Electrochemically Driven Regioselective Synthesis of 4-Sulfenyl-1*H*-isochromen-1-ones from *o*-Alkynyl Benzoates and Diaryl Disulfides

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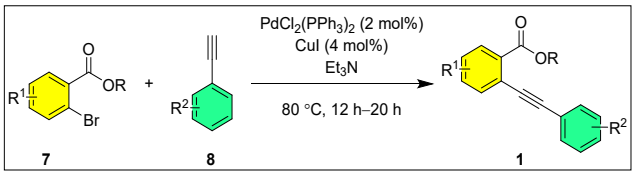
Supporting Information

Experimental:

General methods:

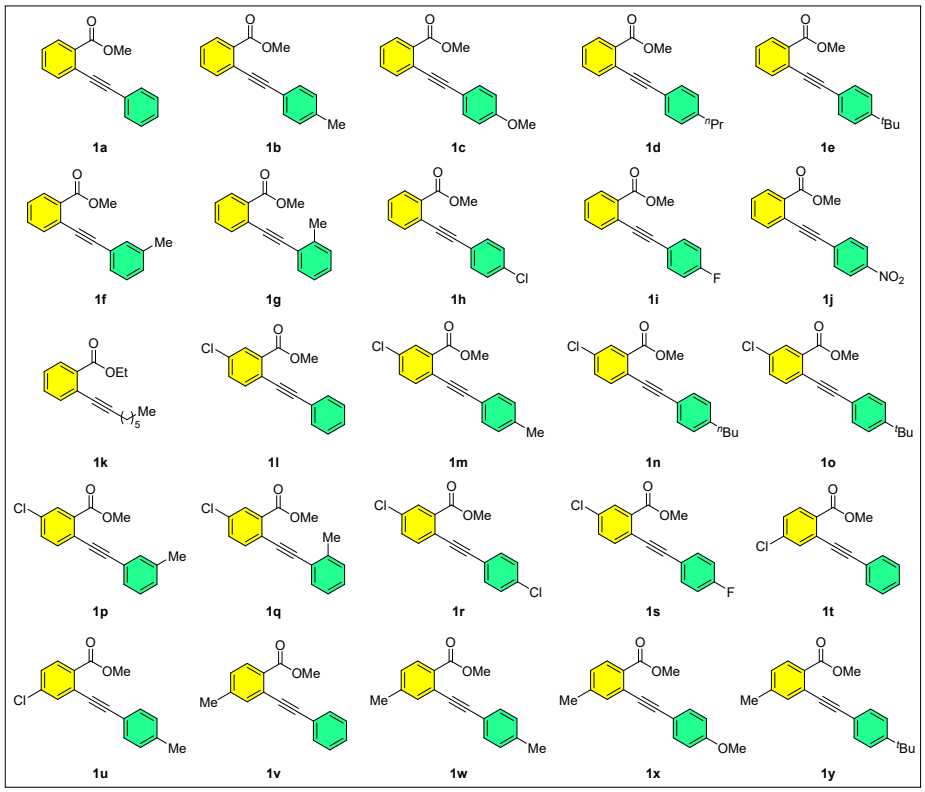
IR spectra were recorded on a Bruker Tensor 37 (FTIR) spectrophotometer. ^{1}H NMR spectra were recorded on Bruker Advance 400 (400 MHz) and 600 (600 MHz) spectrometers at 295 K in $CDCl_3$; chemical shifts (δ ppm) and coupling constants (Hz) are reported in standard fashion concerning either internal standard tetramethylsilane (TMS) (δH = 0.00 ppm) *or* $CDCl_3$ (δH = 7.26 ppm). $^{13}C\{^{1}H\}$ NMR spectra were recorded on Bruker Advance 400 (101 MHz) and 600 (151 MHz) spectrometers at RT in $CDCl_3$. Chemical shifts (δ ppm) are reported relative to $CDCl_3$ [δ = 77.16 ppm (central line of the triplet)]. In the $^{13}C\{^{1}H\}$ NMR, the nature of carbons (C, CH, CH_2, and CH_3) was determined by recording the DEPT-135 spectra and is given in parentheses and noted as s = singlet (for C), d = doublet (for CH), t = triplet (for CH_2) and q = quartet (for CH_3). In the ^{1}H-NMR, the following abbreviations were used throughout: s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, sept = septet, dd = doublet of doublet, m = multiplet and br. s = broad singlet. The assignment of signals was confirmed by ^{1}H, $^{13}C\{^{1}H\}$ CPD, and DEPT spectra. High-resolution mass spectra (HRMS) were recorded on an Agilent 6538 UHD Q-TOF electron spray ionization (ESI) mode and atmospheric pressure chemical ionization (APCI) modes. Melting points are recorded using Tempo and Mettler FP1 melting point apparatus in capillary tubes and are uncorrected. A single crystal of **3cc** was selected and mounted on an Oxford SuperNova, Dual, Cu at zero, Eos diffractometer. The crystal was kept at 298 K during data collection. Using Olex2, the structure was solved with the olex2.solve structure solution program using direct methods and refined with the olex2. refinement package using Gauss−Newton minimization. Electrolysis reactions were conducted using ElectraSyn 2.0 Package supply purchased from IKA Instruments. Reactions were monitored by TLC on silica gel using a combination of hexane and ethyl acetate as eluents. Solvents were distilled before use; petroleum ether the boiling range of 60−80 °C was used. Cyclic voltammetry (CV) analysis was performed using a platinum disk electrode as the working electrode, a platinum wire electrode as a counter electrode and Ag/AgCl electrode as a reference electrode. The Cyclic voltammogram was recorded at 50 mV/s scan rate. The phenylacetylenes, electrolytes and solvents were purchased from Sigma-Aldrich/Avra/BLD/TCI/local sources and used as received. Acme's silica gel (60–120 mesh) was used for column chromatography (approximately 20 g per one gram of crude material).

General Procedure - 1 (GP-1) for the preparation of starting material **1**:[1]

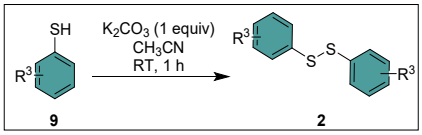


To an oven-dried Schlenk tube equipped with a magnetic stir bar, were added alkyl 2-bromobenzoate **7** (1.5 mmol, 1 equiv), $PdCl_2(PPh_3)_2$ (21 mg, 2 mol%), CuI (12 mg, 4 mol%) and Et_3N (5 mL). The resultant reaction mixture was allowed to stir at room temperature for 10 min. Then, a solution of phenylacetylene **8** (1.8 mmol, 1.2 equiv) in triethylamine (2.0 mL) was added dropwise under an argon atmosphere over 5 minutes *via* syringe and the reaction mixture was left to stir at 80 °C for 12 h. Upon completion of the reaction, the mixture was poured into an aqueous NH_4Cl solution (30 mL) and extracted with ethyl acetate (3 × 10 mL) and the combined organic phase was washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate, 95:05–90:10), furnished **1** (80% to 94% yields), as a yellow/brown liquid *or* colorless solid (Table S1).

Table S1: The following starting material is prepared by using the literature reports **1a-1y**.[1]

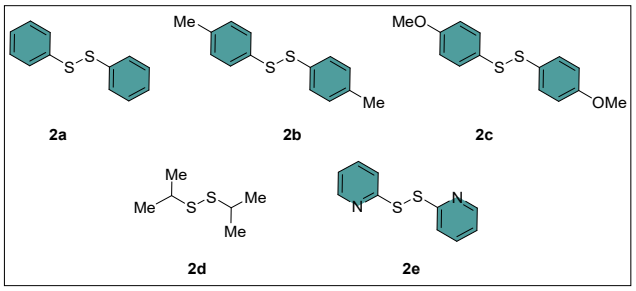


General Procedure - 2 (GP-2) for the preparation of starting material **2**:[2]

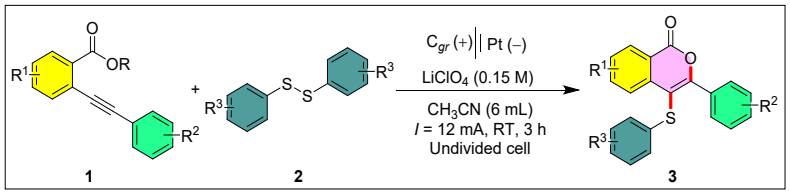


An oven-dried 25 mL round bottom flask equipped with a magnetic stir bar, were added thiol **9** (1.0 equiv), anhydrous potassium carbonate (1.0 equiv), and CH_3CN (5 mL) and the mixture was stirred at rt for 1 h. The progress of the reaction was monitored by TLC. Upon completion of the reaction, mixture was quenched with EtOAc and extracted with ethyl acetate (3 × 10 mL) and the combined organic phase was washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate, 100:00–95:05) furnished **2** (70% to 80% yields), as a yellow *or* colorless solid (Table S2).

Table S2: The following starting material **2a-2e** is prepared by using literature reports.[2]

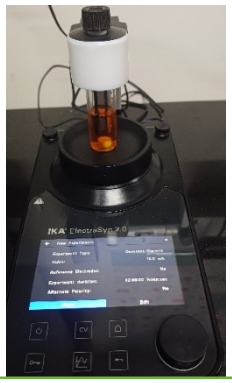


General Procedure - 3 (GP-3) for the Preparation of 4-sulfenyl-1*H*-isochromen-1-ones **3**:



To an oven dried ElectraSyn vial (10 mL) equipped with a magnetic stirring bar, were added *o*-alkynylbenzoate **1** (0.25 mmol), diaryl disulfide **2** (0.30 mmol, 1.2 equiv), $LiClO_4$ (0.15 M), and CH_3CN (6 mL). The ElectraSyn vial cap fitted with the anode (Graphite) and cathode (Platinum) and was inserted into the reaction mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 12 mA for 3 h. After completion of the reaction, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (2 mL), and the mixture was poured into an aqueous NH_4Cl solution (40 mL) and

extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate, 95:05–90:10) furnished the desired product **3** (72 to 87% yields), as a yellow liquid *or* yellow (*or* white) solids.



'Zoomed' photo of the experiments.

Cyclic Voltammograms:

The cyclic voltammetry (CV) studies were carried out to insight the reaction mechanism, and below Figure S1 shows the cyclic voltammetry (CV) curves with 0.1 M $LiClO_4$ solution in CH_3CN as a background. The voltammogram was obtained at a scan rate of 50 mV/s with Pt wire as a counter electrode, Pt disk electrode as a working electrode and Ag/AgCl as a reference electrode which is submerged in saturated aqueous KCl solution. Within the scanning window (0 to 2.5 V). An evident there is no peak for blank (Curve 1, black line). The CV of methyl 2-(phenylethynyl)benzoate **1a** displayed oxidation peak at 1.77 V *vs* Ag/AgCl (Curve 2, red line). When testing diphenyl disulfide **2a**, an oxidation peak was seemed at 1.65 V *vs* Ag/AgCl in Curve 3 (blue line). The mixture of methyl 2-(phenylethynyl)benzoate **1a** and diphenyl diselenide **2a** showed two oxidation signals at 1.81 V and 1.52 V as shown in curve 4 (pink line), respectively.

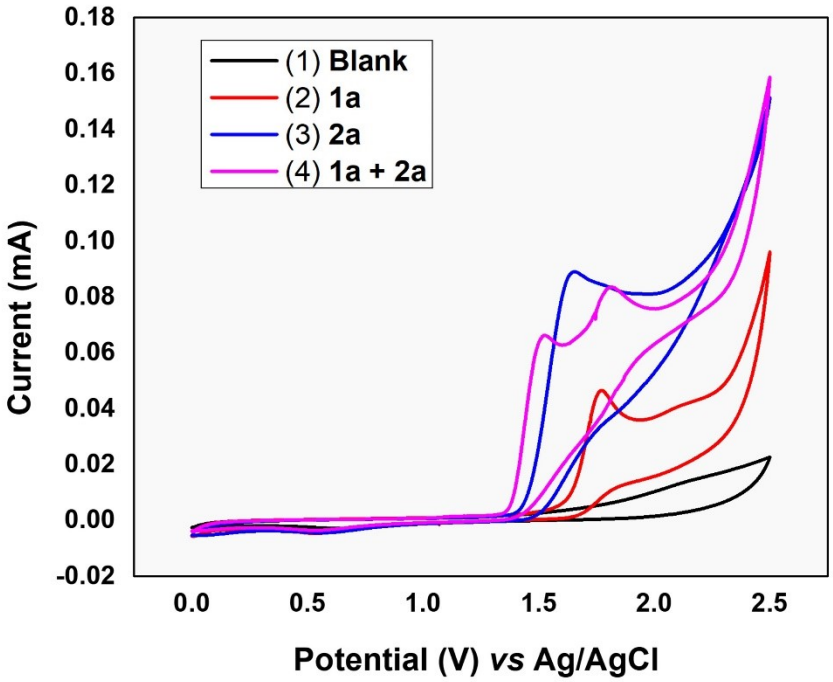
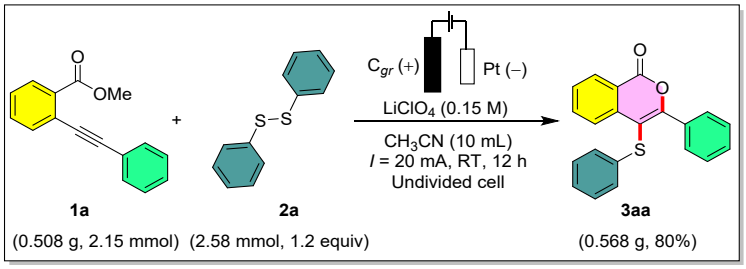


Figure S1. Cyclic voltammograms of reactants and their mixtures in 0.1 M $LiClO_4$ solution in CH_3CN at room temperature: 1) Blank (black line); 2) **1a** (0.01 M) (red line); 3) **2a** (0.01 M) (blue line); 4) **1a** (0.01 M) + **2a** (0.01 M) (pink line); The voltammogram was obtained at a scan rate of 50 mV/s with Pt wire as a counter electrode, Ag/AgCl as a reference electrode, and Pt disk electrode as a working electrode.

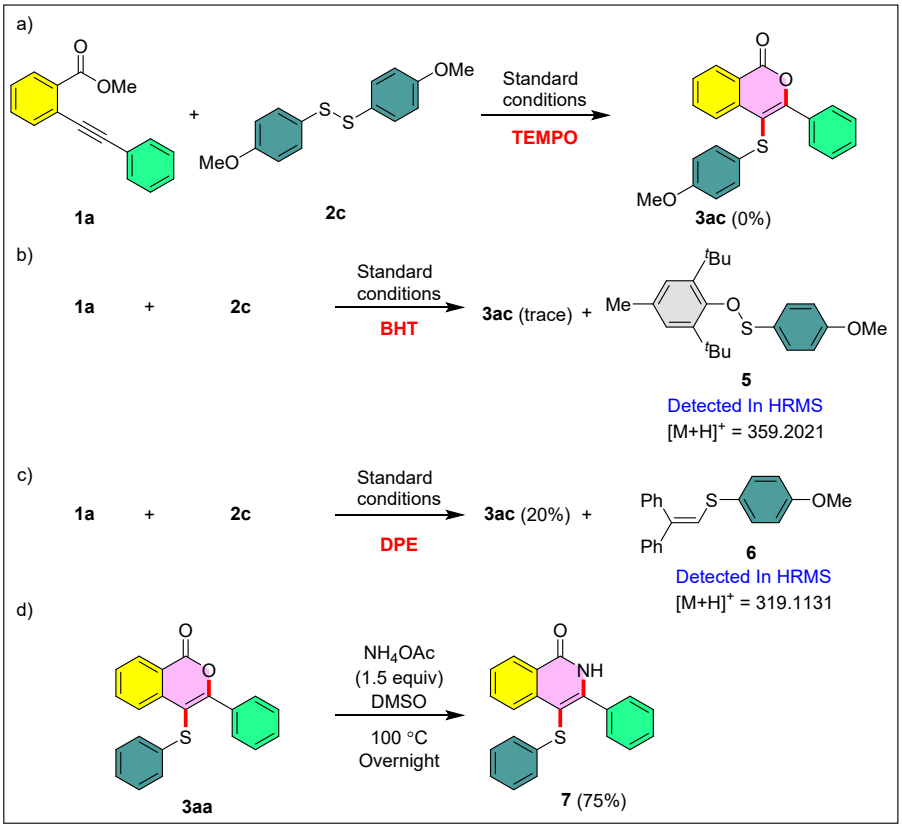
Scheme S1: Scale up reaction.



To an oven dried ElectraSyn vial (10 mL) equipped with a magnetic stirring bar, were added methyl 2-(phenylethynyl)benzoate **1a** (508 mg, 2.15 mmol), 1,2-diphenyldisulfane **2a** (2.58 mmol, 1.2 equiv), $LiClO_4$ (159 mg, 0.15 M), and CH_3CN (10 mL). The ElectraSyn vial cap fitted with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 20 mA for 12 h. After completion of the reaction, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (5 mL) and the mixture was poured into aqueous NH_4Cl solution (50 mL) and extracted with DCM (5 × 10 mL). The combined organic layers were washed with brine and

dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure and purification of the residue by silica gel column chromatography using petroleum ether/ethyl acetate as the eluent furnished the desired product **3aa** (0.568 g, 80%), as a colorless solid.

Scheme S2: Control Experiments



a) To an oven dried ElectraSyn vial (10 mL) equipped with a magnetic stirring bar, were added methyl 2-(phenylethynyl)benzoate **1a** (59 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (0.30 mmol, 1.2 equiv), TEMPO (0.50 mmol, 2 equiv), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (10 mL). The ElectraSyn vial cap fitted with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 12 mA for 3 h. After completion of the reaction, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (5 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure.

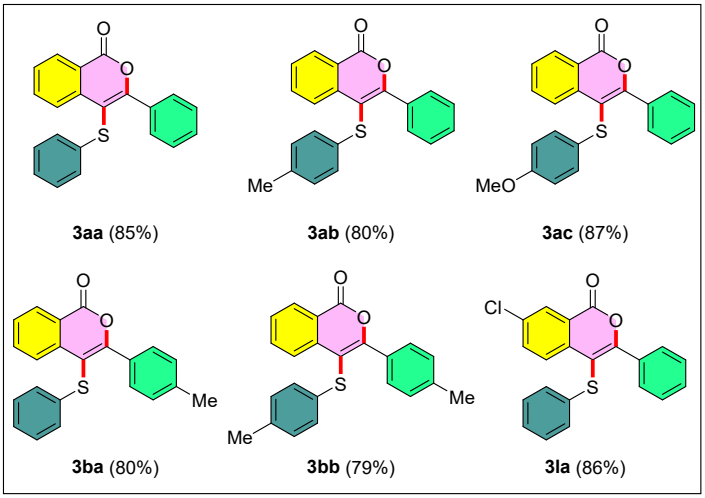
b) To an oven dried ElectraSyn vial (10 mL) equipped with a magnetic stirring bar, were added methyl 2-(phenylethynyl)benzoate **1a** (59 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (0.30 mmol, 1.2 equiv), BHT (0.50 mmol, 2 equiv), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (10 mL). The ElectraSyn vial cap fitted with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 12 mA for 3 h. After completion of the reaction, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (5 mL) and the mixture was poured into aqueous NH_4Cl solution (40 mL) and extracted with DCM (3 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure. The crude mixture was subjected to mass spectrometric analysis to confirm the formation of (2,6-di-tert-butyl-4-methylphenoxy)(4-methoxyphenyl)sulfane **5**. HRMS-ESI (m/z): calcd for $C_{22}H_{31}O_2S^+$ $[M + H]^+$ 359.2039; found 359.2021.

c) To an oven dried ElectraSyn vial (10 mL) equipped with a magnetic stirring bar, were added methyl 2-(phenylethynyl)benzoate **1a** (59 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (0.30 mmol, 1.2 equiv), 1,1-diphenylethylene (DPE) (0.50 mmol, 2 equiv), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (10 mL). The ElectraSyn vial cap fitted with anode (graphite) and cathode (platinum) were inserted into the mixture. The reaction mixture was electrolyzed at ambient temperature under a constant current of 12 mA for 3 h. After completion of the reaction, the ElectraSyn vial cap was removed, electrodes were rinsed with DCM (5 mL) and the mixture was poured into aqueous NH_4Cl solution (50 mL) and extracted with DCM (5 × 10 mL). The combined organic layers were washed with brine and dried over Na_2SO_4. Evaporation of the solvent(s) under reduced pressure. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ac** (18 mg, 20%), as a colorless solid. The crude mixture was subjected to mass spectrometric analysis to confirm the formation of (2,2-diphenylvinyl)(4-methoxyphenyl)sulfane **6**. HRMS-ESI (m/z): calcd for $C_{21}H_{19}OS^+$ $[M + H]^+$ 319.1151; found 319.1131.

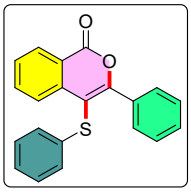
d) To an oven-dried 10 mL round bottom flask equipped with a magnetic stir bar, were added 3-phenyl-4-(phenylthio)-1H-isochromen-1-one **3aa** (50 mg, 1.0 equiv), ammonium acetate (18 mg, 1.5 equiv), and DMSO (3 mL). The mixture was stirred at 100 °C for overnight under an argon atmosphere. The progress of the reaction was

monitored by TLC. After completion of the reaction, the mixture was poured into aqueous NH_4Cl solution (50 mL) and extracted with ethyl acetate (3 × 10 mL). The combined organic phase was washed with brine and dried over Na_2SO_4. Evaporation of the solvent under reduced pressure and purification of the residue by silica gel column chromatography (petroleum ether/ethyl acetate, 60:40) furnished the 3-phenyl-4-(phenylthio)isoquinolin-1(2*H*)-one **7** (37 mg, 75% yields).

Table-S3. The following 4-Sulfenyl-1*H*-isochromen-1-ones (**3aa**, **3ab**, **3ac**, **3ba**, **3bb**, and **3la)** are known in the literature as shown in Table-S3.[3–5]



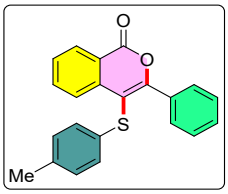
Characterization of compounds:



3-Phenyl-4-(phenylthio)-1*H*-isochromen-1-one (3aa):

GP3 was carried out with methyl 2-(phenylethynyl)benzoate **1a** (59 mg, 0.25 mmol), 1,2-diphenyldisulfane **2a** (65 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M)), and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3aa** (70 mg, 85%), as a colorless solid , mp = 140–142 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.50, R_f (**2a**) = 0.90, R_f (**3aa**) = 0.48, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3050, 2930, 2840, 1737, 1595, 1475, 1220, 1070, 1000, 790, 699 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.37 (dd, J = 7.9, 0.9 Hz, 4H), 7.97 (d, J = 8.1 Hz, 1H), 7.77 – 7.68 (m, 3H), 7.57 (ddd, J = 7.2, 6.9, 1.1 Hz, 1H), 7.47 – 7.37 (m, 3H), 7.26 – 7.20 (m, 2H), 7.16 – 7.10 (m, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.6, 160.3, 138.1, 136.7, 135.5, 132.9, 130.5, 129.9, 129.5 (2 × Ar–CH), 129.46 (2 × Ar–CH), 128.9, 128.1 (2 × Ar–CH), 126.2, 126.1 (2 × Ar–CH), 125.8, 121.0, 106.6 ppm.

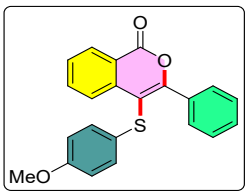


3-Phenyl-4-(*p*-tolylthio)-1*H*-isochromen-1-one (3ab):

GP3 was carried out with methyl 2-(phenylethynyl)benzoate **1a** (59 mg, 0.25 mmol), 1,2-di-*p*-tolyldisulfane **2b** (74 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ab** (69 mg, 80%), as a colorless solid , mp = 170–172 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.50, R_f (**2b**) = 0.90, R_f (**3ab**) = 0.48, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3062, 2922, 2860, 1738, 1601, 1484, 1229, 1082, 1020, 804, 760, 692 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (dd, J = 7.9, 1.0 Hz, 1H), 7.98 (d, J = 8.1 Hz, 1H), 7.77 – 7.72 (m, 2H), 7.70 (dd, J = 8.1, 1.4 Hz, 1H), 7.54 (ddd, J = 7.2, 6.9, 1.1

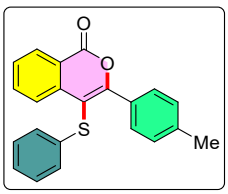
Hz, 1H), 7.47 – 7.37 (m, 3H), 7.07 – 6.99 (m, 4H), 2.27 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.6, 160.1, 138.2, 135.8, 135.5, 133.1, 132.9, 130.4, 130.2 (2 × Ar–CH), 129.9, 129.6 (2 × Ar–CH), 128.8, 128.1 (2 × Ar–CH), 126.3 (2 × Ar–CH), 126.2, 121.0, 107.0, 21.0 ppm.



4-((4-Methoxyphenyl)thio)-3-phenyl-1*H*-isochromen-1-one (3ac):

GP3 was carried out with methyl 2-(phenylethynyl)benzoate **1a** (59 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ac** (78 mg, 87%), as a colorless solid , mp = 112–114 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1a**) = 0.50, R_f (**2c**) = 0.50, R_f (**3ac**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3058, 2947, 2835, 1732, 1597, 1483, 1235, 1079, 1020, 823, 733, 688 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.35 (dd, J = 7.8, 0.8 Hz, 1H), 8.04 (d, J = 8.1 Hz, 1H), 7.77 (dd, J = 8.0, 1.7 Hz, 2H), 7.72 (ddd, J = 7.4, 6.9, 0.6 Hz, 1H), 7.53 (t, J = 7.6 Hz, 1H), 7.47 – 7.39 (m, 3H), 7.07 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.73 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 161.6, 159.7, 158.4, 138.2, 135.3, 132.9, 130.3, 129.8, 129.7 (2 × Ar–CH), 128.7, 128.5 (2 × Ar–CH), 128.0 (2 × Ar–CH), 126.9, 126.2, 120.9, 115.1 (2 × Ar–CH), 108.0, 55.4 ppm.

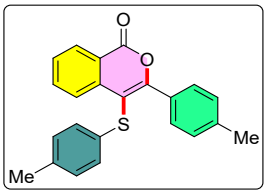


4-(Phenylthio)-3-(*p*-tolyl)-1*H*-isochromen-1-one (3ba):

GP3 was carried out with methyl 2-(*p*-tolylethynyl)benzoate **1b** (62 mg, 0.25 mmol), 1,2-diphenyldisulfane **2a** (65 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ba** (69 mg, 80%), as a colorless solid , mp =

110–112 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1b**) = 0.50, R_f (**2a**) = 0.90, R_f (**3ba**) = 0.48, UV detection].

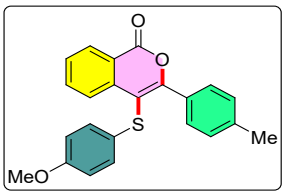
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3061, 2922, 2835, 1737, 1597, 1471, 1268, 1081, 1019, 826, 752, 690 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.36 (dd, J = 7.9, 1.0 Hz, 1H), 7.95 (d, J = 8.1 Hz, 1H), 7.70 (ddd, J = 7.2, 6.8, 1.4 Hz, 1H), 7.65 (d, J = 8.2 Hz, 2H), 7.53 (ddd, J = 6.9, 6.3, 1.1 Hz, 1H), 7.26 – 7.19 (m, 4H), 7.16 – 7.10 (m, 3H), 2.39 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.7, 160.5, 140.8, 138.2, 136.8, 135.5, 130.0, 129.9, 129.5 (2 × Ar–CH), 129.4 (2 × Ar–CH), 128.8 (2 × Ar–CH), 128.7, 126.1, 126.0 (2 × Ar–CH), 125.8, 120.9, 106.0, 21.6 ppm.



3-(*p*-Tolyl)-4-(*p*-tolylthio)-1*H*-isochromen-1-one (3bb):

GP3 was carried out with methyl 2-(*p*-tolylethynyl)benzoate **1b** (62 mg, 0.25 mmol), 1,2-di-*p*-tolyldisulfane **2b** (74 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3bb** (71 mg, 79%), as a colorless solid , mp = 152–154 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1b**) = 0.50, R_f (**2b**) = 0.90, R_f (**3bb**) = 0.48, UV detection].

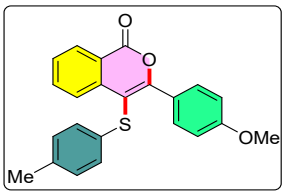
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3028, 2920, 2861, 1734, 1599, 1480, 1080, 1017, 809, 762, 691 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.35 (dd, J = 7.9, 1.0 Hz, 1H), 7.96 (d, J = 8.1 Hz, 1H), 7.70 (ddd, J = 7.2, 6.9, 1.4 Hz, 1H), 7.66 (d, J = 8.2 Hz, 2H), 7.52 (ddd, J = 7.1, 6.9, 1.1 Hz, 1H), 7.21 (d, J = 7.9 Hz, 2H), 7.06 – 6.98 (m, 4H), 2.39 (s, 3H), 2.28 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.7, 160.3, 140.8, 138.3, 135.7, 135.4, 133.2, 130.2 (2 × Ar–CH), 130.1, 129.9, 129.5 (2 × Ar–CH), 128.8 (2 × Ar–CH), 128.6, 126.2 (2 × Ar–CH), 120.9, 106.4, 21.6, 21.0 ppm.



4-((4-Methoxyphenyl)thio)-3-(*p*-tolyl)-1*H*-isochromen-1-one (3bc):

GP3 was carried out with methyl 2-(*p*-tolylethynyl)benzoate **1b** (62 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3bc** (73 mg, 78%), as a yellow solid , mp = 100–102 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1b**) = 0.50, R_f (**2c**) = 0.50, R_f (**3bc**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2930, 2840, 1732, 1597, 1482, 1237, 1076, 1019, 761, 731, 690 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.34 (dd, J = 7.9, 1.0 Hz, 1H), 8.02 (d, J = 8.0 Hz, 1H), 7.71 (dd, J = 7.3, 1.2 Hz, 1H), 7.68 (d, J = 8.2 Hz, 2H), 7.51 (ddd, J = 7.2, 7.0, 1.0 Hz, 1H), 7.23 (d, J = 8.0 Hz, 2H), 7.07 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.73 (s, 3H), 2.40 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 161.7, 159.9, 158.3, 140.7, 138.3, 135.3, 130.1, 129.8, 129.6 (2 × Ar–CH), 128.7 (2 × Ar–CH), 128.6, 128.4 (2 × Ar–CH), 127.1, 126.2, 120.9, 115.1 (2 × Ar–CH), 107.4, 55.4, 21.6 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{19}O_3S^+$ $[M+H]^+$: 375.1049, found: 375.1051.

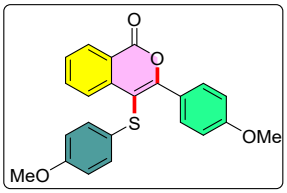


3-(4-Methoxyphenyl)-4-(*p*-tolylthio)-1*H*-isochromen-1-one (3cb):

GP3 was carried out with methyl 2-((4-methoxyphenyl)ethynyl)benzoate **1c** (66 mg, 0.25 mmol), 1,2-di-*p*-tolyldisulfane **2b** (74 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3cb** (69 mg, 74%), as a colorless solid , mp = 136–138 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1c**) = 0.40, R_f (**2b**) = 0.90, R_f (**3cb**) = 0.38, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2922, 2854, 1738, 1602, 1503, 1253, 1178, 1081, 1026, 767 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 8.34 (dd, J = 7.9, 0.9 Hz, 1H), 7.96 (d, J = 7.9 Hz, 1H), 7.75 (d, J = 8.9 Hz, 2H), 7.69 (ddd, J = 7.1, 6.9, 1.4 Hz, 1H), 7.51 (ddd, J = 7.1, 7.0, 1.0

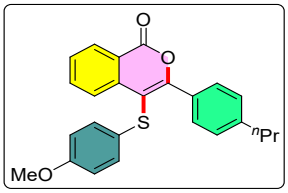
Hz, 1H), 7.05 (d, J = 8.4 Hz, 2H), 7.02 (d, J = 8.5 Hz, 2H), 6.91 (d, J = 8.9 Hz, 2H), 3.84 (s, 3H), 2.28 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.8, 161.2, 160.0, 138.5, 135.7, 135.4, 133.2, 131.3 (2 × Ar–CH), 130.2 (2 × Ar–CH), 129.8, 128.5, 126.1 (2 × Ar–CH), 125.2, 120.8, 113.5 (3 × Ar–CH), 105.7, 55.5, 21.0 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{19}O_3S^+$ $[M+H]^+$: 375.1049, found: 375.1049.



3-(4-Methoxyphenyl)-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3cc):

GP3 was carried out with methyl 2-((4-methoxyphenyl)ethynyl)benzoate **1c** (66 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3cc** (83 mg, 85%), as a colorless solid , mp = 138–140 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1c**) = 0.40, R_f (**2c**) = 0.50, R_f (**3cc**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3066, 2935, 2840, 1734, 1600, 1498, 1245, 1176, 1081, 1024, 828, 764, 692 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.33 (dd, J = 7.9, 0.9 Hz, 1H), 8.01 (d, J = 8.1 Hz, 1H), 7.76 (d, J = 8.9 Hz, 2H), 7.70 (ddd, J = 7.1, 6.9, 1.3 Hz, 1H), 7.51 (ddd, J = 7.2, 7.0, 1.0 Hz, 1H), 7.06 (d, J = 8.9 Hz, 2H), 6.93 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.85 (s, 3H), 3.74 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.8, 161.2, 159.7, 158.3, 138.5, 135.3, 131.4 (2 × Ar–CH), 129.8, 128.4, 128.3 (2 × Ar–CH), 127.2, 126.1, 125.3, 120.8, 115.2 (2 × Ar–CH), 113.4 (2 × Ar–CH), 106.7, 55.5, 55.4 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{19}O_4S^+$ $[M+H]^+$: 391.0999, found: 391.1001.

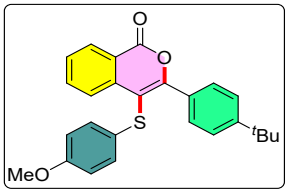


4-((4-Methoxyphenyl)thio)-3-(4-propylphenyl)-1*H*-isochromen-1-one (3dc):

GP3 was carried out with methyl 2-((4-propylphenyl)ethynyl)benzoate **1d** (69 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography

(petroleum ether/ethyl acetate, 95:05) furnished the product **3dc** (86 mg, 86%), as a yellow solid , mp = 100–102 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1d**) = 0.50, R_f (**2c**) = 0.50, R_f (**3dc**) = 0.30, UV detection].

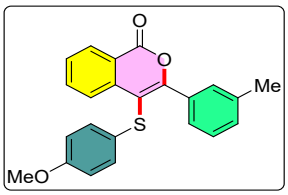
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2926, 2858, 1736, 1599, 1489, 1241, 1079, 1024, 821, 769 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.34 (dd, J = 7.9, 1.0 Hz, 1H), 8.02 (d, J = 8.0 Hz, 1H), 7.74 – 7.66 (m, 3H), 7.52 (ddd, J = 7.1, 7.0, 1.1 Hz, 1H), 7.23 (d, J = 8.2 Hz, 2H), 7.07 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.73 (s, 3H), 2.63 (t, J = 7.8 Hz, 2H), 1.73 – 1.61 (m, 2H), 0.96 (t, J = 7.3 Hz, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.7, 159.9, 158.3, 145.4, 138.4, 135.3, 130.3, 129.8, 129.6 (2 × Ar–CH), 128.6, 128.4 (2 × Ar–CH), 128.2 (2 × Ar–CH), 127.1, 126.2, 120.9, 115.1 (2 × Ar–CH), 107.4, 55.4, 38.0, 24.4, 13.9 ppm. **HRMS:** *m/z* calcd for $C_{25}H_{23}O_3S^+$ $[M+H]^+$: 403.1362, found: 403.1359.



3-(4-(*tert*-Butyl)phenyl)-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3ec):

GP3 was carried out with methyl 2-((4-(*tert*-butyl)phenyl)ethynyl)benzoate **1e** (73 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ec** (83 mg, 80%), as a brown solid , mp = 138–140 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1e**) = 0.50, R_f (**2c**) = 0.50, R_f (**3ec**) = 0.30, UV detection].

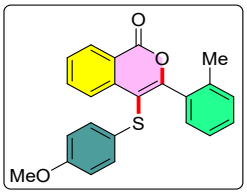
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3063, 2957, 1734, 1599, 1484, 1238, 1081, 1022, 829, 762, 737, 693 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.34 (dd, J = 7.9, 1.0 Hz, 2H), 8.01 (d, J = 8.1 Hz, 1H), 7.73 (d, J = 8.6 Hz, 2H), 7.71 – 7.67 (m, 1H), 7.52 (ddd, J = 7.2, 6.9, 1.1 Hz, 1H), 7.44 (d, J = 8.6 Hz, 2H), 7.08 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 1.34 (s, 9H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.7, 159.9, 158.4, 153.7, 138.4, 135.3, 130.0, 129.8, 129.5 (2 × Ar–CH), 128.6, 128.3 (2 × Ar–CH), 127.2, 126.2, 125.1 (2 × Ar–CH), 121.0, 115.1 (2 × Ar–CH), 107.3, 55.4, 35.0, 31.3 ppm. **HRMS:** *m/z* calcd for $C_{26}H_{25}O_3S^+$ $[M+H]^+$: 417.1519, found: 417.1522.



4-((4-Methoxyphenyl)thio)-3-(*m*-tolyl)-1*H*-isochromen-1-one (3fc):

GP3 was carried out with methyl 2-(*m*-tolylethynyl)benzoate **1f** (62 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3fc** (75 mg, 80%), as a yellow solid , mp = 98–100 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1f**) = 0.50, R_f (**2c**) = 0.50, R_f (**3fc**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2925, 2843, 1733, 1595, 1483, 1284, 1241, 1178, 1085, 1021, 822, 766, 698 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.29 (dd, J = 7.9, 1.2 Hz, 1H), 7.97 (d, J = 8.1 Hz, 1H), 7.69 – 7.62 (m, 1H), 7.52 – 7.44 (m, 3H), 7.26 – 7.18 (m, 2H), 7.00 (d, J = 8.9 Hz, 2H), 6.71 (d, J = 8.9 Hz, 2H), 3.68 (s, 3H), 2.32 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.7, 160.0, 158.4, 138.3, 137.8, 135.4, 132.9, 131.1, 130.2, 129.9, 128.7, 128.6 (2 × Ar–CH), 127.9, 127.1, 126.9, 126.2, 121.0, 115.1 (2 × Ar–CH), 107.9, 55.4, 21.5 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{19}O_3S^+$ $[M+H]^+$: 375.1049, found: 375.1040.

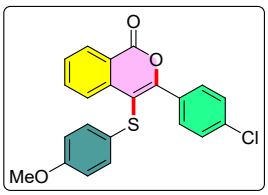


4-((4-Methoxyphenyl)thio)-3-(*o*-tolyl)-1*H*-isochromen-1-one (3gc):

GP3 was carried out with methyl 2-(*o*-tolylethynyl)benzoate **1g** (62 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3gc** (76 mg, 81%), as a colorless solid , mp = 118–120 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1g**) = 0.50, R_f (**2c**) = 0.50, R_f (**3gc**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3062, 2926, 2846, 1735, 1602, 1485, 1240, 1064, 1021, 813, 760, 710 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.37 (dd, J = 7.9, 1.0 Hz, 1H), 8.06 (d, J = 7.8 Hz, 1H), 7.76 (ddd, J = 7.2, 6.8, 1.4 Hz, 1H), 7.57 (ddd, J = 7.2, 6.9, 1.1 Hz, 1H), 7.40 – 7.29 (m, 2H), 7.26 (d, J = 7.6 Hz, 1H), 7.21 (t, J = 7.5 Hz, 1H), 6.98 (d, J = 8.9 Hz, 2H), 6.72

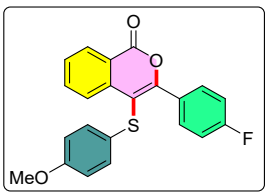
(d, J = 8.9 Hz, 2H), 3.73 (s, 3H), 2.27 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.8, 160.4, 158.6, 137.9, 137.3, 135.4, 133.0, 130.4, 130.1, 130.0, 129.9, 129.7 (2 × Ar–CH), 128.8, 126.5, 126.1, 125.5, 121.2, 114.9 (2 × Ar–CH), 110.4, 55.4, 19.9 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{19}O_3S^+$ $[M+H]^+$: 375.1049, found: 375.1043.



3-(4-Chlorophenyl)-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3hc):

GP3 was carried out with methyl 2-((4-chlorophenyl)ethynyl)benzoate **1h** (68 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3hc** (75 mg, 76%), as a colorless solid , mp = 122–124 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1h**) = 0.50, R_f (**2c**) = 0.50, R_f (**3hc**) = 0.30, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3067, 2944, 2838, 1737, 1599, 1485, 1241, 1086, 1021, 827, 762, 690 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 8.35 (dd, J = 7.9, 0.9 Hz, 1H), 8.03 (d, J = 8.1 Hz, 1H), 7.75 – 7.69 (m, 3H), 7.54 (ddd, J = 7.1, 7.0, 1.1 Hz, 1H), 7.39 (d, J = 8.7 Hz, 2H), 7.04 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 161.4, 158.6, 158.5, 138.0, 136.5, 135.5, 131.4, 131.1 (2 × Ar–CH), 129.9, 128.9, 128.5 (2 × Ar–CH), 128.4 (2 × Ar–CH), 126.6, 126.3, 121.0, 115.2 (2 × Ar–CH), 108.5, 55.5 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{16}ClO_3S^+$ $[M+H]^+$: 395.0503, found: 395.0505.

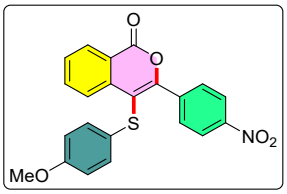


3-(4-Fluorophenyl)-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3ic):

GP3 was carried out with methyl 2-((4-fluorophenyl)ethynyl)benzoate **1i** (63 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ic** (79 mg, 84%), as a colorless

solid , mp = 130–132 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1i**) = 0.50, R_f (**2c**) = 0.50, R_f (**3ic**) = 0.30, UV detection].

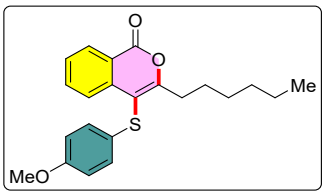
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2924, 2853, 1738, 1600, 1495, 1236, 1082, 1022, 832, 767 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 8.35 (dd, J = 7.9, 0.9 Hz, 1H), 8.03 (d, J = 7.7 Hz, 1H), 7.78 – 7.75 (m, 2H), 7.73 (ddd, J = 7.1, 6.9, 1.4 Hz, 1H), 7.54 (ddd, J = 7.1, 7.0, 1.1 Hz, 1H), 7.12 – 7.08 (m, 2H), 7.04 (d, J = 9.0 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 163.8 (d, J = 251.1 Hz), 161.5, 158.7, 158.5, 138.1, 135.5, 131.9 (d, $J_{C\text{-}F}$ = 8.8 Hz, (2 × Ar–CH)), 129.9, 129.1 (d, J = 3.4 Hz), 128.9, 128.5 (2 × Ar–CH), 126.7, 126.3, 120.9, 115.25 (d, $J_{C\text{-}F}$ = 21.9 Hz, (2 × Ar–CH)), 115.2 (2 × Ar–CH), 108.1, 55.5 ppm. **^{19}F NMR** (376 MHz, $CDCl_3$) δ = –109.3 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{16}FO_3S^+$ $[M+H]^+$: 379.0799, found: 379.0794.



4-((4-Methoxyphenyl)thio)-3-(4-nitrophenyl)-1*H*-isochromen-1-one (3jc):

GP3 was carried out with methyl 2-((4-nitrophenyl)ethynyl)benzoate **1j** (70 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3jc** (79 mg, 78%), as a yellow solid, mp = 138–140 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1j**) = 0.40, R_f (**2c**) = 0.50, R_f (**3jc**) = 0.20, UV detection].

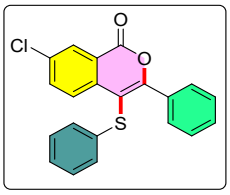
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2948, 2844, 1742, 1590, 1494, 1311, 1257, 1139, 1021, 829, 767, 661 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.38 (dd, J = 7.9, 1.0 Hz, 1H), 8.27 (d, J = 8.9 Hz, 2H), 8.08 (d, J = 7.8 Hz, 1H), 7.95 (d, J = 8.9 Hz, 2H), 7.78 (ddd, J = 7.2, 6.9, 1.4 Hz, 1H), 7.60 (ddd, J = 7.3, 6.9, 1.0 Hz, 1H), 7.04 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.75 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.9, 158.8, 156.9, 148.5, 138.9, 137.6, 135.7, 130.8 (2 × Ar–CH), 130.1, 129.6, 128.6 (2 × Ar–CH), 126.5, 126.0, 123.3 (2 × Ar–CH), 121.2, 115.4 (2 × Ar–CH), 110.3, 55.5 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{16}NO_5S^+$ $[M+H]^+$: 406.0744, found: 406.0716.



3-Hexyl-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3kc):

GP3 was carried out with ethyl 2-(oct-1-yn-1-yl)benzoate **1k** (64 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3kc** (79 mg, 86%), as a brown liquid. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1k**) = 0.65, R_f (**2c**) = 0.50, R_f (**3kc**) = 0.50, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2925, 2858, 1734, 1602, 1484, 1288, 1241, 1179, 1026, 822, 767, 693 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.28 (dd, J = 7.9, 1.0 Hz, 1H), 7.92 (d, J = 8.0 Hz, 1H), 7.67 (ddd, J = 7.1, 7.0, 1.4 Hz, 1H), 7.46 (ddd, J = 7.1, 7.0, 1.0 Hz, 1H), 7.09 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.99 (t, 7.7 Hz, 2H), 1.74 – 1.66 (m, 2H), 1.39 – 1.31 (m, 2H), 1.30 – 1.24 (m, 4H), 0.86 (t, J = 7.1 Hz, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 164.6, 162.0, 158.4, 138.1, 135.3, 129.8, 128.5 (2 × Ar–CH), 128.1, 126.7, 125.4, 120.7, 115.0 (2 × Ar–CH), 107.3, 55.4, 32.8, 31.6, 29.0, 27.9, 22.6, 14.1 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{25}O_3S^+$ $[M+H]^+$: 369.1519, found: 369.1493.

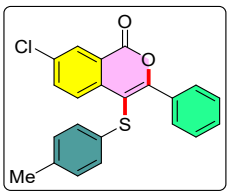


7-Chloro-3-phenyl-4-(phenylthio)-1*H*-isochromen-1-one (3la):

GP3 was carried out with methyl 5-chloro-2-(phenylethynyl)benzoate **1l** (68 mg, 0.25 mmol), 1,2-diphenyldisulfane **2a** (65 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3la** (78 mg, 86%), as a colorless solid , mp = 118–120 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1l**) = 0.60, R_f (**2a**) = 0.90, R_f (**3la**) = 0.58, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3062, 2923, 2856, 1738, 1588, 1474, 1267, 1213, 1081, 865, 738, 693 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.32 (d, J = 2.3 Hz, 1H), 7.90 (d, J = 8.7 Hz, 1H), 7.73 (dd, J = 8.2, 1.5 Hz, 2H), 7.63 (dd, J = 8.7, 2.3 Hz, 1H), 7.48 – 7.38 (m, 3H),

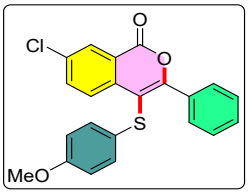
7.27 – 7.21 (m, 2H), 7.17 – 7.12 (m, 1H), 7.10 (dd, J = 8.4, 1.2 Hz, 2H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.4, 136.6, 136.2, 135.8, 134.9, 132.5, 130.7, 129.6 (2 × Ar–CH), 129.5 (2 × Ar–CH, 1 × Ar–C), 129.3, 128.1 (2 × Ar–CH), 127.9, 126.15 (2 × Ar–CH), 126.1, 122.2, 106.2 ppm.



7-Chloro-3-phenyl-4-(*p*-tolylthio)-1*H*-isochromen-1-one (3lb):

GP3 was carried out with methyl 5-chloro-2-(phenylethynyl)benzoate **1l** (68 mg, 0.25 mmol), 1,2-di-*p*-tolyldisulfane **2b** (74 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3lb** (78 mg, 82%), as a colorless solid , mp = 138–140 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1l**) = 0.60, R_f (**2b**) = 0.90, R_f (**3lb**) = 0.58, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3063, 2921, 2865, 1740, 1595, 1479, 1314, 1268, 1214, 1083, 814, 769, 695 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.32 (d, J = 2.3 Hz, 1H), 7.92 (d, J = 8.7 Hz, 1H), 7.75 (dd, J = 8.1, 1.4 Hz, 2H), 7.63 (dd, J = 8.7, 2.3 Hz, 1H), 7.49 – 7.38 (m, 3H), 7.05 (d, J = 8.2 Hz, 2H), 7.01 (d, J = 8.4 Hz, 2H), 2.28 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.5, 160.2, 136.7, 136.1, 135.7, 134.8, 132.6, 132.5, 130.6, 130.3 (2 × Ar–CH), 129.6 (2 × Ar–CH), 129.2, 128.12 (2 × Ar–CH), 128.1, 126.4 (2 × Ar–CH), 122.2, 106.6, 21.0 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{16}ClO_2S^+$ $[M+H]^+$: 379.0554, found: 379.0544.

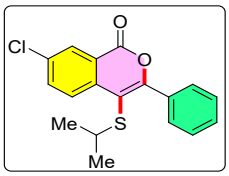


7-Chloro-4-((4-methoxyphenyl)thio)-3-phenyl-1*H*-isochromen-1-one (3lc):

GP3 was carried out with methyl 5-chloro-2-(phenylethynyl)benzoate **1l** (68 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3lc** (82 mg, 83%), as a colorless

solid , mp = 102–104 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1l**) = 0.60, R_f (**2c**) = 0.50, R_f (**3lc**) = 0.40, UV detection].

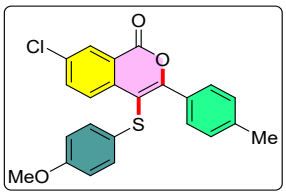
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3061, 2945, 2835, 1737, 1589, 1479, 1244, 1080, 1030, 820, 734, 692 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.30 (d, J = 2.2 Hz, 1H), 7.98 (d, J = 8.8 Hz, 1H), 7.76 (dd, J = 8.1, 1.5 Hz, 2H), 7.64 (dd, J = 8.7, 2.3 Hz, 1H), 7.49 – 7.40 (m, 3H), 7.04 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 160.4, 159.8, 158.6, 136.7, 135.6, 134.7, 132.6, 130.5, 129.7 (2 × Ar–CH), 129.2, 128.7 (2 × Ar–CH), 128.1 (2 × Ar–CH), 128.0, 126.4, 122.2, 115.2 (2 × Ar–CH), 107.7, 55.4 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{16}ClO_3S^+$ $[M+H]^+$: 395.0503, found: 395.0494.



7-Chloro-4-(isopropylthio)-3-phenyl-1*H*-isochromen-1-one (3ld):

GP3 was carried out with methyl 5-chloro-2-(phenylethynyl)benzoate **1l** (68 mg, 0.25 mmol), 1,2-diisopropyldisulfane **2d** (45 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3ld** (69 mg, 84%), as a pale yellow solid , mp = 78–80 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1l**) = 0.60, R_f (**2d**) = 0.90, R_f (**3ld**) = 0.60, UV detection].

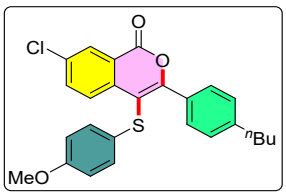
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2922, 2865, 1741, 1594, 1463, 1317, 1215, 1083, 771, 696 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.29 (d, J = 2.2 Hz, 1H), 8.24 (d, J = 8.7 Hz, 1H), 7.82 – 7.72 (m, 3H), 7.47 – 7.41 (m, 3H), 2.88 (hept, J = 6.7 Hz, 1H), 1.04 (s, 3H), 1.02 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.6, 159.1, 137.8, 135.4, 134.5, 133.1, 130.3 (2 × Ar–CH), 130.1, 129.1, 128.1, 127.9 (2 × Ar–CH), 121.9, 109.0, 39.6, 22.9 (2 × $-CH_3$) ppm. **HRMS:** *m/z* calcd for $C_{18}H_{16}ClO_2S^+$ $[M+H]^+$: 331.0554, found: 331.0533.



7-Chloro-4-((4-methoxyphenyl)thio)-3-(*p*-tolyl)-1*H*-isochromen-1-one (3mc):

GP3 was carried out with methyl 5-chloro-2-(*p*-tolylethynyl)benzoate **1m** (71 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3mc** (80 mg, 78%), as a yellow solid , mp = 158–160 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1m**) = 0.60, R_f (**2c**) = 0.50, R_f (**3mc**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2954, 2842, 1738, 1595, 1480, 1250, 1083, 1029, 820, 739, 672 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.30 (d, J = 2.2 Hz, 1H), 7.95 (d, J = 8.7 Hz, 1H), 7.67 (d, J = 8.2 Hz, 2H), 7.63 (dd, J = 8.7, 2.3 Hz, 1H), 7.23 (d, J = 8.0 Hz, 2H), 7.04 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.40 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.6, 160.1, 158.5, 141.0, 136.9, 135.6, 134.6, 131.0, 129.7 (2 × Ar–CH), 129.2, 128.8 (2 × Ar–CH), 128.5 (2 × Ar–CH), 128.0, 126.6, 122.2, 115.2 (2 × Ar–CH), 107.1, 55.5, 21.7 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{18}ClO_3S^+$ $[M+H]^+$: 409.0660, found: 409.0654.

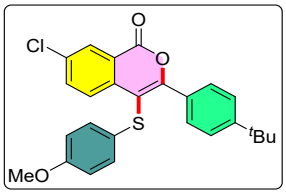


3-(4-Butylphenyl)-7-chloro-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3nc):

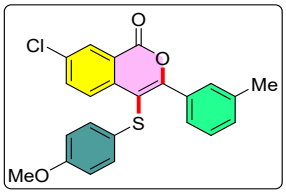
GP3 was carried out with methyl 2-((4-butylphenyl)ethynyl)-5-chlorobenzoate **1n** (82 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3nc** (84 mg, 75%), as a yellow solid , mp = 100–102 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1n**) = 0.60, R_f (**2c**) = 0.50, R_f (**3nc**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2927, 2860, 1738, 1593, 1247, 1080, 1025, 816, 737, 696 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.29 (d, J = 2.2 Hz, 1H), 7.96 (d, J = 8.7 Hz, 1H), 7.69 (d, J = 8.3 Hz, 2H), 7.63 (dd, J = 8.7, 2.3 Hz, 1H), 7.24 (d, J = 8.3 Hz, 2H), 7.05 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.66 (t, J = 8 Hz, 2H), 1.66 – 1.58 (m, 2H),

1.41 – 1.32 (m, 2H), 0.93 (t, J = 7.3 Hz, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.6, 160.1, 158.5, 145.9, 136.9, 135.6, 134.5, 129.9, 129.7 (2 × Ar–CH), 129.2, 128.6 (2 × Ar–CH), 128.2 (2 × Ar–CH), 128.0, 126.6, 122.2, 115.2 (2 × Ar–CH), 107.0, 55.4, 35.7, 33.4, 22.5, 14.1 ppm. **HRMS:** *m/z* calcd for $C_{26}H_{24}ClO_3S^+$ $[M+H]^+$: 451.1129, found: 451.1126.



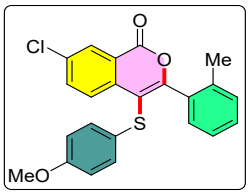
3-(4-(*tert*-Butyl)phenyl)-7-chloro-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3oc):
GP3 was carried out with methyl 2-((4-(*tert*-butyl)phenyl)ethynyl)-5-chlorobenzoate **1o** (82 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3oc** (96 mg, 85%), as a colorless solid , mp = 146–148 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1o**) = 0.60, R_f (**2c**) = 0.50, R_f (**3oc**) = 0.40, UV detection].
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3058, 2960, 1739, 1595, 1479, 1250, 1081, 1028, 826, 733, 670 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 8.29 (d, J = 2.2 Hz, 1H), 7.95 (d, J = 8.7 Hz, 1H), 7.74 (d, J = 8.5 Hz, 2H), 7.62 (dd, J = 8.7, 2.3 Hz, 1H), 7.45 (d, J = 8.6 Hz, 2H), 7.06 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 1.35 (s, 9H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.5, 159.9, 158.5, 153.9, 136.9, 135.5, 134.5, 129.6, 129.5 (2 × Ar–CH), 129.1, 128.4 (2 × Ar–CH), 127.9, 126.6, 125.1 (2 × Ar–CH), 122.2, 115.2 (2 × Ar–CH), 106.9, 55.4, 35.0, 31.3 ppm. **HRMS:** *m/z* calcd for $C_{26}H_{24}ClO_3S^+$ $[M+H]^+$: 451.1129, found: 451.1128.



7-Chloro-4-((4-methoxyphenyl)thio)-3-(*m*-tolyl)-1*H*-isochromen-1-one (3pc):
GP3 was carried out with methyl 5-chloro-2-(*m*-tolylethynyl)benzoate **1p** (71 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3pc** (84 mg, 82%), as a colorless

solid , mp = 118–120 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1p**) = 0.60, R_f (**2c**) = 0.50, R_f (**3pc**) = 0.40, UV detection].

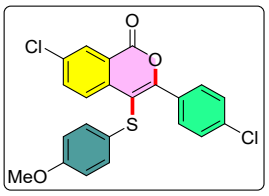
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2932, 2840, 1737, 1589, 1480, 1241, 1086, 1036, 832, 787, 701 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.30 (d, J = 2.3 Hz, 1H), 7.97 (d, J = 8.7 Hz, 1H), 7.64 (dd, J = 8.7, 2.3 Hz, 1H), 7.57 – 7.50 (m, 2H), 7.33 – 7.26 (m, 2H), 7.04 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.38 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 160.5, 160.1, 158.6, 137.9, 136.8, 135.6, 134.7, 132.5, 131.4, 130.2, 129.2, 128.7 (2 × Ar–CH), 128.1, 127.9, 126.9, 126.6, 122.2, 115.2 (2 × Ar–CH), 107.6, 55.4, 21.5 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{18}ClO_3S^+$ $[M+H]^+$: 409.0660, found: 409.0658.



7-Chloro-4-((4-methoxyphenyl)thio)-3-(*o*-tolyl)-1*H*-isochromen-1-one (3qc):

GP3 was carried out with methyl 5-chloro-2-(*o*-tolylethynyl)benzoate **1q** (71 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3qc** (81 mg, 79%), as a brown solid , mp = 148–150 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1q**) = 0.60, R_f (**2c**) = 0.50, R_f (**3qc**) = 0.40, UV detection].

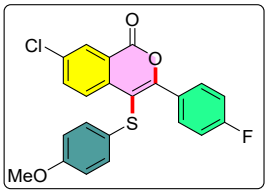
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3066, 2951, 2842, 1739, 1599, 1483, 1212, 1072, 1037, 827, 764 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.32 (d, J = 2.2 Hz, 1H), 8.00 (d, J = 8.6 Hz, 1H), 7.68 (dd, J = 8.6, 2.3 Hz, 1H), 7.40 – 7.36 (m, 1H), 7.33 (dd, J = 10.9, 1.3 Hz, 1H), 7.31 – 7.27 (m, 1H), 7.22 (t, J = 7.5 Hz, 1H), 6.96 (d, J = 8.9 Hz, 2H), 6.72 (d, J = 8.9 Hz, 2H), 3.73 (s, 3H), 2.26 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.7, 160.5, 158.8, 137.3, 136.5, 135.6, 134.9, 132.7, 130.4, 130.3, 129.9, 129.8 (2 × Ar–CH), 129.4, 127.9, 125.9, 125.5, 122.4, 114.9 (2 × Ar–CH), 110.1, 55.5, 19.9 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{18}ClO_3S^+$ $[M+H]^+$: 409.0660, found: 409.0658.



7-Chloro-3-(4-chlorophenyl)-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3rc):

GP3 was carried out with methyl 5-chloro-2-((4-chlorophenyl)ethynyl)benzoate **1r** (76 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3rc** (77 mg, 72%), as a colorless solid , mp = 174–176 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1r**) = 0.60, R_f (**2c**) = 0.50, R_f (**3rc**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2958, 2836, 1736, 1586, 1476, 1248, 1078, 1015, 804, 710 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.29 (d, J = 1.8 Hz, 1H), 7.97 (d, J = 8.6 Hz, 1H), 7.71 (d, J = 8.6 Hz, 2H), 7.65 (dd, J = 8.7, 2.3 Hz, 1H), 7.40 (d, J = 8.6 Hz, 2H), 7.03 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.2, 158.7, 158.6, 136.7, 136.5, 135.7, 135.0, 131.1 (2 × Ar–CH), 130.9, 129.3, 128.6 (2 × Ar–CH), 128.4 (2 × Ar–CH), 128.1, 126.1, 122.2, 115.3 (2 × Ar–CH), 108.1, 55.5 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{15}Cl_2O_3S^+$ $[M+H]^+$: 429.0113, found: 429.0123.

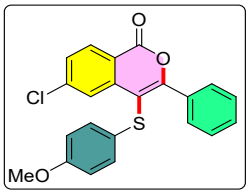


7-Chloro-3-(4-fluorophenyl)-4-((4-methoxyphenyl)thio)-1*H*-isochromen-1-one (3sc):

GP3 was carried out with methyl 5-chloro-2-((4-fluorophenyl)ethynyl)benzoate **1s** (72 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3sc** (82 mg, 80%), as a yellow solid , mp = 144–146 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1s**) = 0.60, R_f (**2c**) = 0.50, R_f (**3sc**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2924, 2851, 1741, 1599, 1496, 1236, 1169, 1084, 1034, 832, 790 cm^{-1}. **^{1}H NMR** (600 MHz, $CDCl_3$) δ = 8.30 (d, J = 2.2 Hz, 1H), 7.97 (d, J = 8.6 Hz, 1H), 7.79 – 7.75 (m, 2H), 7.65 (dd, J = 8.7, 2.3 Hz, 1H), 7.14 – 7.08 (m, 2H), 7.02 (d, J = 9.0 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ =

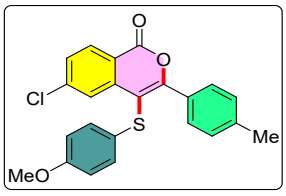
163.9 (d, $J_{C\text{-}F}$ = 251.8 Hz), 160.3, 158.8, 158.7, 136.7, 135.7, 134.9, 131.9 (d, $J_{C\text{-}F}$ = 8.6 Hz, (2 × Ar–CH)), 129.3, 128.7 (d, $J_{C\text{-}F}$ = 3.4 Hz), 128.6 (2 × Ar–CH), 128.1, 126.2, 122.2, 115.3 (d, $J_{C\text{-}F}$ = 22.2 Hz, (2 × Ar–CH)), 115.3 (2 × Ar–CH), 107.7, 55.5 ppm. **^{19}F NMR** (376 MHz, $CDCl_3$) δ = –108.9 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{15}ClFO_3S^+$ $[M+H]^+$: 413.0409, found: 413.0420.



6-Chloro-4-((4-methoxyphenyl)thio)-3-phenyl-1*H*-isochromen-1-one (3tc):

GP3 was carried out with methyl 4-chloro-2-(phenylethynyl)benzoate **1t** (68 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3tc** (73 mg, 74%), as a yellow solid , mp = 108–110 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1t**) = 0.60, R_f (**2c**) = 0.50, R_f (**3tc**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2926, 2846, 1738, 1594, 1489, 1458, 1294, 1243, 1177, 1083, 1033, 825, 769, 695 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.27 (d, J = 8.4 Hz, 1H), 8.05 (d, J = 2.0 Hz, 1H), 7.75 (dd, J = 8.1, 1.5 Hz, 2H), 7.50 – 7.40 (m, 4H), 7.05 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.75 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 160.99, 160.9, 158.7, 142.5, 140.0, 132.7, 131.6, 130.6, 129.8 (2 × Ar–CH), 129.3, 128.9 (2 × Ar–CH), 128.1 (2 × Ar–CH), 126.3, 126.0, 119.3, 115.3 (2 × Ar–CH), 107.6, 55.5 ppm. **HRMS:** *m/z* calcd for $C_{22}H_{16}ClO_3S^+$ $[M+H]^+$: 395.0503, found: 395.0500.

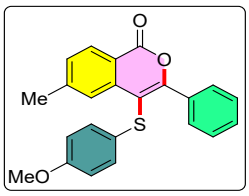


6-Chloro-4-((4-methoxyphenyl)thio)-3-(*p*-tolyl)-1*H*-isochromen-1-one (3uc):

GP3 was carried out with methyl 4-chloro-2-(*p*-tolylethynyl)benzoate **1u** (71 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography

(petroleum ether/ethyl acetate, 95:05) furnished the product **3uc** (74 mg, 72%), as a colorless solid , mp = 148–150 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1u**) = 0.60, R_f (**2c**) = 0.50, R_f (**3uc**) = 0.40, UV detection].

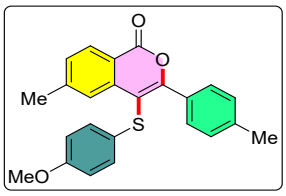
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2932, 2843, 1737, 1594, 1291, 1243, 1085, 1033, 825, 737 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.25 (d, J = 8.4 Hz, 1H), 8.03 (d, J = 2.0 Hz, 1H), 7.67 (d, J = 8.2 Hz, 2H), 7.46 (dd, J = 8.5, 2.0 Hz, 1H), 7.23 (d, J = 7.9 Hz, 2H), 7.06 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.40 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.2, 160.9, 158.6, 142.4, 141.0, 140.1, 131.5, 129.8, 129.7 (2 × Ar–CH), 129.1, 128.8 (2 × Ar–CH), 128.7 (2 × Ar–CH), 126.4, 125.9, 119.2, 115.2 (2 × Ar–CH), 106.9, 55.4, 21.6 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{17}ClO_3S^+$ $[M]^+$: 408.0581, found: 408.0582.



4-((4-Methoxyphenyl)thio)-6-methyl-3-phenyl-1*H*-isochromen-1-one (3vc):

GP3 was carried out with methyl 4-methyl-2-(phenylethynyl)benzoate **1v** (62 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3vc** (79 mg, 84%), as a yellow solid , mp = 120–122 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1v**) = 0.60, R_f (**2c**) = 0.50, R_f (**3vc**) = 0.40, UV detection].

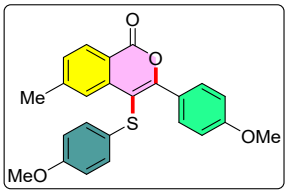
IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 3057, 2927, 2842, 1732, 1603, 1487, 1240, 1081, 1032, 823, 767, 689 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.24 (d, J = 8.0 Hz, 1H), 7.84 (s, 1H), 7.73 (dd, J = 8.0, 1.6 Hz, 2H), 7.47 – 7.38 (m, 3H), 7.35 (dd, J = 8.1, 1.1 Hz, 1H), 7.05 (d, J = 8.9 Hz, 2H), 6.77 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.44 (s, 3H) ppm. **^{13}C{H} NMR** (101 MHz, $CDCl_3$) δ = 161.7, 159.9, 158.4, 146.6, 138.3, 133.1, 130.3, 130.1, 129.9, 129.7 (2 × Ar–CH), 128.5 (2 × Ar–CH), 128.0 (2 × Ar–CH), 127.2, 126.2, 118.6, 115.1 (2 × Ar–CH), 55.4, 22.5 ppm. **HRMS:** *m/z* calcd for $C_{23}H_{19}O_3S^+$ $[M+H]^+$: 375.1049, found: 375.1039.



4-((4-Methoxyphenyl)thio)-6-methyl-3-(*p*-tolyl)-1*H*-isochromen-1-one (3wc):

GP3 was carried out with methyl 4-methyl-2-(*p*-tolylethynyl)benzoate **1w** (66 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3wc** (78 mg, 80%), as a colorless solid , mp = 136–138 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1w**) = 0.60, R_f (**2c**) = 0.50, R_f (**3wc**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2924, 2852, 1736, 1604, 1492, 1179, 1082, 1034, 825, 780, 729, 682 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.23 (d, J = 8.0 Hz, 1H), 7.82 (s, 1H), 7.65 (d, J = 8.2 Hz, 2H), 7.33 (dd, J = 8.1, 1.0 Hz, 1H), 7.21 (d, J = 8.0 Hz, 2H), 7.06 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.74 (s, 3H), 2.43 (s, 3H), 2.39 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.8, 160.2, 158.3, 146.6, 140.6, 138.4, 130.2, 129.99, 129.9, 129.6 (2 × Ar–CH), 128.7 (2 × Ar–CH), 128.3 (2 × Ar–CH), 127.3, 126.1, 118.5, 115.1 (2 × Ar–CH), 107.3, 55.4, 22.5, 21.6 ppm. **HRMS:** *m/z* calcd for $C_{24}H_{21}O_3S^+$ $[M+H]^+$: 389.1206, found: 389.1196.

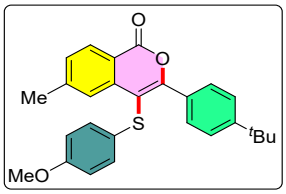


3-(4-Methoxyphenyl)-4-((4-methoxyphenyl)thio)-6-methyl-1*H*-isochromen-1-one (3xc):

GP3 was carried out with methyl 2-((4-methoxyphenyl)ethynyl)-4-methylbenzoate **1x** (70 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3xc** (83 mg, 82%), as a yellow solid , mp = 140–142 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1x**) = 0.50, R_f (**2c**) = 0.50, R_f (**3xc**) = 0.20, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2927, 2844, 1732, 1602, 1492, 1246, 1176, 1083, 1032, 830, 779, 677 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.22 (d, J = 8.0 Hz, 1H), 7.81 (s, 1H), 7.74 (d, J = 9.0 Hz, 2H), 7.32 (dd, J = 8.1, 1.0 Hz, 1H), 7.06 (d, J = 8.9 Hz, 2H), 6.92 (d, J =

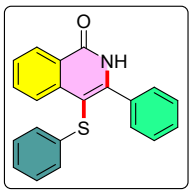
9.0 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.84 (s, 3H), 3.74 (s, 3H), 2.42 (s, 3H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.8, 161.1, 159.9, 158.3, 146.6, 138.6, 131.4 (2 × Ar–CH), 129.8 (2 × Ar–CH), 128.2 (2 × Ar–CH), 127.4, 126.0, 125.4, 118.4, 115.1 (2 × Ar–CH), 113.4 (2 × Ar–CH), 106.7, 55.5, 55.4, 22.5 ppm. **HRMS:** *m/z* calcd for $C_{24}H_{21}O_4S^+$ $[M+H]^+$: 405.1155, found: 405.1145.



3-(4-(*tert*-Butyl)phenyl)-4-((4-methoxyphenyl)thio)-6-methyl-1*H*-isochromen-1-one (3yc):

GP3 was carried out with methyl 2-((4-(*tert*-butyl)phenyl)ethynyl)-4-methylbenzoate **1y** (77 mg, 0.25 mmol), 1,2-bis(4-methoxyphenyl)disulfane **2c** (83 mg, 0.30 mmol), $LiClO_4$ (96 mg, 0.15 M) and CH_3CN (6 mL) for 3 h. Purification of the crude material by silica-gel column chromatography (petroleum ether/ethyl acetate, 95:05) furnished the product **3yc** (84 mg, 78%), as a colorless solid , mp = 138–140 °C. [TLC control (petroleum ether/ethyl acetate 95:05), R_f (**1y**) = 0.60, R_f (**2c**) = 0.50, R_f (**3yc**) = 0.40, UV detection].

IR: (MIR-ATR, 4000–600 cm^{-1}) ν_{max} = 2957, 2864, 1732, 1602, 1484, 1280, 1238, 1079, 1030, 829, 734, 680 cm^{-1}. **^{1}H NMR** (400 MHz, $CDCl_3$) δ = 8.23 (d, J = 8.0 Hz, 1H), 7.82 (s, 1H), 7.71 (d, J = 8.7 Hz, 2H), 7.43 (d, J = 8.7 Hz, 2H), 7.33 (dd, J = 8.1, 1.1 Hz, 1H), 7.07 (d, J = 8.9 Hz, 2H), 6.78 (d, J = 8.9 Hz, 2H), 3.75 (s, 3H), 2.42 (s, 3H), 1.34 (s, 9H) ppm. **^{13}C{H} NMR** (151 MHz, $CDCl_3$) δ = 161.7, 160.0, 158.3, 153.6, 146.5, 138.4, 130.2, 129.96, 129.9, 129.4 (2 × Ar–CH), 128.3 (2 × Ar–CH), 127.4, 126.1, 125.0 (2 × Ar–CH), 118.5, 115.1 (2 × Ar–CH), 107.2, 55.4, 35.0, 31.3, 22.5 ppm. **HRMS:** *m/z* calcd for $C_{27}H_{27}O_3S^+$ $[M+H]^+$: 431.1675, found: 431.1666.

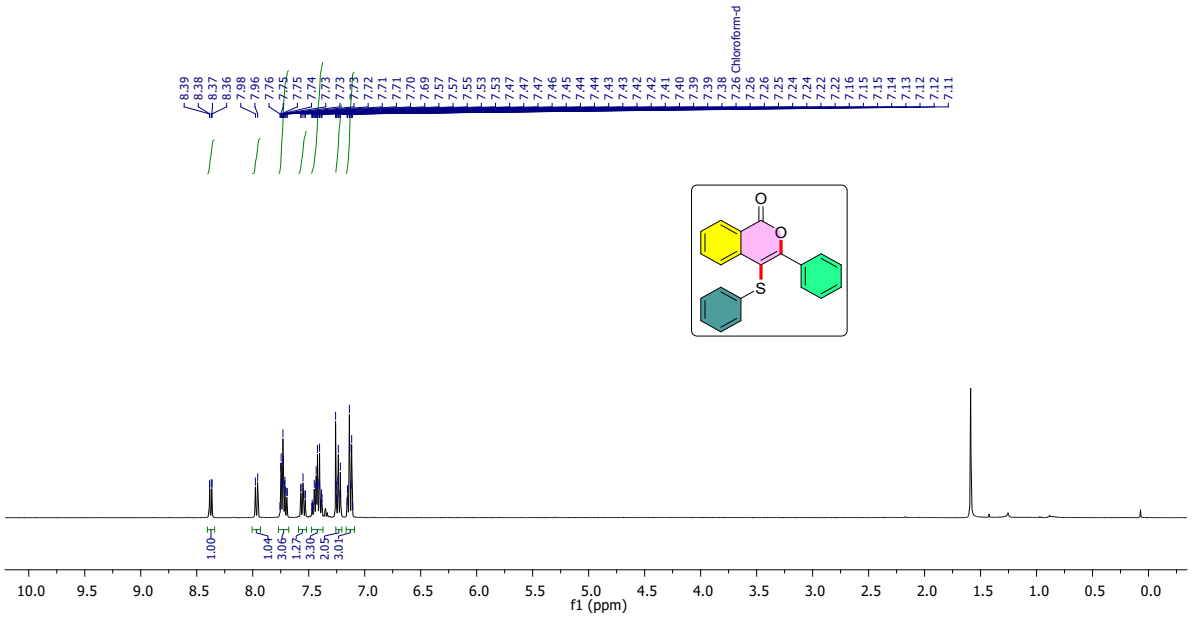


3-Phenyl-4-(phenylthio)isoquinolin-1(2*H*)-one (7):

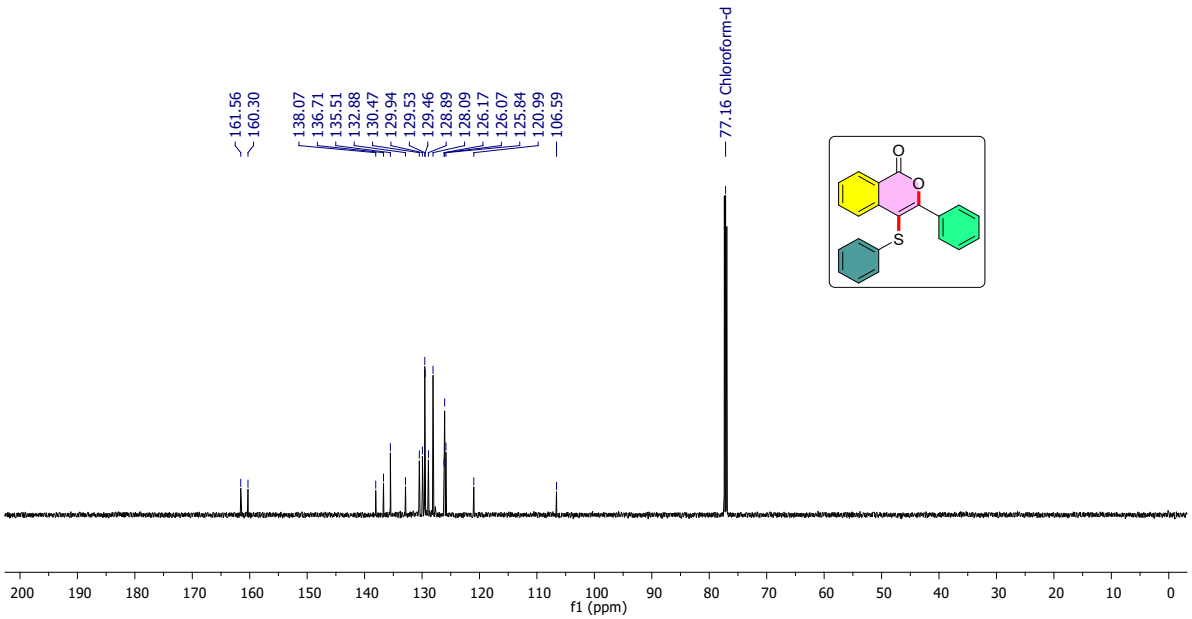
To an oven-dried 10 mL round bottom flask equipped with a magnetic stir bar, were added 3-phenyl-4-(phenylthio)-1*H*-isochromen-1-one **3aa** (50 mg, 1.0 equiv), ammonium acetate (18 mg, 1.5 equiv), and DMSO (3 mL). The mixture was stirred at 100 °C for overnight under an argon atmosphere. Purification of the residue by silica-gel column chromatography (petroleum ether/ethyl acetate, 60:40) furnished the product **7** (37 mg, 75% yields), as a colorless solid, mp = 310-312 °C. [TLC control (petroleum ether/ethyl acetate 60:40), R_f (**3aa**) = 0.98, R_f (**7**) = 0.50, UV detection]. **^{1}H NMR** (400 MHz, DMSO-d_6) δ = 11.97 (s, 1H), 8.30 (dd, J = 8.0, 0.9 Hz, 1H), 7.93 (d, J = 7.9 Hz, 1H), 7.75 – 7.68 (m, 1H), 7.57 – 7.52 (m, 1H), 7.49 – 7.38 (m, 5H), 7.21 (dd, J = 10.6, 4.7 Hz, 2H), 7.12 – 6.99 (m, 3H) ppm. **^{13}C{H} NMR** (101 MHz, DMSO-d_6) δ = 161.8, 149.0, 138.0, 137.8, 134.7, 133.3, 129.3, 129.2 (2 × Ar–CH), 129.1 (2 × Ar–CH), 127.8 (2 × Ar–CH), 127.2, 126.9, 125.9, 125.2, 125.2 (2 × Ar–CH), 125.1, 101.8 ppm. **HRMS:** *m/z* calcd for $C_{21}H_{16}NOS^+$ $[M+H]^+$: 330.0947, found: 330.0917.

NMR Spectra:

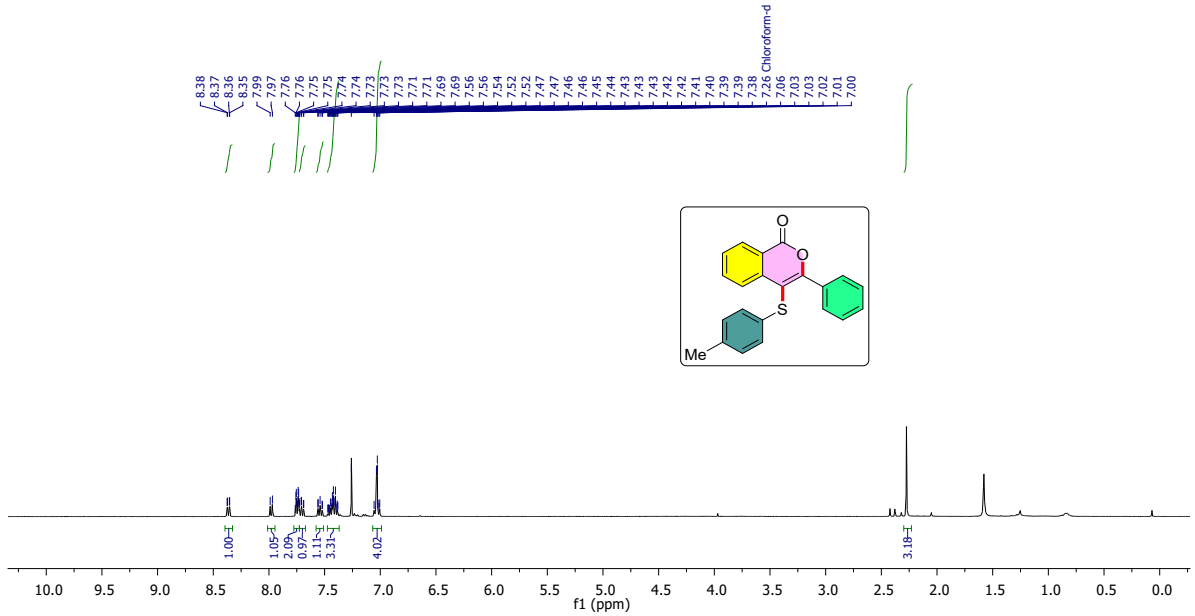
^{1}H-NMR (400 MHz) spectrum of **3aa** in $CDCl_3$



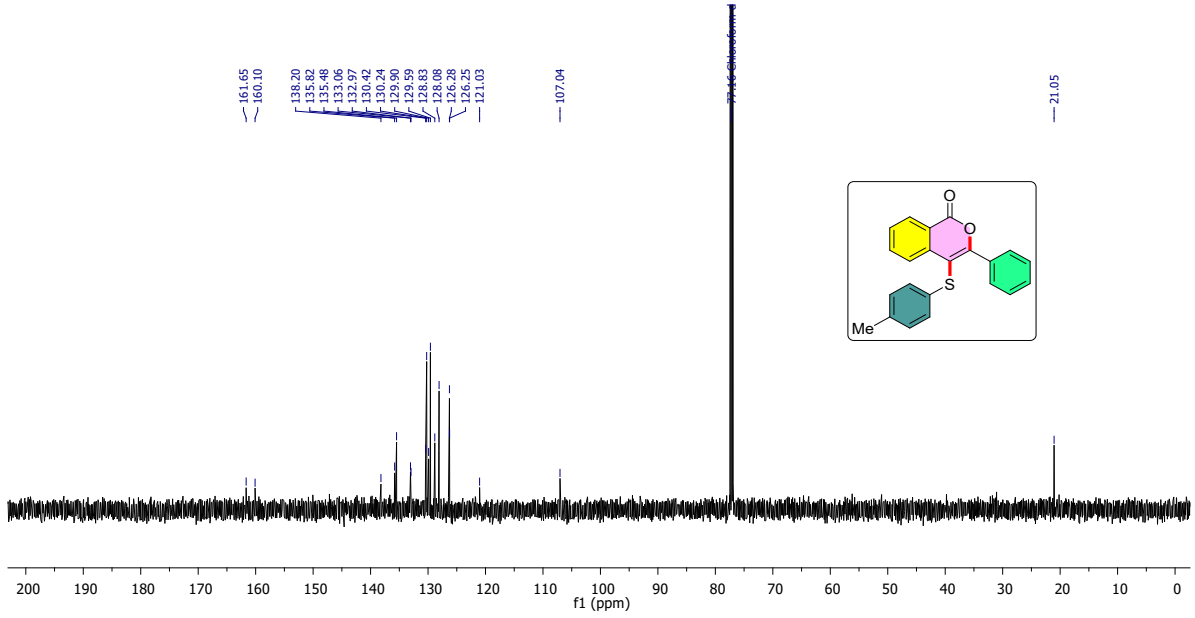
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3aa** in $CDCl_3$



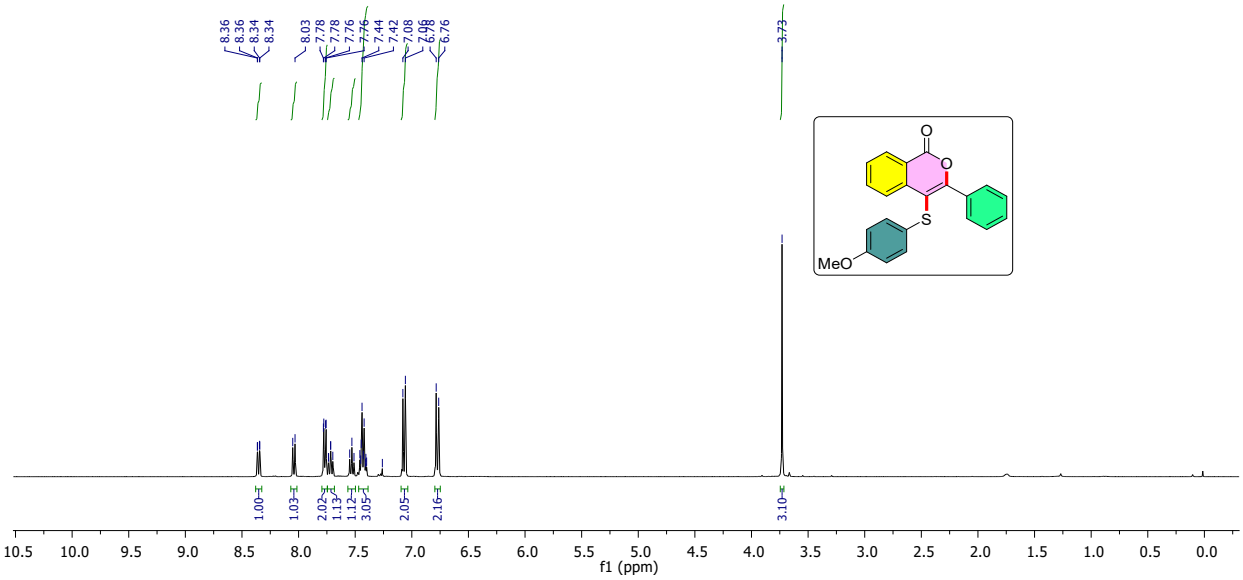
^{1}H-NMR (400 MHz) spectrum of **3ab** in $CDCl_3$



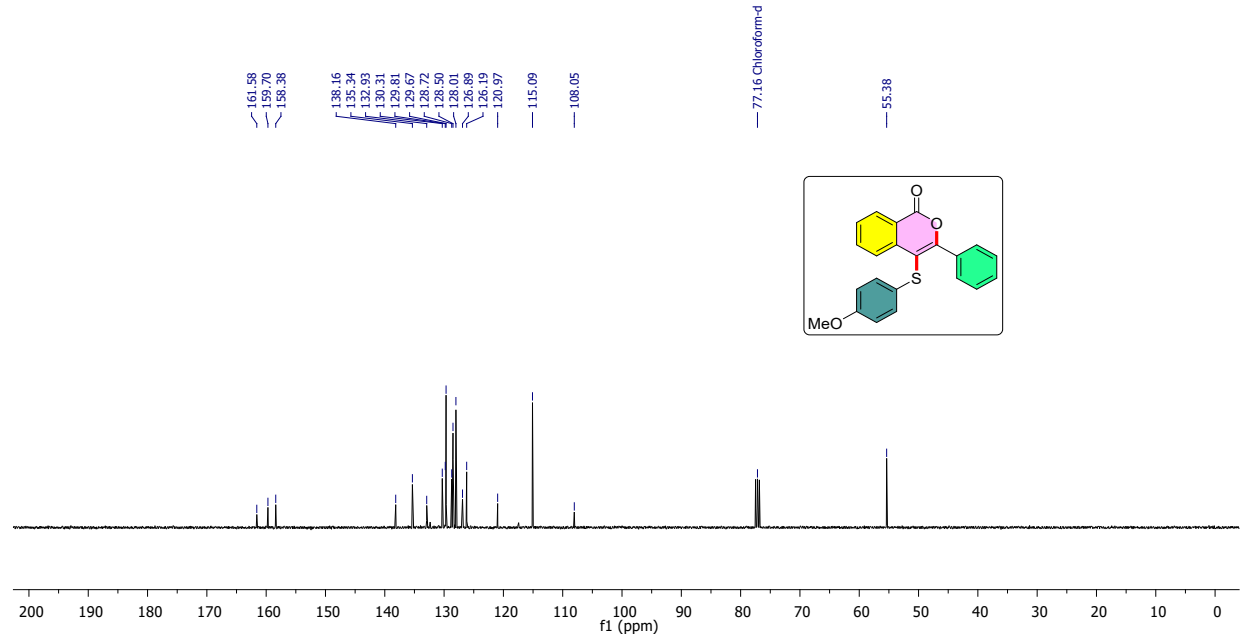
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3ab** in $CDCl_3$



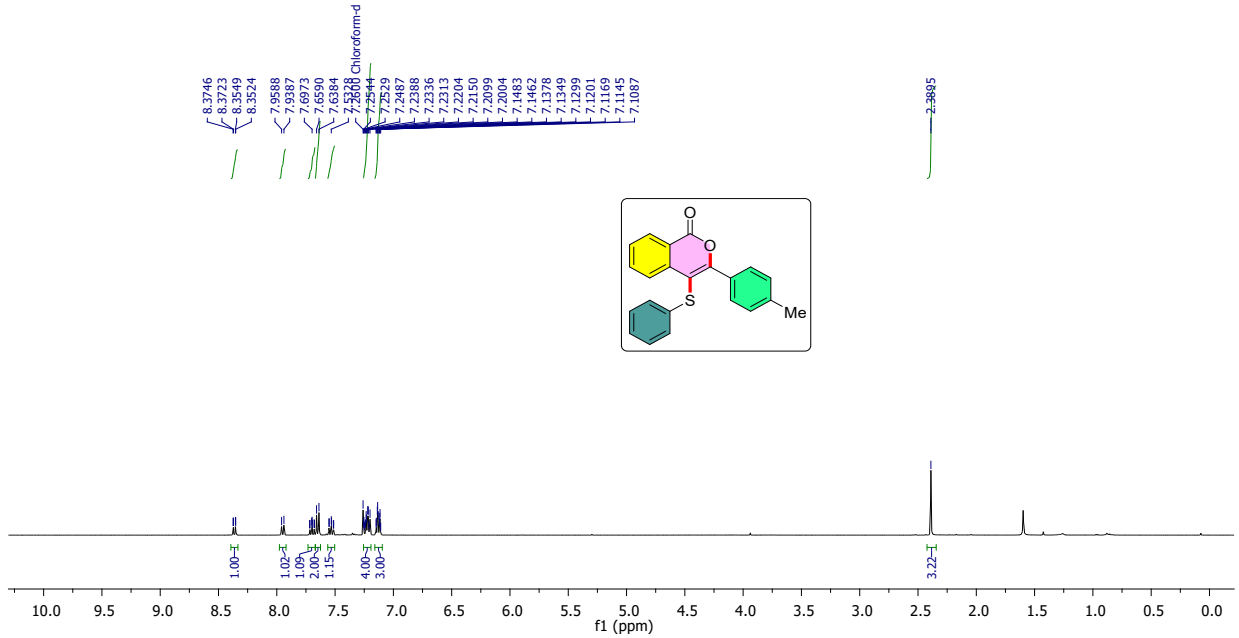
^{1}H-NMR (400 MHz) spectrum of **3ac** in $CDCl_3$



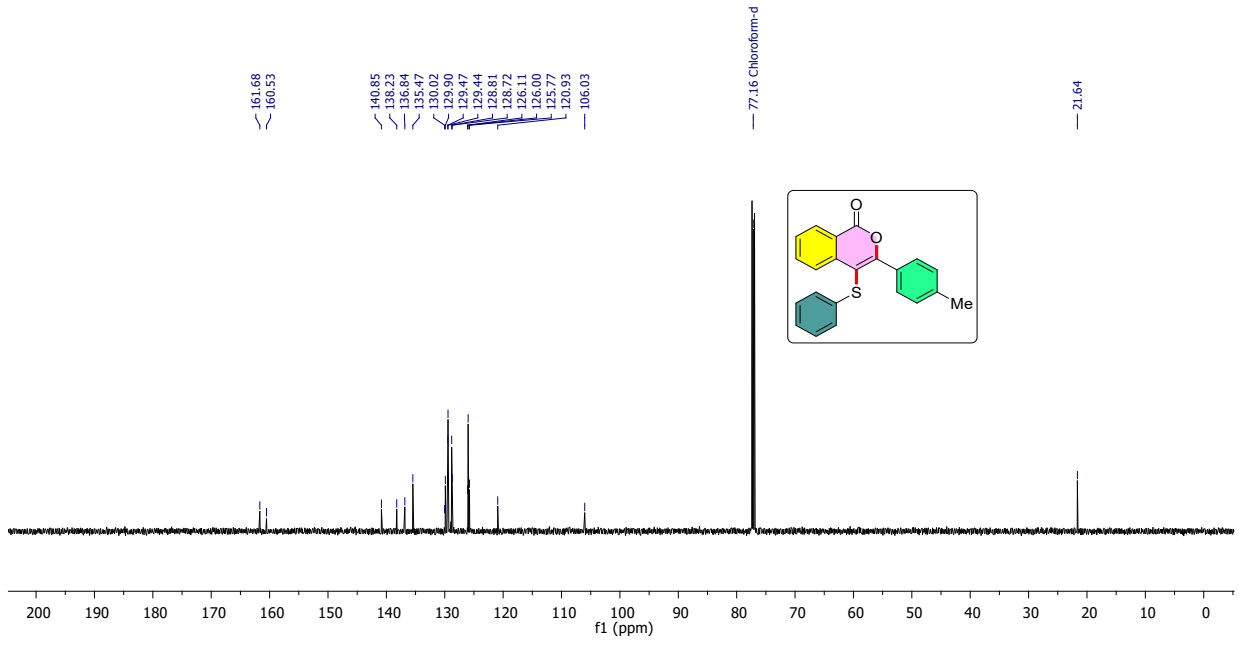
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3ac** in $CDCl_3$



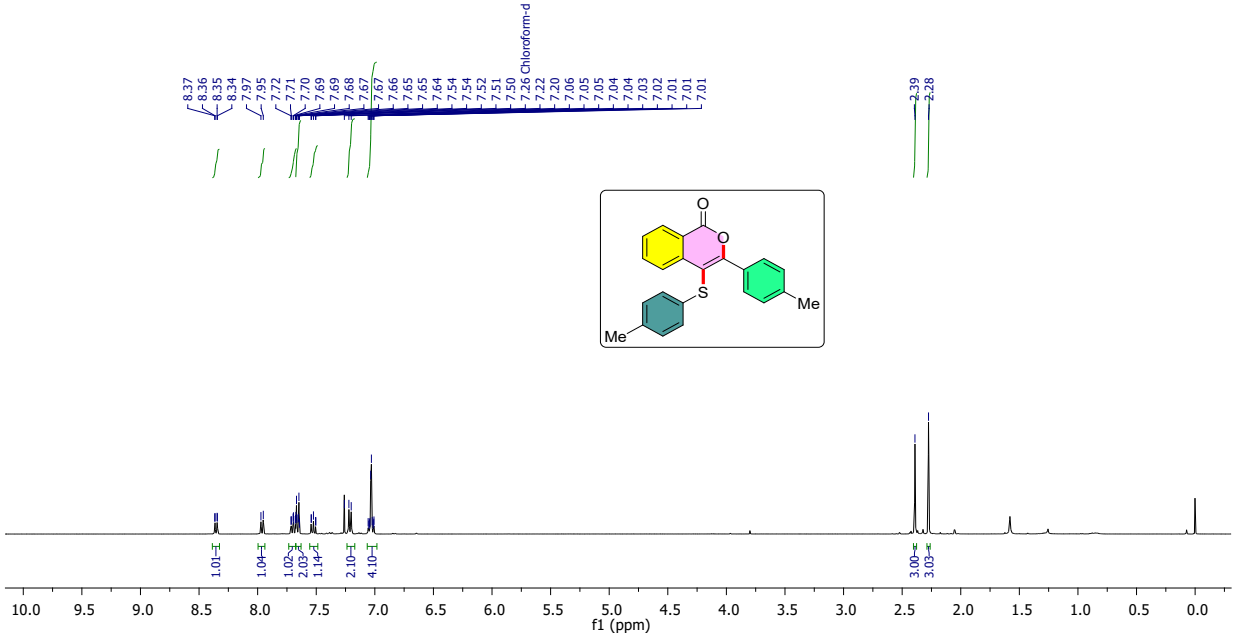
^{1}H-NMR (400 MHz) spectrum of **3ba** in $CDCl_3$



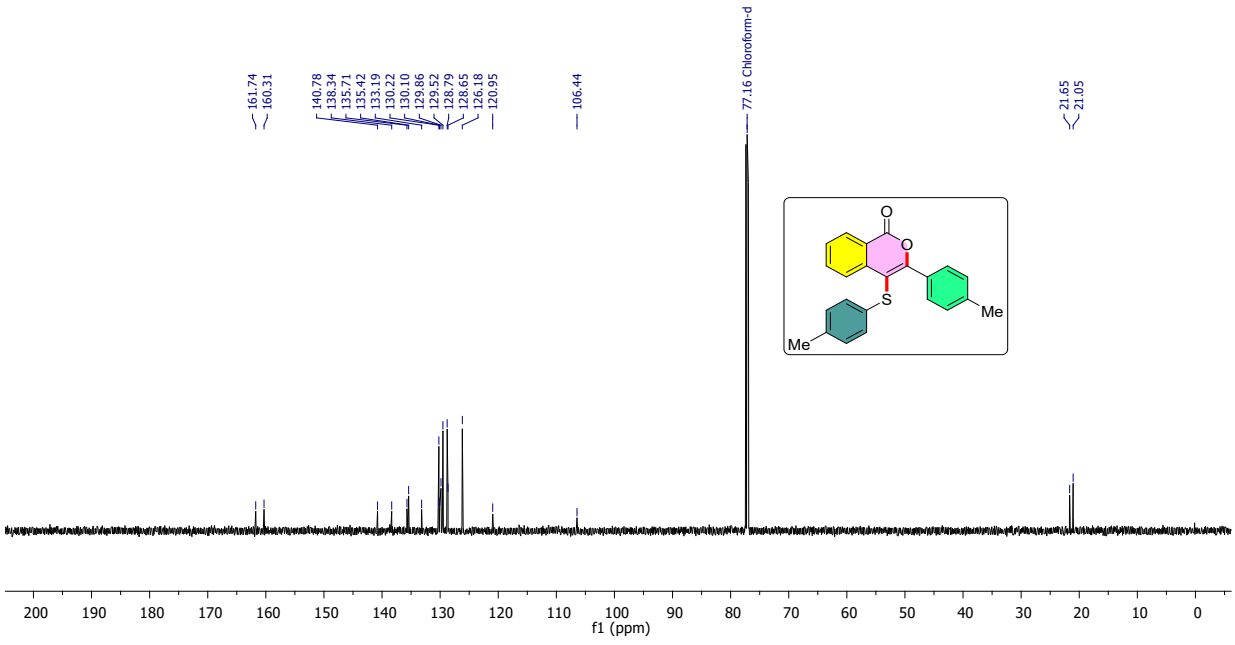
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3ba** in $CDCl_3$



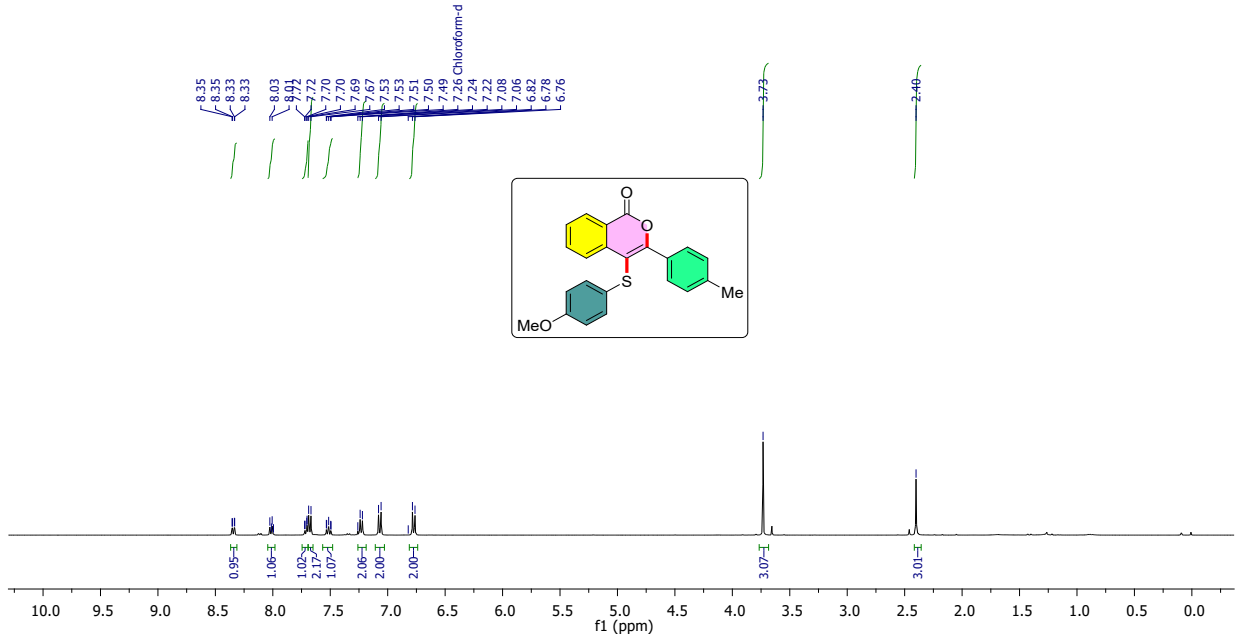
^{1}H-NMR (400 MHz) spectrum of **3bb** in $CDCl_3$



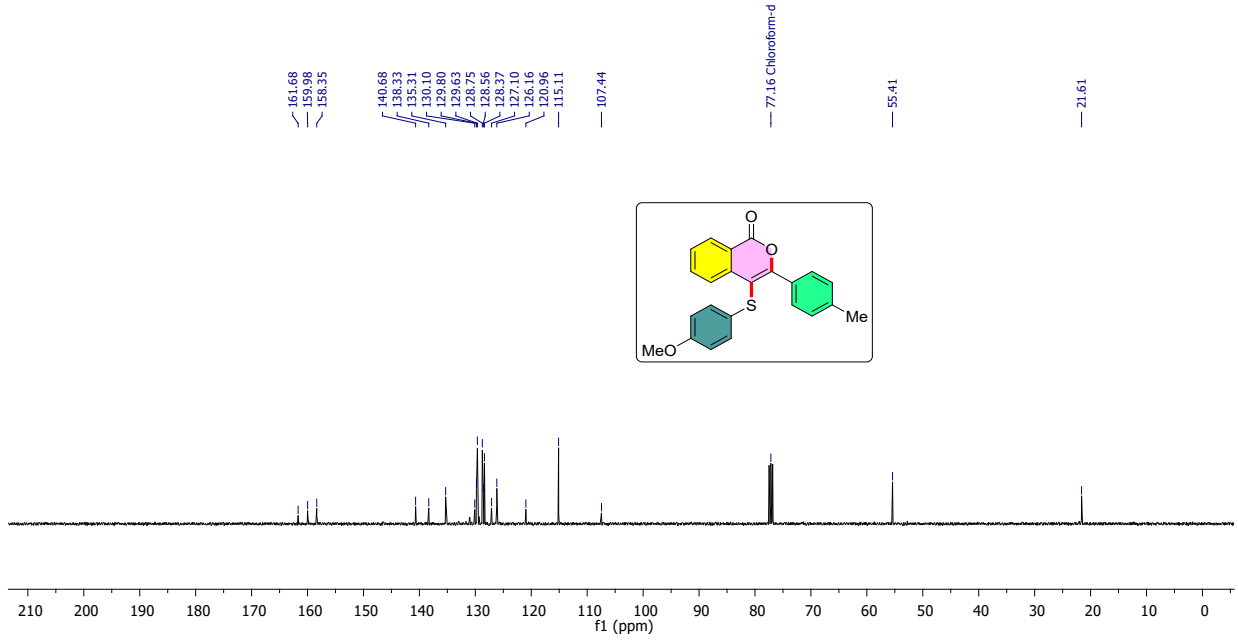
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3bb** in $CDCl_3$



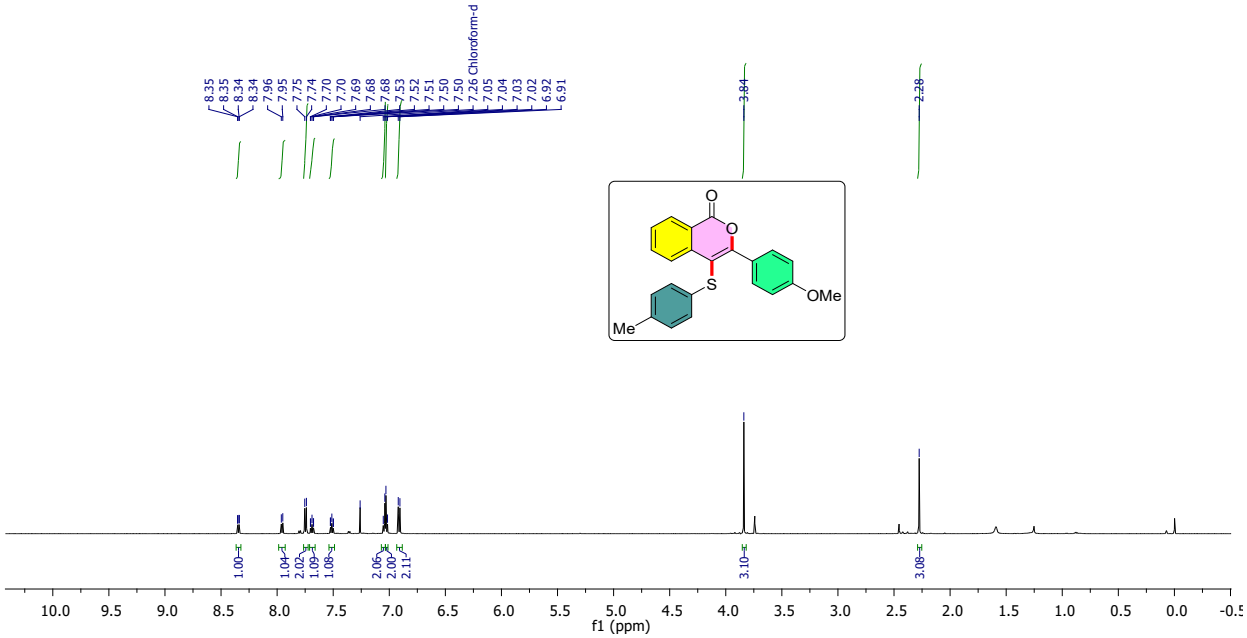
^{1}H-NMR (400 MHz) spectrum of **3bc** in $CDCl_3$



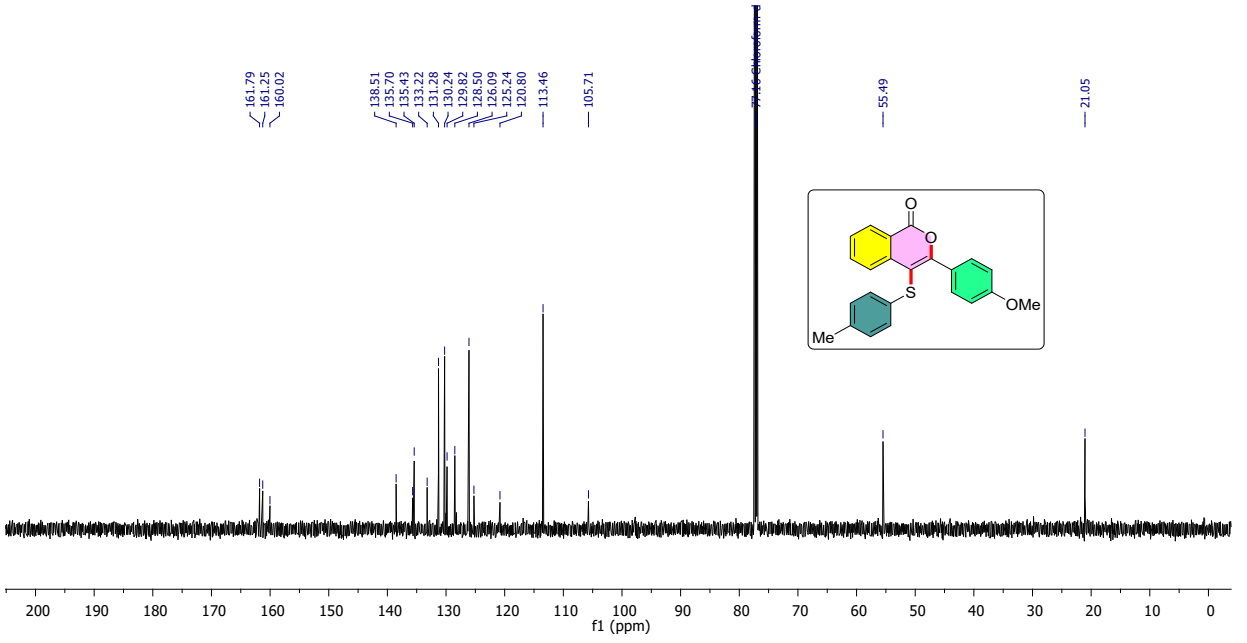
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3bc** in $CDCl_3$



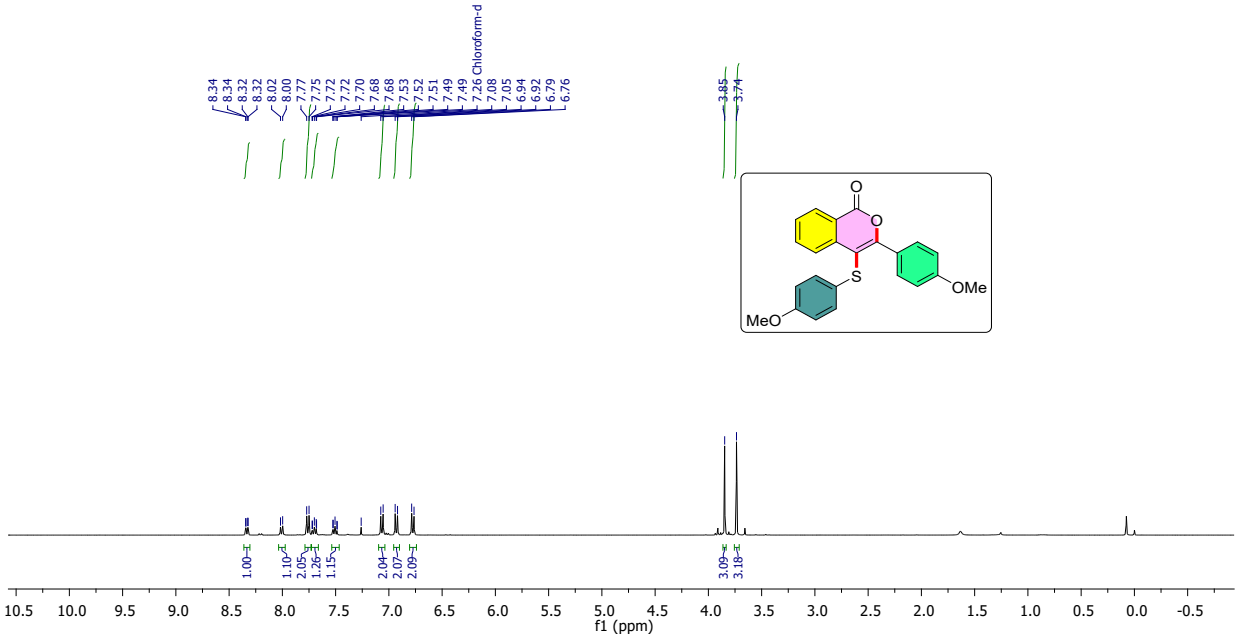
^{1}H-NMR (600 MHz) spectrum of **3cb** in $CDCl_3$



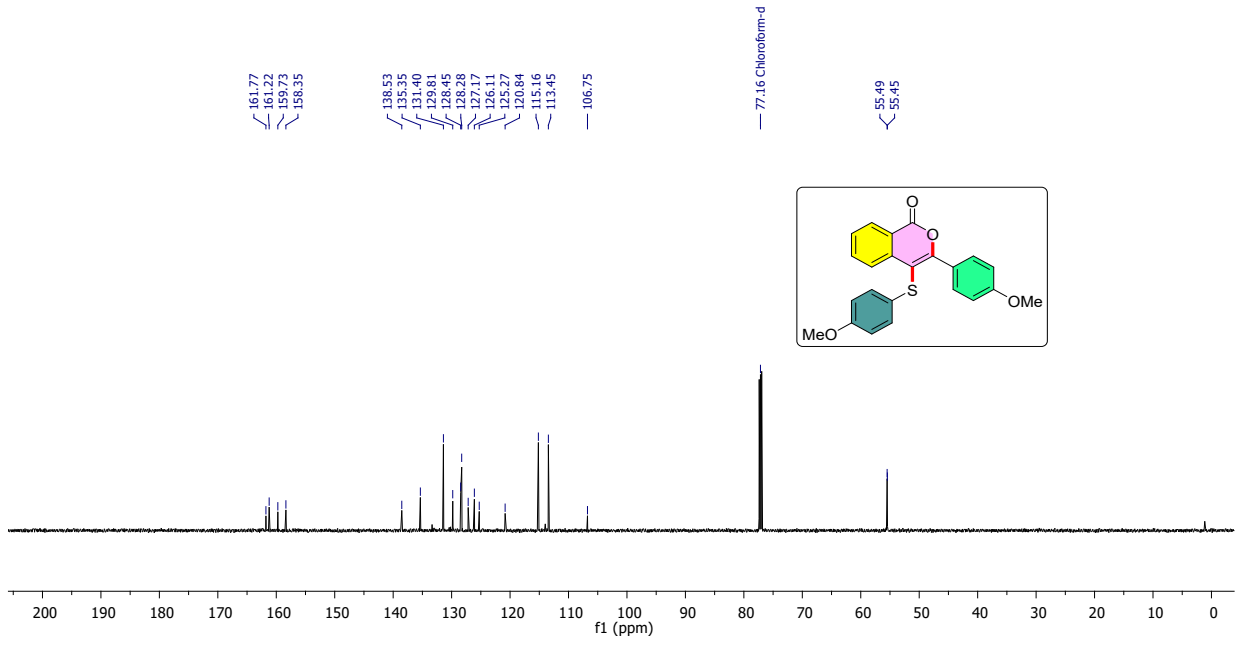
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3cb** in $CDCl_3$



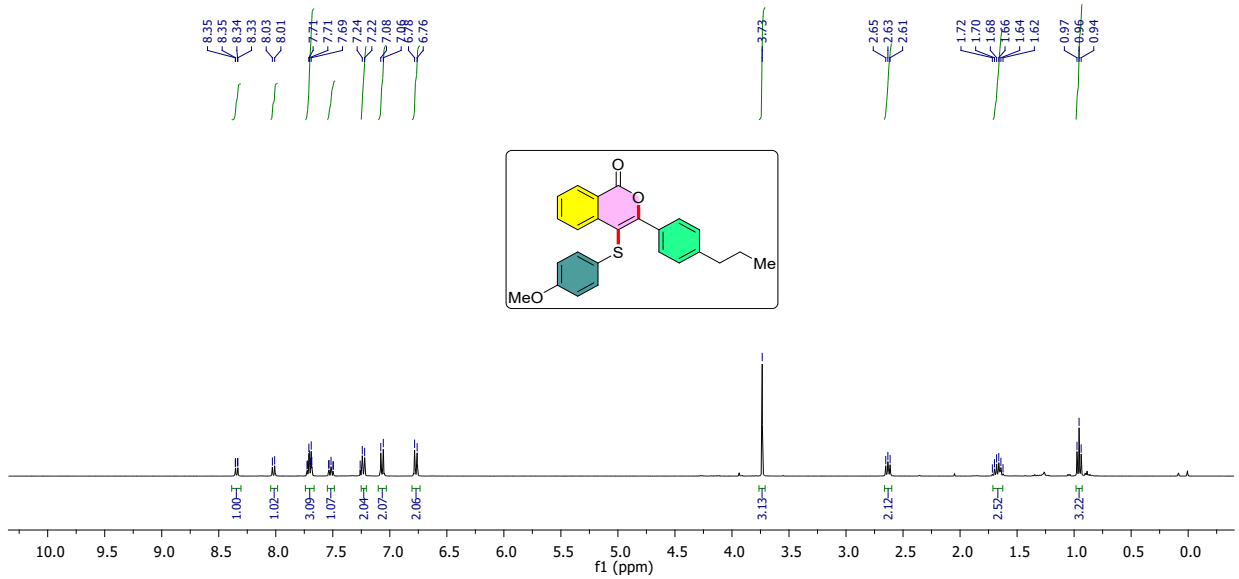
^{1}H-NMR (400 MHz) spectrum of **3cc** in $CDCl_3$



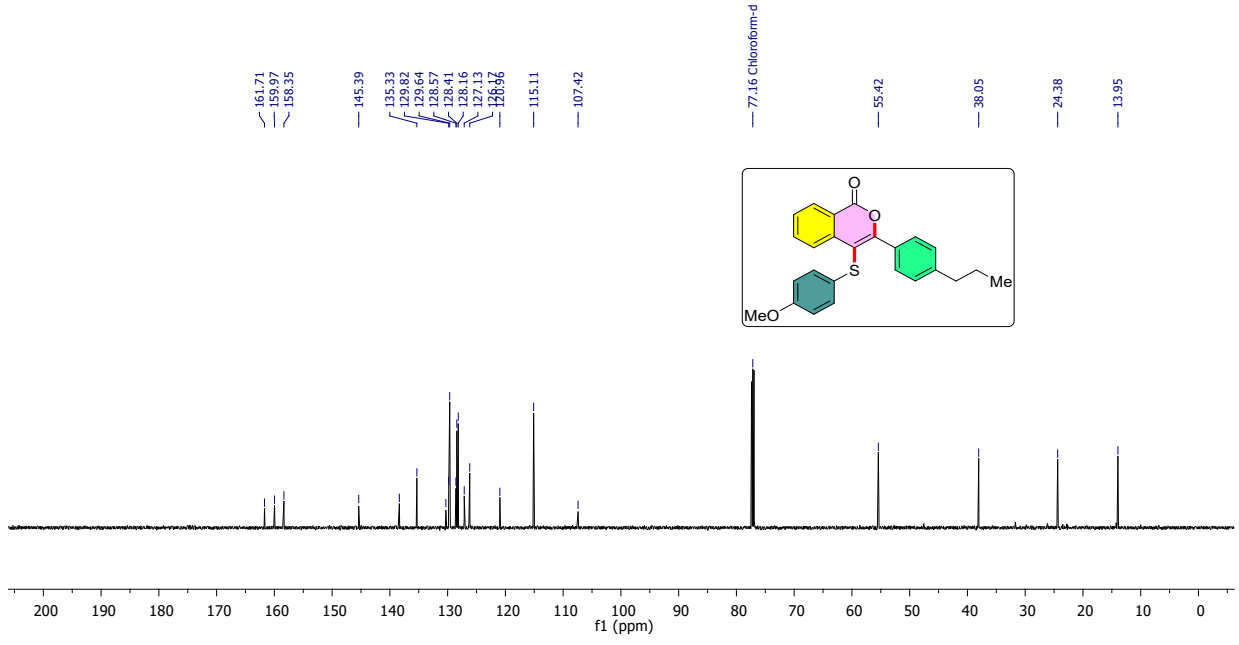
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3cc** in $CDCl_3$



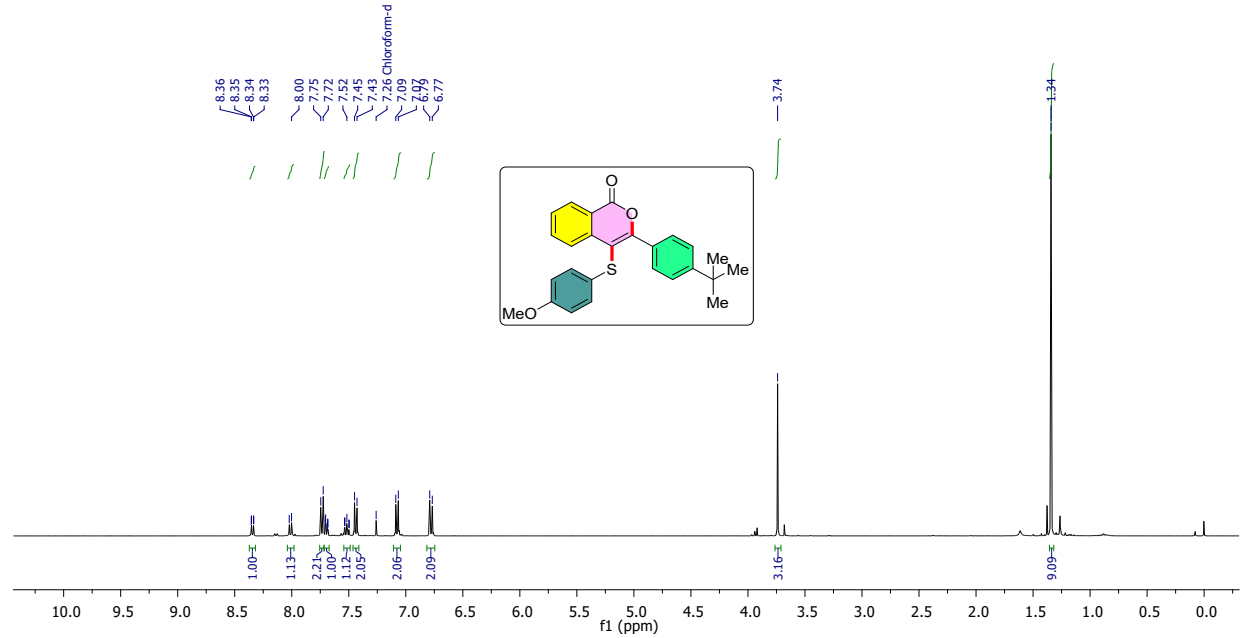
^{1}H-NMR (400 MHz) spectrum of **3dc** in $CDCl_3$



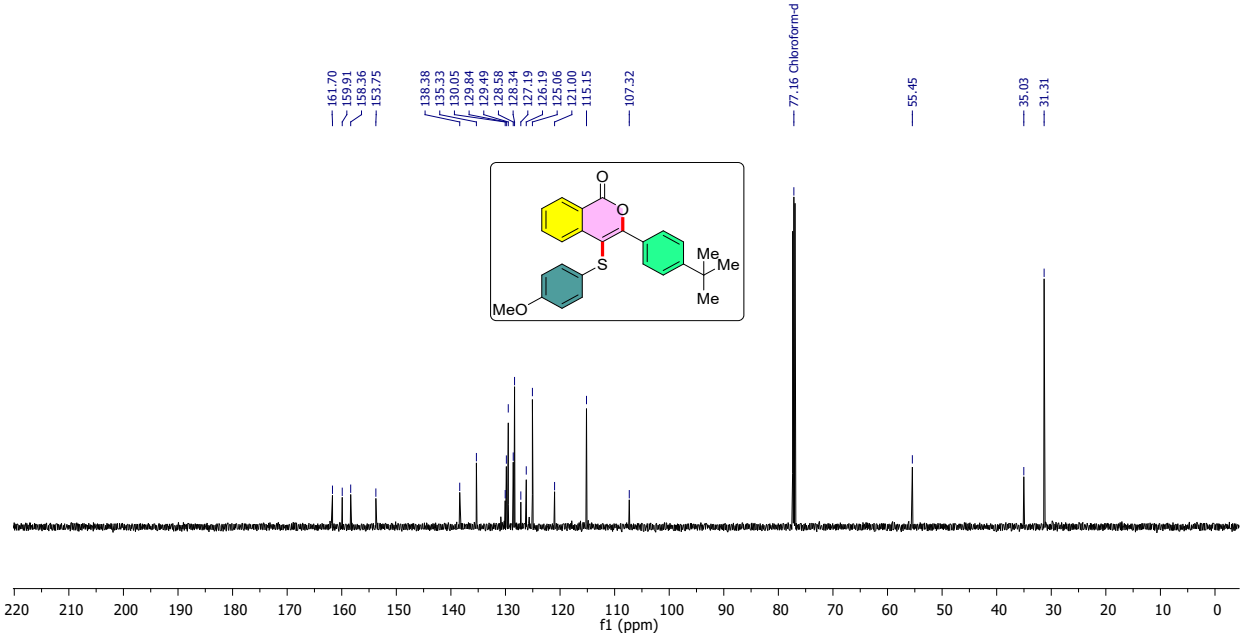
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3dc** in $CDCl_3$



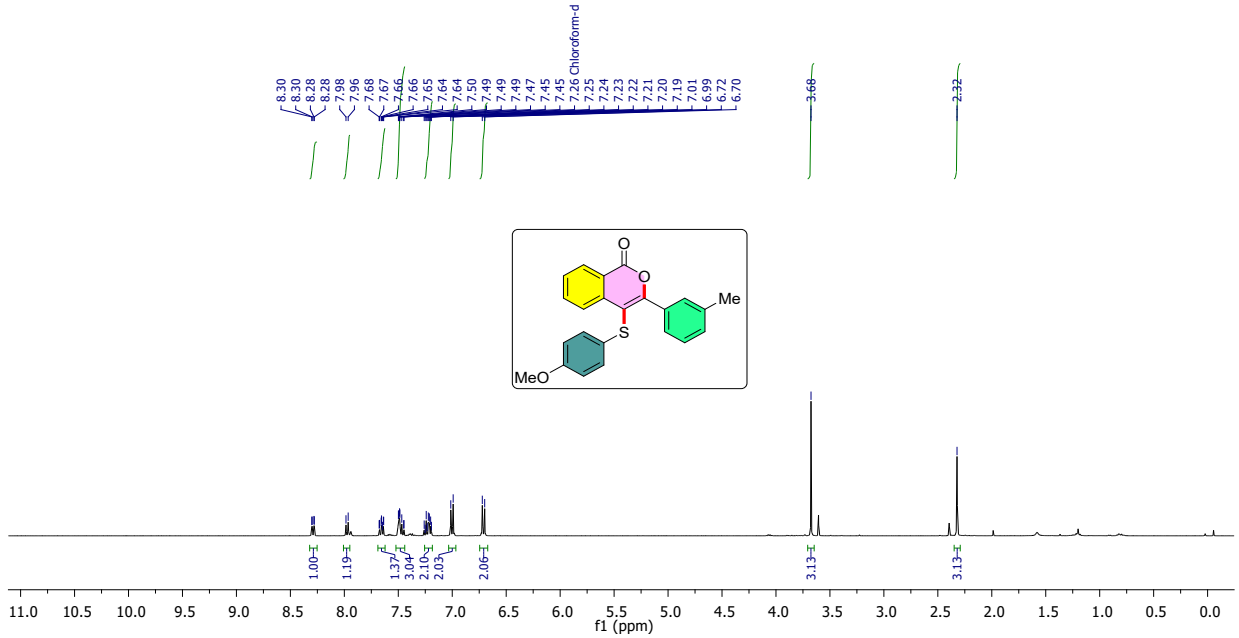
^{1}H-NMR (400 MHz) spectrum of **3ec** in $CDCl_3$



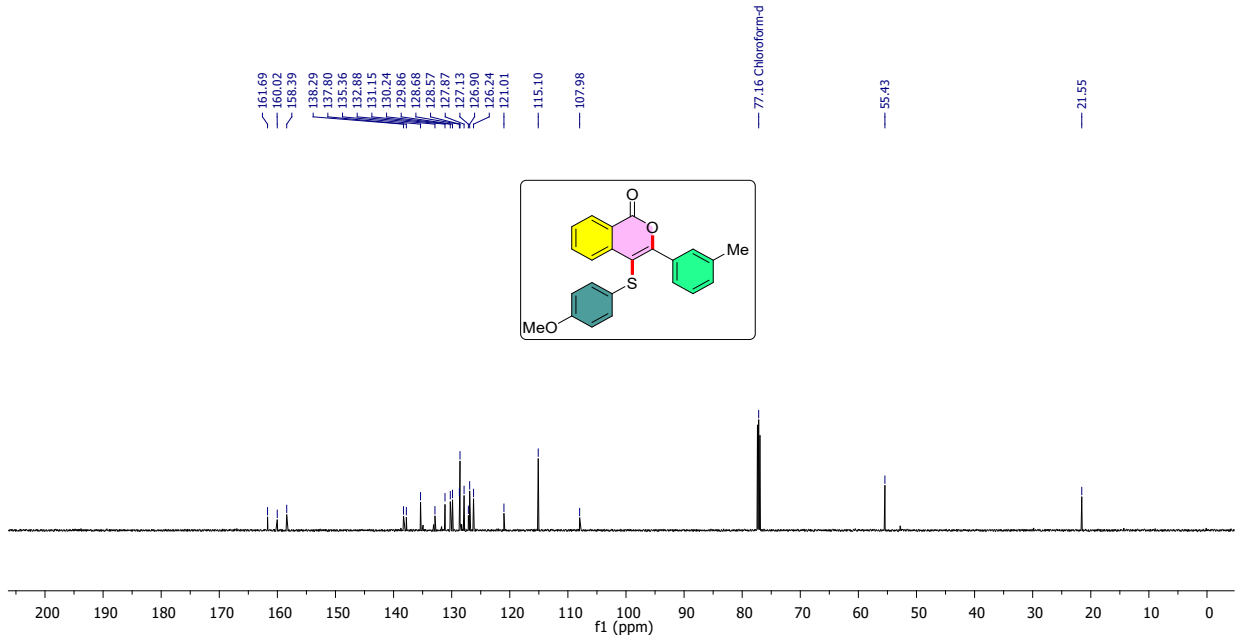
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3ec** in $CDCl_3$



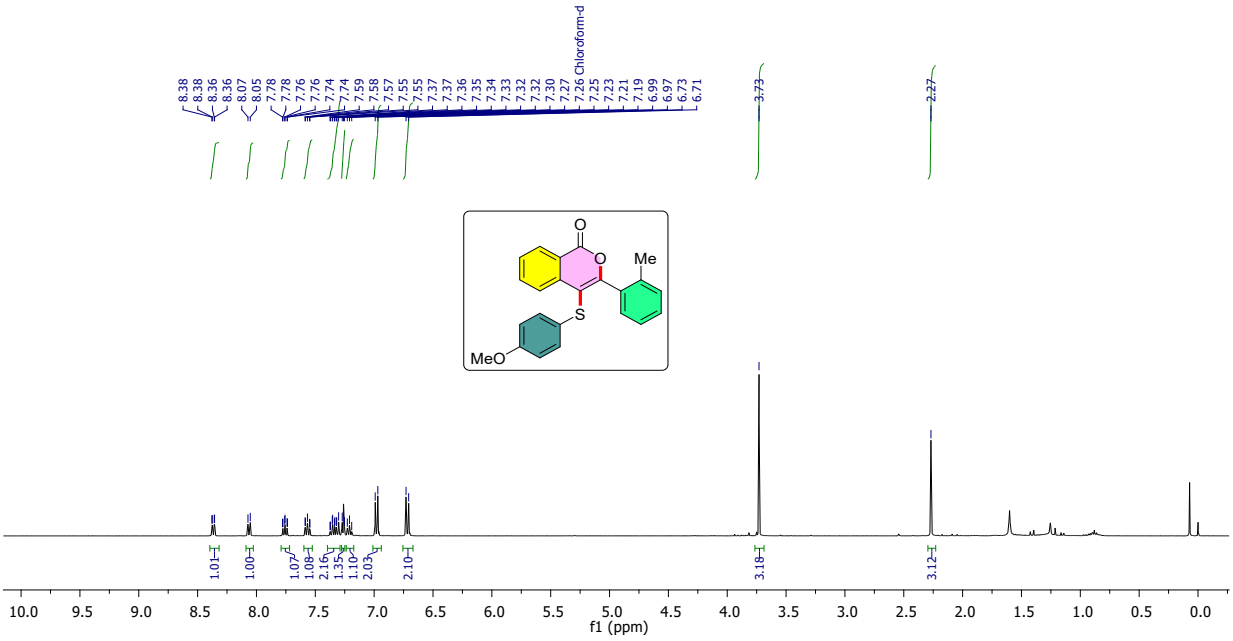
^{1}H-NMR (400 MHz) spectrum of **3fc** in $CDCl_3$



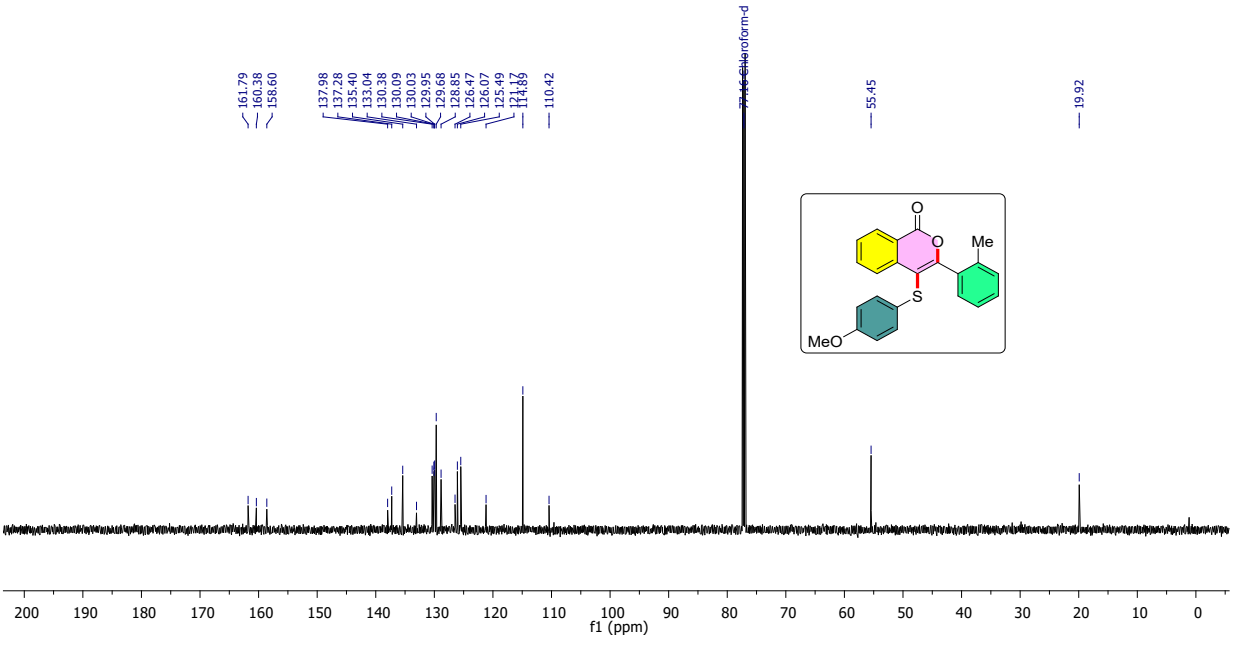
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3fc** in $CDCl_3$



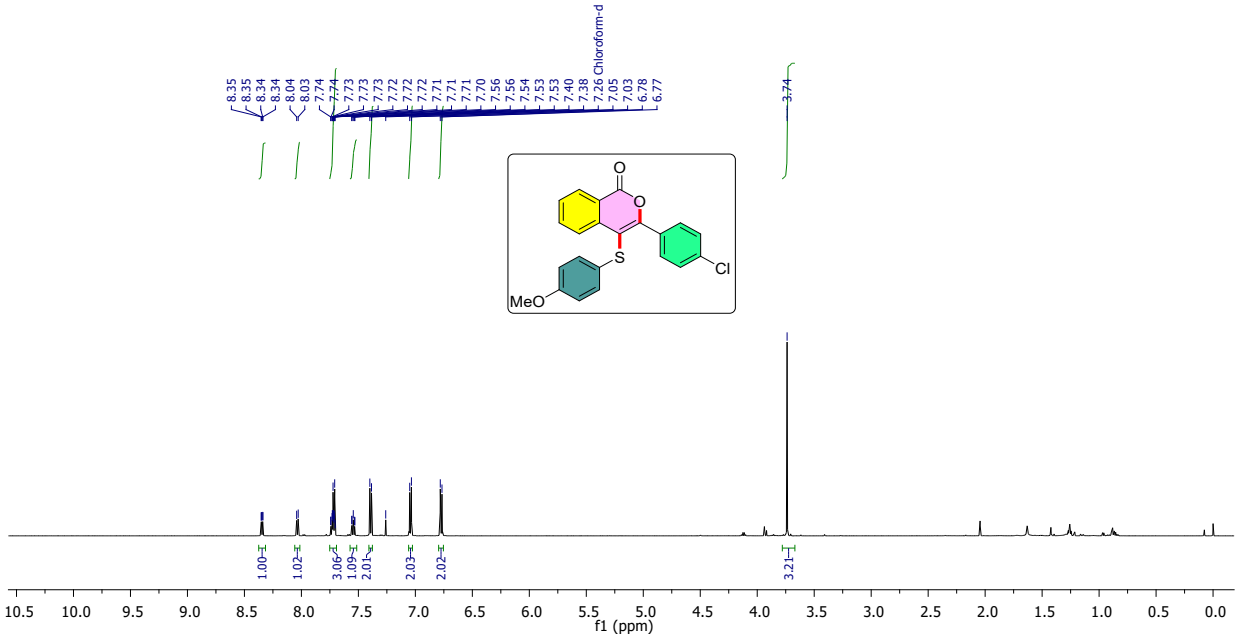
^{1}H-NMR (400 MHz) spectrum of **3gc** in $CDCl_3$



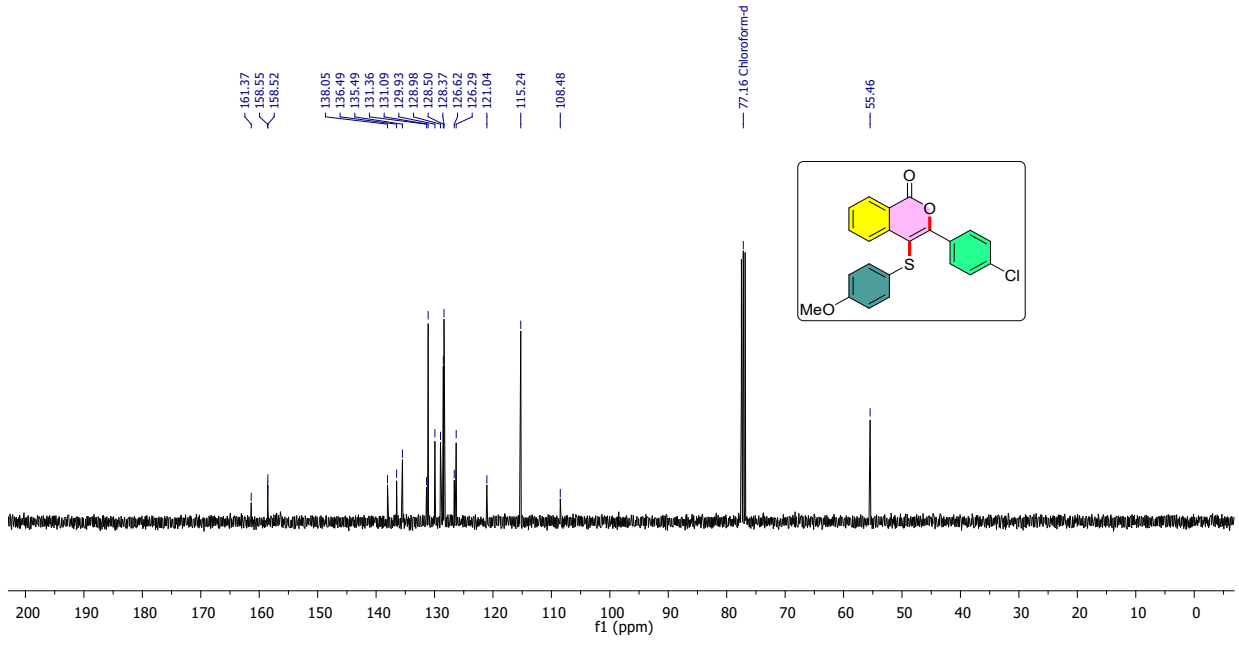
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3gc** in $CDCl_3$



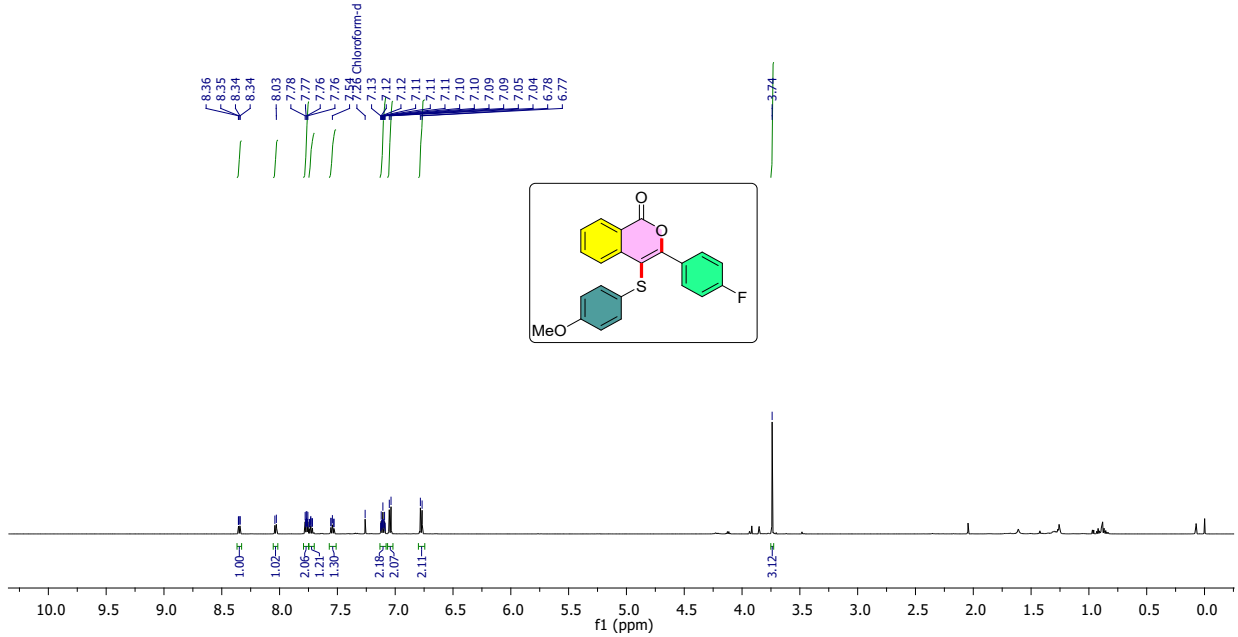
^{1}H-NMR (600 MHz) spectrum of **3hc** in $CDCl_3$



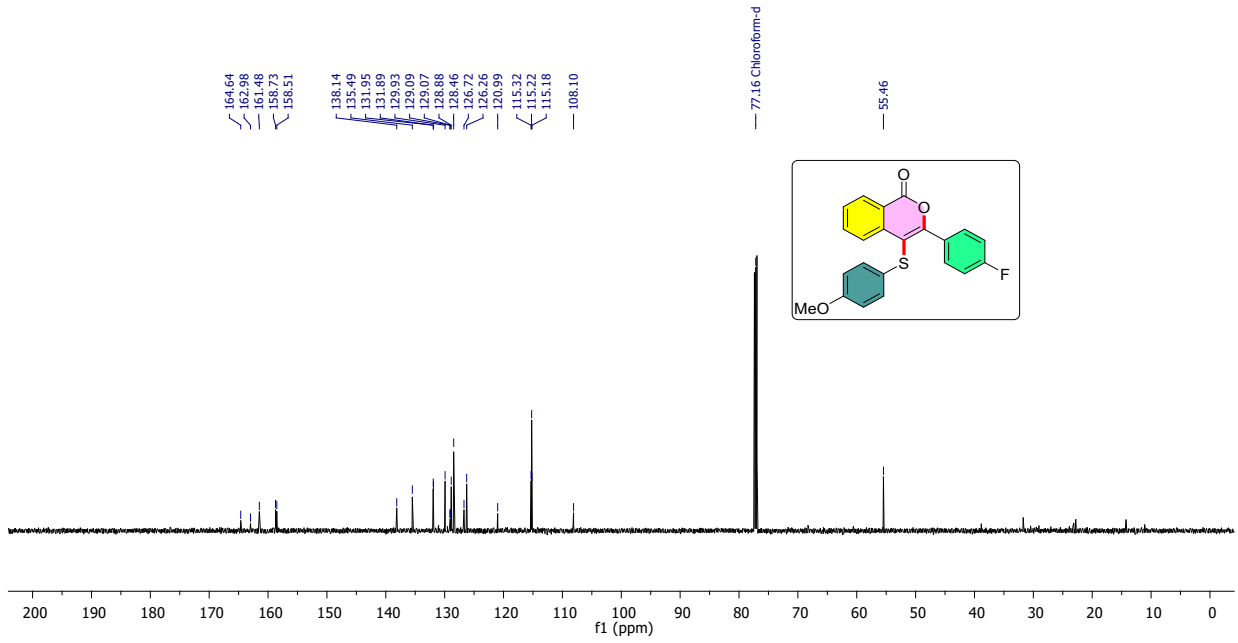
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3hc** in $CDCl_3$



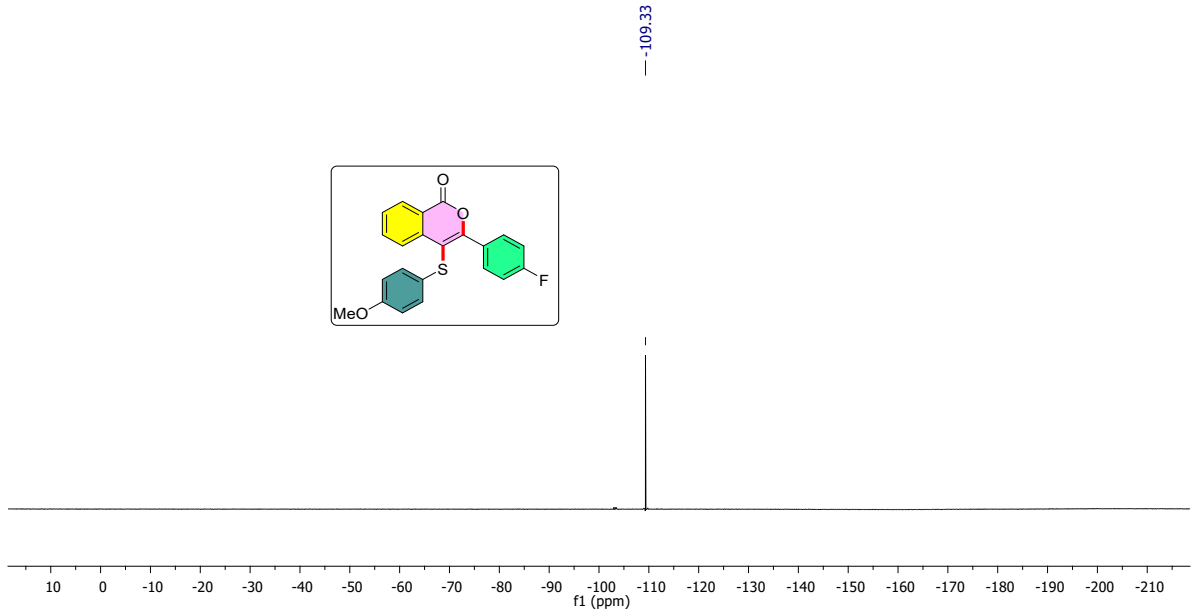
^{1}H-NMR (600 MHz) spectrum of **3ic** in $CDCl_3$



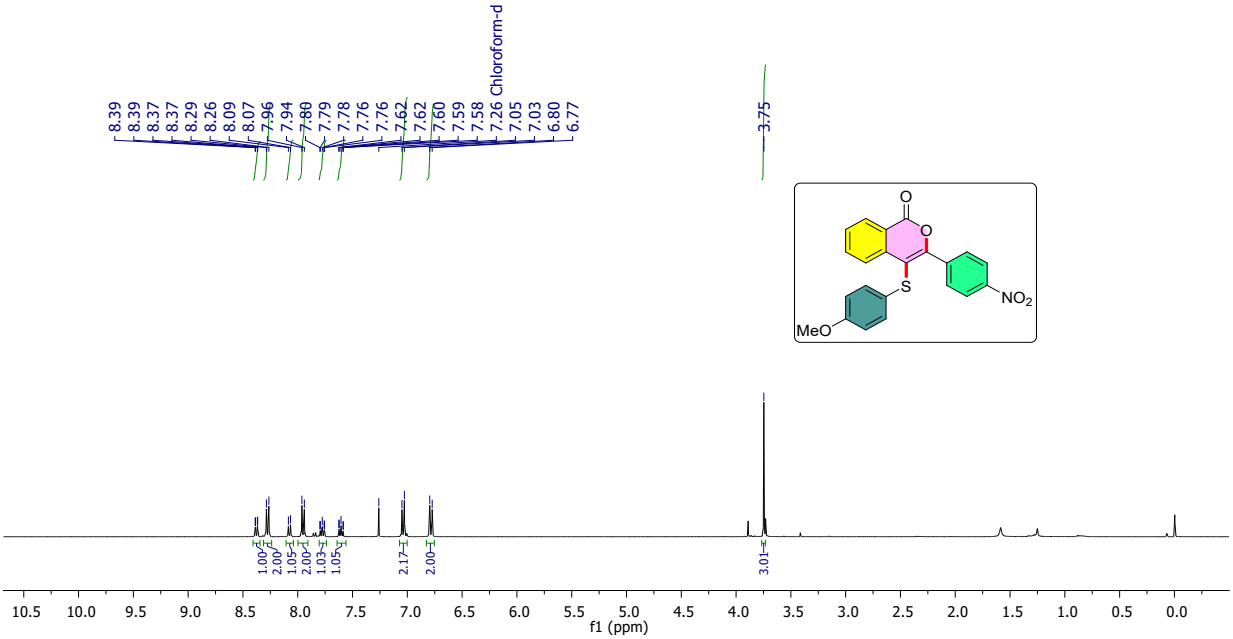
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3ic** in $CDCl_3$



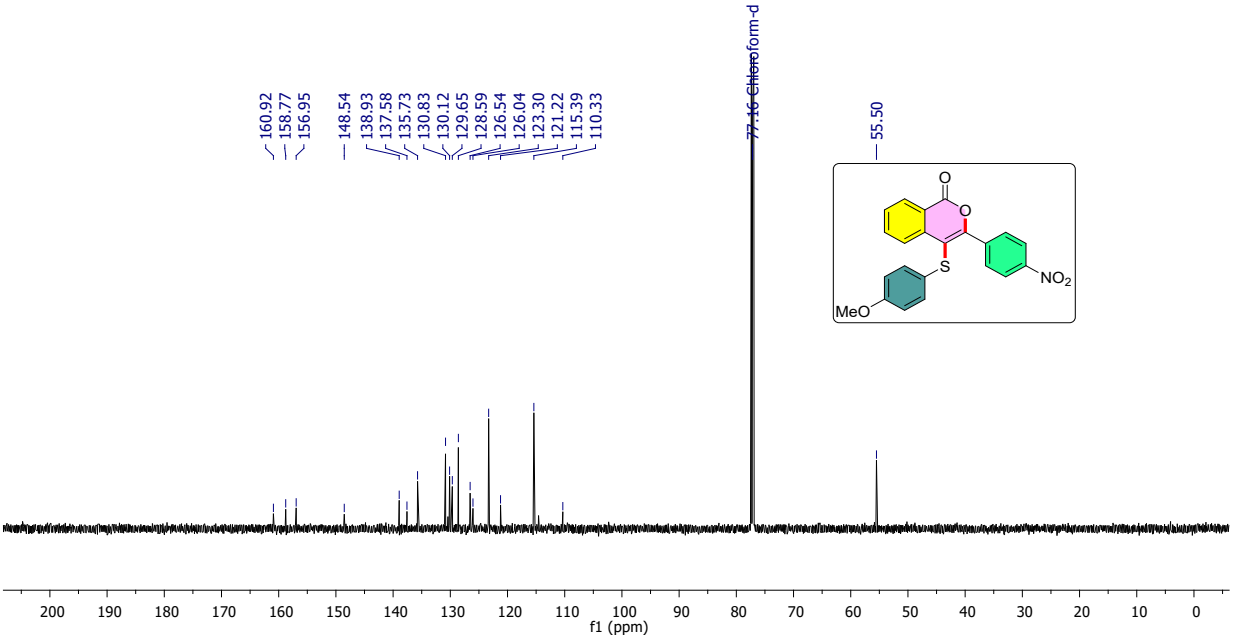
^{19}F-NMR (376 MHz) spectrum of **3ic** in $CDCl_3$



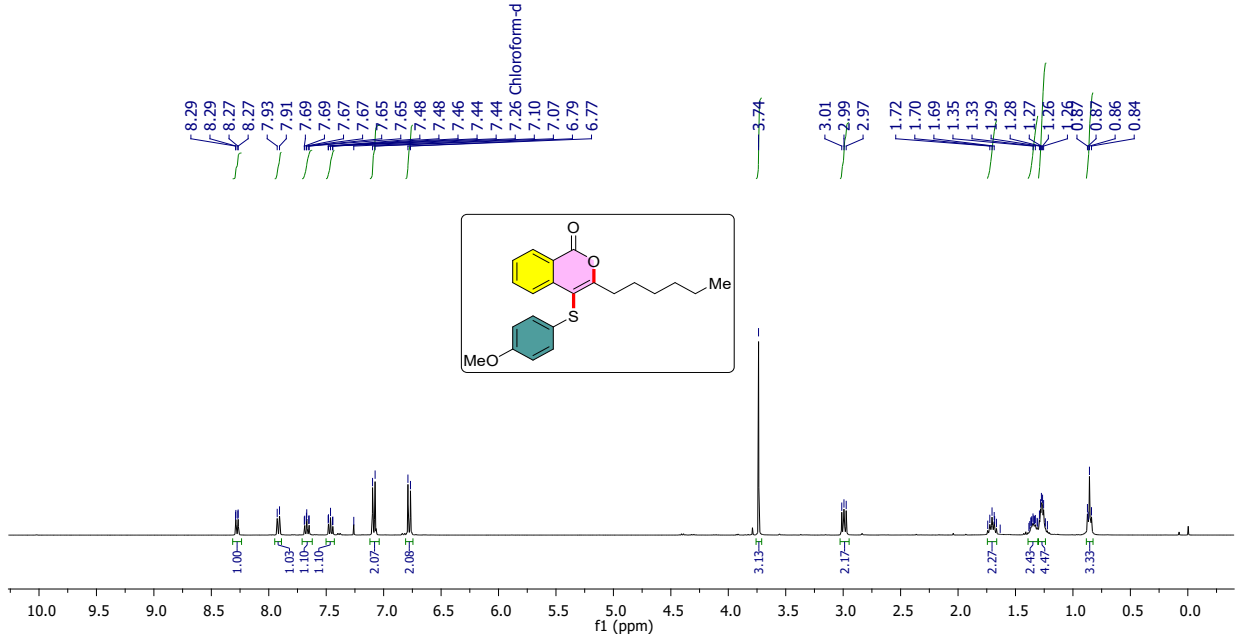
^{1}H-NMR (400 MHz) spectrum of **3jc** in $CDCl_3$



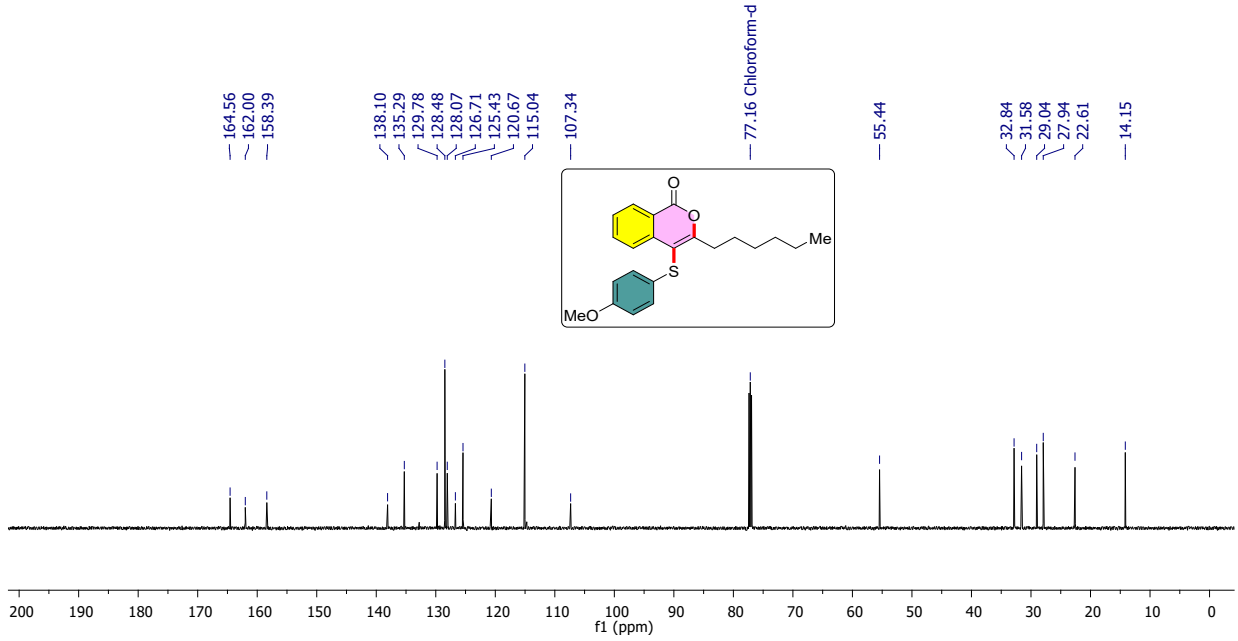
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3jc** in $CDCl_3$



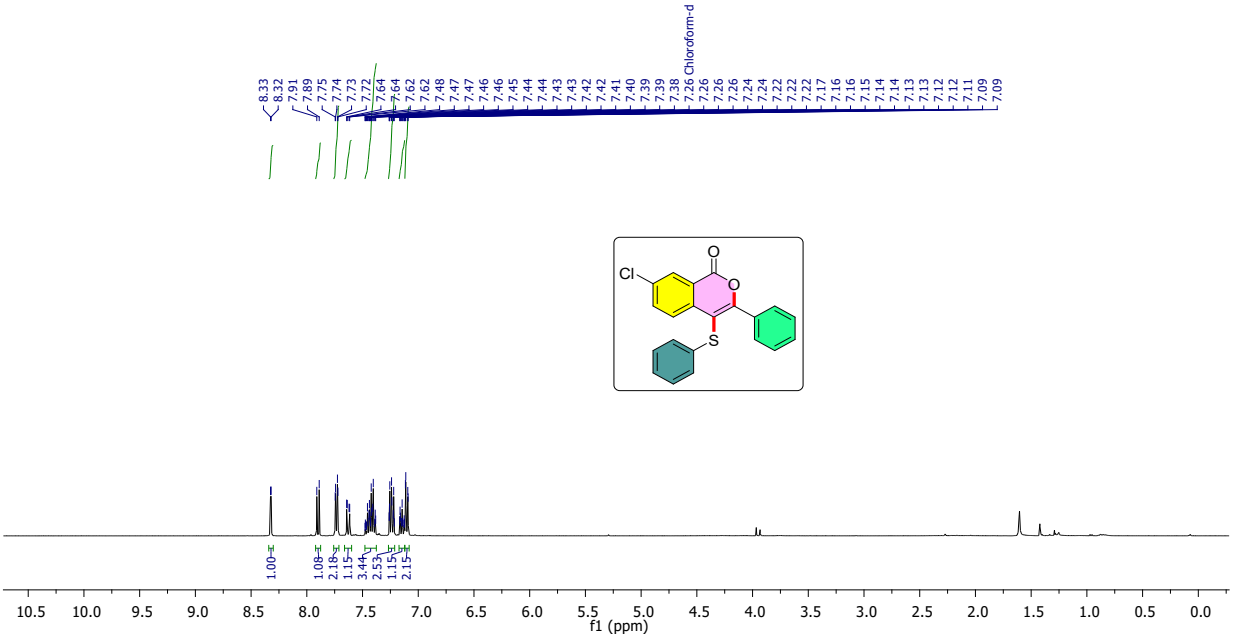
^{1}H-NMR (400 MHz) spectrum of **3kc** in $CDCl_3$



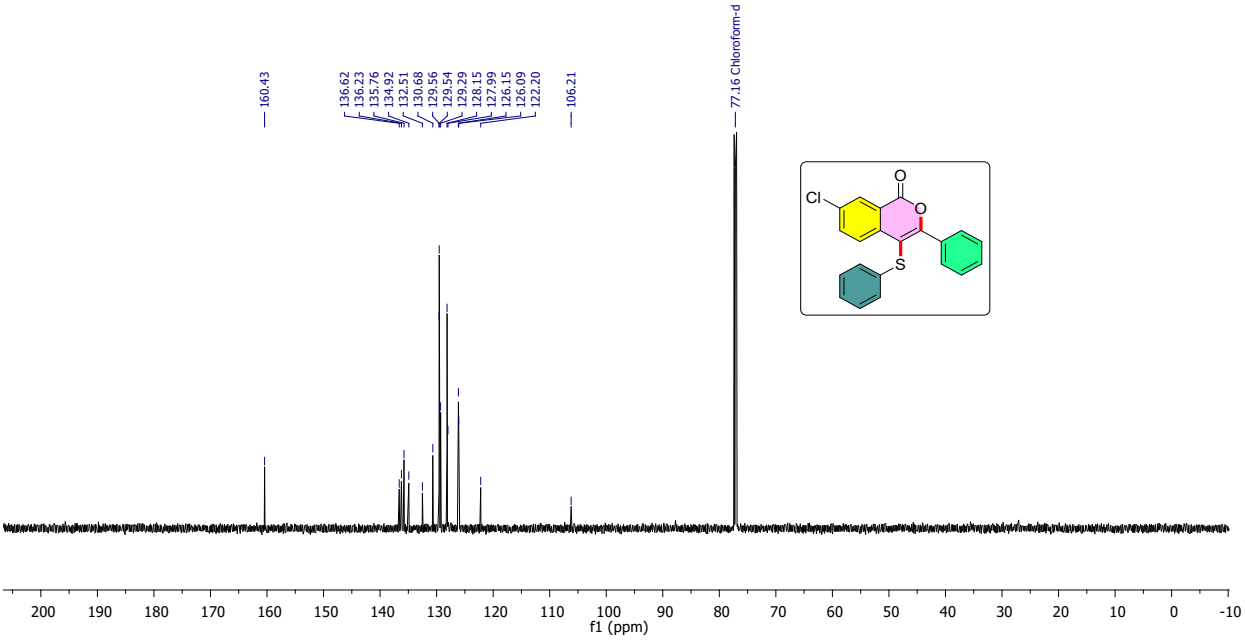
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3kc** in $CDCl_3$



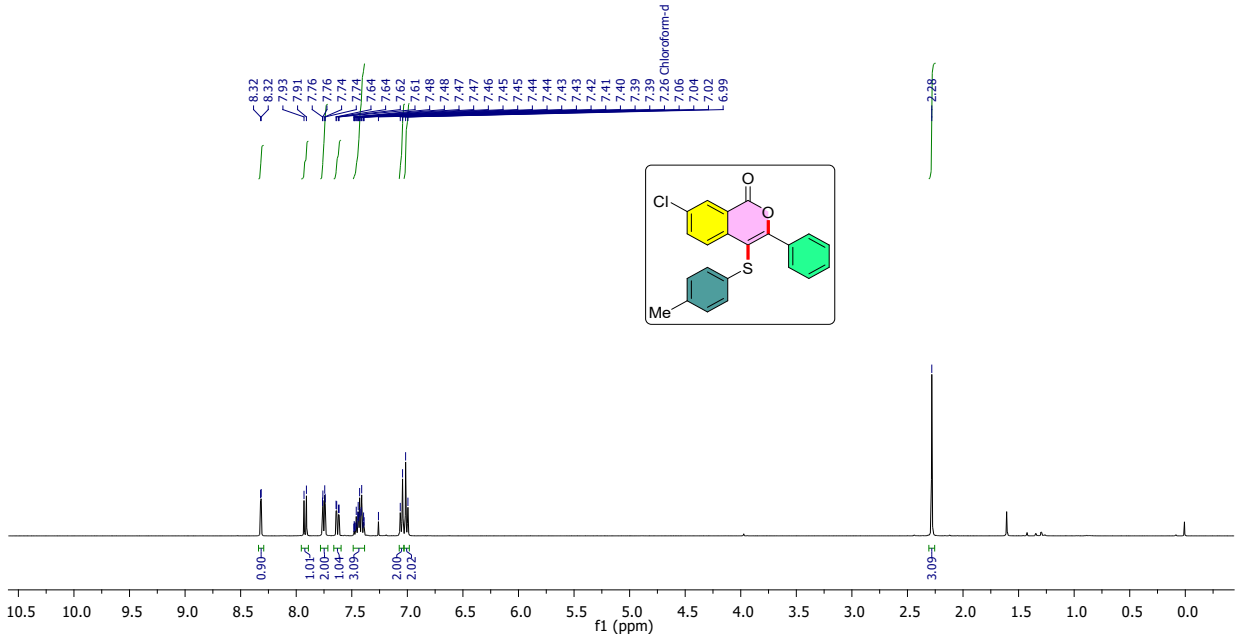
^{1}H-NMR (400 MHz) spectrum of **3la** in $CDCl_3$



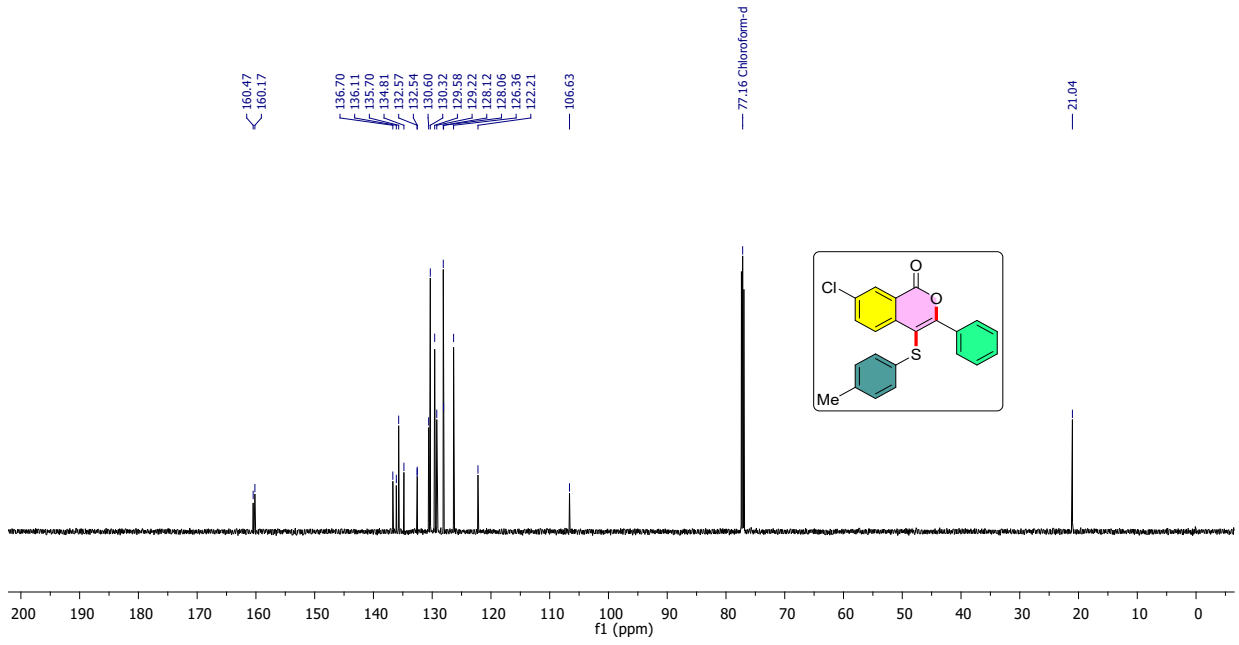
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3la** in $CDCl_3$



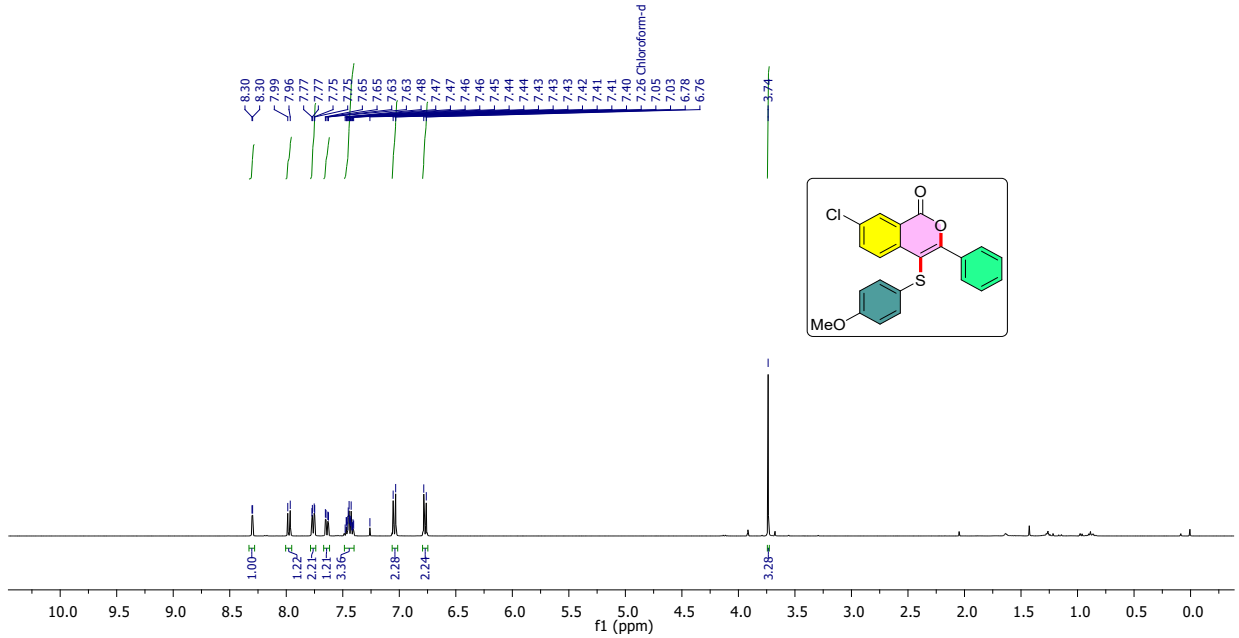
^{1}H-NMR (400 MHz) spectrum of **3lb** in $CDCl_3$



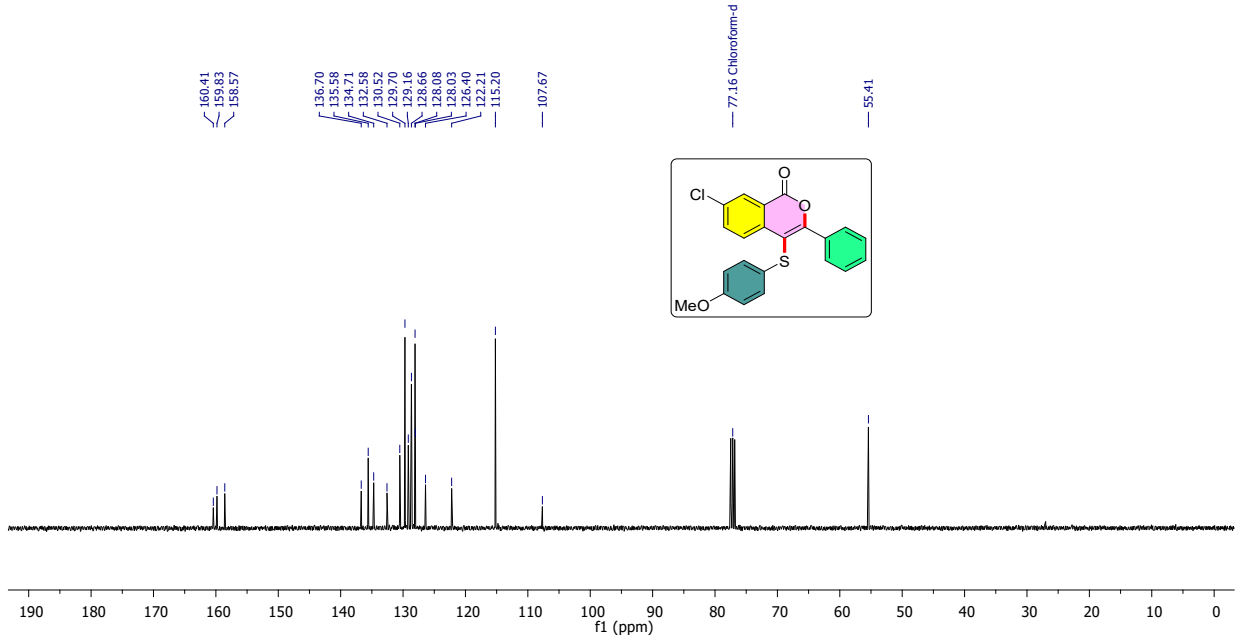
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3lb** in $CDCl_3$



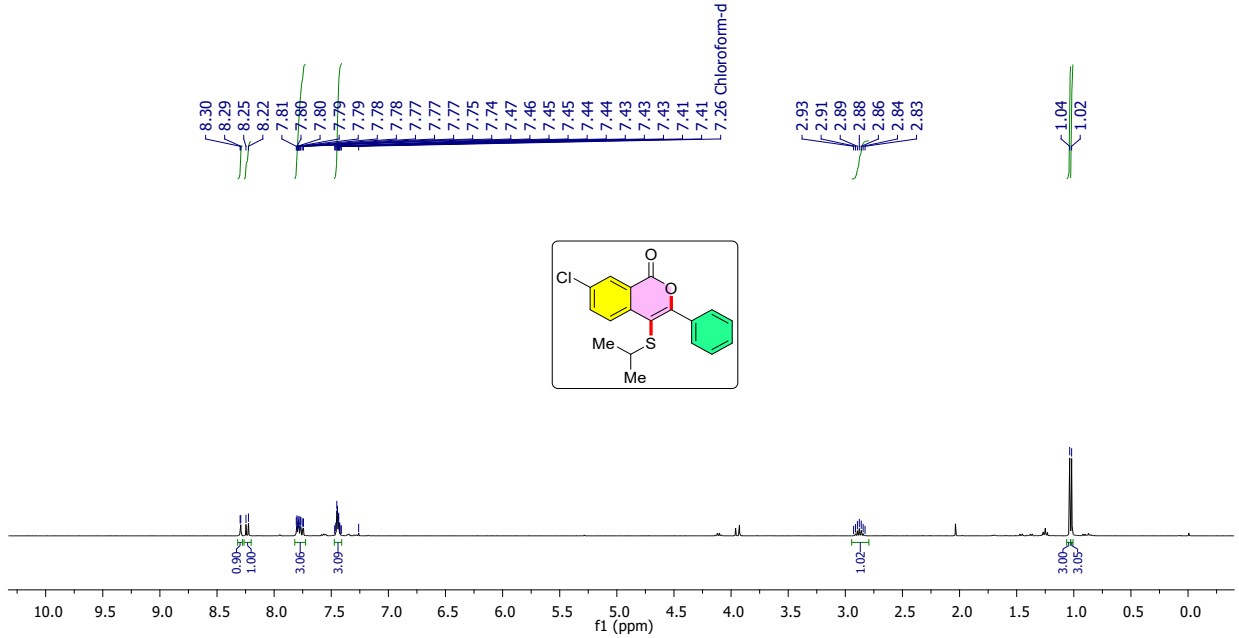
^{1}H-NMR (400 MHz) spectrum of **3lc** in $CDCl_3$



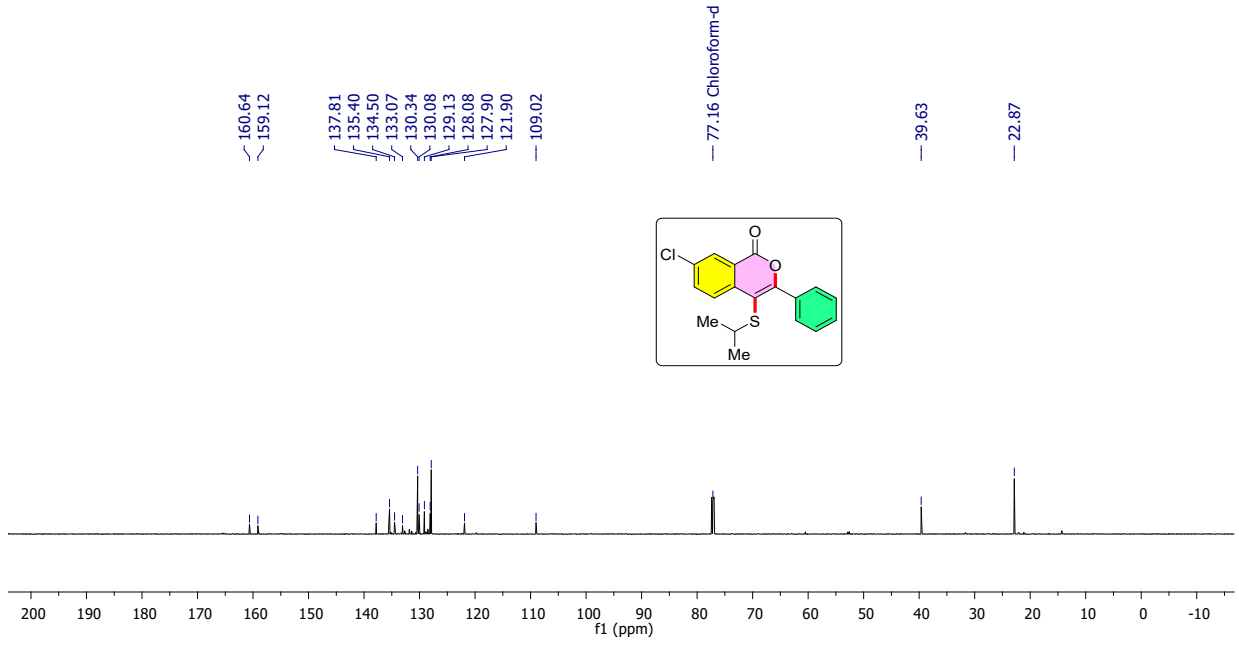
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3lc** in $CDCl_3$



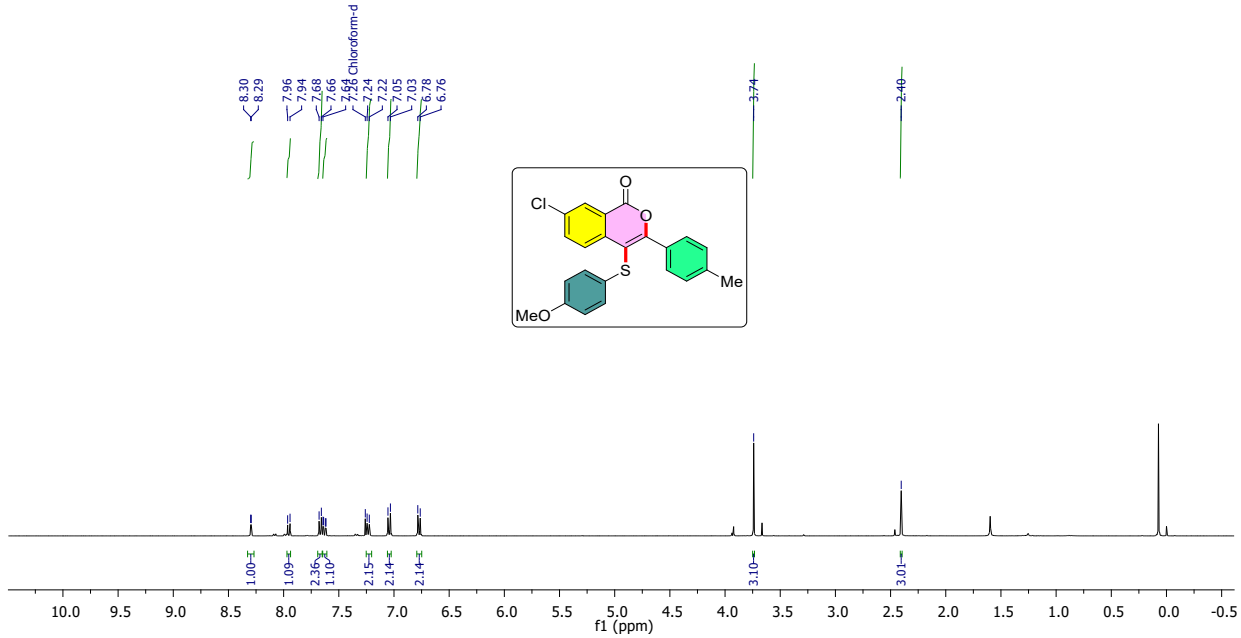
^{1}H-NMR (400 MHz) spectrum of **3ld** in $CDCl_3$



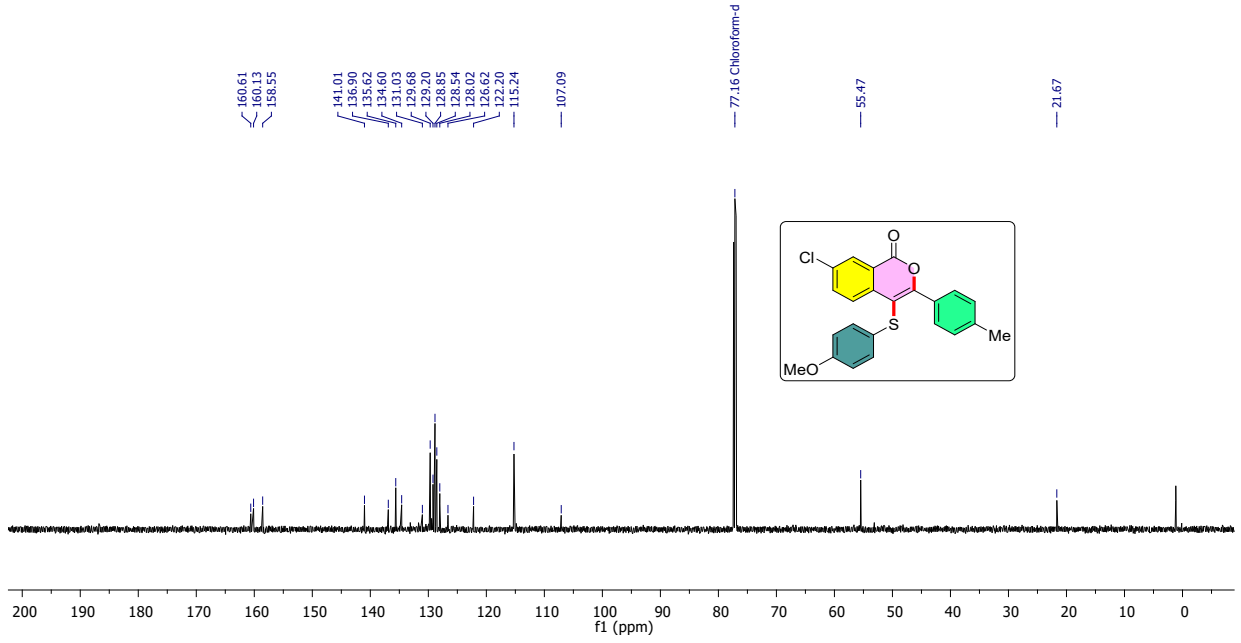
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3ld** in $CDCl_3$



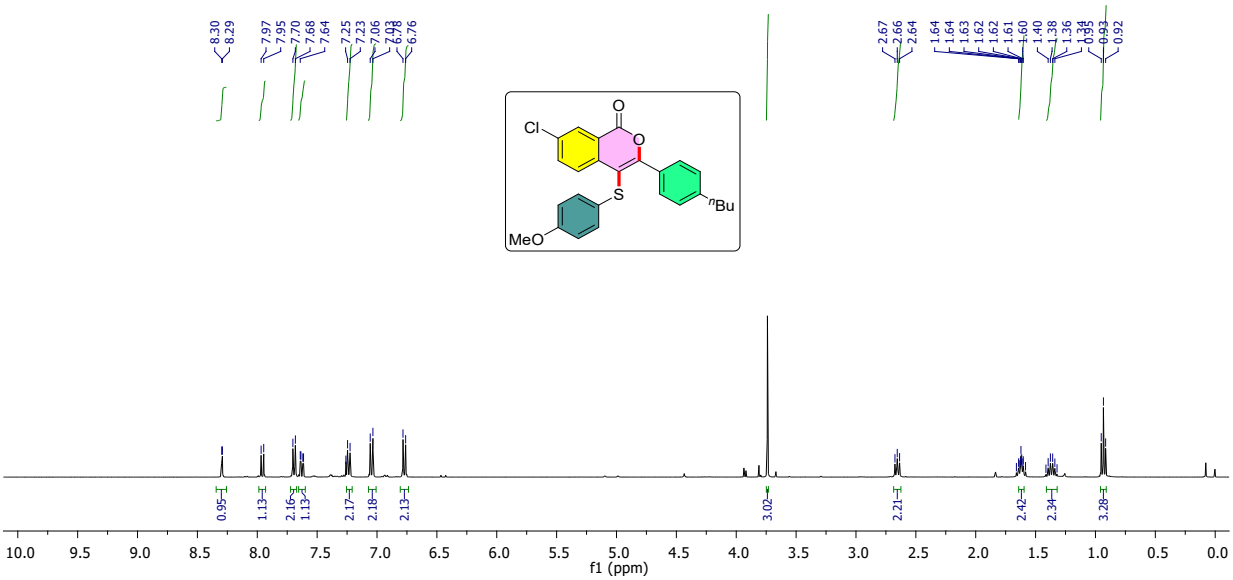
^{1}H-NMR (400 MHz) spectrum of **3mc** in $CDCl_3$



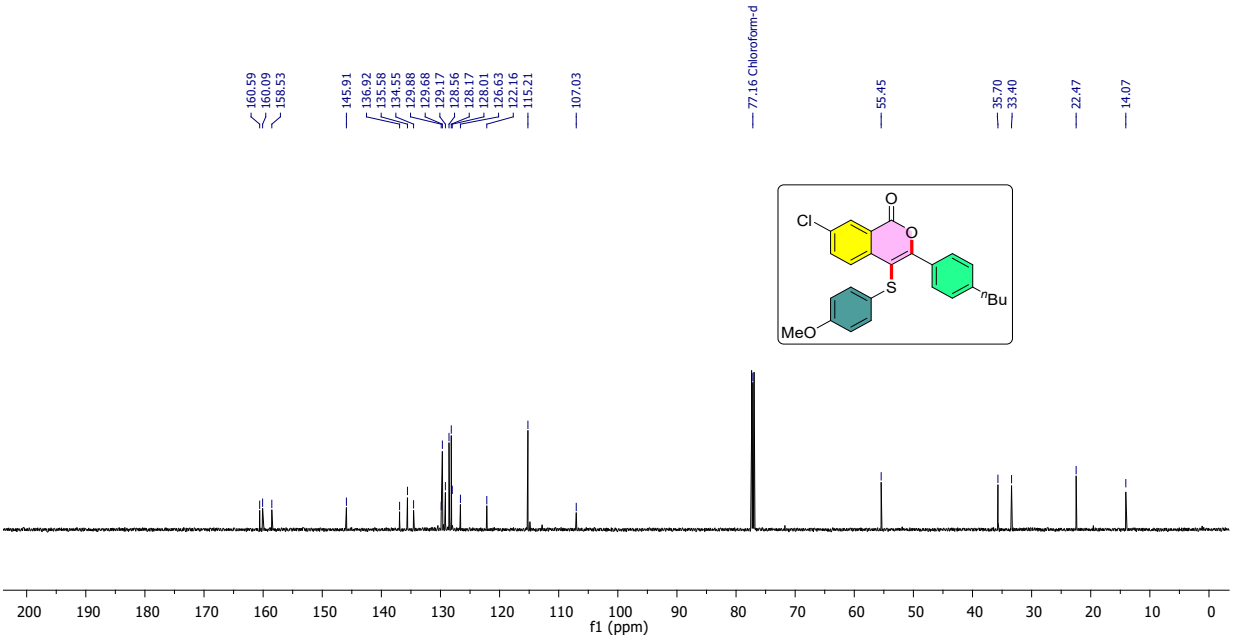
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3mc** in $CDCl_3$



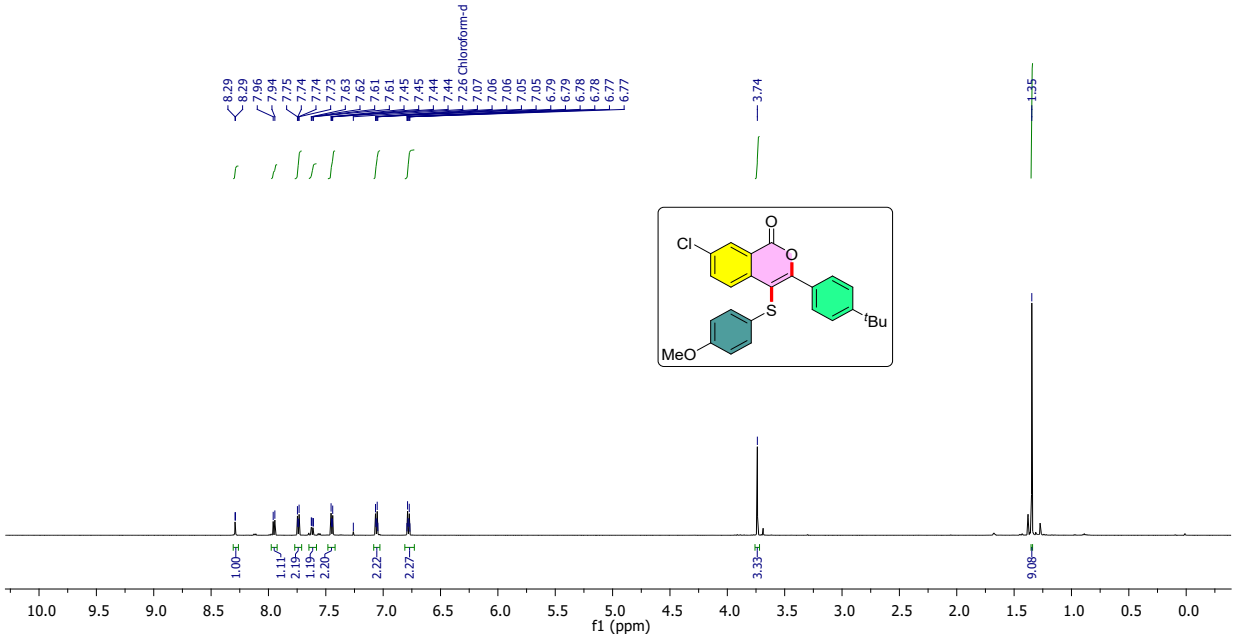
^{1}H-NMR (400 MHz) spectrum of **3nc** in $CDCl_3$



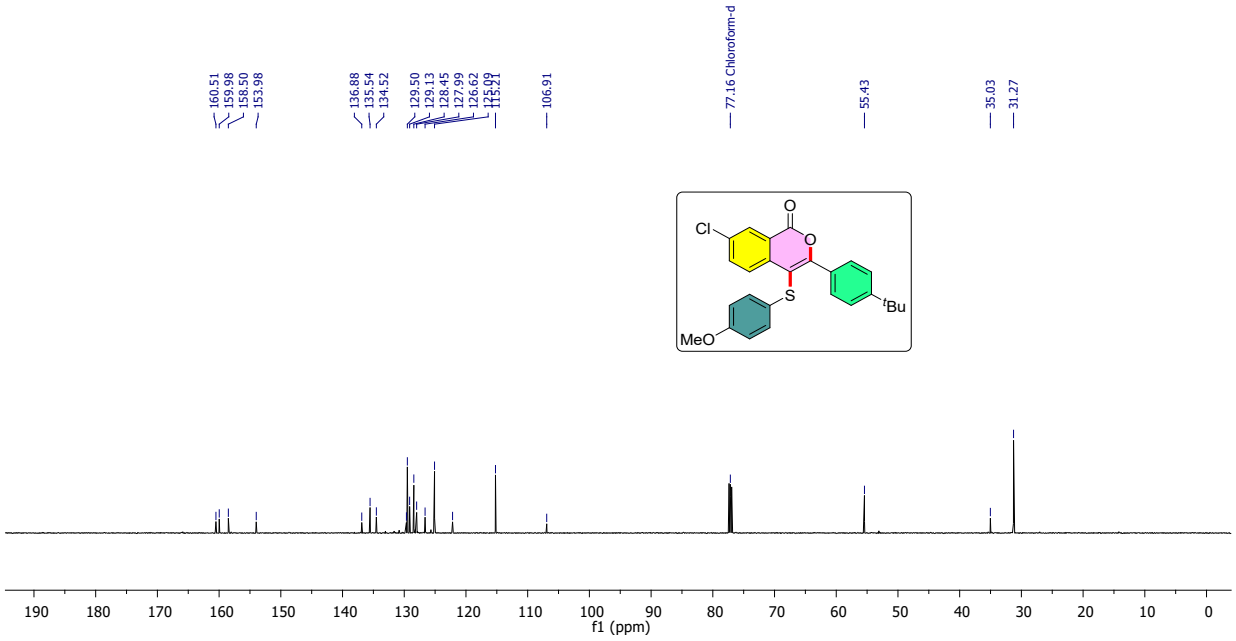
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3nc** in $CDCl_3$



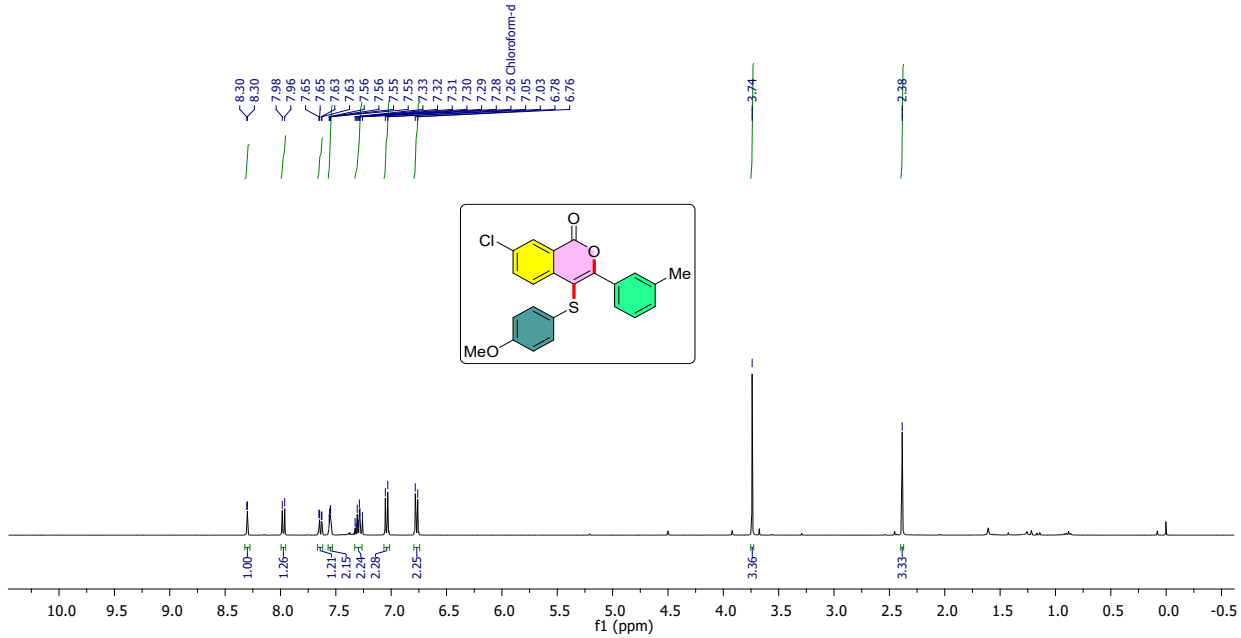
^{1}H-NMR (600 MHz) spectrum of **3oc** in $CDCl_3$



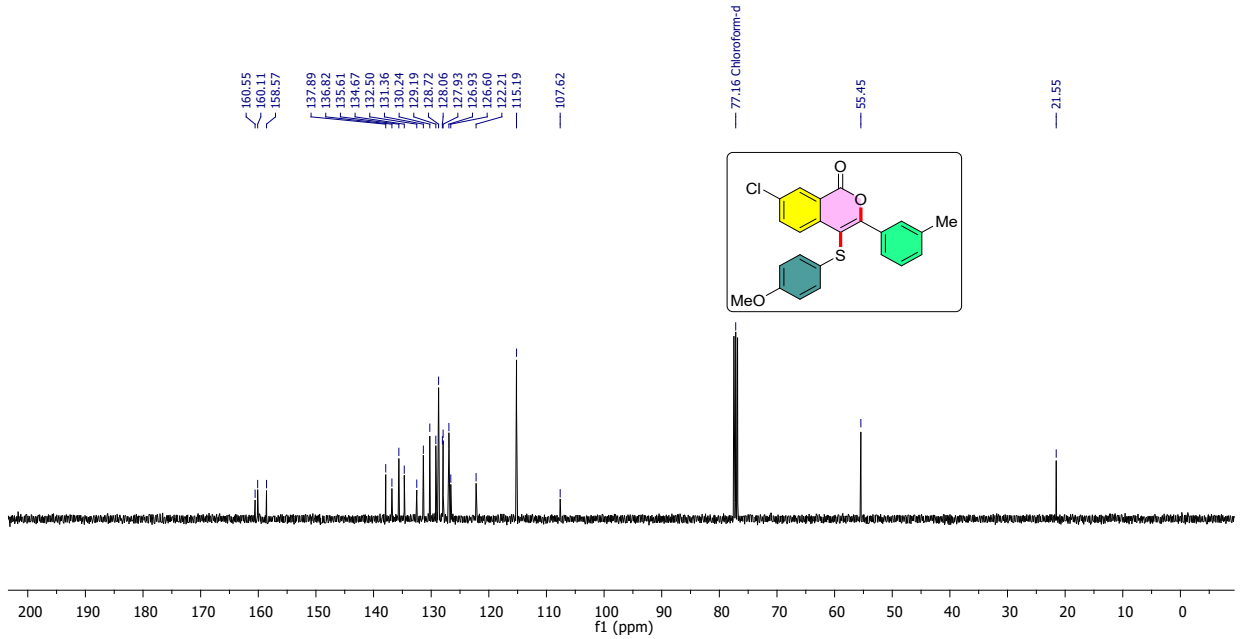
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3oc** in $CDCl_3$



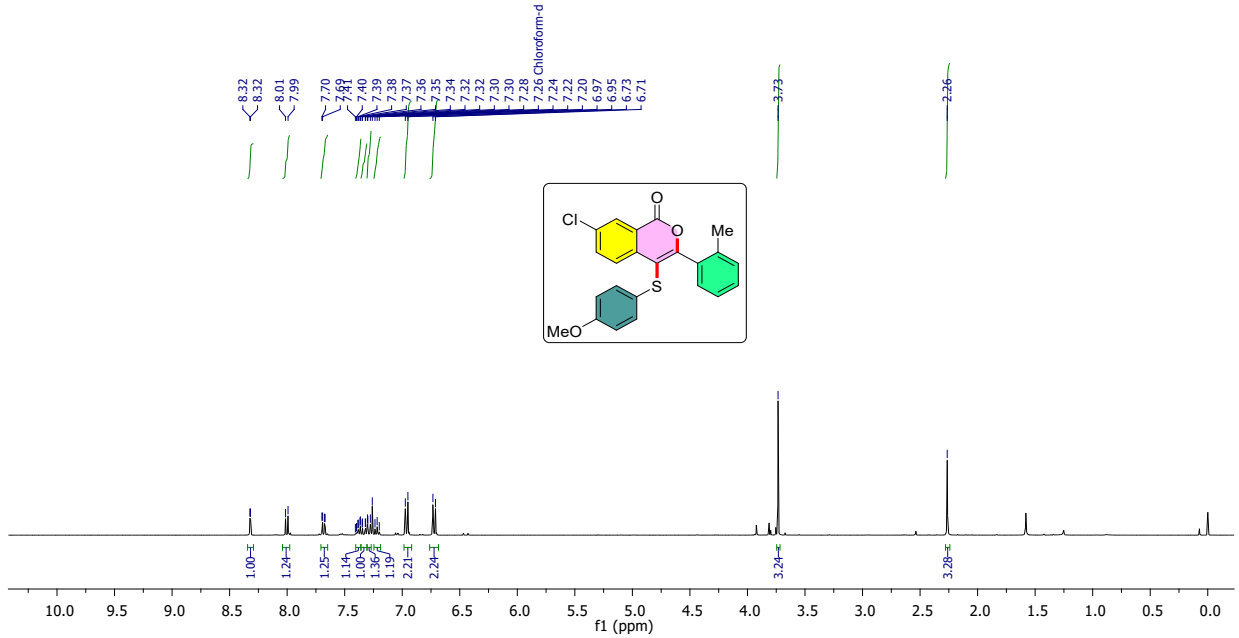
^{1}H-NMR (400 MHz) spectrum of **3pc** in $CDCl_3$



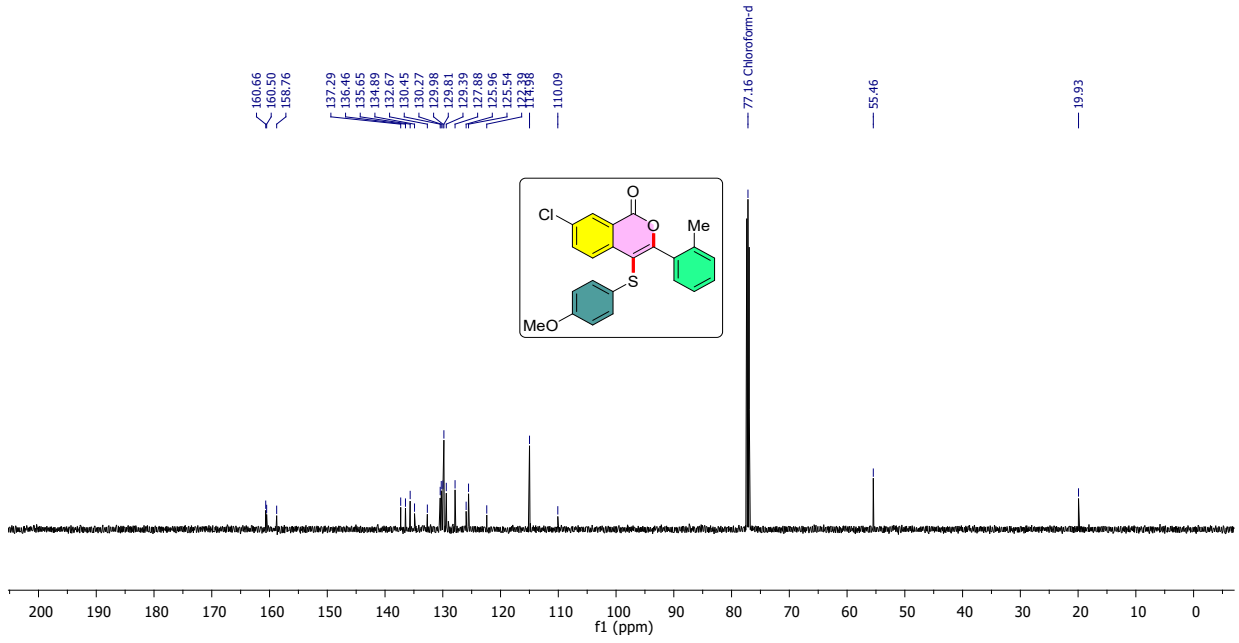
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3pc** in $CDCl_3$



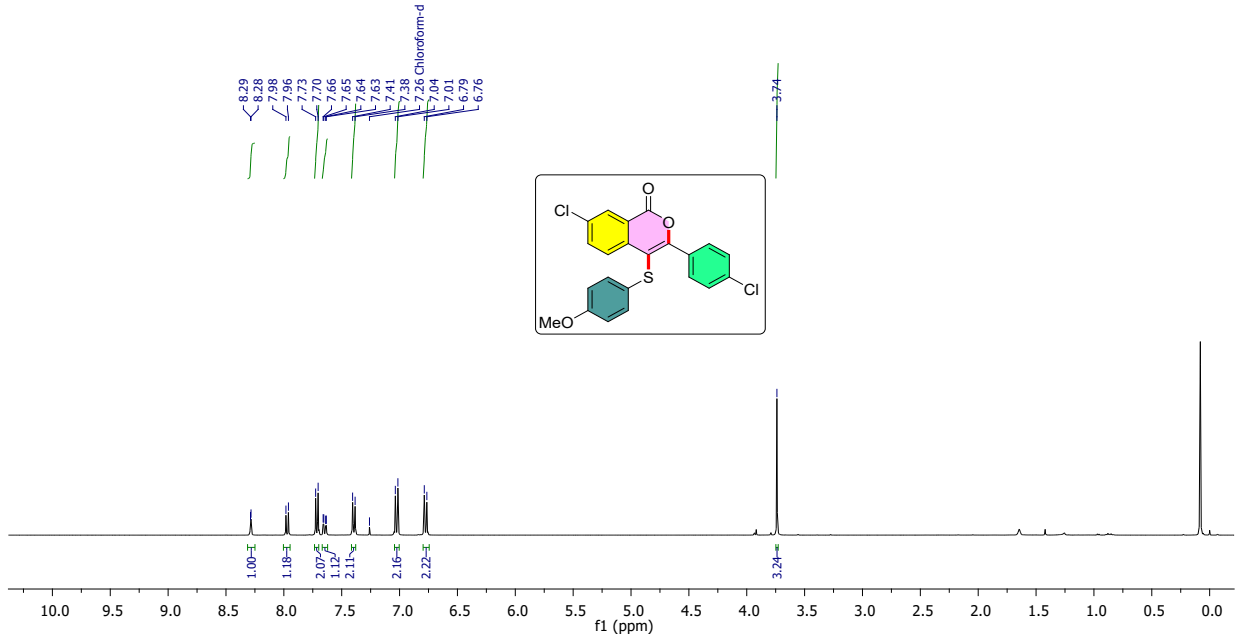
^{1}H-NMR (400 MHz) spectrum of **3qc** in $CDCl_3$



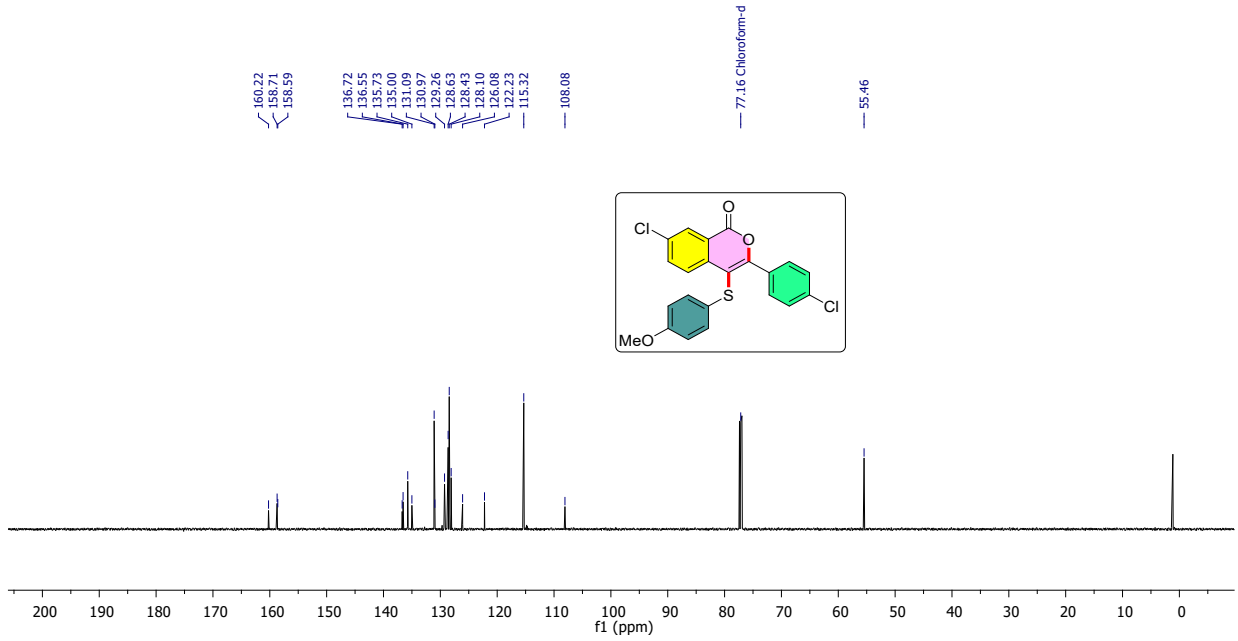
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3qc** in $CDCl_3$



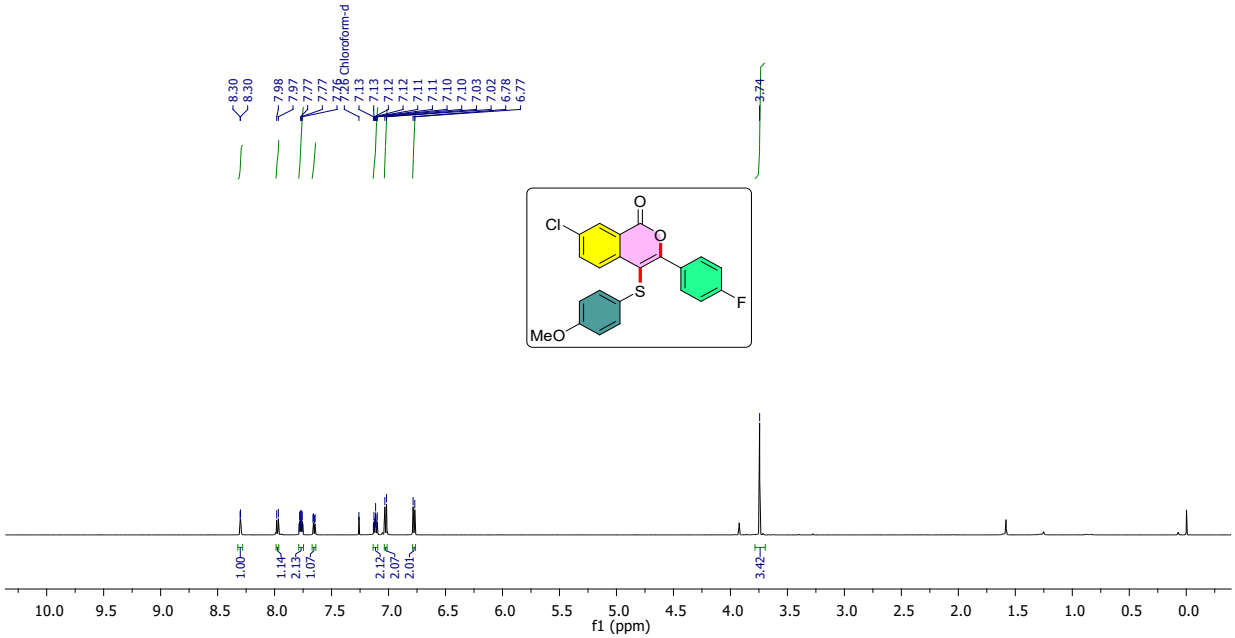
^{1}H-NMR (400 MHz) spectrum of **3rc** in $CDCl_3$



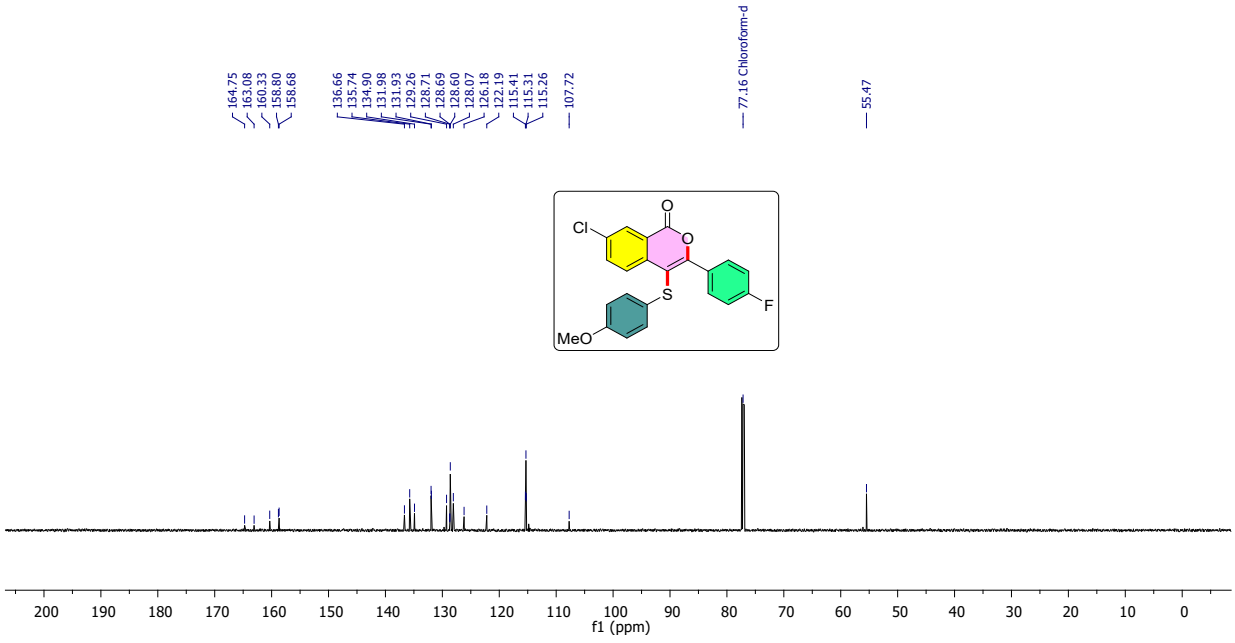
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3rc** in $CDCl_3$



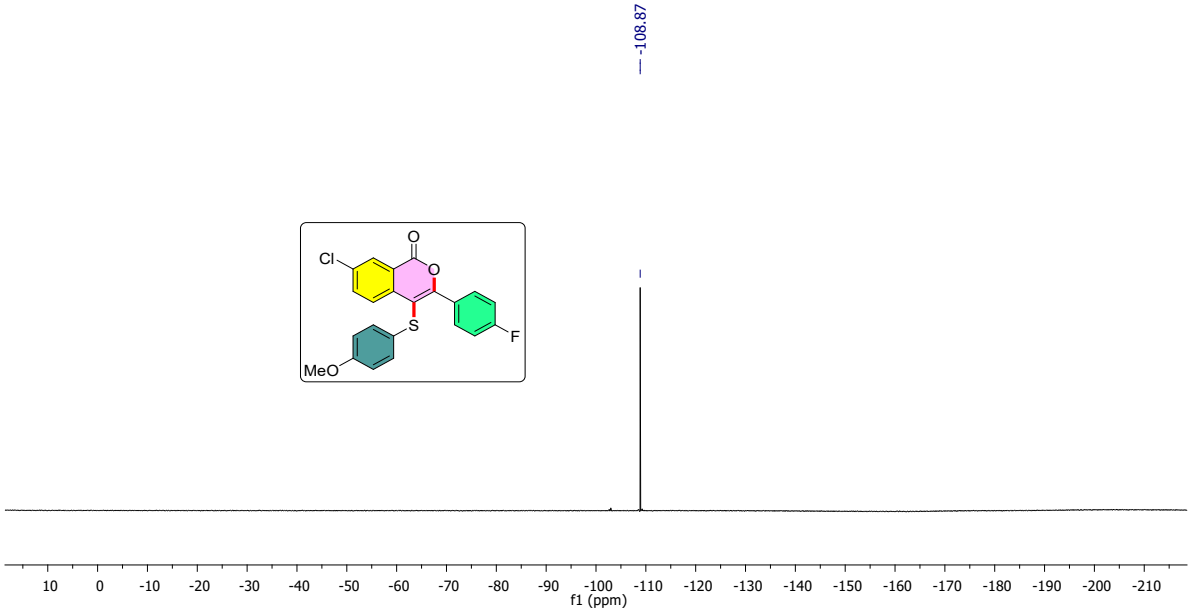
^{1}H-NMR (600 MHz) spectrum of **3sc** in $CDCl_3$



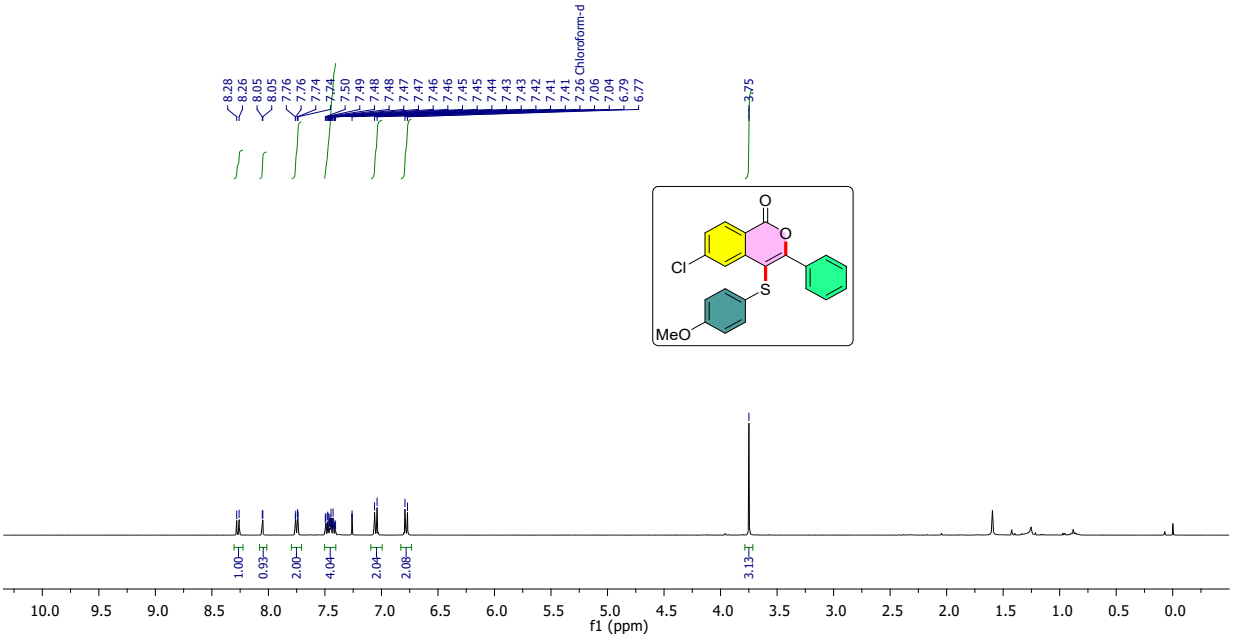
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3sc** in $CDCl_3$



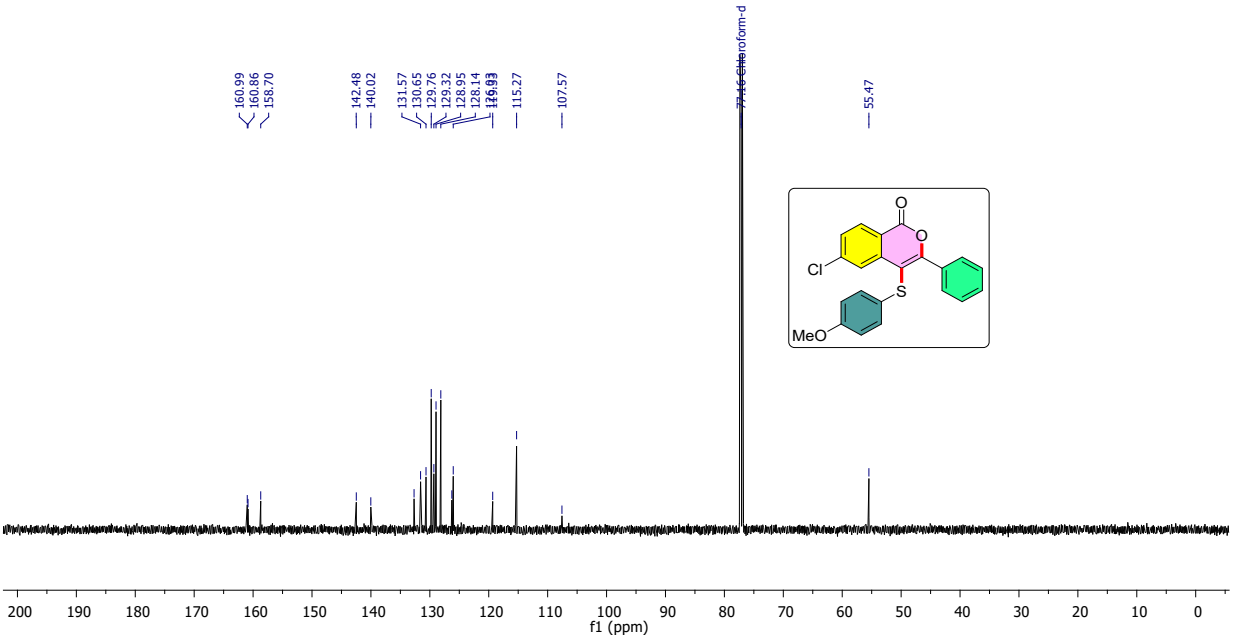
^{19}F-NMR (376 MHz) spectrum of **3sc** in $CDCl_3$



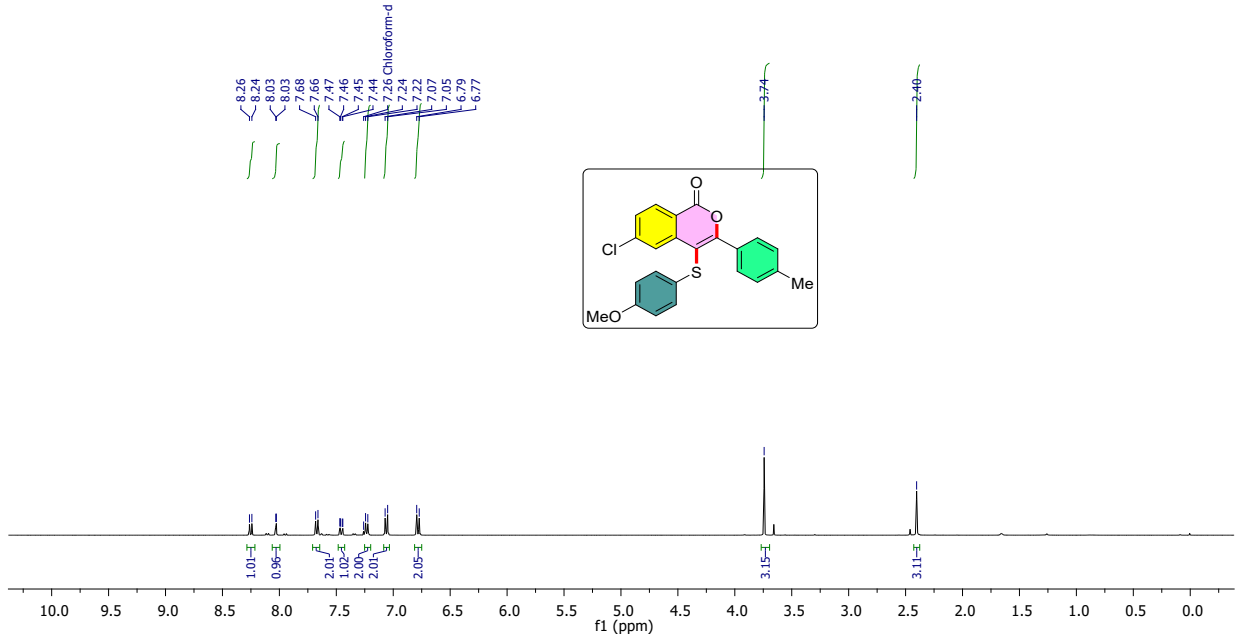
^{1}H-NMR (400 MHz) spectrum of **3tc** in $CDCl_3$



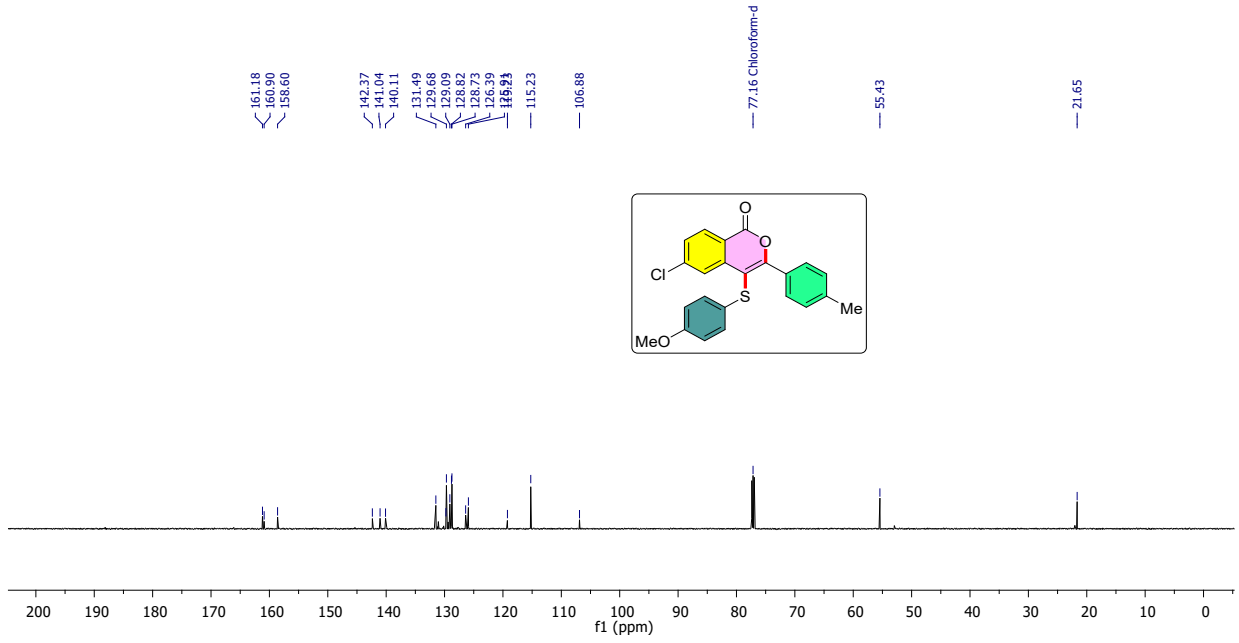
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3tc** in $CDCl_3$



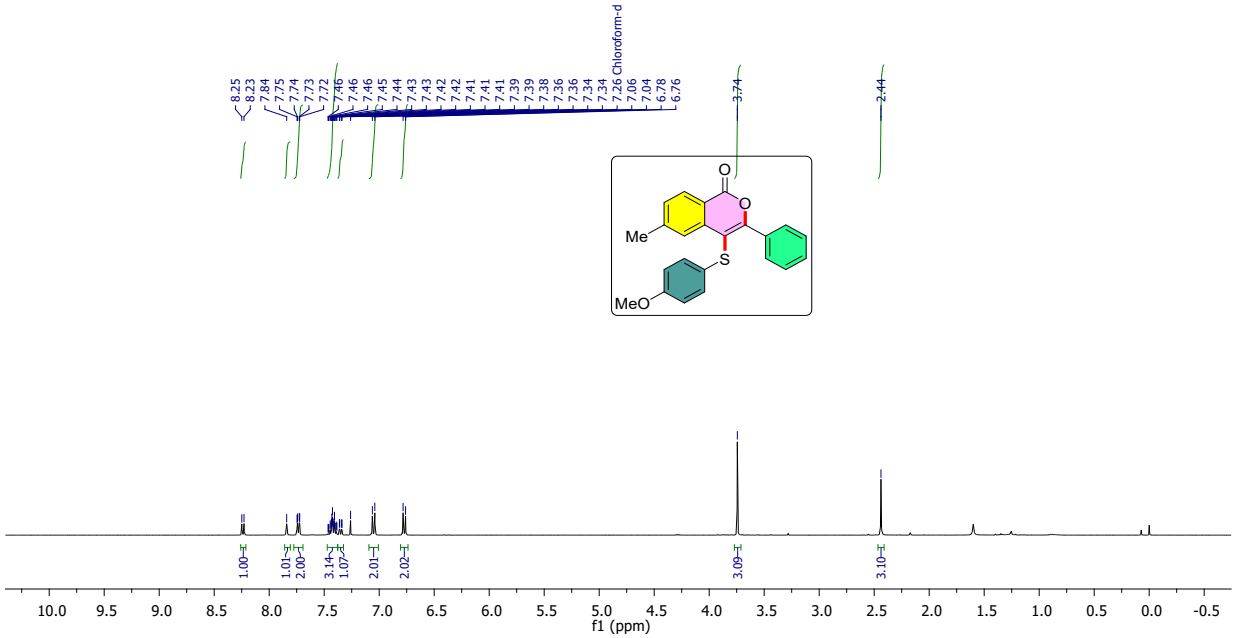
^{1}H-NMR (400 MHz) spectrum of **3uc** in $CDCl_3$



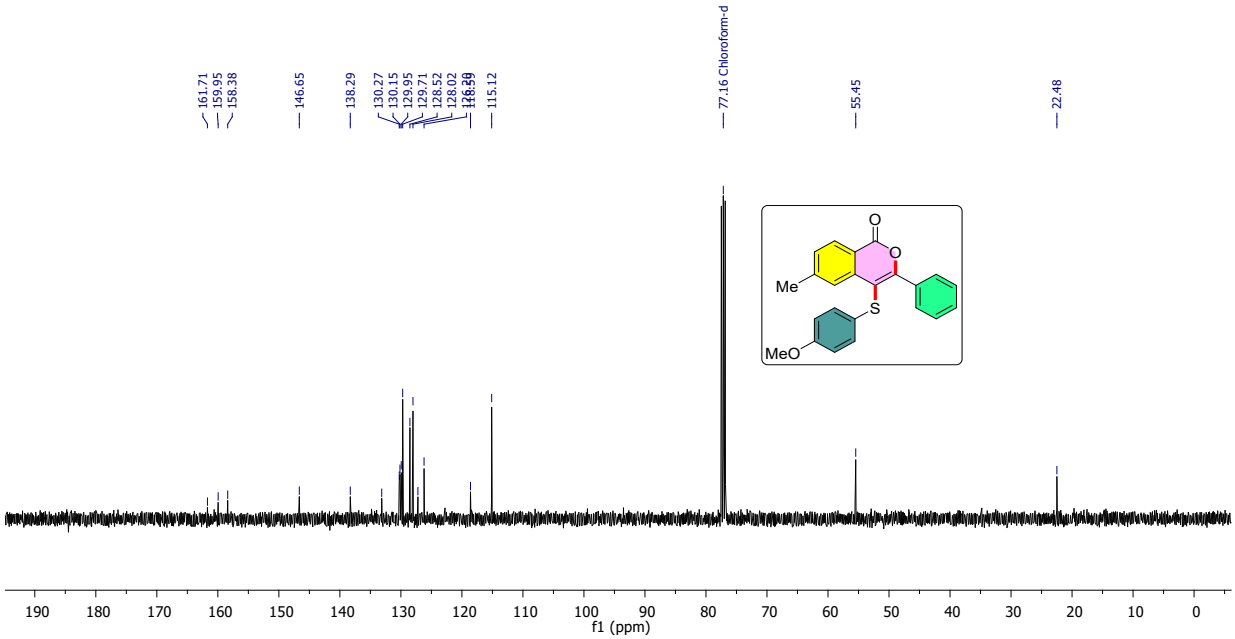
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3uc** in $CDCl_3$



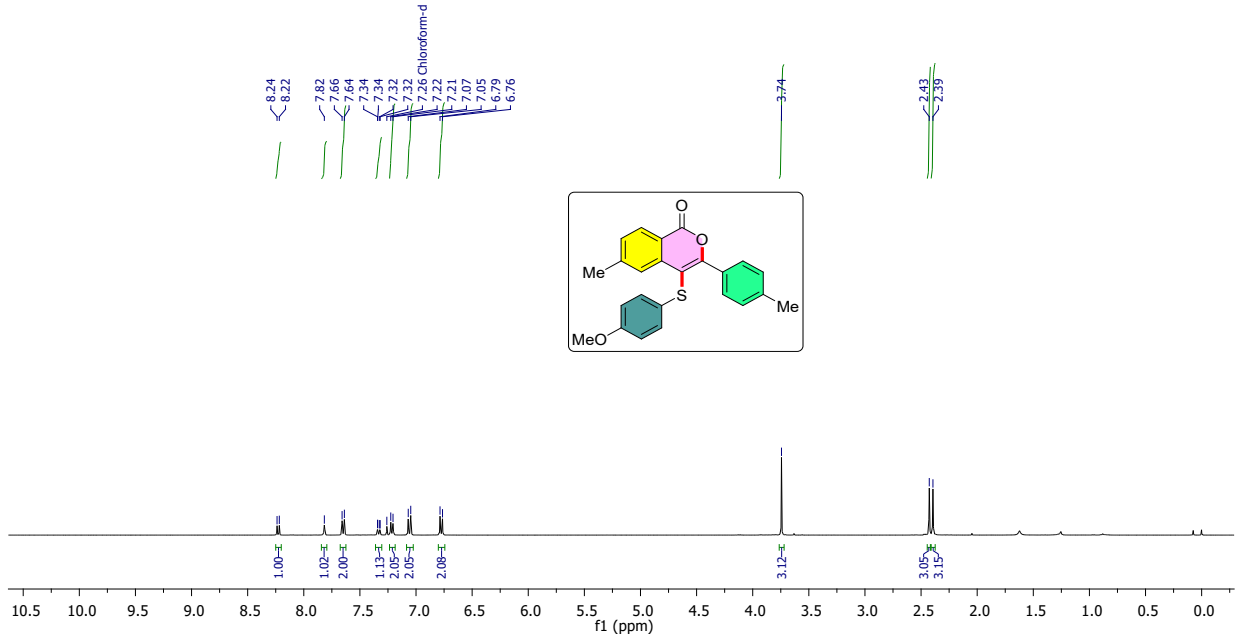
^{1}H-NMR (400 MHz) spectrum of **3vc** in $CDCl_3$



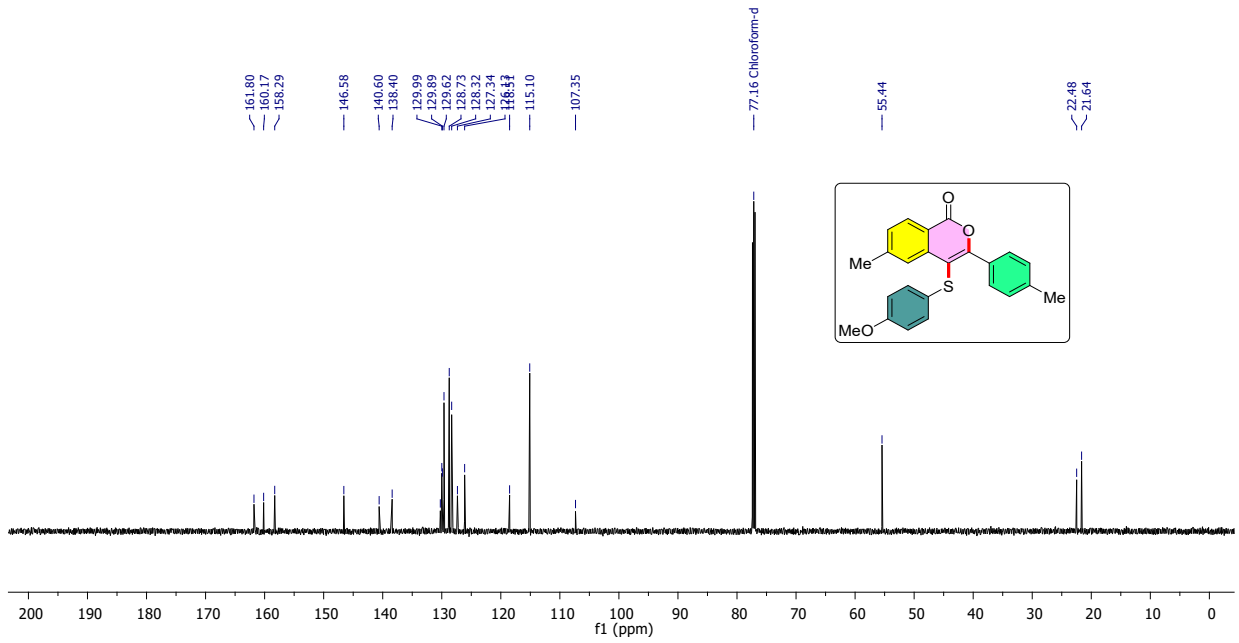
^{13}C{^{1}H}-NMR (101 MHz) spectrum of **3vc** in $CDCl_3$



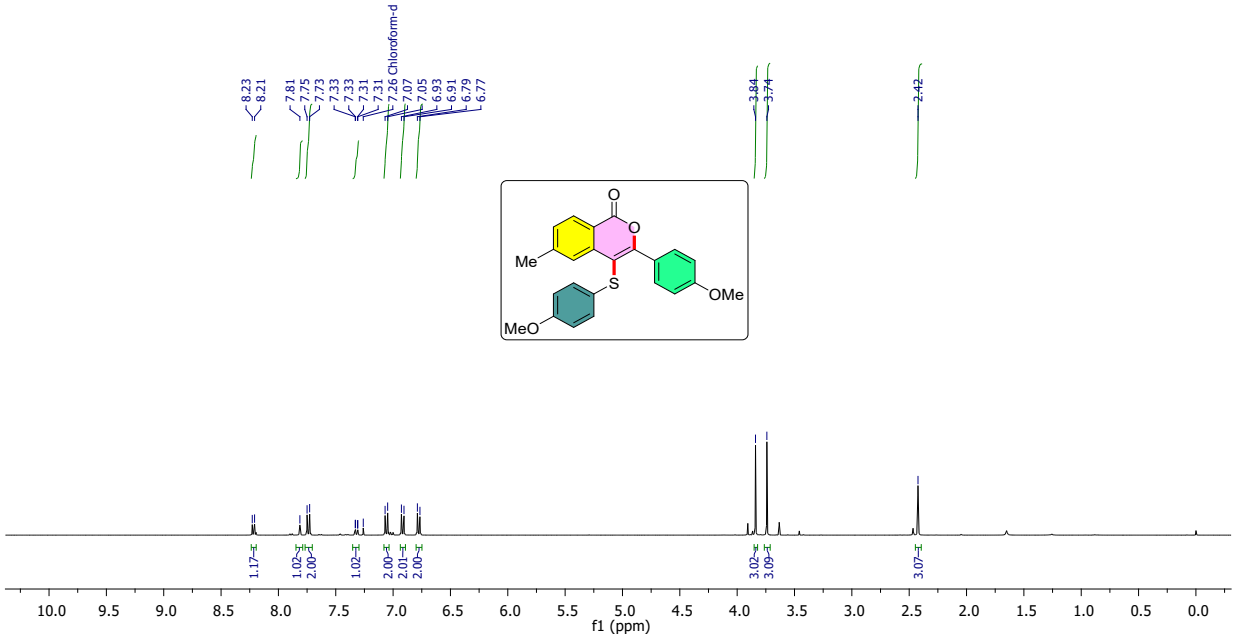
^{1}H-NMR (400 MHz) spectrum of **3wc** in $CDCl_3$



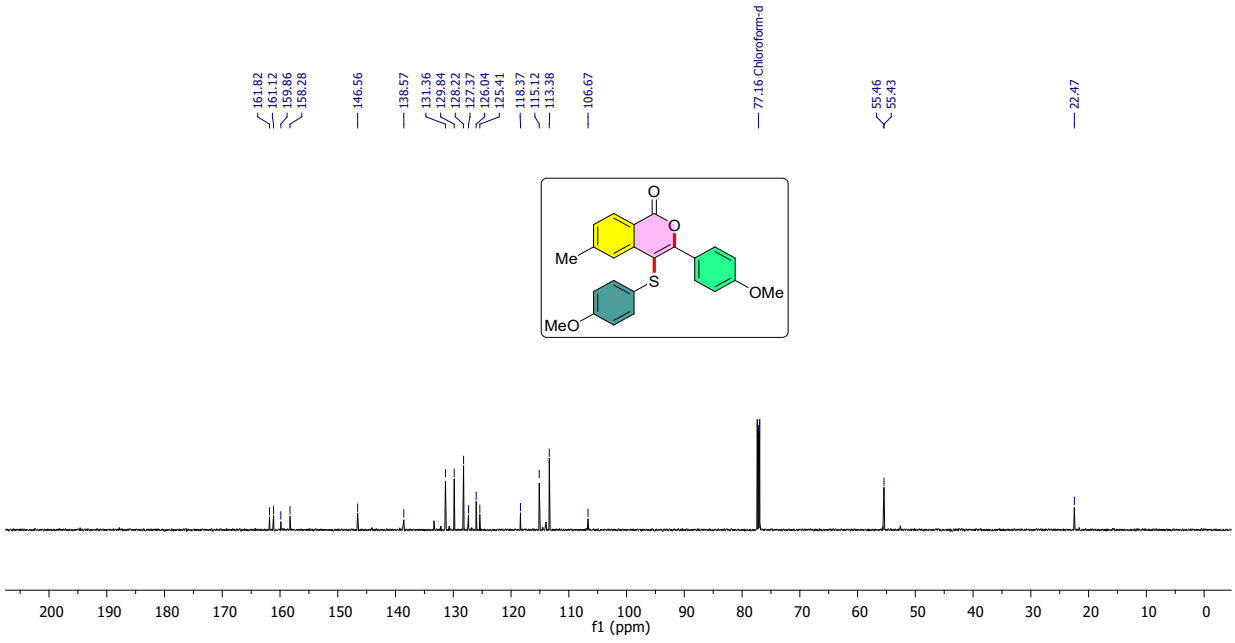
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3wc** in $CDCl_3$



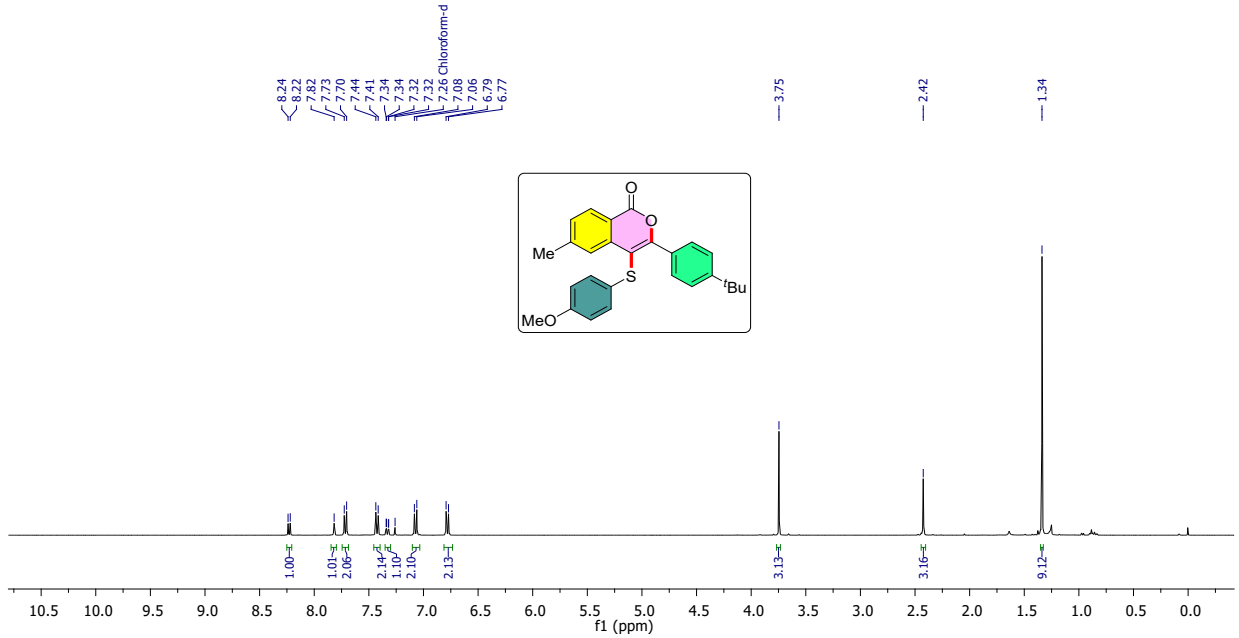
^{1}H-NMR (400 MHz) spectrum of **3xc** in $CDCl_3$



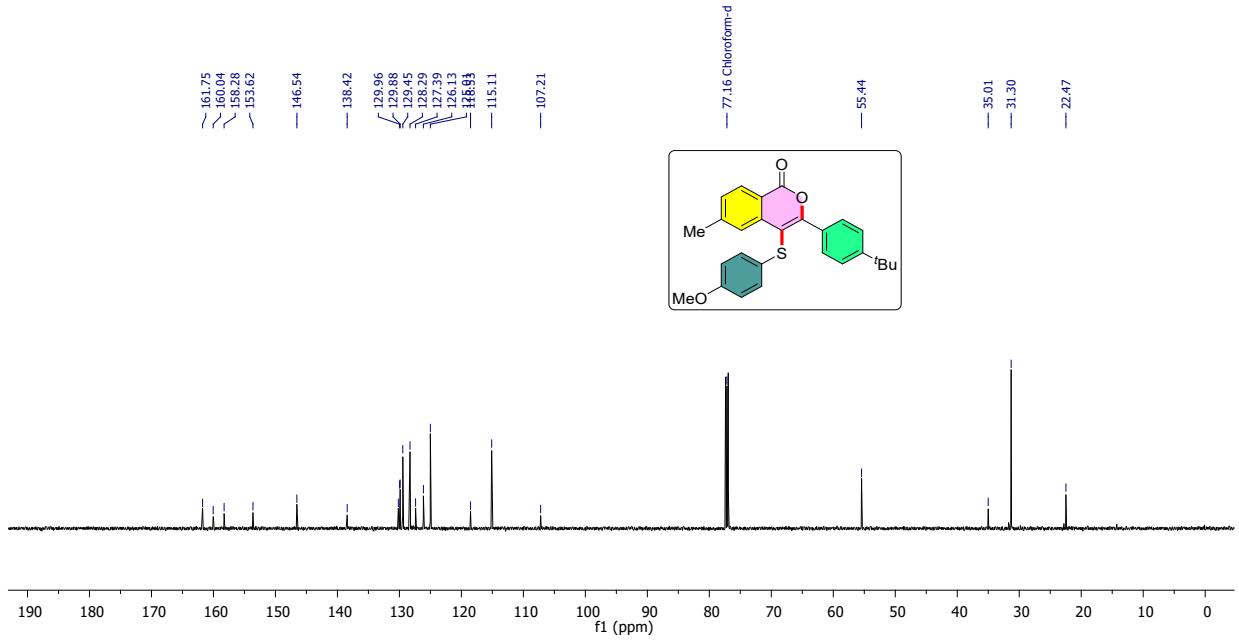
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3xc** in $CDCl_3$



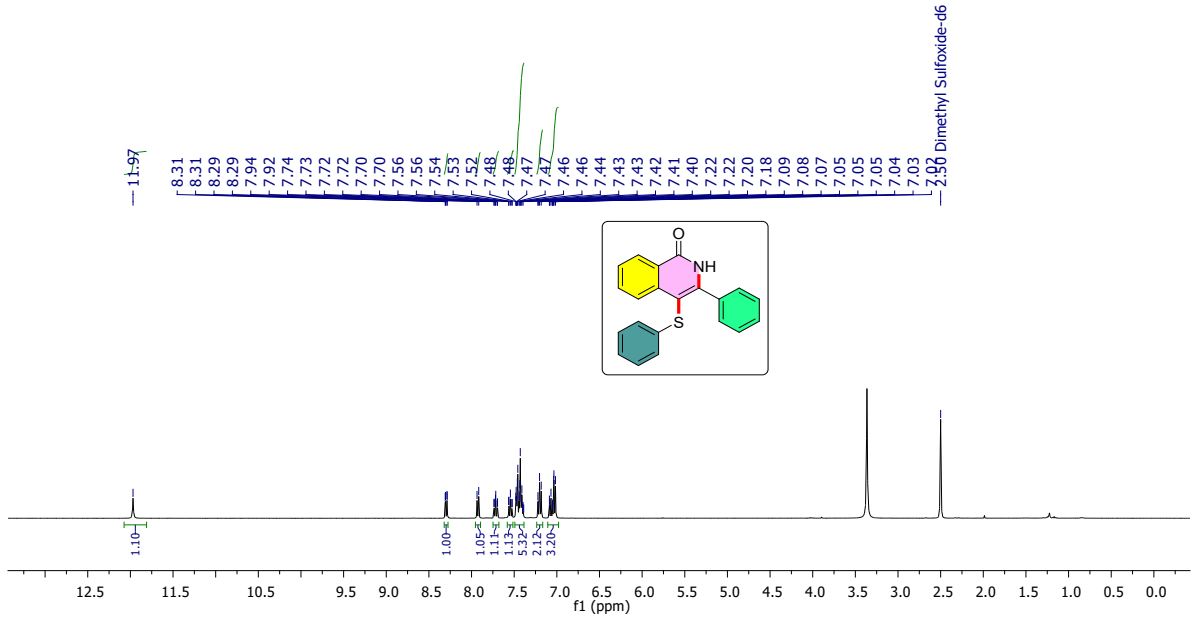
^{1}H-NMR (400 MHz) spectrum of **3yc** in $CDCl_3$



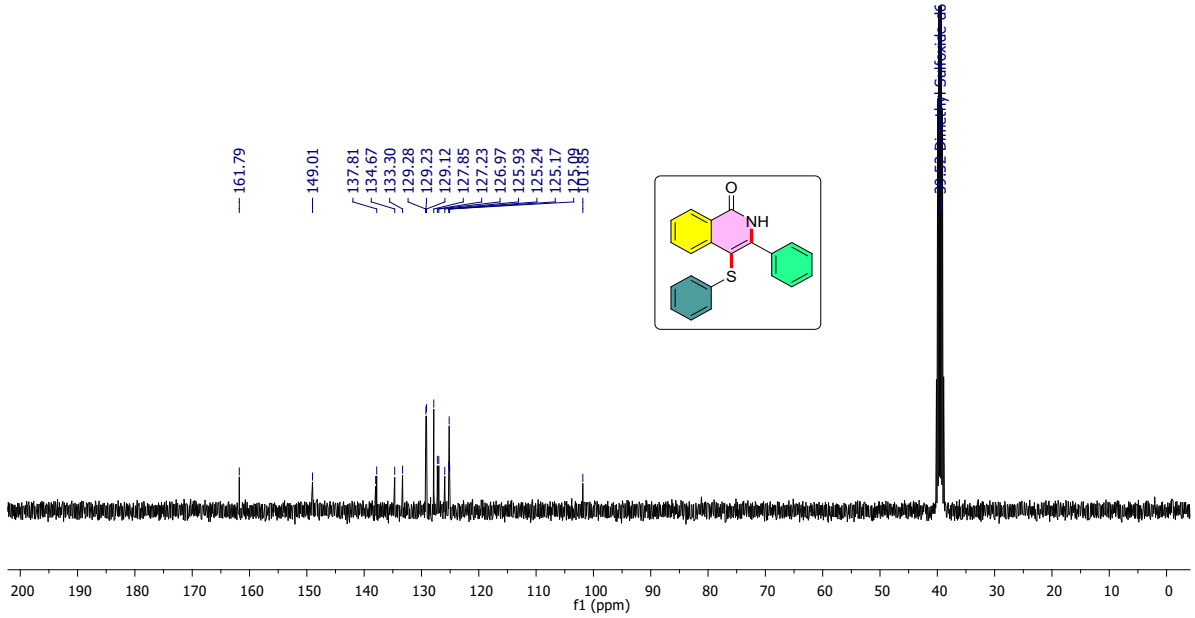
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **3yc** in $CDCl_3$



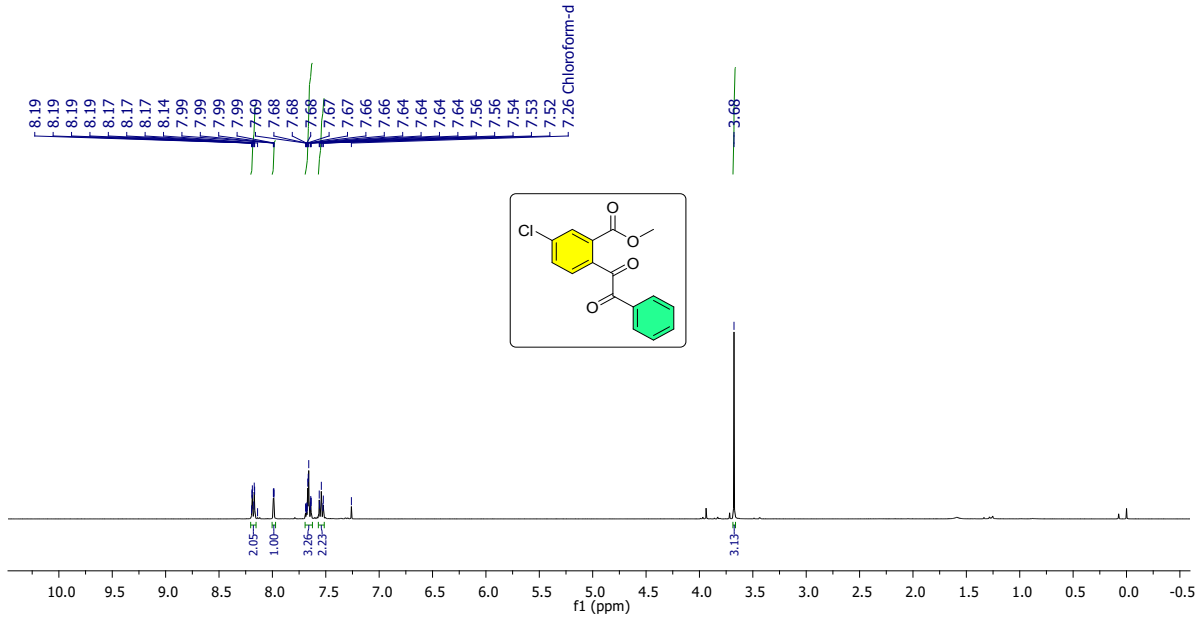
^{1}H-NMR (400 MHz) spectrum of **7** in DMSO-d_6



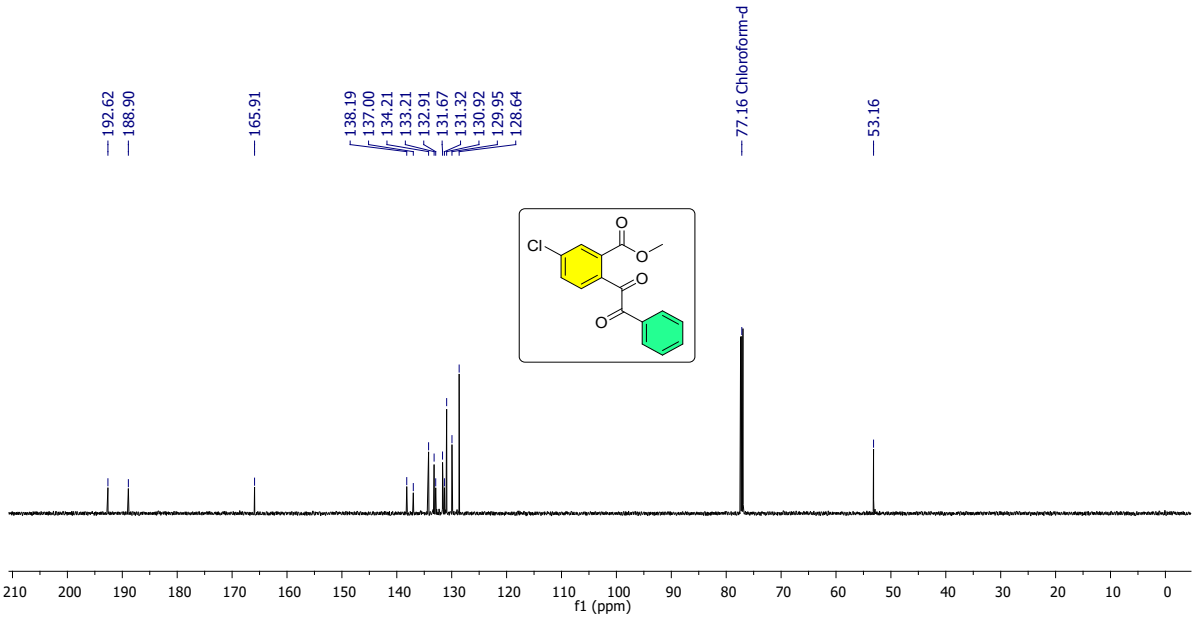
^{13}C{^{1}H}-NMR (151 MHz) spectrum of **7** in DMSO-d_6



^{1}H-NMR (400 MHz) spectrum of **9** in $CDCl_3$



^{13}C{^{1}H}-NMR (151 MHz) spectrum of **9** in $CDCl_3$



Crystal structure data:

X-Ray crystal structure of compound **3cc**: Crystal of compound **3cc** were obtained by dissolving the product in CH_2Cl_2/CH_3CN (1:1) and allowing the solvent to slowly evaporate at room temperature. The crystal structure information for this compound has been deposited at the Cambridge Crystallographic Data Centre. **CCDC No.** 2344684 contains the crystal structure information of this compound and can be obtained free of charge *via* http://www.ccdc.cam.ac.uk

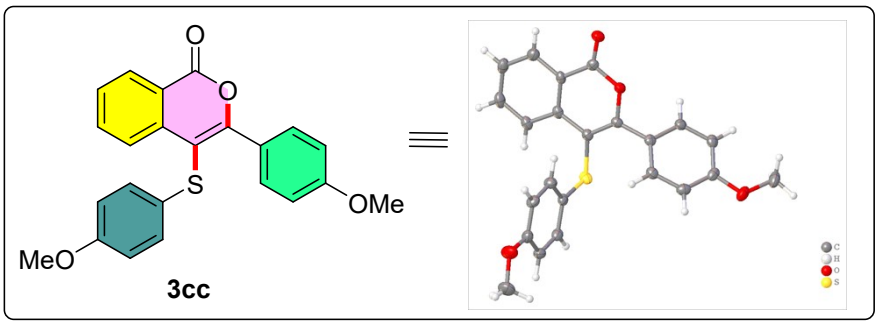


Figure S2: X-ray structure of the product **3cc** with the ellipsoids drawn at the 50% probability level (**CCDC**-2344684).

Table 4S: Crystal data and structure refinement for **3cc** (**CCDC**-2344684)

Table 1 Crystal data and structure refinement for 3cc.	
Identification code	**3cc**
Empirical formula	$C_{23}H_{18}O_4S$
Formula weight	390.43
Temperature/K	149.00
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	8.3863(19)
b/Å	18.506(3)
c/Å	12.780(2)
α/°	90
β/°	108.742(7)
γ/°	90
Volume/Å^3	1878.3(6)
Z	4
ρ_{calc}g/cm^3	1.381
μ/mm^{-1}	0.200
F(000)	816.0
Crystal size/mm^3	0.045 × 0.036 × 0.012
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	4.402 to 49.99
Index ranges	$-9 \le h \le 9$, $-21 \le k \le 22$, $-15 \le l \le 15$
Reflections collected	22124

Independent reflections	3304 [R_{int} = 0.0981, R_{sigma} = 0.0593]
Data/restraints/parameters	3304/0/256
Goodness-of-fit on F^2	1.015
Final R indexes [I>=2σ (I)]	R_1 = 0.0421, wR_2 = 0.0895
Final R indexes [all data]	R_1 = 0.0659, wR_2 = 0.1004
Largest diff. peak/hole / e $Å^{-3}$	0.22/-0.23

References:

[1] P. Zhou, W.-T. Yang, W.-J. Hao, B. Jiang, *Org. Lett.* **2023**, *25*, 8495–8500.
[2] X. Zhang, C. Liu, W. Pang, X. Gu, W. Wei, Z. Zhang, H. Chen, T. Liang, *Org. Chem. Front.* **2024**, *11*, 871–884.
[3] Z. Li, J. Hong, L. Weng, X. Zhou, *Tetrahedron* **2012**, *68*, 1552–1559.
[4] L. Xing, Y. Zhang, B. Li, Y. Du, *Org. Lett.* **2019**, *21*, 3620–3624.
[5] A. Saha, E. Ramesh, A. K. Sahoo, *Adv. Synth. Catal.* **2022**, *364*, 3496–3500.