Supporting Information

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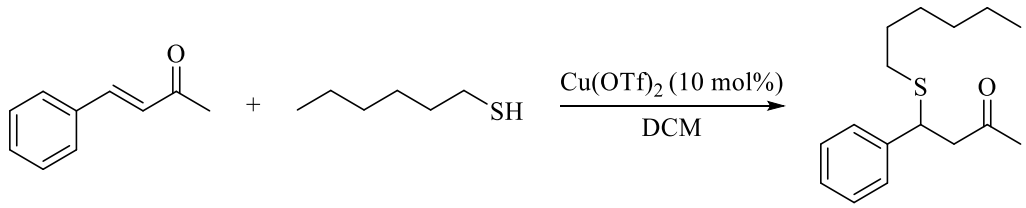
General Information

All commercially available reagents were used without further purification. Column chromatography was performed on silica gel (200-300 mesh). Thin-layer chromatography (TLC) was performed on silica gel plates. ^{1}H NMR (500 MHz), ^{13}C NMR (126 MHz), and ^{19}F NMR (471 MHz) spectra were recorded on a JEOL ECZ500R NMR spectrometer. High-resolution mass spectra (HRMS) were recorded on a Bruker Micro TOF ESI mass spectrometer. Chemical shifts (δ) were reported in ppm, and coupling constants (*J*) were given in Hertz (Hz). Data were reported as s – singlet, d – doublet, t – triplet, q – quartet, dd – doublet of doublets, m – multiplet, dm – doublet of multiplets.

Experimental Section

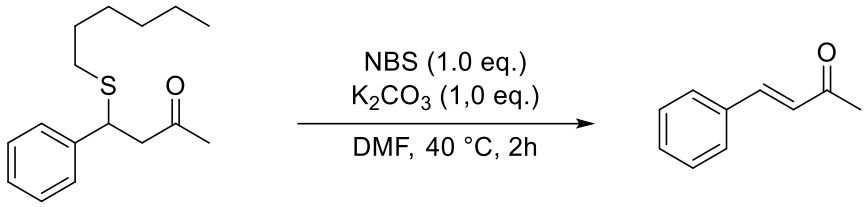
Synthesis of β-keto sulfide[1]



Take the synthesis of **1a** as an example. To a dry round-bottomed flask was added 10% mmol $Cu(OTf)_2$ (2 mmol, 72.3 mg), 10 mL DCM, and trans-4-phenyl-3-buten-2-one (2 mmol, 292.1 mg). Then, hexane-1-thiol (2 mmol, 236.5 mg) was slowly added to the stirring mixture solution. The mixture was stirred overnight. After the reaction was complete (monitored by TLC), the residual was extracted with ethyl acetate. The combined organic phases were washed with brine, dried over Na_2SO_4, and concentrated

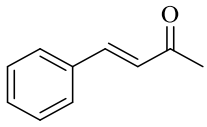
by rotary evaporation. The crude was further purified by silica gel column chromatography to afford **1a**.

Synthesis of Enone



Take the synthesis of **2a** as an example. To a dry glass tube was added 1 mL DMF, 0.2 mmol NBS (1 eq, 35.6 mg), 0.2 mmol **1a** (1 eq, 52.8 mg), and 0.2 mmol K_2CO_3 (1 eq, 27.6 mg). The mixed solution was stirred at 40 °C for two hours. After the reaction was complete (monitored by TLC), the residual was extracted with ethyl acetate. The combined organic phases were washed with brine, dried over Na_2SO_4, and concentrated by rotary evaporation. The crude was further purified by silica gel column chromatography to afford **2a**.

Characterization data



(*E*)-4-phenylbut-3-en-2-one (2a)

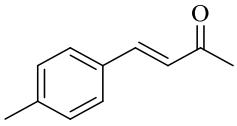
Prepared according to the general procedure from 4-(hexylthio)-4-phenylbutan-2-one **1a** (52.8 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a colourless oil (25.9 mg, 88%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.57 – 7.53 (m, 2H, Ar-*H*), 7.51 (d, *J* = 16.4 Hz, 1H, ArC*H*=CH), 7.40 (m, 3H, Ar-*H*), 6.72 (m, 1H, C*H*CO), 2.38 (d, *J* = 0.8 Hz, 3H, COCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.6, 143.6, 134.5, 130.6, 129.1 (2C), 128.4 (2C), 127.3, 27.6.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{10}H_{10}O$: 147.0804; found: 147.0800.

This data was concordant with literature values.[2]



(*E*)-4-(p-tolyl)but-3-en-2-one (2b)

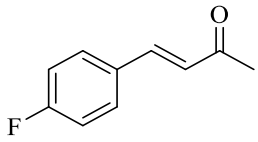
Prepared according to the general procedure from 4-(hexylthio)-4-(p-tolyl)butan-2-one **1b** (55.6 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (23.2 mg, 72%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.48 (d, *J* = 16.3 Hz, 1H, ArC*H*=CH), 7.43 (d, *J* = 8.1 Hz, 2H, Ar-*H*), 7.19 (d, *J* = 7.9 Hz, 2H, Ar-*H*), 6.66 (d, *J* = 16.3 Hz, 1H, C*H*CO), 2.36 (s, 3H, ArCH_3), 2.36 (s, 3H, COCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.6, 143.6, 141.1, 131.8, 129.8 (2C), 128.4 (2C),26.4, 27.5, 21.6.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{11}H_{12}O$: 161.0961; found: 161.0959.

This data was concordant with literature values.[2]



(*E*)-4-(4-fluorophenyl)but-3-en-2-one (2c)

Prepared according to the general procedure from 4-(4-fluorophenyl)-4-(hexylthio)butan-2-one **1c** (56.43 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (25.4 mg, 77%).

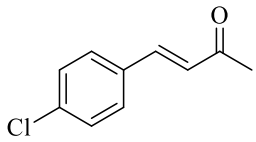
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.56 – 7.50 (m, 2H, Ar-*H*), 7.47 (d, *J* = 16.3 Hz, 1H, ArC*H*=CH), 7.11 – 7.05 (m, 2H, Ar-*H*), 6.63 (d, *J* = 16.3 Hz, 1H, C*H*CO), 2.36 (s, 3H, ArCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.3, 164.1 (d, *J* = 251.8 Hz), 142.2, 130.8 (d, *J* = 3.2 Hz), 130.3 (2C, d, *J* = 8.5 Hz), 127.0, 116.3 (2C, d, *J* = 22.1 Hz), 27.7.

^{19}F NMR (471 MHz, Chloroform-*d*) δ = -113.8.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{10}H_9FO$: 165.0710; found: 165.0703.

This data was concordant with literature values. [2]



(*E*)-4-(4-chlorophenyl)but-3-en-2-one (2d)

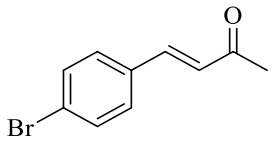
Prepared according to the general procedure from 4-(4-chlorophenyl)-4-(hexylthio)butan-2-one **1d** (59.6 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (22.4 mg, 62%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.49 – 7.42 (m, 3H, Ar-*H*, ArC*H*=CH), 7.39 – 7.33 (m, 2H, Ar-*H*), 6.68 (d, *J* = 16.3 Hz, 1H, C*H*CO), 2.37 (s, 3H, ArCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.2, 142.0, 136.5, 133.0, 129.5 (2C), 129.4(2C), 127.6, 27.8.

HRMS (ESI): *m*/z $[M+H]^+$ calcd for $C_{10}H_9ClO$: 181.0415; found: 181.0415.

This data was concordant with literature values. [2]



(*E*)-4-(4-bromophenyl)but-3-en-2-one (2e)

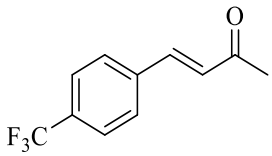
Prepared according to the general procedure from 4-(4-bromophenyl)-4-(hexylthio)butan-2-one **1e** (68.4 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (36.0 mg, 80%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.52 (d, *J* = 8.5 Hz, 2H, Ar-*H*), 7.43 (d, *J* = 16.3 Hz, 1H, ArC*H*=CH), 7.41 – 7.37 (m, 2H, Ar-*H*), 6.69 (d, *J* = 16.3 Hz, 1H, C*H*CO), 2.37 (s, 3H, ArCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.2, 142.1, 133.5, 132.3 (2C), 129.7 (2C), 127.7, 124.9, 27.8.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{10}H_9BrO$: 224.9910; found: 224.9899.

This data was concordant with literature values. [2]



(*E*)-4-(4-(trifluoromethyl)phenyl)but-3-en-2-one (2f)

Prepared according to the general procedure from 4-(hexylthio)-4-(4-(trifluoromethyl)phenyl)butan-2-one **1f** (66.4 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (26.2 mg, 61%).

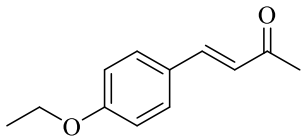
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.69-7.58 (m, 4H, Ar-*H*), 7.52 (d, *J* = 16.3 Hz, 1H, Ar-C*H*=CH), 6.77 (d, *J* = 16.3 Hz, 1H, C*H*CO), 2.40 (s, 3H, COCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.0, 141.4, 138.0, 132.0 (q, *J* = 32.7 Hz), 129.2, 128.4 (2C), 126.0 (2C, q, *J* = 3.8 Hz), 123.9 (d, *J* = 272.1 Hz), 27.9.

^{19}F NMR (471 MHz, Chloroform-*d*) δ = -62.8.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{11}H_9F_3O$: 215.0678; found: 215.0672.

This data was concordant with literature values.[3]



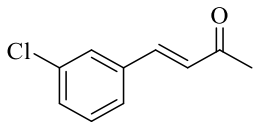
(*E*)-4-(4-ethoxyphenyl)but-3-en-2-one (2g)

Prepared according to the general procedure from 4-(4-ethoxyphenyl)-4-(hexylthio)butan-2-one **1g** (61.6 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a yellow solid (30.9 mg, 81%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.49 – 7.44 (m, 3H, Ar-*H*, ArC*H*=CH), 6.93 – 6.83 (m, 2H, Ar-*H*), 6.59 (d, *J* = 16.2 Hz, 1H, C*H*CO), 4.05 (q, *J* = 7.0 Hz, 2H, CH_2O), 2.35 (s, 3H, CH_3CH$_2$), 1.42 (t, *J* = 7.0 Hz, 3H, COCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.6, 161.1, 143.5, 130.1 (2C), 126.9, 125.0, 115.0 (2C), 63.7, 27.5, 14.8.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{12}H_{14}O_2$: 191.1067; found: 191.1067.



(*E*)-4-(3-chlorophenyl)but-3-en-2-one (2h)

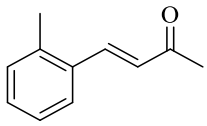
Prepared according to the general procedure from 4-(3-chlorophenyl)-4-(hexylthio)butan-2-one **1h** (59.6 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a colourless oil (31.5 mg, 87%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.5 (t, *J* = 1.9 Hz, 1H, Ar-*H*), 7.5 – 7.4 (m, 2H, Ar-*H*, Ar-C*H*=CH), 7.4 – 7.3 (m, 2H, Ar-*H*), 6.7 (d, *J* = 16.2 Hz, 1H, C*H*CO), 2.4 (s, 3H, COCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.1, 141.7, 136.4, 135.1, 130.4, 130.3, 128.3, 128.1, 126.5, 27.9.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{10}H_9ClO$: 181.0415; found: 181.0411.

This data was concordant with literature values.[3]



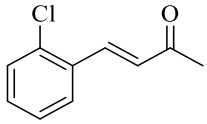
(*E*)-4-(o-tolyl)but-3-en-2-one (2i)

Prepared according to the general procedure from 4-(hexylthio)-4-(o-tolyl)butan-2-one **1i** (55.6 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a colourless oil (29.0 mg, 90%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.8 (d, *J* = 16.1 Hz, 1H, Ar-C*H*=CH), 7.6 (d, *J* = 7.5 Hz, 1H, AR-*H*), 7.3 – 7.3 (m, 1H, AR-*H*), 7.2 (m, 2H, AR-*H*), 6.6 (d, *J* = 16.1 Hz, 1H, C*H*CO), 2.4 (s, 3H, Ar-CH_3), 2.4 (s, 3H, COCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.5, 140.9, 137.9, 133.4, 131.0, 130.3, 128.1, 126.5, 126.5, 27.9, 19.8.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{11}H_{12}O$: 161.0961; found: 161.0962.



(*E*)-4-(2-chlorophenyl)but-3-en-2-one (2j)

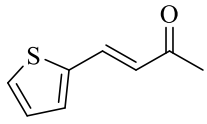
Prepared according to the general procedure from 4-(2-chlorophenyl)-4-(hexylthio)butan-2-one **1j** (59.6 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (34.4 mg, 95%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.9 (d, *J* = 16.4 Hz, 1H, Ar-C*H*=CH), 7.64-7.58 (m, 1H, Ar-*H*), 7.4 – 7.4 (m, 1H, Ar-*H*), 7.3 – 7.2 (m, 2H, Ar-*H*), 6.6 (d, *J* = 16.3 Hz, 1H, C*H*CO), 2.4 (s, 3H, COCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.5, 139.3, 135.2, 132.7, 131.4, 130.3, 129.7, 127.7, 127.3, 27.3.

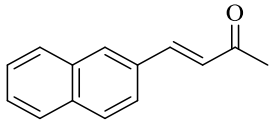
HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{10}H_9ClO$: 181.0415; found: 181.0412.

This data was concordant with literature values.[4]

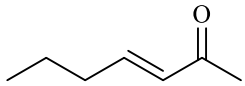


(*E*)-4-(thiophen-2-yl)but-3-en-2-one (2k)

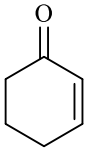
Prepared according to the general procedure from 4-(hexylthio)-4-(thiophen-2-yl)butan-2-one **1k** (54.0 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a yellow oil (29.9 mg, 97%).
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.68-7.58 (m, 1H, Ar-C*H*=CH), 7.44-7.37 (m, 1H, Ar-*H*), 7.33 – 7.26 (m, 1H, Ar-*H*), 7.11-7.02 (m, 1H, Ar-*H*), 6.53 (d, *J* = 15.9 Hz, 1H, C*H*CO), 2.34 (s, 3H, COCH_3).
^{13}C NMR (126 MHz, Chloroform-*d*) δ = 197.9, 139.9, 135.9, 131.7, 129.1, 128.4, 125.9, 27.8.
HRMS (ESI): m/z $[M+H]^+$ calcd for C_8H_8OS: 153.0369; found: 153.0357.
This data was concordant with literature values.[4]



(*E*)-4-(naphthalen-2-yl)but-3-en-2-one (2l)
Prepared according to the general procedure from 4-(hexylthio)-4-(naphthalen-2-yl)butan-2-one **1l** (62.8 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (35.6 mg, 91%).
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.97 – 7.91 (m, 1H, Ar-*H*), 7.87 – 7.80 (m, 3H, Ar-*H*), 7.70 – 7.63 (m, 2H, Ar-*H*), 7.55 – 7.49 (m, 2H, Ar-C*H*=CH, Ar-*H*), 6.82 (d, *J* = 16.3 Hz, 1H, C*H*CO), 2.42 (s, 3H, COCH_3).
^{13}C NMR (126 MHz, Chloroform-*d*) δ = 198.5, 143.6, 134.4, 133.4, 132.0, 130.5, 128.9, 128.7, 127.9, 127.5, 127.3, 126.9, 123.6, 27.7.
HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{14}H_{12}O$: 197.0961; found: 197.0959.
This data was concordant with literature values.[5]



(E)-hept-3-en-2-one (2m)
Prepared according to the general procedure from 4-(hexylthio)heptan-2-one **1m** (92.0 mg, 0.4 mmol) and NBS (71.2 mg, 0.4 mmol), The product was isolated as a pale yellow liquid (22.0 mg, 49%).
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 6.73 (ddd, *J* = 16.0, 7.6, 6.3 Hz, 1H), 5.99 (dt, *J* = 16.0, 1.5 Hz, 1H), 2.17 (d, *J* = 1.2 Hz, 3H), 2.13 (qd, *J* = 7.0, 1.5 Hz, 2H), 1.43 (h, *J* = 7.4 Hz, 2H), 0.87 (t, *J* = 7.4 Hz, 3H).
^{113}C NMR (126 MHz, Chloroform-*d*) δ = 198.9, 148.5, 131.5, 34.5, 26.8, 21.4, 13.7.
HRMS (ESI): m/z $[M+H]^+$ calcd for $C_7H_{12}O$: 113.0961; found: 113.0961.
This data was concordant with literature values.[6]



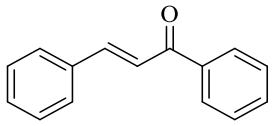
cyclohex-2-en-1-one (2n)
Prepared according to the general procedure from 3-(hexylthio)cyclohexan-1-one **1n** (85.6 mg, 0.4 mmol) and NBS (71.2 mg, 0.4 mmol), The product was isolated as a pale yellow liquid (16.2 mg, 42%).
^{1}H NMR (500 MHz, Chloroform-*d*) δ 6.92 (dt, *J* = 10.4, 4.1 Hz, 1H), 5.92 (dt, *J* = 10.2, 2.1 Hz, 1H),

2.37 – 2.30 (t, *J* = 7.0 Hz, 2H), 2.27 (m,, 2H), 1.93 (p, *J* = 6.3 Hz, 2H).

^{13}C NMR (126 MHz, Chloroform-*d*) δ 199.8, 150.9, 129.9, 38.1, 25.7, 22.8.

HRMS (ESI): m/z $[M+H]^+$ calcd for C_6H_8O: 97.0648; found: 97.0649.

This data was concordant with literature values.[7]



(*E*)-chalcone (2o)

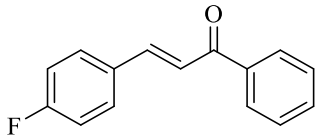
Prepared according to the general procedure from 3-(hexylthio)-1,3-diphenylpropan-1-one **1o** (65.2 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol), The product was isolated as a white solid (36.0 mg, 86%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 8.07 – 8.01 (m, 2H, Ar-C*H*=CH,), 7.83 (d, *J* = 15.7 Hz, 1H, Ar-*H*), 7.65 (ddd, *J* = 5.2, 3.1, 1.9 Hz, 2H, Ar-*H*), 7.62 – 7.47 (m, 4H, Ar-*H*), 7.46 – 7.38 (m, 3H, Ar-*H* , C*H*CO).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 190.7, 145.0, 138.3, 135.0, 132.9, 130.7, 129.1 (2C), 128.7 (2C), 128.6 (2C), 128.6 (2C), 122.2.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{12}O$: 209.0961; found: 209.0958.

This data was concordant with literature values.[8]



(*E*)-3-(4-fluorophenyl)-1-phenylprop-2-en-1-one (2p)

Prepared according to the general procedure from 3-(4-fluorophenyl)-3-(hexylthio)-1-phenylpropan-1-one **1p** (68.8 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (41.8 mg, 92%).

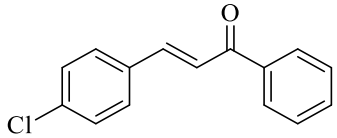
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 8.06 – 7.99 (m, 2H, Ar-*H*), 7.78 (d, *J* = 15.7 Hz, 1H, Ar-C*H*=CH), 7.67 – 7.56 (m, 3H, Ar-*H*), 7.54 – 7.44 (m, 3H, Ar-*H*, C*H*CO), 7.15 – 7.06 (m, 2H, Ar-*H*).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 190.4, 164.2 (d, *J* = 251.8 Hz), 163.2, 143.6, 138.2, 133.0, 131.2, 131.2, 130.5, 130.4, 128.8, 128.6, 121.8, 116.3, 116.2.

^{19}F NMR (471 MHz, Chloroform-*d*) δ = -108.9.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{11}FO$: 227.0867; found: 227.0860.

This data was concordant with literature values.[9]



(*E*)-3-(4-chlorophenyl)-1-phenylprop-2-en-1-one (2q)

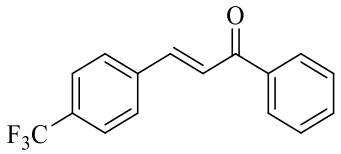
Prepared according to the general procedure from 3-(4-chlorophenyl)-3-(hexylthio)-1-phenylpropan-1-one **1q** (72.0 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (40.8 mg, 84%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 8.06 – 7.98 (m, 2H, Ar-*H*), 7.75 (d, *J* = 15.7 Hz, 1H, Ar-C*H*=CH), 7.63 – 7.55 (m, 3H, Ar-*H*), 7.54 – 7.48 (m, 3H, Ar-*H*, C*H*CO), 7.42 – 7.36 (m, 2H, Ar-*H*).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 190.3, 143.4, 138.1, 136.5, 133.5, 133.1, 129.7 (2C), 129.4 (2C), 128.8 (2C), 128.6 (2C), 122.5.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{11}ClO$: 243.0571; found: 243.0570.

This data was concordant with literature values.[8]



(*E*)-1-phenyl-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one (2r)

Prepared according to the general procedure from 3-(hexylthio)-1-phenyl-3-(4-(trifluoromethyl) phenyl) propan-1-one **1r** (78.8 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (29.9 mg, 54%).

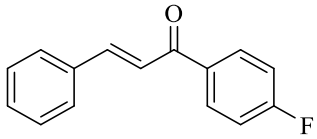
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 8.08-7.97 (m, 2H, Ar-*H*), 7.81 (d, *J* = 15.8 Hz, 1H, Ar-C*H*=CH), 7.74 (d, *J* = 8.1 Hz, 2H, Ar-*H*), 7.67 (d, *J* = 8.2 Hz, 2H, Ar-*H*), 7.64 – 7.56 (m, 2H, Ar-*H*, C*H*CO), 7.52 (t, *J* = 7.6 Hz, 2H, Ar-*H*).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 190.1, 142.8, 138.4, 137.9, 133.3, 132.2 – 131.5 (m), 128.9 (2C), 128.7 (2C), 128.6 (2C), 126.0, 125.0, 124.3, 123.9 (d, *J* = 272.1 Hz).

^{19}F NMR (471 MHz, Chloroform-*d*) δ = -62.7.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{16}H_{11}F_3O$: 277.0835; found: 277.0838.

This data was concordant with literature values.[9]



(*E*)-1-(4-fluorophenyl)-3-phenylprop-2-en-1-one (2s)

Prepared according to the general procedure from 1-(4-fluorophenyl)-3-(hexylthio)-3-phenylpropan-1-one **1s** (68.8 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (41.8 mg, 92%).

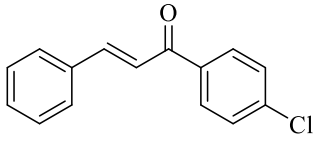
^{1}H NMR (500 MHz, Chloroform-*d*) δ = 8.10 – 8.04 (m, 2H, Ar-*H*), 7.82 (d, *J* = 15.7 Hz, 1H, Ar-C*H*=CH), 7.68 – 7.61 (m, 2H, Ar-*H*), 7.51 (d, *J* = 15.7 Hz, 1H, C*H*CO), 7.46 – 7.39 (m, 3H, Ar-*H*), 7.22 – 7.15 (m, 2H, Ar-*H*).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 188.9, 165.7 (d, *J* = 254.6 Hz), 145.2, 134.9, 134.6, 131.2 (2C, d, *J* = 9.2 Hz), 130.8, 129.1 (2C), 128.6 (2C), 121.6, 115.9, 115.8.

^{19}F NMR (471 MHz, Chloroform-*d*) δ = -105.4.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{11}FO$: 227.0867; found: 227.0869.

This data was concordant with literature values.[8]



(*E*)-1-(4-chlorophenyl)-3-phenylprop-2-en-1-one (2t)

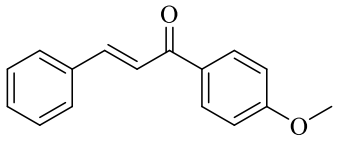
Prepared according to the general procedure from 1-(4-chlorophenyl)-3-(hexylthio)-3-phenylpropan-1-one **1t** (72.0 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (39.4 mg, 81%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.99 – 7.94 (m, 2H, Ar-*H*), 7.82 (d, *J* = 15.6 Hz, 1H, Ar-C*H*=CH), 7.68 – 7.61 (m, 2H, Ar-*H*), 7.53 – 7.46 (m, 3H, Ar-*H*, C*H*CO), 7.45 – 7.40 (m, 3H, Ar-*H*).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 189.3, 145.5, 139.3, 136.6, 134.8, 130.0, 130.0 (2C), 129.1 (2C), 129.1 (2C), 128.6 (2C), 121.5.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{15}H_{11}ClO$: 243.0571; found: 243.0572.

This data was concordant with literature values.[8]



(*E*)-1-(4-methoxyphenyl)-3-phenylprop-2-en-1-one (2u)

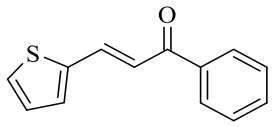
Prepared according to the general procedure from 3-(hexylthio)-1-(4-methoxyphenyl)-3-phenylpropan-1-one **1u** (71.2 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a white solid (39.2 mg, 82%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 8.06 – 7.97 (m, 2H, Ar-*H*), 7.79 (d, *J* = 15.6 Hz, 1H, Ar-C*H*=CH), 7.62 – 7.55 (m, 3H, Ar-*H*), 7.53 – 7.48 (m, 2H, Ar-*H*), 7.42 (d, *J* = 15.7 Hz, 1H, C*H*CO), 7.00 – 6.86 (m, 2H, Ar-*H*), 3.85 (s, 3H, OCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 190.7, 161.8, 144.8, 138.6, 132.7, 130.4 (2C), 128.7 (2C), 128.5 (2C), 127.7, 119.9, 114.5 (2C), 55.5.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{16}H_{14}O_2$: 239.1067; found: 239.1073.

This data was concordant with literature values.[8]



(*E*)-1-phenyl-3-(thiophen-2-yl)prop-2-en-1-one (2v)

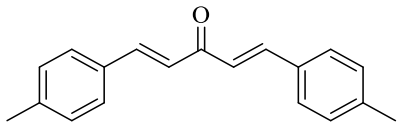
Prepared according to the general procedure from 3-(hexylthio)-1-phenyl-3-(thiophen-2-yl)propan-1-one **1v** (66.4 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a brown solid (42.1 mg, 98%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 8.04 – 7.98 (m, 2H, Ar-*H*), 7.98-7.91 (m, 1H, Ar-C*H*=CH), 7.62 – 7.55 (m, 1H, Ar-*H*), 7.54 – 7.46 (m, 2H, Ar-*H*), 7.44-7.39 (m, 1H, COC*H*), 7.38 – 7.30 (m, 2H, Ar-*H*), 7.12-7.06 (m, 1H, Ar-*H*).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 190.0, 140.5, 138.2, 137.3, 132.9, 132.2, 128.9, 128.7 (2C), 128.5 (2C), 128.5, 120.8.

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{10}OS$: 215.0525; found: 215.0515.

This data was concordant with literature values.[8]



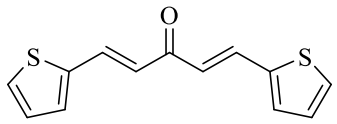
(1*E*,4*E*)-1,5-di-p-tolylpenta-1,4-dien-3-one (4a)

Prepared according to the general procedure from (E)-5-(hexylthio)-1,5-di-p-tolylpent-1-en-3-one **3a** (76.0 mg, 0.2 mmol) and NBS (35.6mg, 0.2 mmol). The product was isolated as a yellow solid (45.8 mg, 88%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.72 (d, *J* = 15.9 Hz, 2H, Ar-C*H*=CH), 7.52 (d, *J* = 8.1 Hz, 4H, Ar-*H*), 7.22 (d, *J* = 7.9 Hz, 4H, Ar-*H*), 7.05 (d, *J* = 15.9 Hz, 2H, Ar-C*H*=CH), 2.39 (s, 6H, ArCH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 189.2, 143.3 (2C), 141.1 (2C), 132.2 (2C), 129.8 (4C), 128.5 (4C), 124.7 (2C), 21.6 (2C).

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{19}H_{18}O$: 263.1430; found: 263.1435.



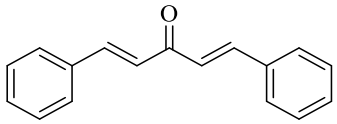
(1*E*,4*E*)-1,5-di(thiophen-2-yl)penta-1,4-dien-3-one (4b)

Prepared according to the general procedure from (E)-5-(hexylthio)-1,5-di(thiophen-2-yl)pent-1-en-3-one **3b** (72.8 mg, 0.2 mmol) and NBS (35.6 mg, 0.2 mmol). The product was isolated as a yellow solid (45.9 mg, 93%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.90-7.75 (m, 2H, Ar-C*H*=CH), 7.45-7.25 (m, 4H, Ar-*H*), 7.13 – 7.01 (m, 2H, Ar-*H*), 6.86 – 6.72 (m, 2H, C*H*CO).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 187.8, 140.4 (2C), 135.7 (2C), 132.0 (2C), 128.9 (2C), 128.4 (2C), 124.5 (2C).

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{13}H_{10}OS_2$: 247.0246; found: 247.0248.



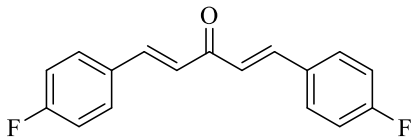
(1*E*,4*E*)-1,5-diphenylpenta-1,4-dien-3-one (4c)

Prepared according to the general procedure from 1,5-bis(hexylthio)-1,5-diphenylpentan-3-one **3c** (94.0 mg, 0.2 mmol) and NBS (71.2 mg, 0.4 mmol). The product was isolated as a white solid (43.7 mg, 93%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.75 (d, *J* = 15.9 Hz, 2H, Ar-C*H*=CH), 7.66 – 7.57 (m, 4H, Ar-*H*), 7.46-7.37 (m, 6H, Ar-*H*), 7.09 (d, *J* = 15.9 Hz, 2H, C*H*CO).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 189.0, 143.4 (2C), 134.8 (2C), 130.6 (2C), 129.0 (4C), 128.5 (4C), 125.5 (2C).

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{17}H_{14}O$: 235.1117; found: 235.1119.



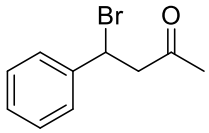
(1*E*,4*E*)-1,5-bis(4-fluorophenyl)penta-1,4-dien-3-one (4d)

Prepared according to the general procedure from 1,5-bis(4-fluorophenyl)-1,5-bis(hexylthio)pentan-3-one **3d** (101.3 mg, 0.2 mmol) and NBS (71.2 mg, 0.4 mmol). The product was isolated as a yellow solid (39.6 mg, 73%).

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.69 (d, *J* = 15.9 Hz, 2H, Ar-C*H*=CH), 7.64 – 7.53 (m, 4H, Ar-*H*), 7.09 (t, *J* = 8.6 Hz, 4H, Ar-*H*), 6.98 (d, *J* = 15.9 Hz, 2H, C*H*CO).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 188.5, 164.1 (2C, d, *J* = 252.0 Hz),142.1 (2C), 131.0 (2C, d, *J* = 3.2 Hz), 130.4 (4C, d, *J* = 8.5 Hz), 125.1 (2C), 116.2 (4C, d, *J* = 21.9 Hz)

HRMS (ESI): m/z $[M+H]^+$ calcd for $C_{17}H_{12}F_2O$: 271.0929; found: 271.0925.



4-bromo-4-phenylbutan-2-one (A)

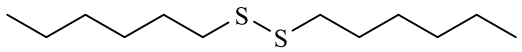
Isolated from products in Scheme **6c6**

^{1}H NMR (500 MHz, Chloroform-*d*) δ = 7.43 – 7.37 (m, 2H, Ar-*H*), 7.37 – 7.29 (m, 2H, Ar-*H*), 7.31 – 7.23 (m, 1H, Ar-*H*), 5.45 – 5.39 (m, 1H, C*H*Br), 3.53 (dd, *J* = 17.3, 8.7 Hz, 1H, CH_2CO), 3.25 (dd, *J* = 17.3, 5.7 Hz, 1H, CH_2CO), 2.17 (s, 3H, CH_3).

^{13}C NMR (126 MHz, Chloroform-*d*) δ = 204.4, 141.2, 129.0 (2C), 128.8, 127.3 (2C), 52.8, 47.3, 30.8.

HRMS (ESI): m/z $[M+Na]^+$ calcd for $C_{10}H_{11}BrO$: 248.9885; found: 248.9888.

This data was concordant with literature values.[10]



1,2-dihexyldisulfane (B)

Isolated from products in Scheme **6c**

^{1}H NMR (400 MHz, Chloroform-*d*) δ = 2.68 (t, *J* = 7.5 Hz, 4H), 1.67 (tt, *J* = 7.6, 6.4 Hz, 4H), 1.48 – 1.17 (m, 12H), 0.89 (t, *J* = 6.8 Hz, 6H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ = 39.2, 31.5, 29.2, 28.2, 22.6, 14.1.

This data was concordant with literature values.[11–13]

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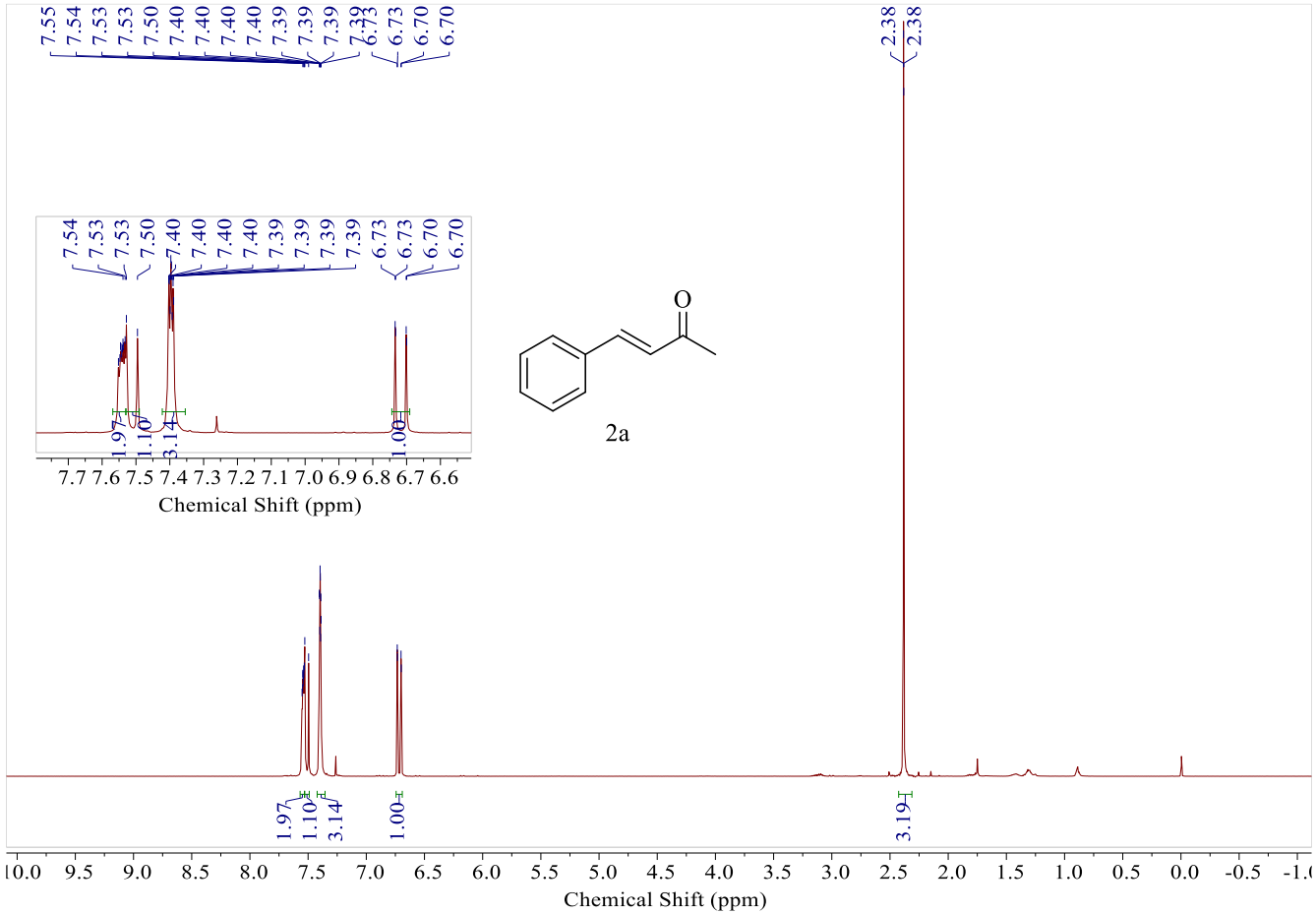
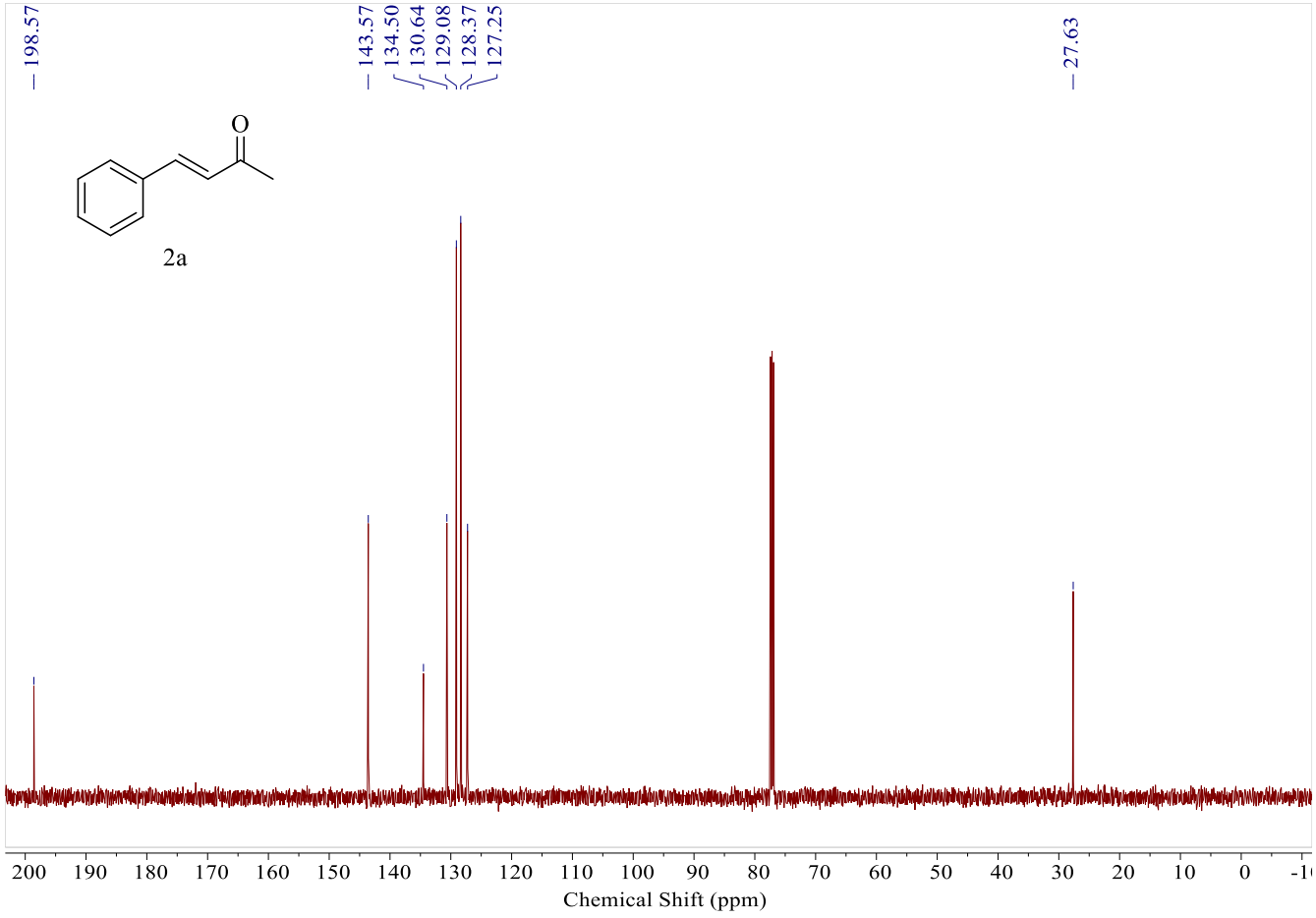
NMR Spectra of Compounds

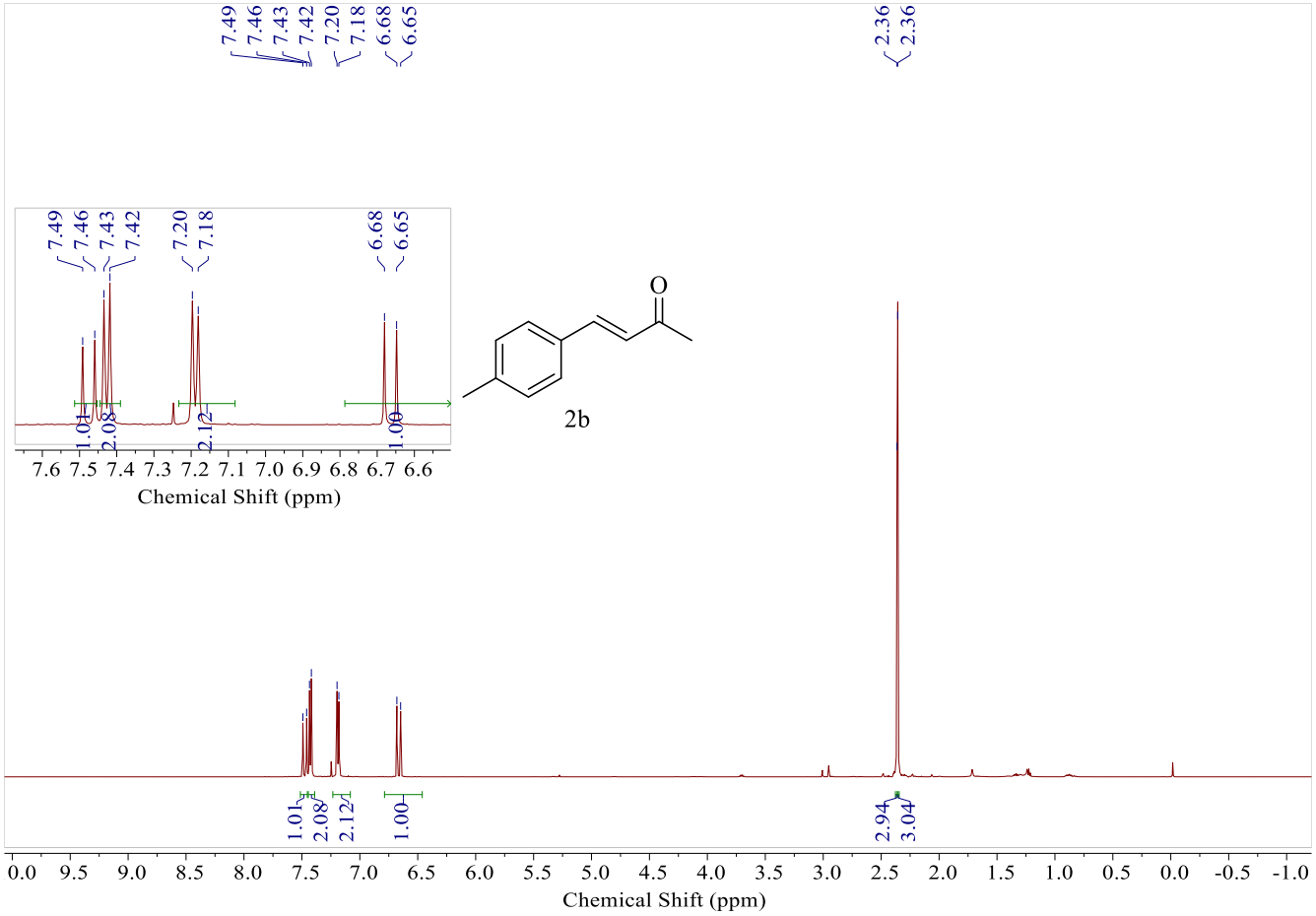
^{1}H NMR of compound **2a**



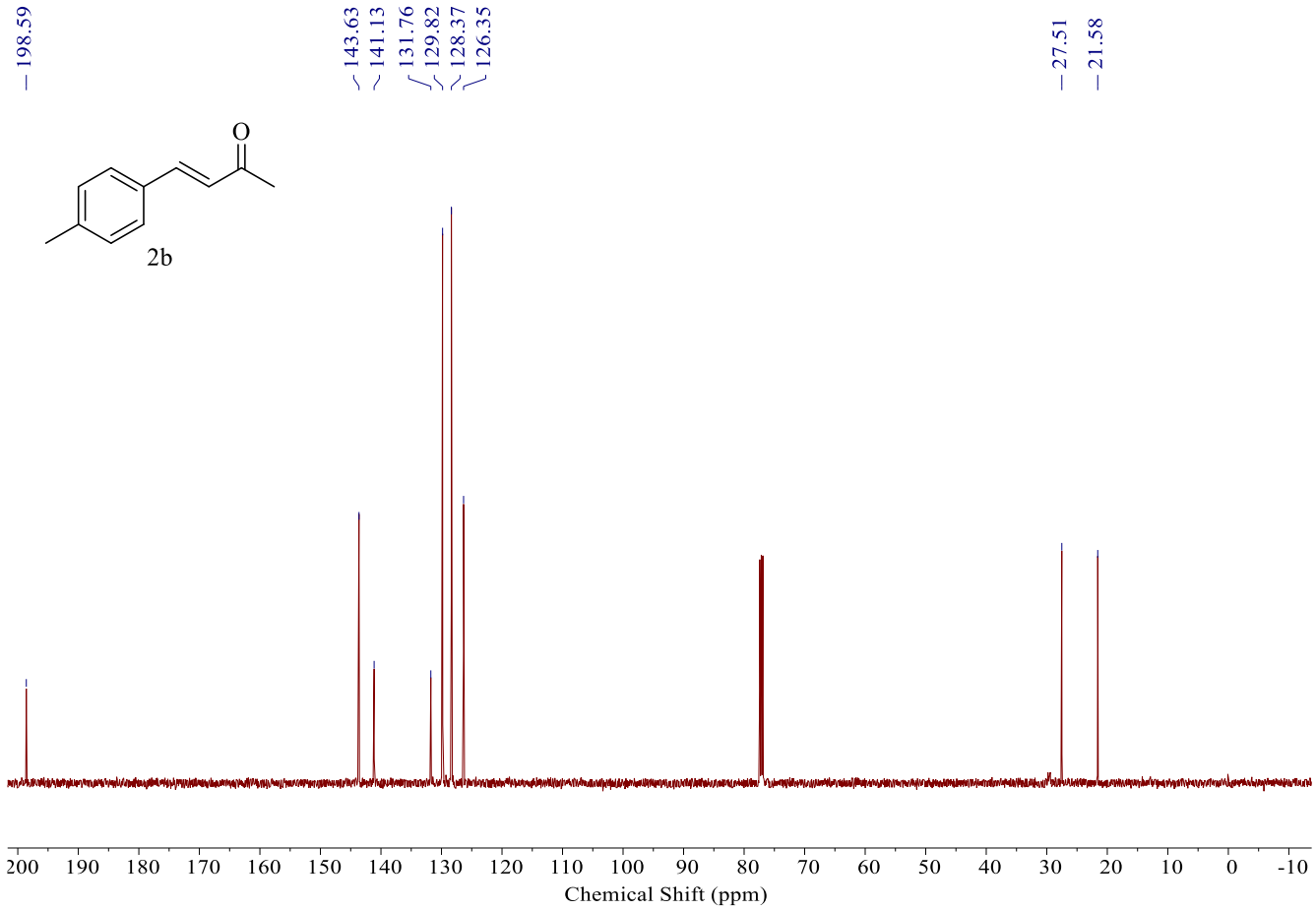
^{13}C NMR of compound **2a**



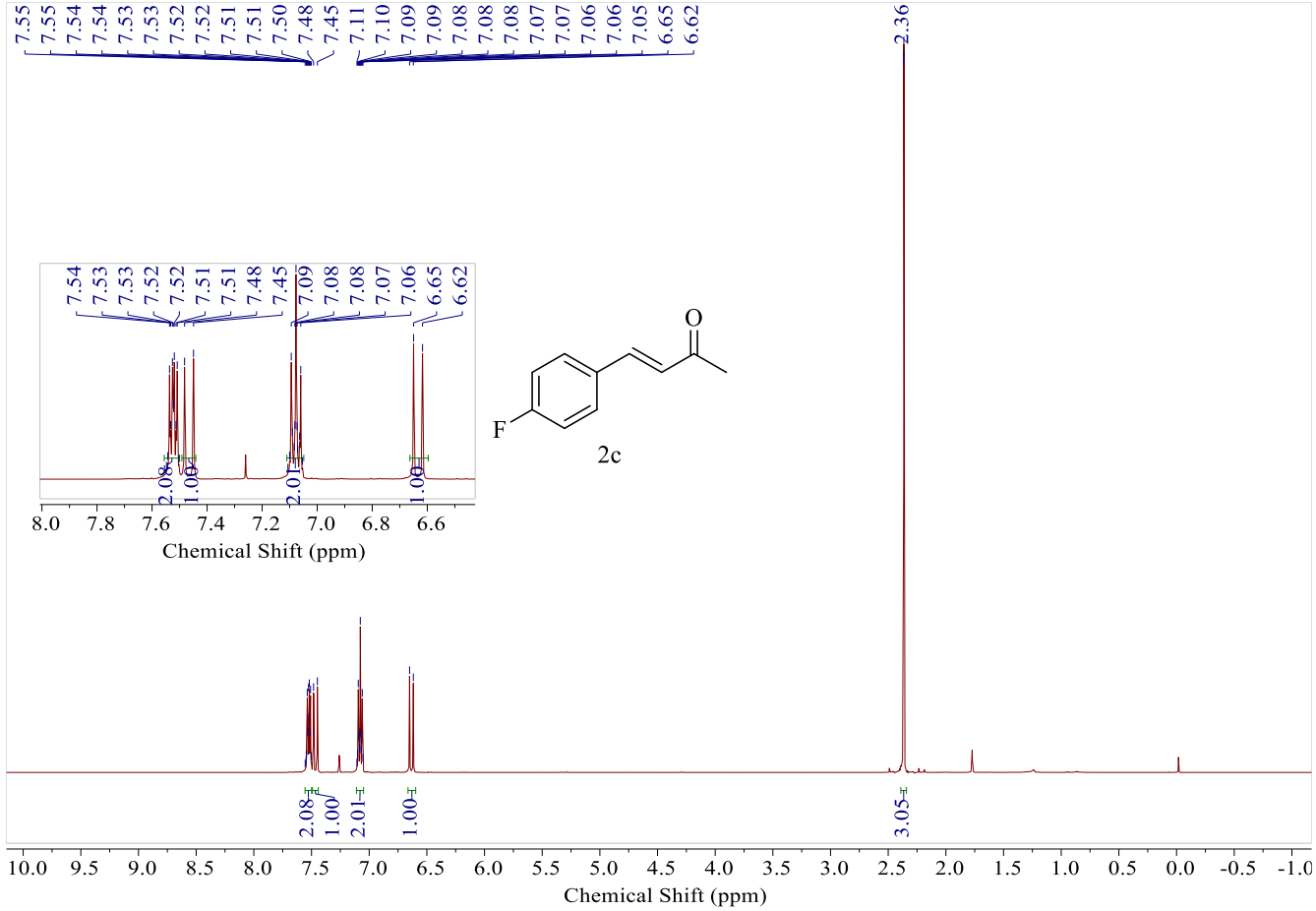
^{1}H NMR of compound **2b**



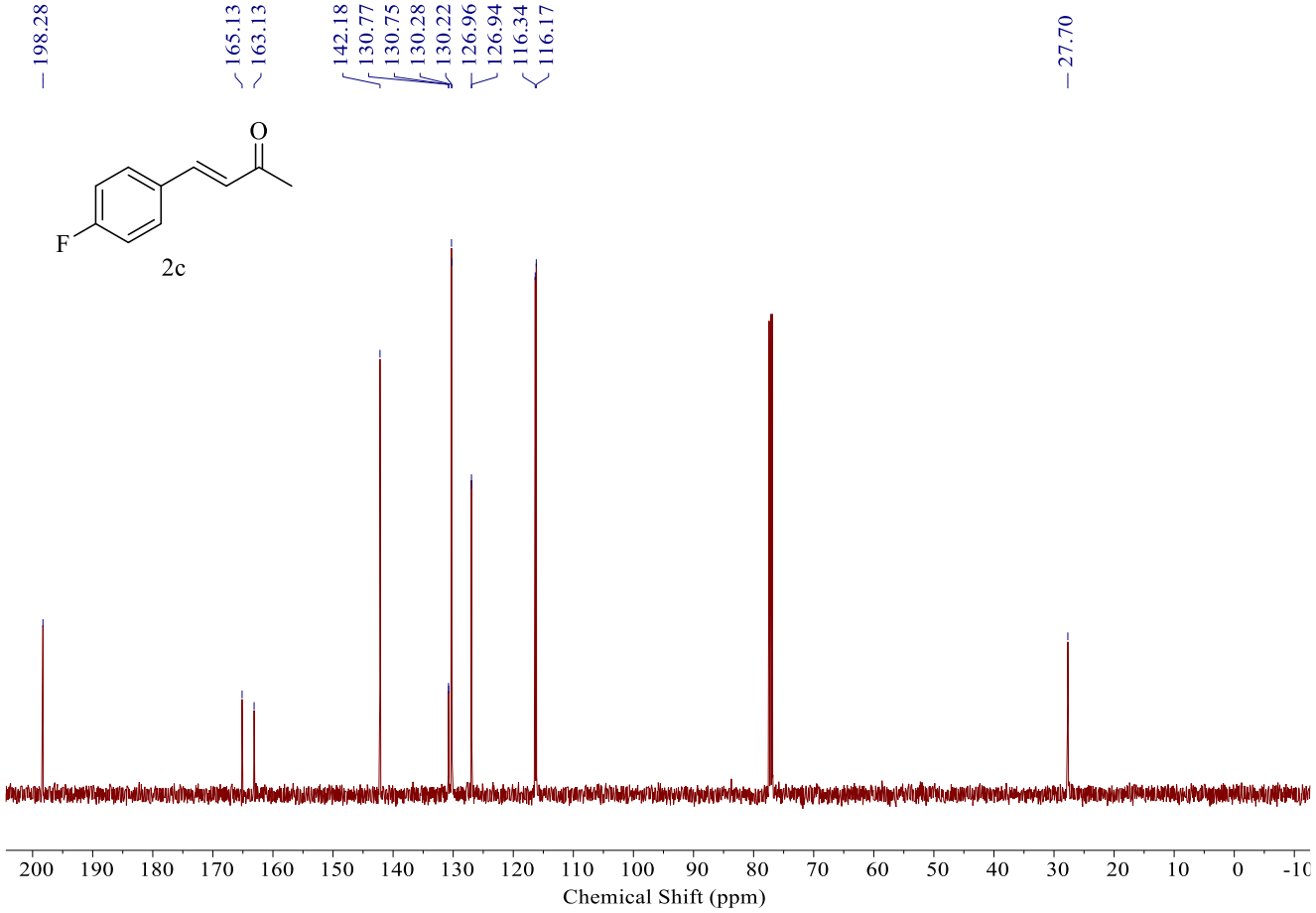
^{13}C NMR of compound **2b**



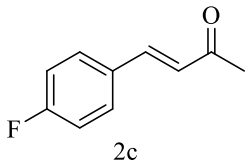
[1]H NMR of compound **2c**



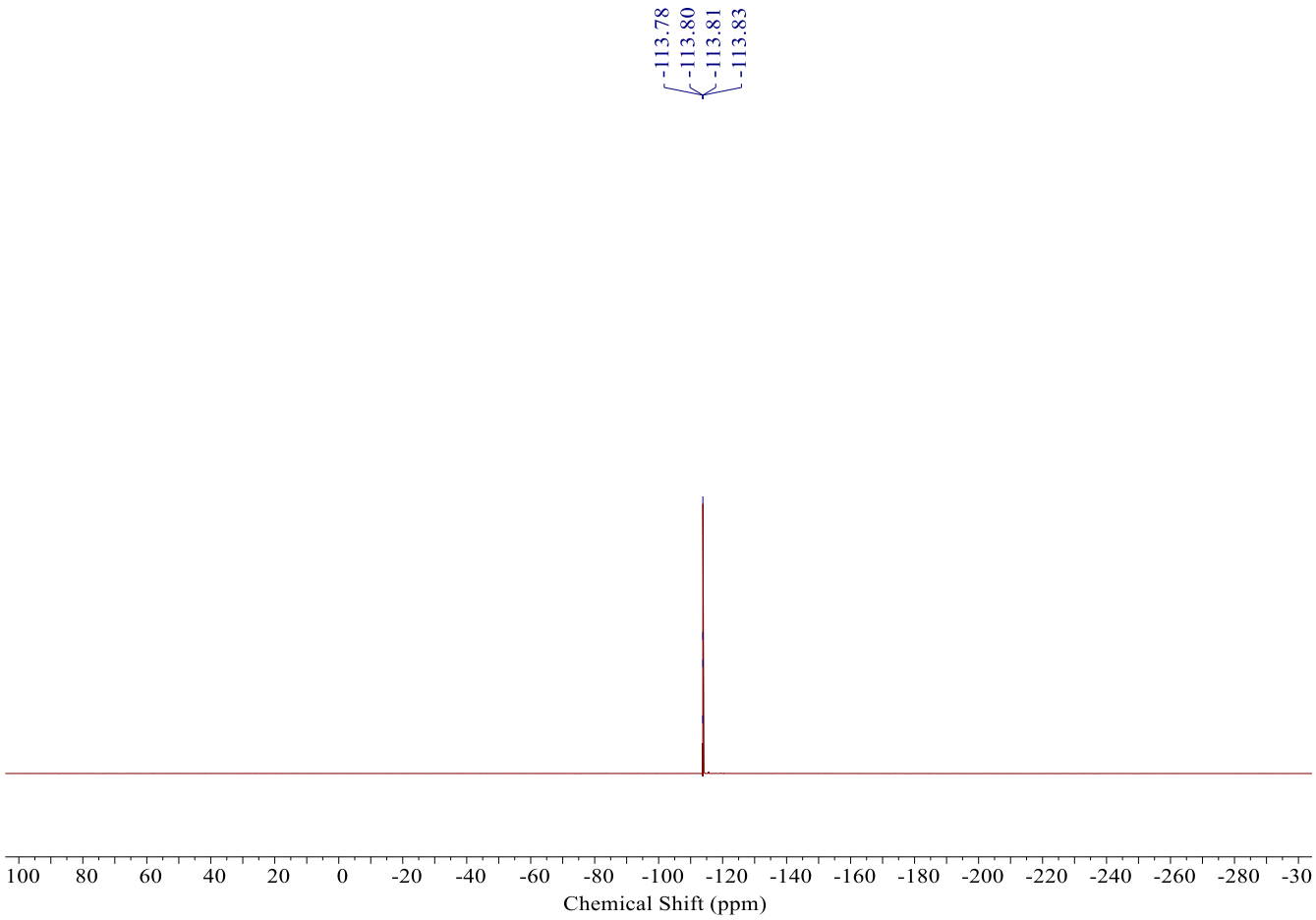
^{13}C NMR of compound **2c**



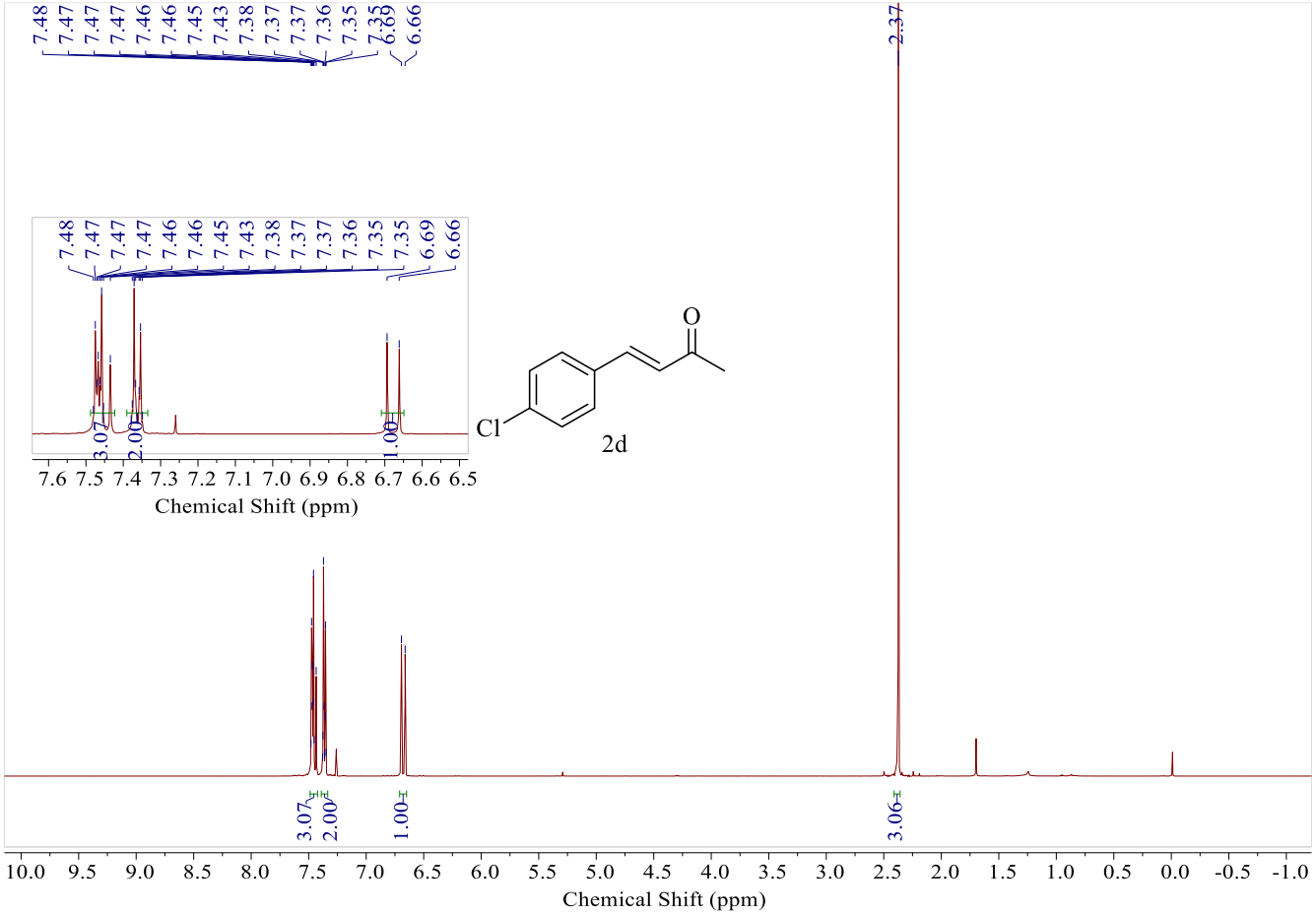
^{19}F NMR of compound **2c**



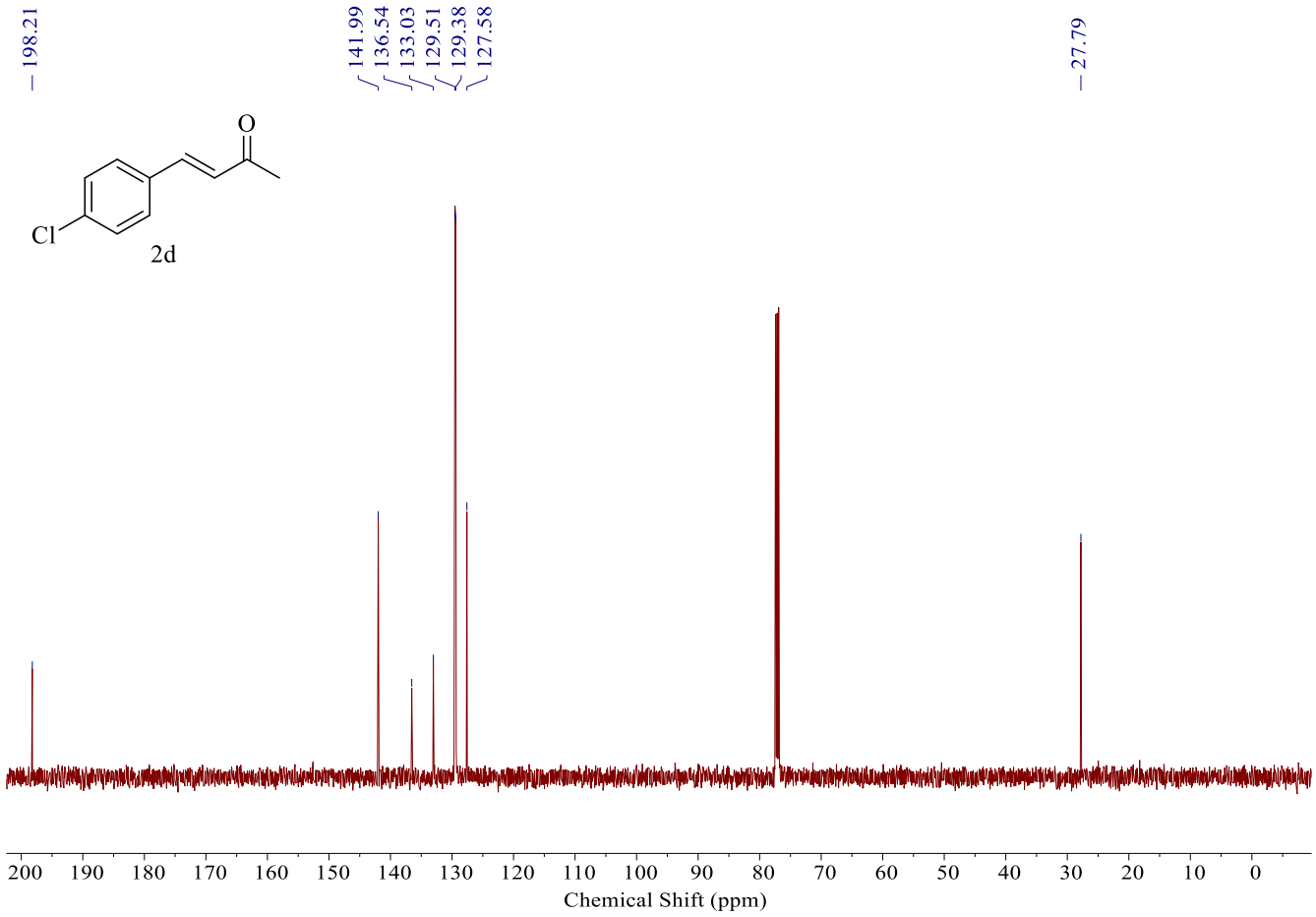
2c



[1]H NMR of compound **2d**



^{13}C NMR of compound **2d**



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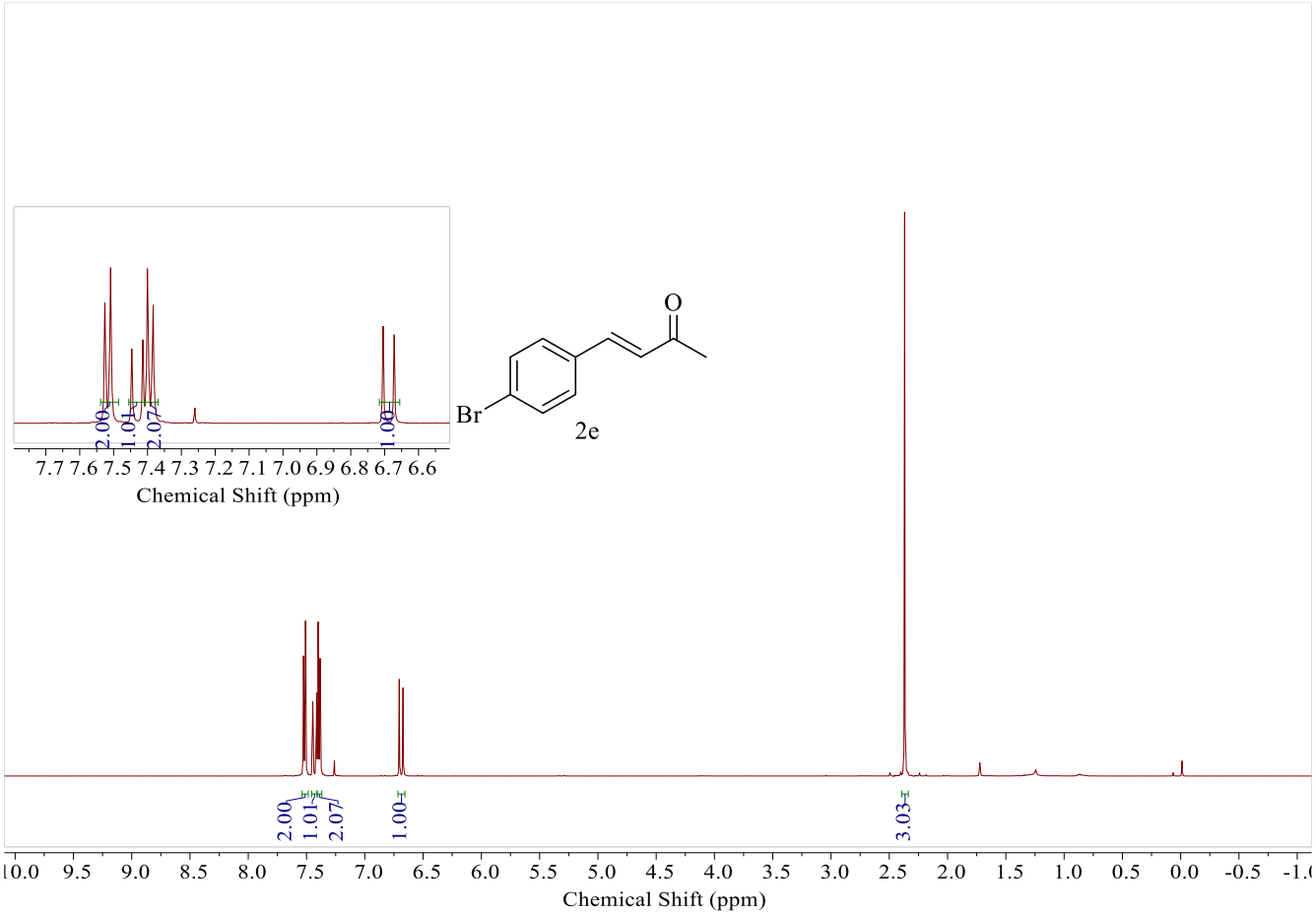
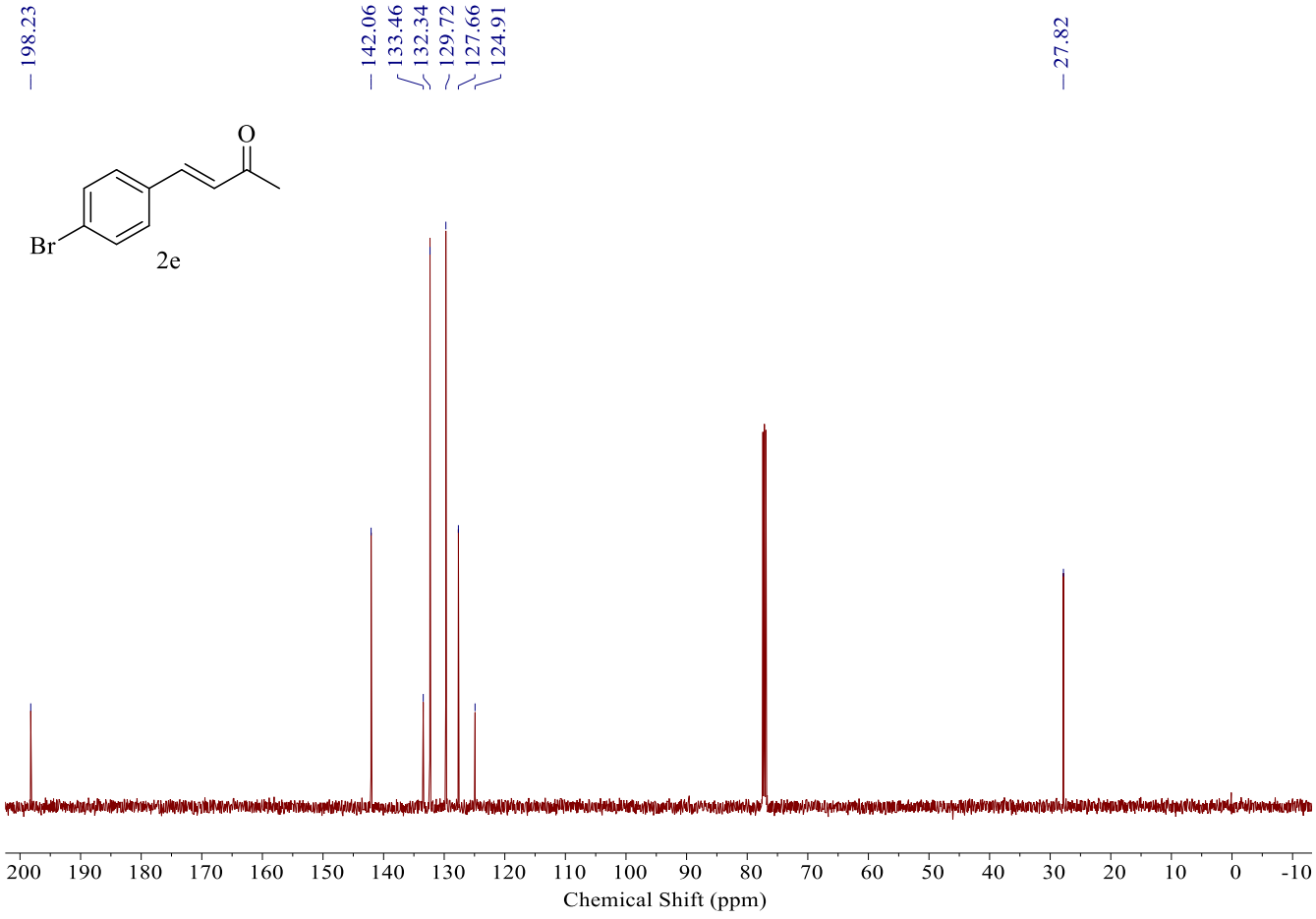
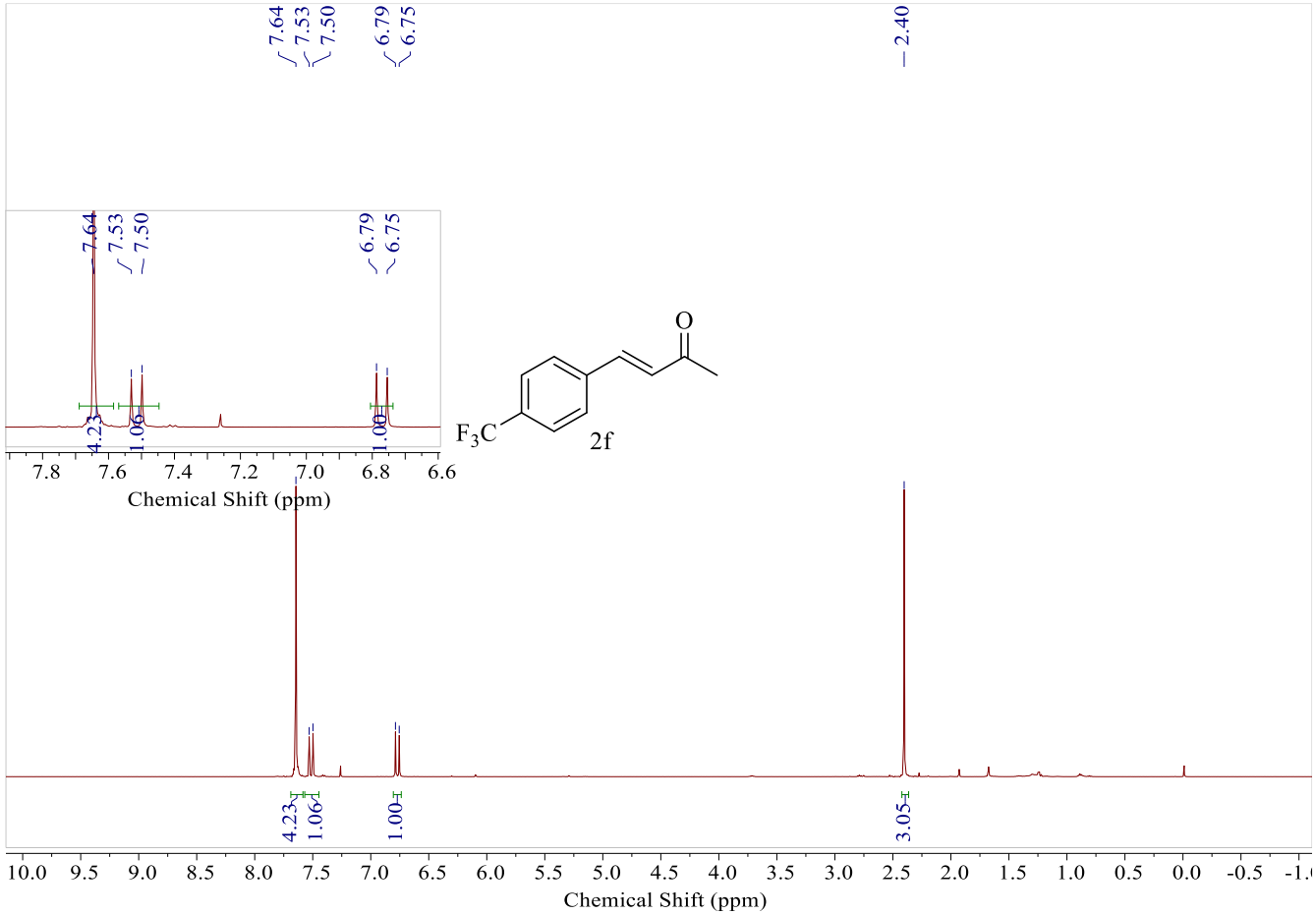


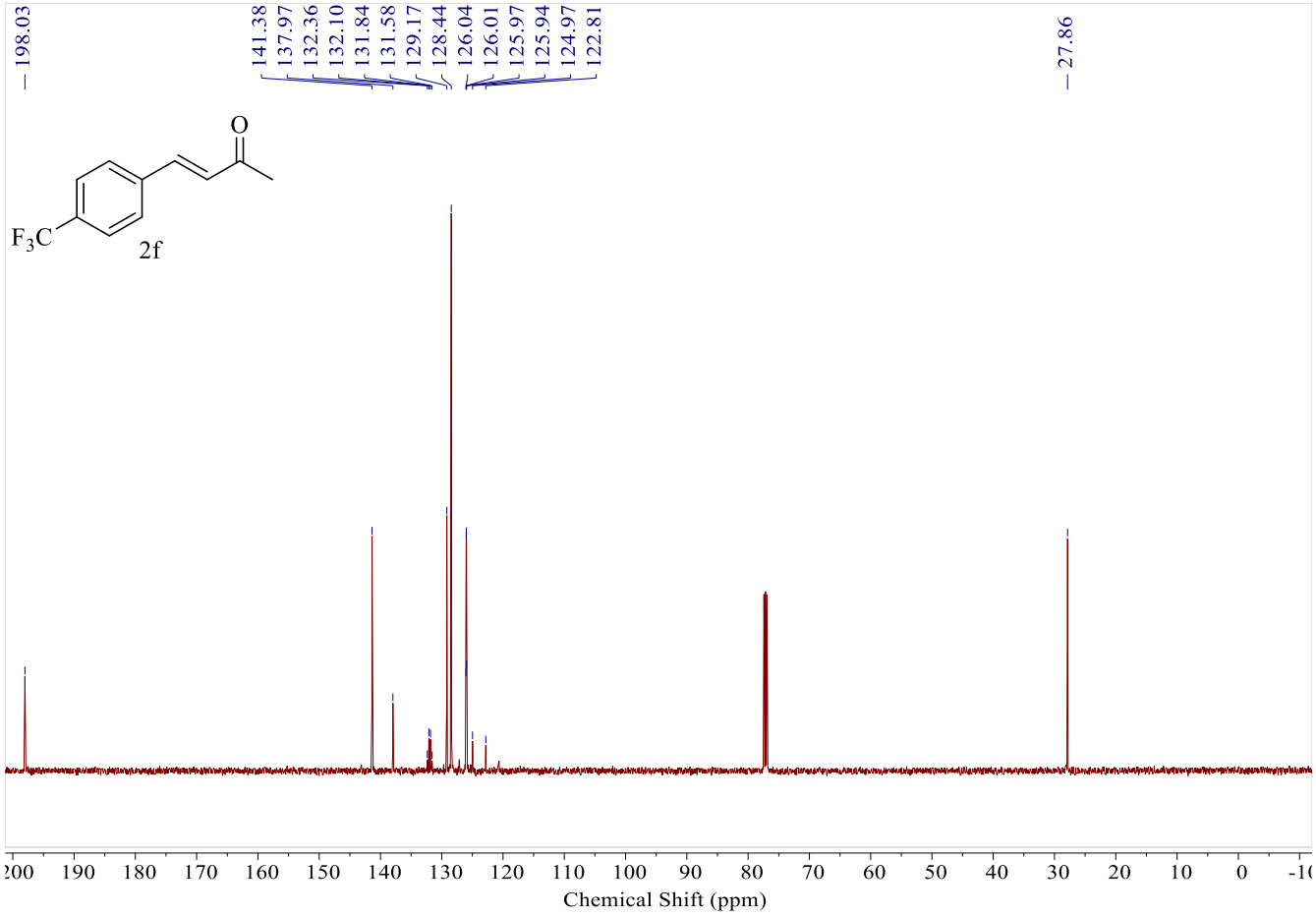
^{13}C NMR of compound **2e**



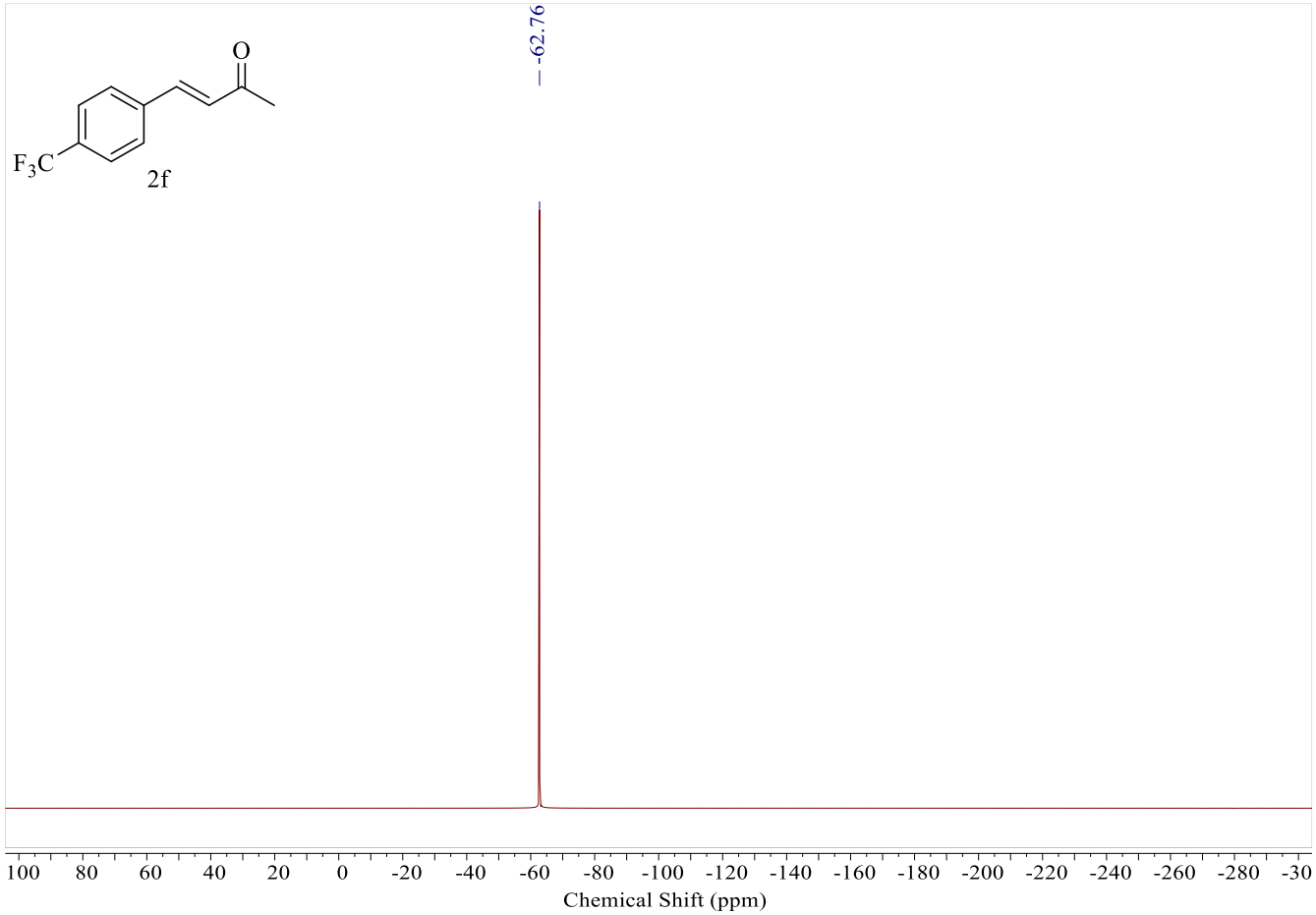
^{1}H NMR of compound **2f**



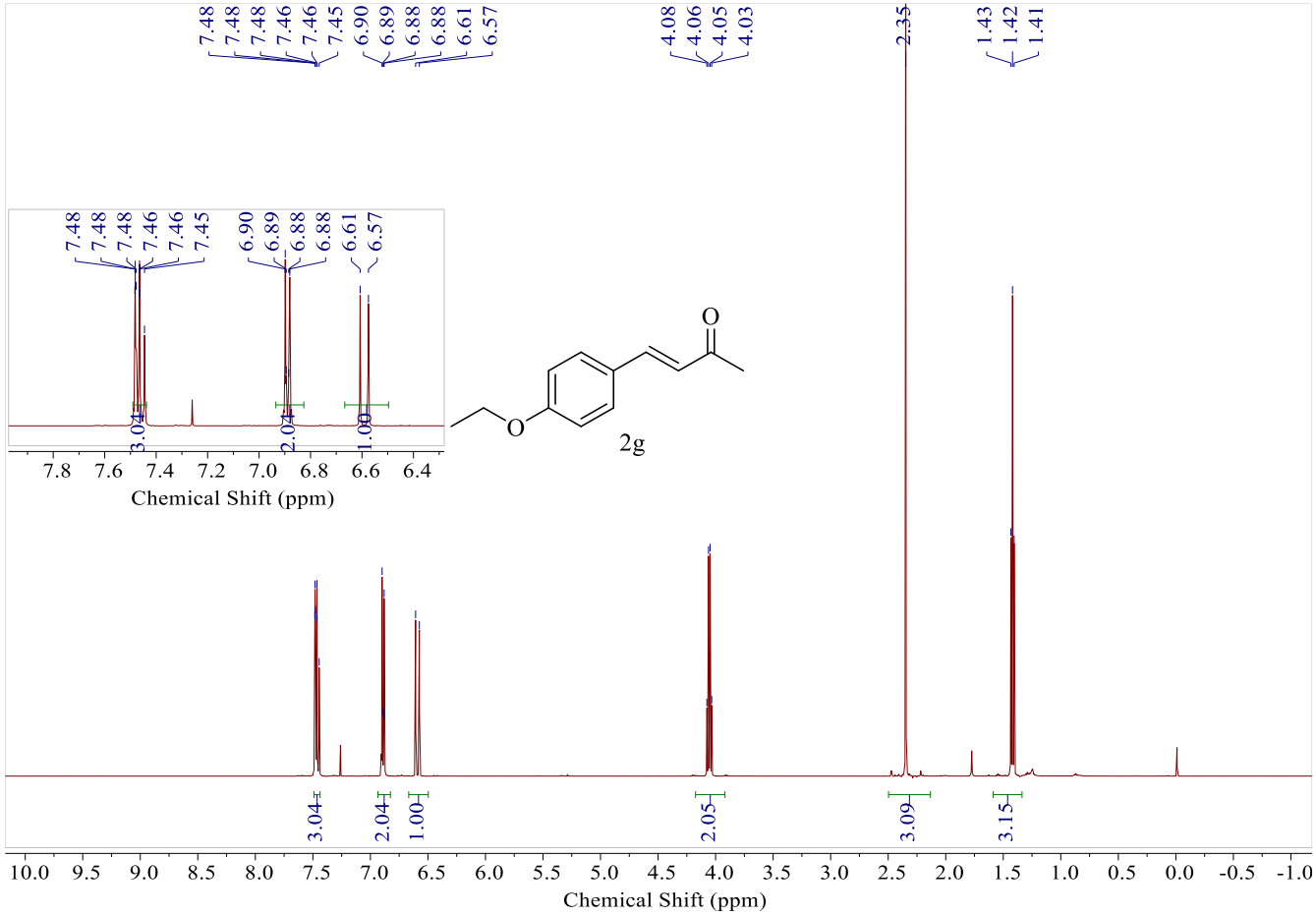
^{13}C NMR of compound **2f**



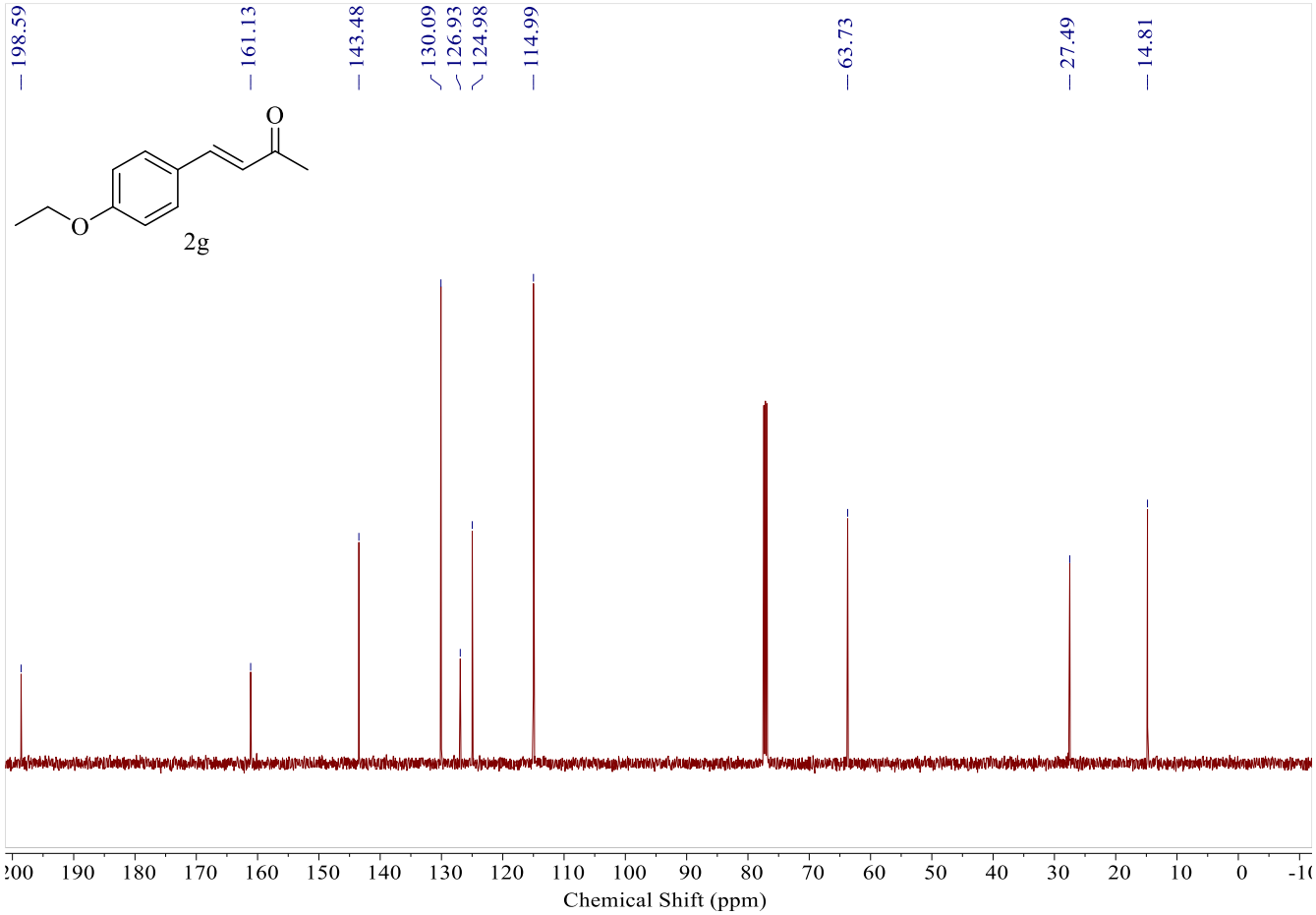
^{19}F NMR of compound **2f**



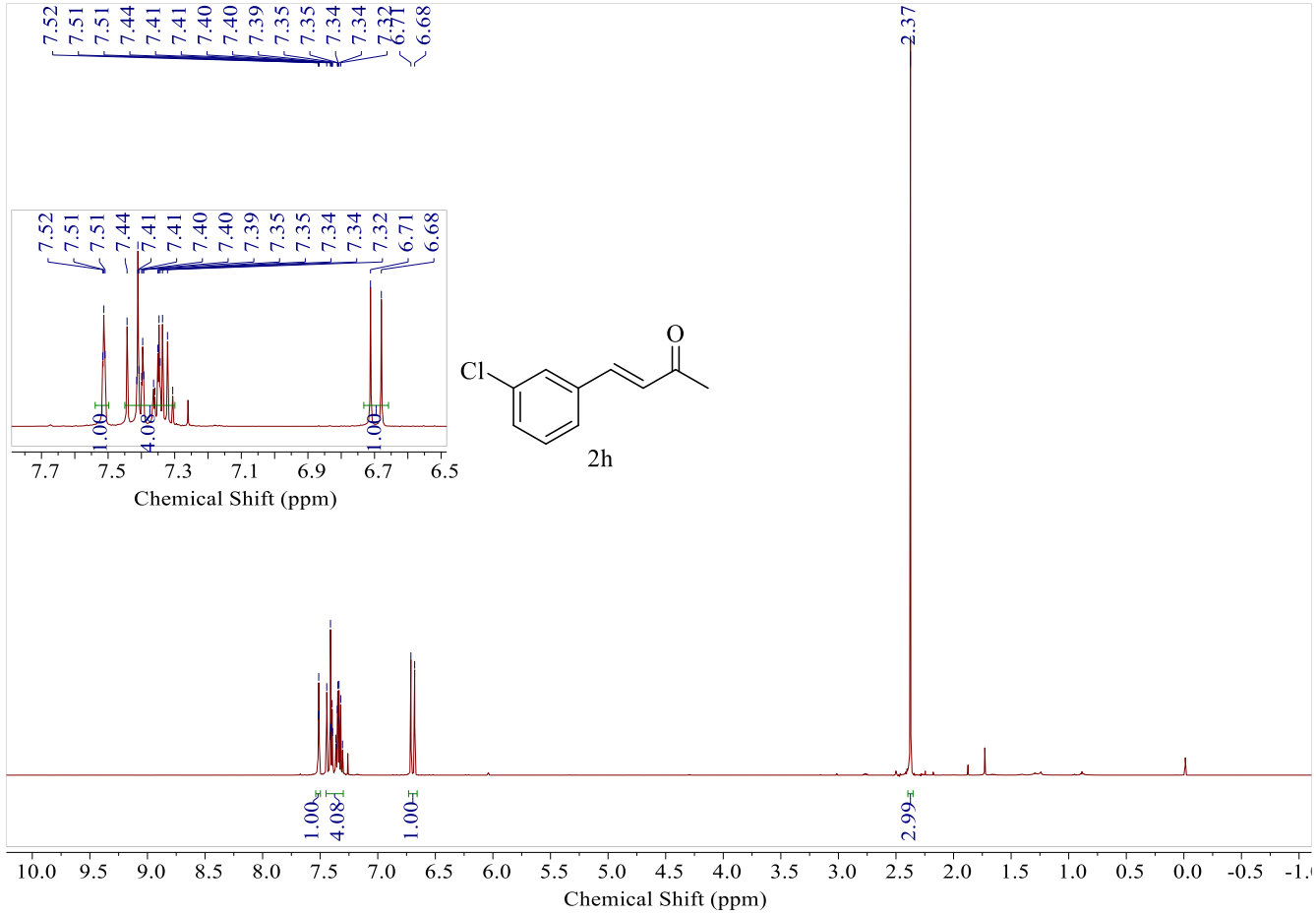
^{1}H NMR of compound **2g**



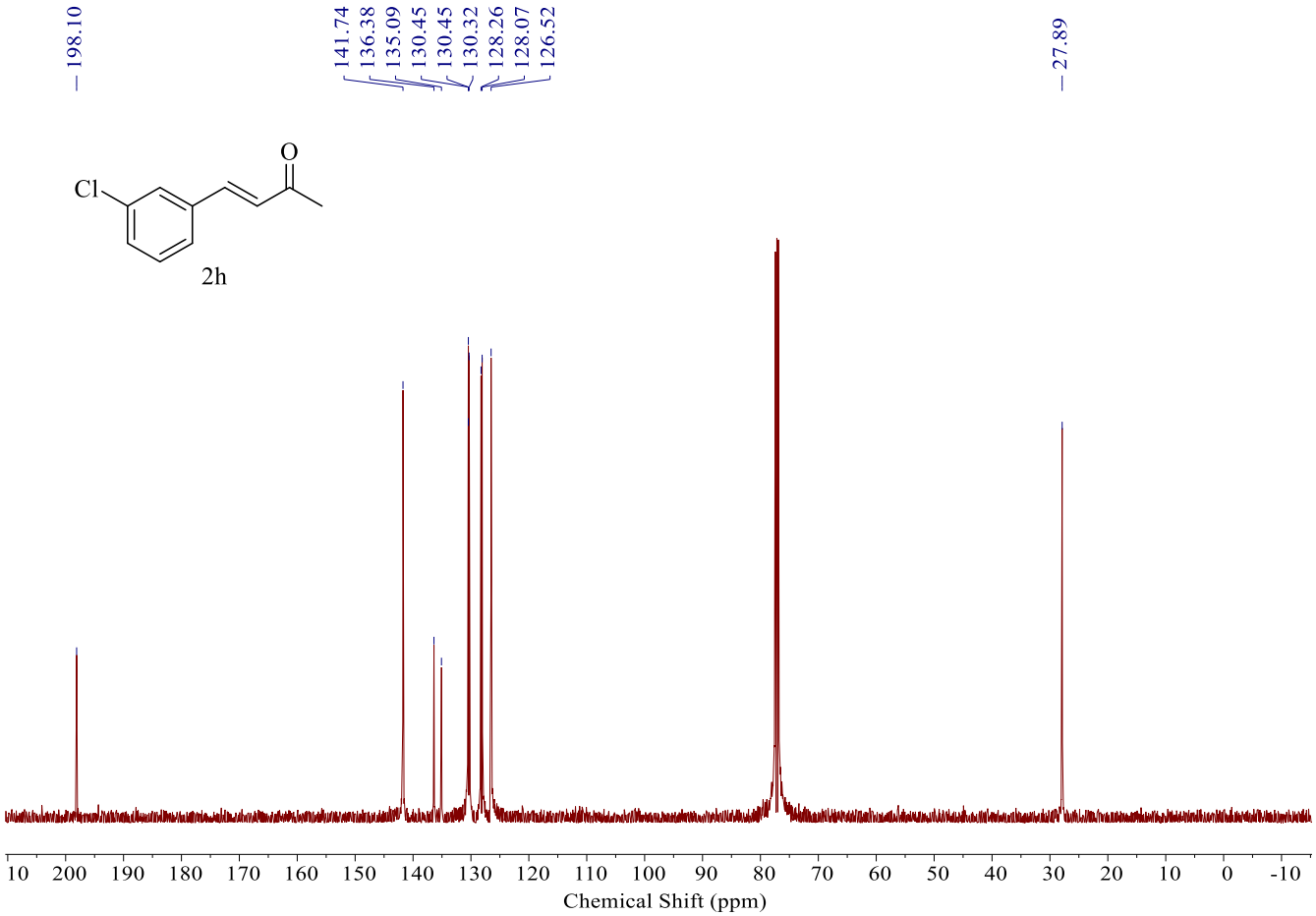
^{13}C NMR of compound **2g**



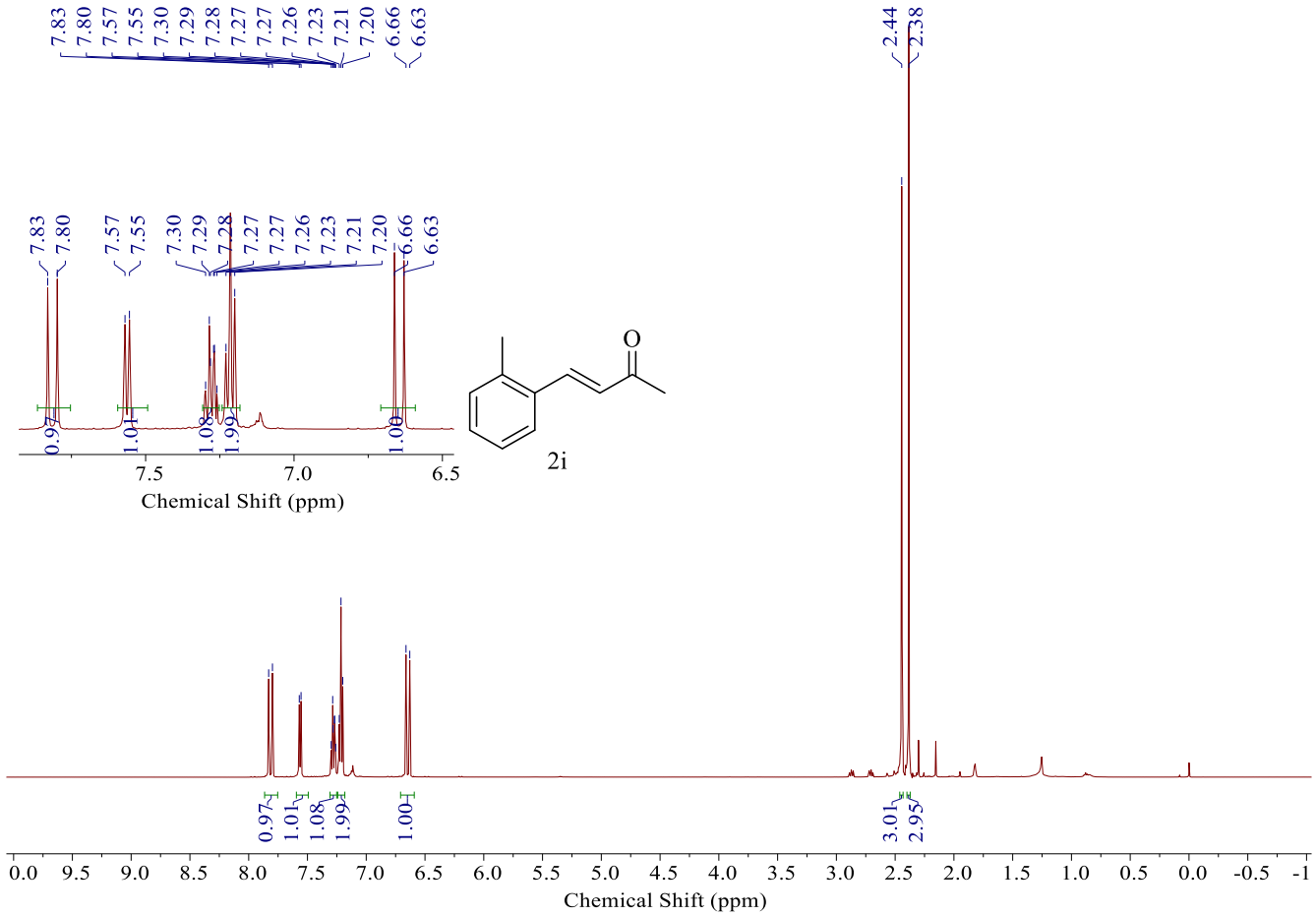
^{1}H NMR of compound **2h**



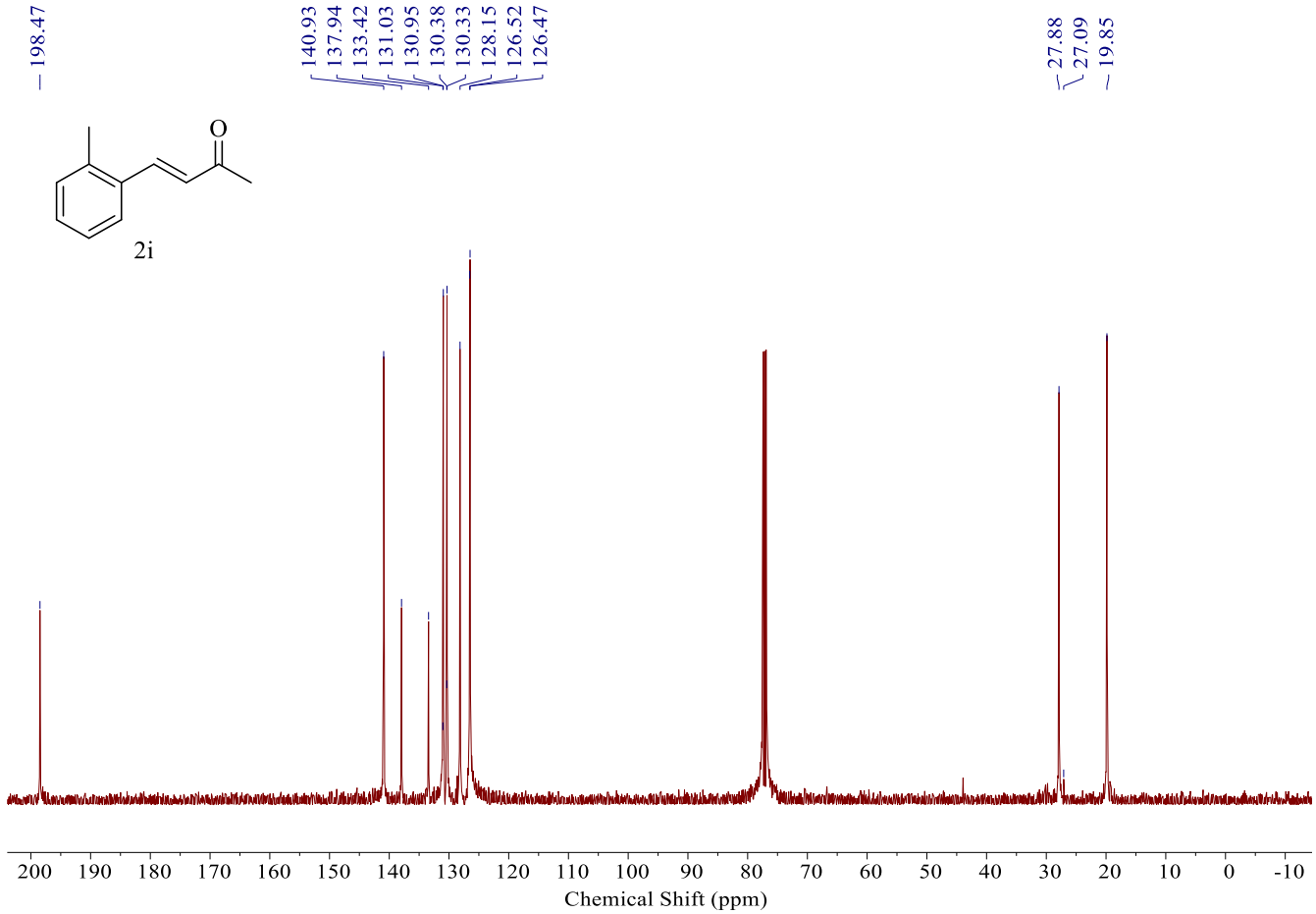
^{13}C NMR of compound **2h**



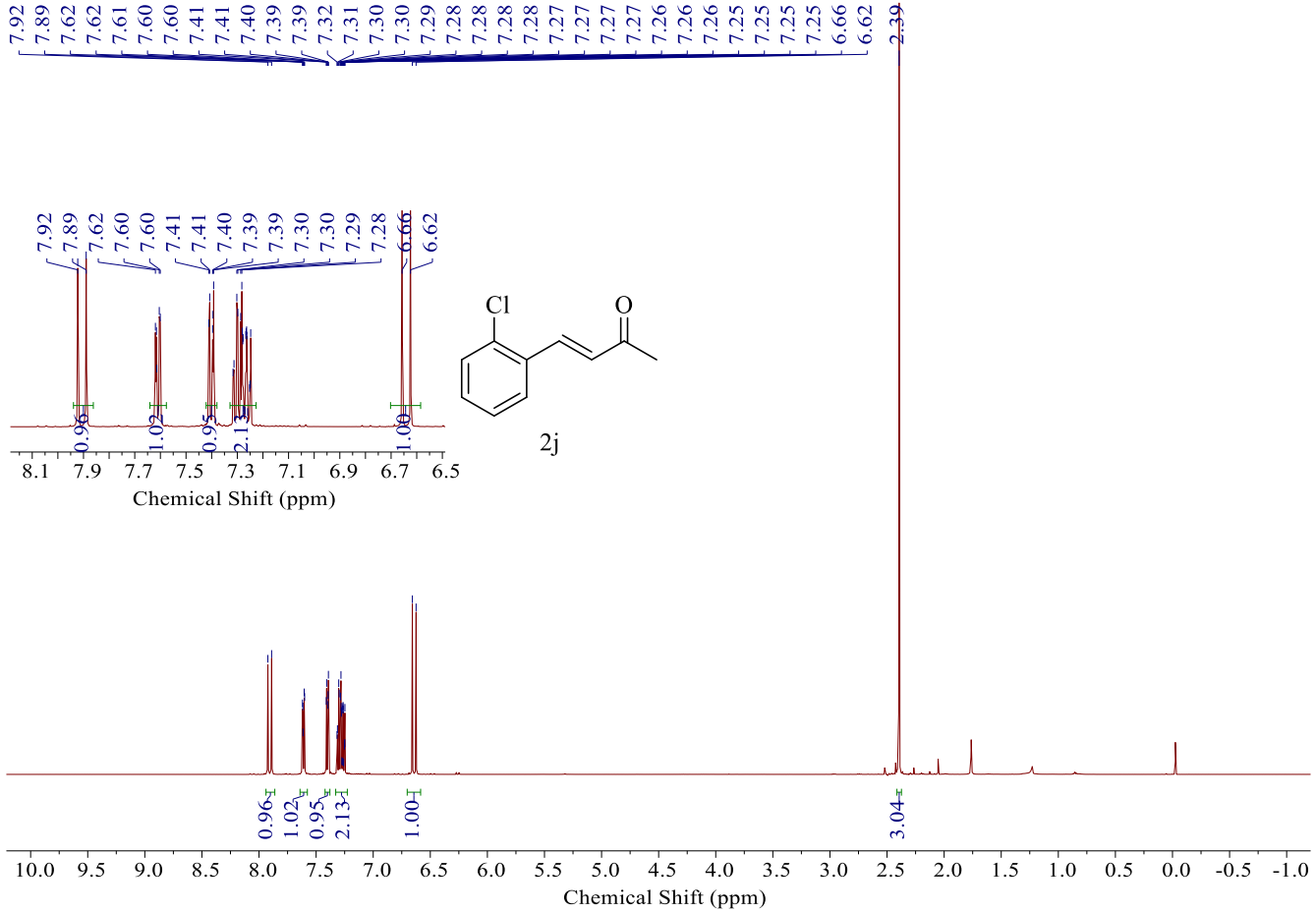
[1]H NMR of compound **2i**



^{13}C NMR of compound **2i**



[1]H NMR of compound **2j**



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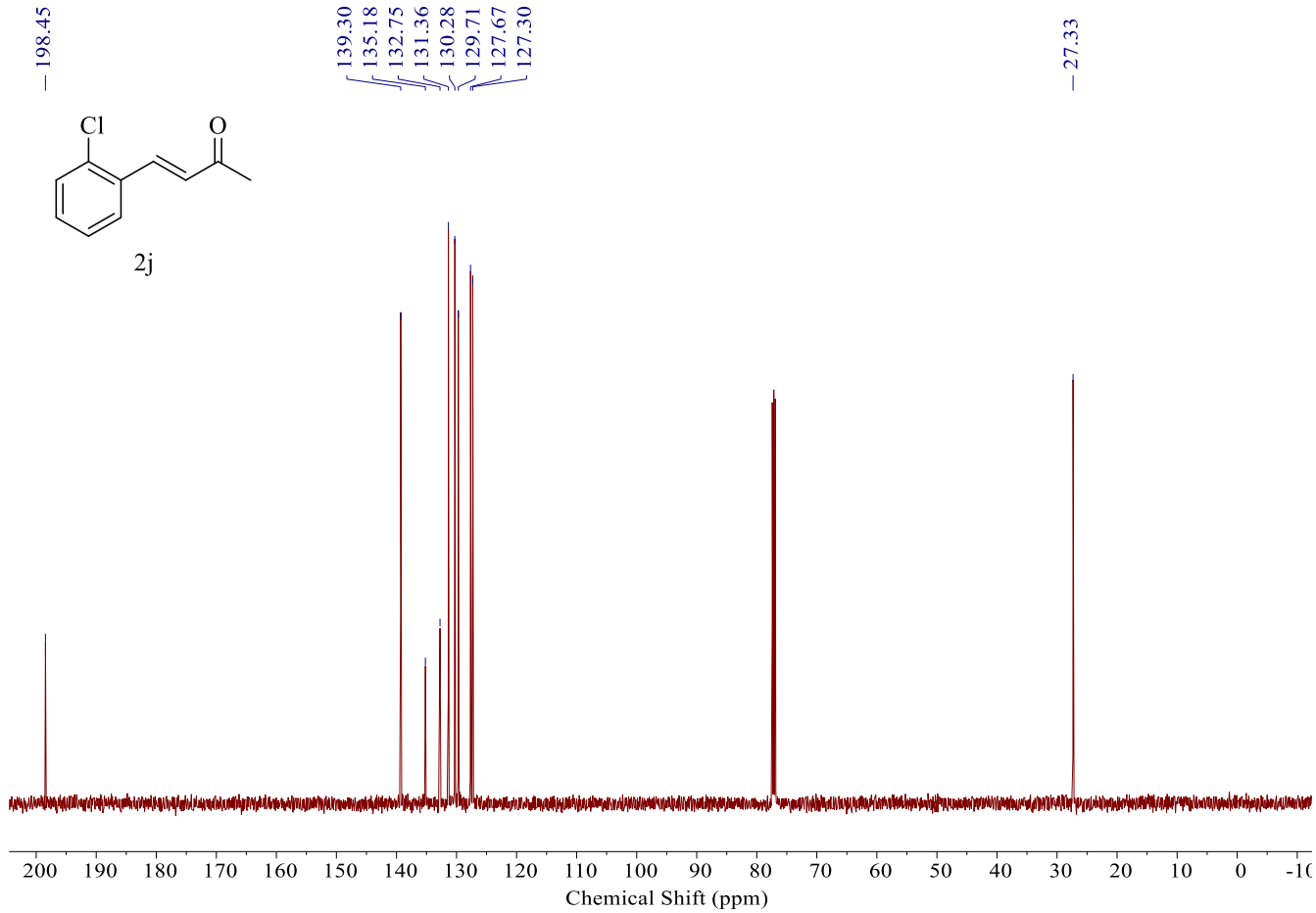

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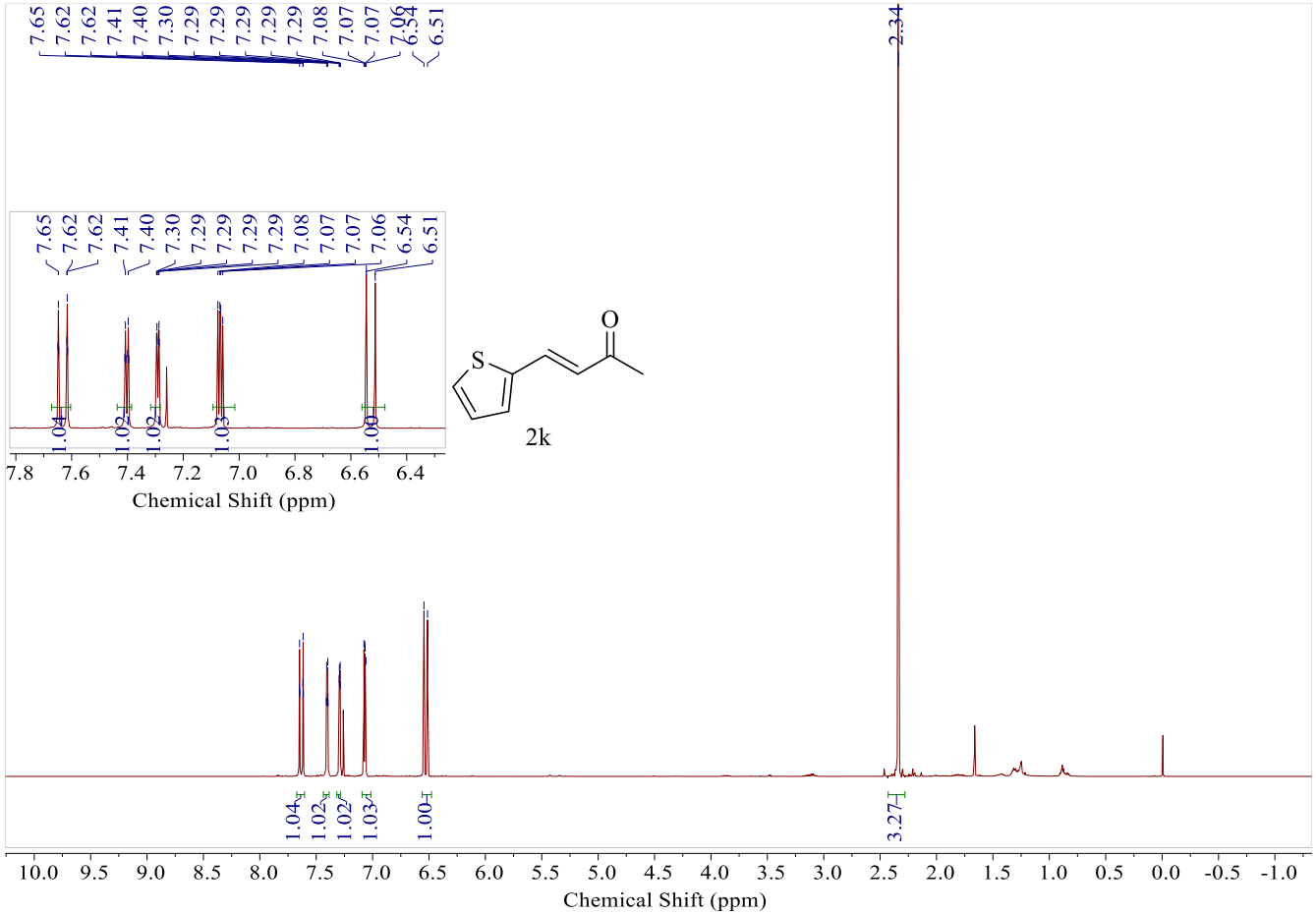
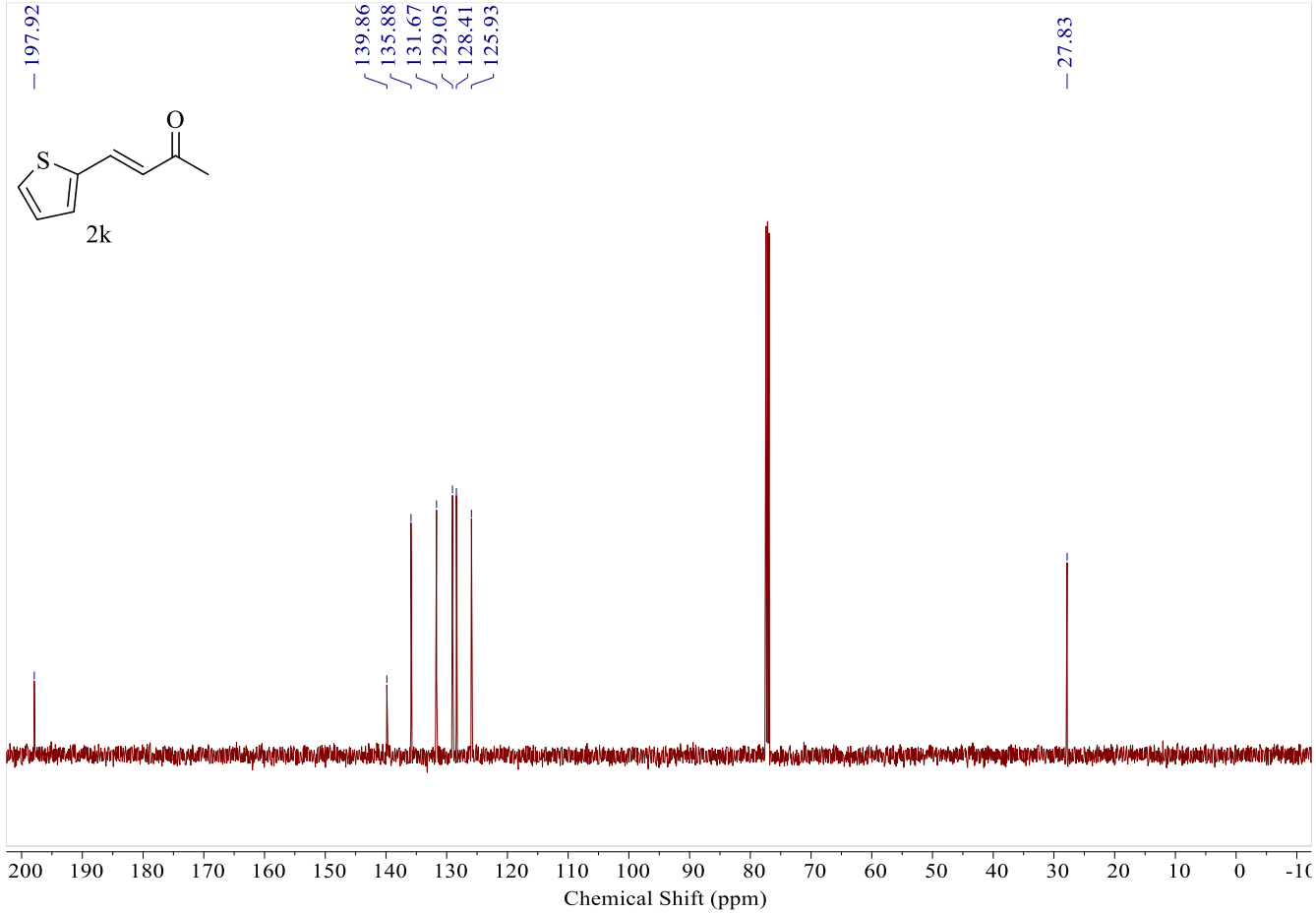
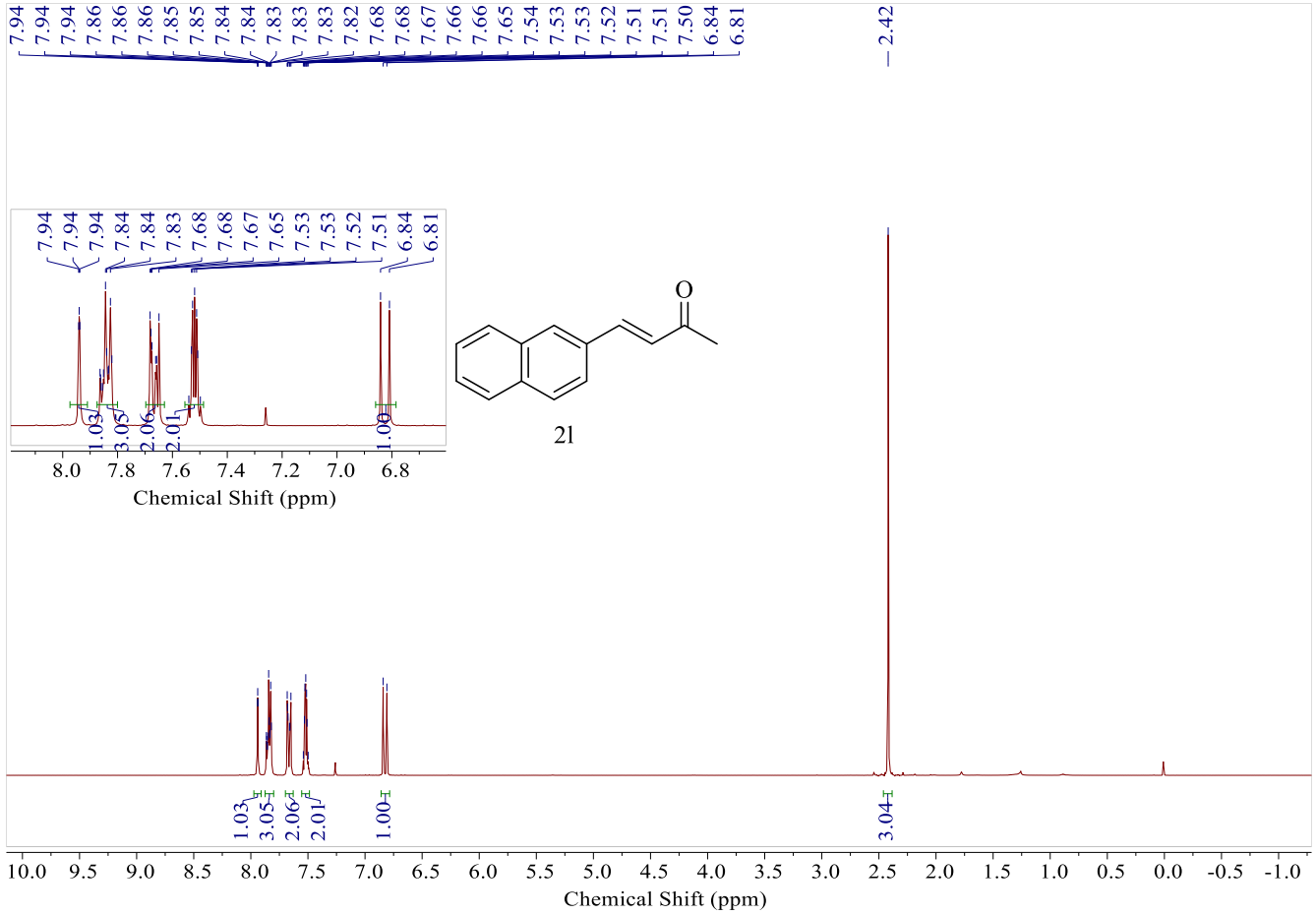


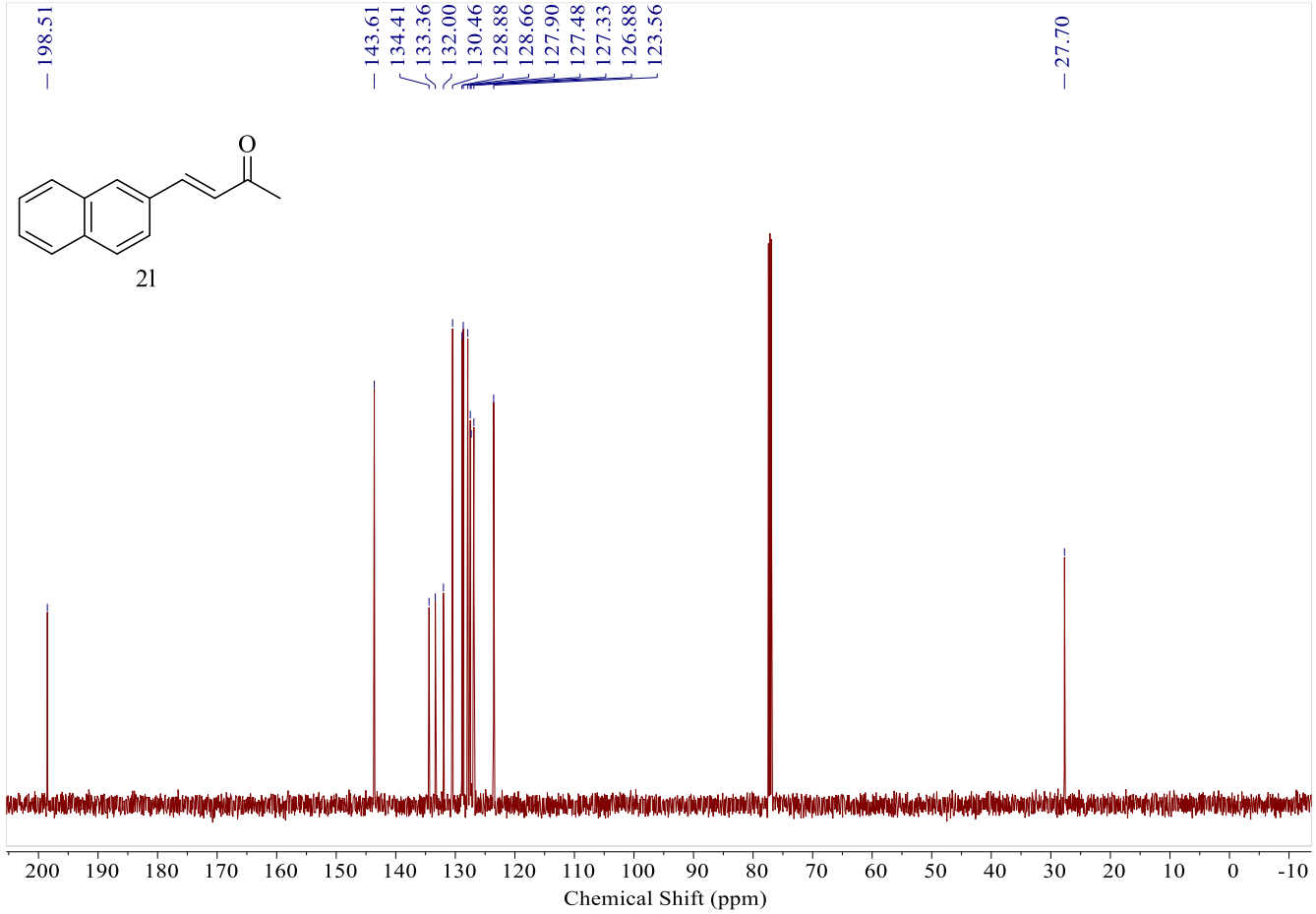
^{13}C NMR of compound **2k**



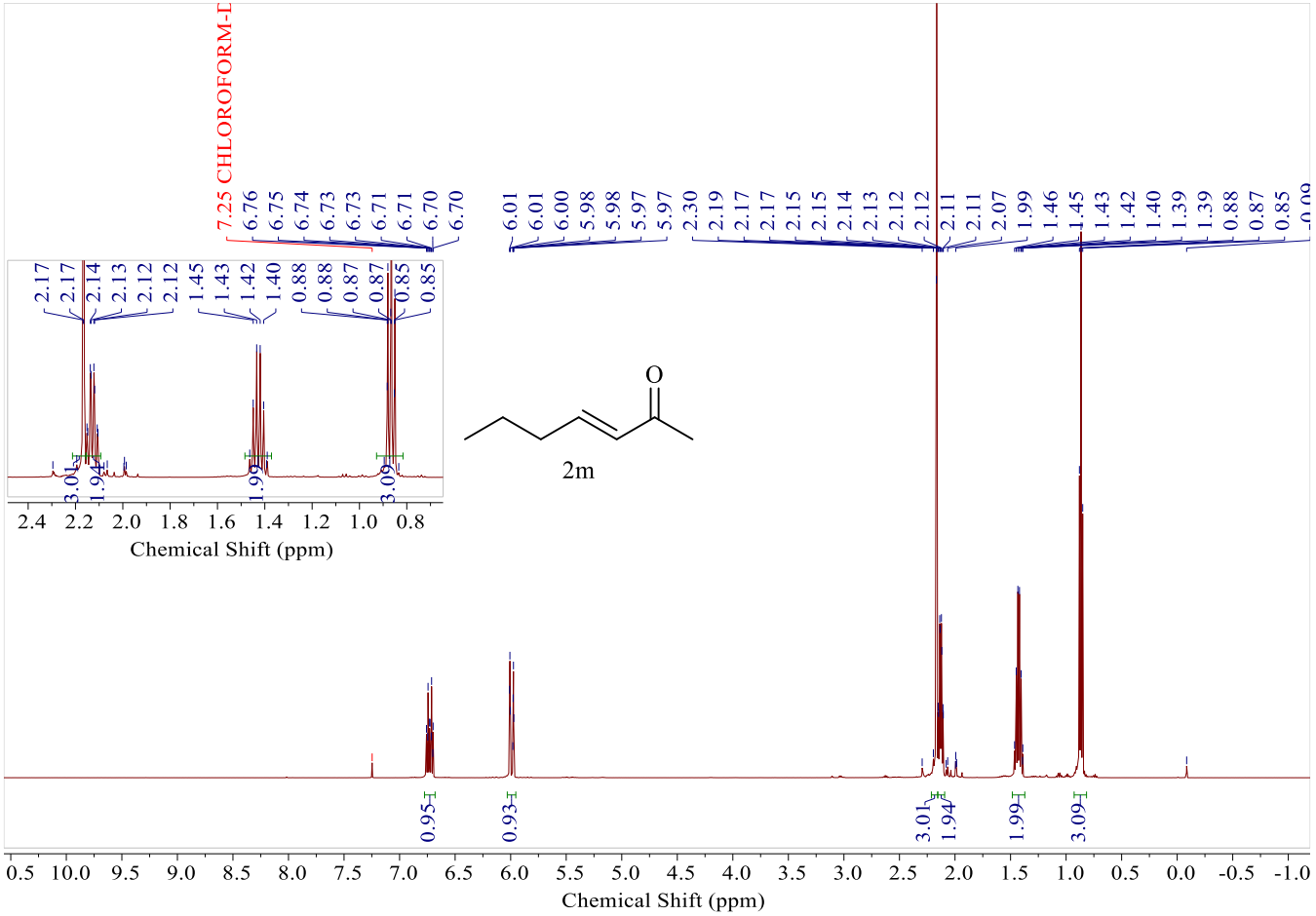
^{1}H NMR of compound **2l**



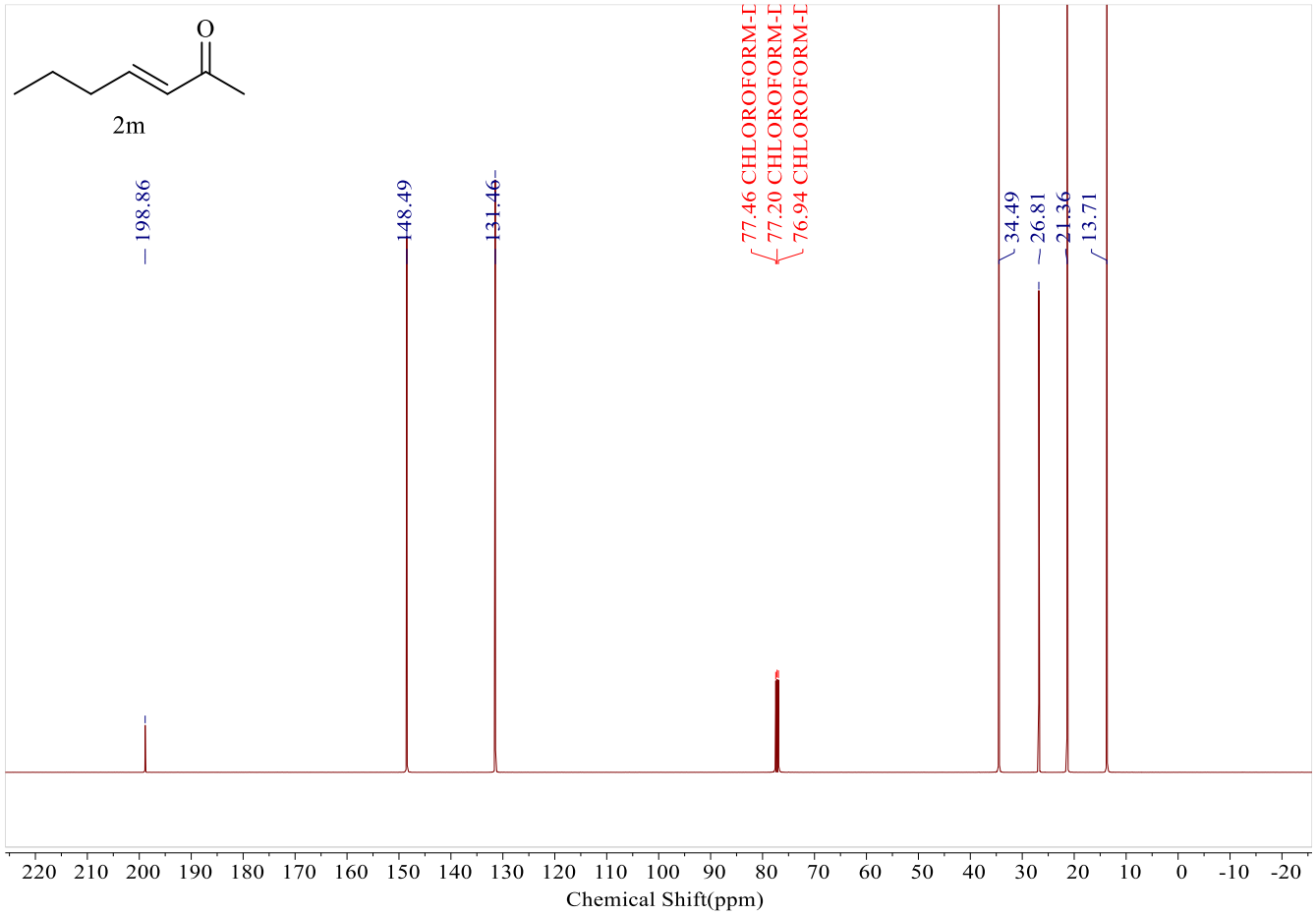
^{13}C NMR of compound **2l**



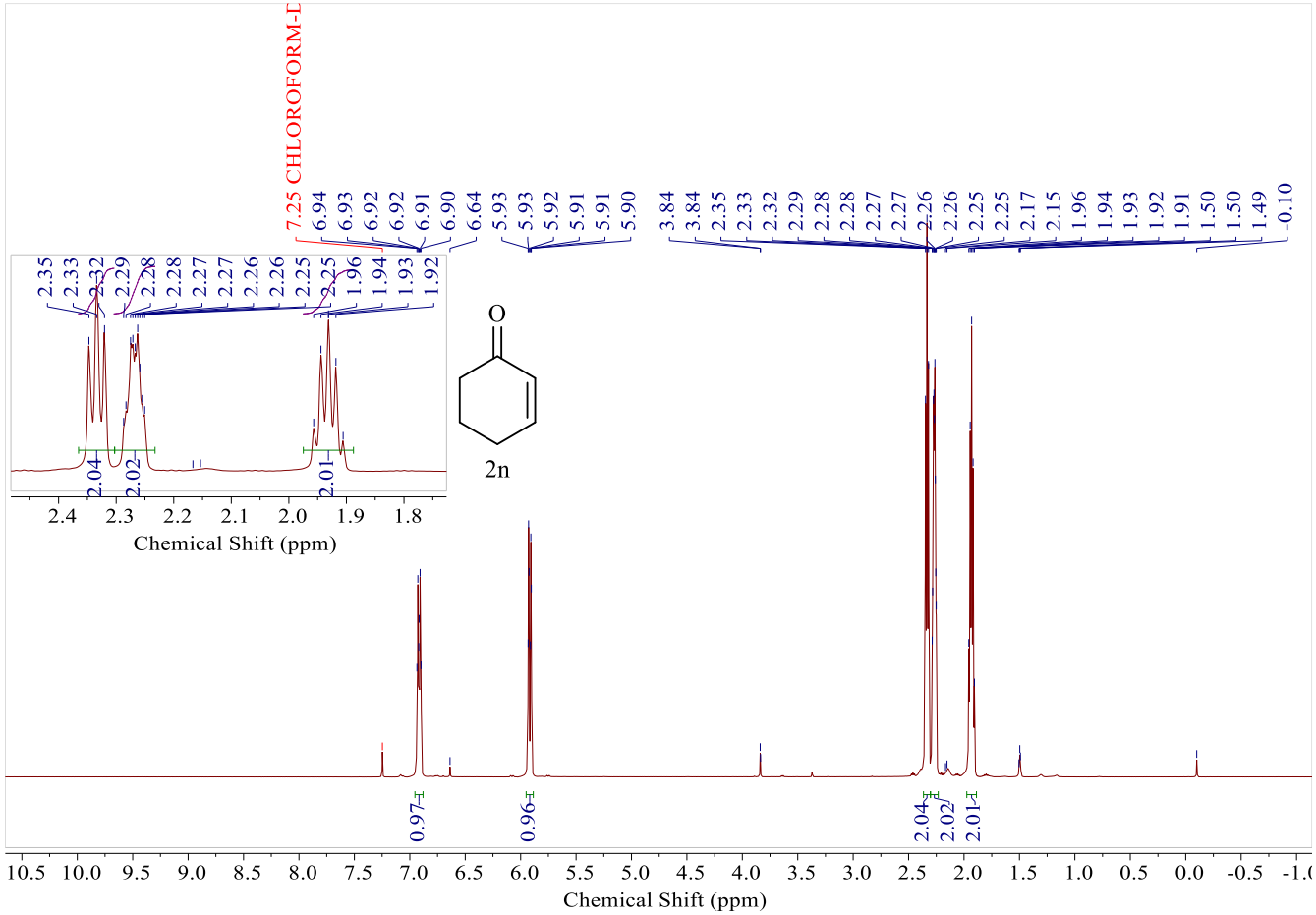
[1]H NMR of compound **2m**



^{13}C NMR of compound **2m**



^{1}H NMR of compound **2n**



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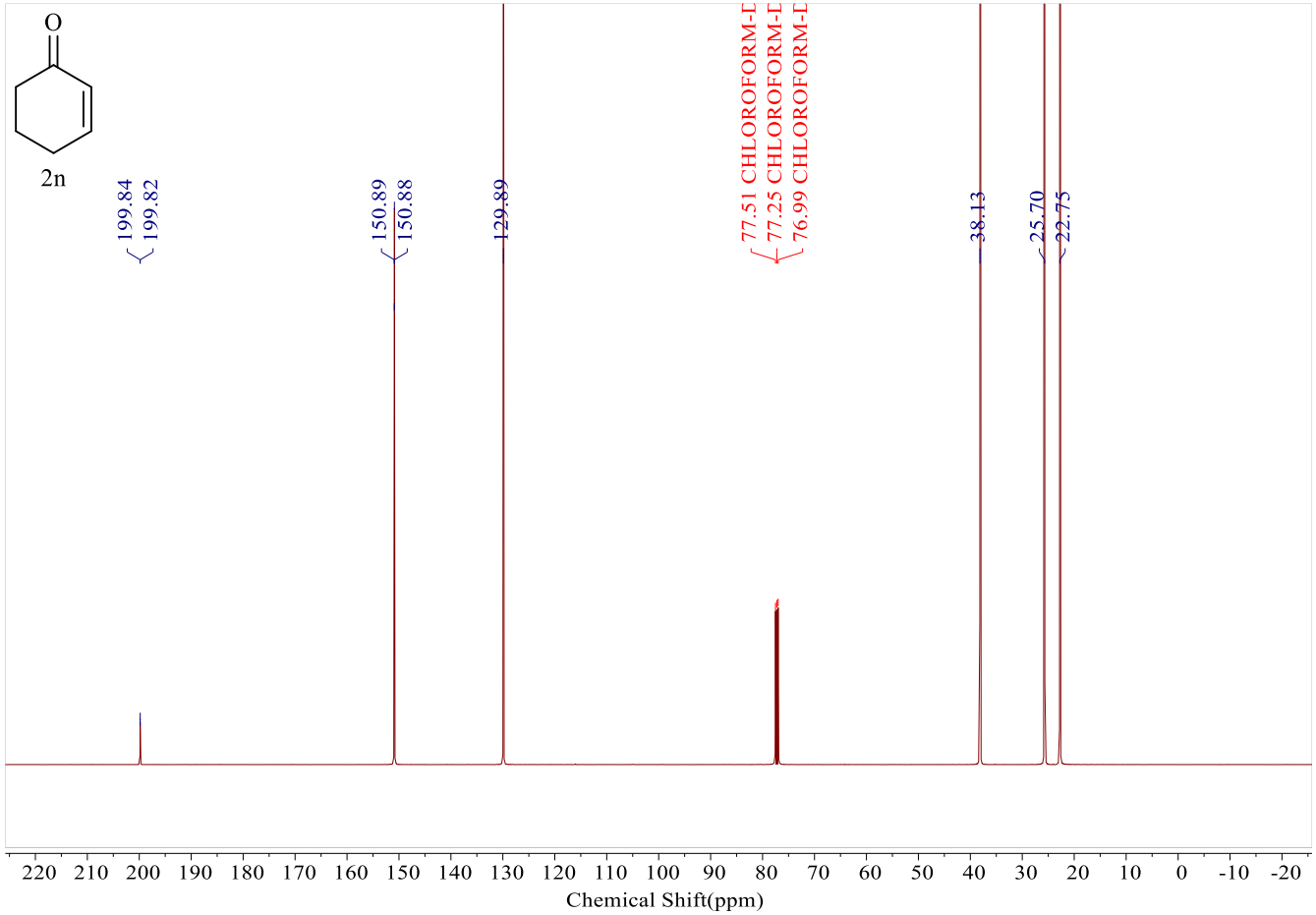
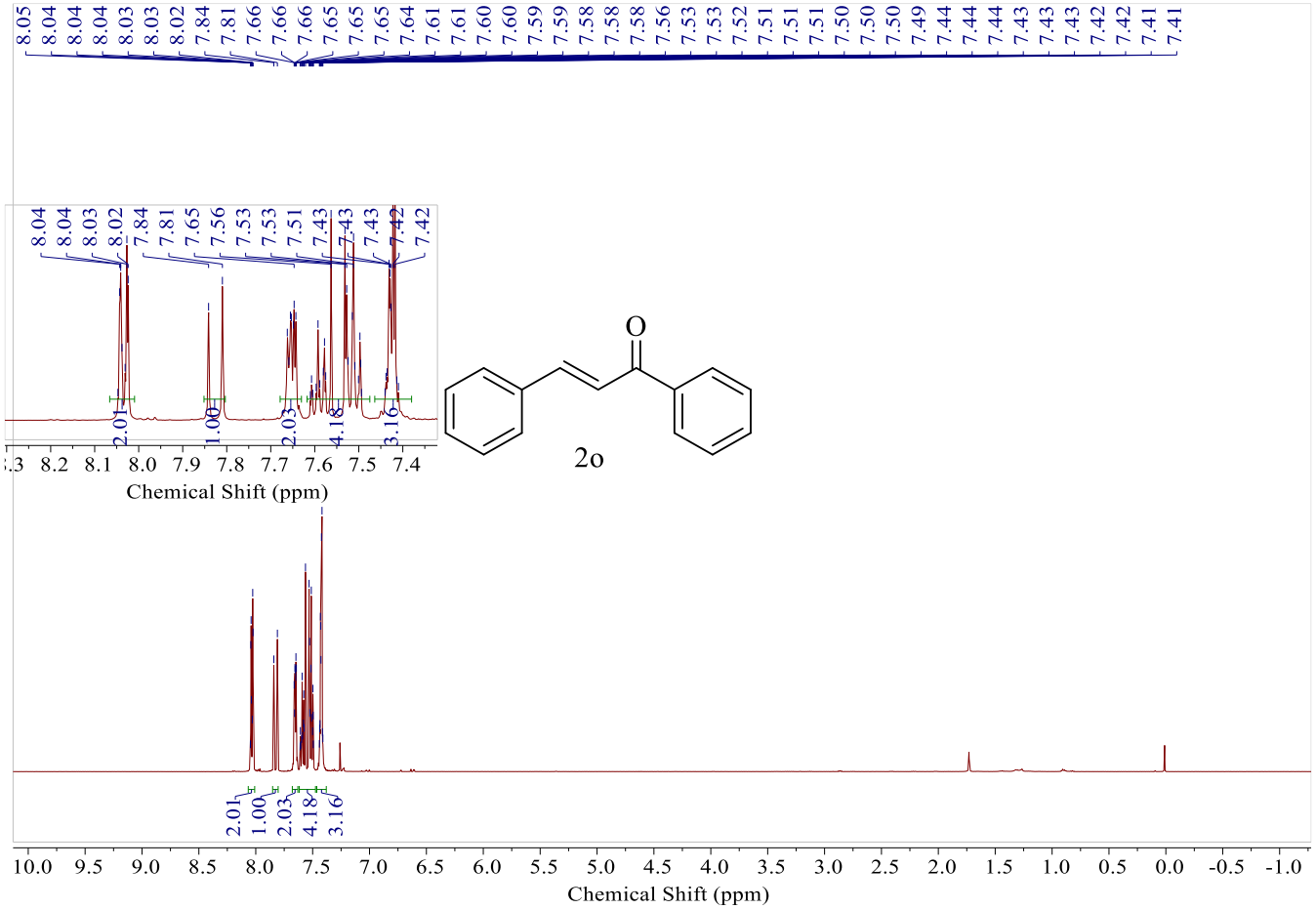
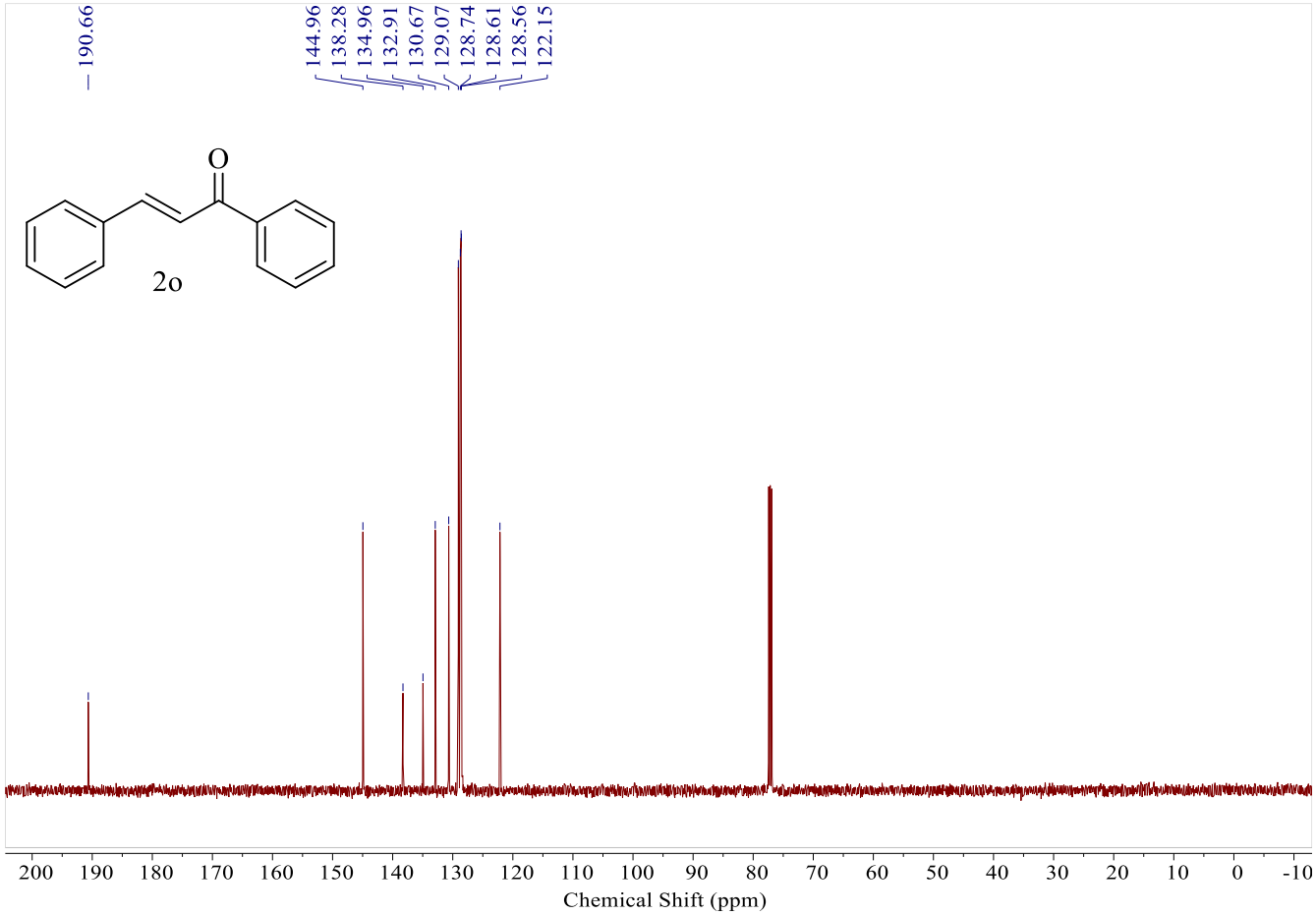


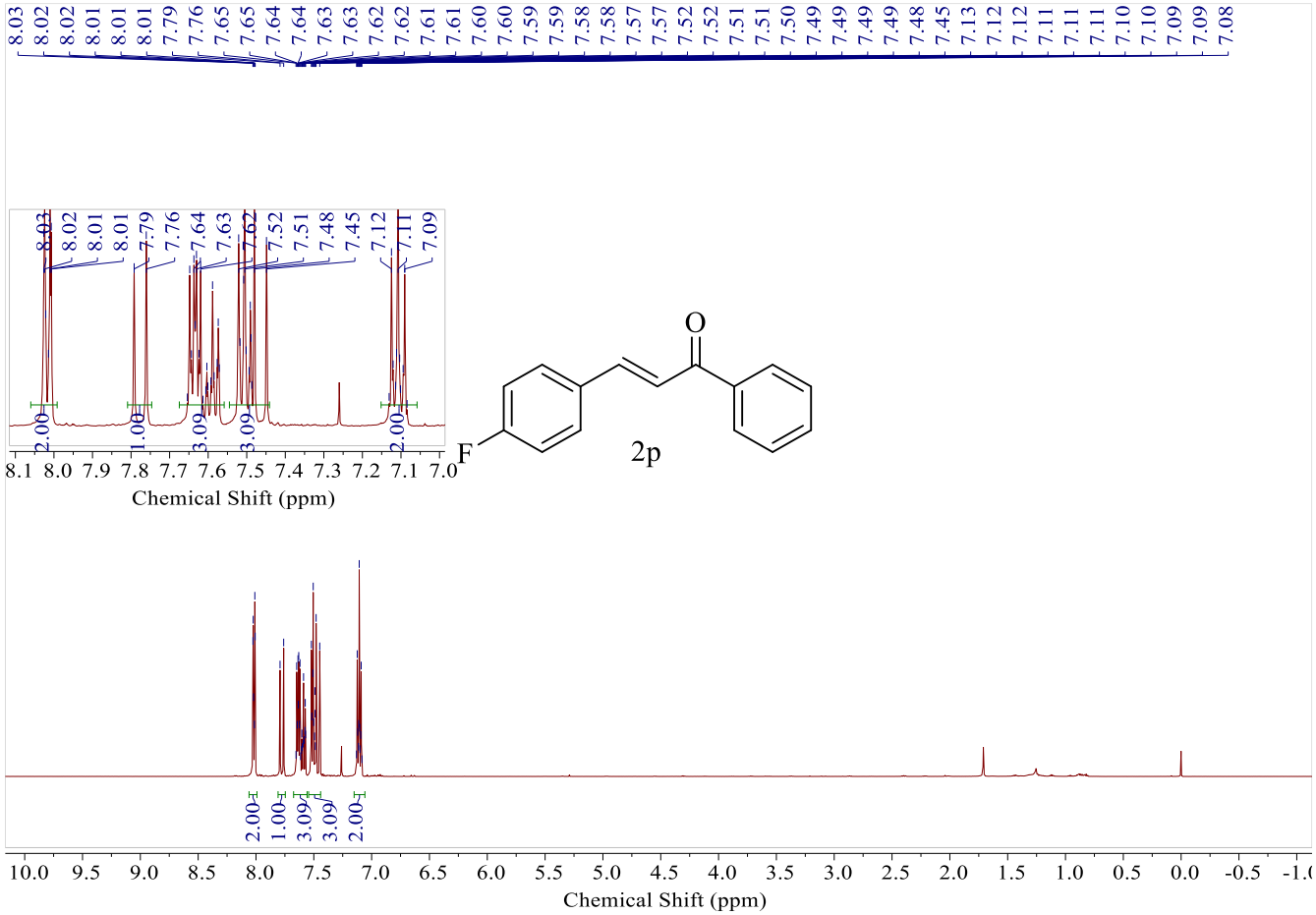
^{1}H NMR of compound **2o**



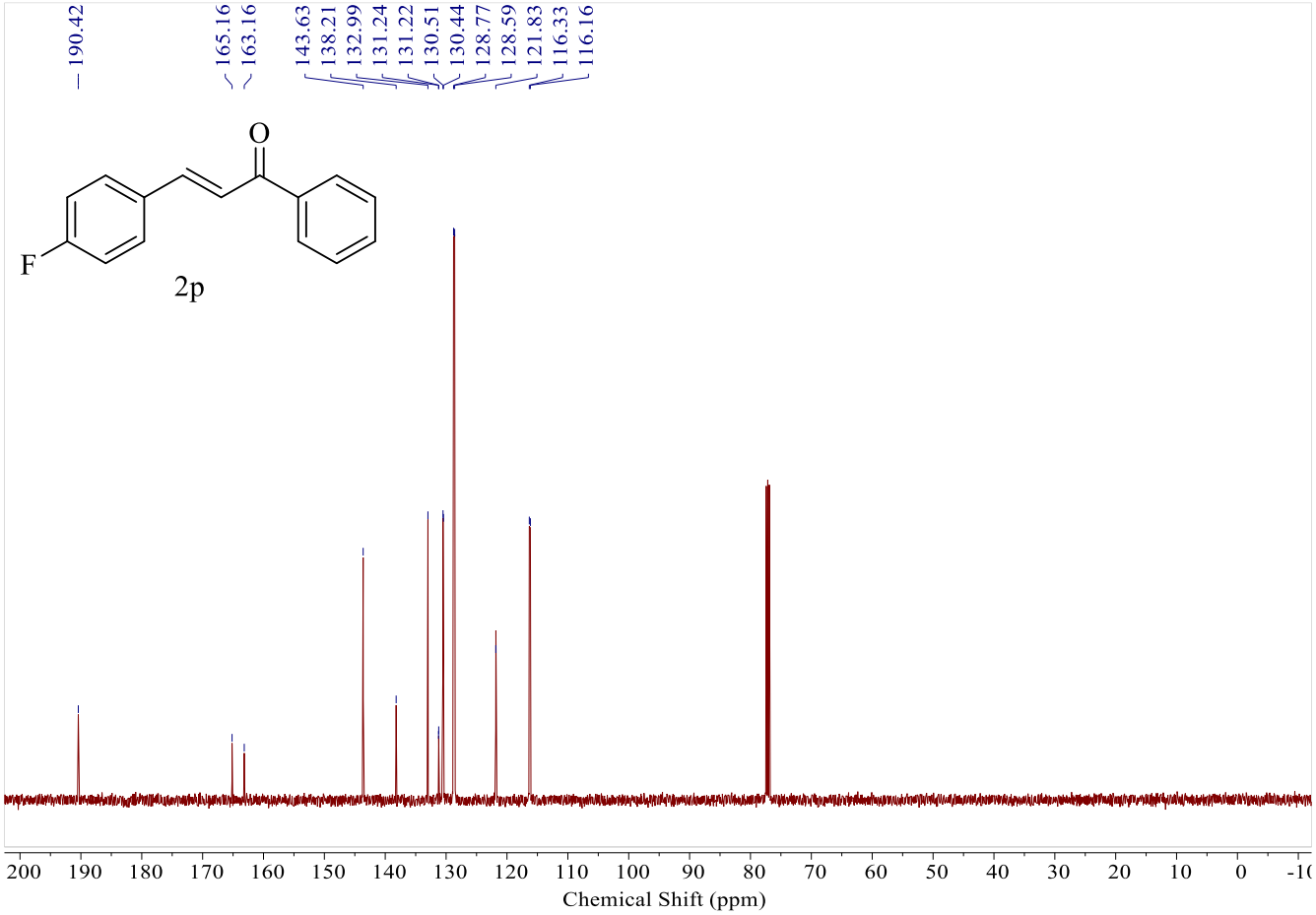
^{13}C NMR of compound **2o**



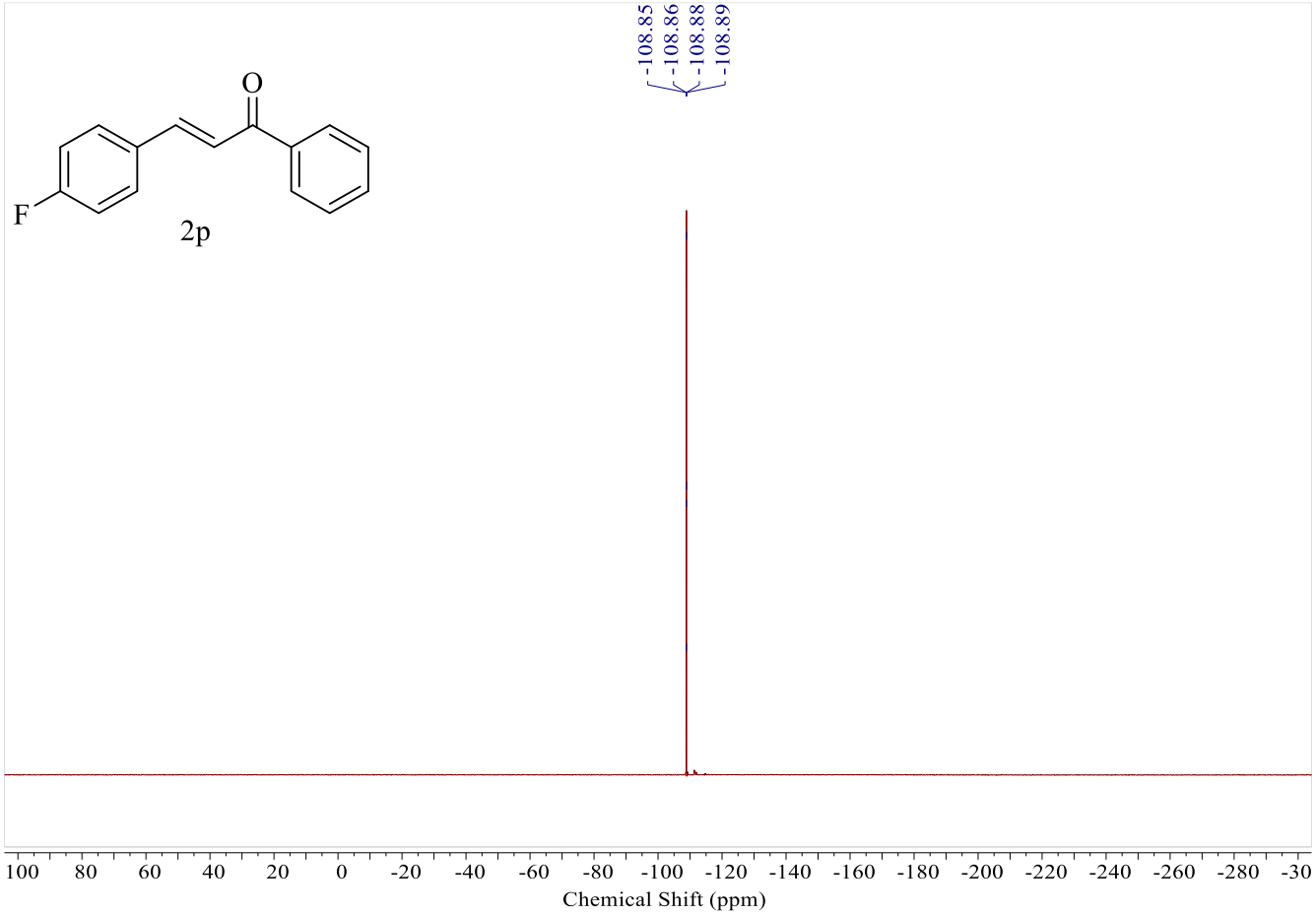
^{1}H NMR of compound **2p**



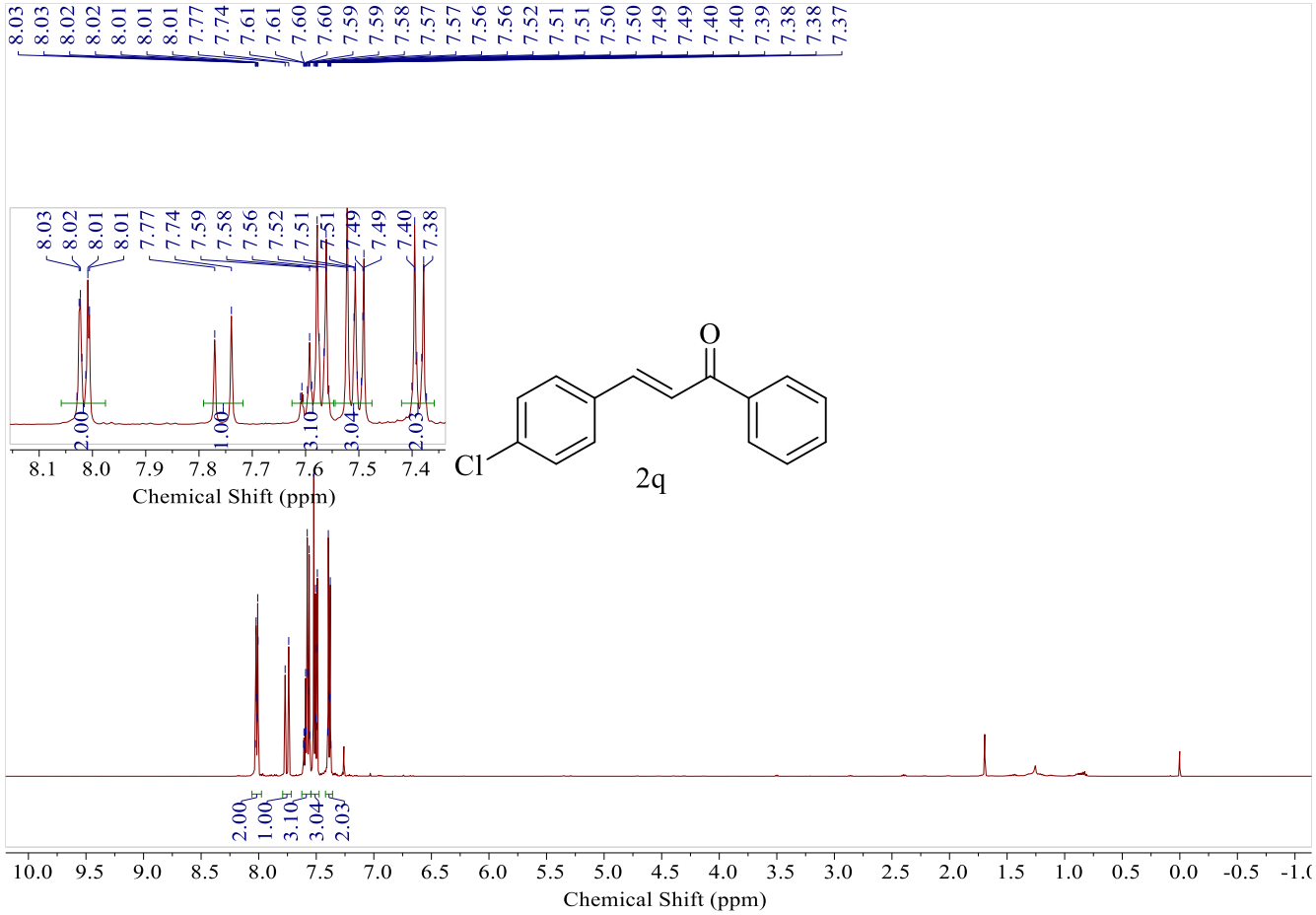
^{13}C NMR of compound **2p**



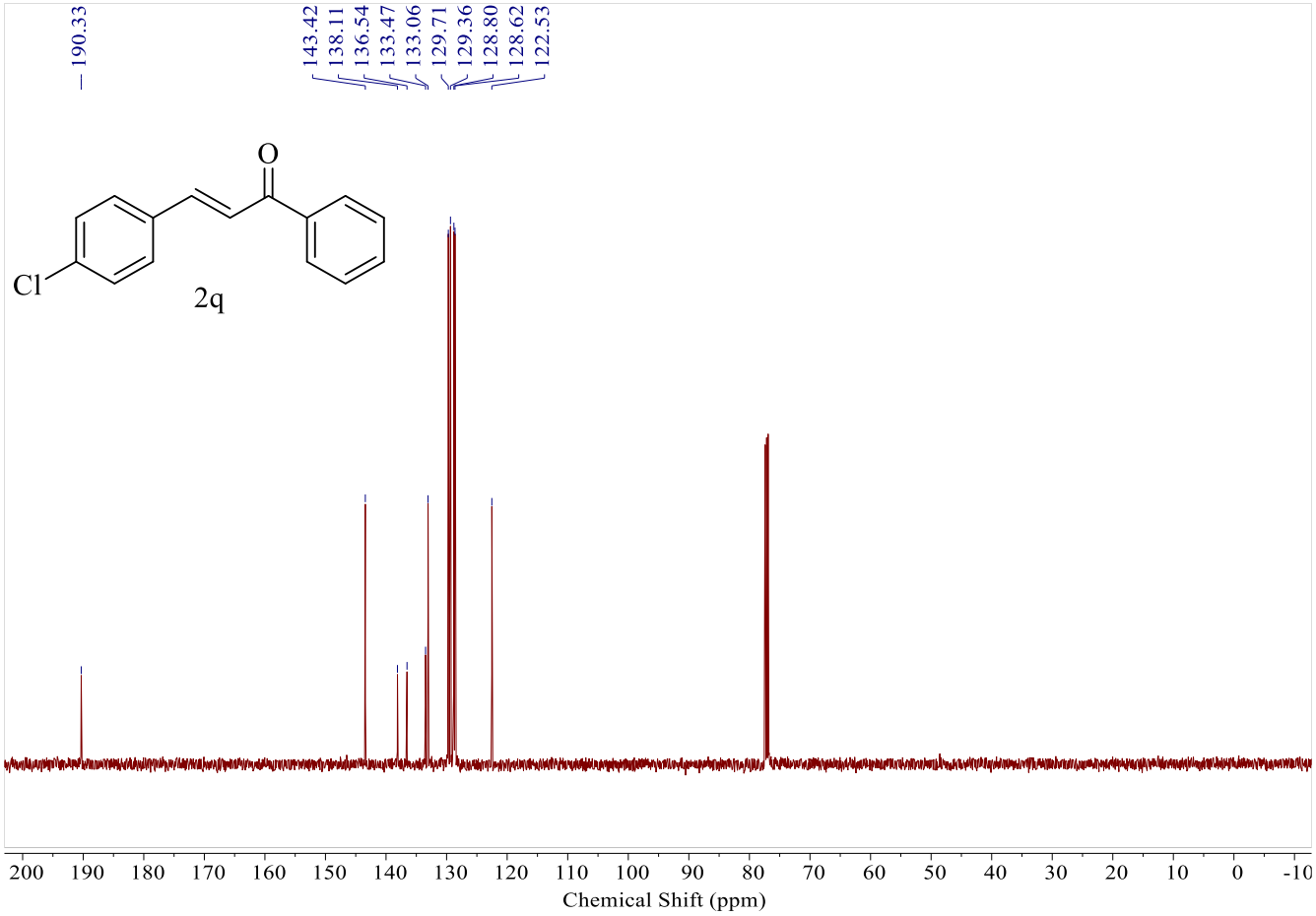
^{19}F NMR of compound **2p**



^{1}H NMR of compound **2q**



^{13}C NMR of compound **2q**



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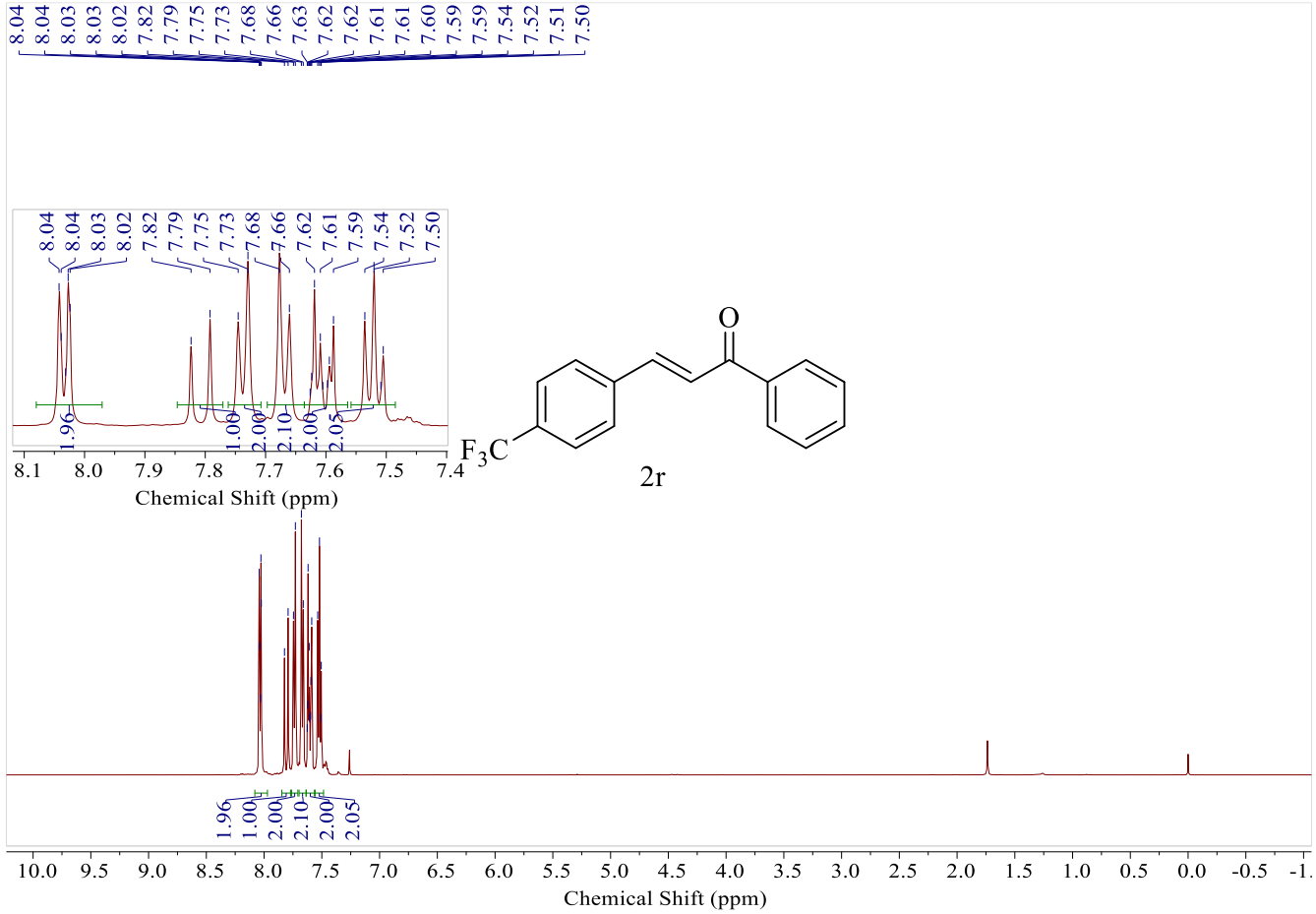
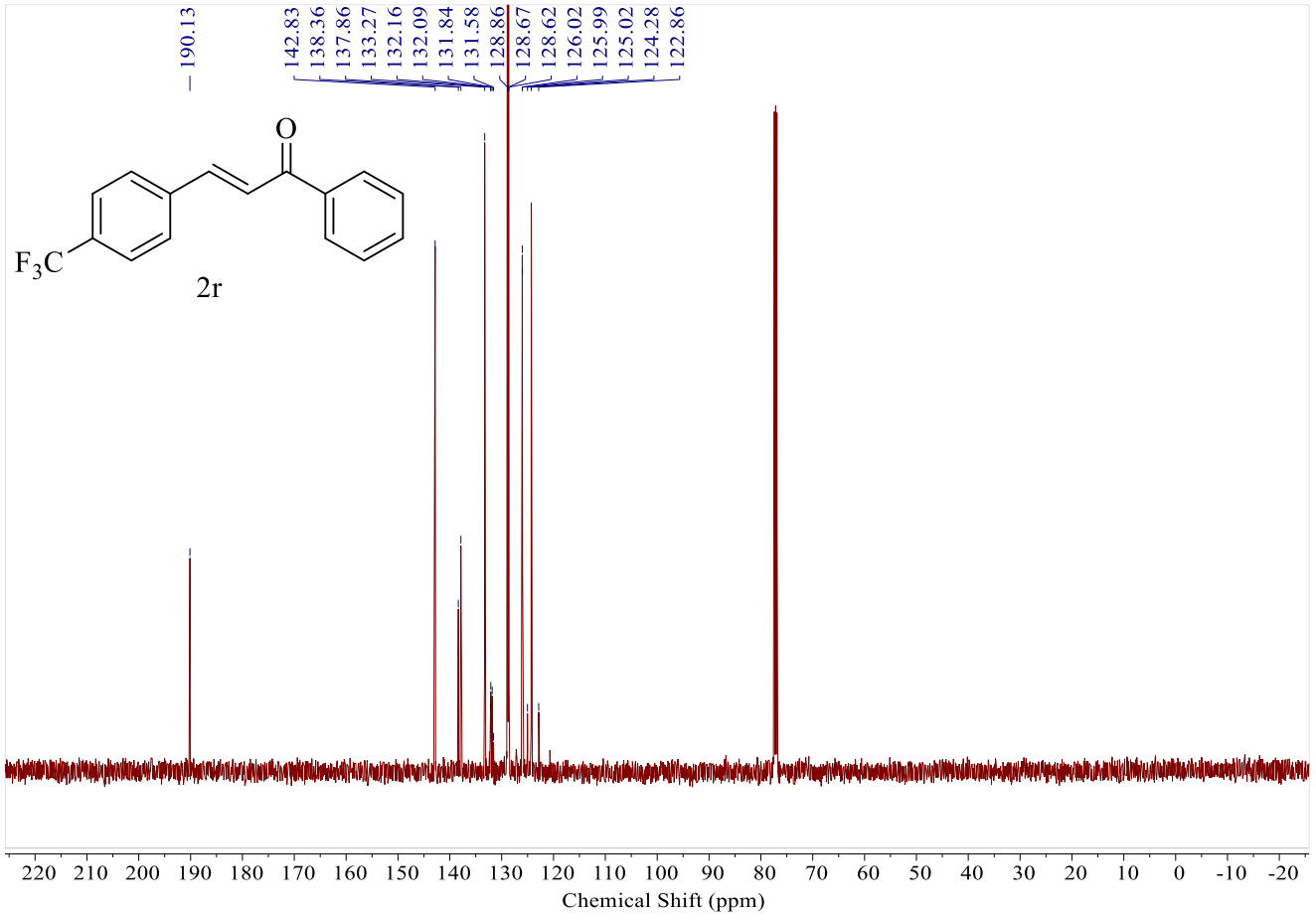
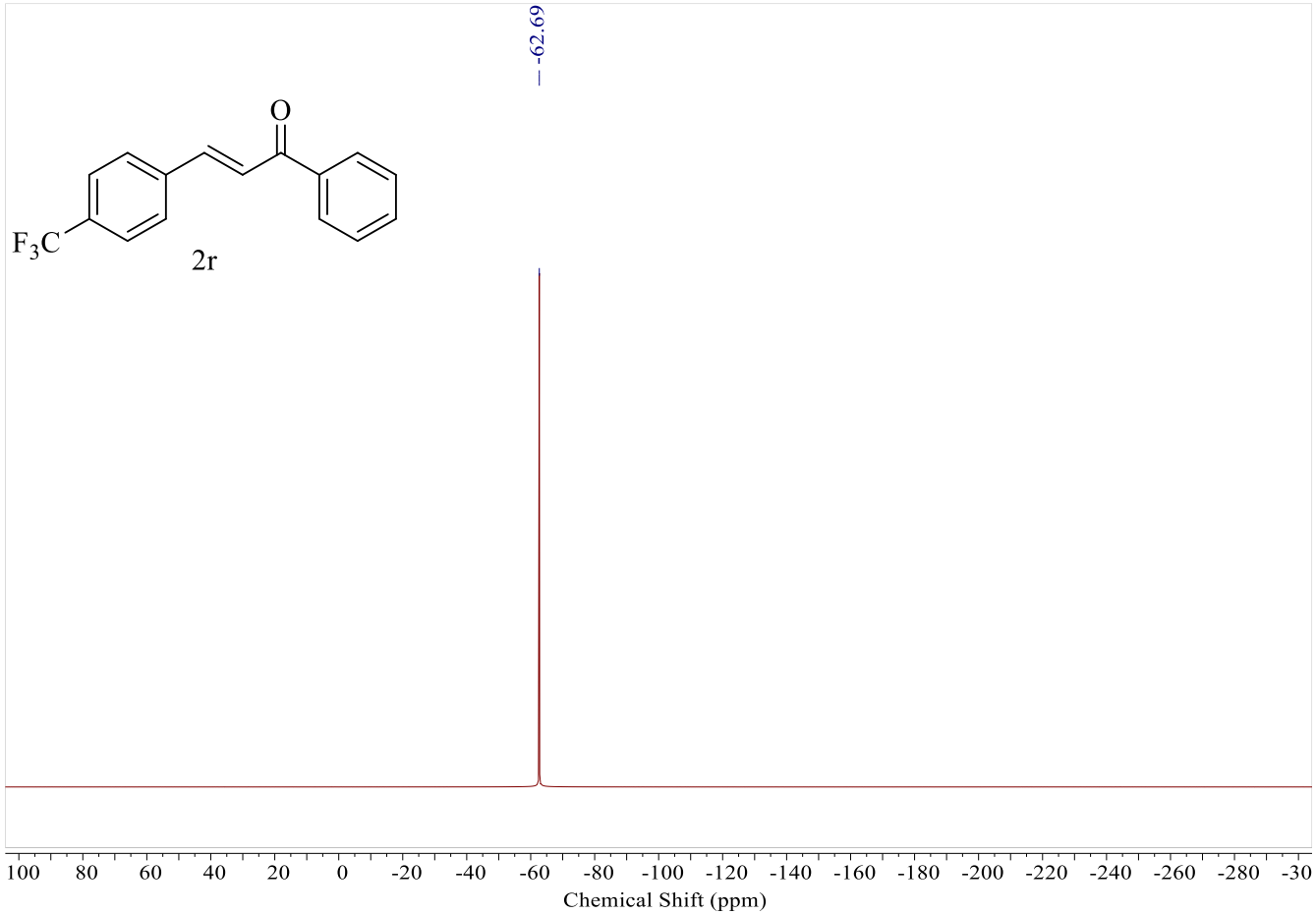


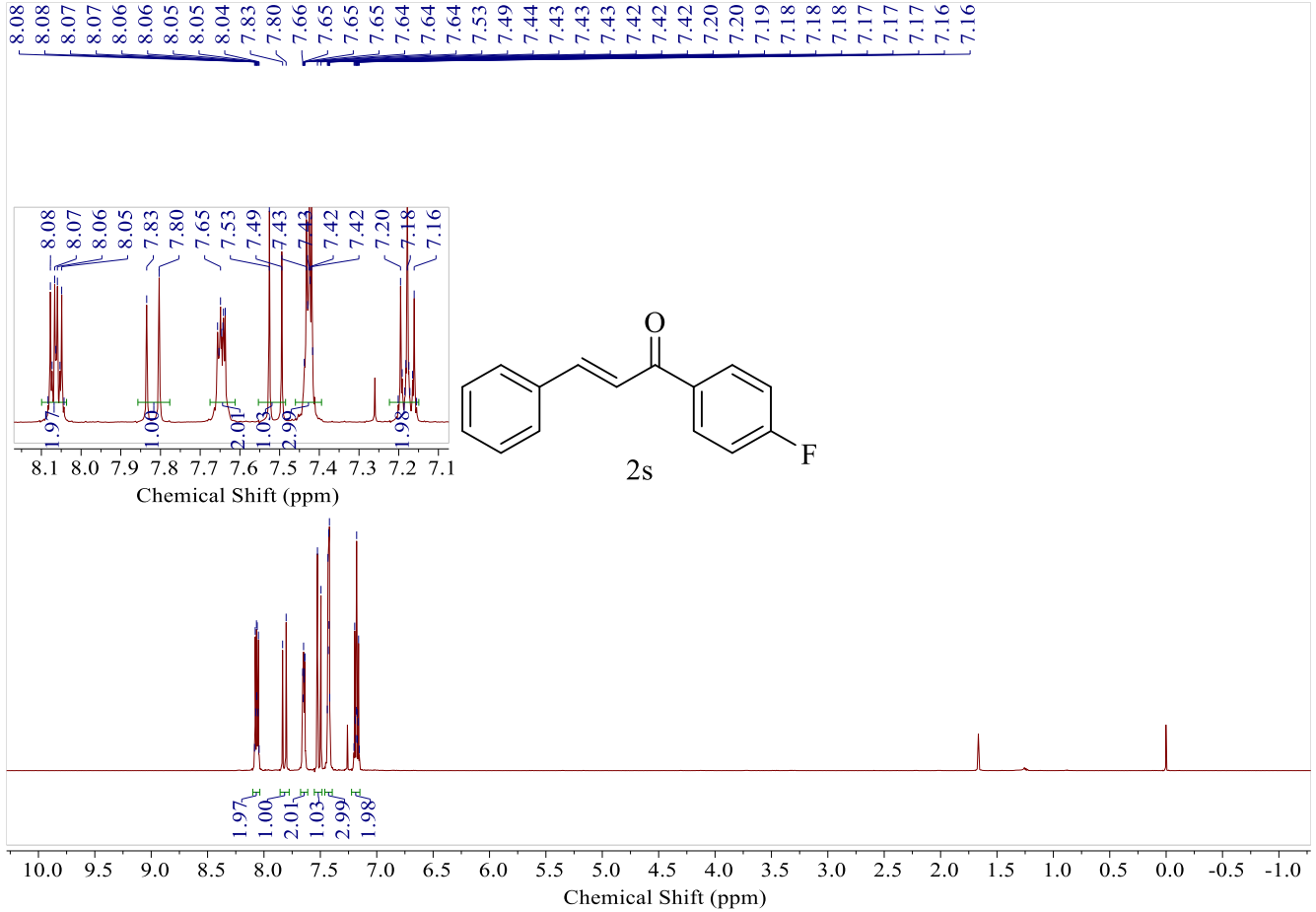
^{13}C NMR of compound **2r**



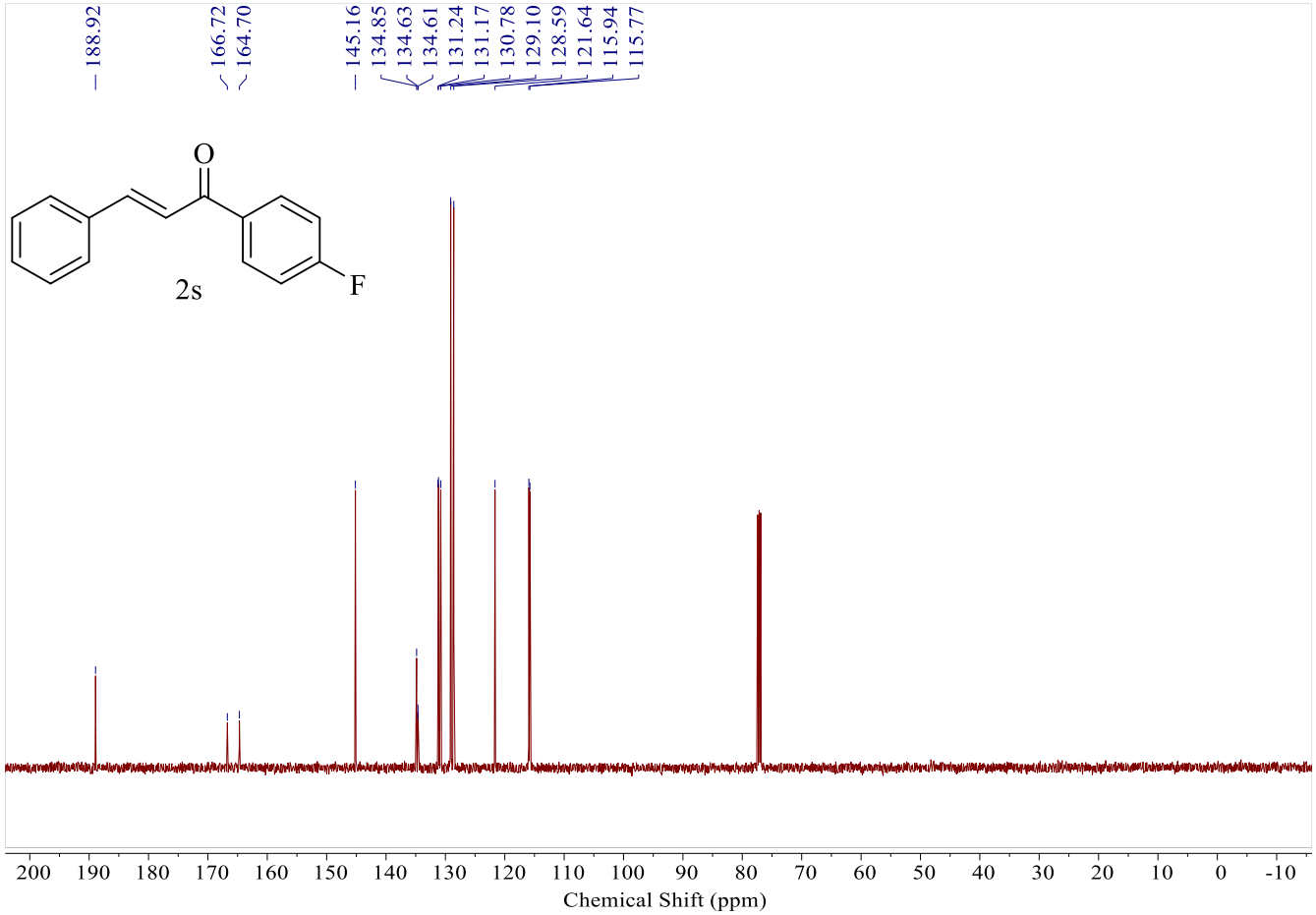
^{19}F NMR of compound **2r**



^{1}H NMR of compound **2s**



^{13}C NMR of compound **2s**



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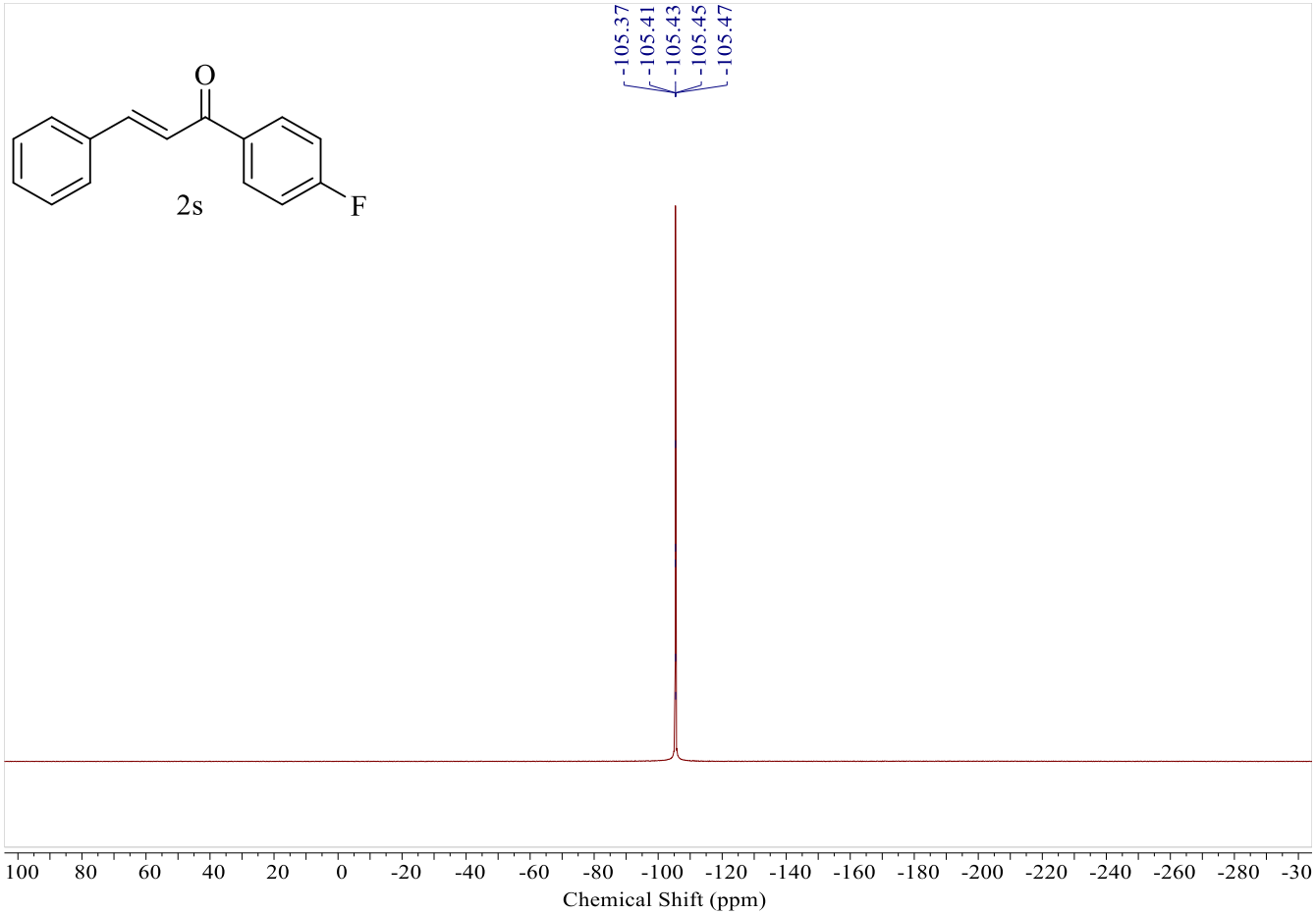
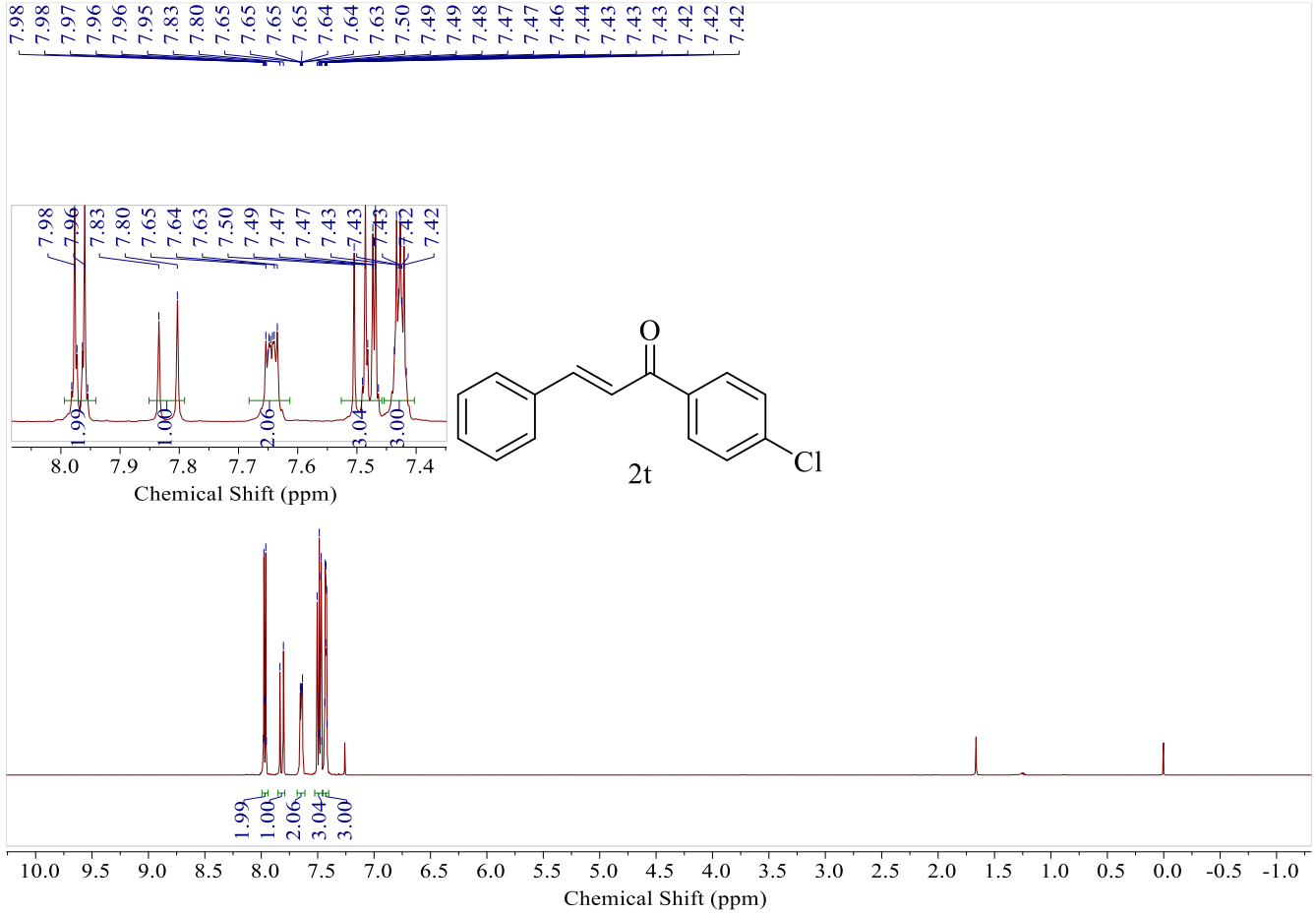
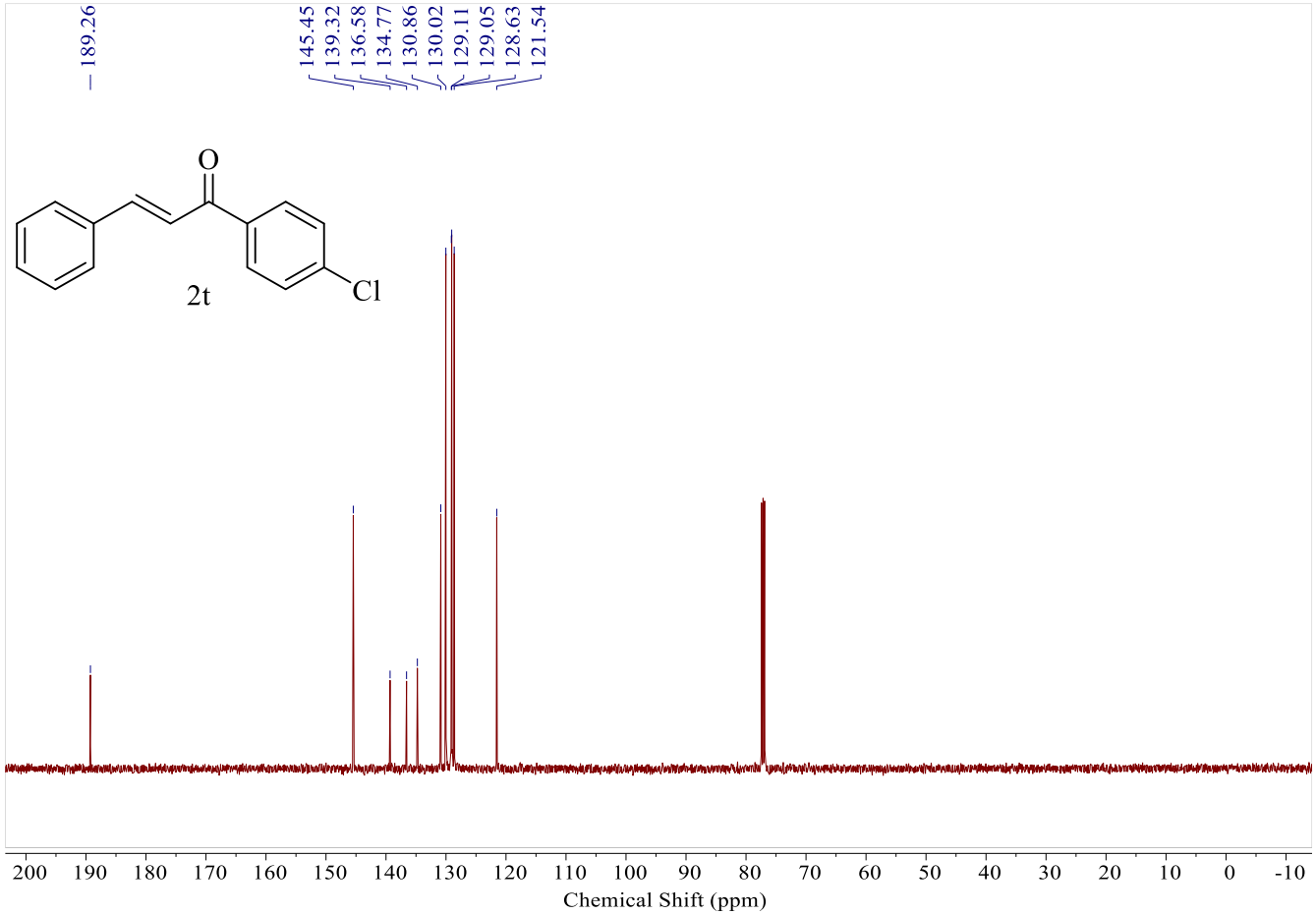


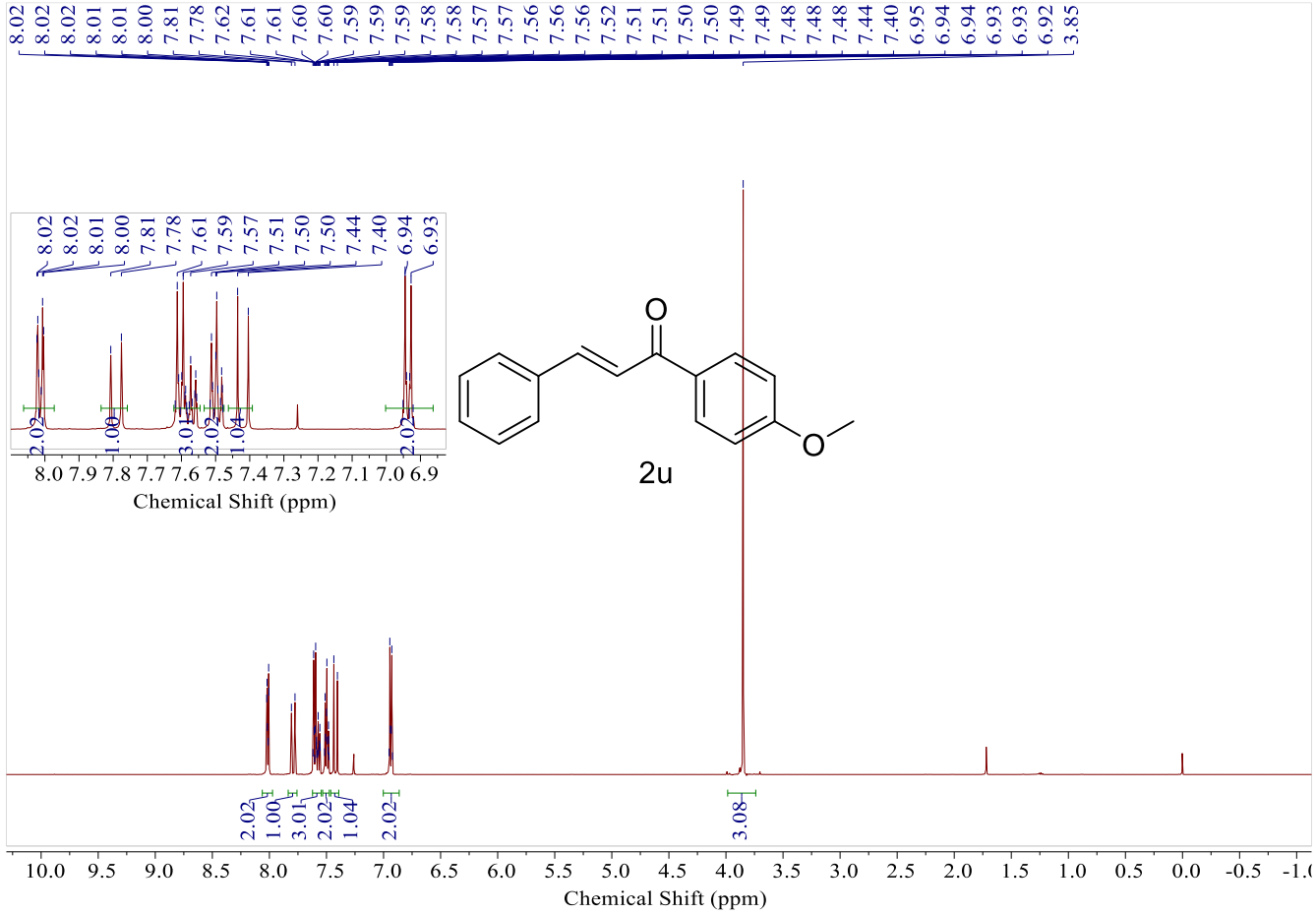
^{1}H NMR of compound **2t**



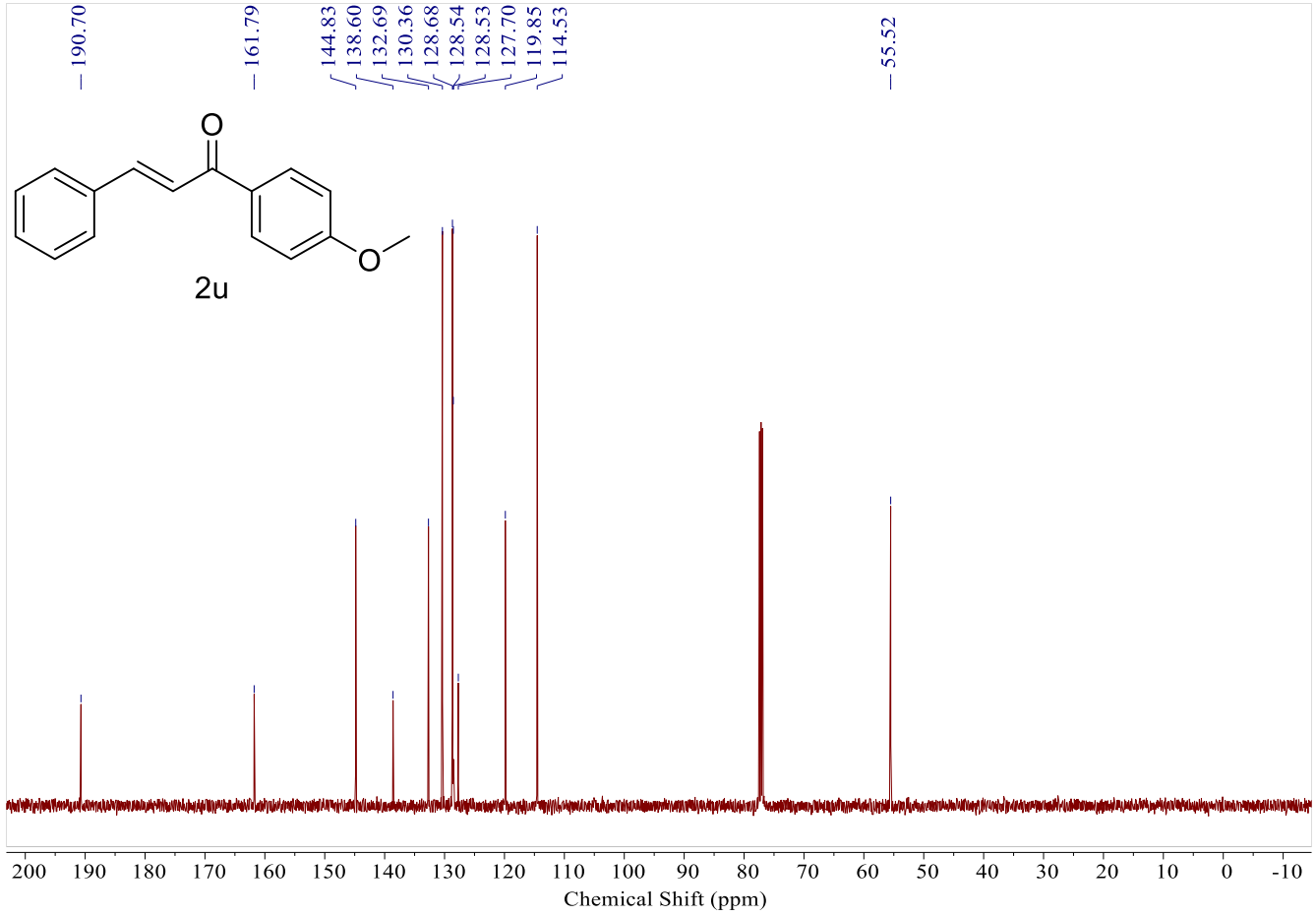
^{13}C NMR of compound **2t**



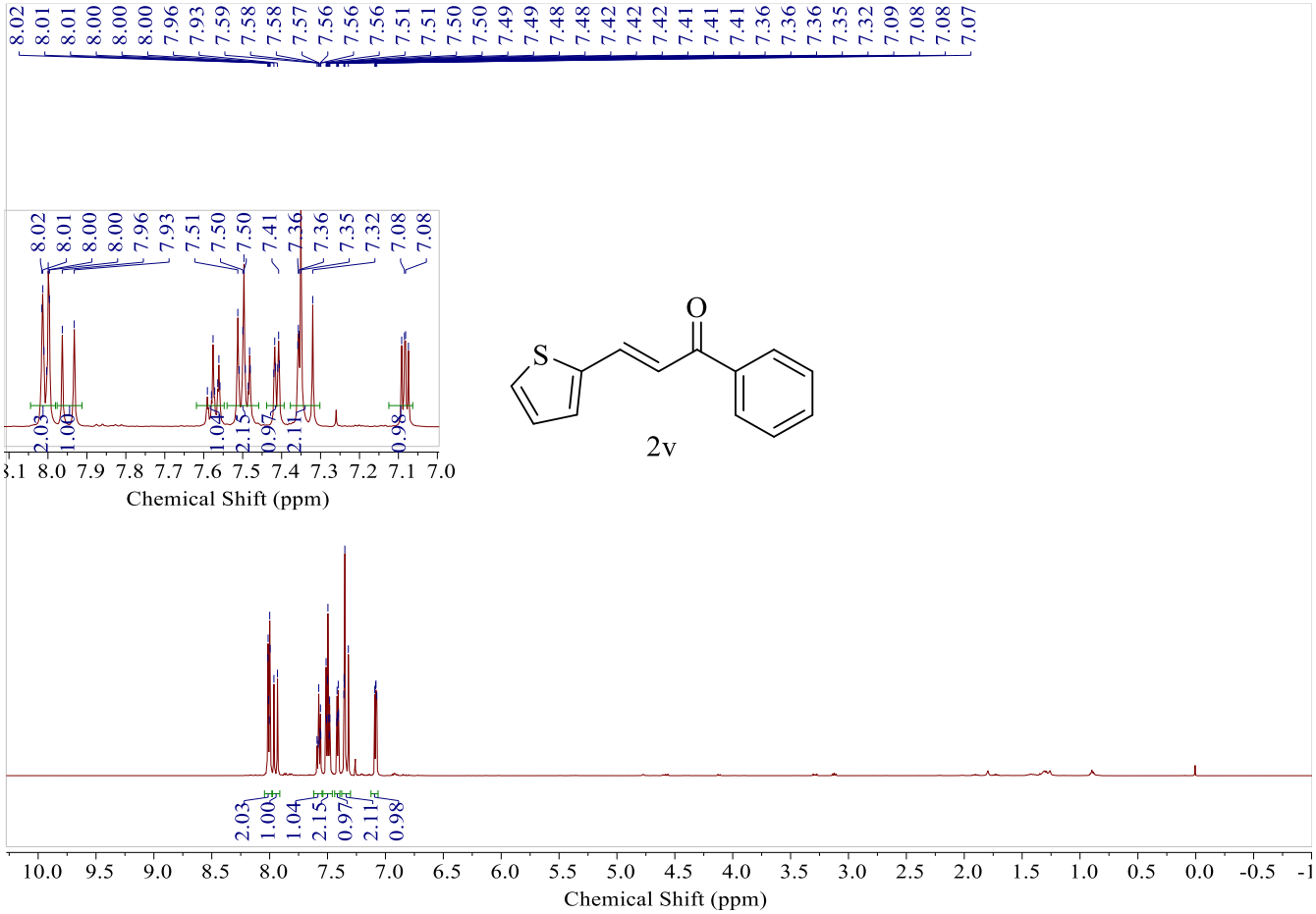
[1]H NMR of compound **2u**



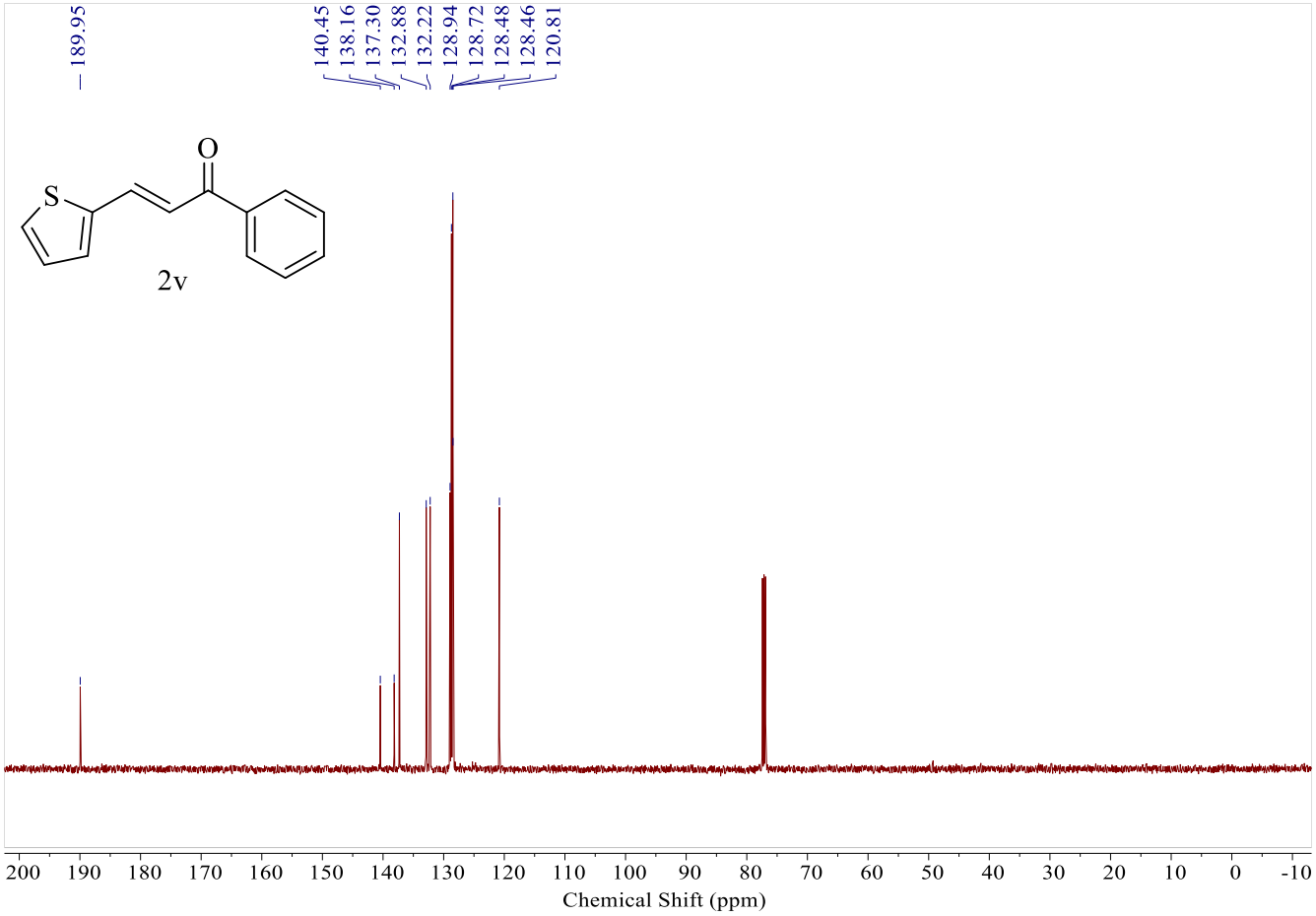
^{13}C NMR of compound **2u**



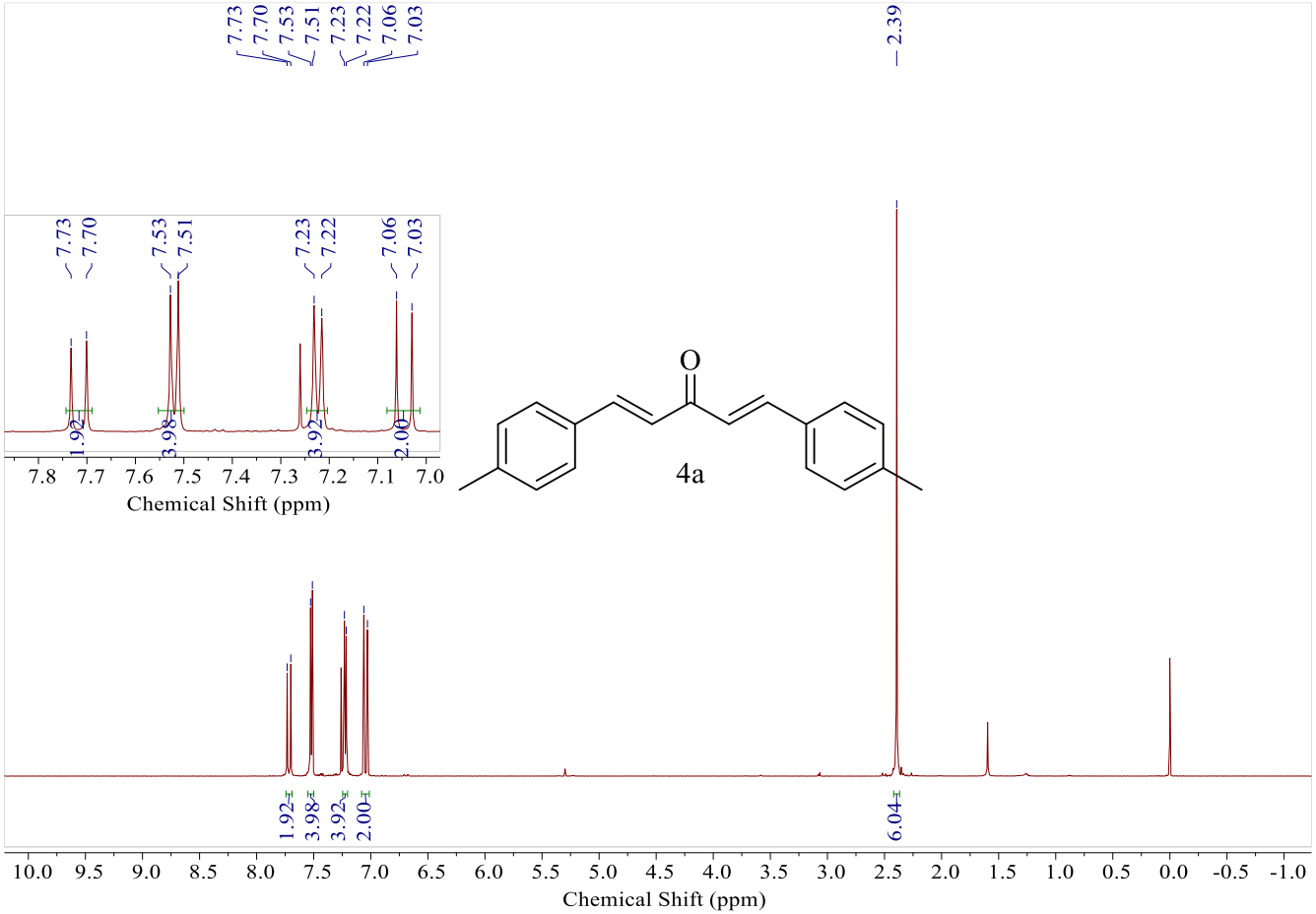
[1]H NMR of compound **2v**



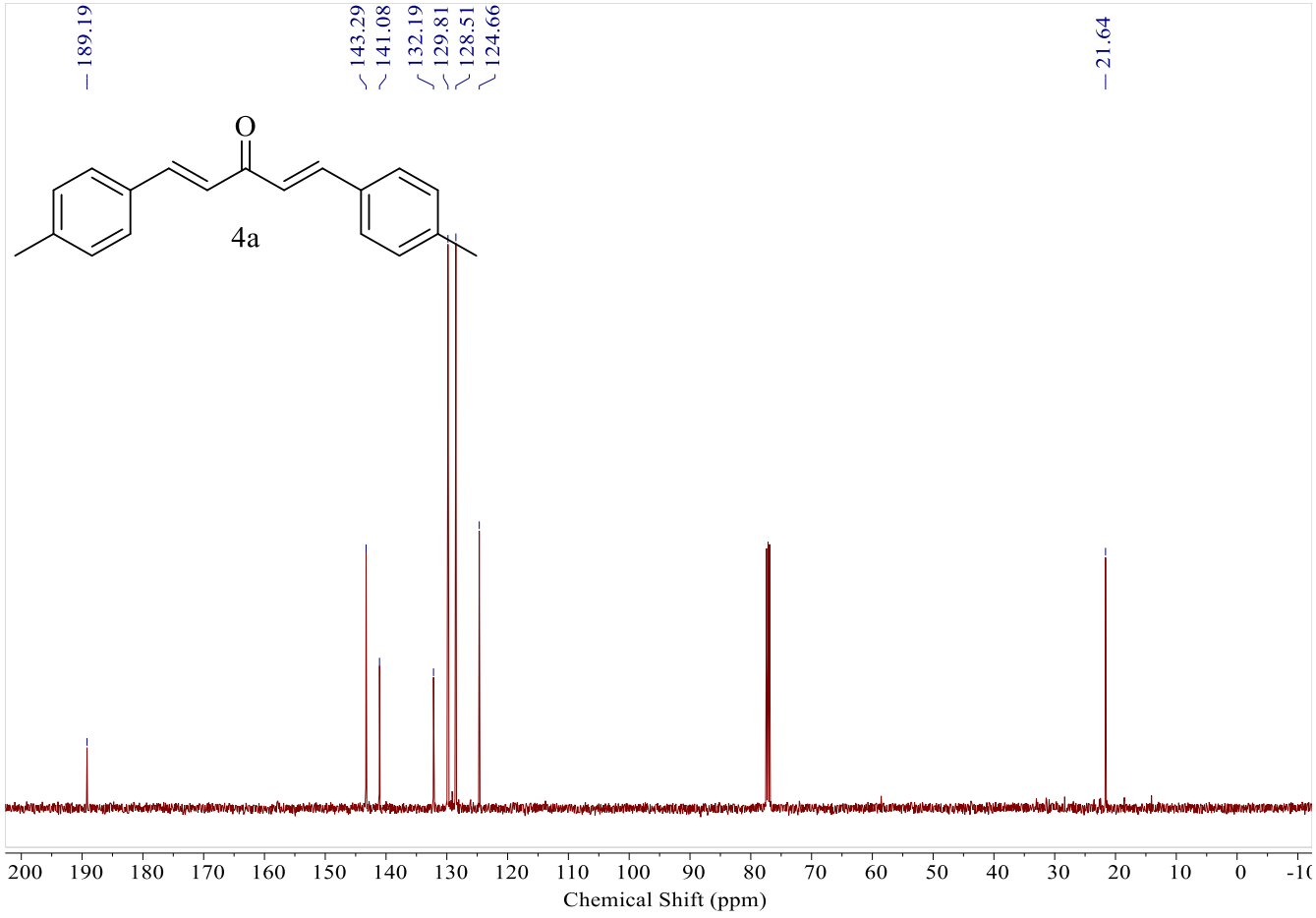
^{13}C NMR of compound **2v**



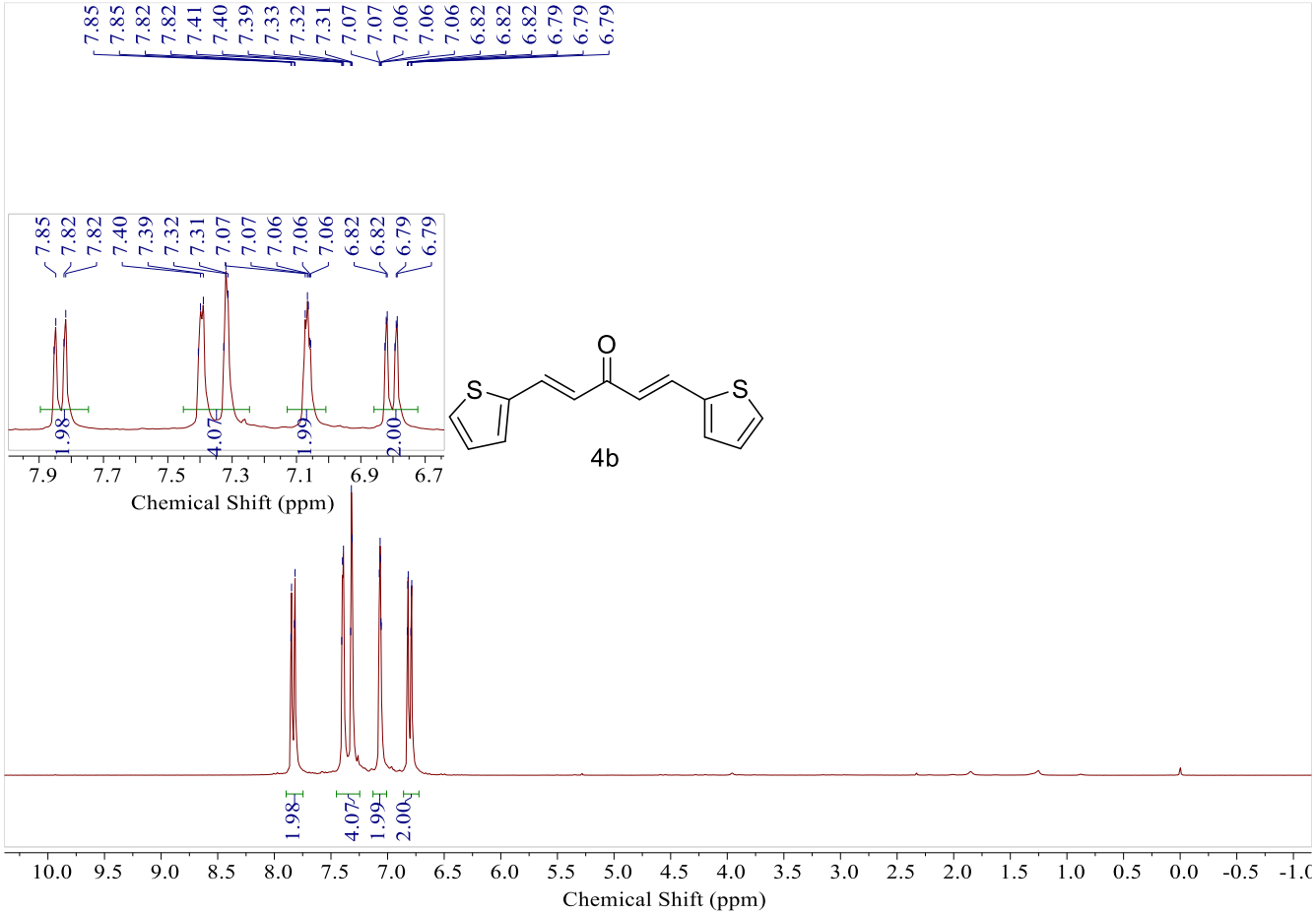
^{1}H NMR of compound **4a**



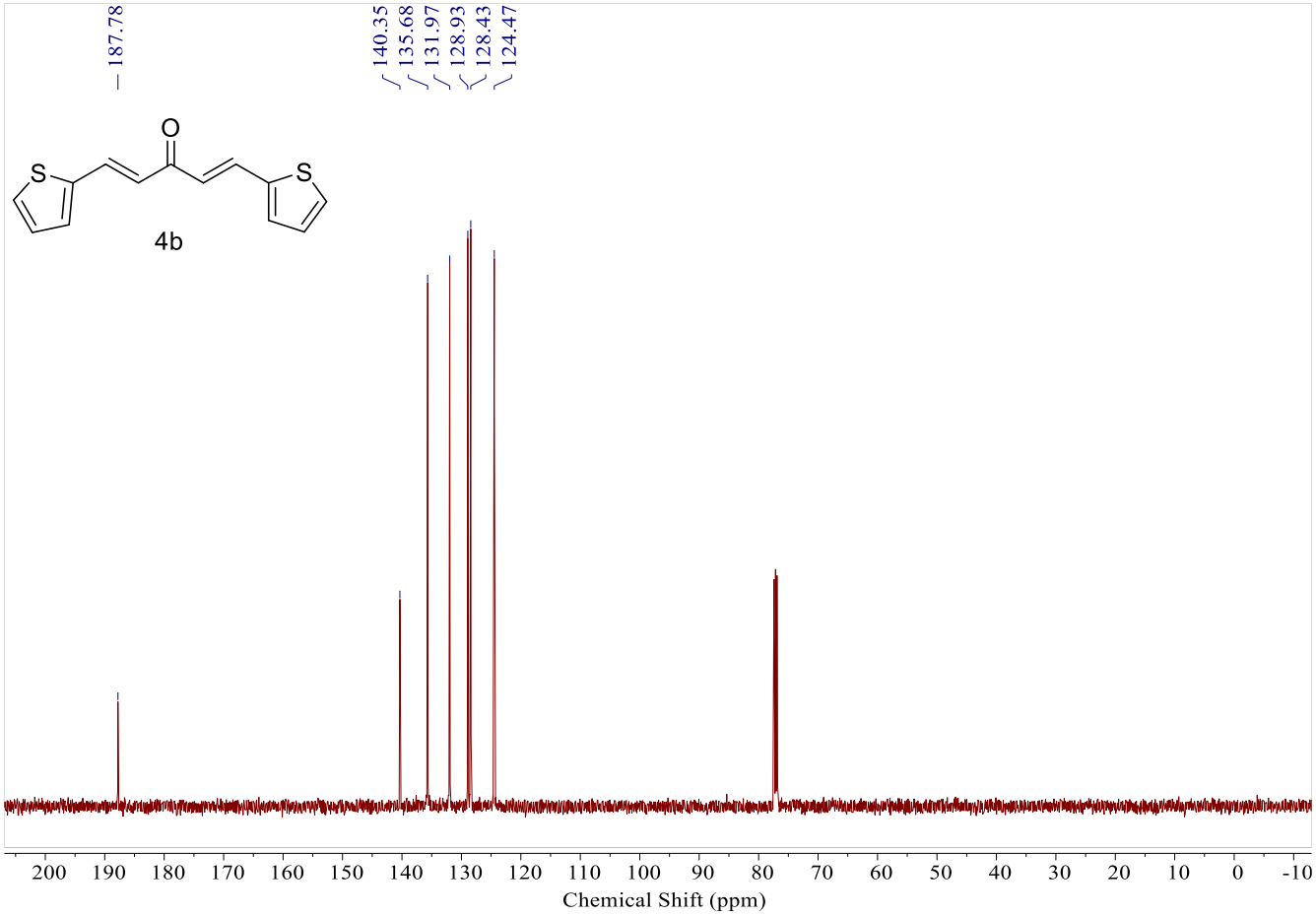
^{13}C NMR of compound **4a**



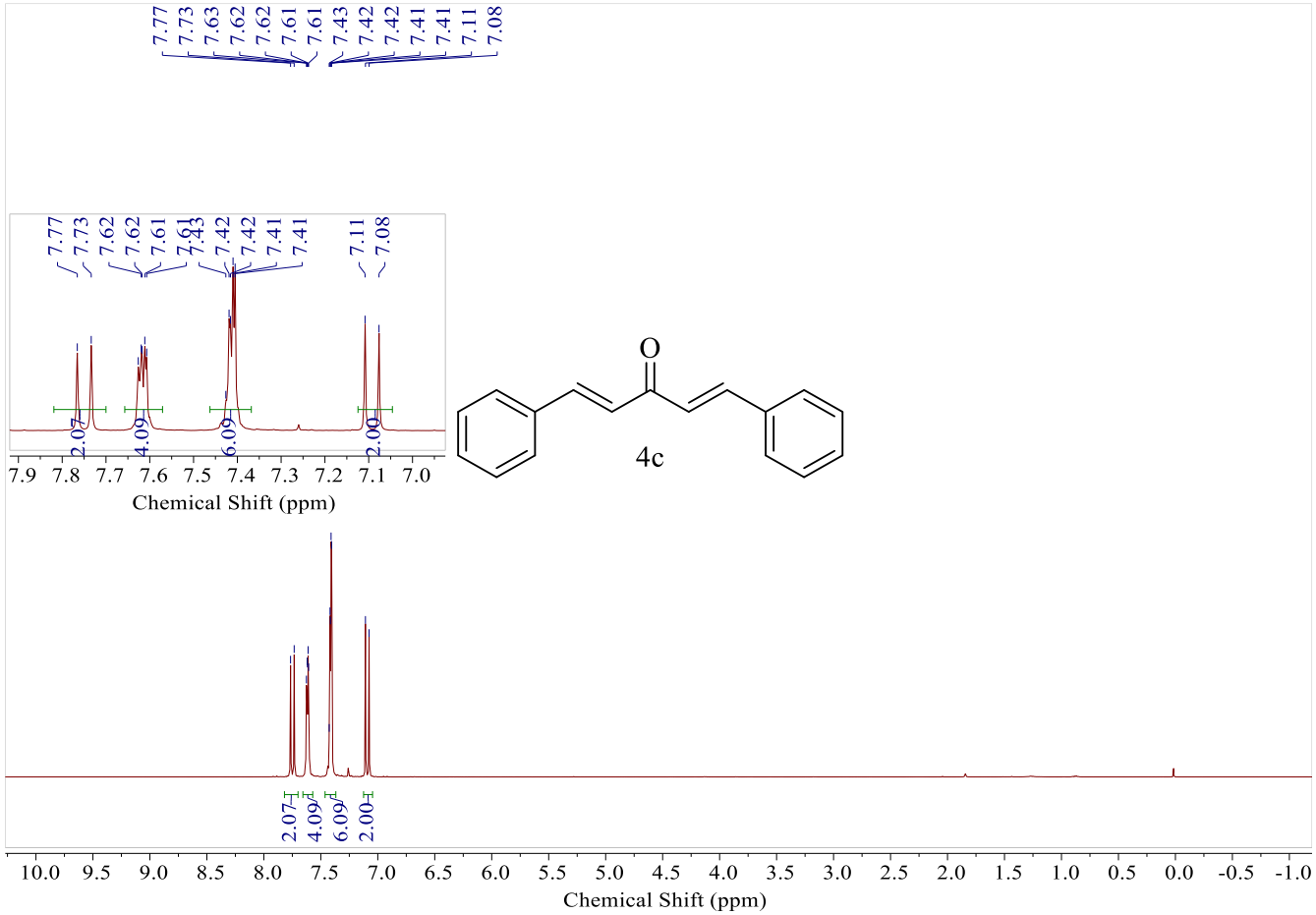
^{1}H NMR of compound **4b**



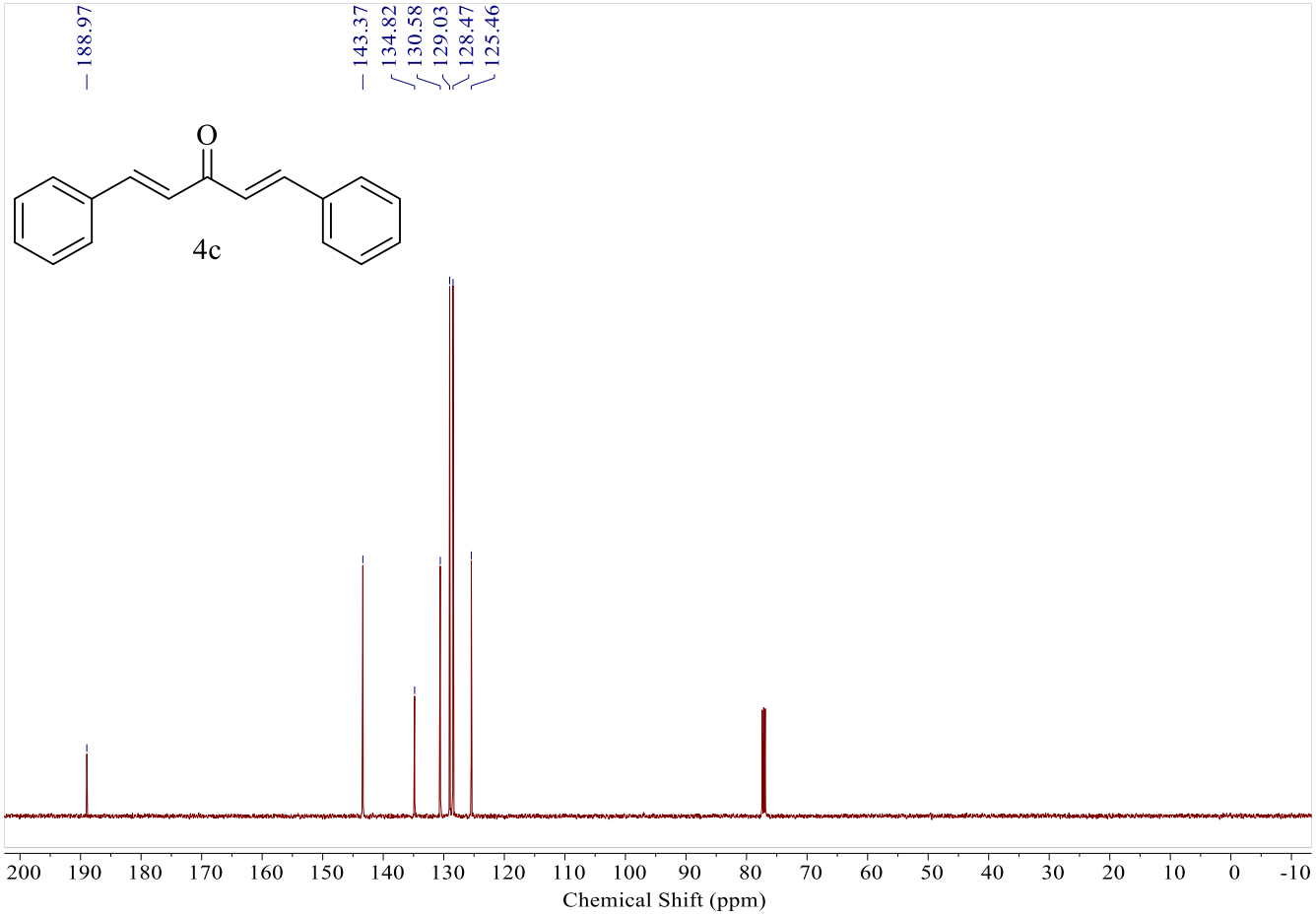
^{13}C NMR of compound **4b**



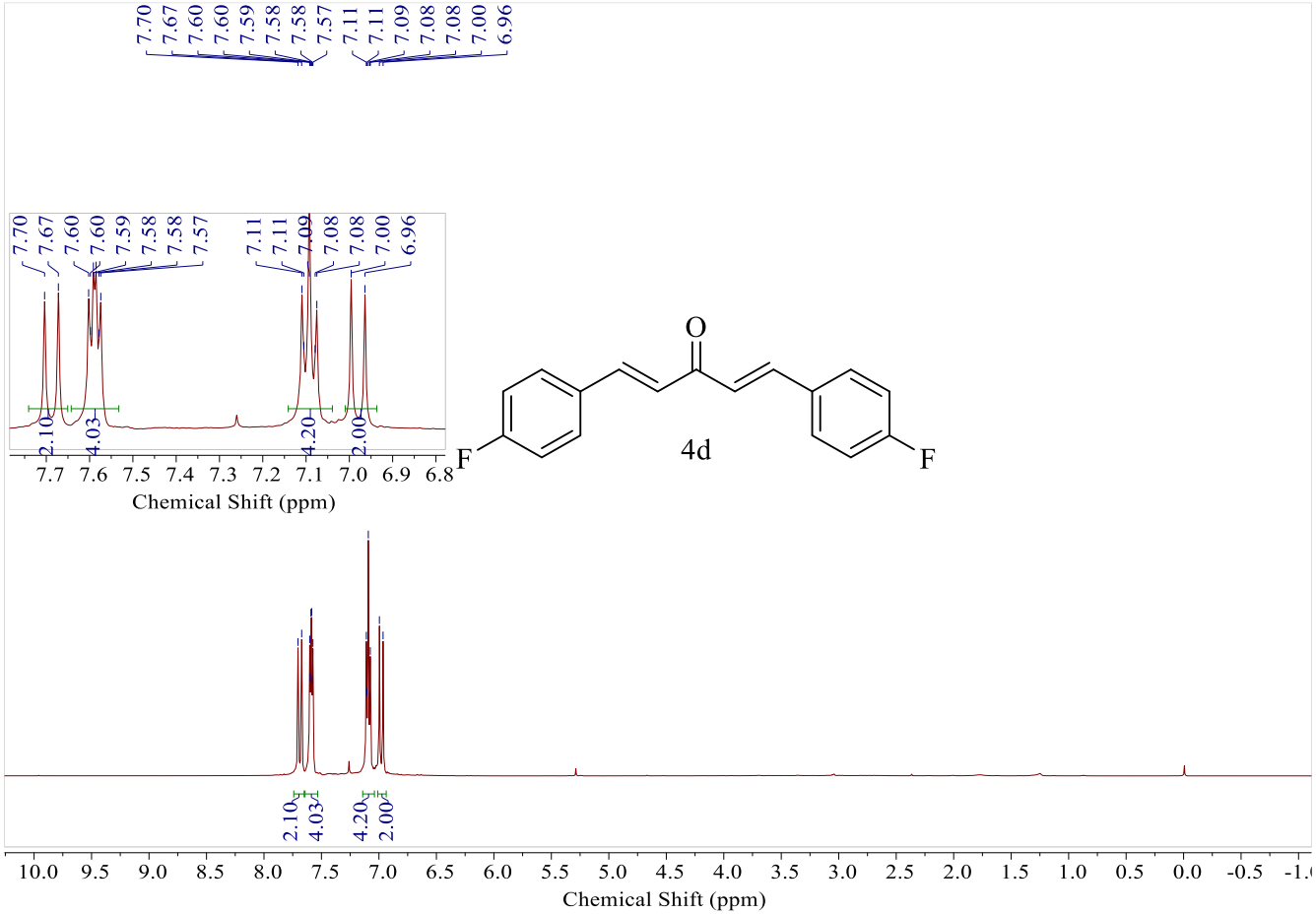
^{1}H NMR of compound **4c**



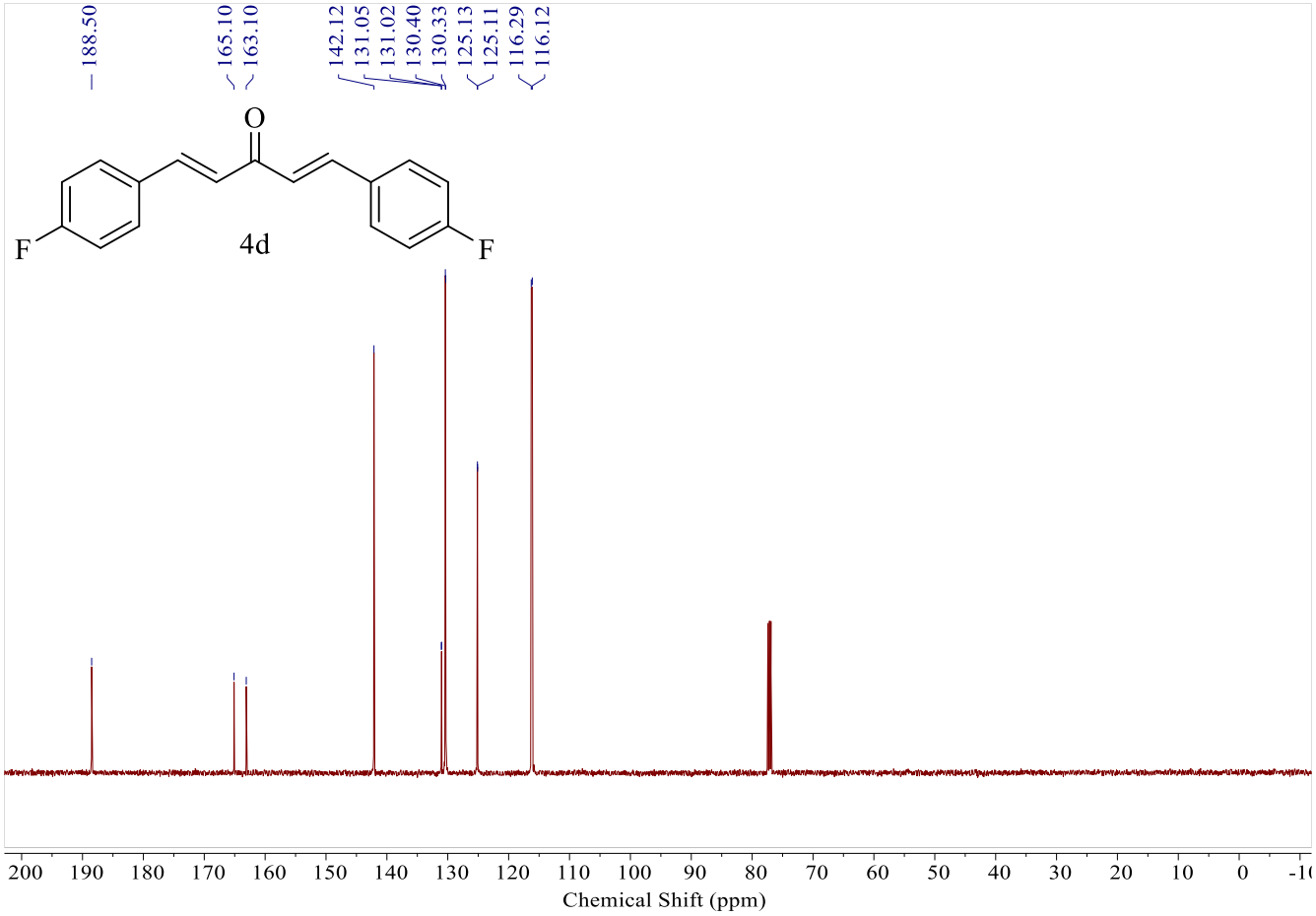
^{13}C NMR of compound **4c**



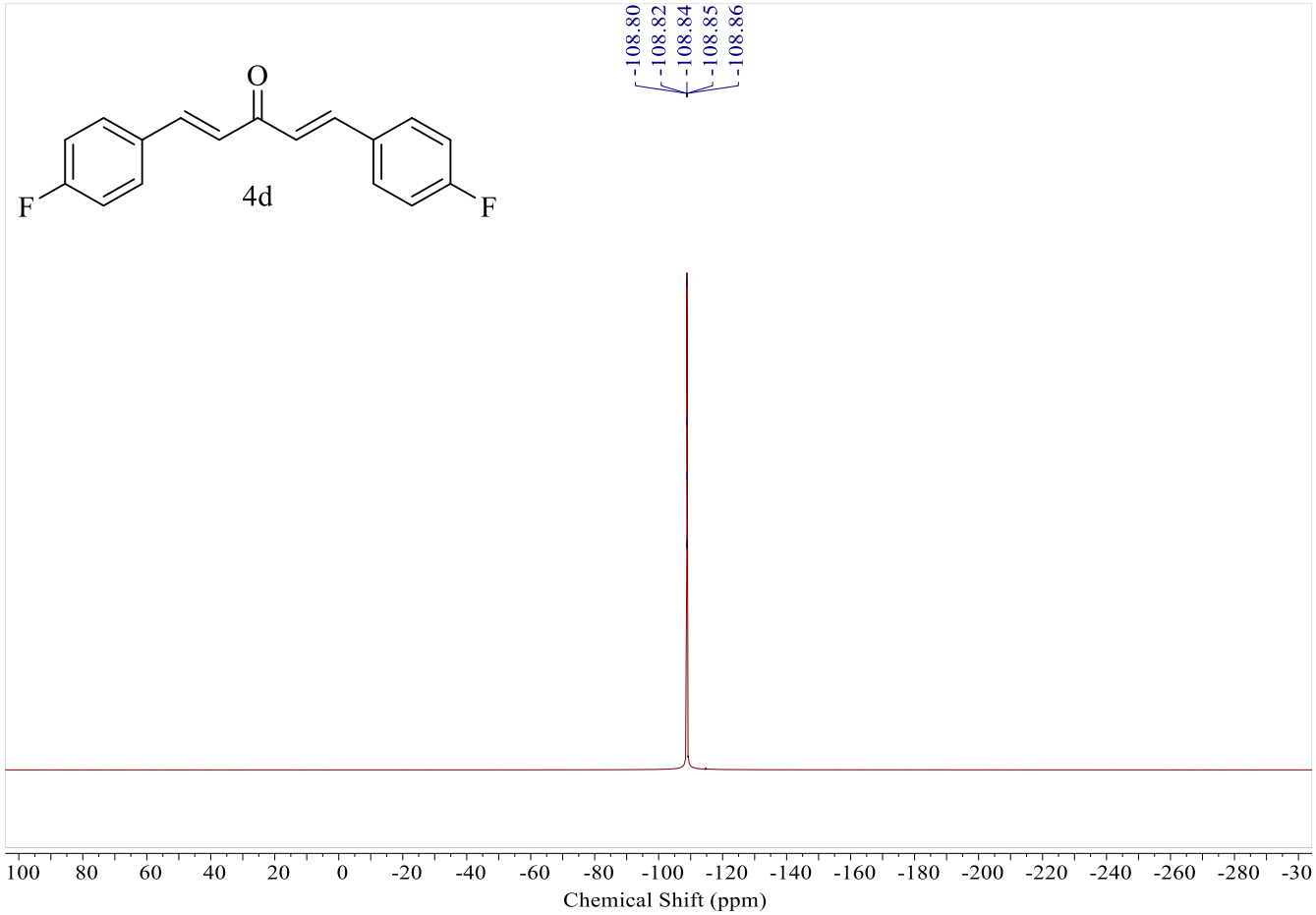
^{1}H NMR of compound **4d**



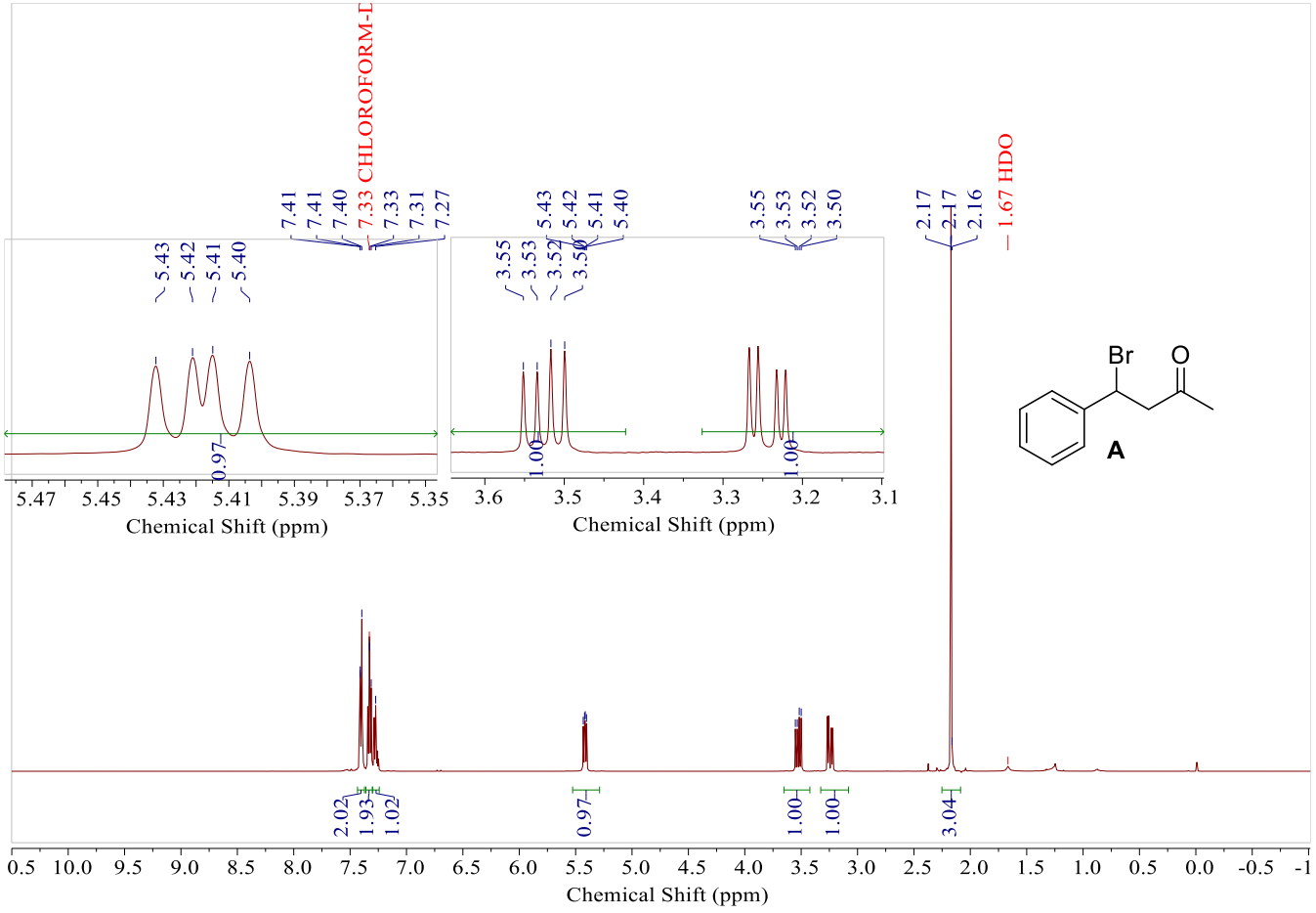
^{13}C NMR of compound **4d**



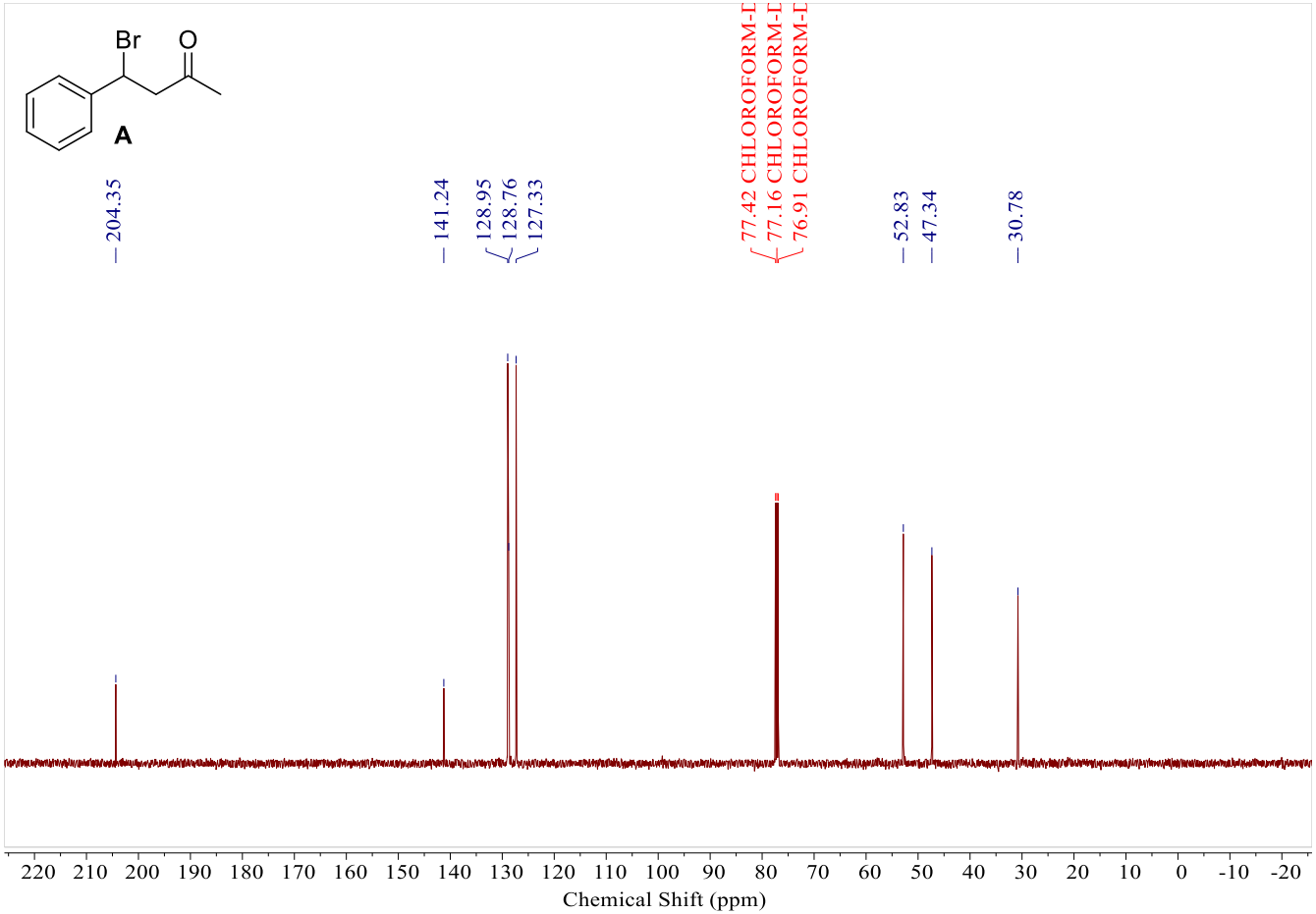
^{19}F NMR of compound **4d**



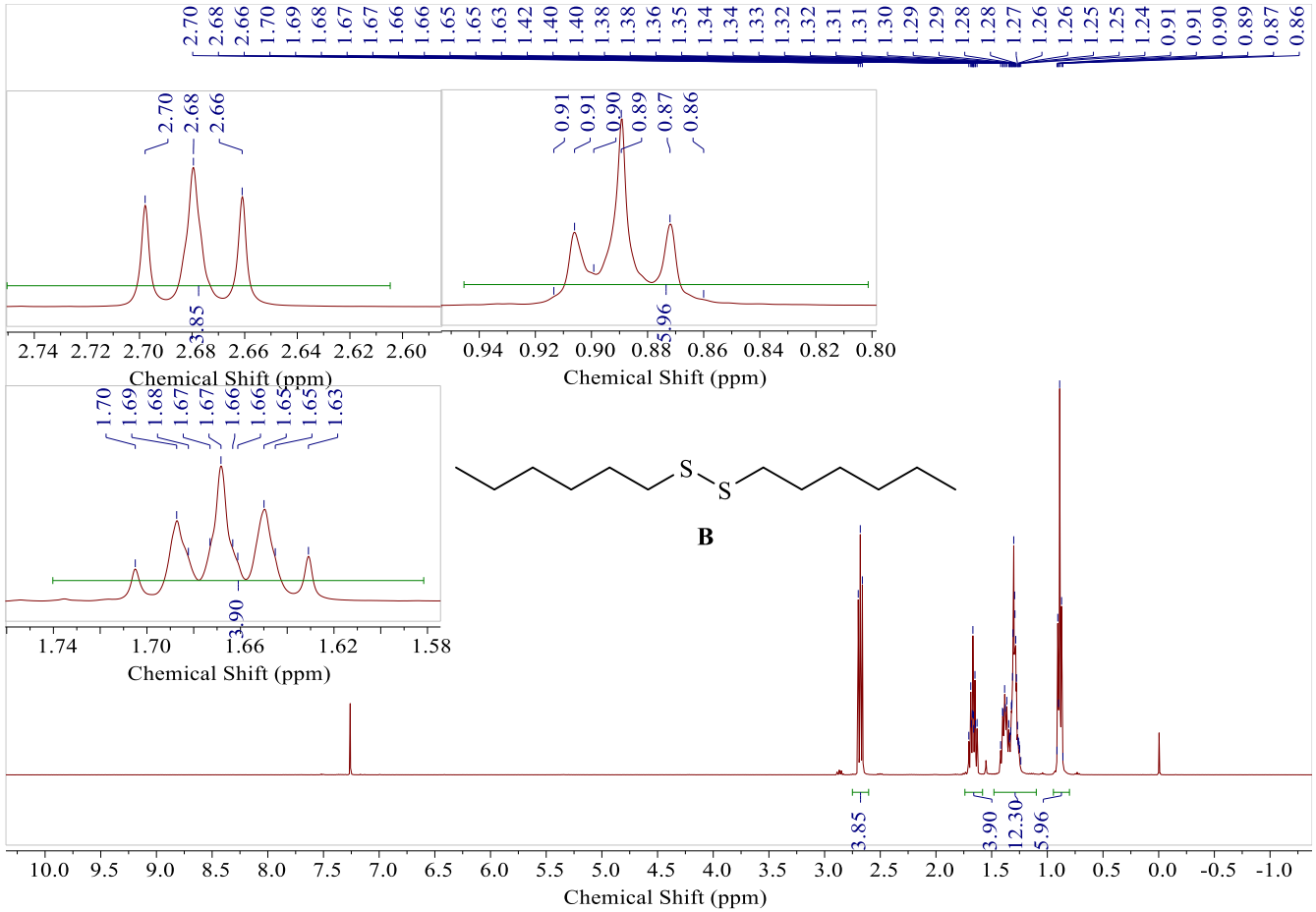
^{1}H NMR of compound **A**



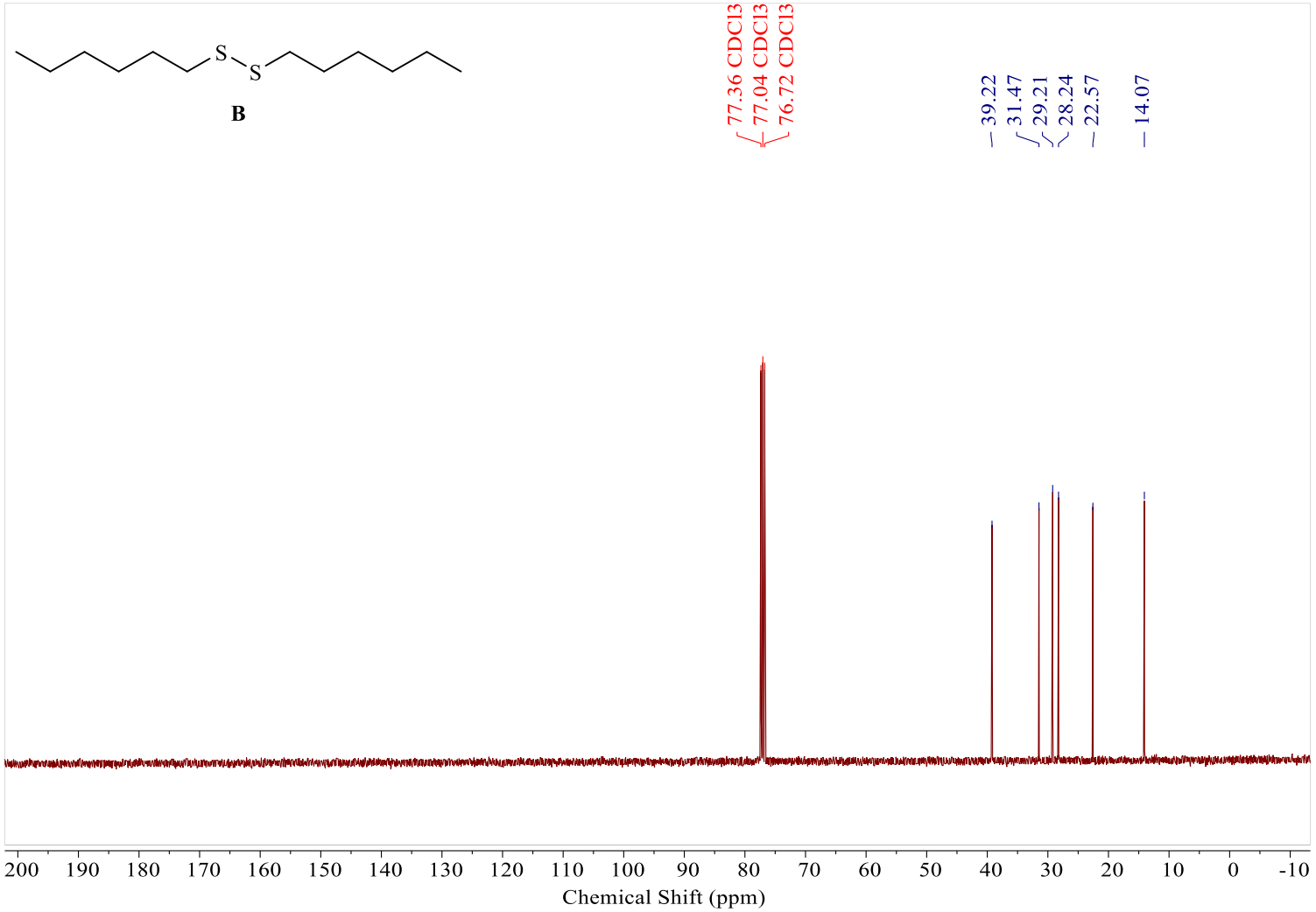
^{13}C NMR of compound **A**

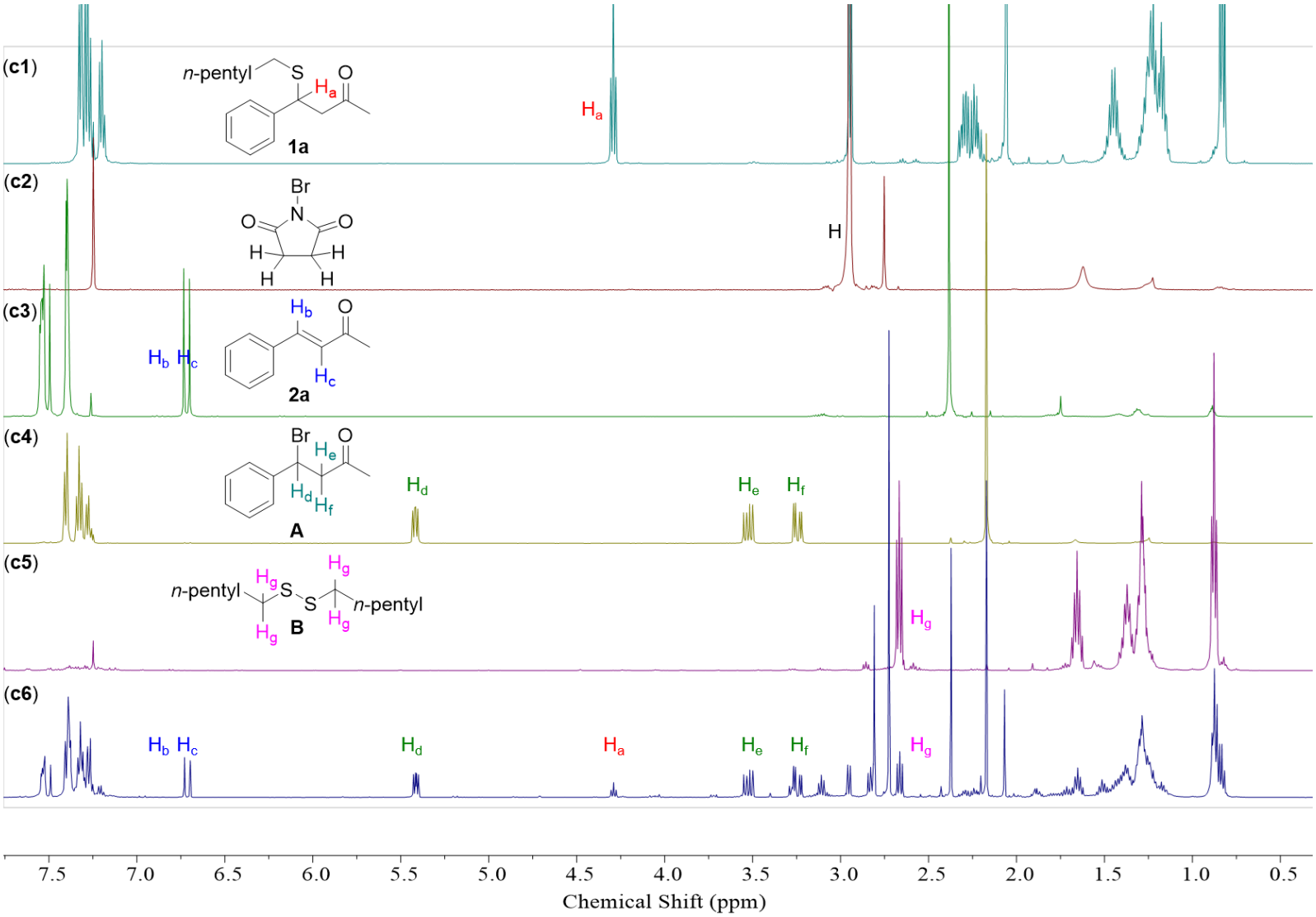


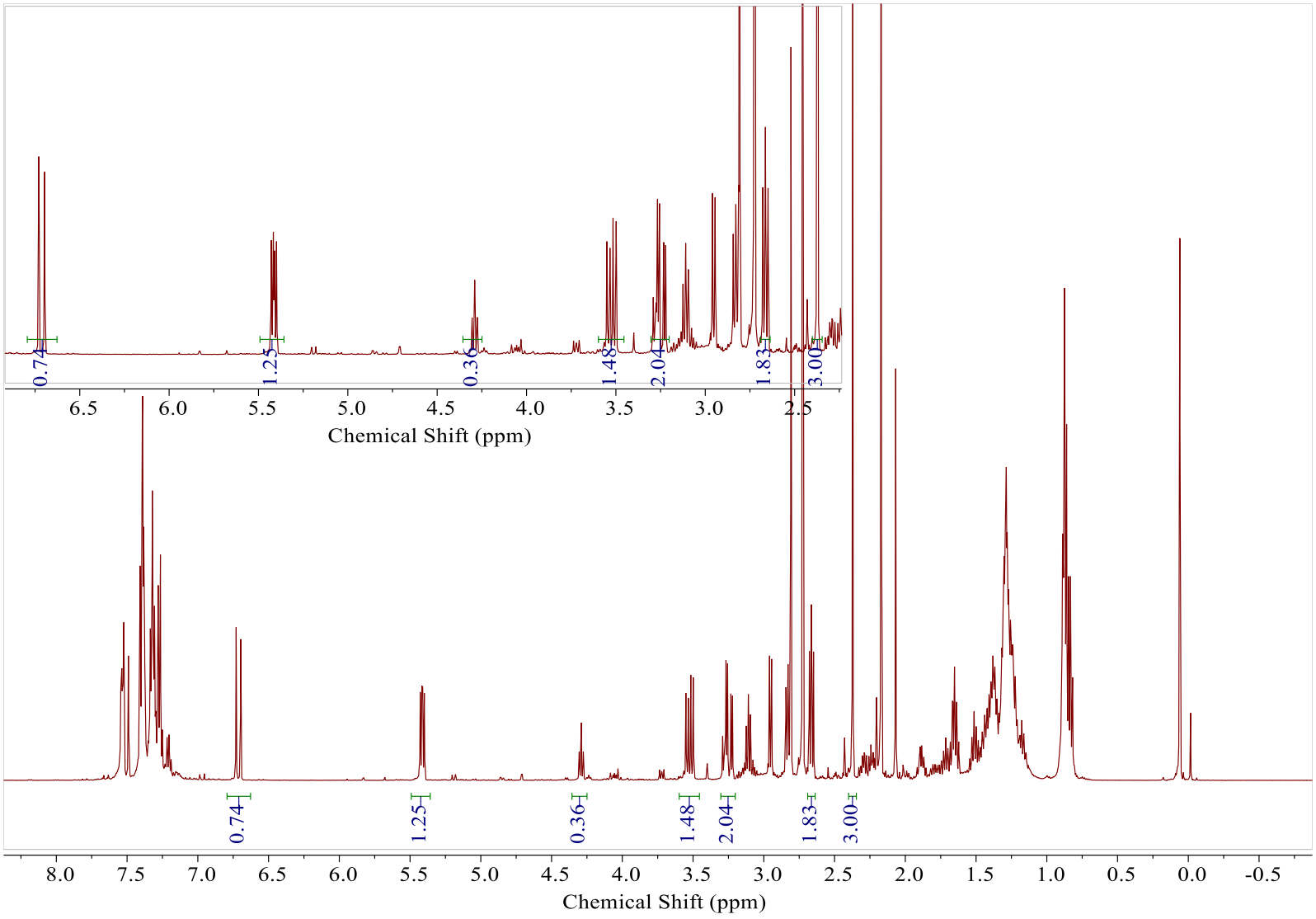
^{1}H NMR of compound **B**



^{13}C NMR of compound **B**




(c1)
n-pentyl
S
O
Ha
1a
Ha
(c2)
Br
N
O
O
H
H
H
H
H
(c3)
Hb
O
Hb Hc
Hc
2a
(c4)
Br
He
O
Hd
Hf
A
Hd
He
Hf
(c5)
Hg
Hg
n-pentyl
S
S
n-pentyl
Hg
Hg
B
Hg
(c6)
Hb Hc
Hd
Ha
He
Hf
Hg
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
Chemical Shift (ppm)


0.74
1.25
0.36
1.48
2.04
1.83
3.00
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
Chemical Shift (ppm)
0.74
1.25
0.36
1.48
2.04
1.83
3.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
Chemical Shift (ppm)