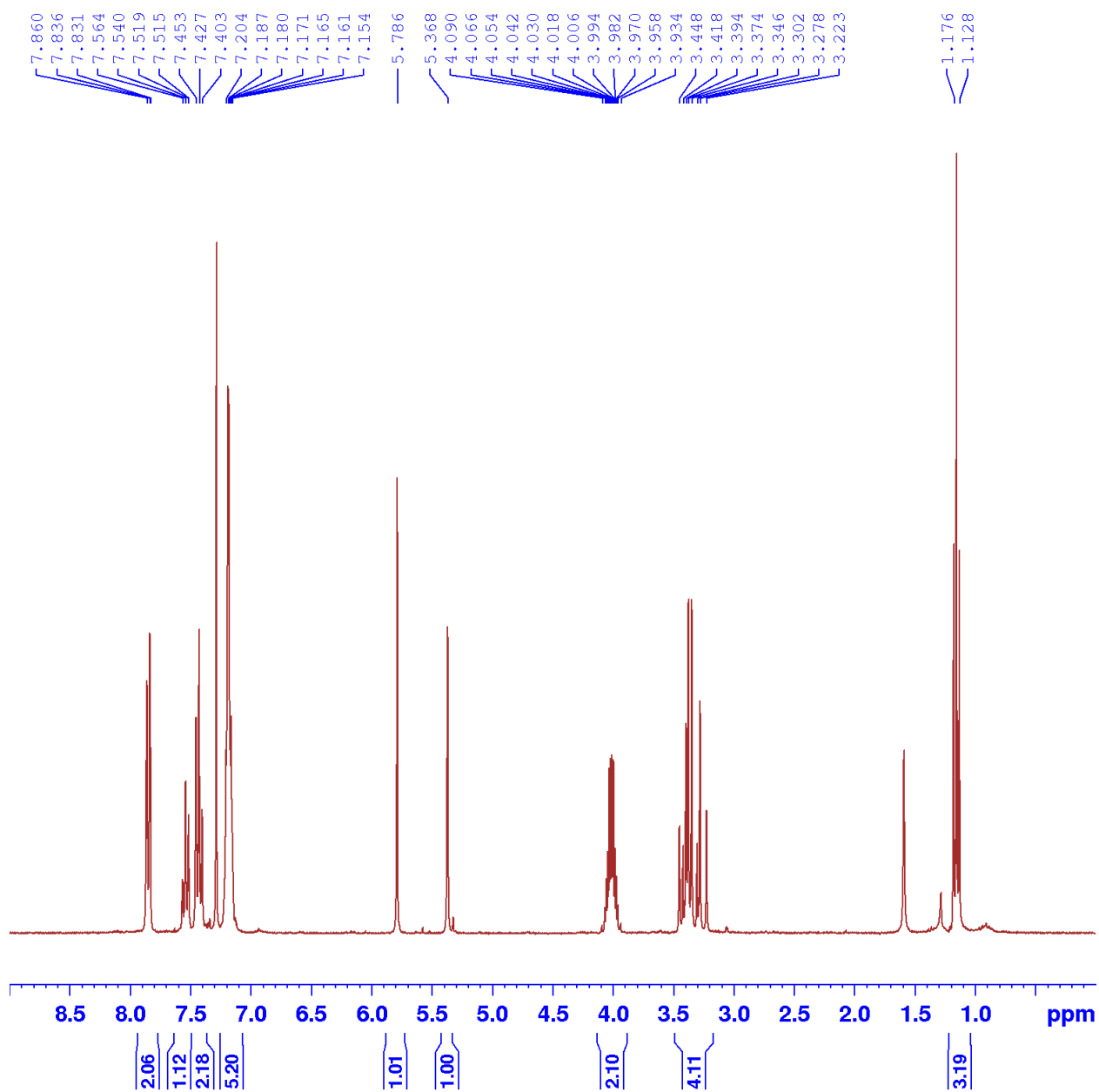
4. NMR Spectra

NMR spectra of compound 5a

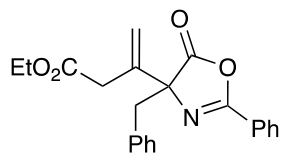
¹H NMR (300 MHz, CDCl₃, 298 K)



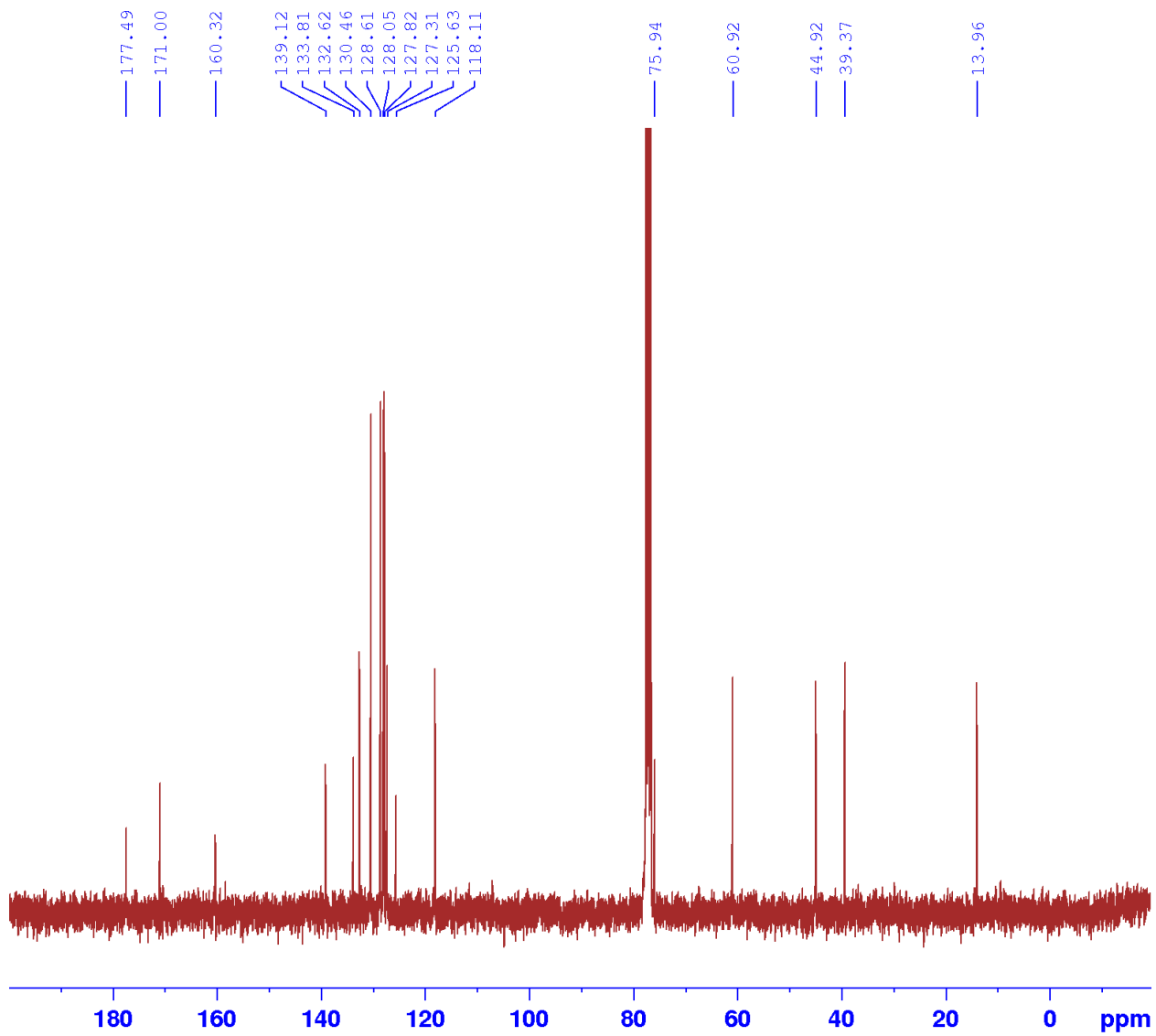
5a



^{13}C NMR (75 MHz, CDCl_3 , 298 K)

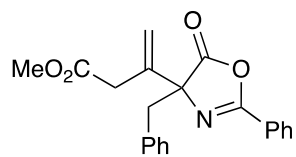


5a

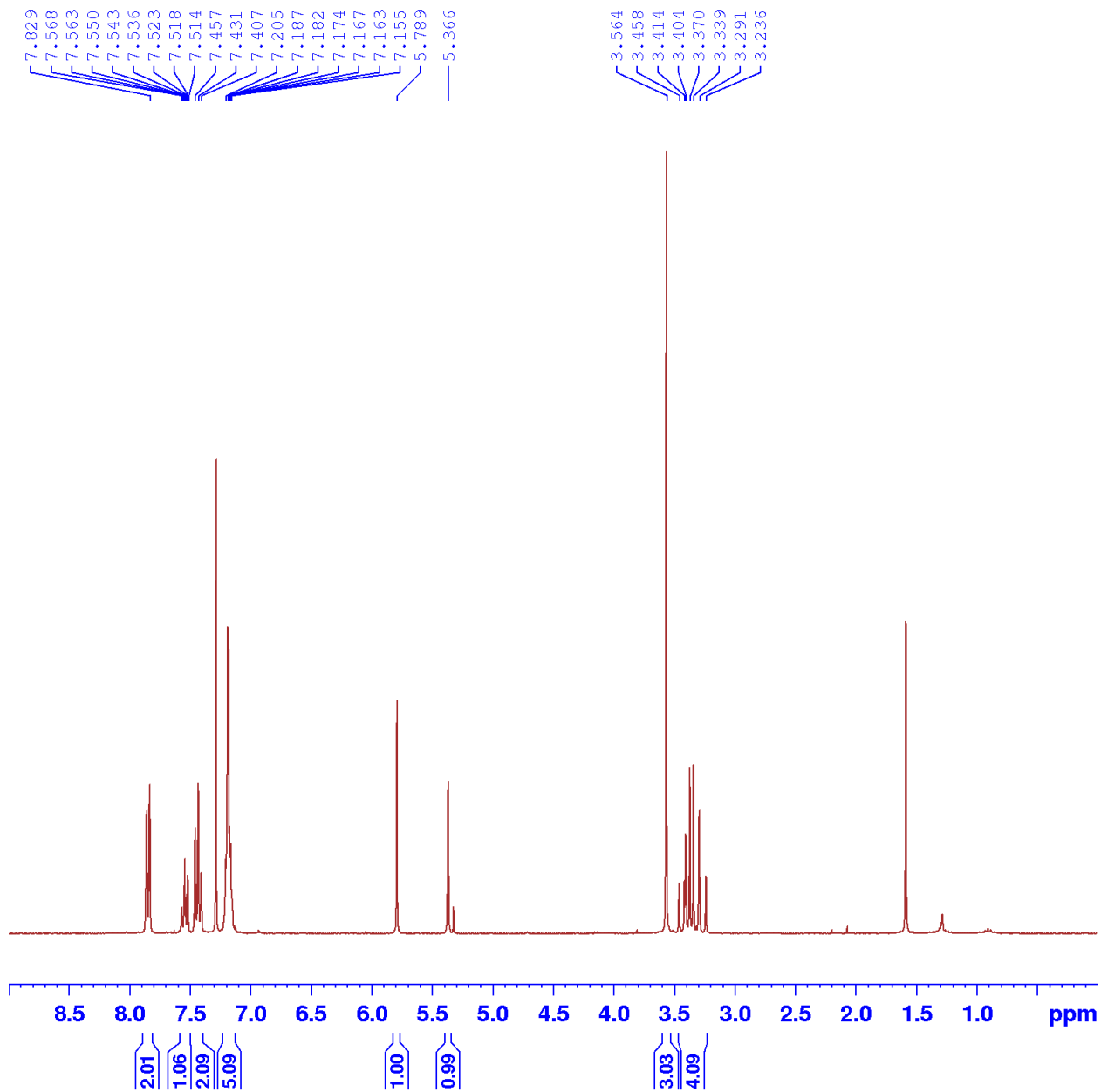


NMR spectra of compound 5b

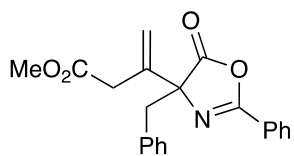
^1H NMR (300 MHz, CDCl_3 , 298 K)



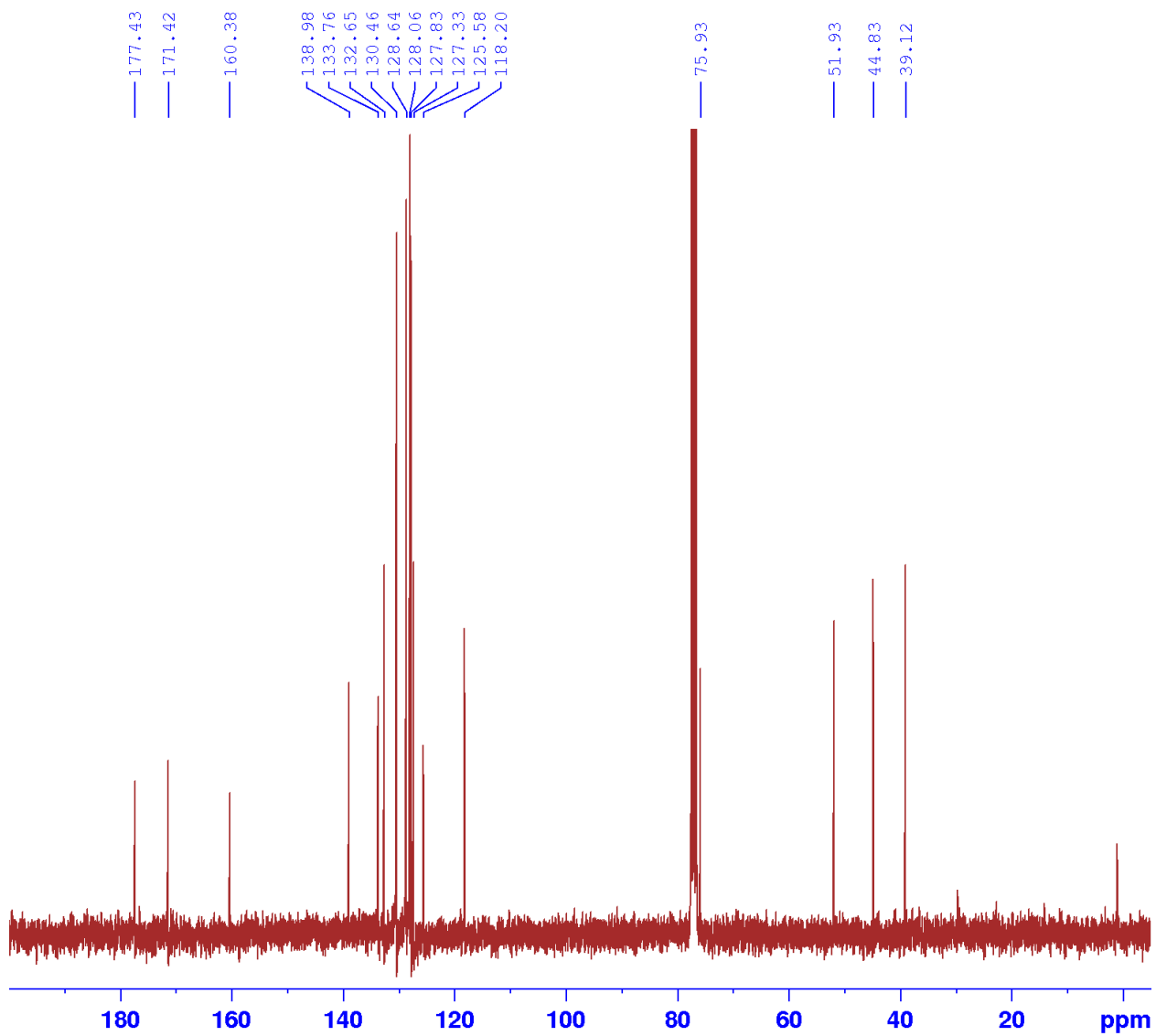
5b



^{13}C NMR (75 MHz, CDCl_3 , 298 K)

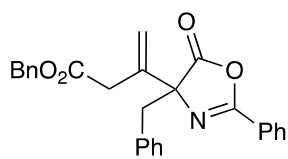


5b

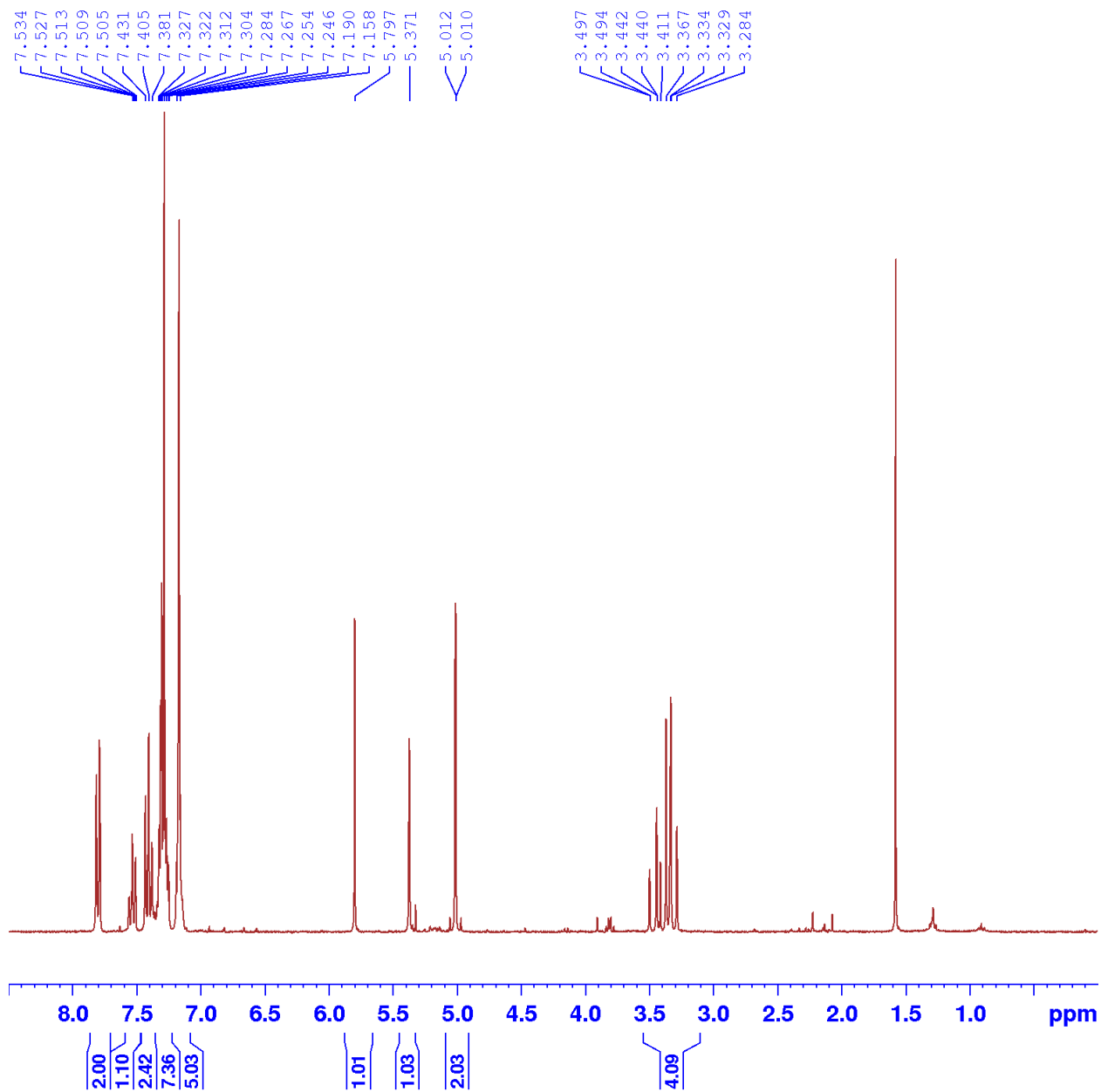


NMR spectra of compound 5c

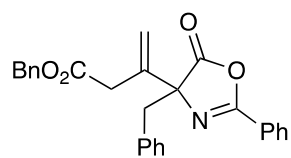
¹H NMR (300 MHz, CDCl₃, 298 K)



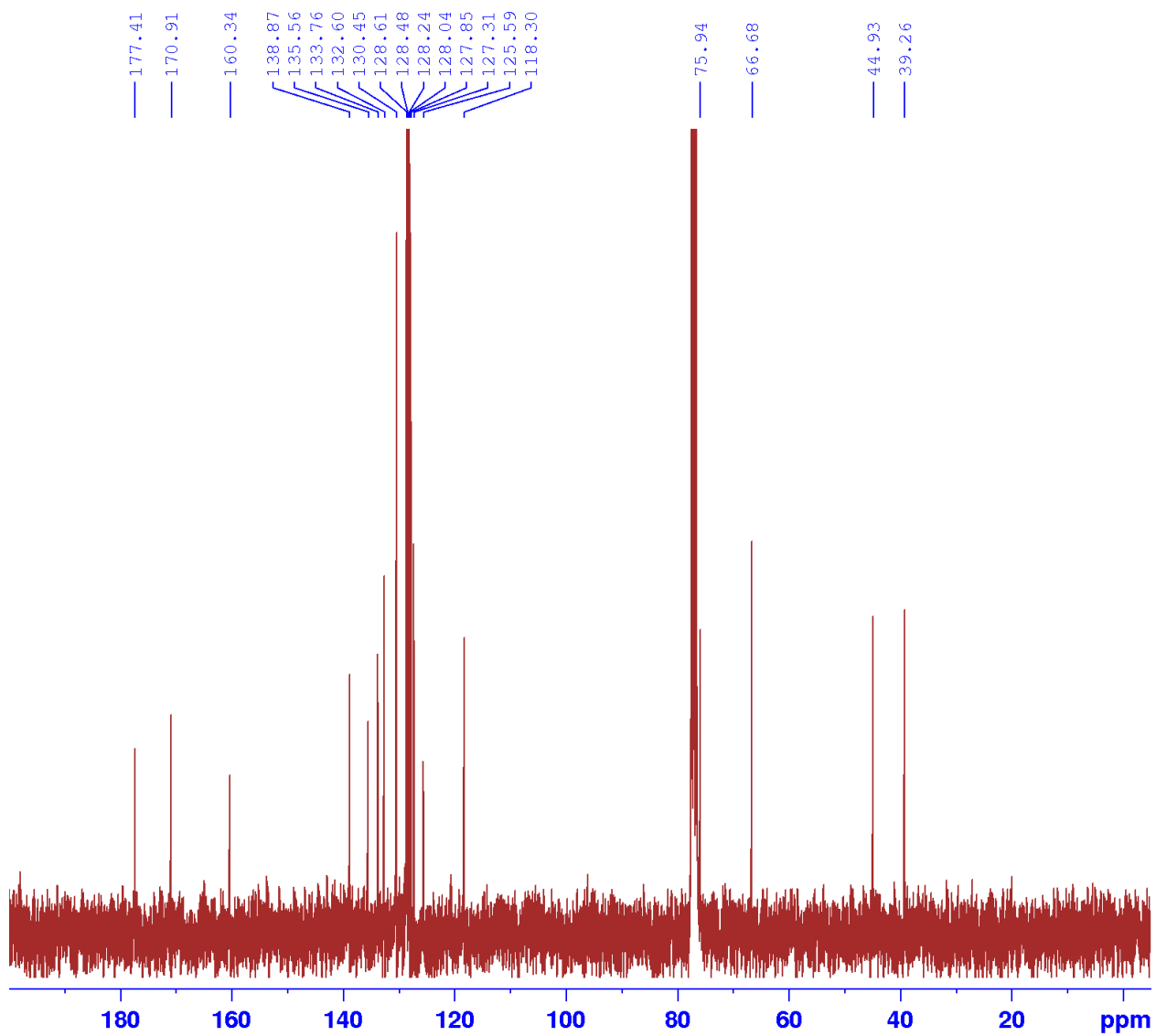
5c



^{13}C NMR (75 MHz, CDCl_3 , 298 K)

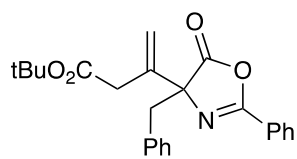


5c

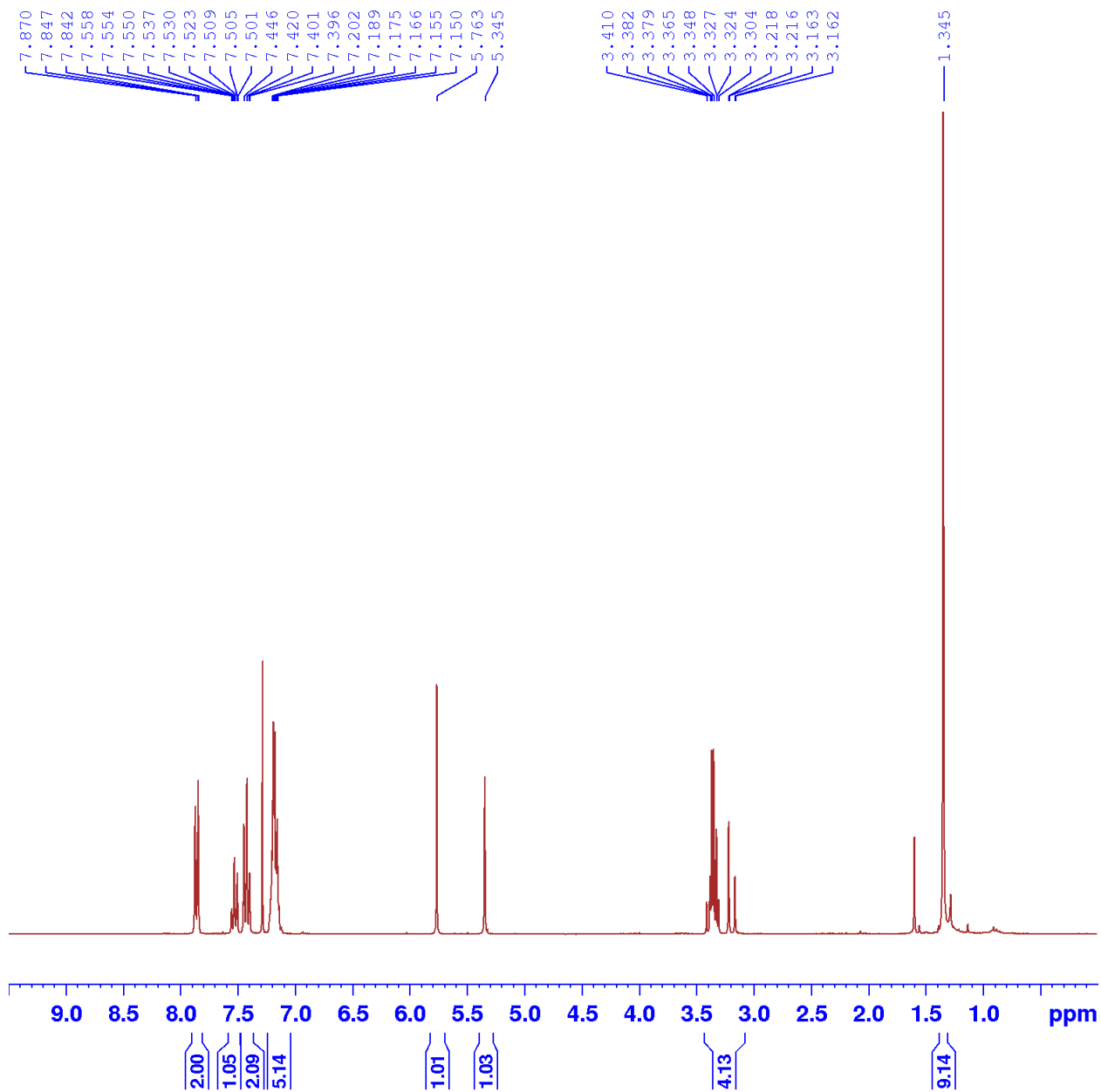


NMR spectra of compound 5d

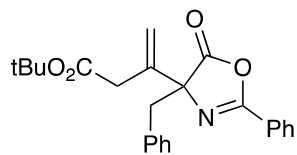
^1H NMR (300 MHz, CDCl_3 , 298 K)



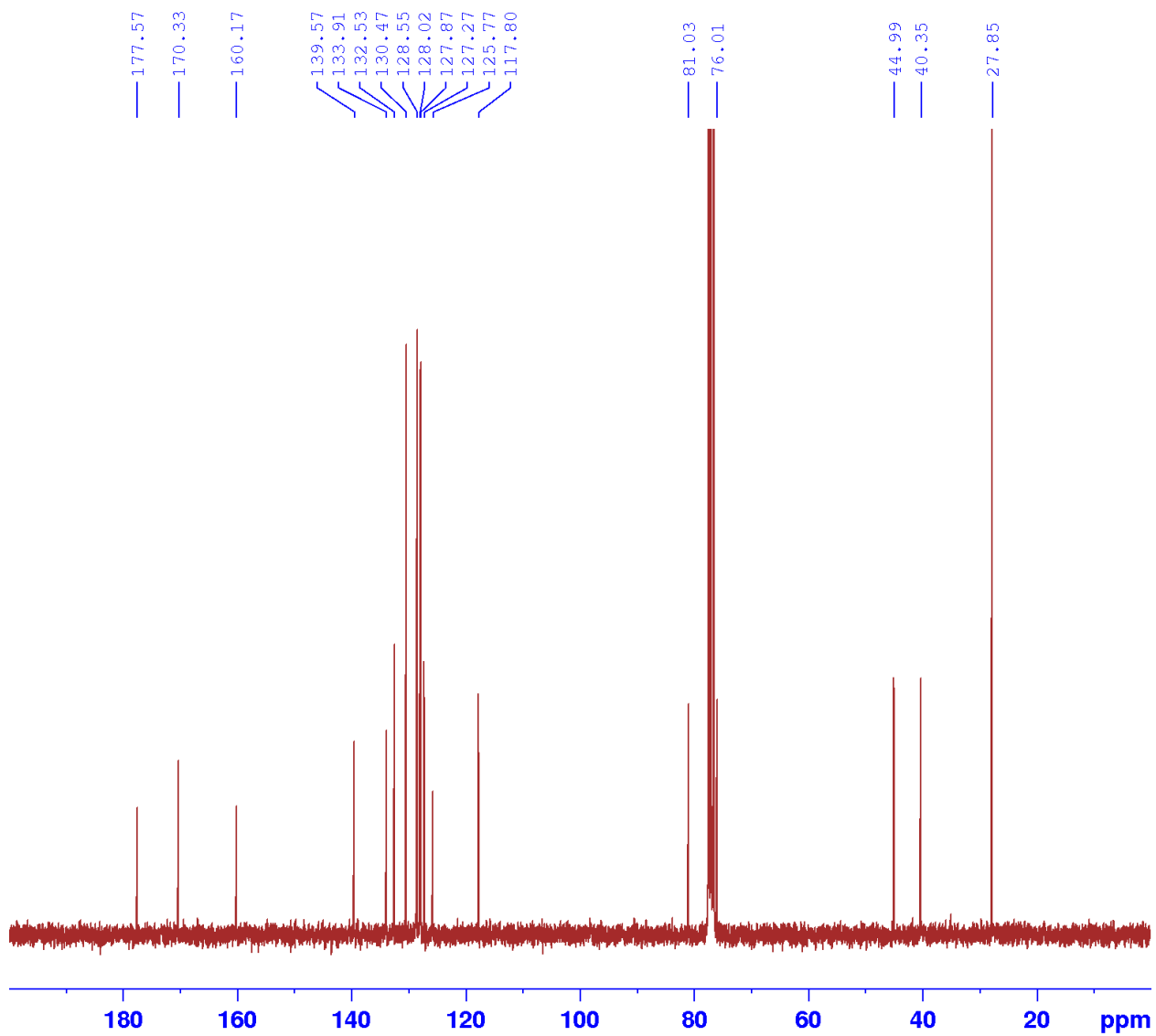
5d



^{13}C NMR (75 MHz, CDCl_3 , 298 K)

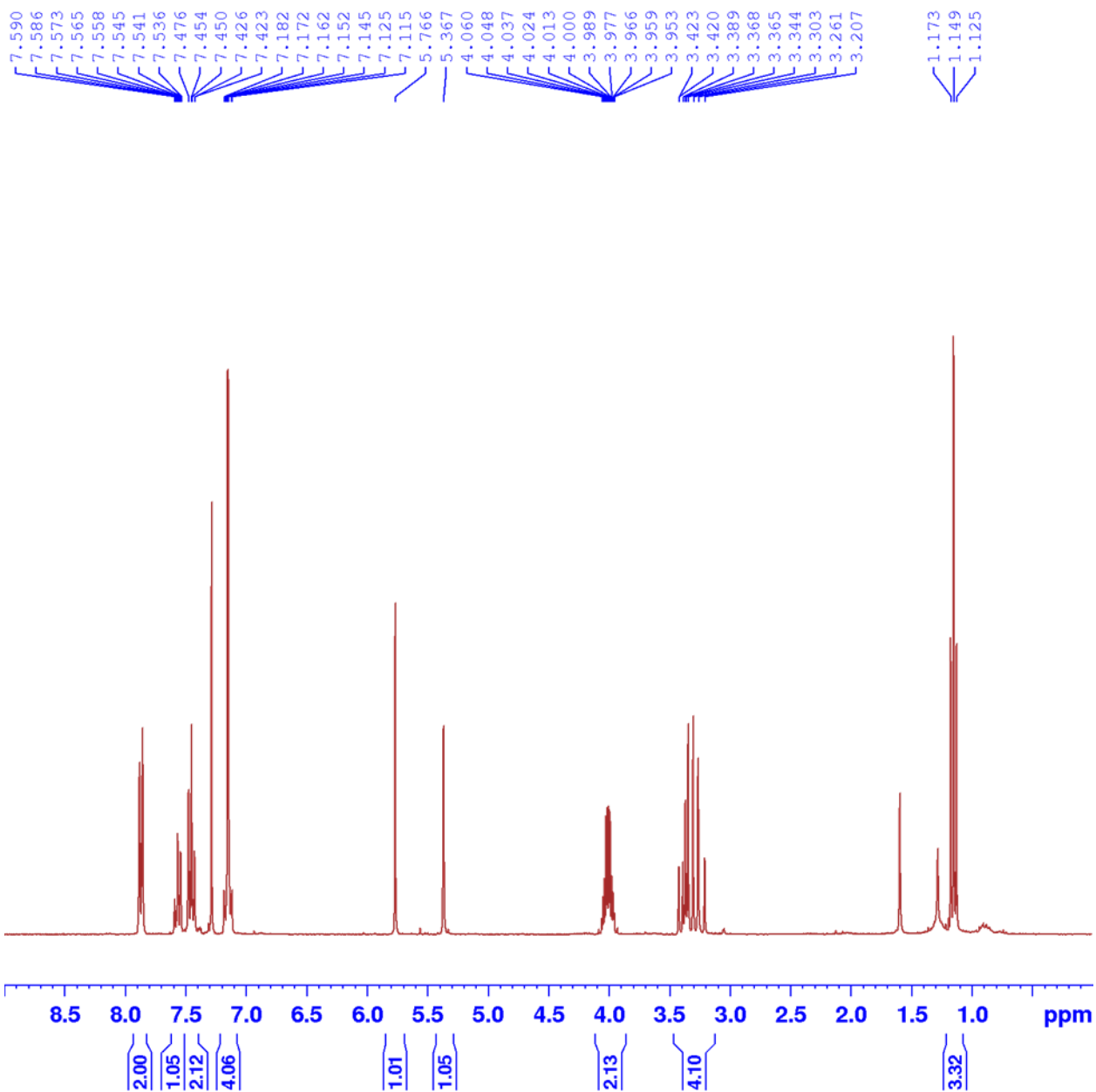
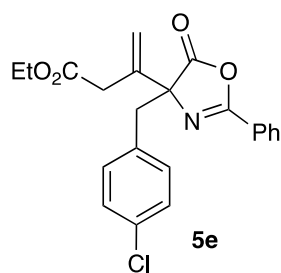


5d

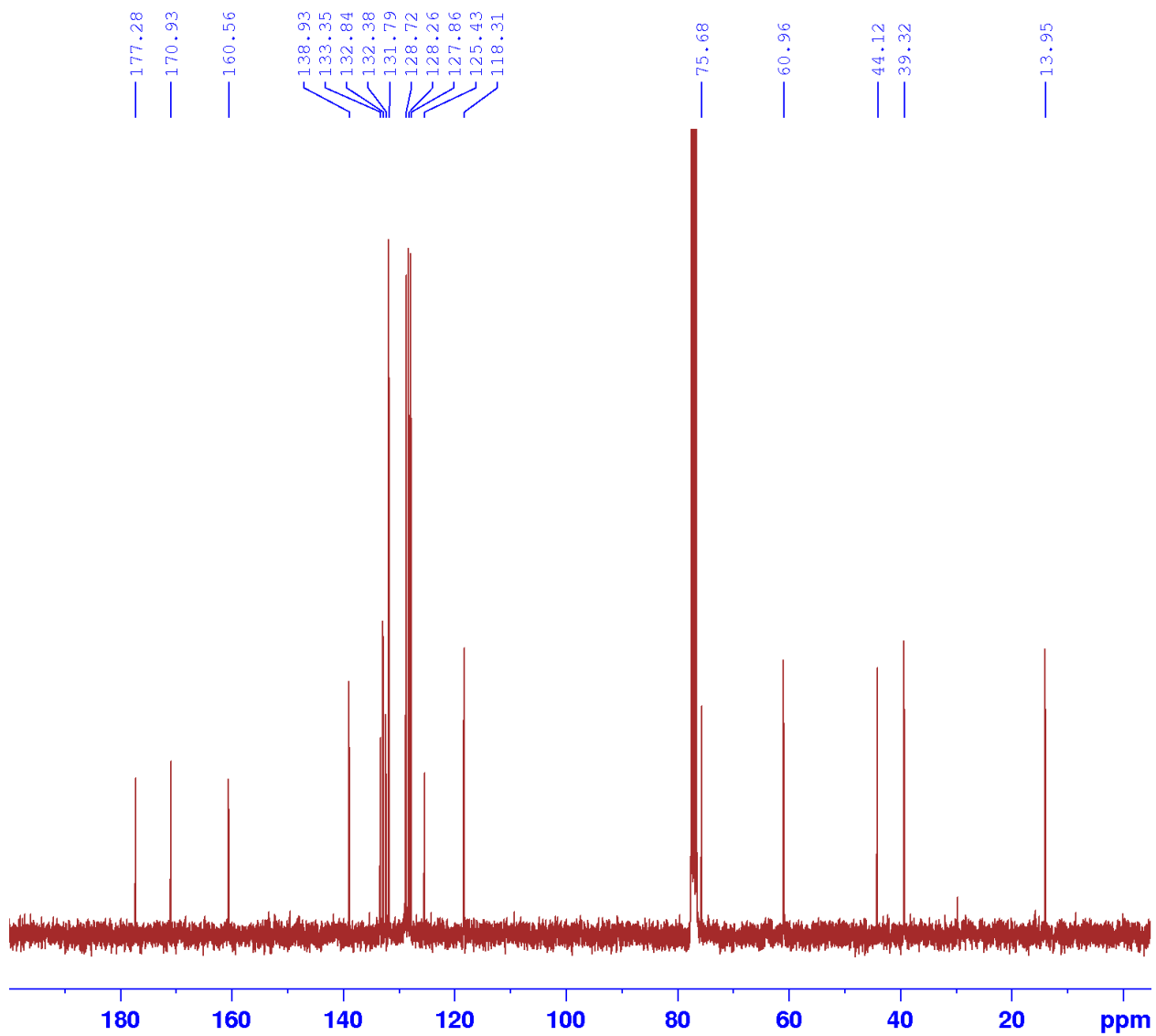
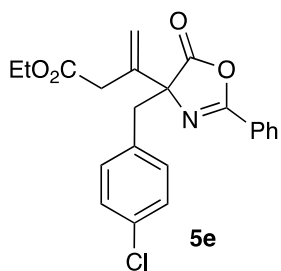


NMR spectra of compound 5e

¹H NMR (300 MHz, CDCl₃, 298 K)

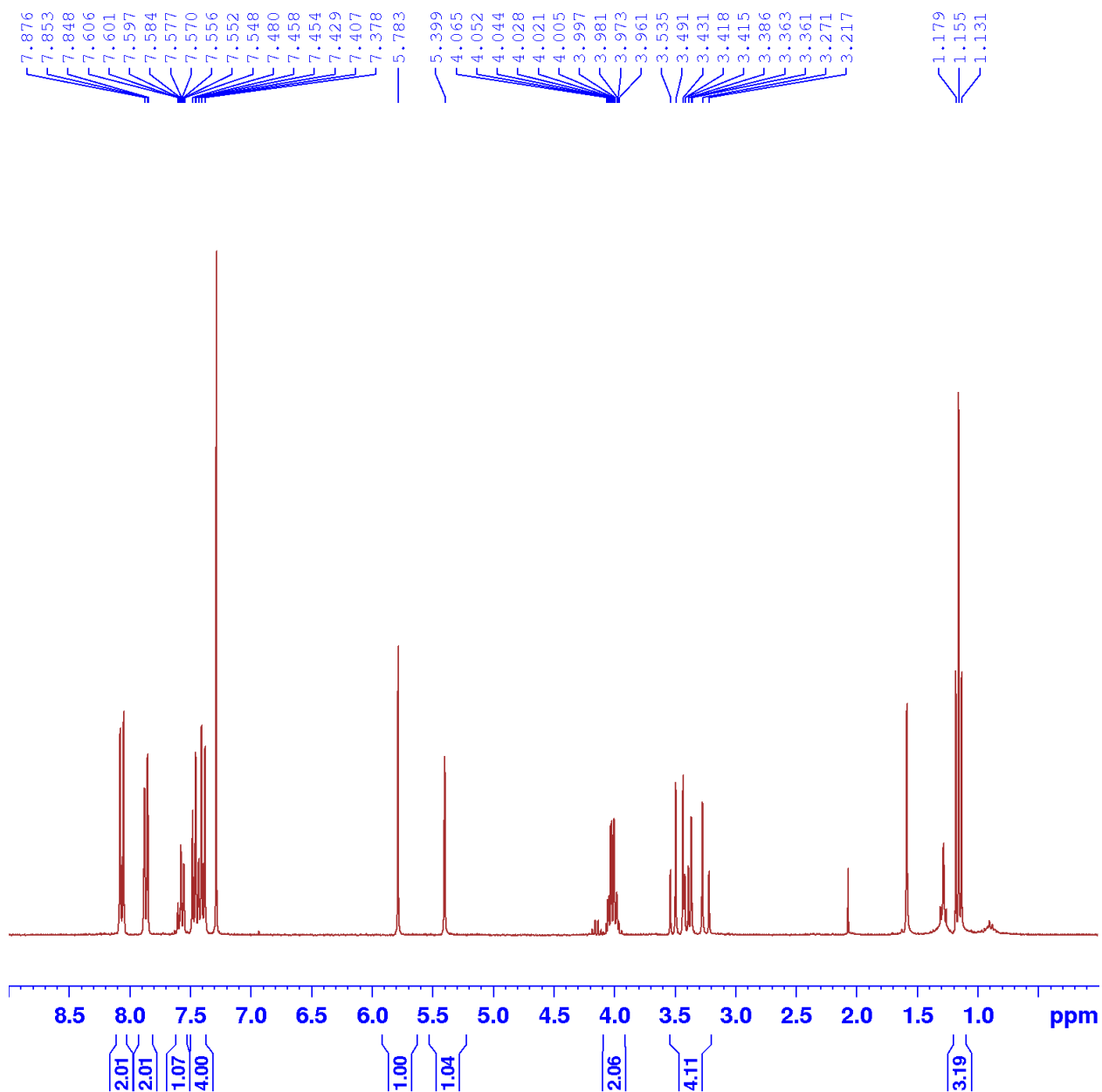
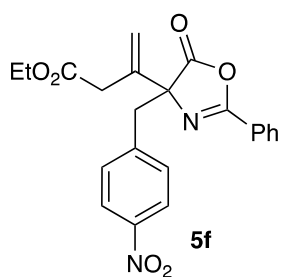


^{13}C NMR (75 MHz, CDCl_3 , 298 K)

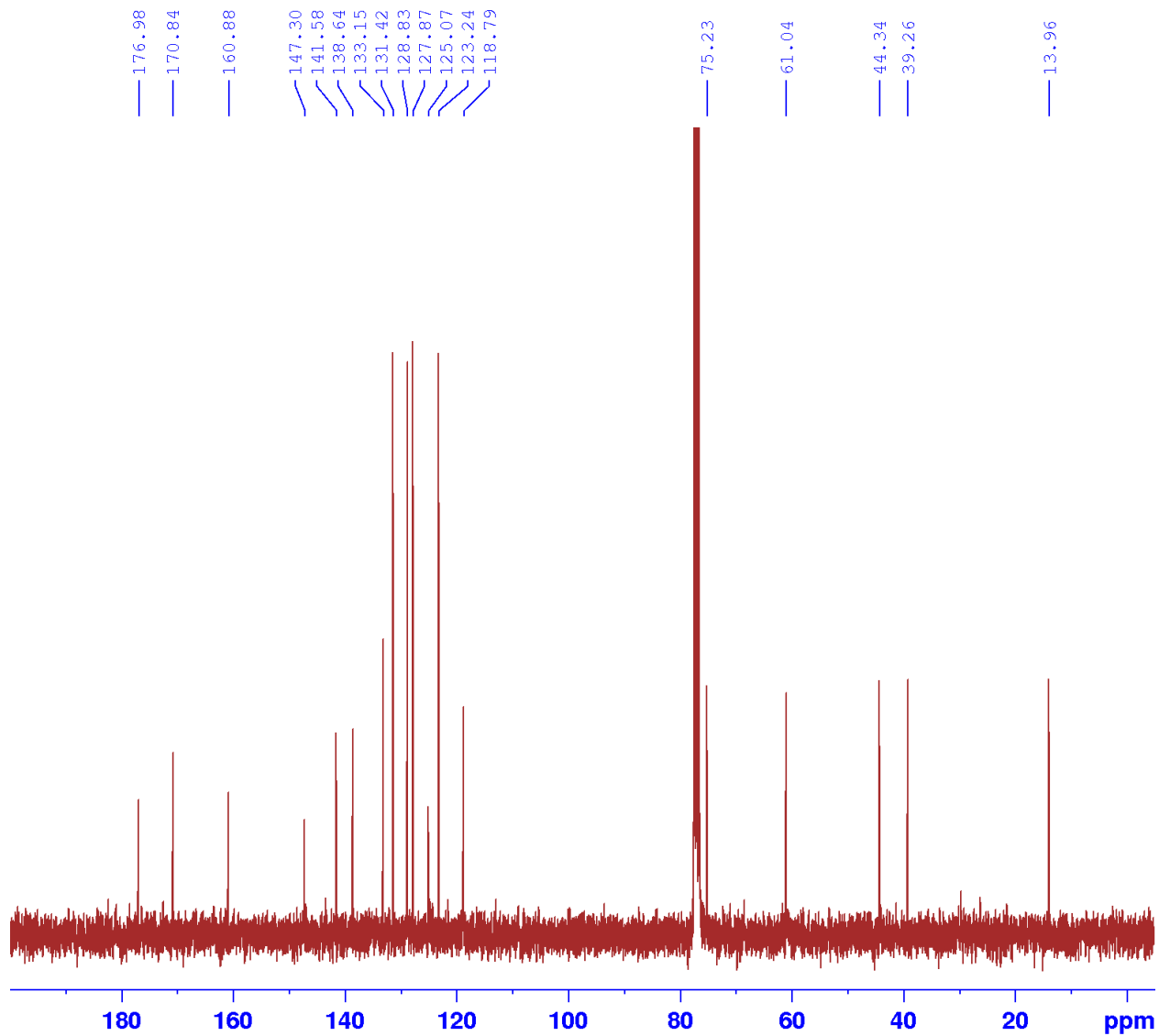
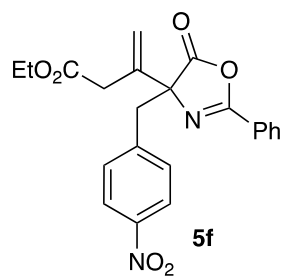


NMR spectra of compound 5f

¹H NMR (300 MHz, CDCl₃, 298 K)

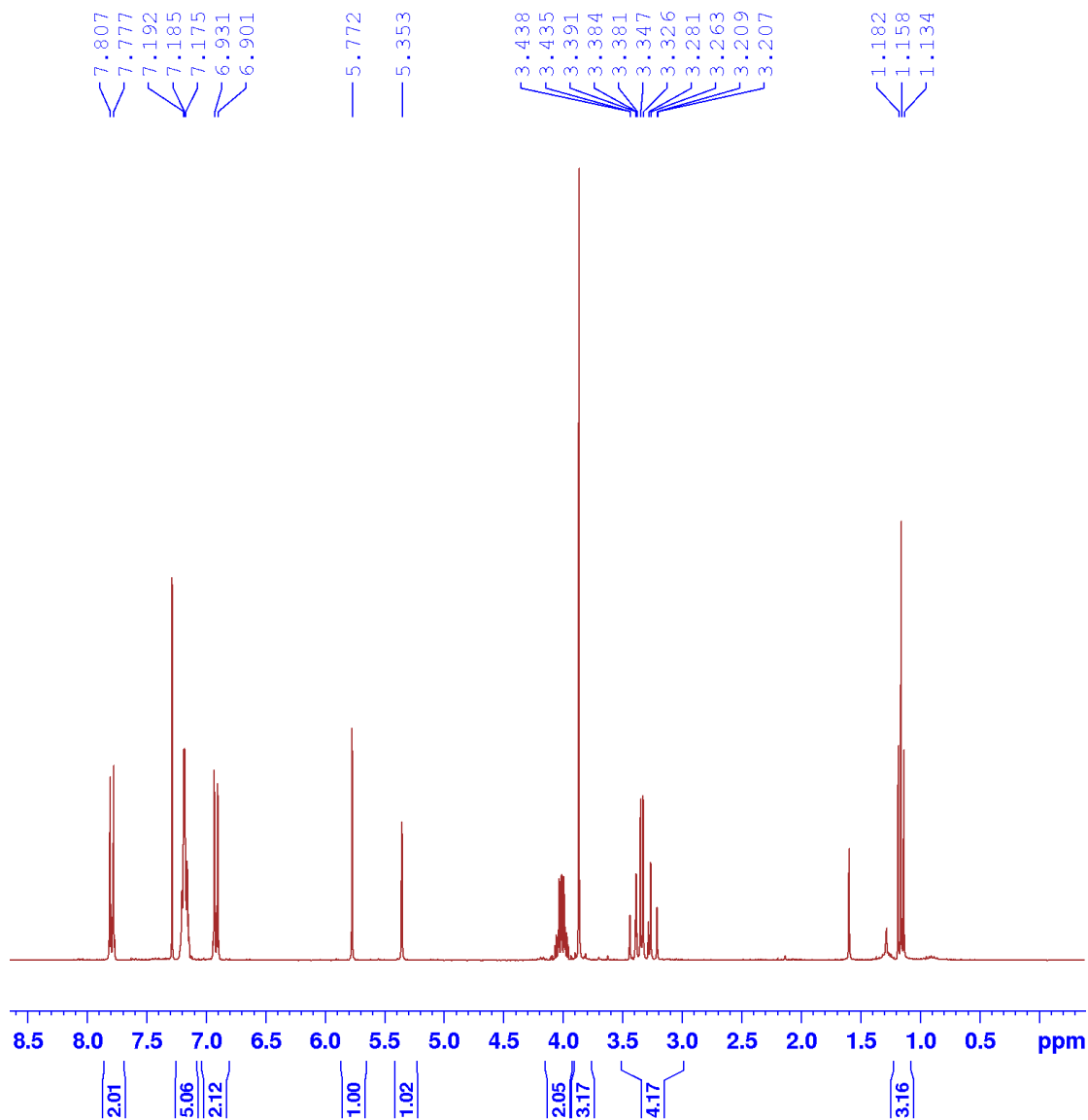
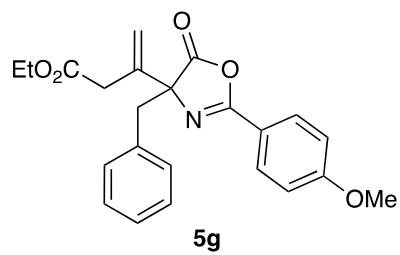


¹³C NMR (75 MHz, CDCl₃, 298 K)

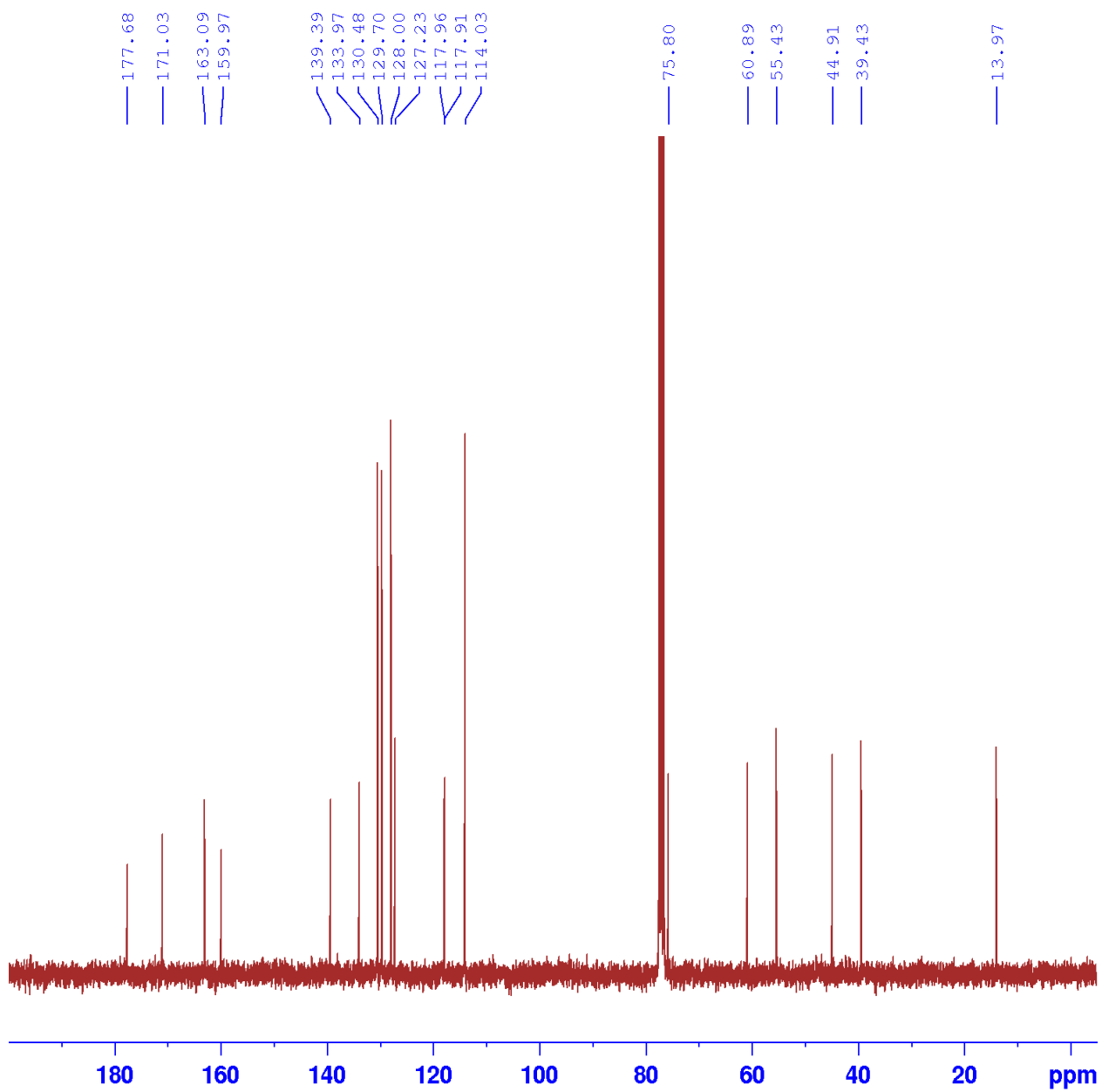
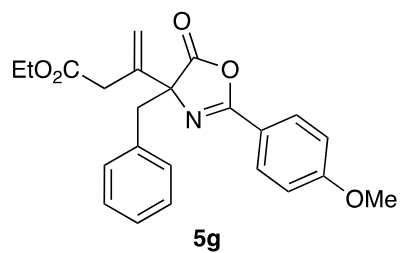


NMR spectra of compound 5g

¹H NMR (300 MHz, CDCl₃, 298 K)

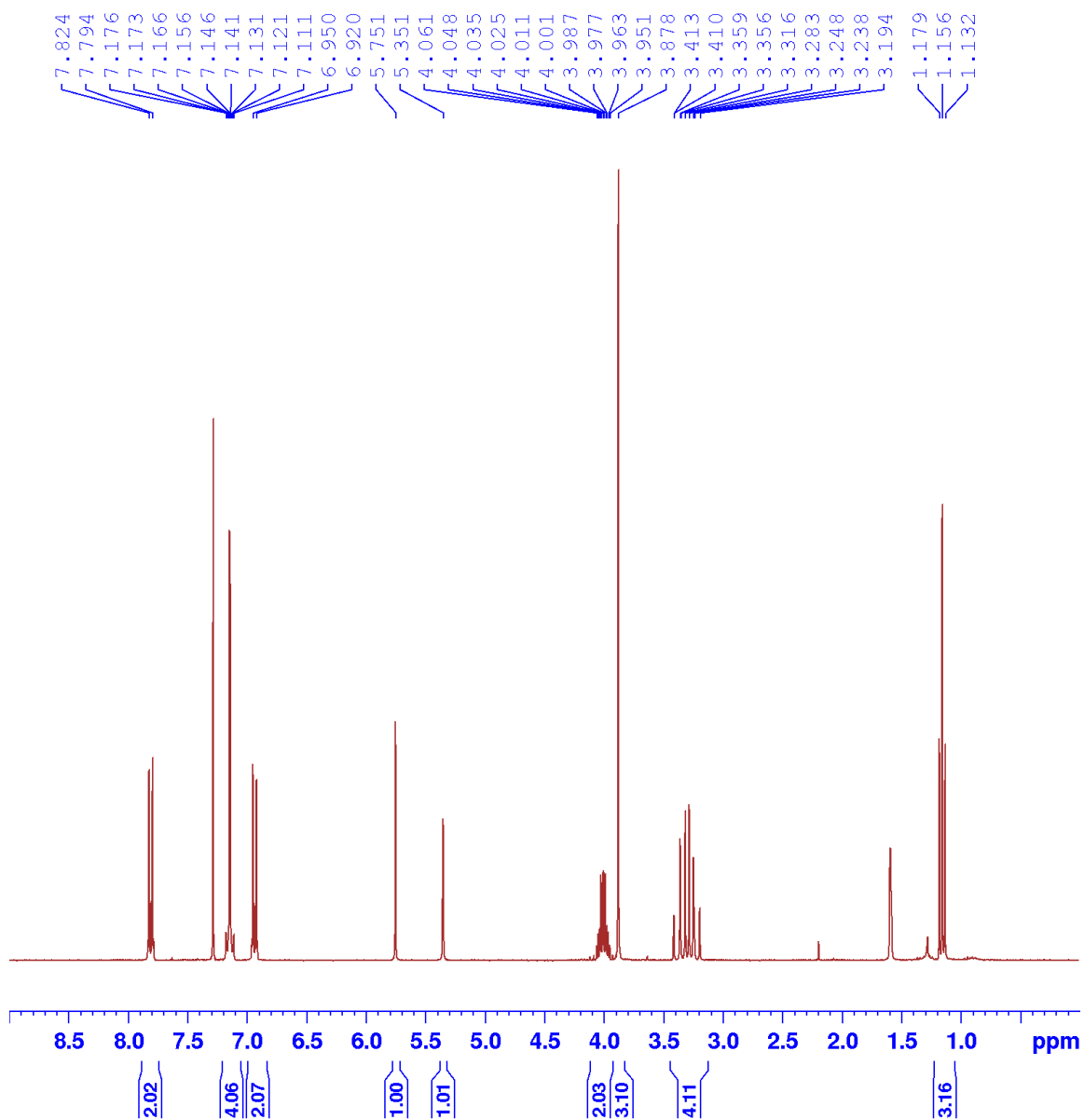
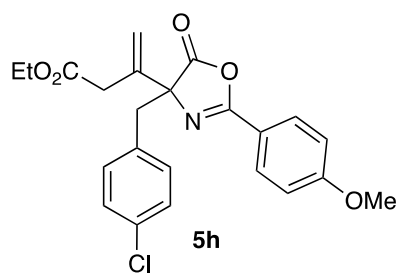


^{13}C NMR (75 MHz, CDCl_3 , 298 K)

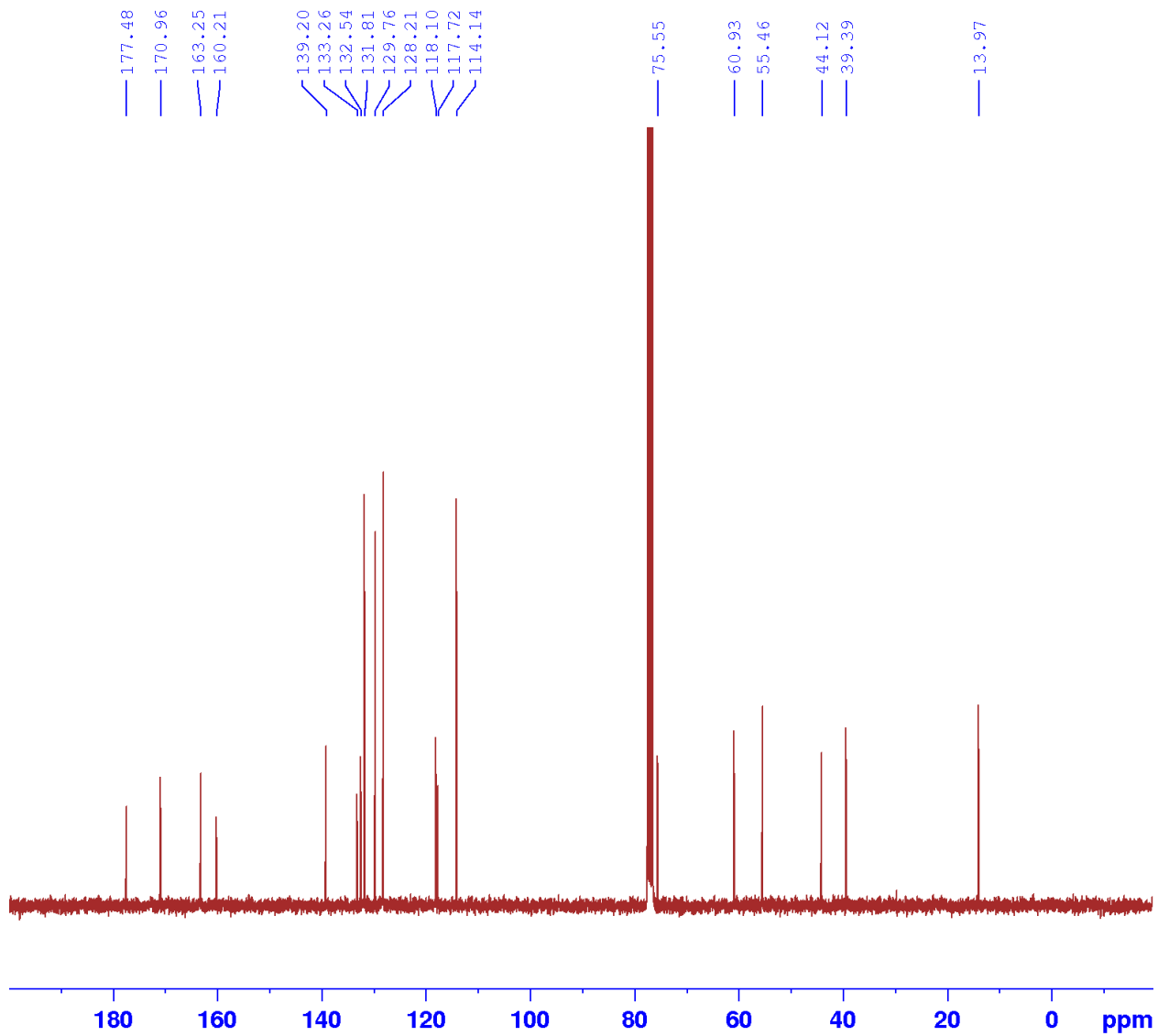
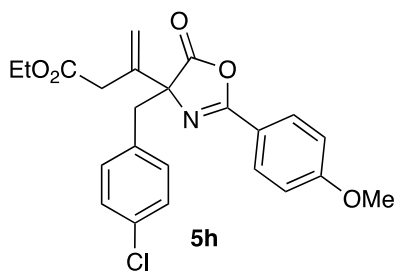


NMR spectra of compound 5h

¹H NMR (300 MHz, CDCl₃, 298 K)

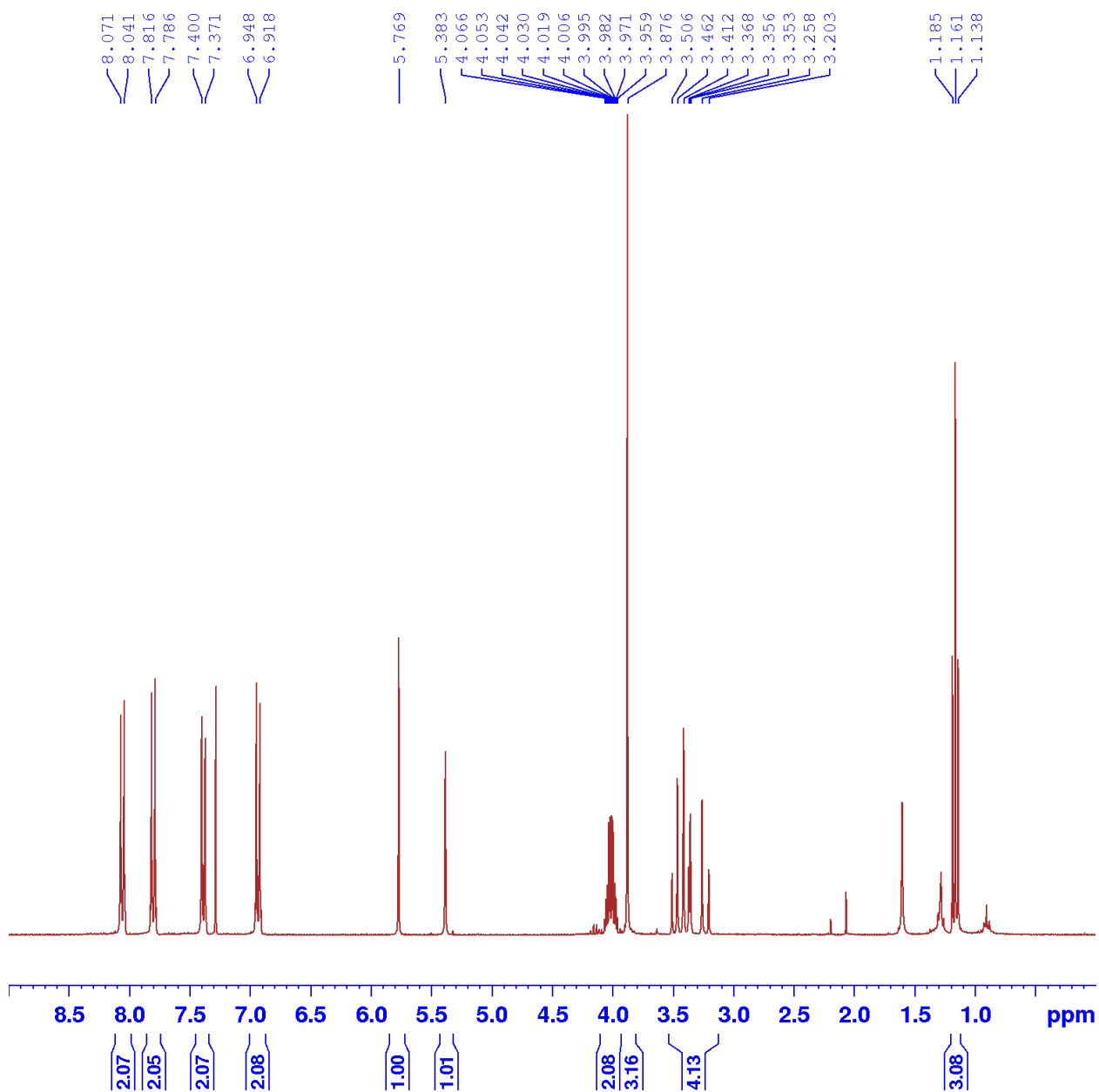
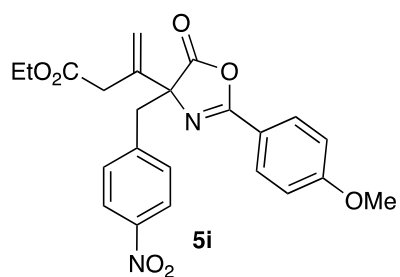


¹³C NMR (75 MHz, CDCl₃, 298 K)

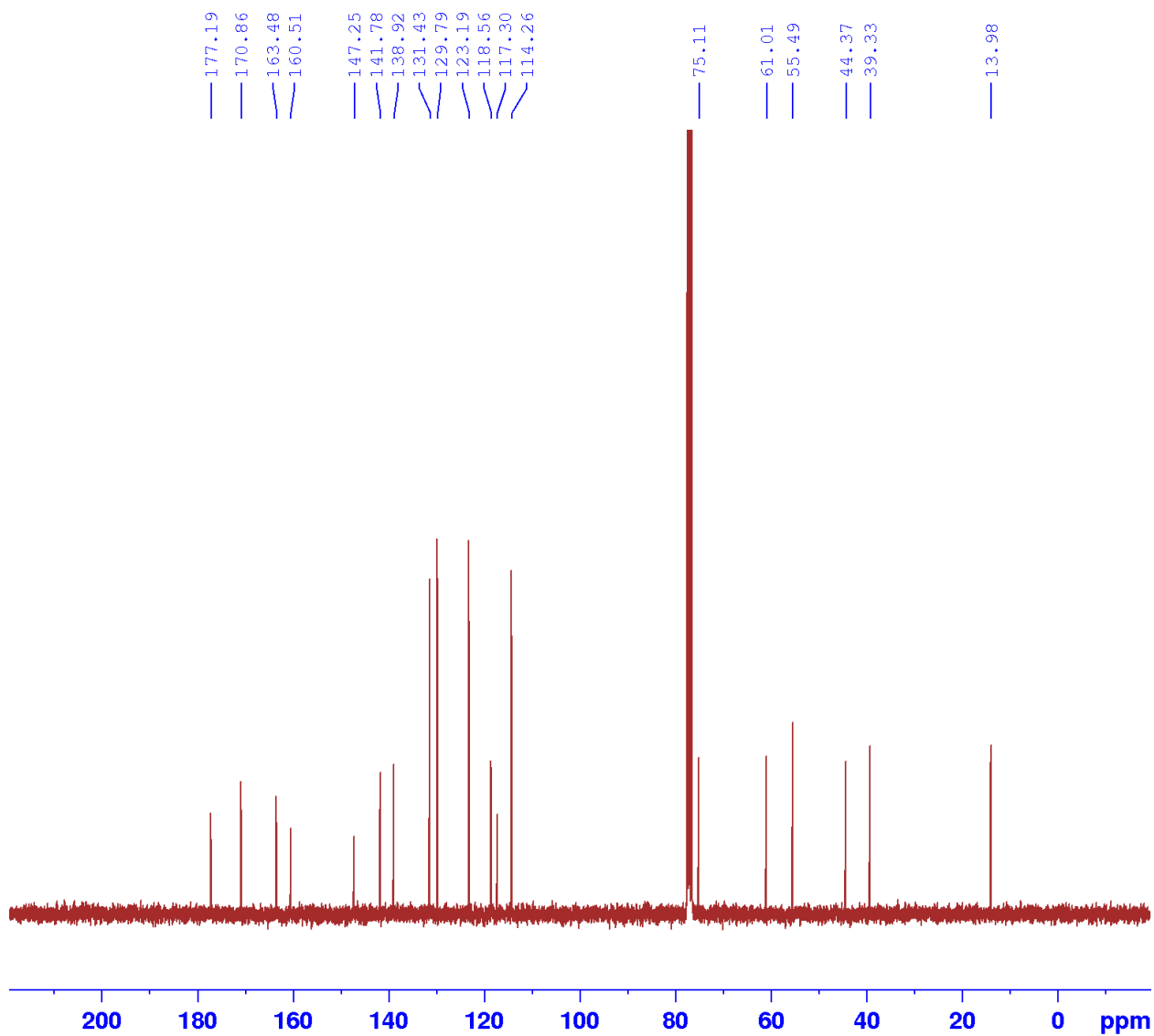
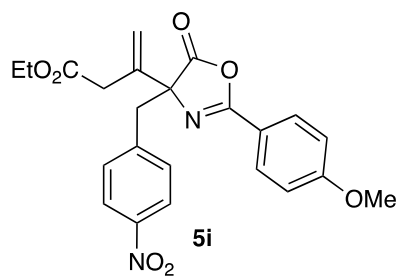


NMR spectra of compound 5i

¹H NMR (300 MHz, CDCl₃, 298 K)

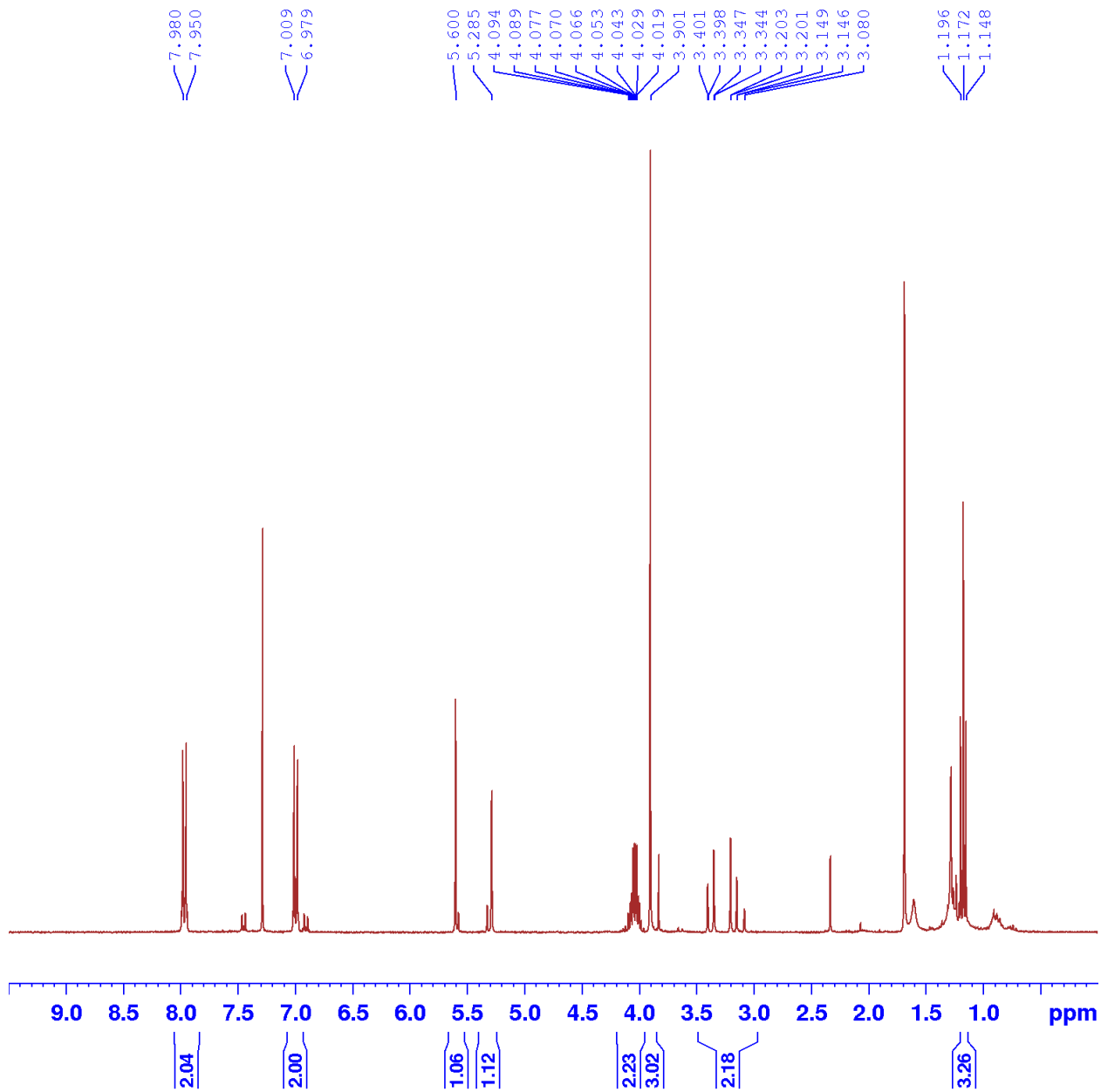
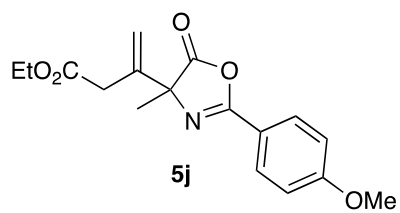


¹³C NMR (75 MHz, CDCl₃, 298 K)

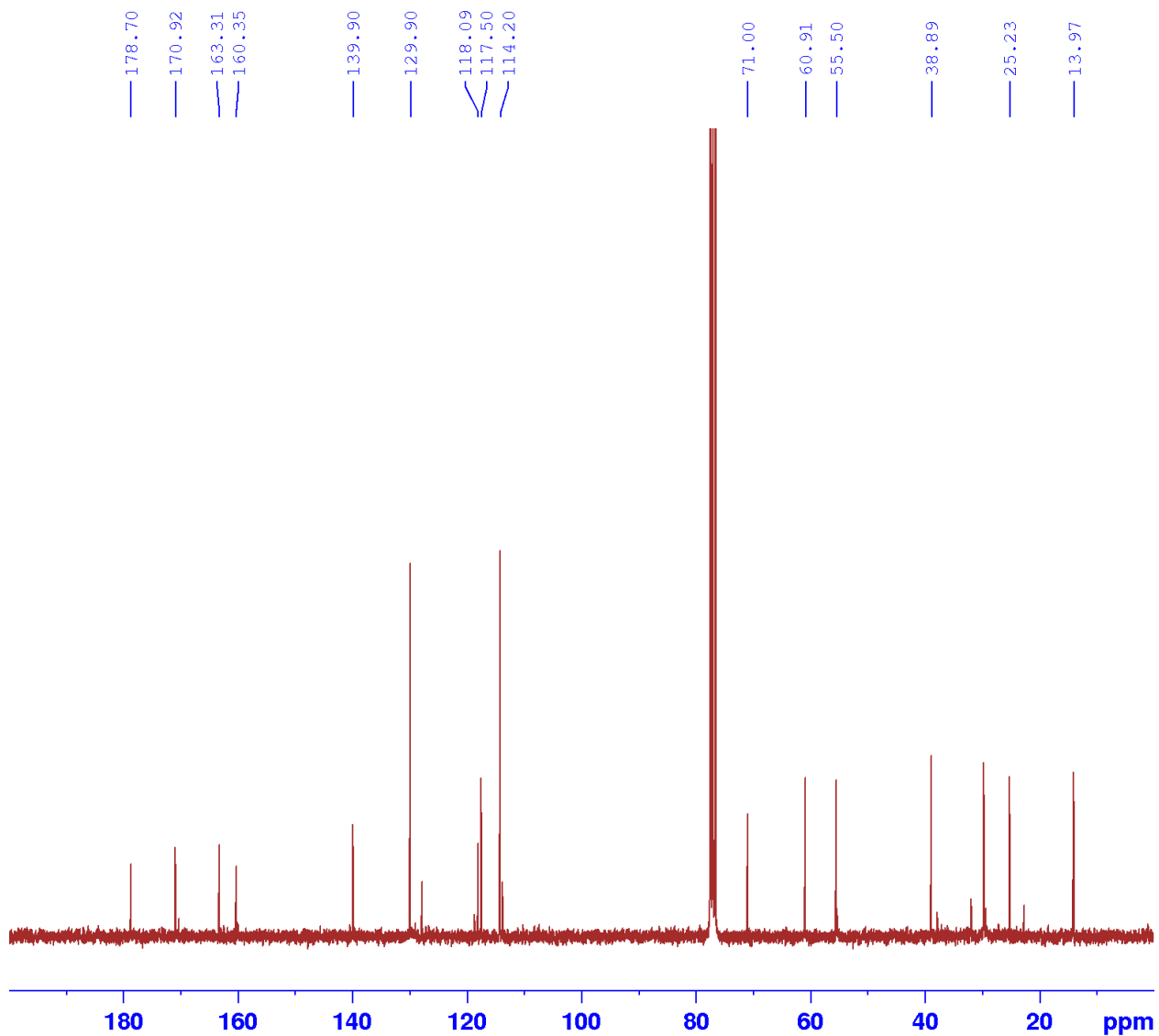
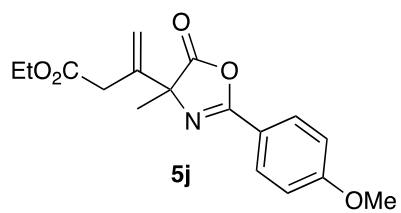


NMR spectra of compound 5j (containing traces of ring-opened product)

¹H NMR (300 MHz, CDCl₃, 298 K)

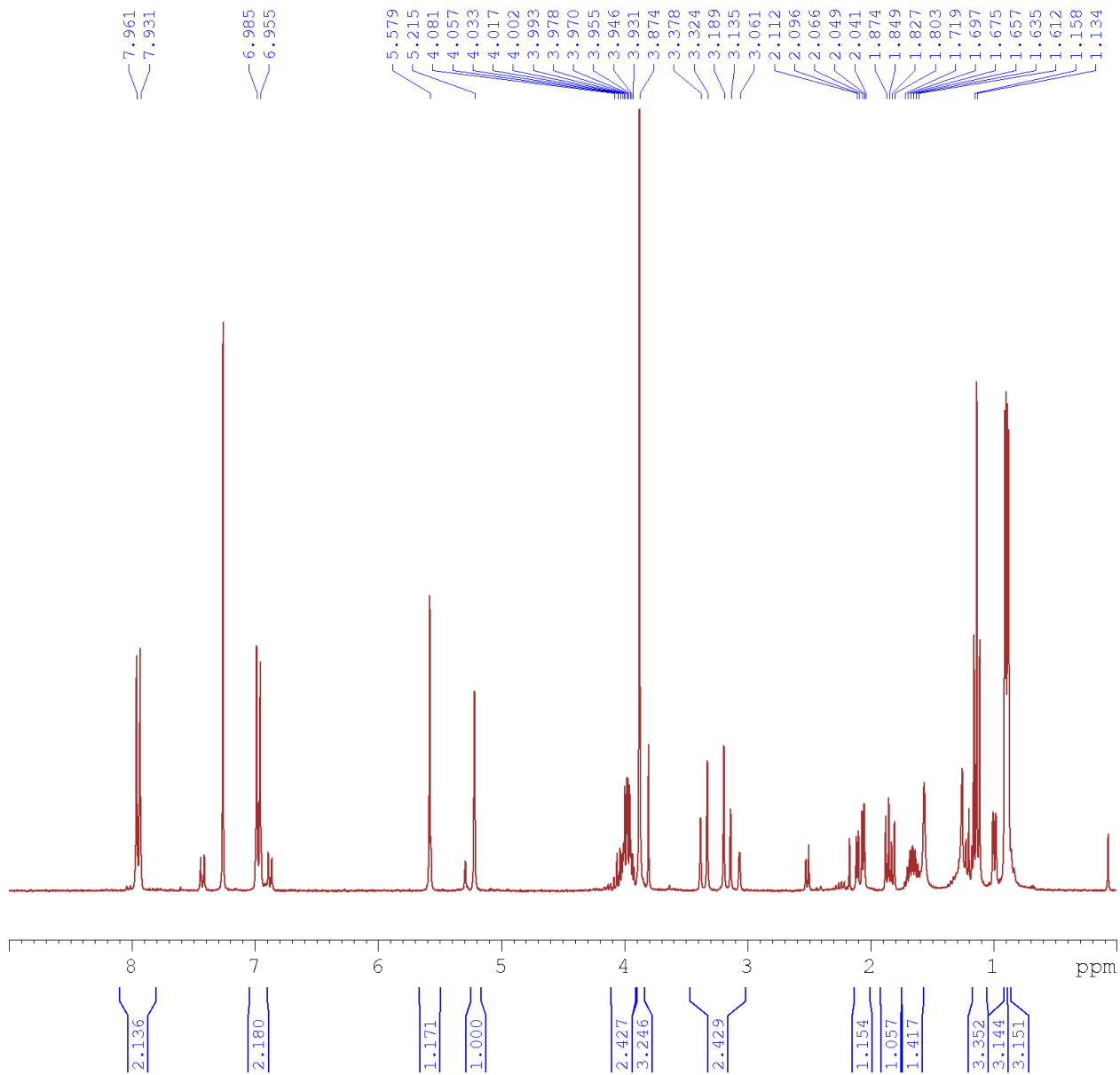
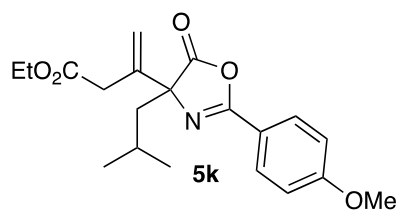


¹³C NMR (75 MHz, CDCl₃, 298 K)

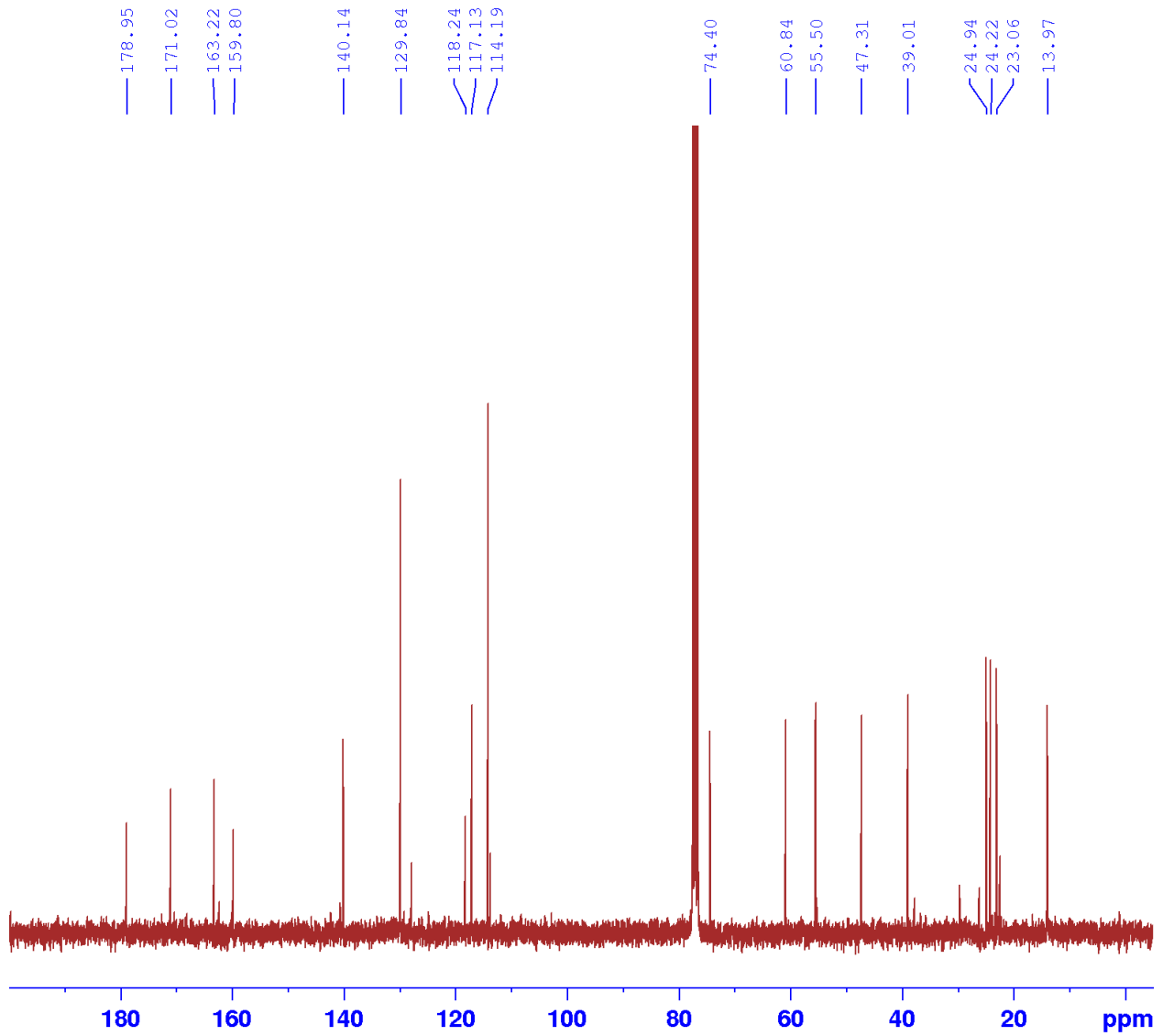
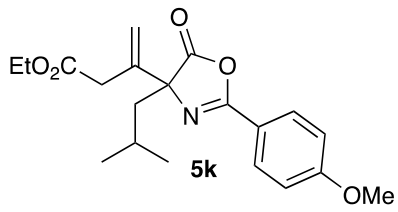


NMR spectra of compound 5k (containing traces of ring-opened product)

¹H NMR (300 MHz, CDCl₃, 298 K)

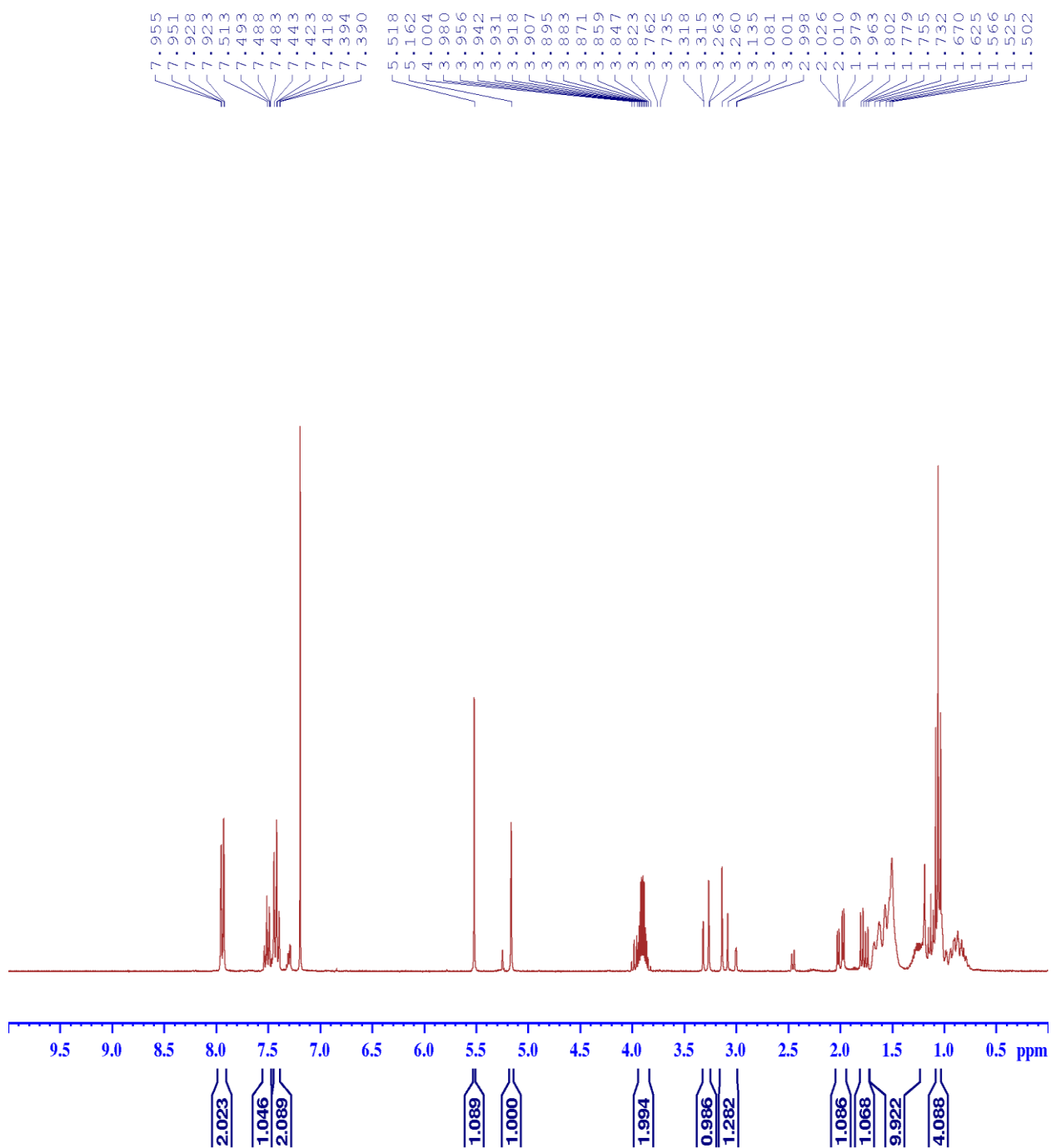
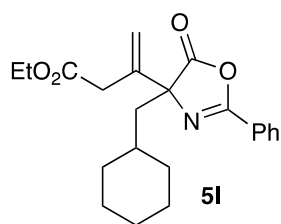


¹³C NMR (75 MHz, CDCl₃, 298 K)

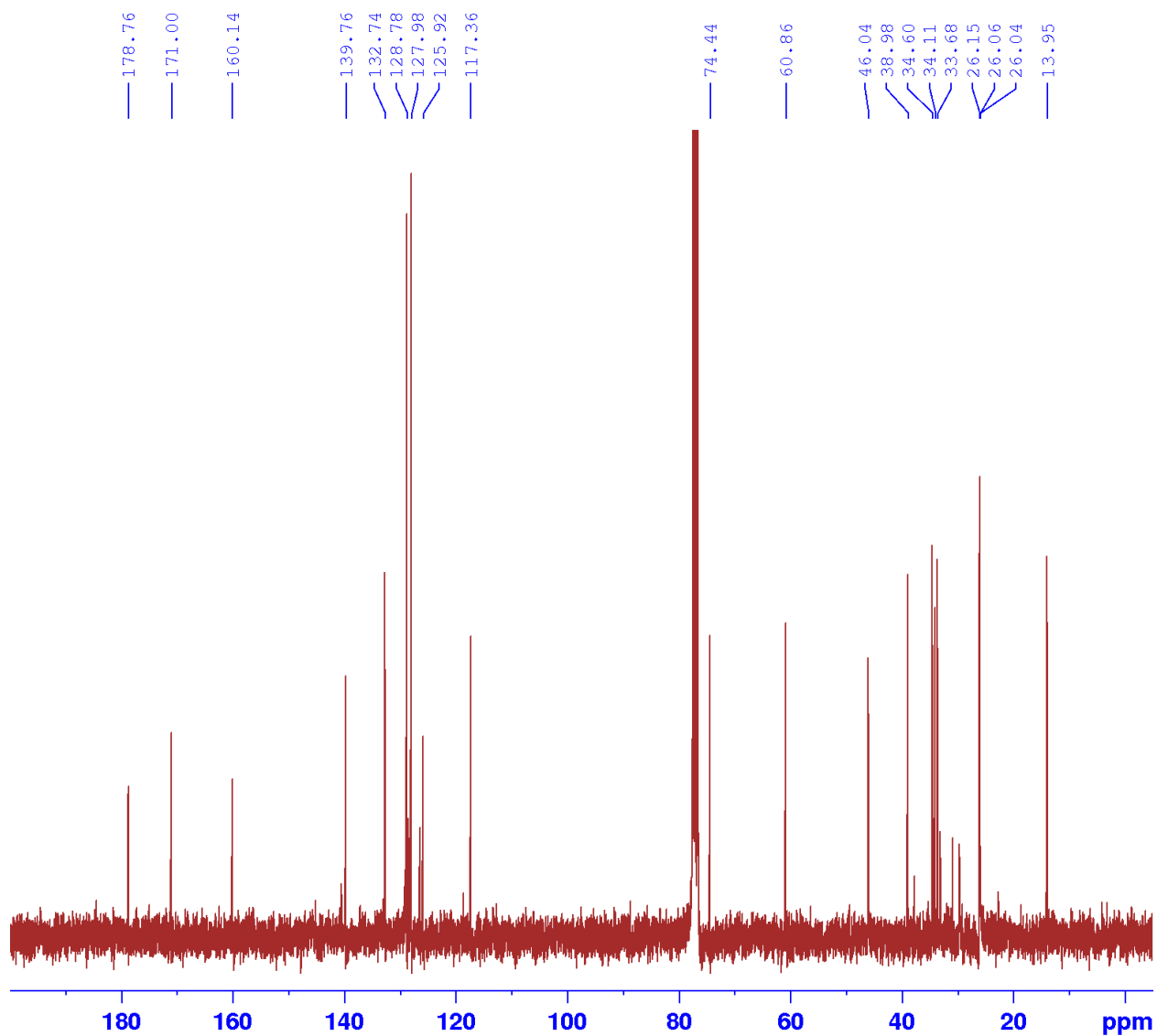
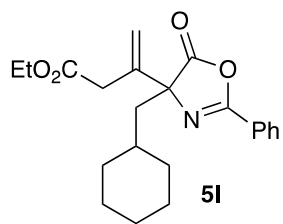


NMR spectra of compound 5I

¹H NMR (300 MHz, CDCl₃, 298 K)

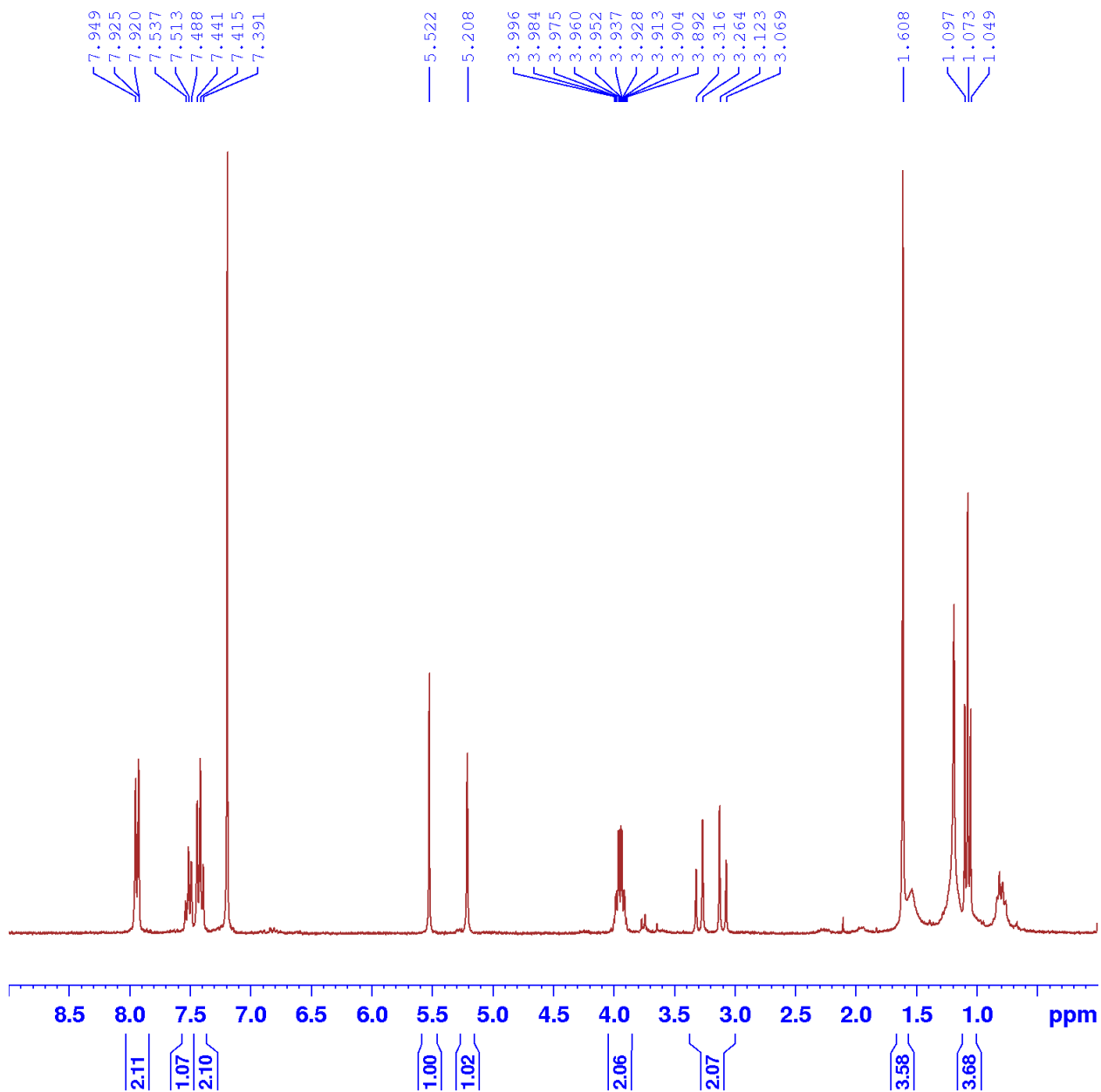
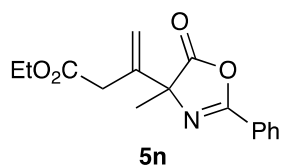


¹³C NMR (75 MHz, CDCl₃, 298 K)

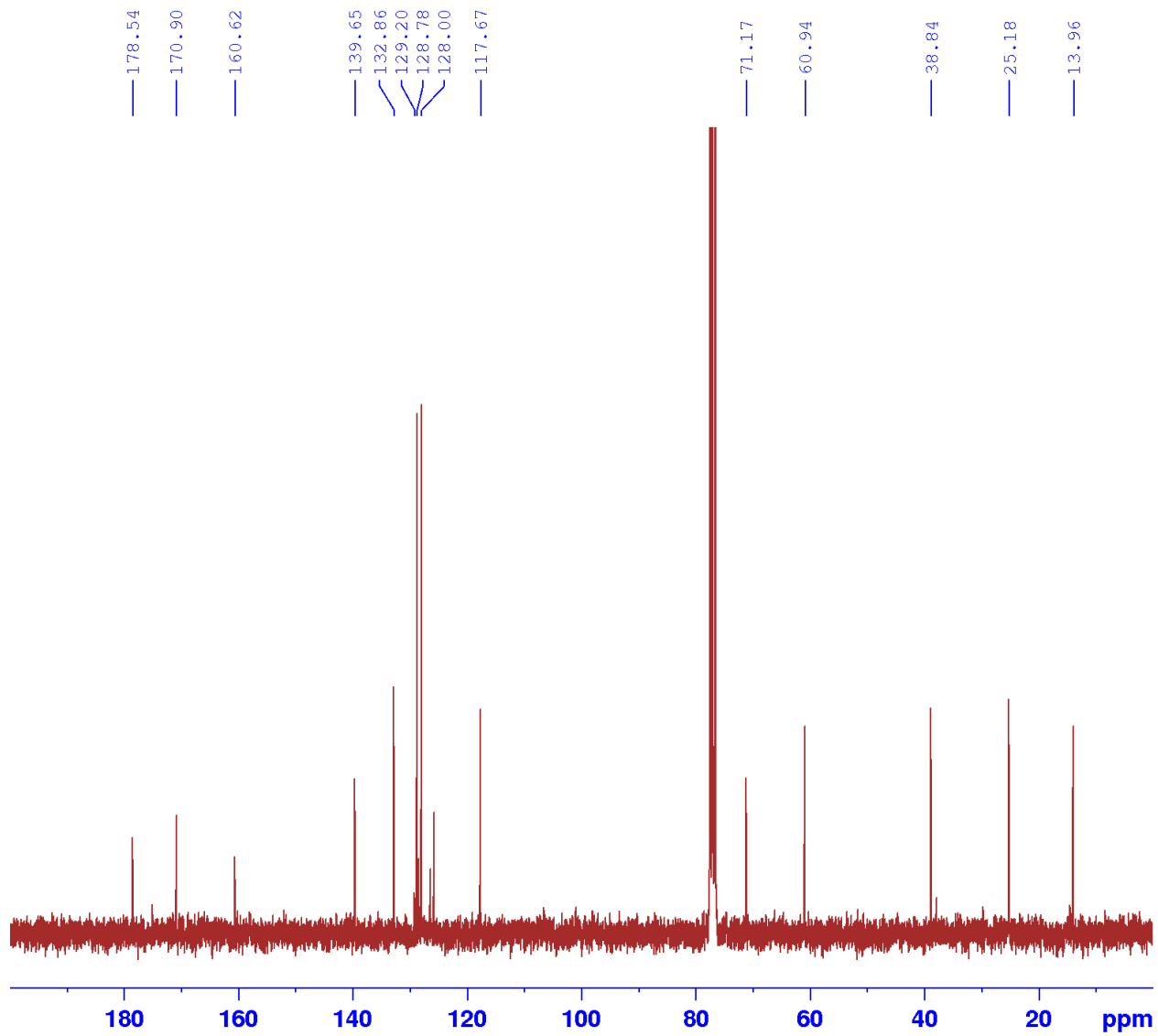
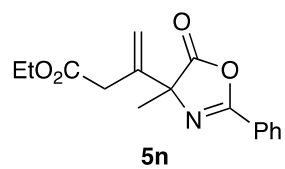


NMR spectra of compound 5n

¹H NMR (300 MHz, CDCl₃, 298 K)

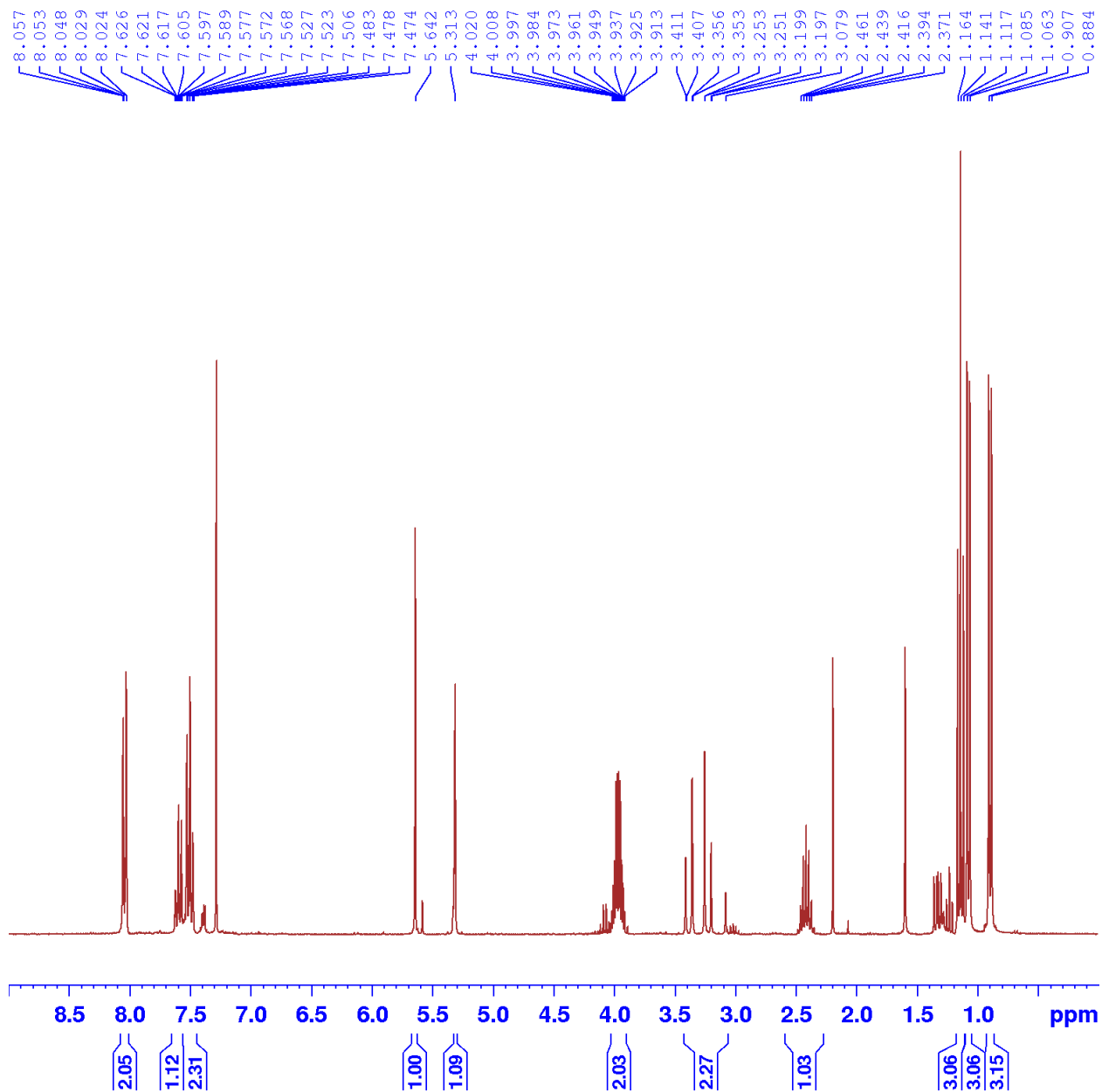
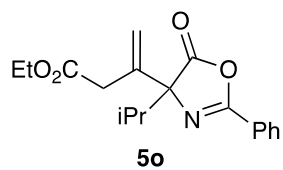


^{13}C NMR (75 MHz, CDCl_3 , 298 K)

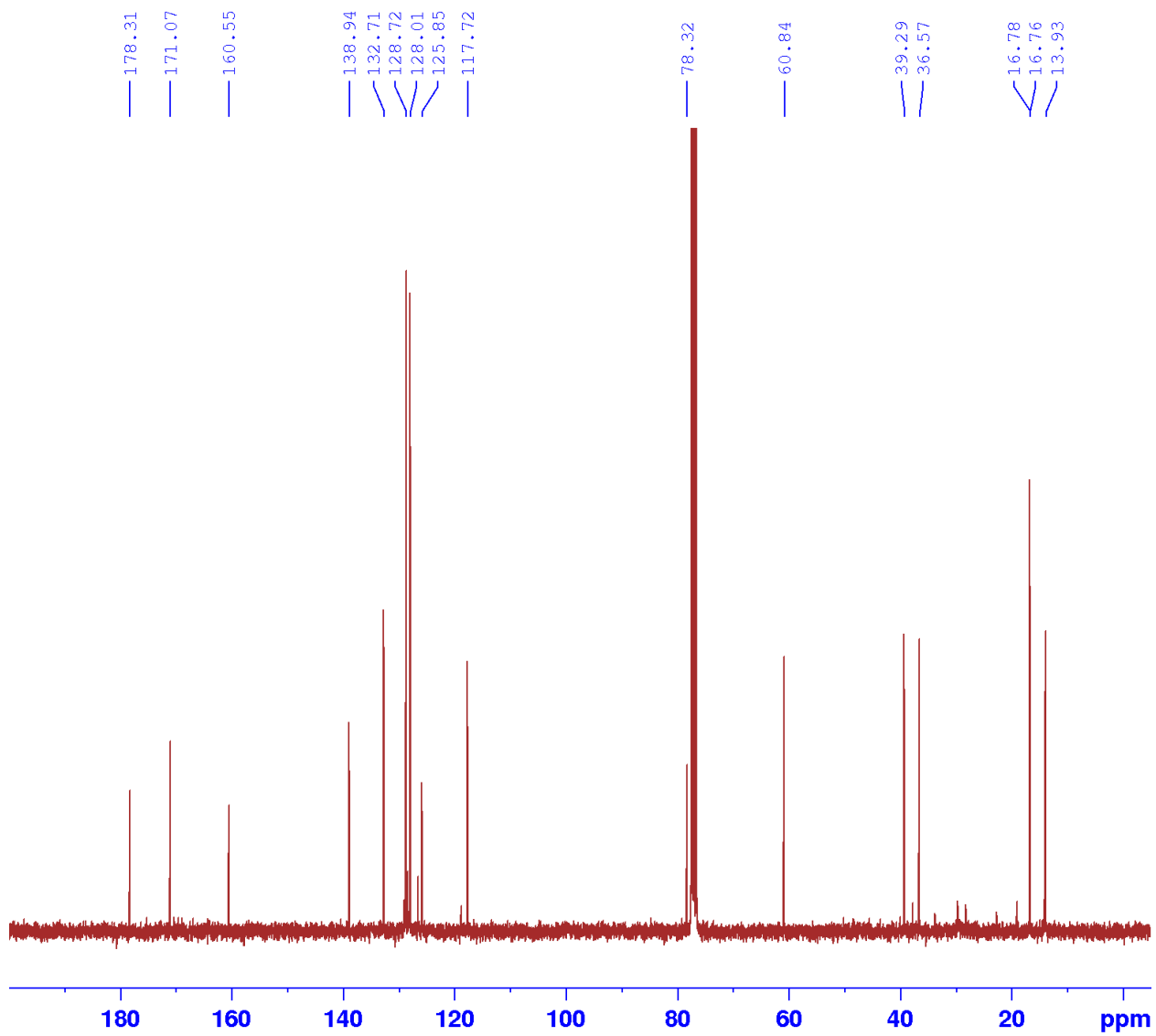
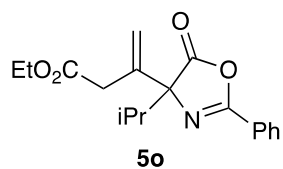


NMR spectra of compound 5o (containing traces of ring-opened product)

¹H NMR (300 MHz, CDCl₃, 298 K)

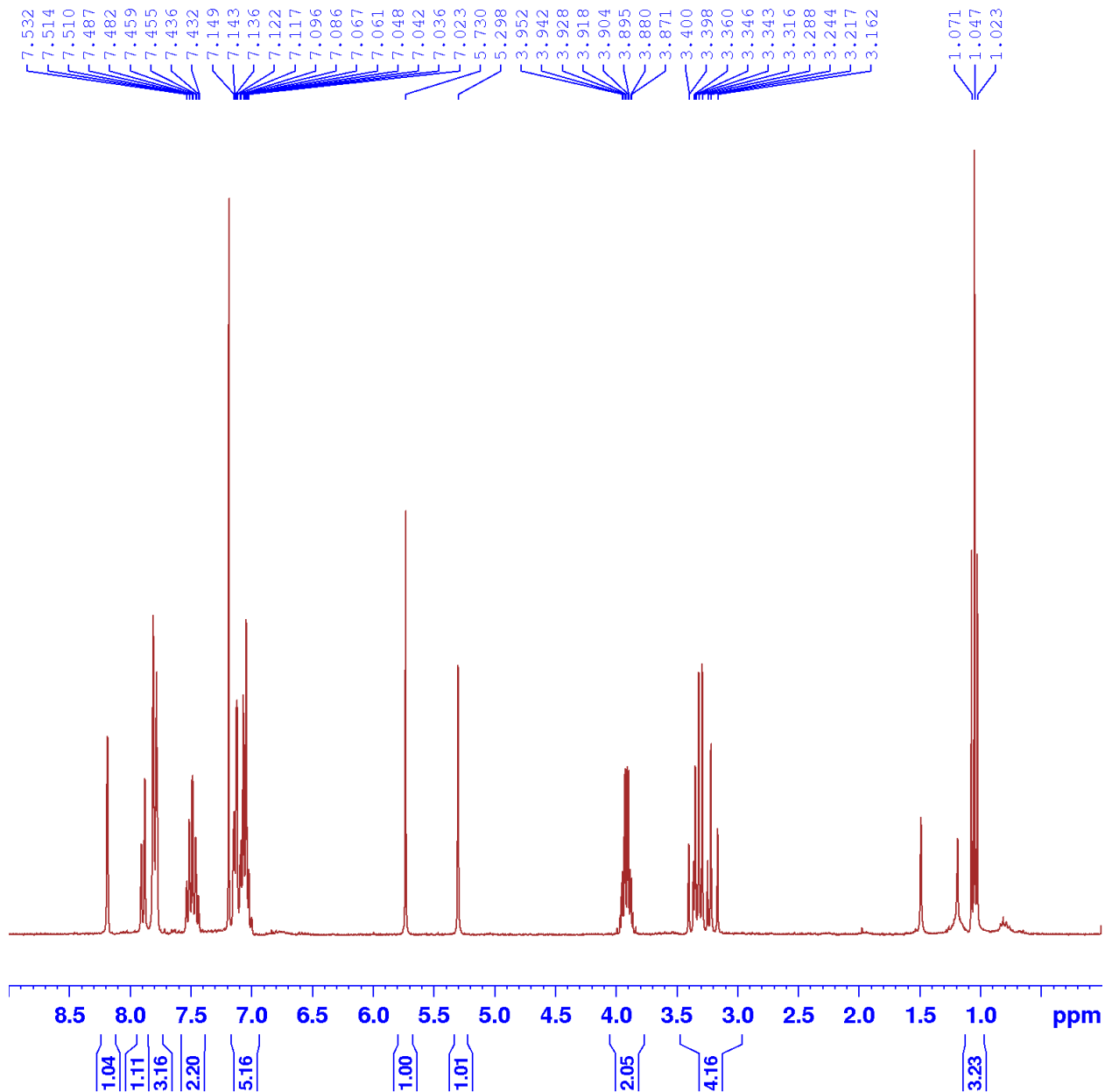
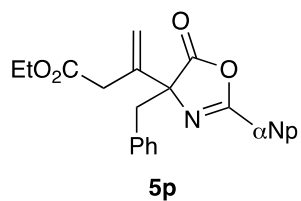


^{13}C NMR (75 MHz, CDCl_3 , 298 K)

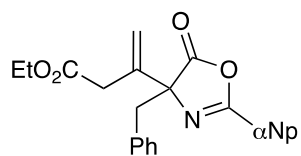


NMR spectra of compound 5p

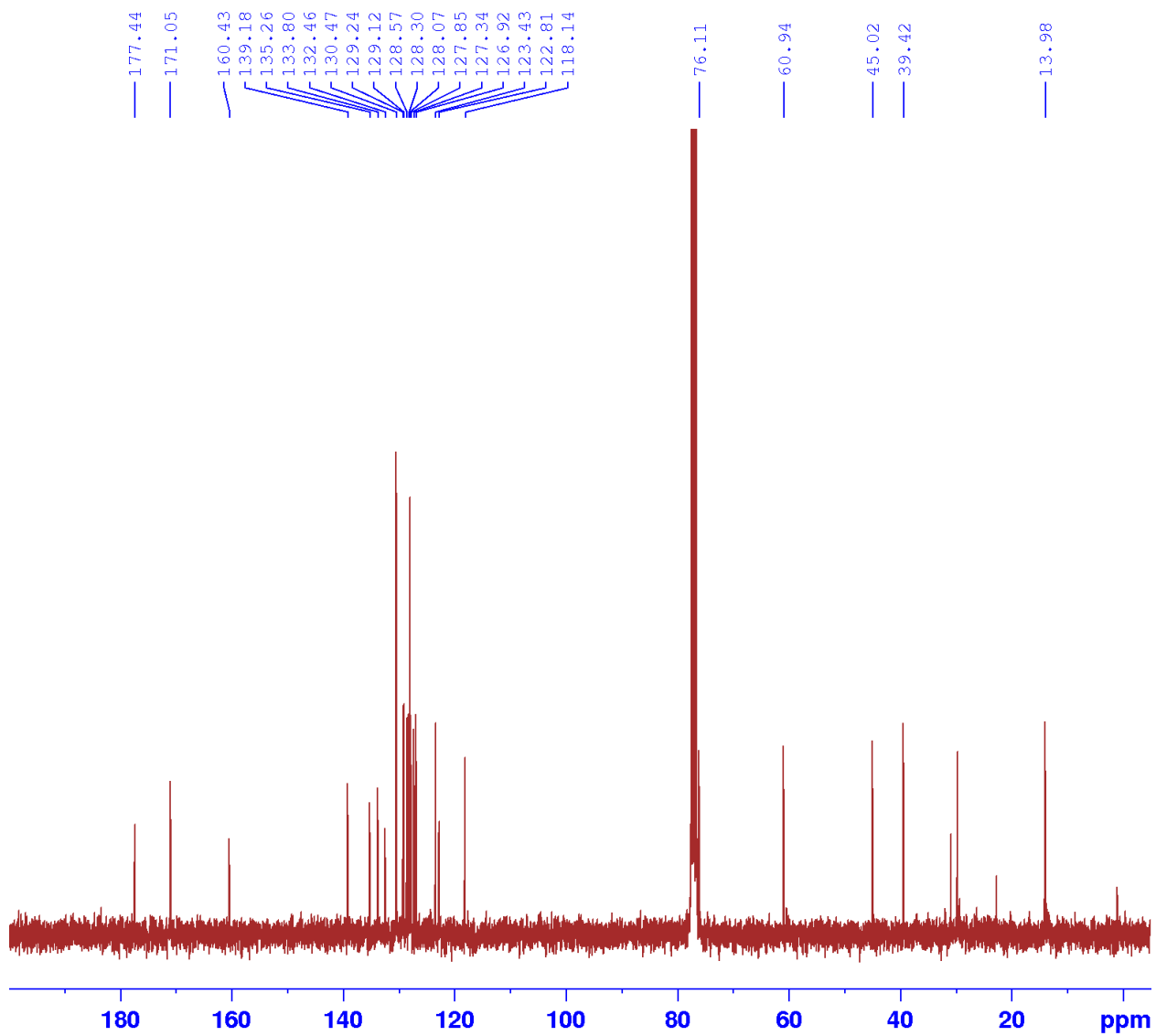
¹H NMR (300 MHz, CDCl₃, 298 K)



^{13}C NMR (75 MHz, CDCl_3 , 298 K)

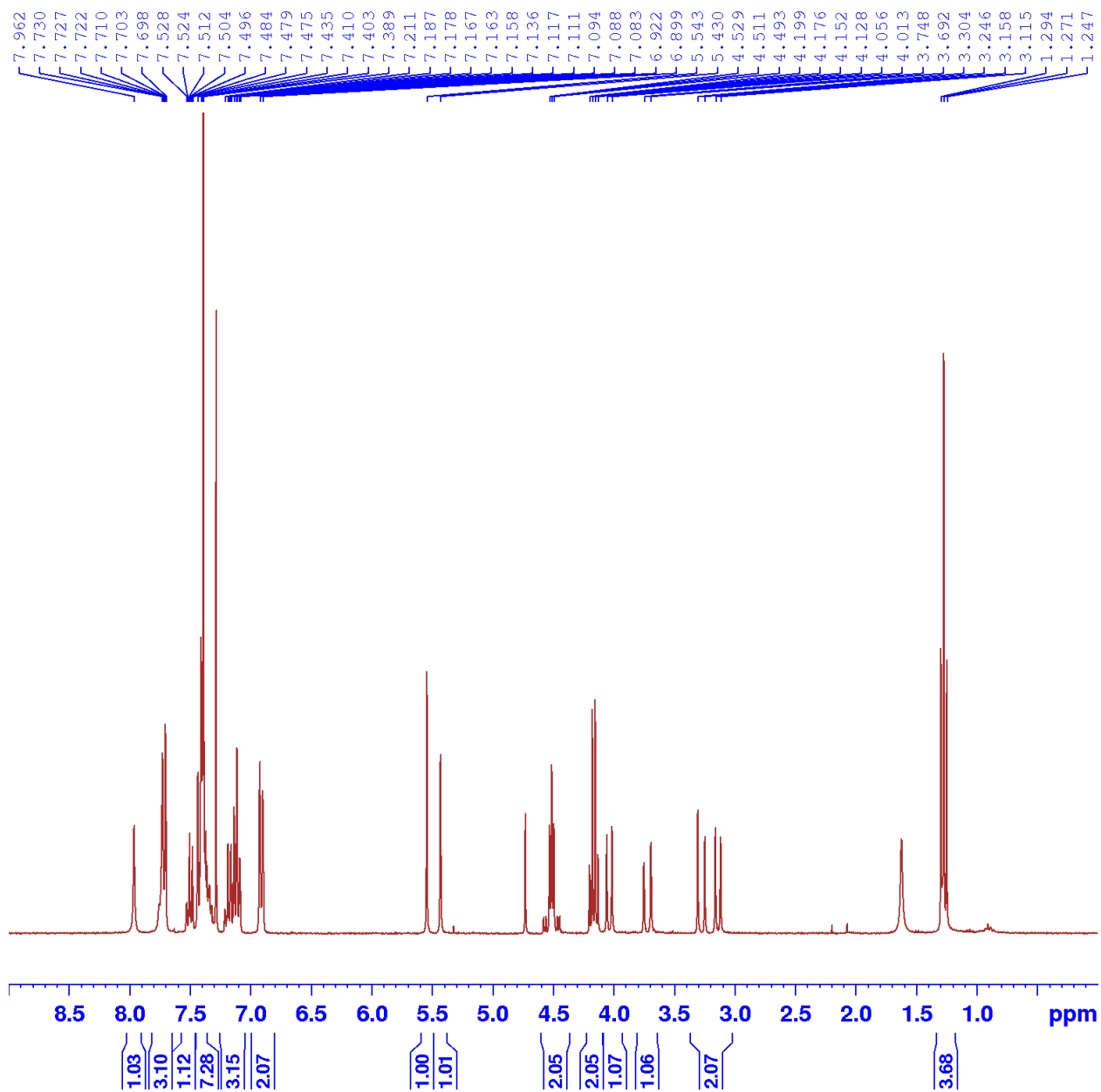
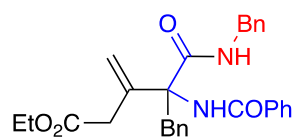


5p

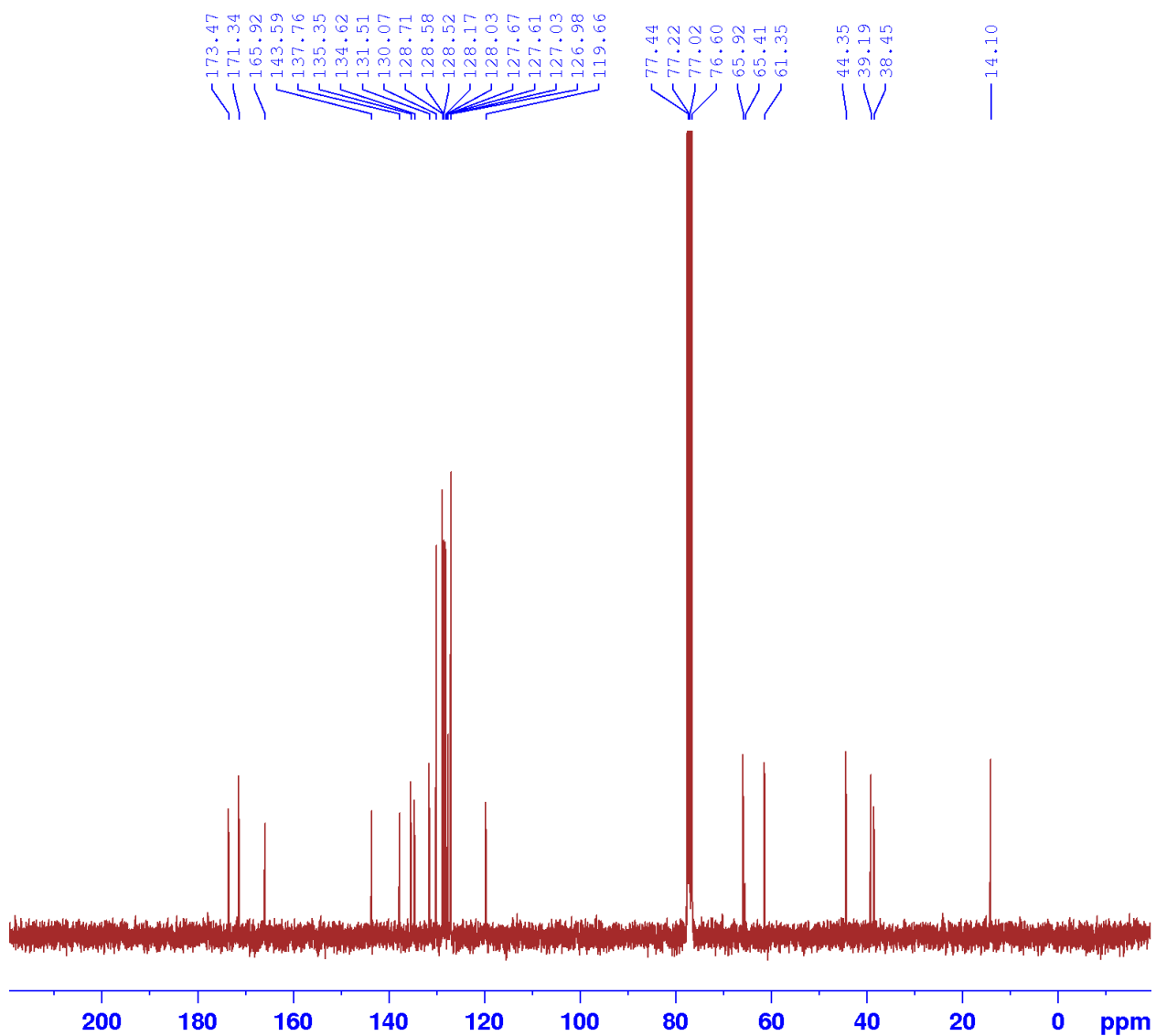
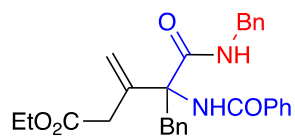


NMR spectra of compound 6a

¹H NMR (300 MHz, CDCl₃, 298 K)

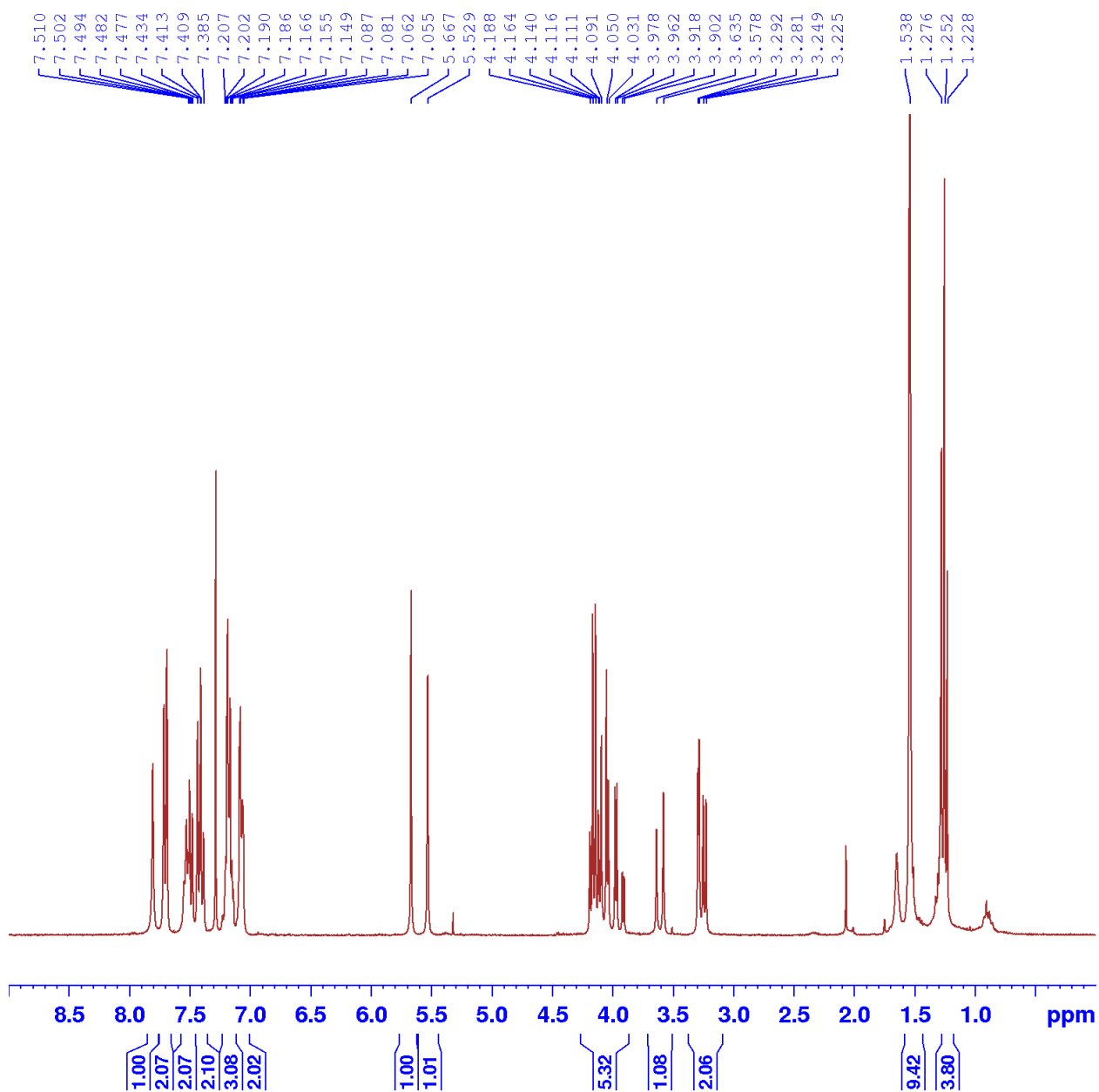
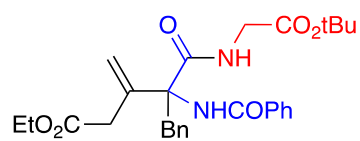


^{13}C NMR (75 MHz, CDCl_3 , 298 K)

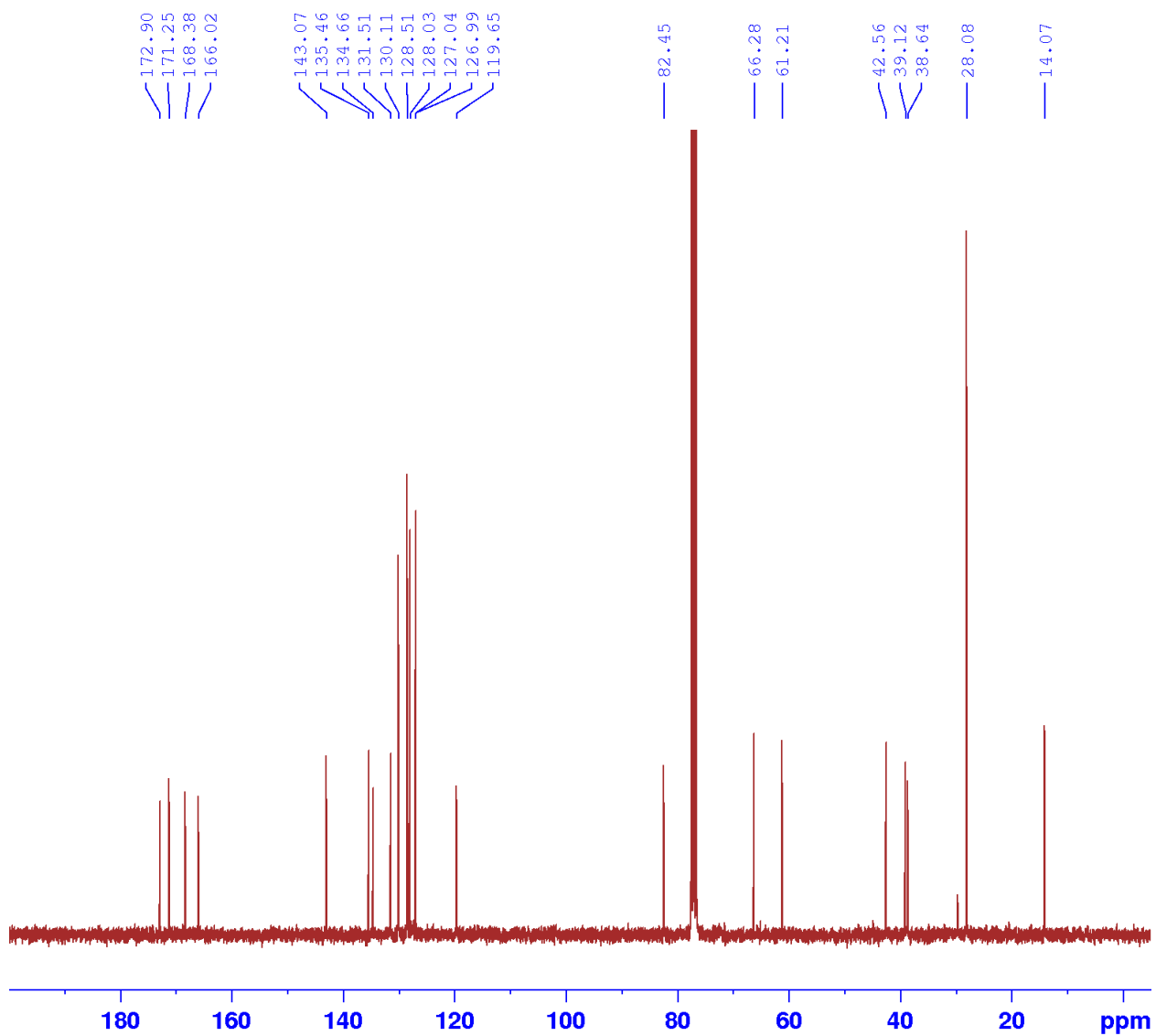
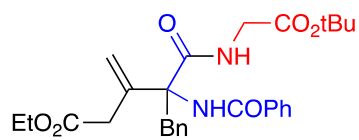


NMR spectra of compound 6b

¹H NMR (300 MHz, CDCl₃, 298 K)

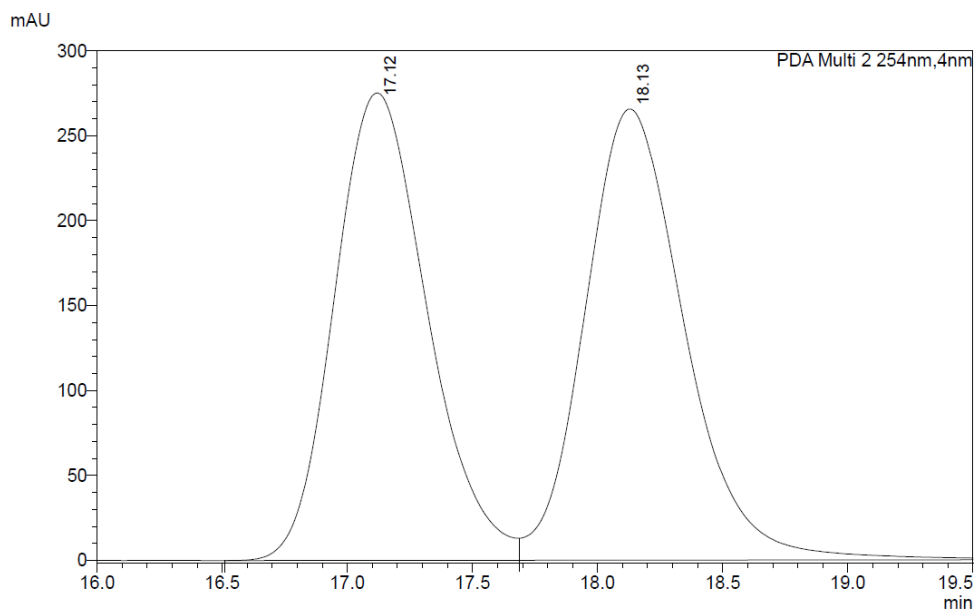
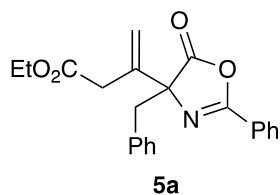


^{13}C NMR (75 MHz, CDCl_3 , 298 K)



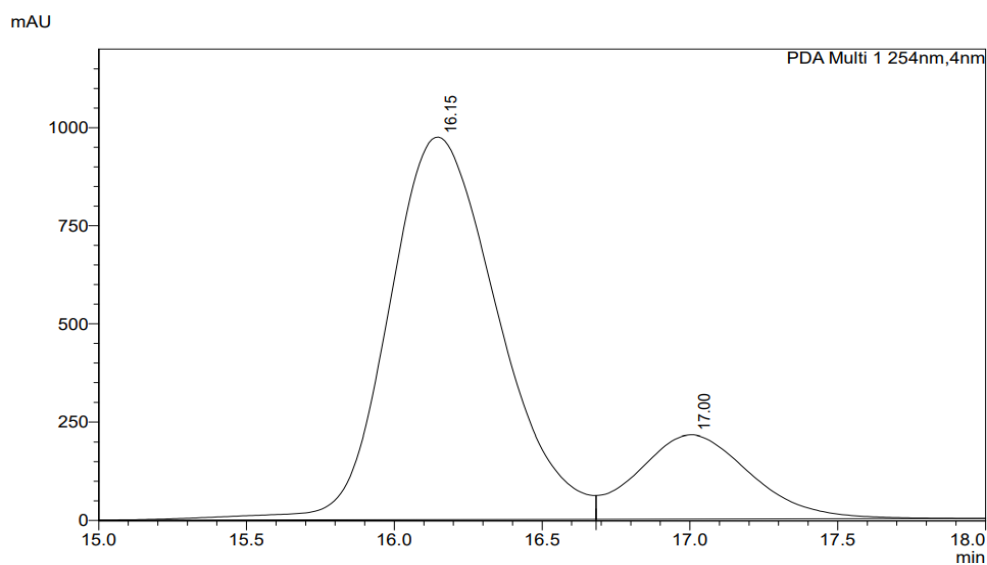
5. HPLC Chromatograms

HPLC traces of compound 5a



Peak Table

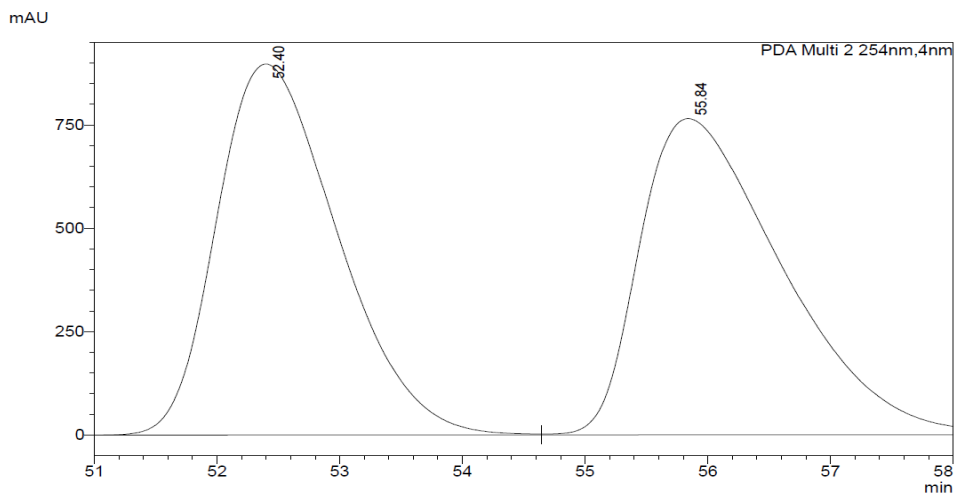
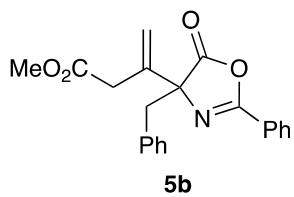
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 17.12 | 7090681 | 48.98 |
| 2 | 18.13 | 7387342 | 51.02 |
| Total | | 14478023 | 100.00 |



Peak Table

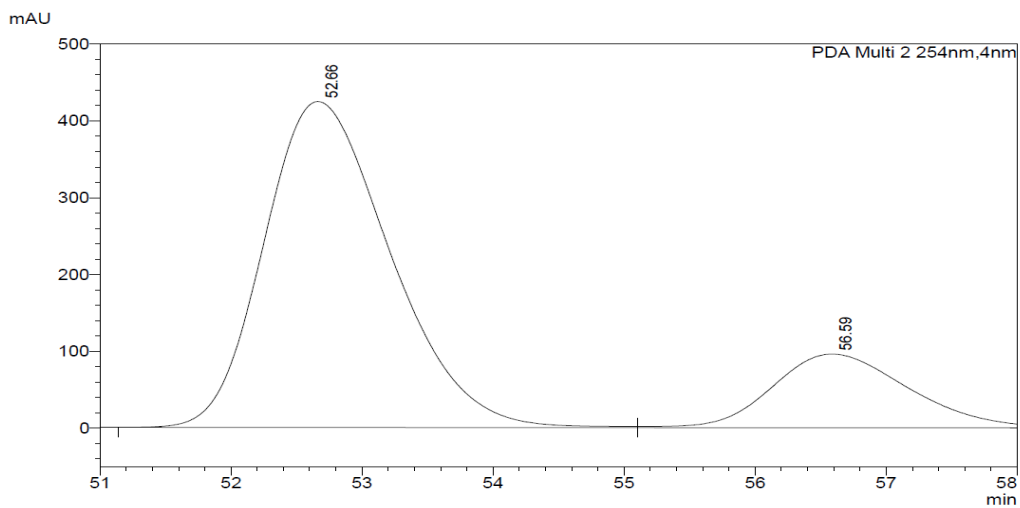
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 16.15 | 24915611 | 81.41 |
| 2 | 17.00 | 5689961 | 18.59 |
| Total | | 30605572 | 100.00 |

HPLC traces of compound 5b



Peak Table

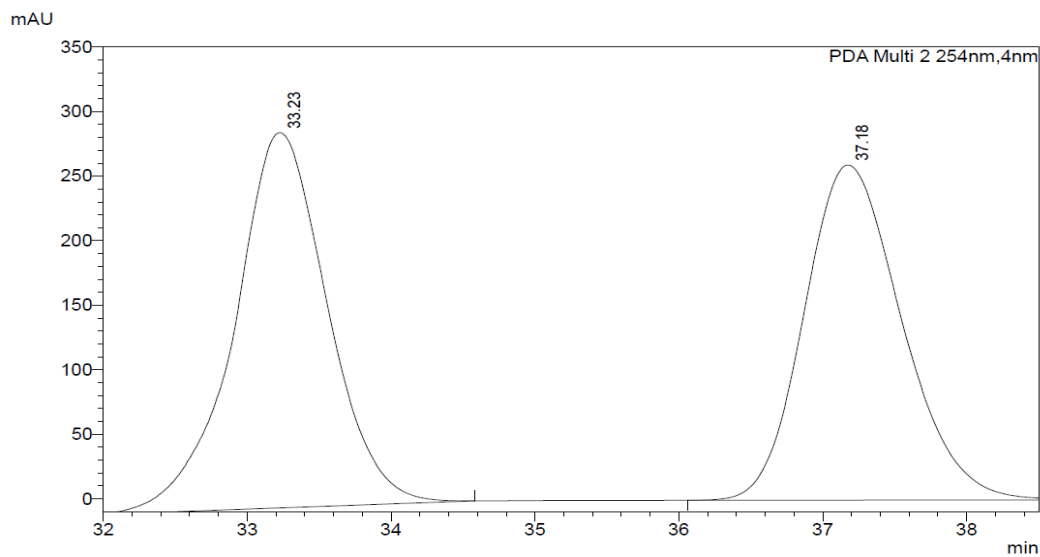
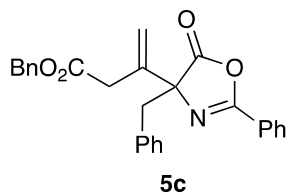
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|-----------|--------|
| 1 | 52.40 | 61451526 | 49.72 |
| 2 | 55.84 | 62152914 | 50.28 |
| Total | | 123604440 | 100.00 |



Peak Table

| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 52.66 | 28371001 | 80.41 |
| 2 | 56.59 | 6913381 | 19.59 |
| Total | | 35284382 | 100.00 |

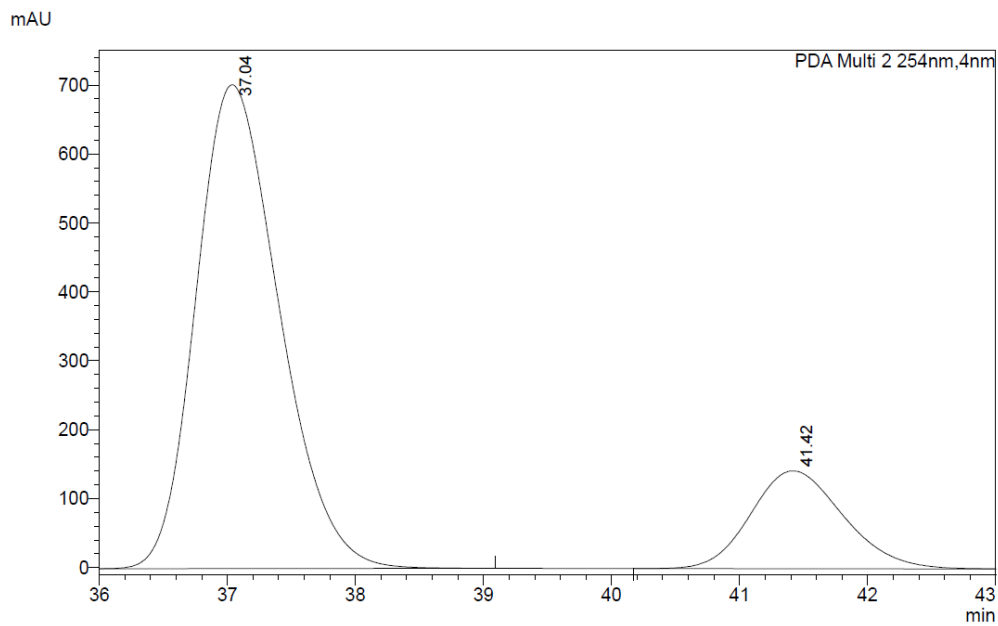
HPLC traces of compound 5c



Peak Table

PDA Ch2 254nm

| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 33.23 | 13050646 | 51.61 |
| 2 | 37.18 | 12234954 | 48.39 |
| Total | | 25285600 | 100.00 |

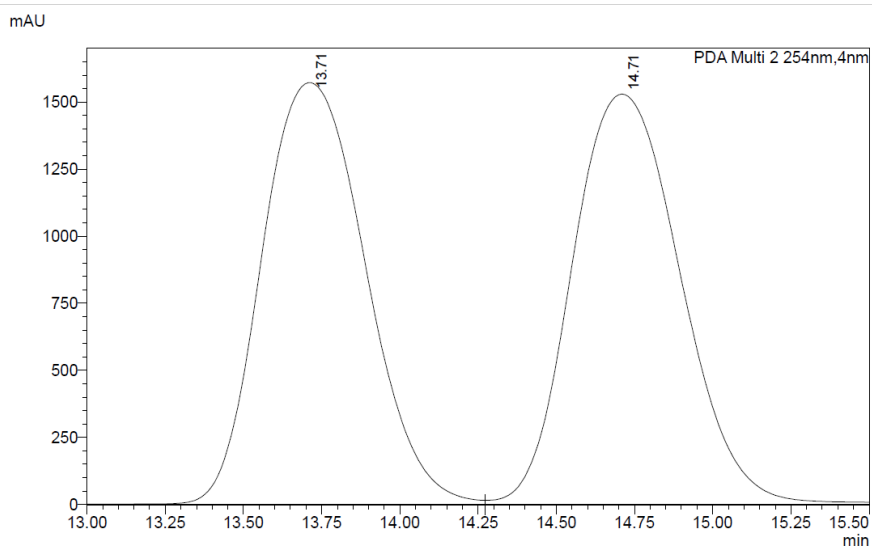
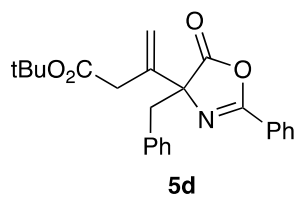


Peak Table

PDA Ch2 254nm

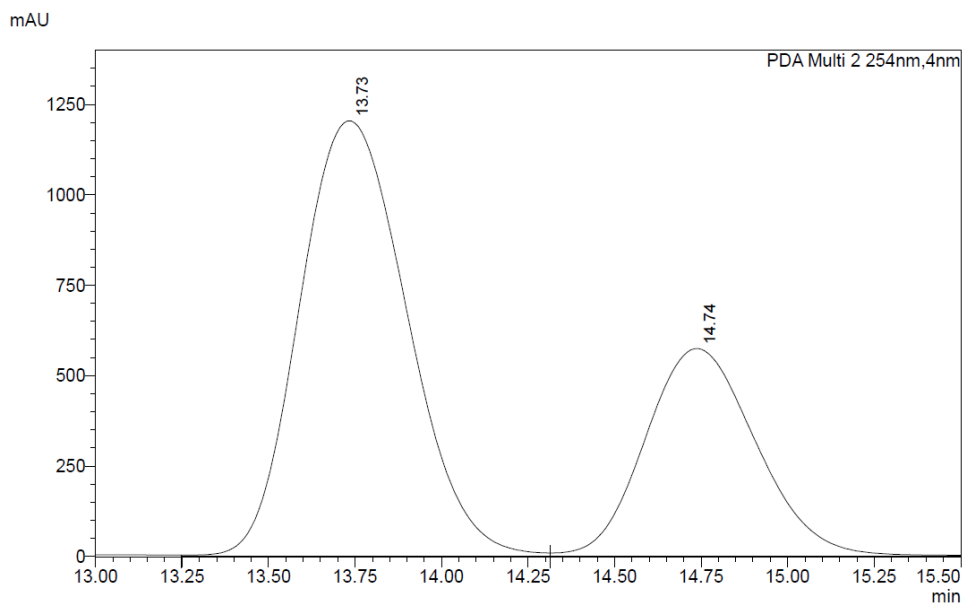
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 37.04 | 31666267 | 81.58 |
| 2 | 41.42 | 7149212 | 18.42 |
| Total | | 38815479 | 100.00 |

HPLC traces of compound 5d



Peak Table

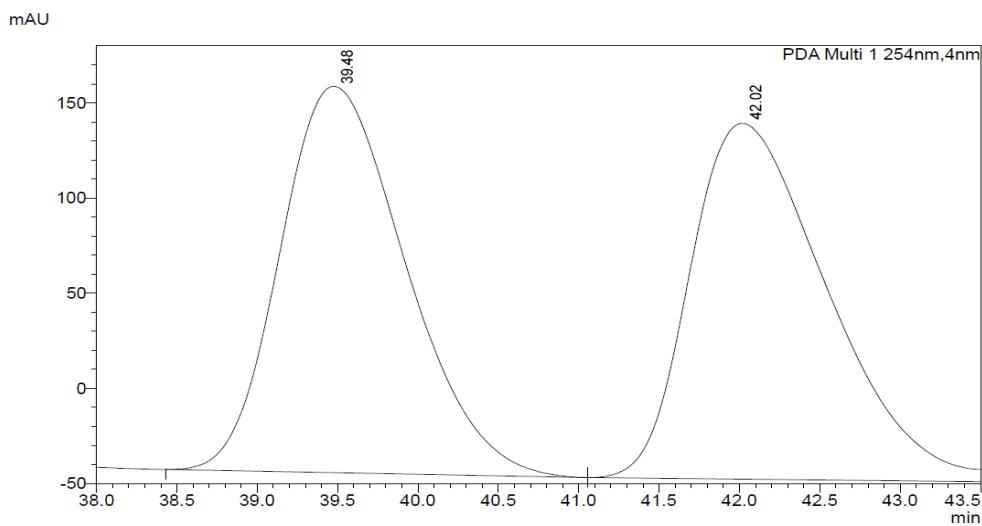
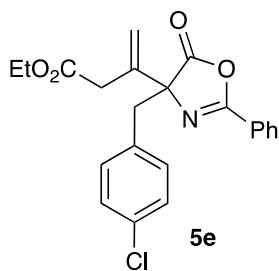
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 13.71 | 36239310 | 49.23 |
| 2 | 14.71 | 37369328 | 50.77 |
| Total | | 73608638 | 100.00 |



Peak Table

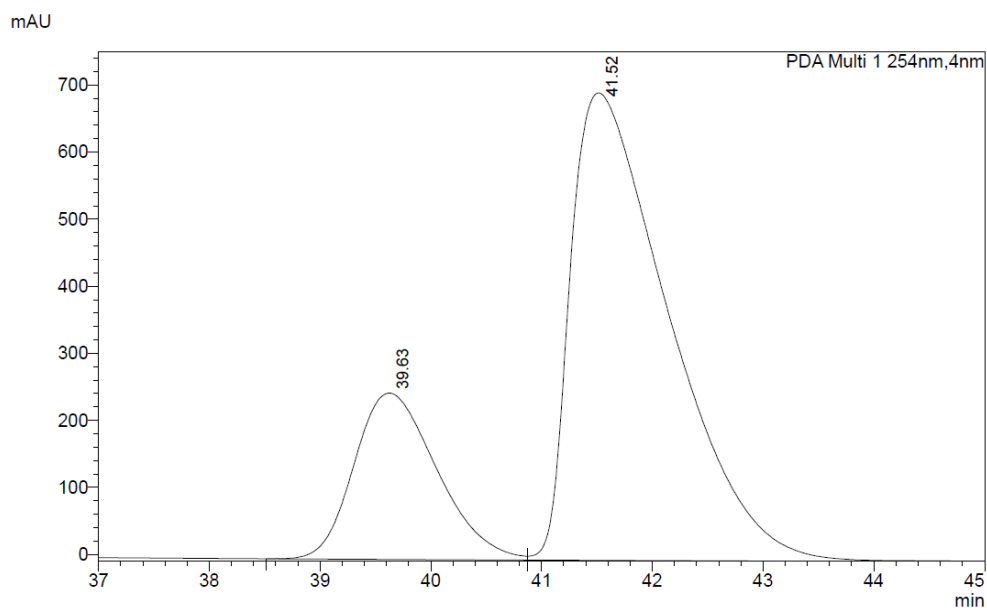
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 13.73 | 26022533 | 66.86 |
| 2 | 14.74 | 12898901 | 33.14 |
| Total | | 38921434 | 100.00 |

HPLC traces of compound 5e



Peak Table

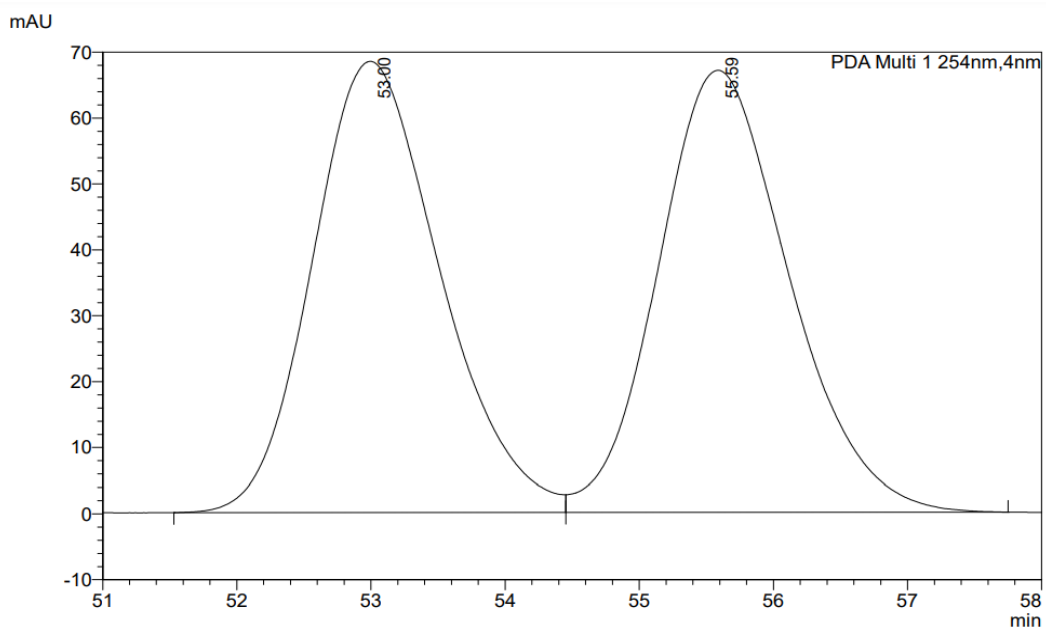
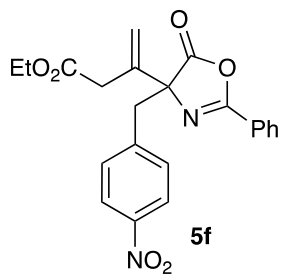
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 39.48 | 11003087 | 49.96 |
| 2 | 42.02 | 11022192 | 50.04 |
| Total | | 22025279 | 100.00 |



Peak Table

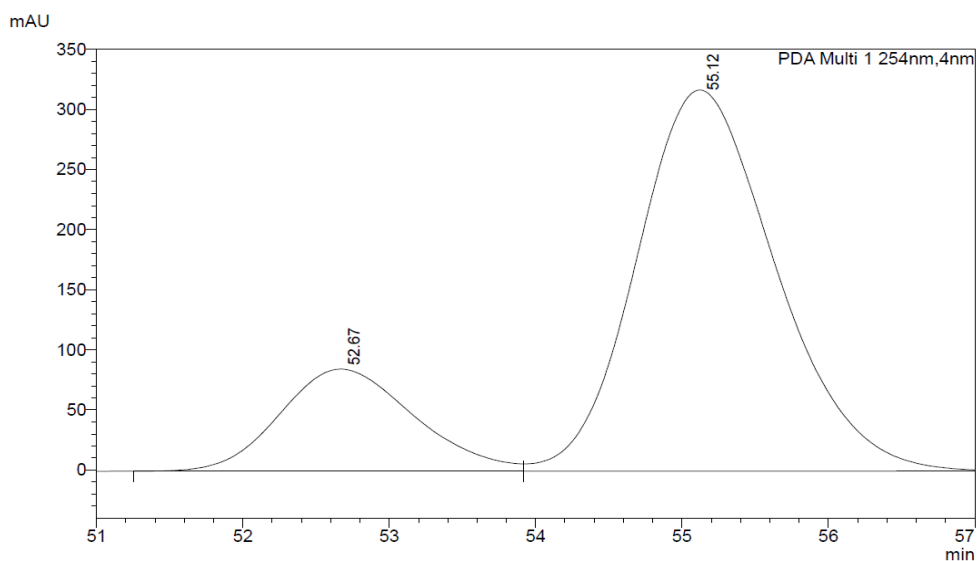
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 39.63 | 12888472 | 23.05 |
| 2 | 41.52 | 43032898 | 76.95 |
| Total | | 55921370 | 100.00 |

HPLC traces of compound 5f



Peak Table

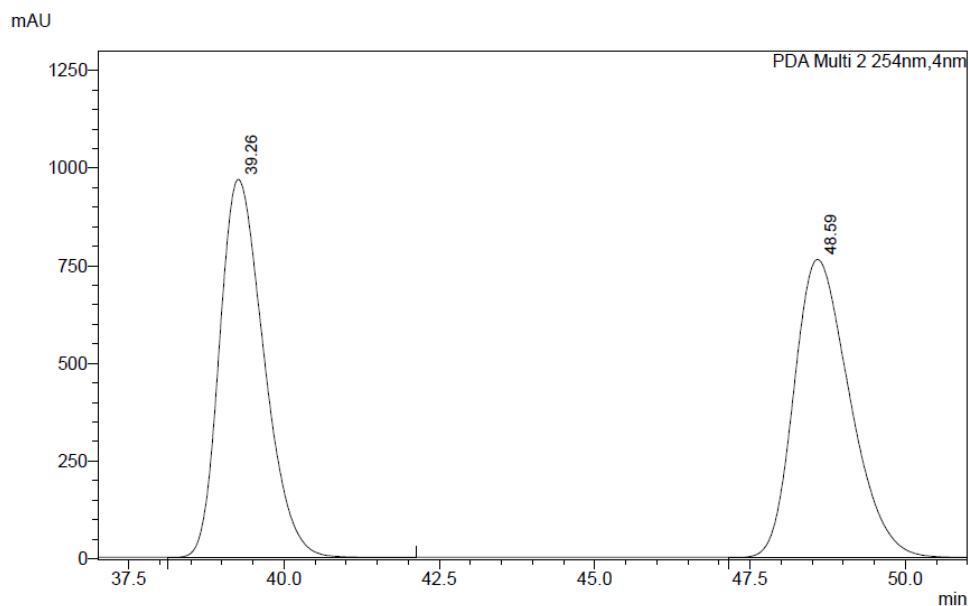
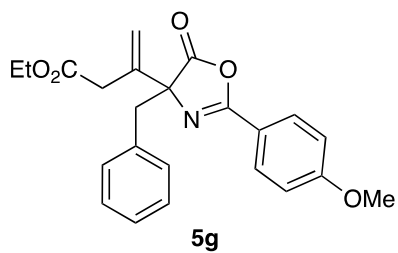
| PDA Ch1 254nm | | | |
|---------------|-----------|---------|--------|
| Peak# | Ret. Time | Area | Area% |
| 1 | 53.00 | 4512895 | 49.85 |
| 2 | 55.59 | 4539681 | 50.15 |
| Total | | 9052576 | 100.00 |



Peak Table

| PDA Ch1 254nm | | | |
|---------------|-----------|----------|--------|
| Peak# | Ret. Time | Area | Area% |
| 1 | 52.67 | 5420086 | 20.54 |
| 2 | 55.12 | 20967253 | 79.46 |
| Total | | 26387339 | 100.00 |

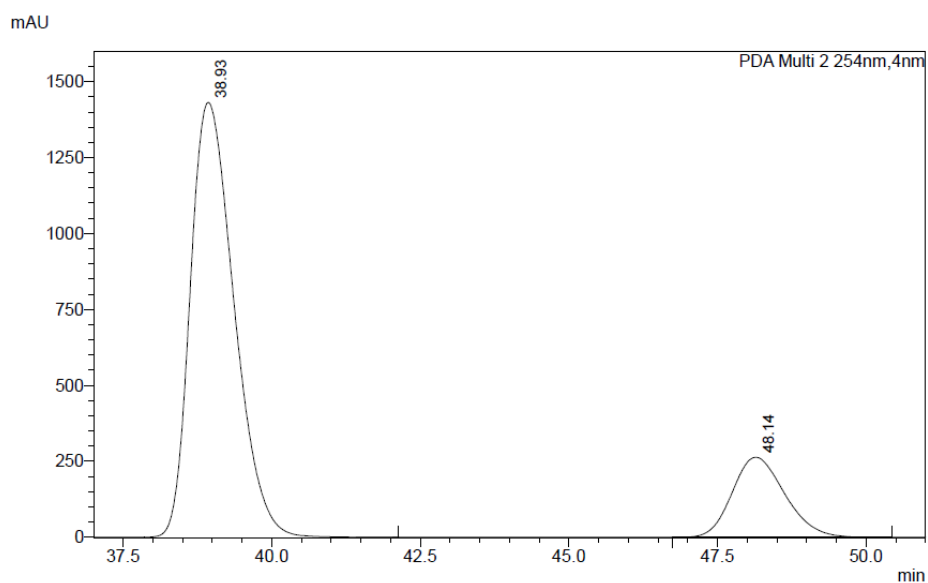
HPLC traces of compound 5g



Peak Table

PDA Ch2 254nm

| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 39.26 | 47885898 | 50.13 |
| 2 | 48.59 | 47646696 | 49.87 |
| Total | | 95532594 | 100.00 |

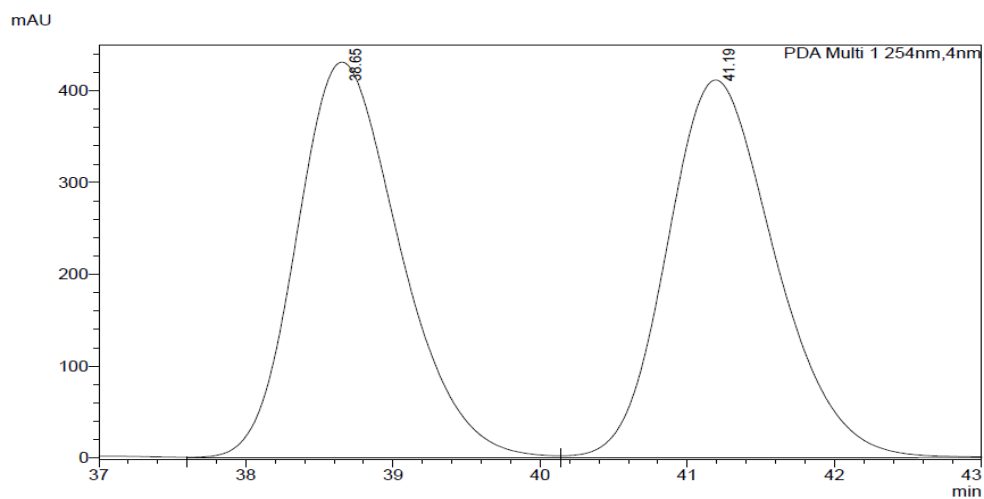
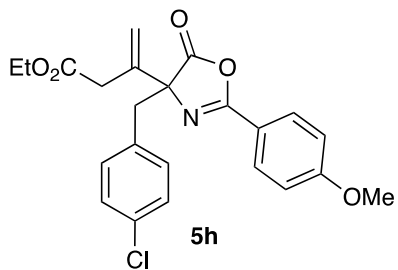


Peak Table

PDA Ch2 254nm

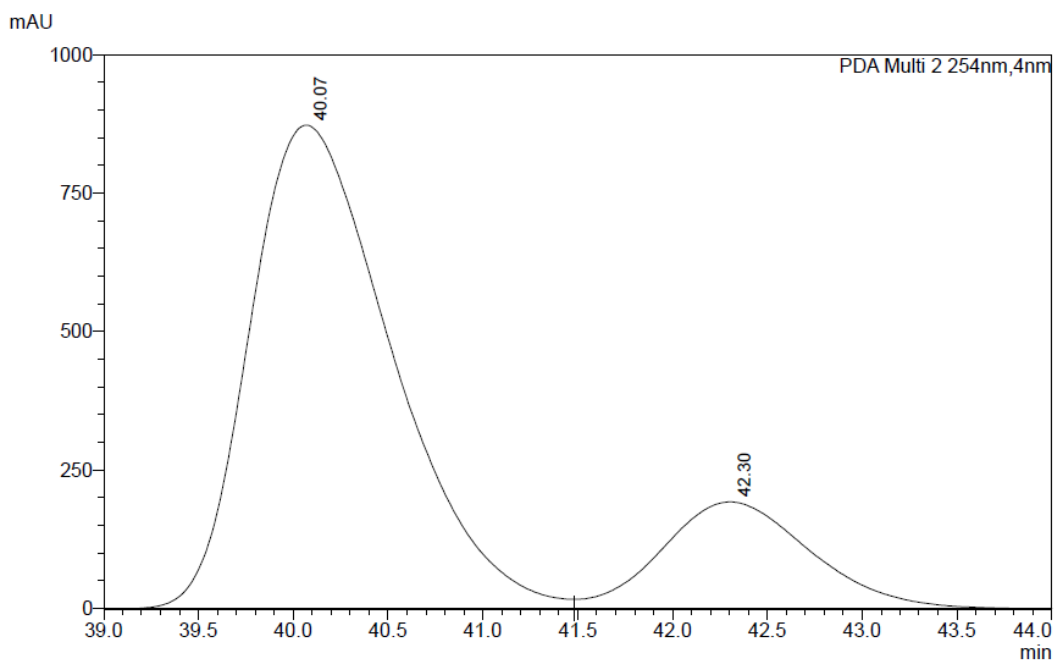
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 38.93 | 73851534 | 81.96 |
| 2 | 48.14 | 16253845 | 18.04 |
| Total | | 90105379 | 100.00 |

HPLC traces of compound 5h



Peak Table

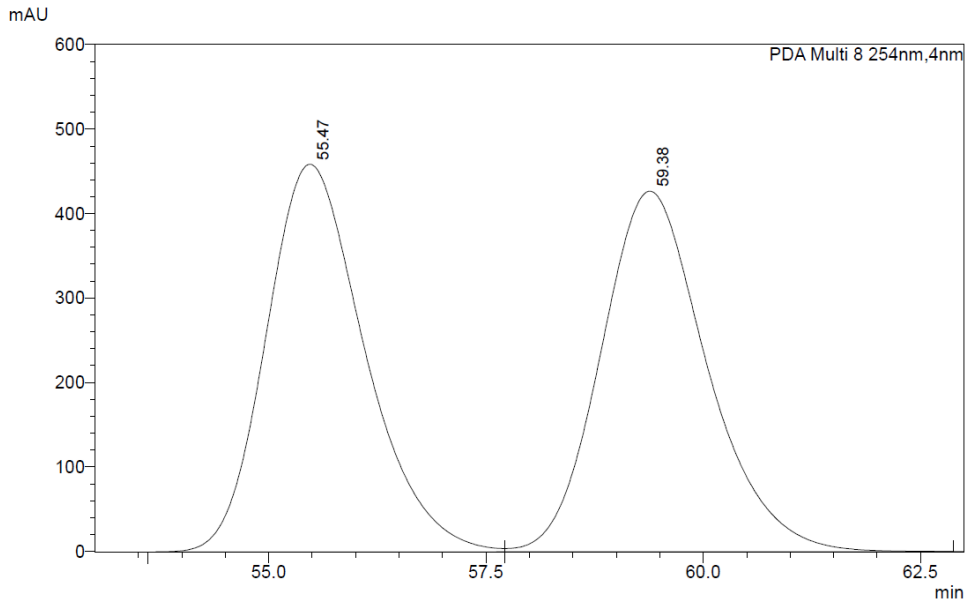
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 38.65 | 21021081 | 49.93 |
| 2 | 41.19 | 21081792 | 50.07 |
| Total | | 42102873 | 100.00 |



Peak Table

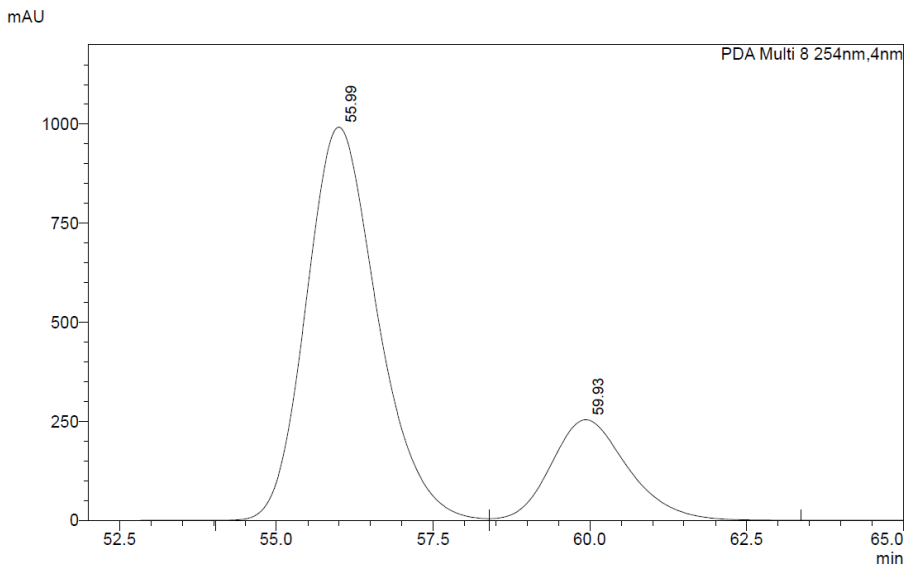
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 40.07 | 45563752 | 81.18 |
| 2 | 42.30 | 10559991 | 18.82 |
| Total | | 56123743 | 100.00 |

HPLC traces of compound 5i



Peak Table

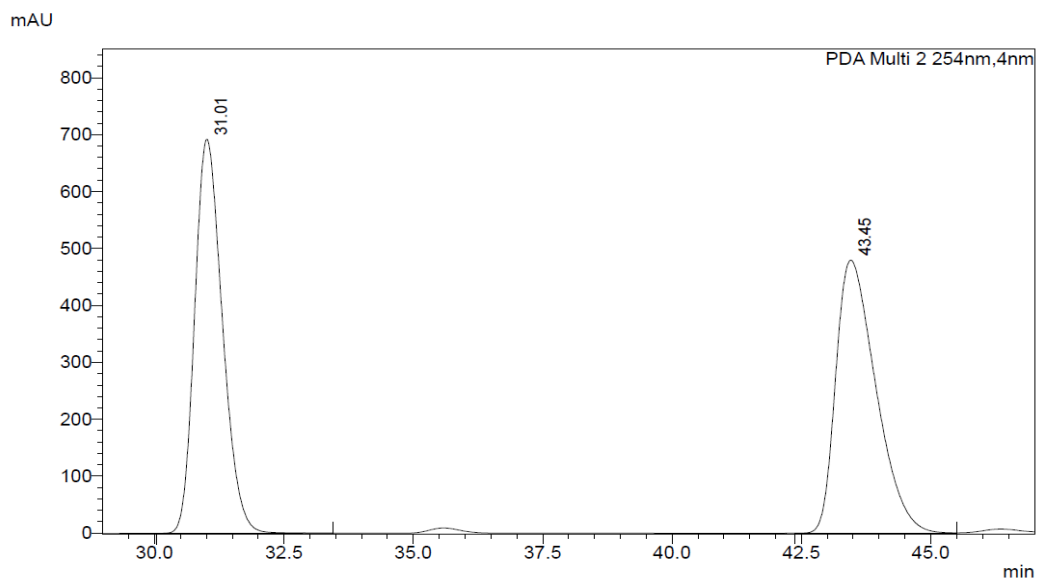
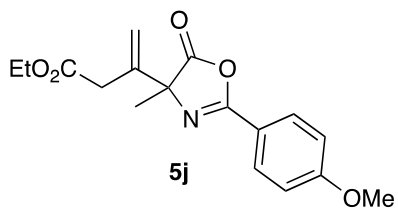
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 55.47 | 36122440 | 49.97 |
| 2 | 59.38 | 36164191 | 50.03 |
| Total | | 72286631 | 100.00 |



Peak Table

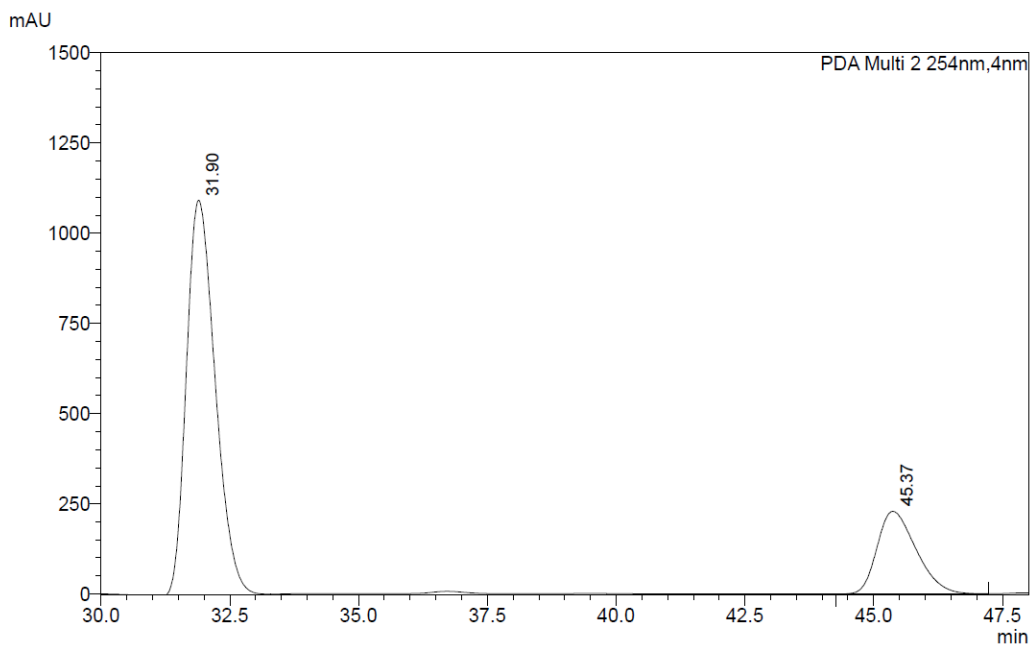
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|-----------|--------|
| 1 | 55.99 | 78354208 | 78.33 |
| 2 | 59.93 | 21670718 | 21.67 |
| Total | | 100024926 | 100.00 |

HPLC traces of compound 5j



Peak Table

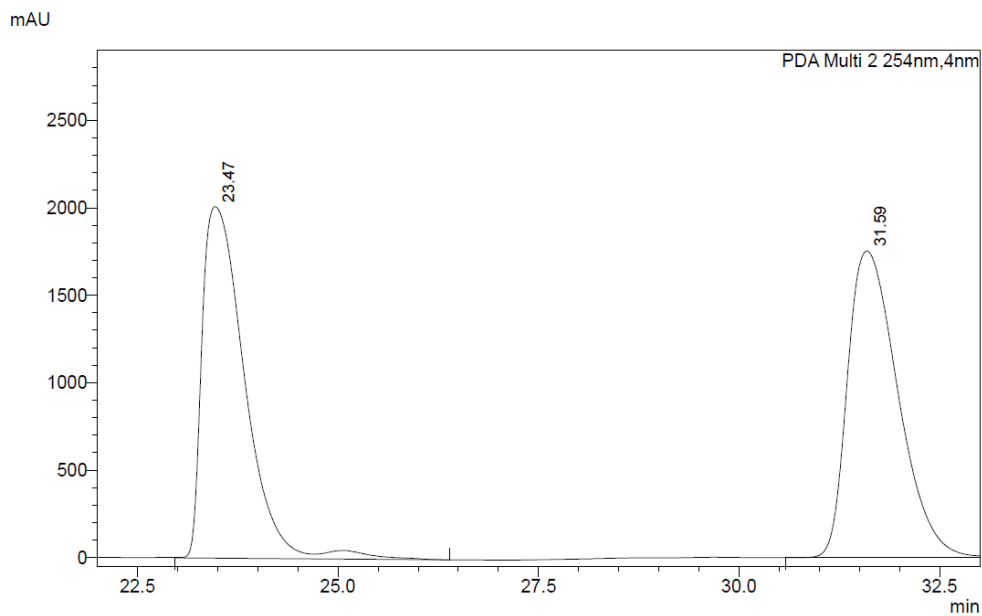
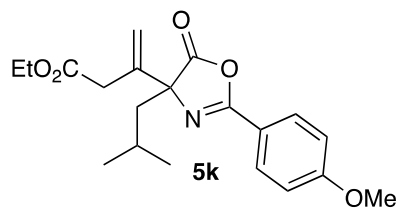
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 31.01 | 25809245 | 49.95 |
| 2 | 43.45 | 25859036 | 50.05 |
| Total | | 51668281 | 100.00 |



Peak Table

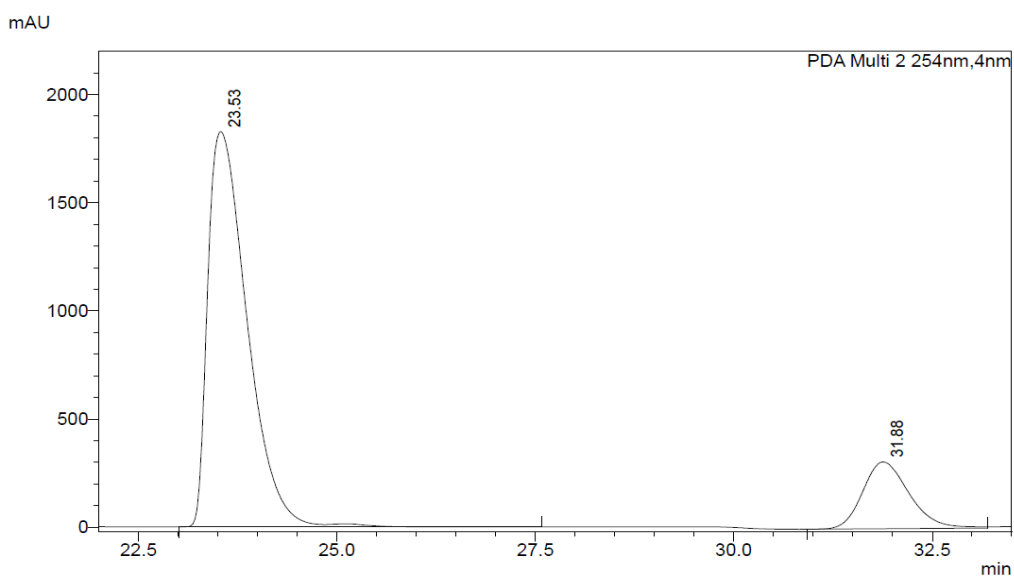
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 31.90 | 43027961 | 78.00 |
| 2 | 45.37 | 12135252 | 22.00 |
| Total | | 55163214 | 100.00 |

HPLC traces of compound 5k



Peak Table

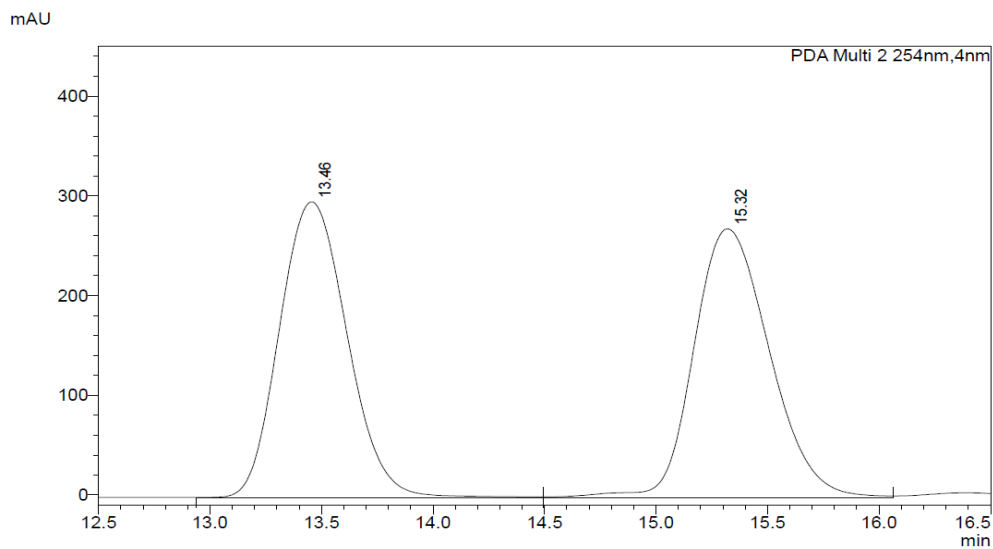
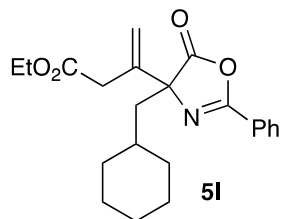
| PDA Ch2 254nm | | | |
|---------------|-----------|-----------|--------|
| Peak# | Ret. Time | Area | Area% |
| 1 | 23.47 | 74530914 | 49.30 |
| 2 | 31.59 | 76659495 | 50.70 |
| Total | | 151190409 | 100.00 |



Peak Table

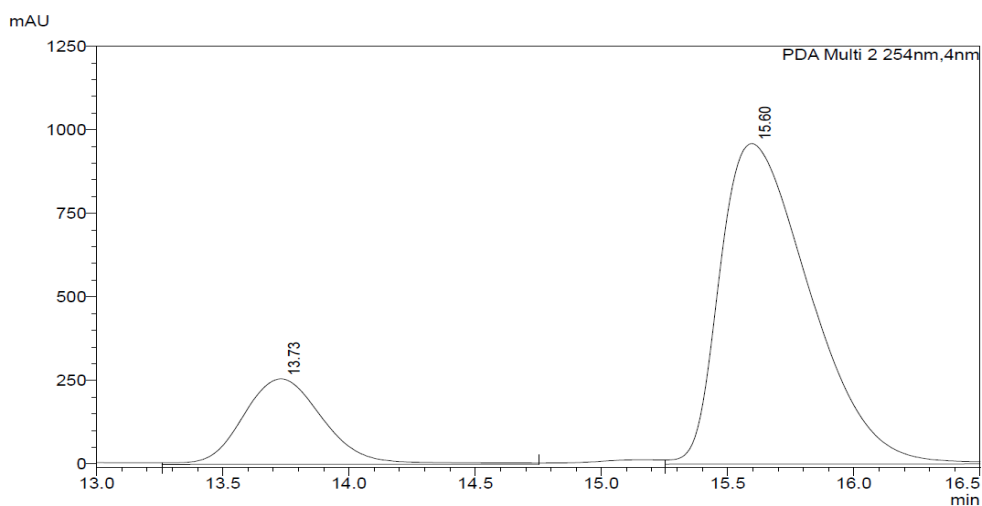
| PDA Ch2 254nm | | | |
|---------------|-----------|----------|--------|
| Peak# | Ret. Time | Area | Area% |
| 1 | 23.53 | 63048120 | 83.36 |
| 2 | 31.88 | 12583478 | 16.64 |
| Total | | 75631598 | 100.00 |

HPLC traces of compound 51



Peak Table

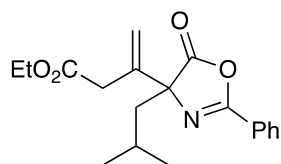
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 13.46 | 6258547 | 49.59 |
| 2 | 15.32 | 6362149 | 50.41 |
| Total | | 12620696 | 100.00 |



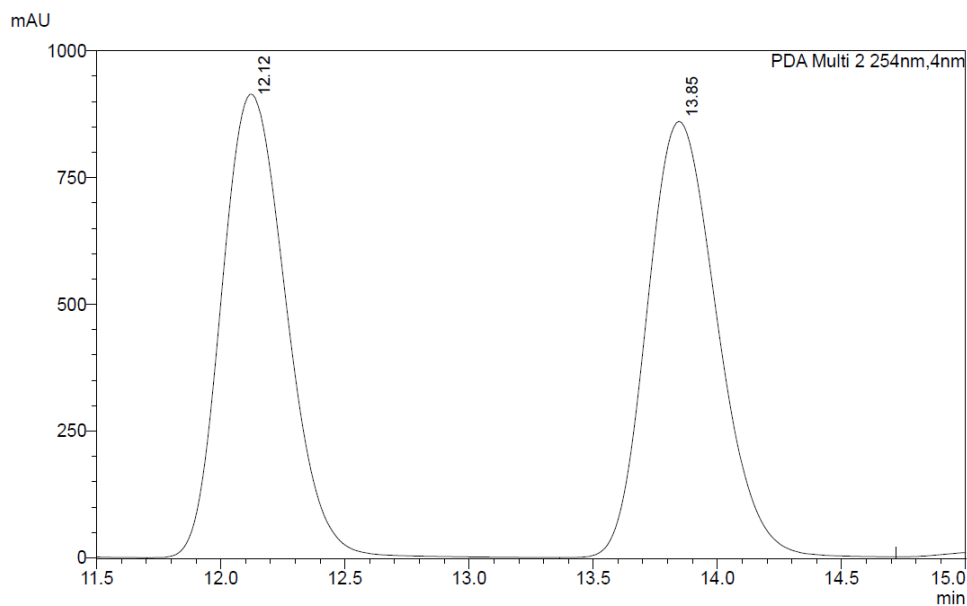
Peak Table

| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 13.73 | 5681974 | 19.09 |
| 2 | 15.60 | 24075662 | 80.91 |
| Total | | 29757636 | 100.00 |

HPLC traces of compound 5m

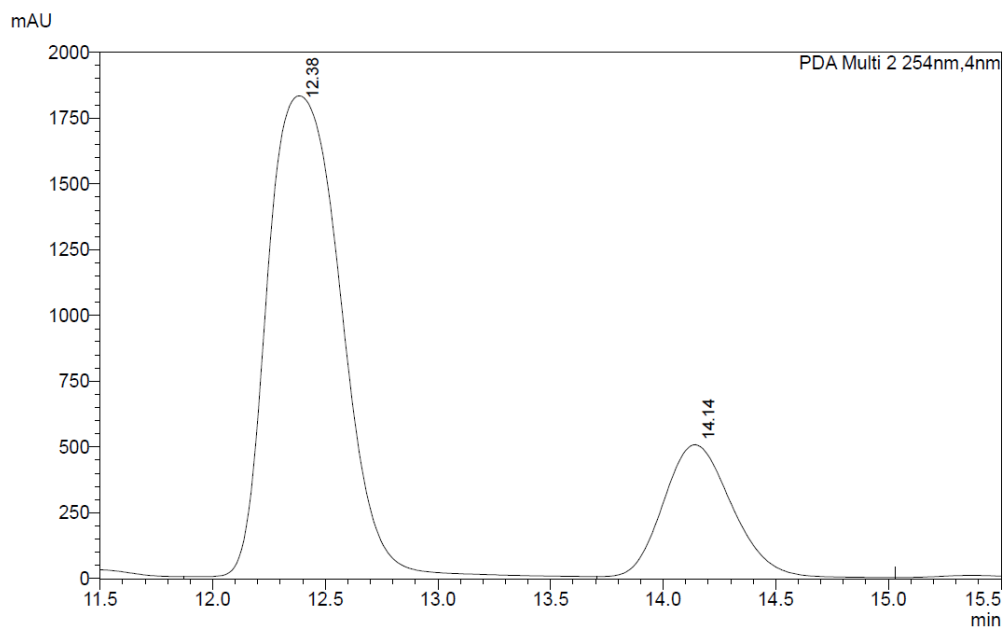


5m



Peak Table

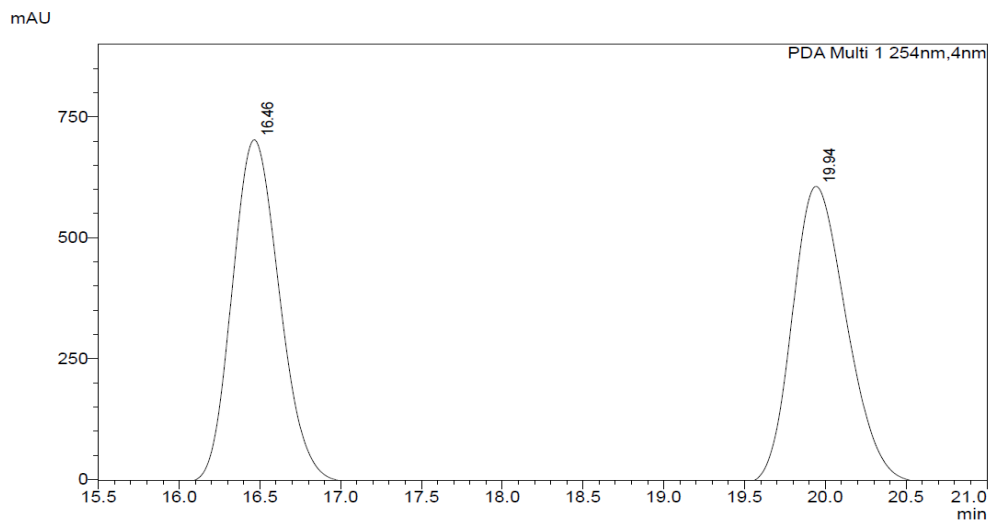
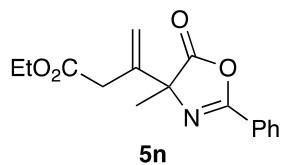
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 12.12 | 16858574 | 49.63 |
| 2 | 13.85 | 17106534 | 50.37 |
| Total | | 33965107 | 100.00 |



Peak Table

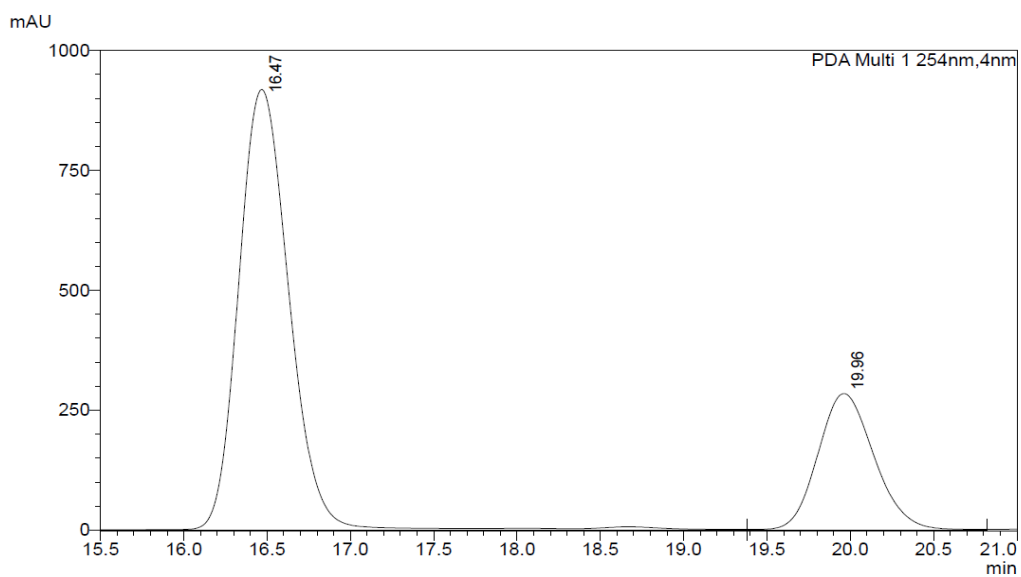
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 12.38 | 42338909 | 79.09 |
| 2 | 14.14 | 11193623 | 20.91 |
| Total | | 53532533 | 100.00 |

HPLC traces of compound 5n



Peak Table

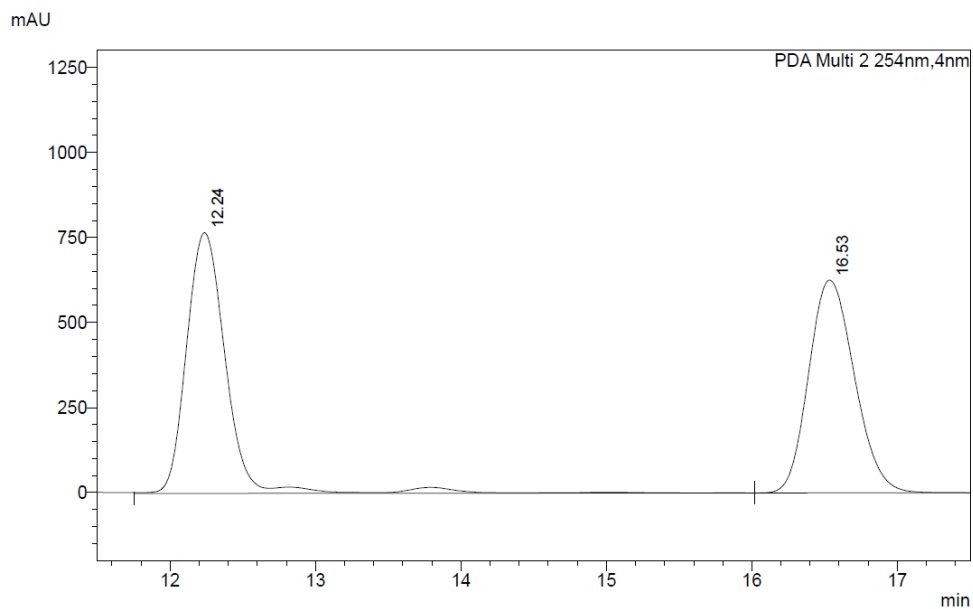
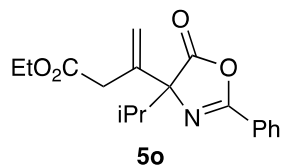
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 16.46 | 14552051 | 49.75 |
| 2 | 19.94 | 14698394 | 50.25 |
| Total | | 29250445 | 100.00 |



Peak Table

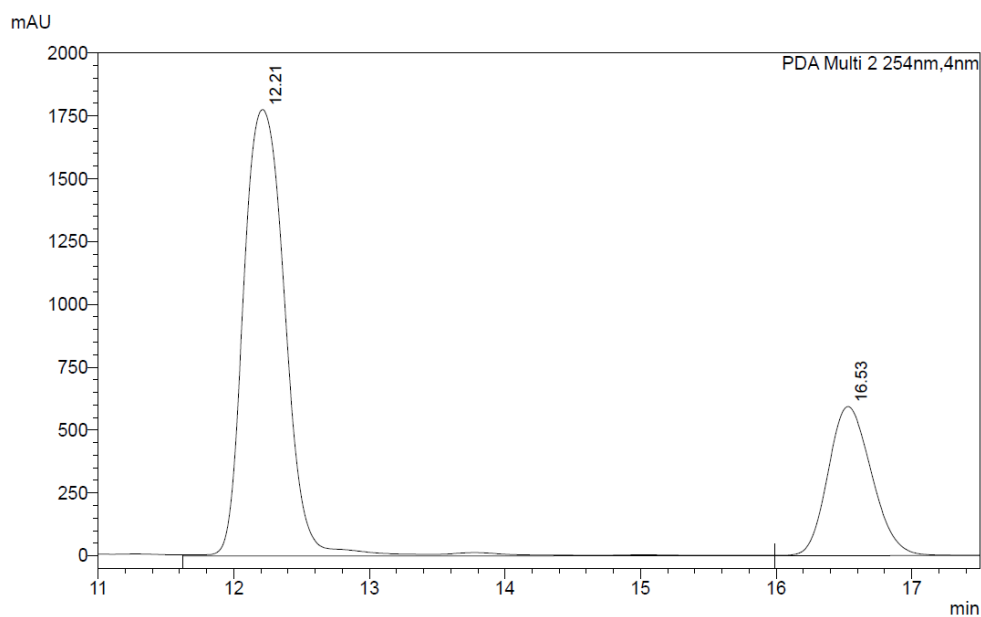
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 16.47 | 19567857 | 74.55 |
| 2 | 19.96 | 6678516 | 25.45 |
| Total | | 26246373 | 100.00 |

HPLC traces of compound 5o



Peak Table

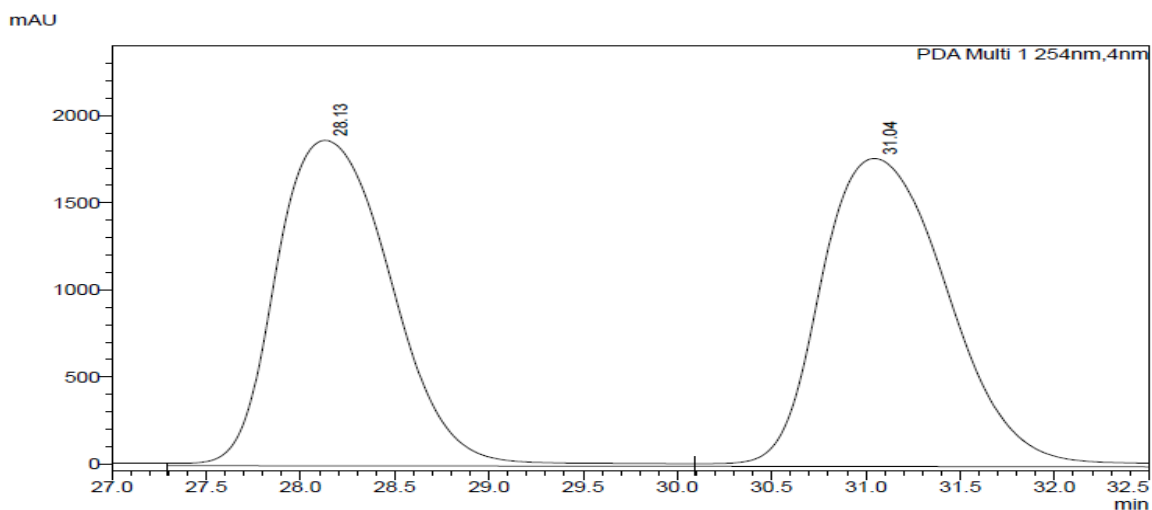
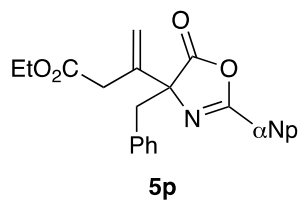
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 12.24 | 14733960 | 51.77 |
| 2 | 16.53 | 13726796 | 48.23 |
| Total | | 28460757 | 100.00 |



Peak Table

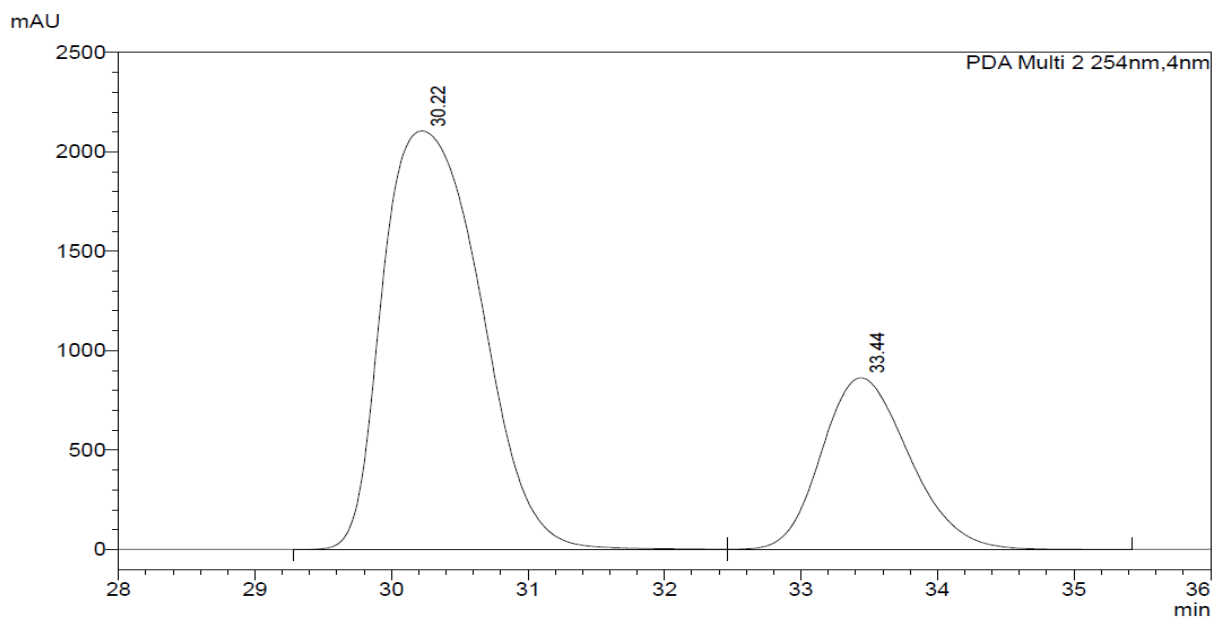
| Peak# | Ret. Time | Area | Area% |
|-------|-----------|----------|--------|
| 1 | 12.21 | 38245981 | 74.18 |
| 2 | 16.53 | 13315363 | 25.82 |
| Total | | 51561344 | 100.00 |

HPLC traces of compound 5p



Peak Table

| Peak# | Ret. Time | Area | Area% |
|-------|-----------|-----------|--------|
| 1 | 28.13 | 79700929 | 46.66 |
| 2 | 31.04 | 91121437 | 53.34 |
| Total | | 170822366 | 100.00 |



Peak Table

| Peak# | Ret. Time | Area | Area% |
|-------|-----------|-----------|--------|
| 1 | 30.22 | 105150834 | 73.15 |
| 2 | 33.44 | 38586996 | 26.85 |
| Total | | 143737830 | 100.00 |