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Photophysical properties of 2′-hydroxychalcones of 2-cinnamoyl-4-nitro-1-naphthol series

Sergey D. Batalin

Experimental Section

General information: TLC was performed on Sorbfil plates and displayed with the ultraviolet lamp (λmax=254 nm), eluent was mixture hexane-ethyl acetate with 5:1 ratio. The IR spectra were measured on FT-IR spectrometer Shimadzu IR Affinity-1S equipped with ATR accessory. NMR spectra were recorded on a Bruker Avance-III spectrometer (^{1}H NMR 400 MHz and ^{13}C NMR at 100 MHz) equipped with a BBO probe. Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual deuterium in the solvent (^{1}H NMR: $CDCl_3$ at 7.26 ppm). High-resolution mass spectra were recorded with a Bruker Maxis spectrometer (TOF, electrospray ionization, in MeCN solution, using HCO_2Na-HCO_2H for calibration). Melting points were measured with Stuart SMP30 apparatus. UV/Vis spectra were recorded on UV-spectrophotometer Hitachi-2900. Excitation and emission spectra in solution were recorded on FluoroMax-4P spectrofluorometer. The excitation gap was 10, the emission gap was 10 for solid state. The excitation gap was 1, the emission gap was 1 for solutions of compound **3j**. Solvents were hexane, toluene, tetrachloromethane, chloroform, dichloromethane, ethyl acetate, THF, methanol, ethanol, acetonitrile and DMSO with a sample concentration of 1×10^{-6} mol dm^{-3}. All reactions were performed in air with constant magnetic stirring. 4-nitro-1-hydroxy-2-acetonaphthone was prepared by known method.[S1] For systems **3a**,**c**,**e**,**i** ^{1}H and ^{13}C NMR spectra correspond to those previously presented.[S2] The relative fluorescence quantum yield for compound **3j** in solution was calculated using Rhodamine 6G in EtOH as a standard (λ_{ex}=480 nm, QY=95%)

General procedure[S2] for the preparation of 3: A mixture of 1-hydroxy-4-nitro-2-acetonaphthone **1** (2.31 g, 10 mmol), aromatic aldehyde **2** (13 mmol), sodium hydroxide (1.68 g, 30 mmol) and PEG-400 (20 mL) was stirred at 100°C for 60 min. The reaction was monitored by thin-layer chromatography. After completion of the reaction, the reaction mixture was cooled; mixture water-acetic acid (5:1, 100 mL) was added to the reaction mixture, and this was stirred for 30 min. The resulting solid product was filtered, rinsed with ice cold water (50 mL) and twice recrystallized first from EtOH-THF mixture (at 3:1 ratio), second from acetic acid.

(*E*)-1-(1-Hydroxy-4-nitronaphthalen-2-yl)-3-phenylprop-2-en-1-one (**3a**).
Yellow-orange solid, yield 89%, 2.84 g; m.p.= 210.3-211.6°C (210-212°C).[S2] FTIR (ν_{max}, cm^{-1}): 3362, 3001, 1626, 1589, 1579, 1505. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 15.55 (s, 1H), 8.95 (s, 1H), 8.80 (d, *J* = 8.7 Hz, 1H), 8.63 (d, *J* = 8.4 Hz, 1H), 8.09 (d, *J* = 15.3 Hz, 1H), 7.89 (t, *J* = 7.8 Hz, 1H), 7.78-7.43 (m, 4H), 7.52-7.45 (m, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 192.6, 168.6, 147.6, 134.0, 133.3, 131.6, 129.2, 129.2, 129.1, 127.7, 127.3, 125.9, 125.4, 125.1, 123.9, 118.7, 111.1. HRMS (ESI TOF) *m/z*: (M - H)$^-$ calculated for $C_{19}H_{12}NO_4$ 318.0772; Found 318.0756.

(*E*)-1-(1-Hydroxy-4-nitronaphthalen-2-yl)-3-(2-methoxyphenyl)prop-2-en-1-one (**3b**).
Yellow-orange solid, yield 88%, 3.07 g; m.p.= 234.9-236.2°C. FTIR (ν_{max}, cm^{-1}): 3352, 3064, 2921, 1643, 1576, 1509. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 15.75 (s, 1H), 9.01 (s, 1H) 8.85 (d, *J* =

8.8 Hz, 1H), 8.66 (d, *J* = 8.5 Hz, 1H), 8.40 (d, *J* = 15.5 Hz, 1H), 7.96-7.87 (m, 2H), 7.78-7.68 (m, 2H), 7.49 (t, *J* = 8.0 Hz, 1H), 7.30-7.25 (m, 2H), 7.10 (t, *J* = 7.6 Hz, 1H), 7.03 (d, *J* = 8.4 Hz, 1H), 4.02 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 193.3, 168.6, 156.7, 143.3, 133.2, 133.0, 130.4, 129.5, 127.3, 126.1, 125.8, 125.2, 123.9, 121.0, 119.4, 111.4, 55.7 HRMS (ESI TOF) *m/z*: (M - H)$^-$ calculated for $C_{20}H_{14}NO_5$ 348.0877; Found 348.0861.

(*E*)-1-(1-Hydroxy-4-nitronaphthalen-2-yl)-3-(4-methoxyphenyl)prop-2-en-1-one (**3c**).
Yellow-orange solid, yield 85%, 2.96 g; m.p. = 219.4-220.6°C (220-221°C).[S2] FTIR (ν_{max}, cm^{-1}): 3350, 3071, 2945, 1635, 1580, 1527. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 15.77 (s, 1H), 8.95 (s, 1H), 8.81 (d, *J* = 8.2 Hz, 1H), 8.62 (d, *J* = 8.4 Hz, 1H), 8.06 (d, *J* = 15.2 Hz, 1H), 7.88 (t, *J* = 7.8 Hz, 1H), 7.76-7.64 (m, 3H), 7.58 (d, *J* = 15.2 Hz, 1H), 7.00 (d, *J* = 7.7 Hz, 1H), 3.89 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 192.5, 168.8, 162.6, 147.5, 133.2, 131.2, 129.4, 129.1, 127.3, 126.8, 125.5, 125.1, 123.8, 116.0, 114.7, 55.5. HRMS (ESI TOF) *m/z*: (M - H)$^-$ calculated for $C_{20}H_{14}NO_5$ 348.0877; Found 348.0858.

(*E*)-3-(2,3-Dimethoxyphenyl)-1-(1-hydroxy-4-nitronaphthalen-2-yl)prop-2-en-1-one (**3d**).
Yellow-orange, yield 84%, 3.18 g; m.p. = 274.6-275.8°C. FTIR (ν_{max}, cm^{-1}): 3321, 3052, 2909, 1621, 1568, 1507. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 15.64 (s, 1H), 8.99 (s, 1H), 8.84 (d, *J* = 8.7 Hz, 1H), 8.66 (d, *J* = 8.4 Hz, 1H), 8.41 (d, *J* = 15.5 Hz, 1H), 7.92 (t, *J* = 7.8 Hz, 1H), 7.84 (d, *J* = 15.5 Hz, 1H), 7.72 (t, *J* = 7.6 Hz, 1H), 7.41 (d, *J* = 7.8 Hz, 1H), 7.20 (t, *J* = 8.0 Hz, 1H), 7.08 (d, *J* = 7.7 Hz, 1H), 3.97 (d, *J* = 17.3 Hz, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 193.0, 165.6, 153.2, 148.5, 142.5, 133.3, 127.3, 125.6, 125.2, 124.4, 123.9, 120.1, 120.0, 115.3, 61.5, 55.9. HRMS (ESI TOF) *m/z*: (M - H)$^-$ calculated for $C_{21}H_{16}NO_6$ 378.0973; Found 378.0959.

(*E*)-3-(3,4-Dimethoxyphenyl)-1-(1-hydroxy-4-nitronaphthalen-2-yl)prop-2-en-1-one (**3e**).
Orange solid, yield 79%, 3.00 g; m.p. = 231.5.-232.6°C (232-233°C).[S2] FTIR (ν_{max}, cm^{-1}): 3331, 3071, 2941, 1638, 1598, 1574, 1511. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, J/Hz): 15.78 (s, 1H), 8.94 (s, 1H), 8.79 (d, *J* = 8.7 Hz, 1H), 8.61 (d, *J* = 8.3 Hz, 1H), 8.04 (d, *J* = 15.2 Hz, 1H), 7.88 (t, *J* = 7.8 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.54 (d, *J* = 15.1 Hz, 1H), 7.35 (d, *J* = 8.2 Hz, 1H), 7.22 (s, 1H), 6.95 (d, *J* = 8.2 Hz, 1H), 4.01 (s, 3H), 3.96 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 192.4, 168.6, 152.5, 149.4, 147.8, 137.1, 133.2, 129.1, 127.3, 127.1, 125.9, 125.4, 125.1, 124.4, 123.8, 116.1, 111.2, 111.1, 110.6, 56.1, 56.0. HRMS (ESI TOF) *m/z*: (M - H)$^-$ calculated for $C_{21}H_{16}NO_6$ 378.0983; Found 378.0956.

(*E*)-1-(1-Hydroxy-4-nitronaphthalen-2-yl)-3-(2,3,4-trimethoxyphenyl)prop-2-en-1-one (**3f**).
Orange solid, yield 83%, 3.39 g; m.p. = 171.8-172.9°C. FTIR (ν_{max}, cm^{-1}): 3371, 3092, 2936, 1632, 1577, 1516. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 15.74 (s, 1H), 8.90 (s, 1H), 8.75 (d, *J* = 8.7 Hz, 1H), 8.56 (d, *J* = 8.1 Hz, 1H), 8.21 (d, *J* = 15.4 Hz, 1H), 7.81 (t, *J* = 7.1 Hz, 1H), 7.70 (d, *J* = 15.4 Hz, 1H), 7.61 (t, *J* = 7.1 Hz, 1H), 7.44 (d, *J* = 8.8 Hz, 1H), 6.72 (d, *J* = 8.8 Hz, 1H), 3.97 (s, 3H), 3.89 (s, 3H), 3.85 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 191.9, 167.6, 158.3, 155.9, 153.4, 142.0, 136.0, 132.1, 128.1, 126.2, 124.9, 124.6, 124.1, 123.8, 122.8, 120.2, 116.5, 110.2, 106.6, 60.5, 59.9, 55.1. HRMS (ESI TOF) *m/z*: (M - H)$^-$ calculated for $C_{22}H_{18}NO_7$ 408.1089; Found 408.1077.

(*E*)-1-(1-Hydroxy-4-nitronaphthalen-2-yl)-3-(2,4,5-trimethoxyphenyl)prop-2-en-1-one (**3g**).
Orange solid, yield 79%, 3.23 g; m.p. = 215.3-216.4°C. FTIR (ν_{max}, cm^{-1}): 3391, 3051, 2921 1642, 1594, 1527. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 15.99 (s, 1H), 8.93 (s, 1H), 8.78 (d, *J* = 8.4 Hz, 1H), 8.59 (d, *J* = 8.2 Hz, 1H), 8.32 (d, *J* = 15.5 Hz, 1H), 7.85 (t, *J* = 7.8 Hz, 1H), 7.69 – 7.61 (m, 2H), 7.12 (s, 1H), 6.50 (s, 1H), 3.98 – 3.87 (m, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 192.8, 168.6, 155.8, 153.7, 143.4, 143.1, 136.9, 132.9, 129.0, 127.1, 125.9, 125.7, 125.0, 123.7, 116.1, 114.7, 112.1, 111.3, 56.6, 56.2, 56.1. HRMS (ESI TOF) *m/z*: (M - H)$^-$ calculated for $C_{22}H_{18}NO_7$ 408.1089; Found 408.1057.

(*E*)-1-(1-Hydroxy-4-nitronaphthalen-2-yl)-3-(2,4,6-trimethoxyphenyl)prop-2-en-1-one (**3h**).
Yellow-orange solid, yield 93%, 3.80 g; m.p. = 276.9-277.8°C. FTIR (ν_{max}, cm^{-1}): 3309, 3052, 2931, 1625, 1576, 1521. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 16.22 (s, 1H), 9.03 (s, 1H), 8.85 (d, *J* = 8.8 Hz, 1H), 8.65 (d, *J* = 8.3 Hz, 1H), 8.58 (d, *J* = 15.4 Hz, 1H), 8.15 (d, *J* = 15.4 Hz, 1H), 7.89 (t, *J* = 7.1 Hz, 1H), 7.69 (t, *J* = 7.3 Hz, 1H), 6.19 (s, 2H), 4.02 (s, 6H), 3.92 (s, 3H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 189.8, 162.9, 152.0, 150.2, 139.0, 138.8, 137.4, 132.8, 127.0, 126.3, 126.0, 125.1, 124.9, 123.8, 120.4, 117.7, 90.6, 56.0, 55.5. HRMS (ESI TOF) *m/z*: $(M - H)^-$ calculated for $C_{22}H_{18}NO_7$ 408.1089; Found 408.1082.

(*E*)-1-(1-Hydroxy-4-nitronaphthalen-2-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one (**3i**).
Dark orange solid, yield 72%, 2.95 g; m.p.= 173.6-175.2°C (173-175°C).[S2] FTIR (ν_{max}, cm^{-1}): 3220, 3007, 2949, 2928, 1624, 1587, 1504. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, J/Hz): 15.65 (s, 1H), 8.91 (s, 1H), 8.76 (d, *J* = 8.7 Hz, 1H), 8.60 (d, *J* = 8.3 Hz, 1H), 7.99 (d, *J* = 15.2 Hz, 1H), 7.87 (t, *J* = 7.9 Hz, 1H), 7.68 (t, *J* = 7.6 Hz, 1H), 7.55 (d, *J*= 15.2 Hz, 1H), 6.94 (s, 2H), 4.00-3.91 (m, 9H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 192.4, 168.6, 153.3, 147.8, 141.5, 137.2, 133.2, 129.4, 129.1, 127.3, 125.8, 125.1, 123.8, 117.6, 111.0, 106.0, 61.0, 56.3. HRMS (ESI TOF) *m/z*: $(M - H)^-$ calculated for $C_{22}H_{18}NO_7$ 408.1089; Found 408.1066.

(*E*)-3-(4-Dimethylaminophenyl)-1-(1-hydroxy-4-nitronaphthalen-2-yl)prop-2-en-1-one (**3j**).
Dark red solid, yield 67%, 2.43 g; m.p.= 250.5-251.3°C. FTIR (ν_{max}, cm^{-1}): 3402, 3092, 2971 1649, 1584, 1527. ^{1}H NMR ($CDCl_3$, 400 MHz, δ, ppm, *J*/Hz): 16.27 (s, 1H), 9.00 (s, 1H), 8.85 (d, *J* = 8.7 Hz, 1H), 8.64 (d, *J* = 8.3 Hz, 1H), 8.10 (d, *J* = 14.9 Hz, 1H), 7.89 (t, *J* = 7.8 Hz, 1H), 7.73 – 7.64 (m, 3H), 7.50 (d, *J* = 14.9 Hz, 1H), 6.75 (d, *J* = 8.8 Hz, 2H), 3.12 (s, 6H). ^{13}C NMR ($CDCl_3$, 100 MHz, δ, ppm): 189.4, 166.2, 150.3, 146.1, 130.3, 129.1, 127.6, 126.5, 124.5, 123.5, 123.2, 122.5, 121.3, 119.3, 109.8, 109.2, 108.9, 37.5. HRMS (ESI TOF) *m/z*: $(M + H)^+$ calculated for $C_{21}H_{19}N_2O_4$ 363.1339; Found 363.1331.

Table S1. Photophysical properties of compounds **3a-j** in the solid state.

Compound	**λex**	**λem**	Stokes shift, nm
3a	470	600	130
3b	470	602	132
3c	470	614	144
3d	460	602	142
3e	470	624	154
3f	470	604	134
3g	470	626	156
3h	470	600	130
3i	460	602	142
3j	470	672	202

Table S2. Photophysical properties of compound **3j** in various solvent at a concentration of sample 1×10^{-6} mol dm^{-3}

Solvent	λ_{abs}/nm ($\varepsilon\times10^5$/dm^3 mol^{-1} cm^{-1})	λ_{ex}/nm	λ_{em}/nm	$\Delta\lambda$/nm	QY[a], %
Hexane	453 (1.37)	467	590	123	3.4
Toluene	468 (1.23)	467	611	144	2.7
CCl_4	462 (1.3)	467	601	134	0.25
$CHCl_3$	478 (1.18)	467	616	149	4.44
CH_2Cl_2	480 (1.13)	467	623	156	1.46
EtOAc	467 (1.11)	467	616	149	6.05
THF	471 (1.09)	467	626	159	6.42
MeOH	470 (1.17)	-	-	-	-
EtOH	470 (1.17)	-	-	-	-
CH_3CN	475 (1.08)	-	-	-	-
DMSO	483 (1.46)	-	-	-	-

[a] The relative fluorescence quantum yield (QY) calculated using Rhodamine 6G in EtOH as a standard (λ_{ex}=480 nm, QY=95%)[S3]

References

S1 I. R. Green, *J. Chem. Educ.,* 1982, **59**, 698.
S2 S. Batalin, *Dyes Pigm.*, 2023, **208**, 110850.
S3 R. F. Kubin, A. N. Fletcher, *J. Lumin*., 1982, **27**, 455.

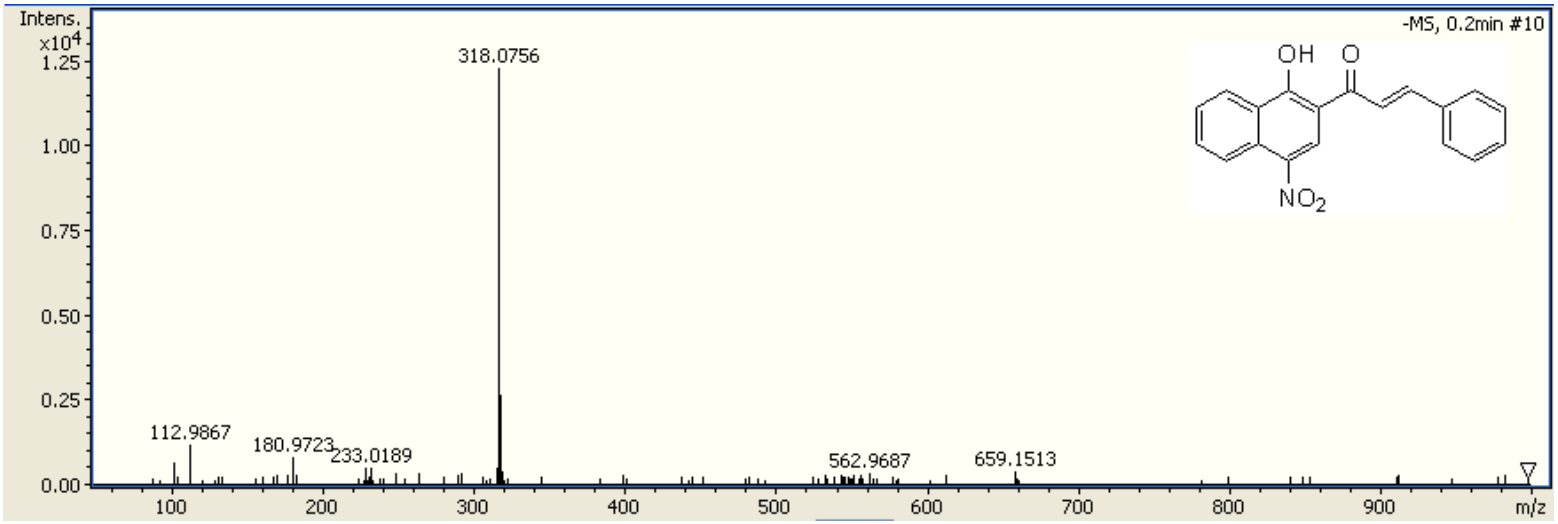


Figure S1 HRMS spectra of **3a**

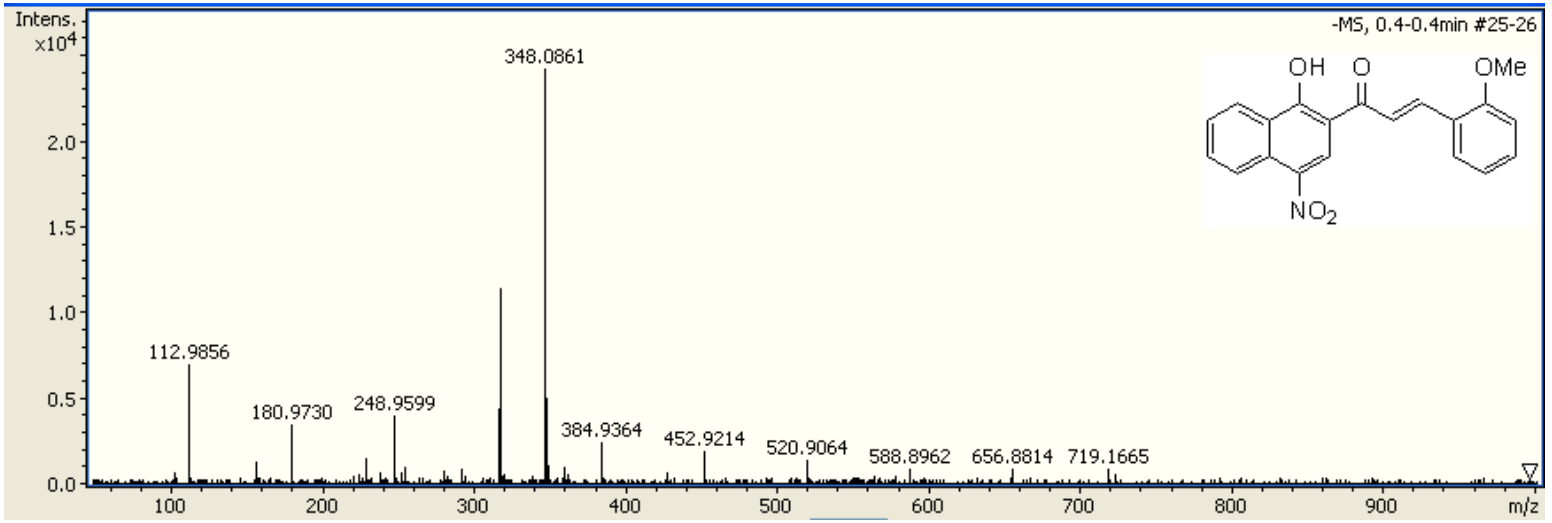


Figure S2 HRMS spectra of **3b**

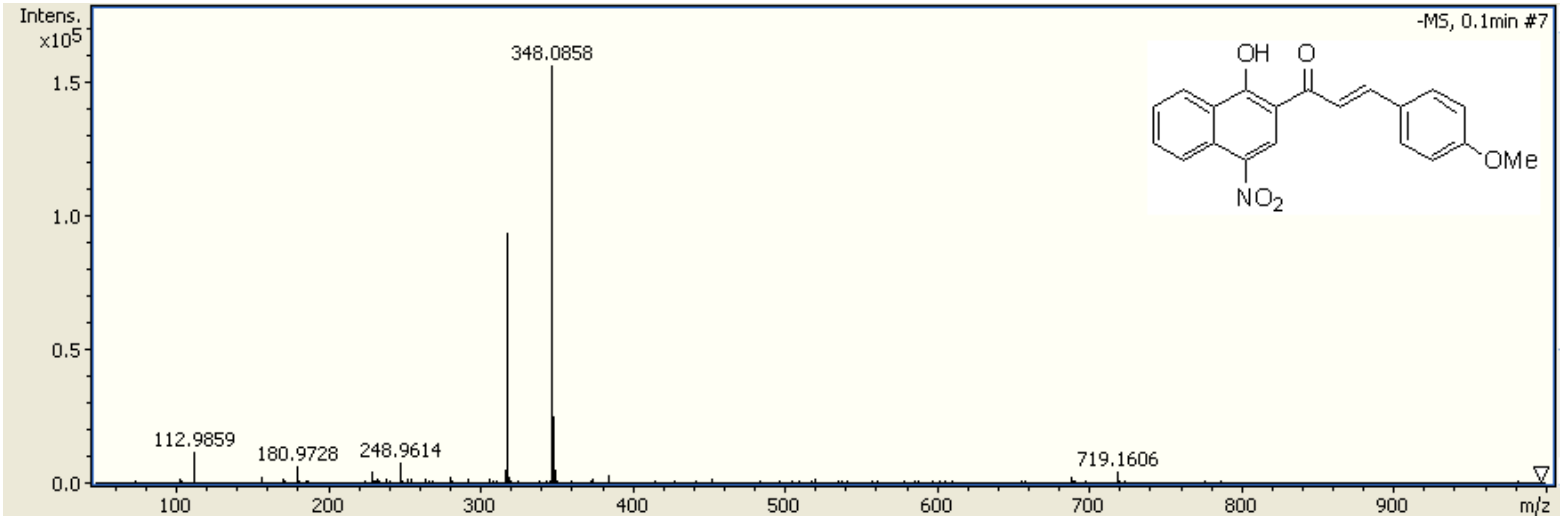


Figure S3 HRMS spectra of **3c**

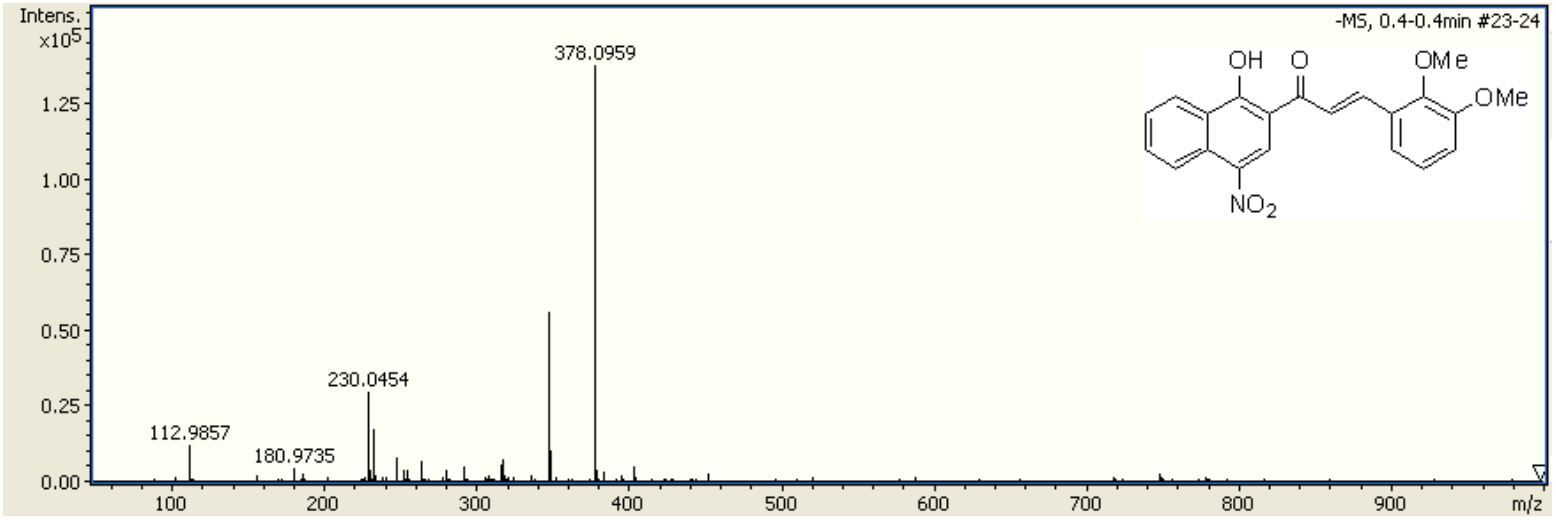


Figure S4 HRMS spectra of **3d**

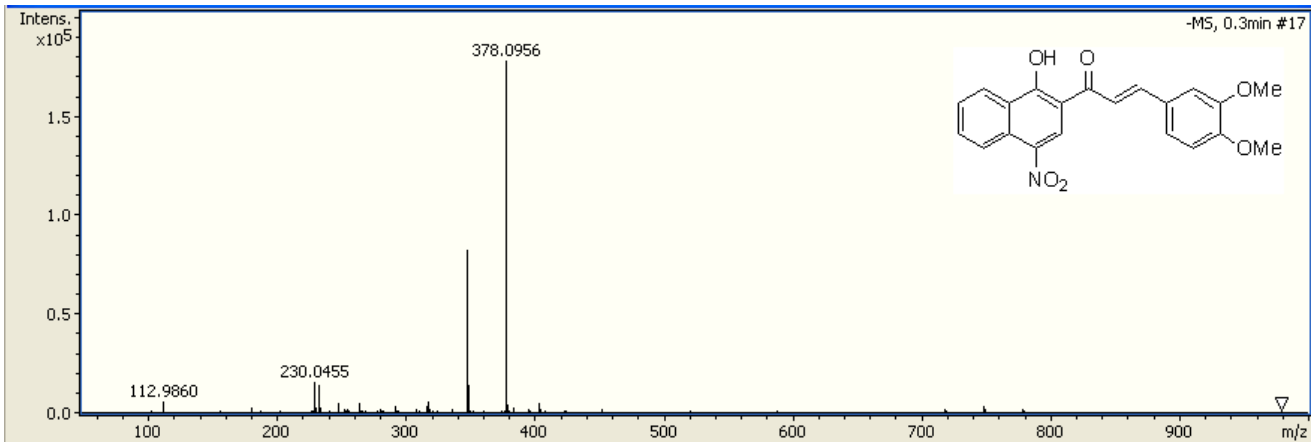


Figure S5 HRMS spectra of **3e**

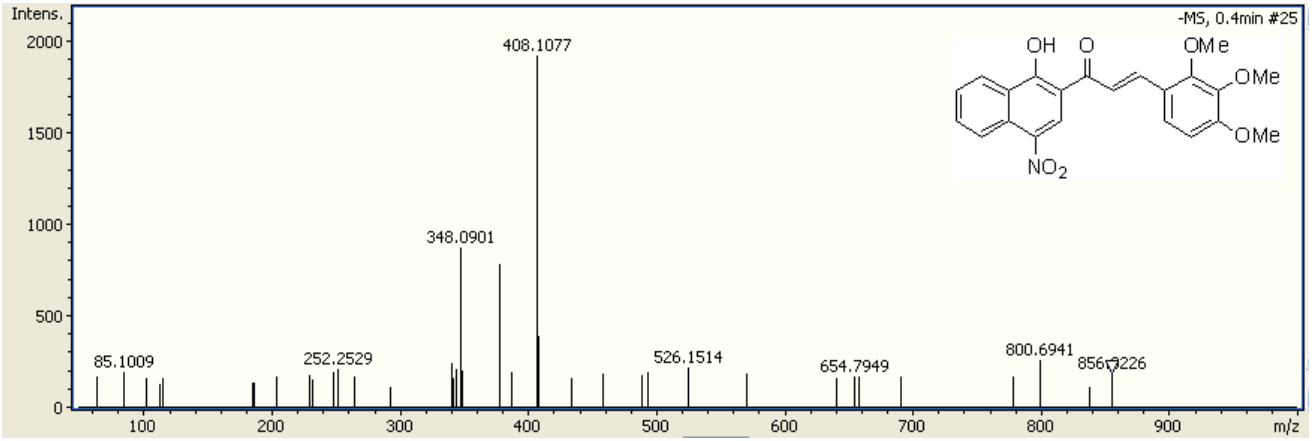


Figure S6 HRMS spectra of **3f**

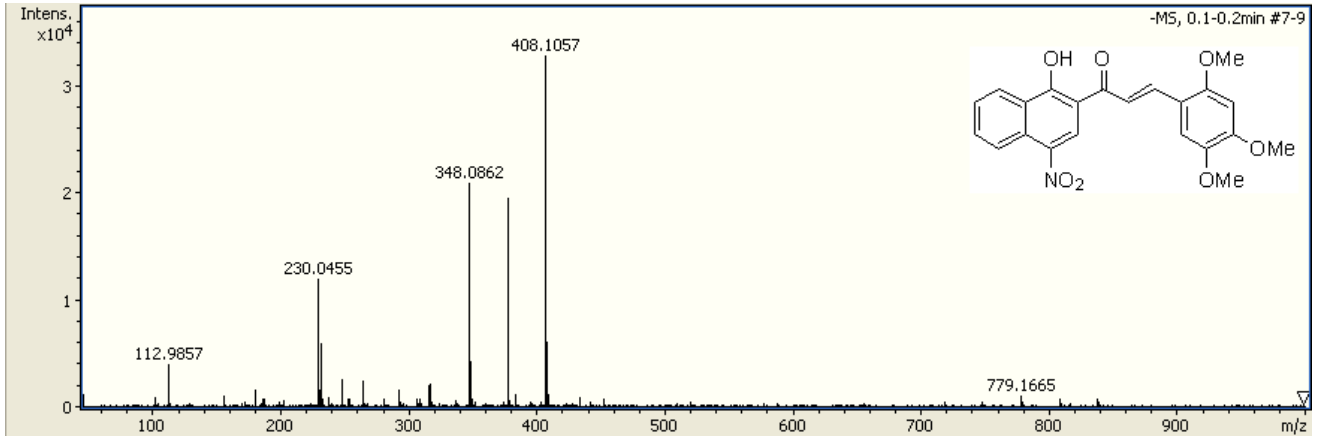


Figure S7 HRMS spectra of **3g**

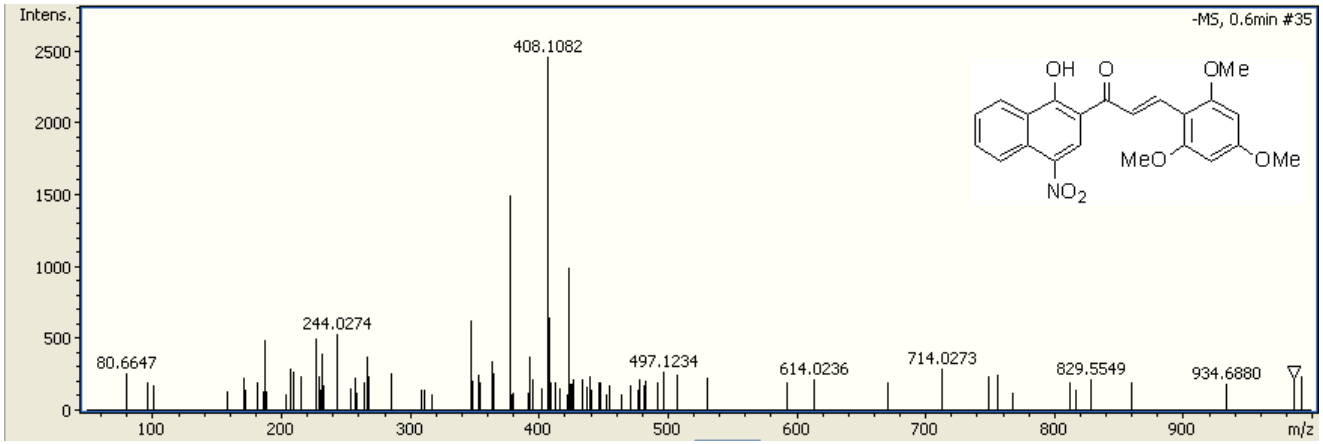


Figure S8 HRMS spectra of **3h**

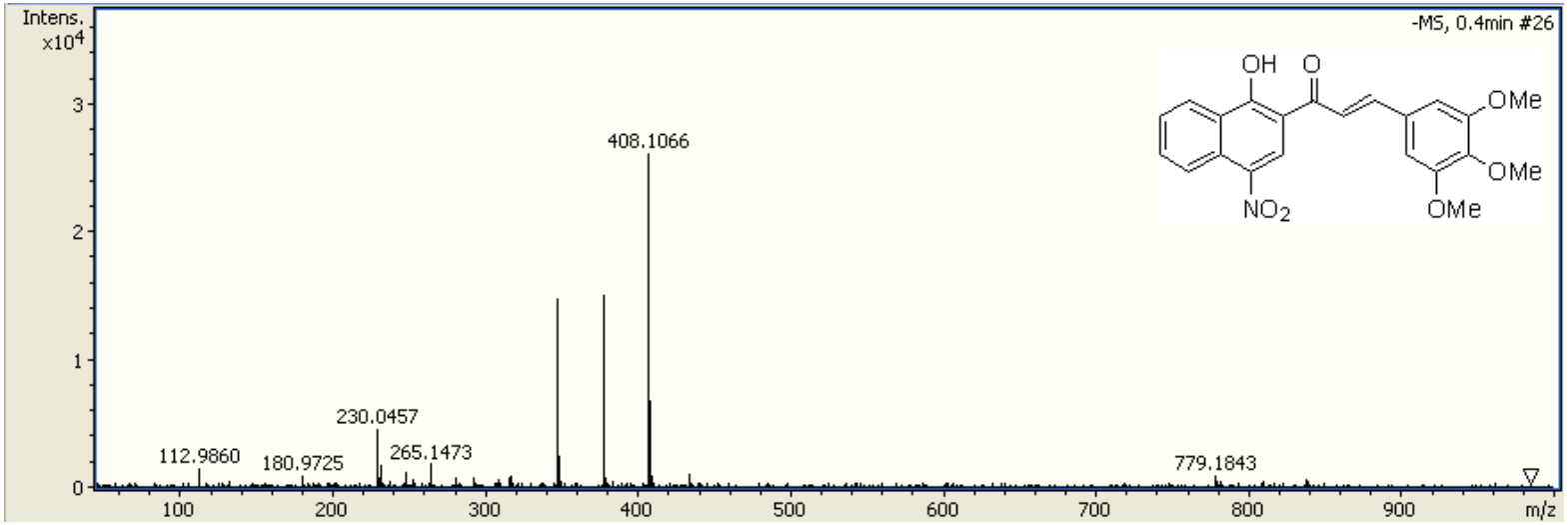


Figure S9 HRMS spectra of **3i**

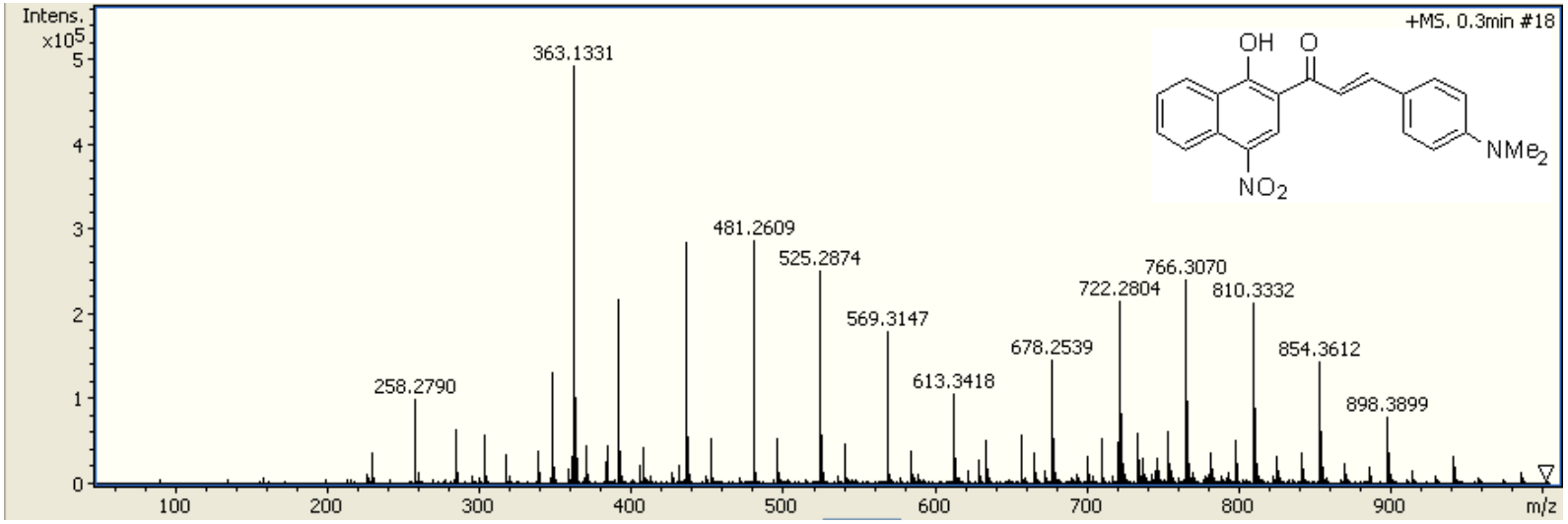


Figure S10 HRMS spectra of **3j**

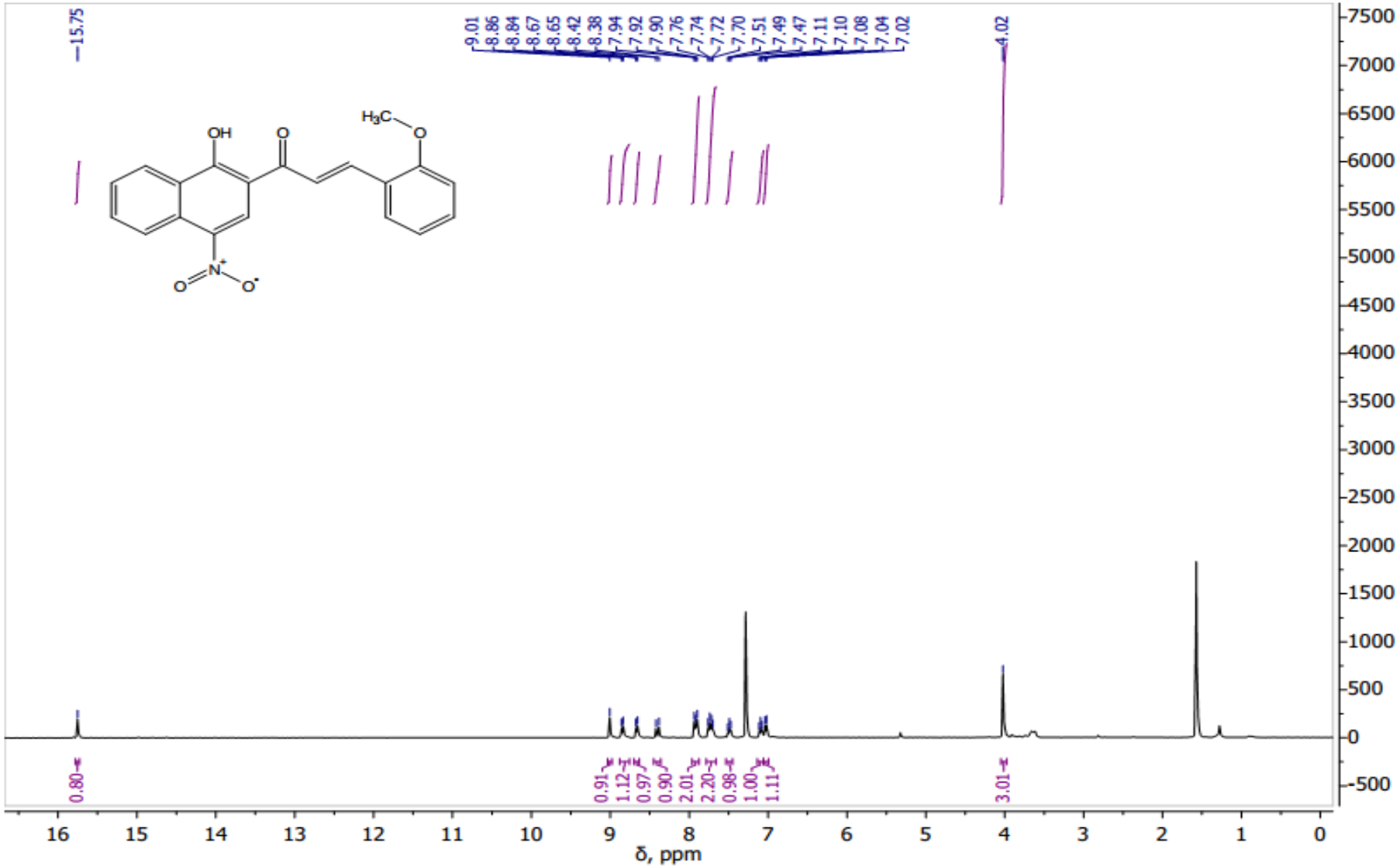


Figure S11 NMR ^{1}H spectra of **3b**

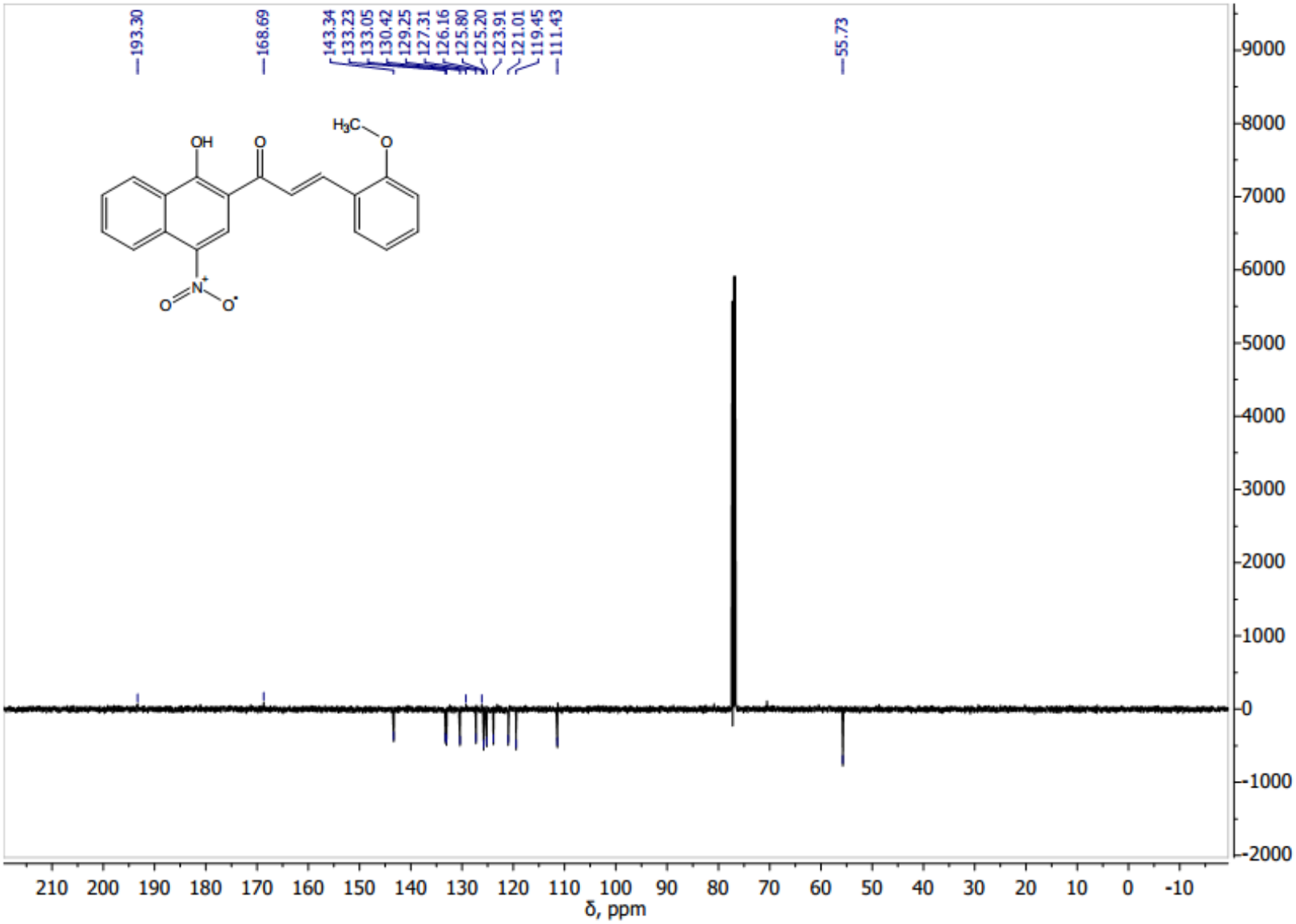


Figure S12 NMR ^{13}C spectra of **3b**

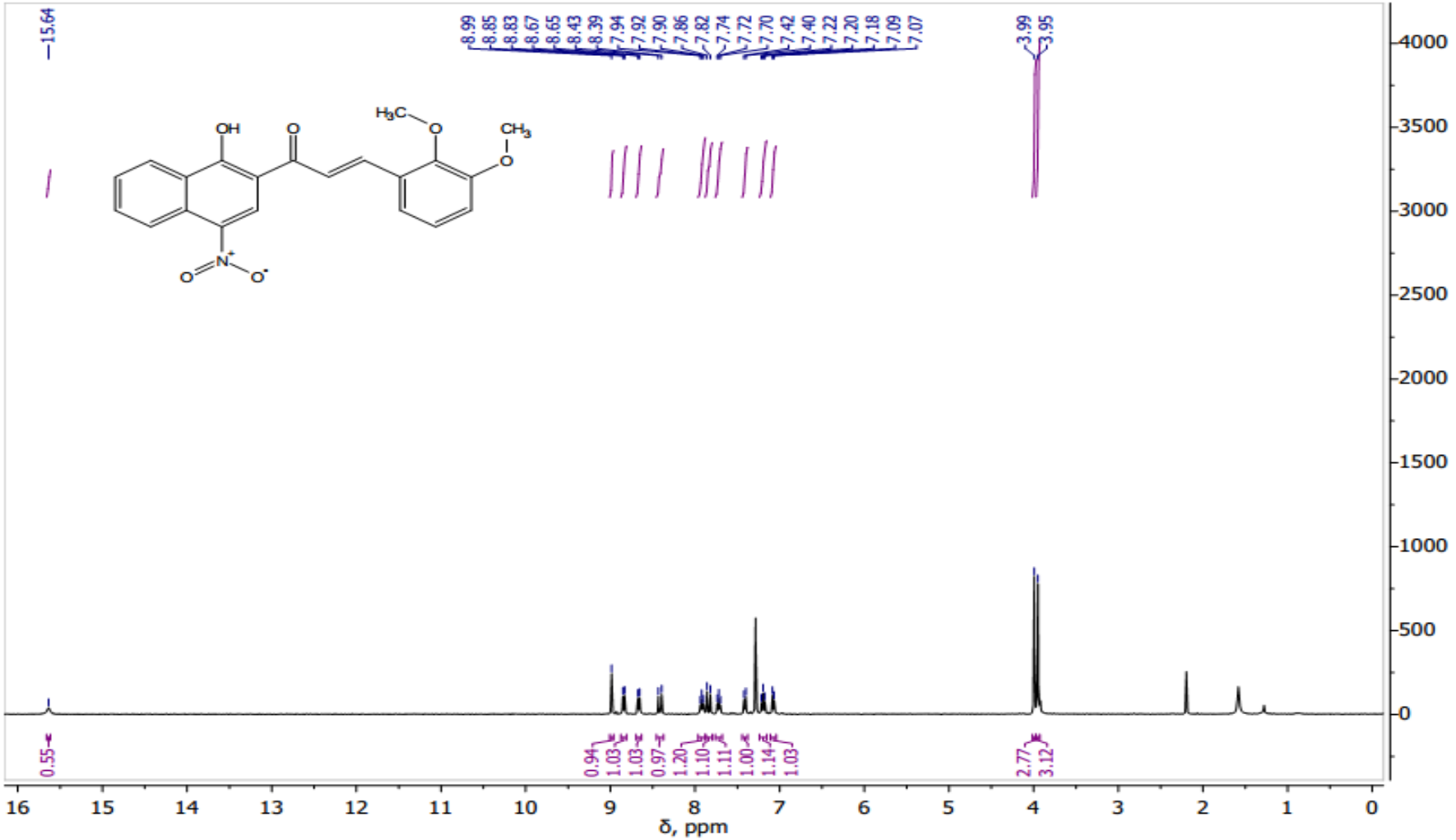


Figure S13 NMR ^{1}H spectra of **3d**

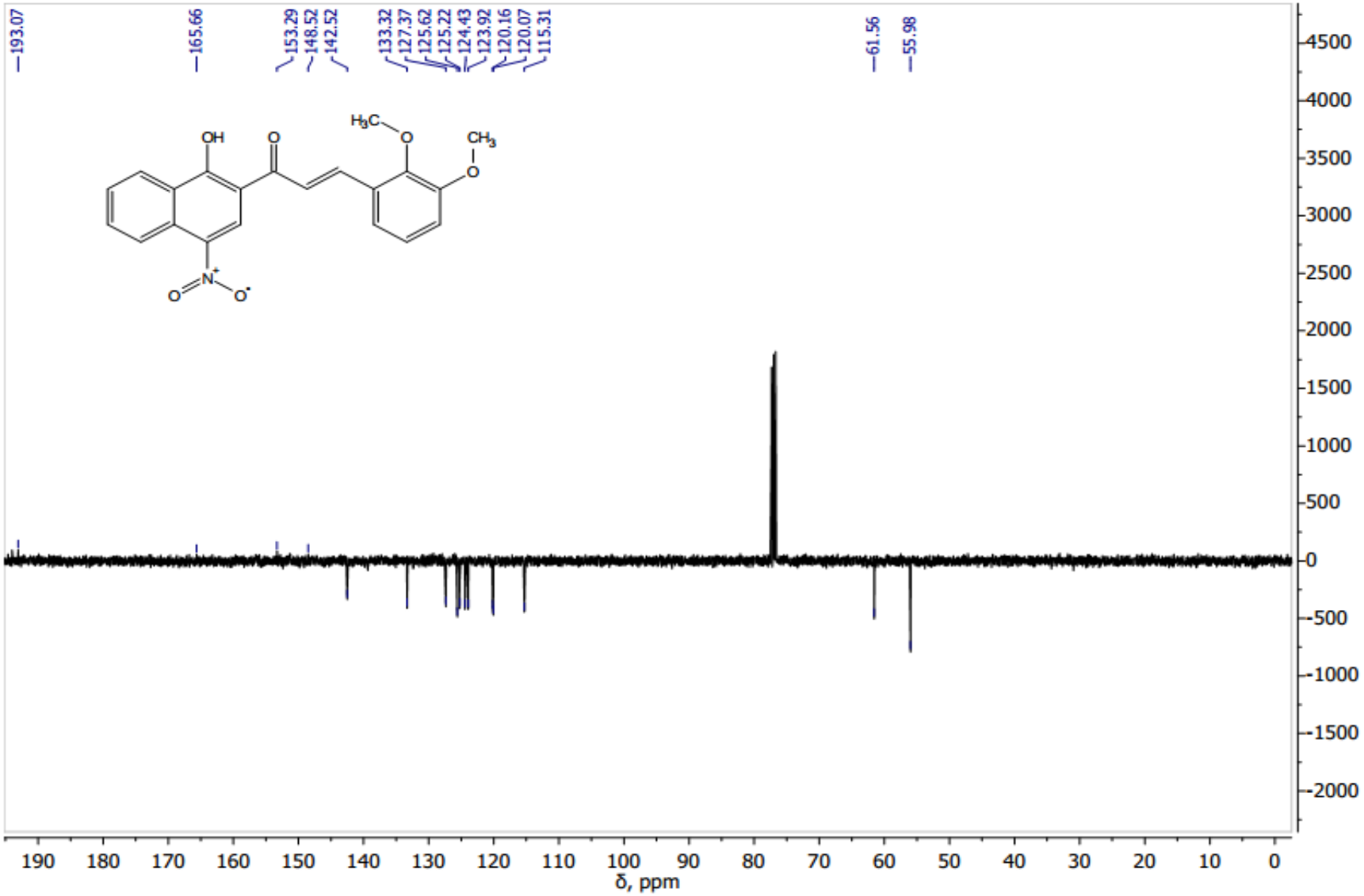


Figure S14 NMR ^{13}C spectra of **3b**

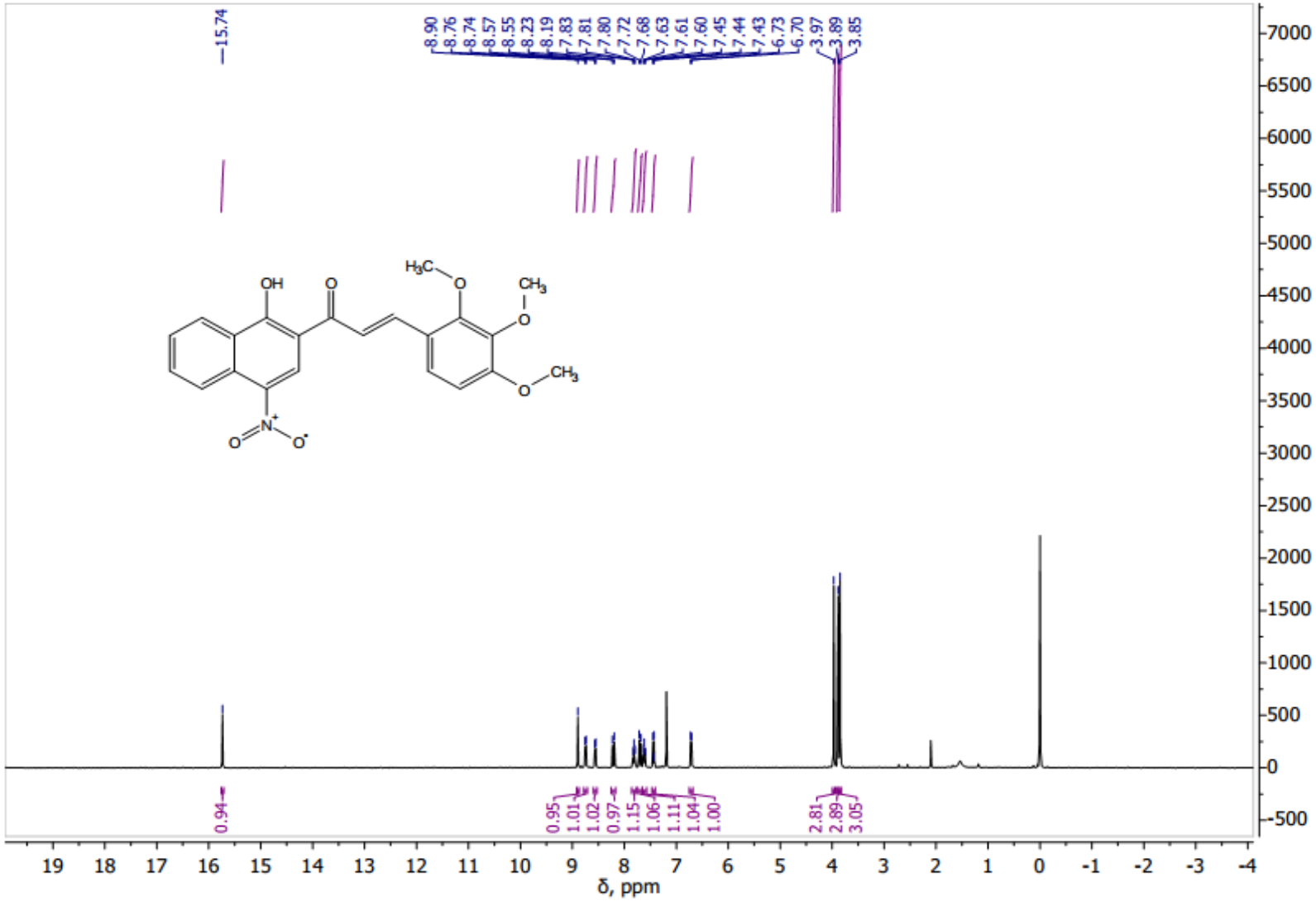


Figure S15 NMR ^{1}H spectra of **3f**

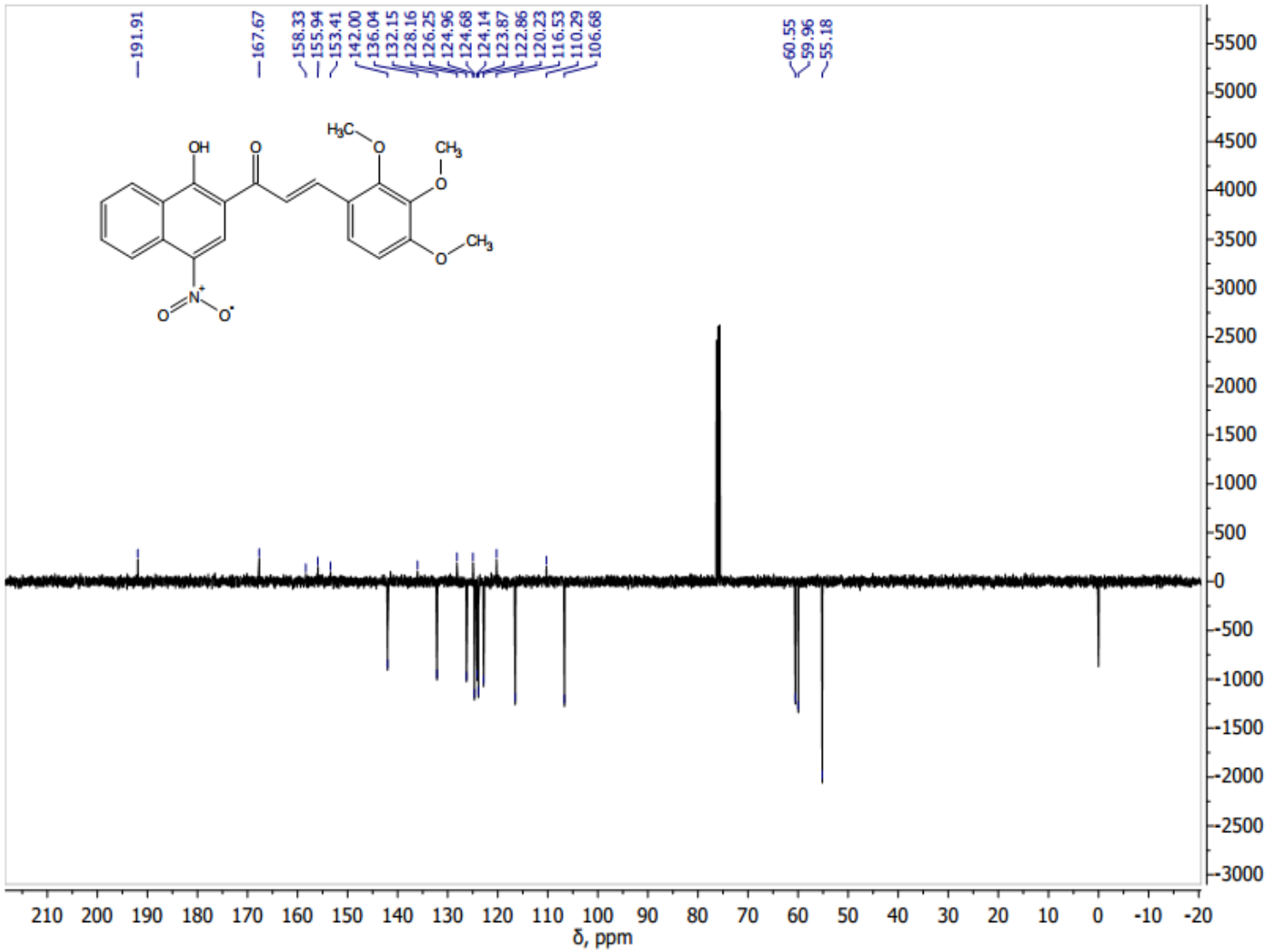


Figure S16 NMR ^{13}C spectra of **3f**

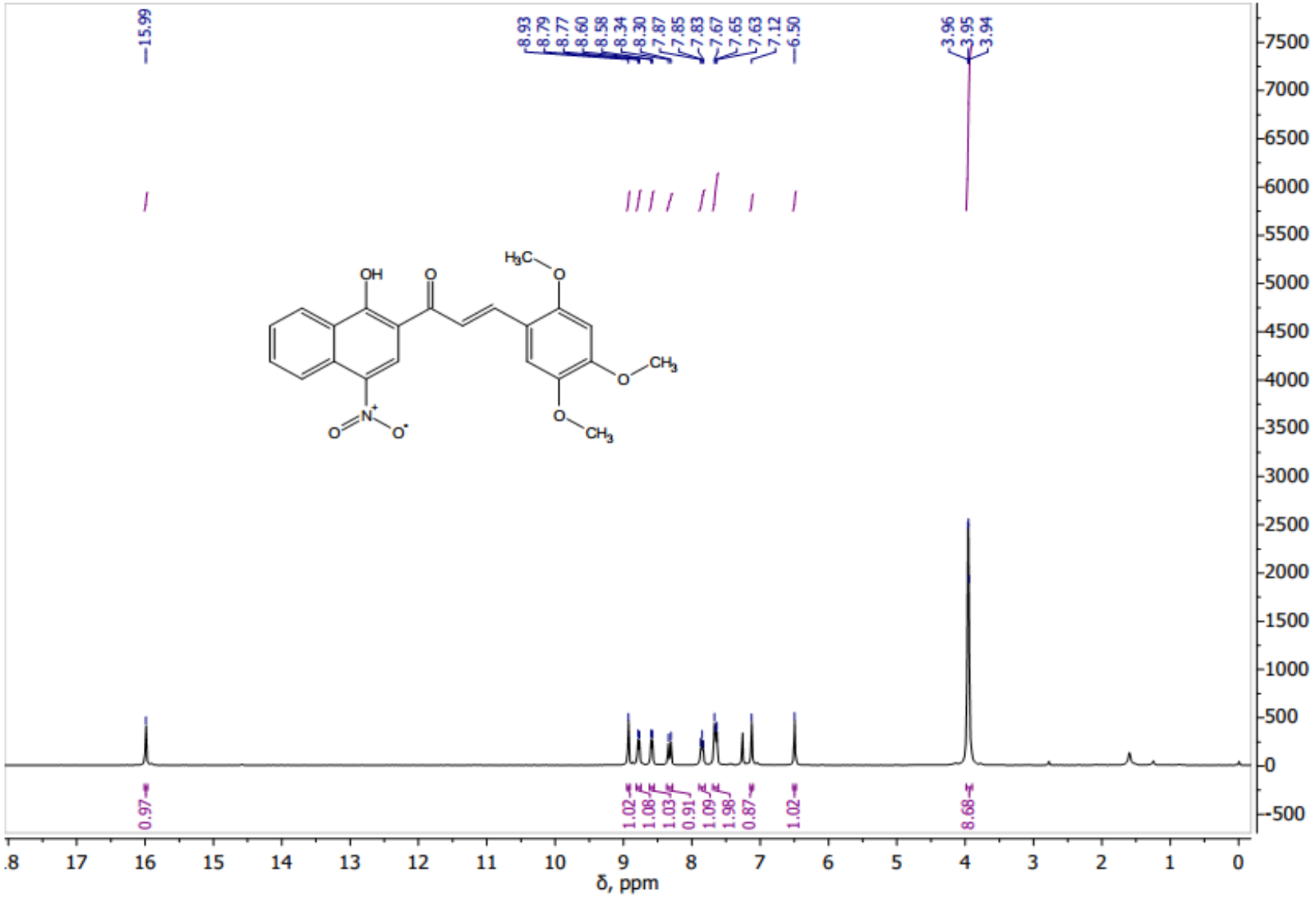


Figure S17 NMR ^{1}H spectra of **3g**

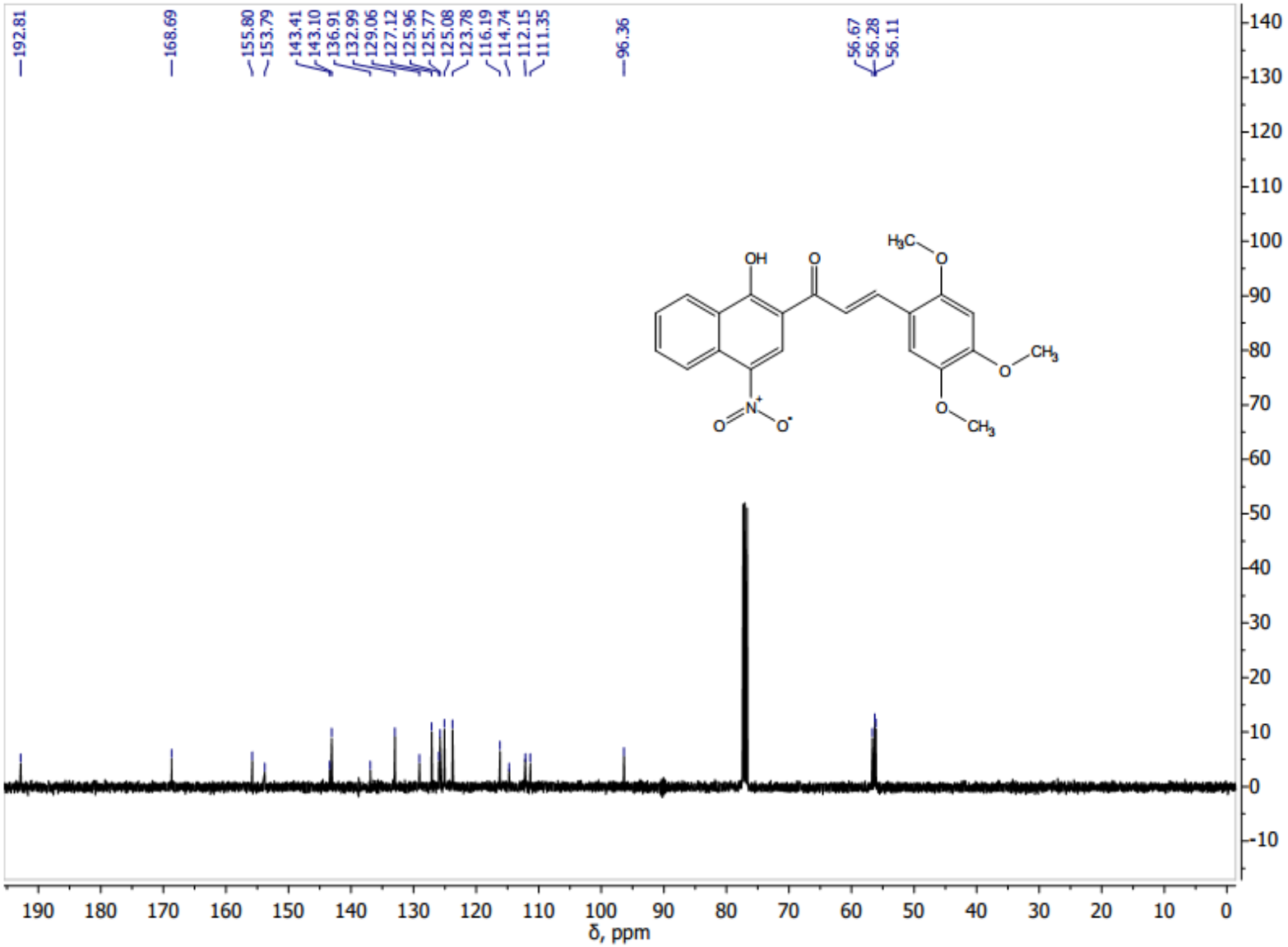


Figure S18 NMR ^{13}C spectra of **3g**

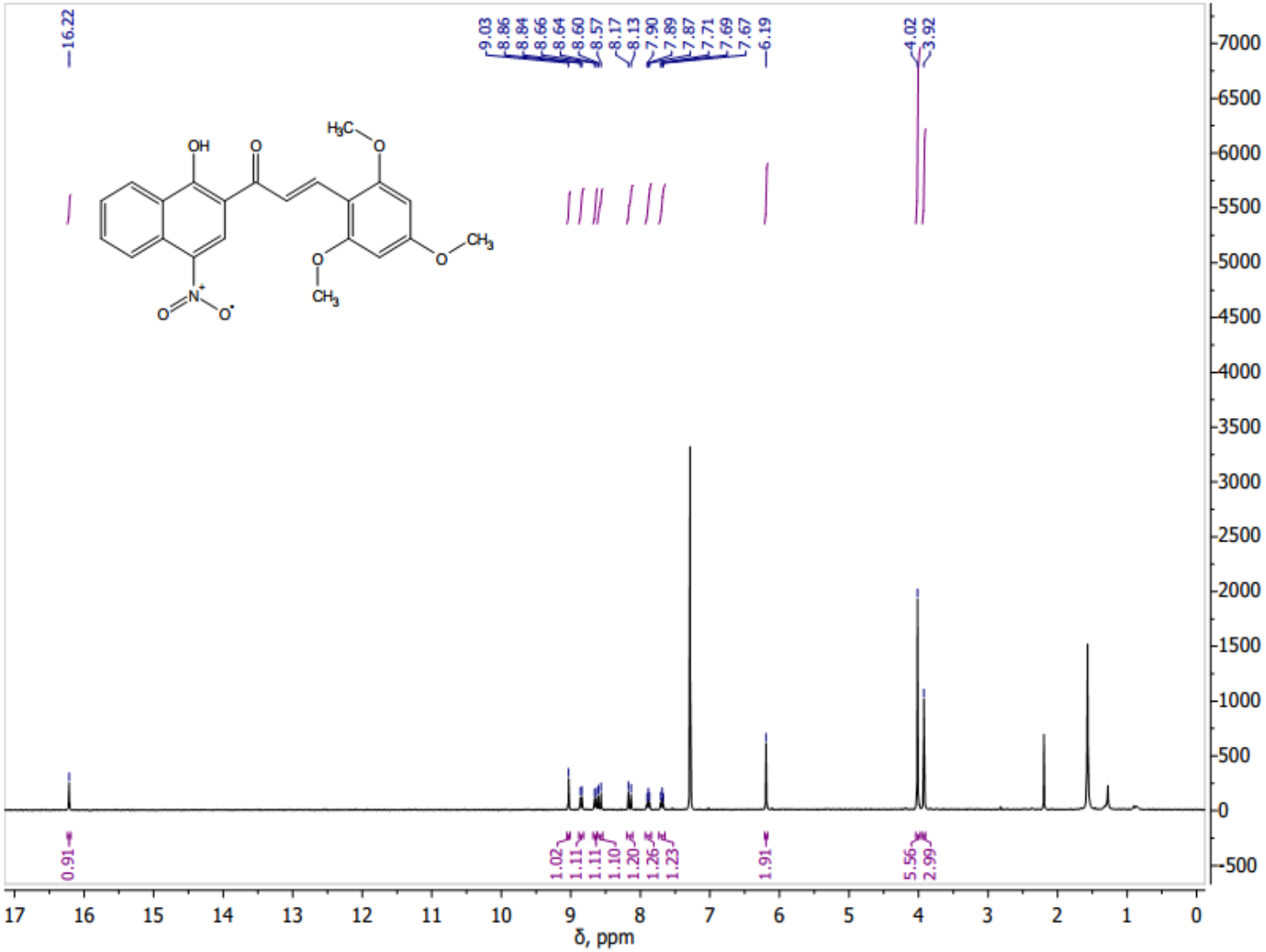


Figure S19 NMR ^{1}H spectra of **3h**

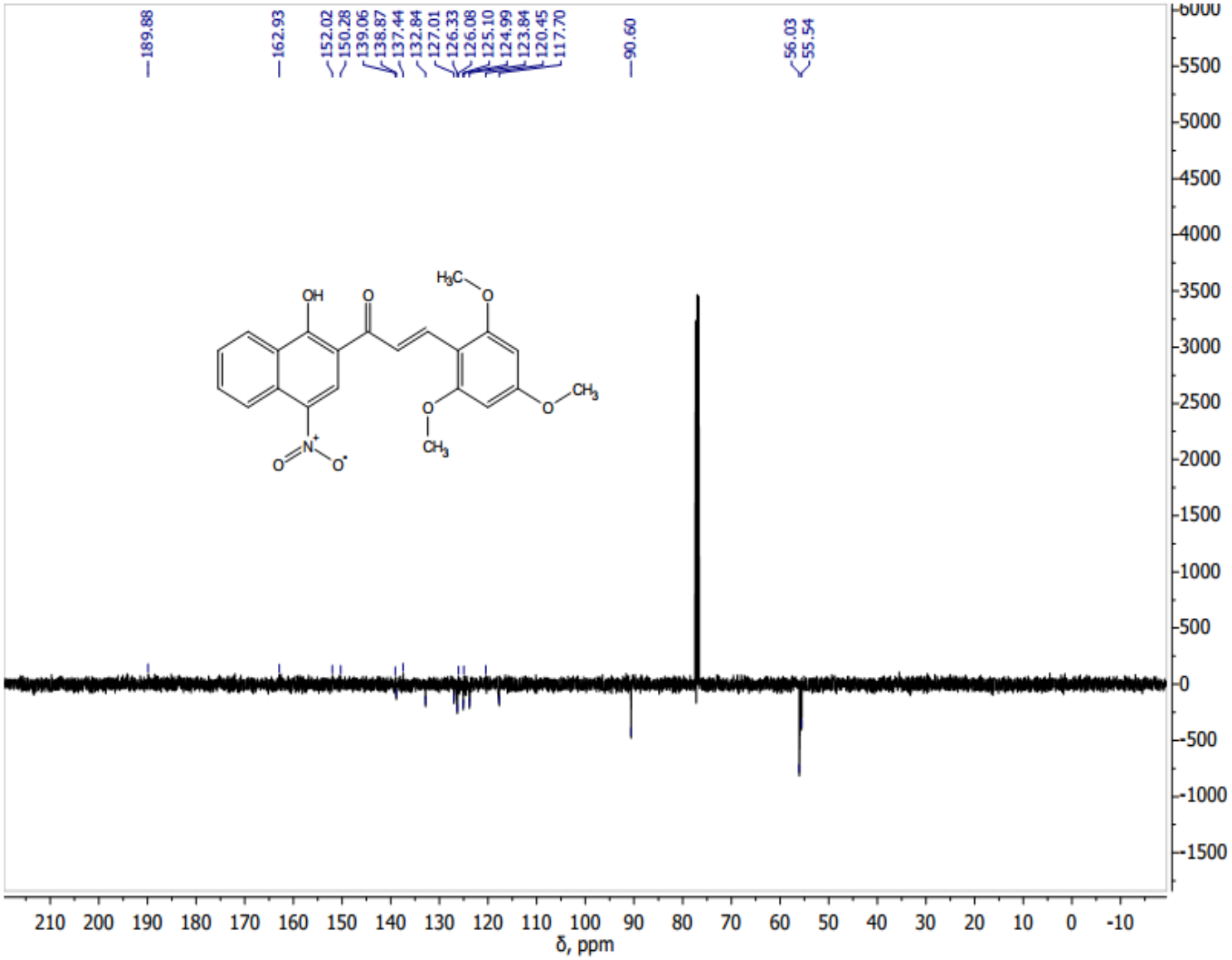


Figure S20 NMR ^{13}C spectra of **3h**

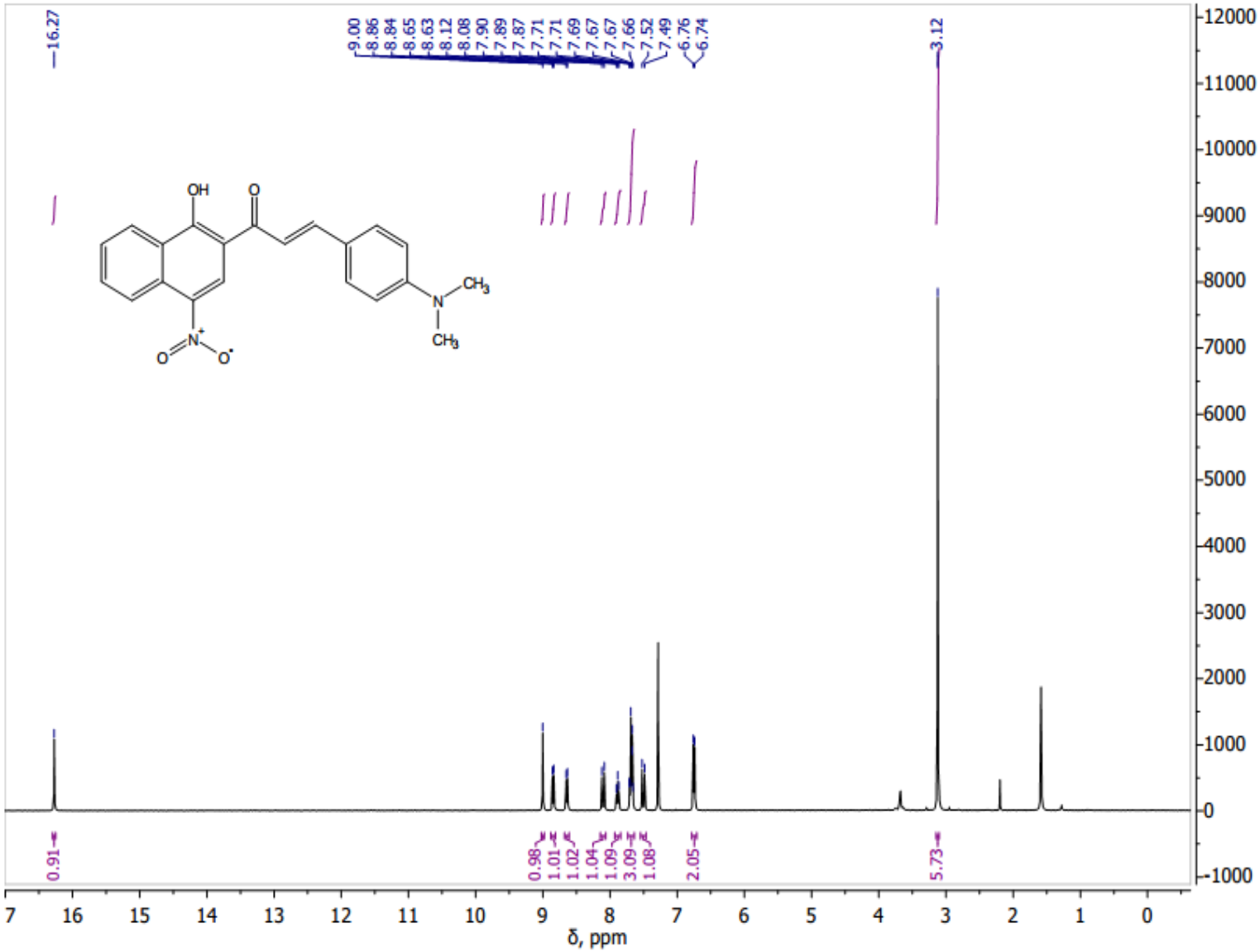


Figure S21 NMR ^{1}H spectra of **3j**

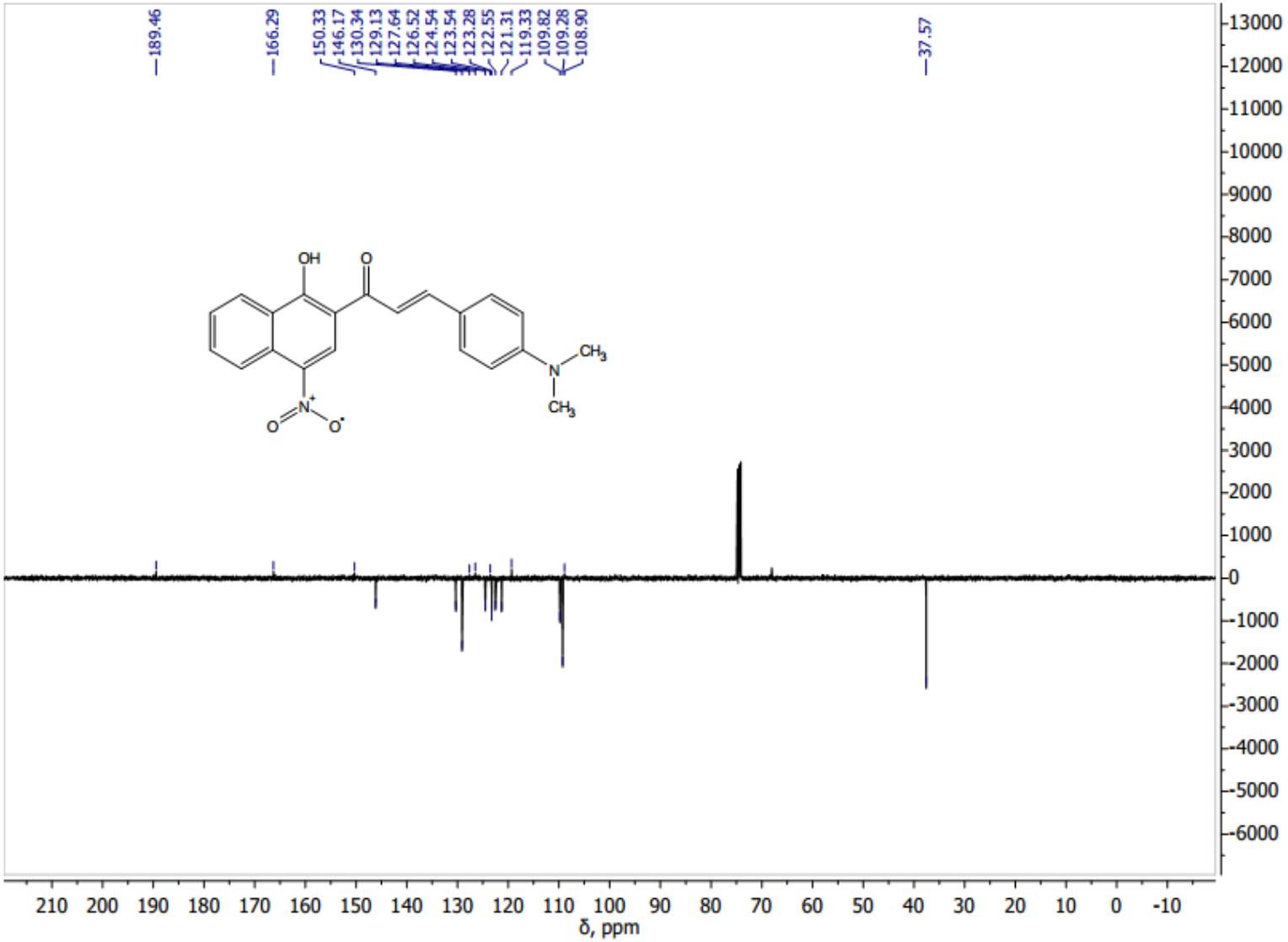
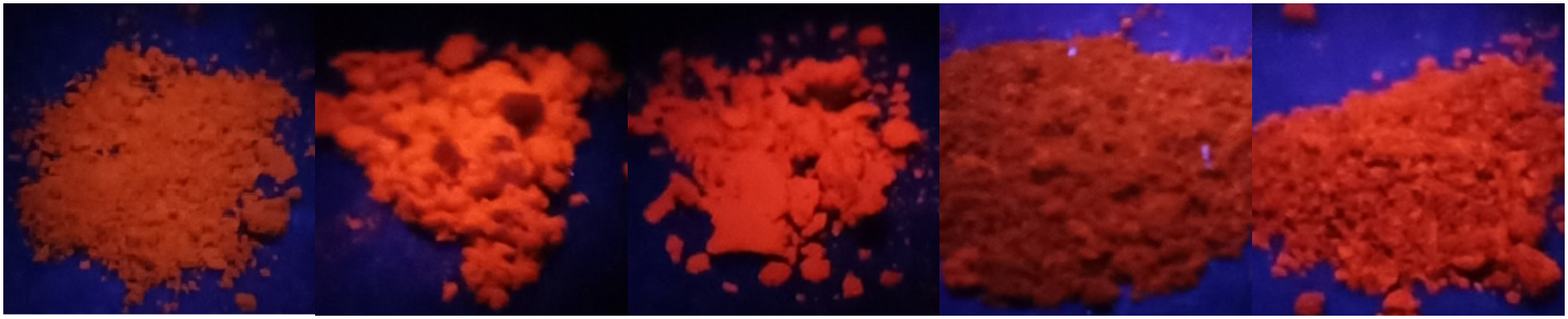


Figure S22 NMR ^{13}C spectra of **3j**

Figure S23 Pictures of compounds **3** under sunlight.

3a **3b** **3c** **3d** **3e**



3f **3g** **3h** **3i** **3j**

Figure S24 Pictures of compounds **3** under UV irradiation