SUPPORTING INFORMATION

Novel Styryl-thiazole hybrids as potential anti-Alzheimer's agents

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EXPERIMENTAL PART

General method for the synthesis of cinnamamides 2a-h

A solution of cinnamic acids **1a-h** (1.00 mmol) and *N*-hydroxysuccinimide (0.13 g, 1.12 mmol) in dry CH_2Cl_2 (2.5 mL) was cooled at 0°C. Then EDC·HCl (0.19, 1.00 mmol) was added and the reaction mixture was stirred at room temperature for 18 h. Then CH_2Cl_2 was added and the organic phase was washed with H_2O, $NaHCO_3$ and brine, dried over Na_2SO_4, and concentrated under reduced pressure. The crude ester was dissolved in a mixture of $CHCl_3$/CH_3CN 1:2 (6 mL), cooled at 0°C and 25% aq. NH_3 (~2 mL) was added dropwise under intense stirring. The biphasic system was stirred for 1h at room temperature, diluted with ethyl acetate and washed with H_2O. The organic phase was dried over Na_2SO_4 and concentrated in vacuo to give the cinnamamides **2a-h** in pure form.

Cinnamamide, 2a

Following the general method , using **1a** (1.26 g, 8.60 mmol) **2a** was obtained as white solid (1.10 g) in 87% yield. R_f= 0.28 (CH_2Cl_2/MeOH 9.8:0.2). ^{1}H NMR (400 MHz, DMSO) δ 7.65-7.55 (m, 3H), 7.47 – 7.34 (m, 4H), 7.09 (bs, 1H), 6.61 (d, *J* = 15.9 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 166.6, 139.1, 134.9, 129.4, 128.9, 127.5, 122.3. MS (ESI) m/z calculated for $C_9H_{10}NO^+$ $[M+H]^+$ 148.1, found 148.1.

(*E*)-3-(p-tolyl)acrylamide, 2b

Following the general method D, using **1b** (1.31 g, 8.10 mmol) **2b** was obtained as white solid (0.99 g) in 76% yield. R_f= 0.67 (CH_2Cl_2/MeOH 9.8:0.2) ^{1}H NMR (200 MHz, DMSO) δ 7.60-7.43 (m, 3H), 7.33 (s, 1H), 7.30-7.21 (m, 2H), 7.06 (bs, 1H), 6.54 (d, *J* = 15.9 Hz, 1H), 2.31 (s, 3H). ^{13}C NMR (50 MHz, DMSO) δ 166.8, 139.2, 139.1, 132.1, 129.5, 127.5, 121.3, 21.0. MS (ESI) m/z calculated for $C_{10}H_{12}NO^+$ $[M+H]^+$ 162.1, found 162.1.

(*E*)-3-(4-methoxyphenyl)acrylamide, 2c

Following the general method D, using **1c** (0.83 g, 4.60 mmol) **2c** was obtained as white solid (0.66 g) in 83% yield. R_f= 0.29 (CH_2Cl_2/MeOH 9.8:0.2) ^{1}H NMR (200 MHz, DMSO) δ 7.60-7.47 (m, 3H), 7.34 (d, *J* = 15.8 Hz, 1H), 7.04 (bs, 1H), 6.96 (d, *J* = 8.7 Hz, 2H), 6.47 (d, *J* = 15.8 Hz, 1H), 3.77 (s, 3H). ^{13}C NMR (50 MHz, DMSO) δ 167.0, 160.3, 138.9, 129.1, 127.4, 119.8, 114.4, 55.3. MS (ESI) m/z calculated for $C_{10}H_{12}NO_2^+$ $[M+H]^+$ 178.1, found 178.2.

(*E*)-3-(3,4-dimethoxyphenyl)acrylamide, 2d

Following the general method D, using **1d** (0.96 g, 4.60 mmol) **2d** was obtained as white solid (0.75 g) in 79% yield. R_f= 0.26 (CH_2Cl_2/MeOH 9.8:0.2). ^{1}H NMR (200 MHz, DMSO) δ 7.45 (s, 1H), 7.37 (d, *J* = 15.8 Hz, 1H), 7.19 – 6.92 (m, 4H), 6.51 (d, *J* = 15.8 Hz, 1H), 3.79 (s, 3H), 3.78 (s, 3H). ^{13}C NMR (50 MHz, DMSO) δ 167.1, 150.1, 148.9, 139.3, 127.7, 121.5, 120.0, 111.7, 109.9, 55.6, 55.4. MS (ESI) m/z calculated for $C_{11}H_{14}NO_3^+$ $[M+H]^+$ 208.1, found 208.2.

(*E*)-3-(3,4,5-trimethoxyphenyl)acrylamide, 2e

Following the general method D, using **1e** (1.00 g, 4.20 mmol) **2e** was obtained as white solid (0.81 g) in 81% yield. R_f= 0.50 (CH_2Cl_2/MeOH 9.8:0.2). ^{1}H NMR (200 MHz, DMSO) δ 7.45 (bs, 1H), 7.36 (d, *J* = 15.8 Hz, 1H), 7.05 (bs, 1H), 6.88 (s, 2H), 6.56 (d, *J* = 15.8 Hz, 1H), 3.81 (s, 6H), 3.68 (s, 3H). ^{13}C NMR (50 MHz, DMSO) δ 166.8, 153.1, 139.5, 138.6, 130.6, 121.7, 104.9, 60.1, 55.9. MS (ESI) m/z calculated for $C_{12}H_{16}NO_4^+$ $[M+H]^+$ 238.1, found 238.3.

(*E*)-3-(4-chlorophenyl)acrylamide, 2f

Following the general method D, using **1f** (0.59 g, 3.20 mmol) **2f** was obtained as white solid (0.47 g) in 81% yield. R_f= 0.65 (CH_2Cl_2/MeOH 9.8:0.2). ^{1}H NMR (200 MHz, DMSO) δ 7.58 (d, *J* = 8.0 Hz, 3H), 7.46 (d, *J* = 6.7 Hz, 2H), 7.37 (s, 1H), 7.16 (bs, 1H), 6.62 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR (50 MHz, DMSO) δ 166.5, 137.8, 133.9, 129.3, 129.0, 123.2. MS (ESI) m/z calculated for $C_9H_7ClNO^-$ $[M-H]^-$ 180.0, found 180.0.

(*E*)-3-(4-fluorophenyl)acrylamide, 2g

Following the general method D, using **1g** (1.50 g, 9.00 mmol) **2g** was obtained as white solid (1.16 g) in 78% yield. R_f= 0.25 (CH_2Cl_2/MeOH 9.8:0.2 ^{1}H NMR (200 MHz, DMSO) δ 7.69 – 7.57 (m, 2H), 7.57 (bs, 1H), 7.42 (d, *J* = 15.8 Hz, 1H), 7.24 (t, *J* = 8.9 Hz, 2H), 7.16 (bs, 1H), 6.56 (d, *J* = 15.9 Hz, 1H). ^{13}C NMR (50 MHz, DMSO) δ 166.7, 162.8 (d, *J* = 239.4 Hz), 138.0, 131.5 (d, *J* = 3.2 Hz), 129.7 (d, *J* = 8.6 Hz), 122.3, 115.9 (d, *J* = 21.8 Hz). MS (ESI) m/z calculated for $C_9H_7FNO^-$ $[M-H]^-$ 164.1, found 164.2.

(*E*)-3-(4-nitrophenyl)acrylamide, 2h

Following the general method D, using **1h** (1.00 g, 5.20 mmol) **2h** was obtained as white solid (0.82 g) in 82% yield. R_f= 0.48 (CH_2Cl_2/MeOH 9.8:0.2). ^{1}H NMR (200 MHz, DMSO) δ 8.25 (d, *J* = 8.7 Hz, 2H), 7.82 (d, *J* = 8.6 Hz, 2H), 7.68 (bs, 1H), 7.52 (d, *J* = 15.9 Hz, 1H), 7.31 (bs, 1H), 6.80 (d, *J* = 15.9 Hz, 1H). ^{13}C NMR (50 MHz, DMSO) δ 166.0, 147.5, 141.5, 136.9, 128.6, 126.7, 124.1. MS (ESI) m/z calculated for $C_9H_7N_2O_3^-$ $[M-H]^-$ 191.1, found 191.3.

General method for the synthesis of thioamides 3a-h

In a flask containing cinnamamide **2a-h** (1.00 mmol), phosphorus pentasulfide P_2S_5 (0.10 g, 0.22 mmol) and dry dioxane (13 mL) were added and the mixture was heated to 80°C for 2h, the reaction progress being monitored by TLC. Then, the reaction mixture was concentrated under reduced pressure and the crude material was purified by column chromatography using ethyl acetate and petroleum ether mixtures as eluent to give the desired product **3a-h**.

((*E*)-3-phenylprop-2-enethioamide, 3a

Following the general method E, using **2a** (0.10 g, 0.68 mmol) **3a** was obtained as yellow solid (0.06 g) in 50% yield. R_f= 0.68 (AcOEt/PE (40-60°C) 1:1. ^{1}H NMR (200 MHz, CD_3OD) δ 7.79 (d, *J* = 15.5 Hz, 1H), 7.57 (dd, *J* = 6.6, 2.9 Hz, 2H), 7.45 – 7.26 (m, 5H), 7.03 (d, *J* = 15.5 Hz, 1H). ^{13}C NMR (50 MHz, CD_3OD) δ 199.5, 144.2, 136.2, 131.0, 130.0, 129.1, 127.4.

MS (ESI) m/z calculated for $C_9H_8NS^-$ $[M-H]^-$ 162.0, found 162.2.

(*E*)-3-(p-tolyl)prop-2-enethioamide, 3b

Following the general method E, using **2b** (0.28 g, 1.80 mmol) **3b** was obtained as yellow solid (0.19 g) in 49% yield. R_f= 0.67 (AcOEt/PE (40-60°C) 1:1). ^{1}H NMR (200 MHz, DMSO) δ 9.47 (bs, 1H), 9.28 (bs, 1H), 7.30 – 7.10 (m, 6H), 2.27 (s, 3H). ^{13}C NMR (50 MHz, DMSO) δ 204.2, 139.8, 136.4, 129.7, 129.0, 127.9, 127.1, 20.7. MS (ESI) m/z calculated for $C_{10}H_{12}NS^+$ $[M+MeOH+H]^+$ 210.1, found 210.1.

(*E*)-3-(4-methoxyphenyl)prop-2-enethioamide, 3c

Following the general method E, using **2c** (0.03 g, 0.14 mmol) **3c** was obtained as yellow solid (0.01 g) in 37% yield. R_f= 0.48 (AcOEt/PE (40-60°C) 1:1). ^{1}H NMR (400 MHz, DMSO) δ 9.39 (bs, 1H), 9.13 (bs, 1H), 7.62 (d, *J* = 15.4 Hz, 1H), 7.54 (d, *J* = 8.5 Hz, 2H), 6.99 (d, *J* = 8.6 Hz, 2H), 6.89 (d, *J* = 15.5 Hz, 1H), 3.80 (s, 3H). ^{13}C NMR (101 MHz, DMSO) δ 196.5, 160.6, 141.3, 129.5, 127.3, 125.1, 114.5, 55.3. MS (ESI) m/z calculated for $C_{10}H_{10}NOS^-$ $[M-H]^-$ 192.1, found 192.0.

(*E*)-3-(3,4-dimethoxyphenyl)prop-2-enethioamide, 3d

Following the general method E, using **2d** (0.70 g, 3.40 mmol) **3d** was obtained as yellow solid (0.08 g) in 27% yield. R_f= 0.32 (AcOEt/PE (40-60°C) 1:1). ^{1}H NMR (400 MHz, DMSO) δ 9.39 (bs, 1H), 9.11 (bs, 1H), 7.60 (d, *J* = 15.4 Hz, 1H), 7.18 (s, 1H), 7.15 (d, *J* = 8.3 Hz, 1H), 7.00 (d, *J* = 8.3 Hz, 1H), 6.91 (d, *J* = 15.4 Hz, 1H), 3.80 (s, 3H), 3.79 (s, 3H). ^{13}C NMR (101 MHz, DMSO) δ 196.6, 150.5, 149.0, 141.7, 127.5, 125.3, 122.0, 111.8, 110.2, 55.6, 55.5. MS (ESI) m/z calculated for C11H12NO2S- [M-H]- 222.1, found 222.3.

(*E*)-3-(3,4,5-trimethoxyphenyl)prop-2-enethioamide, 3e

Following the general method E, using **2e** (0.47 g, 1.98 mmol) **3e** was obtained as yellow solid (0.30 g) in 60% yield. R_f= 0.72 (AcOEt/PE (40-60°C) 7:3). ^{1}H NMR (200 MHz, CD_3OD) δ 7.76 (s, 1H), 7.68 (s, 1H), 7.00 (s, 1H), 6.93 (s, 1H), 6.90 (s, 2H), 3.87 (s, 6H), 3.79 (s, 3H). ^{13}C NMR (50 MHz, MeOD) δ 199.7, 154.8, 144.2, 141.0, 132.3, 127.2, 106.6, 61.2, 56.7. MS (ESI) m/z calculated for $C_{12}H_{14}NO_3S^-$ $[M-H]^-$ 252.1, found 251.9.

(*E*)-3-(4-chlorophenyl)prop-2-enethioamide, 3f

Following the general method E, using **2f** (0.35 g, 1.90 mmol) **3e** was obtained as yellow solid (0.12 g) in 32% yield. R_f= 0.58 (AcOEt/PE (40-60°C) 1:1). ^{1}H NMR (400 MHz, DMSO) δ 9.58 (s, 1H), 9.27 (s, 1H), 7.63 (s, 1H), 7.60 (d, *J* = 7.4 Hz, 2H), 7.48 (d, *J* = 8.5 Hz, 2H), 7.00 (d, *J* = 15.6 Hz, 1H). ^{13}C NMR (101 MHz, DMSO) δ 196.2, 139.6, 134.2, 133.8, 129.5, 129.1, 128.2. MS (ESI) m/z calculated for $C_9H_7ClNS^-$ [M-H]- 196.0, found 196.1.

(*E*)-3-(4-fluorophenyl)prop-2-enethioamide, 3g

Following the general method E, using **2g** (0.40 g, 2.40 mmol) **3g** was obtained as yellow solid (0.21 g) in 50% yield. R_f= 0.58 (AcOEt/PE (40-60ºC) 1:1). Rf= 0.78 (AcOEt/PE (40-60ºC) 1:1). 1H NMR (400 MHz, DMSO) δ 9.54 (s, 1H), 9.24 (s, 1H), 7.69 – 7.60 (m, 3H), 7.26-7.23 (m, 2H), 6.95 (d, *J* = 15.5 Hz, 1H). 13C NMR (101 MHz, DMSO) δ 196.3, 162.9 (d, *J* = 247.9 Hz), 140.0, 131.4 (d, *J* = 3.1 Hz), 130.1 (d, *J* = 8.6 Hz), 127.4 (d, *J* = 2.1 Hz), 116.1 (d, *J* = 21.8 Hz). MS (ESI) m/z calculated for $C_9H_9FNS^+$ [M+H]+ 182.0, found 182.2.

(*E*)-3-(4-nitrophenyl)prop-2-enethioamide, 3h

Following the general method E, using **2h** (0.36 g, 1.85 mmol) **3h** was obtained as yellow solid (0.06 g) in 17% yield.Rf= 0.64 (AcOEt/PE (40-60oC) 1:1). 1H NMR (400 MHz, DMSO) δ 9.74 (s, 1H), 9.40 (s, 1H), 8.26 (d, *J* = 8.8 Hz, 2H), 7.86 (d, *J* = 8.8 Hz, 2H), 7.68 (d, *J* = 15.6 Hz, 1H), 7.15 (d, *J* = 15.6 Hz, 1H). 13C NMR (101 MHz, DMSO) δ 195.8, 147.6, 141.4, 138.0, 131.3, 128.9, 124.2. MS (ESI) m/z calculated for C9H7N2O2S- [M-H]- 207.0, found 207.2.

General method for the synthesis of phenacyl bromides 5a-d

To a stirred solution of Br_2 (0.6 mL, 11.00 mmol) in dioxane (12 mL) at 0^oC, a solution of acetophenone or substituted acetophenone **4a-d** (10.00 mmol) in dioxane was added dropwise. The reaction mixture was stirred at room temperature for 30 min. Then the volatiles were removed in vacuo and the crude product was purified by column chromatography using diethyl ether and petroleum ether mixtures as eluent to give the desired product **5a-d.**

Phenacyl bromide, 5a

Following the general method F, using **4a** (1.20 g, 10.00 mmol), **5a** was obtained as white solid (1.38 g) in 70% yield. R_f= 0.76 (Et_2O/PE (40-60^{o}C) 1:3). ^{1}H NMR (200 MHz, $CDCl_3$) δ 7.75-7.71 (m, 2H), 7.40 – 7.29 (m, 1H), 7.27 – 7.15 (m, 2H), 4.29 (s, 2H).^{13}C NMR (101 MHz, $CDCl_3$) δ 190.5, 133.3, 133.2, 128.2, 128.2, 31.6.

4-hydroxy-phenacyl bromide, 5b

Following the general method F, using **4b** (1.36 g, 10.00 mmol), **5b** was obtained as white solid (1.31g) in 62% yield. R_f= 0.48 (Et_2O/PE (40-60^{o}C) 1:1). ^{1}H NMR (200 MHz, DMSO) δ 10.56 (s, 1H), 7.89 (d, *J* = 8.8 Hz, 2H), 6.87 (d, *J* = 8.7 Hz, 2H), 4.78 (s, 2H). ^{13}C NMR (50 MHz, DMSO) δ 190.0, 162.8, 131.6, 125.5, 115.5, 33.7.

4-methoxy-phenacyl bromide, 5c

Following the general method F, using **4c** (1.50 g, 10.00 mmol), **5c** was obtained as white solid (1.17 g) in 56% yield. R_f= 0.64 (Et_2O/PE (40-60^{o}C) 1:1). ^{1}H NMR (400 MHz, DMSO) δ 7.99 (d, *J* = 8.9 Hz, 2H), 7.07 (d, *J* = 7.3 Hz, 2H), 4.83 (s, 2H), 3.86 (s, 3H). ^{13}C NMR (101 MHz, DMSO) δ 190.2, 163.6, 131.2, 126.8, 114.1, 55.6, 33.6.

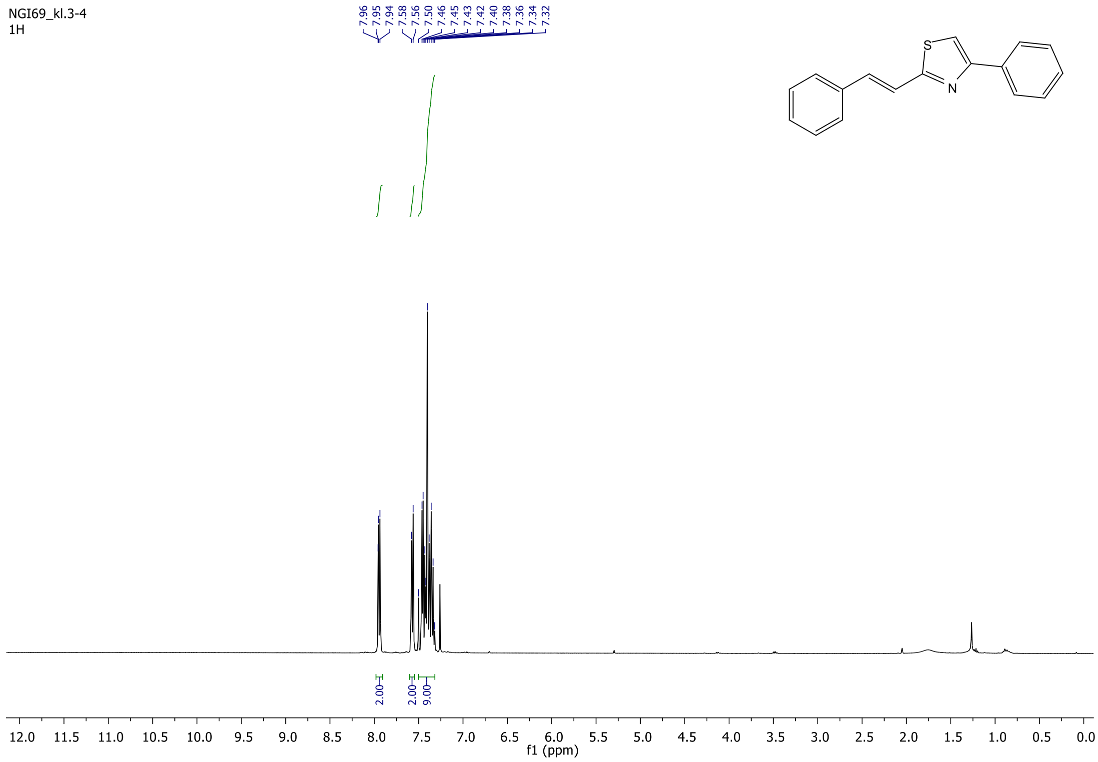


Figure 1 : ^{1}H NMR of **6a** in $CDCl_3$.

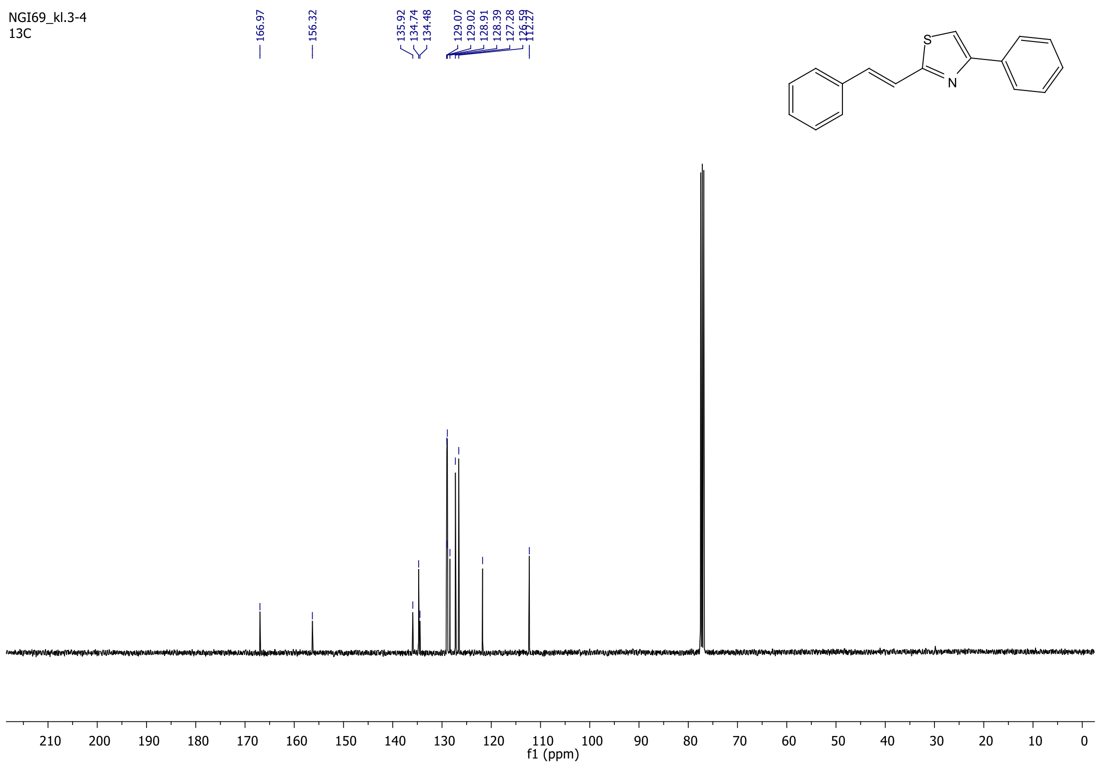


Figure 2 : ^{13}C NMR of **6a** in $CDCl_3$.

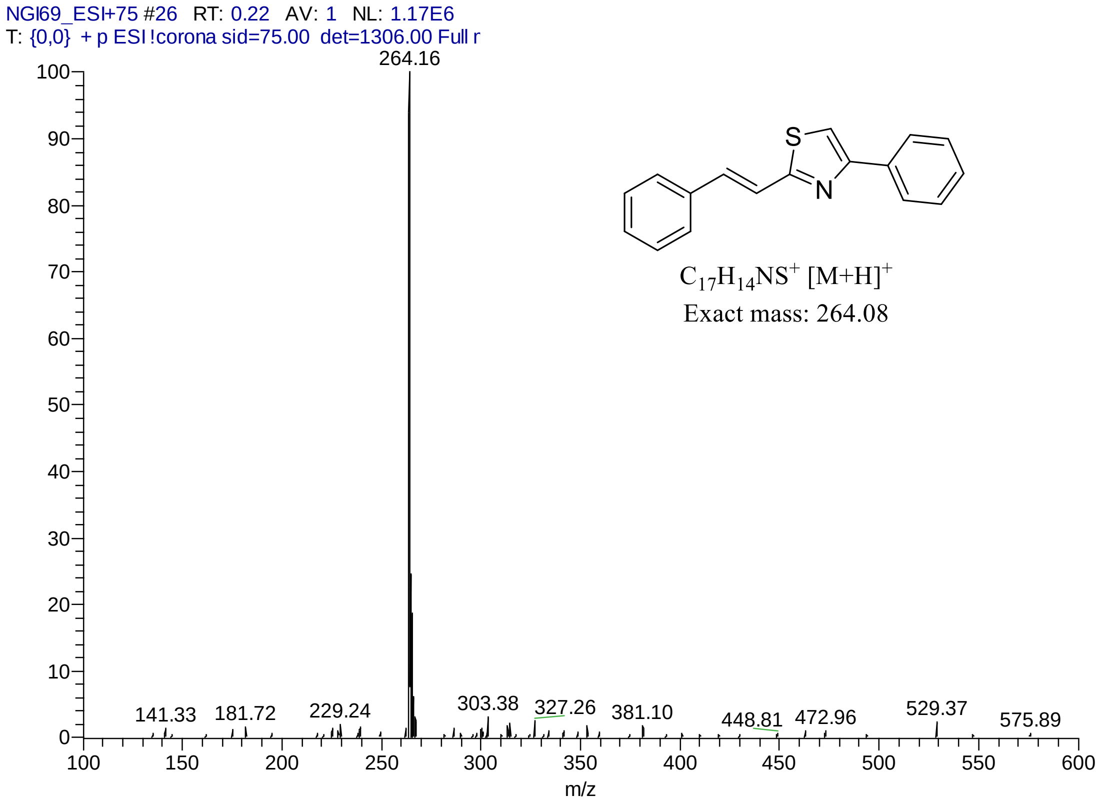


Figure 3: ESI-MS of **6a**.

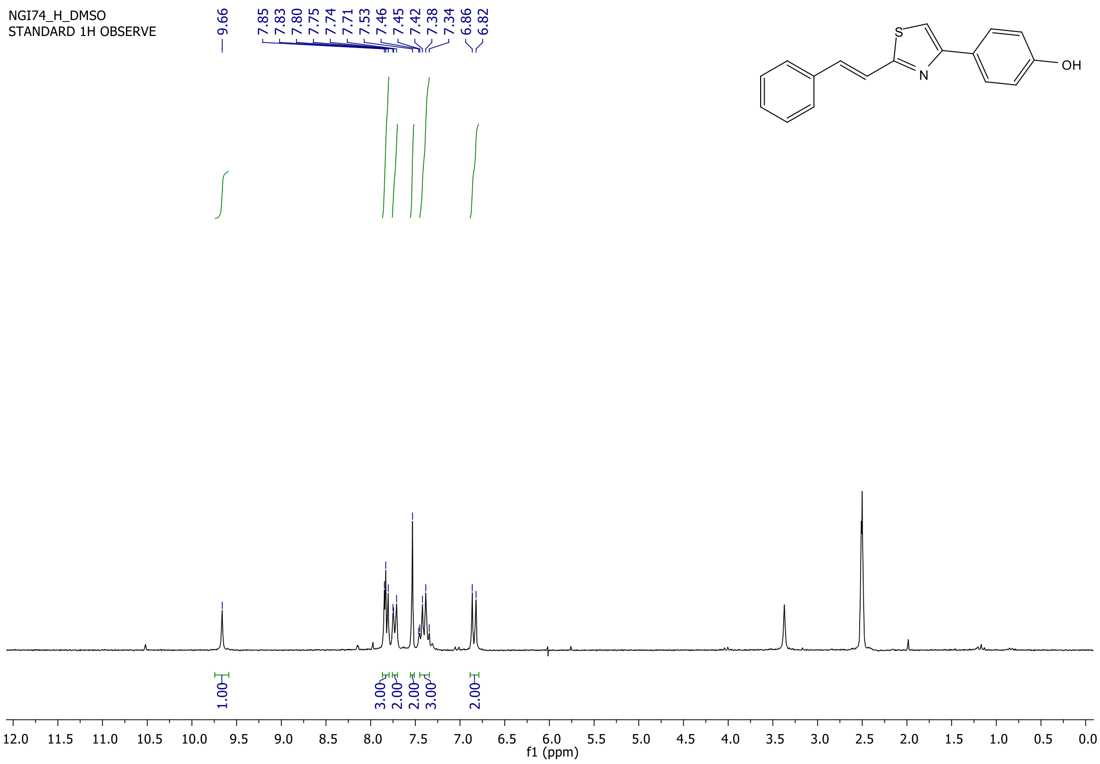


Figure 4: ^{1}H NMR of **6b** in DMSO.

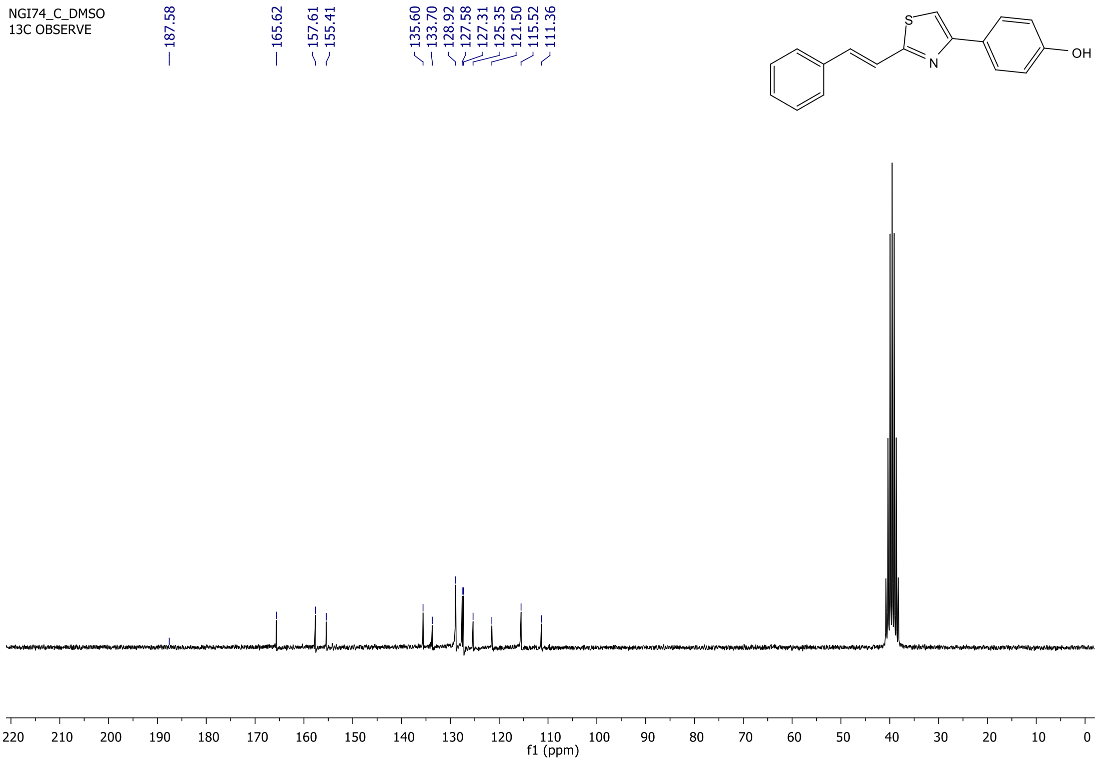


Figure 5: ^{13}C NMR of **6b** in DMSO.

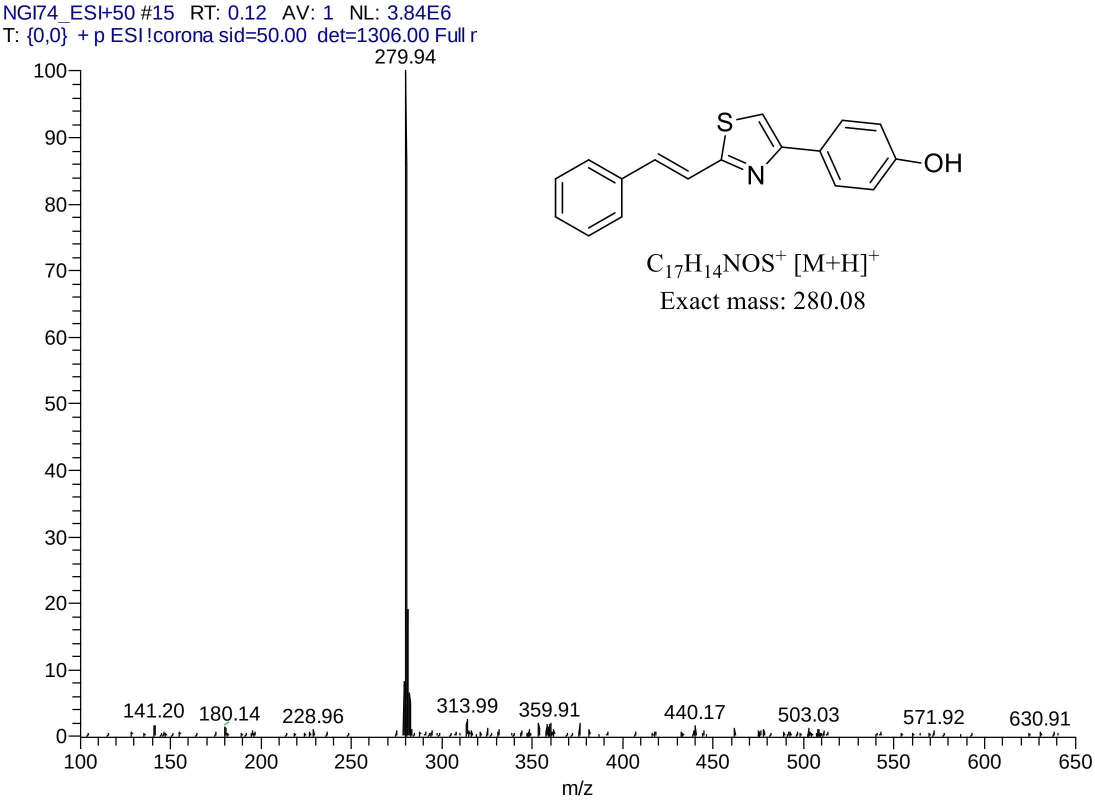


Figure 6: ESI-MS of **6b**.

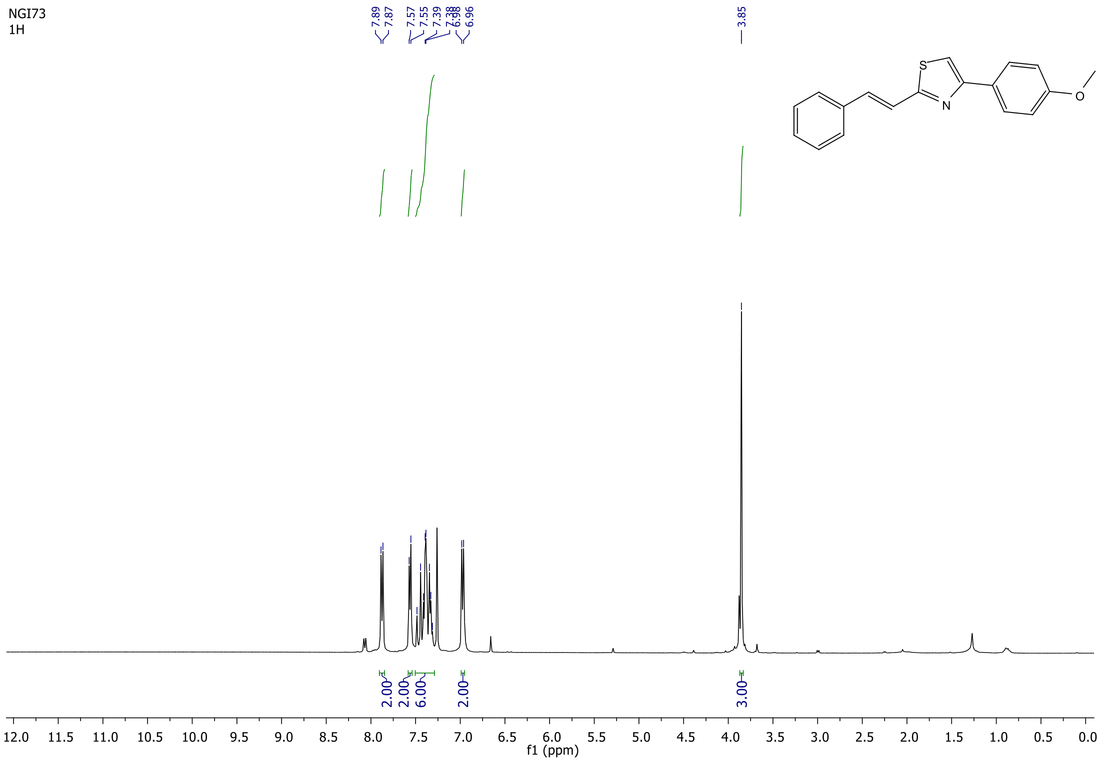


Figure 7: ^{1}H NMR of **6c** in $CDCl_3$.

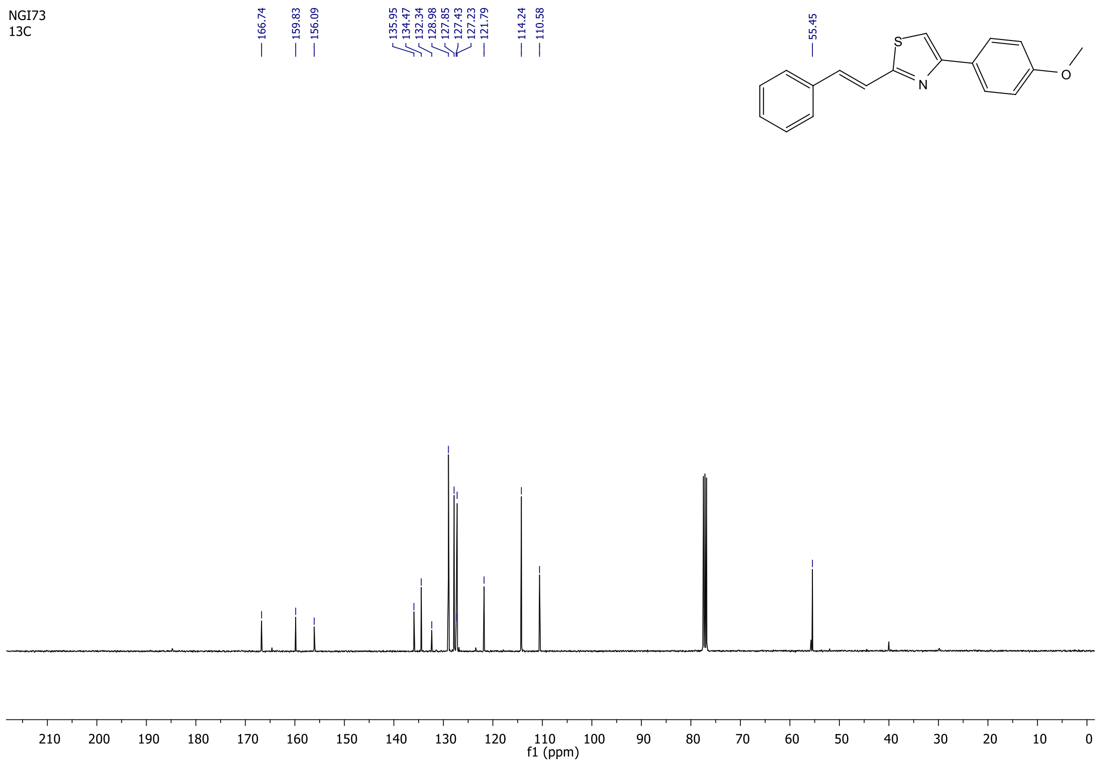


Figure :8 ^{13}C NMR of **6c** in $CDCl_3$.

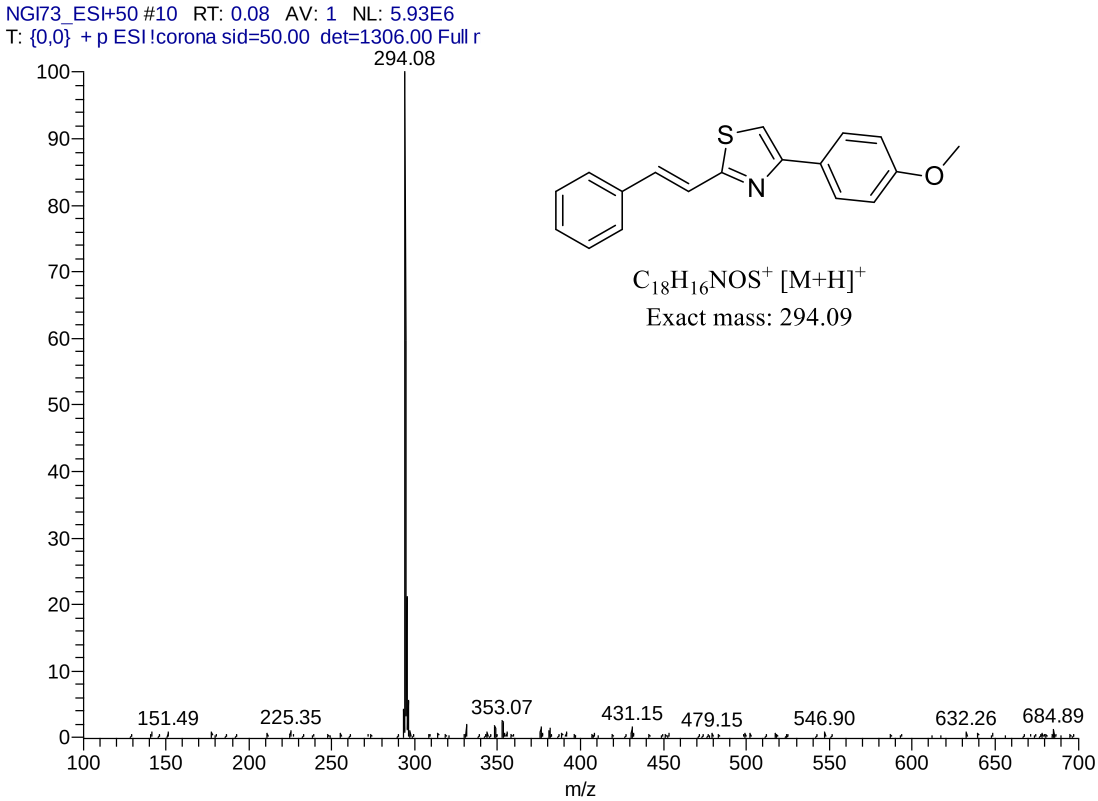


Figure 9: ESI-MS of **6c**.

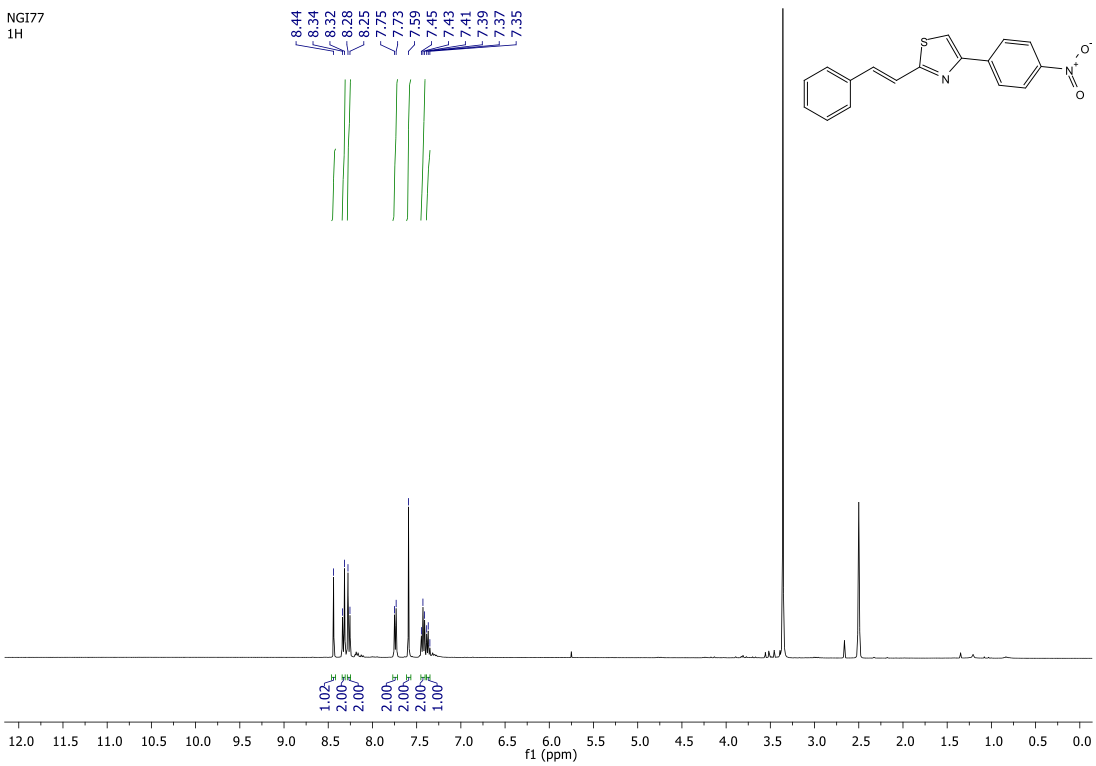


Figure 10: ^{1}H NMR of **6d** in DMSO.

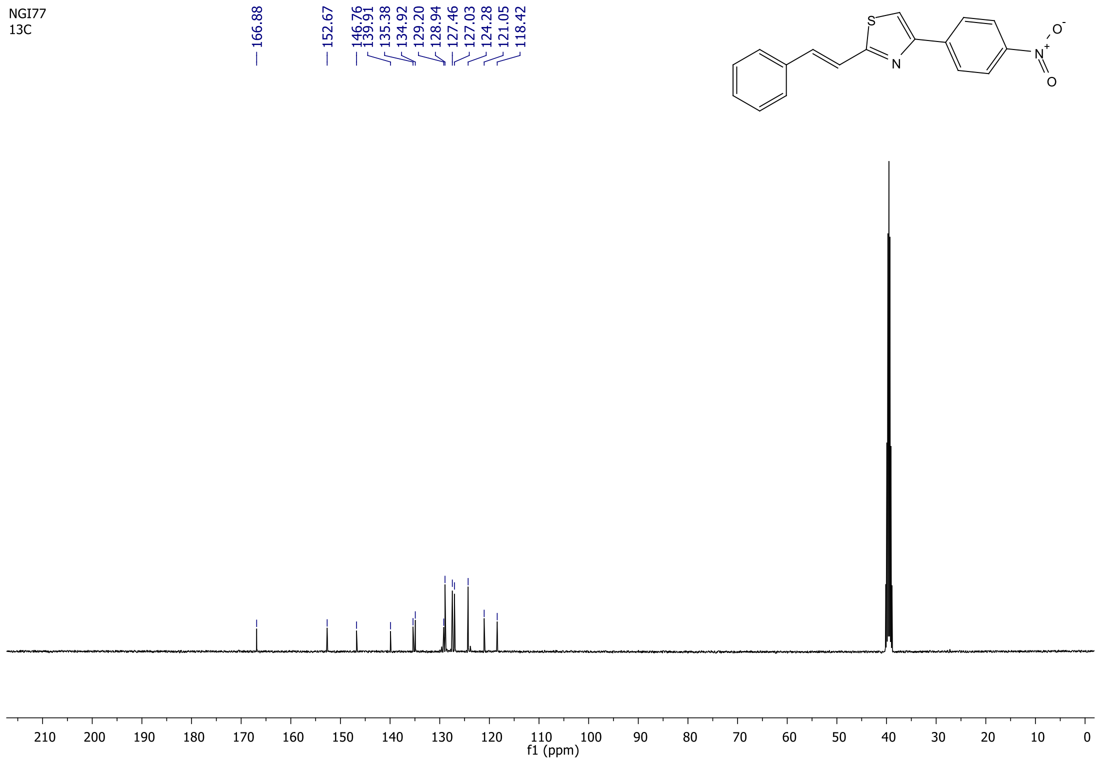


Figure 11: ^{13}C NMR of **6d** in DMSO.

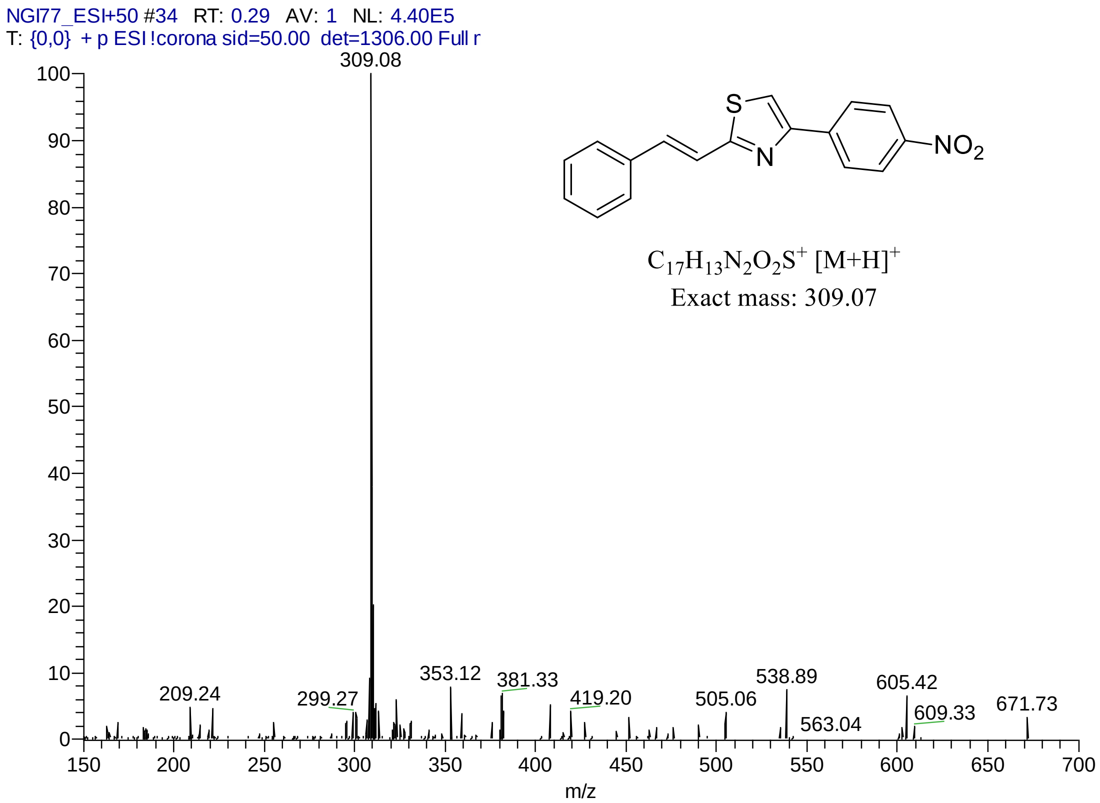


Figure 12: ESI-MS of **6d**.

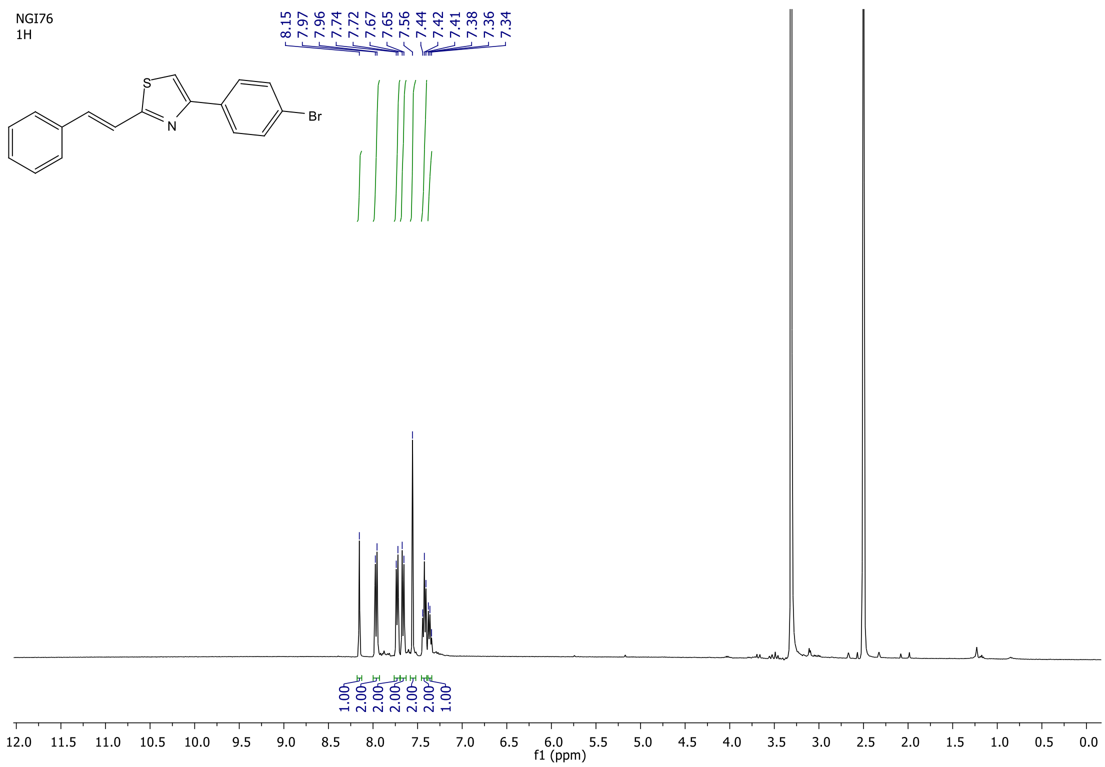


Figure 13: ^{1}H NMR of **6e** in DMSO.

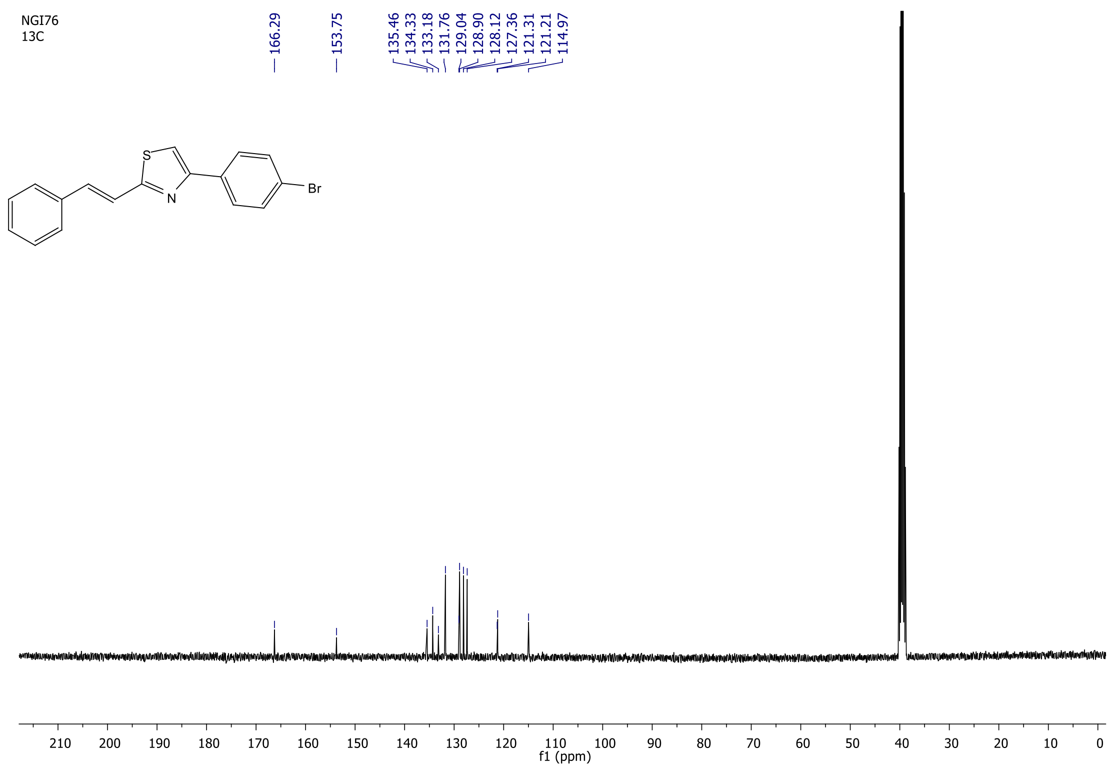


Figure 14: ^{13}C NMR of **6e** in DMSO.

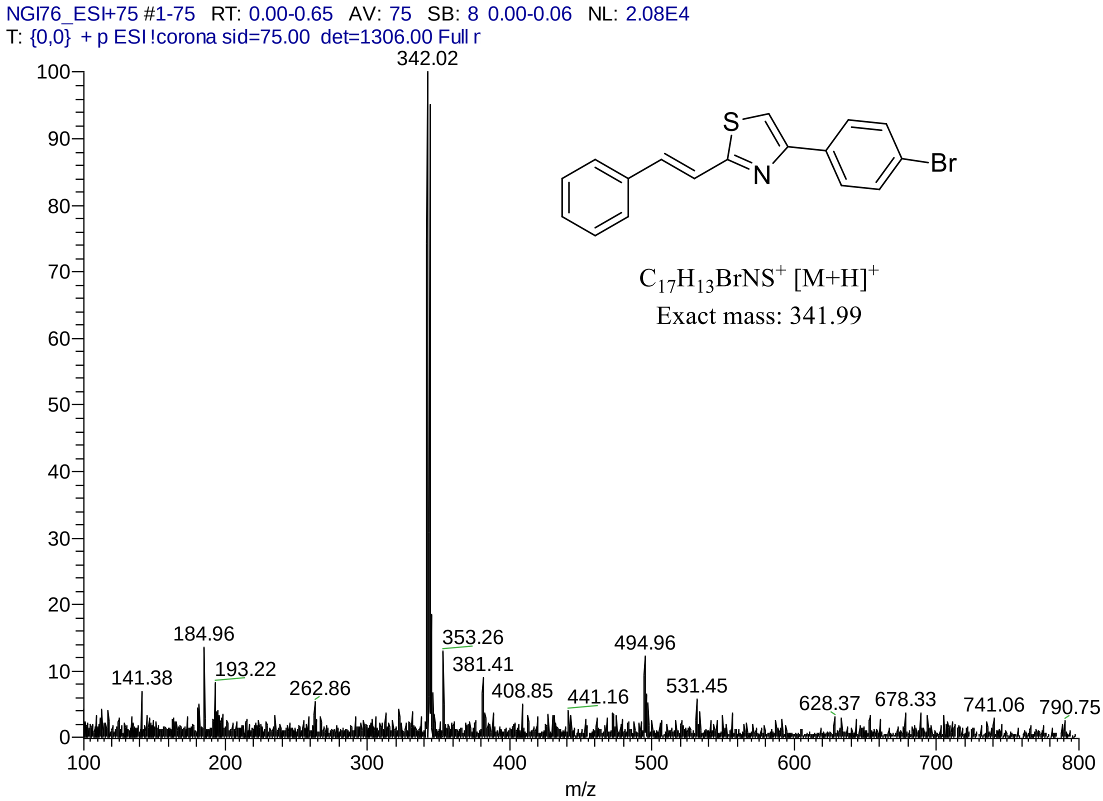


Figure 15: ESI-MS of **6e**.

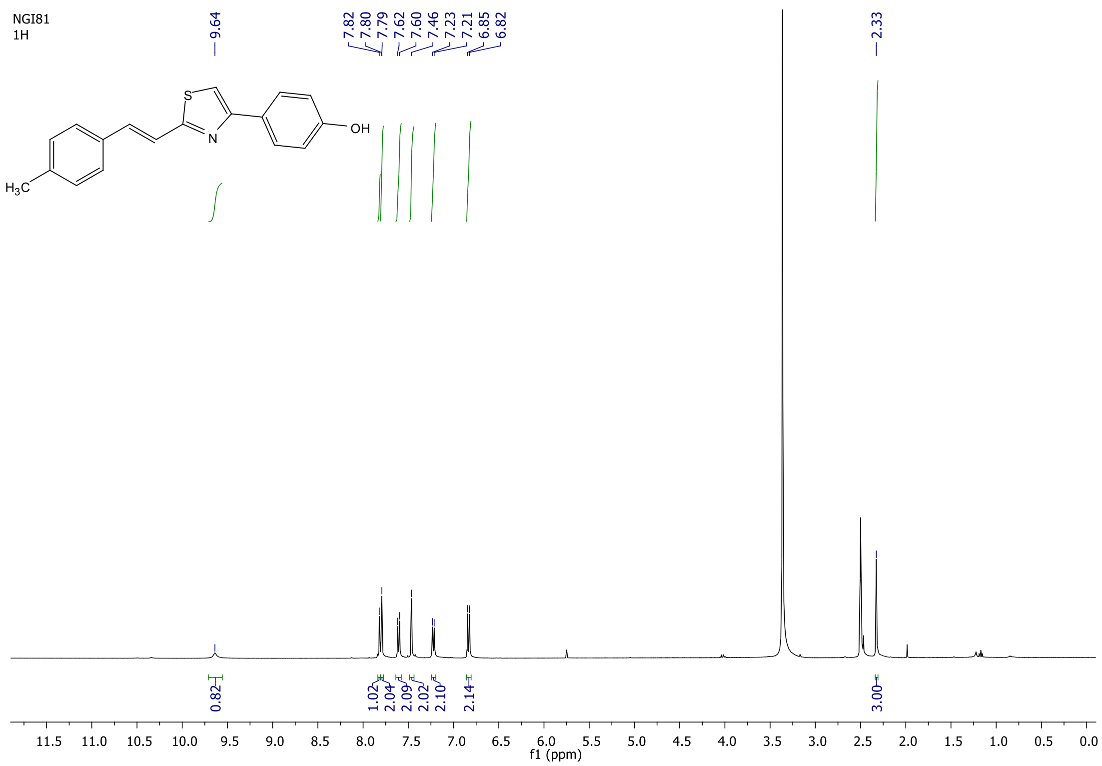


Figure 16: ^{1}H NMR of **6f** in DMSO.

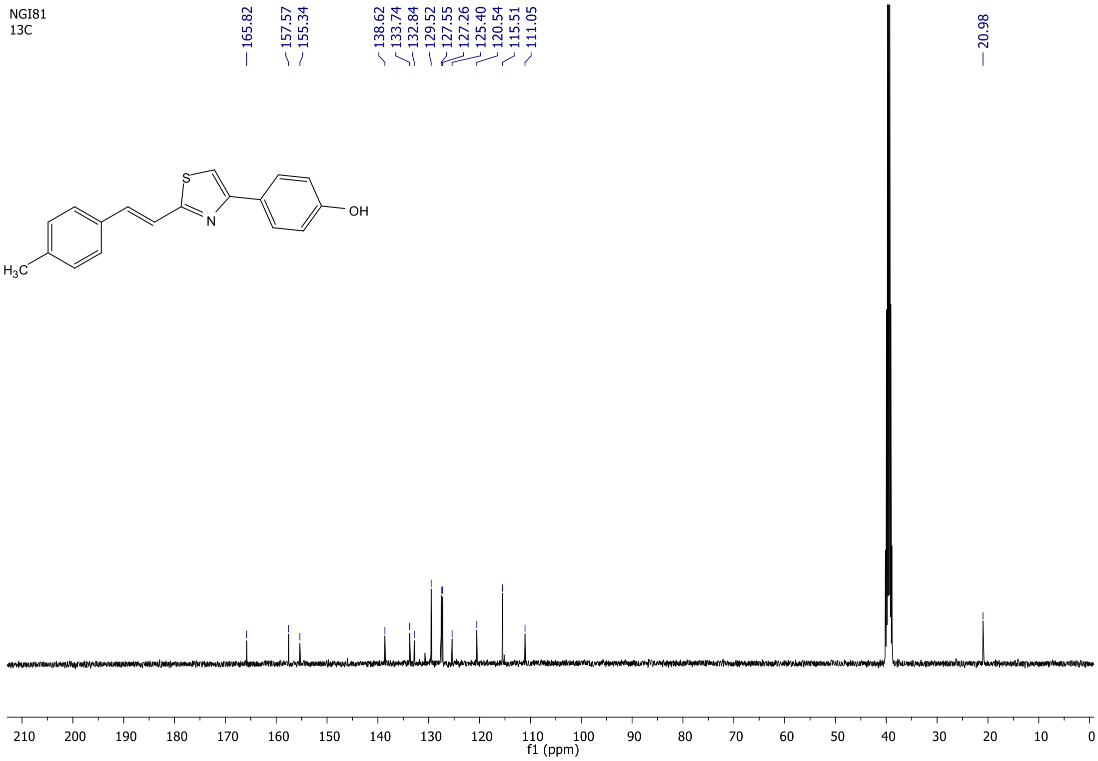


Figure 17: ^{13}C NMR of **6f** in DMSO.

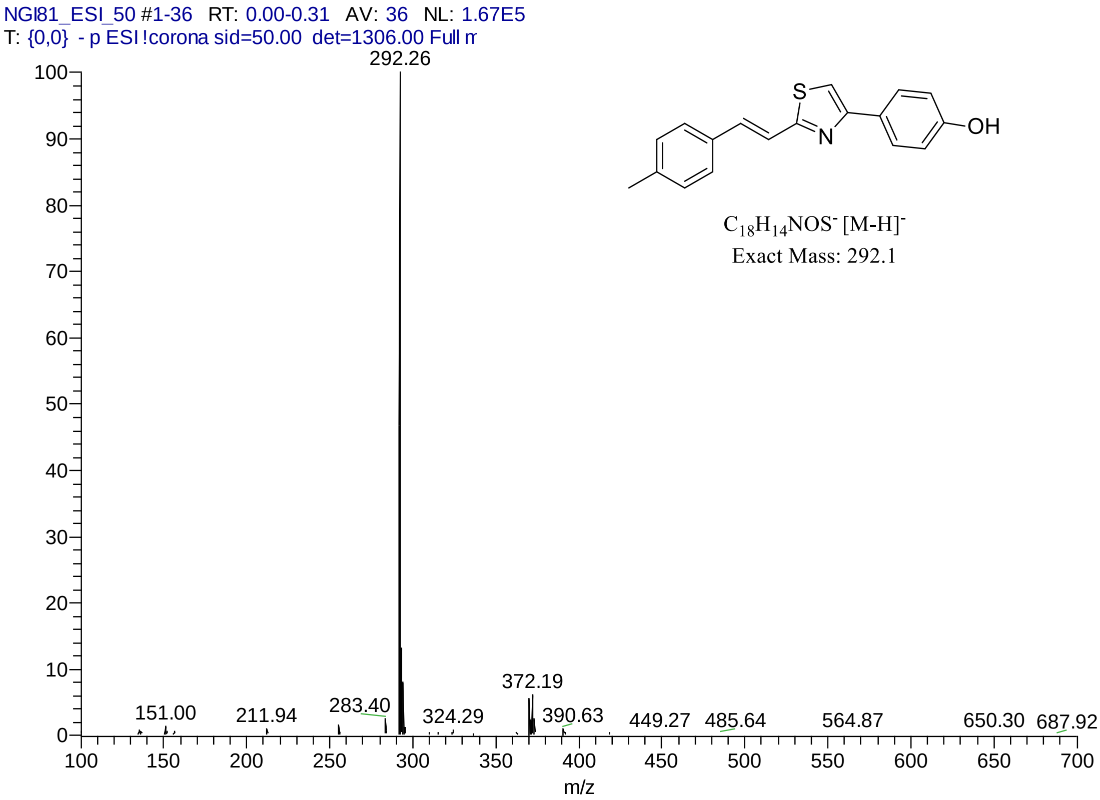


Figure 18: ESI-MS of **6f**.

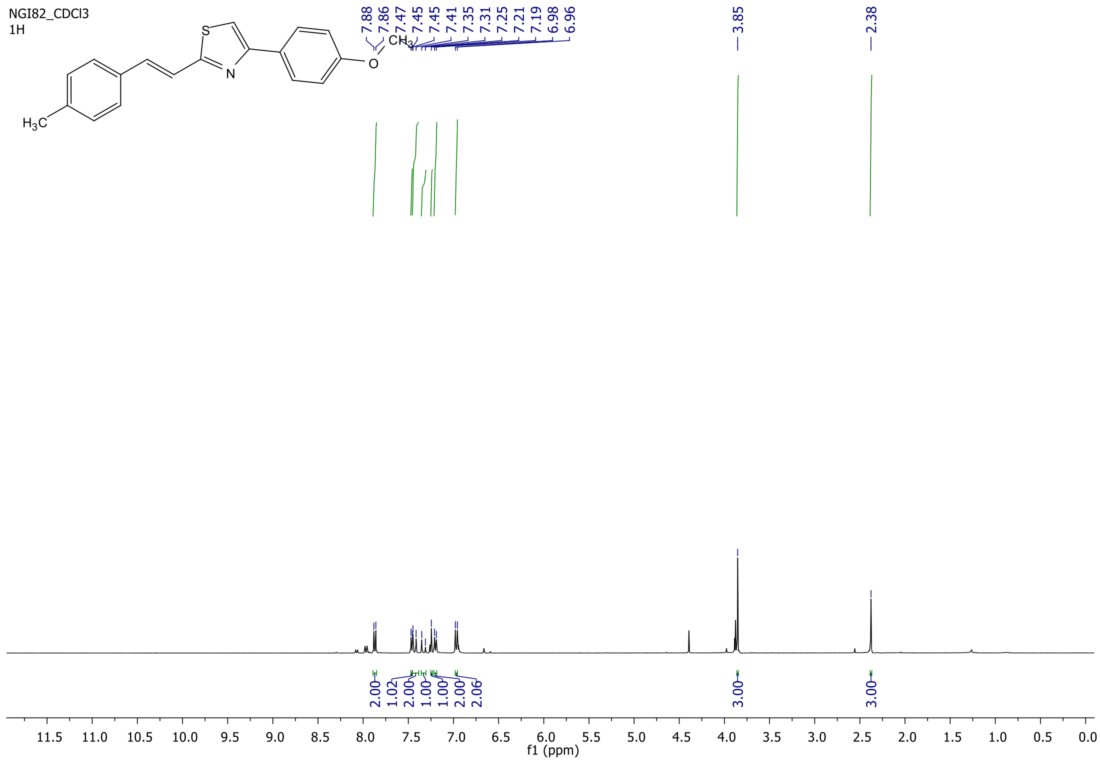


Figure 19: ^{1}H NMR of **6g** in $CDCl_3$.

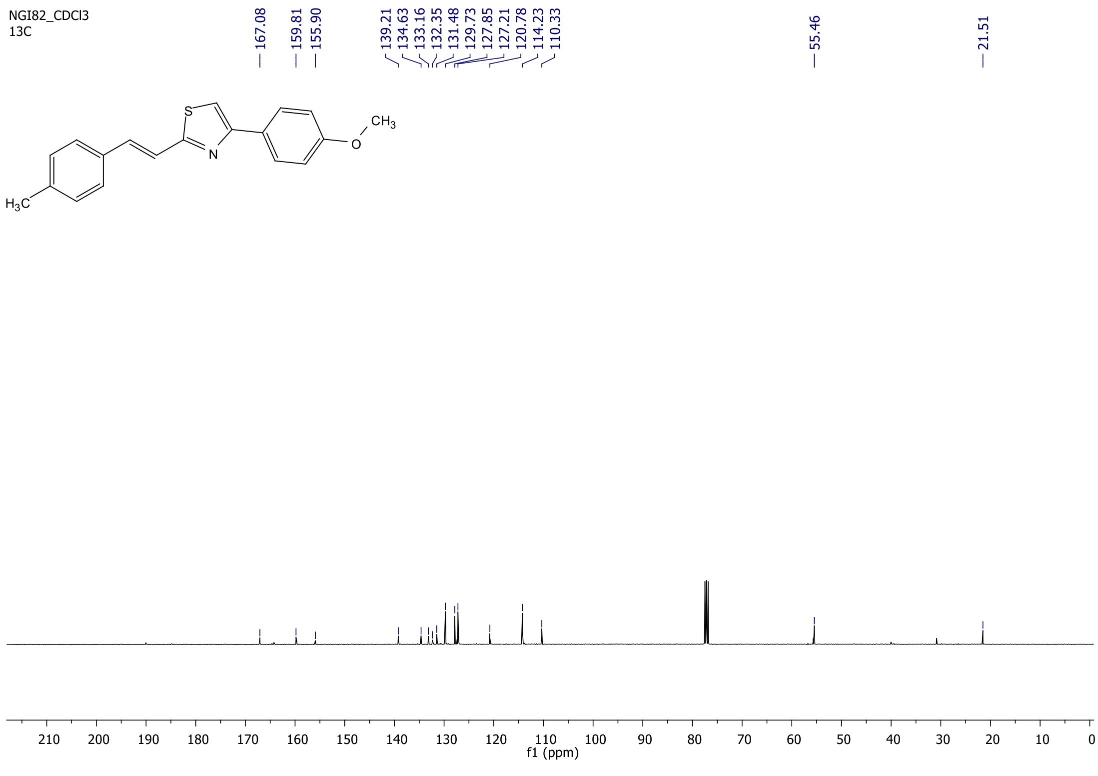


Figure 20: ^{13}C NMR of **6g** in $CDCl_3$.

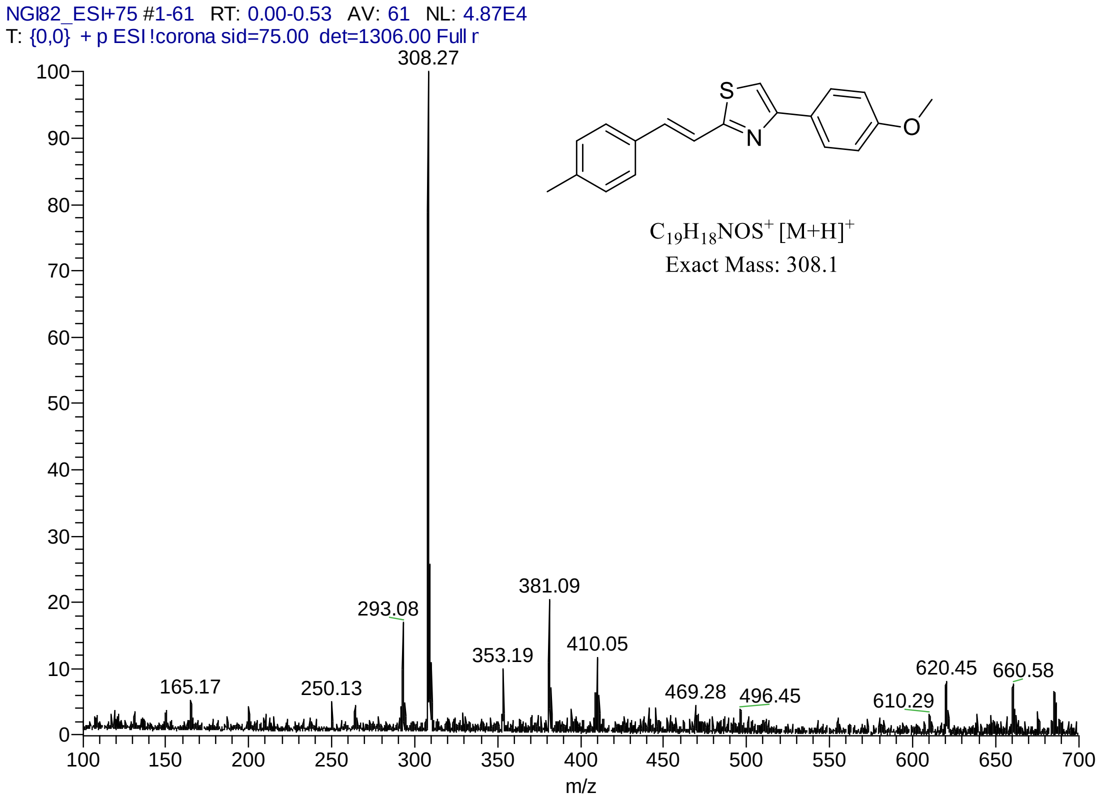


Figure 21: ESI-MS of **6g**.

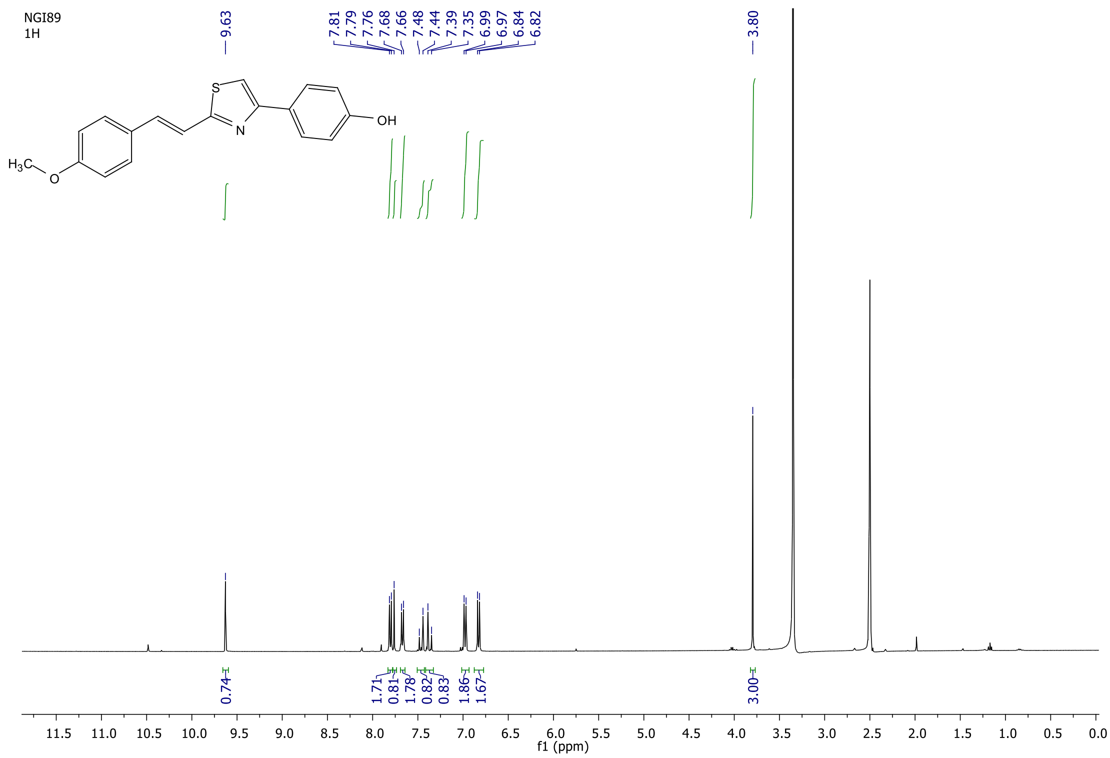


Figure 22: [1]H NMR of **6h** in DMSO.

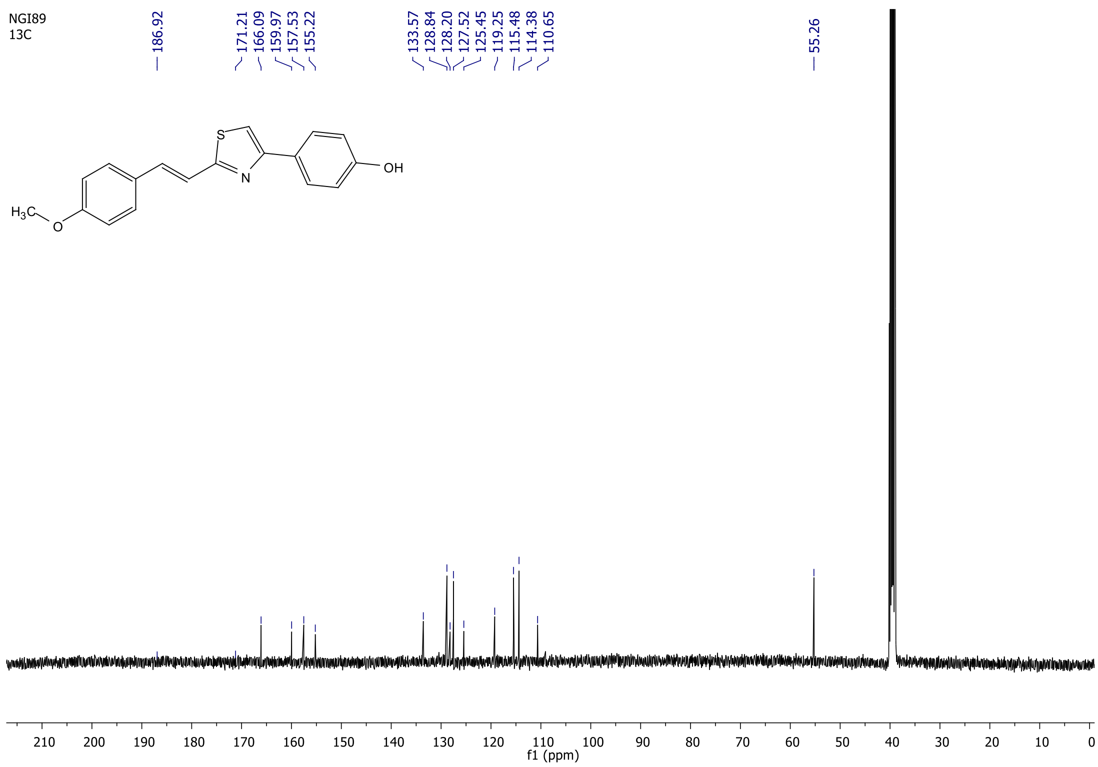


Figure 23: ^{13}C NMR of **6h** in DMSO.

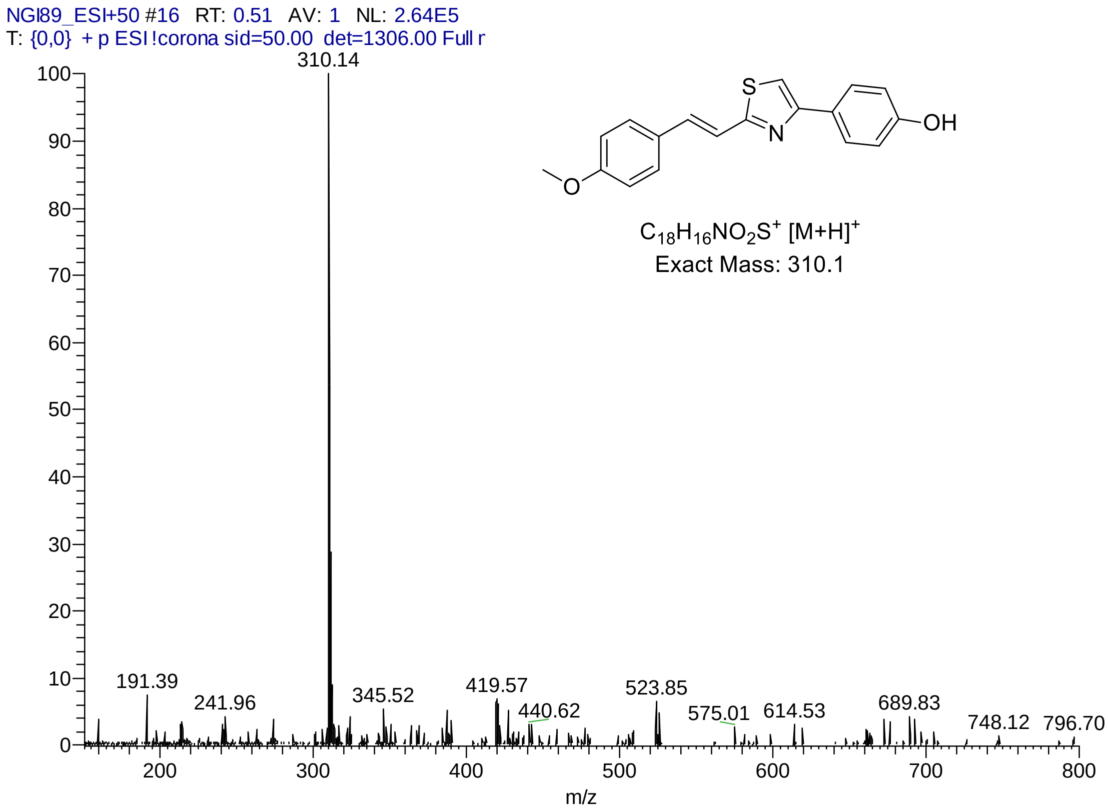


Figure 24: ESI-MS of **6h**.

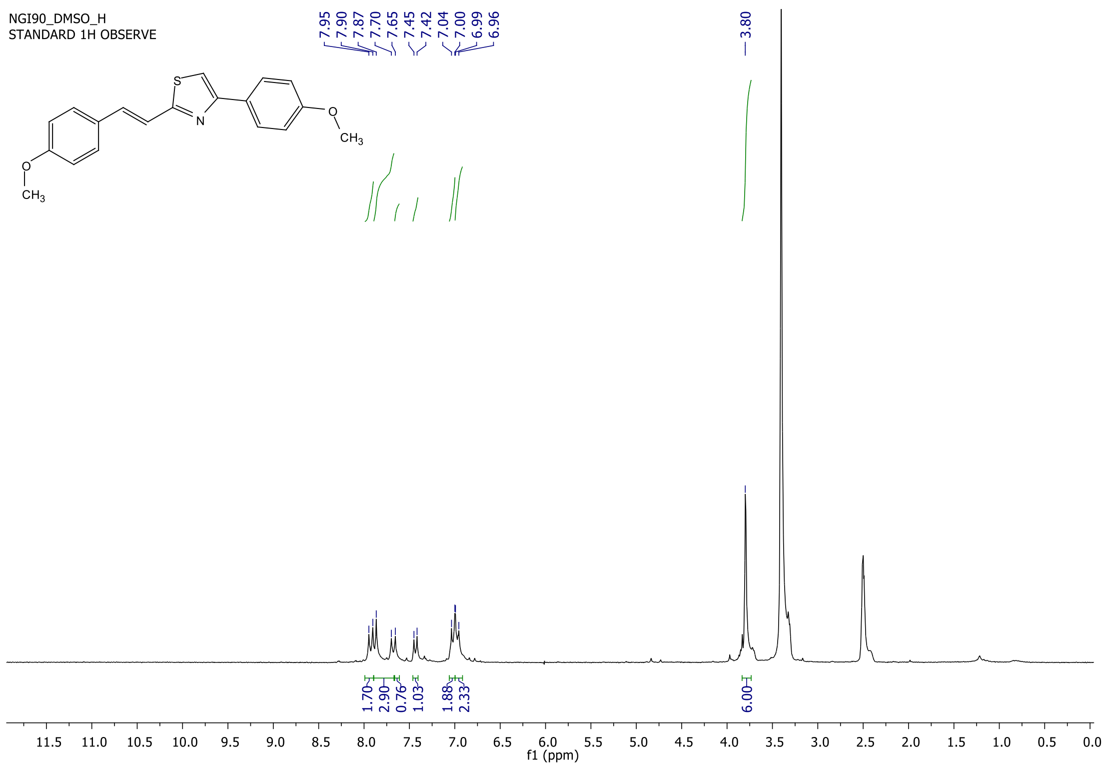


Figure 25: ^{1}H NMR of **6i** in DMSO.

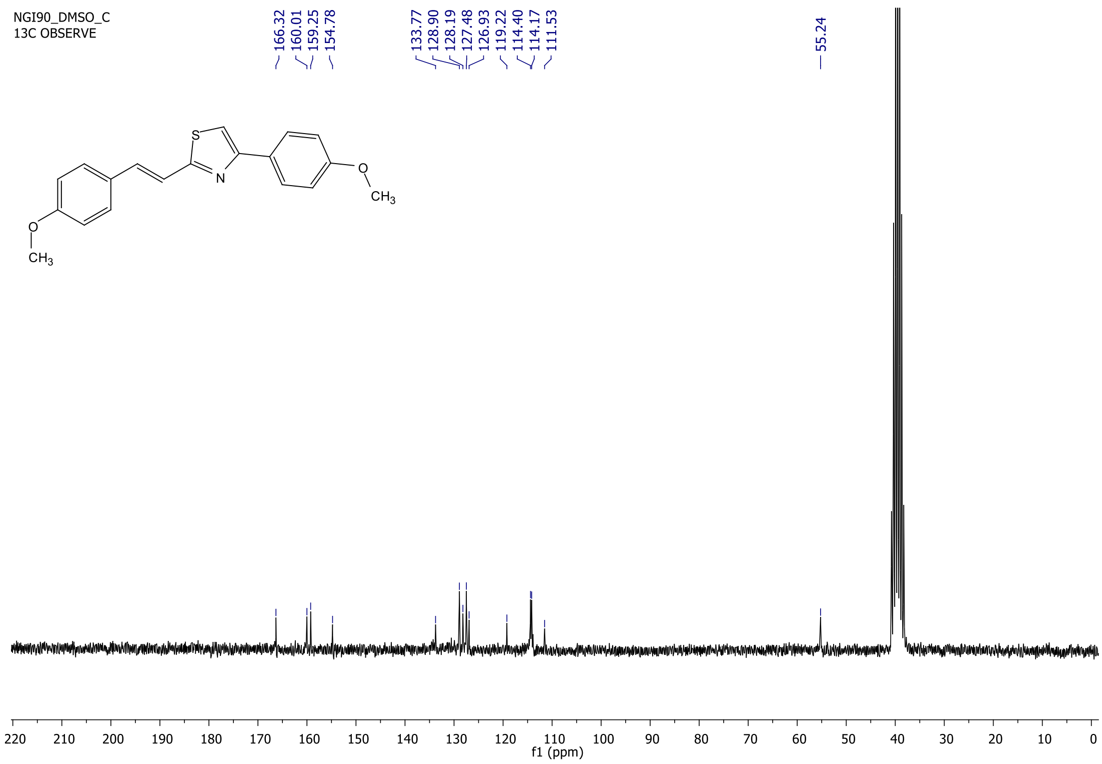


Figure 26: ^{13}C NMR of **6i** in DMSO.

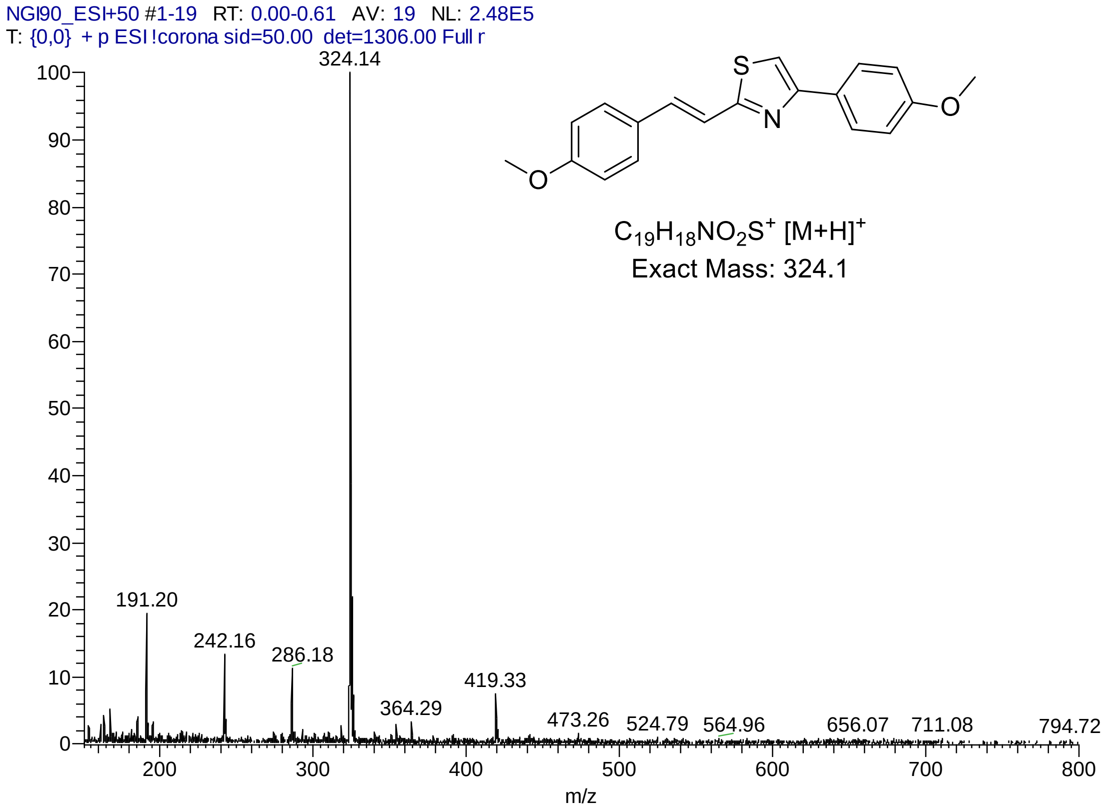


Figure 27: ESI-MS of **6i**.

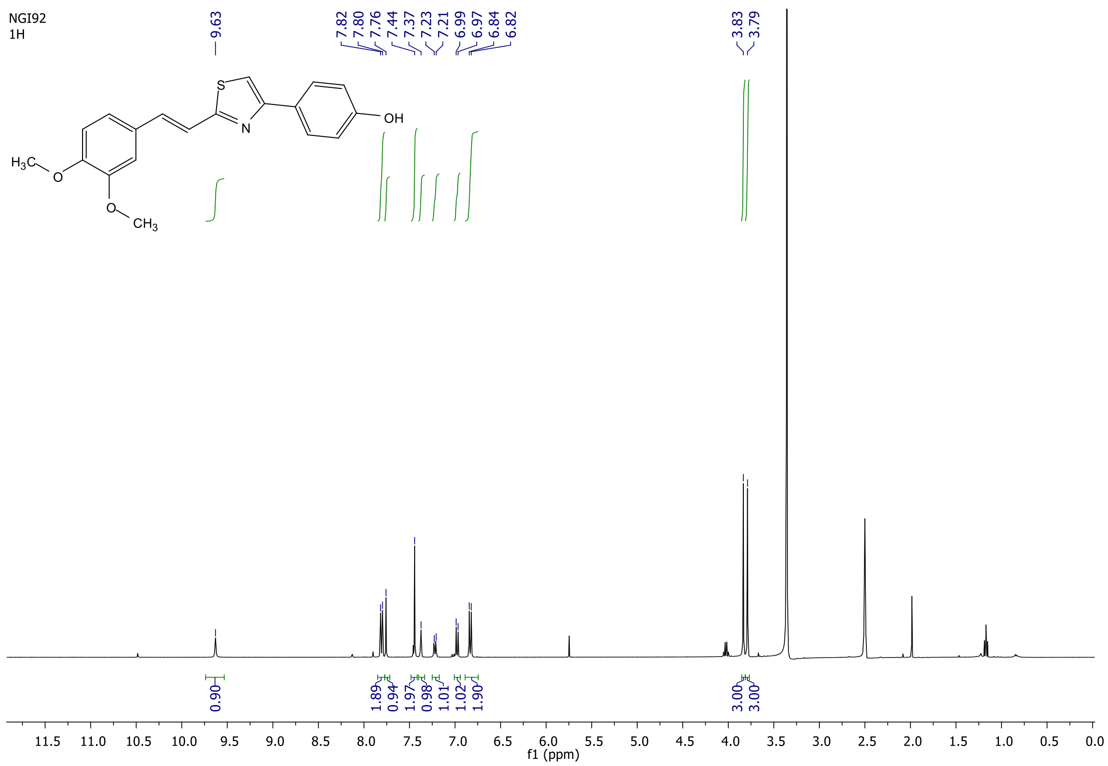


Figure 28: ^{1}H NMR of **6j** in DMSO.

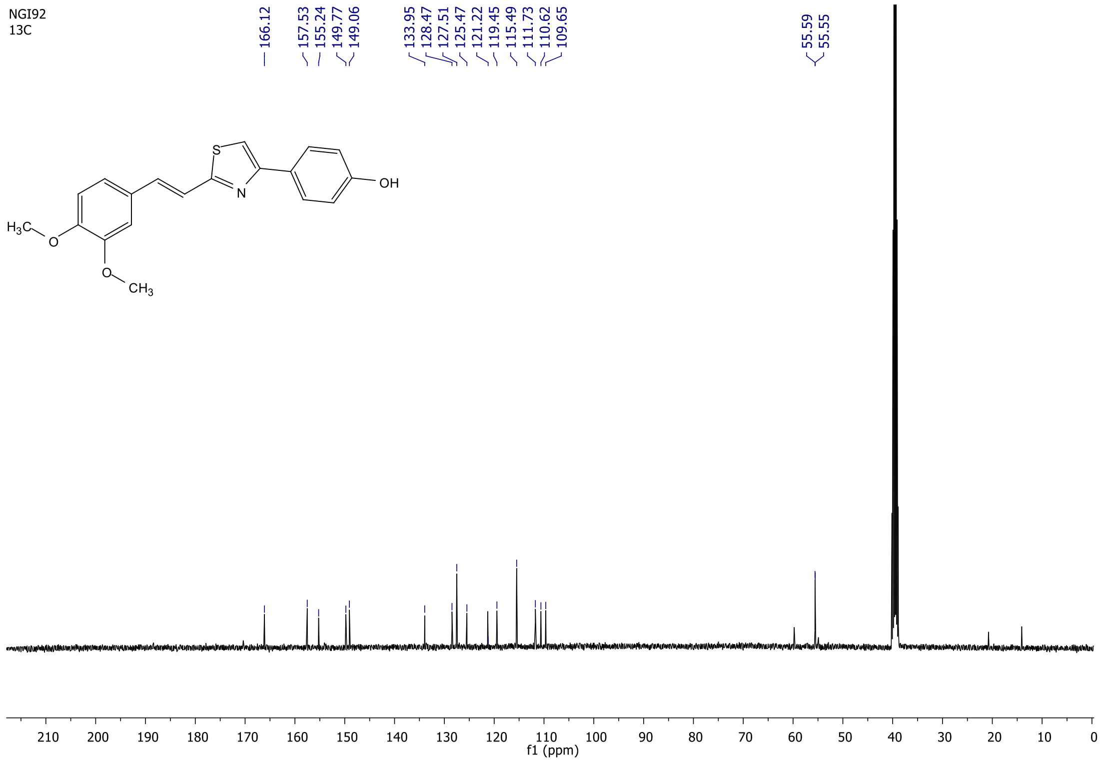


Figure 29: ^{13}C NMR of **6j** in DMSO.

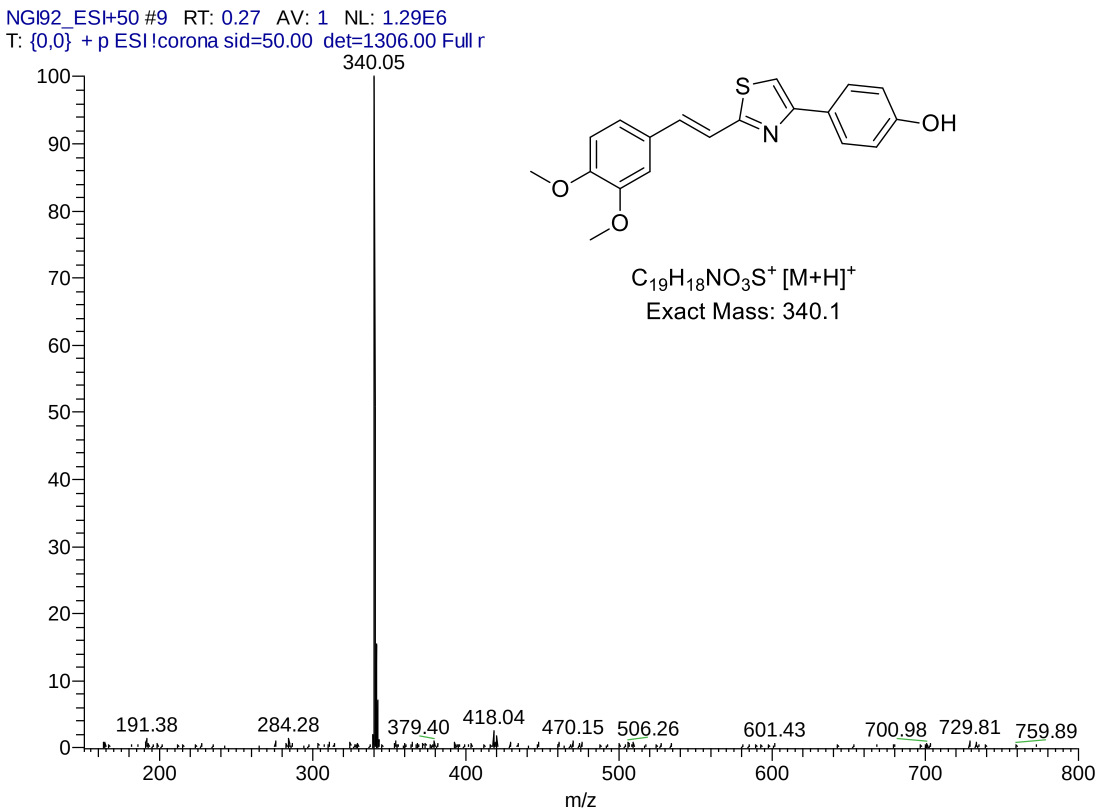


Figure 30: ESI-MS of **6j**.

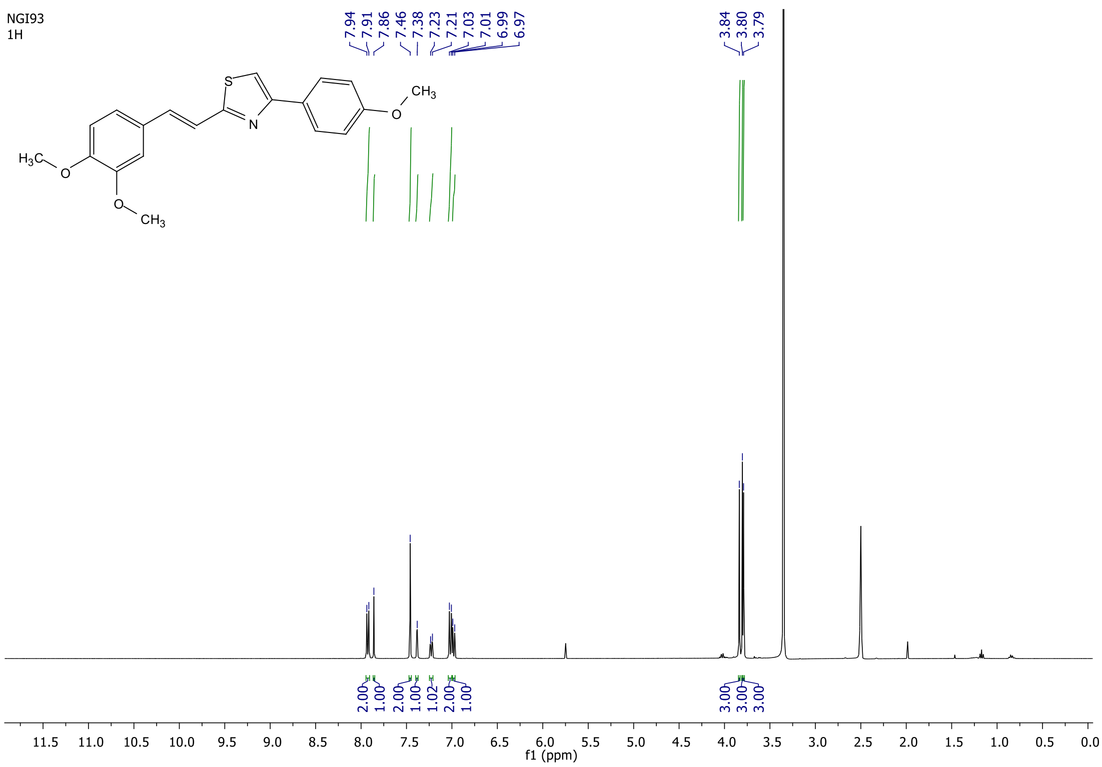


Figure 31: ^{1}H NMR of **6k** in DMSO.

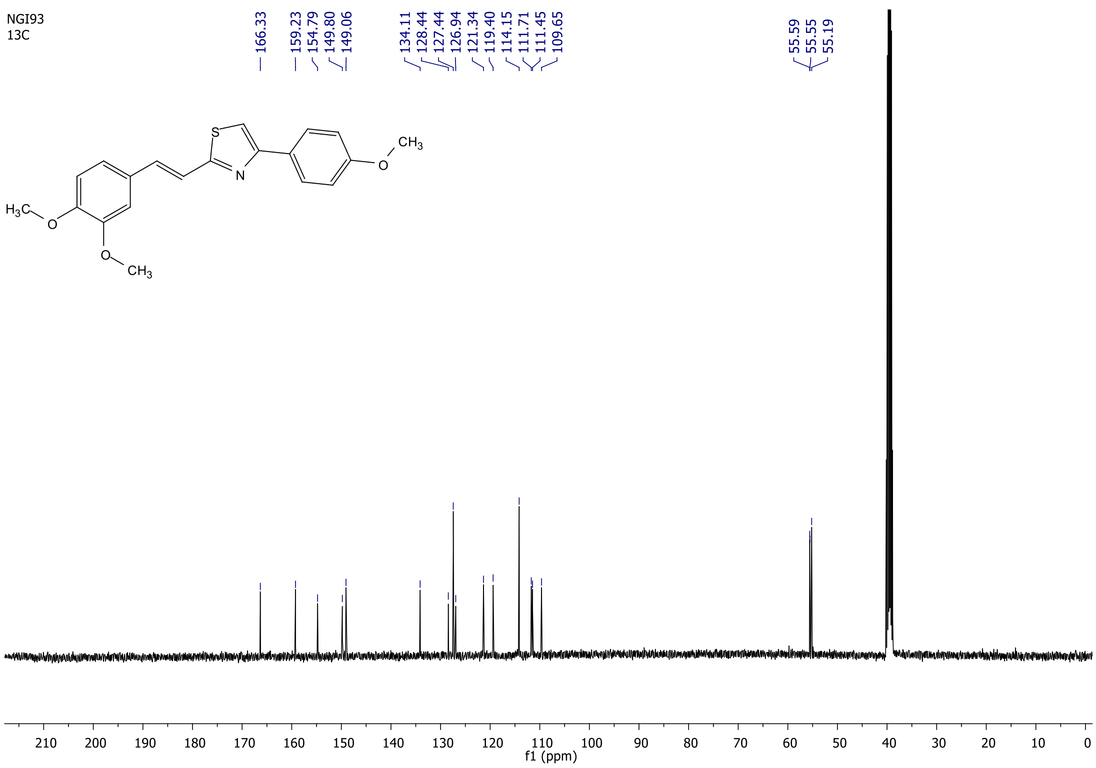


Figure 32: ^{13}C NMR of **6k** in DMSO.

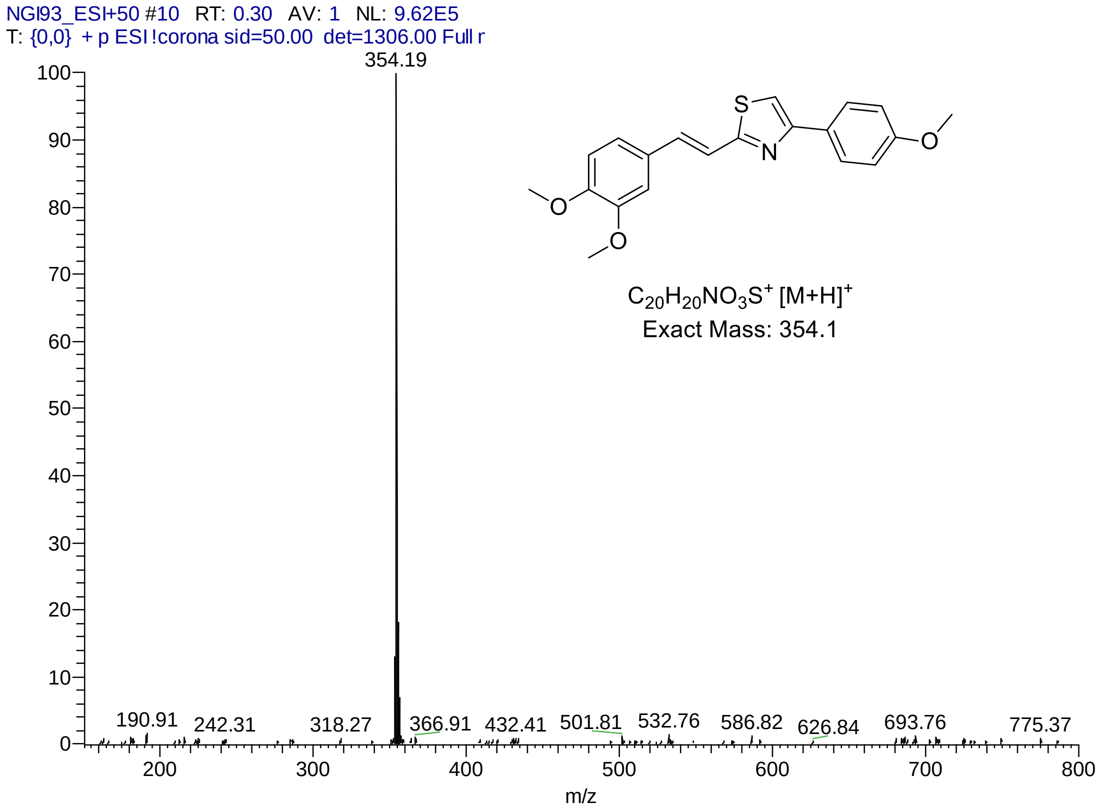


Figure 33: ESI-MS of **6k**.

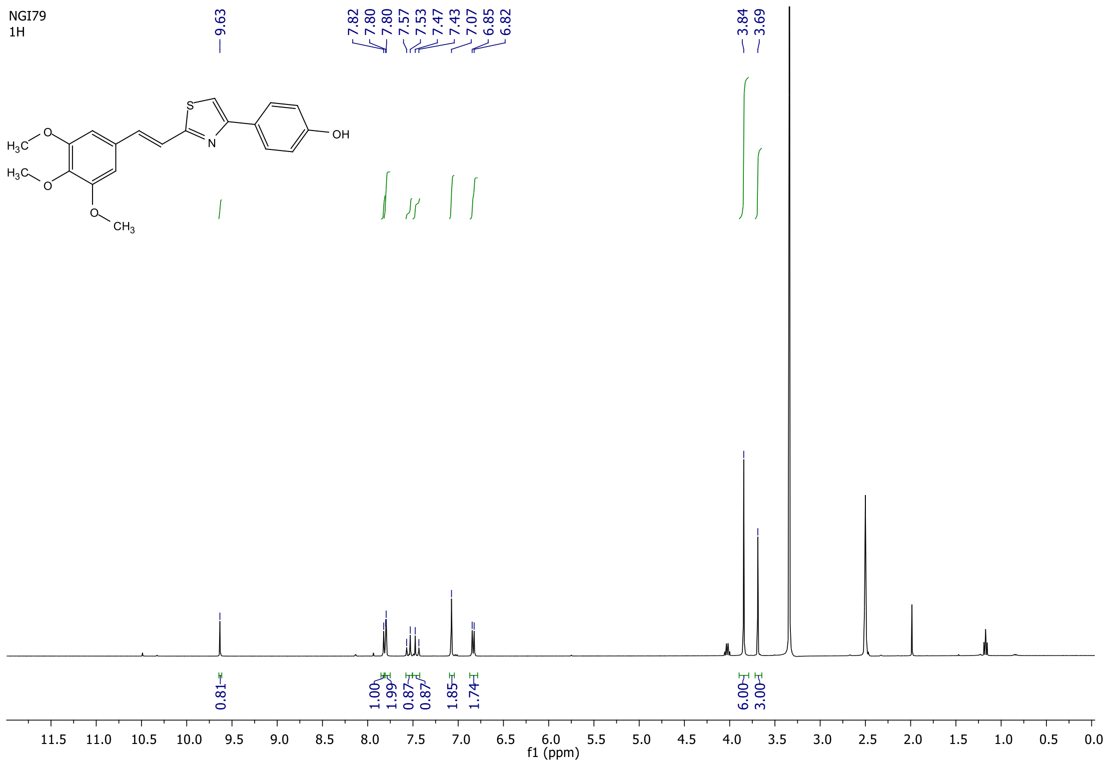


Figure 34: ^{1}H NMR of **6l** in DMSO.

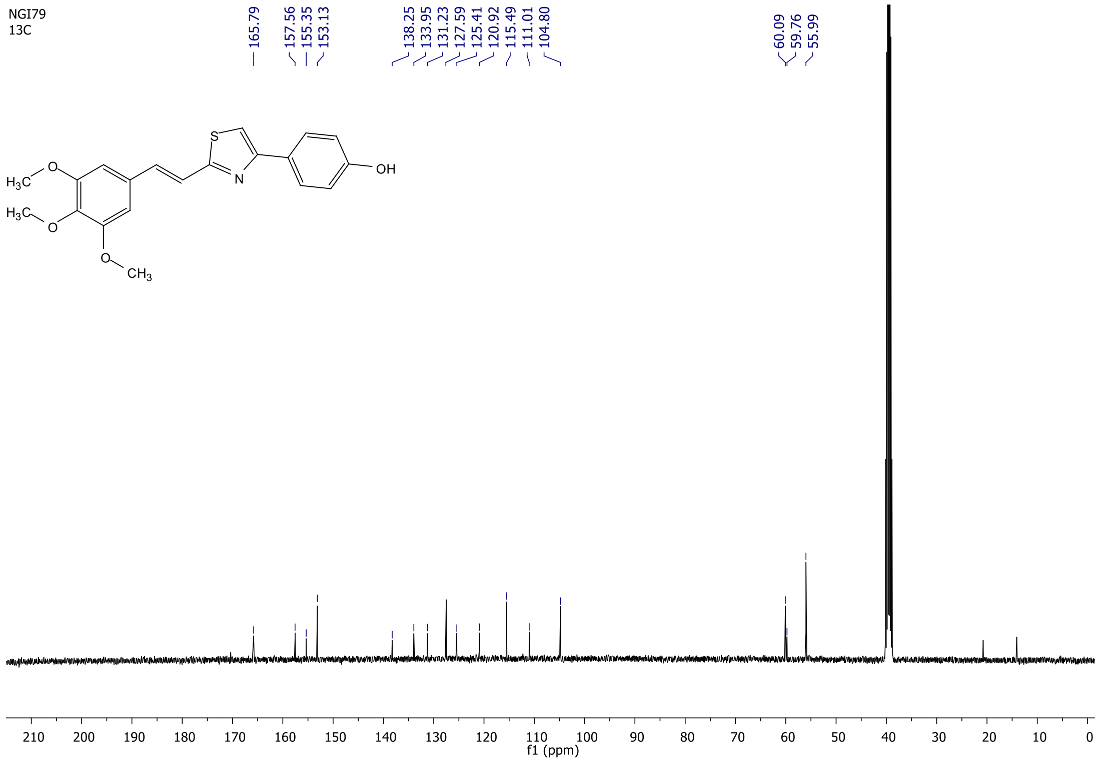


Figure 35: ^{13}C NMR of **6l** in DMSO.

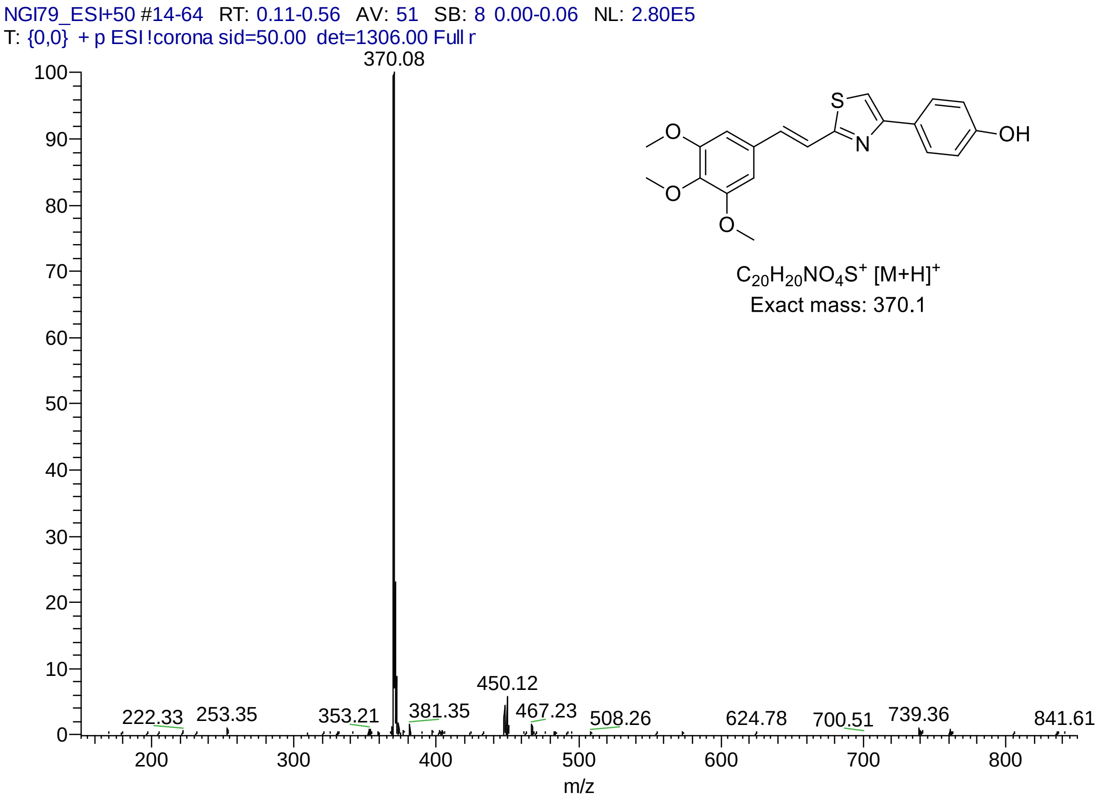


Figure 36: ESI-MS of **6l**.

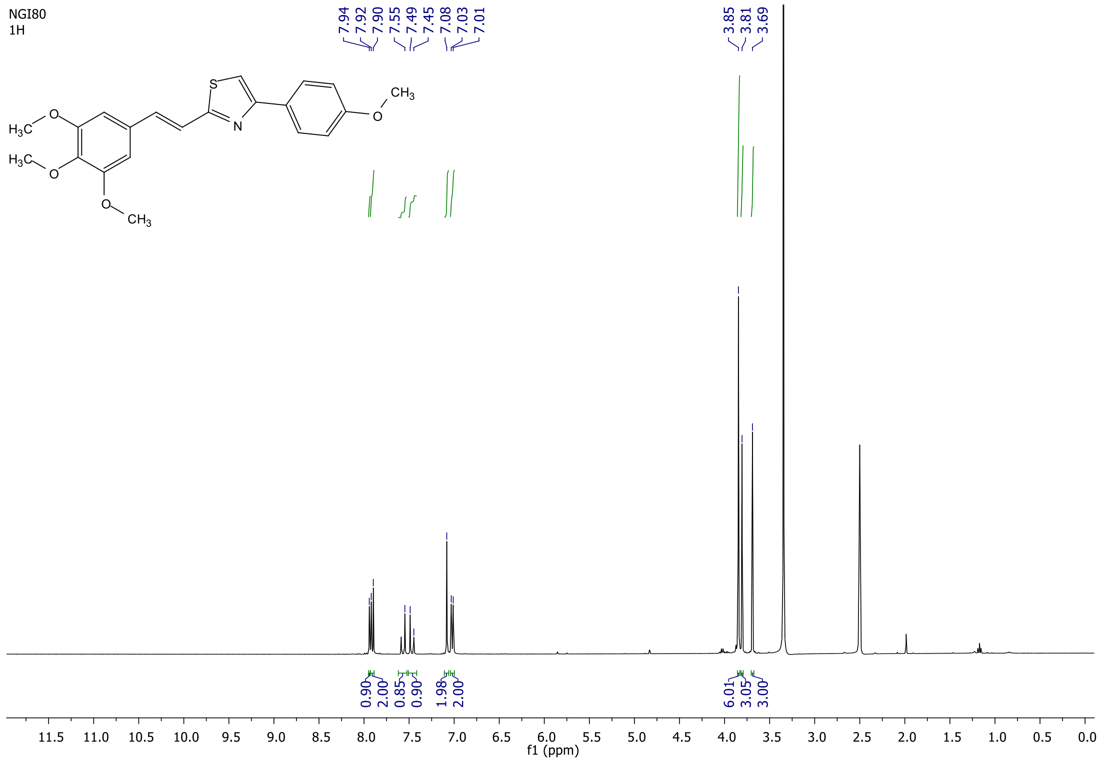


Figure 37: ^{1}H NMR of **6m** in DMSO.

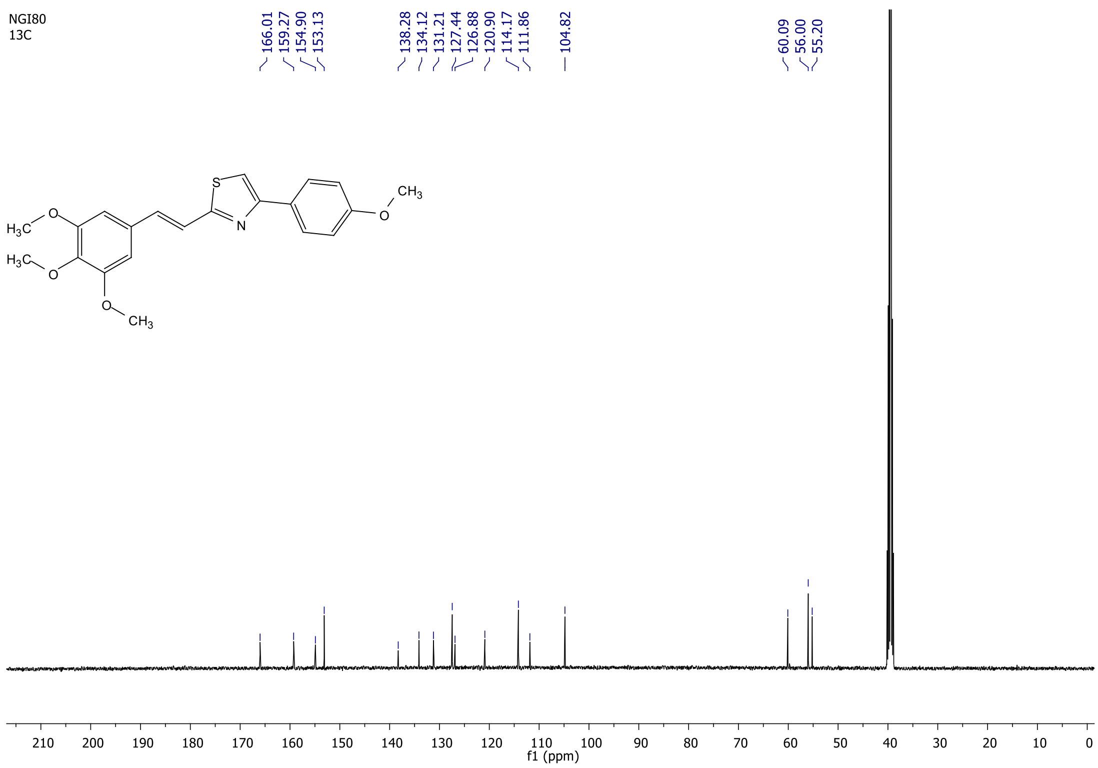


Figure 38: ^{13}C NMR of **6m** in DMSO.

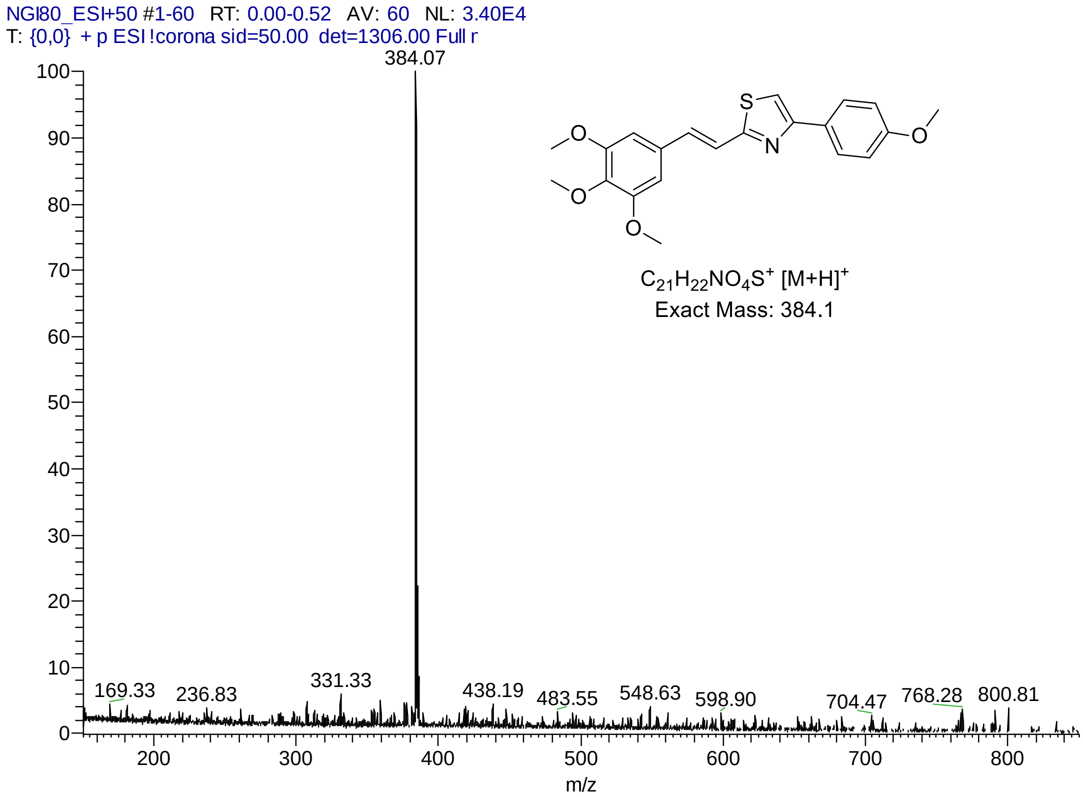


Figure 39: ESI-MS of **6m**.

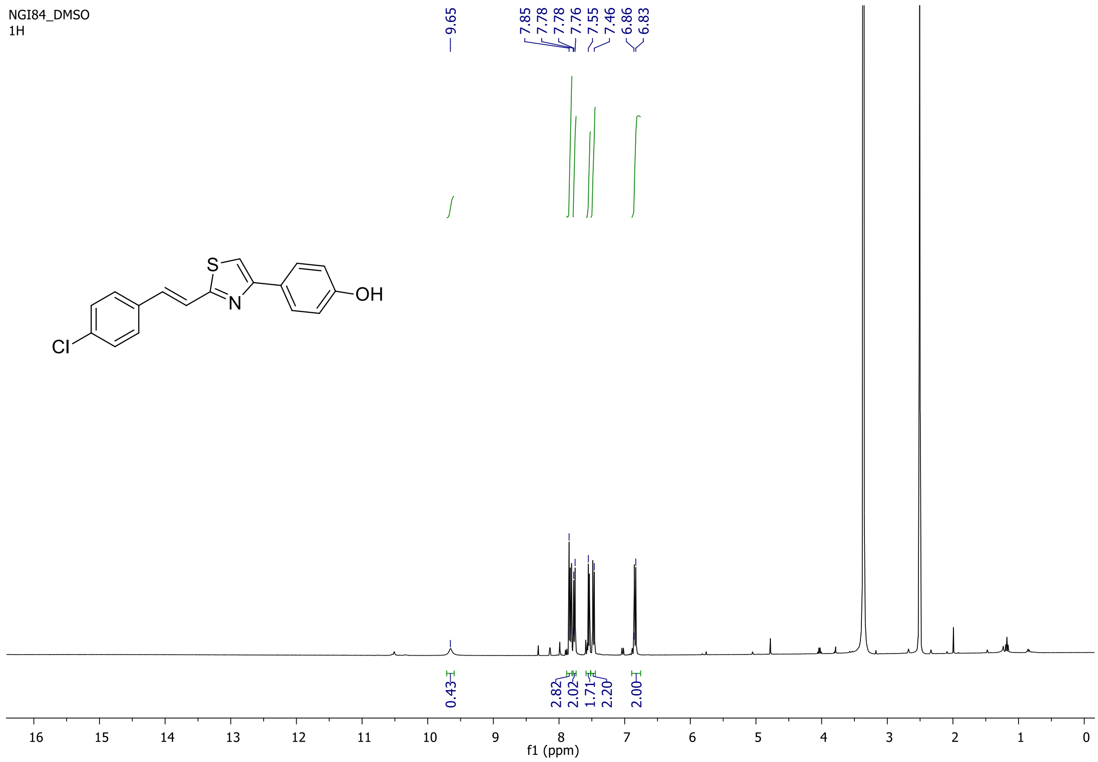


Figure 40: ^{1}H NMR of **6n** in DMSO.

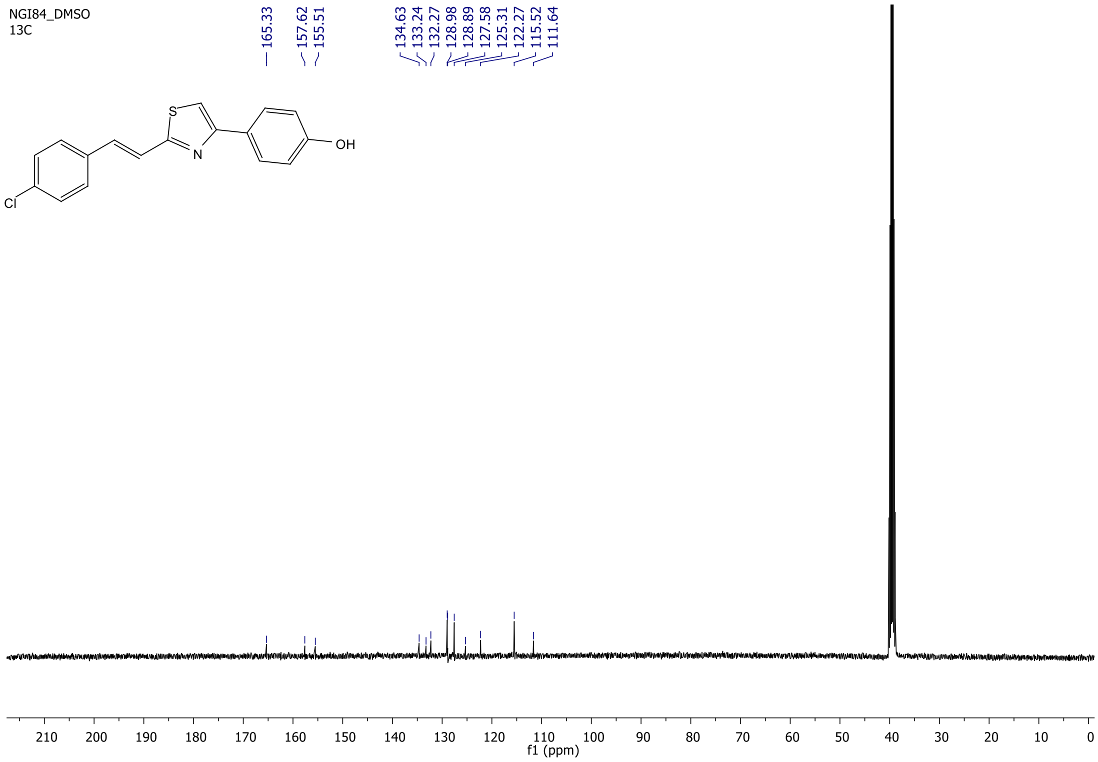


Figure 41: ^{13}C NMR of **6n** in DMSO.

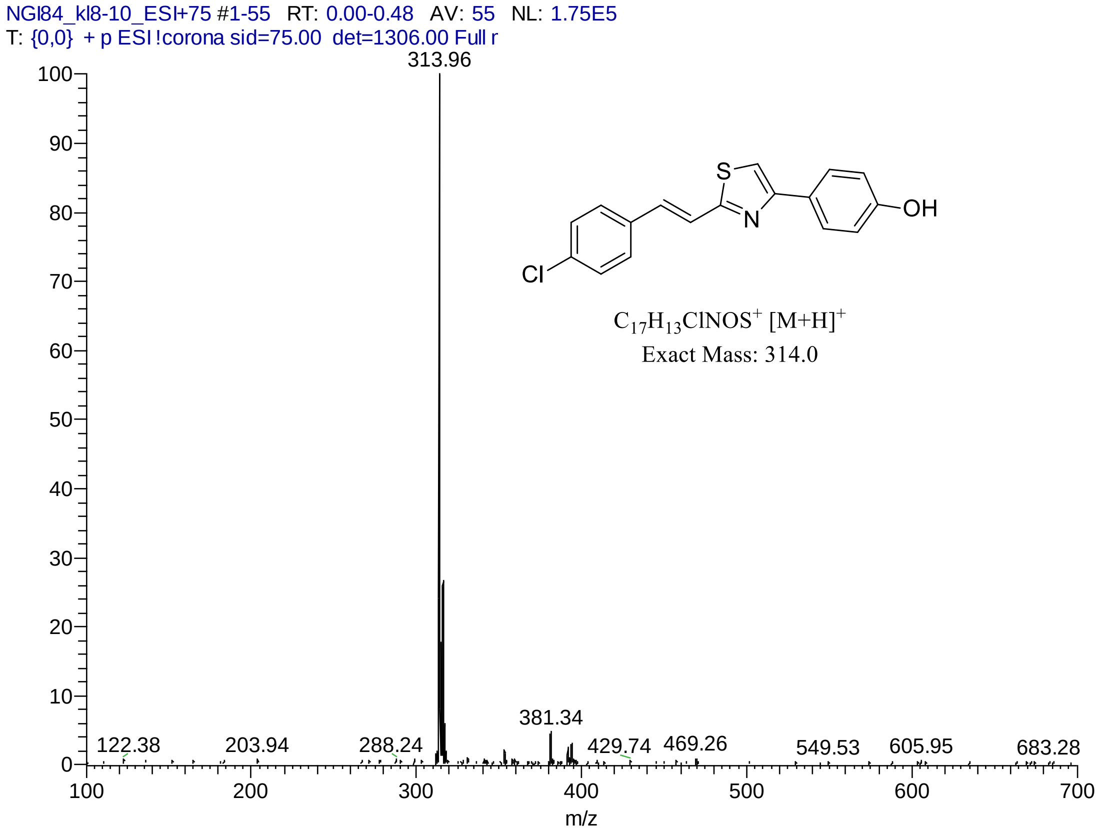


Figure 42: ESI-MS of **6n**.

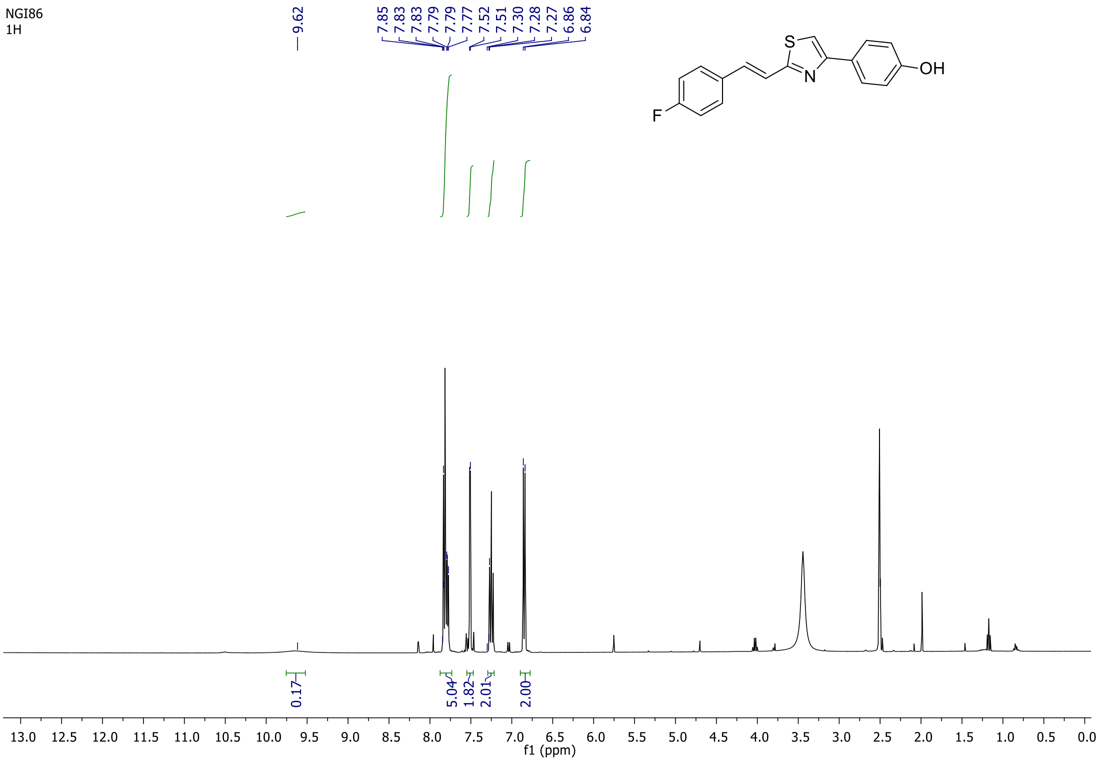


Figure 43: ^{1}H NMR of **6o** in DMSO.

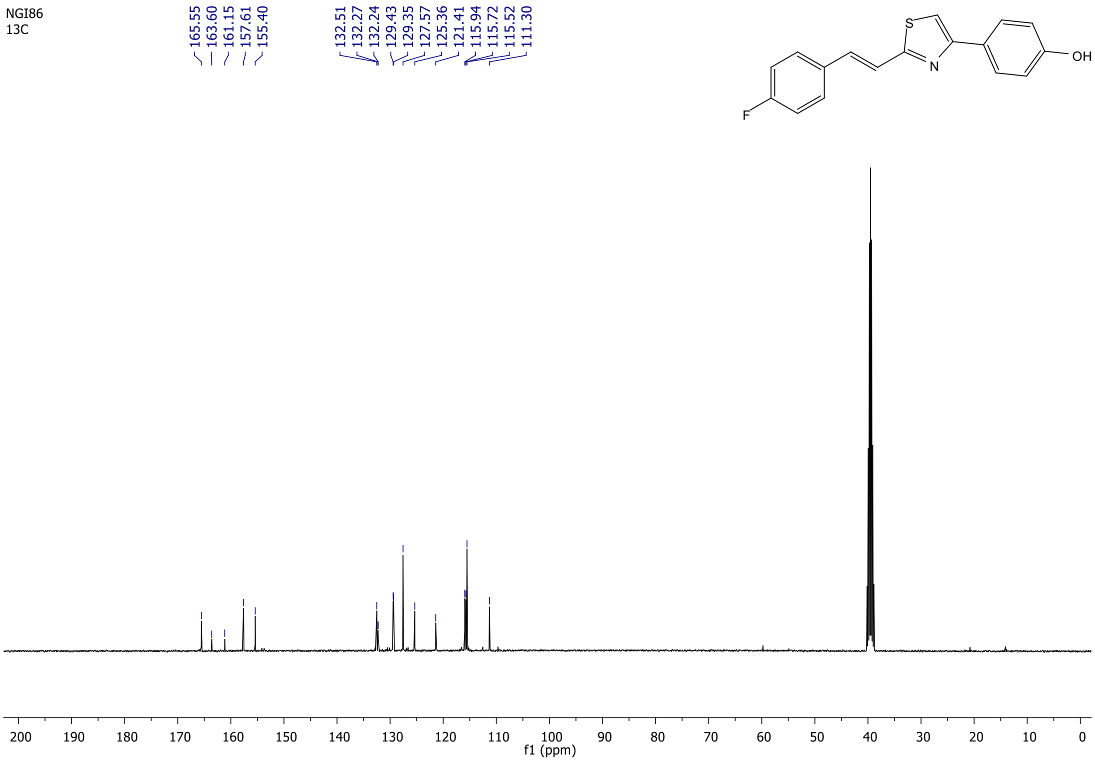


Figure 44: ^{13}C NMR of **6o** in DMSO.

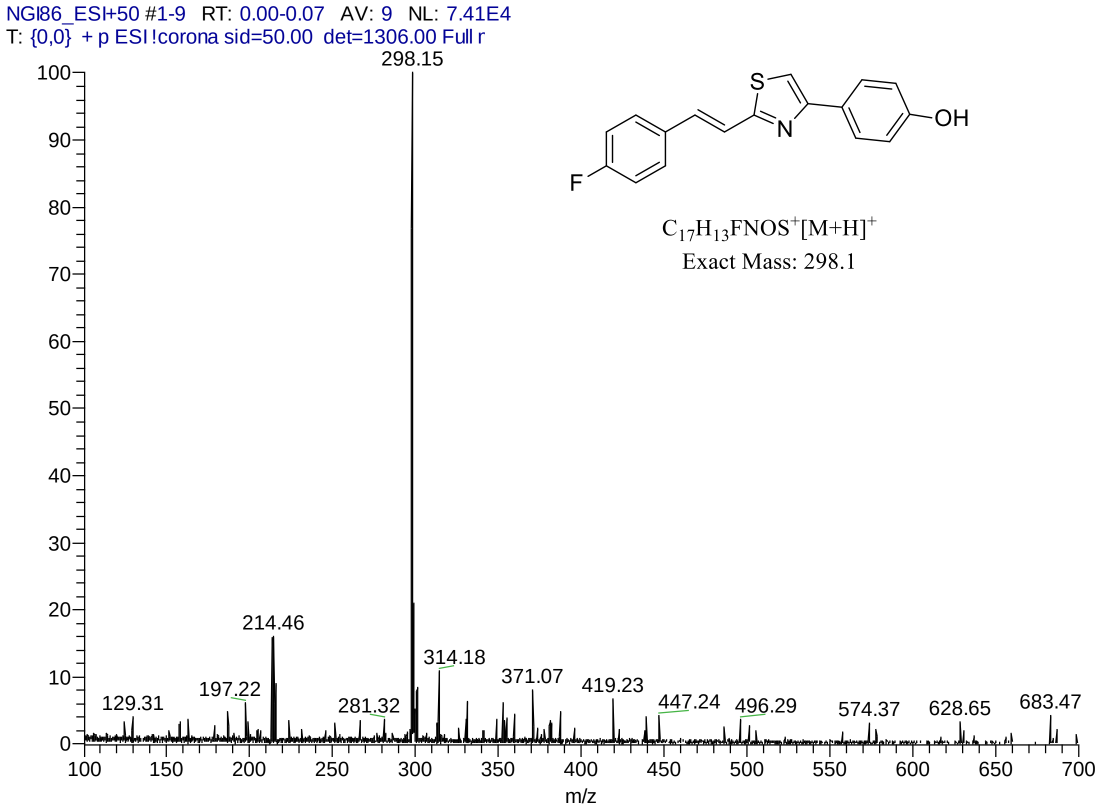


Figure 45: ESI-MS of **6o**.

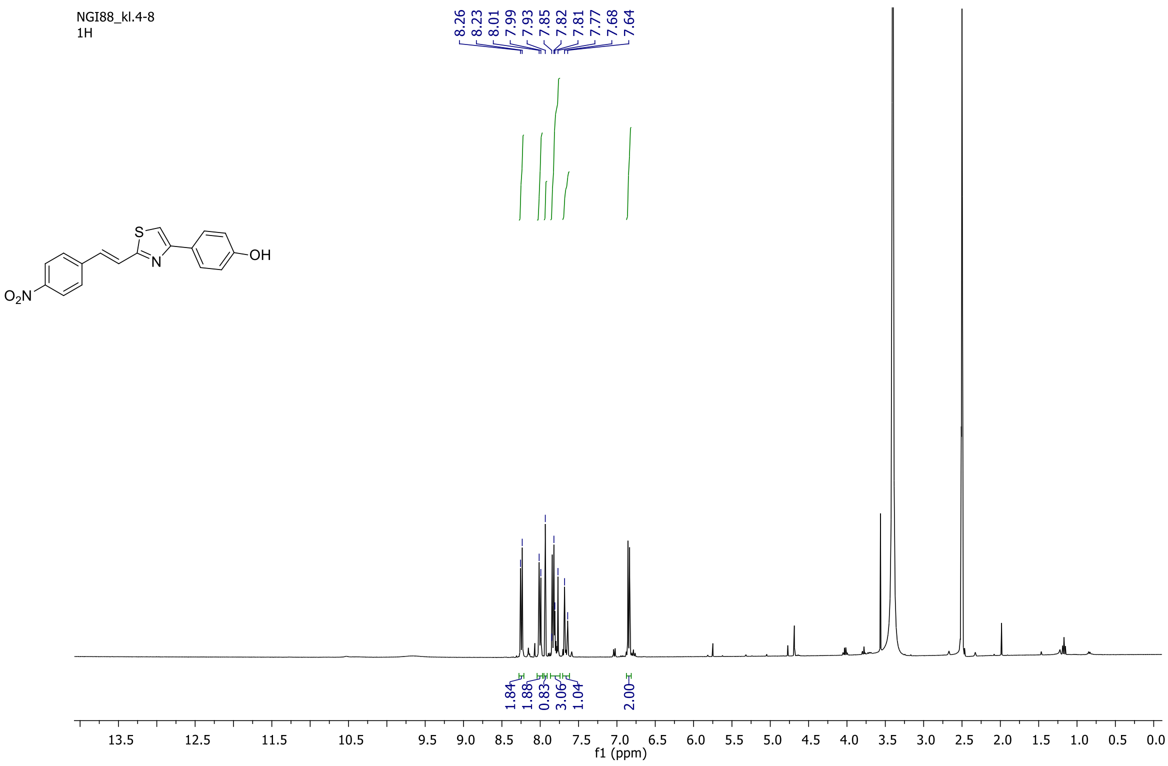


Figure 46: ^{1}H NMR of **6p** in DMSO.

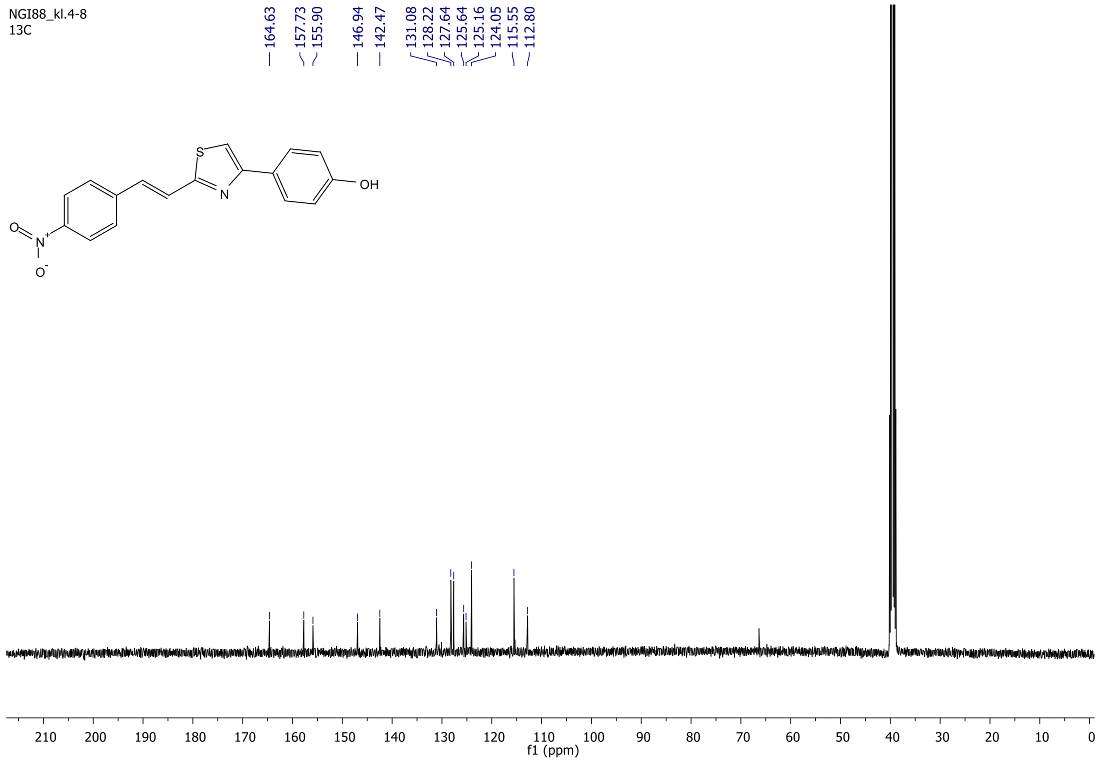


Figure 47: [13]C NMR of **6p** in DMSO.

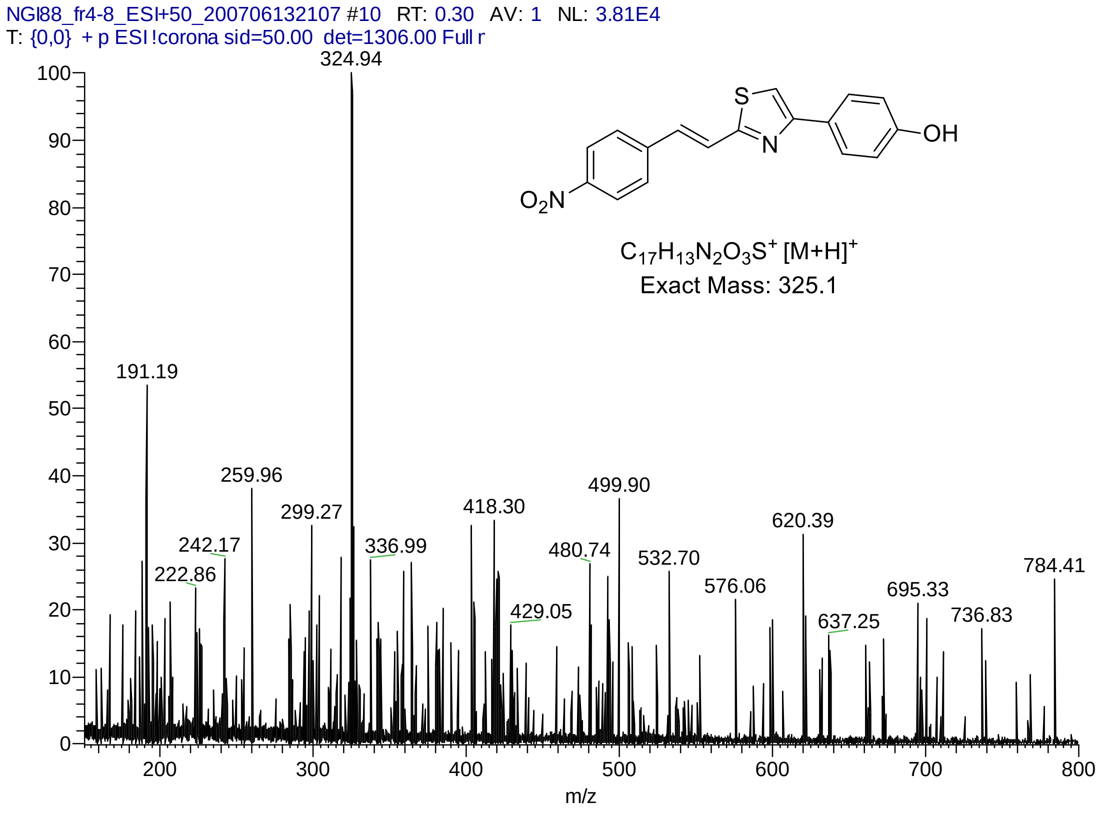
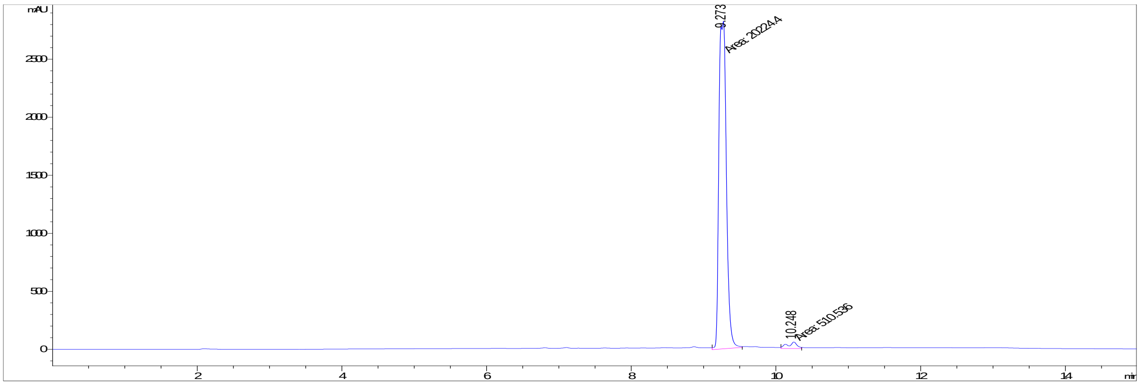
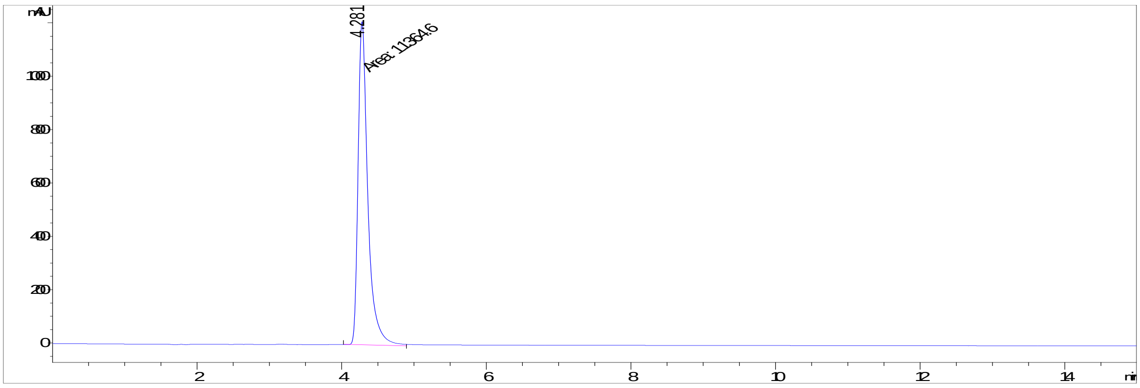


Figure 48: ESI-MS of **6p**.

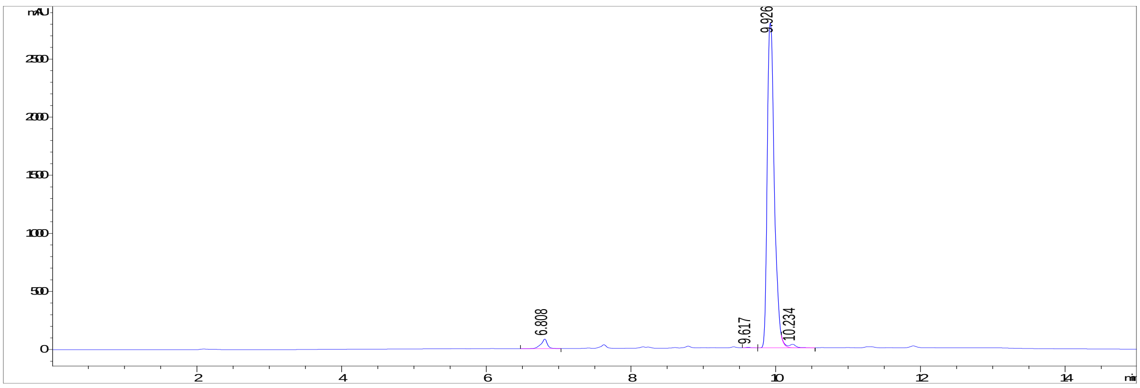
HPLC CHROMATOGRAMS OF **6a-6p**



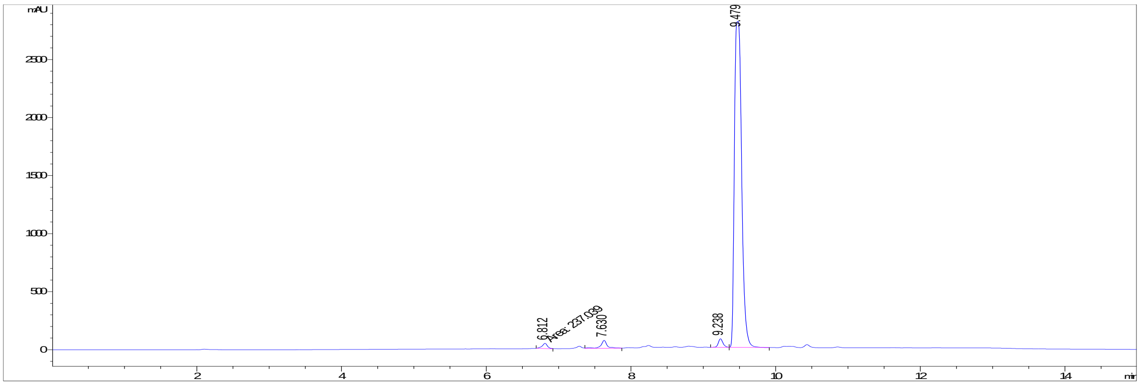
Compound	Retention time	Area%
6a	9.27	>97



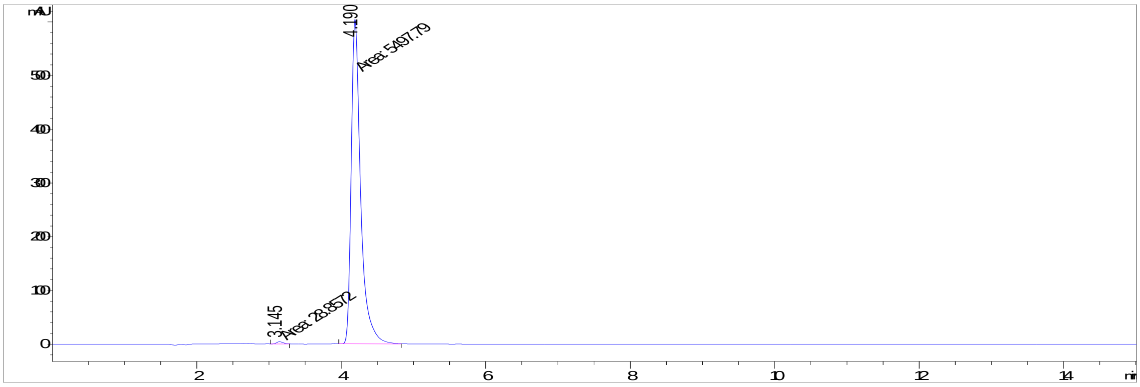
Compound	Retention time	Area%
6b	4.28	>99



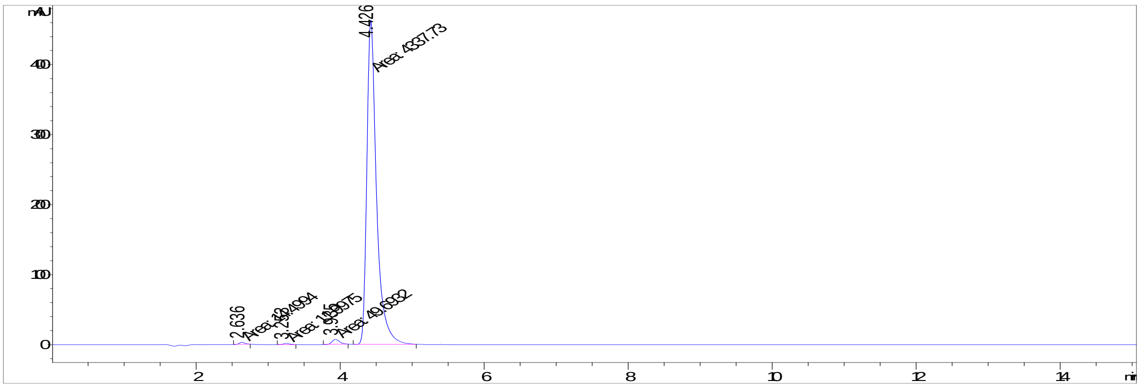
Compound	Retention time	Area%
6c	9.93	>96



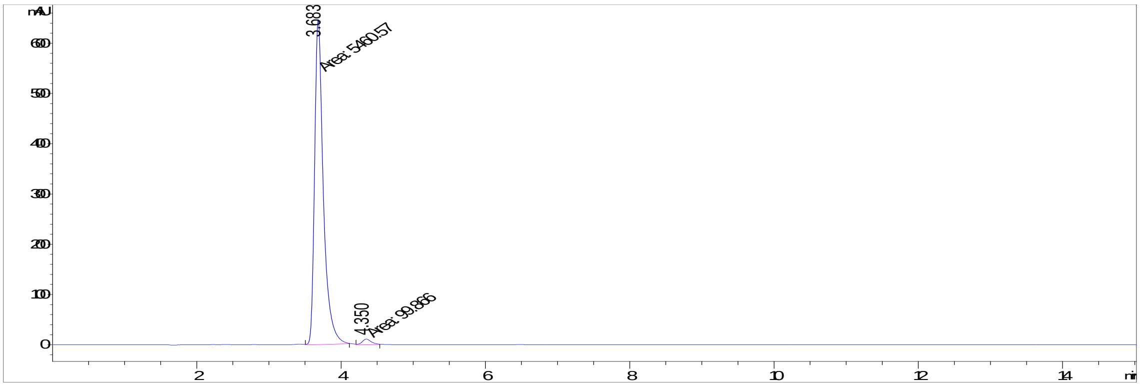
Compound	Retention time	Area%
6d	9.48	>95



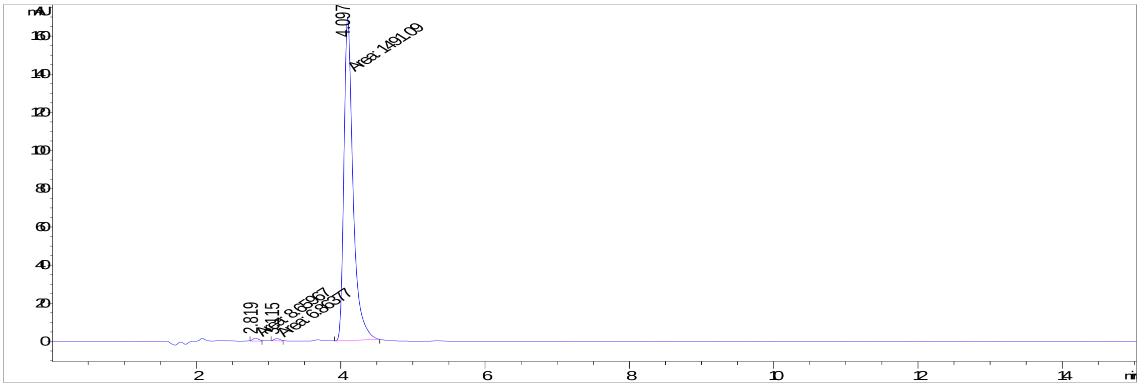
Compound	Retention time	Area%
6e	4.19	>99



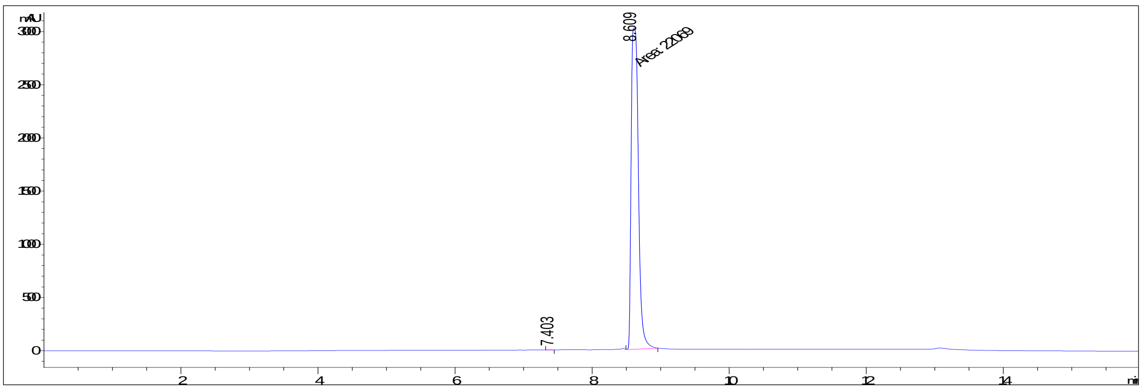
Compound	Retention time	Area%
6f	4.43	>98



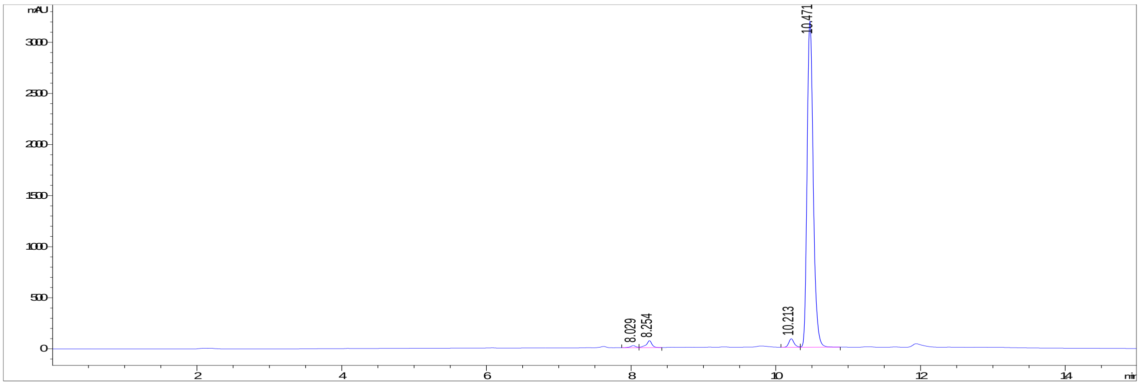
Compound	Retention time	Area%
6g	3.70	>98



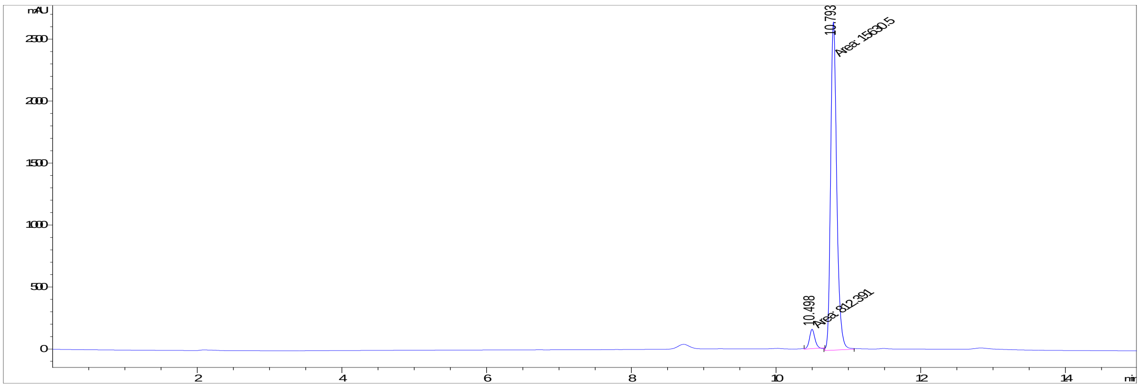
Compound	Retention time	Area%
6h	4.10	>98



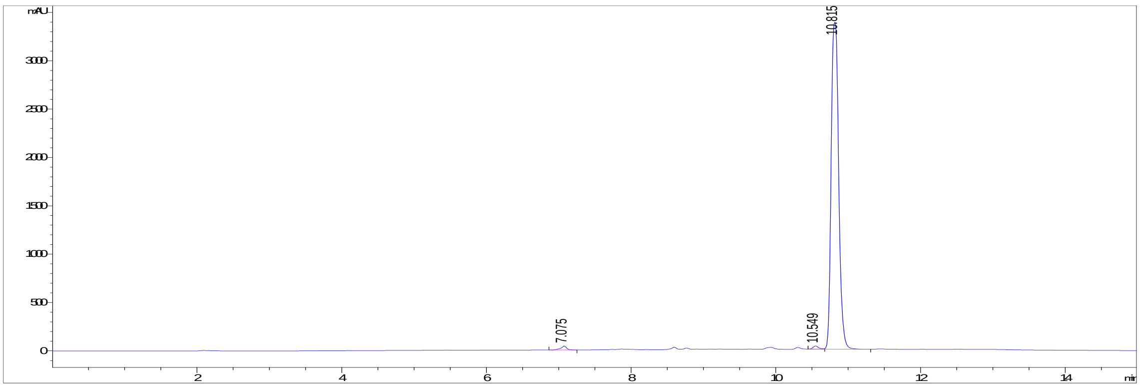
Compound	Retention time	Area%
6i	8.61	>99



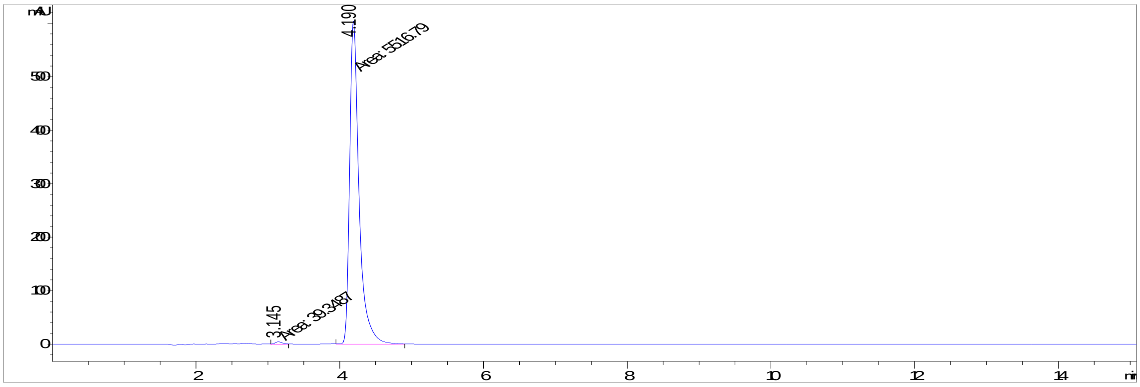
Compound	Retention time	Area%
6j	10.47	>95



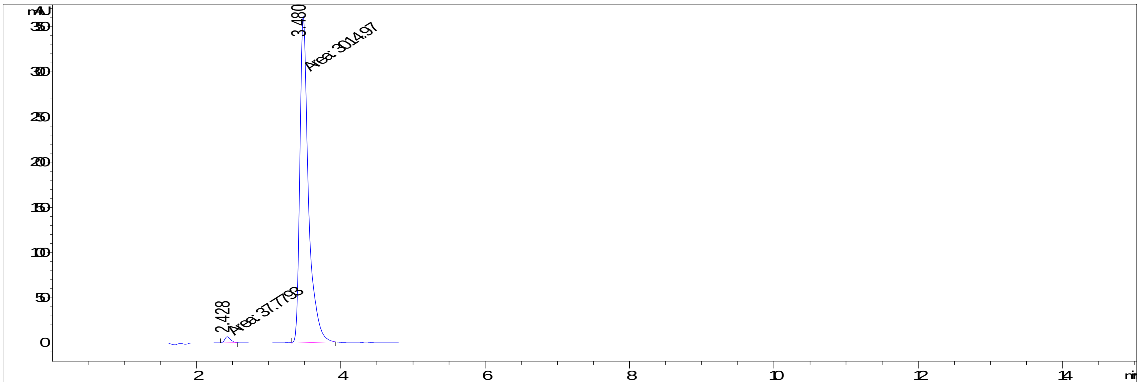
Compound	Retention time	Area%
6k	10.79	>95



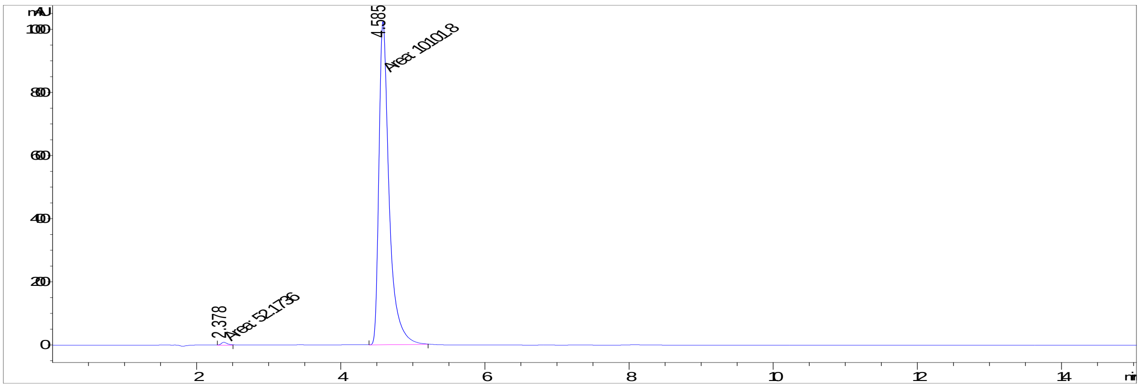
Compound	Retention time	Area%
6l	10.82	>98



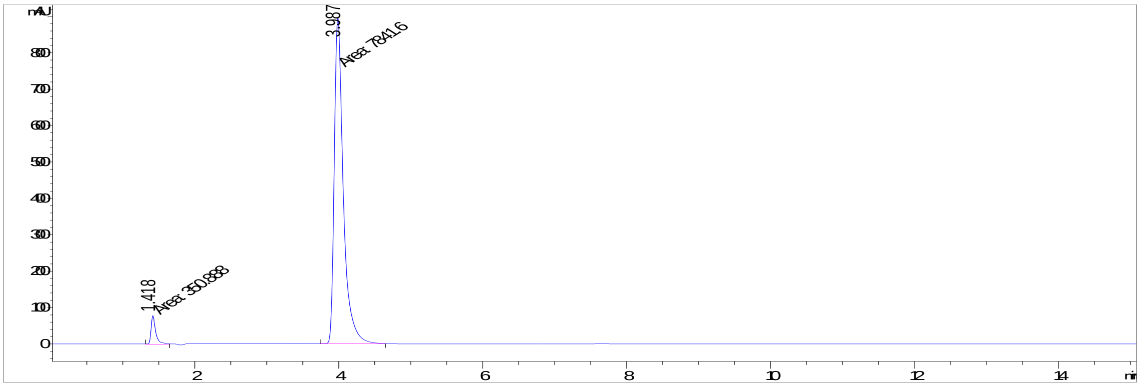
Compound	Retention time	Area%
6m	4.19	>99



Compound	Retention time	Area%
6n	3.48	>98



Compound	Retention time	Area%
6o	4.59	>99



Compound	Retention time	Area%
6p	3.99	>95