Supporting Information

Synthesis of Functionalized 1-Aminoisoquinolines through Cascade Three-Component Reaction of *ortho*-Alkynylbenzaldoximes, 2*H*-Azirines, and Electrophiles

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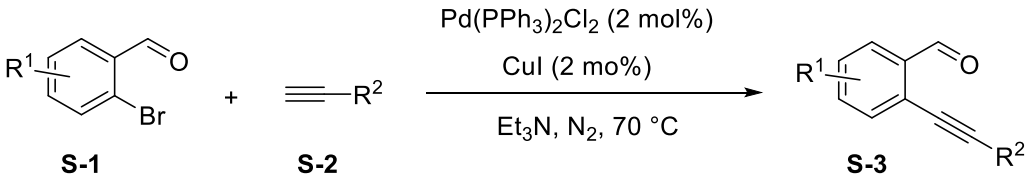
General experimental methods:

^{1}H and ^{13}C NMR spectra were recorded at 300 or 400 MHz and 75 or 100 MHz. Chemical shifts are reported in parts per million (δ/ppm) relative to tetramethylsilane as an internal standard. Resonance patterns are reported with the notations s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). Coupling constants (*J*) are reported in hertz (Hz). For the Mass spectrometry, ion source temperature was 150-250°C. High-resolution ESI-mass spectra were performed with a resolution of 10,000. Melting points were measured using a melting point instrument and were uncorrected. Column chromatography was carried out using 70-230 mesh silica gels. Commercially available reagents were used without additional purification, unless otherwise stated. Sealed tubes were dried in oven for overnight and cooled at room temperature prior to use.

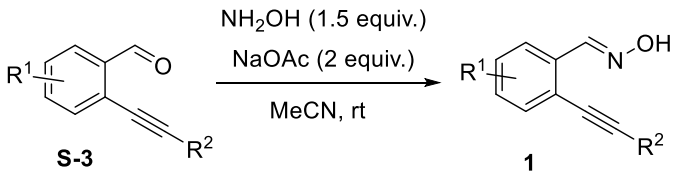
Experimental procedures

General Procedure fo Preparation of *ortho*-Alkynylbenzaldehydes



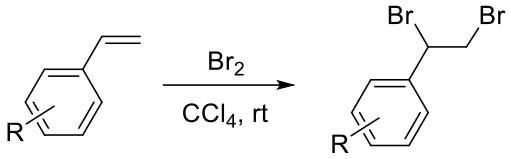
To a solution of the 2-bromobenzaldehyde (**S-1**) (10 mmol, 1 equiv.), $PdCl_2(PPh_3)_2$ (2 mol%), and CuI (2 mol%) in Et_3N (50 mL), the appropriate acetylene (**S-2**) (1.2 equiv.) was added at room temperature under N_2 atmosphere. The reaction mixture was heated at 70 °C in an oil bath for 6-18 h, and monitored by TLC. After the reaction was completed, the mixture was cooled to room temperature, and filtered by celite, washed with acetone. The filtrate was concentrated under a vacuum. The residue was purified by column chromatography on silica gel to afford the *ortho*-alkynylbenzaldoxime (**S-3**) in almost 90% yield.

General Procedure for Preparation of *ortho*-Alkynylbenzaldoximes



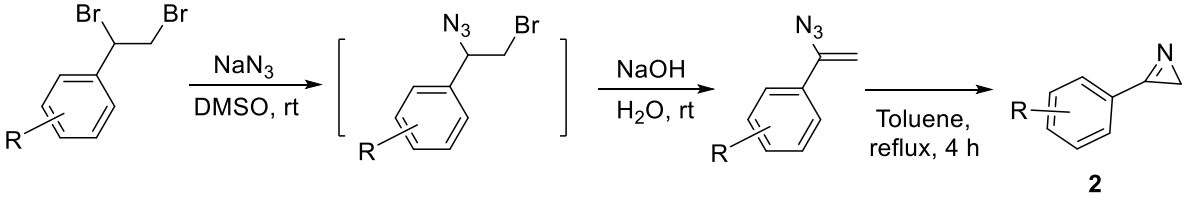
A solution of 2-alkynylbenzaldehyde (**S-3**) (2.0 mmol, 1 equiv.), hydroxylamine hydrochloride (3 mmol, 1.5 equiv.), sodium acetate (4.0 mmol, 2.0 equiv.) in ACN (10 mL) was stirred at room temperature for 12 hours. The solvent was evaporated and then quenched with water (10 mL), extracted with EtOAc (2×30 mL), dried by anhydrate Na_2SO_4. Evaporation of the solvent followed by purification on silica gel provided the corresponding 2-alkynylbenzaldoxime **1** in good yields.

General procedure for the synthesis of 1-(1,2- dibromoethyl) benzene derivatives:



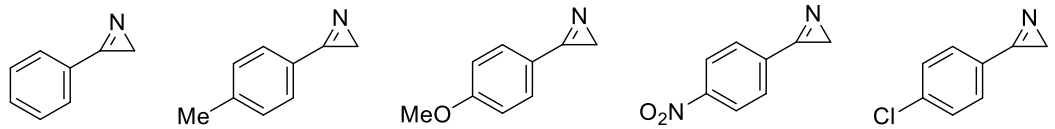
Bromine (8.0 g, 0.05 mol) in 30 mL CCl_4 was added slowly to a stirred and cooled (15-20 ^oC) solution of styrene (0.05 mol) in 40 mL of CCl_4. After the addition was completed, the mixture was stirred for 2 h in 15-20 ^oC, and then the CCl_4 was removed *in vacuo*, remaining the residue of crystalline in high yield.

General procedure for the synthesis of 2-phenyl-aza-methylcyclopropene derivatives:



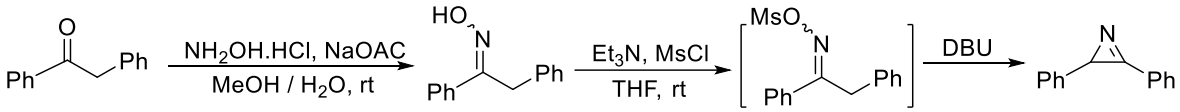
1-(1,2-dibromoethyl) benzene (46 mmol) was dissolved in 70 mL of dimethyl sulfoxide. A slow stream of N_2 was passed through the apparatus, sodium azide (4.9 g, 75 mmol) was slowly added into the solution and for 45 min afterward. The mixture became thick with precipitated azido bromide and was stirred for a further 13 h at 25 ^oC. The reaction mixture was treated with (2.0 g, 50 mmol) of sodium hydroxide in 2.0 mL of deionized water. Stirring was continued at 25 ^oC for 24 h. The mixture was poured into 200 mL of 2% sodium bicarbonate aqueous solution and extracted with CH_2Cl_2. The extracts were washed with deionized water, and the CH_2Cl_2 was removed *in vacuo*, and evaporated to yield crude 1-azidostyrene as red oil. The oil was passed through a column of silicon dioxide using petroleum ether as an eluent. The eluent was removed

in vacuo and the residual was dissolved in 100 mL of toluene. The solution refluxed for 4 h. Removal of the solvent and distillation of the crude product, afforded desired 2*H*-azirine.



2a 61% **2b** 55% **2c** 58% **2d** 45% **2e** 50%

Synthesis of 2,3-diphenyl-2H-azirine:



A mixture solvent of MeOH/H_2O (20:1) was added to a mixture of 2-Phenylacetophenone (1 eq.), $NH_2OH{\cdot}HCl$ (1.5 eq.) and sodium acetate (2 eq.) in a round bottom flask. The resulting solution was stirred at room temperature and monitored by TLC. After reaction completed, the solvent was removed in vacuo and added DCM. Then, the mixture was sequentially washed with sat. $NaHCO_3$ and brine. The Organic layer was dried over Na_2SO4. Concentration led to 1,2-diphenylethan-1-one oxime, which was used directly for the next step.

To a solution of the crude oxime (1 eq.) in dry THF was added triethylamine (1.5 eq.) and methanesulfonyl chloride (1.5 eq.) sequentially at 0 ^oC. The solution got cloudy after the addition of methanesulfonyl chloride. The resulting mixture was stirred for 1 h at rt, and DBU (1.5 eq.) was then added over 1 min. After stirred for additional 2 h, the reaction mixture was passed through a pad of silica gel, washing with Et_2O. The mixture was concentrated in vacuo and the residue was chromatographed to give the 2,3-diphenyl-2H-azirine.

Optimization of the Reaction Conditions

Optimization of the process commenced by subjecting the reaction to several solvents. It was observed that THF, compared with other solvents, offered a higher yield of the product (85%) (Table 1, entry 2). Moreover, although using other bases such as NaOAc, DABCO, and Na_2HPO_4 successfully afforded the expected product **3a**; however, these bases failed to improve the yield of the reaction in comparison to $NaHCO_3$. Consequently, for the model reaction, $NaHCO_3$ was selected as the best base. Screening of different temperatures

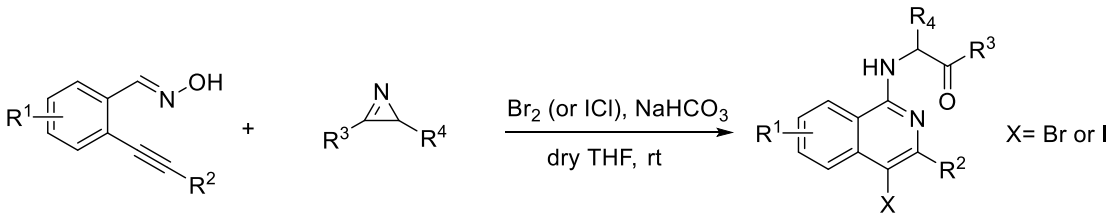
revealed that ambient temperature within 1 h proved to be the optimum temperature condition for this reaction.

Table S1: Optimization of the Reaction Conditions for the synthesis of **3a**

entry	solvent	base	yield[b] (%)
1	DCE	$NaHCO_3$	68
2	**THF**	**$NaHCO_3$**	**85**
3	DMF	$NaHCO_3$	trace
4	Toluene	$NaHCO_3$	43
5	EtOH	$NaHCO_3$	48
6	$CHCl_3$	$NaHCO_3$	53
7	CH_3CN	$NaHCO_3$	43
8[c]	THF	$NaHCO_3$	71
9[d]	THF	$NaHCO_3$	54
10[e]	THF	$NaHCO_3$	79
11	THF	NaOAc	81
12	THF	DABCO	76
13	THF	NaH_2PO_4	79

[a]Reaction conditions: 1a (0.3 mmol), 2a (0.3 mmol), Br_2 (1.1 eq.), solvent (3 mL), temp °C, 1 h. [b]isolated yields. [c]the reaction carried out in 40 °C. [d]the reaction carried out in 0 °C. [e]the reaction carried out during 12 h.

General procedure for the synthesis of final products:



1 **2** **3**

Br_2 or ICl (0.36 mmol, 1.2 eq.) was added to a mixture of *ortho*-alkynylbenzaldoxime **1** (0.30 mmol) and $NaHCO_3$ (0.45 mmol, 1.5 eq.) in THF (3.0 mL). After 30 min, 2*H*-azirine **2** (0.3 mmol, 1.0 eq.) was added, and the reaction was stirred at room temperature. After completion of the reaction as indicated by TLC, saturated aqueous NaS_2O_3 (10 mL) was added to the mixture and

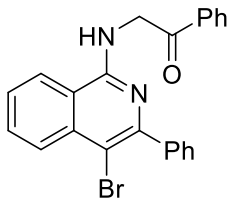
extracted by EtOAc (10 ml). The organic layer was separated and dried by anhydrous Na_2SO_4. After filtration and evaporation of solvent, the residue was purified by column chromatography using hexane/EtOAc as eluent to afford the desired product **3**.

Typical procedure for the synthesis of 3a on 5 mmol scale:

Br_2 (6.0 mmol, 1.2 eq.) was added to a mixture of *ortho*-alkynylbenzaldoxime **1** (5.0 mmol, 1.11 g) and $NaHCO_3$ (7.5 mmol, 630 mg) in THF (15 mL). After 30 min, 2*H*-azirine **2** (5.0 mmol, 586 mg) was added, and the reaction was stirred at room temperature. After completion of the reaction as indicated by TLC, saturated aqueous NaS_2O_3 (50 mL) was added to the mixture and extracted by EtOAc (50 ml). The organic layer was separated and dried by anhydrous Na_2SO_4. After filtration and evaporation of solvent, the residue was purified by column chromatography using hexane/EtOAc as eluent to afford the desired product **3a** (1.50 g, 72% yield).

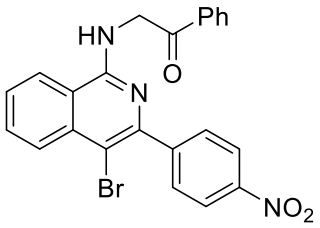
Characterization of final products:

3a: 2-((4-bromo-3-phenylisoquinolin-1-yl) amino)-1-phenylethan-1-one



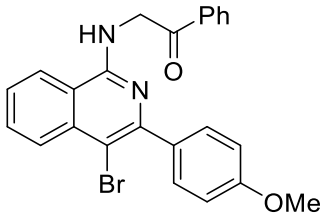
White solid, 160 mg (0.3 mmol, yield 85%), m.p.179-181 ºC, R_f : 0.23 (Ethyl acetate : n-hexane / 1:2) ; **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.27 (d, *J* = 8.6 Hz, 1H, H-Ar), 8.08 (d, *J* = 7.3 Hz, 2H, H-Ar), 8.02 (d, *J* = 8.3 Hz, 1H, H-Ar), 7.82- 7.69 (m, 3H, H-Ar), 7.67 – 7.55 (m, 2H, H-Ar), 7.55 – 7.39 (m, 5H, H-Ar), 6.66 (t, *J* = 3.9 Hz, 1H, NH), 5.09 (d, *J* = 3.9 Hz, 2H, CH_2). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 196.0, 152.9, 150.0, 141.6, 136.5, 134.8, 134.0, 130.9, 130.1, 128.8, 128.4, 128.1, 127.9, 127.6, 126.7, 121.9, 118.6, 106.3, 48.5. **HRMS** (ESI): Calc. for $C_{23}H_{18}N_2O^{79}Br$ $[M+H]^+$ 417.0597, found 417.0596.

3b: 2-((4-bromo-3-(4-nitrophenyl) isoquinolin-1-yl) amino)-1-phenylethan-1-one



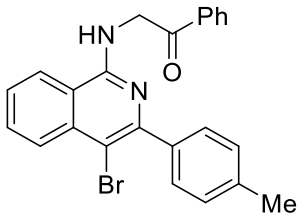
Yellow solid, 93 mg (0.3 mmol, yield 67%), m.p. 158-160 ºC, R_f : 0.63 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.31 (d, *J* = 8.9 Hz, 2H, H-Ar), 8.27 (d, *J* = 7.8 Hz, 1H, H-Ar), 8.13 – 8.00 (m, 3H, H-Ar), 7.92 (d, *J* = 8.3 Hz, 2H, H-Ar), 7.80 (dd, *J* = 8.3, 6.9 Hz, 1H, H-Ar), 7.65 (t, *J* = 7.1 Hz, 1H, H-Ar), 7.55 (d, *J* = 6.6 Hz, 1H, H-Ar), 7.52 (d, *J* = 7.4 Hz, 1H, H-Ar), 6.72 (t, *J* = 3.9 Hz, 1H, -NH), 5.05 (d, *J* = 3.9 Hz, 2H, -CH_2). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.6, 153.1, 147.9, 147.7, 147.2, 136.2, 134.7, 134.1, 131.2, 131.0, 128.9, 128.0, 127.7, 127.4, 122.9, 122.0, 119.0, 106.6, 48.3. **HRMS** (ESI): Calc. for $C_{23}H_{17}N_3O_3{}^{79}Br$ $[M+H]^+$ 462.0448, found 462.0449.

3c: 2-((4-bromo-3-(4-methoxyphenyl) isoquinolin-1-yl) amino)-1-phenylethan-1-one



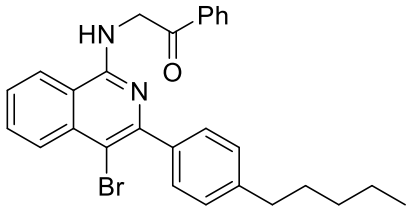
White solid, 95 mg (0.3 mmol, yield 71%), m.p. 194-196 ºC, R_f : 0.13 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.26 (dd, *J* = 8.4, 1.2 Hz, 1H, H-Ar), 8.09 (d, *J* = 8.7 Hz, 2H, H-Ar), 8.01 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.81 – 7.68 (m, 3H, H-Ar), 7.69 – 7.41 (m, 4H, H-Ar), 7.01 (d, *J* = 8.7 Hz, 2H, H-Ar), 6.63 (t, *J* = 4.1 Hz, 1H, NH), 5.09 (d, *J* = 4.1 Hz, 2H, CH_2), 3.90 (s, 3H, OCH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.9, 159.4, 152.8, 149.5, 136.6, 134.9, 134.0, 131.6, 131.3, 131.0, 128.8, 128.1, 127.5, 126.5, 121.9, 118.5, 113.1, 106.1, 55.3, 48.5. **HRMS** (ESI): Calc. for $C_{24}H_{20}N_2O_2{}^{79}Br$ $[M+Na]^+$ 469.0522, found 469.0527.

3d: 2-((4-bromo-3-(p-tolyl) isoquinolin-1-yl) amino)-1-phenylethan-1-one



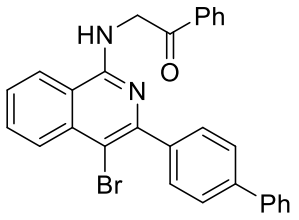
White solid, 89 mg (0.3 mmol, yield 69%), m.p. 123-125 ºC, R_f : 0.03 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.26 (dd, *J* = 8.2, 0.9 Hz, 1H, H-Ar), 8.08 (dd, *J* = 8.4, 1.4 Hz, 2H, H-Ar), 8.02 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.78 – 7.45 (m, 7H, H-Ar), 7.30 (d, *J* = 7.8 Hz, 2H, H-Ar), 6.65 (t, *J* = 4.1 Hz, 1H, -NH), 5.08 (d, *J* = 4.1 Hz, 2H, -CH_2), 2.47 (s, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.9, 152.8, 150.0, 138.8, 137.8, 136.5, 134.8, 133.9, 130.0, 129.9, 128.8, 128.4, 128.1, 127.5, 126.6, 121.9, 118.5, 106.2, 48.5, 21.4. **HRMS** (ESI): Calc. for $C_{24}H_{20}N_2O^{79}Br$ $[M+H]^+$ 437.0754, found 437.0759.

3e: 2-((4-bromo-3-(4-pentylphenyl) isoquinolin-1-yl) amino)-1-phenylethan-1-one



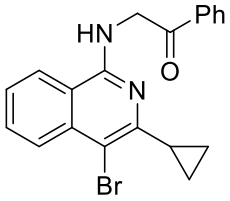
White solid, 115 mg (0.3 mmol, yield 79%), m.p. 115-118 ºC, R_f : 0.15 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.25 (d, *J* = 8.4 Hz, 1H, H-Ar), 8.07 (d, *J* = 7.2 Hz, 2H, H-Ar), 7.98 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.79- 7.69 (m, 3H, H-Ar), 7.63 (dd, *J* = 8.6, 6.4 Hz, 1H, H-Ar), 7.59 – 7.44 (m, 2H, H-Ar), 7.31 (d, *J* = 7.8 Hz, 2H, H-Ar), 6.69 (t, *J* = 4.0 Hz, 1H, -NH), 5.07 (d, *J* = 4.0 Hz, 2H, -CH_2), 2.73 (t, *J* = 7.8 Hz, 2H, CH_2-Aliphatic), 1.81 – 1.65 (m, 2H, H-Aliphatic), 1.58 – 1.28 (m, 4H, H-Aliphatic), 0.98 (t, *J* = 6.4 Hz, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 196.1, 152.8, 149.9, 142.8, 138.9, 136.5, 134.9, 133.9, 130.1, 129.9, 128.8, 128.1, 127.7, 127.5, 126.5, 121.9, 118.5, 106.2, 48.5, 35.9, 31.7, 31.1, 22.6, 14.1. **HRMS** (ESI): Calc. for $C_{28}H_{28}N_2O^{79}Br$ $[M+H]^+$ 487.1380, found 487.1377.

3f: 2-((3-([1,1'-biphenyl]-4-yl)-4-bromoisoquinolin-1-yl) amino)-1-phenylethan-1-one



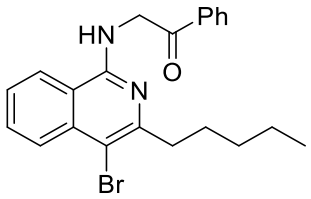
White solid, 87 mg (0.3 mmol, yield 59%), m.p. 216-218 ºC, R_f : 0.28 (Ethyl acetate : n-hexane / 1:2) ; **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.28 (d, *J* = 8.3 Hz, 1H, H-Ar), 8.10 (d, *J* = 7.2 Hz, 2H, H-Ar), 8.02 (d, *J* = 8.2 Hz, 1H, H-Ar), 7.89 (d, *J* = 8.3 Hz, 2H, H-Ar), 7.80 – 7.66 (m, 6H, H-Ar), 7.67 – 7.55 (m, 1H, H-Ar), 7.59 – 7.42 (m, 4H, H-Ar), 7.43 – 7.35 (m, 1H, H-Ar), 6.68 (t, *J* = 4.1 Hz, 1H, NH), 5.10 (d, *J* = 4.1 Hz, 2H, CH_2). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.9, 152.9, 149.5, 140.9, 140.7, 140.5, 136.5, 134.9, 134.0, 133.9, 130.6, 130.4, 128.8, 128.1, 128.0, 127.6, 127.3, 127.1, 126.7, 126.5, 126.3, 122.0, 118.7, 106.3, 48.5. **HRMS** (ESI): Calc. for $C_{29}H_{22}N_2O^{79}Br$ $[M+H]^+$ 493.0909, found 493.0900.

3g: 2-((4-bromo-3-cyclopropylisoquinolin-1-yl) amino)-1-phenylethan-1-one



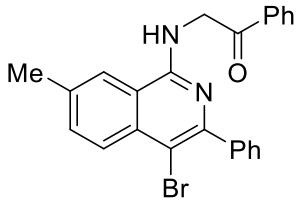
White solid, 140 mg (0.3 mmol, yield 97%), m.p. 200-203 ºC, R_f : 0.73 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.19 – 7.97 (m, 3H, H-Ar), 7.85 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.71 – 7.58 (m, 2H, H-Ar), 7.54 (dd, *J* = 8.4, 6.8 Hz, 2H, H-Ar), 7.42 (dd, *J* = 8.2, 6.9, 1H, H-Ar), 6.42 (t, *J* = 4.4 Hz, 1H, NH), 4.93 (d, *J* = 4.4 Hz, 2H, CH_2), 2.72 – 2.61 (m, 1H, CH), 1.17 – 1.09 (m, 2H, CH- Aliphatic), 1.01 – 0.79 (m, 2H, CH- Aliphatic). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.8, 152.8, 151.3, 136.1, 134.9, 133.8, 130.6, 128.9, 127.9, 126.4, 125.4, 121.8, 118.1, 106.7, 48.1, 16.1, 8.9. **HRMS** (ESI): Calc. for $C_{24}H_{20}N_2O^{79}Br$ $[M+H]^+$ 437.0754, found 437.0759.

3h: 2-((4-bromo-3-pentylisoquinolin-1-yl) amino)-1-phenylethan-1-one



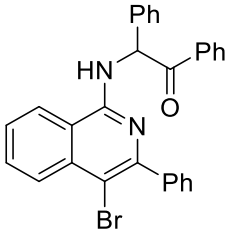
Orange oil, 113 mg (0.3 mmol, yield 92%), R_f : 0.45 (Ethyl acetate : n-hexane / 1:2) ; **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 88.25 – 8.06 (m, 3H, H-Ar), 7.93 (d, *J* = 8.3 Hz, 1H, H-Ar), 7.69 – 7.59 (m, 2H, H-Ar), 7.58 – 7.43 (m, 3H, H-Ar), 6.49 (t, *J* = 4.1 Hz, 1H, NH), 5.06 (d, *J* = 4.1 Hz, 2H, CH_2), 2.98 (t, *J* = 7.8 Hz, 2H, CH_2), 1.92 – 1.66 (m, 3H, H-Aliphatic), 1.52 – 1.30 (m, 5H, H-Aliphatic), 0.92 (t, *J* = 6.9 Hz, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 196.1, 154.5, 152.7, 152.3, 150.9, 150.9, 136.1, 135.0, 130.5, 128.8, 128.0, 125.7, 118.1, 107.0, 48.4, 37.8, 31.7, 28.3, 22.6, 14.0. **HRMS** (ESI): Calc. for $C_{22}H_{24}N_2O^{79}Br$ $[M+H]^+$ 411.1067, found 411.1068.

3i: 2-((4-bromo-6-methyl-3-phenylisoquinolin-1-yl) amino)-1-phenylethan-1-one



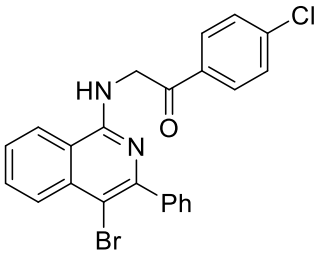
White solid, 116 mg (0.3 mmol, yield 90%), m.p. 143-145 ºC, R_f : 0.38 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.21 – 7.97 (m, 3H, H-Ar), 7.89 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.83 – 7.68 (m, 2H, H-Ar), 7.66 – 7.57 (m, 1H, H-Ar), 7.55 – 7.43 (m, 5H, H-Ar), 7.38 (dd, *J* = 8.4, 1.7 Hz, 1H, H-Ar), 6.62 (t, *J* = 4.2 Hz, 1H, NH), 5.06 (d, *J* = 4.2 Hz, 2H, CH_2), 2.58 (s, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 196.1, 152.8, 150.0, 141.7, 141.4, 136.6, 134.9, 130.2, 129.9, 128.6, 128.3, 127.6, 126.9, 126.7, 122.0, 121.7, 116.8, 106.0, 48.5, 22.1. **HRMS** (ESI): Calc. for $C_{24}H_{20}N_2O^{79}Br$ $[M+H]^+$ 431.0754, found 431.0756.

3j: 2-((4-bromo-3-phenylisoquinolin-1-yl) amino)-1,2-diphenylethan-1-one



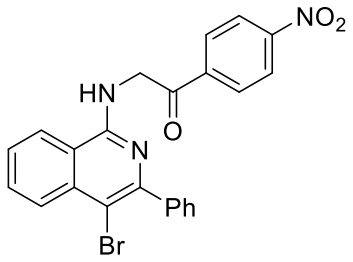
Pale yellow, 81 mg (0.3 mmol, yield 55%), recrystallization solvent: *n*-hexane/dichloromethane, m.p. 178-180 ºC, R_f : 0.7 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (400 MHz, $CDCl_3$)** δ 8.13 (d, *J* = 8.1 Hz, 1H, H-Ar), 8.00 – 7.92 (m, 2H, H-Ar), 7.90 (d, *J* = 8.3 Hz, 1H, H-Ar), 7.63 (t, *J* = 7.7 Hz, 1H), 7.54 – 7.36 (m, 6H, H-Ar), 7.32 – 7.10 (m, 8H, H-Ar), 6.84 (brs, 1H, NH), 6.79 (brs, 1H, CH- Aliphatic). **^{13}C {^{1}H} NMR (101 MHz, $CDCl_3$)** δ 197.7, 152.0, 137.5, 136.6, 135.0, 133.5, 131.1, 130.2, 129.1, 129.0, 128.9, 128.6, 128.3, 127.8, 127.6, 127.4, 126.7, 122.0, 118.6, 106.4, 60.5. **HRMS** (ESI): Calc. for $C_{29}H_{22}N_2O^{79}Br$ $[M+H]^+$ 493.0909, found 493.0909.

3k: 2-((4-bromo-3-phenylisoquinolin-1-yl) amino)-1-(4-chlorophenyl) ethan-1-one



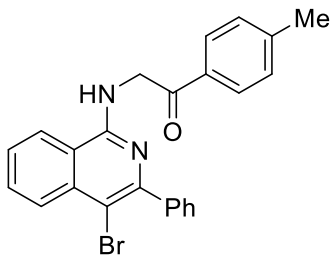
White solid, 115 mg (0.3 mmol, yield 85%), m.p. 155-157 ºC, R_f : 0.43 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.26 (d, *J* = 8.4 Hz, 1H, H-Ar), 8.04 – 7.92 (m, 3H, H-Ar), 7.80 - 7.66 (m, 3H, H-Ar), 7.59 (t, *J* = 7.6 Hz, 1H, H-Ar), 7.52- 7.34 (m, 5H, H-Ar), 6.58 (t, *J* = 4.0 Hz, 1H, NH), 5.03 (d, *J* = 4.0 Hz, 2H, CH_2). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 194.9, 152.7, 149.9, 141.5, 140.4, 136.4, 133.1, 130.1, 129.6, 129.2, 129.1, 128.0, 128.0, 127.7, 127.6, 118.5, 106.5, 48.4. **HRMS** (ESI): Calc. for $C_{23}H_{17}ClN_2O^{79}Br$ $[M+H]^+$ 451.0208, found 451.0201.

3l: 2-((4-bromo-3-phenylisoquinolin-1-yl) amino)-1-(4-nitrophenyl) ethan-1-one



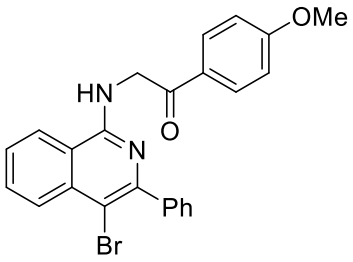
Yellow solid, 124 mg (0.3 mmol, yield 90%), m.p. 148-150, R_f : 0.3 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.32 (d, *J* = 8.9 Hz, 2H, H-Ar), 8.28 (d, *J* = 7.8 Hz, 1H, H-Ar), 8.13 – 8.01 (m, 3H, H-Ar), 7.98 – 7.88 (m, 2H, H-Ar), 7.81 (dd, *J* = 8.3, 6.9, 1H, H-Ar), 7.66 (dd, *J* = 7.0, 1.4 Hz, 2H, H-Ar), 7.54 (dd, *J* = 8.3, 7.0 Hz, 2H, H-Ar), 6.73 (t, *J* = 3.9 Hz, 1H, NH), 5.06 (d, *J* = 3.9 Hz, 2H, CH_2). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.9, 159.4, 152.8, 149.5, 136.6, 134.9, 134.0, 133.9, 131.3, 130.8, 130.6, 128.8, 128.3, 128.1, 127.5, 126.5, 118.5, 106.1, 48.5. **HRMS** (ESI): Calc. for $C_{23}H_{17}N_3O_3{}^{79}Br$ $[M+H]^+$ 462.0448, found 462.0445.

3m: 2-((4-bromo-3-phenylisoquinolin-1-yl) amino)-1-(p-tolyl) ethan-1-one



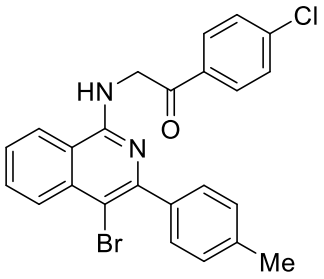
White solid, 101 mg (0.3 mmol, yield 78%), m.p. 143-145 ºC R_f : 0.38 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.26 (dd, *J* = 8.4, 1.1 Hz, 1H, H-Ar), 8.01 (d, *J* = 7.5 Hz, 1H, H-Ar), 7.97 (d, *J* = 8.2 Hz, 2H, H-Ar), 7.83 – 7.65 (m, 3H, H-Ar), 7.61 – 7.38 (m, 4H, H-Ar), 7.30 (d, *J* = 7.5 Hz, 2H, H-Ar), 6.70 (t, *J* = 4.1 Hz, 1H, NH), 5.04 (d, *J* = 4.1 Hz, 2H, CH_2), 2.45 (s, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.4, 152.9, 150.0, 144.9, 141.7, 136.4, 132.3, 130.2, 129.9, 129.5, 128.3, 128.0, 127.6, 126.6, 122.0, 121.8, 118.6, 106.2, 48.3, 21.8. **HRMS** (ESI): Calc. for $C_{24}H_{20}N_2O^{79}Br$ $[M+H]^+$ 431.0754, found 431.0759.

3n: 2-((4-bromo-3-phenylisoquinolin-1-yl) amino)-1-(4-methoxyphenyl) ethan-1-one



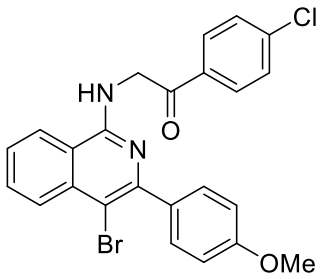
White solid, 94 mg (0.3 mmol, yield 70%), m.p. 156-158 ºC, R_f : 0.35 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.27 (dd, *J* = 8.4, 1.2 Hz, 1H, H-Ar), 8.10 (dd, *J* = 7.1, 1.4 Hz, 2H, H-Ar), 8.02 (d, *J* = 8.2 Hz, 1H, H-Ar), 7.81 – 7.70 (m, 3H, H-Ar), 7.68 – 7.48 (m, 4H, H-Ar), 7.02 (d, *J* = 8.7 Hz, 1H, H-Ar), 6.64 (t, *J* = 4.1 Hz, 1H, NH), 5.10 (d, *J* = 4.1 Hz, 2H, CH_2), 3.91 (s, 3H, OCH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.0, 159.4, 152.6, 149.4, 140.3, 136.5, 133.9, 133.2, 131.6, 131.3, 129.5, 129.2, 129.1, 127.7, 121.9, 121.6, 118.4, 113.1, 112.9, 106.2, 55.3, 48.3. **HRMS** (ESI): Calc. for $C_{24}H_{20}N_2O_2{}^{79}Br$ $[M+H]^+$ 447.0703, found 447.0705.

3o: 2-((4-bromo-3-(p-tolyl) isoquinolin-1-yl) amino)-1-(4-chlorophenyl) ethan-1-one



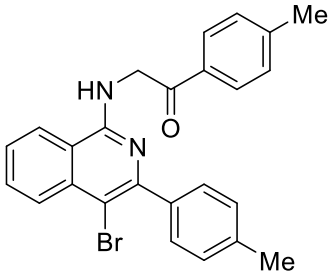
White solid, 104 mg (0.3 mmol, yield 75%), m.p. 170-173 ºC, R_f : 0.43 (Ethyl acetate : n-hexane / 1:2) ; **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.07 – 7.95 (m, 3H, H-Ar), 7.89 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.73 (dd, *J* = 7.9, 1.7 Hz, 2H, H-Ar), 7.53 – 7.35 (m, 6H, H-Ar), 6.51 (t, *J* = 4.2 Hz, 1H, NH), 5.02 (d, *J* = 4.2 Hz, 2H, CH_2), 2.59 (s, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 194.9, 152.7, 150.0, 141.7, 141.6, 140.4, 136.6, 133.2, 130.1, 129.8, 129.6, 129.2, 128.6, 128.0, 127.6, 127.0, 126.8, 122.0, 116.7, 106.2, 48.4, 22.1. **HRMS** (ESI): Calc. for $C_{24}H_{19}ClN_2O^{79}Br$ $[M+H]^+$ 465.0364, found 465.363.

3p: 2-((4-bromo-3-(4-methoxyphenyl) isoquinolin-1-yl) amino)-1-(4-chlorophenyl) ethan-1-one



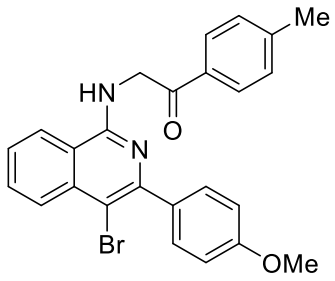
White solid, 133 mg (0.3 mmol, yield 92%), m.p. 140-142 ºC, R_f : 0.38 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.21 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.96 (d, *J* = 8.5 Hz, 2H, H-Ar), 7.92 (d, *J* = 8.3 Hz, 1H, H-Ar), 7.80 – 7.61 (m, 3H, H-Ar), 7.52 (d, *J* = 8.0 Hz, 1H, H-Ar), 7.54 (d, *J* = 8.6 Hz, 2H, H-Ar), 6.99 (d, *J* = 8.7 Hz, 2H, H-Ar), 6.57 (t, *J* = 4.1 Hz, 1H, NH), 4.98 (d, *J* = 4.1 Hz, 2H, CH_2), 3.89 (s, 3H, OCH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.0, 159.4, 152.6, 149.4, 140.3, 136.5, 133.9, 133.2, 131.6, 131.2, 129.5, 129.1, 127.5, 126.5, 121.9, 121.6, 118.3, 113.1, 112.9, 106.2, 55.3, 48.3. **HRMS** (ESI): Calc. for $C_{24}H_{19}ClN_2O_2{}^{79}Br$ $[M+H]^+$ 481.0313, found 481.0313.

3q: 2-((4-bromo-3-(p-tolyl) isoquinolin-1-yl) amino)-1-(p-tolyl) ethan-1-one



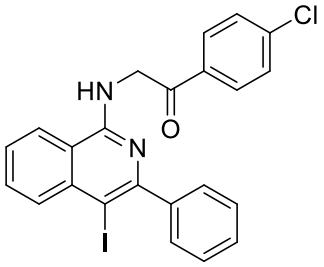
White solid, 120 mg (0.3 mmol, yield 90%), m.p. 208-210 ºC, R_f : 0.5 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.05 – 7.93 (m, 1H, H-Ar), 7.97 (d, *J* = 8.3 Hz, 2H, H-Ar), 7.90 (d, *J* = 8.5 Hz, 1H, H-Ar), 7.78 (dd, *J* = 8.0, 1.7 Hz, 2H, H-Ar), 7.53 – 7.42 (m, 3H, H-Ar), 7.39 (dd, *J* = 8.5, 1.7 Hz, 1H, H-Ar), 7.30 (d, *J* = 8.0 Hz, 2H, H-Ar), 6.63 (t, *J* = 4.1 Hz, 1H, NH), 5.03 (d, *J* = 4.1 Hz, 2H, CH_2), 2.59 (s, 3H, CH_3), 2.44 (s, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.5, 152.8, 150.0, 144.9, 141.8, 141.4, 136.5, 132.3, 130.2, 129.9, 129.5, 128.5, 128.3, 127.6, 126.6, 126.5, 122.1, 121.8, 116.8, 105.9, 48.3, 22.0, 21.8. **HRMS** (ESI): Calc. for $C_{25}H_{22}N_2O^{79}Br$ $[M+H]^+$ 445.0910, found 445.0905.

3r: 2-((4-bromo-3-(4-methoxyphenyl) isoquinolin-1-yl) amino)-1-(p-tolyl) ethan-1-one



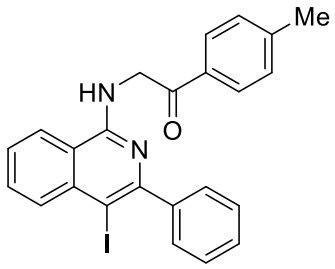
White solid, 135 mg (0.3 mmol, yield 98%), m.p. 126-128 ºC, R_f : 0.38 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.25 (dd, *J* = 8.6, 1.1 Hz, 1H, H-Ar), 8.03 – 7.88 (m, 3H, H-Ar), 7.80 – 7.66 (m, 3H, H-Ar), 7.58 (dd, *J* = 8.3, 6.9, 1H, H-Ar), 7.37 – 7.23 (m, 2H, H-Ar), 7.02 (d, *J* = 8.8 Hz, 1H, H-Ar), 6.64 (t, *J* = 4.0 Hz, 1H, NH), 5.06 (d, *J* = 4.0 Hz, 2H, CH_2), 3.90 (s, 3H, OCH_3), 2.44 (s, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.4, 159.4, 152.8, 149.5, 144.9, 136.6, 134.0, 132.3, 131.6, 131.2, 129.5, 129.2, 128.3, 128.0, 118.5, 113.1, 112.9, 106.0, 48.3, 21.8. **HRMS** (ESI): Calc. for $C_{25}H_{22}N_2O_2{}^{79}Br$ $[M+H]^+$ 461.0859, found 461.0858.

4a: 1-(4-chlorophenyl)-2-((4-iodo-3-phenylisoquinolin-1-yl) amino) ethan-1-one



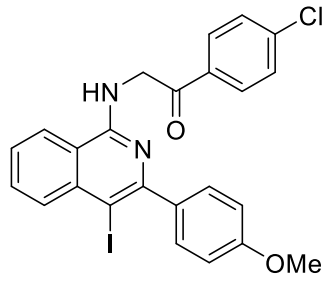
White solid, 105 mg (0.3 mmol, yield 70%), m.p. 152-154 ºC, R_f : 0.48 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 9.18 (s, 1H, H-Ar), 8.22 (d, *J* = 8.5 Hz, 1H, H-Ar), 8.14 (d, *J* = 8.3 Hz, 1H, H-Ar), 7.98- 7.88 (m, 3H, H-Ar), 7.85- 7.76 (m, 2H, H-Ar), 7.72- 7.59 (m, 5H, H-Ar), 7.55 – 7.33 (m, 5H, H-Ar), 6.66 (t, *J* = 4.3 Hz, 1H, NH), 4.95 (d, *J* = 4.3 Hz, 2H, CH_2). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.0, 157.0, 154.8, 153.6, 152.0, 144.5, 143.7, 140.3, 138.5, 133.1, 132.8, 132.3, 130.0, 129.8, 129.1, 128.0, 127.6, 121.8, 118.2, 83.8, 48.4. **HRMS** (ESI): Calc. for $C_{23}H_{17}ClIN_2O$ $[M+H]^+$ 499.0069, found 499.0060.

4b: 2-((4-iodo-3-phenylisoquinolin-1-yl) amino)-1-(p-tolyl) ethan-1-one



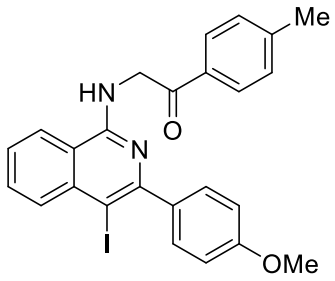
White solid, 108 mg (0.3 mmol, yield 75%), m.p. 160-162 ºC, R_f : 0.62 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, CDCl$_3$)** δ 8.17 (dd, *J* = 8.5, 1.2 Hz, 1H, H-Ar), 7.96 (d, *J* = 8.4 Hz, 1H, H-Ar), 7.95 (d, *J* = 8.2 Hz, 2H, H-Ar), 7.75 – 7.71 (m, 1H, H-Ar), 7.70 – 7.64 (m, 2H, H-Ar), 7.60 – 7.55 (m, 1H, H-Ar), 7.54 – 7.43 (m, 3H, H-Ar), 7.28 (d, *J* = 8.0 Hz, 2H, H-Ar), 6.73 (t, *J* = 4.0 Hz, 1H, NH), 5.02 (d, *J* = 4.0 Hz, 2H, CH_2), 2.44 (s, 3H, CH_3). **^{13}C {^{1}H} NMR (75 MHz, CDCl$_3$)** δ 195.4, 155.0, 153.7, 144.9, 144.6, 138.5, 132.8, 132.3, 131.3, 130.0, 129.5, 128.2, 127.9, 127.6, 126.8, 121.9, 118.4, 83.4, 48.4, 21.8. **HRMS** (ESI): Calc. for $C_{24}H_{20}IN_2O$ $[M+H]^+$ 479.0615, found 479.0616.

4c: 1-(4-chlorophenyl)-2-((4-iodo-3-(4-methoxyphenyl) isoquinolin-1-yl)amino)ethan-1-one



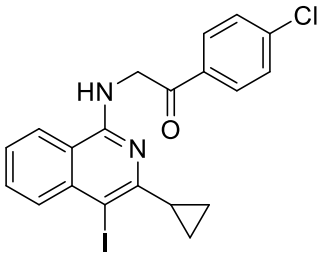
White solid, 135 mg (0.3 mmol, yield 85%), m.p. 163-165 ºC, R_f : 0.33 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, CDCl$_3$)** δ 8.16 (d, *J* = 8.5 Hz, 1H, H-Ar), 8.00 (d, *J* = 8.5 Hz, 2H, H-Ar), 7.93 (d, *J* = 8.3 Hz, 1H, H-Ar), 7.71 (t, *J* = 7.1 Hz, 1H, H-Ar), 7.65-7.54 (m, 3H, H-Ar), 7.50-7.43 (m, 2H, H-Ar), 7.02-6.94 (m, 2H, H-Ar), 6.59 (t, *J* = 3.9 Hz, 1H, NH), 5.01 (d, *J* = 3.9 Hz, 2H, CH_2), 3.90 (s, 3H, OCH_3). **^{13}C {^{1}H} NMR (75 MHz, CDCl$_3$)** δ 194.9, 159.3, 154.4, 153.5, 140.4, 138.7, 136.9, 133.1, 132.8, 131.5, 131.4, 129.5, 129.2, 126.8, 121.8, 118.2, 112.9, 83.7, 55.3, 48.4. **HRMS** (ESI): Calc. for $C_{24}H_{19}ClIN_2O_2$ $[M+H]^+$ 529.0174, found 529.0173.

4d: 2-((4-iodo-3-(4-methoxyphenyl) isoquinolin-1-yl) amino)-1-(p-tolyl) ethan-1-one



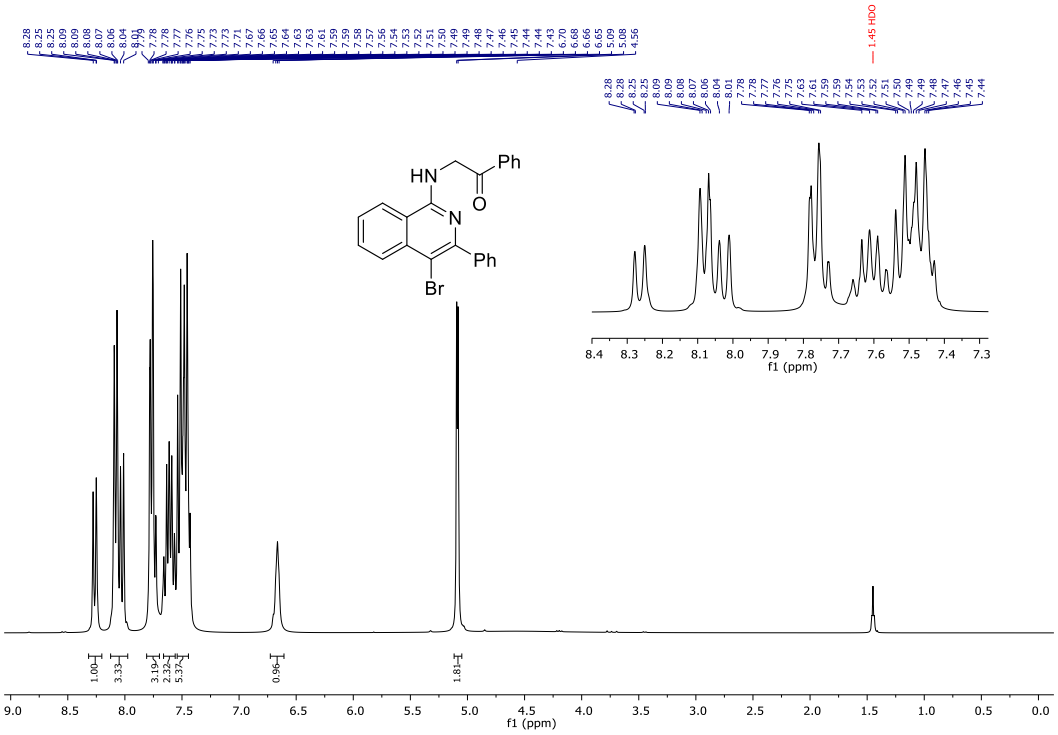
White solid, 145 mg (0.3 mmol, yield 95%), m.p. 148-150 ºC, R_f : 0.55 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.16 (dd, *J* = 8.5, 1.1 Hz, 1H, H-Ar), 7.95 (dd, *J* = 8.1, 4.0 Hz, 3H, H-Ar), 7.70 (t, *J* = 8.3 Hz, 1H, H-Ar), 7.64 (d, *J* = 8.7 Hz, 2H, H-Ar), 7.54 (t, *J* = 8.2 Hz, 1H, H-Ar), 7.29 (d, *J* = 8.1 Hz, 2H, H-Ar), 7.08 – 6.90 (m, 2H, H-Ar), 6.69 (t, *J* = 4.1 Hz, 1H, NH), 5.02 (d, *J* = 4.1 Hz, 2H, CH_2), 3.90 (s, 3H, OCH_3). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 195.4, 159.3, 154.5, 153.7, 144.9, 138.7, 137.1, 132.8, 132.3, 131.4, 131.3, 129.5, 128.1, 126.7, 121.9, 118.3, 112.9, 83.3, 55.3, 48.3, 21.8. **HRMS** (ESI): Calc. for $C_{25}H_{22}IN_2O_2$ $[M+H]^+$ 509.0721, found 509.0720.

4e: 1-(4-chlorophenyl)-2-((3-cyclopropyl-4-iodoisoquinolin-1-yl) amino) ethan-1-one

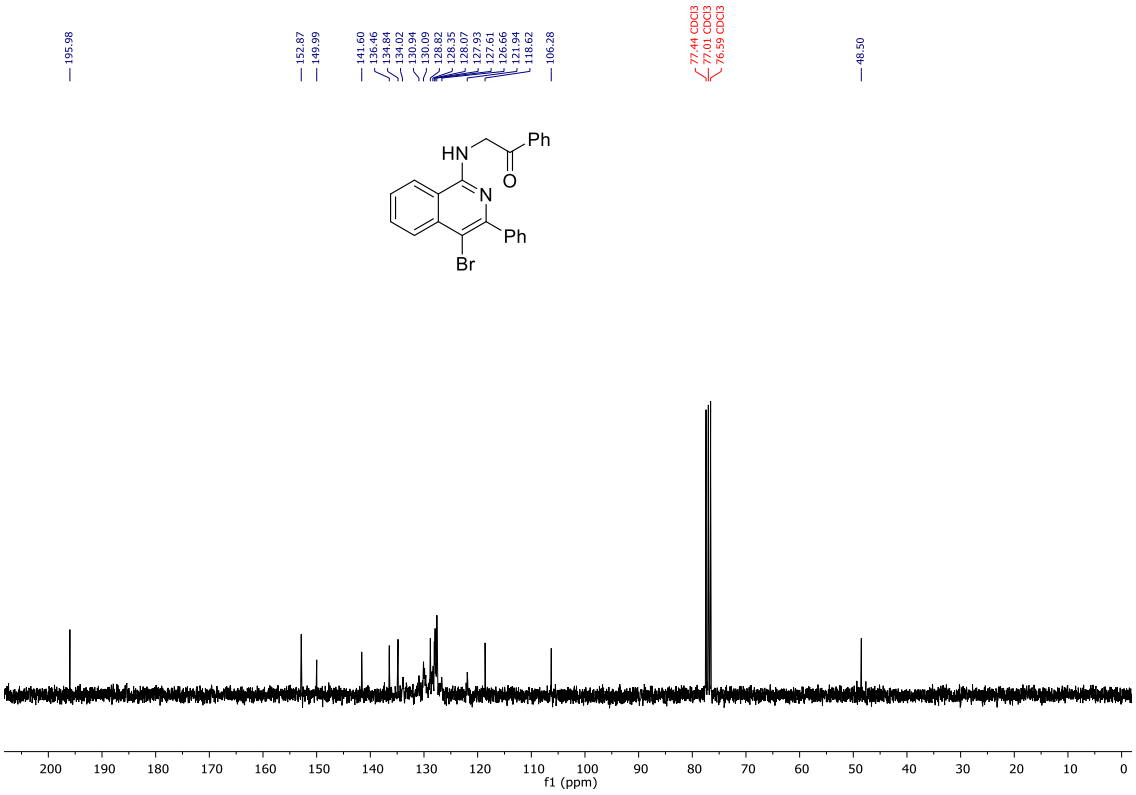


White solid, 69 mg (0.3 mmol, yield 50%), m.p. 142-144 ºC, R_f : 0.55 (Ethyl acetate : n-hexane / 1:2); **^{1}H {^{13}C} NMR (300 MHz, $CDCl_3$)** δ 8.06 – 7.92 (m, 3H, H-Ar), 7.79 (dd, *J* = 8.4, 1.2 Hz, 1H, H-Ar), 7.62 (td, *J* = 7.8, 1.2 Hz, 1H, H-Ar), 7.51 (d, *J* = 8.6 Hz, 2H, H-Ar), 7.43 (td, *J* = 7.6, 1.2 Hz, 1H, H-Ar), 6.36 (t, *J* = 4.3 Hz, 1H, NH), 4.90 (d, *J* = 4.3 Hz, 2H, CH_2), 2.66 (tt, *J* = 8.1, 4.8 Hz, 1H, CH), 1.06 (dt, *J* = 4.8, 2.9 Hz, 2H, H-aliphatic), 0.98 – 0.76 (m, 2H, H-aliphatic). **^{13}C {^{1}H} NMR (75 MHz, $CDCl_3$)** δ 194.7, 154.9, 153.6, 140.4, 138.3, 133.1, 131.6, 131.1, 129.3, 129.2, 125.7, 121.8, 118.2, 84.5, 48.0, 20.9, 9.6. **HRMS** (ESI): Calc. for $C_{20}H_{17}ClIN_2O$ $[M+H]^+$ 463.0069, found 463.0065.

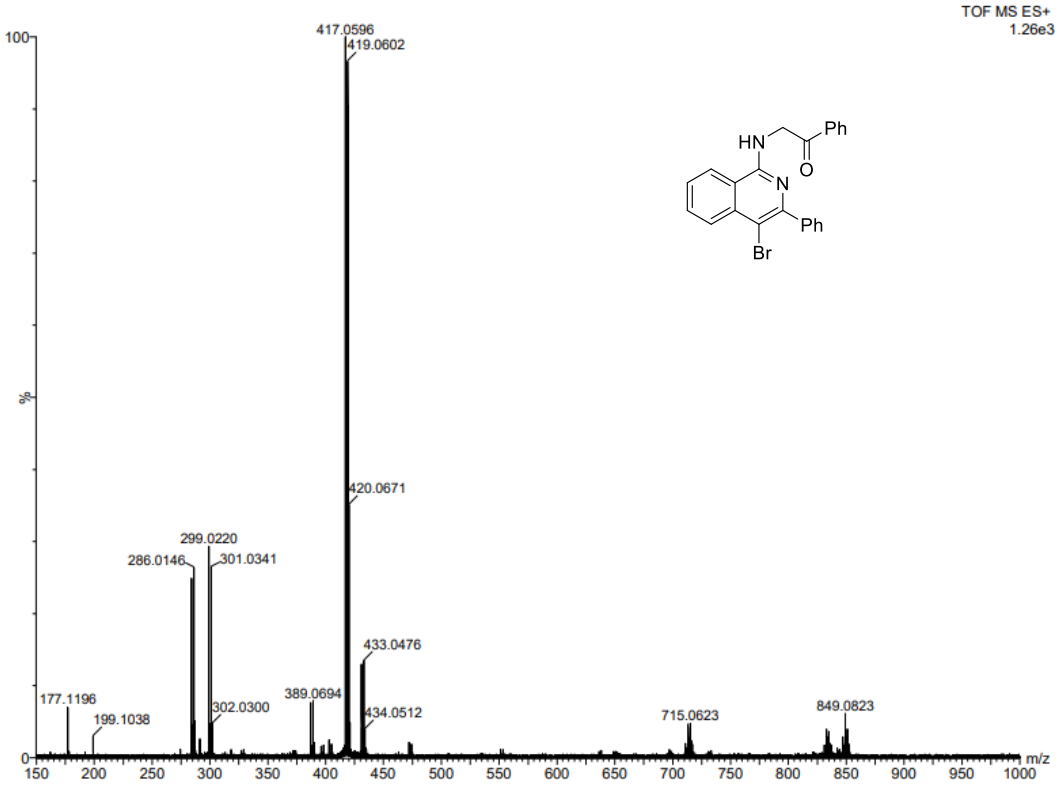
^{1}H NMR, ^{13}C NMR and HRMS Spectra:



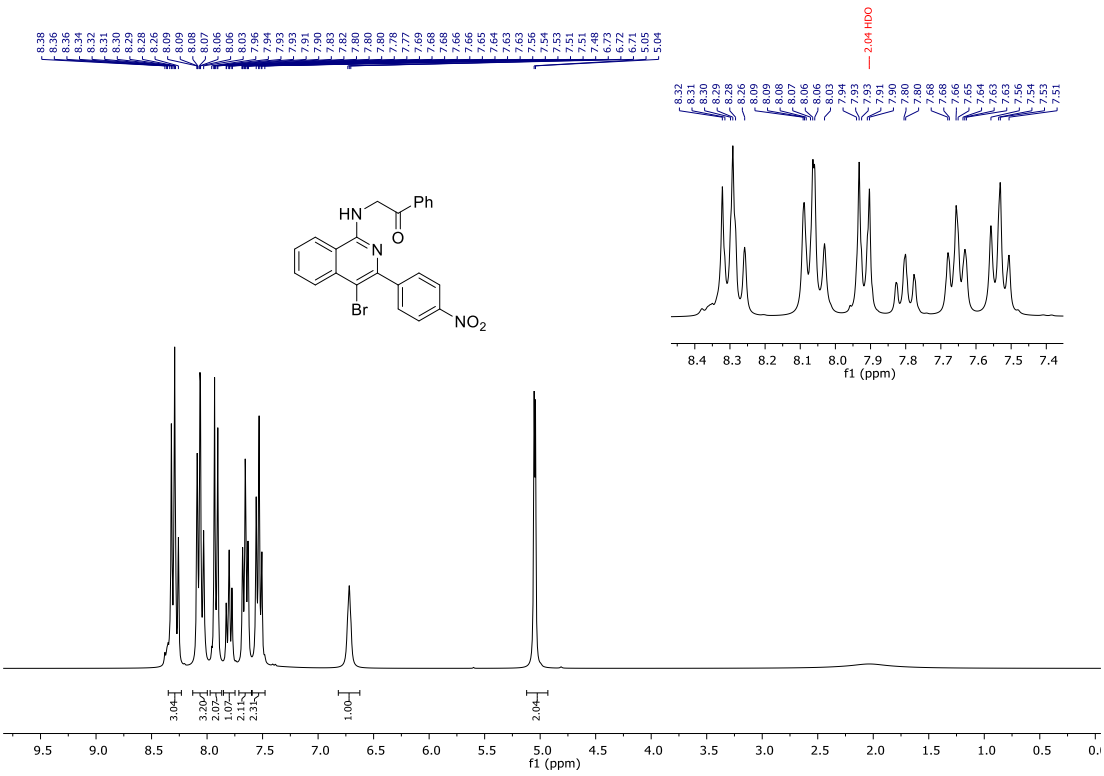
^{1}H-NMR of compound (**3a**) (300 MHz, $CDCl_3$)



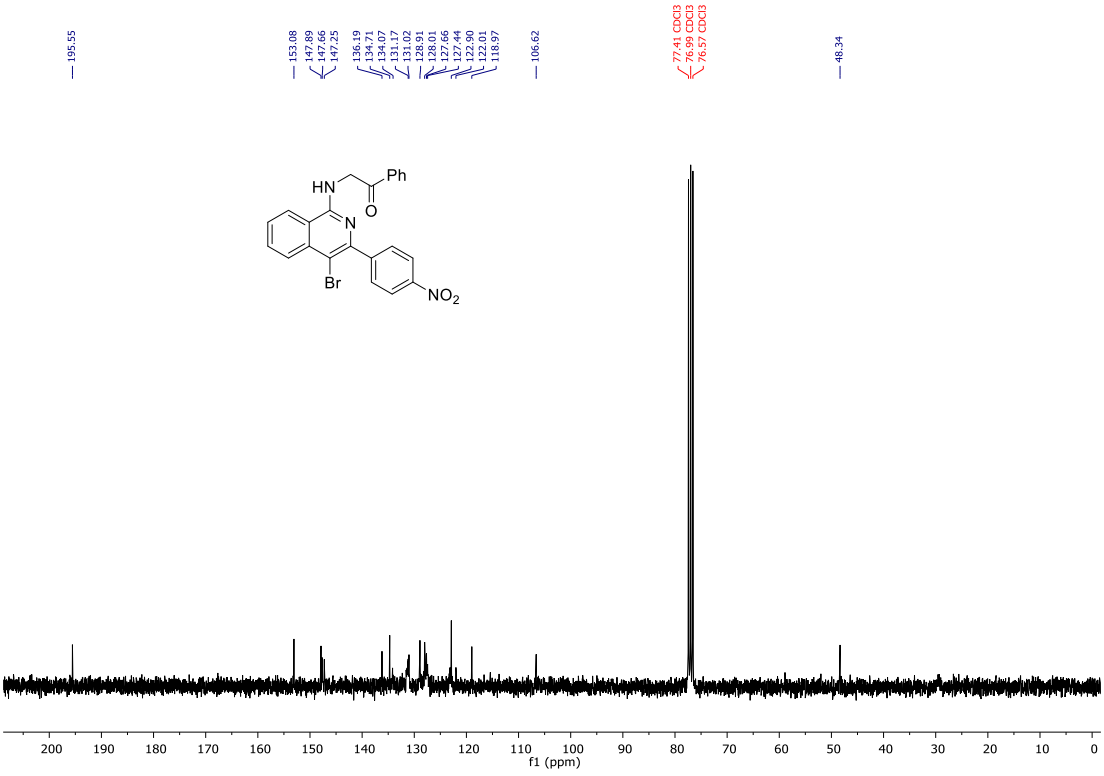
^{13}C-NMR of compound (**3a**) (75 MHz, $CDCl_3$)



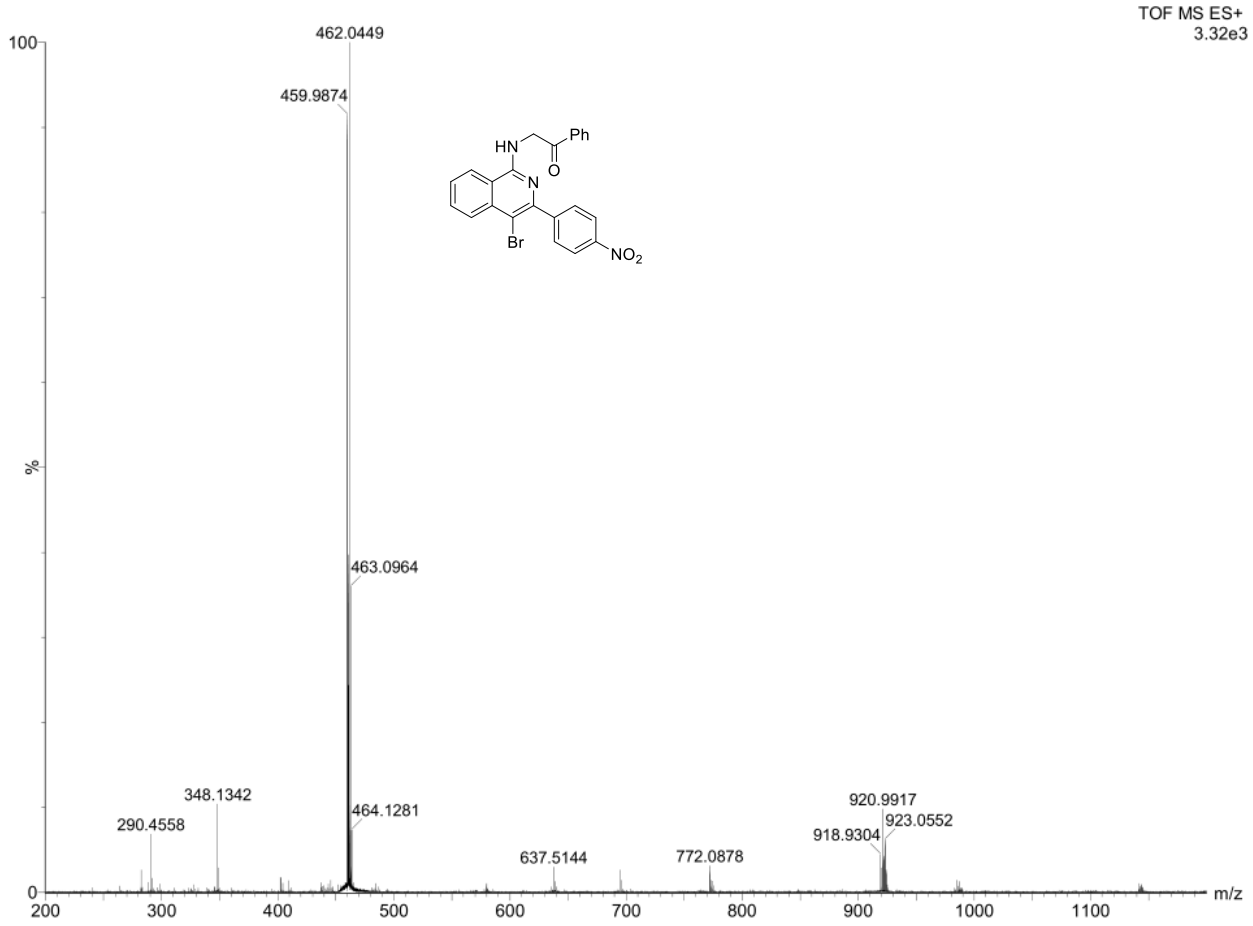
HRMS-ESI (m/z) of (**3a**)



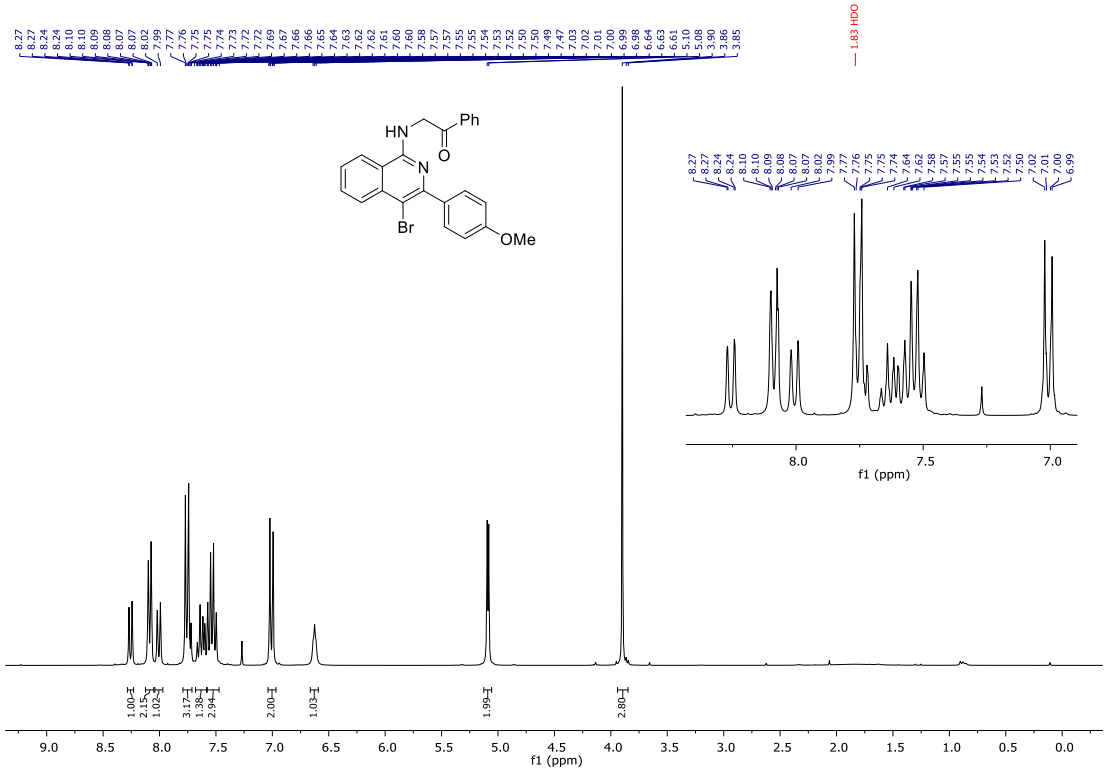
^{1}H-NMR of compound (**3b**) (300 MHz, $CDCl_3$)



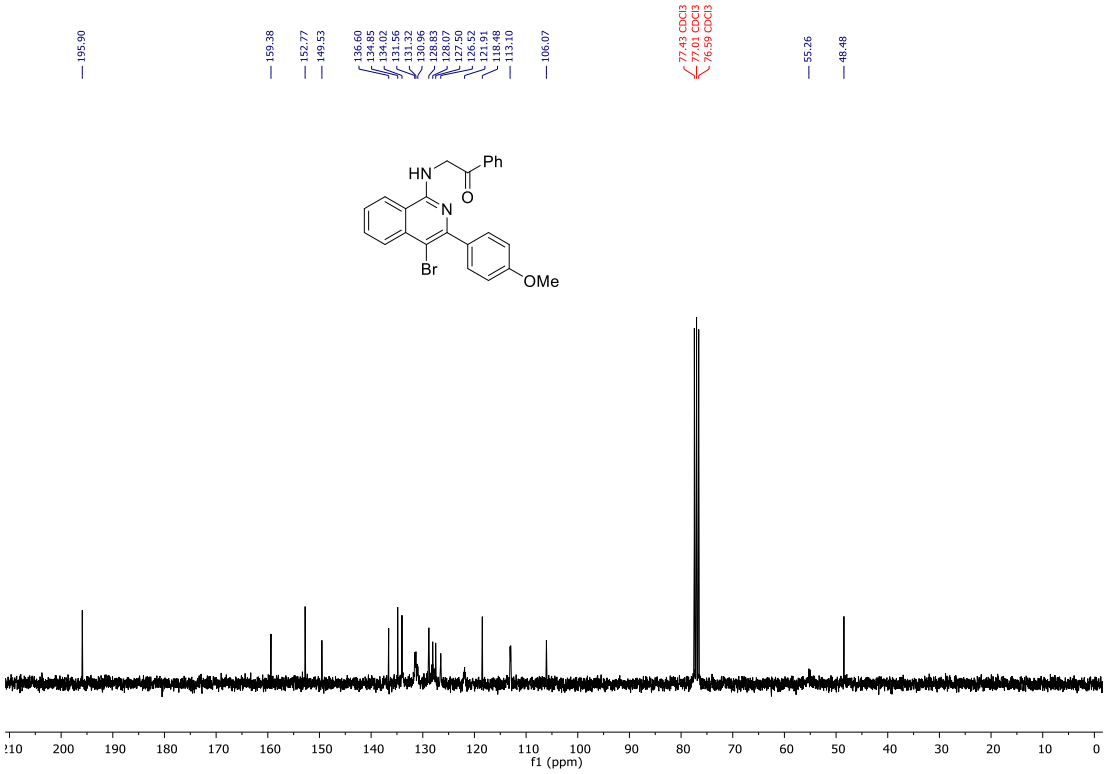
^{13}C-NMR of compound (**3b**) (75 MHz, $CDCl_3$)



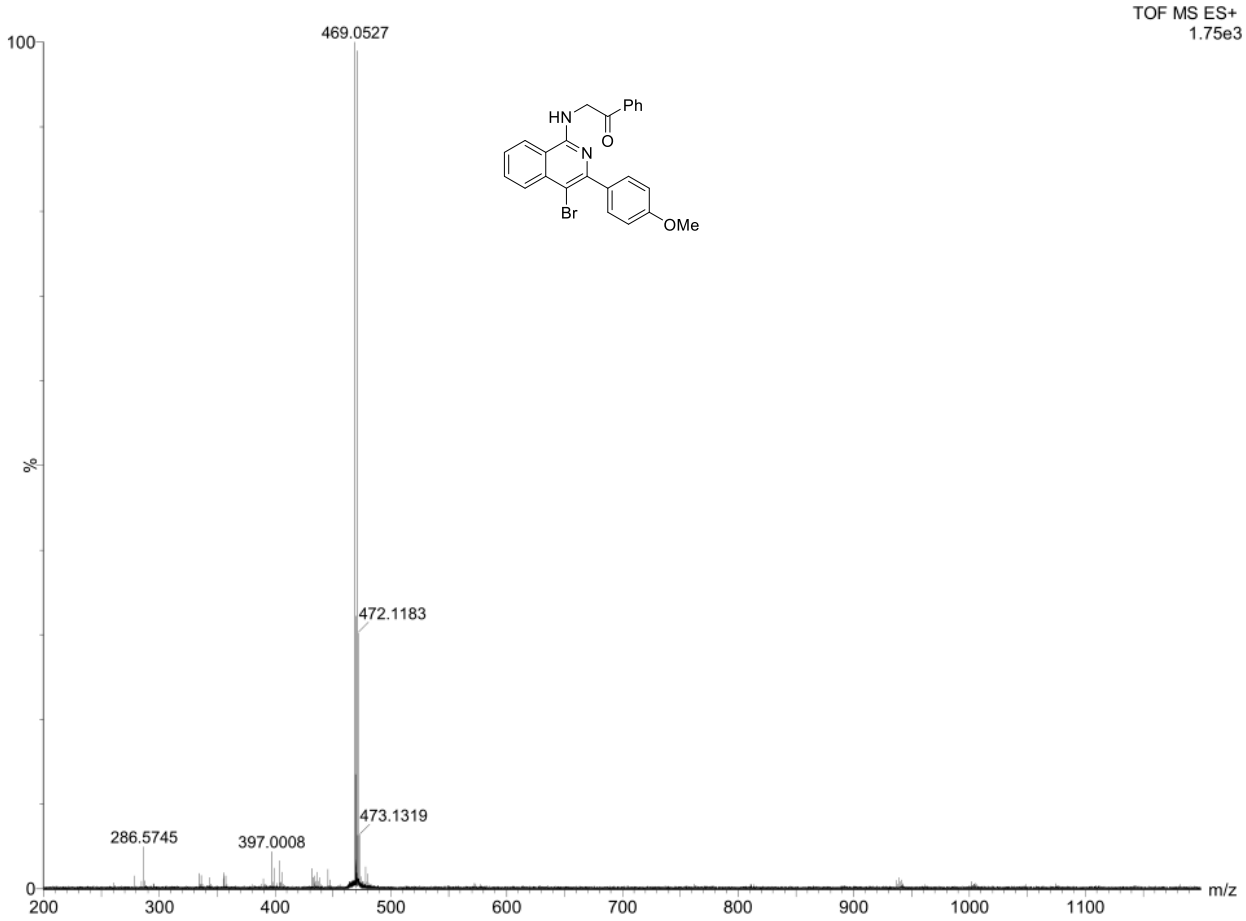
HRMS-ESI (m/z) of (**3b**)



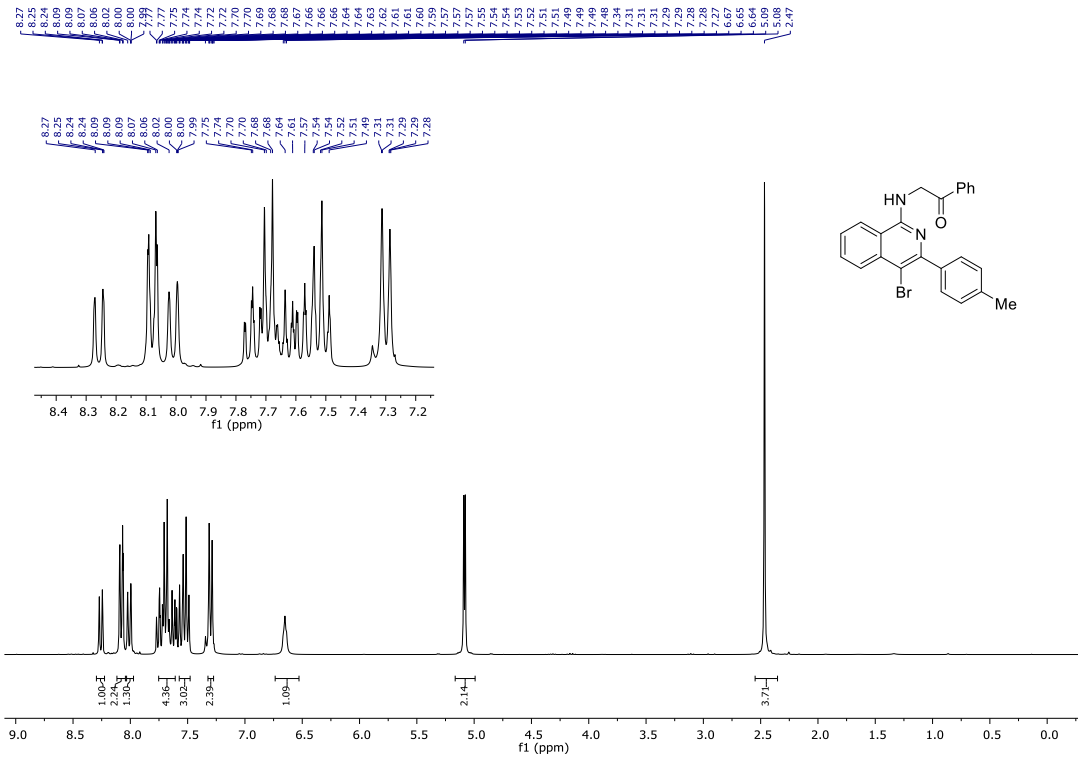
^{1}H-NMR of compound (**3c**) (300 MHz, $CDCl_3$)



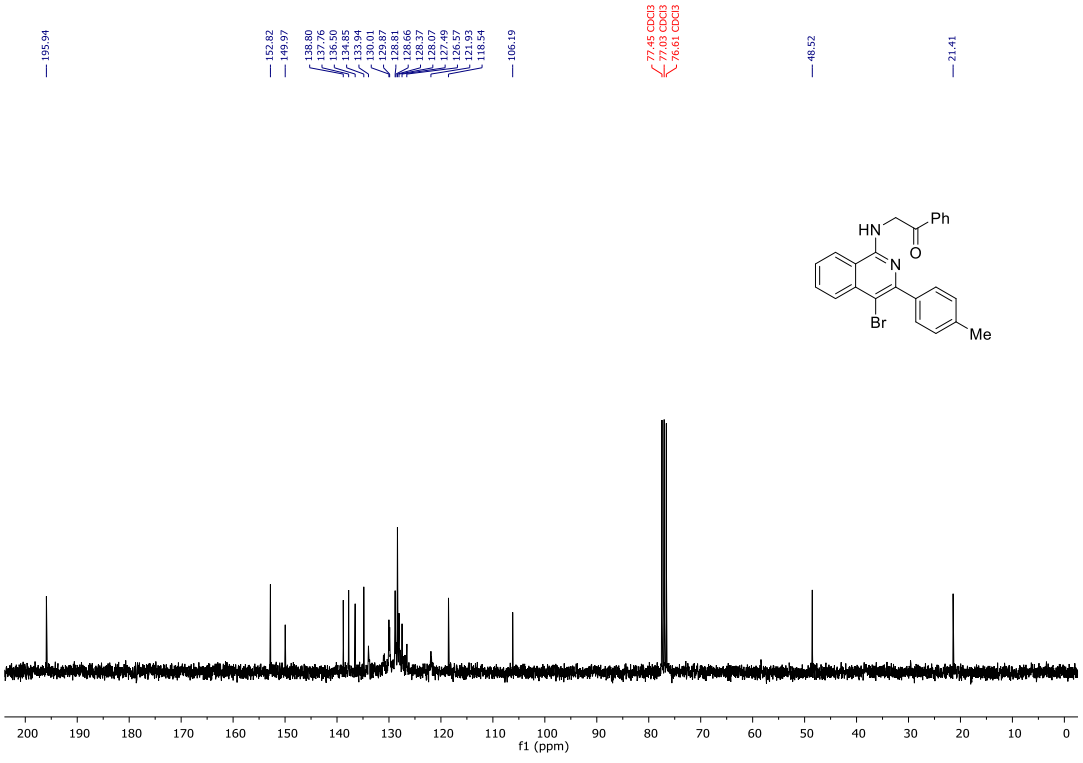
^{13}C-NMR of compound (**3c**) (75 MHz, $CDCl_3$)



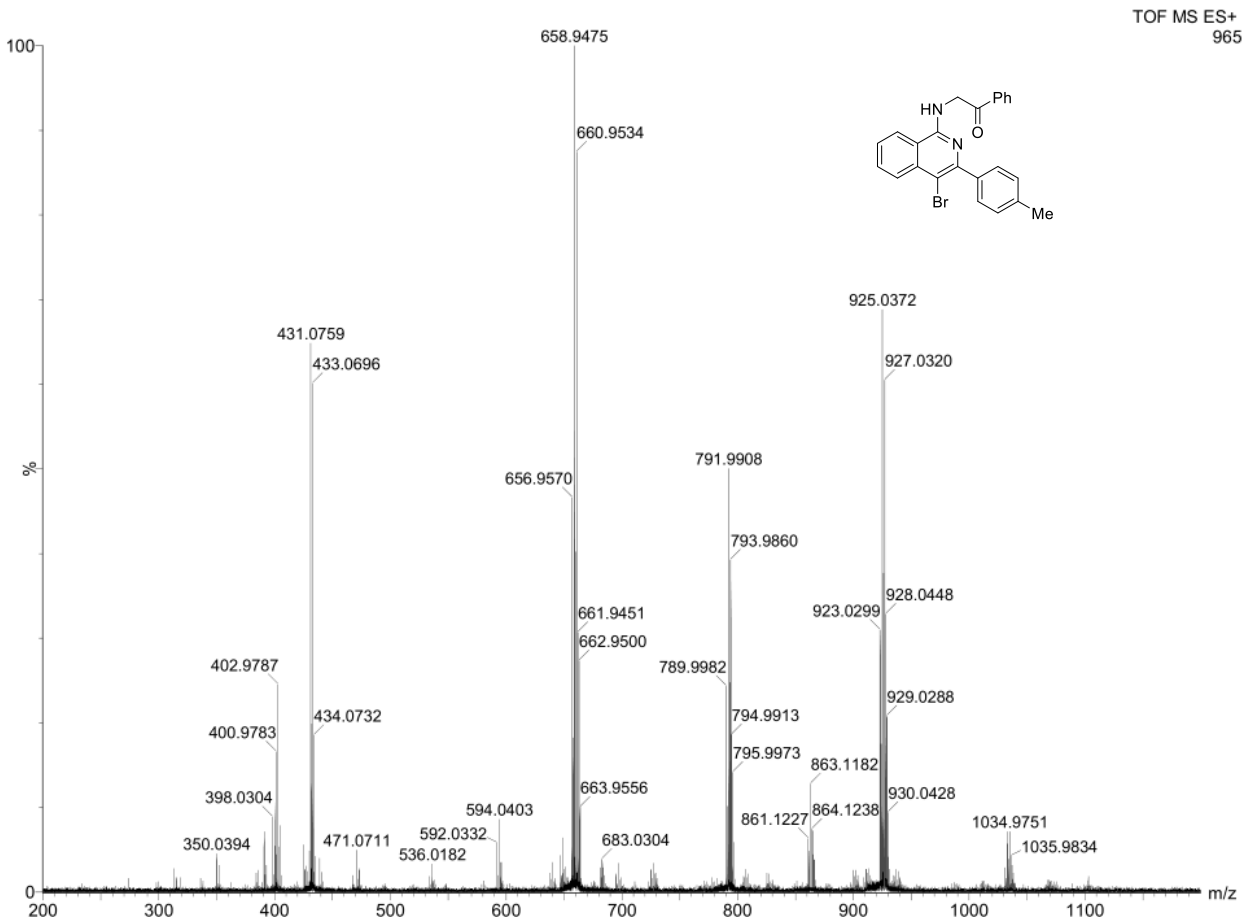
HRMS-ESI (m/z) of (**3c**)



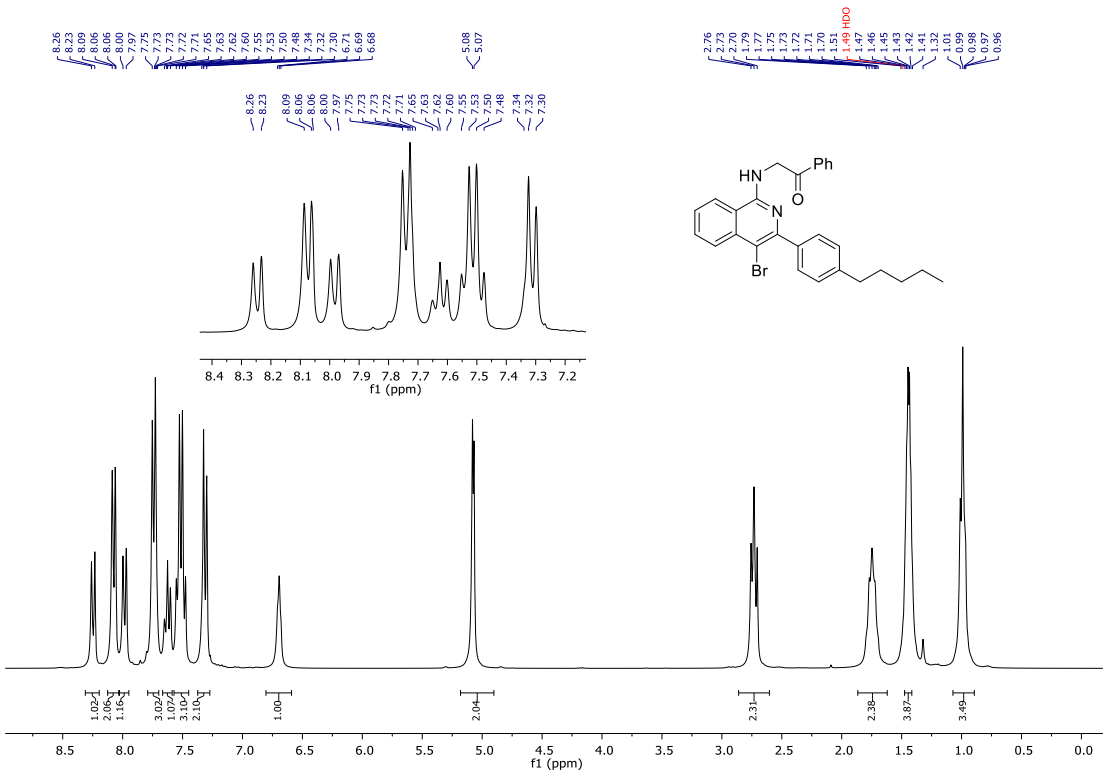
^{1}H-NMR of compound (**3d**) (300 MHz, $CDCl_3$)



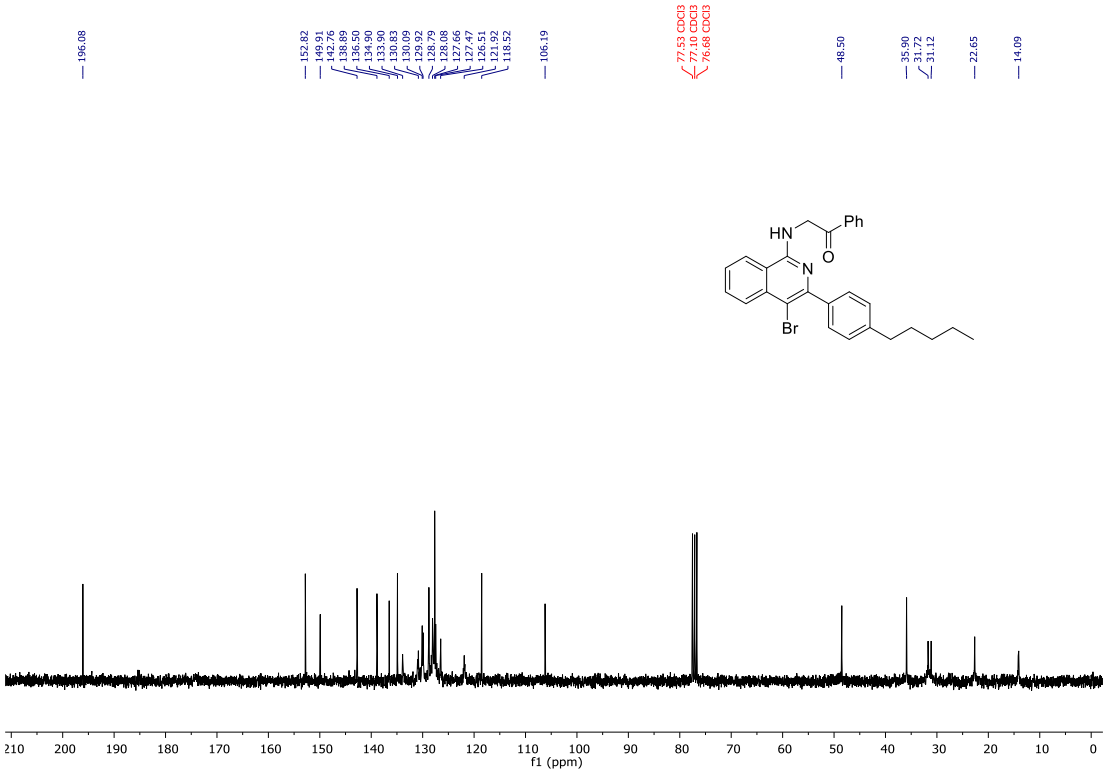
^{13}C-NMR of compound (**3d**) (75 MHz, $CDCl_3$)



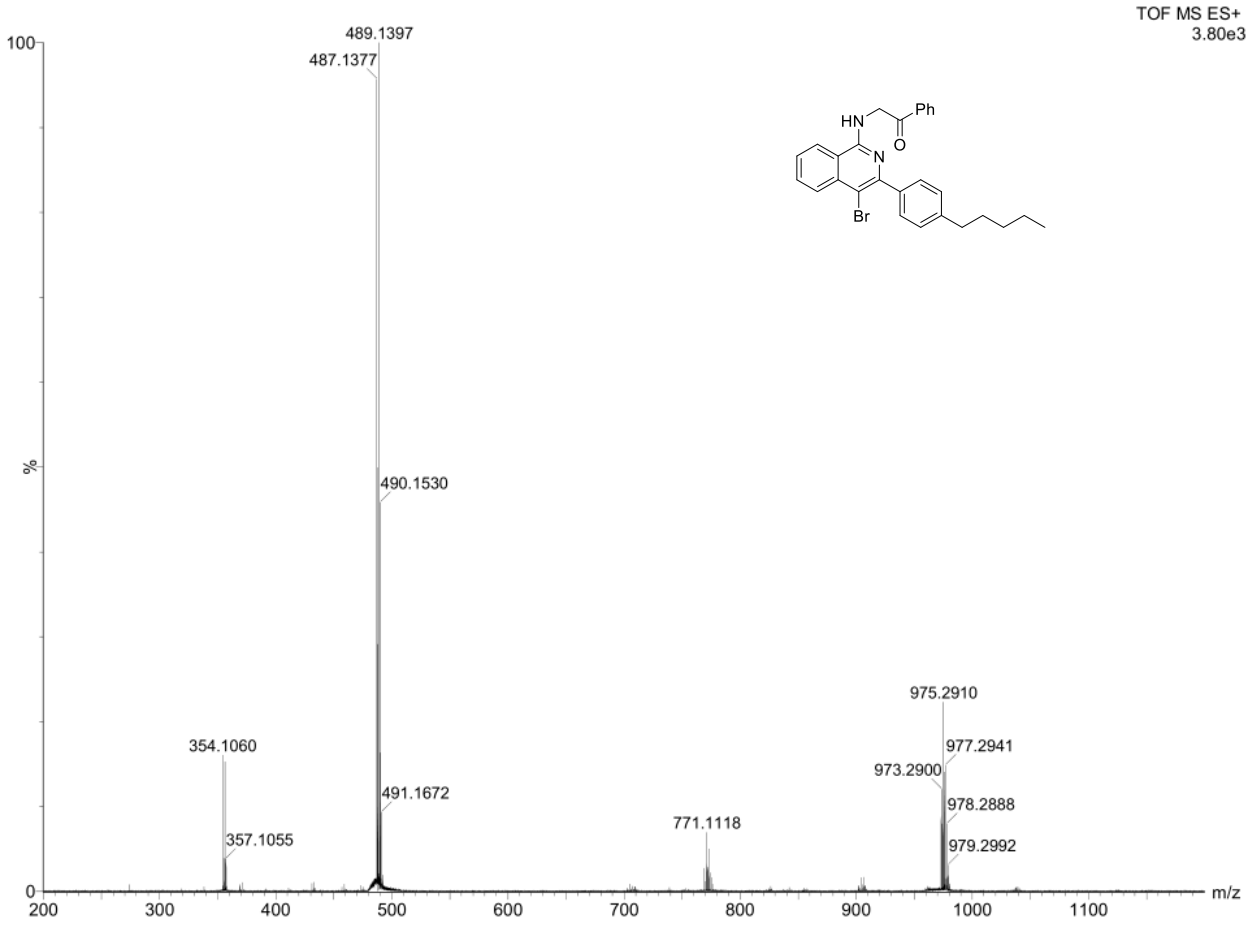
HRMS-ESI (m/z) of (**3d**)



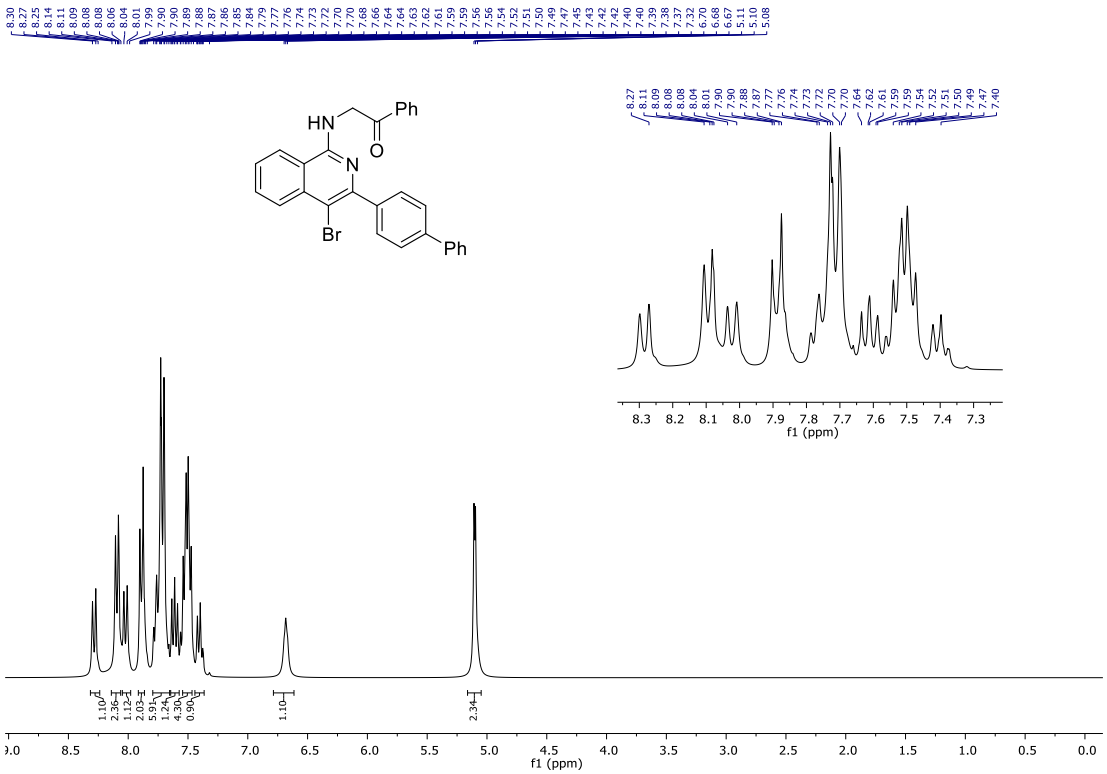
^{1}H-NMR of compound (**3e**) (300 MHz, $CDCl_3$)



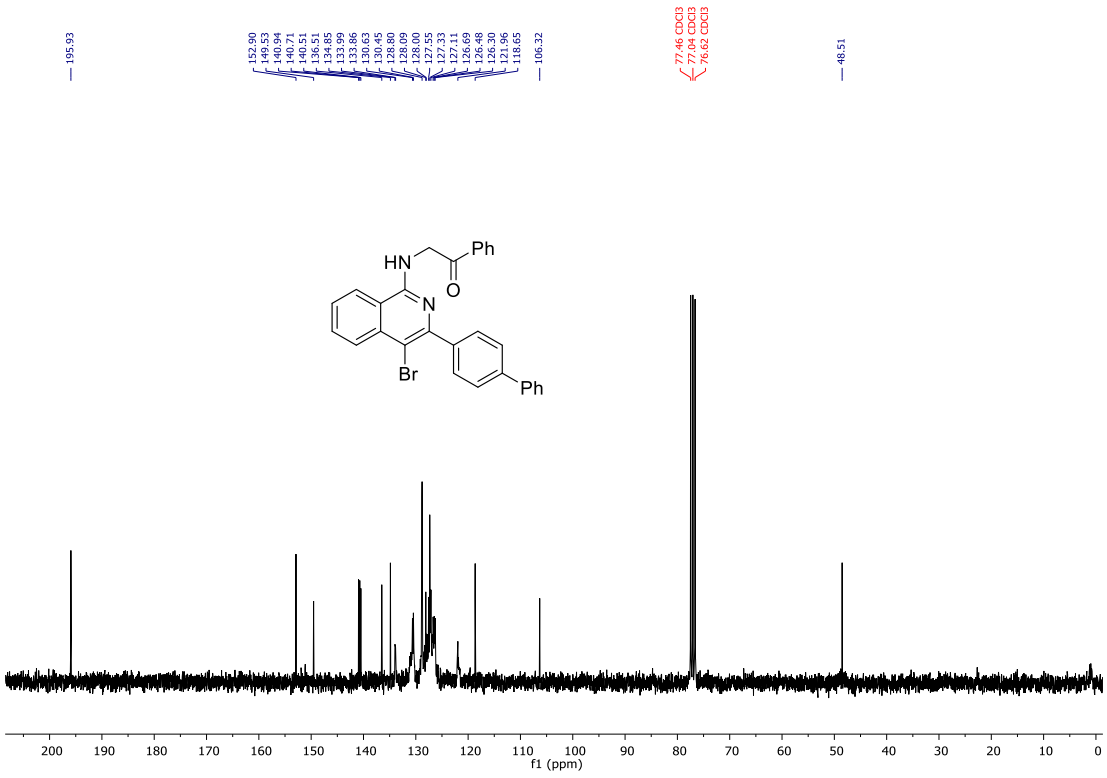
^{13}C-NMR of compound (**3e**) (75 MHz, $CDCl_3$)



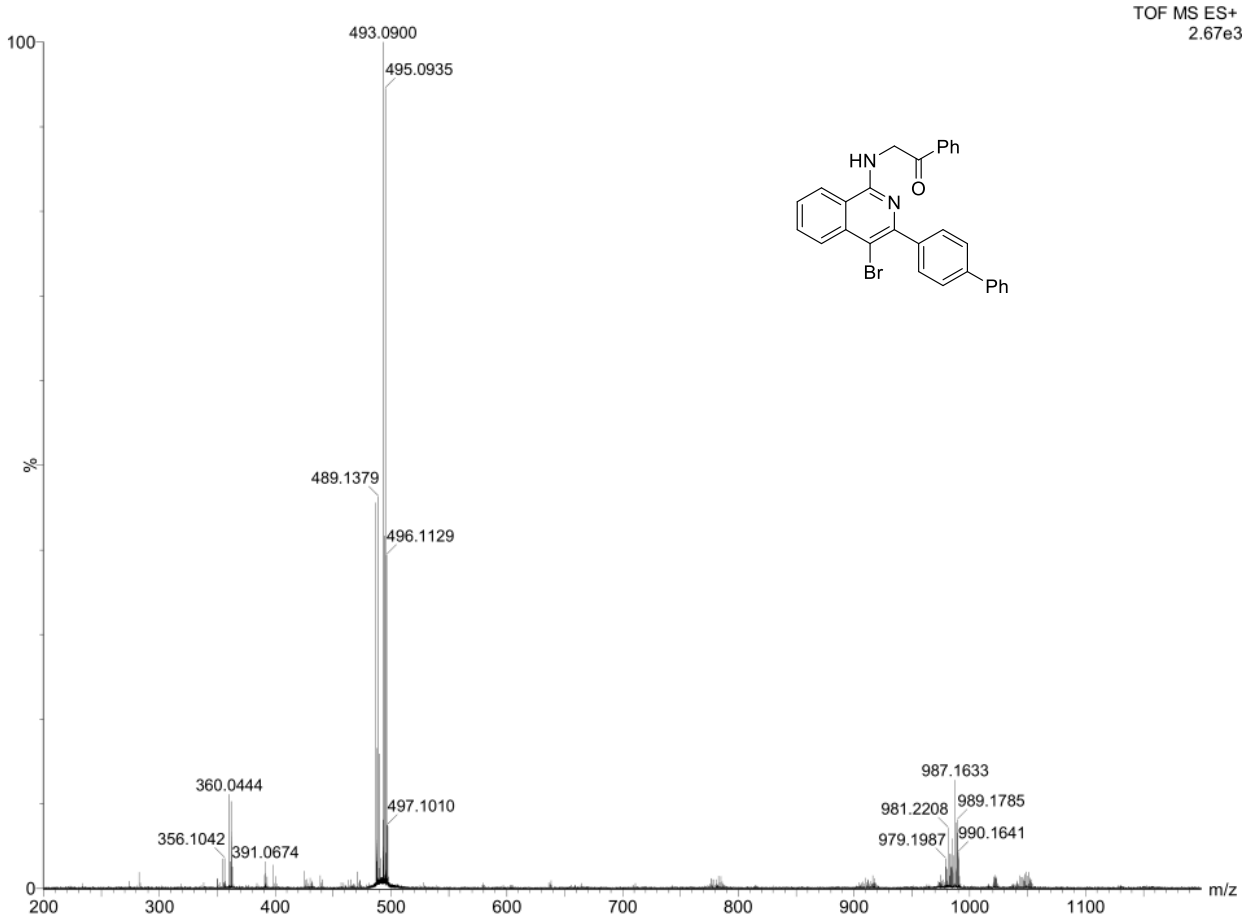
HRMS-ESI (m/z) of (**3e**)



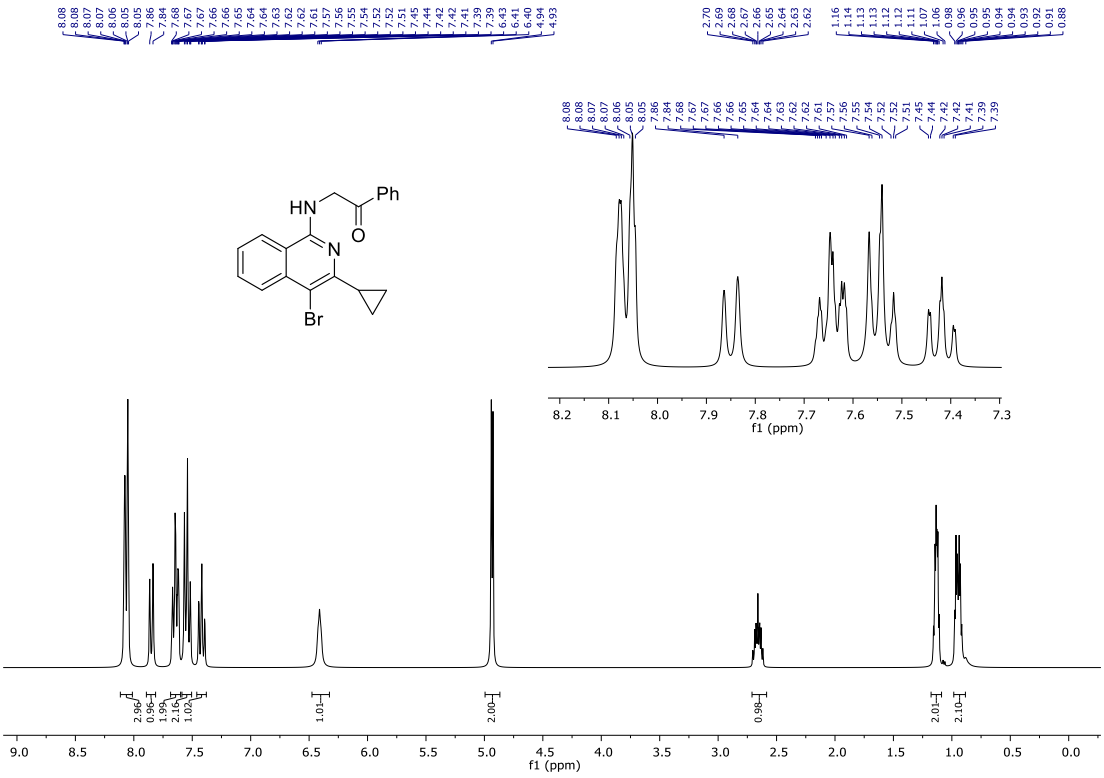
^{1}H-NMR of compound (**3f**) (300 MHz, $CDCl_3$)



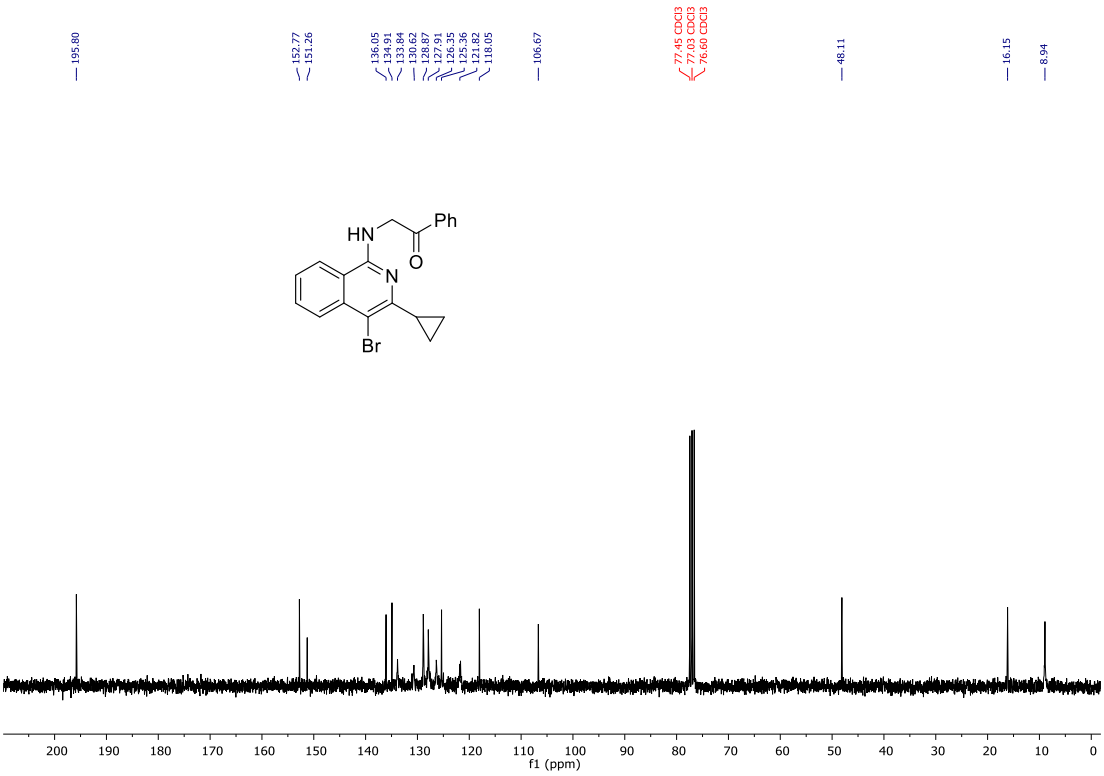
^{13}C-NMR of compound (**3f**) (75 MHz, $CDCl_3$)



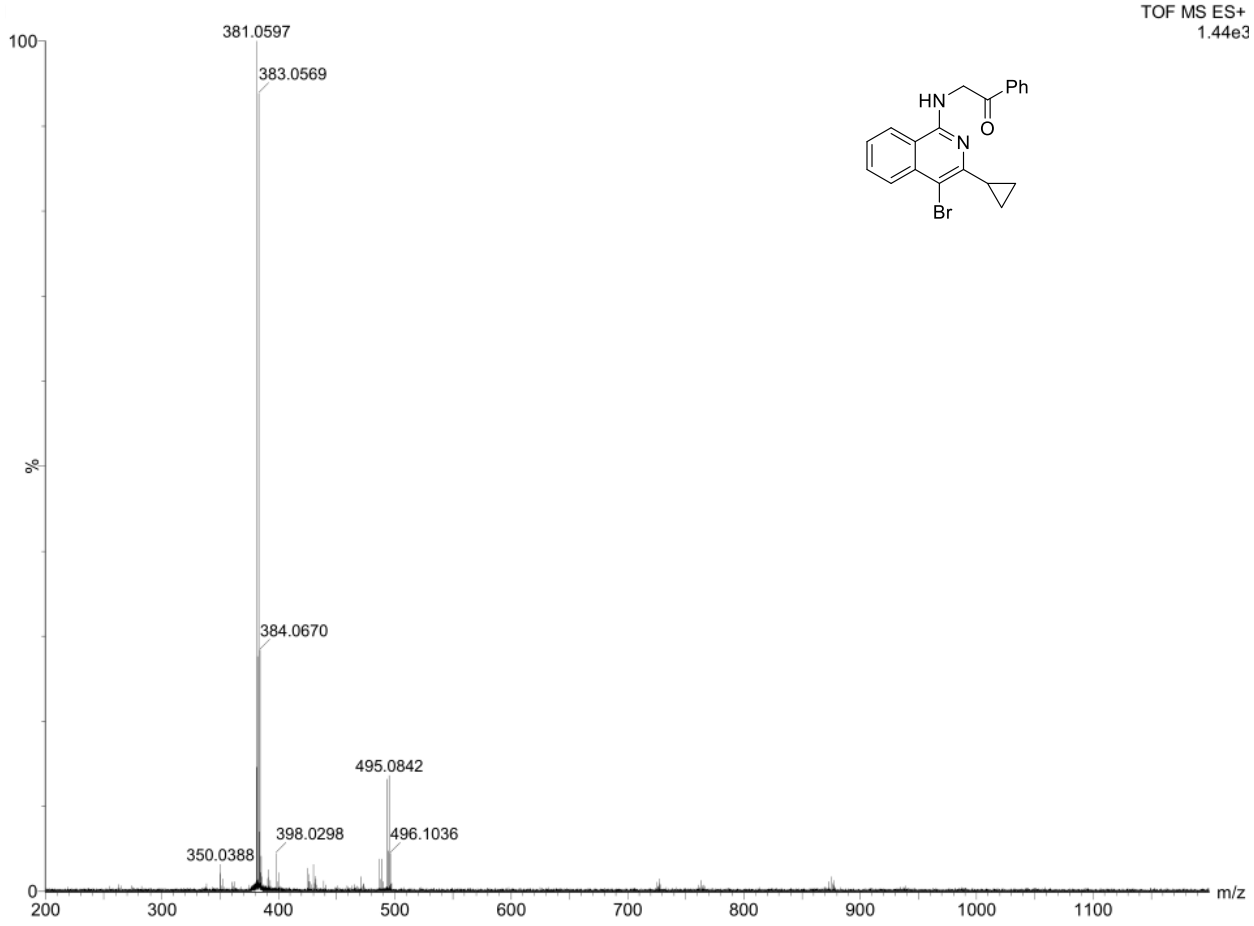
HRMS-ESI (m/z) of (**3f**)



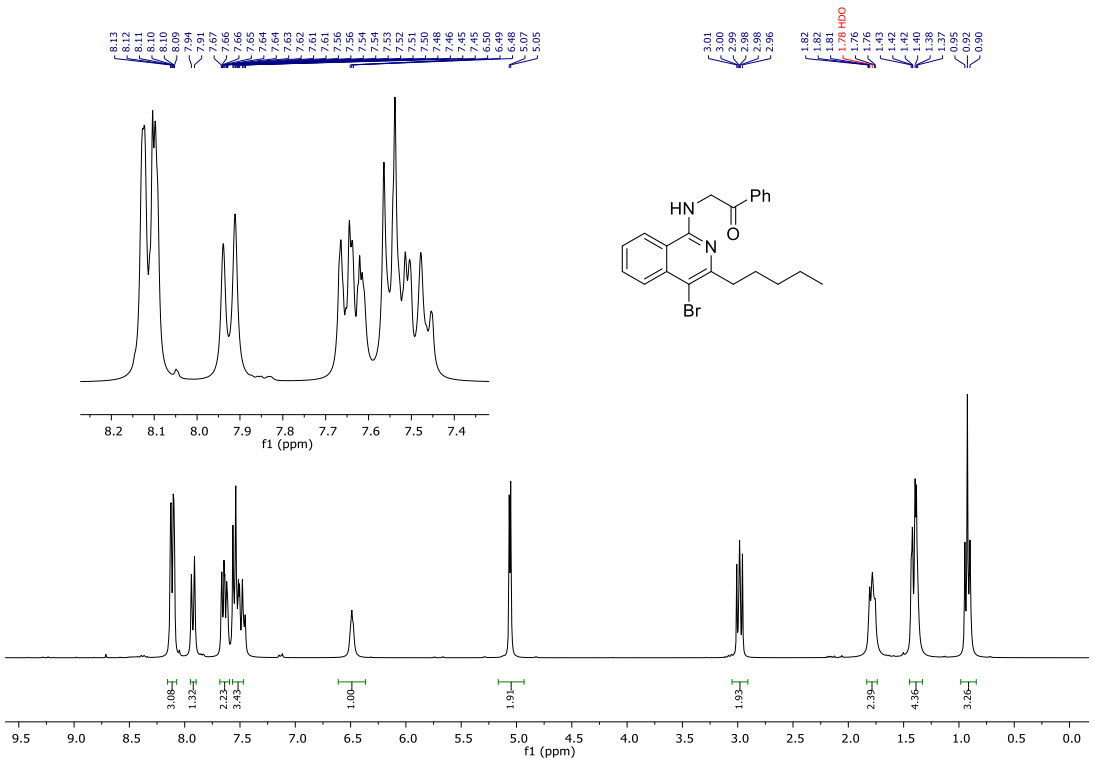
^{1}H-NMR of compound (**3g**) (300 MHz, $CDCl_3$)



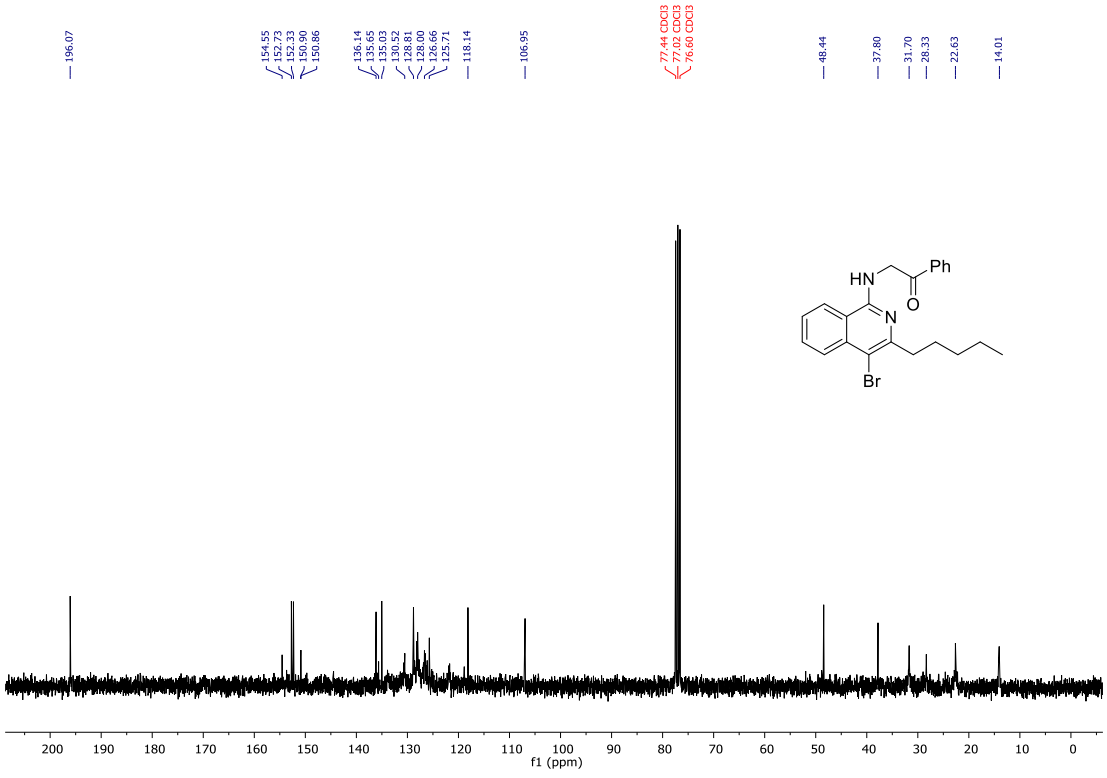
^{13}C-NMR of compound (**3g**) (75 MHz, $CDCl_3$)



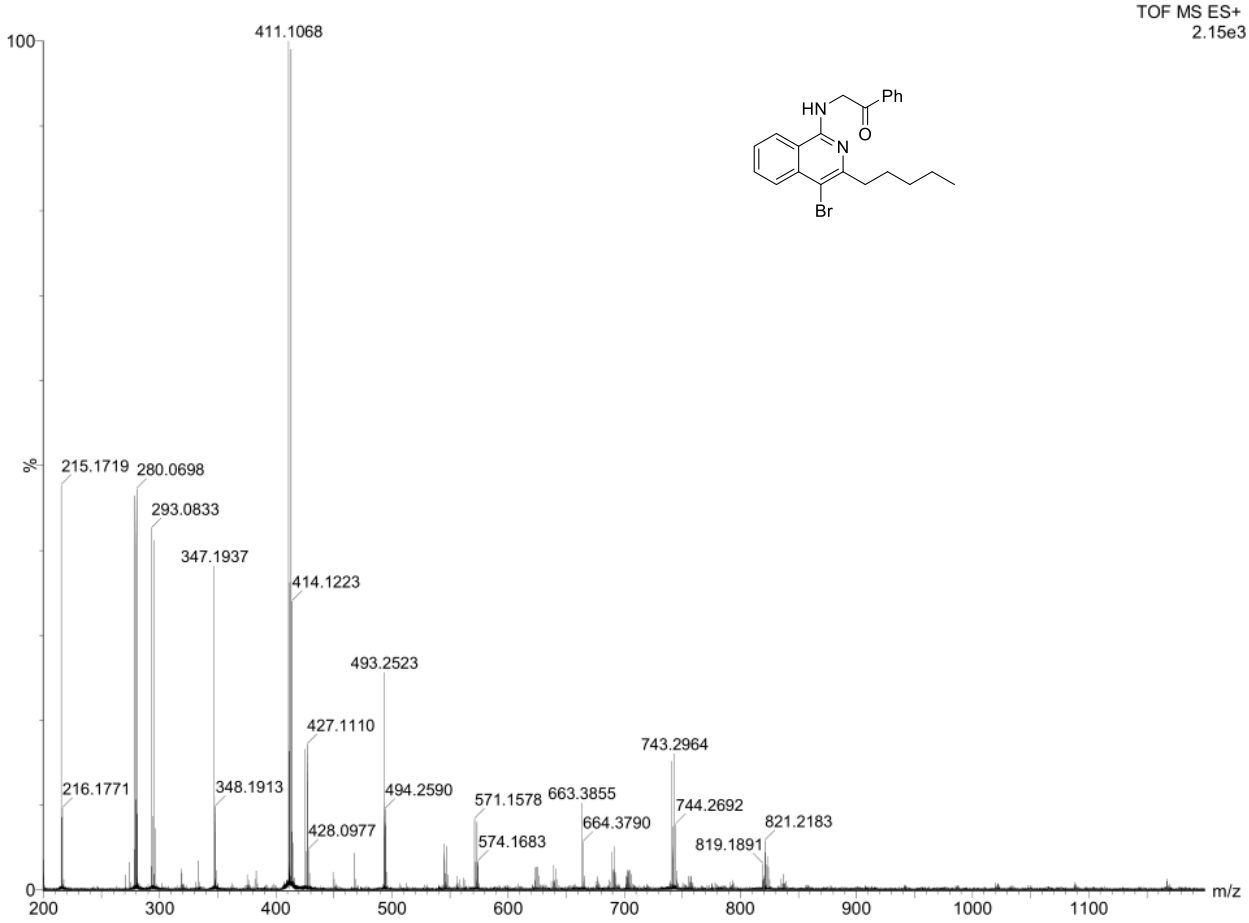
HRMS-ESI (m/z) of (**3g**)



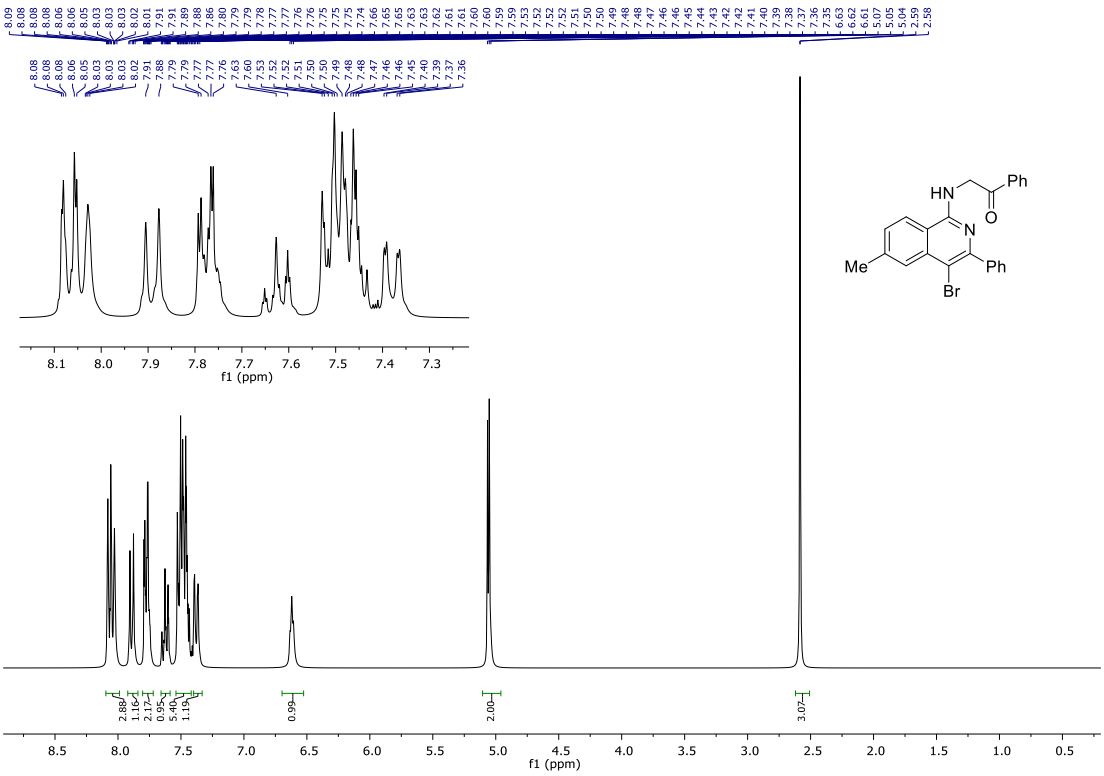
^{1}H-NMR of compound (**3h**) (300 MHz, $CDCl_3$)



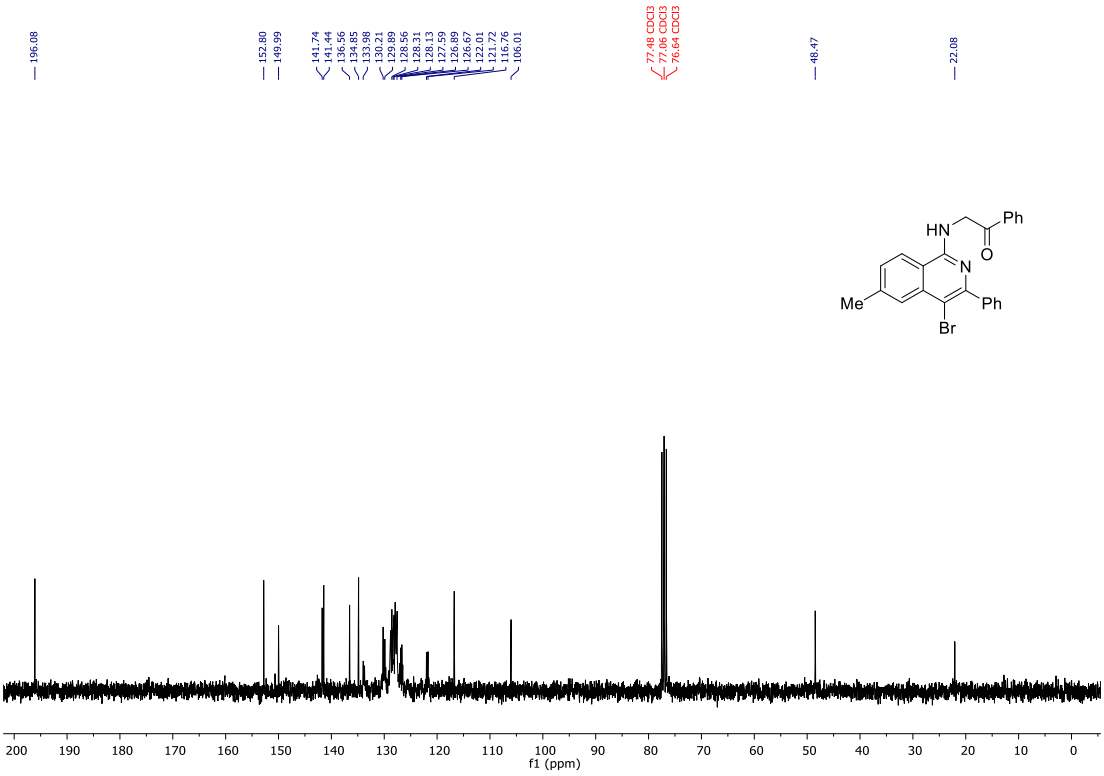
^{13}C-NMR of compound (**3h**) (75 MHz, $CDCl_3$)



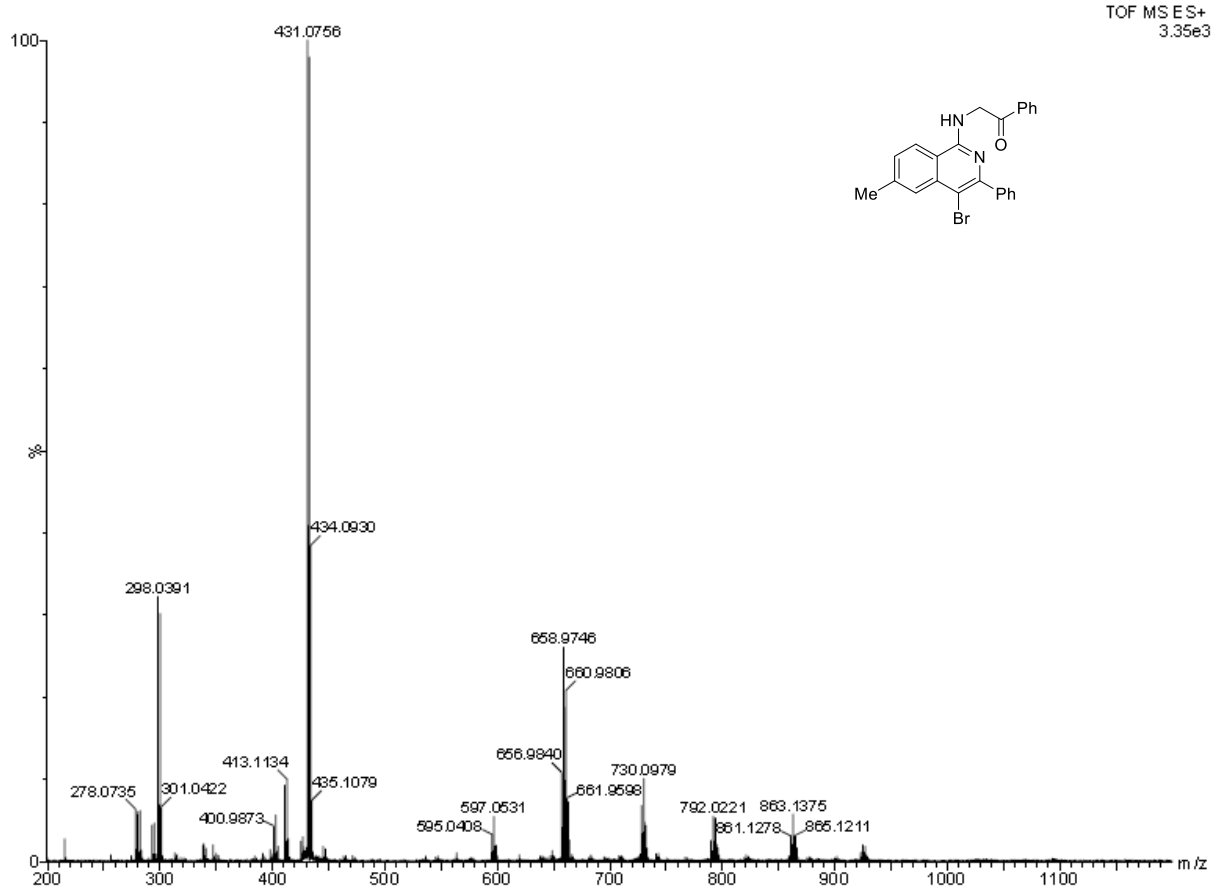
HRMS-ESI (m/z) of (**3h**)



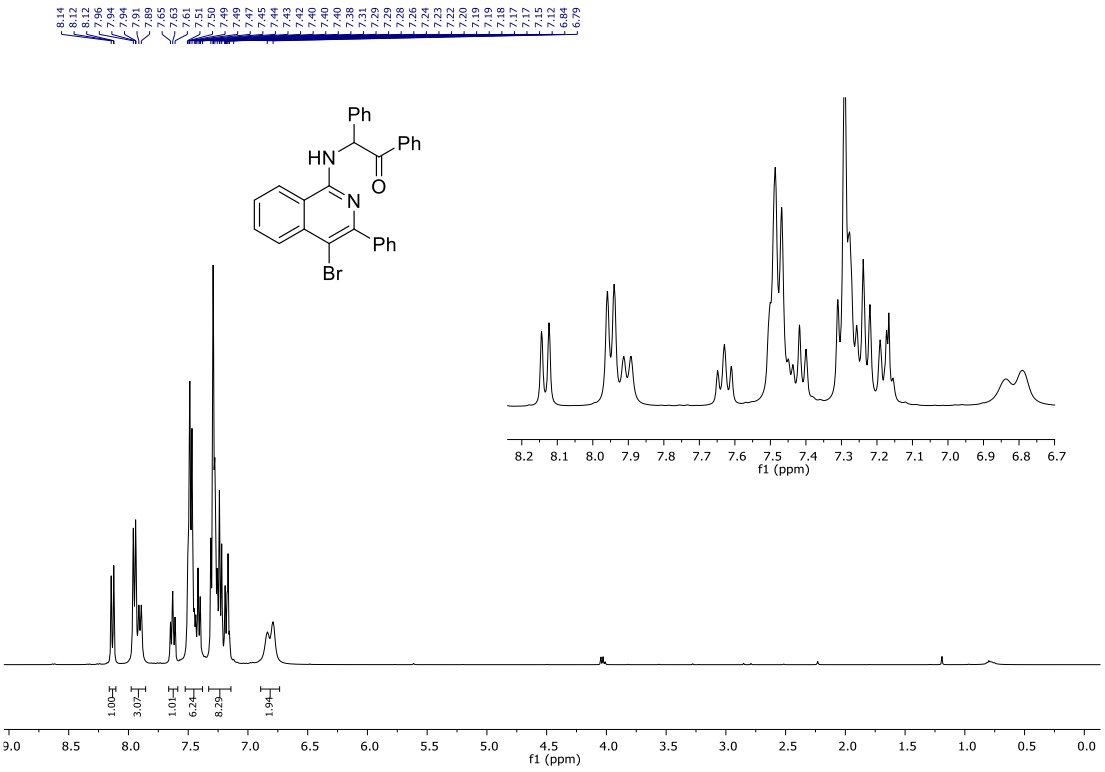
^{1}H-NMR of compound (**3i**) (300 MHz, $CDCl_3$)



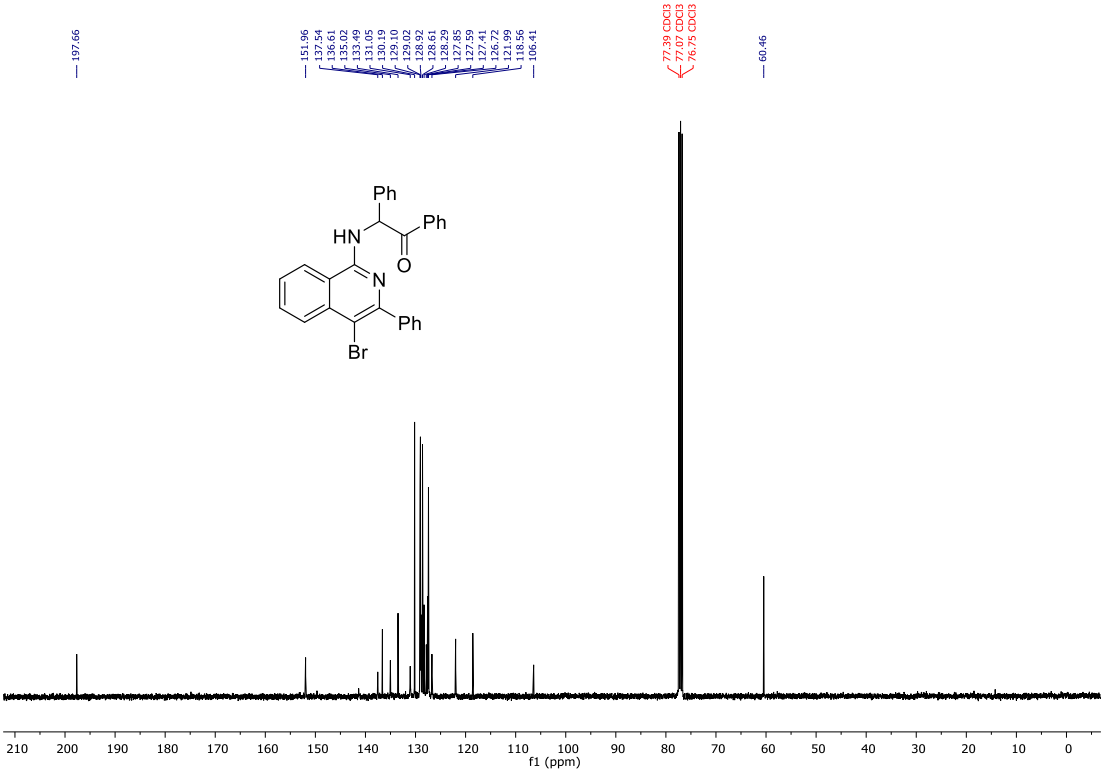
^{13}C-NMR of compound (**3i**) (75 MHz, $CDCl_3$)



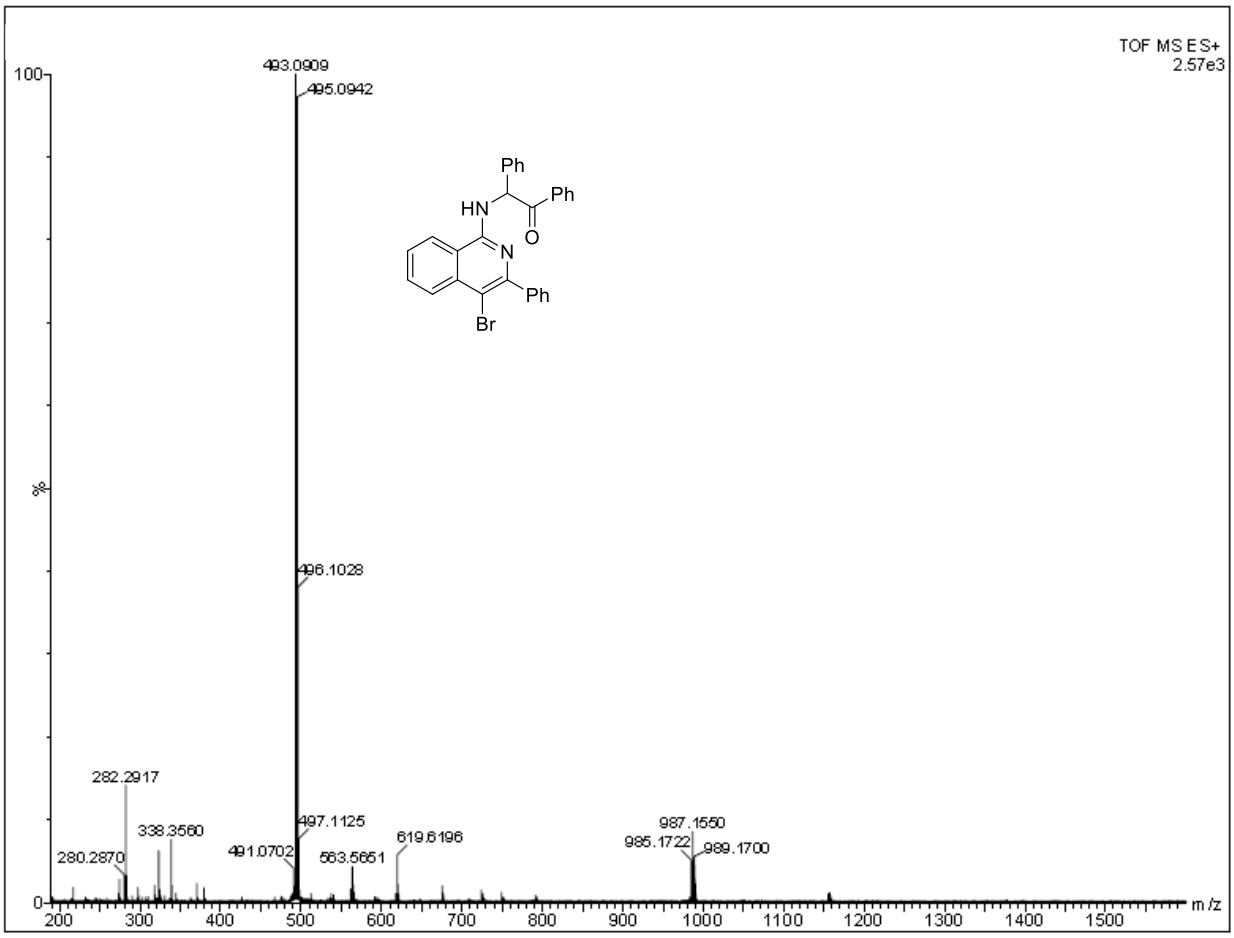
HRMS-ESI (m/z) of (**3i**)



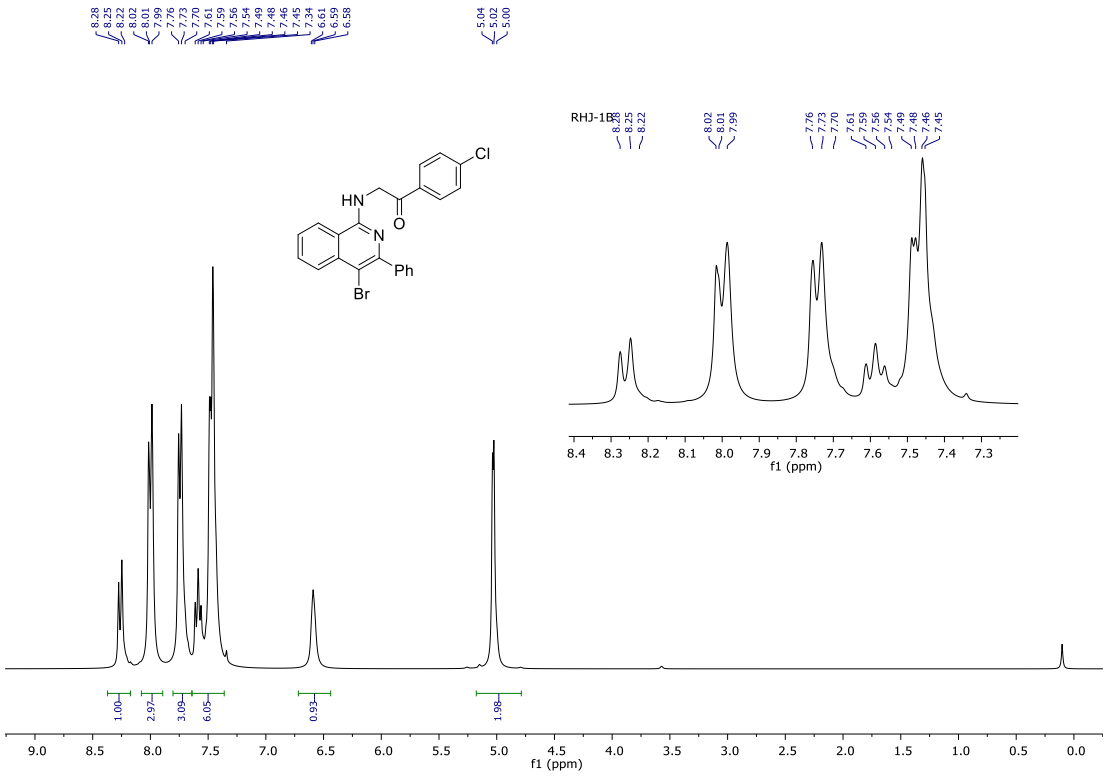
^{1}H-NMR of compound (**3j**) (300 MHz, $CDCl_3$)



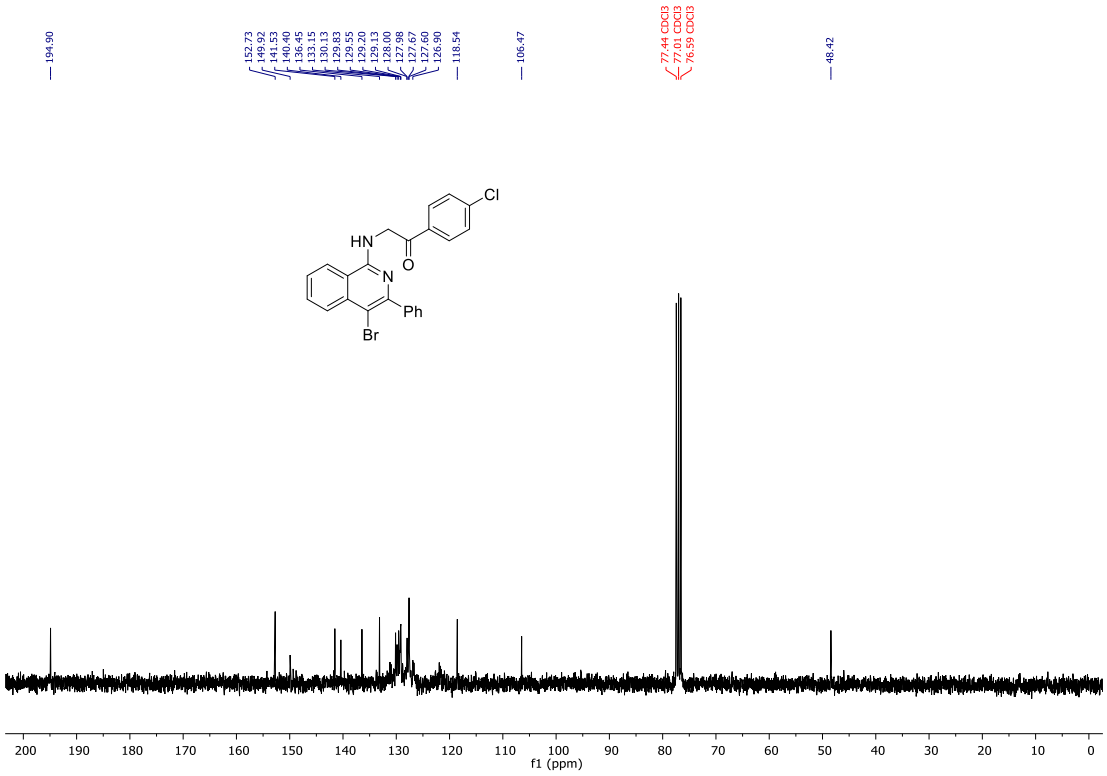
^{13}C-NMR of compound (**3j**) (75 MHz, $CDCl_3$)



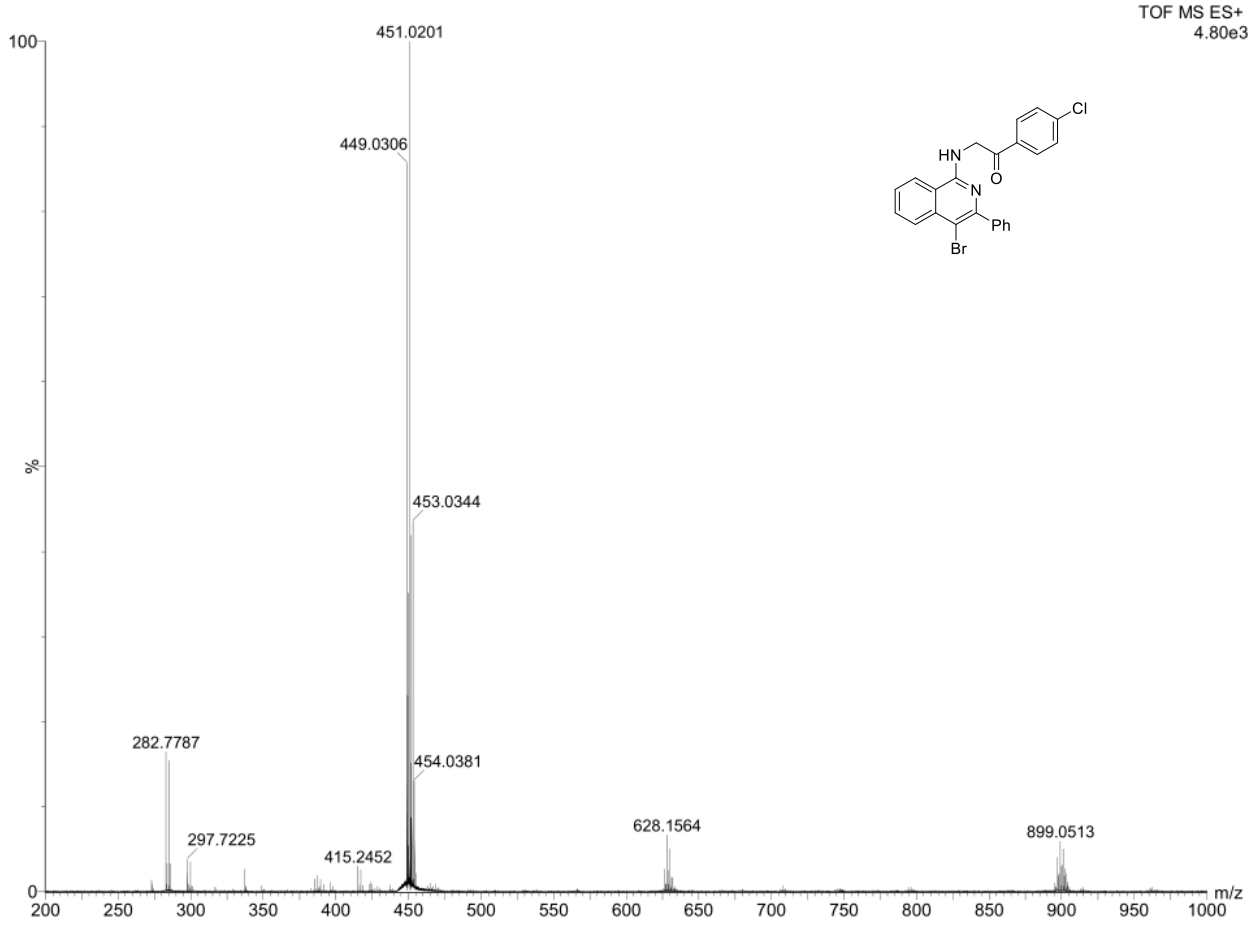
HRMS-ESI (m/z) of (**3j**)



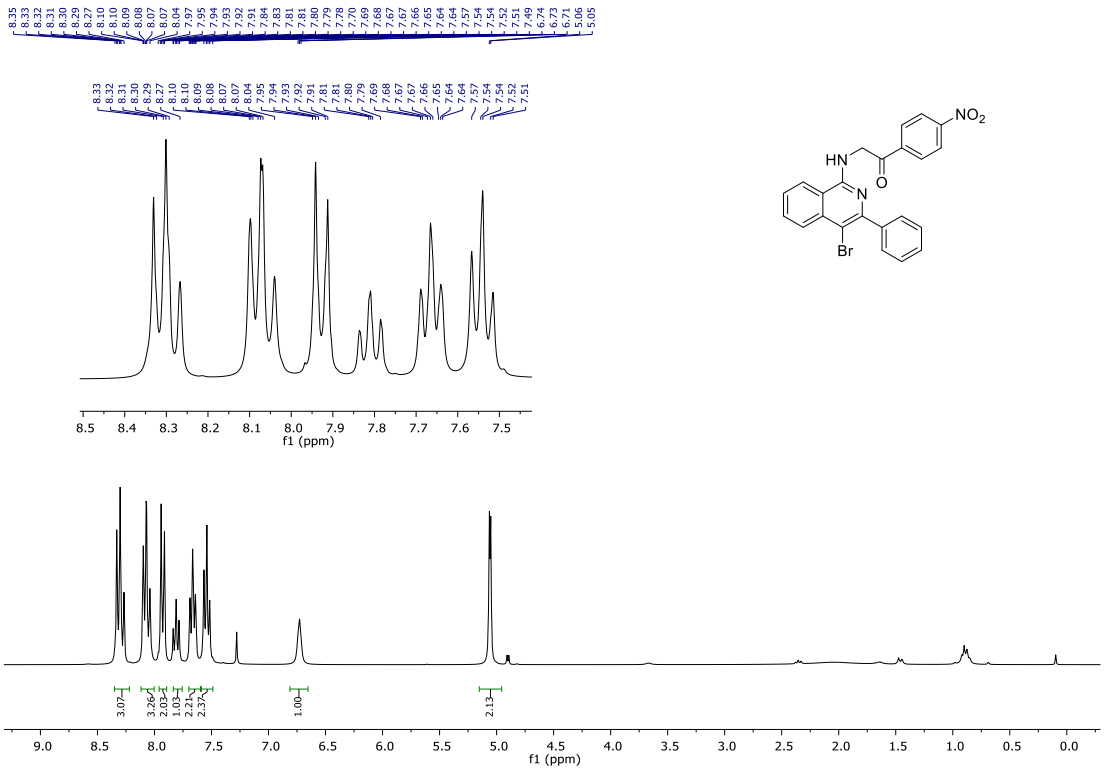
^{1}H-NMR of compound (**3k**) (300 MHz, $CDCl_3$)



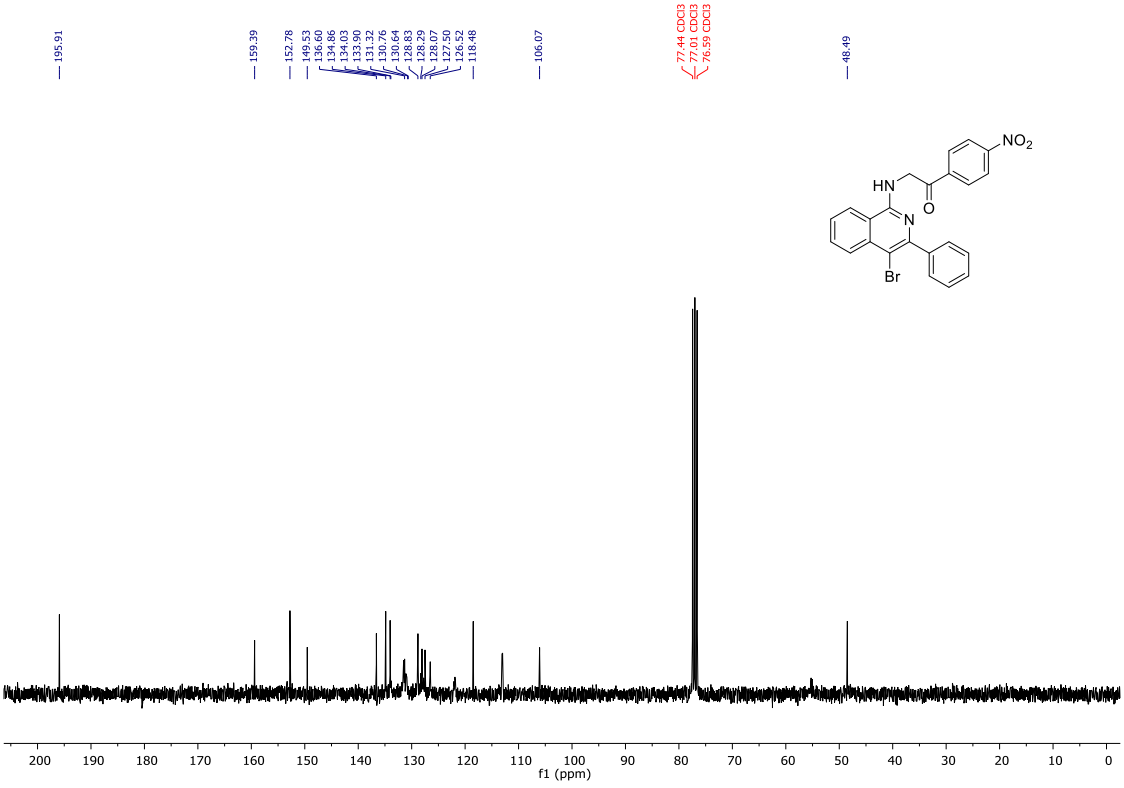
^{13}C-NMR of compound (**3K**) (75 MHz, $CDCl_3$)



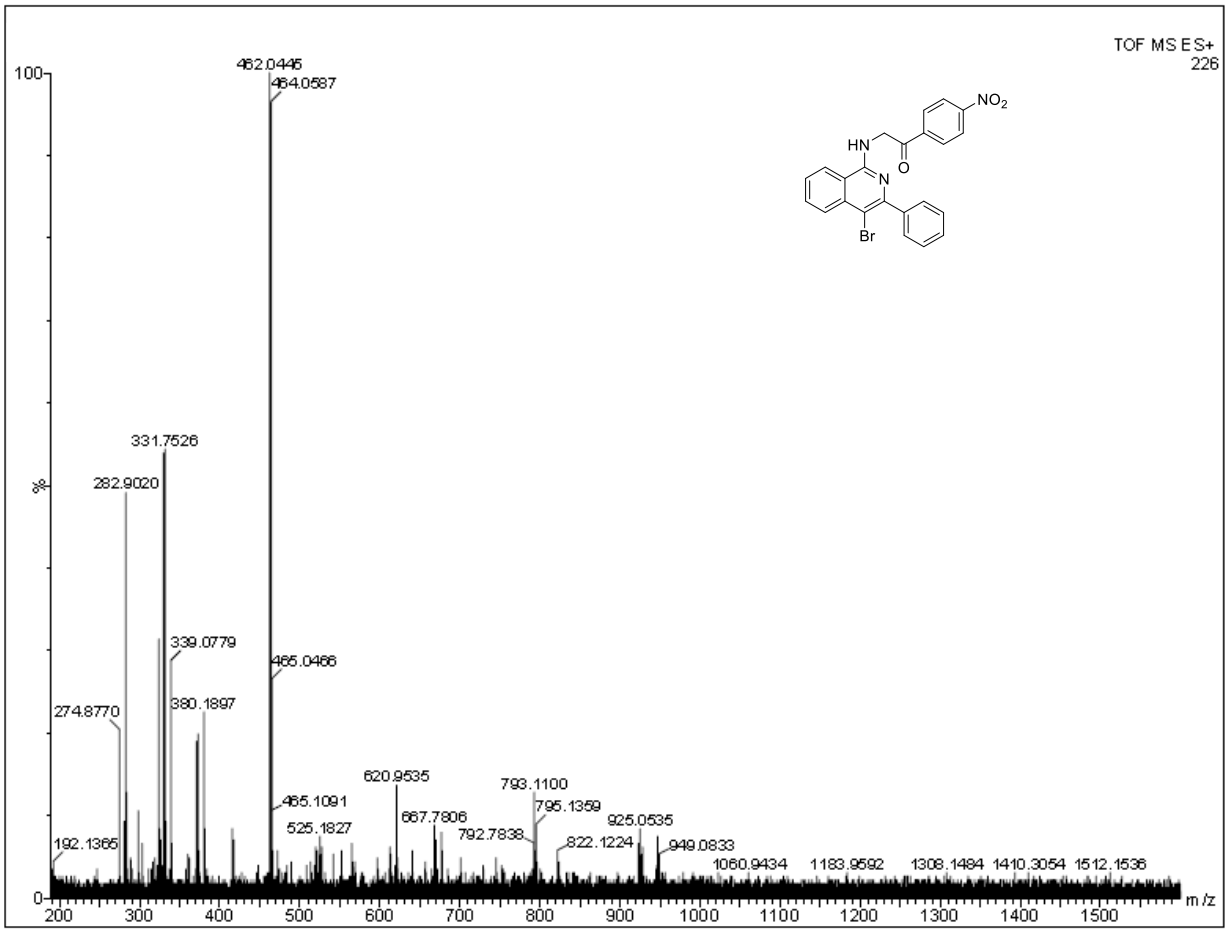
HRMS-ESI (m/z) of (**3k**)



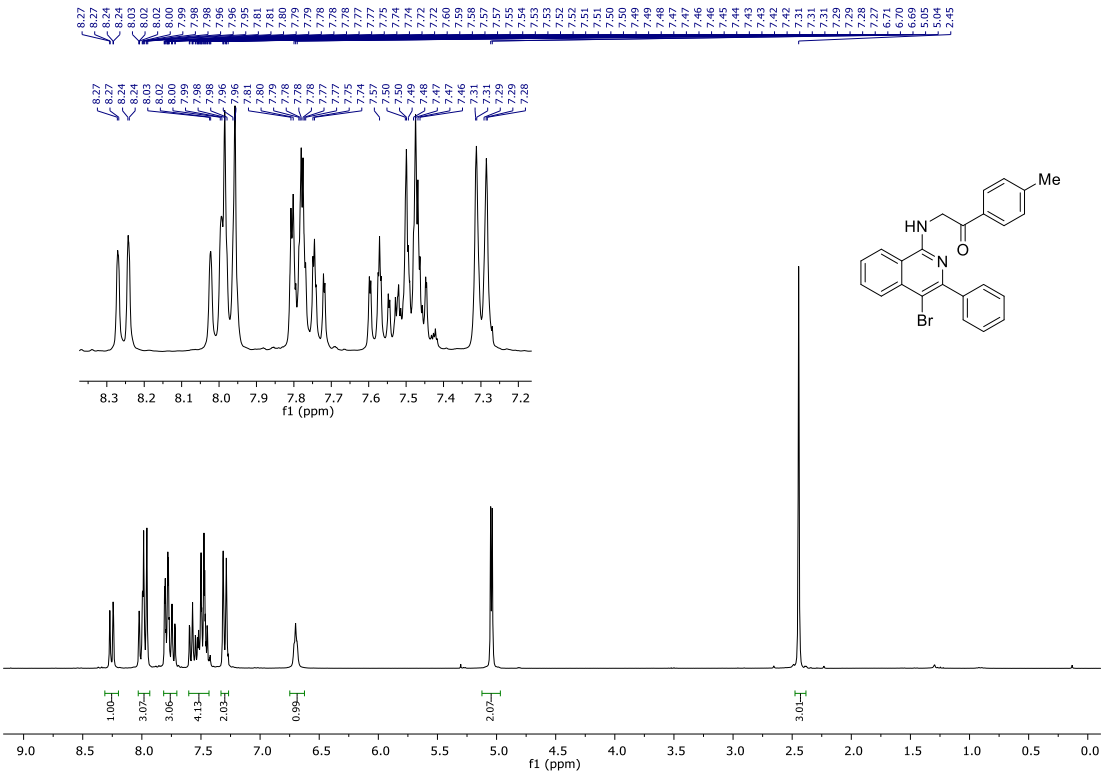
^{1}H-NMR of compound (**3l**) (300 MHz, $CDCl_3$)



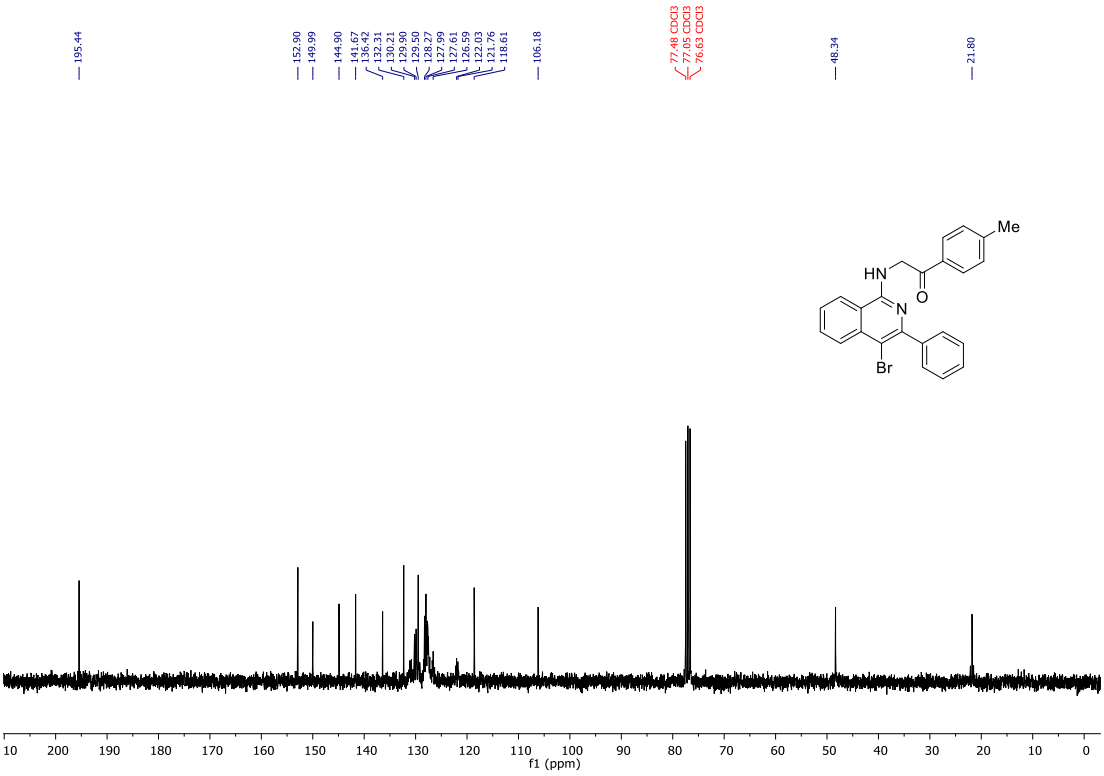
^{13}C-NMR of compound (**3l**) (75 MHz, $CDCl_3$)



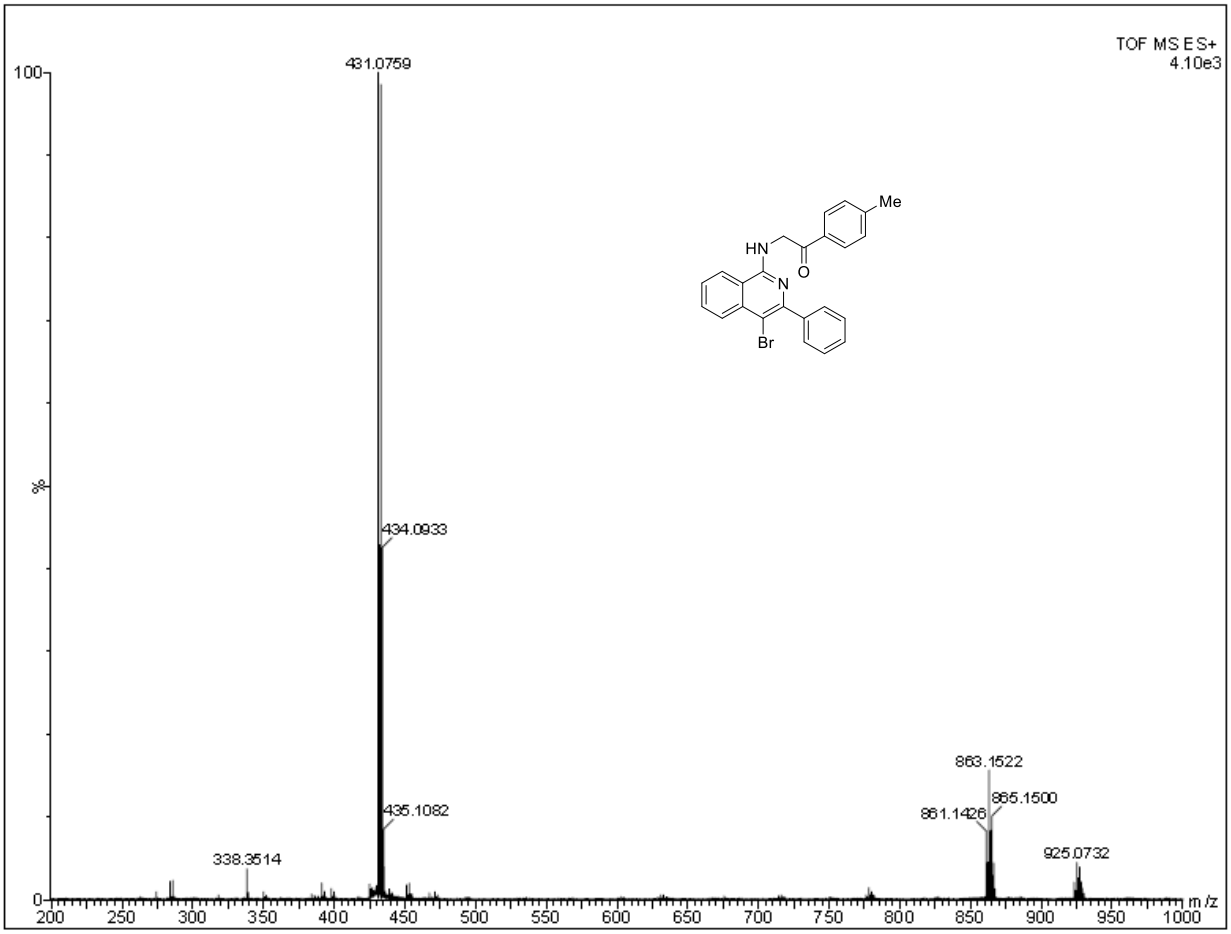
HRMS-ESI (m/z) of (**3l**)



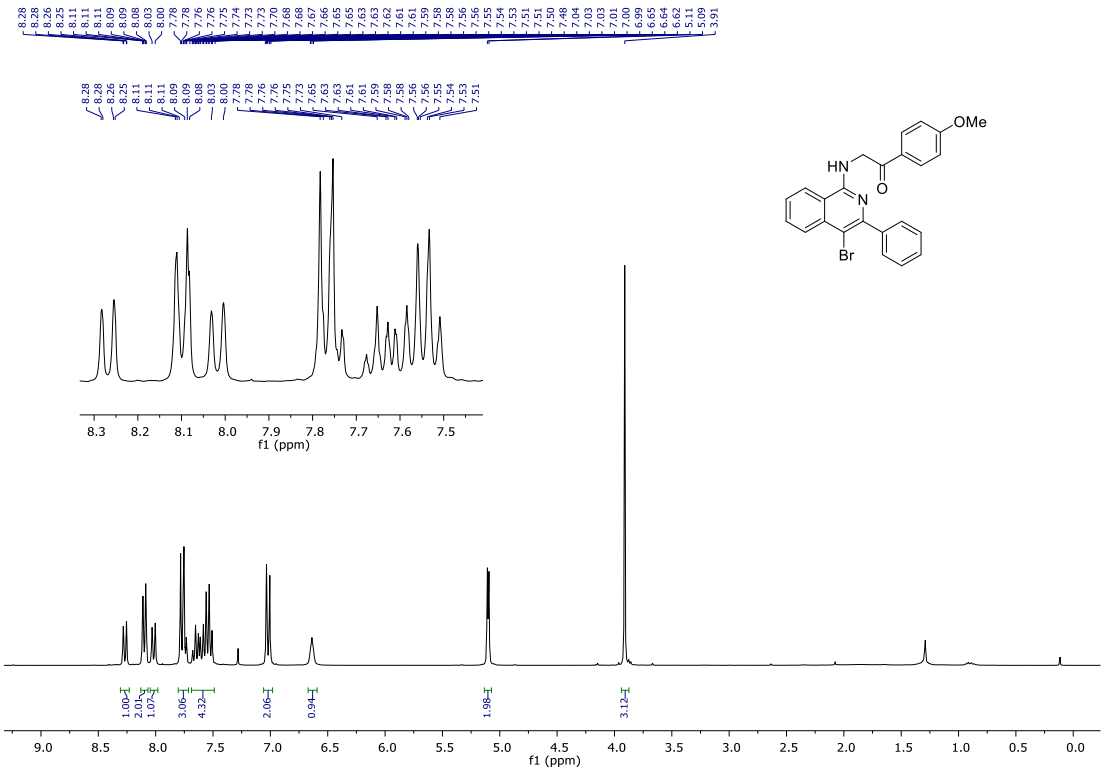
^{1}H-NMR of compound (**3m**) (300 MHz, $CDCl_3$)



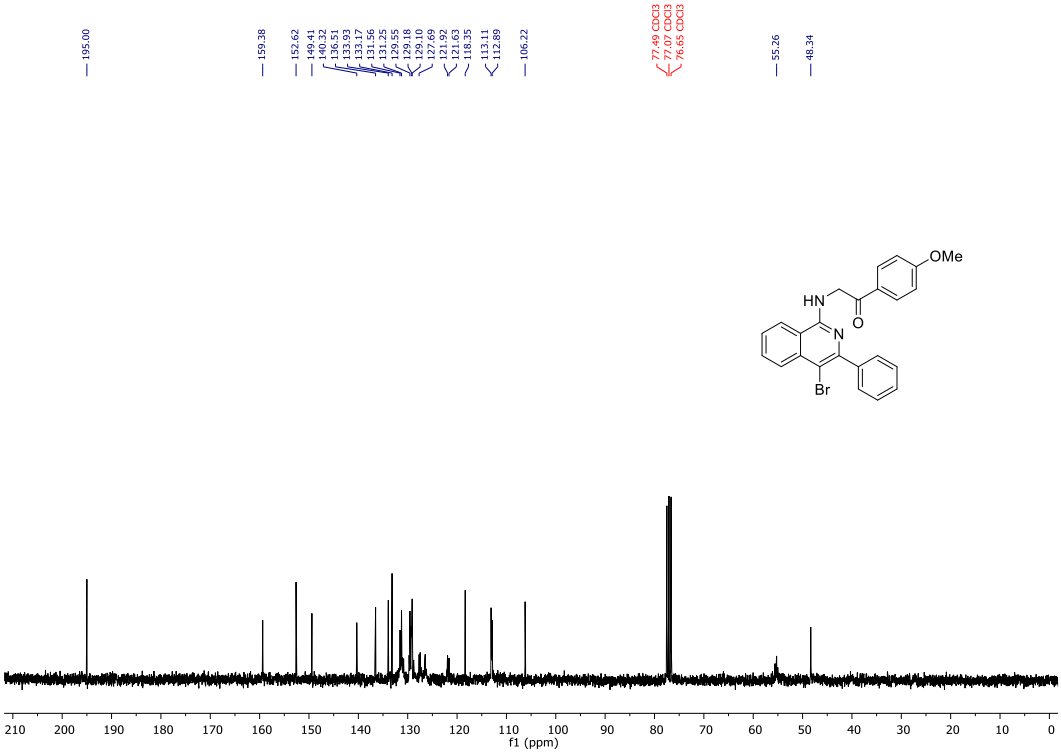
^{13}C-NMR of compound (**3m**) (75 MHz, $CDCl_3$)



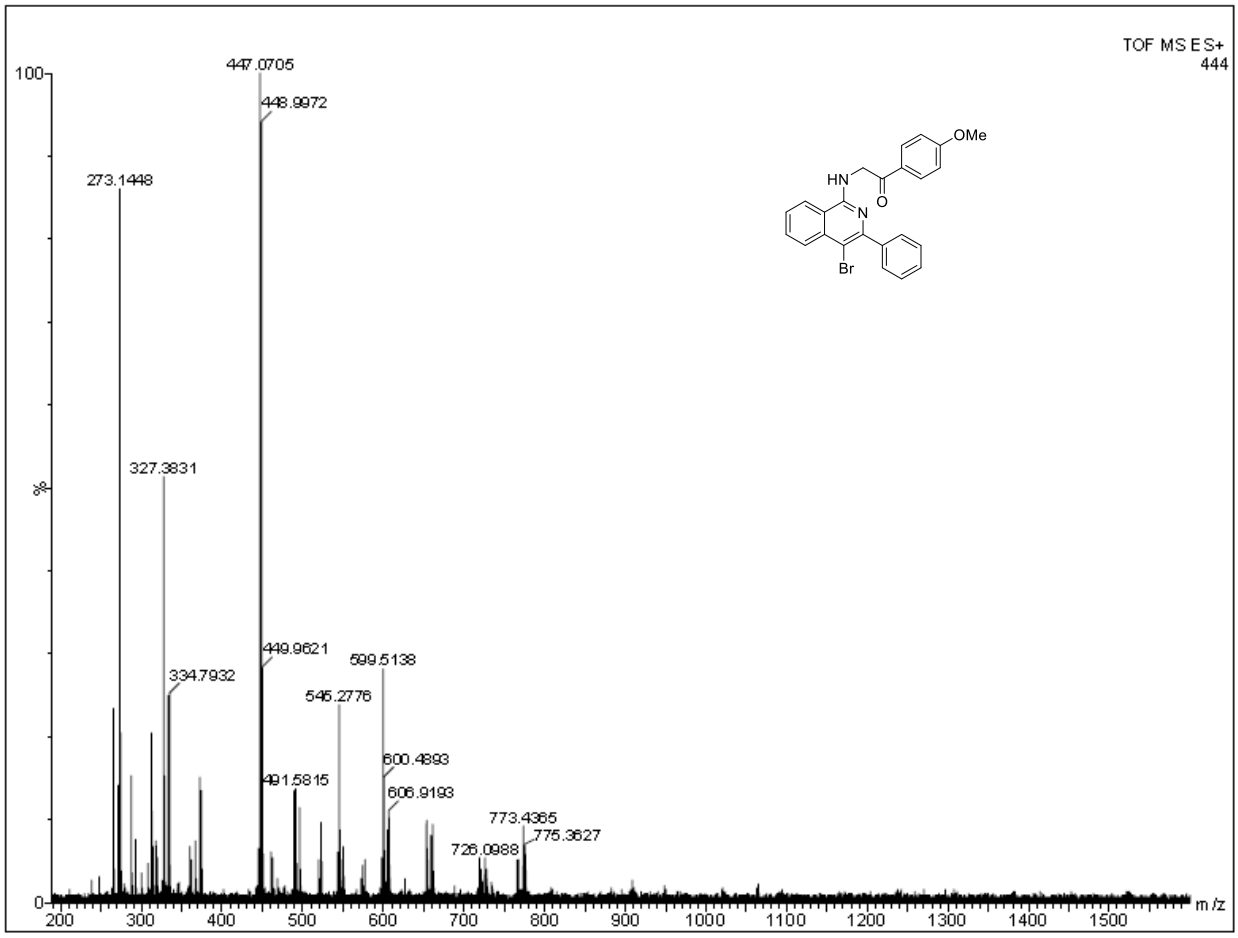
HRMS-ESI (m/z) of (**3m**)



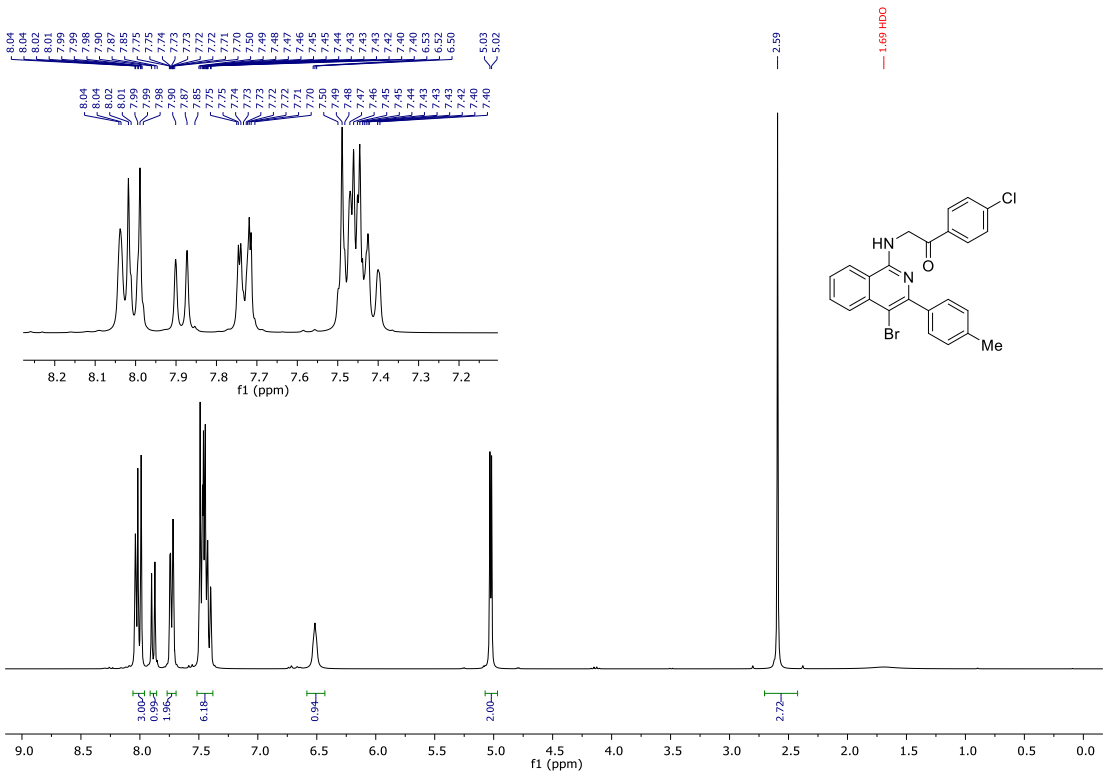
^{1}H-NMR of compound (**3n**) (300 MHz, $CDCl_3$)



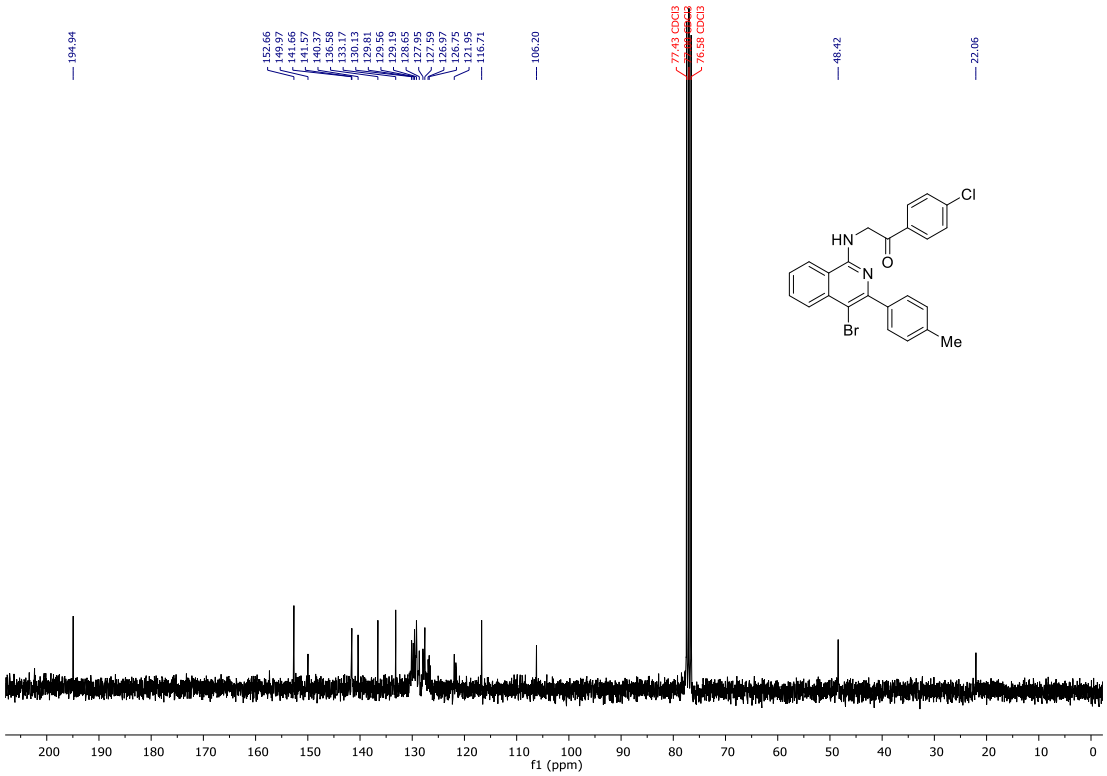
^{13}C-NMR of compound (**3n**) (75 MHz, $CDCl_3$)



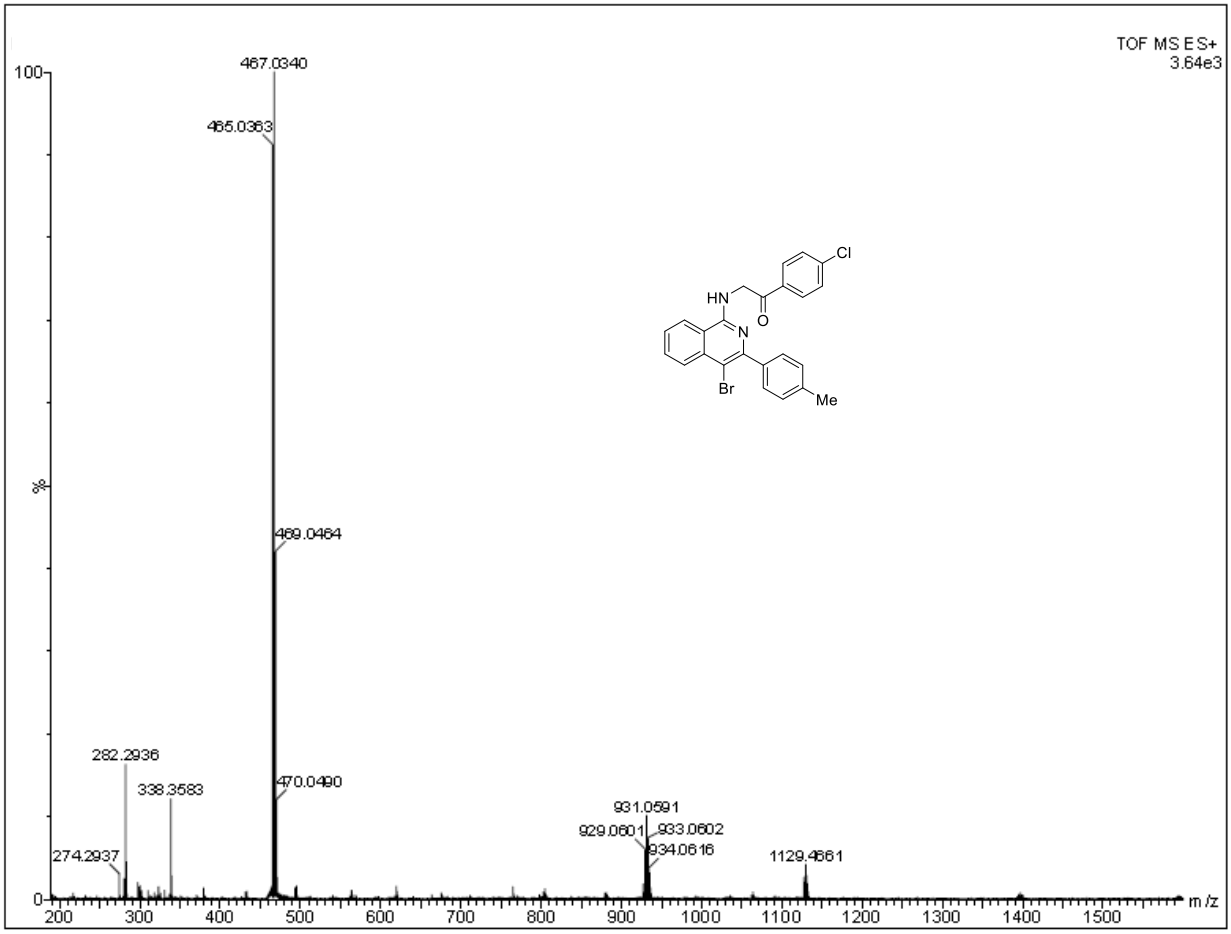
HRMS-ESI (m/z) of (**3n**)



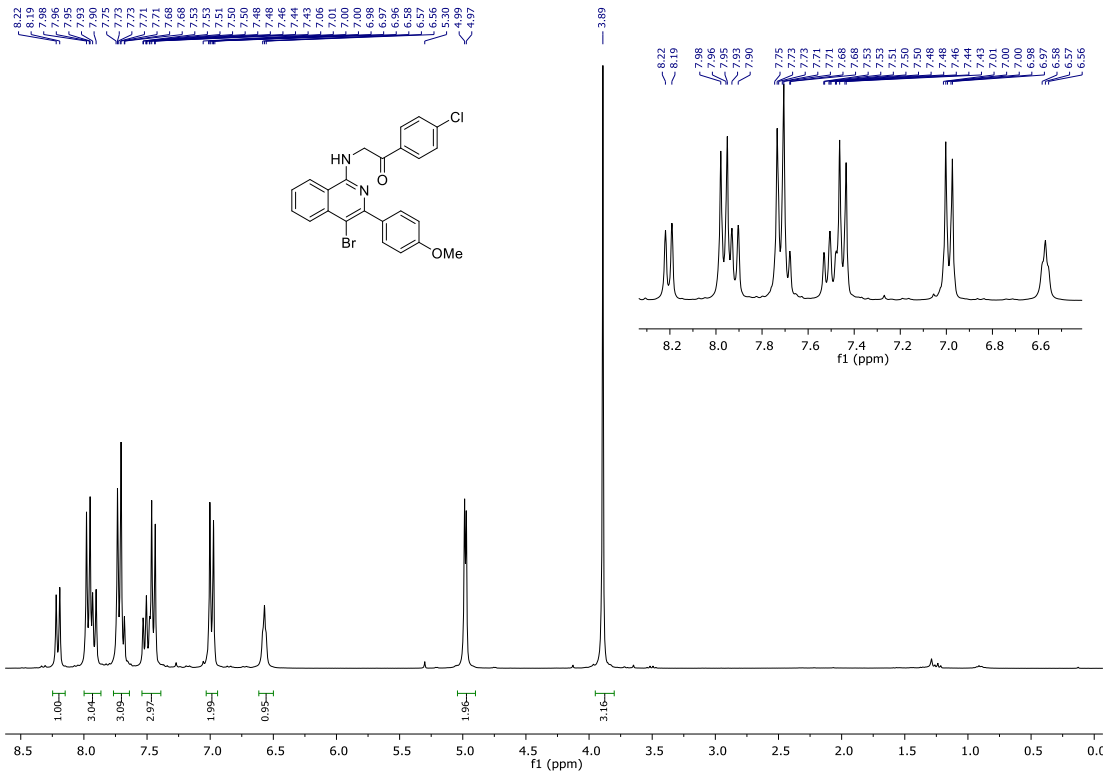
^{1}H-NMR of compound (**3o**) (300 MHz, $CDCl_3$)



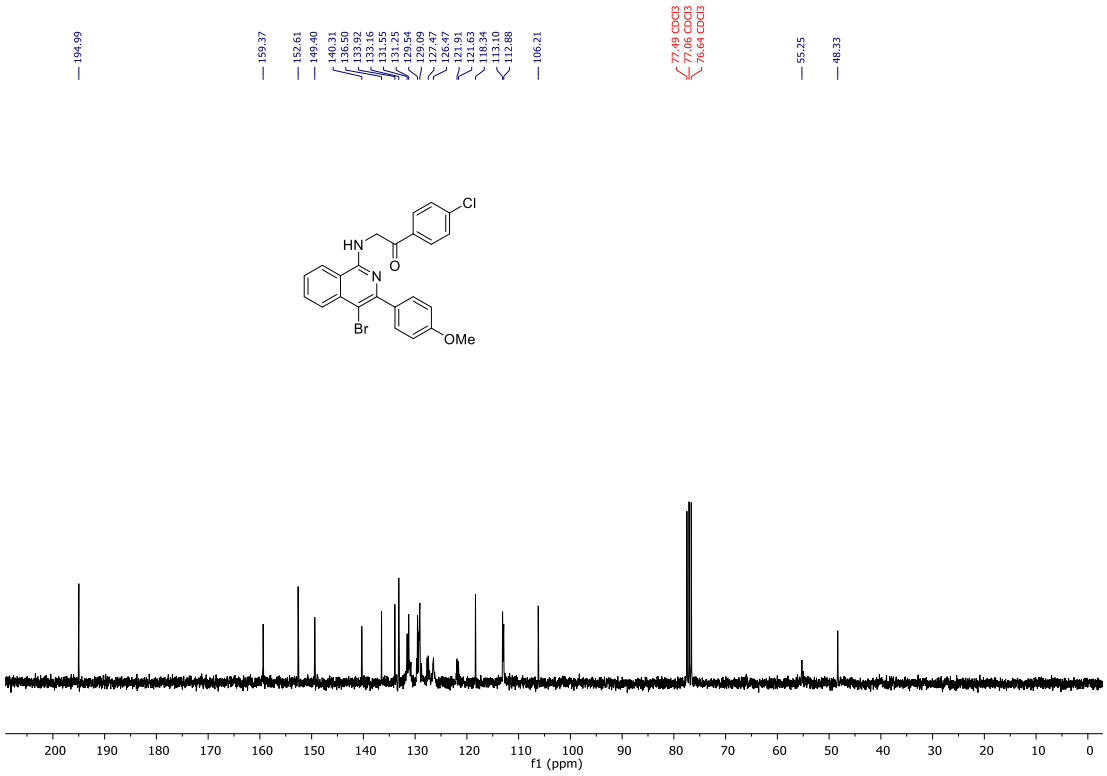
^{13}C-NMR of compound (**3o**) (75 MHz, $CDCl_3$)



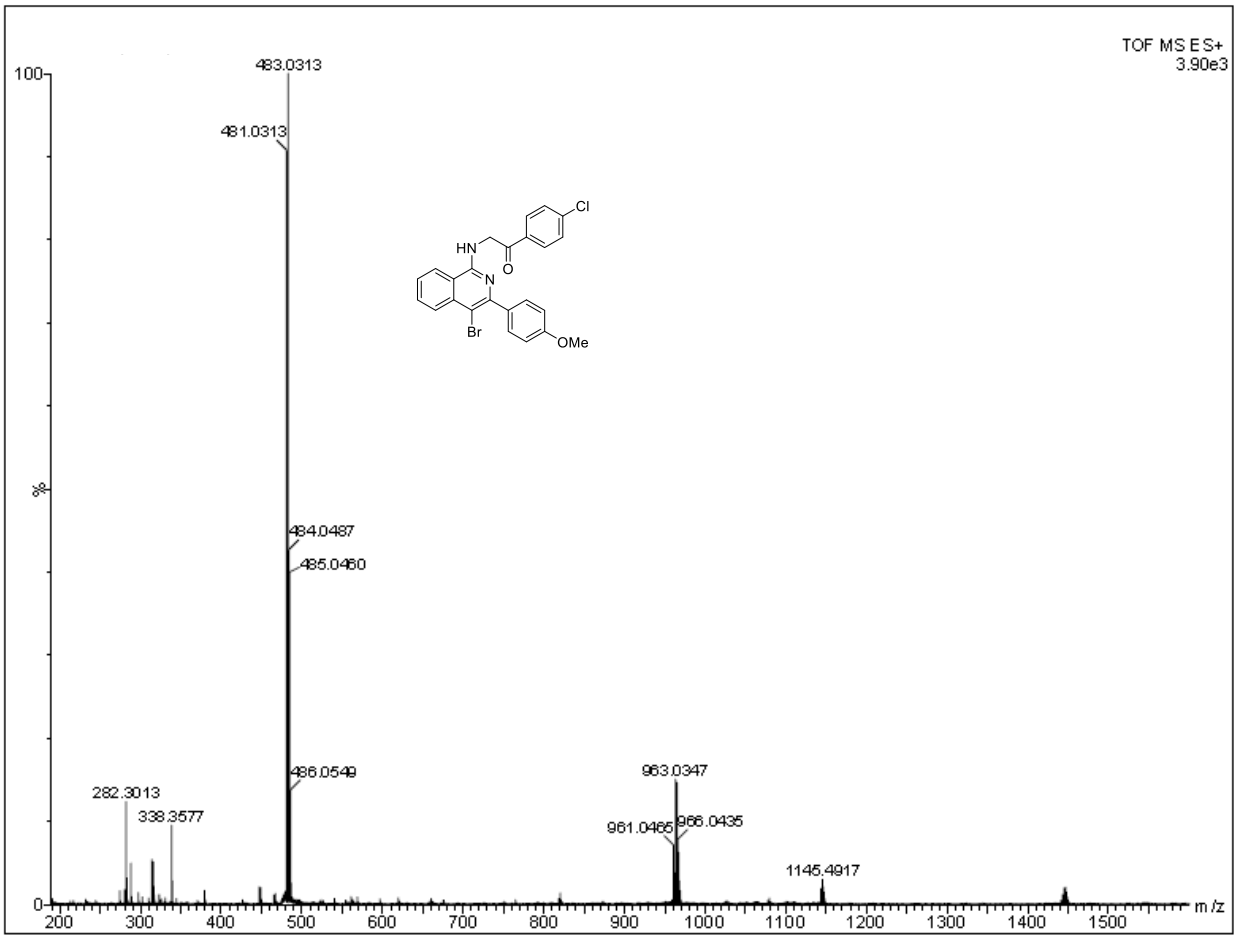
HRMS-ESI (m/z) of (**3o**)



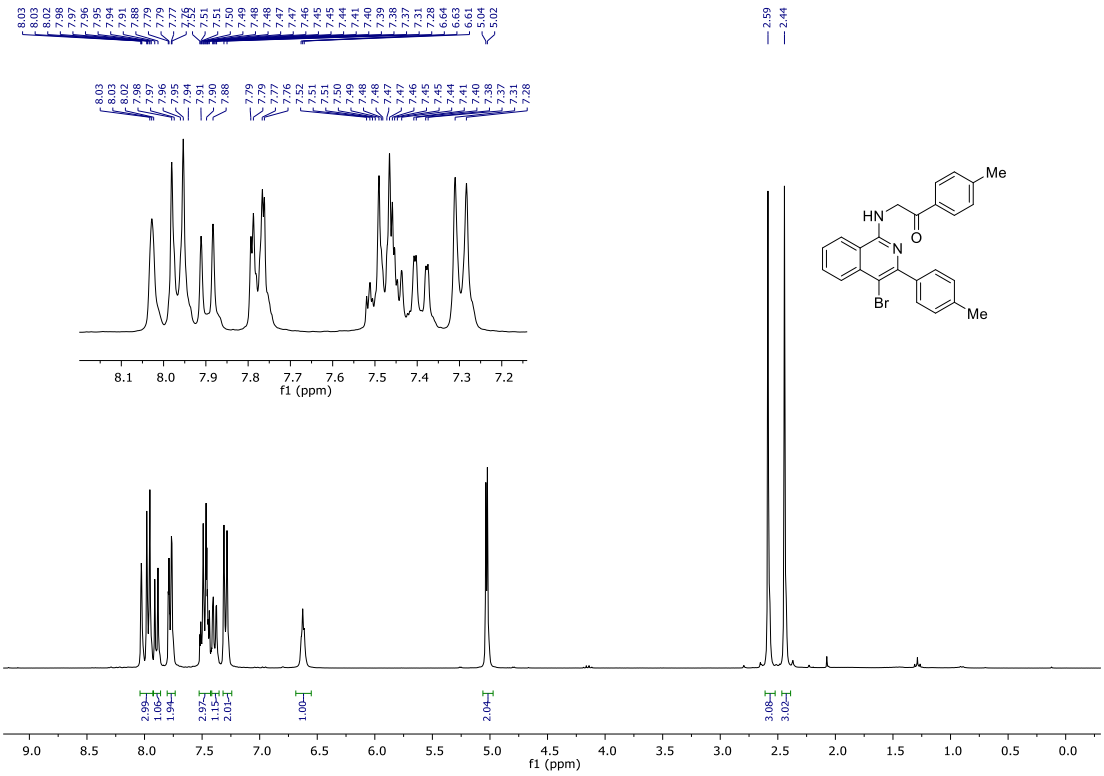
^{1}H-NMR of compound (**3p**) (300 MHz, $CDCl_3$)



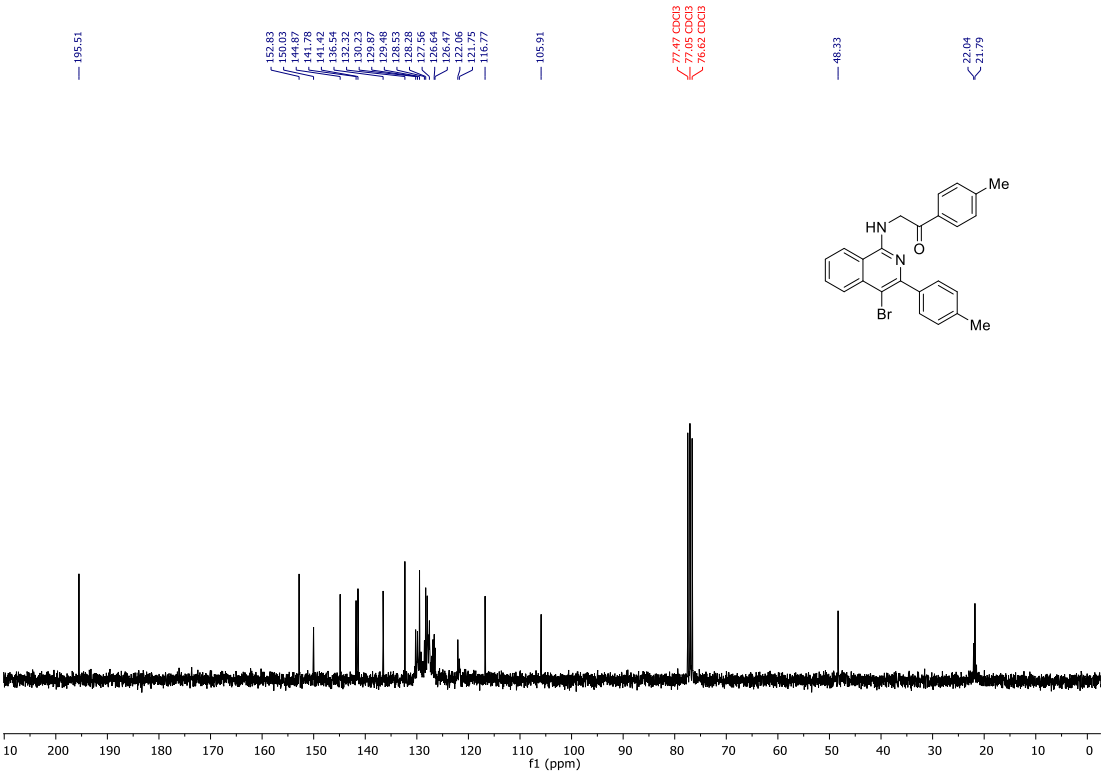
^{13}C-NMR of compound (**3p**) (75 MHz, $CDCl_3$)



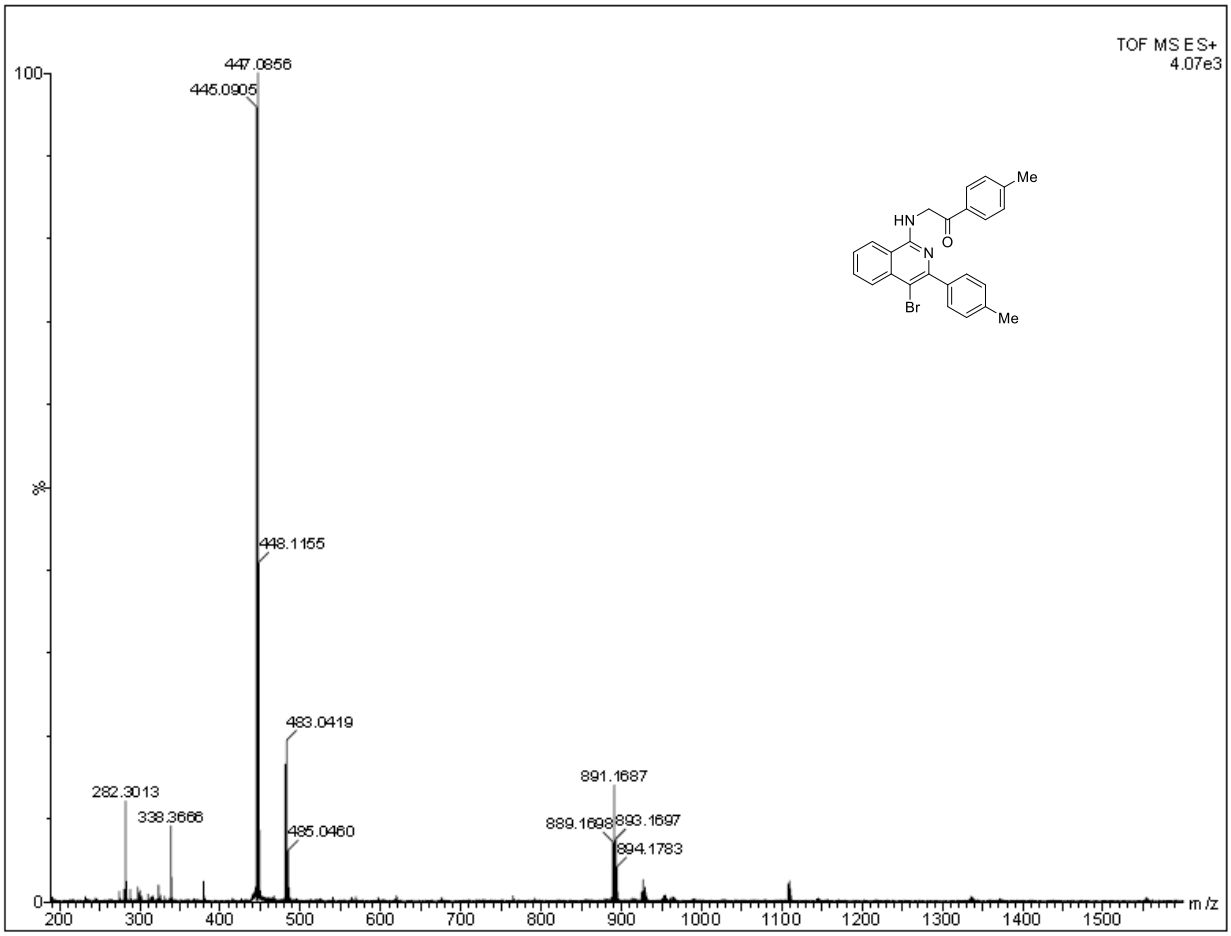
HRMS-ESI (m/z) of (**3p**)



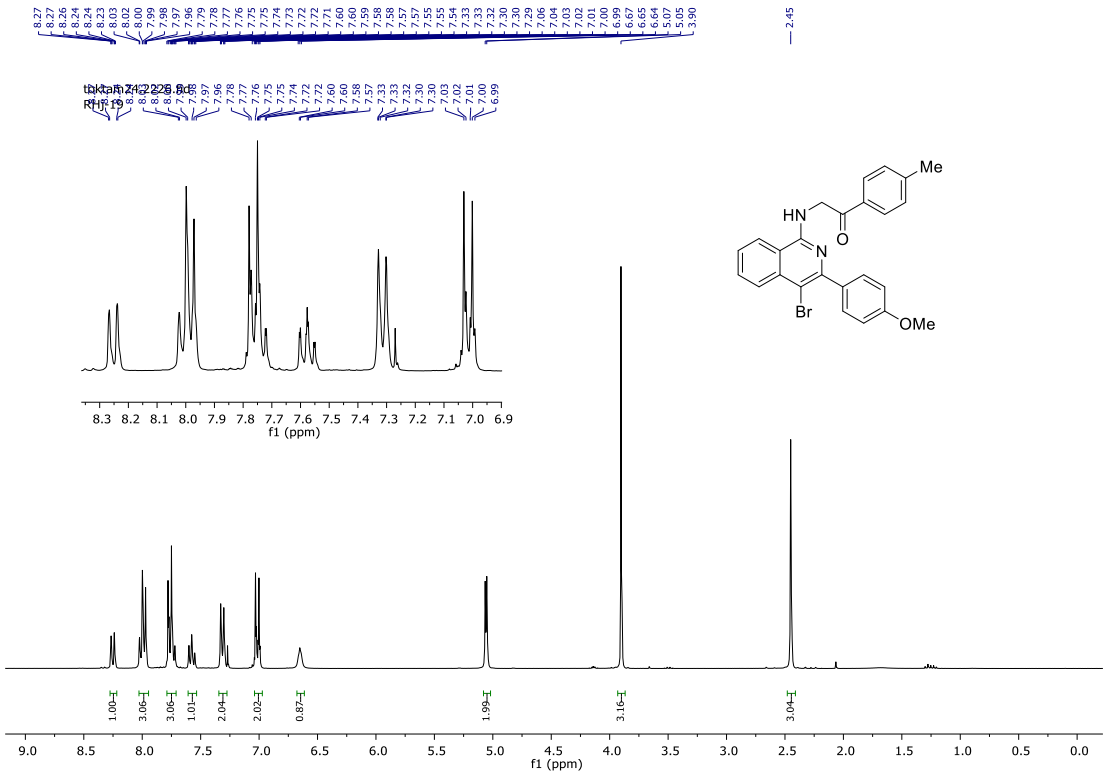
^{1}H-NMR of compound (**3q**) (300 MHz, $CDCl_3$)



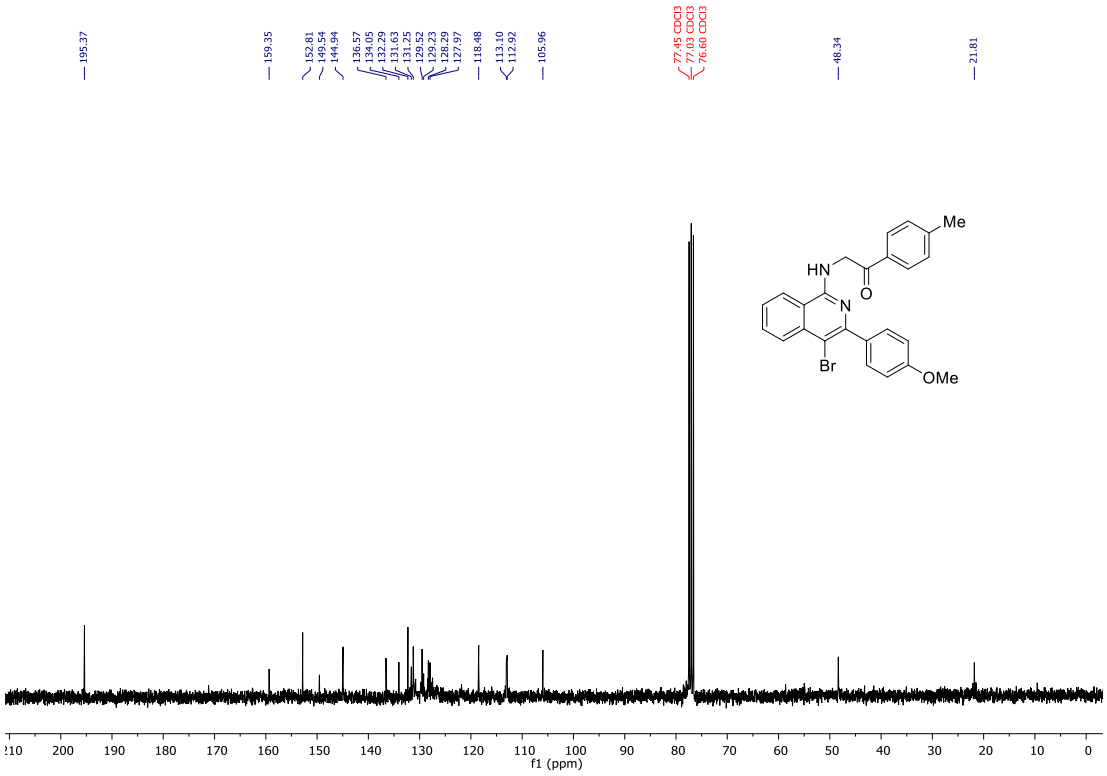
^{13}C-NMR of compound (**3q**) (75 MHz, $CDCl_3$)



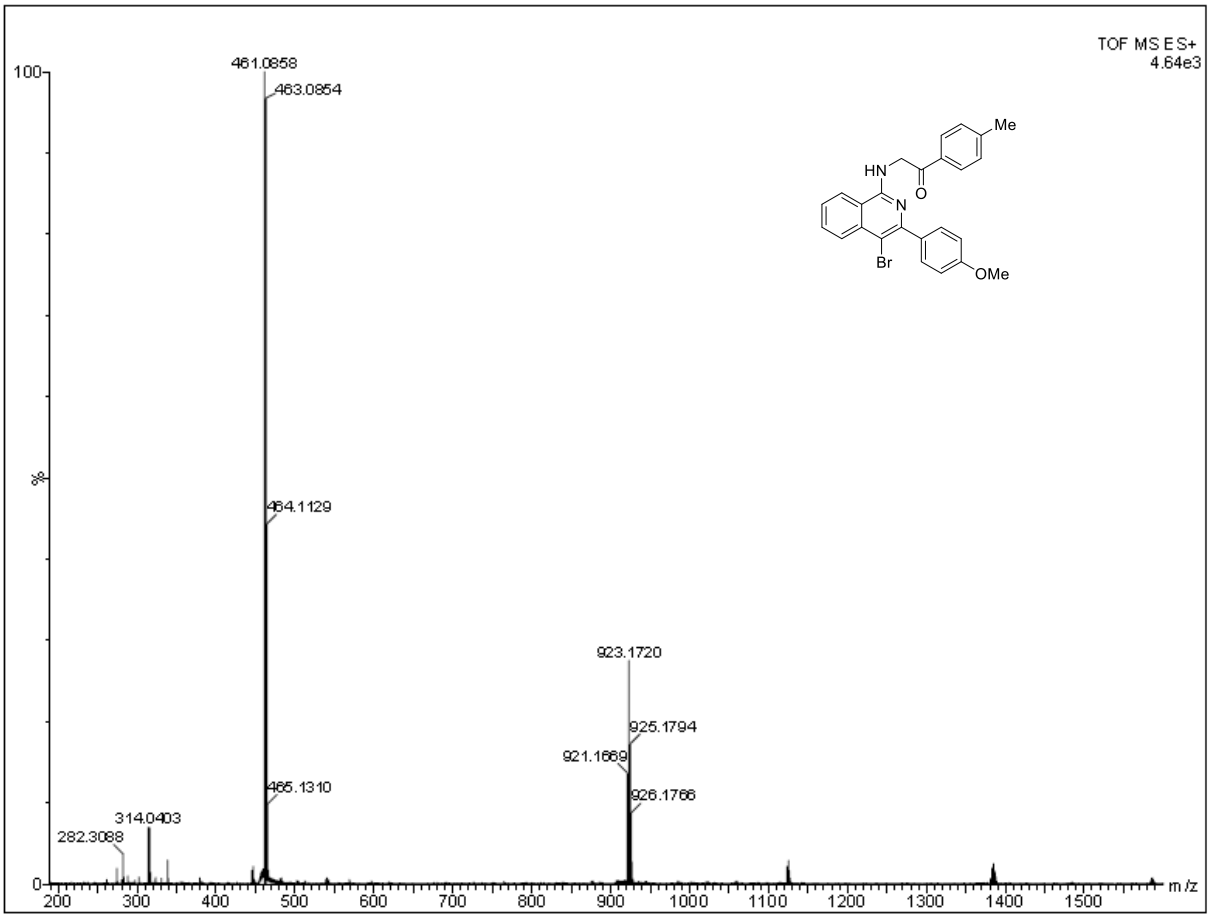
HRMS-ESI (m/z) of (**3q**)



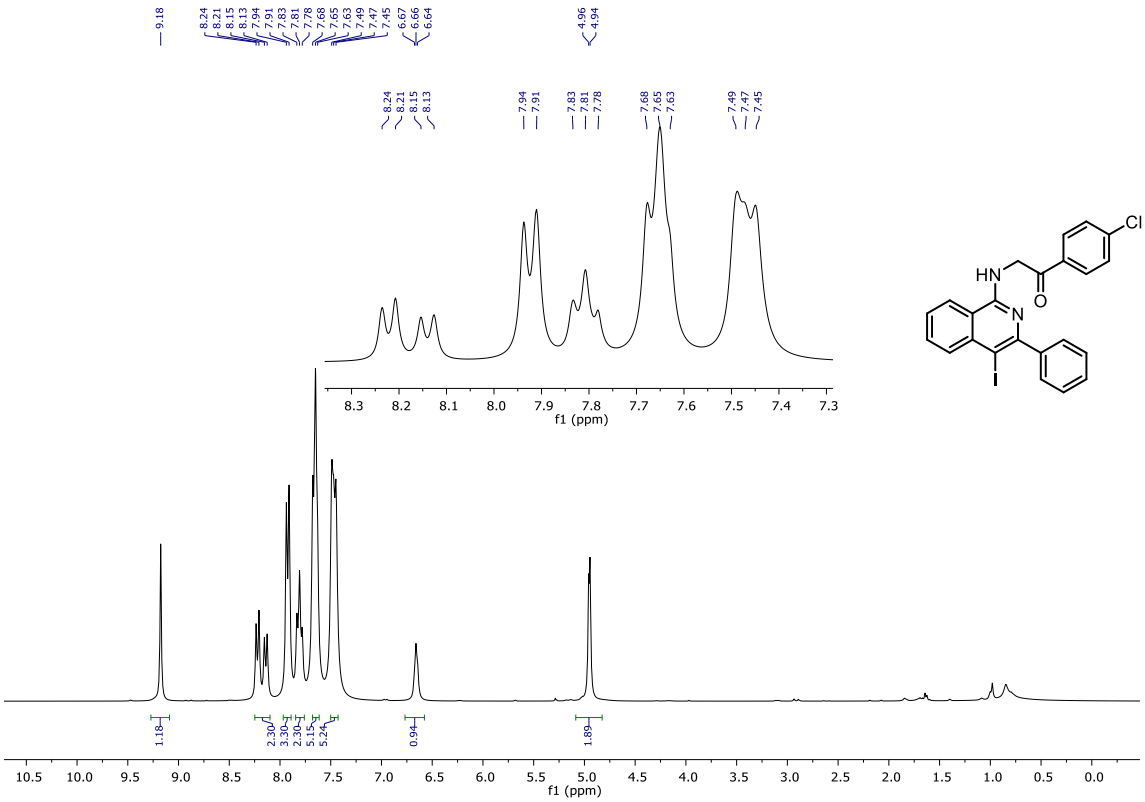
^{1}H-NMR of compound (**3r**) (300 MHz, $CDCl_3$)



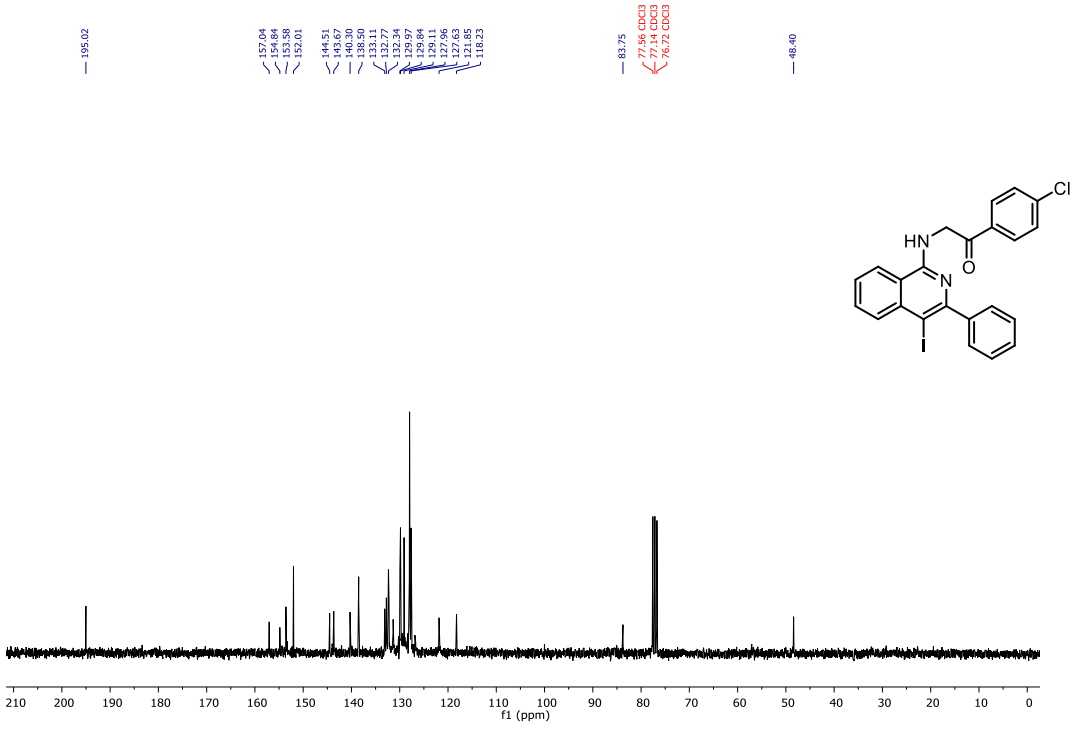
^{13}C-NMR of compound (**3r**) (75 MHz, $CDCl_3$)



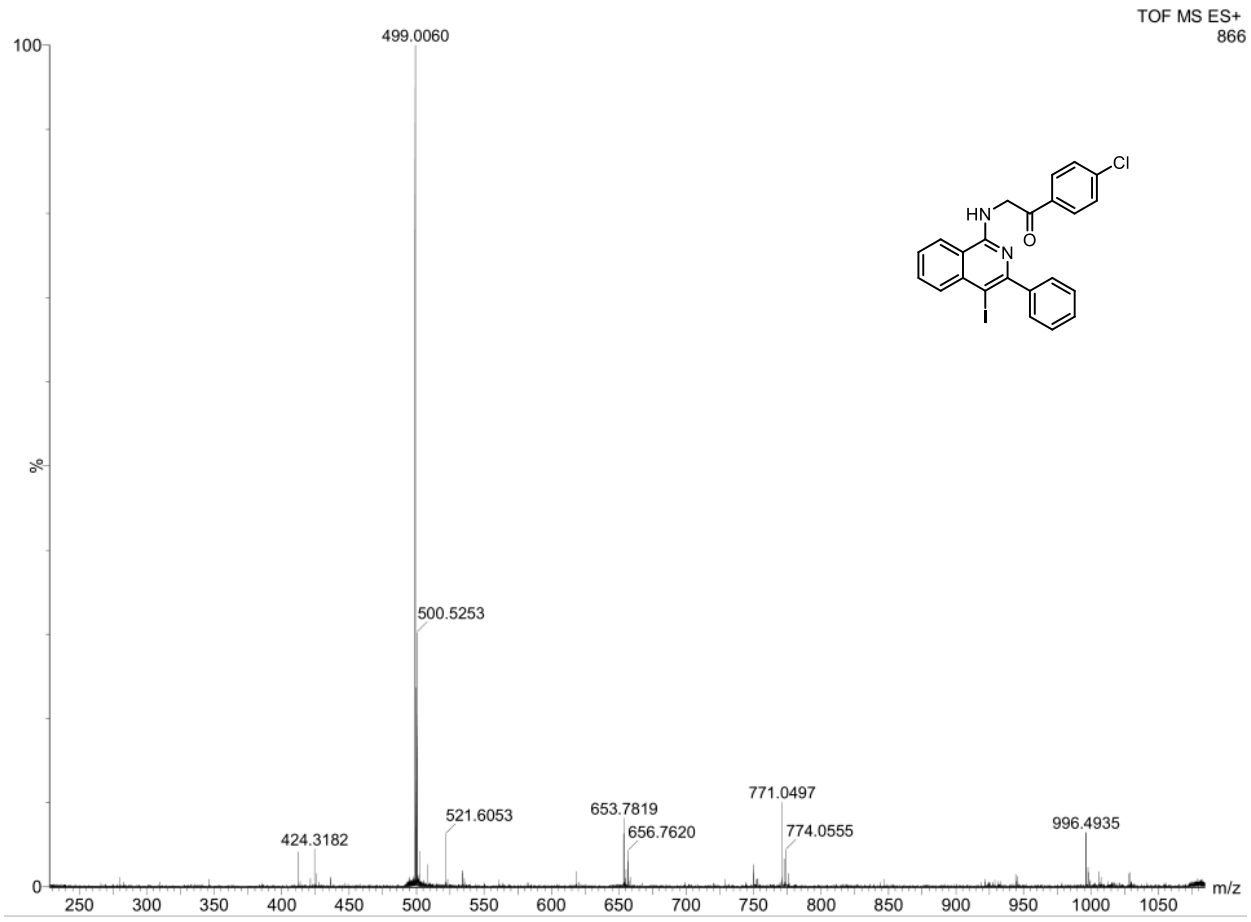
HRMS-ESI (m/z) of (**3r**)



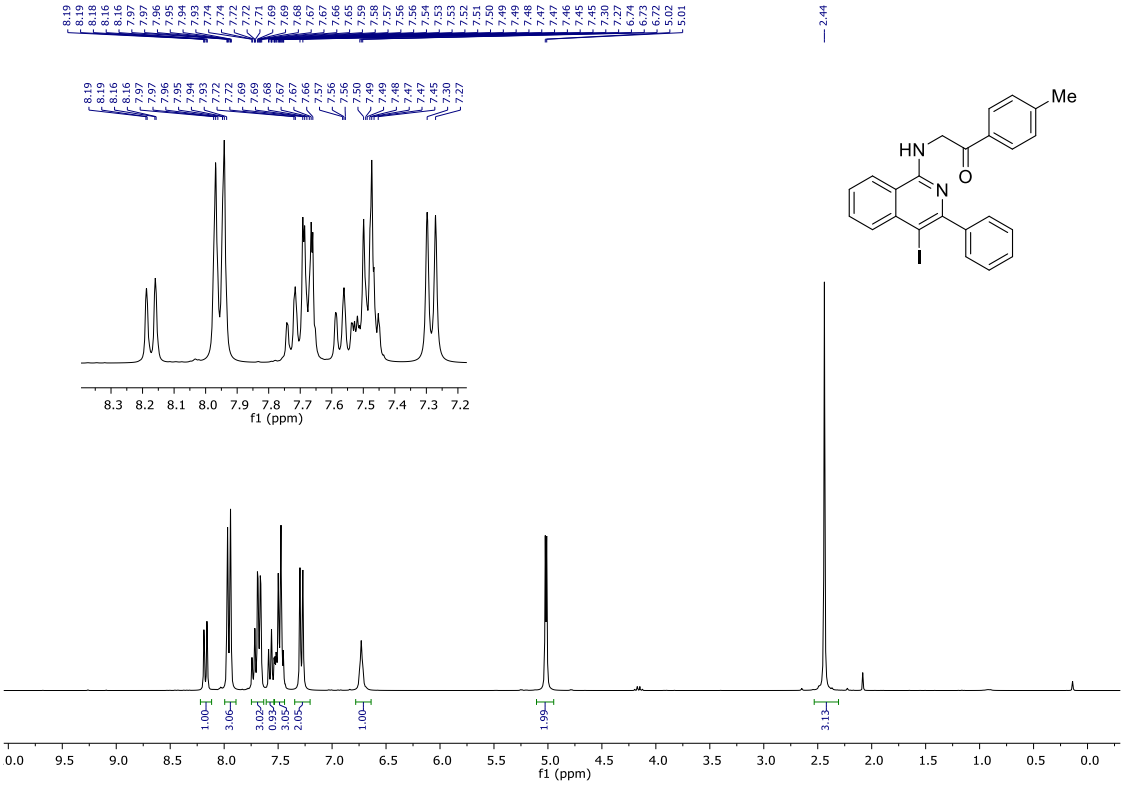
^{1}H-NMR of compound (**4a**) (300 MHz, $CDCl_3$)



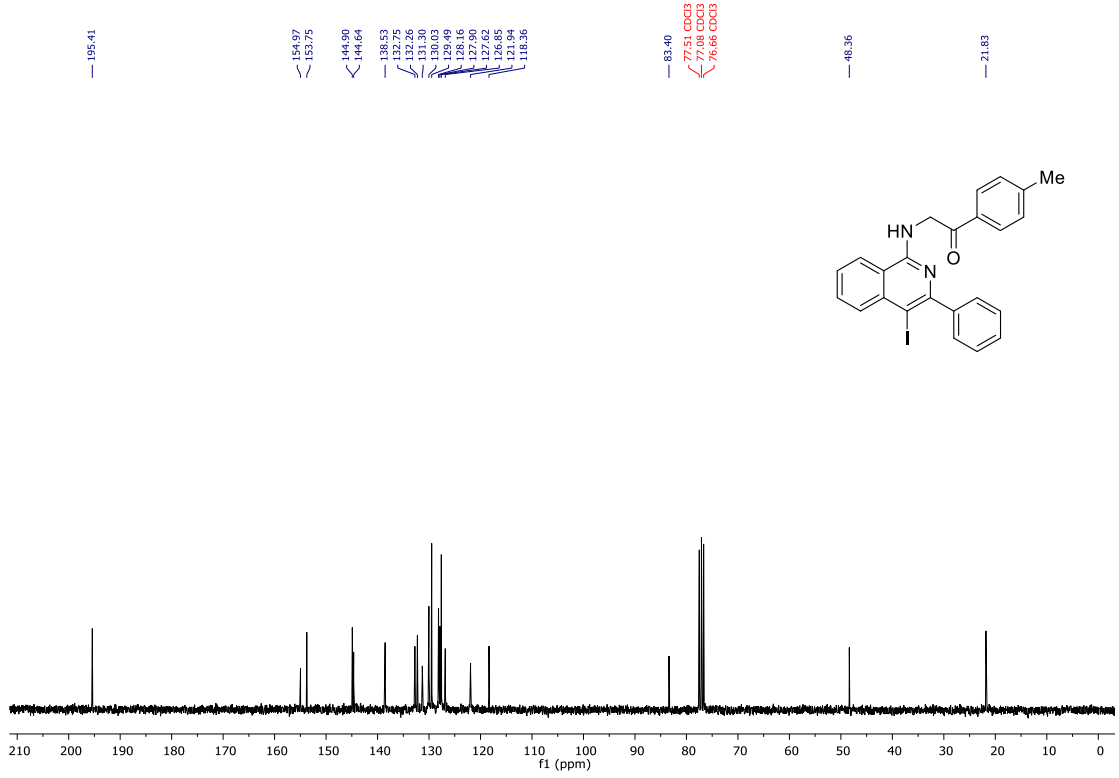
^{13}C-NMR of compound (**4a**) (75 MHz, $CDCl_3$)



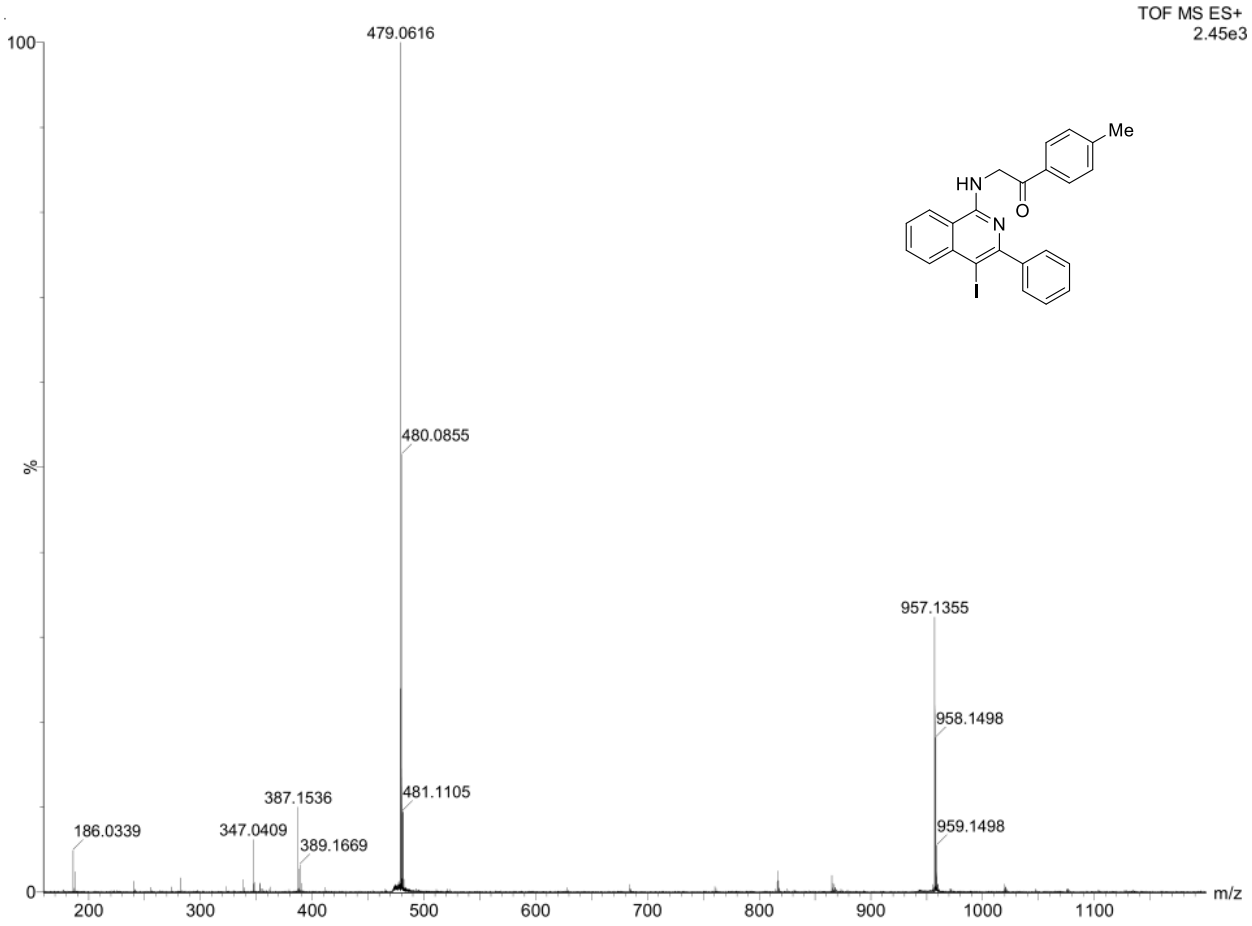
HRMS-ESI (m/z) of (**4a**)



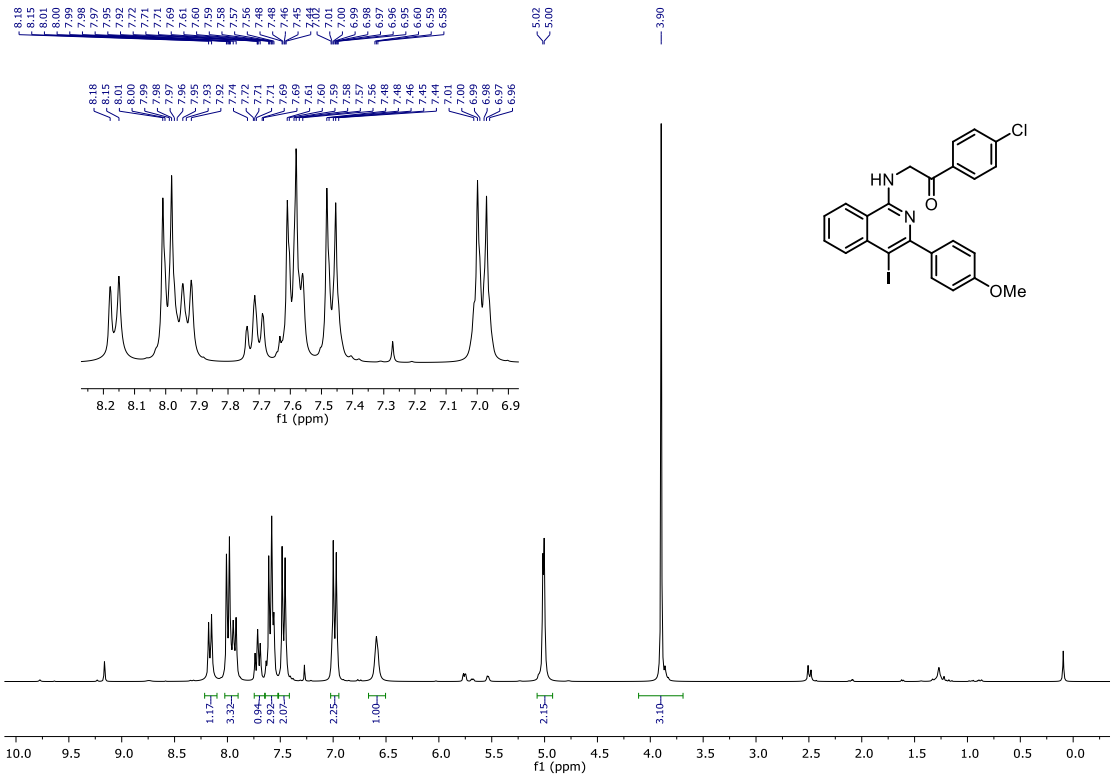
^{1}H-NMR of compound (**4b**) (300 MHz, $CDCl_3$)



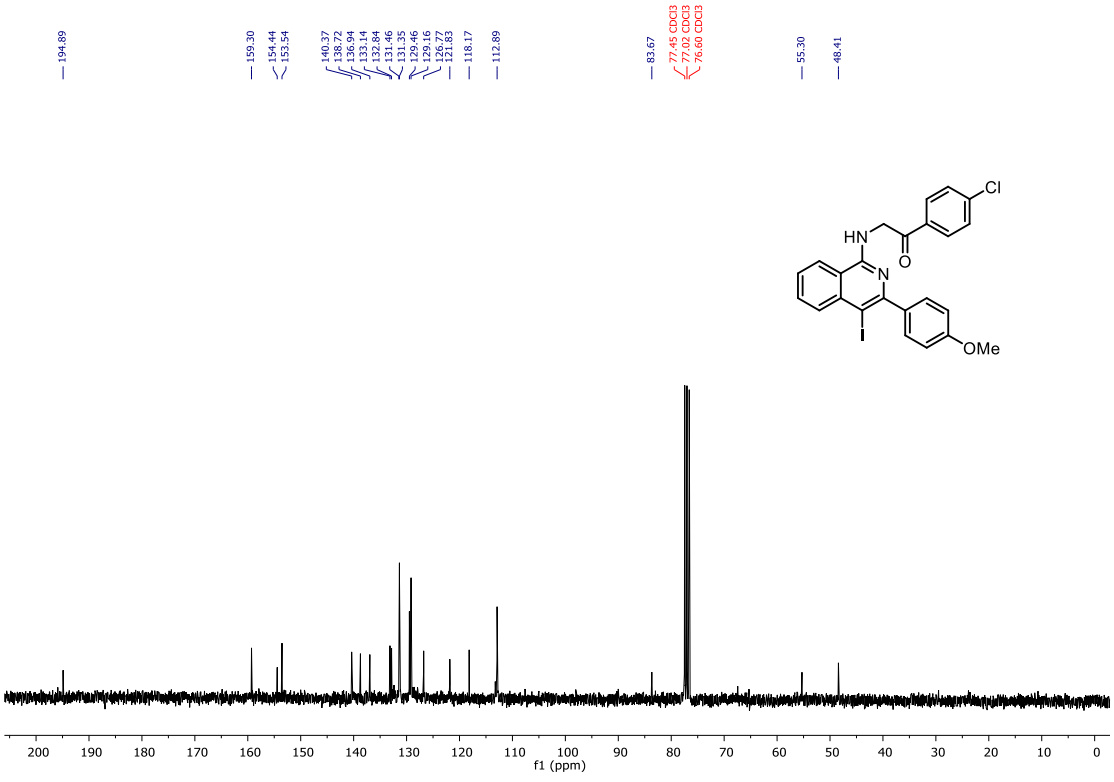
^{13}C-NMR of compound (**4b**) (75 MHz, $CDCl_3$)



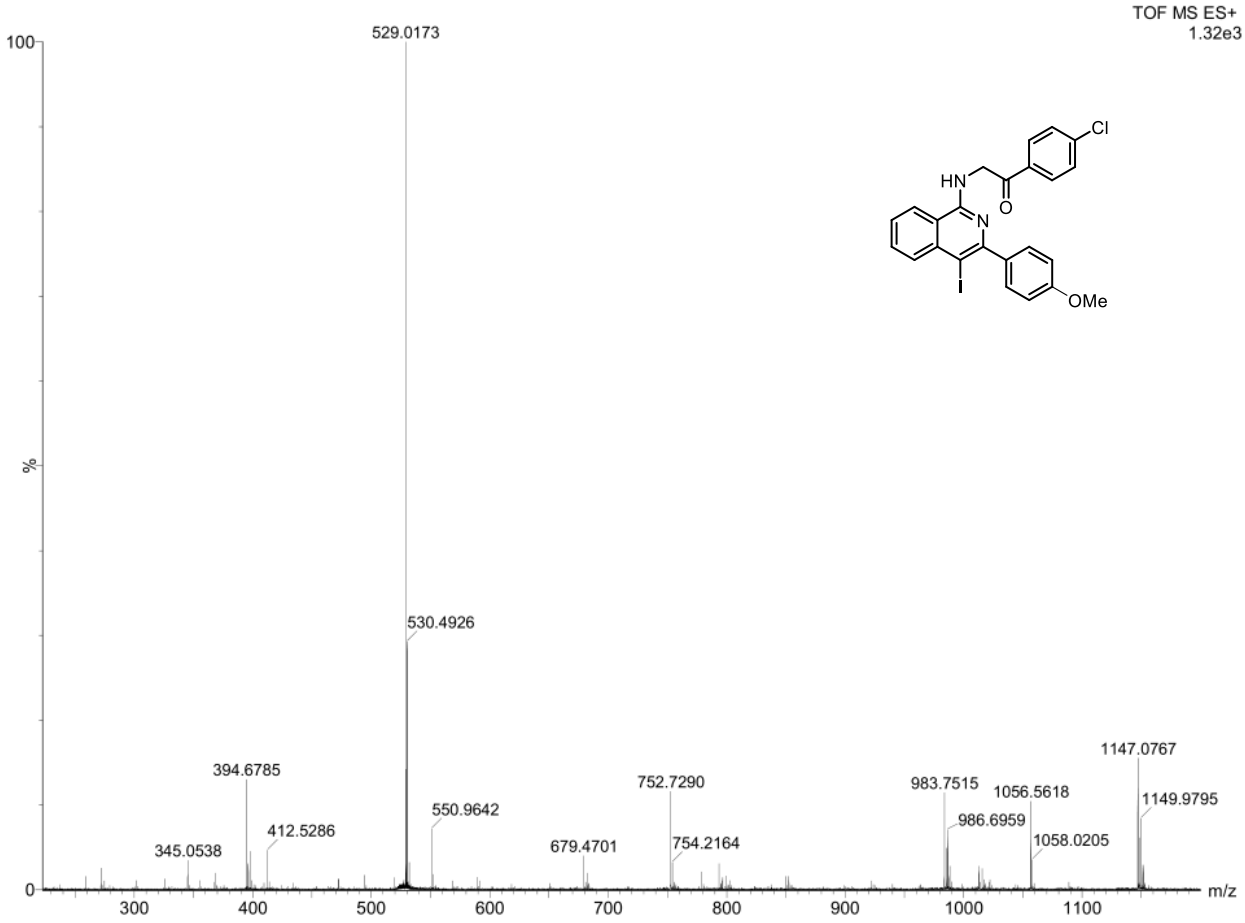
HRMS-ESI (m/z) of (**4b**)



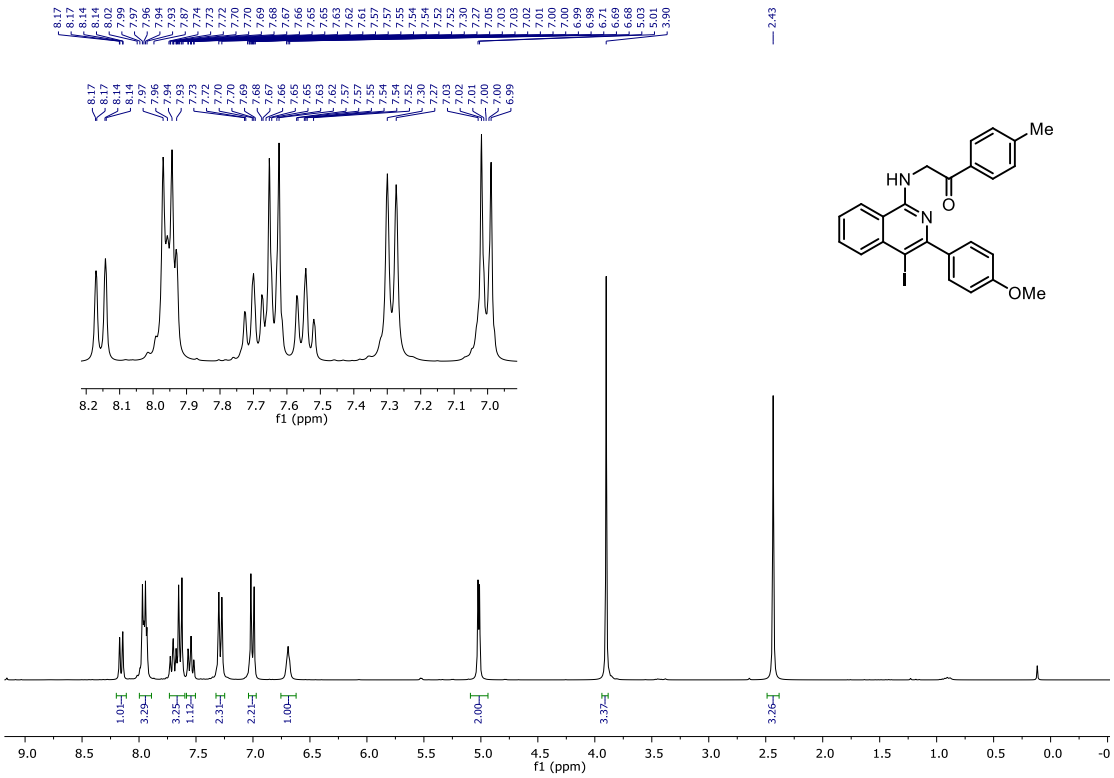
^{1}H-NMR of compound (**4c**) (300 MHz, $CDCl_3$)



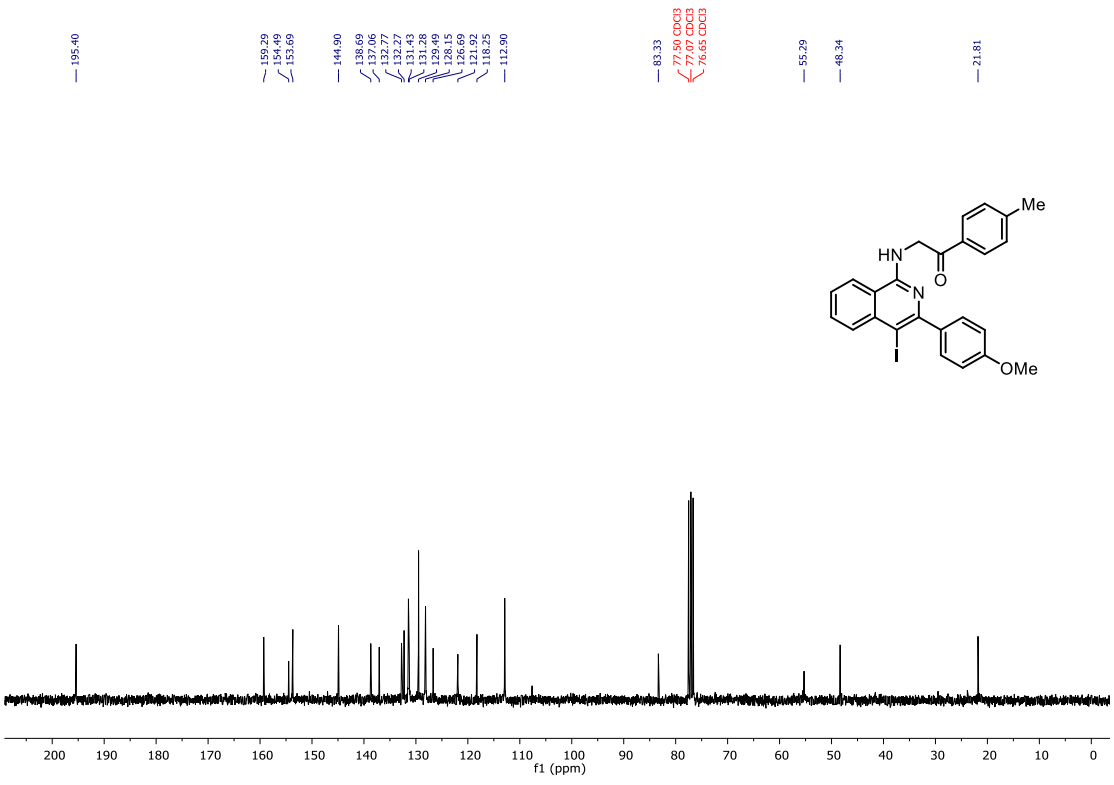
^{13}C-NMR of compound (**4c**) (75 MHz, $CDCl_3$)



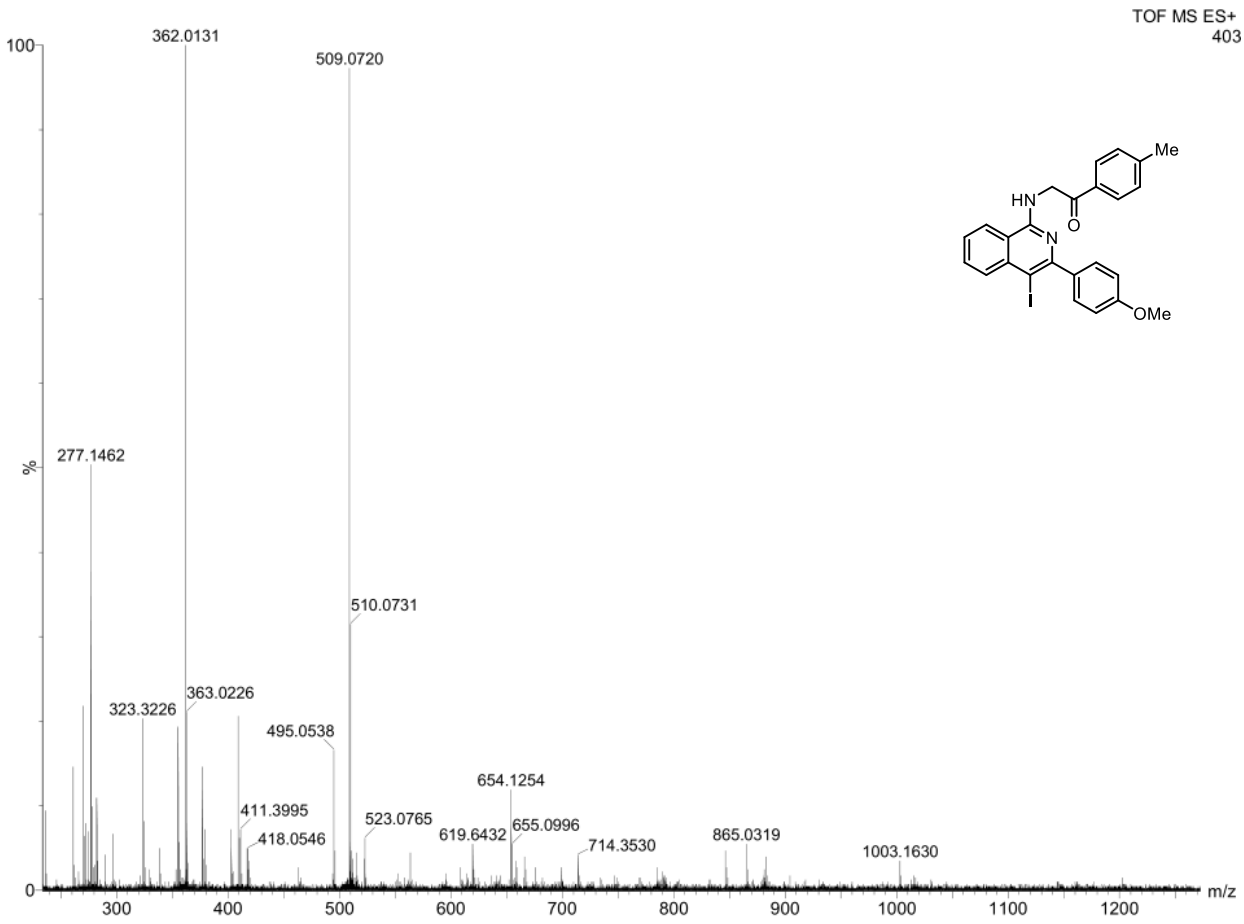
HRMS-ESI (m/z) of (**4c**)



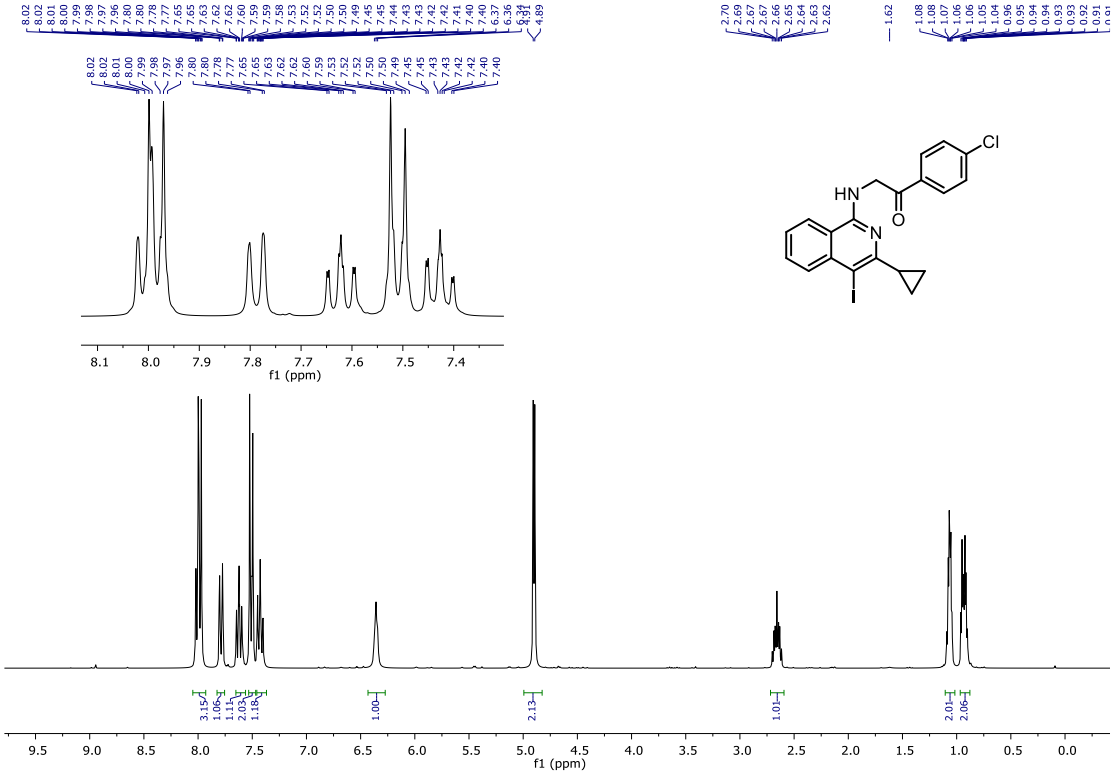
^{1}H-NMR of compound (**4d**) (300 MHz, $CDCl_3$)



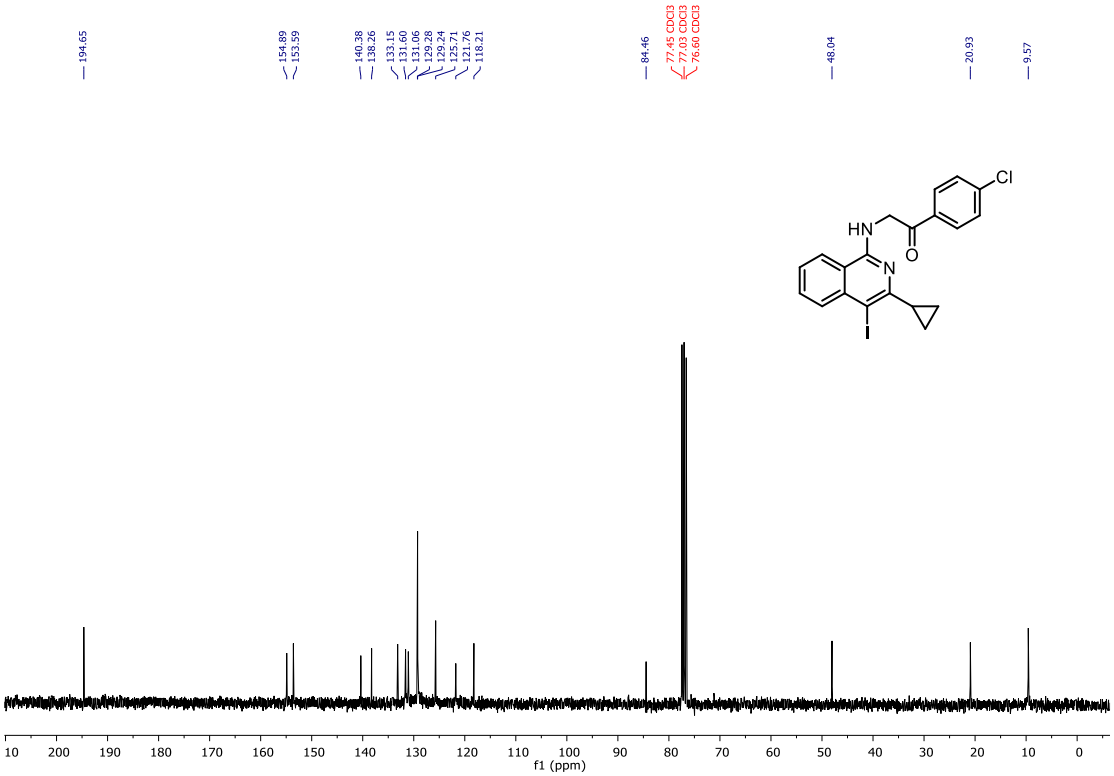
^{13}C-NMR of compound (**4d**) (75 MHz, $CDCl_3$)



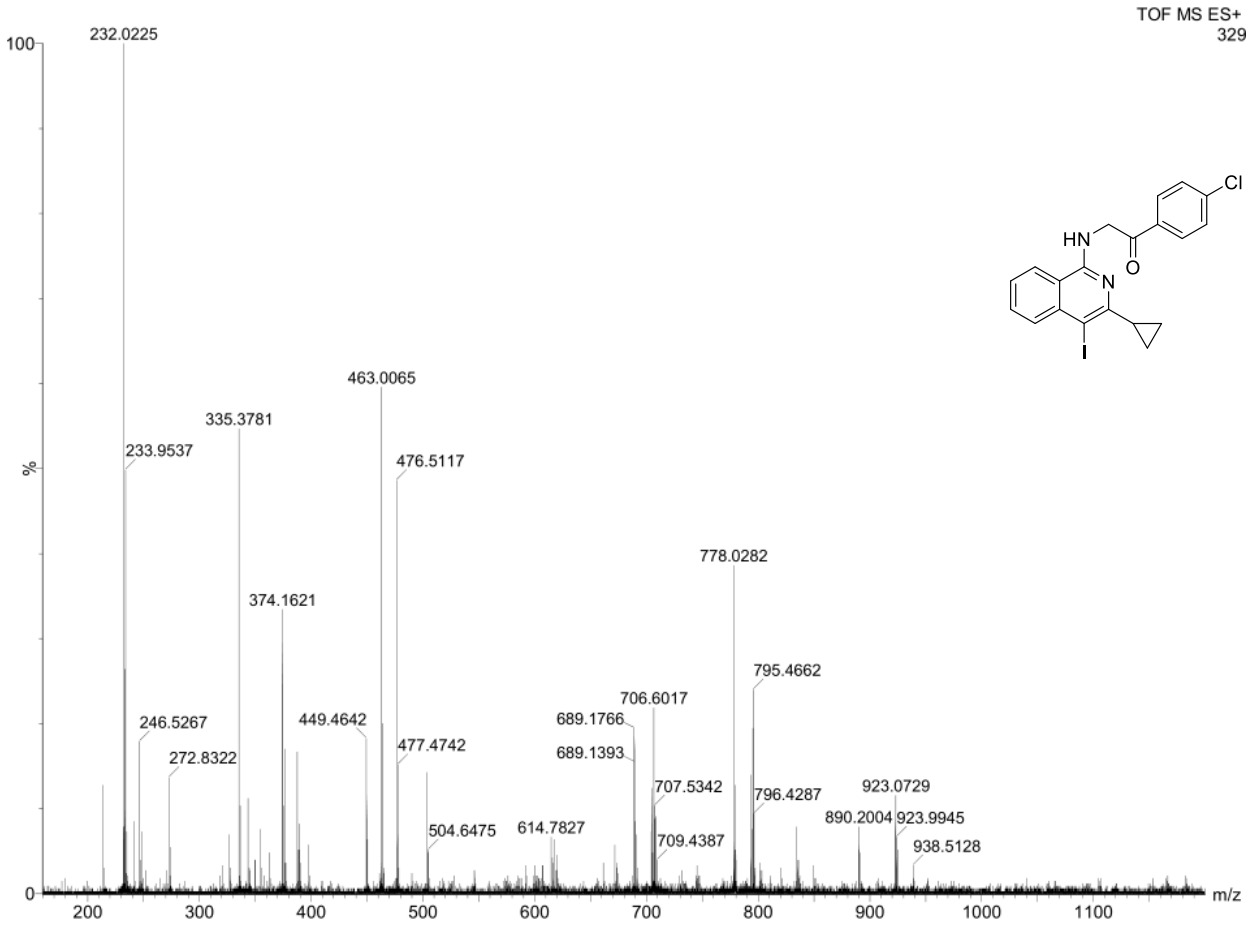
HRMS-ESI (m/z) of (**4d**)



^{1}H-NMR of compound (**4e**) (300 MHz, $CDCl_3$)

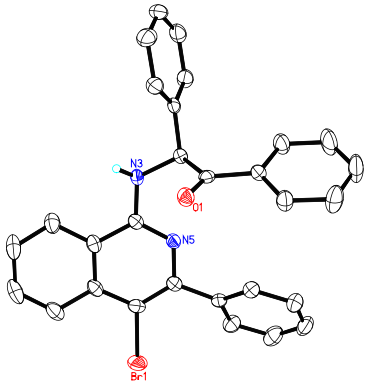


^{13}C-NMR of compound (**4e**) (75 MHz, $CDCl_3$)



HRMS-ESI (m/z) of (**4e**)

Crystallographic Data of Compound **3j**:



Thermal ellipsoid plot for compound **(3j)**, displacement ellipsoids are drawn at the 50% probability level.

Table 1: Crystal data and structure refinement for **3j**.

Identification code	**3j**	
Empirical formula	$C_{29}H_{21}BrN_2O$	
Formula weight	493.39	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	monoclinic	
Space group	C2/c	
Z	8	
Unit cell dimensions	a = 22.7593(14) Å	α = 90 deg.
	b = 10.8788(7) Å	β =111.8321(10) deg.
	c = 19.9065(12) Å	γ = 90 deg.
Volume	4575.2(5) $Å^3$	
Density (calculated)	1.43 g/cm^3	
Absorption coefficient	1.82 mm^{-1}	
Crystal shape	brick	
Crystal size	0.132 x 0.105 x 0.089 mm^3	
Crystal colour	colourless	
Theta range for data collection	1.9 to 28.3 deg.	
Index ranges	-30≤h≤30, -14≤k≤14, -26≤l≤26	
Reflections collected	26581	

Independent reflections	5673 (R(int) = 0.0641)
Observed reflections	3829 (I > 2σ(I))
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.87 and 0.80
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	5673 / 0 / 302
Goodness-of-fit on F^2	1.02
Final R indices (I>2sigma(I))	R1 = 0.046, wR2 = 0.086
Largest diff. peak and hole	1.04 and -1.11 eÅ^{-3}

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suggestion for a short experimental part:

3j: colourless crystal (brick), dimensions 0.132 x 0.105 x 0.089 mm^3, crystal system monoclinic, space group C2/c, Z=8, a=22.7593(14) Å, b=10.8788(7) Å, c=19.9065(12) Å, alpha=90 deg, beta=111.8321(10) deg, gamma=90 deg, V=4575.2(5) Å^3, rho=1.433 g/cm^3, T=200(2) K, Theta$_{max}$= 28.280 deg, radiation MoKa, lambda=0.71073 Å, 0.5 deg omega-scans with CCD area detector, covering the asymmetric unit in reciprocal space with a mean redundancy of 4.58and a completeness of 99.9% to a resolution of 0.75 Å, 26581 reflections measured, 5673 unique (R(int)=0.0641), 3829 observed (I > 2s(I)), intensities were corrected for Lorentz and polarization effects, an empirical scaling and absorption correction was applied using SADABS based on the Laue symmetry of the reciprocal space, mu=1.82mm^{-1}, T$_{min}$=0.80, T$_{max}$=0.87, structure solved with SHELXT-2018/2 (Sheldrick 2015) and refined against F^2 with a Full-matrix least-squares algorithm using the SHELXL-2018/3 (Sheldrick, 2018) software, 302 parameters refined, hydrogen atoms were treated using appropriate riding models, except H3 at N3, which was refined isotropically, goodness of fit 1.02 for observed reflections, final residual values R1(F)=0.046, wR(F^2)=0.086 for observed reflections, residual electron density -1.11 to 1.04 eÅ^{-3}. **CCDC 2130144** contains the supplementary crystallographic data for this paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/structures.[1]

References:

1. (a) Krause, L.; Herbst-Irmer, R.; Sheldrick, G. M.; Stalke, D. Comparison of Silver and Molybdenum Microfocus X-ray Sources for Single-Crystal Structure Determination. *J. Appl. Cryst.* **2015**, *48*, 3-10. (b) Sheldrick, G. M. Shelxt Integrated Space-Group and Crystal Structure Determination. *Acta Cryst.* **2015**, *A71*, 3-8.