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Microwave-Assisted Catalyst-Free Hydride Transfer: Synthesis and Evaluation of Antioxidant properties of N-Benzylindolines

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General information:

All commercially available reagents were used as received. Melting points were measured in open capillary tubes using a Buchi-540 micro melting point apparatus and were uncorrected. Infrared spectra were obtained on a Perkin-Elmer System 2000 FT-IR spectrometer. High-resolution mass spectra (ESI-HRMS) were recorded using an Agilent Accurate-Mass Q-TOF LC/MS 6520. NMR spectra were acquired on a Bruker Avance DPX-400 or DPX-500 spectrometer with tetramethylsilane (TMS) as the internal standard at room temperature. Chemical shifts (δ) are reported in parts per million (ppm), and coupling constants (J) are given in Hertz (Hz). Thin layer chromatography (TLC) was performed on pre-coated silica gel plates (Merck) and visualized under a UV lamp at 254 nm for UV-active compounds. Additional visualization was done using iodine vapor. Column chromatography was carried out on silica gel (100-200 mesh, Merck) with ethyl acetate/hexane as the eluent.

Microwave instrumentation:

All microwave reactions were conducted using a Synthos 3000 (Anton Paar) microwave reactor. This system has a dual magnetron operating at 2.45 GHz, with a maximum power output of 1400 W. The output power is adjustable in 1 W increments and can be controlled continuously without pulsing. A 68xxx series microprocessor system monitors power, pressure, time, and temperature during the reaction. Temperature and pressure were continuously tracked using an infrared detector. The temperature range extends from 0 to 280 °C with an accuracy of ±1%, while pressure can be measured from 0 to 86 bar with an accuracy of ±0.2 bar.

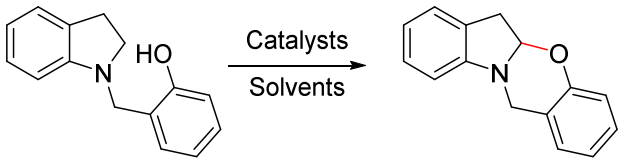
Representative procedure for the synthesis of 4a:

Salicylaldehyde (1.0 mmol, 122 mg), **indoline** (2.0 mmol, 238 mg) were irradiated in a closed vessel under solvent-free conditions inside a microwave reactor at 120 °C for 15 min. The progress of the reaction was monitored by a TLC. Upon completion, the reaction mixture was dissolved in ethyl acetate and washed with water. The organic layer was separated and dried over anhydrous Na_2SO_4. The solvent was then evaporated using a rotary evaporator, and the crude product was purified by column chromatography on silica gel (100-200 mesh) using ethyl acetate/hexane as the eluent. The same procedure has been followed for the synthesis of **9a-b**.

Representative procedure for the synthesis of 10:

Salicylaldehyde (1.0 mmol, 122 mg), **indoline** (2.0 mmol, 238 mg), **TBHP** (70% in H_2O, 1.5 eq., 193 mg), and iodine (10 mol%, 25 mg) were irradiated in a closed vessel under solvent-free conditions at 120 °C for 15 minutes using a microwave reactor. The reaction progress was monitored by thin-layer chromatography (TLC). Once complete, the reaction mixture was dissolved in ethyl acetate and washed with water. The organic layer was separated, dried over anhydrous sodium sulfate, and the solvent was removed using a rotary evaporator. The crude product was purified by column chromatography on silica gel (100-200 mesh) with ethyl acetate/hexane as the eluent.

Table S1. Attempt to synthesize 1,3-oxazines



4 **Expected product**

Entry	Catalyst (mol%)	Oxidant (eq.)	Solvent	Temp. (°C)	Time (h)	Yield (%)
1	I_2 (10)	TBHP (1.5)	---	120	4	n.f.
2	I_2 (10)	TBHP (1.5)	DCE	120	4	n.f.
3	$Cu(OAc)_2$ (10)	TBHP (1.5)	DCE	120	4	n.f.
4	$Cu(OAc)_2$ (10)	TBHP (1.5)	Toluene	120	4	n.f.
5	$FeCl_3.6H_2O$ (10)	TBHP (1.5)	Toluene	120	5	n.f.
6	$FeCl_3.6H_2O$ (10)	TBHP (1.5)	DMSO	120	5	n.f.
7	$CoCl_2$ (10)	TBHP (1.5)	DMSO	120	5	n.f.
8	$CoCl_2$ (10)	H_2O_2 (1.5)	DMSO	120	5	n.f.

Table S2: Absorbance data for compound **4a** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.67	0.87	22.98851	
10	0.336	0.87	61.37931	4.8829
20	0.165	0.87	81.03448	
30	0.075	0.87	91.37931	
40	0.066	0.87	92.41379	
50	0.061	0.87	92.98851	
60	0.059	0.87	93.21839	

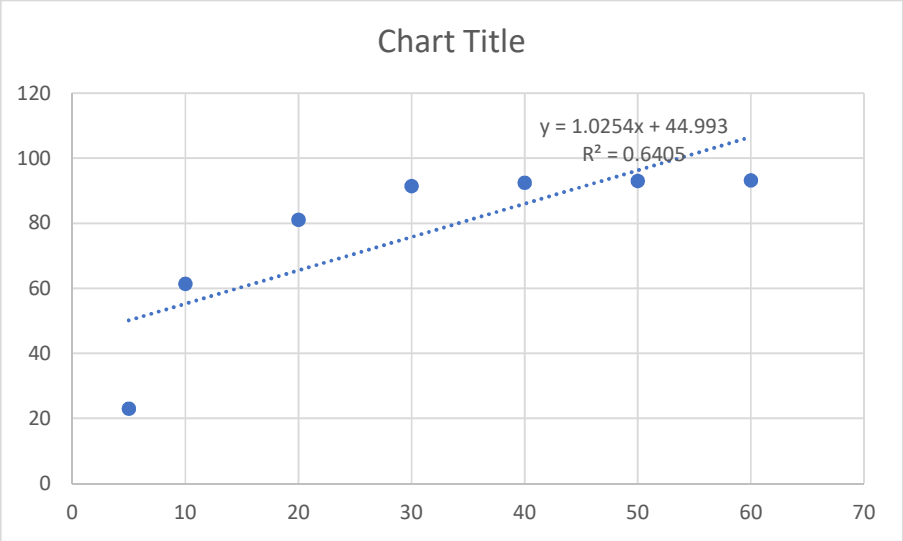


Fig S1: Linear regression curve for the antioxidant activity of compound **4a**

Table S3: Absorbance data for compound **4b** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.803	0.87	7.701149	
10	0.731	0.87	15.97701	
20	0.575	0.87	33.90805	32.28

30	0.443	0.87	49.08046	
40	0.308	0.87	64.5977	
50	0.195	0.87	77.58621	
60	0.129	0.87	85.17241	

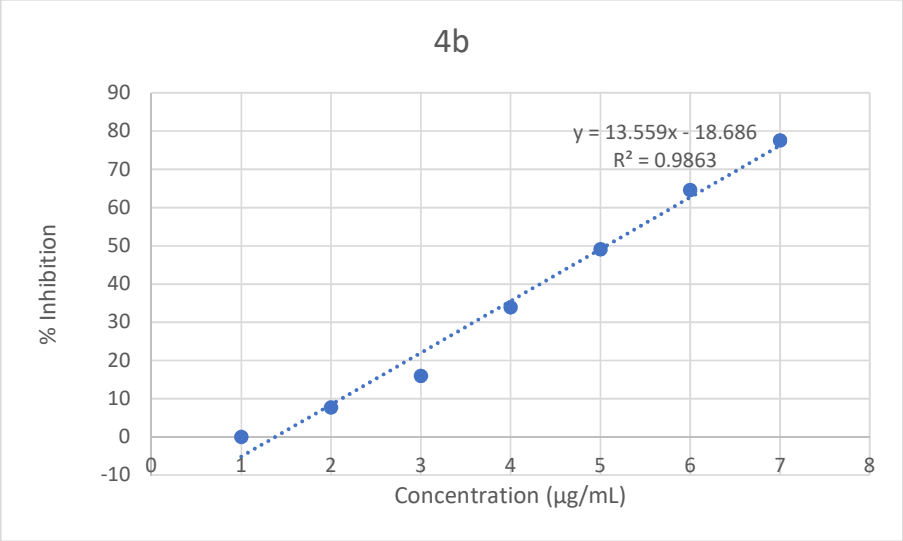


Fig S2: Linear regression curve for the antioxidant activity of compound **4b**

Table S4: Absorbance data for compound **4c** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.649	0.87	25.4023	
10	0.576	0.87	33.7931	17.5083
20	0.399	0.87	54.13793	
30	0.149	0.87	82.87356	
40	0.073	0.87	91.6092	
50	0.062	0.87	92.87356	
60	0.057	0.87	93.44828	

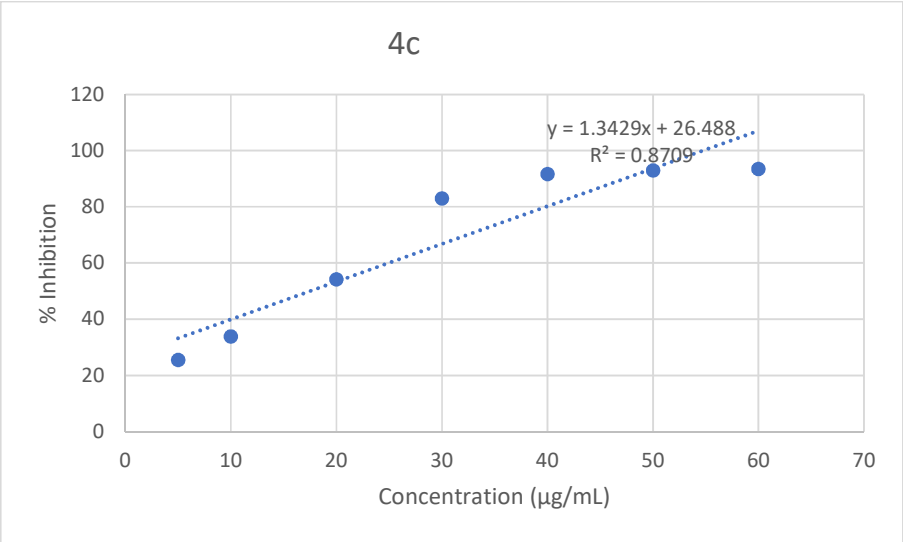


Fig S3: Linear regression curve for the antioxidant activity of compound **4c**

Table S5: Absorbance data for compound **4d** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.74	0.87	14.94253	12.799
10	0.45	0.87	48.27586	
20	0.22	0.87	74.71264	
30	0.082	0.87	90.57471	
40	0.053	0.87	93.90805	
50	0.039	0.87	95.51724	
60	0.016	0.87	98.16092	

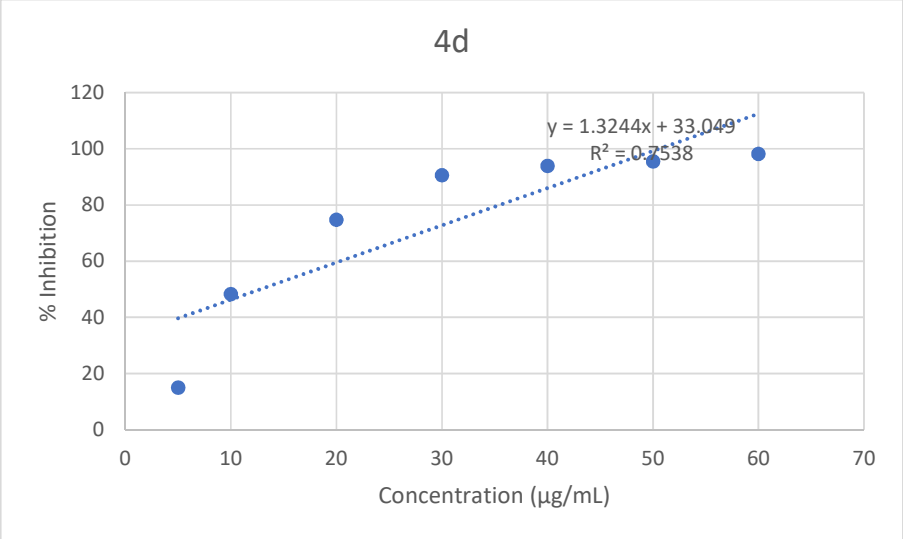


Fig S4: Linear regression curve for the antioxidant activity of compound **4d**

Table S6: Absorbance data for compound **4e** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.679	0.87	21.95402	
10	0.629	0.87	27.70115	26.433
20	0.501	0.87	42.41379	
30	0.378	0.87	56.55172	
40	0.309	0.87	64.48276	
50	0.125	0.87	85.63218	
60	0.088	0.87	89.88506	

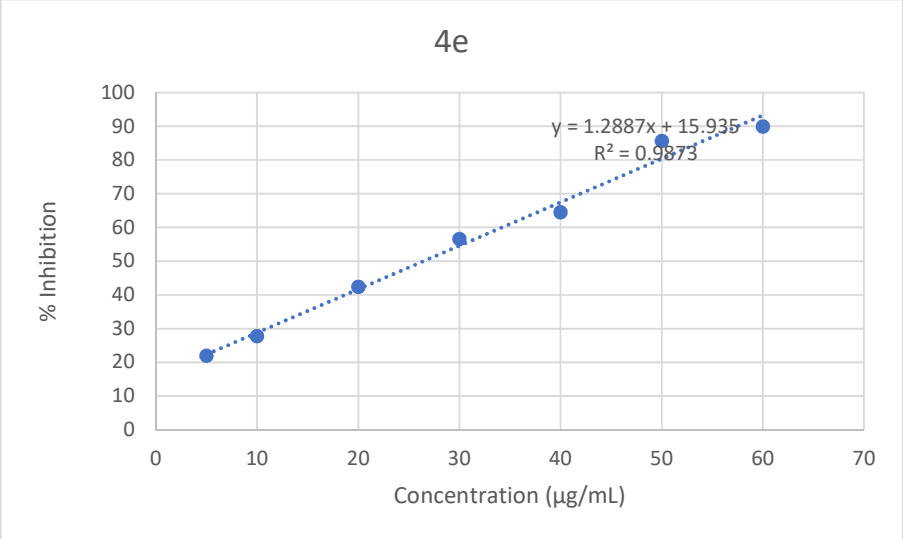


Fig S5: Linear regression curve for the antioxidant activity of compound **4e**

Table S7: Absorbance data for compound **4f** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.807	0.87	7.241379	
10	0.799	0.87	8.16092	124.29
20	0.775	0.87	10.91954	
30	0.744	0.87	14.48276	
40	0.738	0.87	15.17241	
50	0.684	0.87	21.37931	
60	0.613	0.87	29.54023	

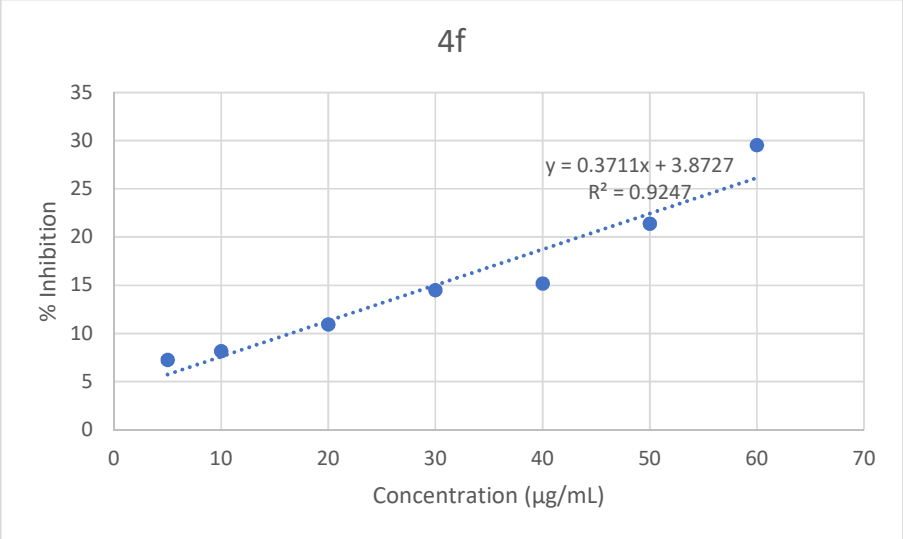


Fig S6: Linear regression curve for the antioxidant activity of compound **4f**

Table S8: Absorbance data for compound **4g** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.551041	0.87	36.662	18.3601
10	0.507294	0.87	41.69038	
20	0.413864	0.87	52.42941	
30	0.370102	0.87	57.45955	
40	0.226746	0.87	73.93729	
50	0.120712	0.87	86.12502	
60	0.051895	0.87	94.03504	

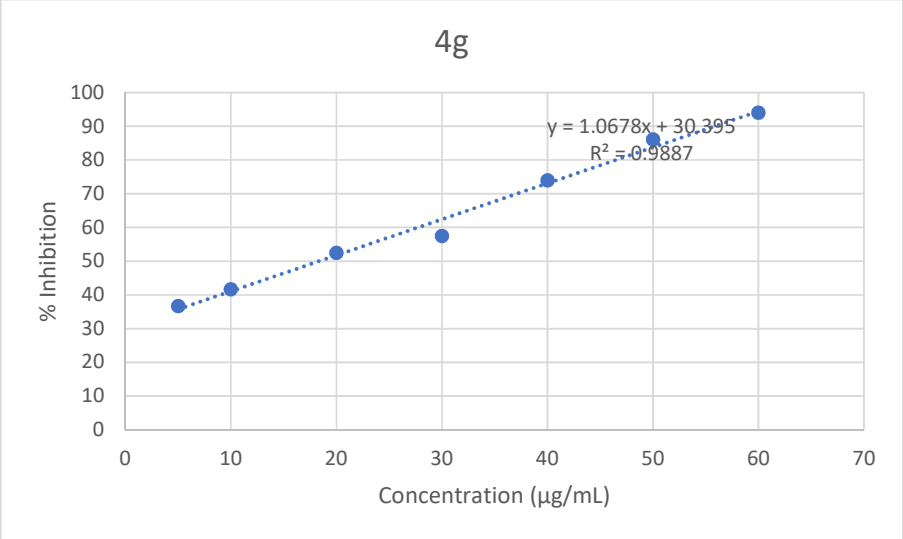


Fig S7: Linear regression curve for the antioxidant activity of compound **4g**

Table S9: Absorbance data for compound **4h** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.612	0.87	29.65517	15.3635
10	0.548	0.87	37.01149	
20	0.326	0.87	62.52874	
30	0.21	0.87	75.86207	
40	0.07	0.87	91.95402	
50	0.061	0.87	92.98851	
60	0.056	0.87	93.56322	

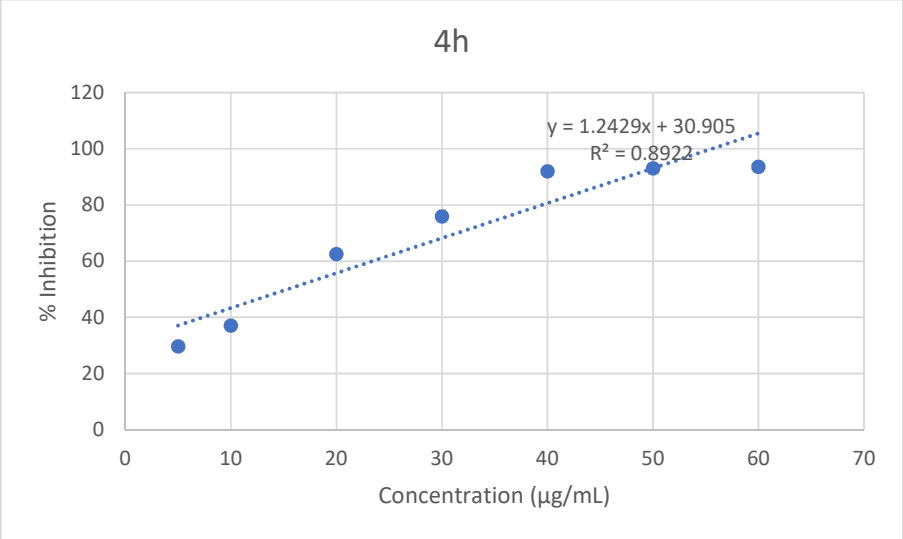


Fig S8: Linear regression curve for the antioxidant activity of compound **4h**

Table S10: Absorbance data for compound **7a** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.59375	0.87	31.75287	37.9411
10	0.579498	0.87	33.391	
20	0.508072	0.87	41.60093	
30	0.497376	0.87	42.8304	
40	0.480972	0.87	44.71583	
50	0.468643	0.87	46.13297	
60	0.194809	0.87	77.60816	

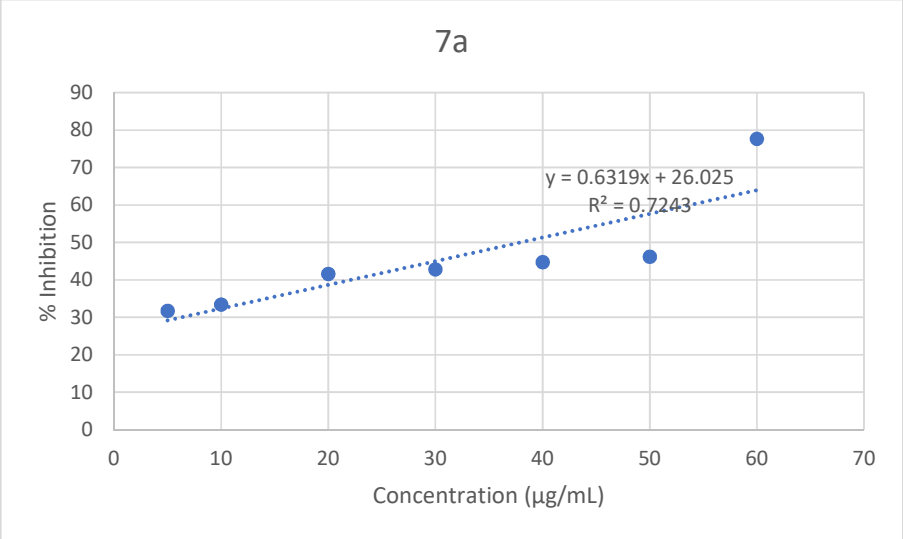


Fig S9: Linear regression curve for the antioxidant activity of compound **7a**

Table S11: Absorbance data for compound **7b** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.612244	0.87	29.62716	83.488
10	0.596649	0.87	31.41963	
20	0.583344	0.87	32.94902	
30	0.557449	0.87	35.92537	
40	0.521973	0.87	40.00314	
50	0.511505	0.87	41.20631	
60	0.490982	0.87	43.56528	

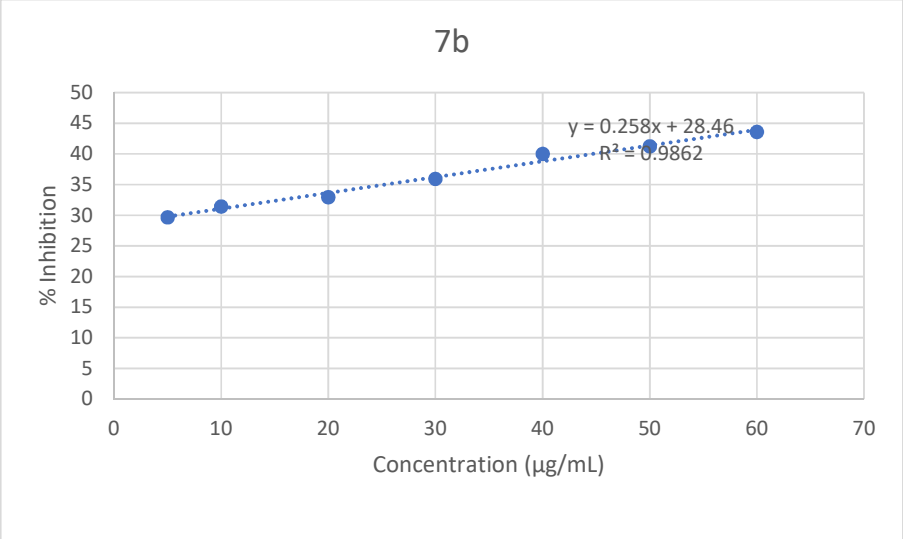


Fig S10: Linear regression curve for the antioxidant activity of compound **7b**

Table S12: Absorbance data for compound **9a** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.847	0.87	2.643678	195.723
10	0.829	0.87	4.712644	
20	0.815	0.87	6.321839	
30	0.786	0.87	9.655172	
40	0.762	0.87	12.41379	
50	0.748	0.87	14.02299	
60	0.729	0.87	16.2069	

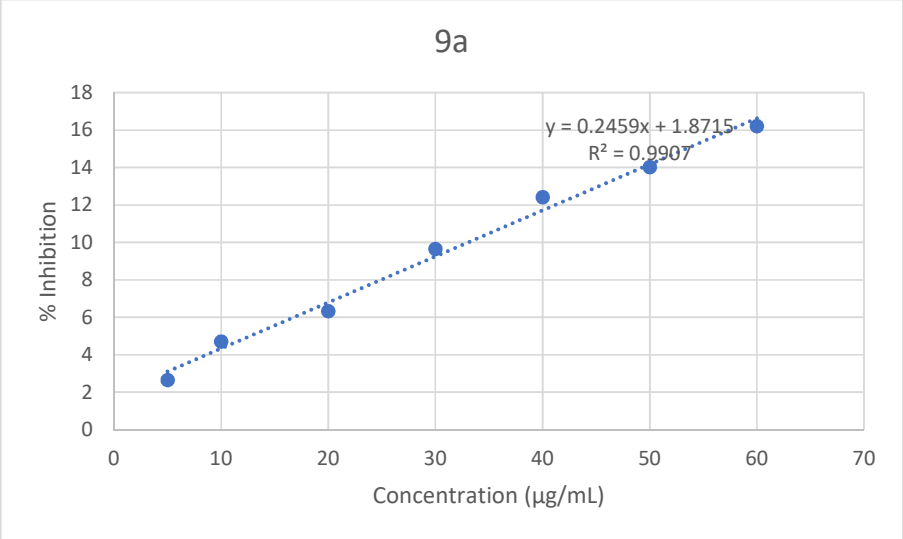


Fig S11: Linear regression curve for the antioxidant activity of compound **9a**

Table S13: Absorbance data for compound **9b** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.836	0.87	3.908046	237.22
10	0.813	0.87	6.551724	
20	0.807	0.87	7.241379	
30	0.798	0.87	8.275862	
40	0.788	0.87	9.425287	
50	0.752	0.87	13.56322	
60	0.731	0.87	15.97701	

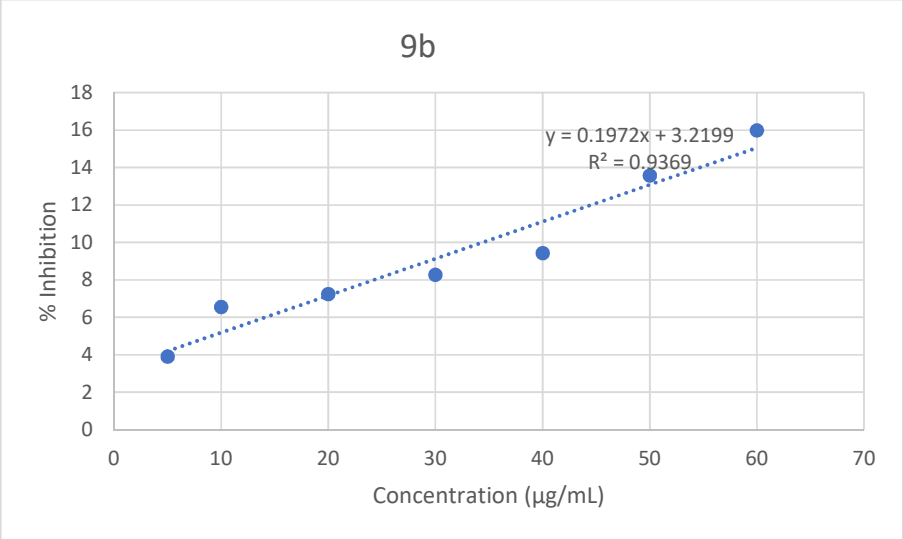


Fig S12: Linear regression curve for the antioxidant activity of compound **9b**

Table S14: Absorbance data for compound **10** in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.856	0.87	1.609195	196.62
10	0.813	0.87	6.551724	
20	0.79	0.87	9.195402	
30	0.787	0.87	9.54023	
40	0.766	0.87	11.95402	
50	0.745	0.87	14.36782	
60	0.721	0.87	17.12644	

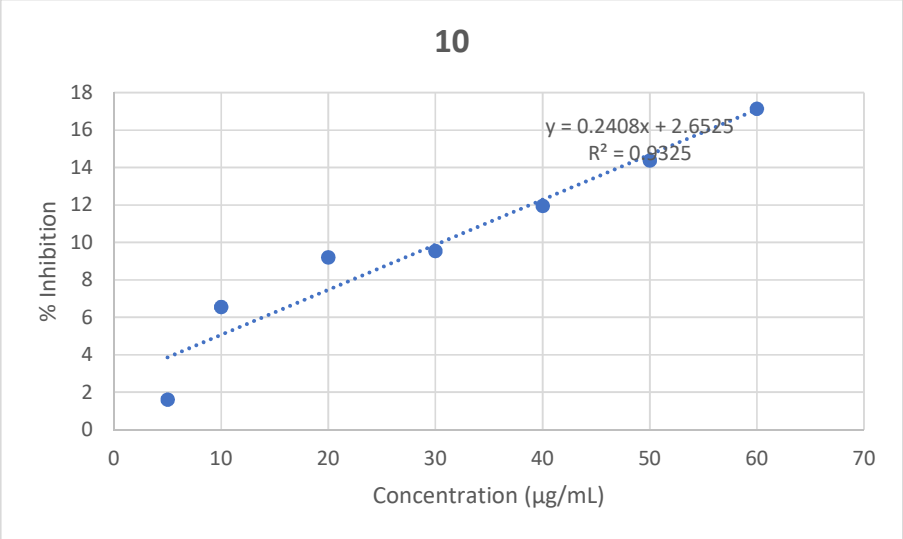


Fig S13: Linear regression curve for the antioxidant activity of compound **10**

Table S15: Absorbance data for Ascorbic Acid in the DPPH assay.

Concentration (µg/mL)	Sample Absorbance	Control Absorbance	% Inhibition	IC50 (µg/mL)
5	0.5879	0.87	32.42529	3.08
10	0.3689	0.87	57.5977	
20	0.1589	0.87	81.73563	
30	0.07954	0.87	90.85747	
40	0.03624	0.87	95.83448	
50	0.03107	0.87	96.42874	
60	0.0211	0.87	97.57471	

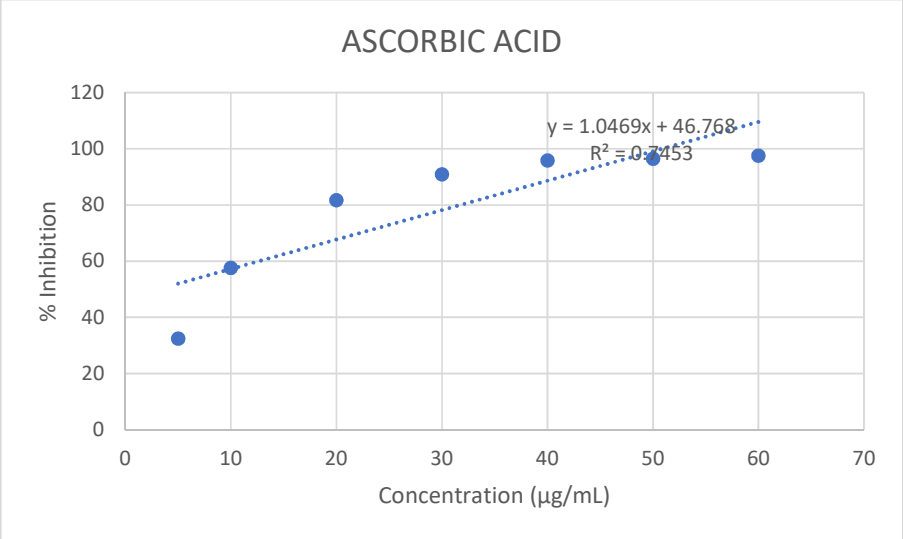
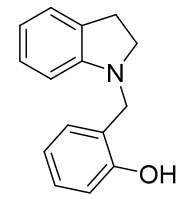
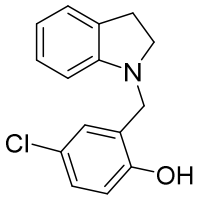
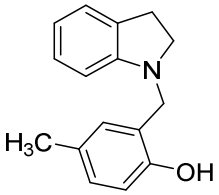
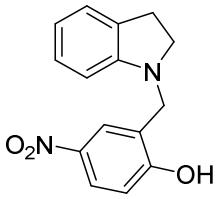
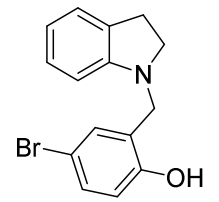
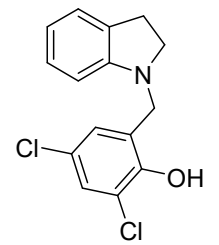
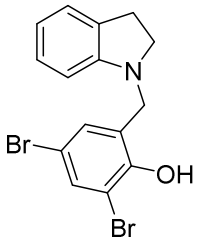
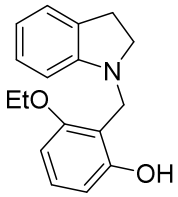
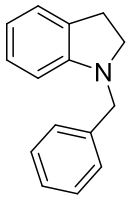
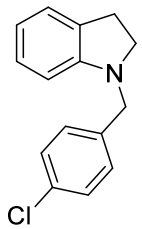


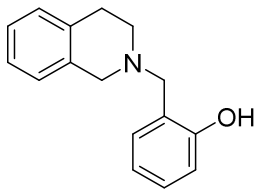
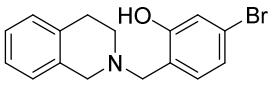
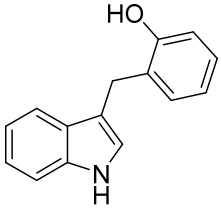
Fig S14: Linear regression curve for the antioxidant activity of Ascorbic Acid

Characterization of the products:

 **4a**	**2-(Indolin-1-ylmethyl)phenol:**[1] Colorless oil, (178 mg, yield-79%); ^{1}H NMR (500 MHz, $CDCl_3$): IR (neat): 3452, 2936, 2857, 1496, 1361, 1092, 750 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 8.96 (bs, 1H), 7.18-7.14 (m, 1H), 7.12-7.11 (m, 1H), 7.08-7.02 (m, 2H), 6.84-6.78 (m, 3H), 6.71-6.69 (m, 1H), 4.23 (s, 2H), 3.17 (t, *J* = 7.9 Hz, 2H), 2.91 (t, *J* = 7.9 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 157.0, 150.9, 131.8, 129.2, 128.6, 127.5, 124.9, 121.6, 121.4, 119.8, 116.3, 111.1, 55.3, 54.9, 28.7; HRMS (ESI) exact mass calculated for $C_{15}H_{15}NO$ $[M+H]^+$: 226.1226; Found: 226.1230.
 **4b**	**4-Chloro-2-(indolin-1-ylmethyl)phenol:**[1] Colorless oil, (197 mg, yield-76%); IR (neat): 3447, 2933, 2863, 1649, 1490, 1343, 1267, 1092, 746 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$): δ 9.12 (bs, 1H), 7.13-7.10 (m, 2H), 7.09-7.06 (m, 1H), 7.02 (m, 1H), 6.85-6.83 (m, 1H), 6.75-6.74 (m, 1H), 6.68-6.67 (m, 1H), 4.18 (s, 2H), 3.15 (t, *J* = 8.1 Hz, 2H), 2.92 (t, *J* = 8.1 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 155.7, 150.8, 131.7, 128.9, 128.1, 127.5, 125.0, 124.3, 123.2, 121.5, 117.6, 111.0, 55.1, 55.0, 28.7; HRMS (ESI) exact mass calculated for $C_{15}H_{14}ClNO$ $[M+H]^+$: 260.0837; Found: 260.0841.

 **4c**	**2-(indolin-1-ylmethyl)-4-methylphenol:** Violet solid, (182 mg, yield-76%); M.P. 123-125 °C; IR (KBr): 3447, 2969, 2843, 1609, 1483, 1345, 932, 760 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 8.81 (bs, 1H), 7.18-7.17 (m, 1H), 7.14-7.11 (m, 1H), 7.03-7.01 (m, 1H), 6.91-6.86 (m, 2H), 6.79-6.74 (m, 2H), 4.25 (s, 2H), 3.21 (t, *J* = 8.0 Hz, 2H), 2.97 (t, *J* = 8.0 Hz, 2H), 2.28 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.6, 151.2, 131.7, 129.5, 129.0, 128.9, 127.4, 124.8, 121.3, 121.1, 110.9, 55.2, 55.0, 28.6, 20.5; HRMS (ESI) exact mass calculated for $C_{16}H_{17}NO$ $[M+H]^+$: 240.1383; Found: 240.1389.
 **4d**	**2-(Indolin-1-ylmethyl)-4-nitrophenol:**[1] Yellow solid, (194 mg, yield-72%); M.P. 131-133 °C; IR (KBr): 3450, 3031, 2922, 2847, 1609, 1510, 1450, 1348, 743 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 10.4 (bs, 1H), 8.17-8.15 (m, 1H), 8.09-8.08 (m, 1H), 7.23-7.22 (m, 1H), 7.18-7.15 (m, 1H), 6.97-6.94 (m, 2H), 6.77-6.75 (m, 1H), 4.38 (s, 2H), 3.27(t, *J* = 8.0 Hz, 2H), 3.04 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.3, 150.2, 140.7, 131.7, 127.6, 125.5, 125.2, 124.5, 122.2, 121.9, 116.8, 111.3, 55.3 (2C), 28.7; HRMS (ESI) exact mass calculated for $C_{15}H_{14}HN_2O_3$ $[M+H]^+$: 271.1077; Found: 271.1075.
 **4e**	**4-Bromo-2-(indolin-1-ylmethyl)phenol:** Red solid, (237 mg, yield-78%); M.P. 135-137 °C; IR (KBr): 3444, 2937, 2841, 1611, 1354, 1235, 806, 744 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 9.12 (bs, 1H), 7.25-7.23 (m, 1H), 7.18-7.16 (m, 1H), 7.13-7.11 (m, 1H), 7.09-7.05 (m, 1H), 6.85-6.82 (m, 1H), 6.71-6.66 (m, 2H), 4.17 (s, 2H), 3.15 (t, *J* = 7.9 Hz, 2H), 2.92 (t, *J* = 7.9 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 156.2, 150.7, 131.8, 131.7, 131.0, 127.5, 125.0, 123.7, 121.6, 118.2, 111.5, 111.0, 55.1, 55.0, 28.7; HRMS (ESI) exact mass calculated for $C_{15}H_{14}BrNO$ $[M+H]^+$: 304.0332; Found: 304.0335.
 **4f**	**2,4-Dichloro-6-(indolin-1-ylmethyl)phenol:** Dark brown Solid, (235 mg, yield-80%); M.P. 129-131 °C; IR (KBr): 3451, 3026, 2933, 2841, 1605, 1321, 1187, 1098, 741 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 9.36 (bs, 1H), 7.32-7.31 (m, 1H), 7.20-7.18 (m, 1H), 7.15-7.12 (m, 1H), 7.05-7.04 (m, 1H), 6.92-6.89 (m, 1H), 6.71-6.69 (m, 1H), 4.26 (s, 2H), 3.26 (t, *J* = 8.0 Hz, 2H), 3.01 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.4, 150.5, 131.5, 128.8, 127.5, 126.7, 125.0, 124.4, 124.3, 121.6 (2C), 110.8, 55.1, 54.7, 28.7; HRMS (ESI) exact mass calculated for $C_{15}H_{13}Cl_2NO$ $[M+H]^+$: 294.0447; Found: 294.0443.

 **4g**	**2,4-Dibromo-6-(indolin-1-ylmethyl)phenol:**[1] Brown oil, (326 mg, yield-85%); IR (neat): 3444, 2938, 2826, 1609, 1334, 1243, 1187, 955, 744 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 9.87 (bs, 1H), 7.61-7.60 (m, 1H), 7.22 (m, 1H), 7.20-7.18 (m, 1H), 7.15-7.12 (m, 1H), 6.92-6.89 (m, 1H), 6.71-6.69 (m, 1H), 4.26 (s, 2H), 3.26 (t, *J* = 8.0 Hz, 2H), 3.01 (t, *J* = 8.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.9, 150.4, 134.3, 131.5, 130.1, 127.5, 125.0, 124.8, 121.7, 111.5, 111.1, 110.9, 55.1, 54.8, 28.7; HRMS (ESI) exact mass calculated for $C_{15}H_{13}Br_2NO$ $[M+H]^+$: 381.9437; Found: 381.9442.
 **4h**	**3-Ethoxy-2-(indolin-1-ylmethyl)phenol:** Light brown solid, (221 mg, yield-82%); M.P. 108-110 °C; IR (KBr): 3446, 3024, 2955, 2842, 1605, 1346, 1236, 1221, 747 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.58 (bs, 1H), 7.12-7.06 (m, 2H), 6.83-6.74 (m, 4H), 6.66-6.64 (m, 1H), 4.29 (s, 2H), 4.10 (q, *J* = 7.0 Hz, 2H), 3.30 (t, *J* = 8.2 Hz, 2H), 2.95 (t, *J* = 8.2 Hz, 2H), 1.45 (t, *J* = 7.0 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 151.8, 146.4, 145.1, 130.8, 127.3, 124.5, 123.0, 120.9, 119.3, 119.2, 111.5, 109.0, 64.4, 54.3, 51.4, 28.6, 14.9; HRMS (ESI) exact mass calculated for $C_{17}H_{19}NO_2$ $[M+H]^+$: 270.1489; Found: 270.1495.
 **7a**	**1-Benzylindoline:**[2] Brown oil, (167 mg, yield-80%); ^1H NMR (500 MHz, $CDCl_3$): IR (neat): 3026, 2908, 2822, 1605, 1368, 742 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.37-7.31 (m, 4H), 7.29-7.25 (m, 1H), 7.10-7.08 (m, 1H), 7.06-7.05 (m, 1H), 6.69-6.65 (m, 1H), 6.51-6.50 (m, 1H), 4.25 (s, 2H), 3.31 (t, *J* = 8.3 Hz, 2H), 2,97 (t, *J* = 8.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.5, 138.5, 130.0, 128.5, 127.9, 127.3, 127.1, 124.5, 117.7, 107.0, 53.6 (2C), 28.5; HRMS (ESI) exact mass calculated for $C_{15}H_{15}N$ $[M+H]^+$: 210.1277; Found: 210.1288.
 **7b**	**1-(4-Chlorobenzyl)indoline:** Dark gummy red, (200 mg, yield-82%); IR (KBr): 3021, 2941, 2831, 1608, 1361, 1188, 747 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.23 (m, 3H), 7.19-7.17 (m, 1H), 7.04-7.02 (m, 1H), 7.00-6.94 (m, 1H), 6.63-6.60 (m, 1H), 6.42-6.40 (m, 1H), 4.14 (s, 2H), 3.23 (t, *J* = 8.4 Hz, 2H), 2.90 (t, *J* = 8.4 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 152.2, 136.9, 132.8, 130.1, 129.2, 128.6, 127.3, 124.6, 118.1, 107.2, 53.6, 53.2, 28.5; HRMS (ESI) exact mass calculated for $C_{15}H_{14}ClN$ $[M+H]^+$: 244.0888; Found: 244.0891.

 **9a**	**2-((3,4-Dihydroisoquinolin-2(1*H*)-yl)methyl)phenol:**[3] White solid, (215 mg, yield-90%); M.P. 155-157 °C; IR (KBr): 3461, 3033, 2941, 2837, 1609, 1340, 981, 743 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 10.75 (bs, 1H), 7.20-7.09 (m, 4H), 7.02-6.97 (m, 2H), 6.84-6.78 (m, 2H), 3.85-3.73 (m, 4H), 2.94-2.83 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.9, 133.5, 133.3, 128.8, 128.6, 126.5, 125.9, 121.2, 119.0, 116.1, 61.1, 55.3, 49.9, 28.6; HRMS (ESI) exact mass calculated for $C_{16}H_{17}NO$ $[M+H]^+$: 240.1383; Found: 240.1387.
 **9b**	**5-Bromo-2-((3,4-dihydroisoquinolin-2(1*H*)-yl)methyl)phenol:** Off white solid, (292 mg, yield-92%); M.P. 166-168 °C; IR (KBr): 3460, 3035, 2946, 2850, 2822, 1607, 1342, 1011, 985, 743 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 11.0 (bs, 1H), 7.29-7.26 (m, 1H), 7.19-7.11 (m, 4H), 7.02-7.00 (m, 1H), 6.72-6.71 (m, 1H), 3.84-3.75 (m, 4H), 2.95-2.86 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.2, 133.4, 133.0, 131.6, 131.2, 128.7, 126.7, 126.6, 126.0, 123.2, 118.1, 110.8, 60.5, 55.3, 49.9, 28.5; HRMS (ESI) exact mass calculated for $C_{16}H_{16}BrNO$ $[M+H]^+$: 318.0488; Found: 318.0483.
 **10**	**2-((1*H*-indol-3-yl)methyl)phenol:**[4] Pale yellow solid, (89 mg, yield-40%); M.P. 141-143 °C; IR (KBr): 3442, 3047, 2962, 2829, 1605, 1022, 743 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$): δ 7.99 (bs, 1H), 7.56-7.54 (m, 1H), 7.35-7.33 (m, 1H), 7.24-7.07 (m, 5H), 6.91-6.87 (m, 2H), 6.82-6.80 (m, 1H), 4.11 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.3, 136.7, 130.5, 127.8, 127.2, 126.4, 122.4 (2C), 120.8, 119.6, 119.1, 115.9, 113.5, 111.2, 26.8; HRMS (ESI) exact mass calculated for $C_{15}H_{13}NO$ $[M+H]^+$: 224.1070; Found: 224.1076.

REFERENCES:

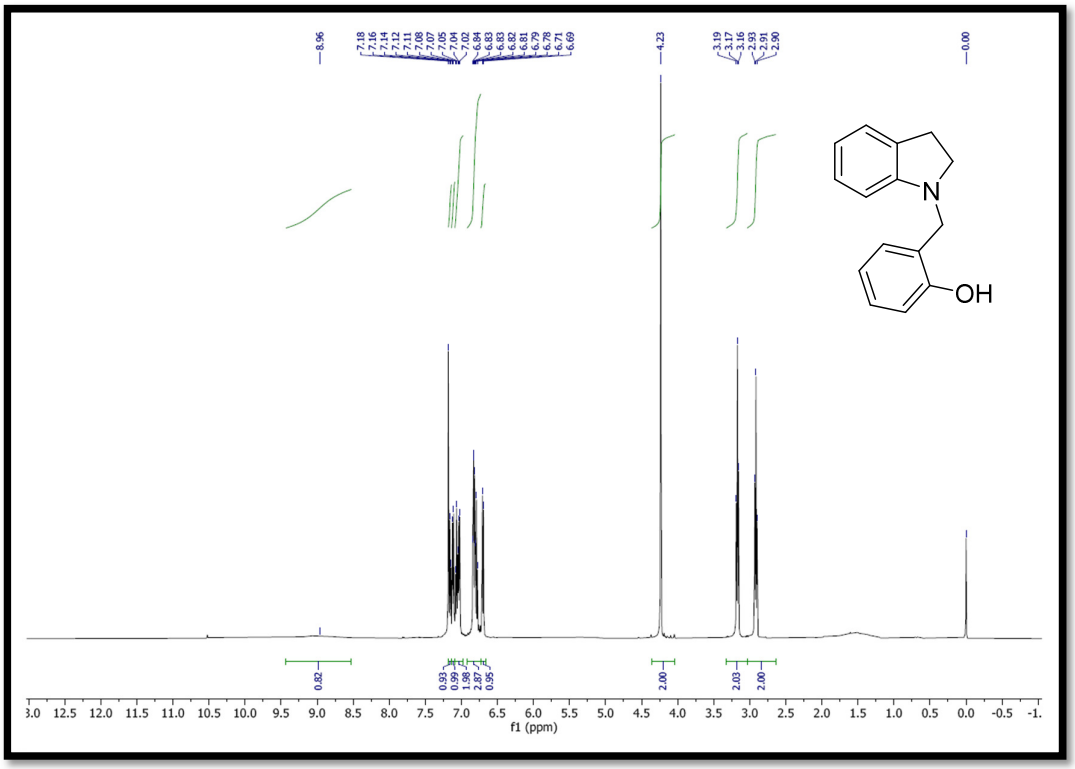
[1] Mao, H.; Xu, R.; Wan, J.; Jiang, Z.; Sun, C.; Pan, Y. *Chem. Eur. J.* **2010**, *16*, 13352-13355.

[2] Maji, M.; Borthakur, I.; Srivastava, S.; Kundu, S. *J. Org. Chem.* **2022**, *87*, 5603–5616.

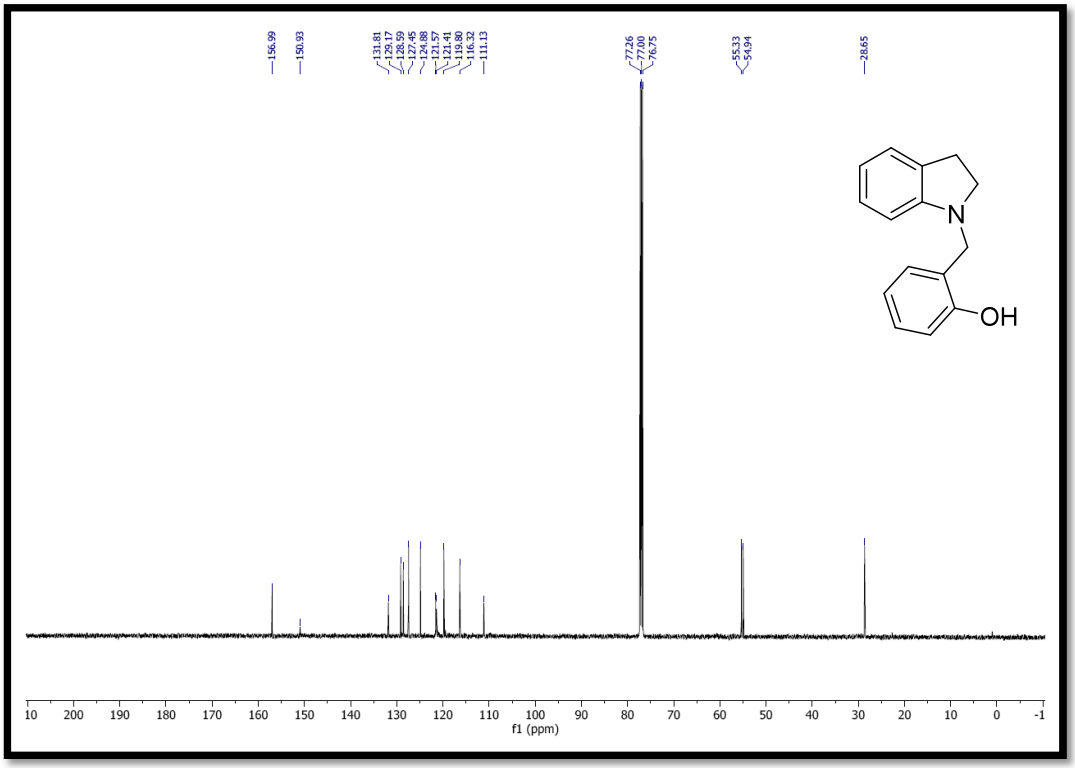
[3] Deb, M. L.; Dey, S. S.; Bento, I.; Barros, M. T.; Maycock, C. D. *Angew. Chem. Int. Ed.* **2013**, *52*, 9791–9795.

[4] Han, L.; Zhang, W.; Shi, X.; You, S. *Adv. Synth. Catal.* **2015**, *357*, 3064–3068.

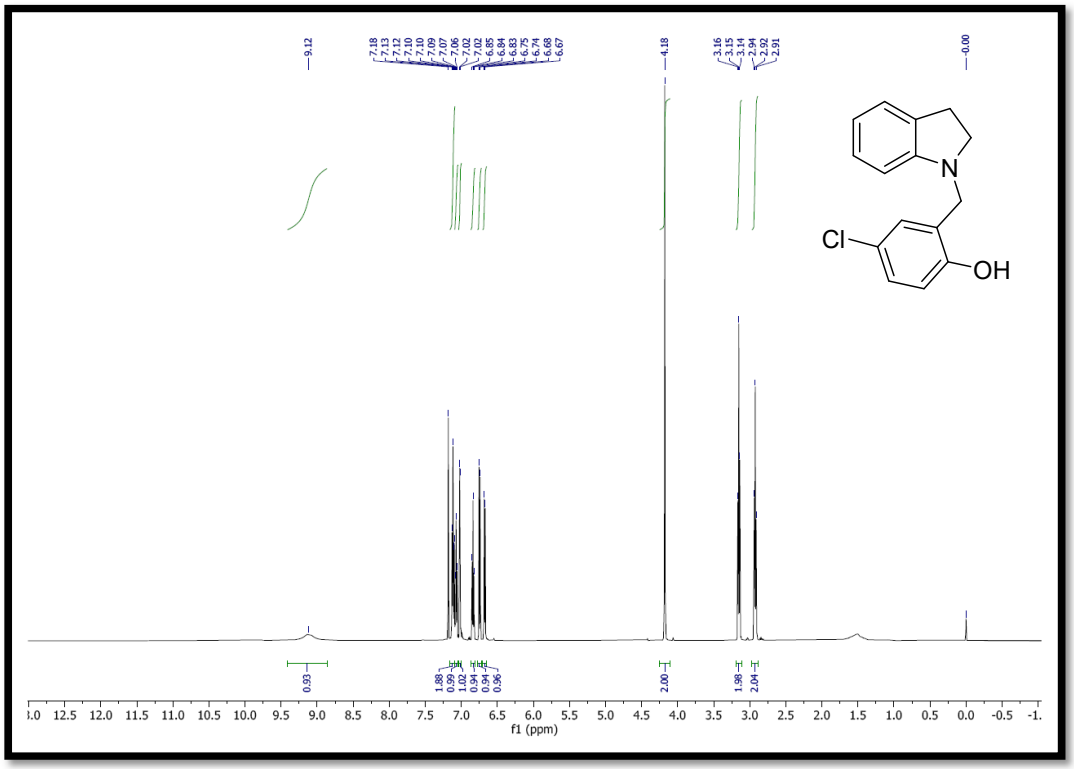
^{1}H-NMR of compound 4a



^{13}C-NMR of compound 4a



^{1}H-NMR of compound 4b



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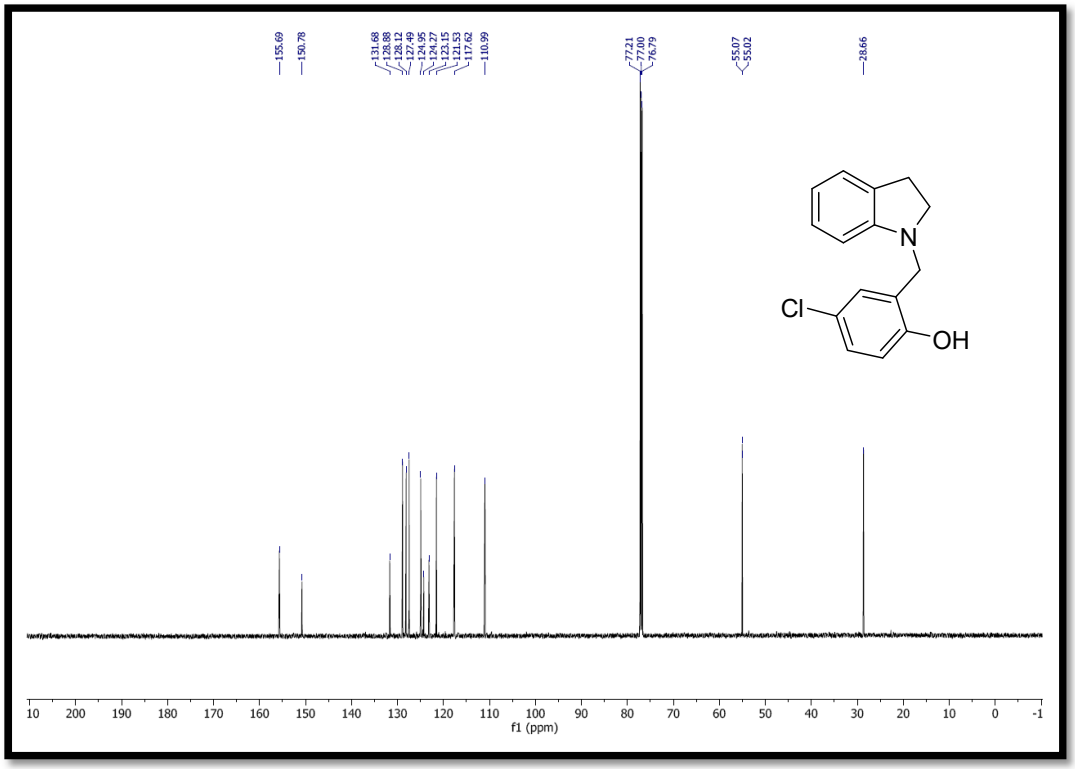

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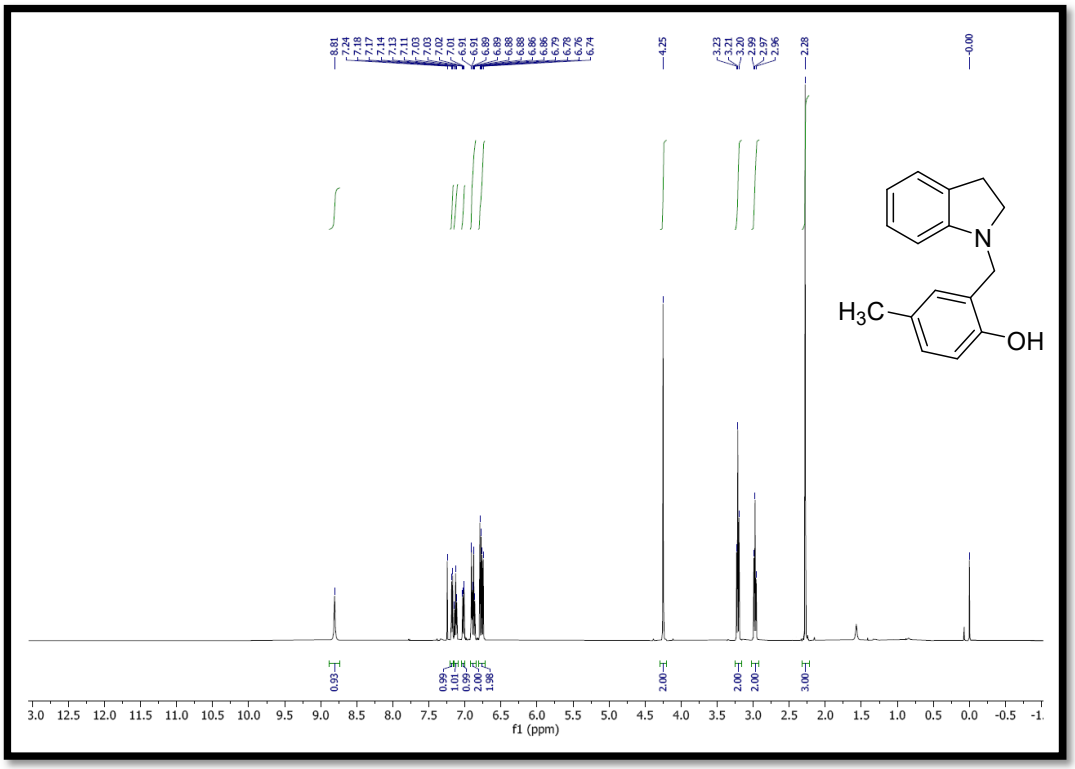

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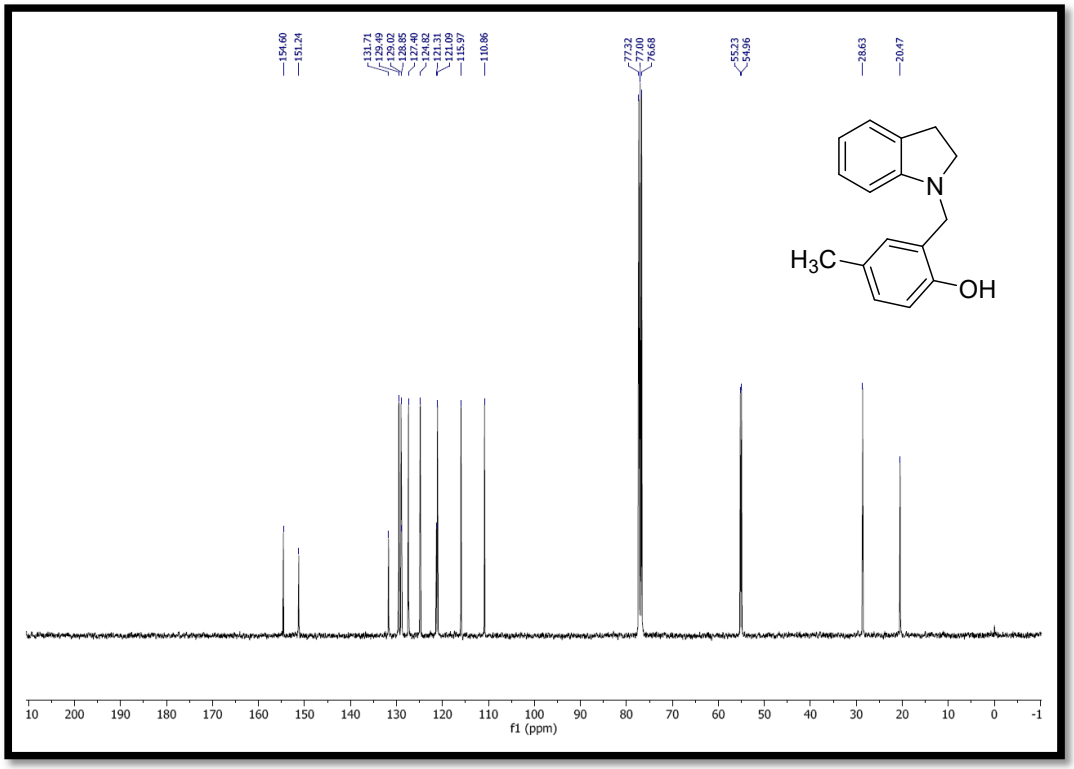

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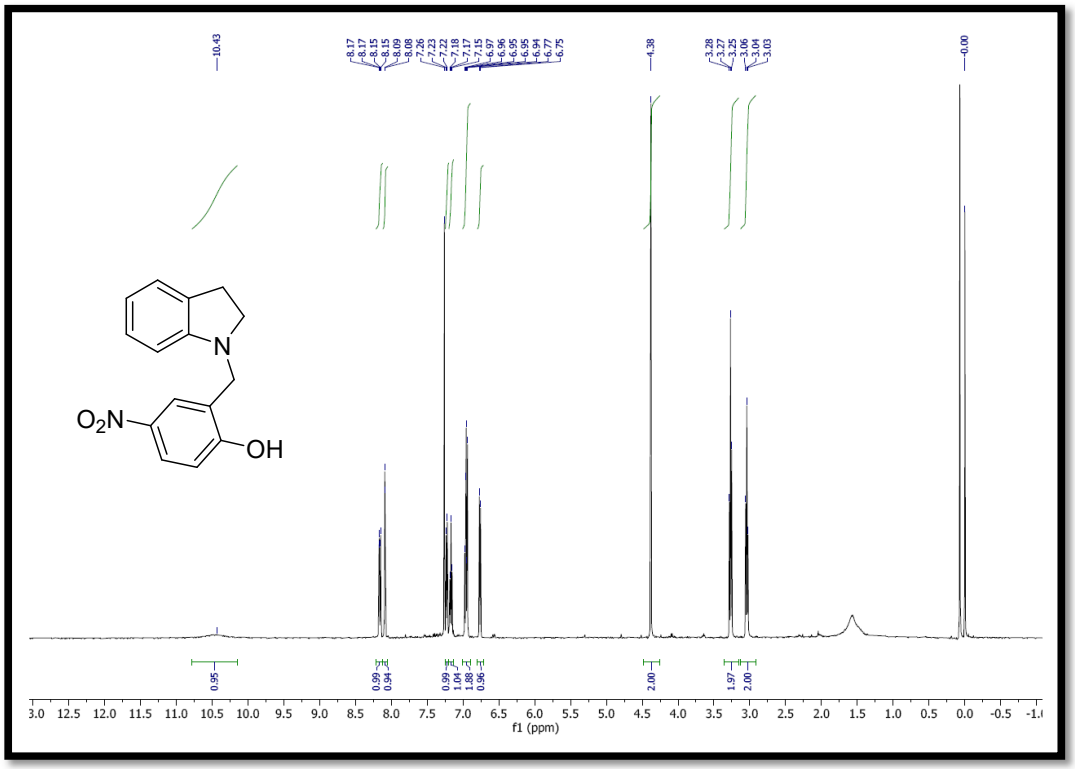

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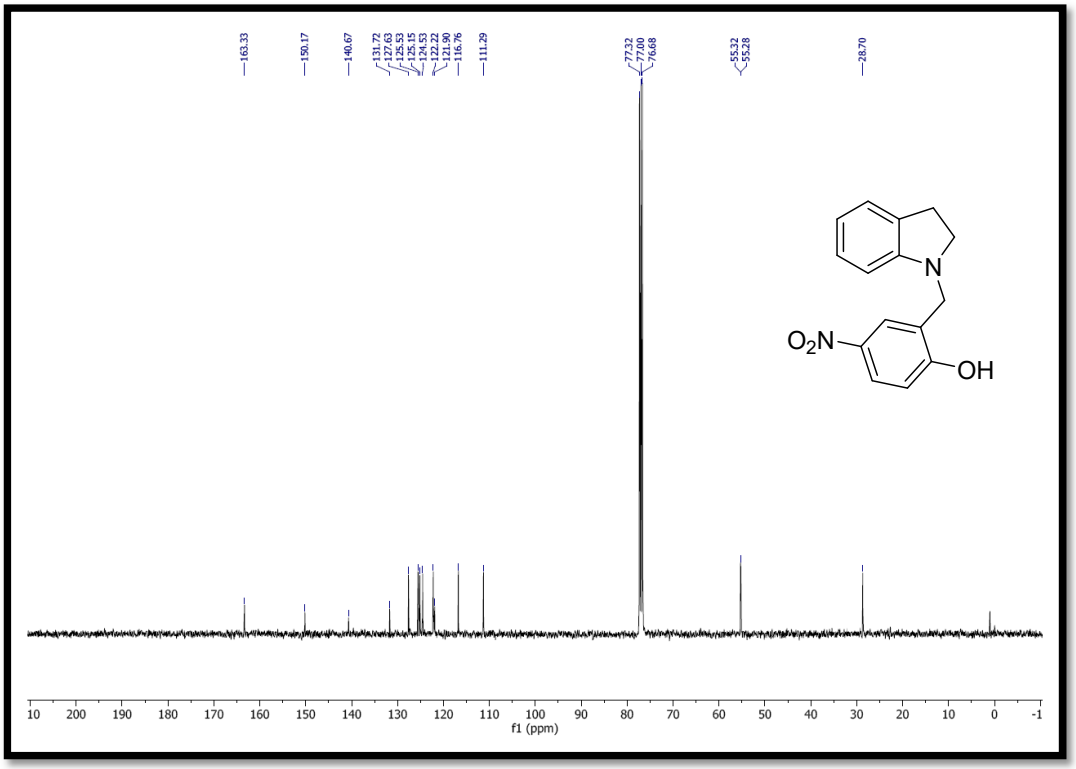

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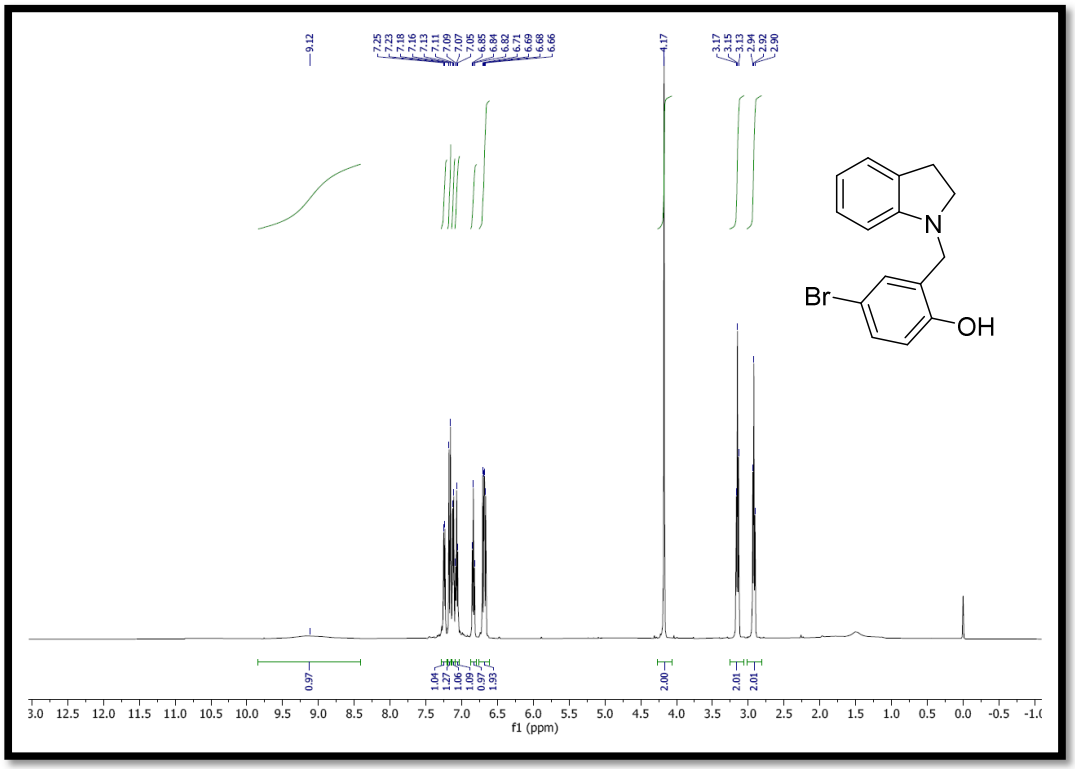

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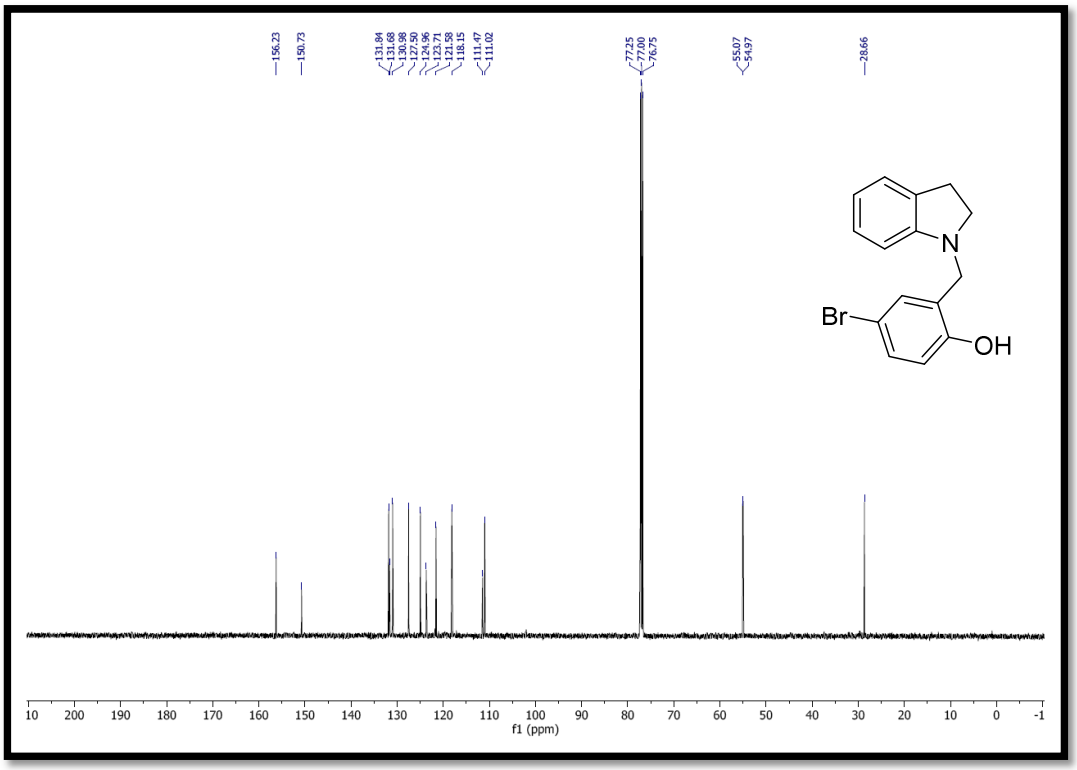

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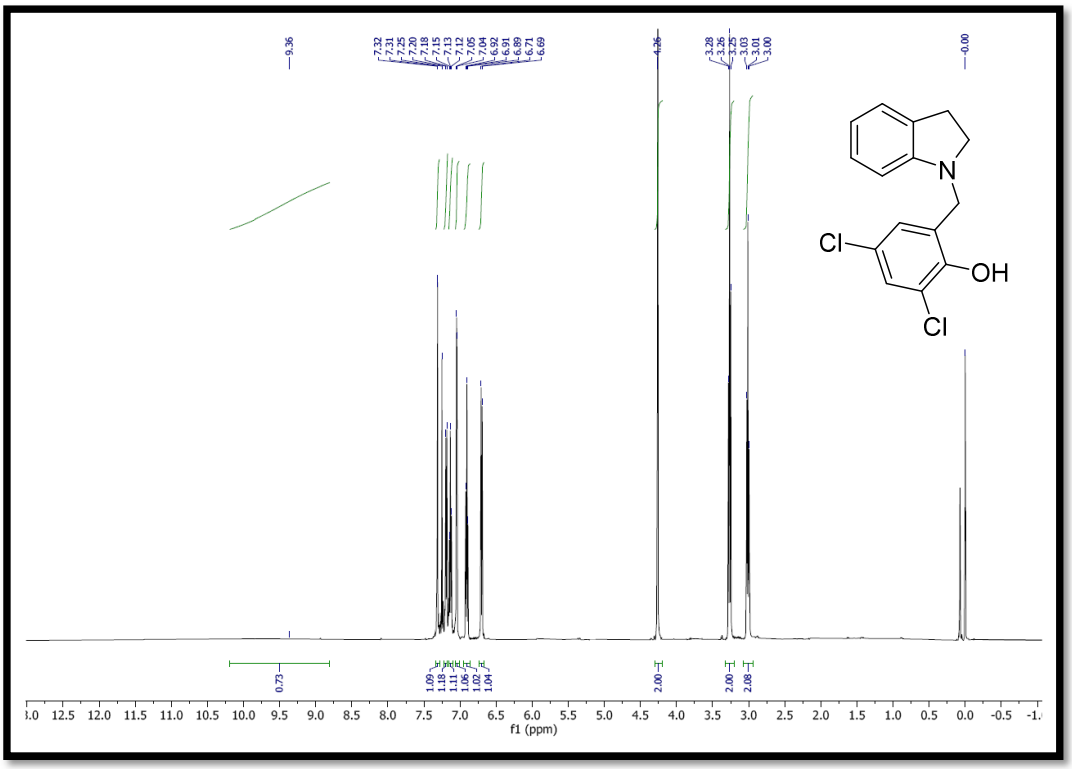

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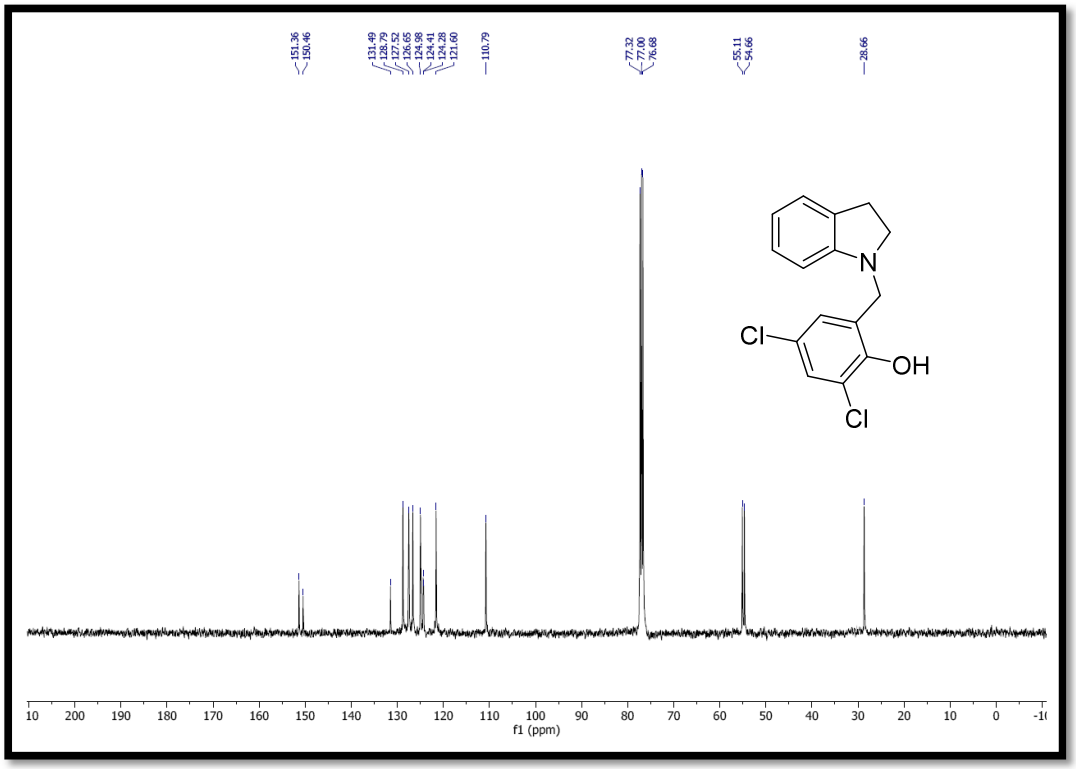

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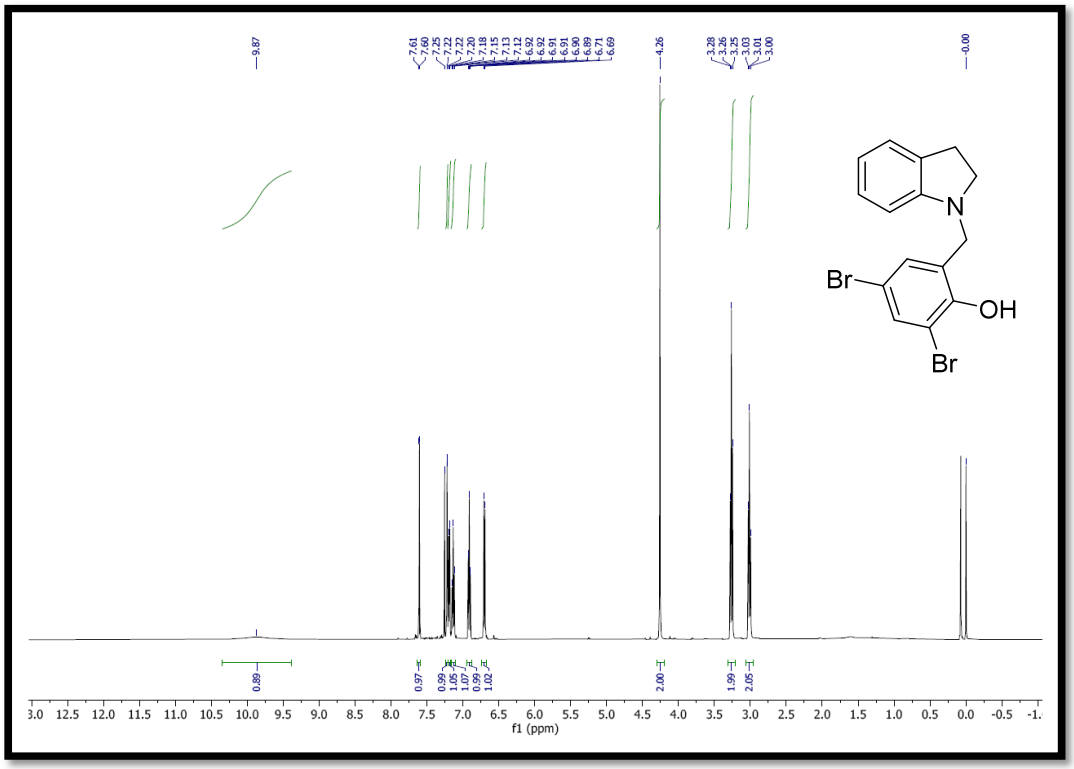

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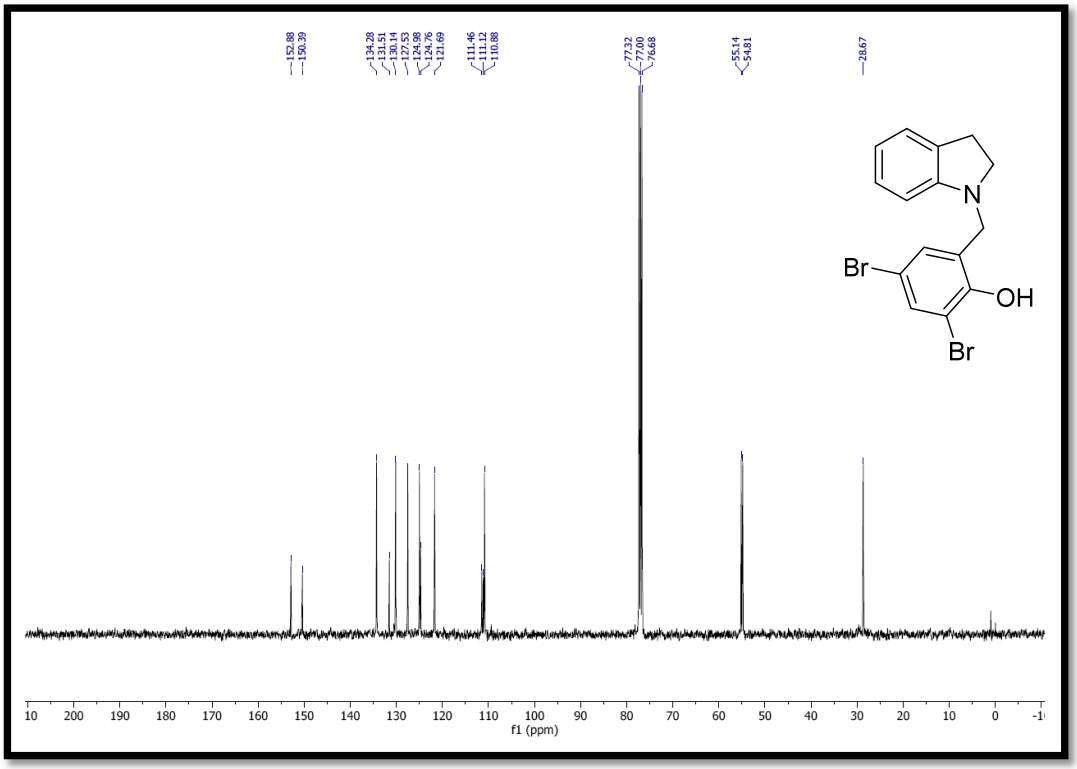

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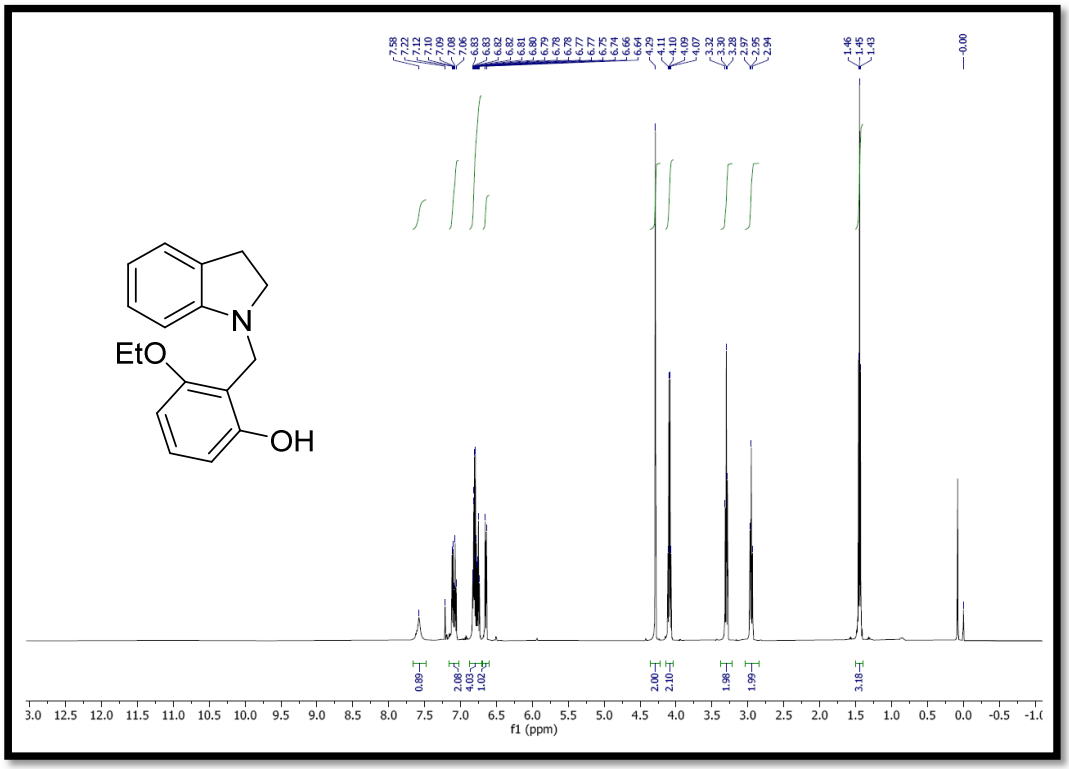

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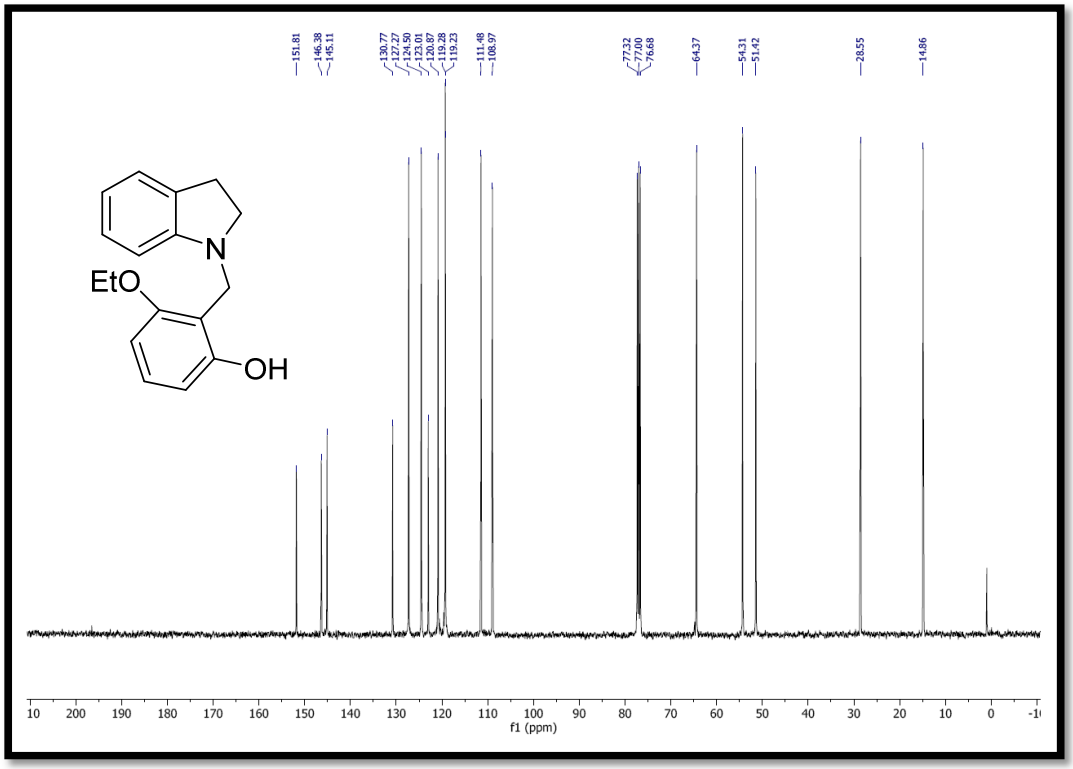
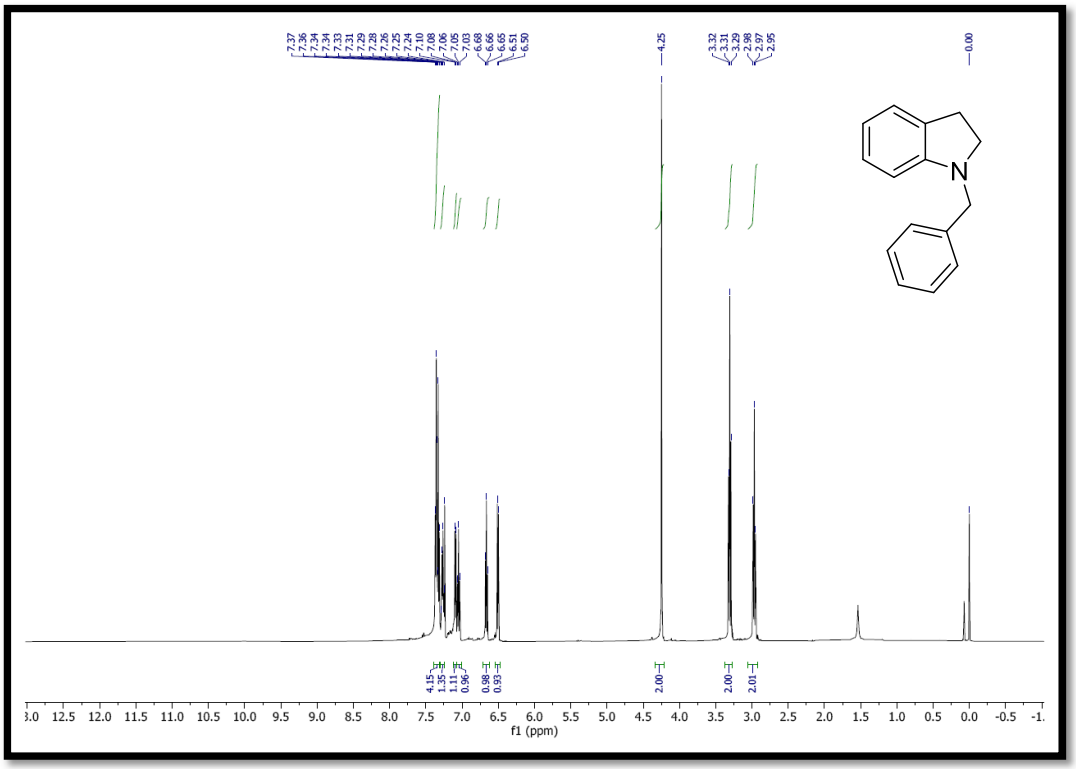


^{1}H-NMR of compound 7a



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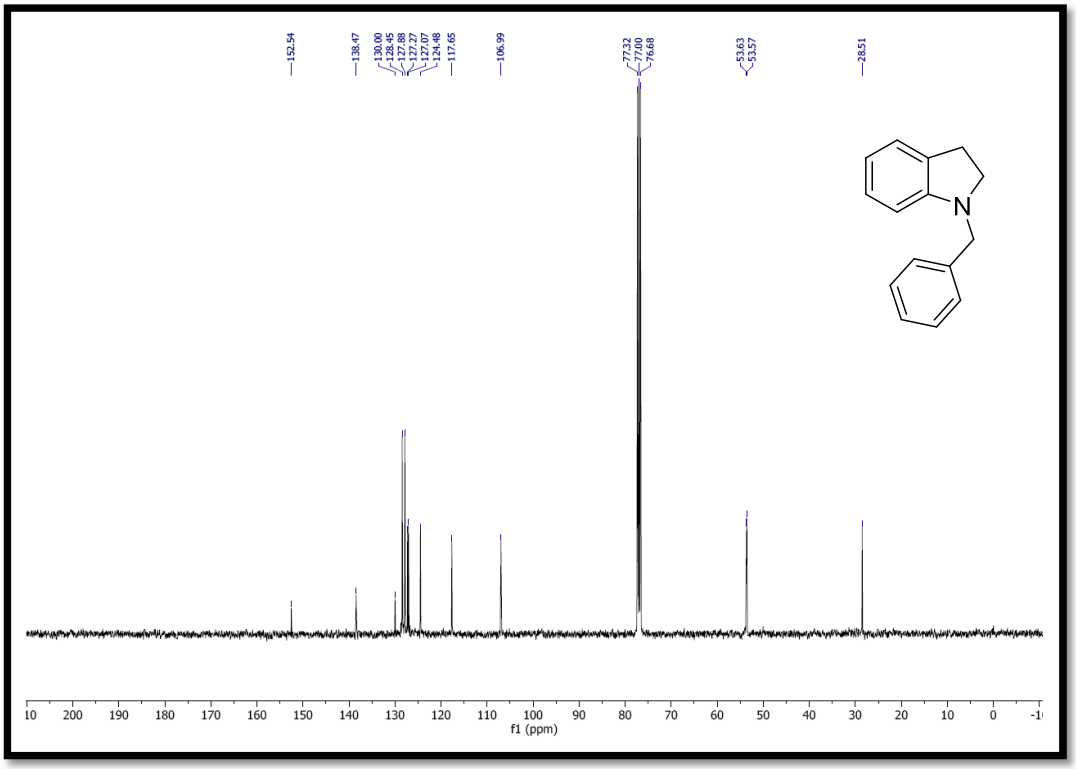

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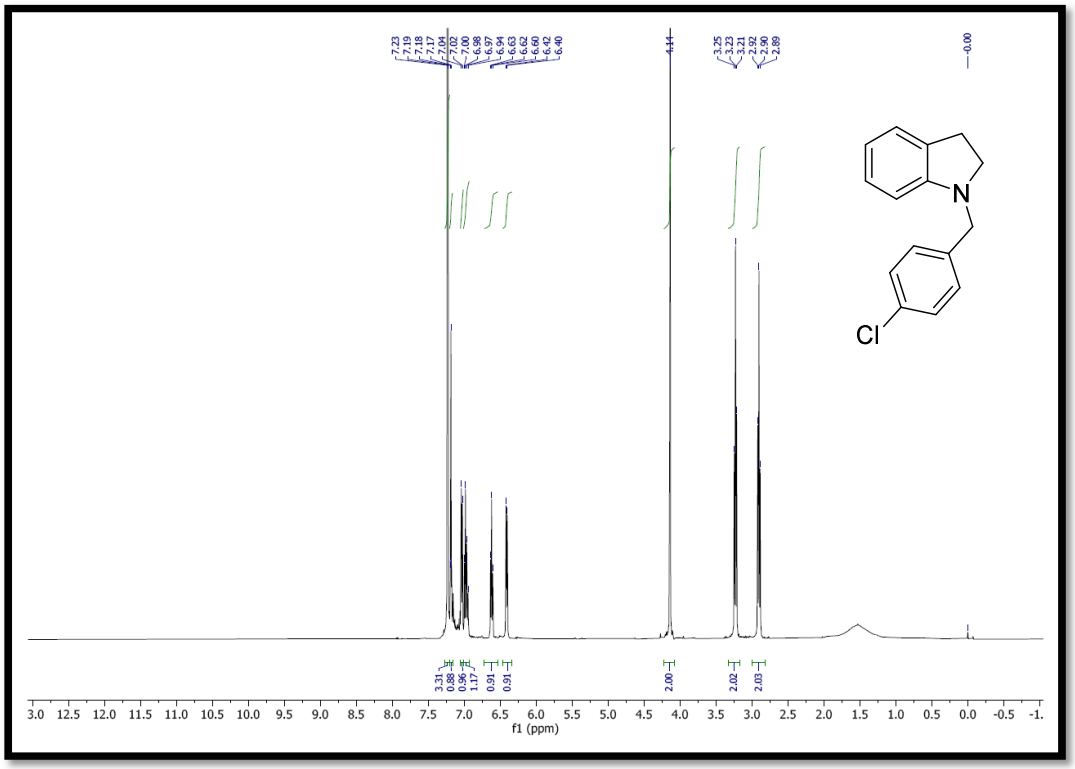

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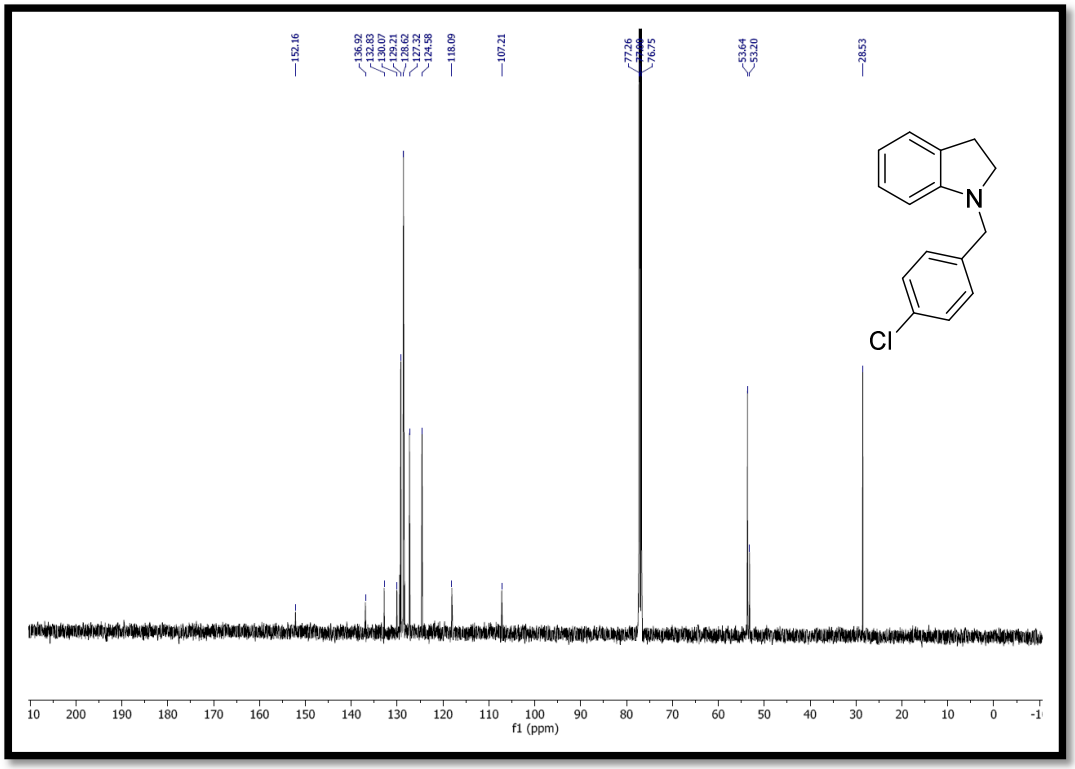

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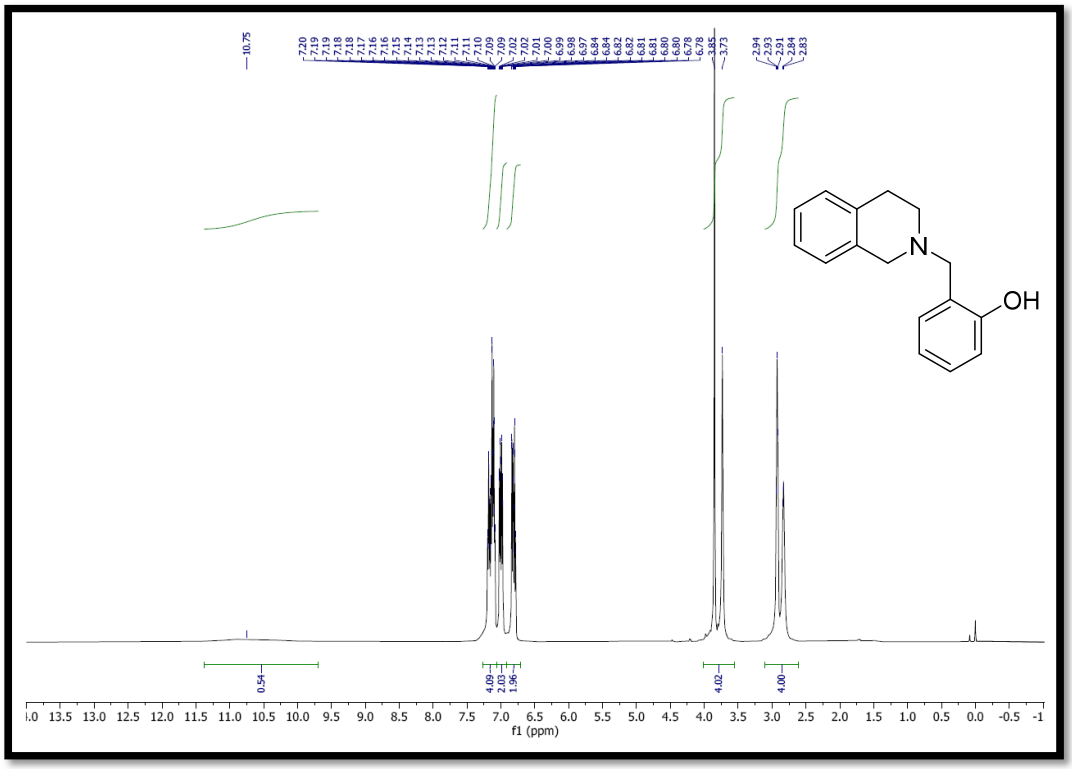
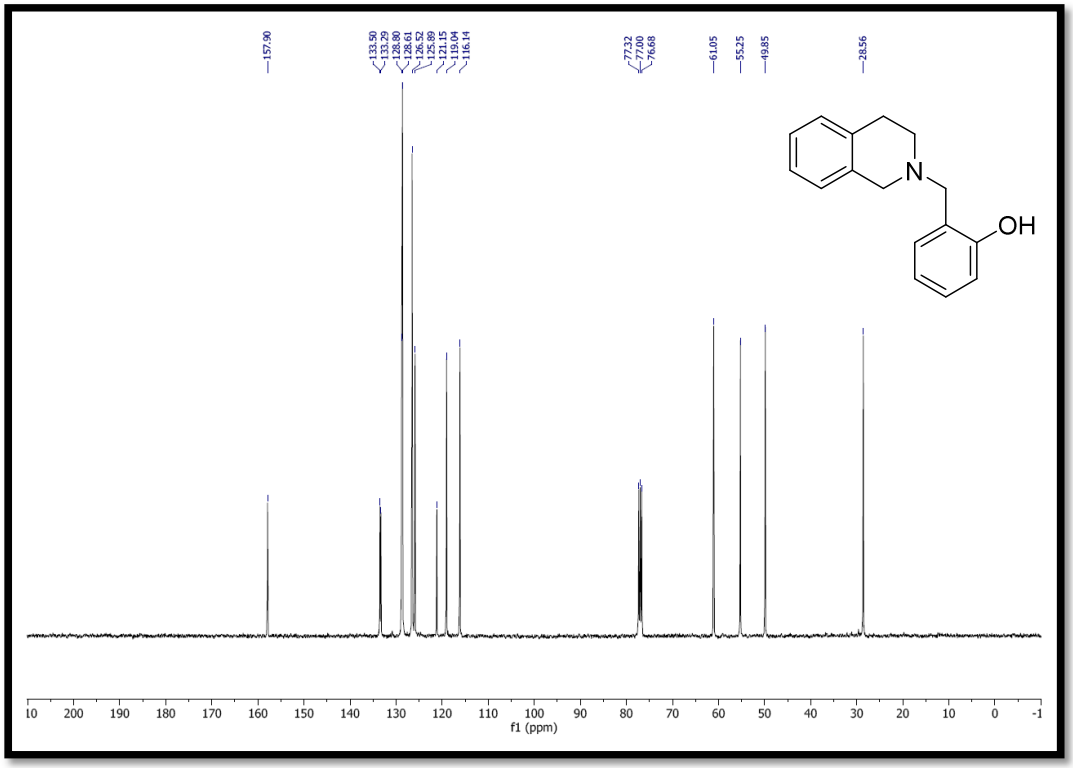
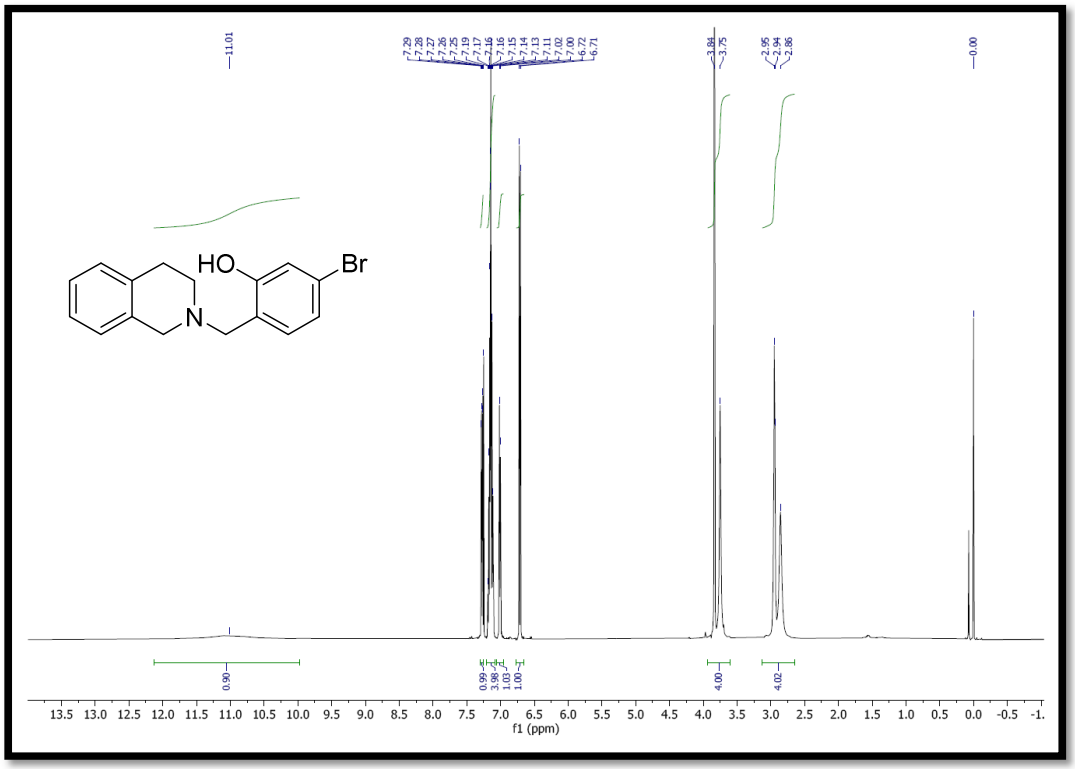


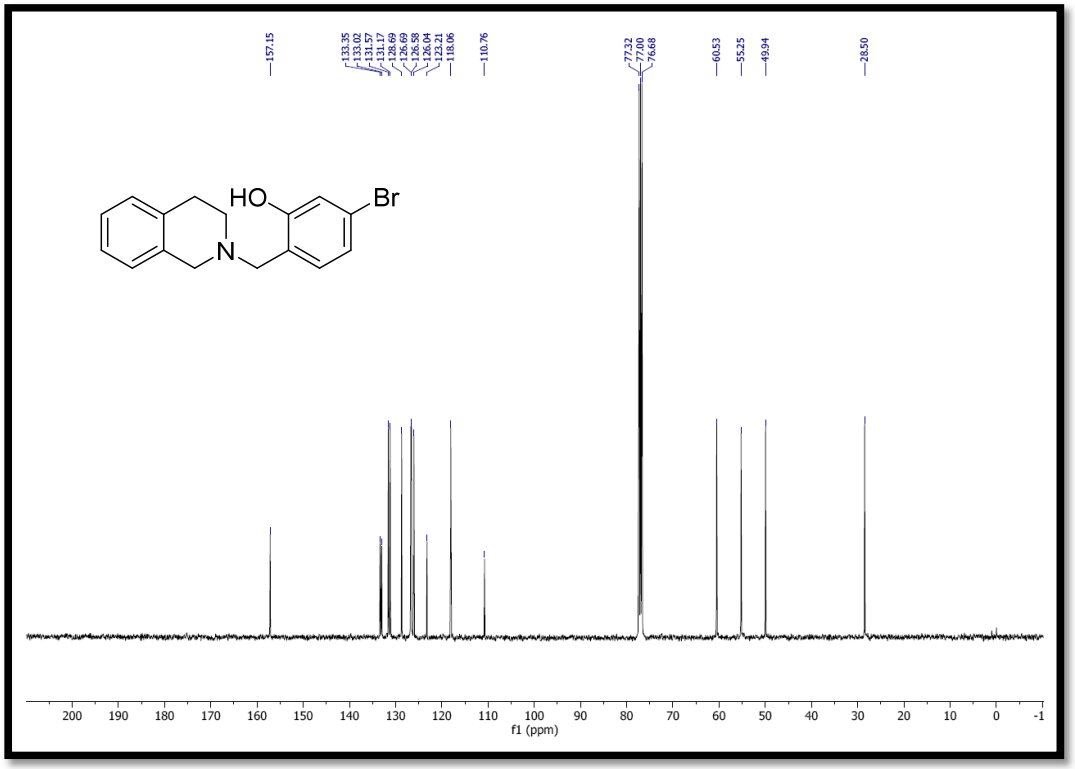
^{13}C-NMR of compound 9a



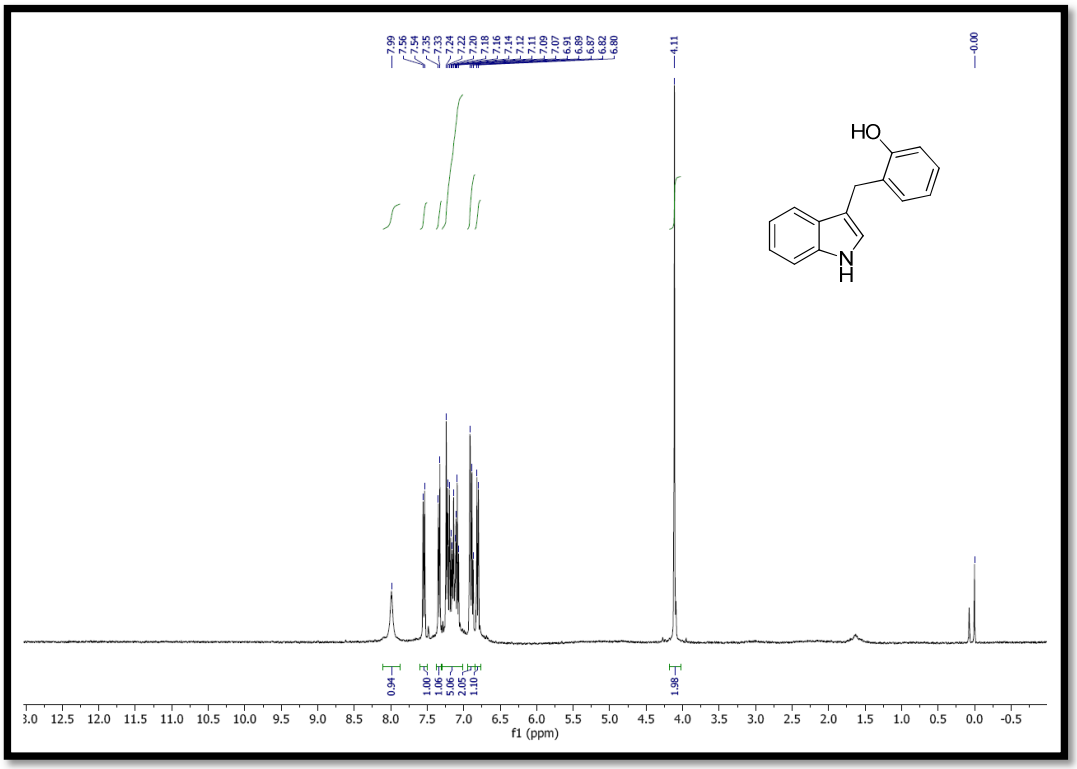
^{1}H-NMR of compound AB-9b



^{13}C-NMR of compound 9b



^{1}H-NMR of compound 10



^{13}C-NMR of compound 10