Supporting Information

For

Ruthenium-catalyzed Heck coupling of 3-arylidene-oxindoles with alkenes: a facile synthesis of 3-allylidene-2(3H)-oxindoles

*Mohan Prabhakaran, Ramesh Sanjana and Kanniyappan Parthasarathy **

Department of Organic Chemistry, University of Madras, Guindy Campus, Tamil Nadu, Chennai-600 025, India,
E-mail: kparthasarathy@unom.ac.in

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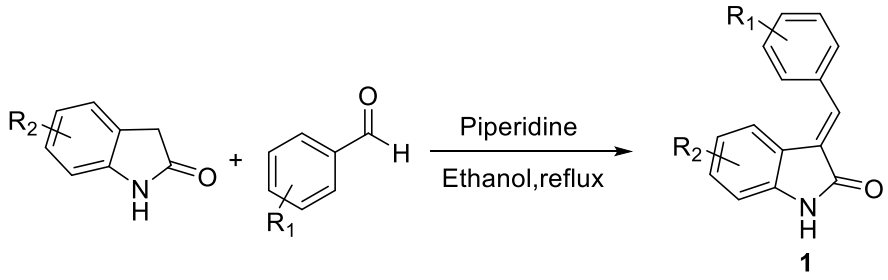
General information

All product mixtures were analyzed by thin layer chromatography using aluminum foil backed silica TLC plates with a fluorescent indicator from Merck. UV-active compounds were detected with a UV lamp (λ = 254 nm). For flash column chromatography, silica gel was used as stationary phase. ^{1}H and ^{13}C NMR spectra were recorded on Bruker 300MHz in deuterated chloroform at 25 °C. Chemical shifts (δ) are reported in ppm, and spin-spin coupling constants (*J*) are given in Hz, while multiplicities are abbreviated by br s (broad singlet), s (singlet), d (doublet), t (triplet), q (quartet) and m (multiplet). High resolution mass spectra (HRMS) were recorded on Waters Xevo G2S QTof (ESI) instrument. All solvents were dried according to known methods and distilled prior to use.[1] The ruthenium catalyst, alkenes and all other reagents were purchased from Sigma-Aldrich, TCI or BLD chemicals and used without further purification.

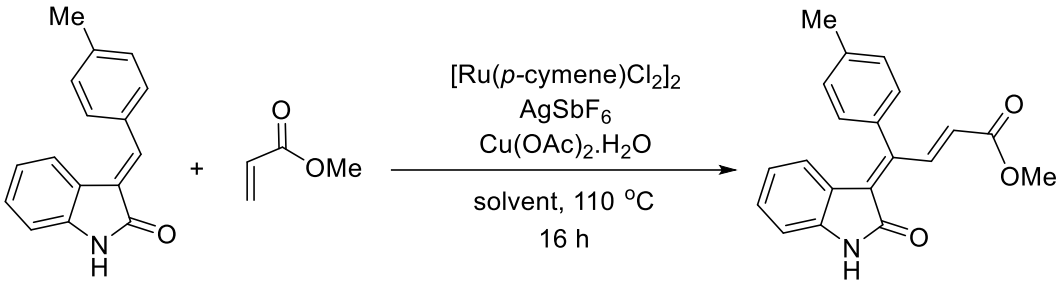
General procedure for the preparation of compound 1.



1

According to the literature procedure:[2] To a solution of oxindole (1g, 1equiv.), aromatic aldehyde (1.1 equiv.) and piperidine (0.1 equiv.) in ethanol (30 mL) was heated under reflux for 2 h. Upon completion (based on TLC), the reaction was allowed to cool room temperature and the solvent was evaporated. The crude was washed with MeOH:hexane (1:9) to give corresponding yellow solid compound **1**.

Effect of solvents [a]

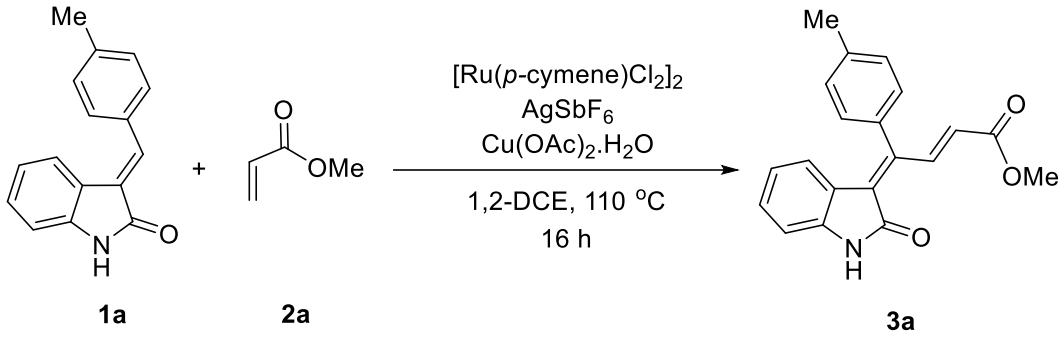


1a **2a** **3a**

entry	Solvent	yield[b] (%)[b]
1	**1,2-DCE**	**78**
2	ACN	45
3	1,4-Dioxane	28
4	Toluene	33
5	THF	16
6	Methanol	10
7	DMF	n.d
8	DMSO	n.d

[a]Reaction condition; all reactions were carried out (*E*)-3-(4-methylbenzylidene)indolin-2-one **1a** (0.30 mmol), methyl acrylate **2a** (0.33 mmol), Ru-cat (2.5 mol%), $Cu(OAc)_2.H_2O$ (1 eq), $AgSbF_6$ (10 mol%), solvent (1.5 mL), at 110 °C for 16 h, under N_2. [b]isolated yields. [n.d] not detected.

General procedure for the ruthenium-catalyzed oxidative vinylic olefination:

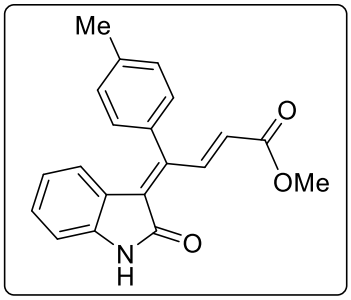


1a **2a** **3a**

A Schlenk tube (20 mL) was loaded with **1a** (0.30 mmol), methyl acrylate **2a** (0.33 mmol), [Ru(*p*-cymene)Cl_2]$_2$ (2.5 mol%), $AgSbF_6$ (10 mol %), and $Cu(OAc)_2.H_2O$ (1 equiv.). To this 1,2-DCE (2 mL) was added through a syringe and the reaction mixture was allowed to stir at 110 °C for 16 h under N_2. The reaction was monitored by TLC and the reaction mixture was diluted with CH_2Cl_2 (10 mL). The mixture was filtered through a Celite pad and the Celite pad was washed with dichloromethane (3 x 20 mL). The combined filtrate was concentrated and the residue was purified by a silica gel column chromatography using hexane/ethyl acetate (92:8) as eluent to give pure product **3a**. Similar experimental procedure was applied for the coupling **3b-z** and **3a-c**.

Spectral data

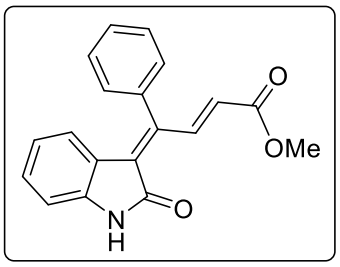
Methyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3a)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (230-232 °C), yield: (74 mg, 73%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.40 (d, *J* = 15.9 Hz, 1H), 8.95 (s, 1H), 7.24 (d, *J* = 7.8 Hz, 1H), 7.05 – 7.00 (m, 3H), 6.78 (d, *J* = 7.5 Hz, 1H), 6.53 (t, *J* = 7.8 Hz, 1H), 5.83 (d, *J* = 7.8 Hz, 1H), 5.68 (d, *J* = 15.6 Hz, 1H), 3.69 (s, 3H), 2.39 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ

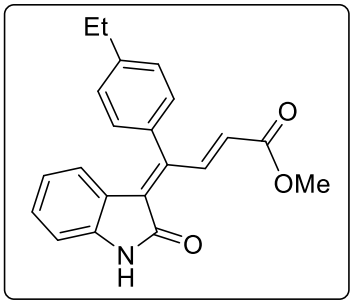
169.3, 167.1, 147.6, 141.6, 141.5, 138.8, 133.3, 130.1, 130.0, 128.5, 128.1, 128.0, 125.0, 123.2, 121.7, 109.8, 51.8, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{20}H_{18}NO_3$ 320.1286, Found 320.1288.

Methyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-phenylbut-2-enoate (3b)



Red orange solid, eluent: (hexane/ethyl acetate, 94:6), m.p. (234-236 ^oC), yield: (65 mg, 67%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.51 (d, *J* = 15.9 Hz, 1H), 9.07 (s, 1H), 7.53 – 7.51 (m, 3H), 7.23 – 7.20 (m, 2H), 7.11 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.5 Hz, 1H), 6.58 (t, *J* = 7.8 Hz, 1H), 5.79 (d, *J* = 8.1 Hz, 1H), 5.74 (d, *J* = 15.9 Hz, 1H), 3.77 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 169.2, 167.0, 147.2, 141.5, 141.3, 136.2, 130.0, 129.4, 128.8, 128.5, 128.2, 128.1, 124.9, 123.0, 121.7, 109.9, 51.8 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{19}H_{16}NO_3$ 306.1130, Found 306.1134.

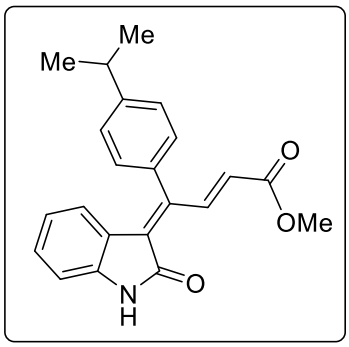
Methyl (*E*)-4-(4-ethylphenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3c)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (158-160 ^oC), yield: (70 mg, 74%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.40 (d, *J* = 15.9 Hz, 1H), 8.77 (s, 1H), 7.27 (d, *J* = 8.1 Hz, 2H), 7.03 (t, *J* = 8.1 Hz, 3H), 6.77 (d, *J* = 7.5 Hz, 1H), 6.53 (t, *J* = 7.8 Hz, 1H), 5.80 (d, *J* = 7.8 Hz, 1H), 5.69 (d, *J* = 15.9 Hz, 1H), 3.70 (s, 3H), 2.69 (q, 2H), 1.25 (t, 3H) ppm. **^{13}C NMR (75**

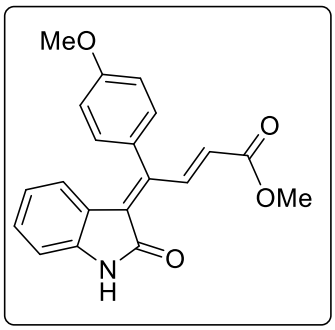
MHz, $CDCl_3$): δ 169.2, 167.1, 147.7, 145.1, 141.6, 141.4, 133.5, 129.9, 128.9, 128.5, 128.2, 128.0, 125.0, 123.2, 121.8, 109.8, 51.8, 28.7, 15.5 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{21}H_{20}NO_3$ 334.1443, Found 334.1448.

Methyl (*E*)-4-(4-isopropylphenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3d)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (184-186 ºC), yield: (65 mg, 71%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.41 (d, *J* = 15.6 Hz, 1H), 8.92 (s, 1H), 7.29 (d, *J* = 8.1 Hz, 2H), 7.06 – 7.00 (m, 3H), 6.78 (d, *J* = 7.5 Hz, 1H), 6.51 (t, *J* = 7.8 Hz, 1H), 5.75 (d, *J* = 8.1 Hz, 1H), 5.71 (d, *J* = 15.6 Hz, 1H), 3.70 (s, 3H), 2.99 – 2.90 (m, 1H), 1.27 (s, 3H), 1.25 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 169.3, 167.1, 149.8, 147.7, 141.6, 141.4, 133.6, 129.9, 128.5, 128.2, 127.4, 124.9, 123.2, 121.7, 109.8, 51.9, 34.0, 24.0 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{22}H_{22}NO_3$ 348.1599, Found 348.1602.

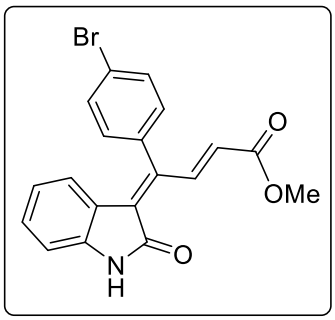
Methyl (*E*)-4-(4-methoxyphenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3e)



Red orange solid, eluent: (hexane/ethyl acetate, 90:10), m.p. (218-220 ºC), yield: (67 mg, 72%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.37 (d, *J* = 15.9 Hz, 1H), 8.71 (s, 1H), 7.08 – 7.04 (m, 3H), 6.99 (t, *J* = 9 Hz, 2H), 6.77 (d, *J* = 7.8 Hz, 1H), 6.56 (t, *J* = 7.8 Hz, 1H), 5.91 (d, *J* = 7.5 Hz,

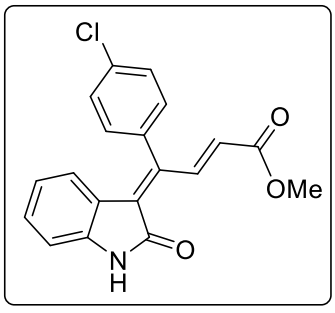
1H), 5.71 (d, *J* = 15.6 Hz, 1H), 3.84 (s, 3H), 3.71 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 169.1, 167.1, 160.0, 147.4, 141.9, 141.3, 129.9, 129.7, 128.5, 128.4, 128.1, 124.9, 123.3, 121.8, 114.8, 109.8, 55.4, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{20}H_{18}NO_4$ 336.1235, Found 336.1234.

Methyl (*E*)-4-(4-bromophenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3f)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (230-232 ºC), yield: (64 mg, 71%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.39 (d, *J* = 15.9 Hz, 1H), 8.59 (s, 1H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.09 – 7.03 (m, 3H), 6.78 (d, *J* = 7.8 Hz, 1H), 6.58 (t, *J* = 7.8 Hz, 1H), 5.85 (d, *J* = 8.1 Hz, 1H), 5.61 (d, *J* = 15.9 Hz, 1H), 3.70 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.8, 166.8, 145.8, 141.5, 141.0, 135.2, 132.8, 130.4, 130.1, 128.5, 128.2, 124.9, 123.2, 122.8, 122.0, 110.0, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{19}H_{15}BrNO_3$ 384.0235, Found 384.0236.

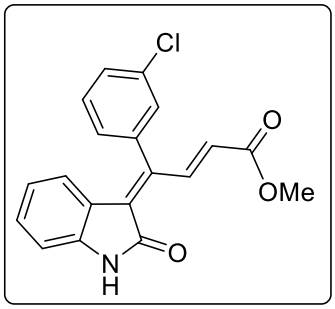
Methyl (*E*)-4-(4-chlorophenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3g)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (238-240 ºC), yield: (65 mg, 70%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.45 (d, *J* = 15.6 Hz, 1H), 8.34 (s, 1H), 7.53 (d, *J* = 8.1 Hz,

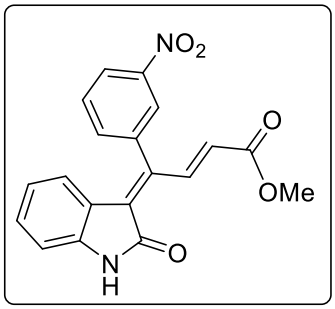
2H), 7.19 – 7.11 (m, 3H), 6.82 (d, *J* = 7.5 Hz, 1H), 6.65 (t, *J* = 7.8 Hz, 1H), 5.91 (d, *J* = 7.8 Hz, 1H), 5.69 (d, *J* = 15.9 Hz, 1H), 3.78 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 168.5, 166.8, 145.8, 141.4, 141.0, 135.1, 134.7, 130.4, 129.9, 129.8, 128.5, 128.1, 124.9, 122.8, 122.0, 109.8, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** [M + H]$^+$ Calcd for $C_{19}H_{15}ClNO_3$ 340.0740, Found 340.0738.

Methyl (*E*)-4-(3-chlorophenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3h)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (216-218 ^{o}C), yield: (56 mg, 60%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.46 (d, *J* = 15.9 Hz, 1H), 8.82 (s, 1H), 7.52 – 7.45 (m, 2H), 7.23 (s, 1H), 7.14 (t, *J* = 7.8 Hz, 2H), 6.86 (d, *J* = 7.8 Hz, 1H), 6.64 (t, *J* = 7.8 Hz, 1H), 5.85 (d, *J* = 7.8 Hz, 1H), 5.70 (d, *J* = 15.9 Hz, 1H), 3.78 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 168.8, 166.8, 145.3, 141.6, 140.8, 138.0, 135.5, 130.9, 130.5, 129.1, 128.5, 128.3, 126.5, 124.9, 122.6, 122.0, 110.0, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** [M + H]$^+$ Calcd for $C_{19}H_{15}ClNO_3$ 340.0740, Found 340.0735.

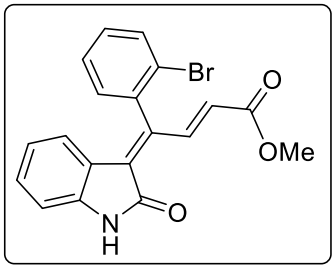
Methyl (*E*)-4-(3-nitrophenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3i)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (214-216 ^{o}C), yield: (38 mg, 41%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.42 (d, *J* = 15.9 Hz, 1H), 8.35 (d, *J* = 8.1 Hz, 1H), 8.30 (s, 1H),

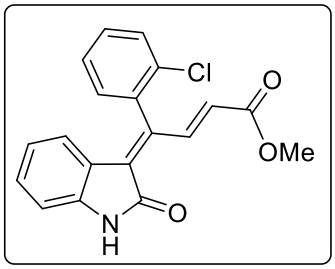
8.07 (s, 1H), 7.70 (t, *J* = 8.1 Hz, 1H), 7.54 (d, *J* = 7.2 Hz, 1H), 7.08 (t, *J* = 7.8 Hz, 1H), 6.77 (d, *J* = 7.5 Hz, 1H), 6.53 (t, *J* = 7.8 Hz, 1H), 5.65 (d, *J* = 7.8 Hz, 1H), 5.52 (d, *J* = 15.9 Hz, 1H), 3.71 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.4, 166.5, 148.9, 143.7, 141.7, 140.5, 137.9, 134.8, 130.9, 130.8, 128.9, 128.5, 124.5, 123.9, 123.6, 122.2, 122.1, 110.3, 52.0 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{19}H_{15}N_2O_5$ 351.0980, Found 351.0986.

Methyl (*E*)-4-(2-bromophenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3j)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (160-162 ºC), yield: (32 mg, 36%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.38 (d, *J* = 15.9 Hz, 1H), 8.59 (s, 1H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.41 (t, *J* = 7.5 Hz, 1H), 7.32 (t, *J* = 7.5 Hz, 1H), 7.12 – 7.03 (m, 2H), 6.77 (d, *J* = 7.8 Hz, 1H), 6.55 (t, *J* = 7.8 Hz, 1H), 5.66 (d, *J* = 7.8 Hz, 1H), 5.58 (d, *J* = 15.9 Hz, 1H), 3.70 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.7, 166.9, 145.3, 141.5, 139.6, 137.0, 133.7, 130.5, 130.4, 129.9, 128.6, 128.5, 127.6, 124.5, 122.7, 122.2, 109.9, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{19}H_{15}BrNO_3$ 384.0235, Found 384.0239.

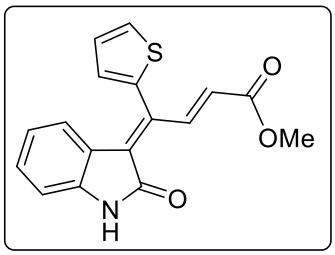
Methyl (*E*)-4-(2-chlorophenyl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3k)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (156-158 ºC), yield: (41 mg, 44%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.48 (d, *J* = 15.6 Hz, 1H), 8.88 (s, 1H), 7.56 (d, *J* = 7.5 Hz, 1H), 7.50 – 7.41 (m, 2H), 7.20 (d, *J* = 7.2 Hz, 1H), 7.14 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 7.8 Hz,

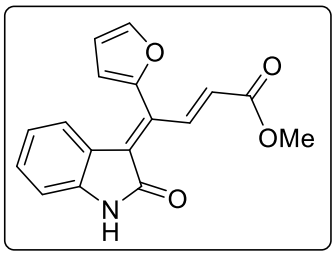
1H), 6.63 (t, *J* = 7.8 Hz, 1H), 5.75 (d, *J* = 7.8 Hz, 1H), 5.65 (d, *J* = 15.6 Hz, 1H), 3.78 ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.9, 166.9, 143.8, 141.6, 139.8, 135.0, 132.6, 130.6, 130.5, 130.4, 129.9, 128.9, 127.9, 127.5, 124.4, 122.7, 122.2, 110.0, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** [M + H]$^+$ Calcd for $C_{19}H_{15}ClNO_3$ 340.0740, Found 340.0743.

Methyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(thiophen-2-yl)but-2-enoate (3l)



Dark red solid, eluent: (hexane/ethyl acetate, 90:10), m.p. (228-230 ^{o}C), yield: (49 mg, 51%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.44 (d, *J* = 15.6 Hz, 1H), 8.75 (s, 1H), 7.58 (d, *J* = 4.8 Hz, 1H), 7.24 – 7.15 (m, 2H), 7.01 (d, *J* = 2.7 Hz, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 6.72 (t, *J* = 7.8 Hz, 1H), 6.01 (d, *J* = 7.5 Hz, 1H), 5.98 (d, *J* = 15.6 Hz, 1H), 3.81 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.5, 167.0, 141.7, 141.6, 139.8, 136.1, 130.6, 130.4, 128.4, 127.9, 127.8, 127.7, 125.2, 122.8, 122.1, 109.9, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** [M + H]$^+$ Calcd for $C_{17}H_{14}NO_3S$ 312.0694, Found 312.0709.

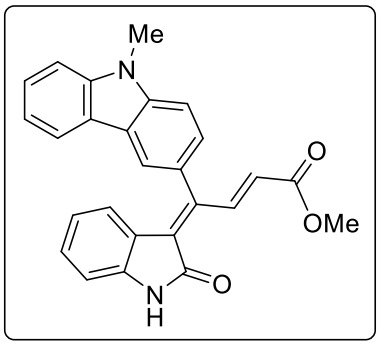
Methyl (*E*)-4-(furan-2-yl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3m)



Dark red solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (222-224 ^{o}C), yield: (46 mg, 49%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.17 (d, *J* = 15.9 Hz, 1H), 8.05 (s, 1H), 7.64 (d, *J* = 1.2 Hz, 1H), 7.18 (t, *J* = 7.5 Hz, 1H), 6.81 (t, *J* = 7.8 Hz, 2H), 6.66 – 6.64 (m, 1H), 6.60 (d, *J* = 3.3 Hz, 1H), 6.15 (d, *J* = 15.9 Hz, 1H), 6.08 (d, *J* = 7.5 Hz, 1H), 3.80 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.3, 166.9, 148.0, 143.5, 141.4, 140.4, 134.5, 130.5, 129.5, 127.8, 124.9, 122.6,

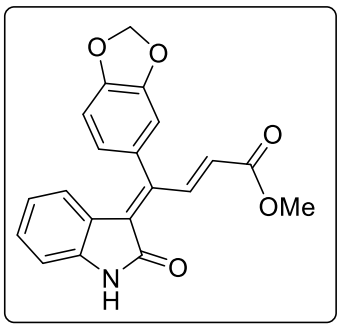
122.2, 113.2, 112.0, 109.7, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{17}H_{14}NO_4$ 296.0922, Found 296.0930.

Methyl (*E*)-4-(9-methyl-9H-carbazol-3-yl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3n)



Red orange solid, eluent: (hexane/ethyl acetate, 88:12), m.p. (250-252 ºC), yield: (47 mg, 53%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.57 (d, *J* = 15.6 Hz, 1H), 8.46 (s, 1H), 8.08 (d, *J* = 7.8 Hz, 1H), 7.98 (s, 1H), 7.58 – 7.52 (m, 2H), 7.48 (d, *J* = 8.4 Hz, 1H), 7.34 – 7.26 (m, 2H), 7.08 (t, *J* = 7.8 Hz, 1H), 6.84 (d, *J* = 7.5 Hz, 1H), 6.48 (t, *J* = 7.5 Hz, 1H), 5.83 (d, *J* = 7.2 Hz, 1H), 5.80 (d, *J* = 15.6 Hz, 1H), 3.98 (s, 3H), 3.78 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 169.2, 167.2, 148.7, 142.5, 141.4, 141.3, 141.0, 129.8, 128.8, 128.3, 126.7, 126.4, 126.0, 125.0, 123.5, 123.4, 122.5, 121.8, 120.6, 120.4, 119.4, 109.7, 109.5, 108.8, 51.8, 29.3 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{26}H_{21}N_2O_3$ 409.1552, Found 409.1551.

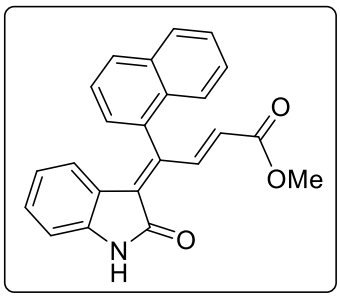
Methyl (*E*)-4-(benzo[*d*][1,3]dioxol-5-yl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3o)



Red orange solid, eluent: (hexane/ethyl acetate, 90:10), m.p. (206-208 ºC), yield: (47 mg, 51%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.43 (d, *J* = 15.6 Hz, 1H), 8.48 (s, 1H), 7.15 (t, *J* = 7.5 Hz, 1H), 6.98 (d, *J* = 7.8 Hz, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 6.72 – 6.70 (m, 3H), 6.12 – 6.08 (m, 3H), 5.84

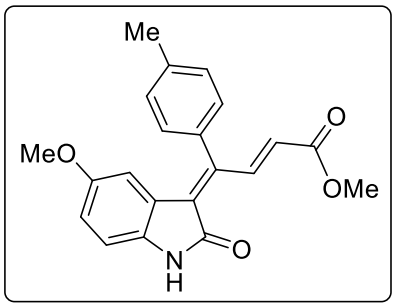
(d, *J* = 15.9 Hz, 1H), 3.80 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.6, 167.0, 148.5, 148.1, 146.9, 141.4, 141.2, 130.1, 129.7, 128.5, 128.0, 125.1, 123.1, 121.9(2C), 109.6, 109.4, 108.9, 101.5, 51.9 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{20}H_{16}NO_5$ 350.1028, Found 350.1029.

Methyl (*E*)-4-(naphthalen-1-yl)-4-((*E*)-2-oxoindolin-3-ylidene)but-2-enoate (3p)



Red orange solid, eluent: (hexane/ethyl acetate, 90:10), m.p. (226-228 ºC), yield: (57 mg, 62%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.68 (d, *J* = 15.6 Hz, 1H), 8.53 (s, 1H), 8.01 (d, *J* = 8.4 Hz, 1H), 7.95 (d, *J* = 8.1 Hz, 1H), 7.61 (q, *J* = 7.8, 8.1 Hz, 2H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.39 (t, *J* = 7.5 Hz, 1H), 7.32 (d, *J* = 6.9 Hz, 1H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.81 (d, *J* = 7.5 Hz, 1H), 6.39 (t, *J* = 7.8 Hz, 1H), 5.60 (d, *J* = 15.9 Hz, 1H), 5.38 (d, *J* = 7.8 Hz, 1H), 3.72 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.8, 167.0, 145.9, 141.3, 140.9, 133.8, 133.7, 130.5, 130.1, 129.1, 128.4, 127.1, 126.6, 126.0, 125.9, 125.0, 124.8, 122.8, 122.0, 109.6, 51.8 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{23}H_{18}NO_3$ 356.1286, Found 356.1286.

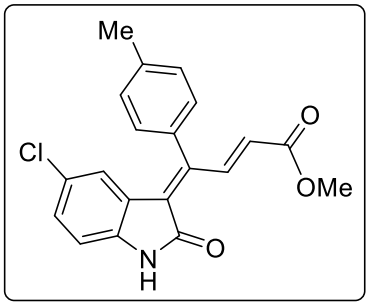
Methyl (*E*)-4-((*E*)-5-methoxy-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3q)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (232-234 ºC), yield: (54 mg, 59%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.38 (d, *J* = 15.9 Hz, 1H), 8.38 (s, 1H), 7.27 (d, *J* = 7.8 Hz,

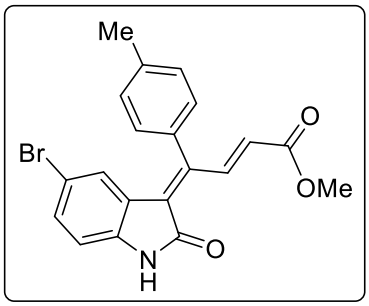
2H), 7.03 (d, *J* = 7.8 Hz, 2H), 6.66 – 6.57 (m, 2H), 5.71 (d, *J* = 15.9 Hz, 1H), 5.36 (d, *J* = 1.5 Hz, 1H), 3.70 (s, 3H), 3.30 (s, 3H), 2.37 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 169.1, 167.0, 154.6, 147.7, 141.4, 138.8, 135.3, 133.2, 130.1, 128.6, 128.4, 128.3, 123.9, 116.3, 110.5, 110.1, 55.1, 51.8, 21.3 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{21}H_{20}NO_4$ 350.1392, Found 350.1400.

Methyl (*E*)-4-((*E*)-5-chloro-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3r)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (240-242 ºC), yield: (56 mg, 61%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.42 (d, *J* = 15.9 Hz, 1H), 8.52 (s, 1H), 7.37 (d, *J* = 7.8 Hz, 2H), 7.10 (d, *J* = 7.8 Hz, 3H), 6.77 (d, *J* = 8.4 Hz, 1H), 5.84 – 5.79 (m, 2H), 3.79 (s, 3H), 2.50 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.5, 166.9, 149.3, 141.1, 139.5, 139.3, 132.7, 130.2, 129.6, 129.5, 127.9, 127.1, 126.9, 125.1, 124.5, 110.5, 51.9, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{20}H_{17}ClNO_3$ 354.0896, Found 354.0898.

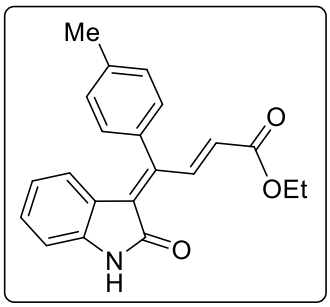
Methyl (*E*)-4-((*E*)-5-bromo-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3s)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (236-238 ºC), yield: (58 mg, 65%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.43 (d, *J* = 15.6 Hz, 1H), 9.09 (s, 1H), 7.38 (d, *J* = 7.8 Hz, 2H), 7.23 (dd, *J* = 8.4, 1.8 Hz, 1H), 7.09 (d, *J* = 7.8 Hz, 2H), 6.77 (d, *J* = 8.4 Hz, 1H), 5.91 (s,

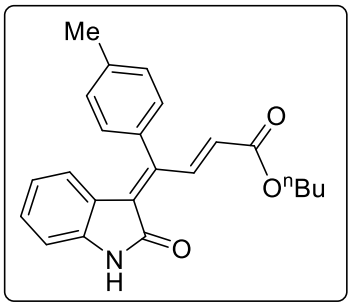
1H), 5.83 (d, *J* = 15.9 Hz, 1H), 3.79 (s, 3H), 2.50 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.8, 166.9, 149.2, 141.1, 140.1, 139.3, 132.7, 132.3, 130.2, 129.5, 127.9 (2C), 127.0, 124.9, 114.4, 111.2, 51.9, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{20}H_{17}BrNO_3$ 398.0391, Found 398.0392.

Ethyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3t)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (188-190 °C), yield: (73 mg, 74%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.41 (d, *J* = 15.6 Hz, 1H), 9.13 (s, 1H), 7.25 (d, *J* = 7.5 Hz, 2H), 7.02 (d, *J* = 7.8 Hz, 3H), 6.78 (d, *J* = 7.5 Hz, 1H), 6.53 (t, *J* = 7.5 Hz, 1H), 5.82 (d, *J* = 7.5 Hz, 1H), 5.67 (d, *J* = 15.6 Hz, 1H), 4.15 (q, 2H), 2.39 (s, 3H), 1.22 (t, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 169.4, 166.7, 147.7, 141.5 (2C), 138.7, 133.3, 130.1, 129.9, 128.9, 128.1, 128.0, 124.9, 123.2, 121.7, 109.9, 60.7, 21.4, 14.2 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{21}H_{20}NO_3$ 334.1443, Found 334.1440.

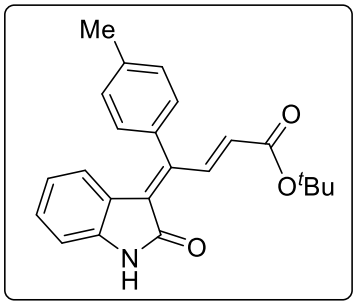
Butyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3u)



Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (174-176 °C), yield: (85 mg, 79%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.41 (d, *J* = 15.9 Hz, 1H), 8.96 (s, 1H), 7.25 (d, *J* = 7.8 Hz, 2H), 7.03 (d, *J* = 7.8 Hz, 3H), 6.77 (d, *J* = 7.5 Hz, 1H), 6.54 (t, *J* = 7.5, 7.8 Hz, 1H), 5.82 (d, *J* =

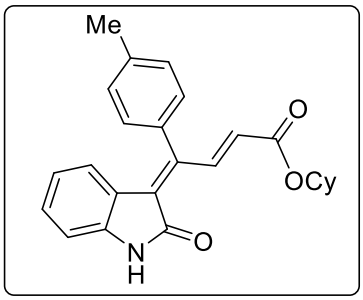
7.8 Hz, 1H), 5.68(d, *J* = 15.6 Hz, 1H), 4.10 (t, *J* = 6.6, 6.9 Hz, 1H), 2.40 (s, 3H), 1.59 (quint, 2H), 1.40 – 1.28 (m, 2H), 0.86 (t, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 169.3, 166.8, 147.8, 141.4, 138.7, 133.3, 130.1, 129.9, 129.5, 129.3, 129.0, 128.1, 127.9, 125.0, 123.2, 121.7, 109.8, 64.6, 30.7, 21.4, 19.1, 13.7 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{23}H_{24}NO_3$ 362.1756, Found 362.1754.

***tert*-Butyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3v)**



Red orange solid, eluent: (hexane/ethyl acetate, 94:6), m.p. (208-210 ^oC), yield: (49 mg, 46%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.42 (d, *J* = 15.6 Hz, 1H), 8.32 (s, 1H), 7.34 (d, *J* = 7.8 Hz, 2H), 7.11 (d, *J* = 7.8 Hz, 3H), 6.82 (d, *J* = 7.8 Hz, 1H), 6.63 (t, *J* = 7.8 Hz, 1H), 5.90 (d, *J* = 7.8 Hz, 1H), 5.69 (d, *J* = 15.6 Hz, 1H), 2.49 (s, 3H), 1.52 (s, 3H) ppm. **^{13}C NMR (75 MHz, $CDCl_3$):** δ 168.9, 165.9, 148.1, 141.1, 140.5, 138.6, 133.5, 131.1, 130.1, 129.7, 128.1, 127.3, 124.9, 123.4, 121.7, 109.5, 28.1, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + Na]^+$ Calcd for $C_{23}H_{23}NNaO_3$ 384.1575, Found 384.1582.

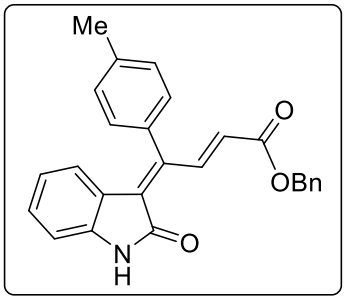
Cyclohexyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3w)



Red orange solid, eluent: (hexane/ethyl acetate, 94:6), m.p. (228-230 ^oC), yield: (48 mg, 42%). **^{1}H NMR (300 MHz, $CDCl_3$):** δ 9.42 (d, *J* = 15.6 Hz, 1H), 8.75 (s, 1H), 7.25 (d, *J* = 7.8 Hz,

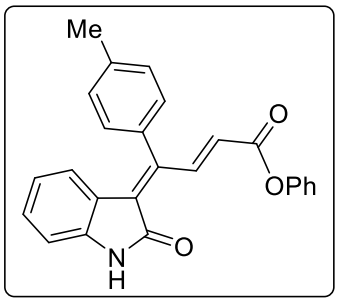
2H), 7.03 (d, *J* = 7.8 Hz, 3H), 6.76 (d, *J* = 7.8 Hz, 1H), 6.54 (t, *J* = 7.8 Hz, 1H), 5.83 (d, *J* = 7.8 Hz, 1H), 5.67 (d, *J* = 15.6 Hz, 1H), 4.81 – 4.76 (m, 1H), 2.40 (s, 3H), 1.80 – 1.78 (m, 2H), 1.70 – 1.67 (m, 2H), 1.46 – 1.18 (m, 6H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 169.2, 166.1, 148.0, 141.3, 141.2, 138.7, 133.4, 130.1, 129.8, 129.6, 128.1, 127.7, 124.9, 123.3, 121.7, 109.7, 31.6, 25.4, 23.7, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + Na]^+$ Calcd for $C_{25}H_{25}NNaO_3$ 410.1732, Found 410.1728.

Benzyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3x)



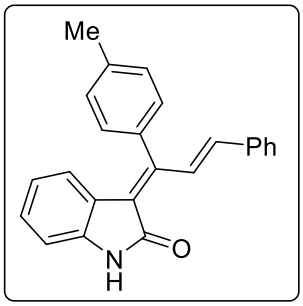
Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (192-194 ^{o}C), yield: (84 mg, 72%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.57 (d, *J* = 15.9 Hz, 1H), 8.94 (s, 1H), 7.43 – 7.32 (m, 7H), 7.13 – 7.10 (m, 3H), 6.82 (d, *J* = 7.8 Hz, 1H), 6.63 (t, *J* = 7.8 Hz, 1H), 5.93 (d, *J* = 7.8 Hz, 1H), 5.81 (d, *J* = 15.6 Hz, 1H), 5.24 (s, 2H), 2.49 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 169.2, 166.5, 147.6, 142.0, 141.4, 138.8, 135.9, 133.3, 130.1, 130.0, 128.6, 128.5, 128.3, 128.2, 128.1, 125.0, 121.7, 109.8, 66.5, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{26}H_{22}NO_3$ 396.1599, Found 396.1593.

Phenyl (*E*)-4-((*E*)-2-oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enoate (3y)



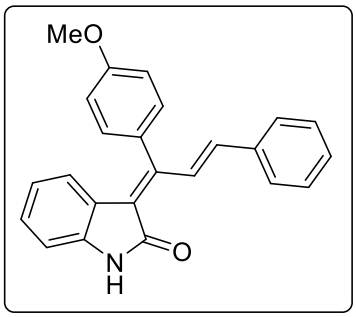
Red orange solid, eluent: (hexane/ethyl acetate, 92:8), m.p. (196-198 ^{o}C), yield: (85 mg, 75%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.71 (d, *J* = 15.9 Hz, 1H), 8.81 (s, 1H), 7.43 – 7.37 (m, 4H), 7.28 – 7.25 (m, 1H), 7.17 – 7.15 (m, 4H), 7.10 (d, *J* = 7.8 Hz, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.64 (t, *J* = 7.8 Hz, 1H), 5.98 – 5.93 (m, 2H), 2.51 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 169.1, 165.1, 150.7, 147.3, 143.2, 141.5, 138.9, 133.2, 130.2, 129.4, 128.5, 128.2, 127.8, 125.8, 125.1, 123.1, 121.8, 121.5, 109.8, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + Na]^+$ Calcd for $C_{25}H_{19}NNaO_3$ 404.1262, Found 404.1263.

(*E*)-3-((*E*)-3-Phenyl-1-(*p*-tolyl)allylidene)indolin-2-one (3z)



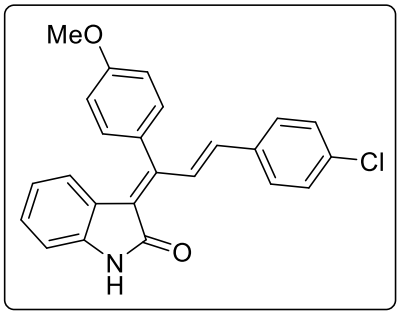
Red orange solid, eluent: (hexane/ethyl acetate, 96:4), m.p. (190-192 ^{o}C), yield: (52 mg, 52%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.18 (d, *J* = 15.9 Hz, 1H), 8.34 (s, 1H), 7.45 (d, *J* = 7.5 Hz, 2H), 7.26 (t, *J* = 7.5 Hz, 3H), 7.22 – 7.17 (m, 2H), 7.09 (d, *J* = 7.8 Hz, 2H), 6.96 (t, *J* = 7.8 Hz, 1H), 6.72 (d, *J* = 7.5 Hz, 1H), 6.52 (t, *J* = 7.8 Hz, 1H), 6.43 (d, *J* = 16.2 Hz, 1H), 5.74 (d, *J* = 7.8 Hz, 1H), 2.43 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 169.7, 152.0, 141.6, 140.0, 138.3, 136.9, 134.5, 129.9, 129.0, 128.7, 128.4, 128.1, 127.9, 127.7, 124.2, 123.9, 122.7, 121.4, 109.2, 21.5 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{24}H_{20}NO$ 338.1544, Found 338.1546.

(*E*)-3-((*E*)-1-(4-Methoxyphenyl)-3-phenylallylidene)indolin-2-one (3aa)



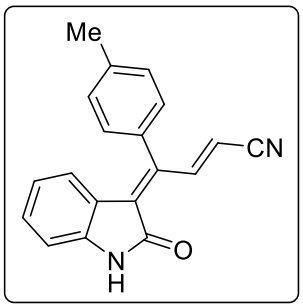
Red orange solid, eluent: (hexane/ethyl acetate, 96:4), m.p. (192-194 ^{o}C), yield: (43 mg, 44%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.15 (d, *J* = 15.9 Hz, 1H), 7.74 (s, 1H), 7.46 (d, *J* = 7.2 Hz, 2H), 7.28 – 7.23 (m, 3H), 7.14 (d, *J* = 8.4 Hz, 2H), 7.02 – 6.96 (m, 3H), 6.71 (d, *J* = 7.5 Hz, 1H), 6.56 (t, *J* = 7.8 Hz, 1H), 6.46 (d, *J* = 15.9 Hz, 1H), 5.83 (d, *J* = 7.8 Hz, 1H), 3.87 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 169.3, 159.7, 151.7, 141.7, 139.8, 136.8, 129.9, 129.7, 129.0, 128.7, 128.1, 127.9, 124.3, 123.9, 122.7, 121.4, 114.6, 109.0, 55.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{24}H_{20}NO_2$ 354.1494, Found 354.1486.

(*E*)-3-((*E*)-3-(4-Chlorophenyl)-1-(4-methoxyphenyl)allylidene)indolin-2-one (3ab)



Red orange solid, eluent: (hexane/ethyl acetate, 96:4), m.p. (194-196 ^{o}C), yield: (41 mg, 38%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.22 (d, *J* = 15.6 Hz, 1H), 8.07 (s, 1H), 7.47 (d, *J* = 7.8 Hz, 2H), 7.32 – 7.28 (m, 2H), 7.23 (d, *J* = 8.1 Hz, 2H), 7.11 – 7.06 (m, 3H), 6.81 (d, *J* = 7.8 Hz, 1H), 6.65 (t, *J* = 7.5 Hz, 1H), 6.48 (d, *J* = 15.6 Hz, 1H), 5.92 (d, *J* = 7.5 Hz, 1H), 3.96 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 159.8, 151.2, 140.0, 135.4, 134.7, 129.9, 129.5, 129.0, 128.9, 128.4, 128.3, 124.2, 123.9, 121.5, 114.6, 109.2, 55.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + H]^+$ Calcd for $C_{24}H_{19}ClNO_2$ 388.1104, Found 388.1083.

(*E*)-4-((*E*)-2-Oxoindolin-3-ylidene)-4-(*p*-tolyl)but-2-enenitrile (3ac)



Red orange solid, eluent: (hexane/ethyl acetate, 96:4), m.p. (220-222 ^{o}C), yield: (36 mg, 43%). **^{1}H NMR (300 MHz, CDCl$_3$):** δ 9.36 (d, *J* = 16.2 Hz, 1H), 8.58 (s, 1H), 7.37 (d, *J* = 7.8 Hz, 2H), 7.19 – 7.09 (m, 3H), 6.86 (d, *J* = 7.5 Hz, 1H), 6.65 (t, *J* = 7.8 Hz, 1H), 5.90 (d, *J* = 7.8 Hz, 1H), 5.25 (d, *J* = 16.5 Hz, 1H), 2.50 (s, 3H) ppm. **^{13}C NMR (75 MHz, CDCl$_3$):** δ 168.7, 147.2, 145.7, 141.6, 139.4, 131.7, 130.6, 130.4, 128.2, 128.1, 125.3, 122.7, 122.0, 118.1, 109.9, 106.1, 21.4 ppm. **HRMS (ESI-TOF) (*m/z*):** $[M + Na]^+$ Calcd for $C_{19}H_{14}N_2ONa$ 309.1003, Found 309.0999.

checkCIF/PLATON report

Structure factors have been supplied for datablock(s) 33

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

Datablock: 33

Bond precision: C-C = 0.0028 A Wavelength=0.71073

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alpha=119.1405(13) beta=91.334(2) gamma=97.5234(17)
Temperature: 296 K

	Calculated	Reported
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Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C20 H16 N O3	?
Sum formula	C20 H16 N O3	C20 H16 N O3
Mr	318.34	318.34
Dx,g cm-3	1.272	1.272
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Mu (mm-1)	0.086	0.086
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F000'	334.16	
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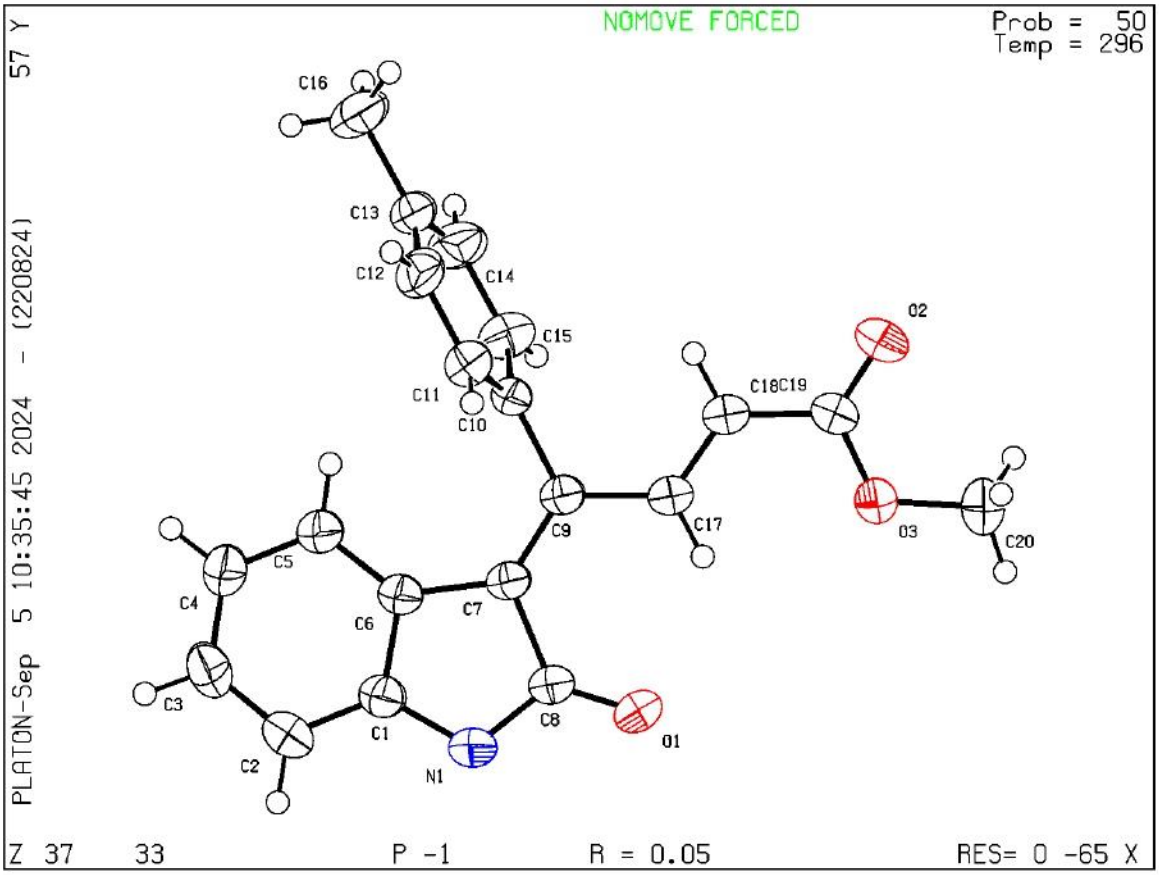
AbsCorr = MULTI-SCAN

Data completeness= 0.998 Theta(max)= 24.995

R(reflections)= 0.0454(2459) wR2(reflections)= 0.1309(2912)

S = 1.033 Npar= 219

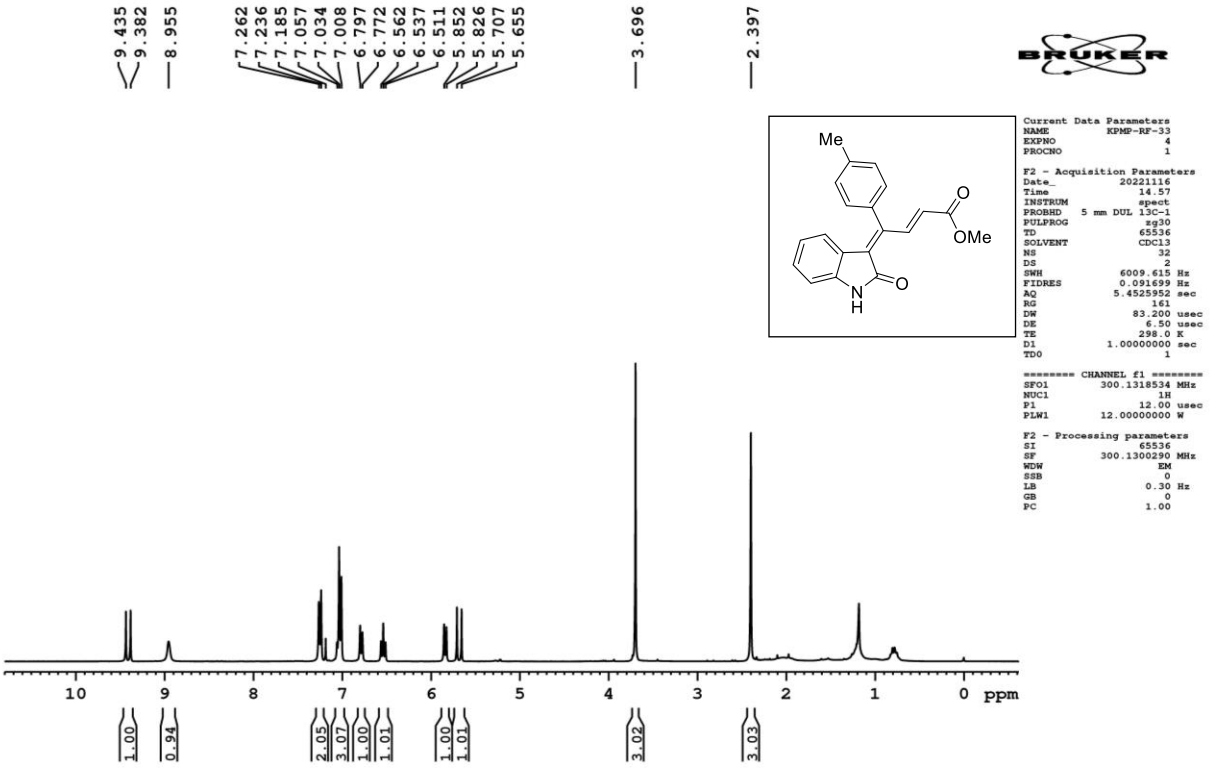
Figure S1: ORTEP representation of compound **3a** displaying thermal elliposoid at 50 probability.

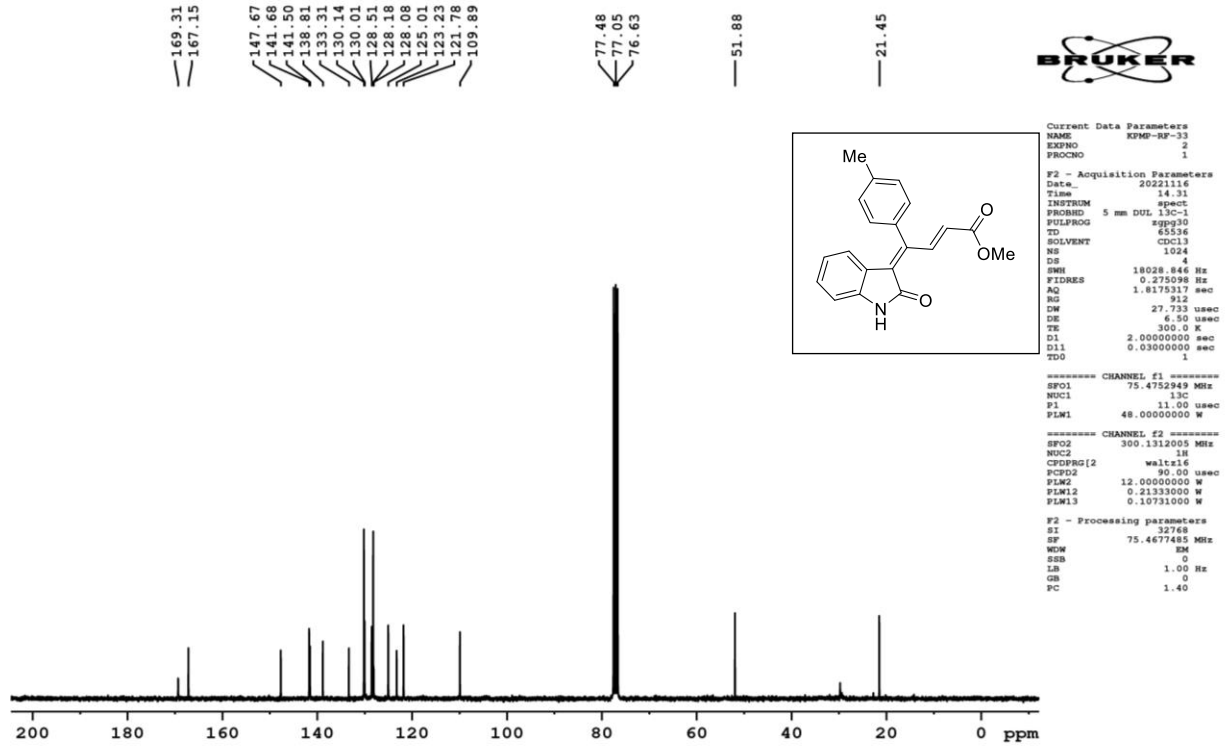


References:

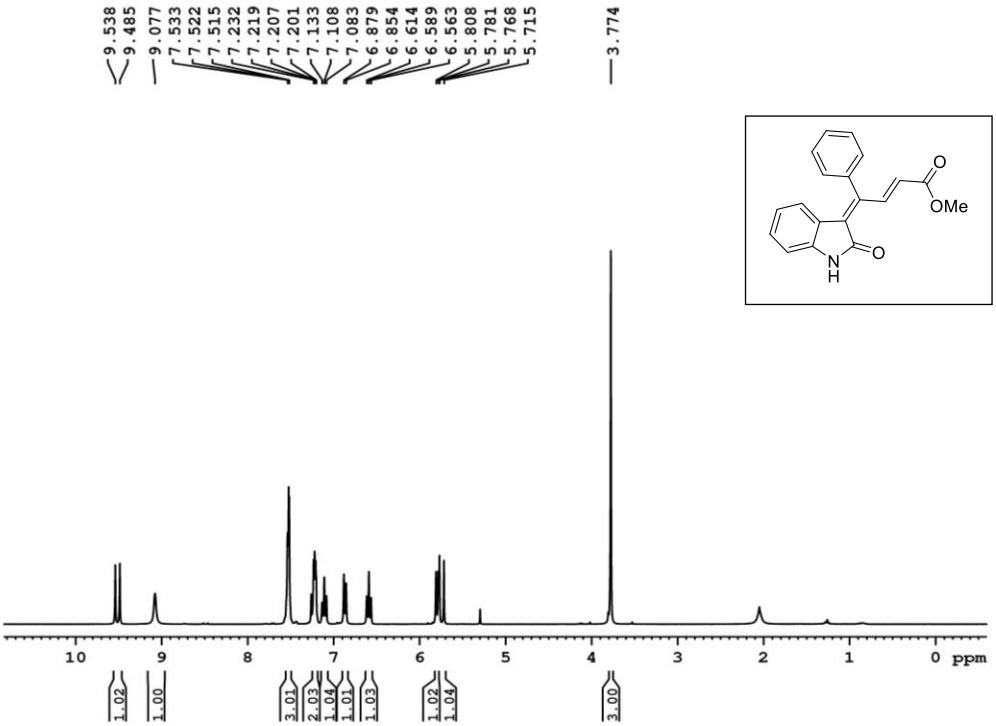
1. D. D. Perrin, W. L. F. Armarego, *In Purification of Laboratory Chemicals*, 3rd ed.; Pergamon Press: New York, 1988.
2. C. R. Reddy, V. Ganesh, A. K. Singh *RSC Adv.* 2020, ***10***, 28630-28634.

^{1}H and ^{13}C NMR spectra of compound **(3a)**





^{1}H and ^{13}C NMR spectra of compound **(3b)**

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TE                   298.0 K
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TD0                         1

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NUC1                       1H
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PLW1            12.00000000 W

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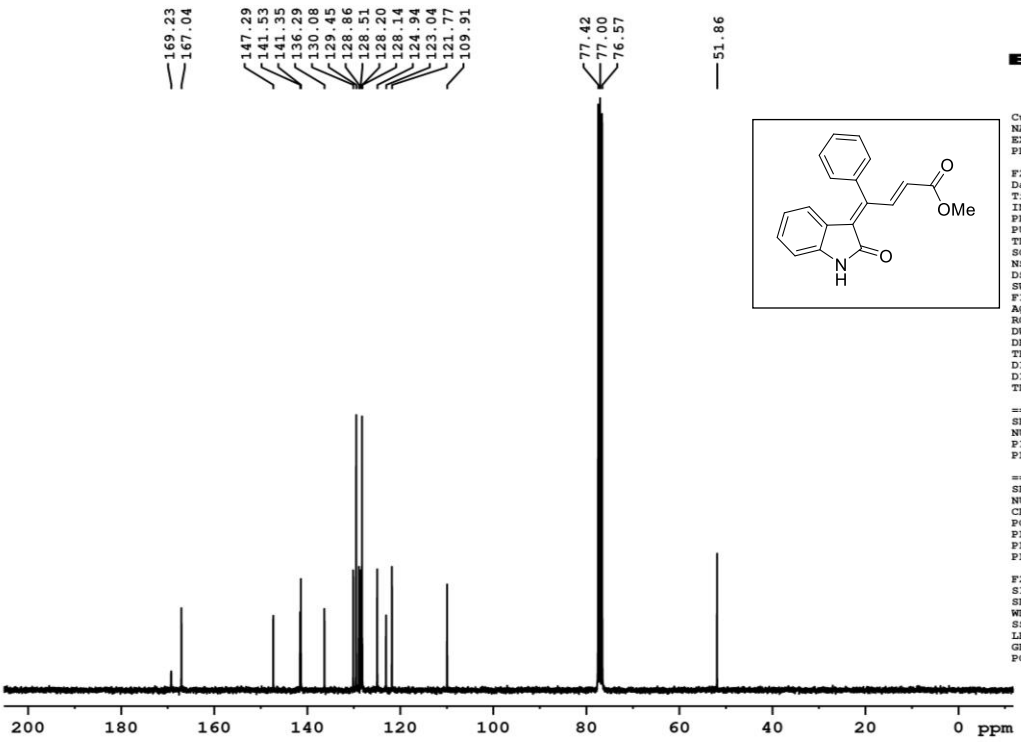


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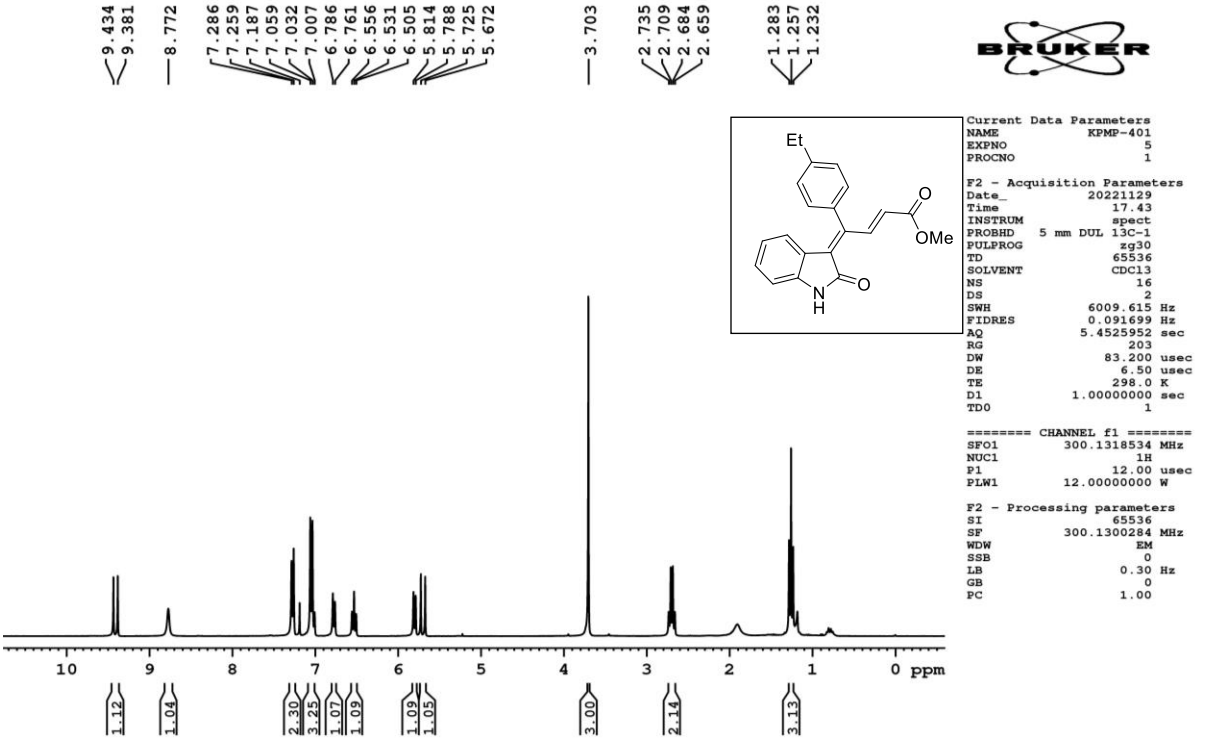
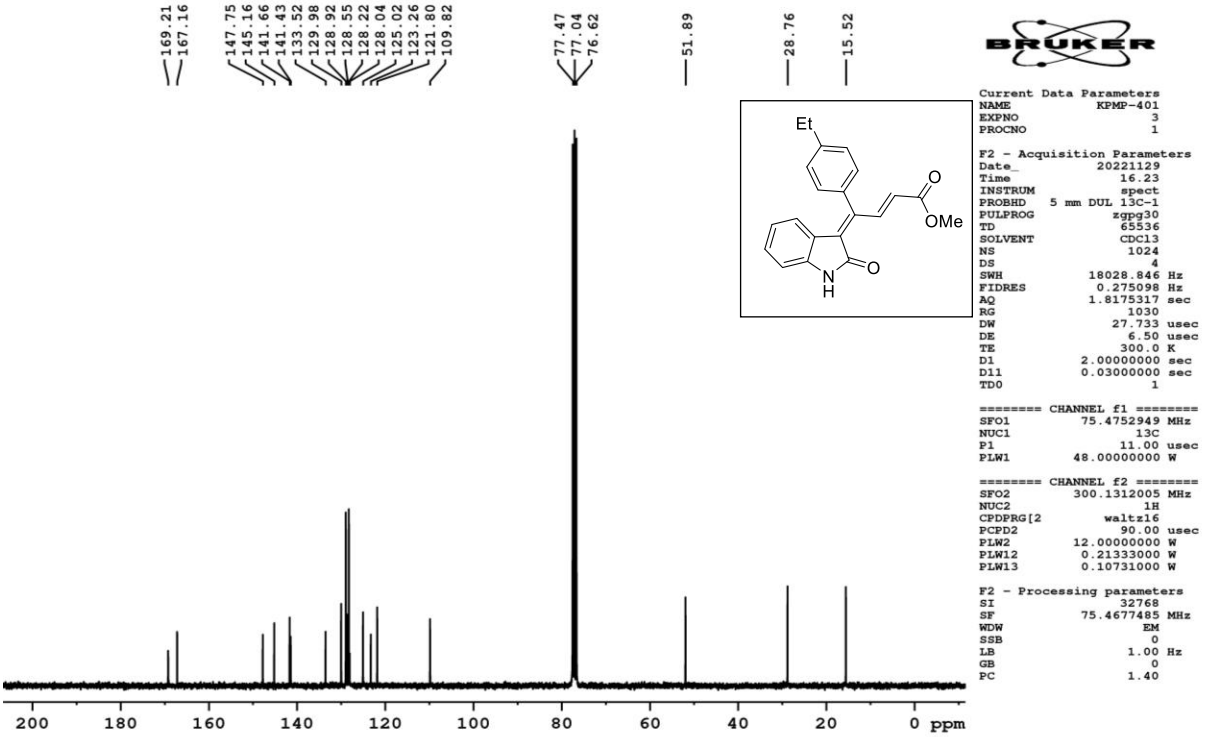
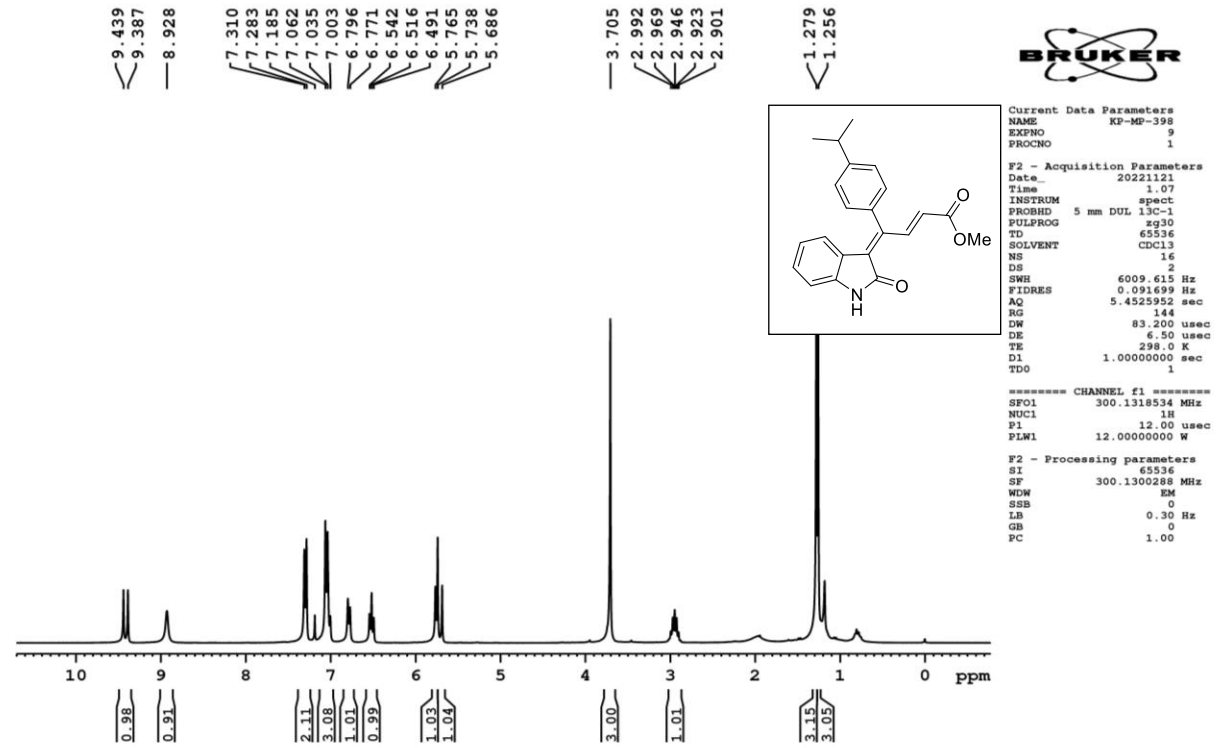
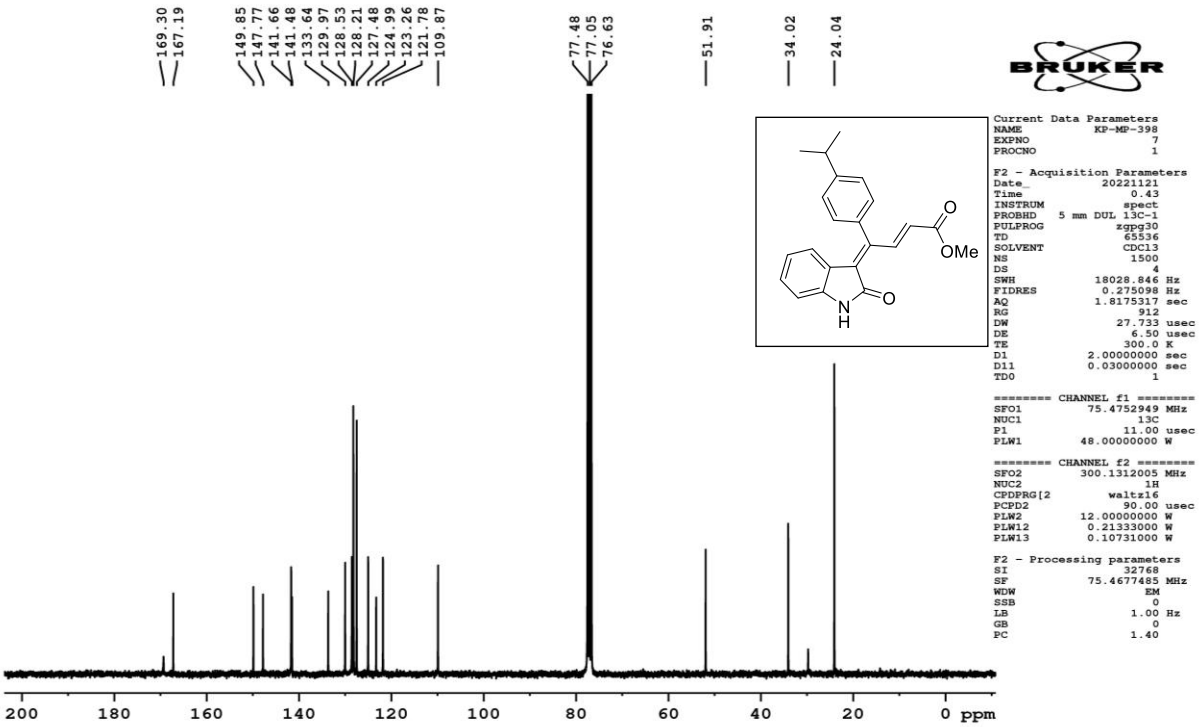
^{1}H and ^{13}C NMR spectra of compound **(3c)**



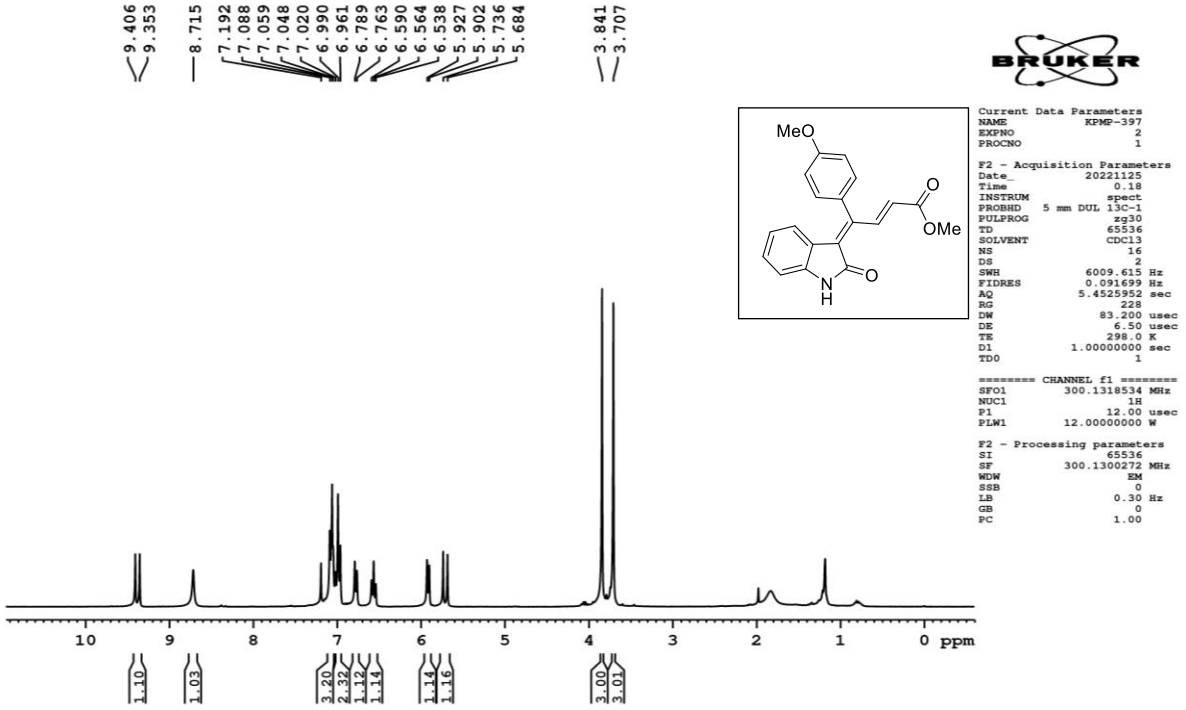


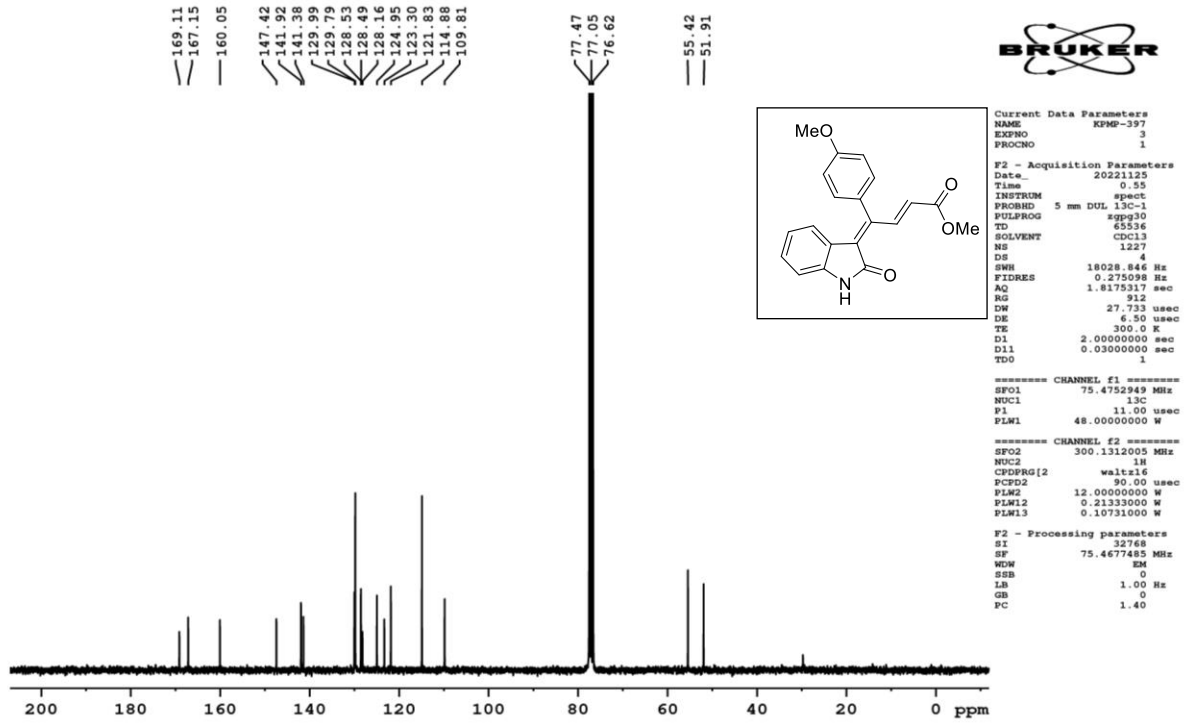
^{1}H and ^{13}C NMR spectra of compound **(3d)**



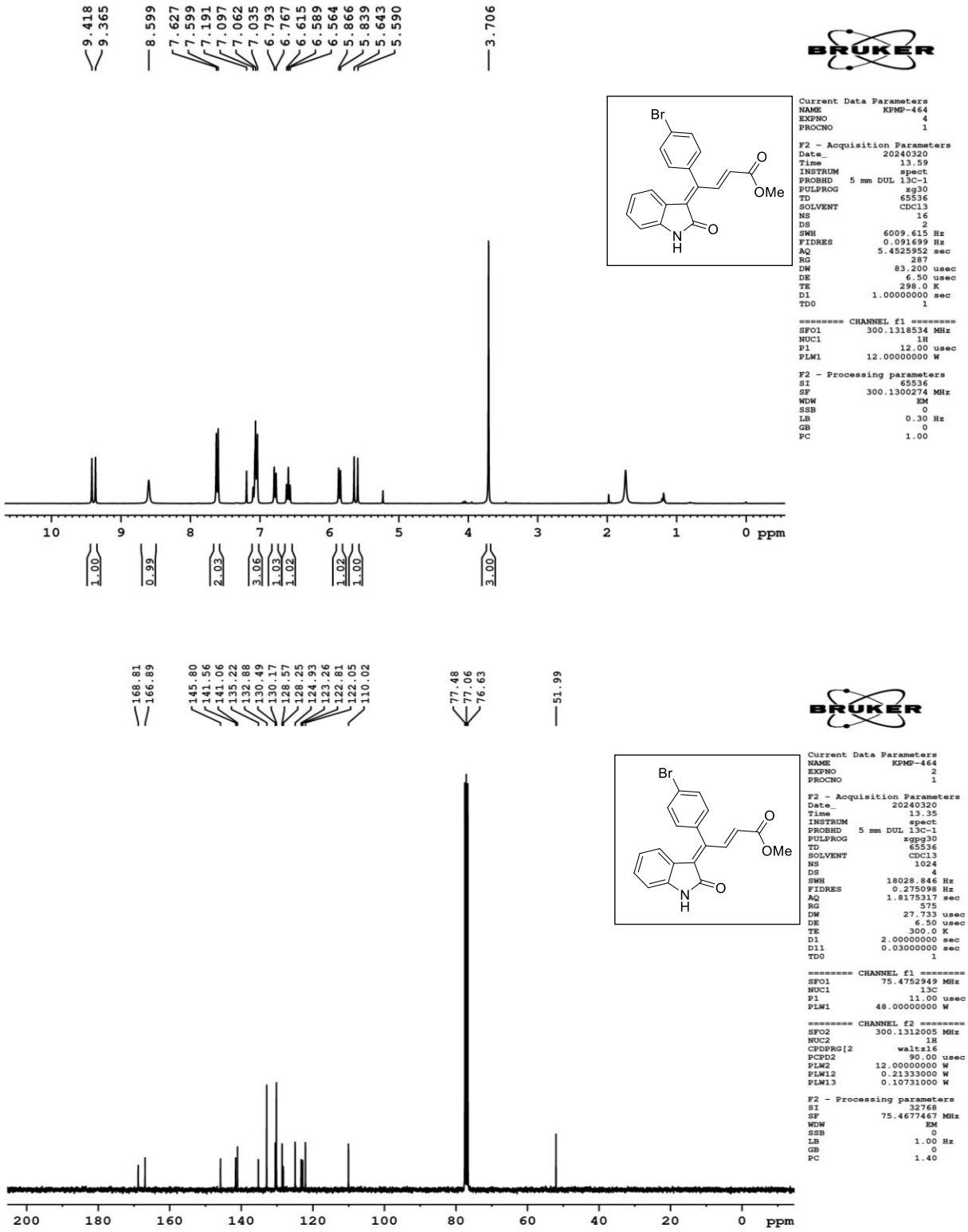


^{1}H and ^{13}C NMR spectra of compound **(3e)**

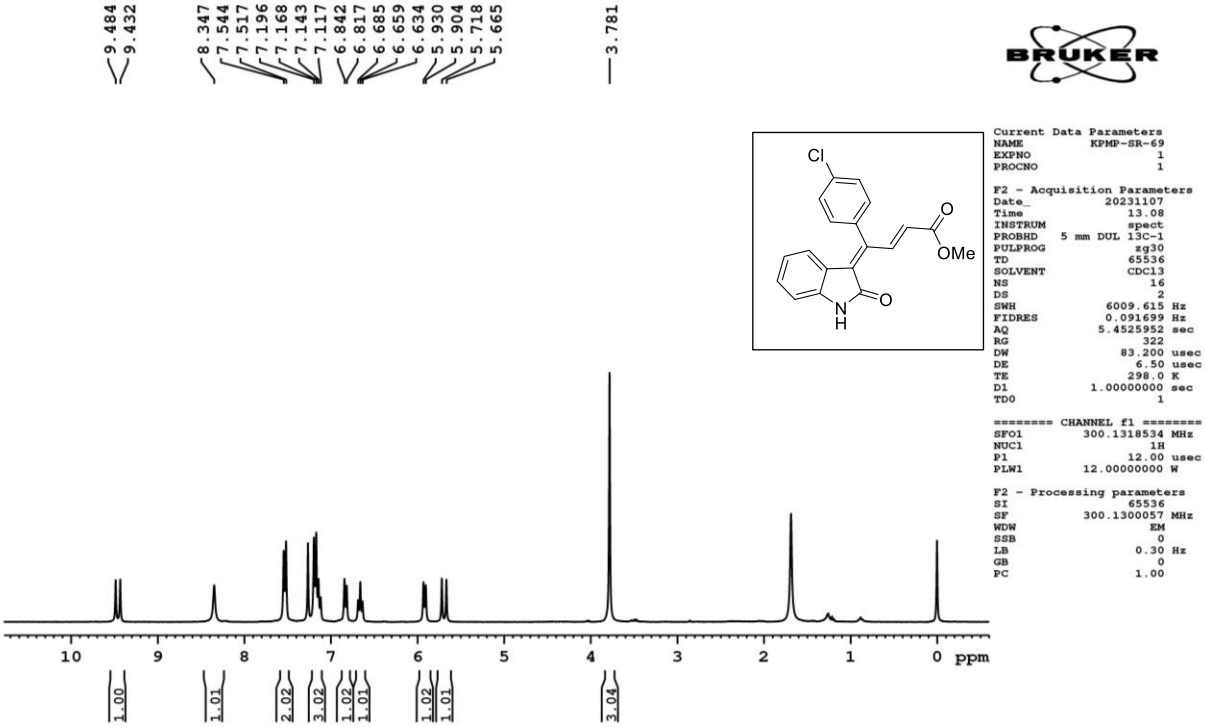


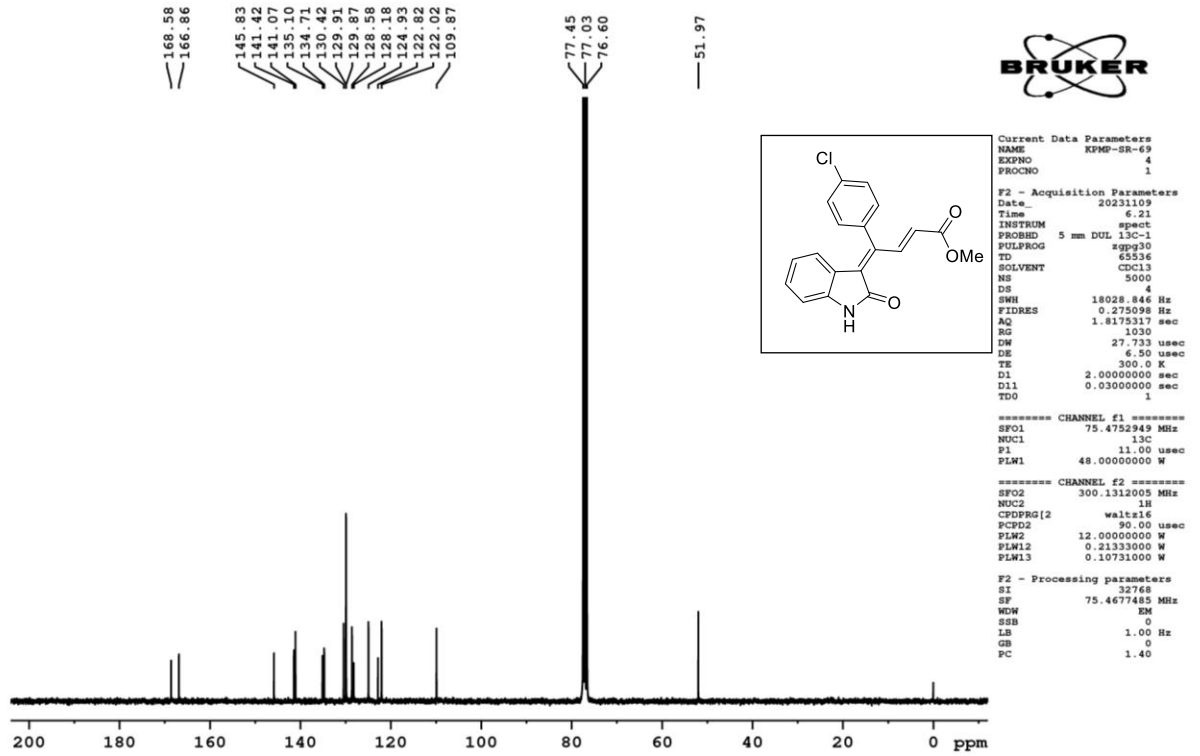


^{1}H and ^{13}C NMR spectra of compound **(3f)**

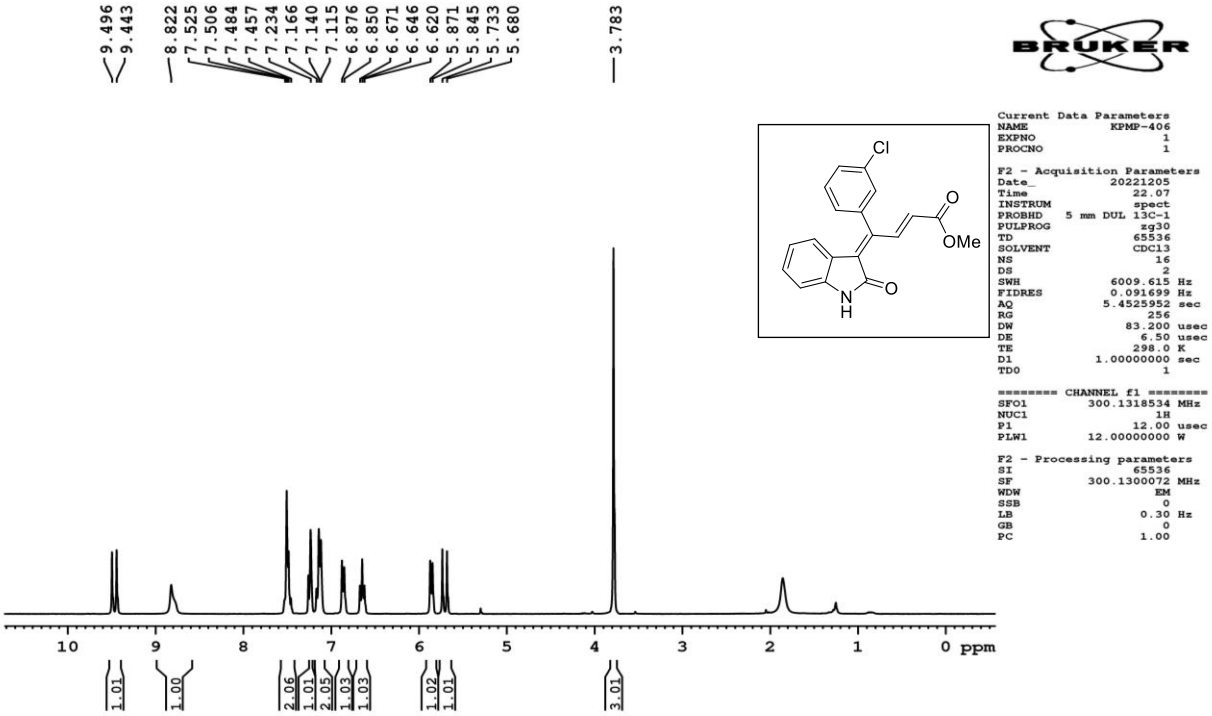


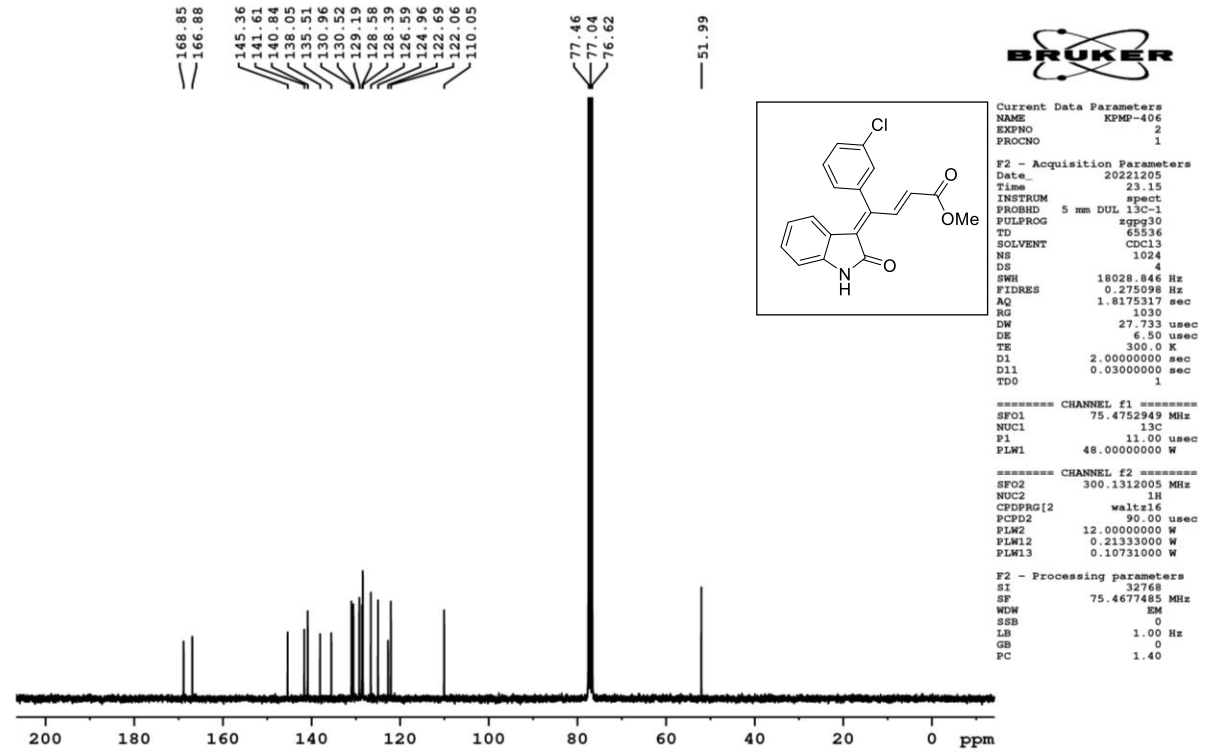
^{1}H and ^{13}C NMR spectra of compound **(3g)**



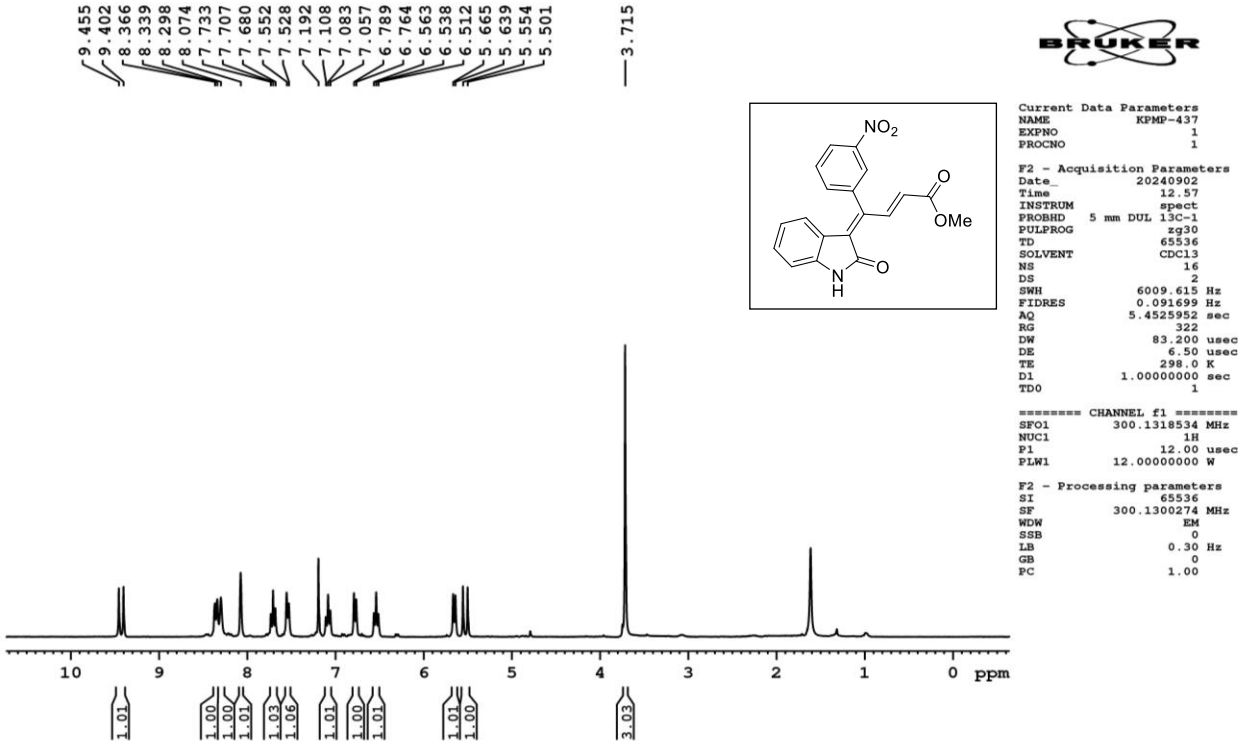


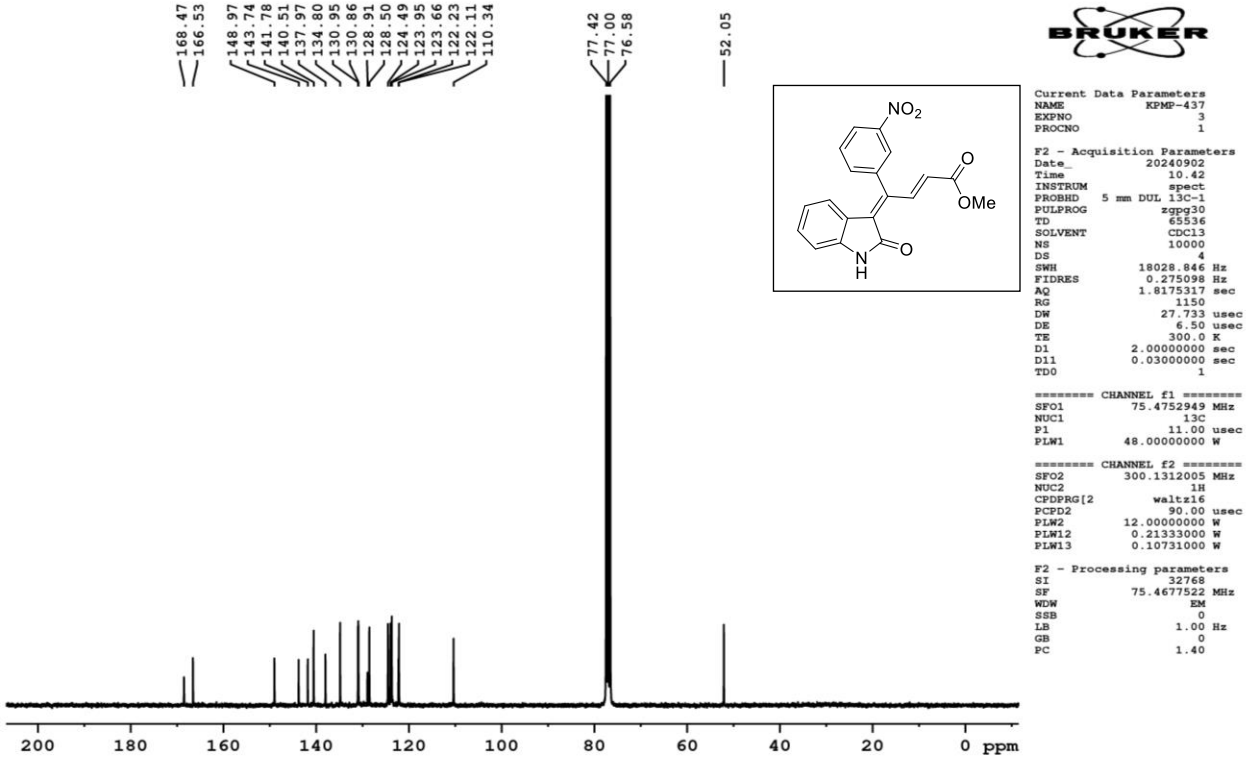
^{1}H and ^{13}C NMR spectra of compound **(3h)**



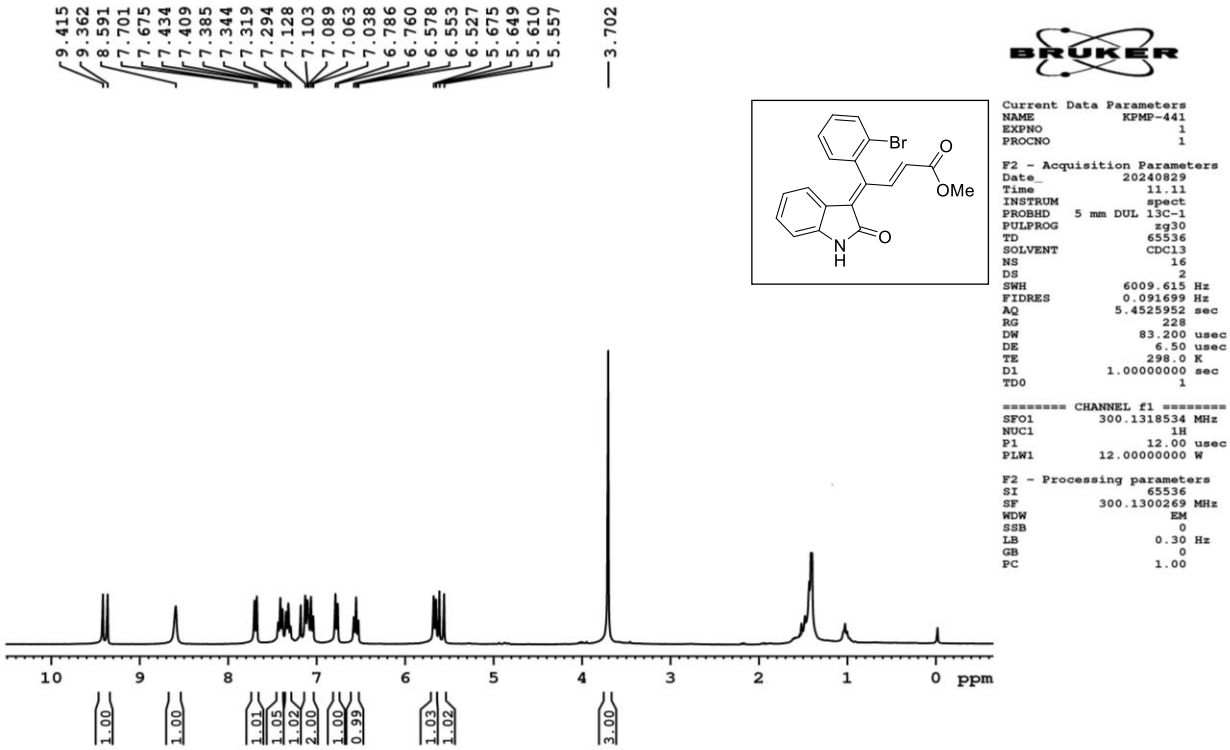


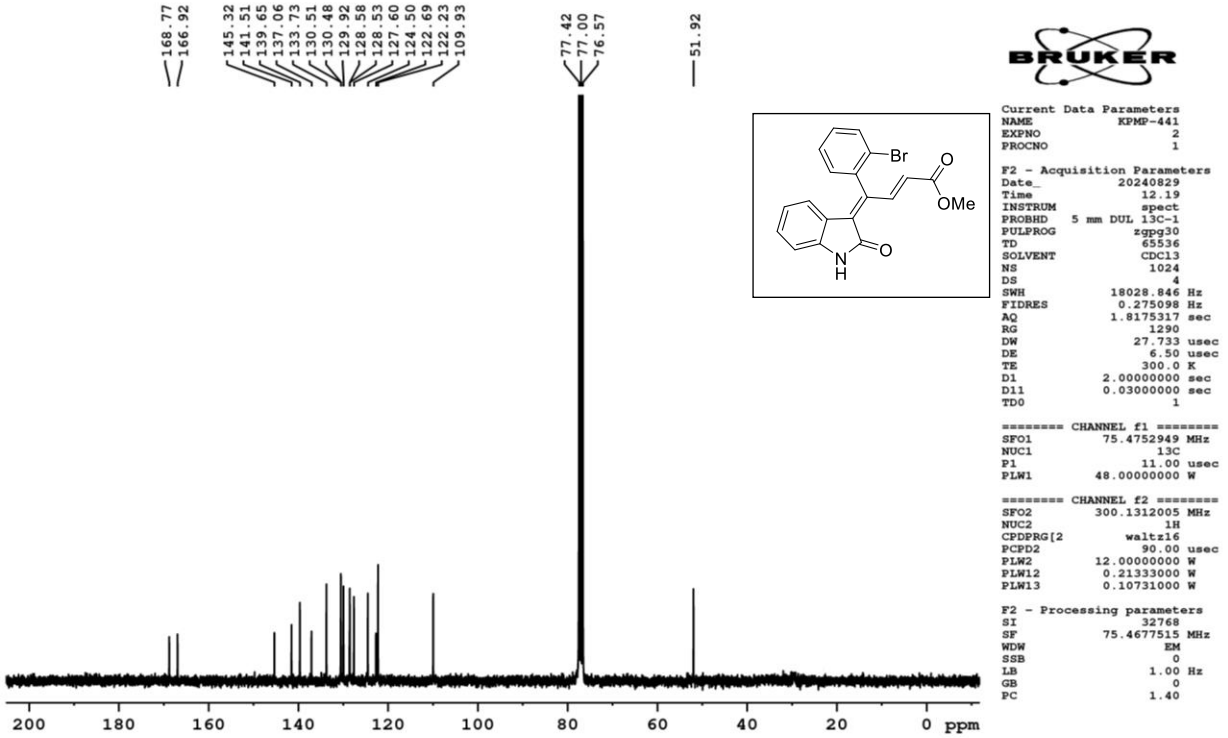
^{1}H and ^{13}C NMR spectra of compound **(3i)**



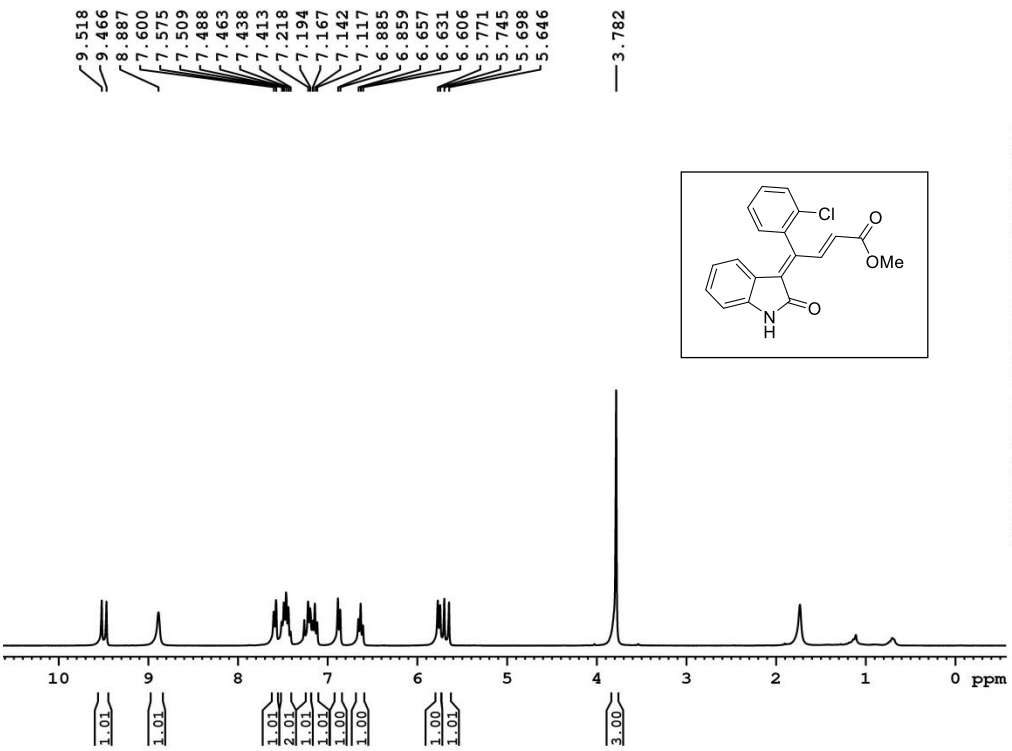


^{1}H and ^{13}C NMR spectra of compound **(3j)**





^{1}H and ^{13}C NMR spectra of compound **(3k)**

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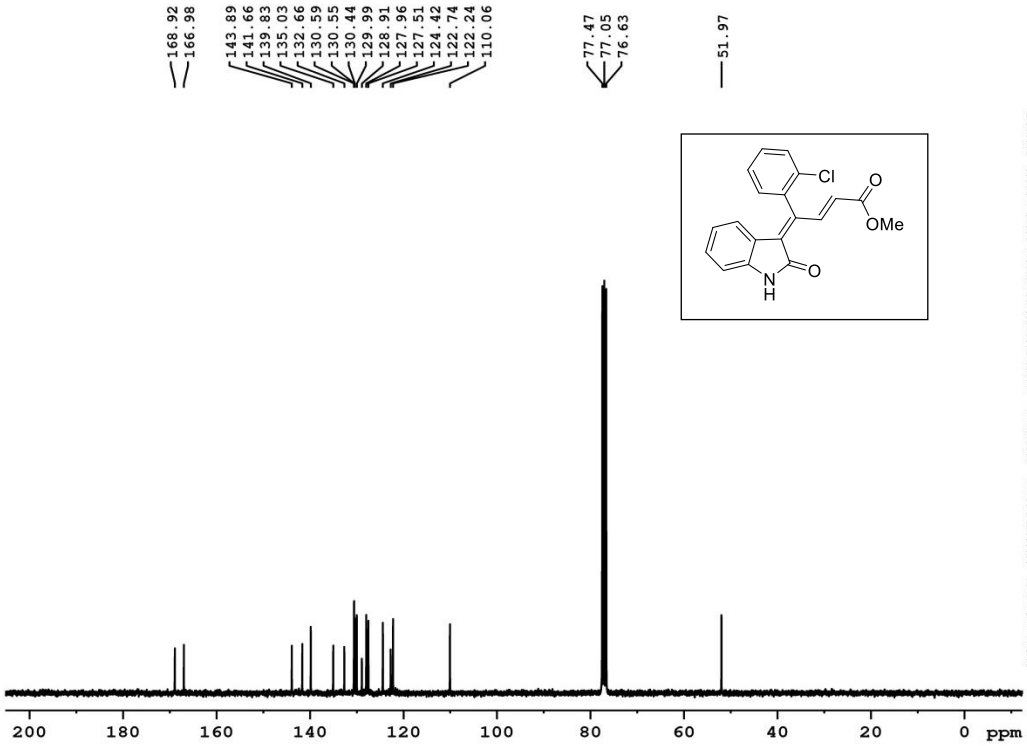


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SWH               18028.846 Hz
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AQ                1.8175317 sec
RG                      645
DW                 27.733 usec
DE                  6.50 usec
TE                  300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
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======== CHANNEL f1 ========
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NUC1                    13C
P1                    11.00 usec
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======== CHANNEL f2 ========
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PLW12           0.21333000 W
PLW13           0.10731000 W

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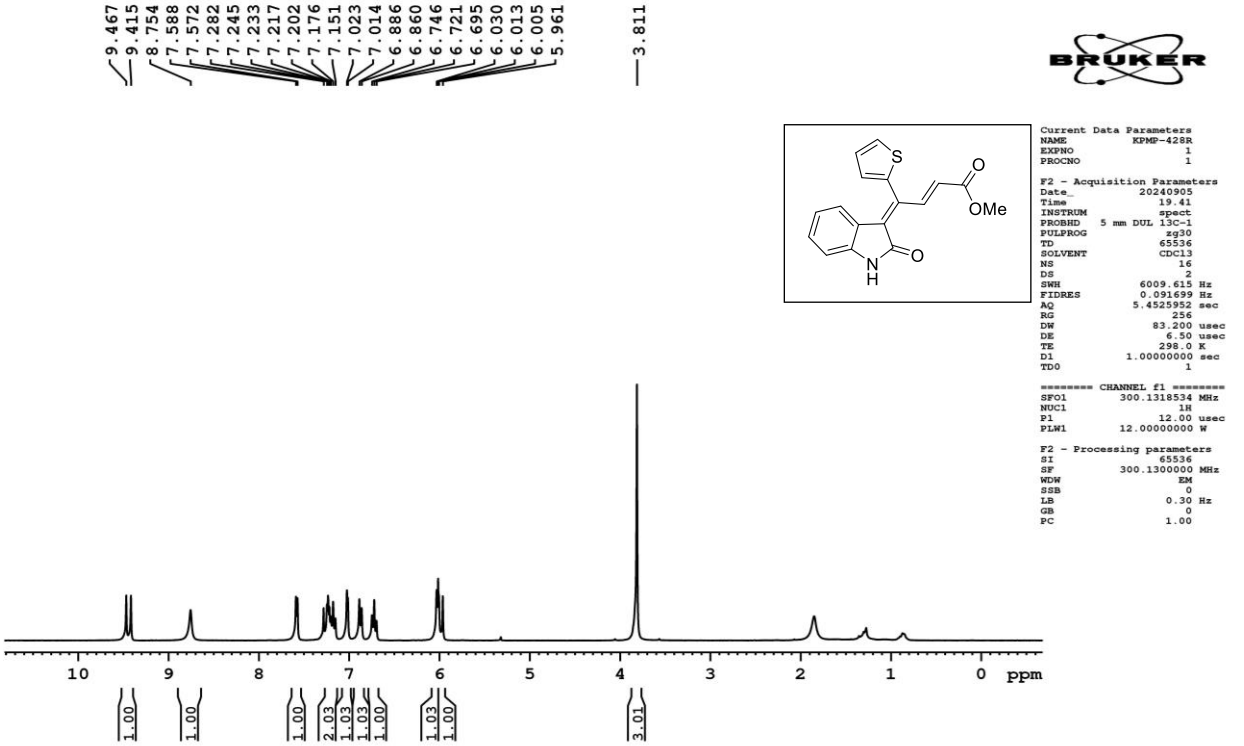
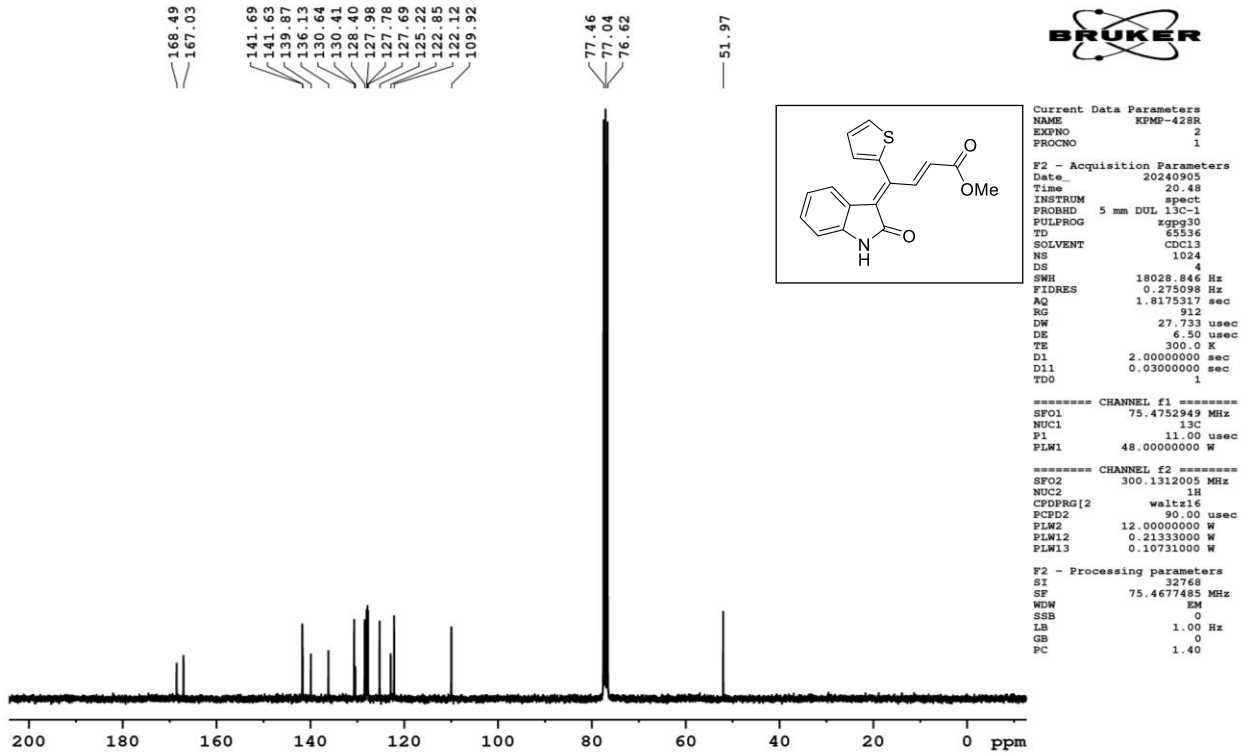
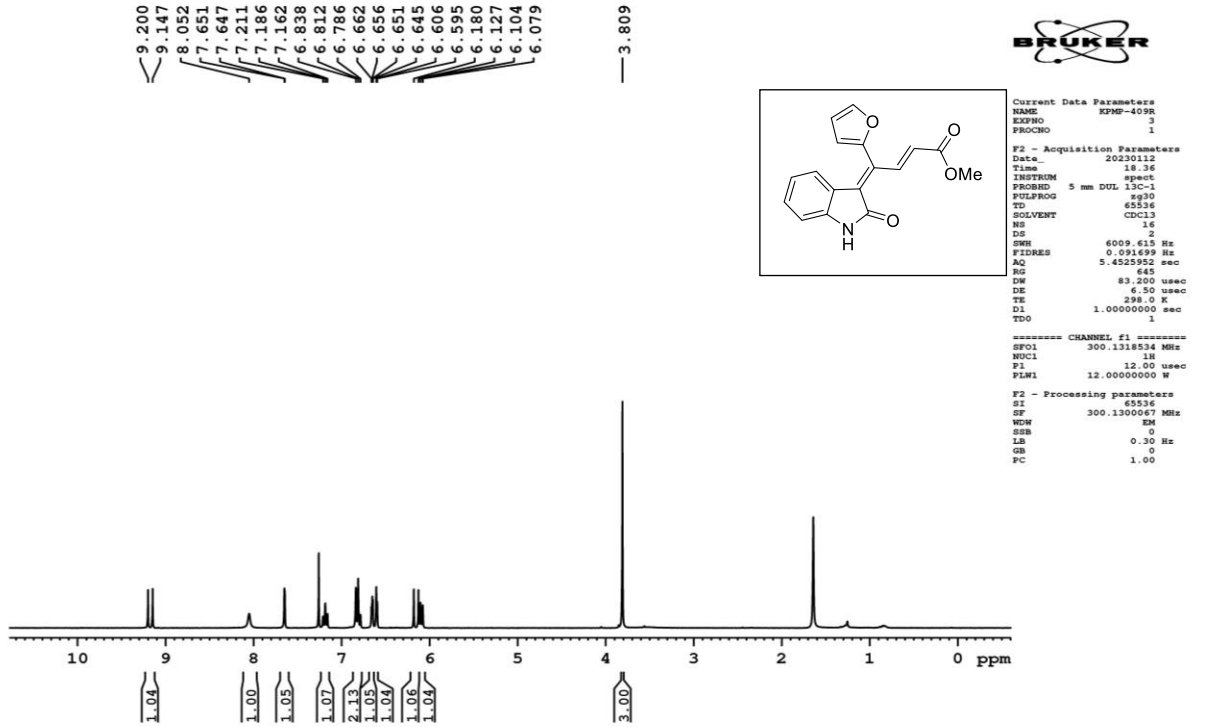
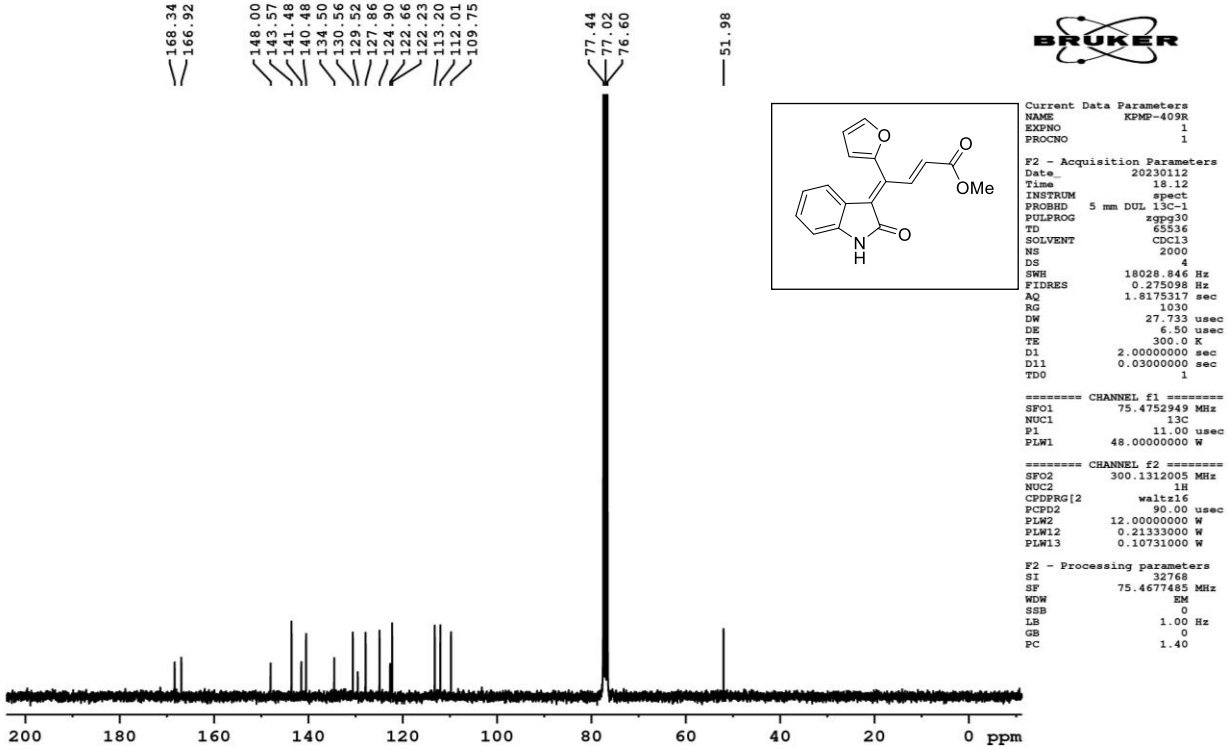
^{1}H and ^{13}C NMR spectra of compound **(3l)**



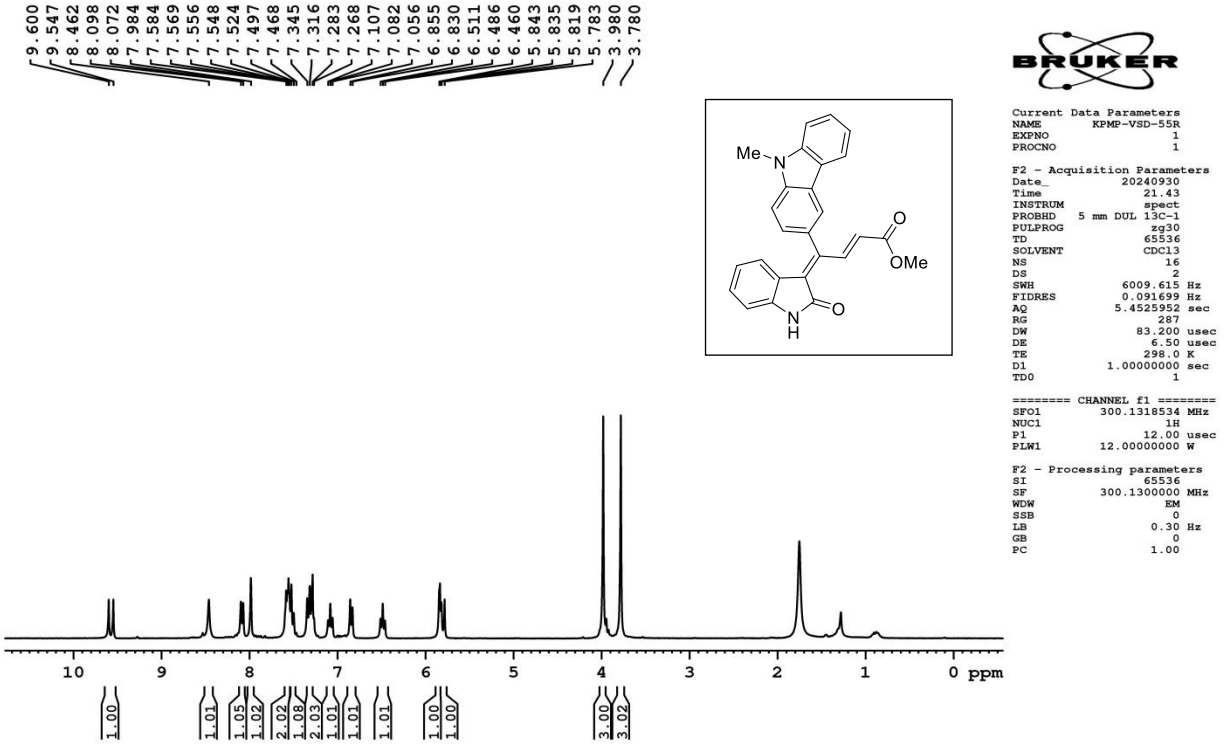


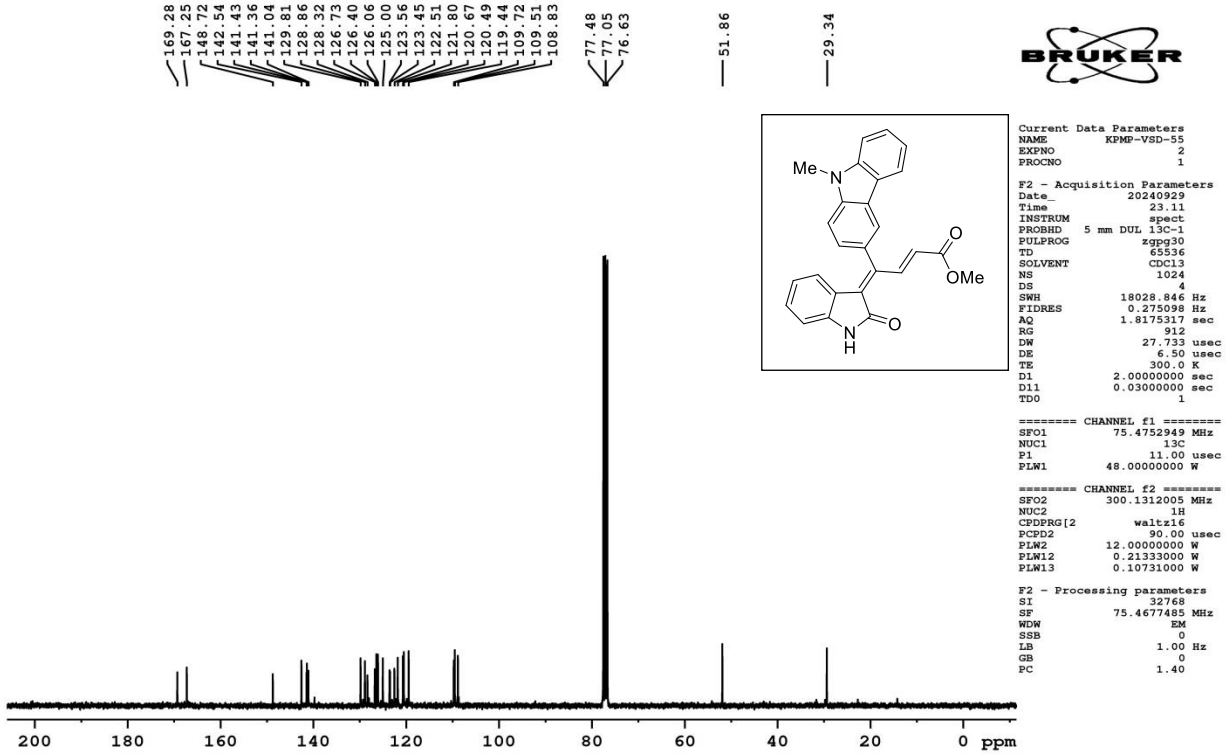
^{1}H and ^{13}C NMR spectra of compound **(3m)**



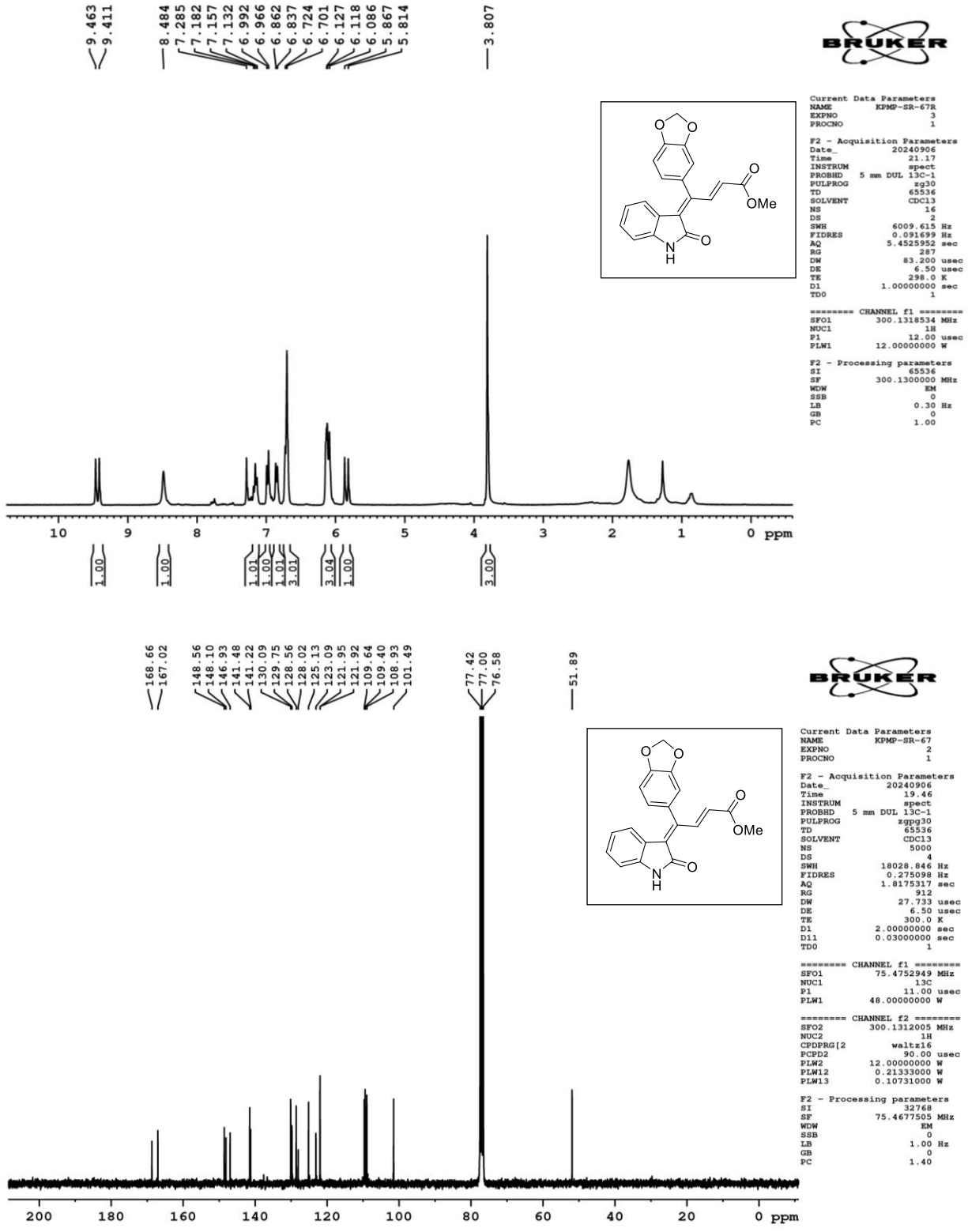


^{1}H and ^{13}C NMR spectra of compound **(3n)**

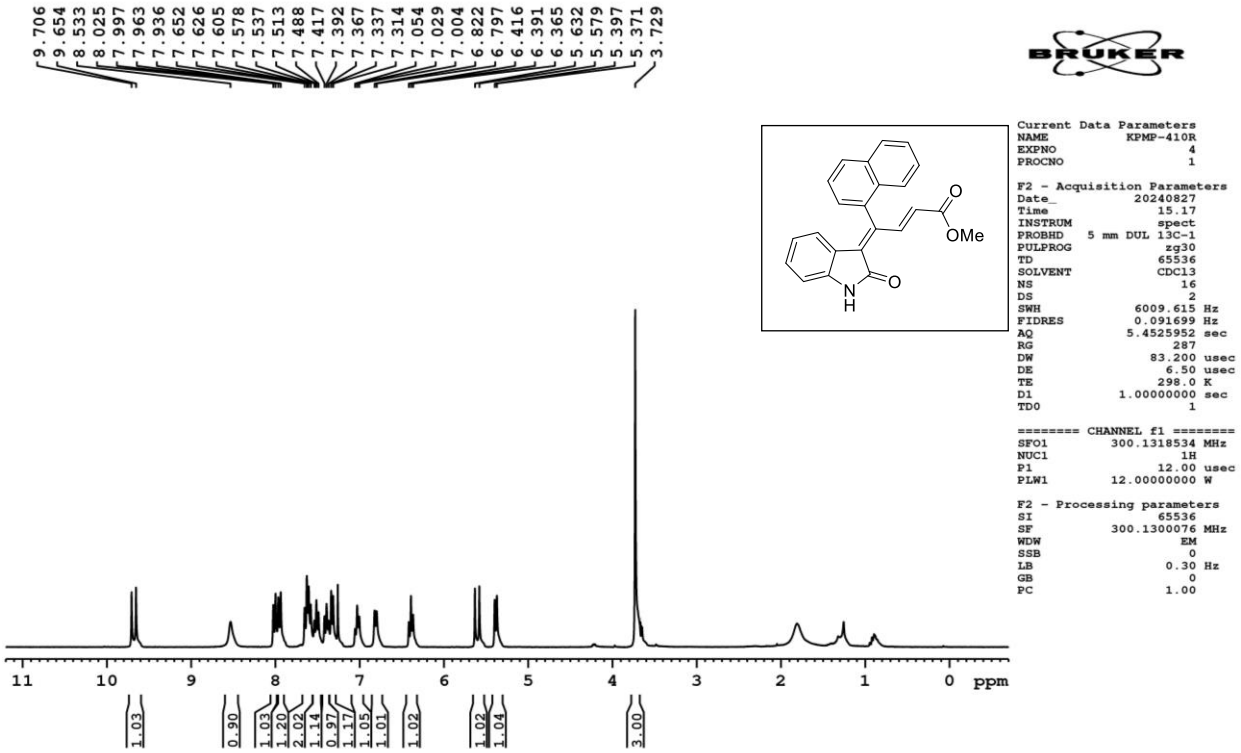


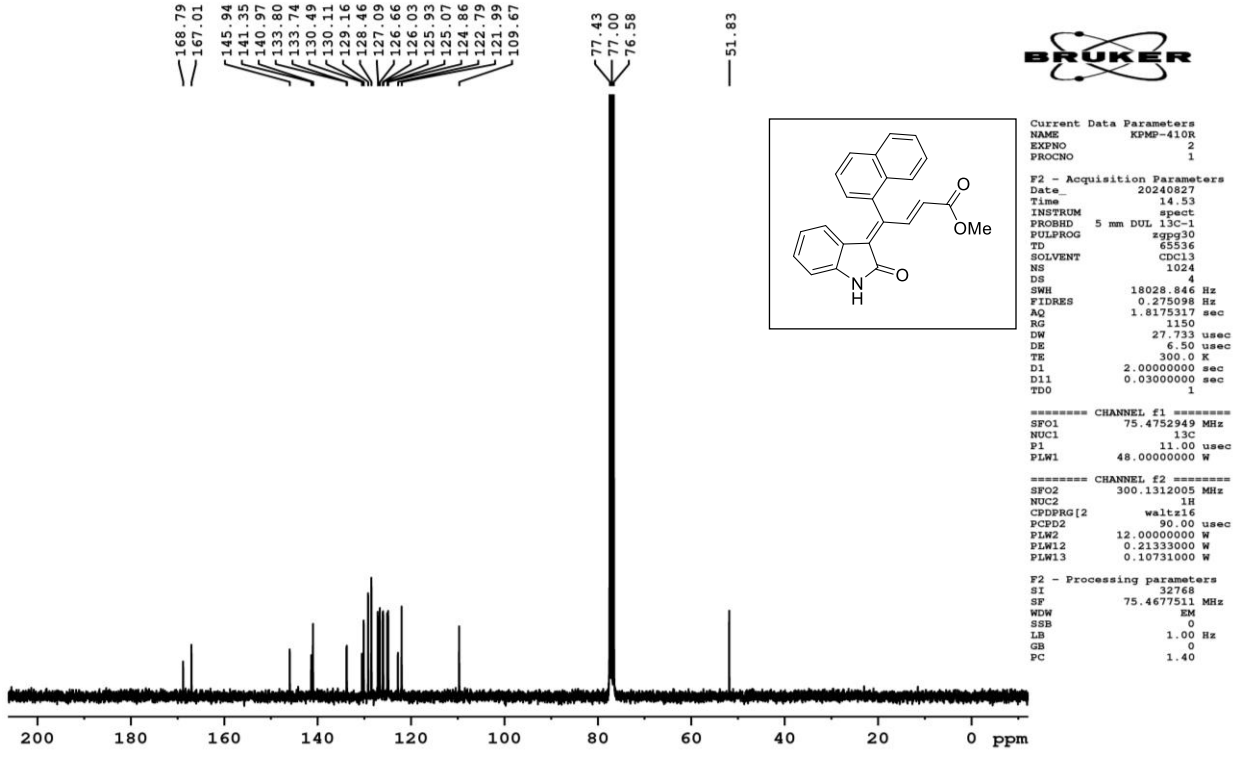


^{1}H and ^{13}C NMR spectra of compound **(3o)**

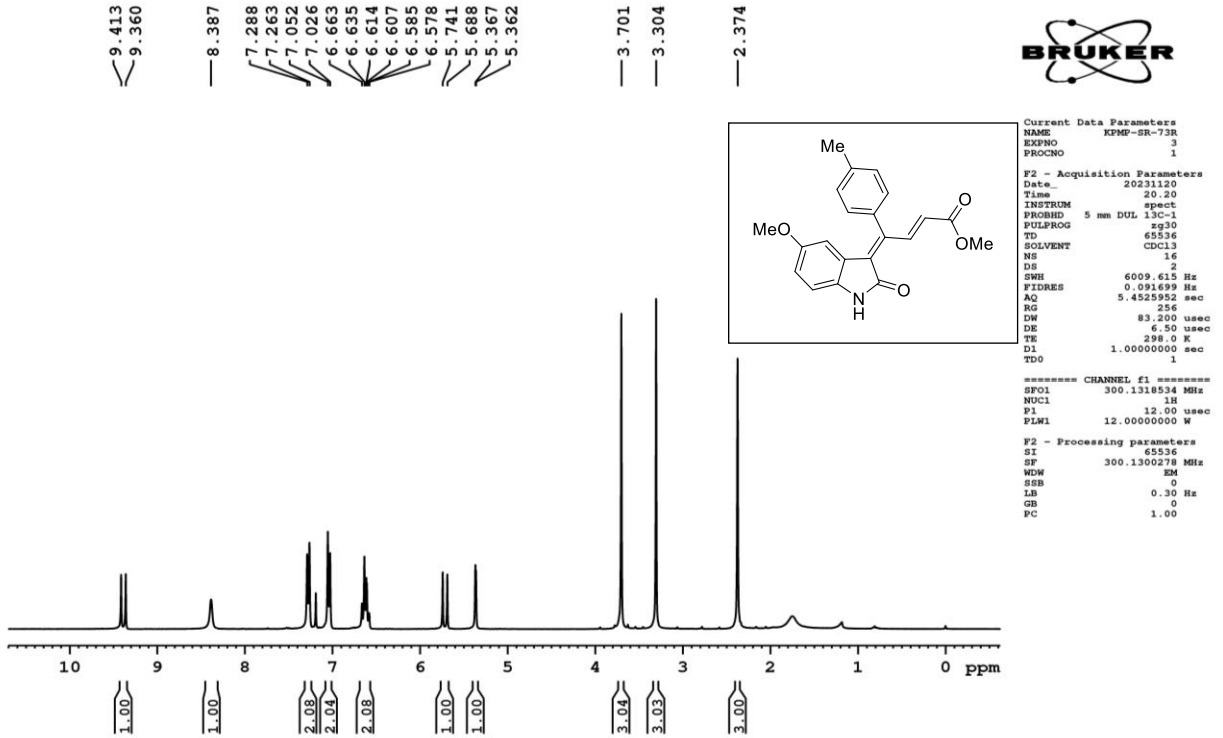


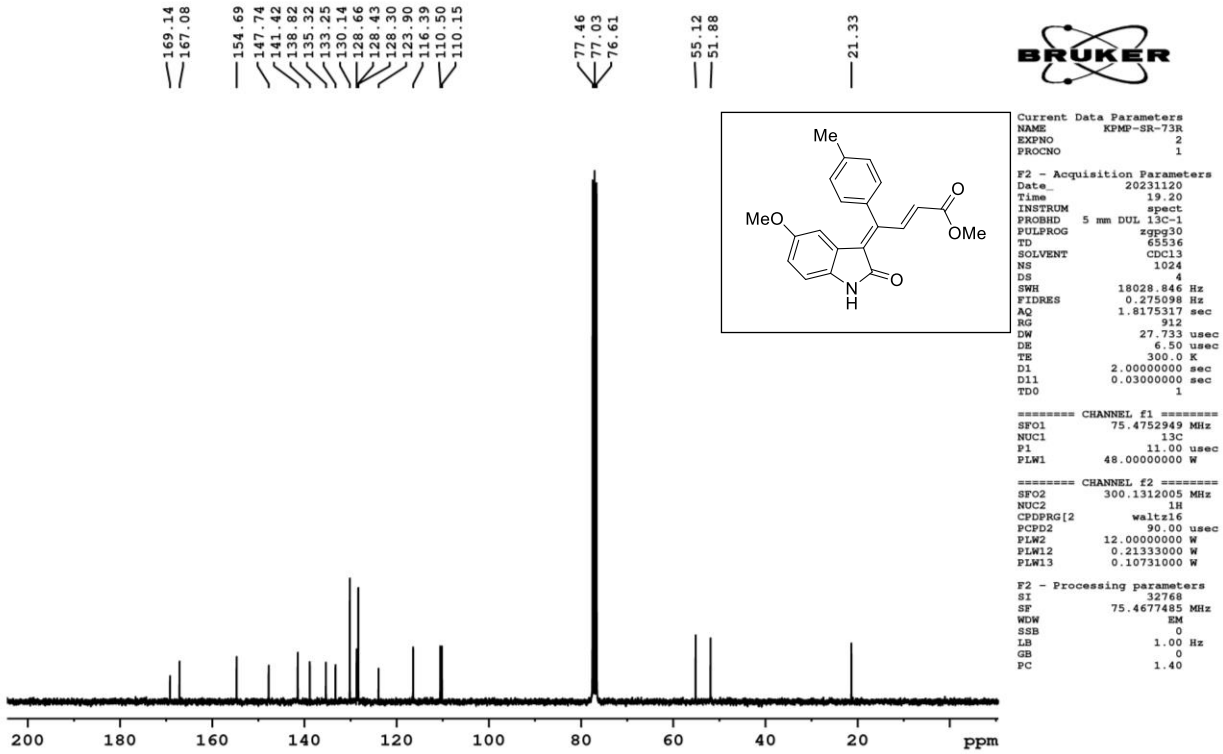
^{1}H and ^{13}C NMR spectra of compound **(3p)**



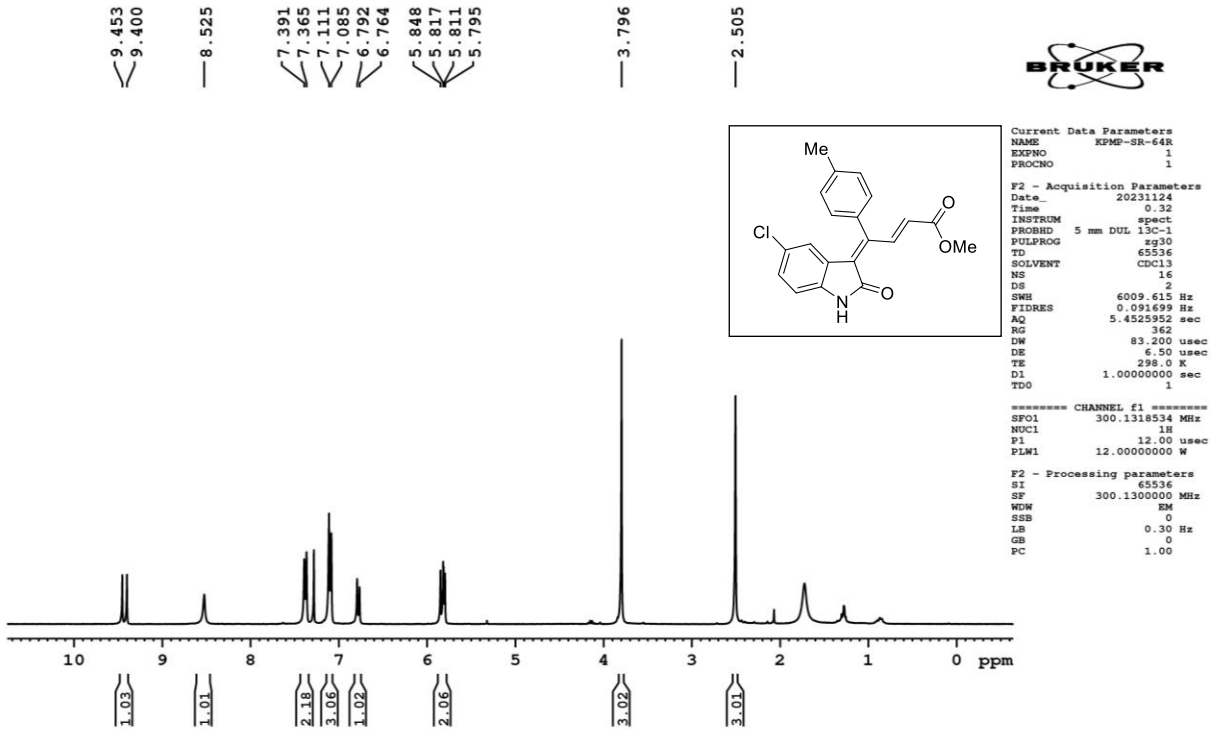


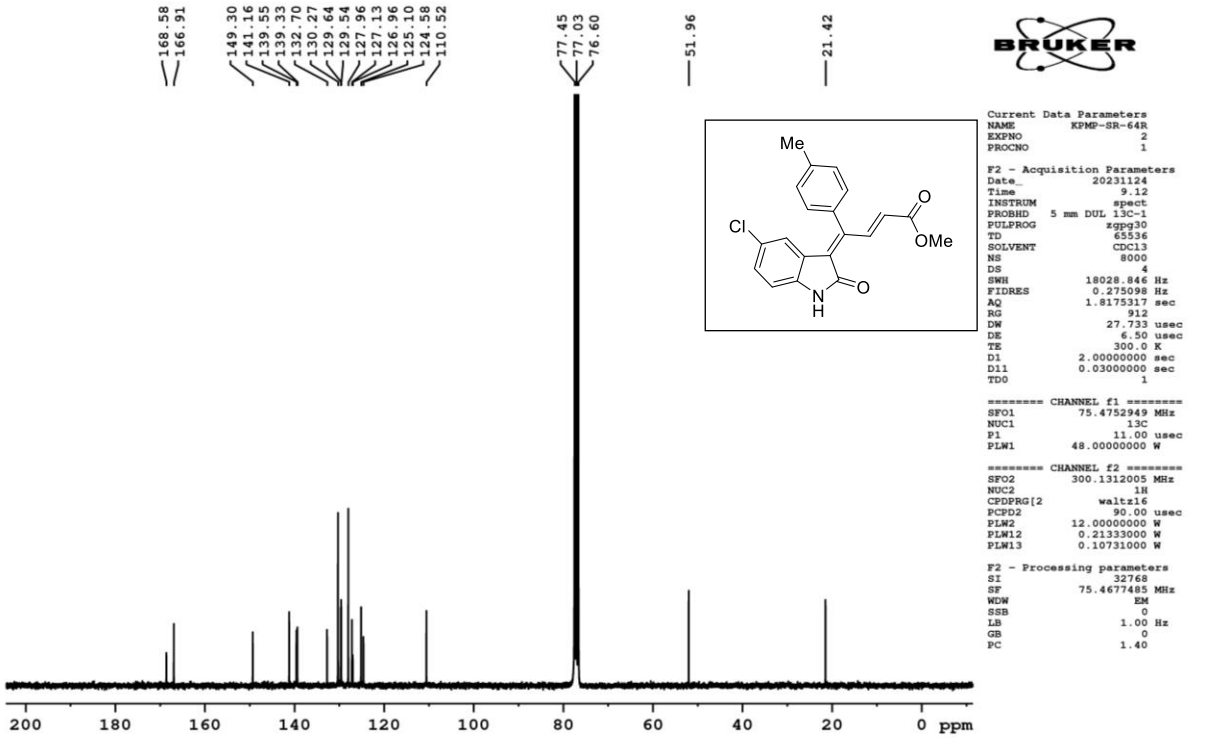
^{1}H and ^{13}C NMR spectra of compound **(3q)**



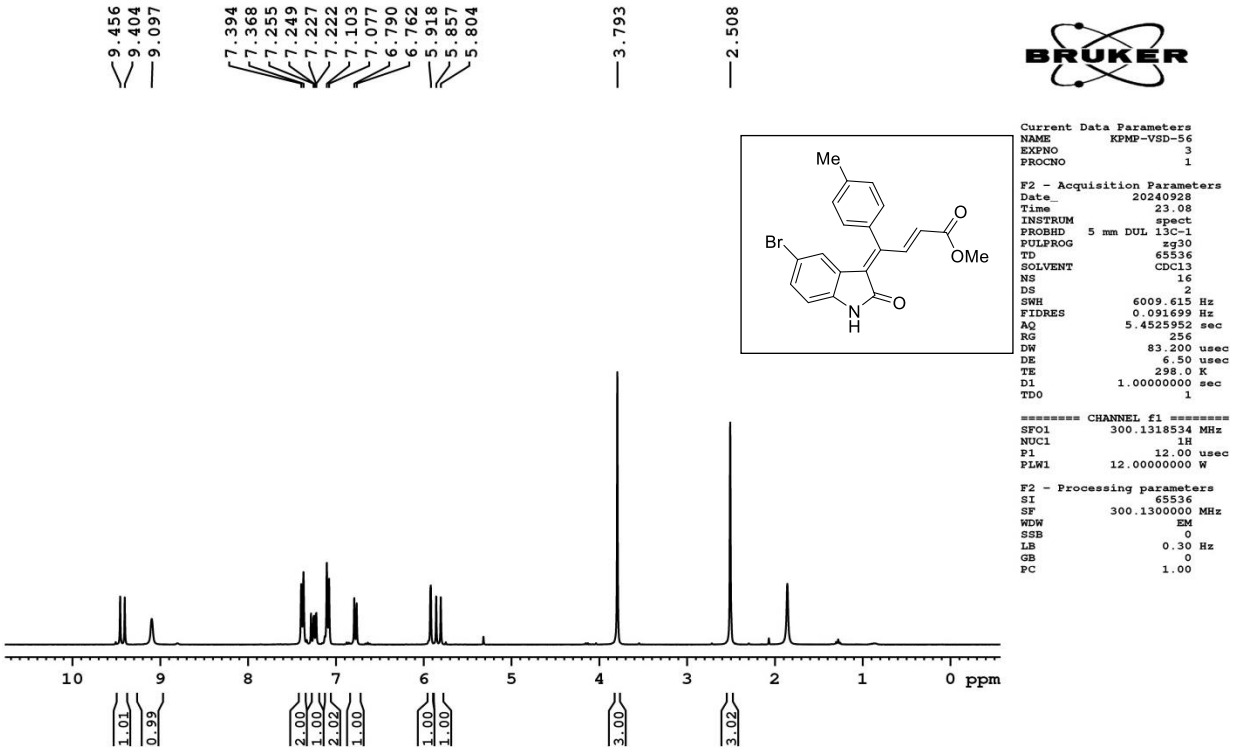


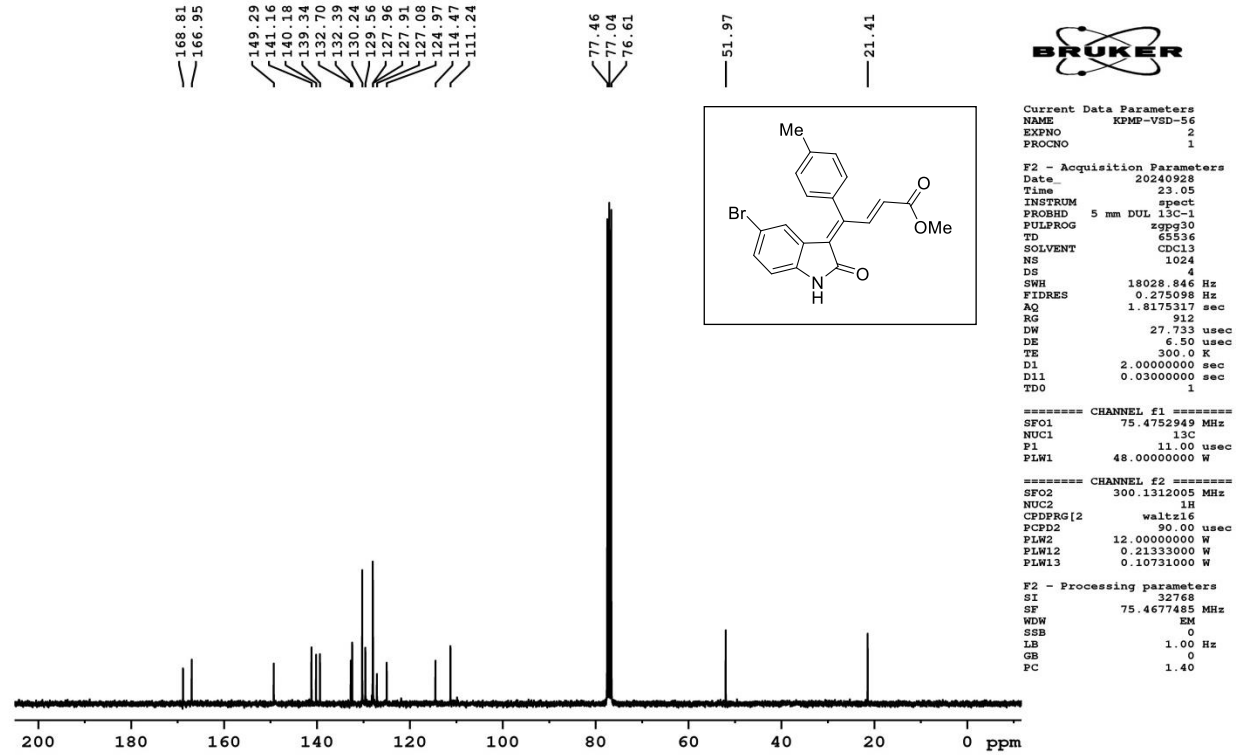
^{1}H and ^{13}C NMR spectra of compound **(3r)**



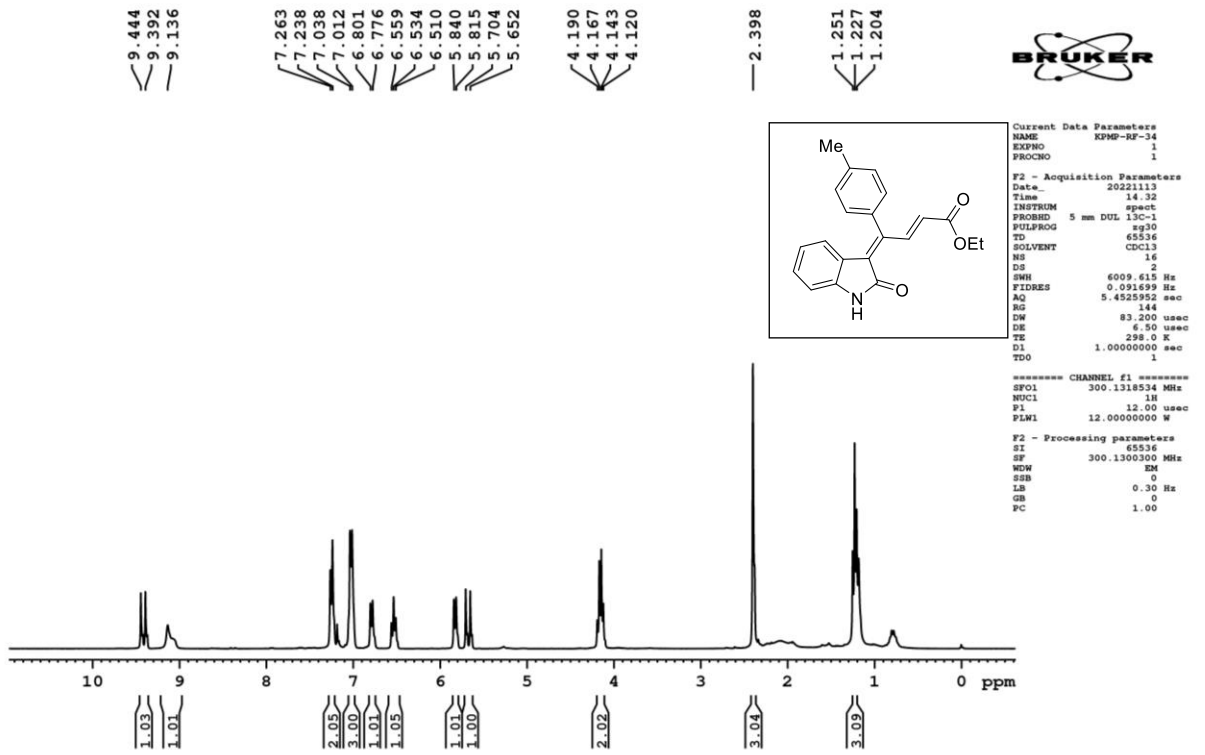


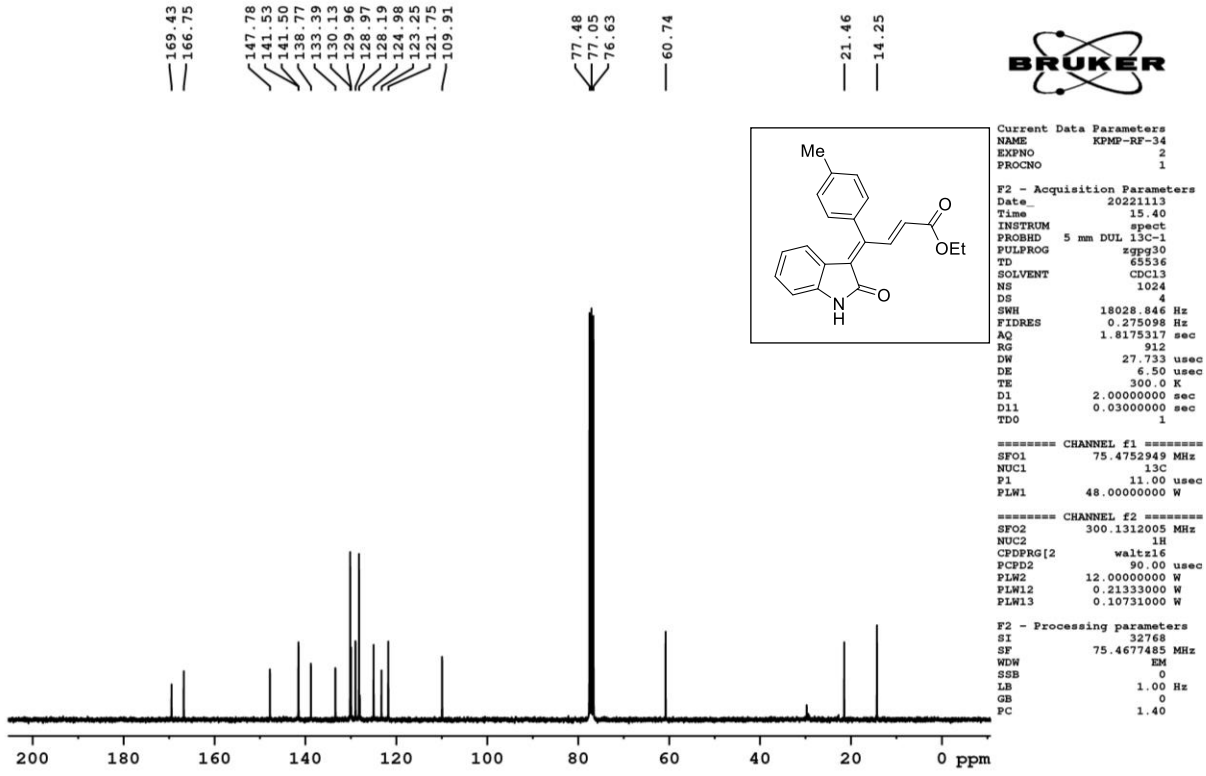
^{1}H and ^{13}C NMR spectra of compound **(3s)**



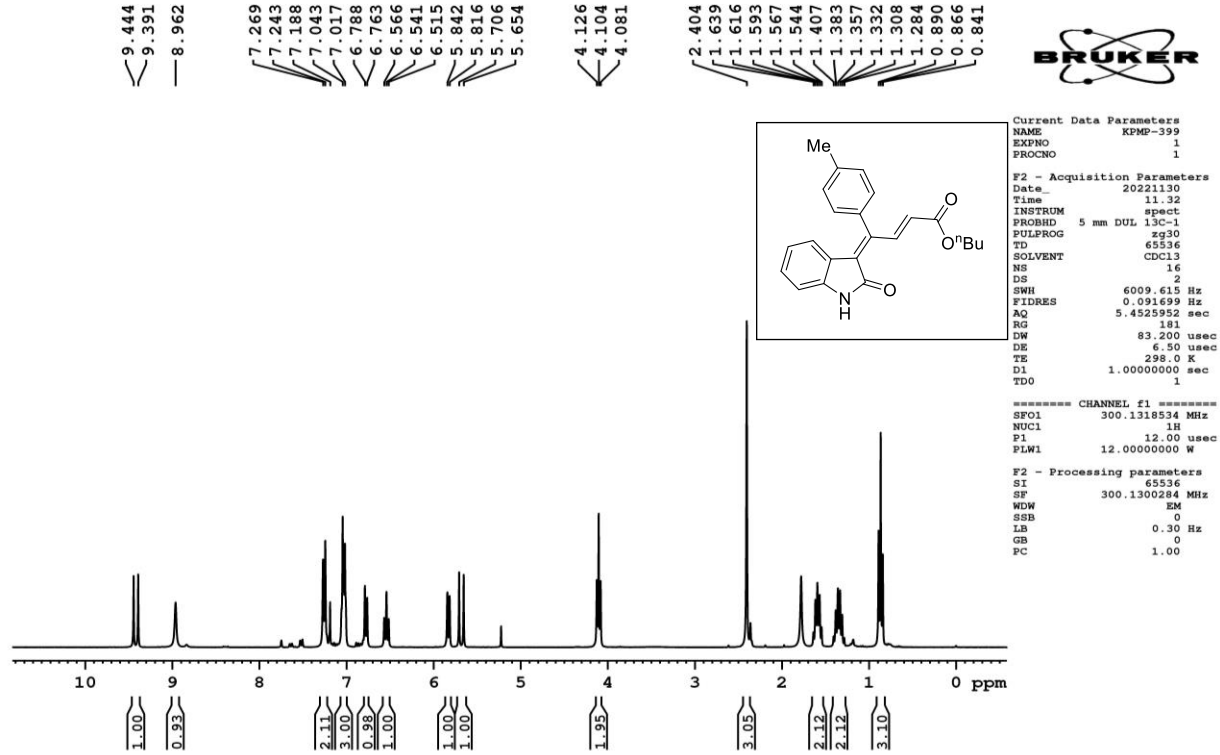


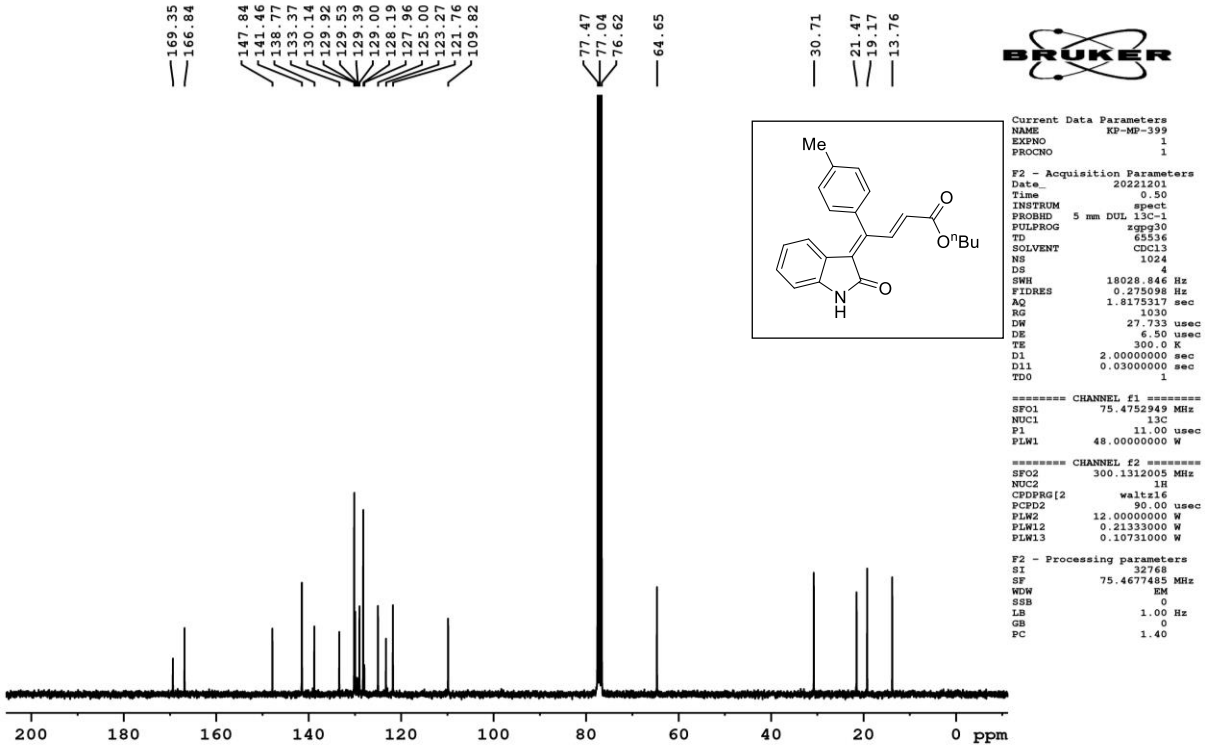
^{1}H and ^{13}C NMR spectra of compound **(3t)**



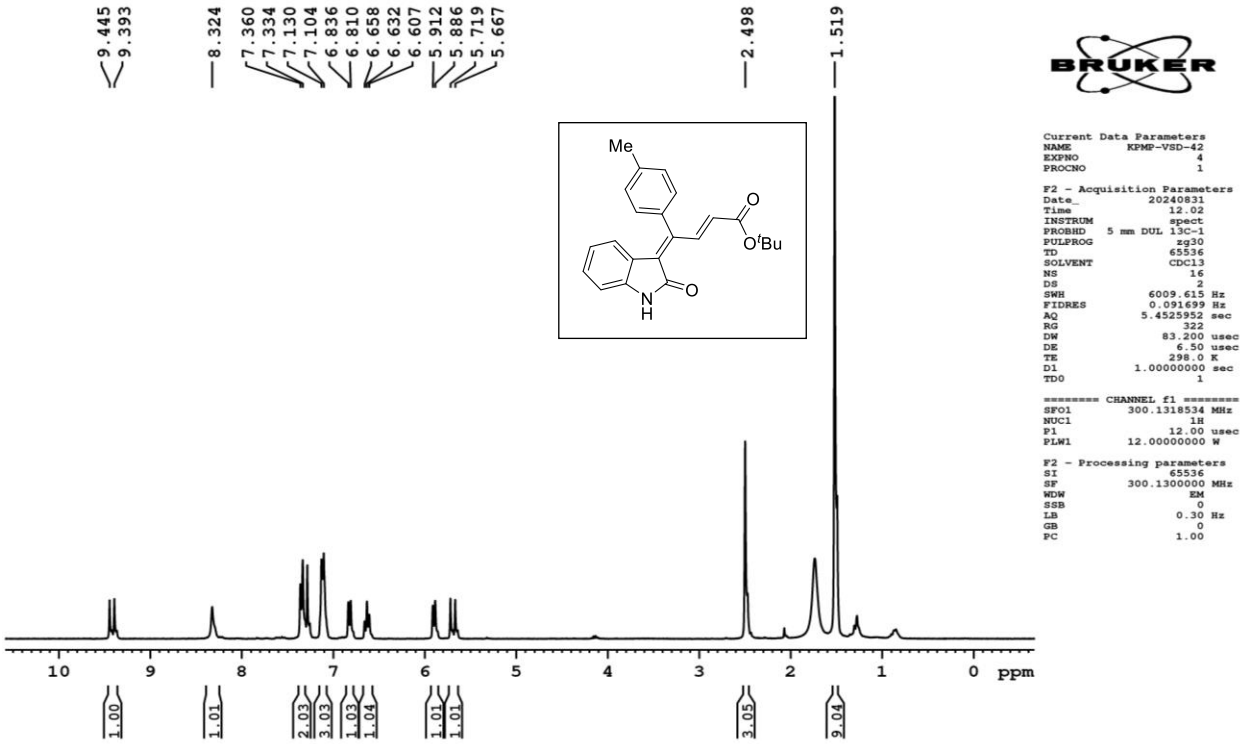


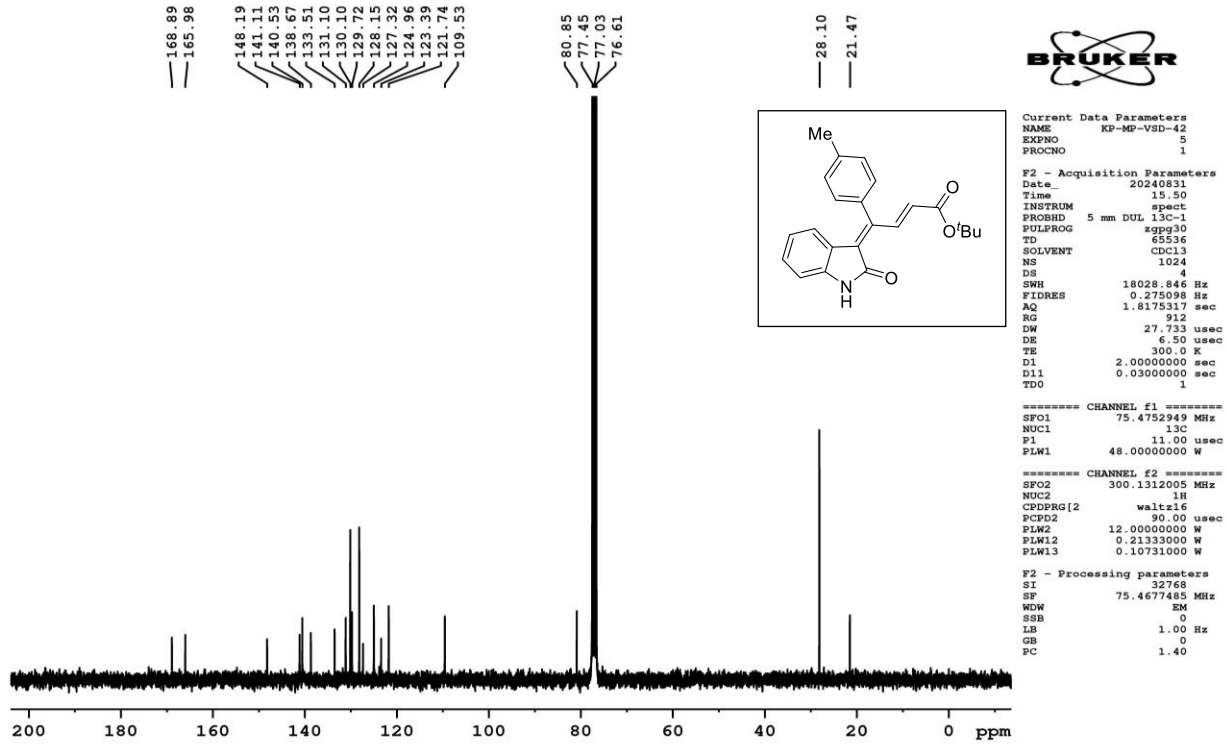
^{1}H and ^{13}C NMR spectra of compound **(3u)**





^{1}H and ^{13}C NMR spectra of compound **(3v)**



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NAME          KPMP-VSD-42
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_            20240831
Time                12.02
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              6009.615 Hz
FIDRES           0.091699 Hz
AQ               5.4525952 sec
RG                    322
DW                 83.200 usec
DE                  6.50 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          300.1318534 MHz
NUC1                   1H
P1                  12.00 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                  65536
SF          300.1300000 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

```
Current Data Parameters
NAME          KP-MP-VSD-42
EXPNO                   5
PROCNO                  1

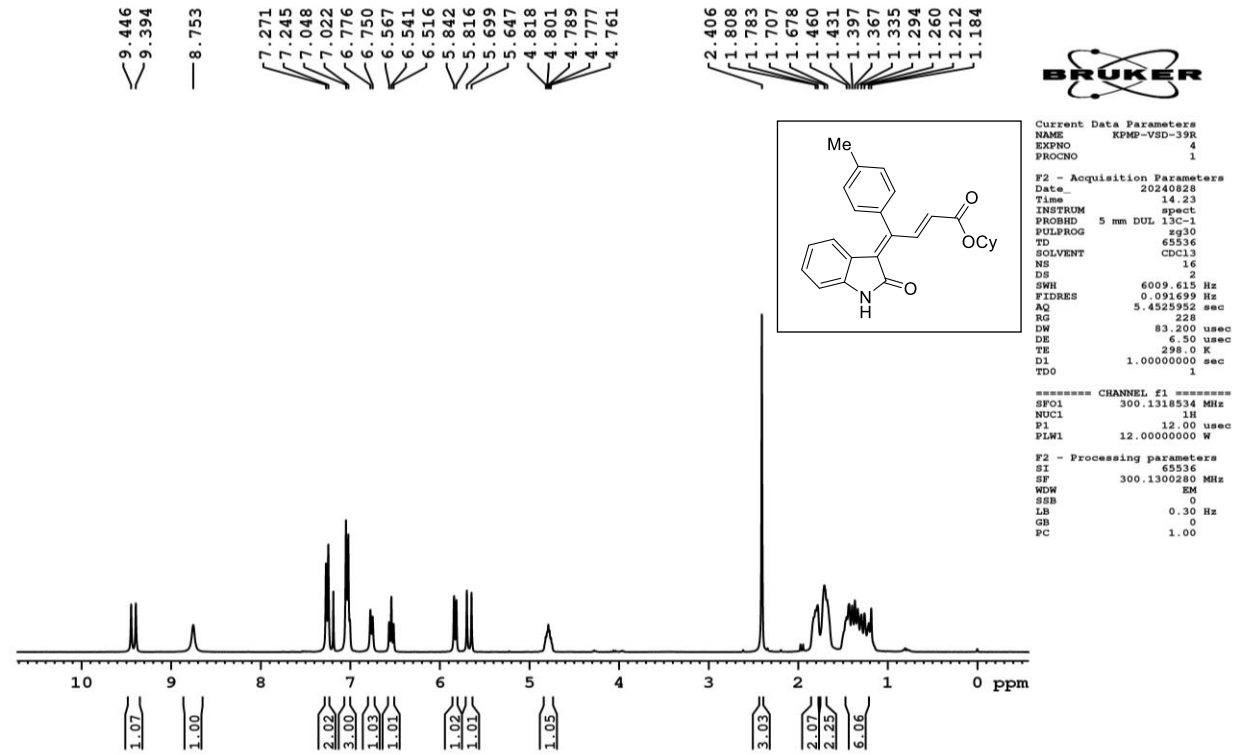
F2 - Acquisition Parameters
Date_            20240831
Time                15.50
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   1024
DS                      4
SWH             18028.846 Hz
FIDRES           0.275098 Hz
AQ               1.8175317 sec
RG                    912
DW                 27.733 usec
DE                  6.50 usec
TE                 300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          75.4752949 MHz
NUC1                   13C
P1                  11.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2         300.1312005 MHz
NUC2                   1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          12.00000000 W
PLW12          0.21333000 W
PLW13          0.10731000 W

F2 - Processing parameters
SI                  32768
SF          75.4677485 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

^{1}H and ^{13}C NMR spectra of compound **(3w)**

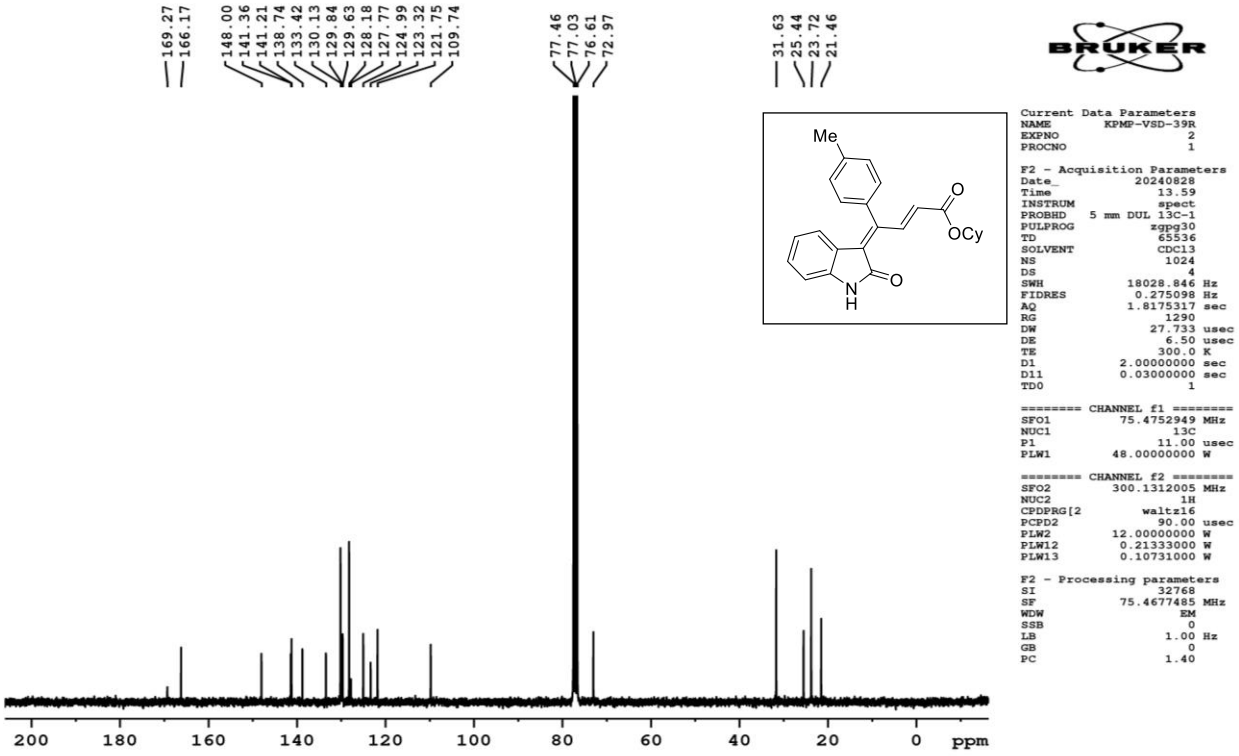



```
Current Data Parameters
NAME                  KPMP-VSD-39R
EXPNO                            4
PROCNO                           1

F2 - Acquisition Parameters
Date_                     20240828
Time                         14.23
INSTRUM                      spect
PROBHD     5 mm DUL 13C-1
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                              16
DS                               2
SWH                     6009.615 Hz
FIDRES                  0.091699 Hz
AQ                      5.4525952 sec
RG                             228
DW                         83.200 usec
DE                          6.50 usec
TE                          298.0 K
D1                      1.00000000 sec
TD0                              1

======== CHANNEL f1 ========
SFO1                300.1318534 MHz
NUC1                           1H
P1                         12.00 usec
PLW1                12.00000000 W

F2 - Processing parameters
SI                          65536
SF                300.1300280 MHz
WDW                            EM
SSB                             0
LB                         0.30 Hz
GB                              0
PC                           1.00
```




```
Current Data Parameters
NAME                  KPMP-VSD-39R
EXPNO                            2
PROCNO                           1

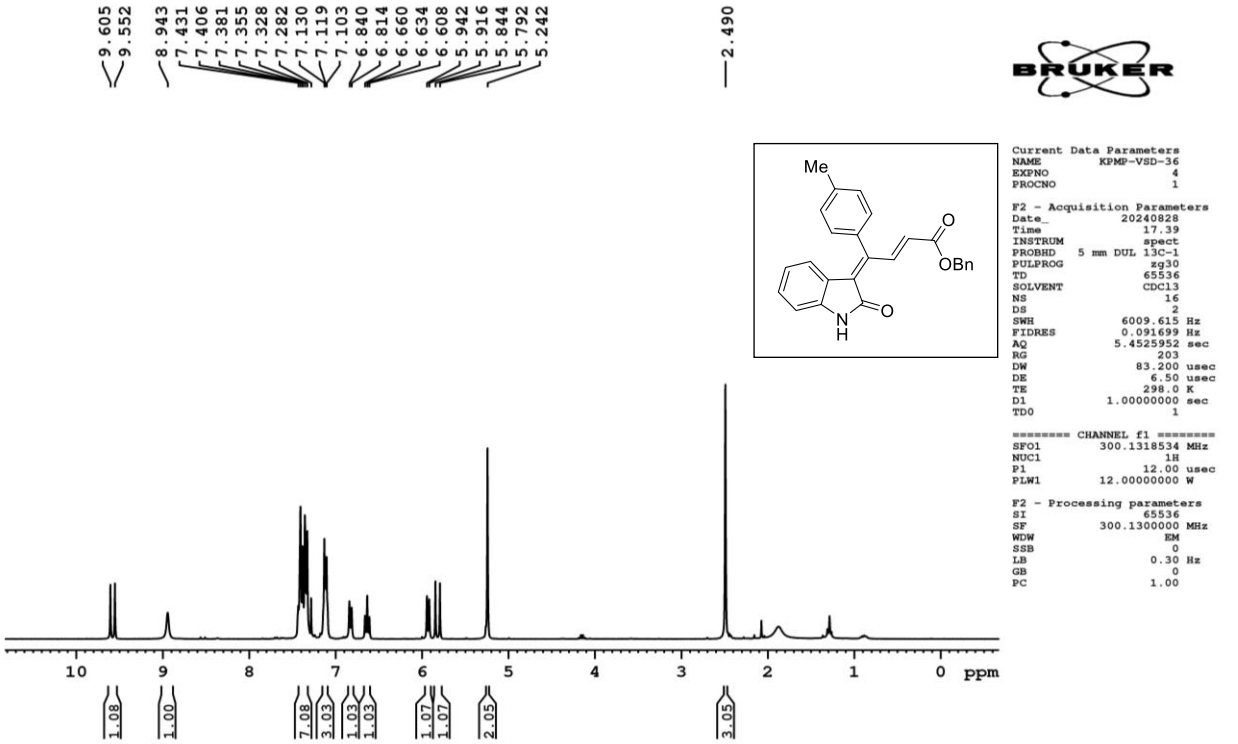
F2 - Acquisition Parameters
Date_                     20240828
Time                         13.59
INSTRUM                      spect
PROBHD     5 mm DUL 13C-1
PULPROG                   zgpg30
TD                          65536
SOLVENT                     CDCl3
NS                            1024
DS                               4
SWH                    18028.846 Hz
FIDRES                  0.275098 Hz
AQ                      1.8175317 sec
RG                            1290
DW                         27.733 usec
DE                          6.50 usec
TE                          300.0 K
D1                      2.00000000 sec
D11                     0.03000000 sec
TD0                              1

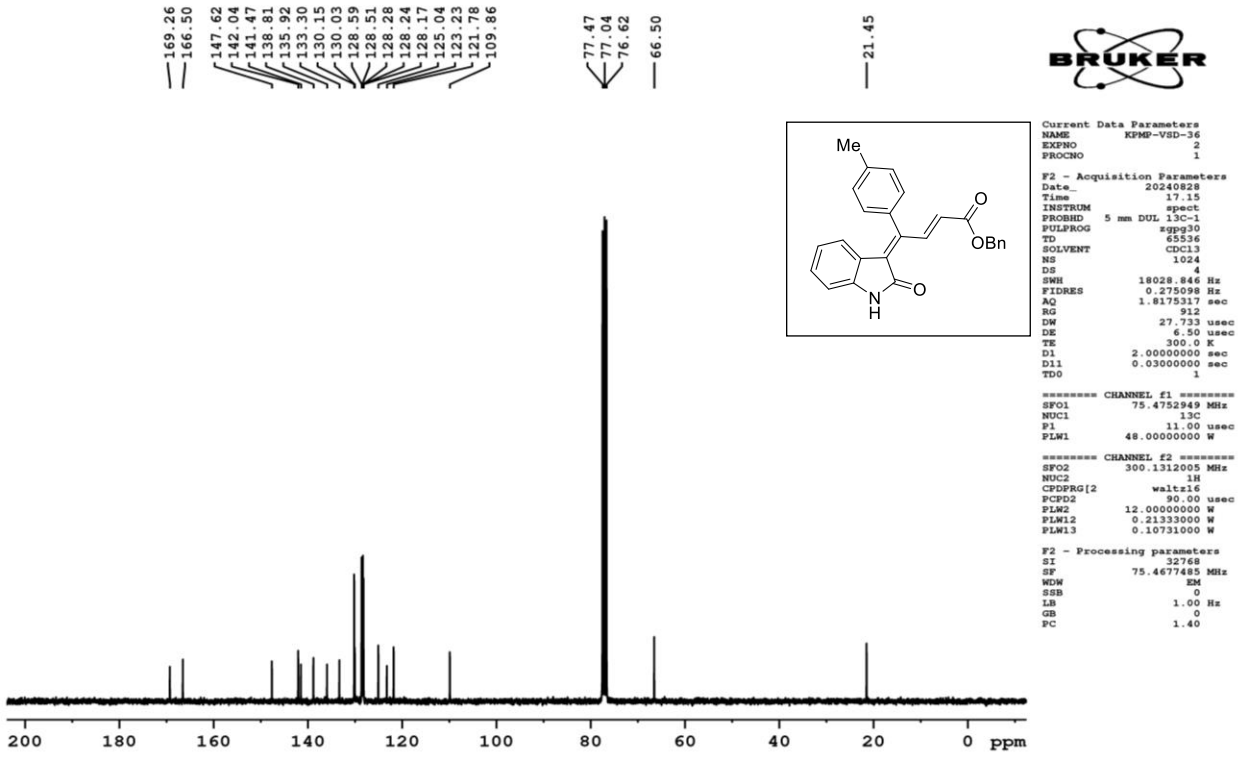
======== CHANNEL f1 ========
SFO1                75.4752949 MHz
NUC1                           13C
P1                         11.00 usec
PLW1                48.00000000 W

======== CHANNEL f2 ========
SFO2               300.1312005 MHz
NUC2                           1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2               12.00000000 W
PLW12               0.21333000 W
PLW13               0.10731000 W

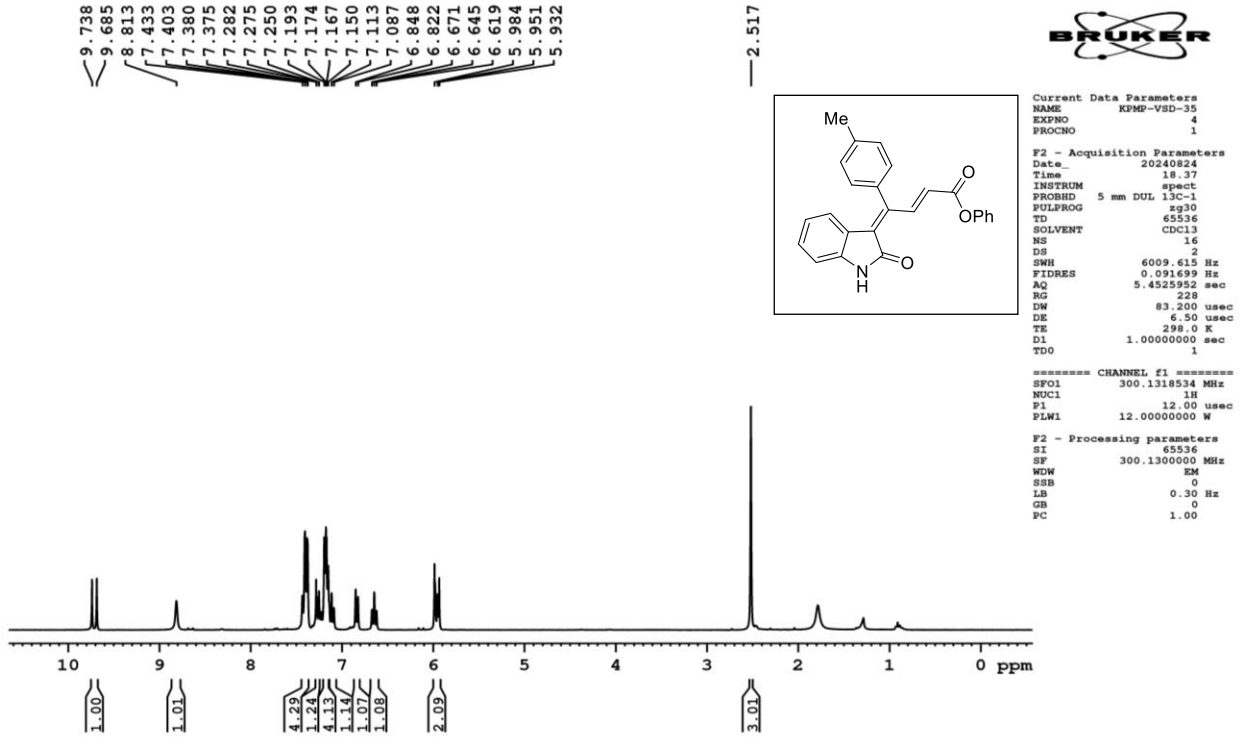
F2 - Processing parameters
SI                          32768
SF                75.4677485 MHz
WDW                            EM
SSB                             0
LB                         1.00 Hz
GB                              0
PC                           1.40
```

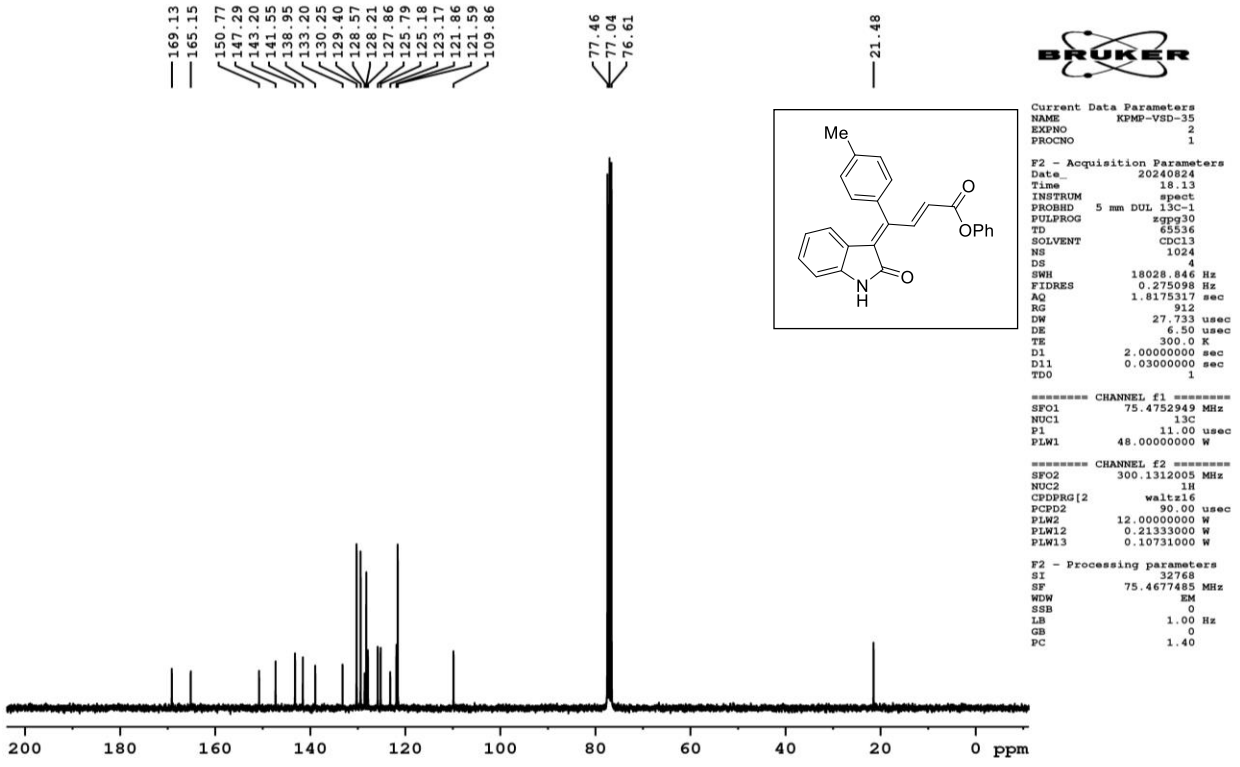
^{1}H and ^{13}C NMR spectra of compound **(3x)**



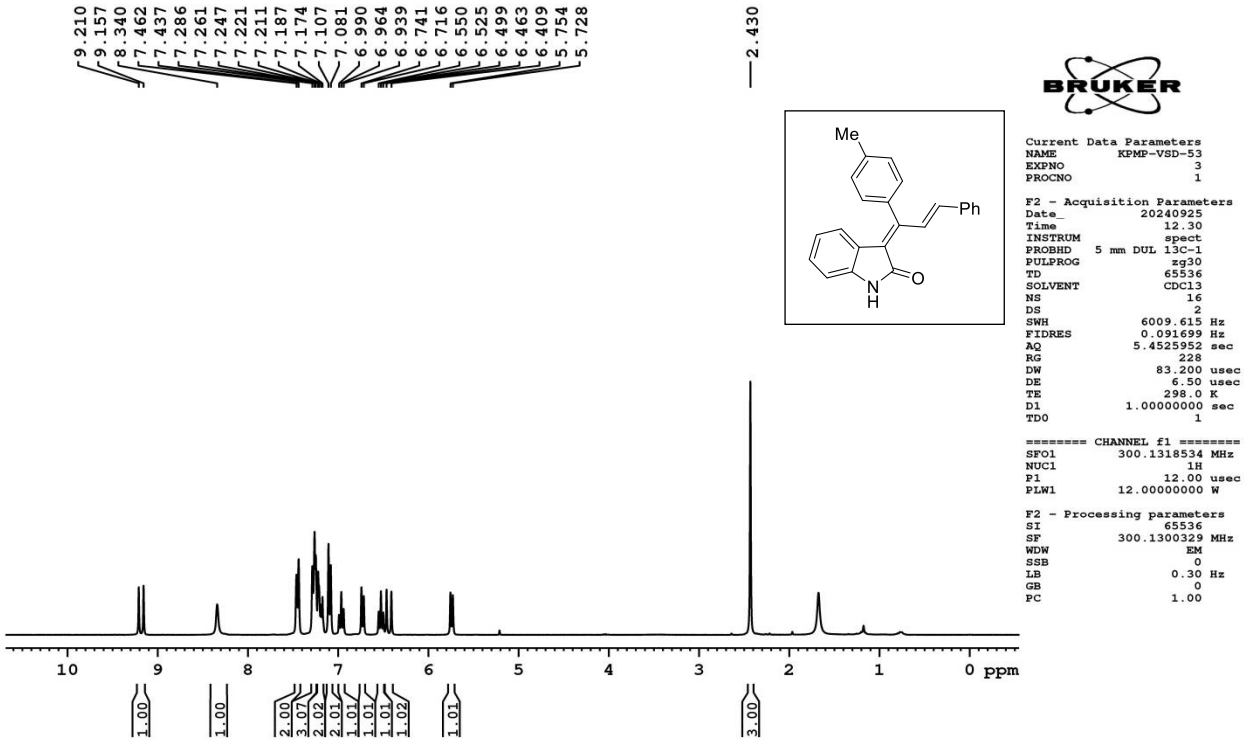


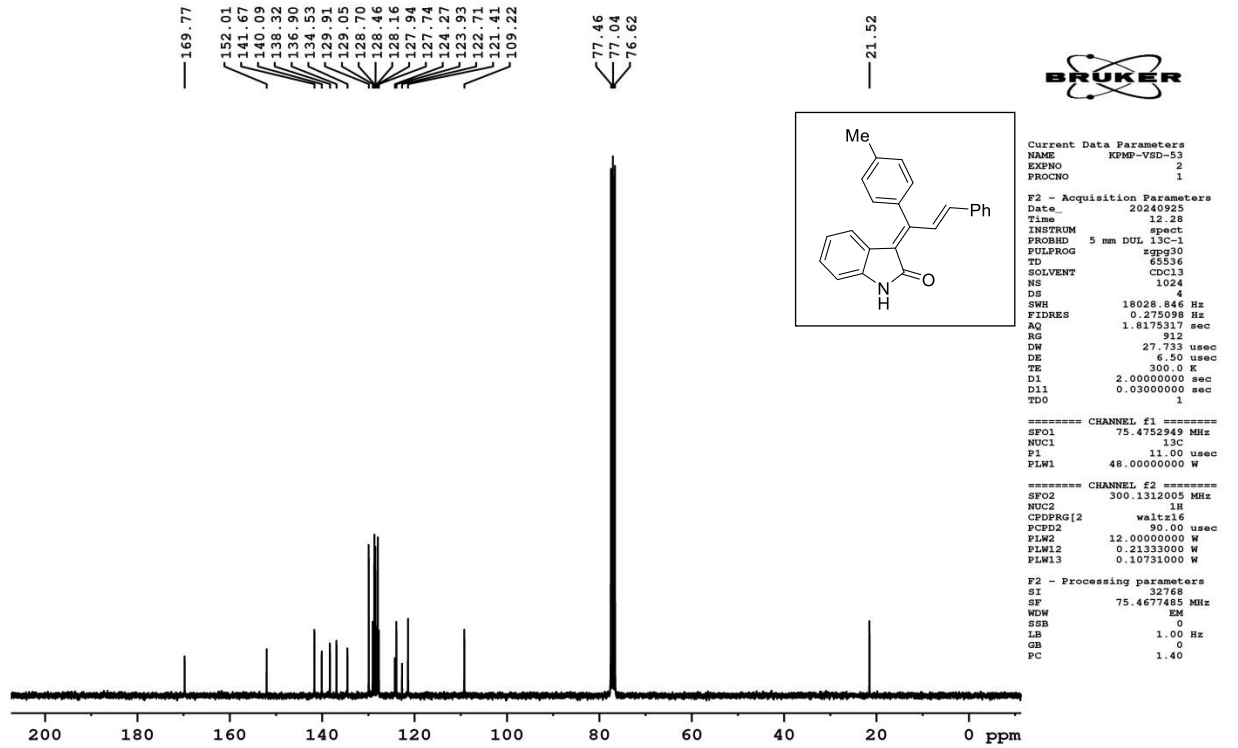
^{1}H and ^{13}C NMR spectra of compound **(3y)**



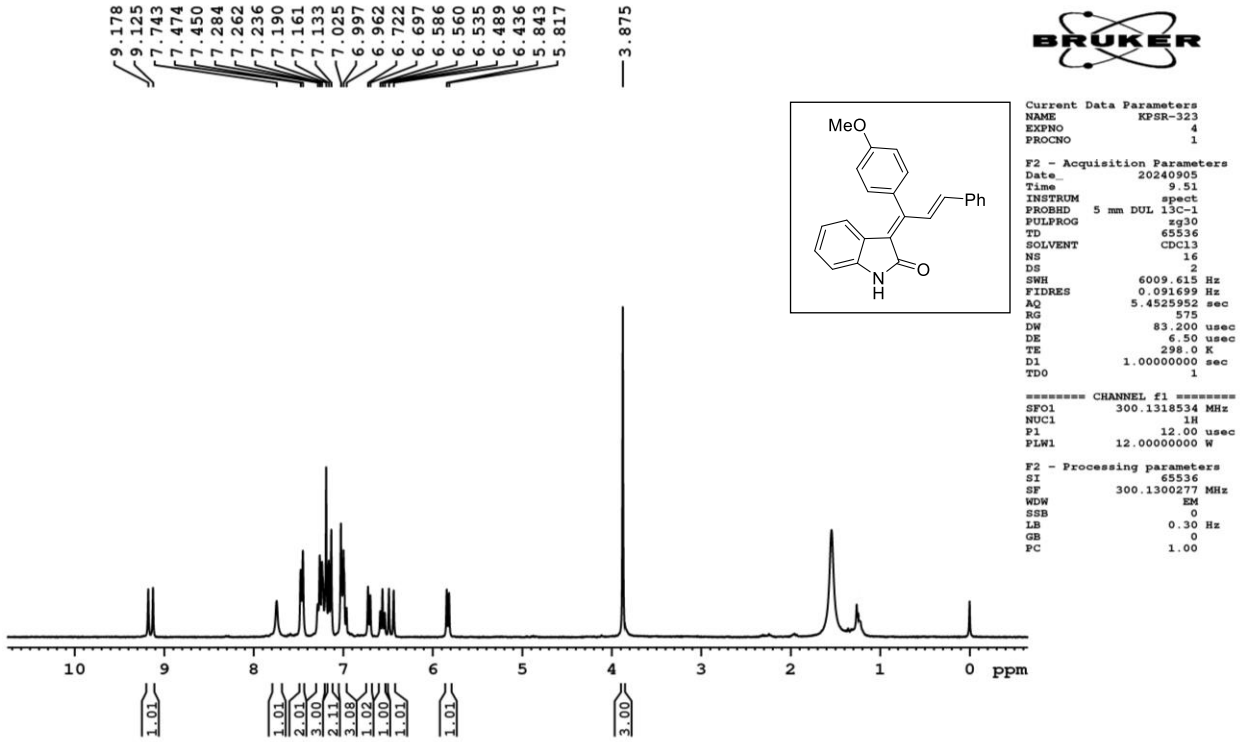


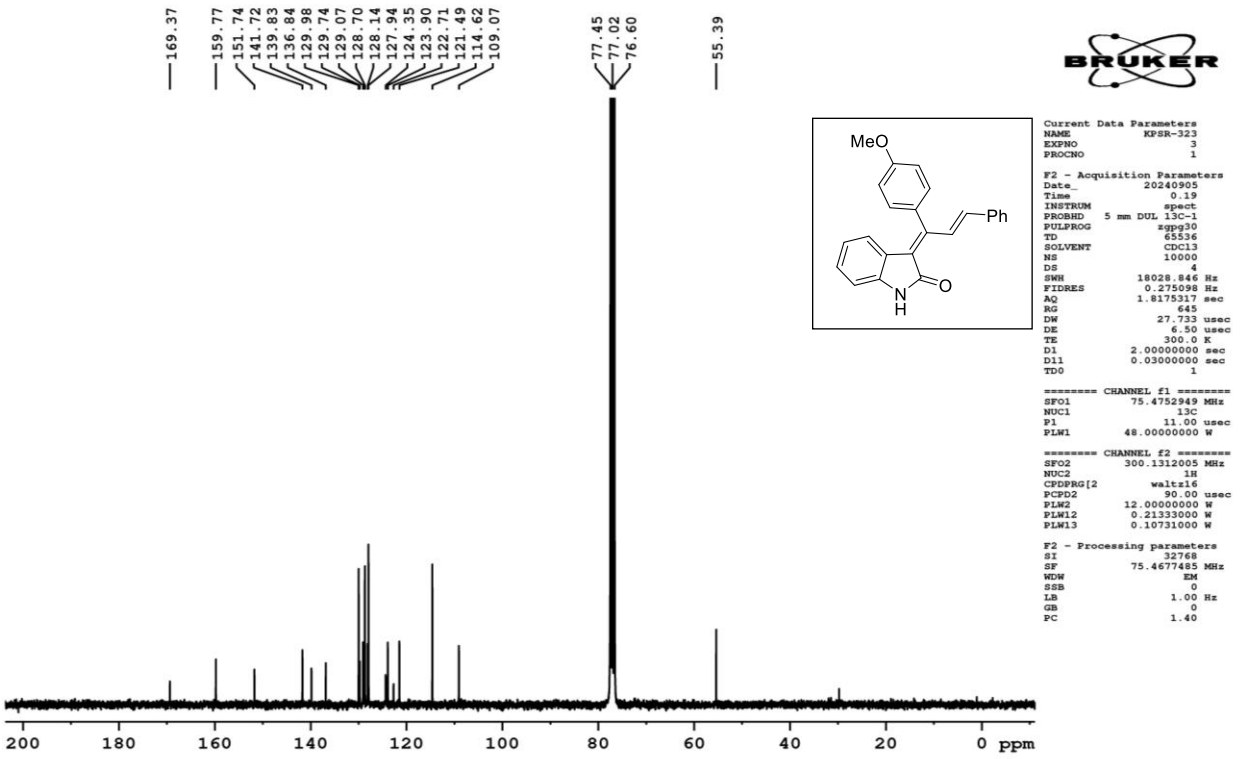
^{1}H and ^{13}C NMR spectra of compound **(3z)**



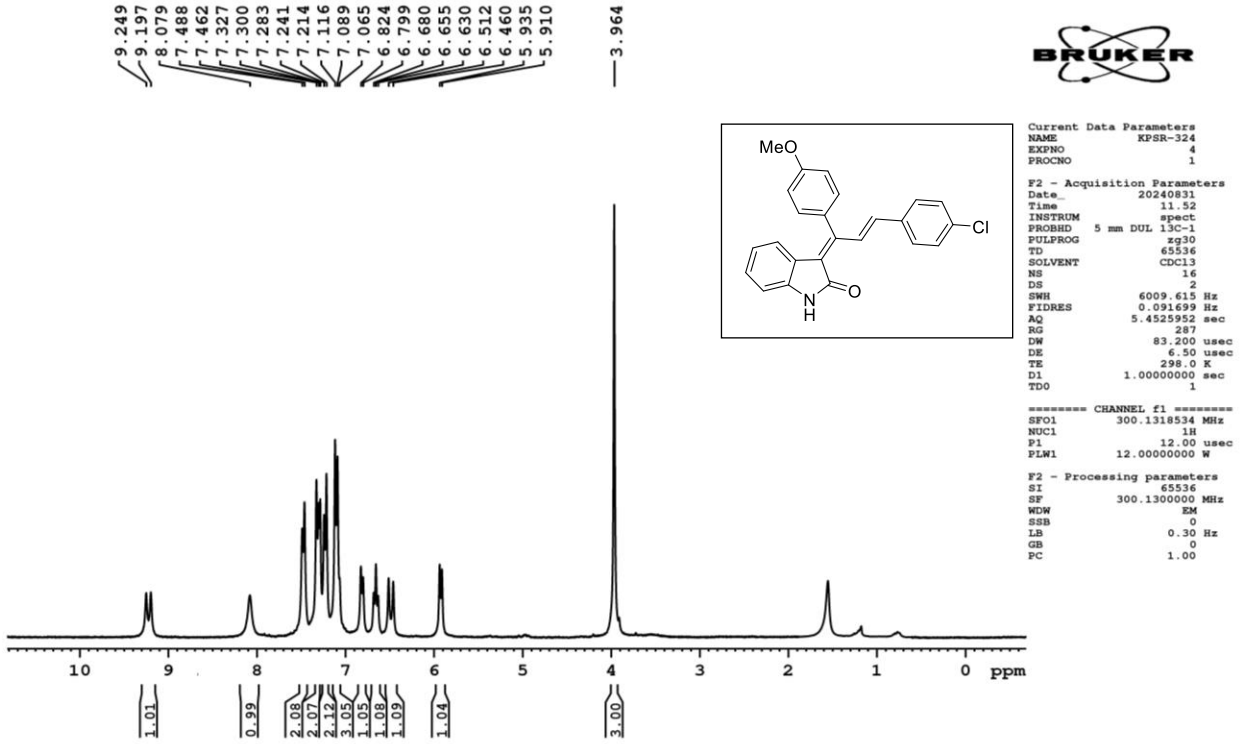


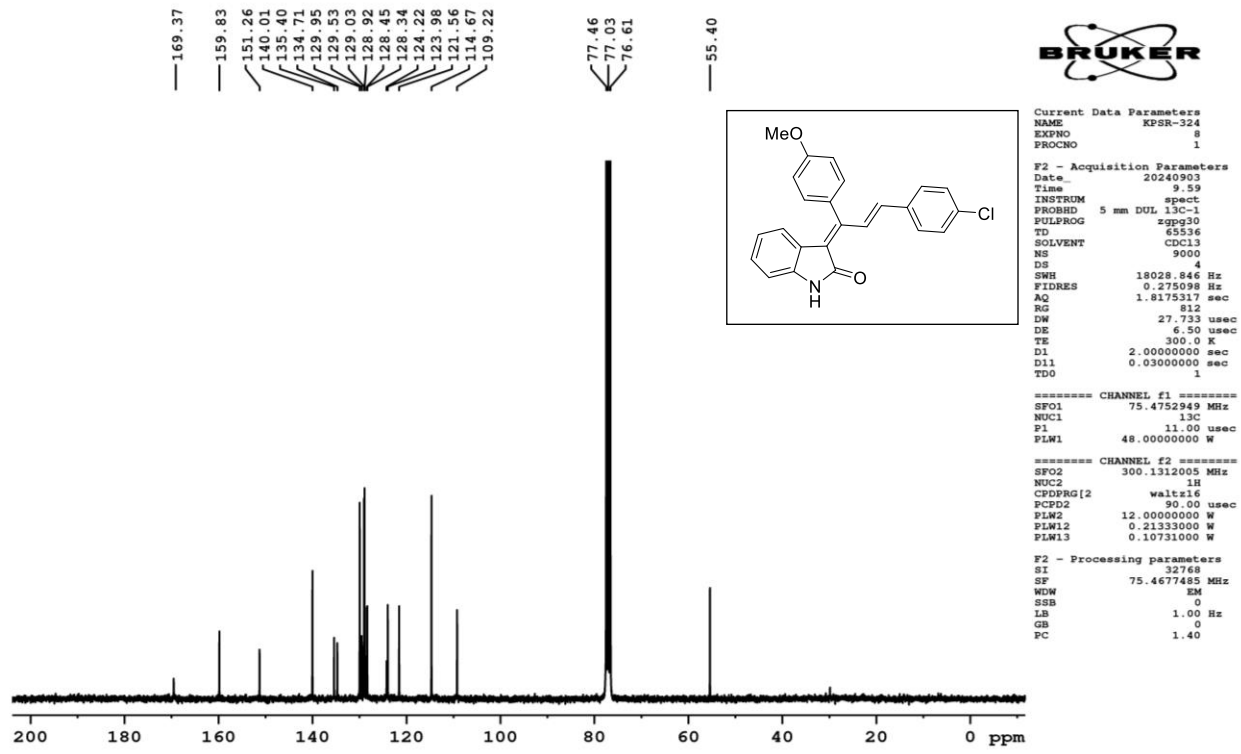
^{1}H and ^{13}C NMR spectra of compound **(3aa)**



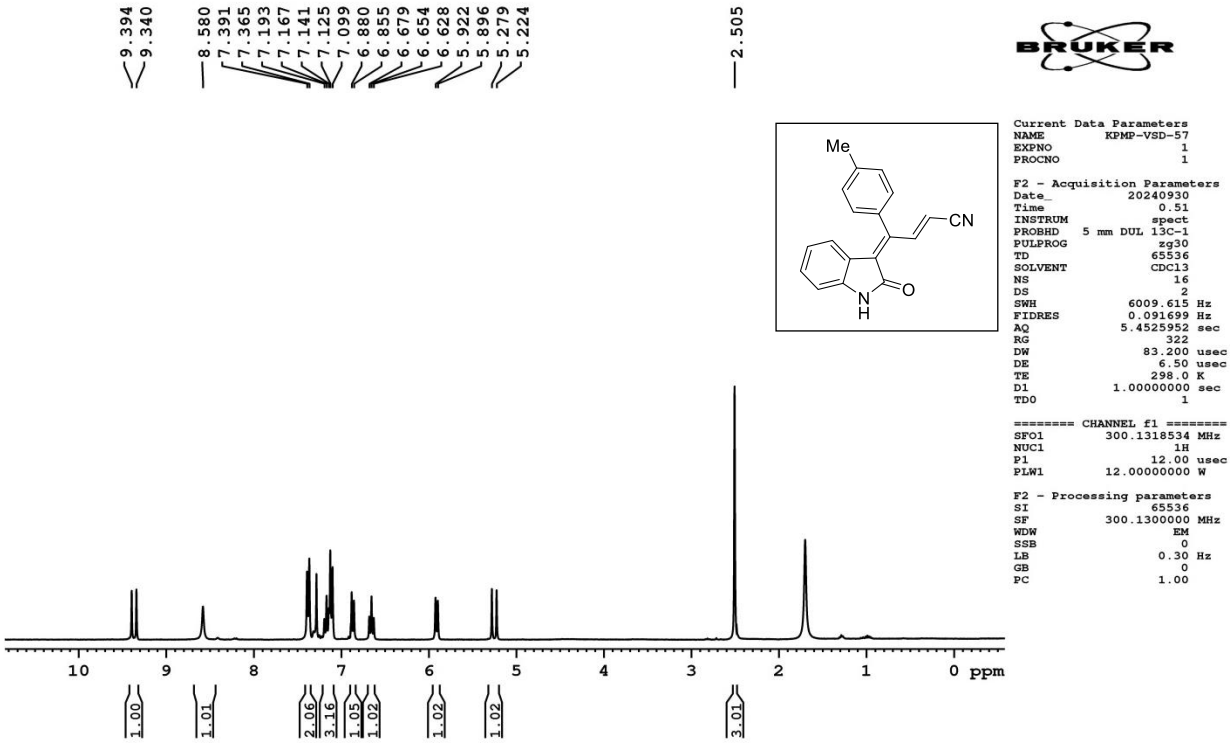


^{1}H and ^{13}C NMR spectra of compound **(3ab)**





^{1}H and ^{13}C NMR spectra of compound **(3ac)**

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EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_              20240930
Time                   0.51
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6009.615 Hz
FIDRES            0.091699 Hz
AQ                5.4525952 sec
RG                      322
DW                   83.200 usec
DE                    6.50 usec
TE                  298.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           300.1318534 MHz
NUC1                     1H
P1                    12.00 usec
PLW1          12.00000000 W

F2 - Processing parameters
SI                    65536
SF               300.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
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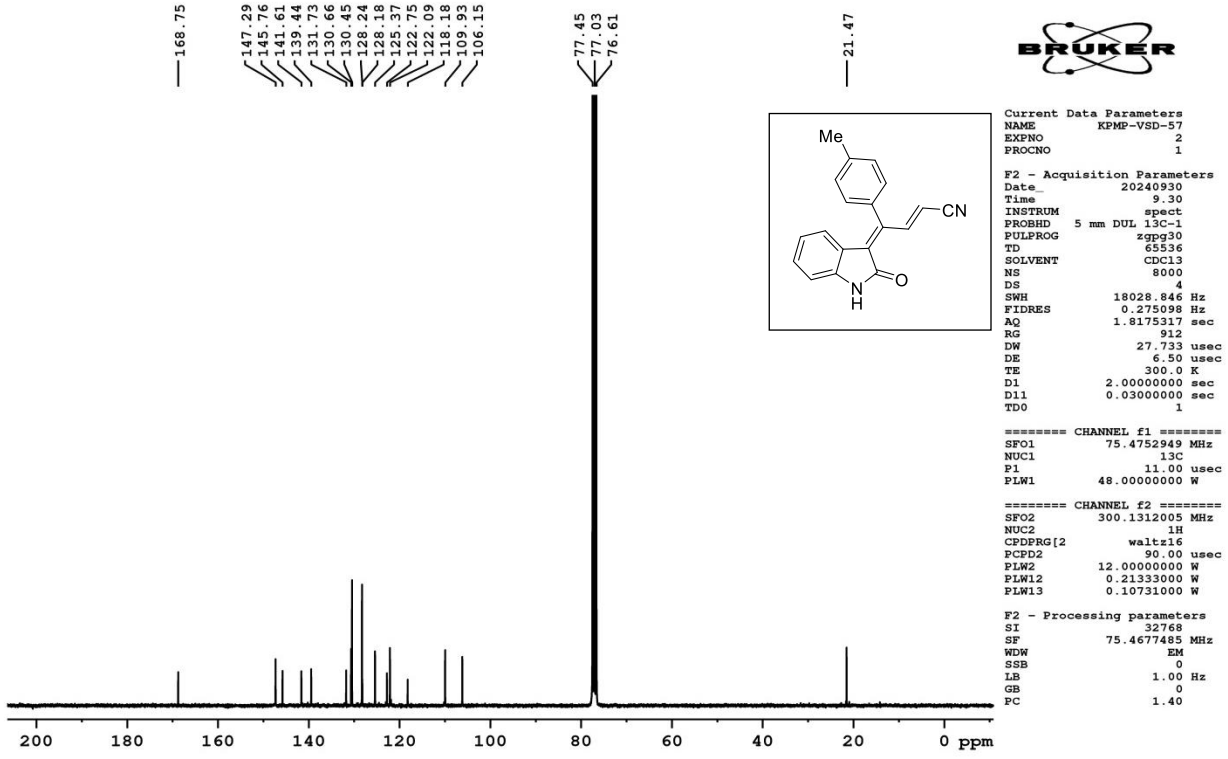



```
Current Data Parameters
NAME          KPMP-VSD-57
EXPNO                   2
PROCNO                  1

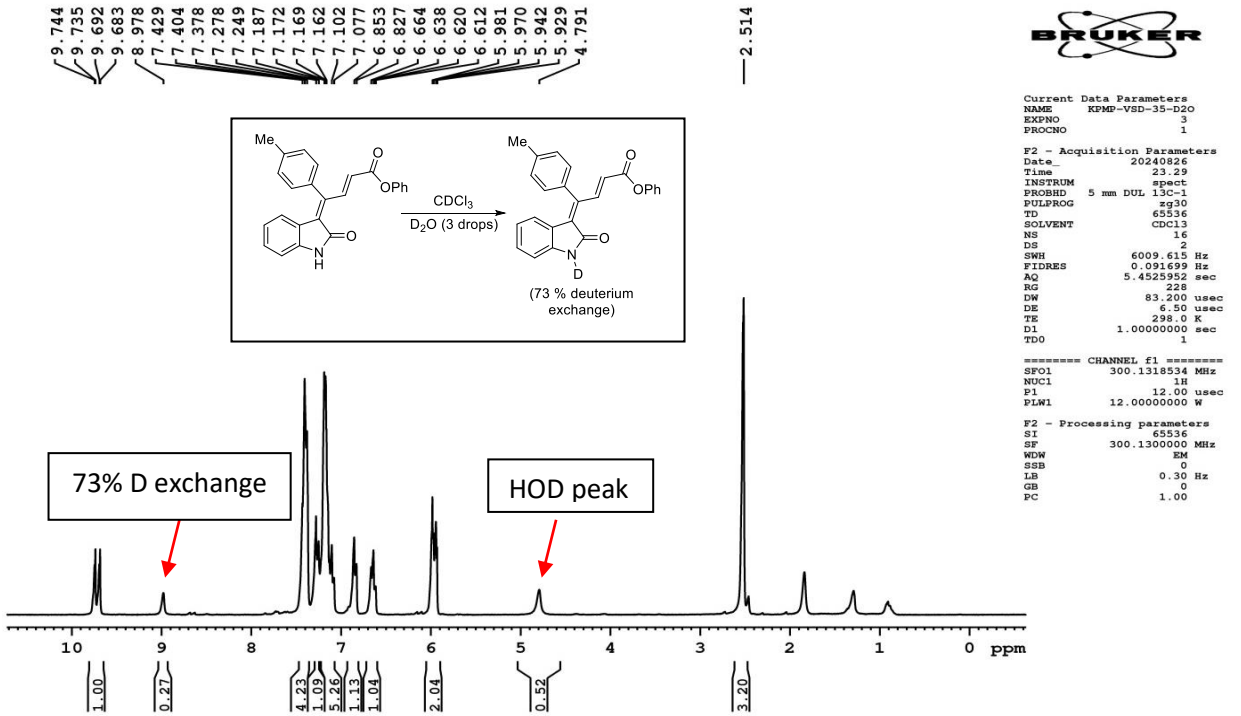
F2 - Acquisition Parameters
Date_              20240930
Time                   9.30
INSTRUM               spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     8000
DS                        4
SWH               18028.846 Hz
FIDRES            0.275098 Hz
AQ                1.8175317 sec
RG                      912
DW                   27.733 usec
DE                    6.50 usec
TE                  300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           75.4752949 MHz
NUC1                    13C
P1                    11.00 usec
PLW1          48.00000000 W

======== CHANNEL f2 ========
SFO2          300.1312005 MHz
NUC2                     1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2          12.00000000 W
PLW12          0.21333000 W
PLW13          0.10731000 W

F2 - Processing parameters
SI                    32768
SF               75.4677485 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

D_2O exchange of the compound **3y**



^1H spectrum of the compound **3y**