

Electronic Supplementary Information
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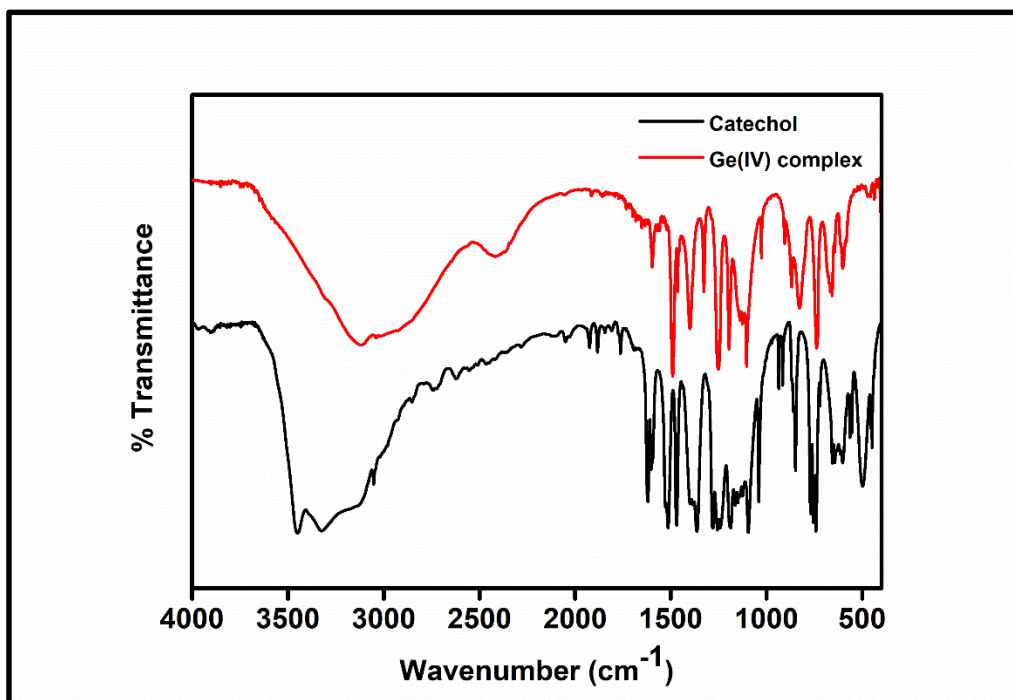


Figure S1: FT-IR spectra of free catechol and complex **1**.

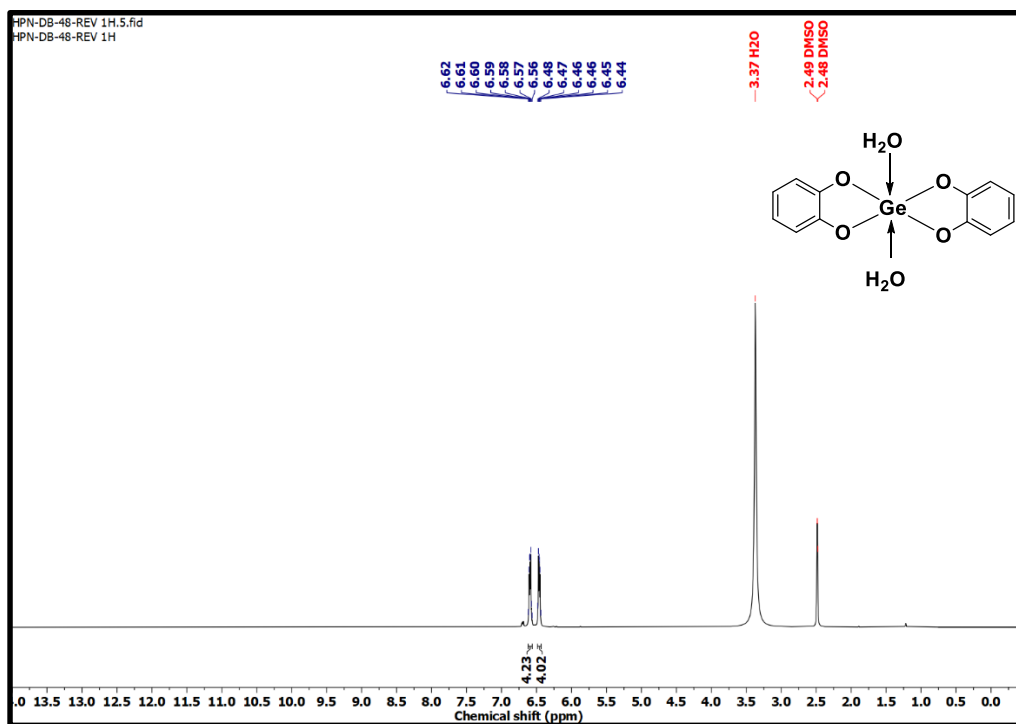


Figure S2: ¹H NMR spectrum of complex **1** recorded in 0.6 mL of DMSO-*d*₆.

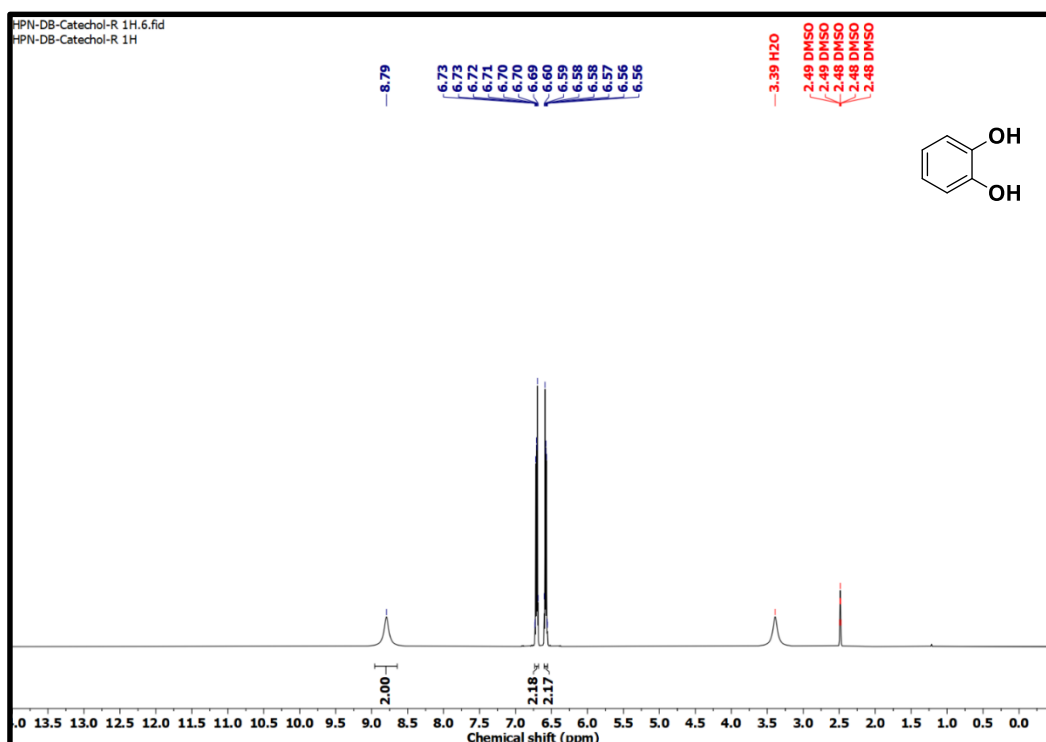


Figure S3: ^1H NMR spectrum of catechol recorded in 0.6 mL of $\text{DMSO-}d_6$.

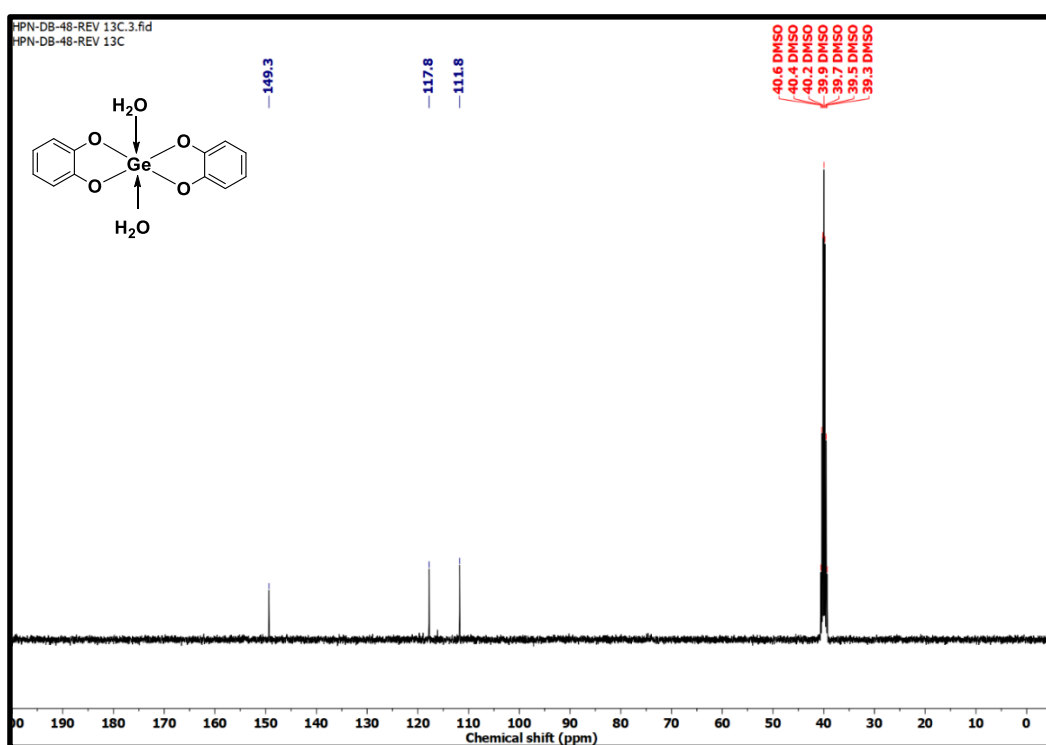


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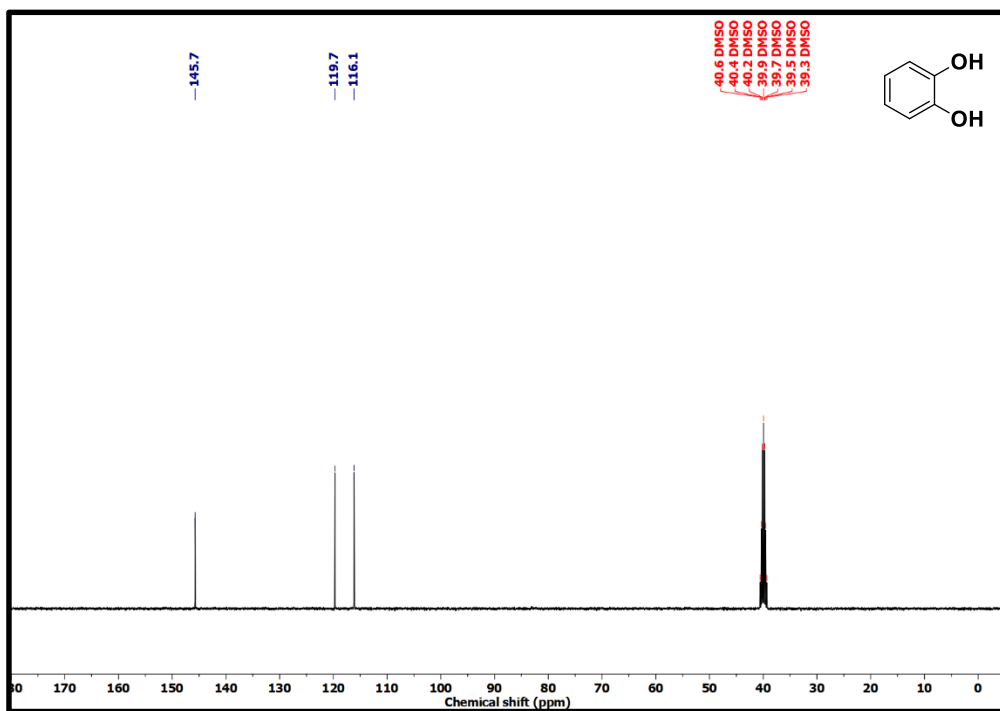
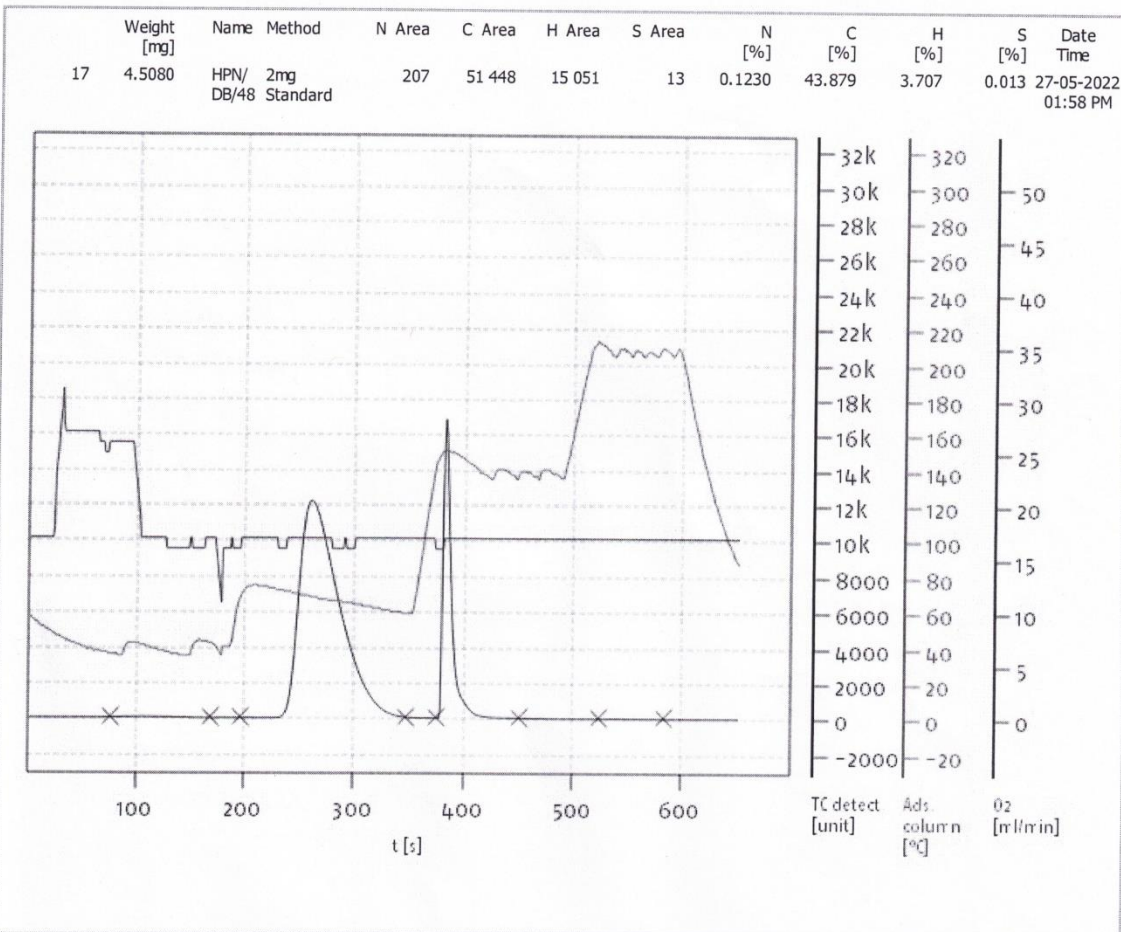


Figure S5: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of catechol recorded in $\text{DMSO-}d_6$.

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Figure S6: Elemental analysis of complex 1.

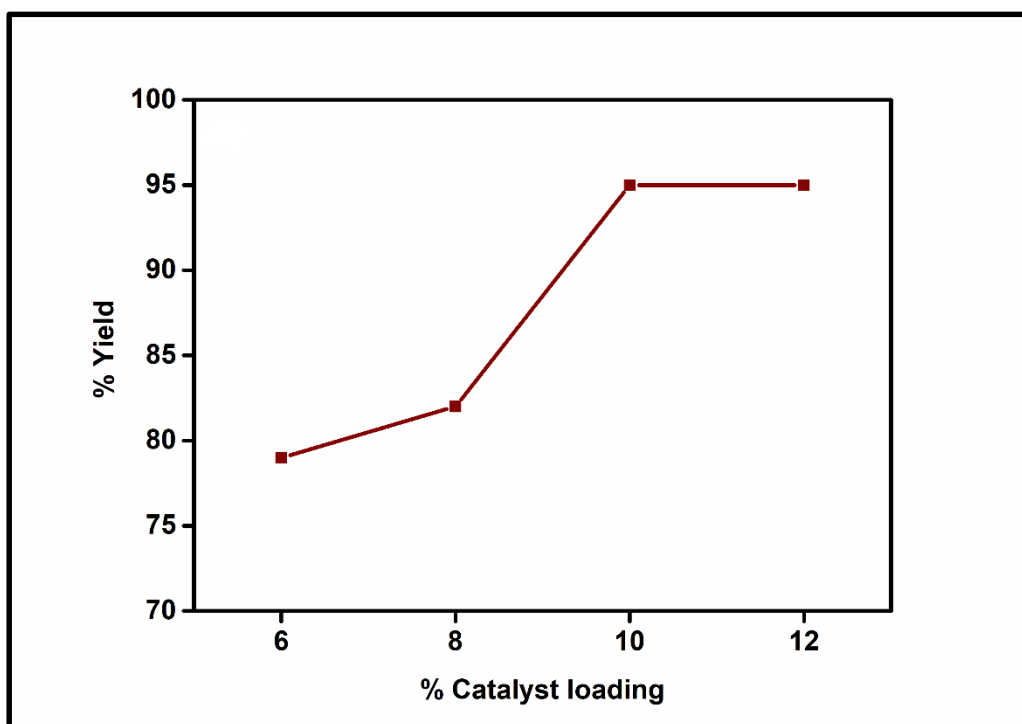


Figure S7: Plot of % yield (isolated) of product vs. catalyst loading for the Friedel-Crafts alkylation reaction between indole and benzaldehyde. Reaction conditions: benzaldehyde (0.24 mmol), indole (0.48 mmol), complex **1** (6-12 mol %). Time: 24 h at room temperature.

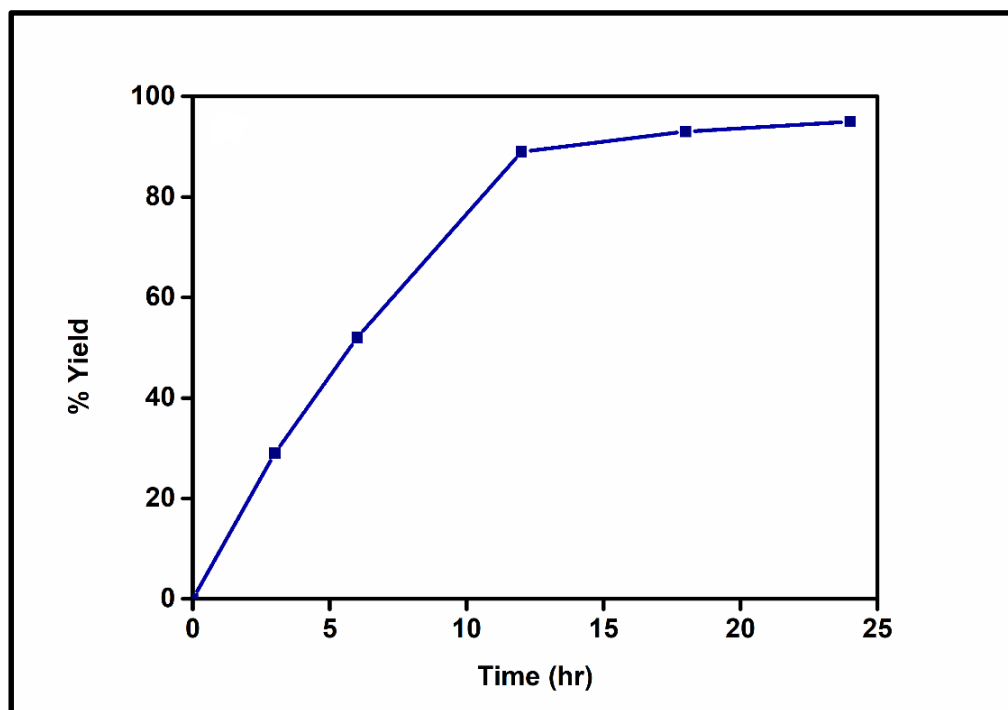


Figure S8: Plot of % yield (isolated) of product vs. time for the Friedel-Crafts alkylation reaction between indole and benzaldehyde. Reaction conditions: benzaldehyde (0.24 mmol), indole (0.48 mmol), complex **1** (10 mol %) at room temperature.

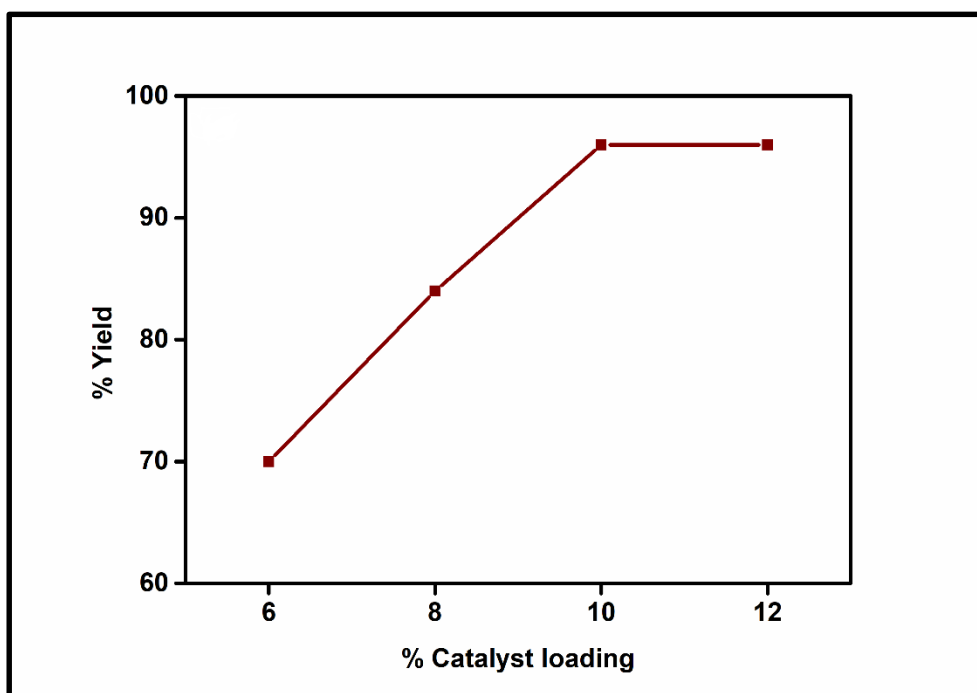


Figure S9: Plot of % yield (isolated) of product vs. catalyst loading for the Friedel-Crafts alkylation reaction between indole and β -nitrostyrene. Reaction condition: β -nitrostyrene (0.40 mmol), indole (0.40 mmol), complex **1** (6-12 mol %). Time: 24 h at rt.

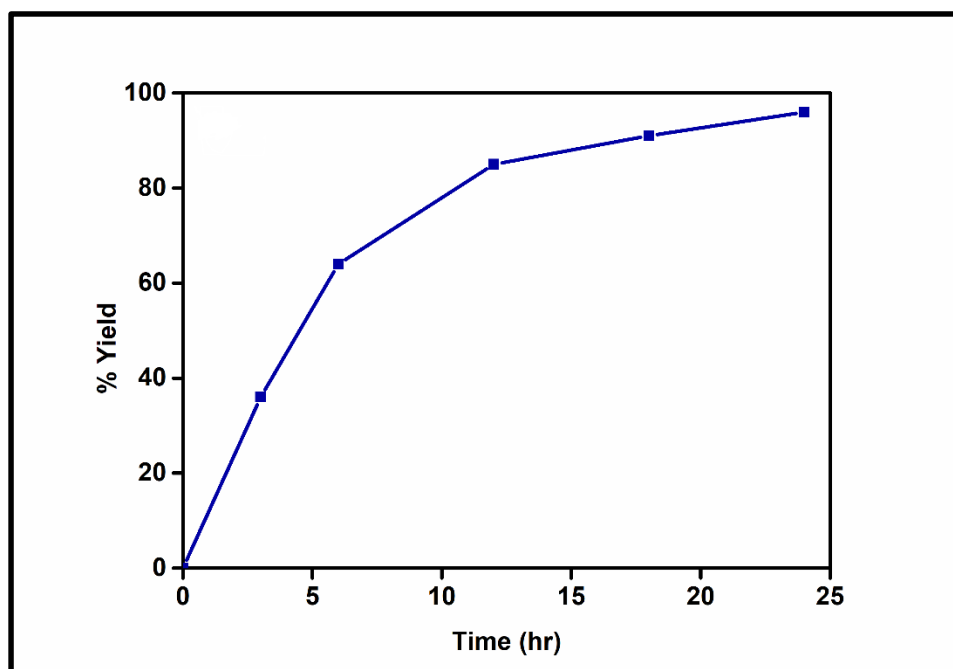
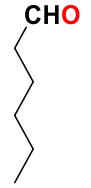
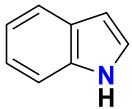
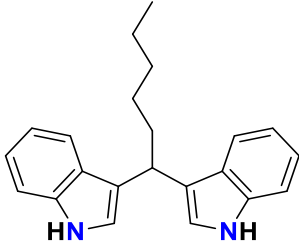
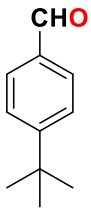
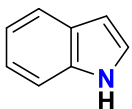
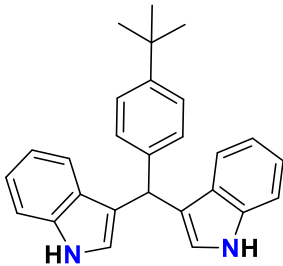
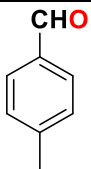
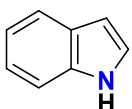
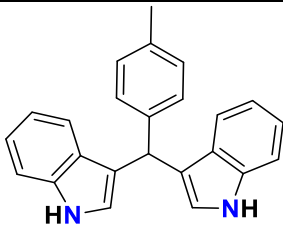
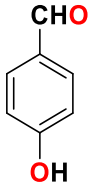
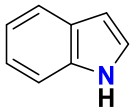
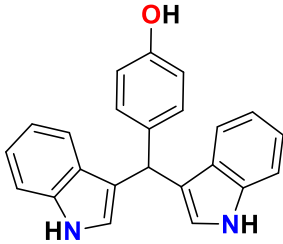
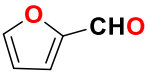
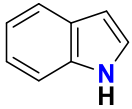
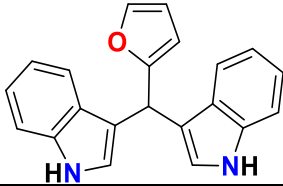
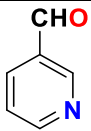
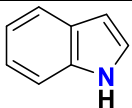
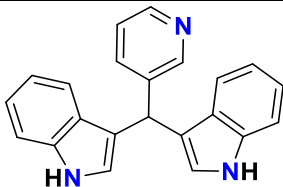
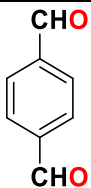
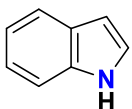
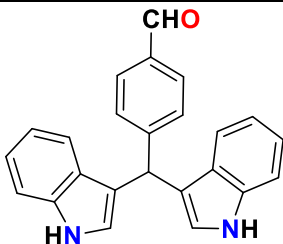


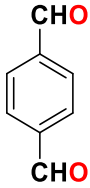
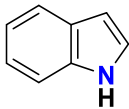
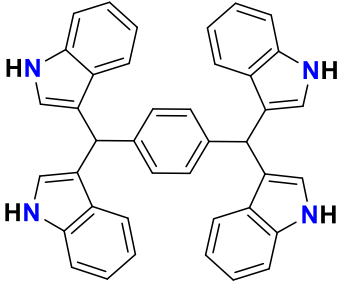
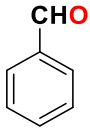
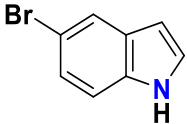
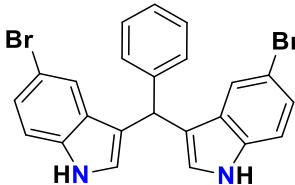
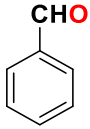
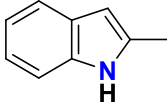
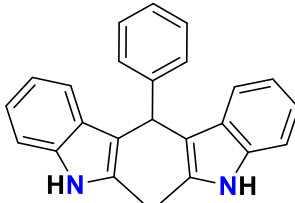
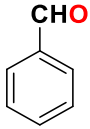
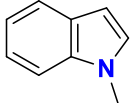
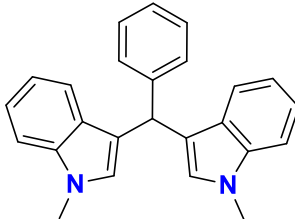
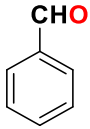
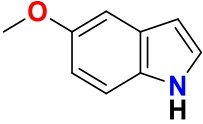
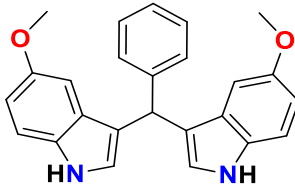
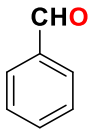
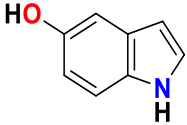
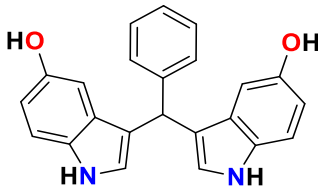
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Table S1: Isolated yields and characterizations of Friedel-Crafts alkylation reaction of various aldehydes and indoles.

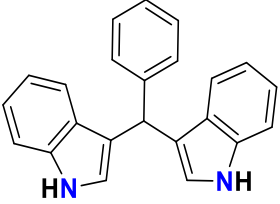
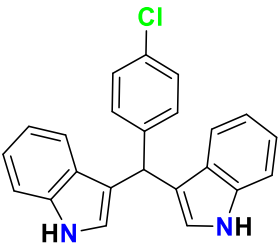
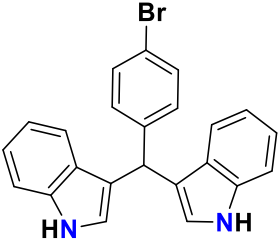
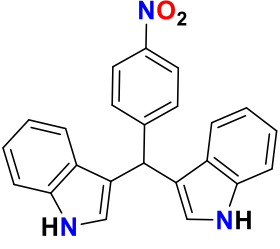
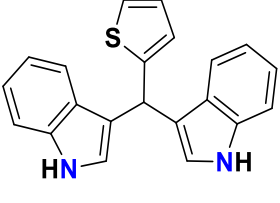
Reaction scheme: $R_1\text{-CHO} + \text{Indole}(R_2) \xrightarrow[\text{H}_2\text{O, rt, 24h}]{\text{Catalyst (10 mol\%)}} \text{Bi-Indole}(R_1, R_2)$

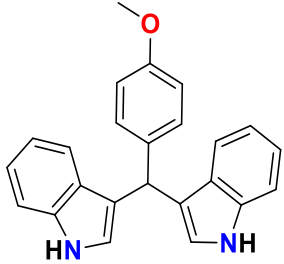
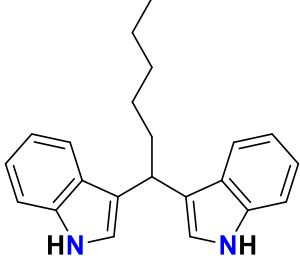
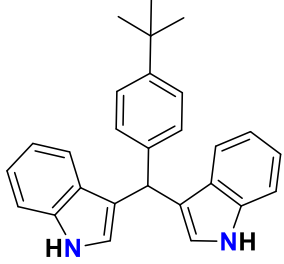
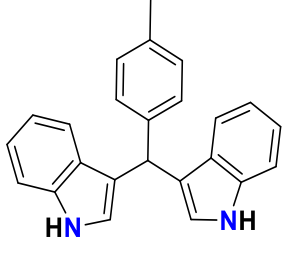
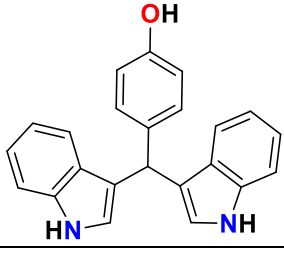
Entry	Aldehydes	Indoles	Product	Code	Yield (%)
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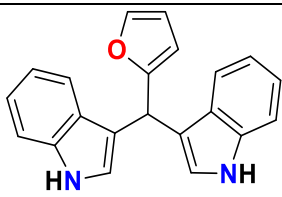
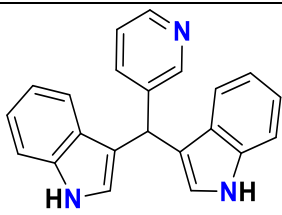
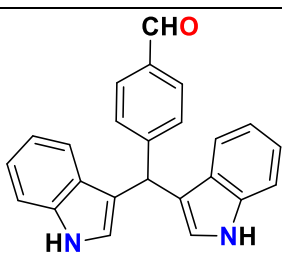
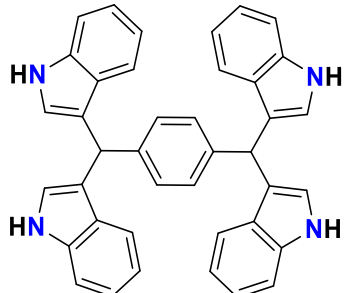
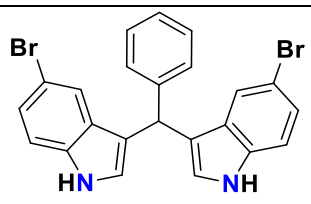
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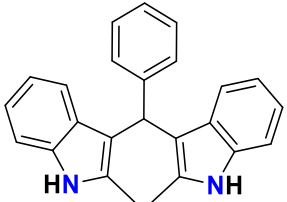
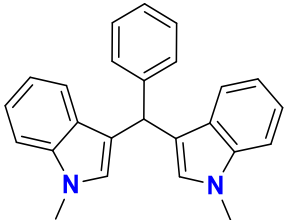
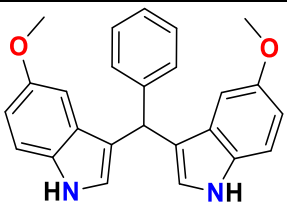
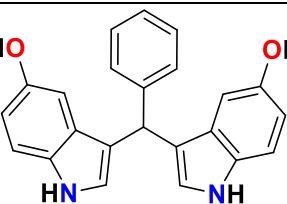
14				2n	29
15				2o	87
16				2p	90
17				2q	91
18				2r	81
19				2s	70

Characterization of isolated bis(indolyl)alkanes:

	<p>2a¹: Red brown solid. $R_f = 0.37$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.79 (s, 2H, -NH), 7.31 (d, $J = 7.9$ Hz, 2H, ArH), 7.26 (d, $J = 5.4$ Hz, 3H, ArH), 7.22 – 7.12 (m, 4H, ArH), 7.09 (t, $J = 7.7$ Hz, 2H, ArH), 6.92 (t, $J = 7.3$ Hz, 2H, ArH), 6.55 – 6.52 (m, 2H, ArH), 5.80 (s, 1H, $-\text{C}_3\text{CH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 144.0, 136.7, 128.7, 128.2, 127.1, 126.2, 123.7, 121.9, 120.0, 119.7, 119.2, 111.1, 40.2. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+ \text{C}_{23}\text{H}_{18}\text{N}_2\text{Na}$ 345.1368, found 345.1350.</p>
	<p>2b¹: Red brown solid. $R_f = 0.37$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.79 (s, 2H, -NH), 7.27 (d, $J = 7.0$ Hz, 3H, ArH), 7.20 – 7.12 (m, 4H, ArH), 7.13 – 6.95 (m, 3H, ArH), 6.95 – 6.87 (m, 2H, ArH), 6.52 (s, 2H, ArH), 5.76 (s, 1H, $-\text{C}_3\text{CH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 142.5, 136.7, 131.8, 130.1, 128.4, 126.9, 123.6, 122.1, 119.8, 119.4, 119.2, 111.2, 39.6. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+ \text{C}_{23}\text{H}_{17}\text{ClN}_2\text{Na}$ 379.0978, found 379.0989.</p>
	<p>2c²: Red crystalline solid. $R_f = 0.3$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.86 (s, 2H, -NH), 7.34 – 7.29 (m, 4H, ArH), 7.18 (s, 1H, ArH), 7.16 – 7.08 (m, 4H, ArH), 6.96 – 6.92 (m, 2H, ArH), 6.81 – 6.72 (m, 1H, ArH), 6.56 (dd, $J = 2.4, 0.9$ Hz, 2H, ArH), 5.77 (s, 1H, $-\text{C}_3\text{CH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 143.1, 136.7, 131.3, 130.5, 126.9, 123.6, 122.1, 119.9, 119.8, 119.4, 119.1, 111.1, 39.7. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+ \text{C}_{23}\text{H}_{18}\text{BrN}_2\text{Na}$ 423.0473, found 423.0477.</p>
	<p>2d³: Yellow solid. $R_f = 0.5$ (20% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, $\text{DMSO}-d_6$) δ 10.92 (d, $J = 2.5$ Hz, 2H, -NH), 8.15 – 8.11 (m, 2H, ArH), 7.61 – 7.57 (m, 2H, ArH), 7.35 (d, $J = 8.1$ Hz, 2H, ArH), 7.26 (d, $J = 7.9$ Hz, 2H, ArH), 7.03 (m, 2H, ArH), 6.88 – 6.84 (m, 4H, ArH), 6.00 (s, 1H, $-\text{C}_3\text{CH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO}-d_6$) δ 153.6, 146.2, 137.0, 129.9, 126.8, 124.3, 123.9, 121.6, 119.4, 118.9, 117.1, 112.1, 40.6. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+ \text{C}_{23}\text{H}_{17}\text{N}_3\text{O}_2\text{Na}$ 390.1218, found 390.1207.</p>
	<p>2e¹: Red brown solid. $R_f = 0.32$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.81 (s, 2H, -NH), 7.38 (d, $J = 7.9$ Hz, 2H, ArH), 7.26 (s, 1H, ArH), 7.12 – 7.03 (m, 4H, ArH), 6.95 (t, $J = 7.5$ Hz, 2H, ArH), 6.84 – 6.81 (m, 2H, ArH), 6.71 (d, $J = 2.3$ Hz, 2H, ArH), 6.07 (s, 1H, $-\text{C}_3\text{CH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 148.7, 136.6, 126.8, 126.5, 125.2, 123.6, 123.2, 122.0, 119.8, 119.7, 119.4, 111.2, 35.3. HRMS (ESI) m/z calcd for $[\text{M}-\text{H}]^+ \text{C}_{21}\text{H}_{15}\text{N}_2\text{S}$ 327.0956, found 327.0861.</p>

	<p>2f¹: Salmon pink solid. $R_f = 0.38$ (20% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.87 (s, 2H, -NH), 7.40 (d, $J = 7.9$ Hz, 2H, ArH), 7.35 (d, $J = 8.1$ Hz, 2H, ArH), 7.26 (d, $J = 2.5$ Hz, 2H, ArH), 7.17 (t, $J = 7.5$ Hz, 2H, ArH), 7.01 (t, $J = 7.4$ Hz, 2H, ArH), 6.83 (d, $J = 8.5$ Hz, 2H, ArH), 6.63 (s, 2H, ArH), 5.85 (s, 1H, $-\text{C}_3\text{CH}$), 3.79 (s, 3H, $-\text{OCH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 157.9, 136.7, 136.2, 129.6, 127.1, 123.5, 121.9, 120.1, 120.0, 119.2, 113.6, 111.0, 55.2, 39.3. HRMS (ESI) m/z calcd for $[\text{M}+\text{K}]^+$ $\text{C}_{24}\text{H}_{20}\text{N}_2\text{OK}$ 391.1213, found 391.1203.</p>
	<p>2g⁴: Red solid. $R_f = 0.5$ (20% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.77 – 7.58 (m, 2H, -NH), 7.51 (d, $J = 7.9$ Hz, 1H, ArH), 7.47 – 7.39 (m, 1H, ArH), 7.29 (m, 1H, ArH), 7.21 – 7.14 (m, 2H, ArH), 7.03 (m, 2H, ArH), 6.98 – 6.93 (m, 1H, ArH), 6.83 (d, $J = 2.4$ Hz, 1H, ArH), 6.75 – 6.66 (m, 1H, ArH), 4.40 – 4.26 (m, 1H, $-\text{C}_3\text{CH}$), 2.15 – 2.03 (m, 2H, $-\text{CH}_2$), 1.24 – 1.16 (m, 6H, $-\text{CH}_2$), 0.77 – 0.74 (m, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 136.6, 127.2, 121.7, 121.4, 120.6, 119.7, 119.0, 111.0, 35.8, 34.0, 32.0, 28.0, 22.7, 14.2. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{22}\text{H}_{24}\text{N}_2\text{Na}$ 339.1837, found 339.1799.</p>
	<p>2h¹: Red solid. $R_f = 0.36$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.63 (s, 2H, -NH), 7.30 (d, $J = 8.1$ Hz, 2H, ArH), 7.17 – 7.11 (m, 6H, ArH), 7.05 (d, $J = 7.1$ Hz, 2H, ArH), 6.89 (t, $J = 7.5$ Hz, 2H, ArH), 6.47 – 6.45 (m, 2H, ArH), 5.74 (s, 1H, $-\text{C}_3\text{CH}$), 1.20 (s, 9H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 148.8, 140.9, 136.7, 128.3, 127.2, 125.1, 123.7, 121.9, 120.0, 119.9, 119.2, 111.1, 39.6, 34.4, 31.5. HRMS (ESI) m/z calcd for $[\text{M}-\text{H}]^+$ $\text{C}_{27}\text{H}_{25}\text{N}_2$ 377.2018, found 377.2038.</p>
	<p>2i¹: Red solid. $R_f = 0.34$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.70 (s, 2H, -NH), 7.30 (d, $J = 7.9$ Hz, 2H, ArH), 7.21 (s, 1H, ArH), 7.13 (d, $J = 8.3$ Hz, 2H, ArH), 7.07 (t, $J = 7.3$ Hz, 2H, ArH), 6.99 (d, $J = 7.9$ Hz, 2H, ArH), 6.91 (t, $J = 7.4$ Hz, 2H, ArH), 6.81 – 6.67 (m, 1H, ArH), 6.53 – 6.49 (m, 2H, ArH), 5.75 (s, 1H, $-\text{C}_3\text{CH}$), 2.23 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 141.0, 136.7, 135.5, 129.0, 128.6, 127.1, 123.6, 121.9, 120.0, 119.9, 119.2, 111.1, 39.8, 21.1. HRMS (ESI) m/z calcd for $[\text{M}-\text{H}]^+$ $\text{C}_{24}\text{H}_{19}\text{N}_2$ 335.1548, found 335.1550. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{24}\text{H}_{20}\text{N}_2\text{Na}$ 359.1524, found 359.1526.</p>
	<p>2j³: Red sticky solid. $R_f = 0.28$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, DMSO) δ 10.75 (d, $J = 2.5$ Hz, 2H, -NH), 9.12 (s, 1H, -OH), 7.31 (dd, $J = 8.1, 1.1$ Hz, 2H, ArH), 7.24 (d, $J = 8.0$ Hz, 2H, ArH), 7.13 – 7.09 (m, 2H, ArH), 7.00 (m, 2H, ArH), 6.83 (m, 2H, ArH), 6.77 – 6.74 (m, 2H, ArH), 6.65 – 6.61 (m, 2H, ArH), 5.68 (s, 1H, $-\text{C}_3\text{CH}$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO-d_6) δ 155.7, 137.0, 135.6,</p>

	129.6, 127.1, 123.8, 121.2, 119.6, 119.1, 118.5, 111.8, 49.1. HRMS (ESI) m/z calcd for $[M+K]^+ C_{23}H_{18}N_2OK$ 377.1056, found 377.1051.
	2k³ : Brown sticky mass. $R_f = 0.2$ (15% Ethyl acetate/Petroleum ether). 1H NMR (400 MHz, $CDCl_3$) δ 7.89 (s, 2H, -NH), 7.47 (d, $J = 7.9$ Hz, 2H, ArH), 7.33 (s, 1H, ArH), 7.31 (s, 1H, ArH), 7.16 (t, $J = 7.5$ Hz, 2H, ArH), 7.03 (t, $J = 7.5$ Hz, 2H, ArH), 6.83 (d, $J = 2.1$ Hz, 2H, ArH), 6.29 (s, 1H, ArH), 6.05 (d, $J = 3.1$ Hz, 1H, ArH), 5.93 (s, 1H, ArH), 5.28 (s, 1H, -C ₃ CH). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 157.0, 141.2, 136.5, 126.7, 123.0, 121.9, 119.7, 119.3, 117.1, 111.1, 111.1, 106.6, 34.1. HRMS (ESI) m/z calcd for $[M-H]^+ C_{21}H_{15}N_2O$ 311.1184, found 311.1219.
	2l⁵ : Violet solid. $R_f = 0.3$ (50% Ethyl acetate/Petroleum ether). 1H NMR (400 MHz, $CDCl_3$) δ 8.64 (s, 1H, -NH), 8.48 (s, 1H, NH, ArH), 8.13 (s, 2H, ArH), 7.65 (dt, $J = 7.9, 1.9$ Hz, 1H, ArH), 7.36 (dd, $J = 8.0, 5.4$ Hz, 4H, ArH), 7.19 (m, 3H, ArH), 7.02 (t, $J = 7.5$ Hz, 2H, ArH), 6.64 (d, $J = 2.4$ Hz, 2H, ArH), 5.92 (s, 1H, -C ₃ CH). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 149.6, 146.8, 136.8, 136.7, 126.7, 123.7, 122.2, 119.6, 119.5, 118.3, 115.7, 111.2, 39.1. HRMS (ESI) m/z calcd for $[M+H]^+ C_{22}H_{18}N_3$ 324.1501, found 324.1482.
	2m⁶ : Red sticky mass. $R_f = 0.46$ (30% Ethyl acetate/Petroleum ether). 1H NMR (400 MHz, $CDCl_3$) δ 9.87 (s, 1H, -CHO), 7.89 (d, $J = 18.5$ Hz, 2H, -NH), 7.69 (d, $J = 8.2$ Hz, 2H, ArH), 7.41 (d, $J = 8.1$ Hz, 2H, ArH), 7.26 (d, $J = 8.8$ Hz, 4H, ArH), 7.11 – 7.06 (m, 2H, ArH), 6.95 – 6.90 (m, 2H, ArH), 6.55 (d, $J = 1.7$ Hz, 2H, ArH), 5.86 (s, 1H, -C ₃ CH). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 192.3, 151.5, 136.7, 134.8, 129.9, 129.4, 126.8, 123.7, 122.2, 119.7, 119.5, 118.5, 111.2, 40.4. HRMS (ESI) m/z calcd for $[M-H]^+ C_{24}H_{17}N_2O$ 349.1341, found 349.1345.
	2n⁷ : Pink solid. $R_f = 0.29$ (30% Ethyl acetate/Petroleum ether). 1H NMR (400 MHz, $DMSO-d_6$) δ 10.76 (d, $J = 2.4$ Hz, 4H, -NH), 7.32 (dt, $J = 8.2, 0.9$ Hz, 4H, ArH), 7.24 (d, $J = 9.0$ Hz, 8H, ArH), 7.01 (m, 4H, ArH), 6.85 – 6.79 (m, 8H, ArH), 5.78 (d, $J = 1.0$ Hz, 2H, -C ₃ CH). $^{13}C\{^1H\}$ NMR (100 MHz, $DMSO-d_6$) δ 142.8, 137.0, 128.5, 127.2, 123.9, 121.3, 119.6, 118.8, 118.6, 111.9, 39.9. HRMS (ESI) m/z calcd for $[M+Na]^+ C_{40}H_{30}N_4Na$ 589.2368, found 589.2369.
	2o³ : Red solid. $R_f = 0.34$ (15% Ethyl acetate/Petroleum ether). 1H NMR (400 MHz, $CDCl_3$) δ 8.07 – 8.02 (m, 2H, -NH), 7.62 – 7.46 (m, 2H, ArH), 7.45 – 7.25 (m, 4H, ArH), 7.23 (d, $J = 1.5$ Hz, 1H, ArH), 7.21 – 7.10 (m, 5H, ArH), 6.58 (dd, $J = 2.4, 1.1$ Hz, 1H, ArH), 5.68 (s, 1H, -C ₃ CH). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ 171.2, 135.3, 133.8, 130.2, 129.2, 128.5, 126.5, 125.0, 124.8, 122.3, 119.1, 112.6, 39.9. HRMS (ESI) m/z calcd for $[M+H]^+ C_{23}H_{17}Br_2N_2$ 478.9759, found 478.9781.

	<p>2p²: Pink solid. $R_f = 0.38$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 10.77 (s, 2H, -NH), 7.31 – 7.21 (m, 7H, ArH), 6.95 – 6.90 (m, 2H, ArH), 6.85 (d, $J = 7.8$ Hz, 2H, ArH), 6.71 (t, $J = 7.0$ Hz, 2H, ArH), 5.97 (s, 1H, -C₃CH), 2.10 (s, 6H, -CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) δ 144.7, 135.6, 132.5, 129.2, 128.8, 128.4, 126.2, 120.0, 119.0, 118.4, 112.7, 110.8, 39.1, 12.4. HRMS (ESI) m/z calcd for $[\text{M-H}]^+ \text{C}_{25}\text{H}_{21}\text{N}_2$ 349.1705, found 349.1699.</p>
	<p>2q³: Pink solid. $R_f = 0.32$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 7.35 (dd, $J = 7.9$, 3.5 Hz, 4H, ArH), 7.31 – 7.22 (m, 4H, ArH), 7.15 (d, $J = 14.5$ Hz, 1H, ArH), 7.09 (t, $J = 7.6$ Hz, 2H, ArH), 6.89 (t, $J = 7.5$ Hz, 2H, ArH), 6.80 (s, 2H, ArH), 5.81 (s, 1H, -C₃CH), 3.67 (s, 6H, -NCH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) δ 145.3, 137.4, 128.7, 128.6, 128.3, 127.4, 126.3, 121.5, 119.7, 118.8, 117.8, 110.1, 39.8, 32.7. HRMS (ESI) m/z calcd for $[\text{M-H}]^+ \text{C}_{25}\text{H}_{21}\text{N}_2$ 349.1705, found 349.1726.</p>
	<p>2r¹: Pink solid. $R_f = 0.3$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, DMSO) δ 10.69 (d, $J = 2.6$ Hz, 2H, -NH), 7.44 – 7.38 (m, 2H, ArH), 7.30 (dd, $J = 14.5$, 8.0 Hz, 4H, ArH), 7.24 – 7.18 (m, 1H, ArH), 6.87 (d, $J = 2.4$ Hz, 2H, ArH), 6.79 – 6.72 (m, 4H, ArH), 5.79 (s, 1H, -C₃CH), 3.63 (s, 6H, -OCH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) δ 153.1, 145.5, 132.3, 128.8, 128.5, 127.5, 126.2, 124.8, 118.2, 112.5, 110.0, 101.9, 55.7, 40.1. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+ \text{C}_{25}\text{H}_{22}\text{N}_2\text{O}_2\text{Na}$ 405.1579, found 405.1598.</p>
	<p>2s²: Pink sticky mass. $R_f = 0.48$ (20% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, $\text{DMSO-}d_6$) δ 9.62 (d, $J = 2.7$ Hz, 2H, -OH), 7.65 (s, 2H, -NH), 6.49 – 6.39 (m, 4H, ArH), 6.31 (dd, $J = 16.0$, 7.9 Hz, 3H, ArH), 5.82 – 5.70 (m, 6H, ArH), 4.73 (s, 1H, -C₃CH). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) δ 150.4, 145.4, 131.6, 128.8, 128.4, 127.8, 126.2, 124.5, 117.6, 112.2, 111.7, 103.7, 40.4. HRMS (ESI) m/z calcd for $[\text{M-H}]^+ \text{C}_{23}\text{H}_{17}\text{N}_2\text{O}_2$ 353.1290, found 353.1289.</p>

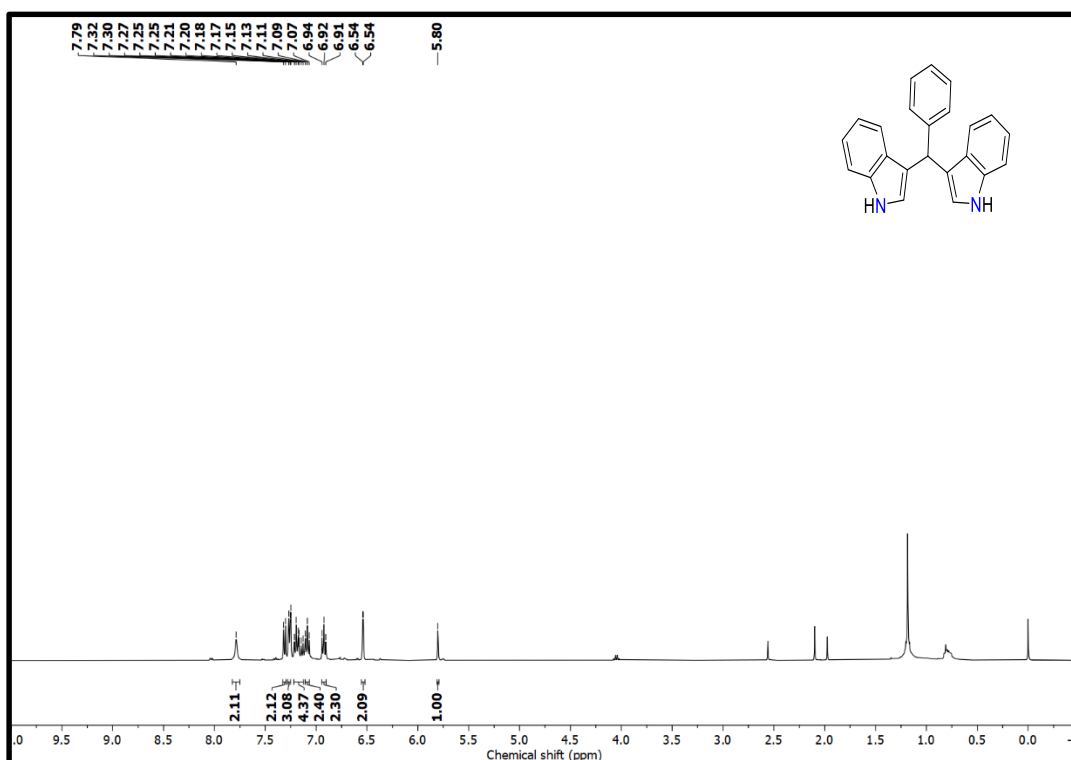


Figure S11: ^1H NMR spectrum of **2a** recorded in CDCl_3 .

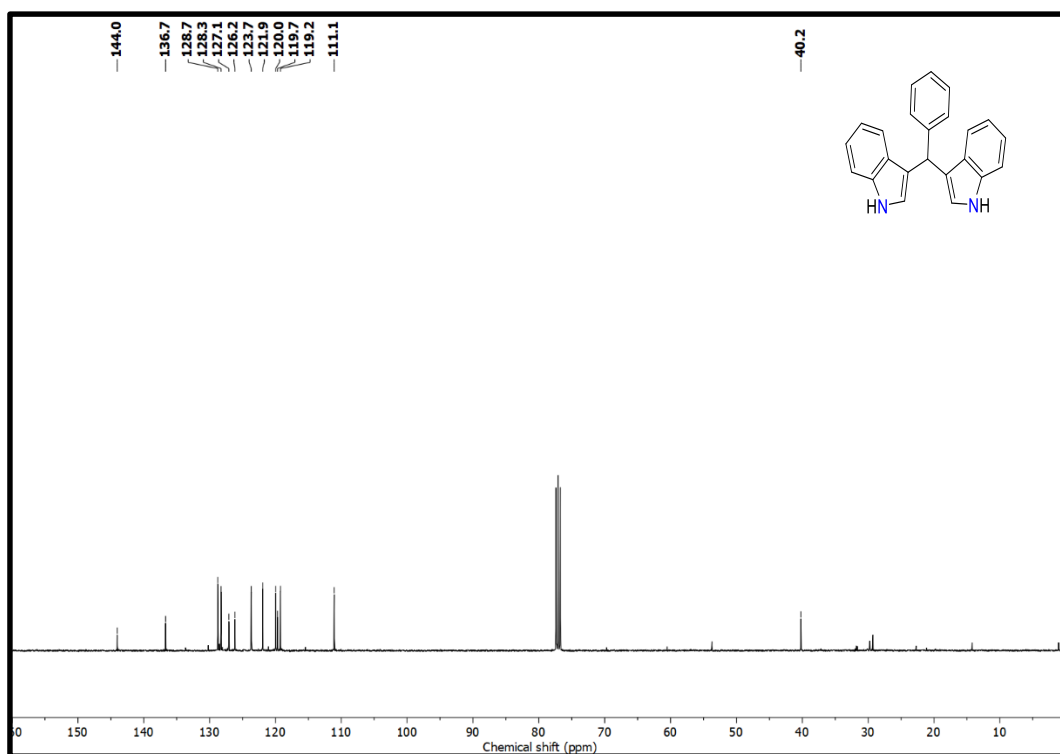


Figure S12: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2a** recorded in CDCl_3 .

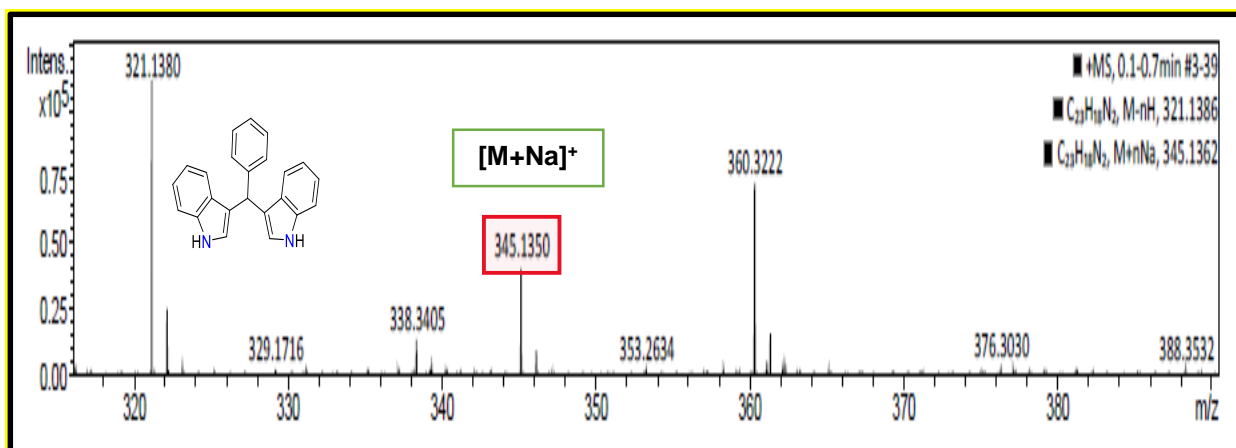


Figure S13: HRMS (ESI) spectrum of **2a**.

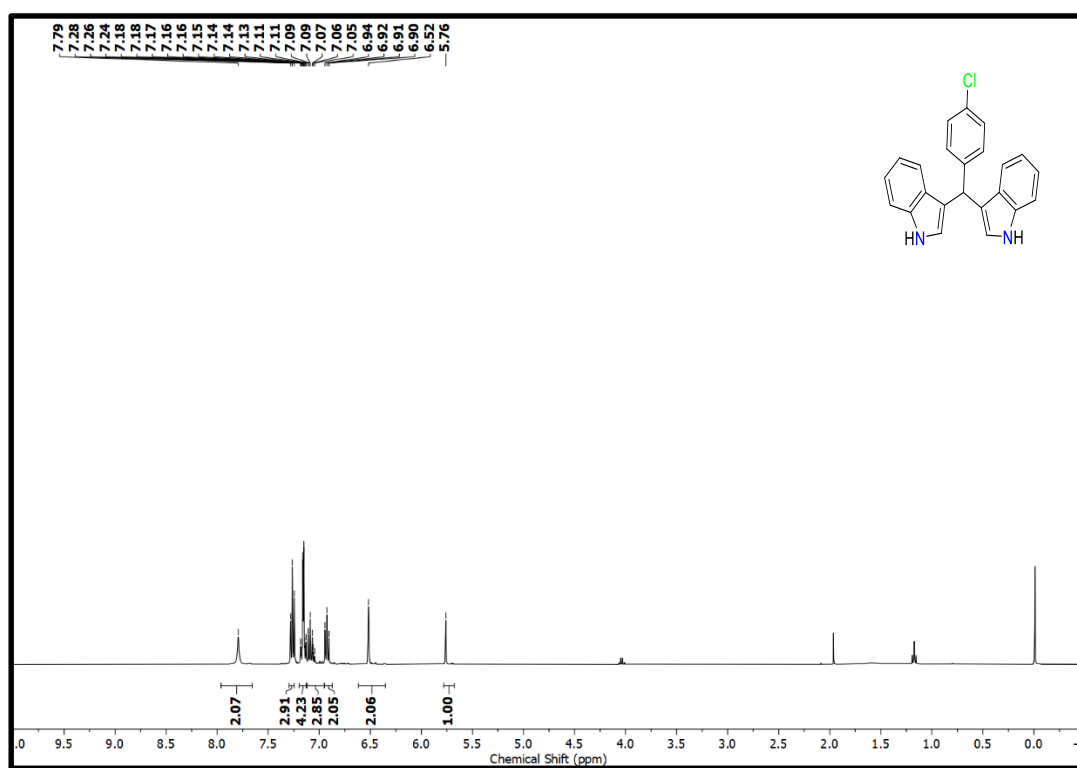


Figure S14: ¹H NMR spectrum of **2b** recorded in CDCl₃.

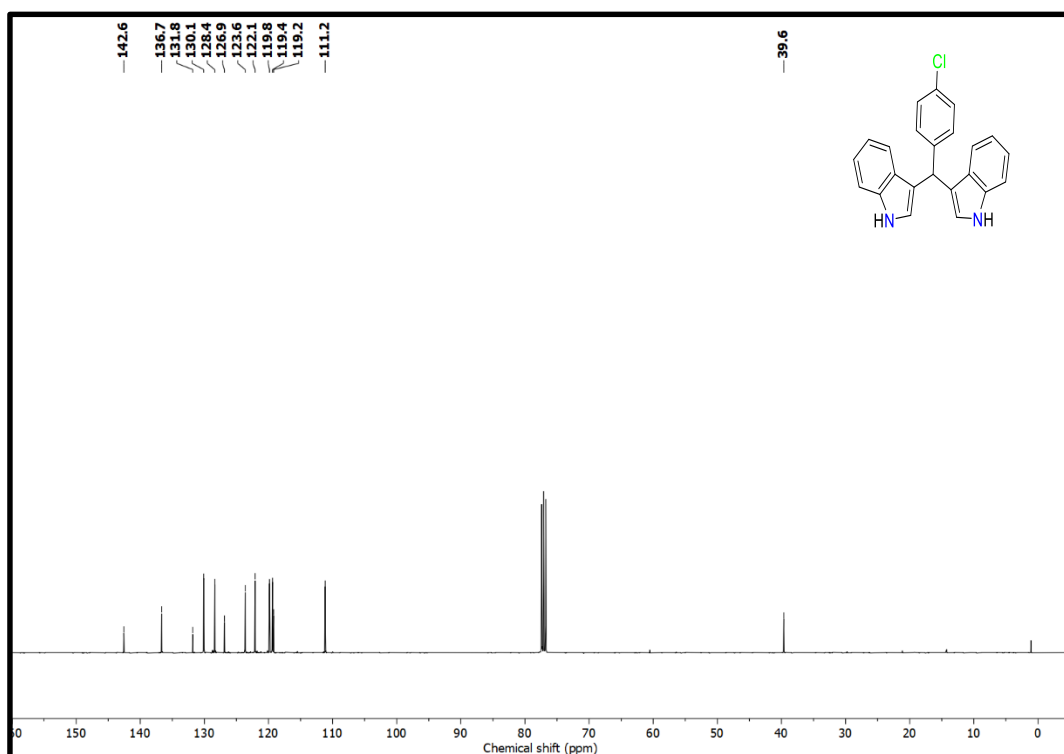


Figure S15: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2b** recorded in CDCl_3 .

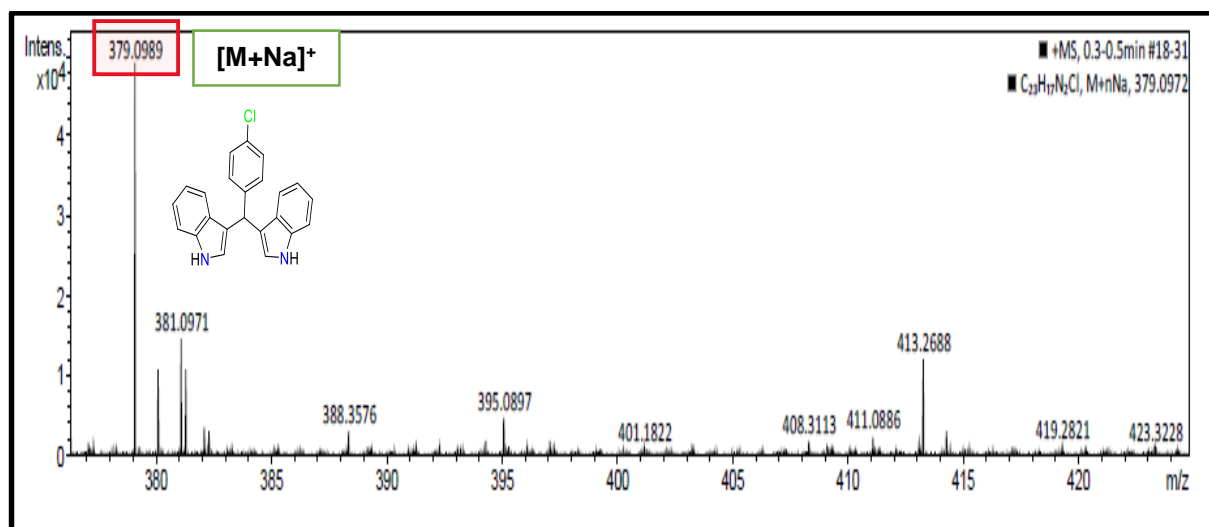


Figure S16: HRMS (ESI) spectrum of **2b**.

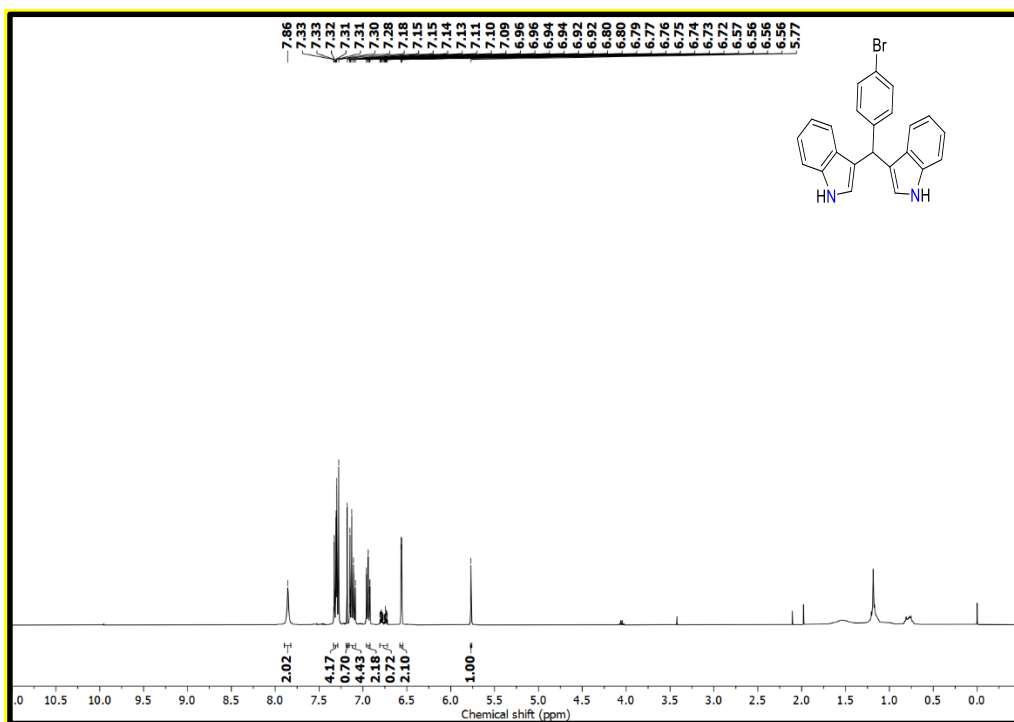


Figure S17: ^1H NMR spectrum of **2c** recorded in CDCl_3 .

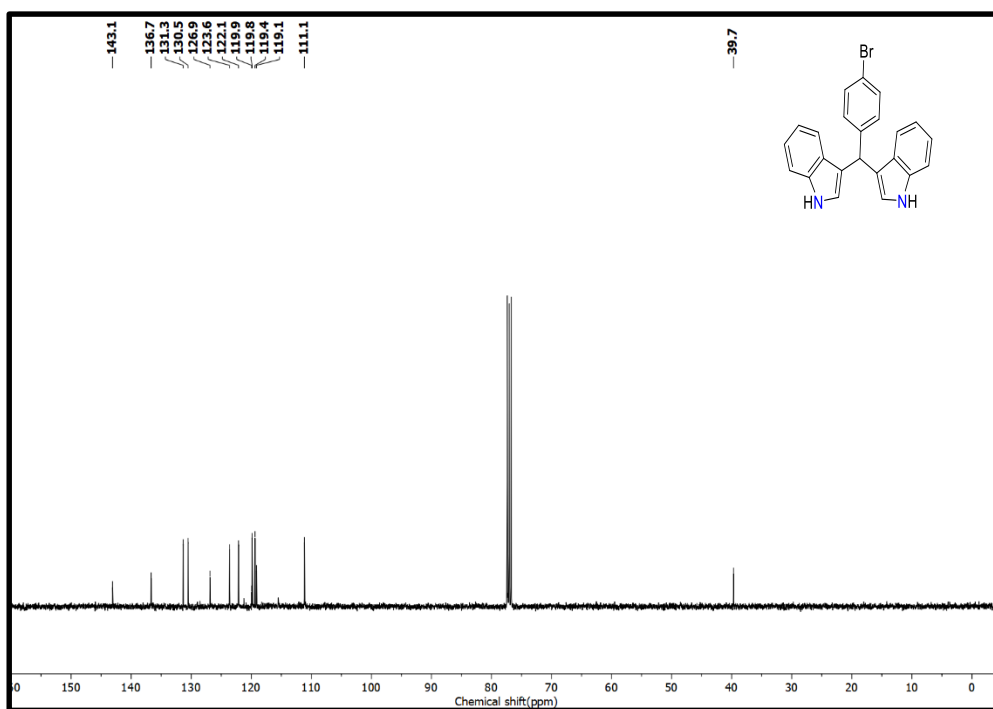


Figure S18: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2c** recorded in CDCl_3 .

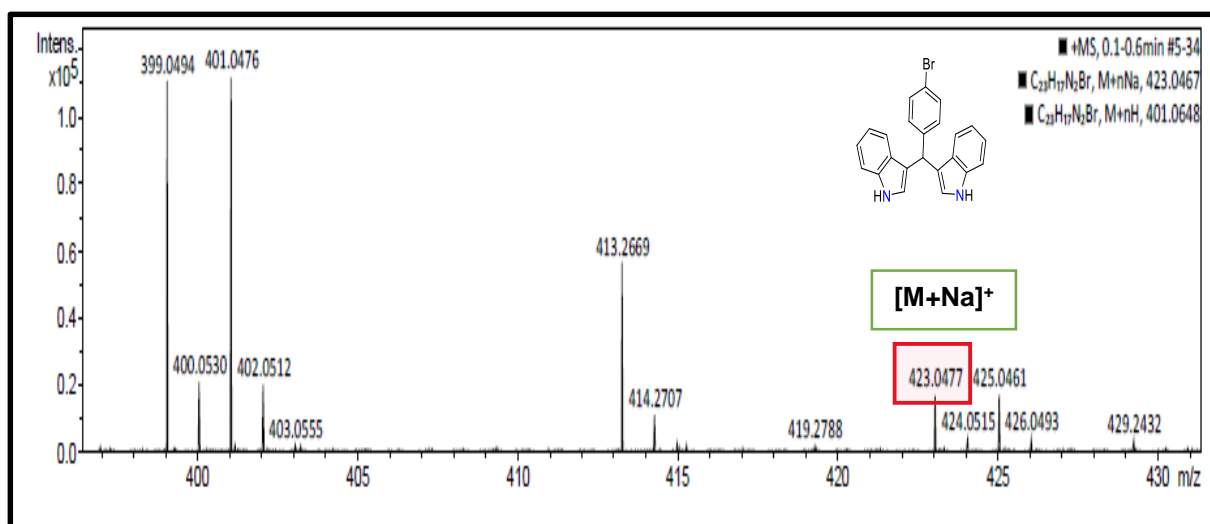


Figure S19: HRMS (ESI) spectrum of **2c**.

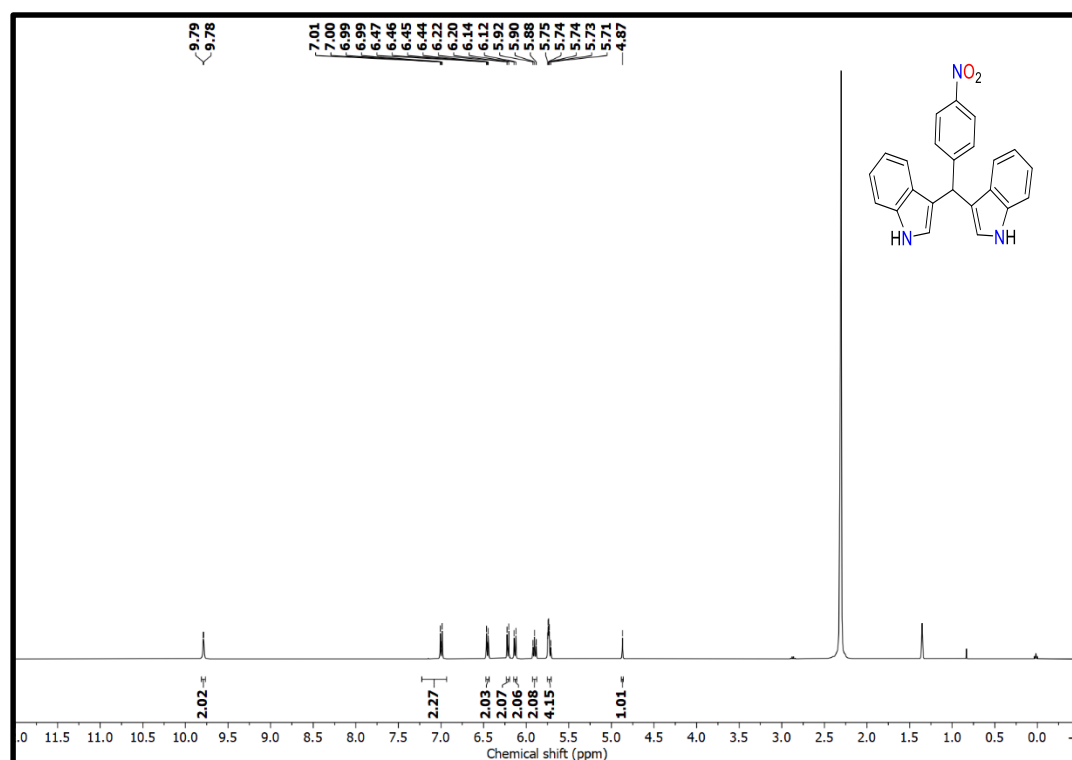


Figure S20: ¹H NMR spectrum of **2d** recorded in DMSO-*d*₆.

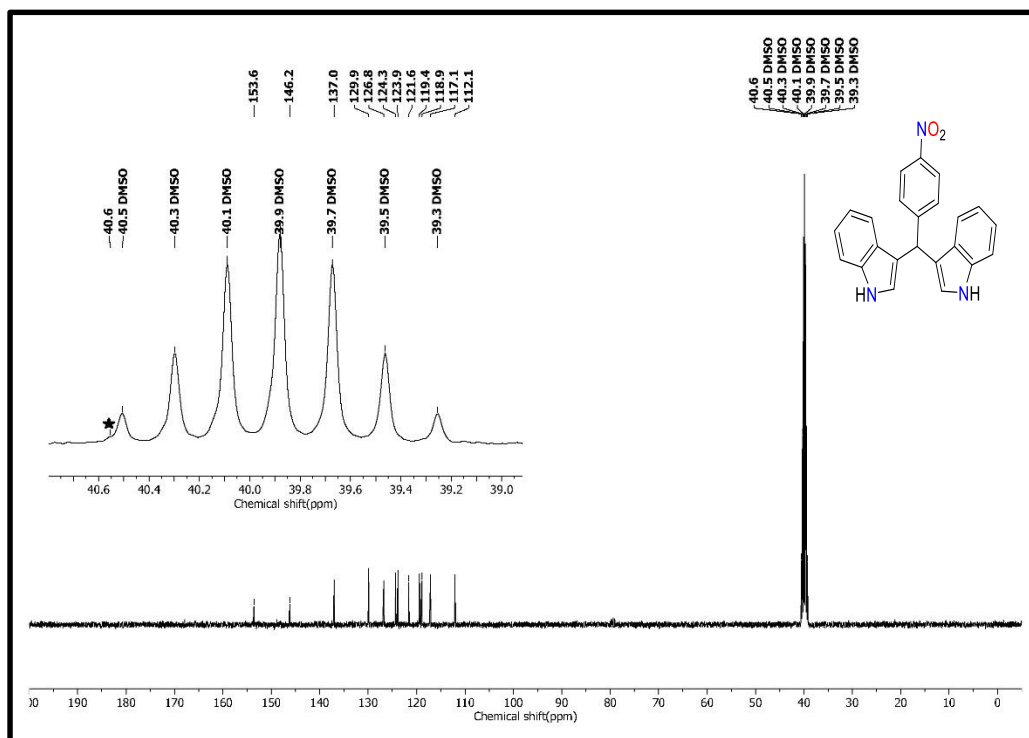


Figure S21: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2d** recorded in $\text{DMSO-}d_6$.

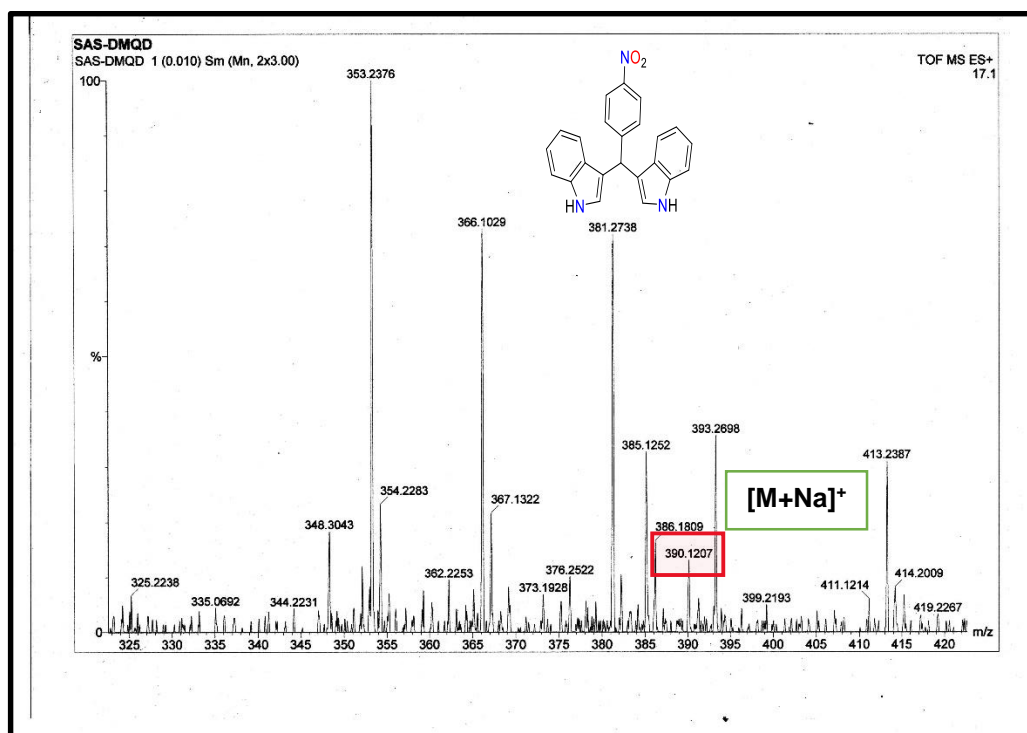


Figure S22: HRMS (ESI) spectrum of **2d**.

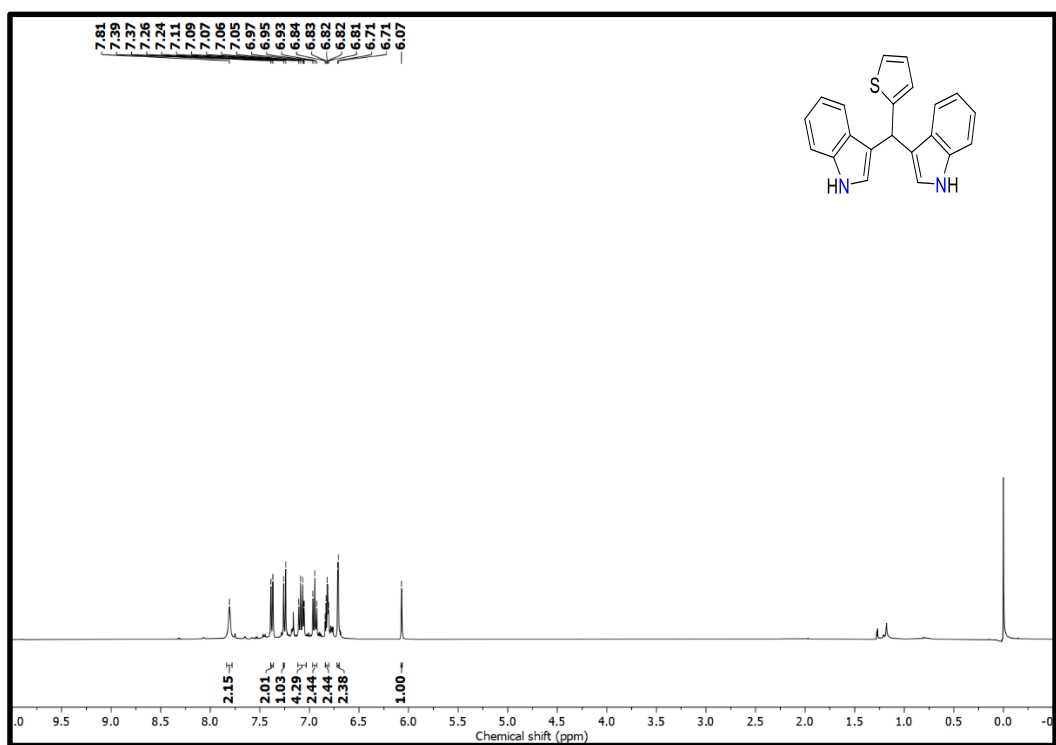


Figure S23: ^1H NMR spectrum of **2e** recorded in CDCl_3 .

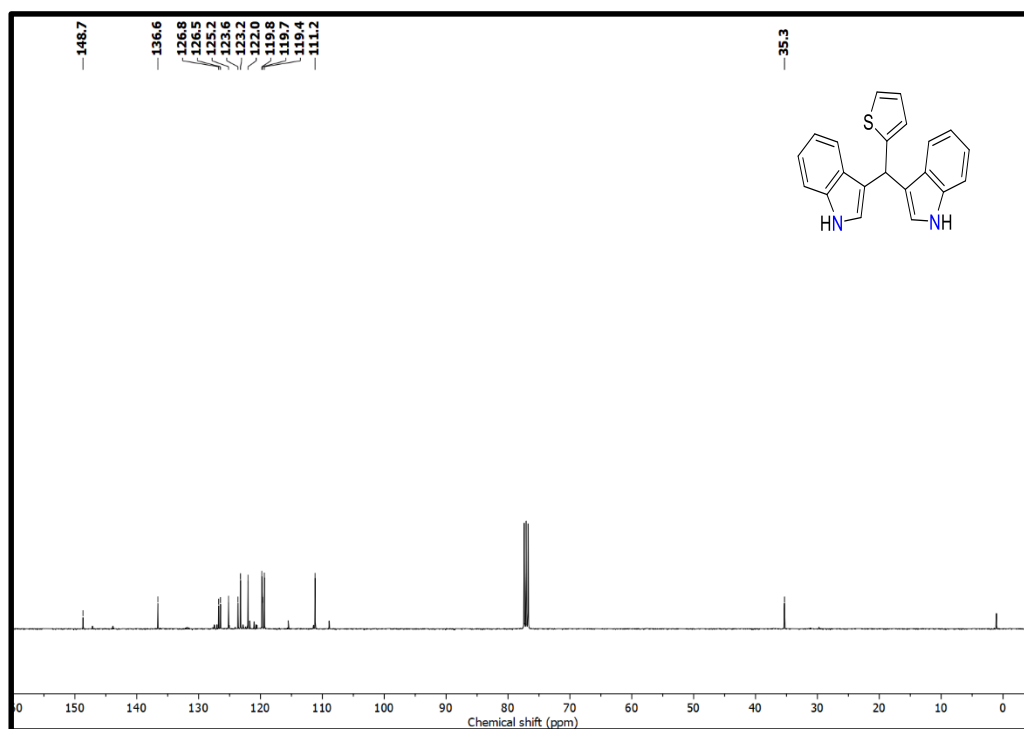


Figure S24: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2e** recorded in CDCl_3 .

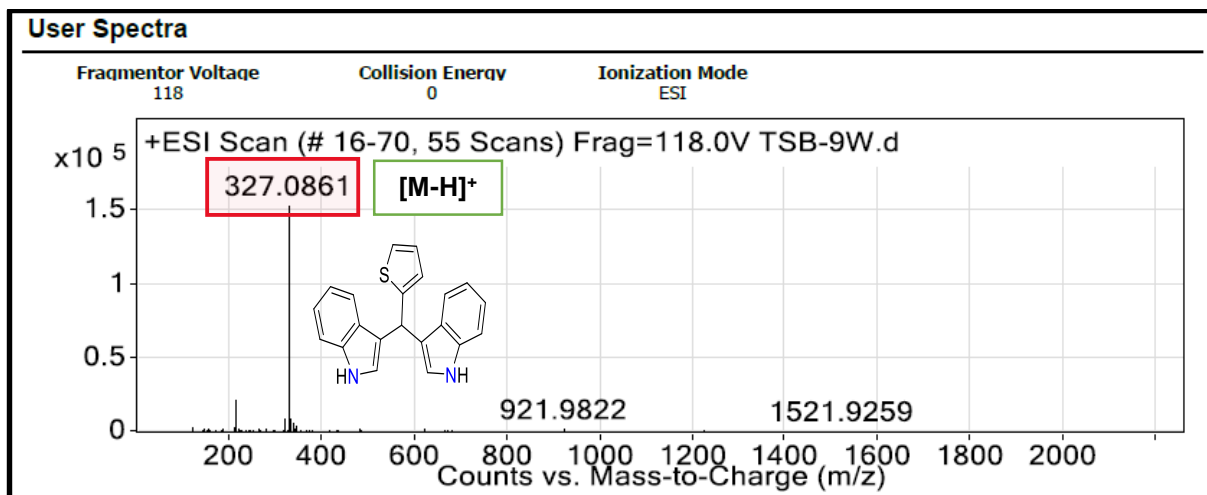


Figure S25: HRMS (ESI) spectrum of **2e**.

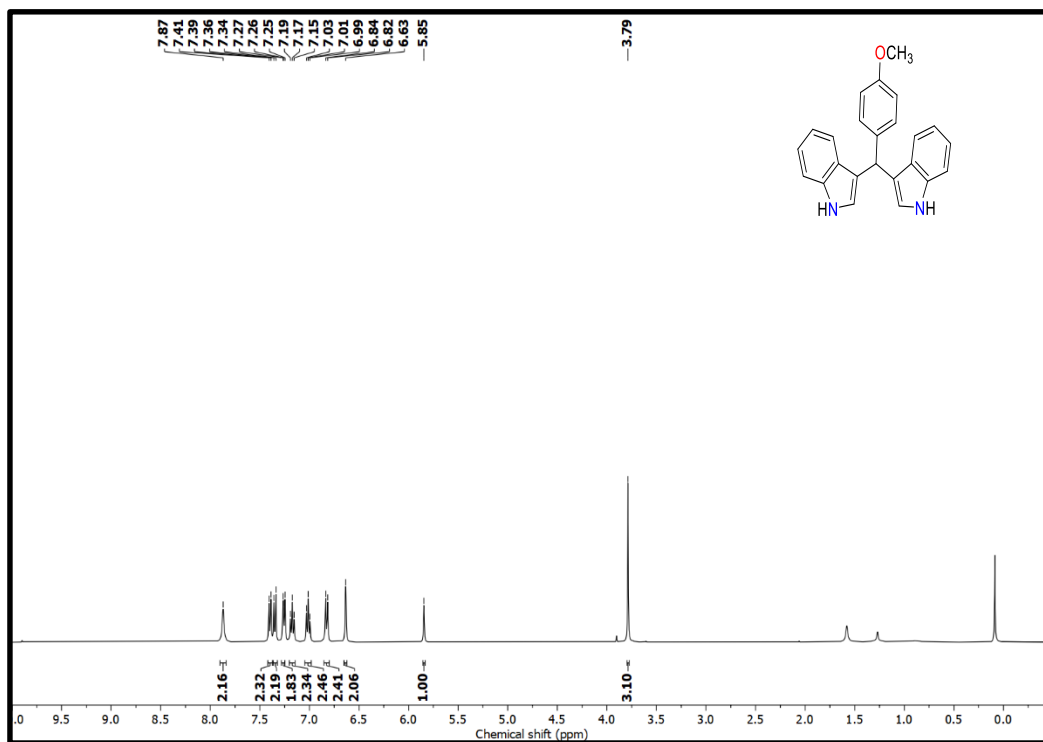


Figure S26: ¹H NMR spectrum of **2f** recorded in CDCl₃.

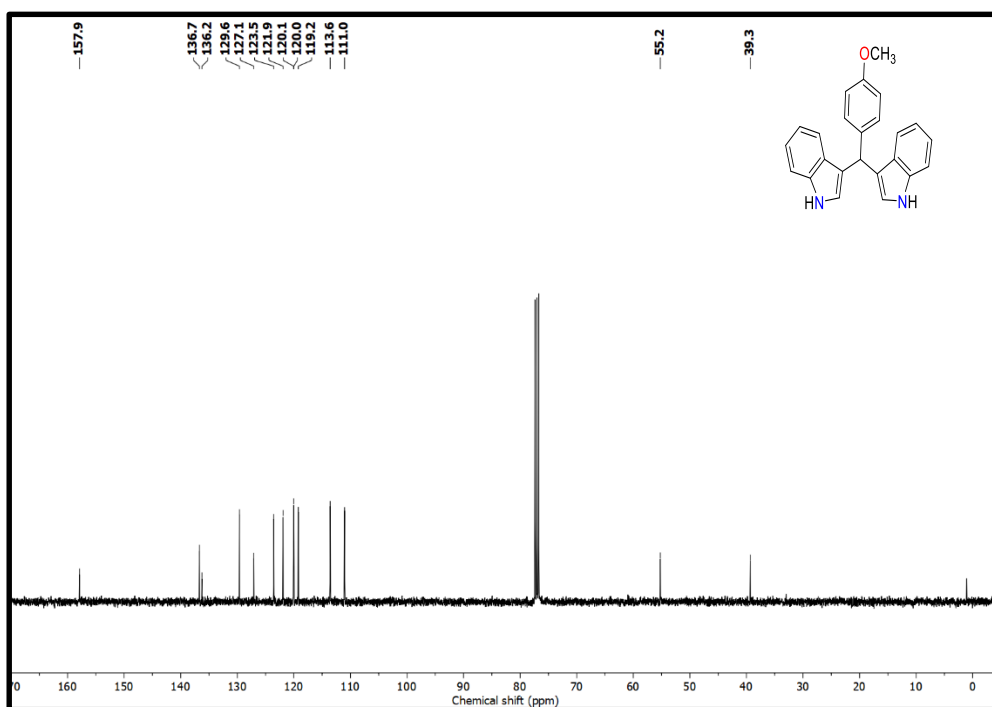


Figure S27: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2f** recorded in CDCl_3 .

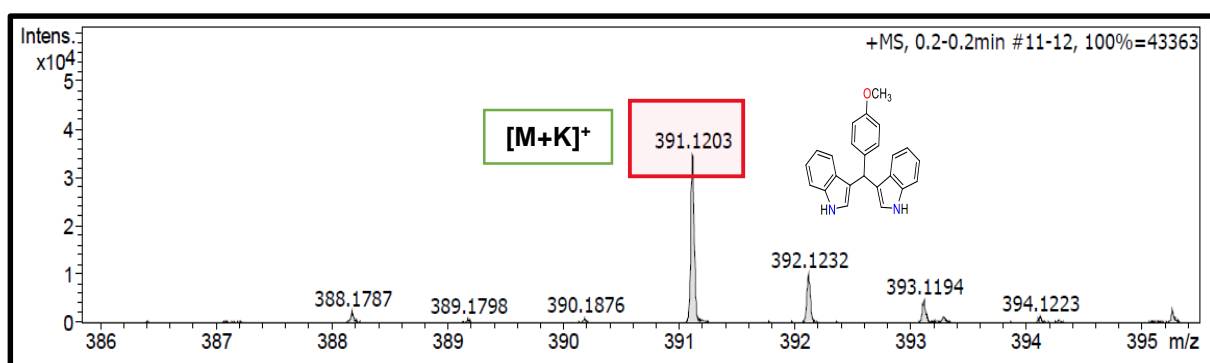


Figure S28: HRMS (ESI) spectrum of **2f**.

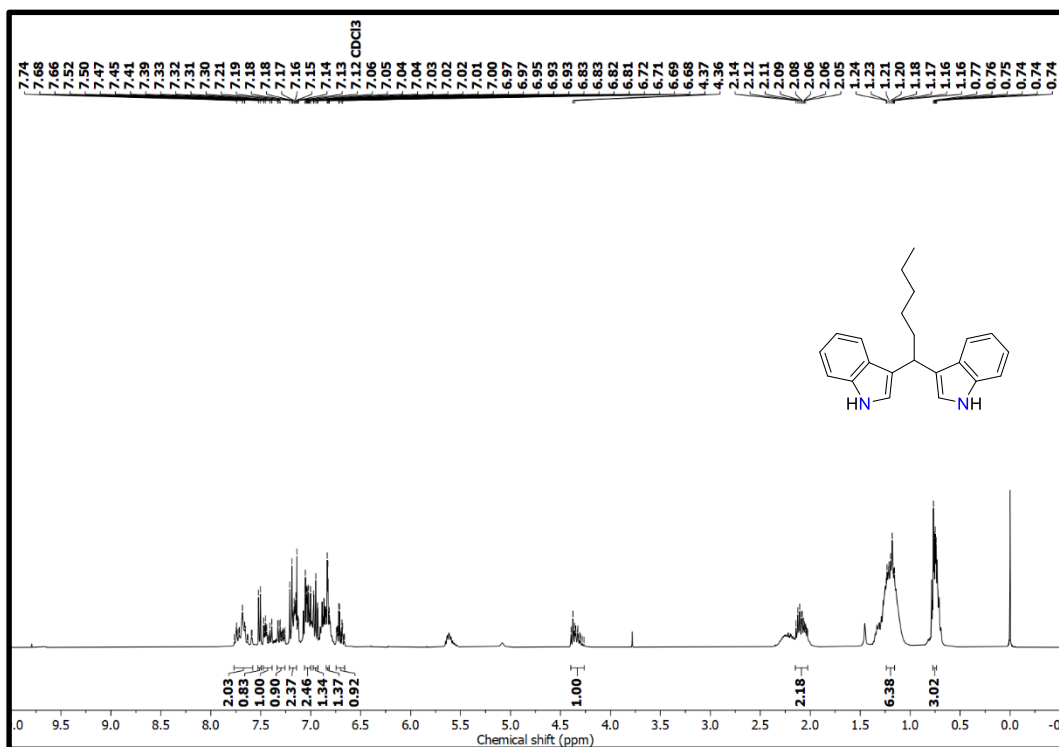


Figure S29: ^1H NMR spectrum of **2g** recorded in CDCl_3 .

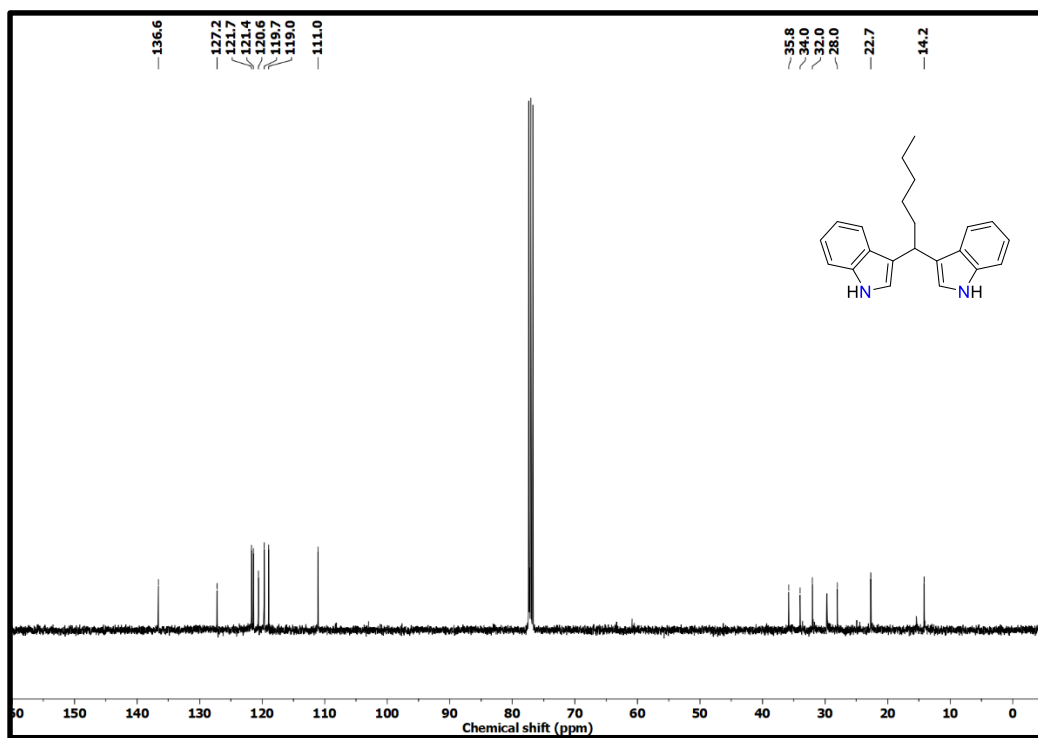


Figure S30: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2g** recorded in CDCl_3 .

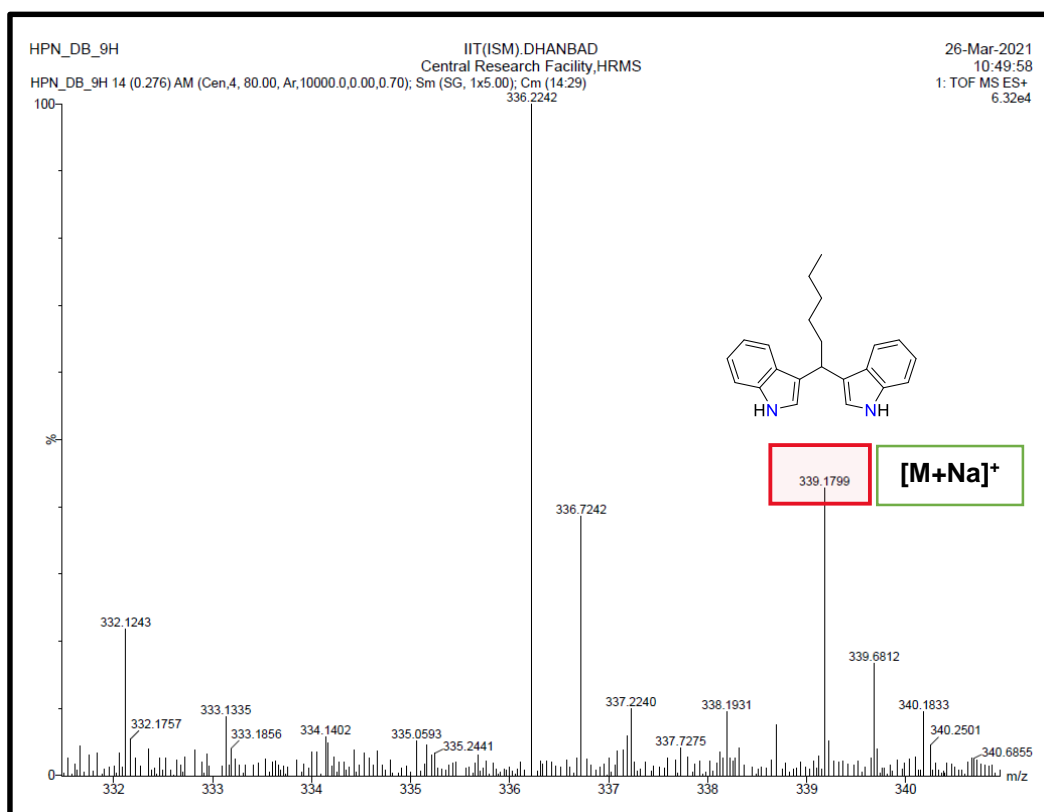


Figure S31: HRMS (ESI) spectrum of 2g.

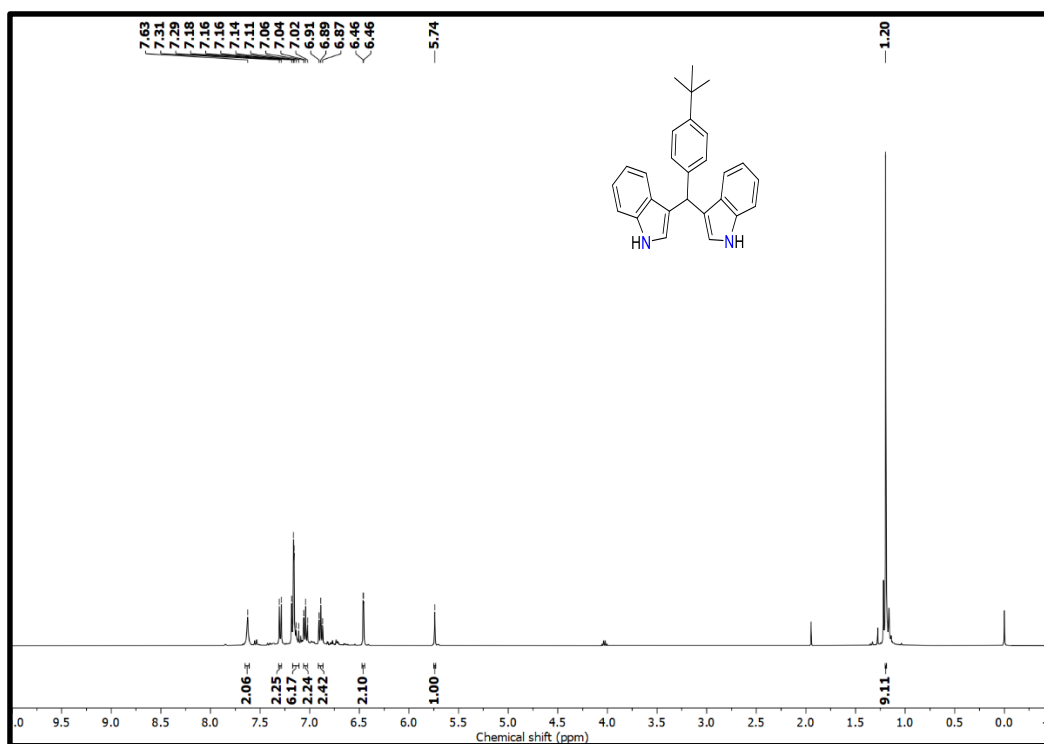


Figure S32: ¹H NMR spectrum of 2h recorded in CDCl₃.

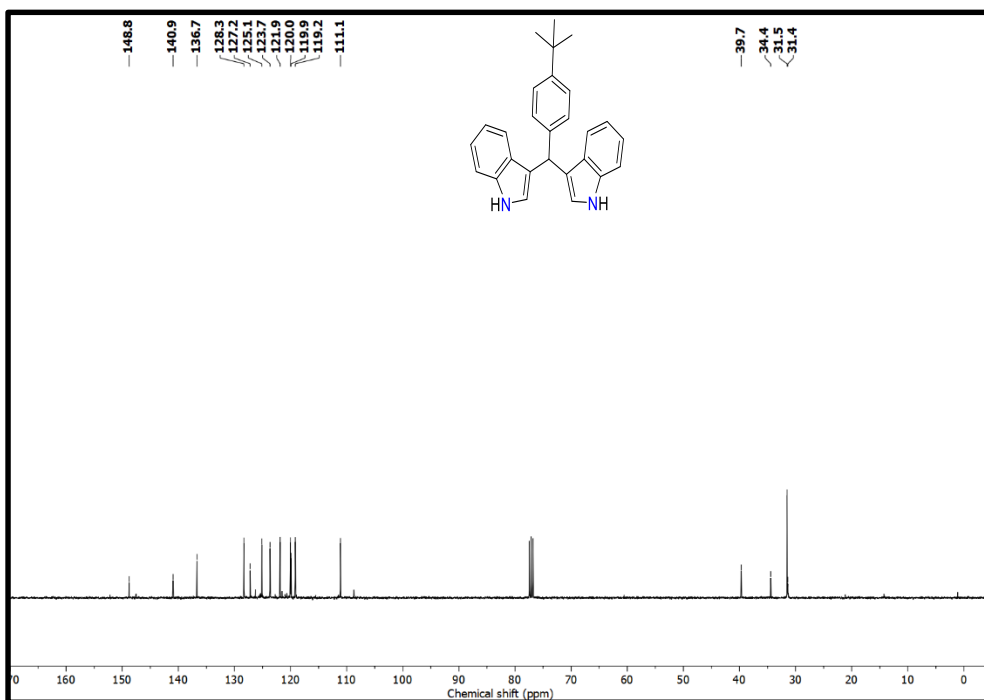


Figure S33: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2h** recorded in CDCl_3 .

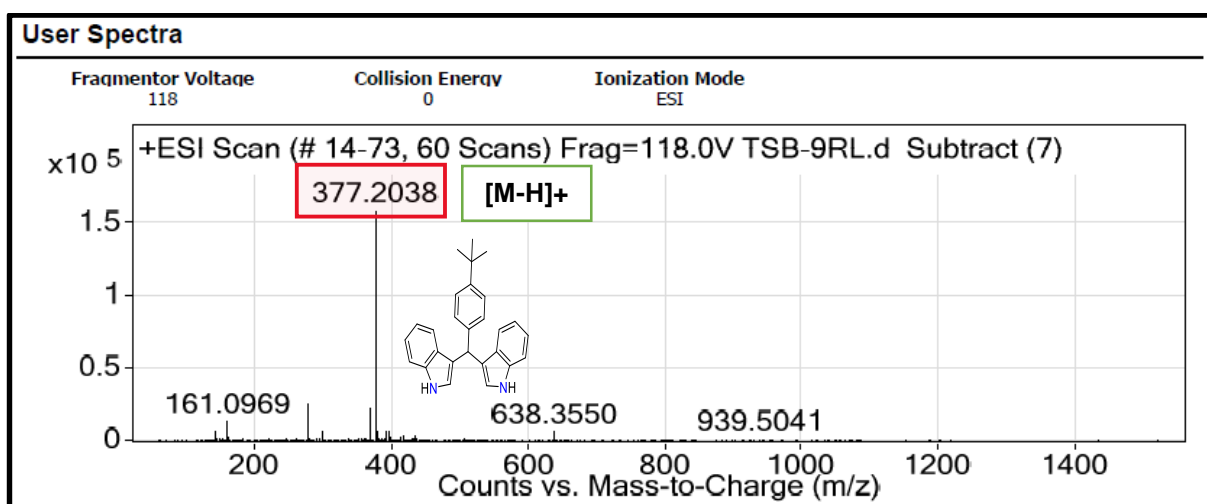


Figure S34: HRMS (ESI) spectrum of **2h**.

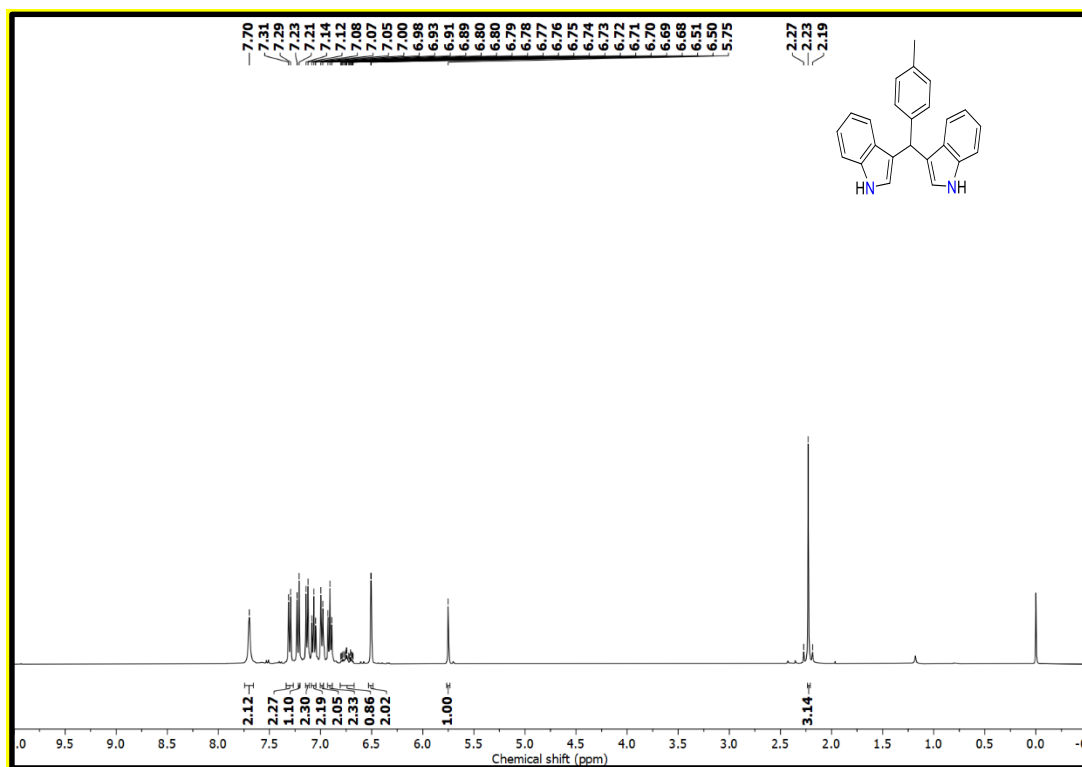


Figure S35: ¹H NMR spectrum of **2i** recorded in CDCl₃.

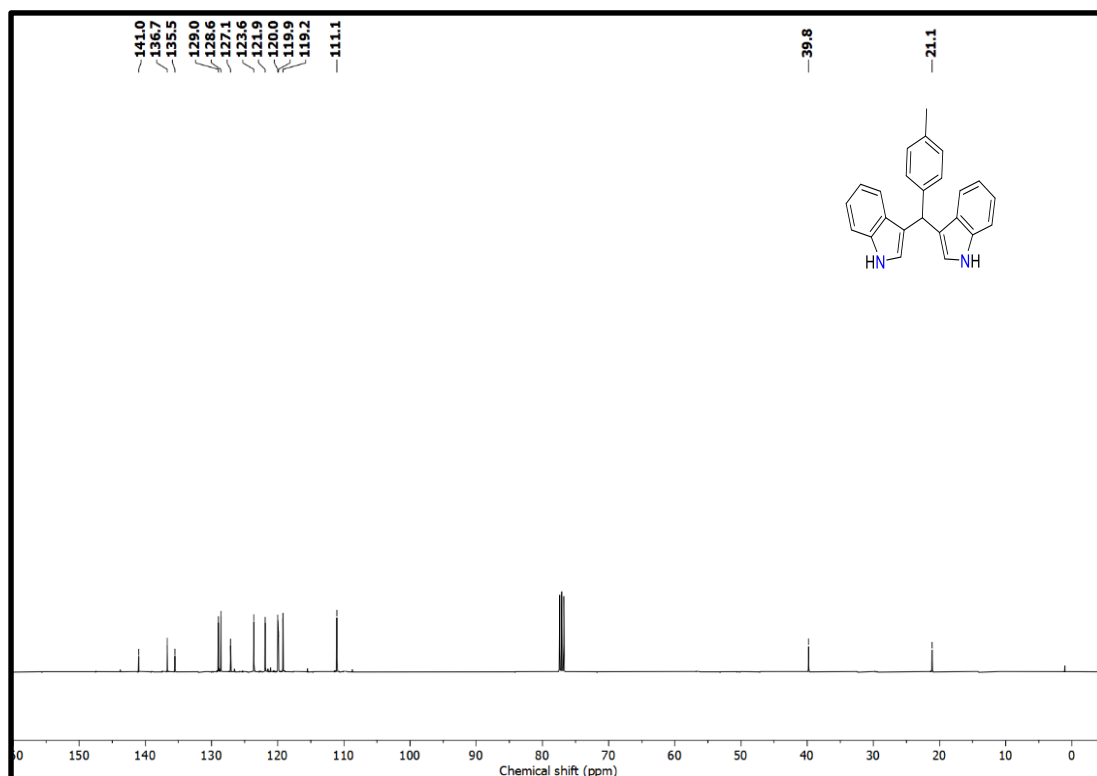


Figure S36: ¹³C{¹H} NMR spectrum of **2i** recorded in CDCl₃.

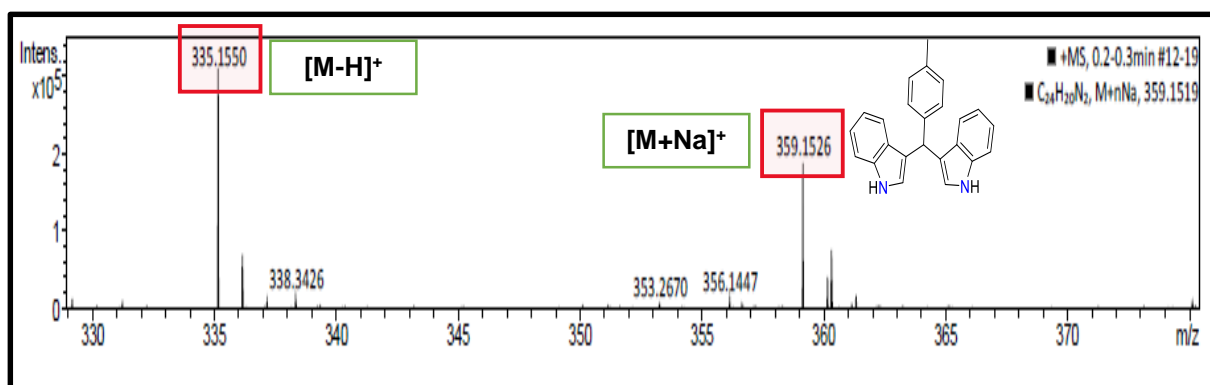


Figure S37: HRMS (ESI) spectrum of **2i**.

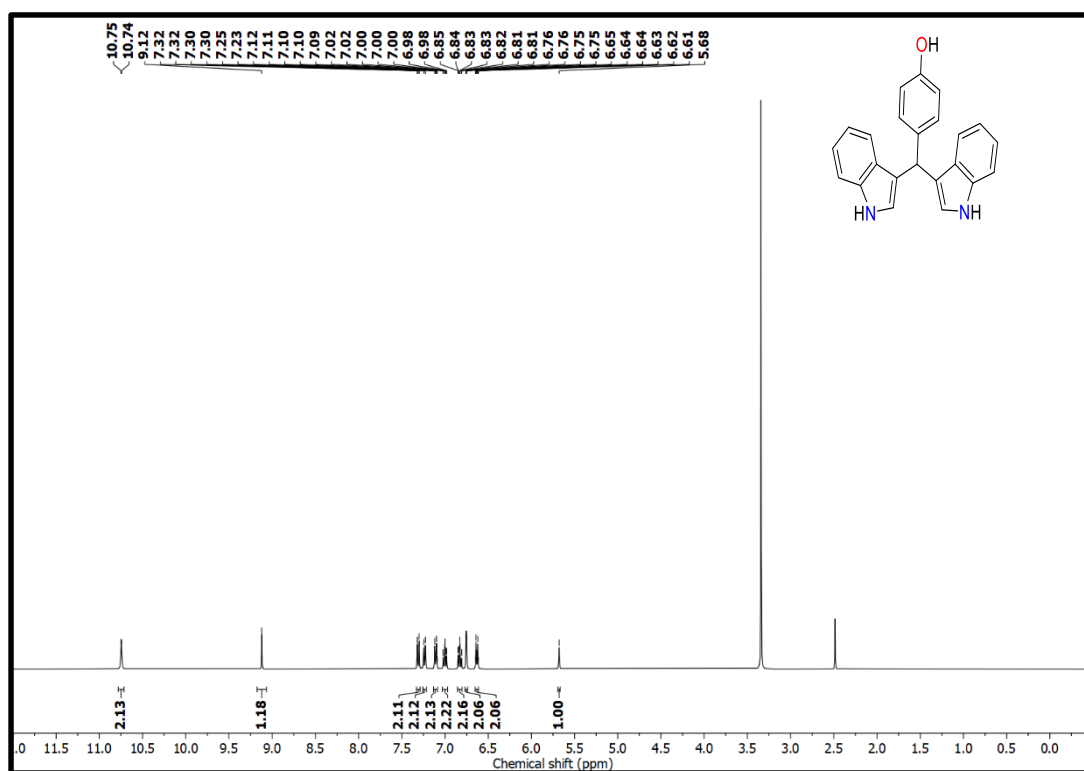


Figure S38: ^1H NMR spectrum of **2j** recorded in $\text{DMSO-}d_6$.

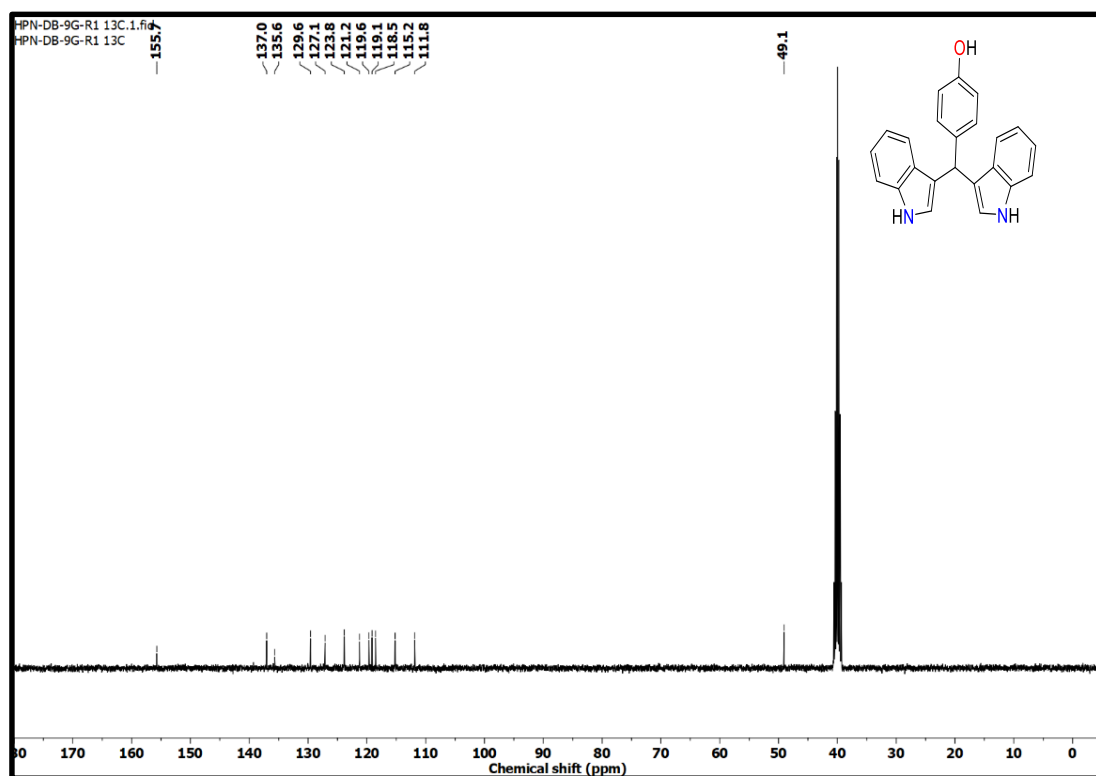


Figure S39: ¹³C{¹H} NMR spectrum of **2j** recorded in DMSO-*d*₆.

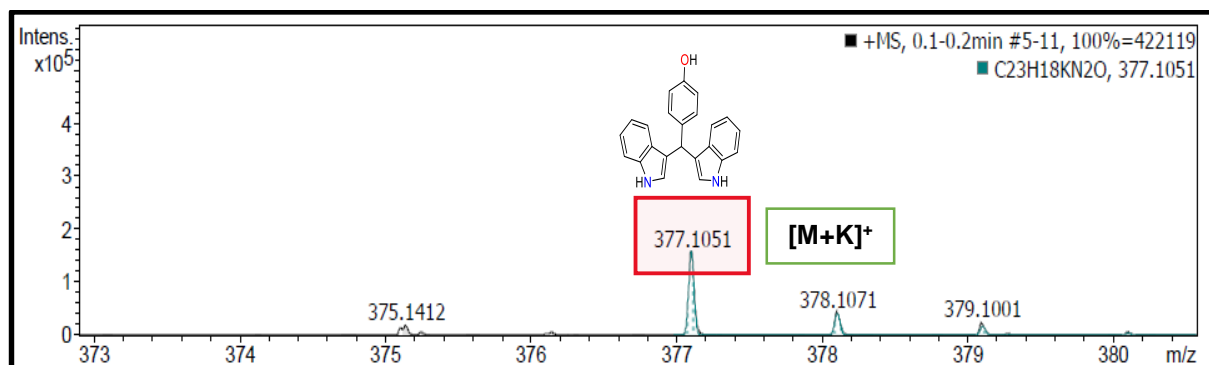


Figure S40: HRMS (ESI) spectrum of **2j**.

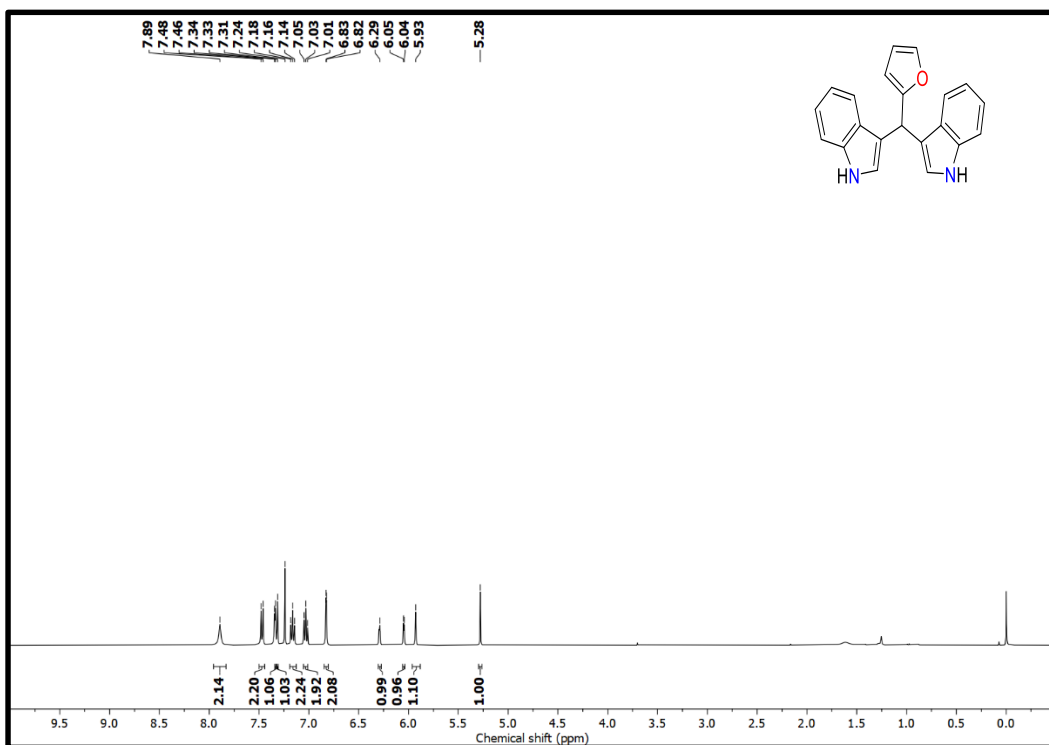


Figure S41: ^1H NMR spectrum of **2k** recorded in CDCl_3 .

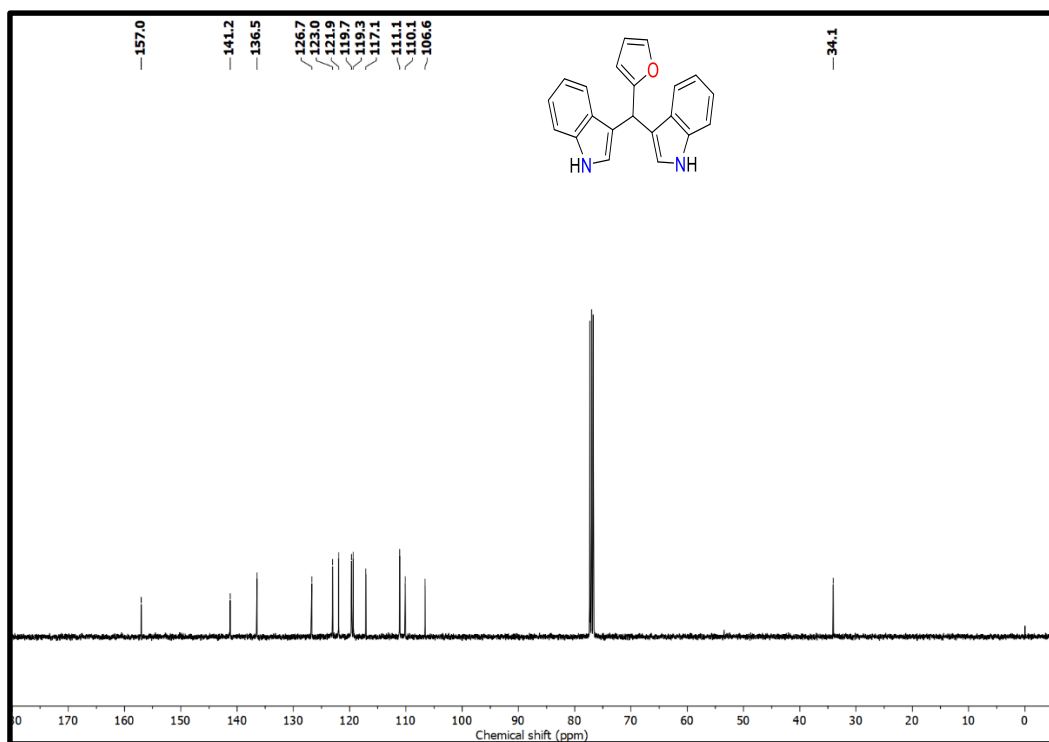


Figure S42: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2k** recorded in CDCl_3 .

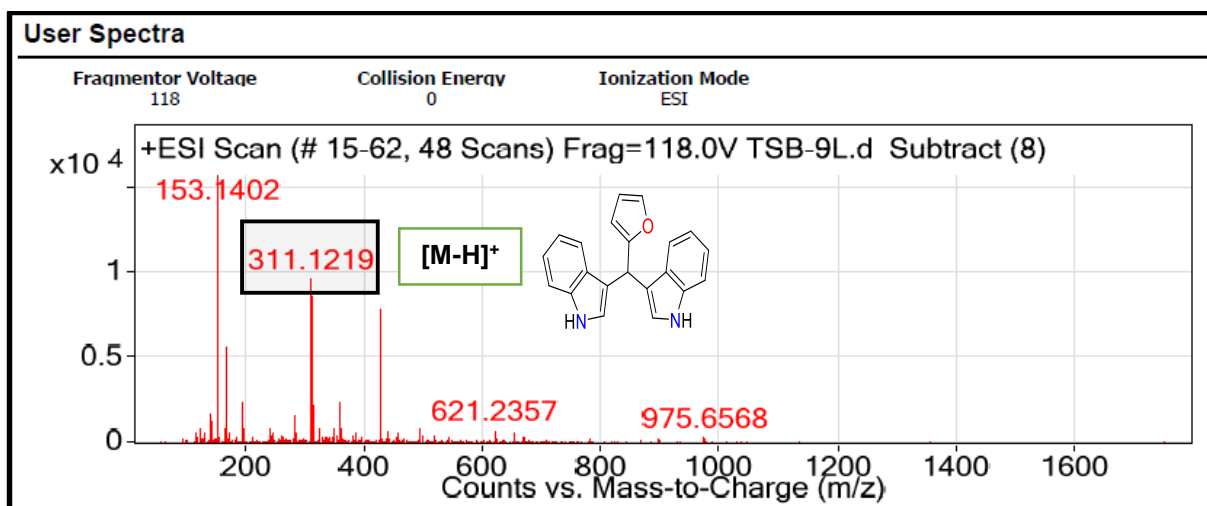


Figure S43: HRMS (ESI) spectrum of **2k**.

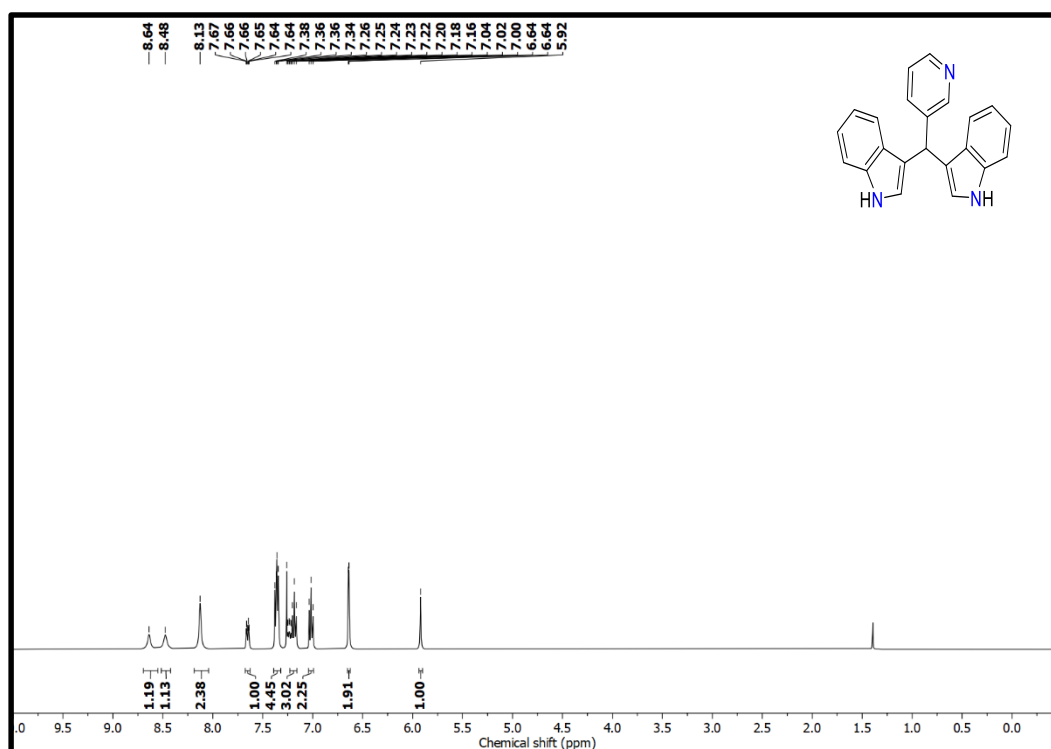


Figure S44: ^1H NMR spectrum of **2l** recorded in CDCl_3 .

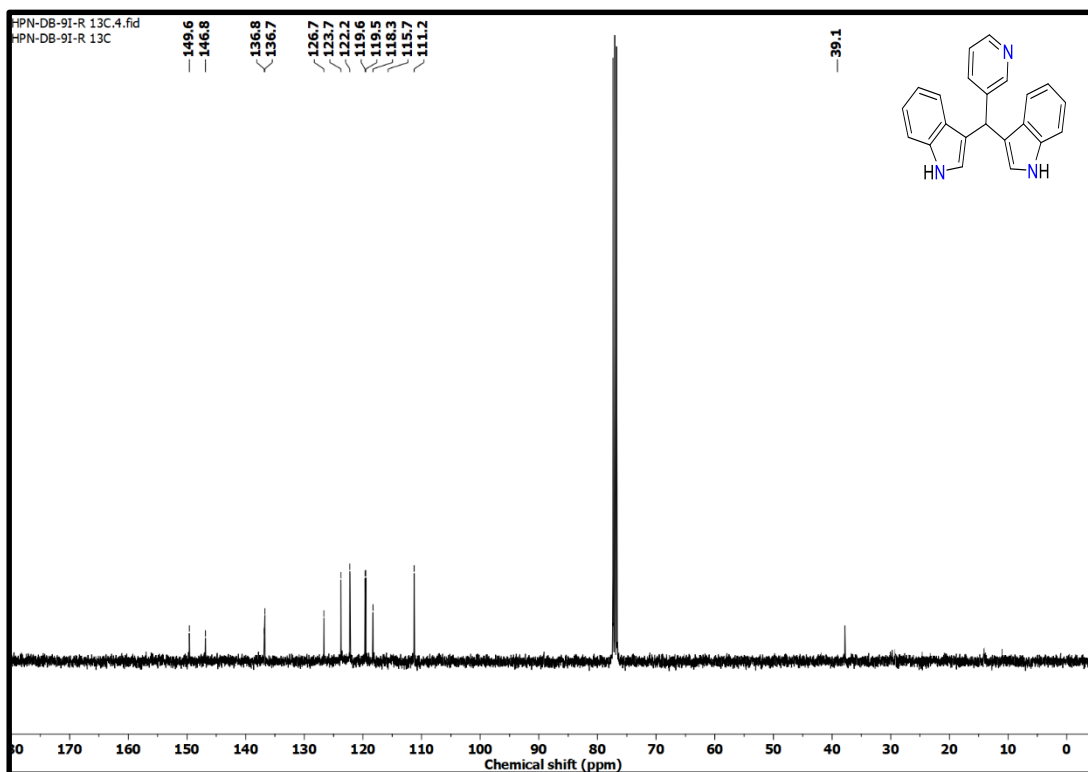


Figure S45: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2l** recorded in CDCl_3 .

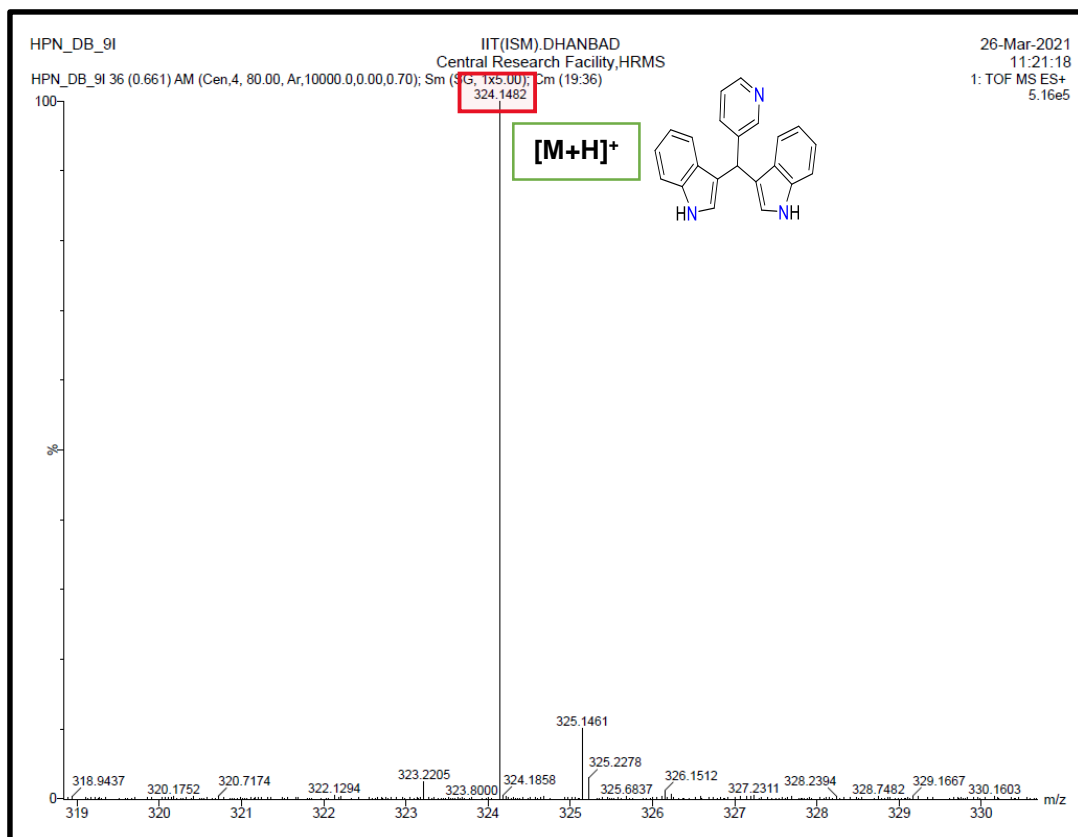


Figure S46: HRMS (ESI) spectrum of **2l**.

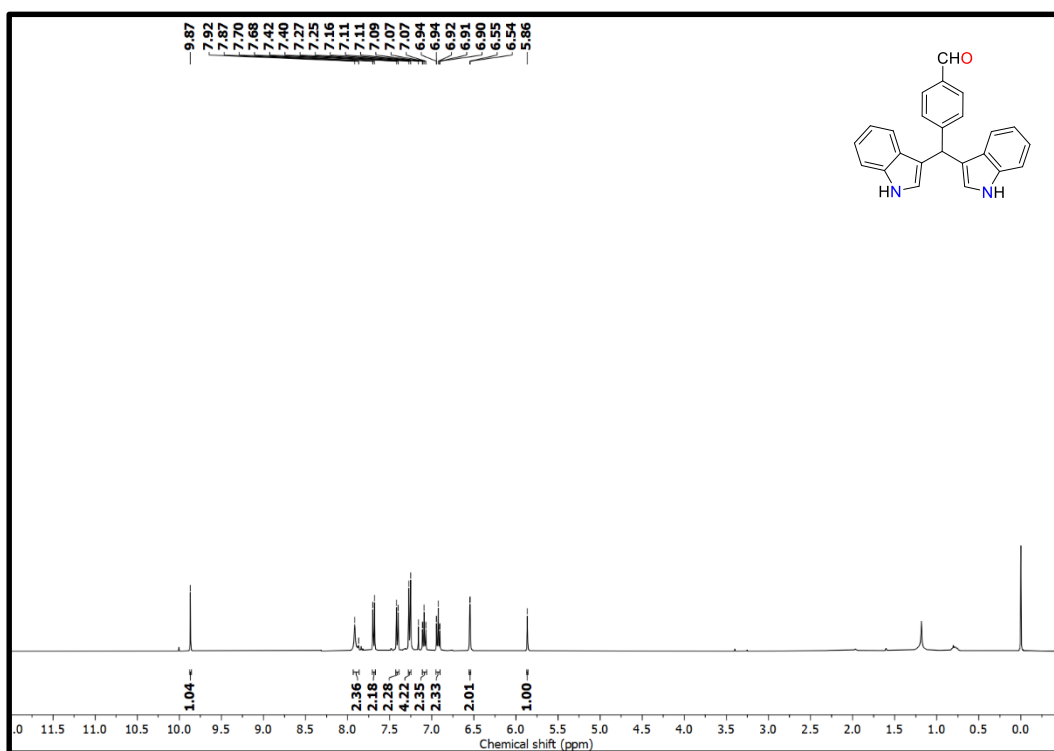


Figure S47: ^1H NMR spectrum of **2m** recorded in CDCl_3 .

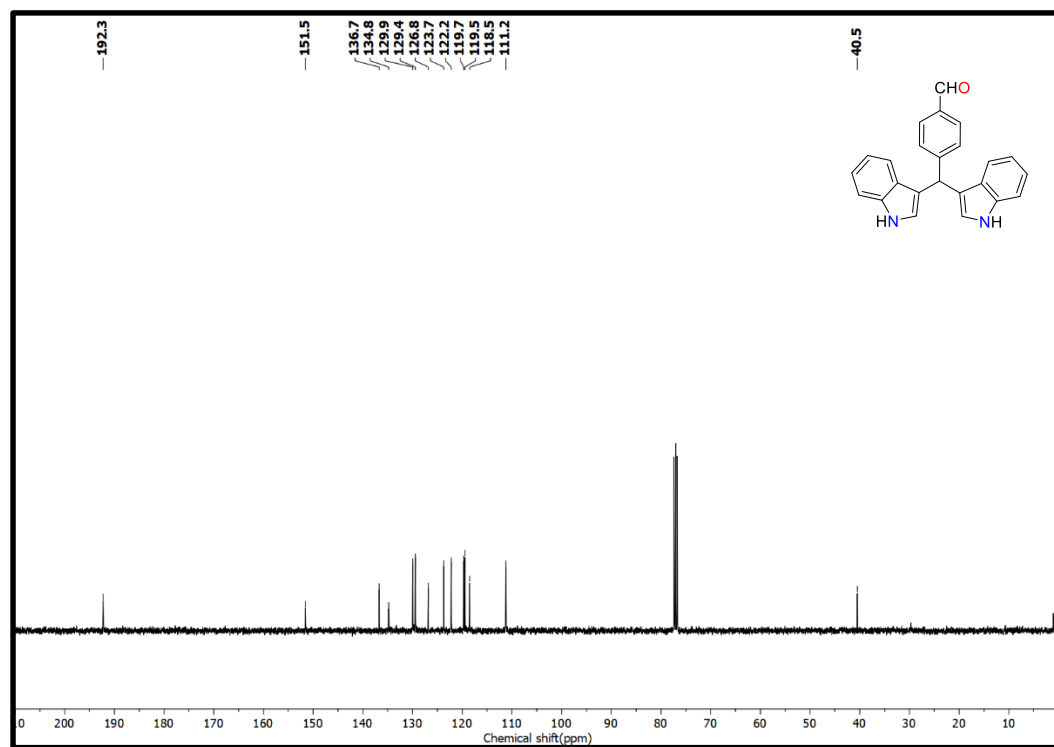


Figure S48: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2m** recorded in CDCl_3 .

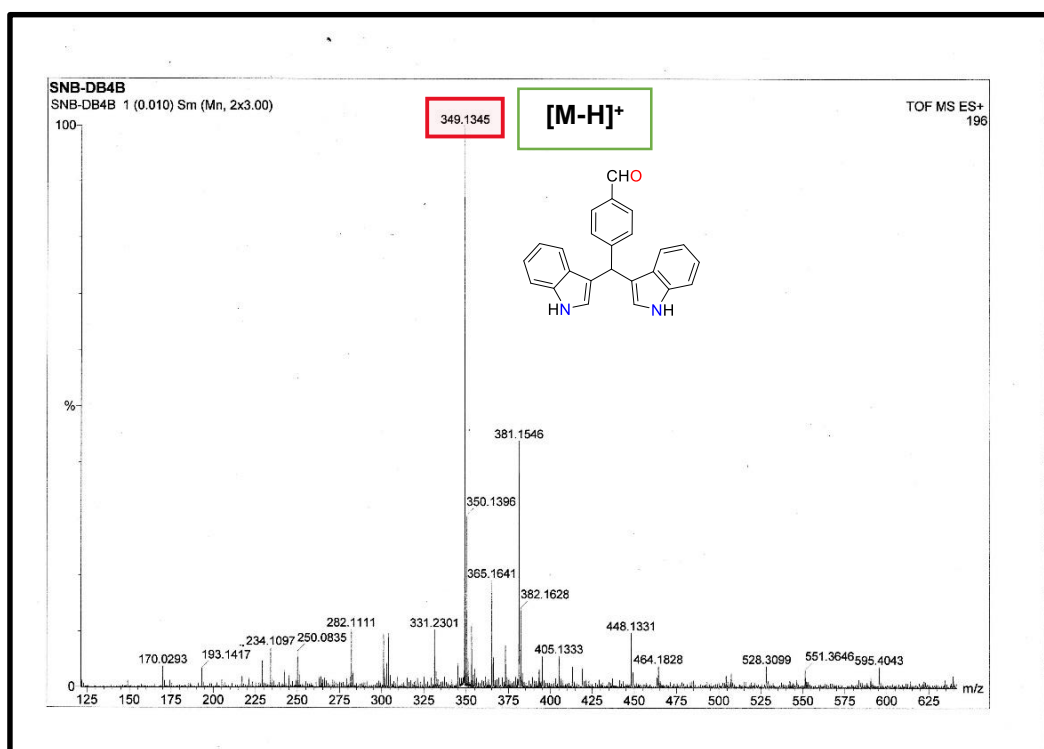


Figure S49: HRMS (ESI) spectrum of **2m**.

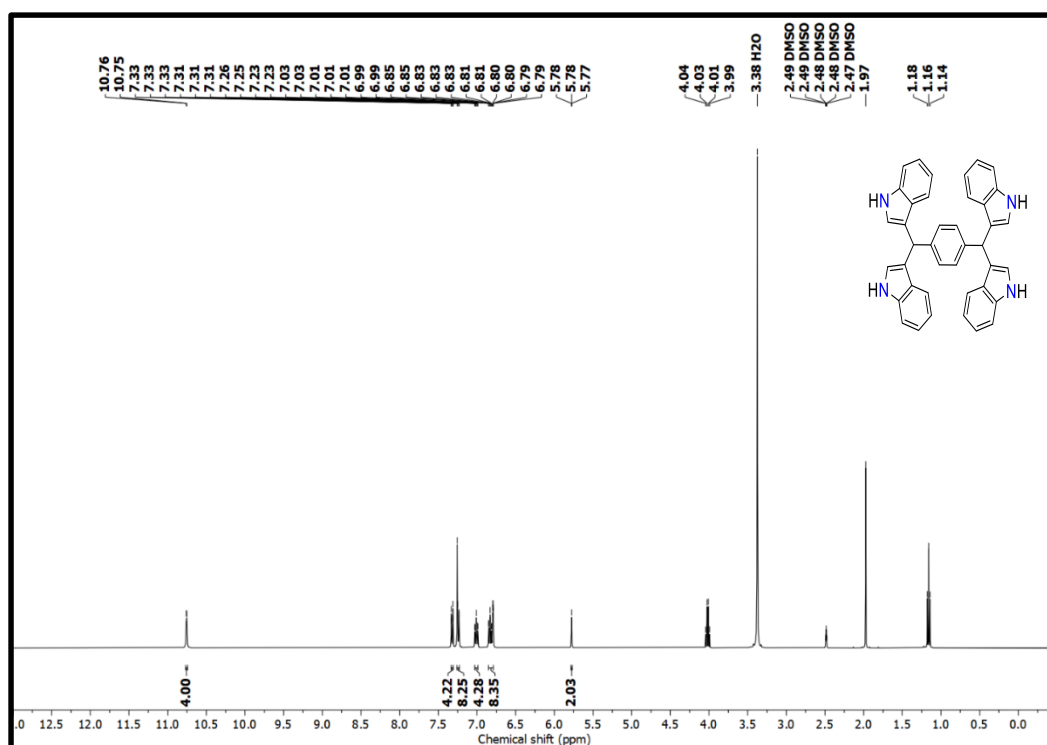


Figure S50: ¹H NMR spectrum of **2n** recorded in DMSO-*d*₆.

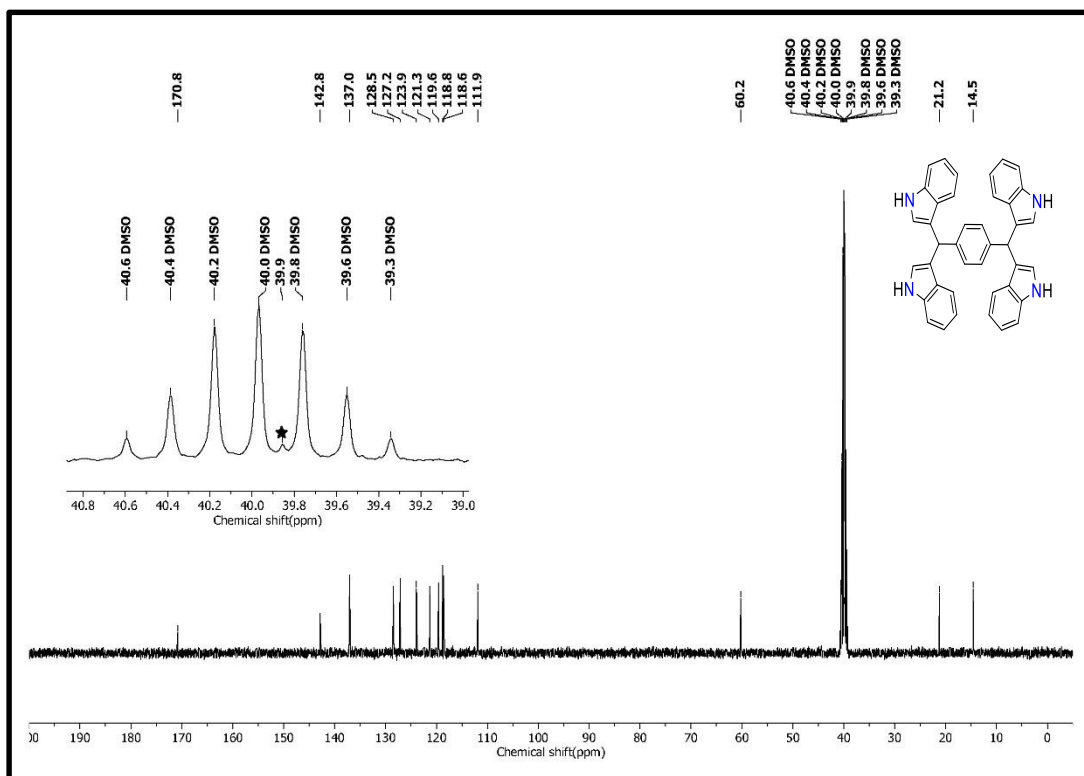


Figure S51: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2n** recorded in $\text{DMSO-}d_6$.

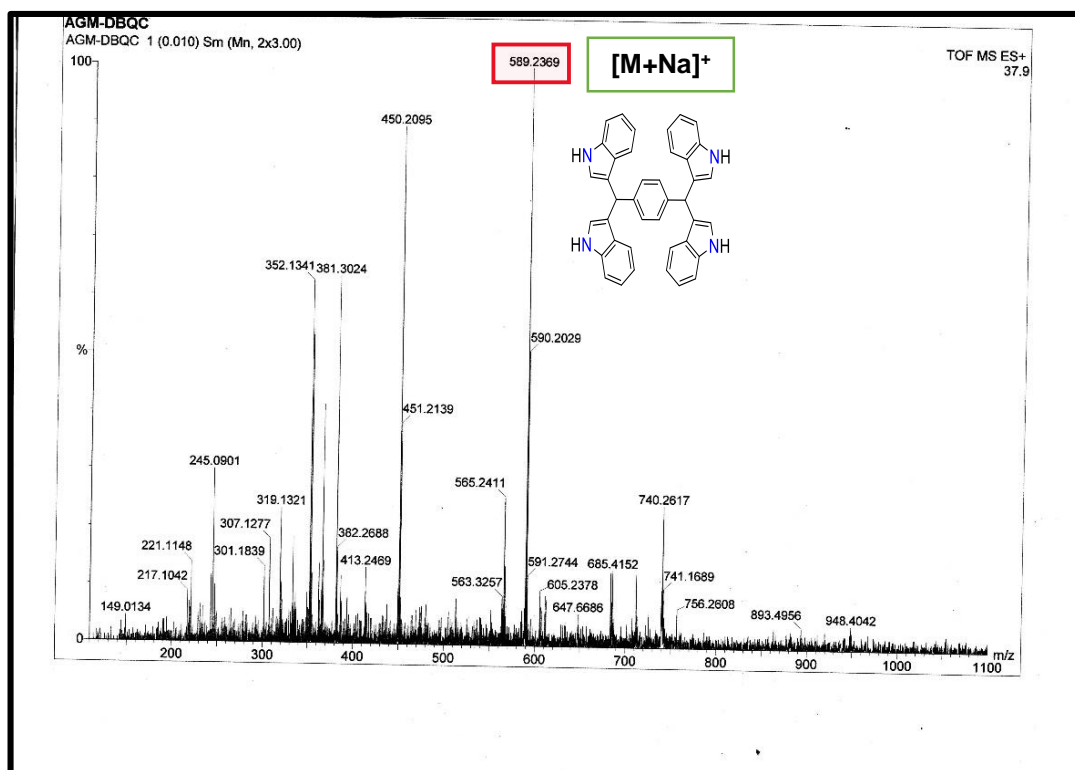


Figure S52: HRMS (ESI) spectrum of **2n**.

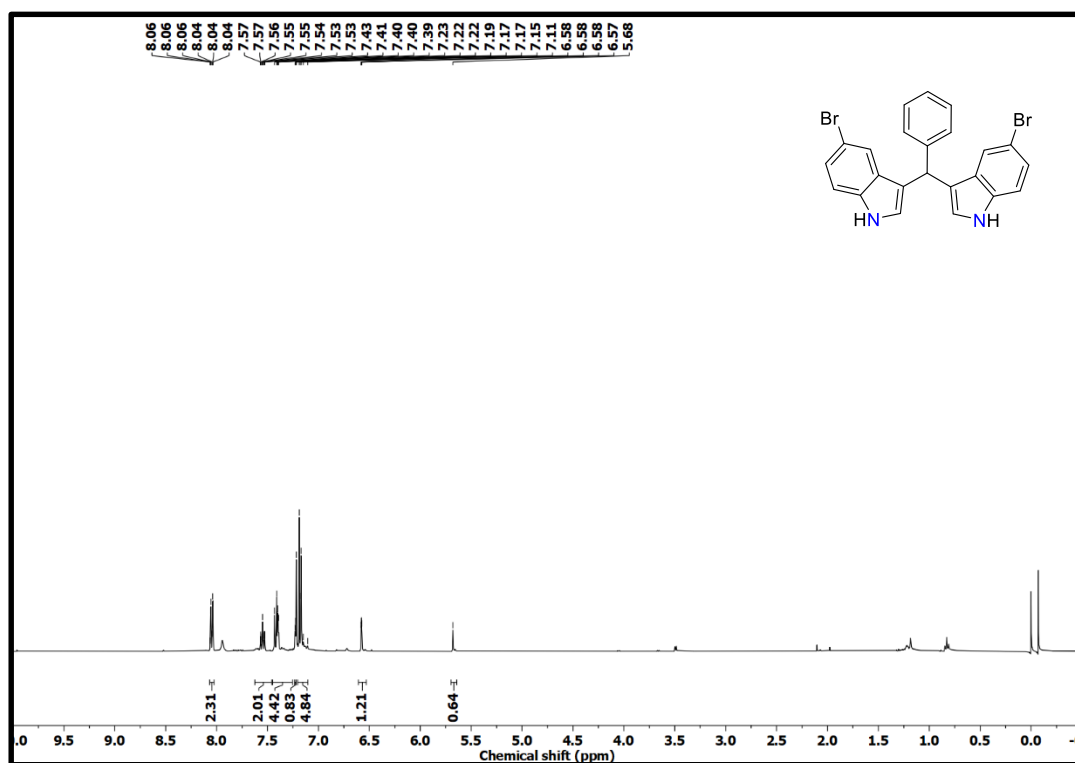


Figure S53: ¹H NMR spectrum of **2o** recorded in CDCl₃.

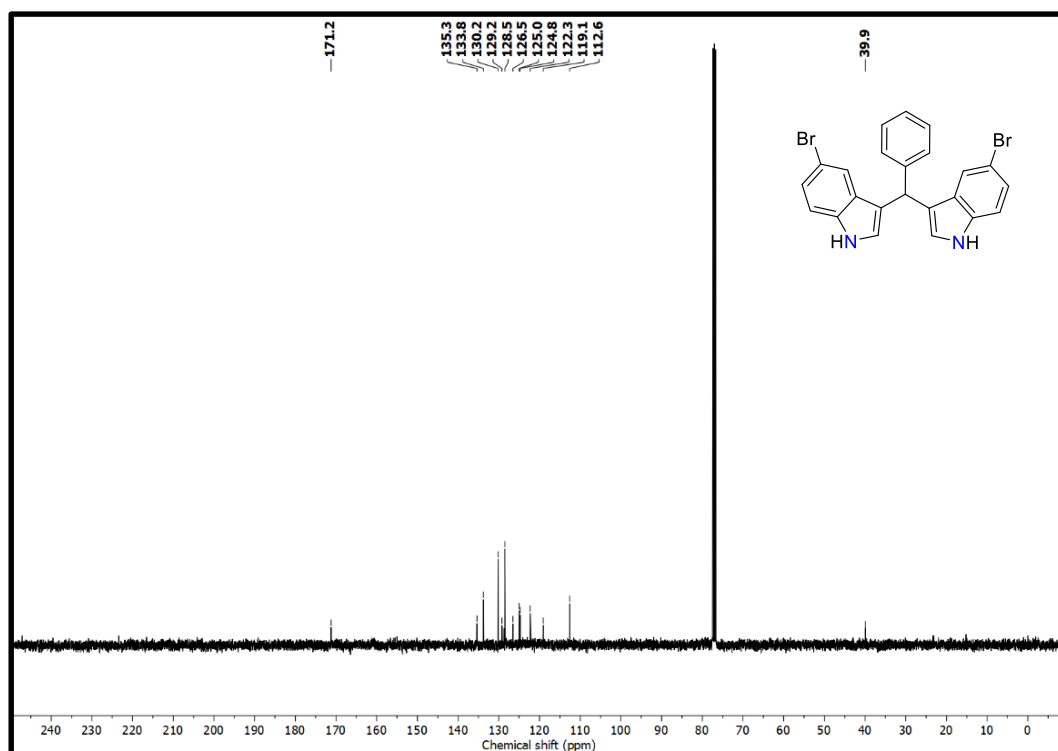


Figure S54: ¹³C{¹H} NMR spectrum of **2o** recorded in CDCl₃.

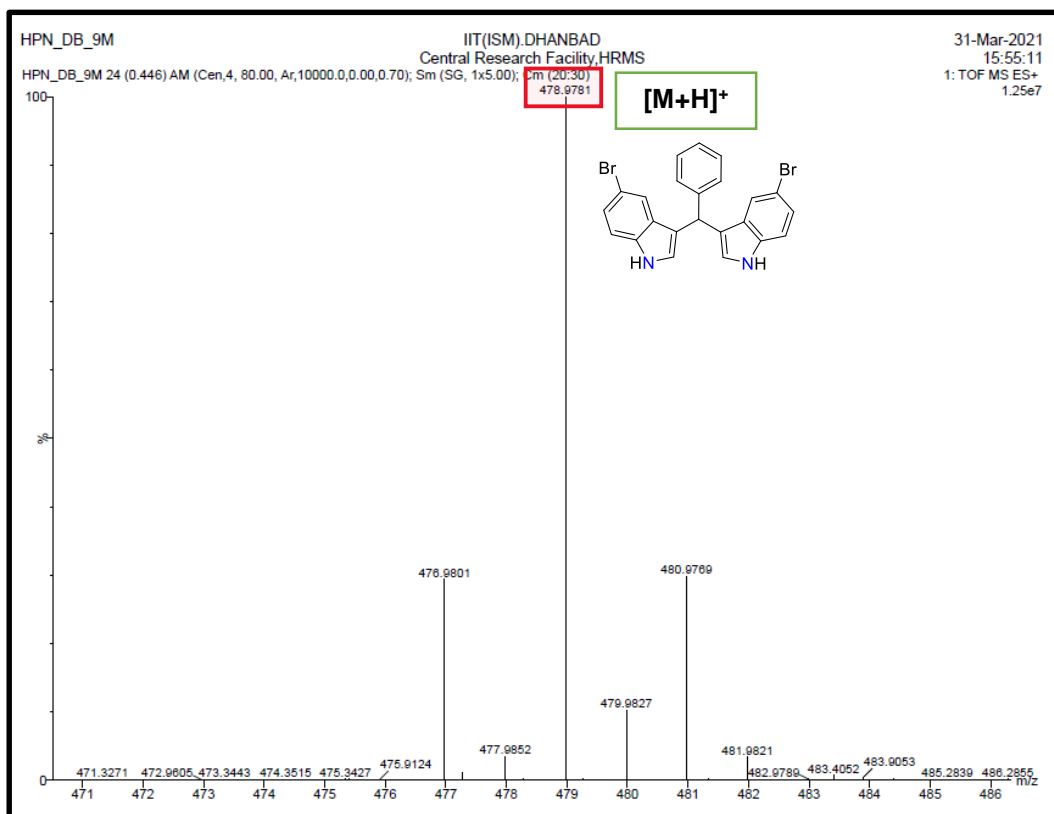


Figure S55: HRMS (ESI) spectrum of **2o**.

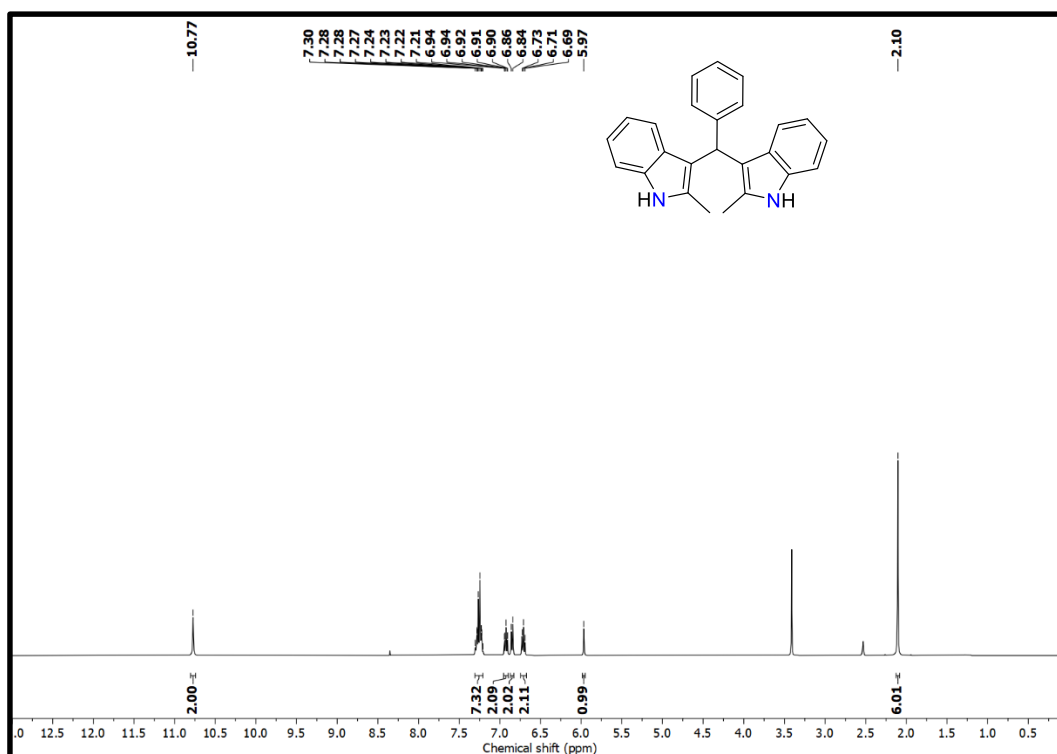


Figure S56: ¹H NMR spectrum of **2p** recorded in DMSO-*d*₆.

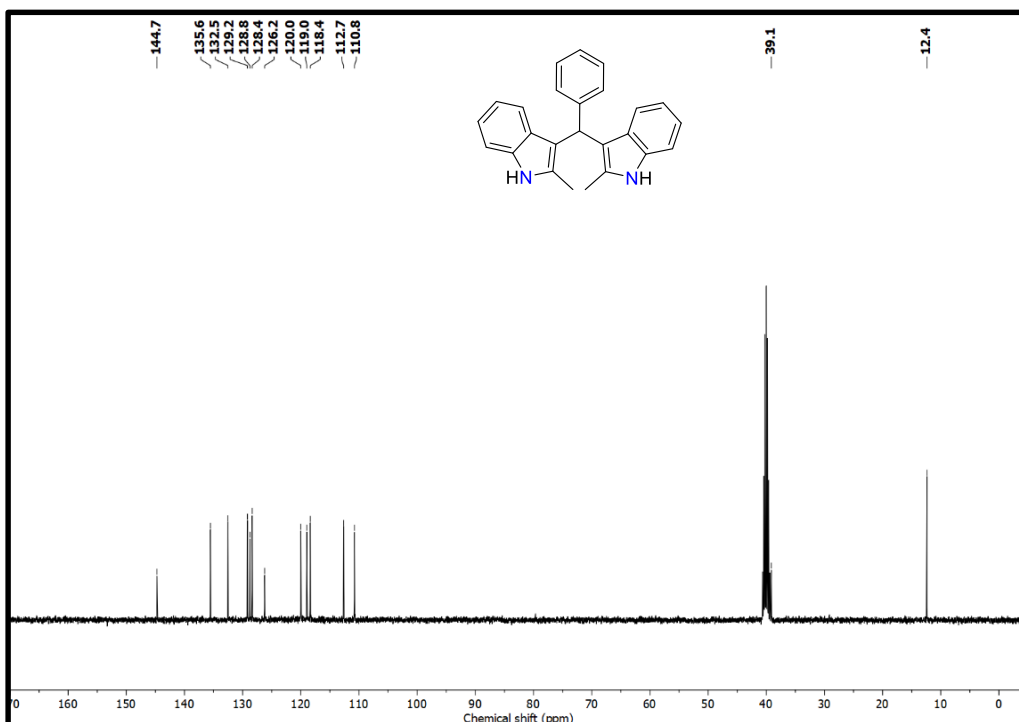


Figure S57: ¹³C{¹H} NMR spectrum of **2p** recorded in DMSO-*d*₆.

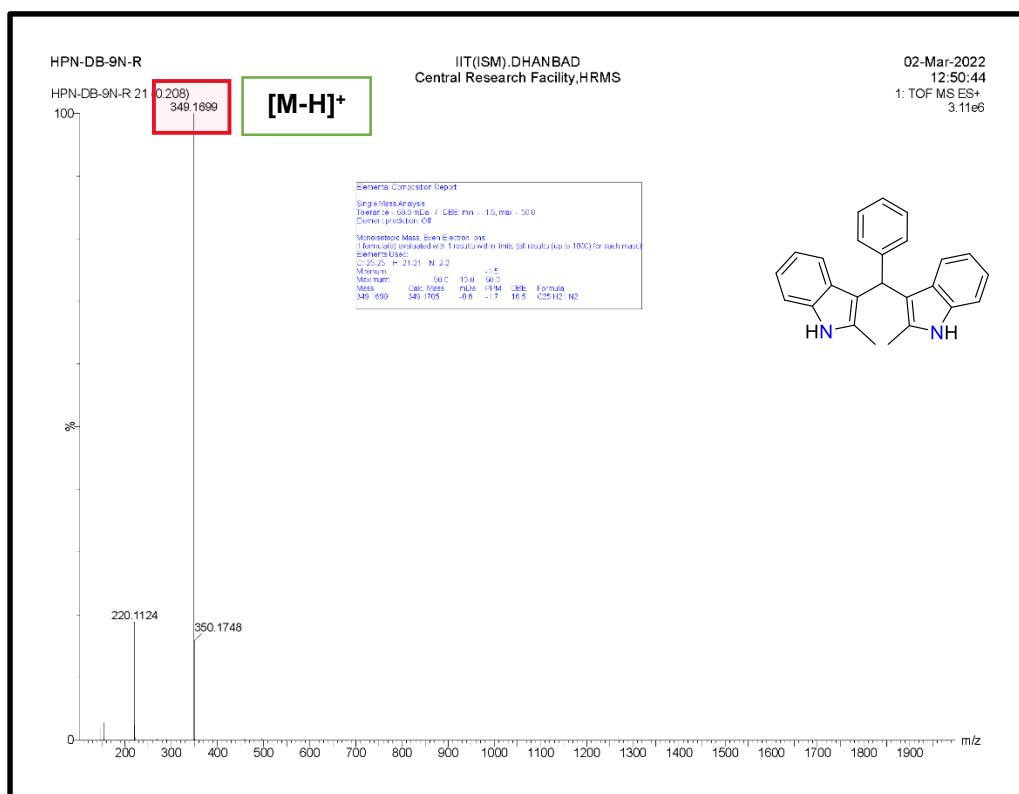


Figure S58: HRMS (ESI) spectrum of **2p**.

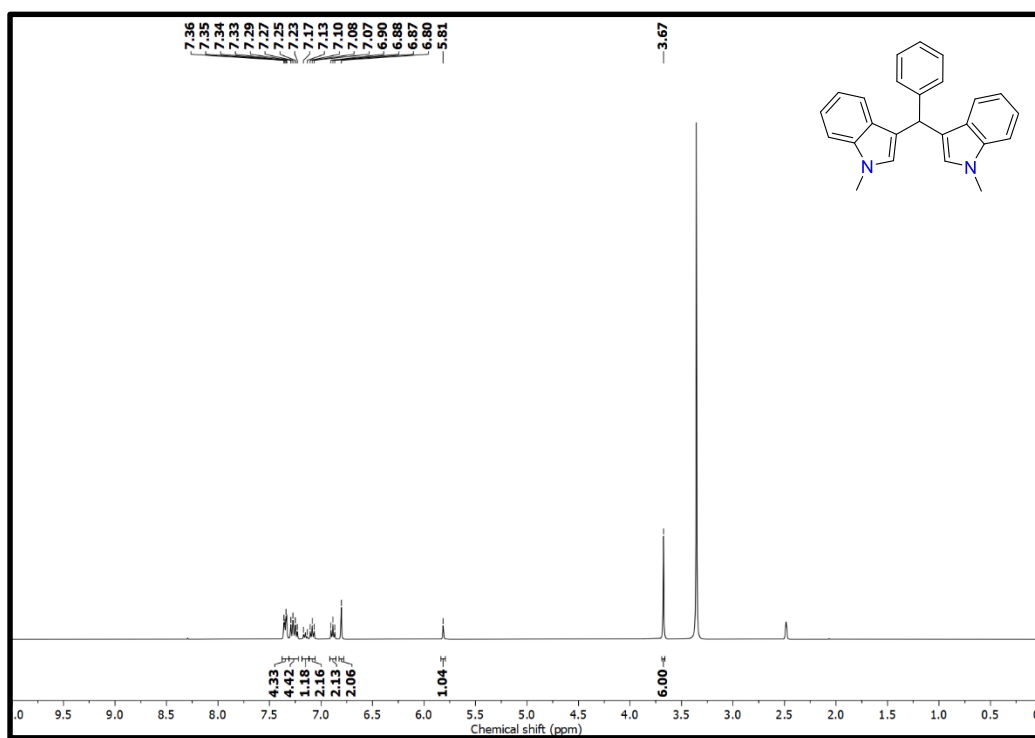


Figure S59: ^1H NMR spectrum of **2q** recorded in $\text{DMSO-}d_6$.

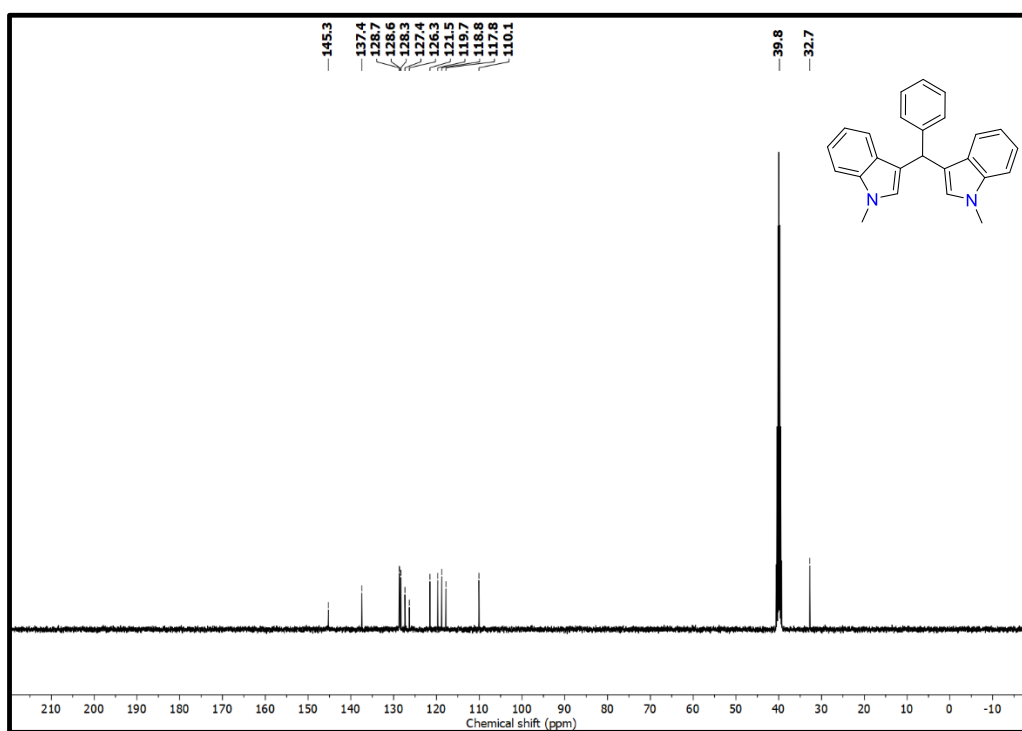


Figure S60: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2q** recorded in $\text{DMSO-}d_6$.

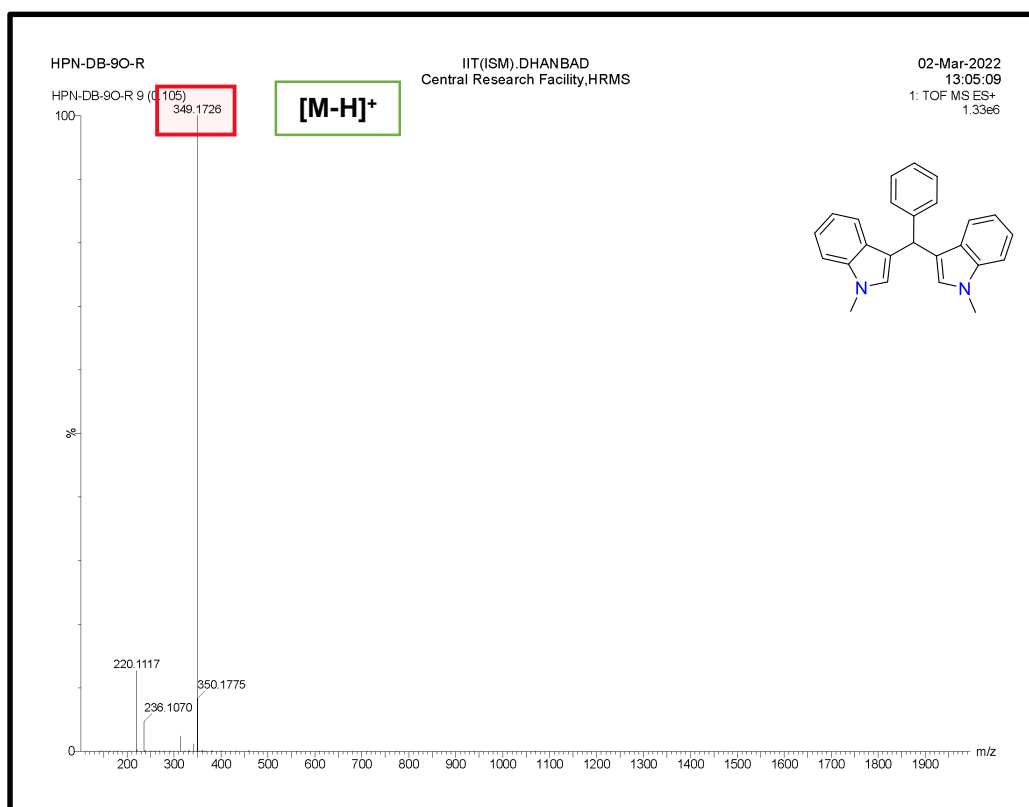


Figure S61: HRMS (ESI) spectrum of **2q**.

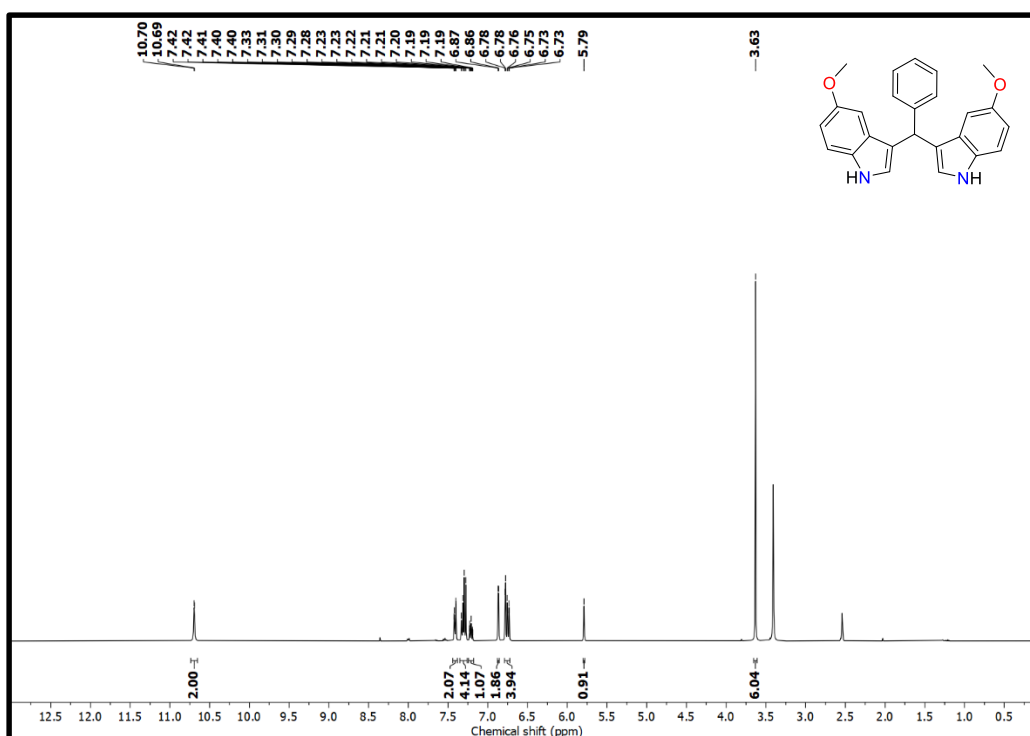


Figure S62: ¹H NMR spectrum of **2r** recorded in DMSO-*d*₆.

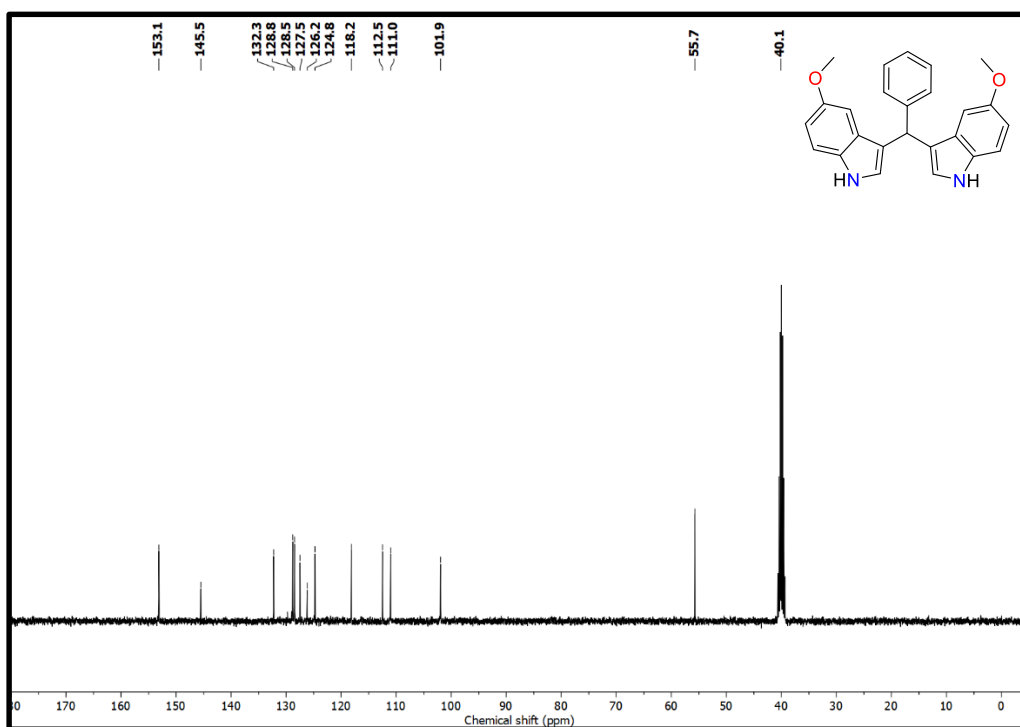


Figure S63: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **2r** recorded in $\text{DMSO-}d_6$.

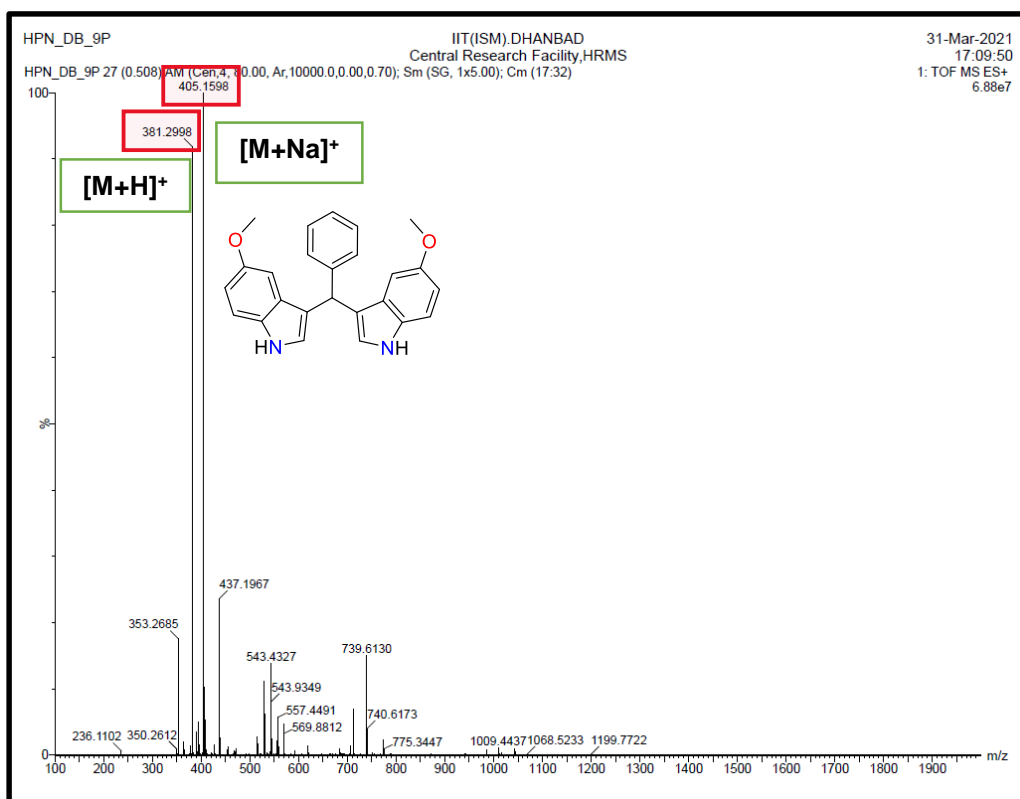


Figure S64: HRMS (ESI) spectrum of **2r**.

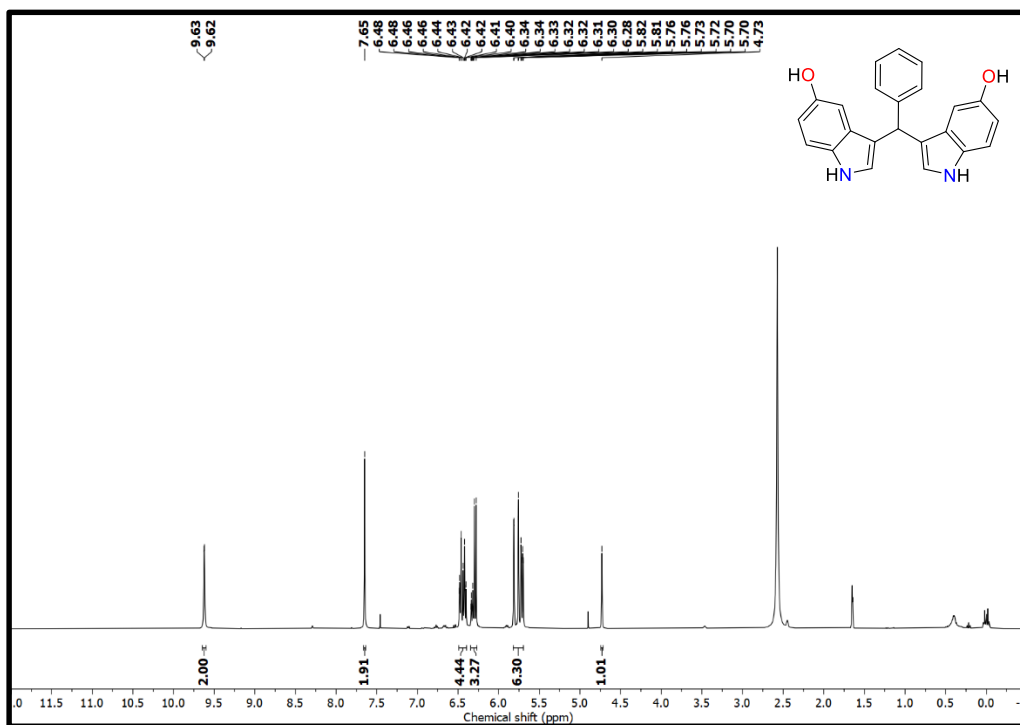


Figure S65: ^1H NMR spectrum of **2s** recorded in $\text{DMSO-}d_6$.

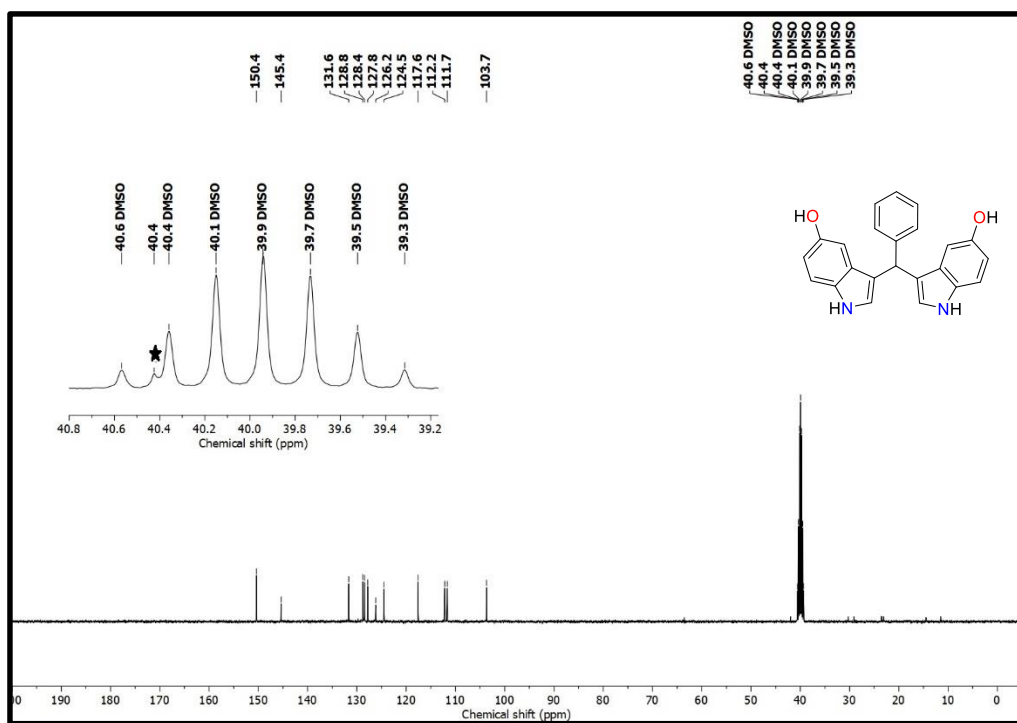


Figure S66: ^{13}C NMR spectrum of **2s** recorded in $\text{DMSO-}d_6$.

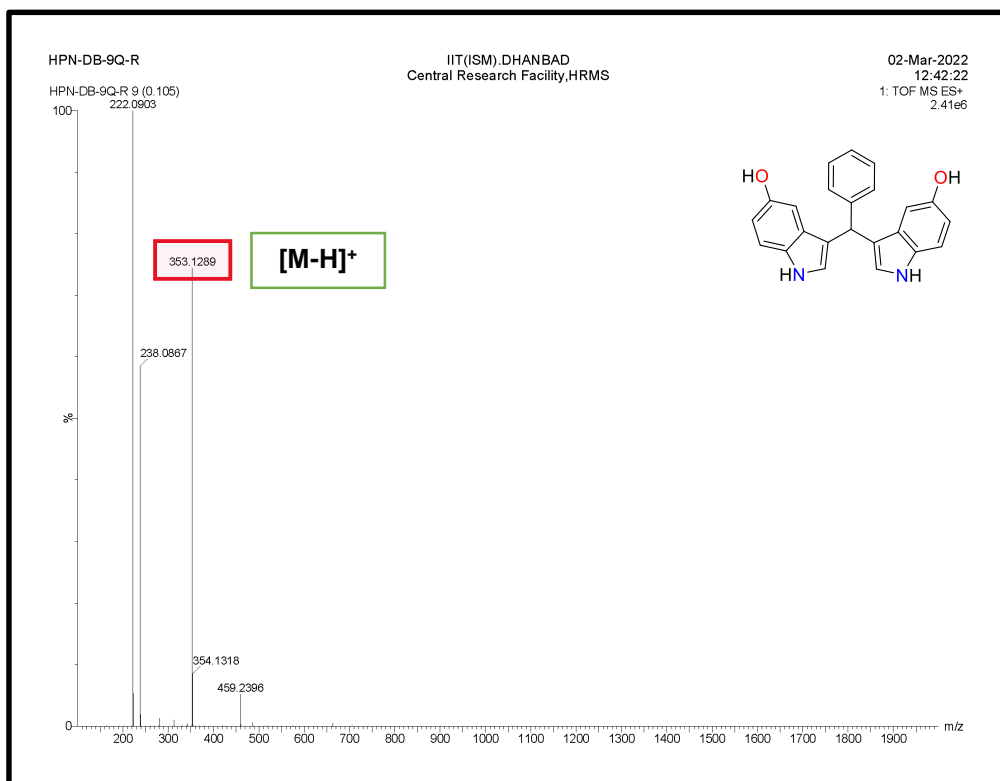
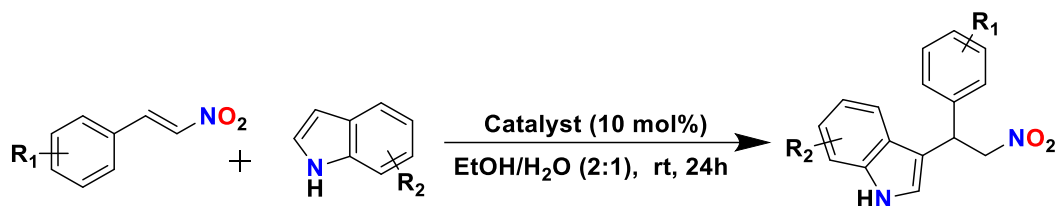
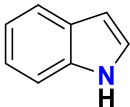
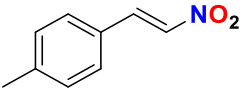
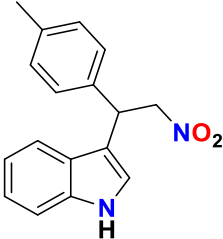
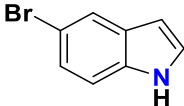
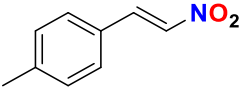
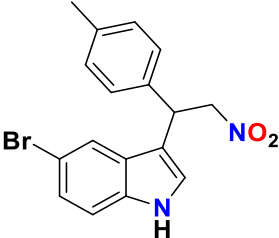
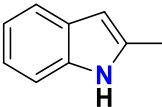
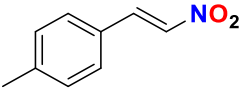
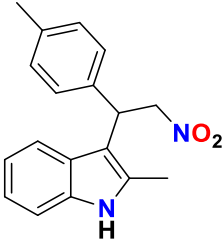
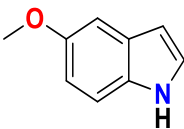
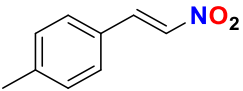
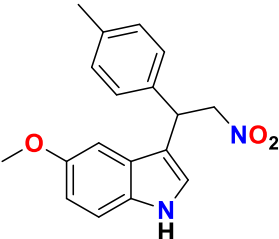
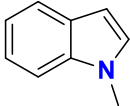
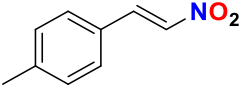
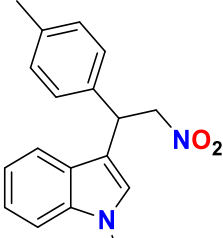
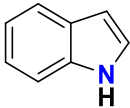
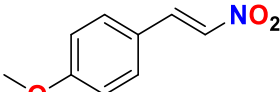
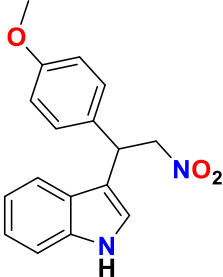


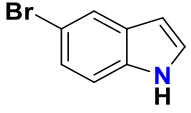
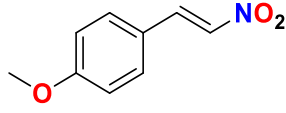
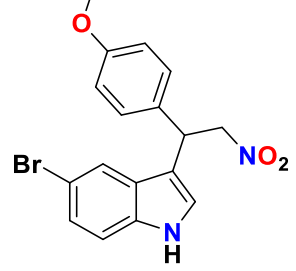
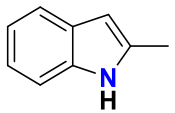
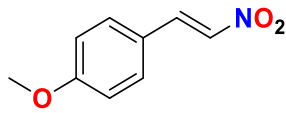
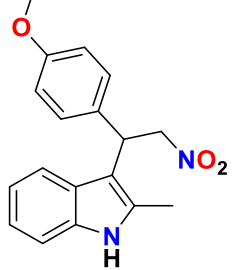
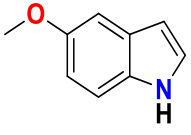
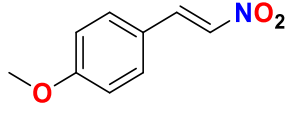
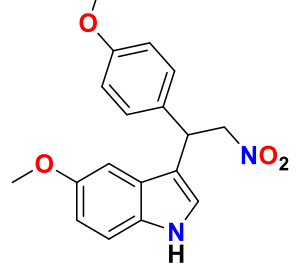
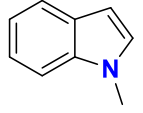
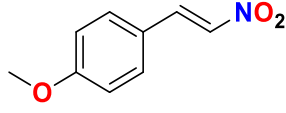
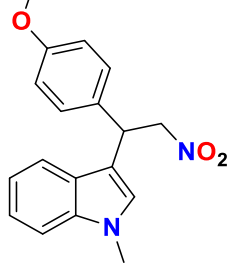
Figure S67: HRMS (ESI) spectrum of **2s**.

Table S2: Isolated yields and characterizations of Friedel-Crafts alkylation reaction of indoles with β -nitrostyrenes.

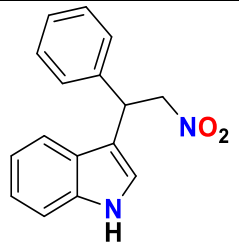


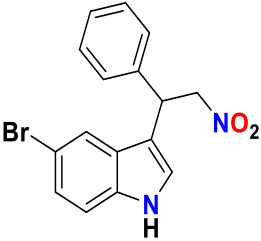
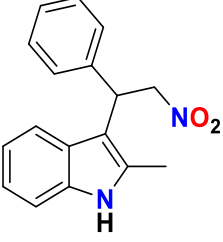
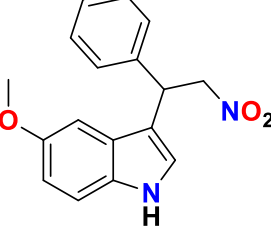
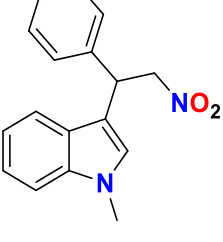
Entry	Indoles	β -nitrostyrenes	Product	Code	Yield (%)
1				3a	96
2				3b	94
3				3c	93
4				3d	91
5				3e	83

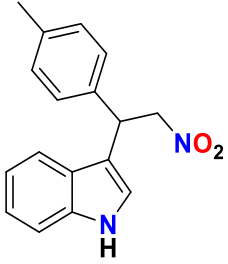
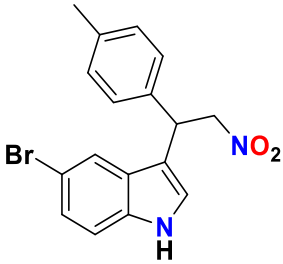
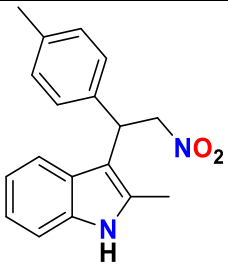
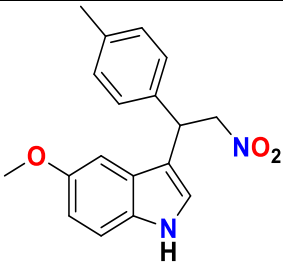
6				3f	92
7				3g	89
8				3h	87
9				3i	88
10				3j	80
11				3k	91

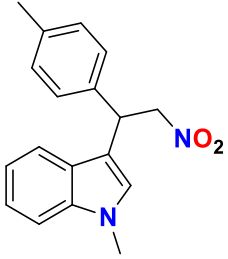
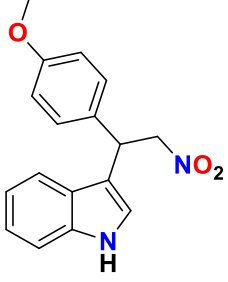
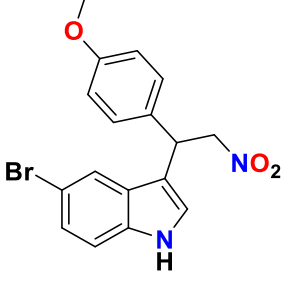
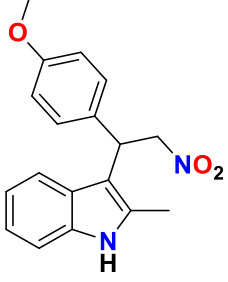
12				3l	87
13				3m	85
14				3n	83
15				3o	78

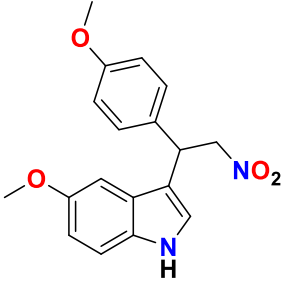
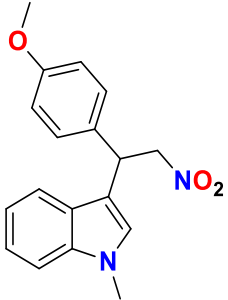
Characterization of isolated nitroalkylated indoles:

	<p>3a⁸: Red solid. $R_f = 0.37$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.90 (s, 1H, -NH), 7.32 (d, $J = 8.0$ Hz, 1H, ArH), 7.20 – 7.10 (m, 6H, ArH), 7.07 (t, $J = 7.6$ Hz, 1H, ArH), 6.95 (t, $J = 7.5$ Hz, 1H, ArH), 6.80 (s, 1H, ArH), 5.05 (t, $J = 8.0$ Hz, 1H, -CH₂), 4.90 (dd, $J = 12.5, 7.6$ Hz, 1H, -CH₂CH), 4.78 (dd, $J = 12.5, 8.4$ Hz, 1H, -CH₂CH). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 139.3, 136.5, 128.9, 127.8, 127.6, 126.1, 122.7, 121.7, 119.9, 118.9, 114.2, 111.5, 79.6, 41.6. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{16}\text{H}_{15}\text{N}_2\text{O}_2$ 267.1134, found 267.1211. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2\text{Na}$ 289.0953, found 289.0997.</p>
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	<p>3b⁸: Red sticky mass. $R_f = 0.38$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.21 (s, 1H, -NH), 7.49 (dd, $J = 25.4, 1.7$ Hz, 1H, ArH), 7.30 – 7.21 (m, 6H, ArH), 7.16 (t, $J = 8.9$ Hz, 1H, ArH), 6.99 (d, $J = 2.1$ Hz, 1H, ArH), 5.08 (t, $J = 8.0$ Hz, 1H, -CH₂), 4.97 (dd, $J = 12.5, 8.0$ Hz, 1H, -CH₂CH), 4.88 (dd, $J = 12.5, 8.0$ Hz, 1H, -CH₂CH). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 138.8, 135.1, 129.1, 127.9, 127.8, 127.7, 125.6, 122.9, 121.4, 113.9, 113.2, 113.0, 79.5, 41.3. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{16}\text{H}_{14}\text{N}_2\text{O}_2\text{Br}$ 345.0238, found 345.0250.</p>
	<p>3c⁸: Brown sticky mass. $R_f = 0.39$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.83 (s, 1H, -NH), 7.35 (d, $J = 7.7$ Hz, 1H, ArH), 7.28 (d, $J = 6.7$ Hz, 2H, ArH), 7.24 (d, $J = 7.9$ Hz, 2H, ArH), 7.19 (d, $J = 8.9$ Hz, 2H, ArH), 7.08 (t, $J = 7.5$ Hz, 1H, ArH), 7.00 (t, $J = 7.5$ Hz, 1H, ArH), 5.22 – 5.02 (m, 3H, -CH₂, -CH₂CH), 2.29 (s, 3H, -CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 139.6, 135.5, 133.0, 128.8, 127.4, 127.1, 126.9, 121.3, 119.8, 118.6, 110.8, 108.8, 78.7, 40.5, 12.0. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_2\text{Na}$ 303.1109, found 303.1098.</p>
	<p>3d⁸: Brown sticky mass. $R_f = 0.37$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.04 (s, 1H, -NH), 7.29 – 7.14 (m, 6H, ArH), 6.90 (d, $J = 2.5$ Hz, 1H, ArH), 6.81 (m, 2H, ArH), 5.09 (t, $J = 8.0$ Hz, 1H, -CH₂), 4.98 (dd, $J = 12.4, 7.5$ Hz, 1H, -CH₂), 4.88 (dd, $J = 12.4, 8.4$ Hz, 1H, -CH₂CH), 3.74 (s, 3H, -OCH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 154.2, 139.2, 131.7, 128.9, 127.8, 127.6, 126.6, 122.4, 114.0, 112.7, 112.2, 100.9, 79.5, 55.9, 41.6. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{17}\text{H}_{16}\text{N}_2\text{O}_3\text{Na}$ 319.1059, found 319.1052.</p>
	<p>3e⁸: Red brown solid. $R_f = 0.38$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.44 (d, $J = 8.0$ Hz, 1H, ArH), 7.31 (q, $J = 7.1$ Hz, 5H, ArH), 7.25 – 7.19 (m, 2H, ArH), 7.10 – 7.01 (m, 1H, ArH), 6.84 (s, 1H, ArH), 5.16 (t, $J = 8.0$ Hz, 1H, -CH₂), 5.02 (dd, $J = 12.5, 7.5$ Hz, 1H, -CH₂), 4.91 (dd, $J = 12.5, 8.5$ Hz, 1H, -CH₂CH), 3.70 (s, 3H, -NCH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 139.4, 137.3, 128.9, 127.8, 127.5, 126.6, 126.4, 122.3, 119.5, 119.0, 112.8, 109.6, 79.5, 41.6, 32.9. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{17}\text{N}_2\text{O}_2$ 281.1290, found 281.1252.</p>

	<p>3f⁹: Red sticky mass. $R_f = 0.32$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.03 (s, 1H, -NH), 7.43 (d, $J = 8.0$ Hz, 1H, ArH), 7.31 (d, $J = 8.2$ Hz, 1H, ArH), 7.20 – 7.15 (m, 3H, ArH), 7.12 – 7.04 (m, 3H, ArH), 6.95 (d, $J = 1.9$ Hz, 1H, ArH), 5.13 (t, $J = 8.0$ Hz, 1H, $-\text{CH}_2$), 5.01 (dd, $J = 12.4, 7.6$ Hz, 1H, $-\text{CH}_2$), 4.89 (dd, $J = 12.4, 8.4$ Hz, 1H, $-\text{CH}_2\text{CH}$), 2.29 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 137.2, 136.4, 136.1, 129.6, 127.6, 126.1, 122.6, 121.5, 119.9, 118.9, 114.5, 111.3, 79.6, 41.2, 21.0. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{17}\text{N}_2\text{O}_2$ 281.129, found 281.1265.</p>
	<p>3g¹⁰: Red sticky mass. $R_f = 0.35$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.27 (s, 1H, -NH), 7.56 (d, $J = 1.9$ Hz, 1H, ArH), 7.24 (d, $J = 1.9$ Hz, 1H, ArH), 7.21 – 7.10 (m, 5H, ArH), 7.01 – 6.95 (m, 1H, ArH), 5.08 (t, $J = 8.0$ Hz, 1H, $-\text{CH}_2$), 4.99 (dd, $J = 12.4, 8.0$ Hz, 1H, $-\text{CH}_2$), 4.88 (dd, $J = 12.3, 8.0$ Hz, 1H, $-\text{CH}_2\text{CH}$), 2.32 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3): $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 137.5, 135.7, 135.1, 129.8, 127.9, 127.6, 125.5, 122.8, 121.4, 114.1, 113.2, 113.0, 79.6, 41.0, 21.1. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{16}\text{BrN}_2\text{O}_2$ 358.0395; observed 359.037.</p>
	<p>3h¹¹: Red solid. $R_f = 0.37$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.85 (s, 1H, -NH), 7.36 (d, $J = 7.9$ Hz, 1H, ArH), 7.21 – 7.16 (m, 3H, ArH), 7.11 – 7.06 (m, 3H, ArH), 7.03 – 6.98 (m, 1H, ArH), 5.18 (dd, $J = 10.0, 5.9$ Hz, 1H, $-\text{CH}_2$), 5.15 – 5.10 (m, 1H, $-\text{CH}_2$), 5.07 (dd, $J = 10.0, 8.0$ Hz, 1H, $-\text{CH}_2\text{CH}$), 2.31 (s, 3H, $-\text{CH}_3$), 2.28 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 136.8, 136.5, 135.4, 132.9, 129.5, 127.2, 126.9, 121.3, 119.7, 118.7, 110.8, 108.9, 78.7, 40.1, 21.0, 12.0. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}_2$ 295.1447, found 295.1460.</p>
	<p>3i: Red sticky mass. $R_f = 0.35$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.05 (s, 1H, -NH), 7.25 – 7.17 (m, 3H, ArH), 7.16 – 7.07 (m, 2H, ArH), 7.00 – 6.90 (m, 1H, ArH), 6.87 (d, $J = 8.4$ Hz, 2H, ArH), 5.11 (t, $J = 8.0$ Hz, 1H, $-\text{CH}_2$), 5.01 (dd, $J = 12.3, 7.5$ Hz, 1H, $-\text{CH}_2$), 4.90 (dd, $J = 12.3, 8.5$ Hz, 1H, $-\text{CH}_2\text{CH}$), 3.80 (s, 3H, $-\text{OCH}_3$), 2.32 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 154.1, 137.2, 136.1, 131.6, 129.6, 127.7, 126.6, 122.3, 114.1, 112.6, 112.2, 100.8, 79.6, 55.9, 41.2, 21.1. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}_3$ 311.1396, found 311.1422.</p>

	<p>3j¹²: Brown solid. $R_f = 0.37$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.45 (d, $J = 8.0$ Hz, 1H, ArH), 7.27 (d, $J = 8.2$ Hz, 1H, ArH), 7.23 – 7.18 (m, 3H, ArH), 7.11 (d, $J = 7.9$ Hz, 2H, ArH), 7.06 (t, $J = 8.0$ Hz, 1H, ArH), 6.83 (s, 1H, ArH), 5.13 (t, $J = 8.0$ Hz, 1H, $-\text{CH}_2$), 5.00 (dd, $J = 12.4, 7.5$ Hz, 1H, $-\text{CH}_2$), 4.88 (dd, $J = 12.4, 8.6$ Hz, 1H, $-\text{CH}_2\text{CH}$), 3.69 (s, 3H, $-\text{NCH}_3$), 2.29 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 137.3, 137.2, 136.4, 129.6, 127.6, 126.6, 126.4, 122.2, 119.5, 119.0, 113.0, 109.6, 79.7, 41.2, 32.9, 21.1. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}_2$ 295.1447, found 295.1425.</p>
	<p>3k¹³: Red solid. $R_f = 0.38$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.11 (s, 1H, $-\text{NH}$), 7.44 (d, $J = 7.9$ Hz, 1H, ArH), 7.37 (d, $J = 8.2$ Hz, 1H, ArH), 7.26 – 7.17 (m, 3H, ArH), 7.10 – 7.02 (m, 2H, ArH), 6.87 – 6.83 (m, 2H, ArH), 5.14 (t, $J = 7.9$ Hz, 1H, $-\text{CH}_2$), 5.05 (dd, $J = 12.3, 7.5$ Hz, 1H, $-\text{CH}_2$), 4.90 (dd, $J = 12.2, 8.4$ Hz, 1H, $-\text{CH}_2\text{CH}$), 3.77 (s, 3H, $-\text{OCH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 158.9, 136.5, 131.7, 128.8, 126.1, 122.7, 121.4, 120.0, 119.0, 114.8, 114.3, 111.4, 79.8, 55.3, 40.9. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{17}\text{H}_{17}\text{N}_2\text{O}_3$ 297.1239, found 297.1221.</p>
	<p>3l¹⁴: Orange solid. $R_f = 0.34$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.15 (s, 1H, $-\text{NH}$), 7.57 – 7.46 (m, 1H, ArH), 7.26 – 7.17 (m, 4H, ArH), 7.05 (d, $J = 1.7$ Hz, 1H, ArH), 6.93 – 6.76 (m, 2H, ArH), 5.07 (t, $J = 7.9$ Hz, 1H, $-\text{CH}_2$), 4.99 (dd, $J = 12.2, 7.9$ Hz, 1H, $-\text{CH}_2$), 4.87 (dd, $J = 12.2, 7.9$ Hz, 1H, $-\text{CH}_2\text{CH}$), 3.78 (s, 3H, $-\text{OCH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 159.0, 135.1, 130.7, 128.7, 127.9, 125.7, 122.6, 121.6, 114.5, 114.4, 113.3, 112.8, 79.6, 55.3, 40.6. HRMS (ESI) m/z calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{17}\text{H}_{15}\text{BrN}_2\text{O}_3\text{Na}$ 397.0164, found 397.0198.</p>
	<p>3m¹⁵: Red sticky mass. $R_f = 0.36$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.88 (s, 1H, $-\text{NH}$), 7.36 (d, $J = 7.9$ Hz, 1H, ArH), 7.27 – 7.22 (m, 3H, ArH), 7.13 – 7.07 (m, 1H, ArH), 7.02 (m, 1H, ArH), 6.86 – 6.78 (m, 2H, ArH), 5.21 – 5.04 (m, 3H, $-\text{CH}_2$, $-\text{CH}_2\text{CH}$), 3.76 (s, 3H, $-\text{OCH}_3$), 2.37 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 158.5, 135.4, 132.7, 131.5, 128.4, 126.9, 121.3, 119.7, 118.7, 114.1, 110.7, 109.1, 78.9, 55.3, 39.8, 12.1. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}_3$ 311.1396, found 311.1392.</p>

	<p>3n¹³: Light pink solid. $R_f = 0.37$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.99 (s, 1H, -NH), 7.28 – 7.18 (m, 3H, ArH), 6.99 – 6.95 (m, 1H, ArH), 6.89 – 6.79 (m, 4H, ArH), 5.11 – 4.98 (m, 2H, -CH₂), 4.88 (dd, $J = 12.1, 8.3$ Hz, 1H, -CH₂CH), 3.77 (d, $J = 2.4$ Hz, 6H, -OCH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 158.9, 154.2, 131.6, 131.1, 128.8, 126.6, 122.2, 114.4, 114.3, 112.7, 112.1, 100.9, 79.7, 55.9, 55.3, 40.9. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}_4$ 327.1345, found 327.1366.</p>
	<p>3o¹⁶: Brown solid. $R_f = 0.36$ (15% Ethyl acetate/Petroleum ether). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.42 (m, 1H, ArH), 7.31 – 7.15 (m, 4H, ArH), 7.05 (m, 1H, ArH), 6.85 – 6.80 (m, 3H, ArH), 5.10 (t, $J = 8.0$ Hz, 1H, -CH₂), 4.99 (dd, $J = 12.3, 7.4$ Hz, 1H, -CH₂), 4.85 (dd, $J = 12.3, 8.6$ Hz, 1H, -CH₂CH), 3.73 (s, 3H, -OCH₃), 3.69 (s, 3H, -CH₃). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, CDCl_3) δ 158.9, 137.4, 131.4, 128.9, 126.6, 126.3, 122.2, 119.5, 119.1, 114.3, 113.2, 109.6, 79.8, 55.3, 40.9, 32.8. HRMS (ESI) m/z calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{18}\text{H}_{19}\text{N}_2\text{O}_3$ 311.1396, found 311.1392.</p>

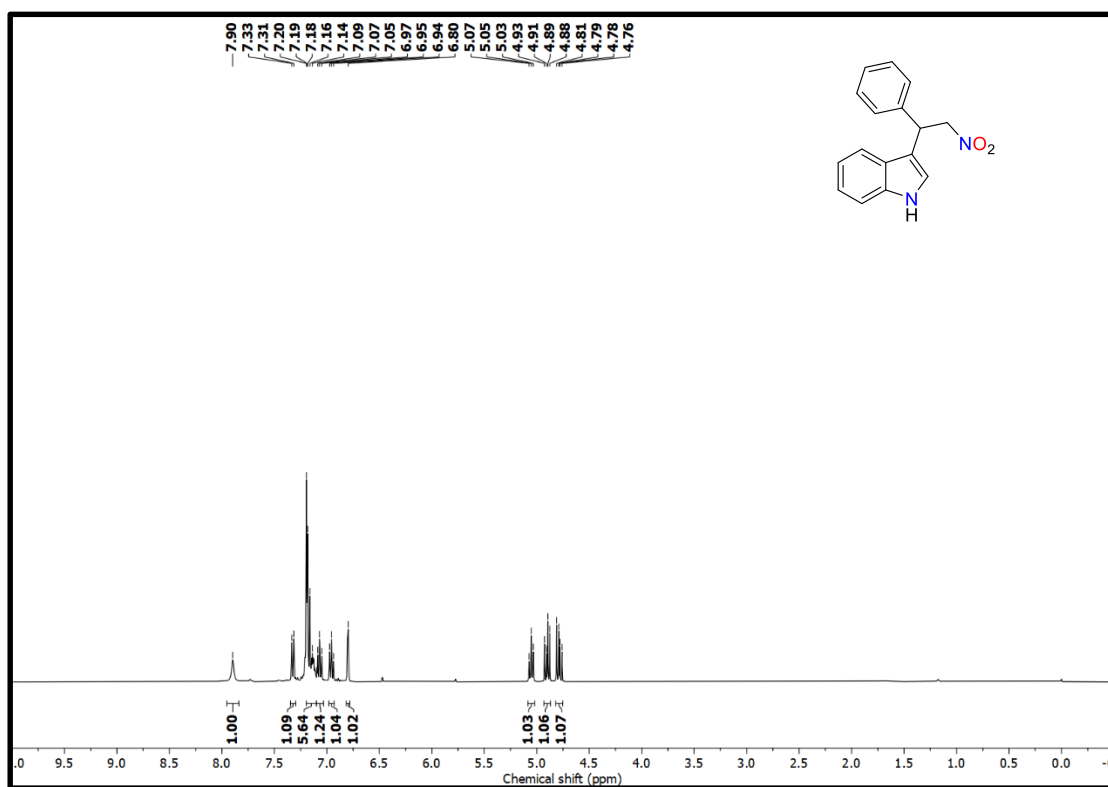


Figure S68: ^1H NMR spectrum of **3a** recorded in CDCl_3 .

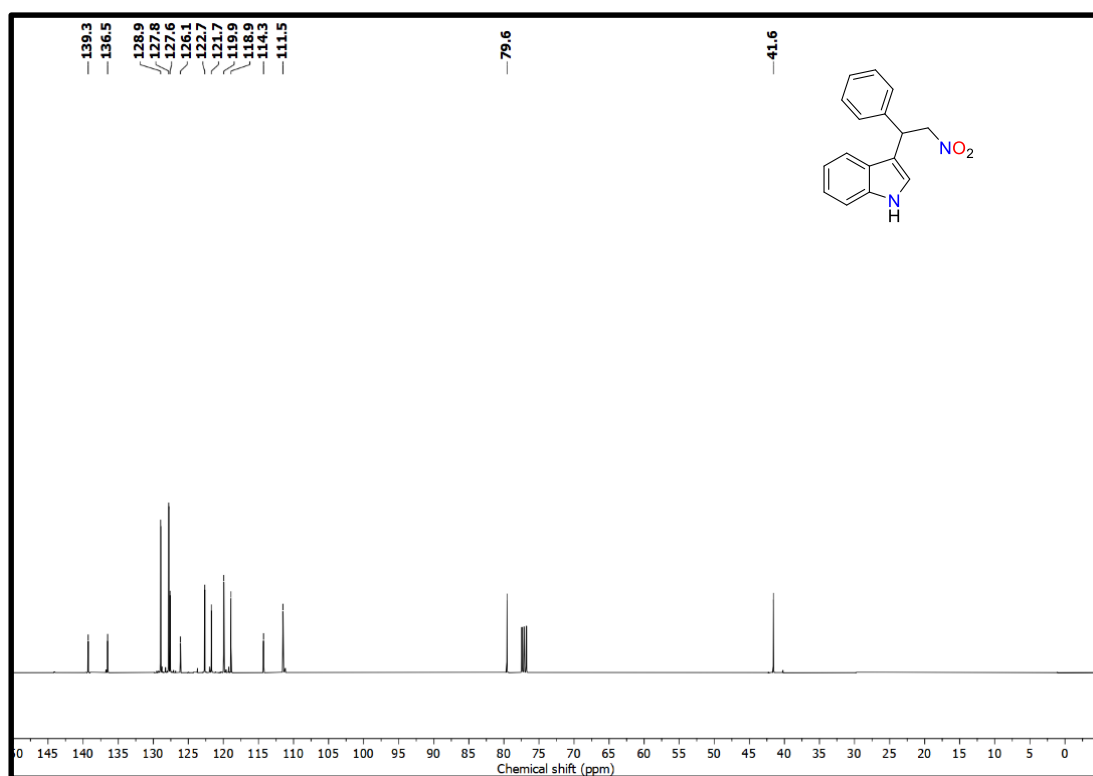


Figure S69: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3a** recorded in CDCl_3 .

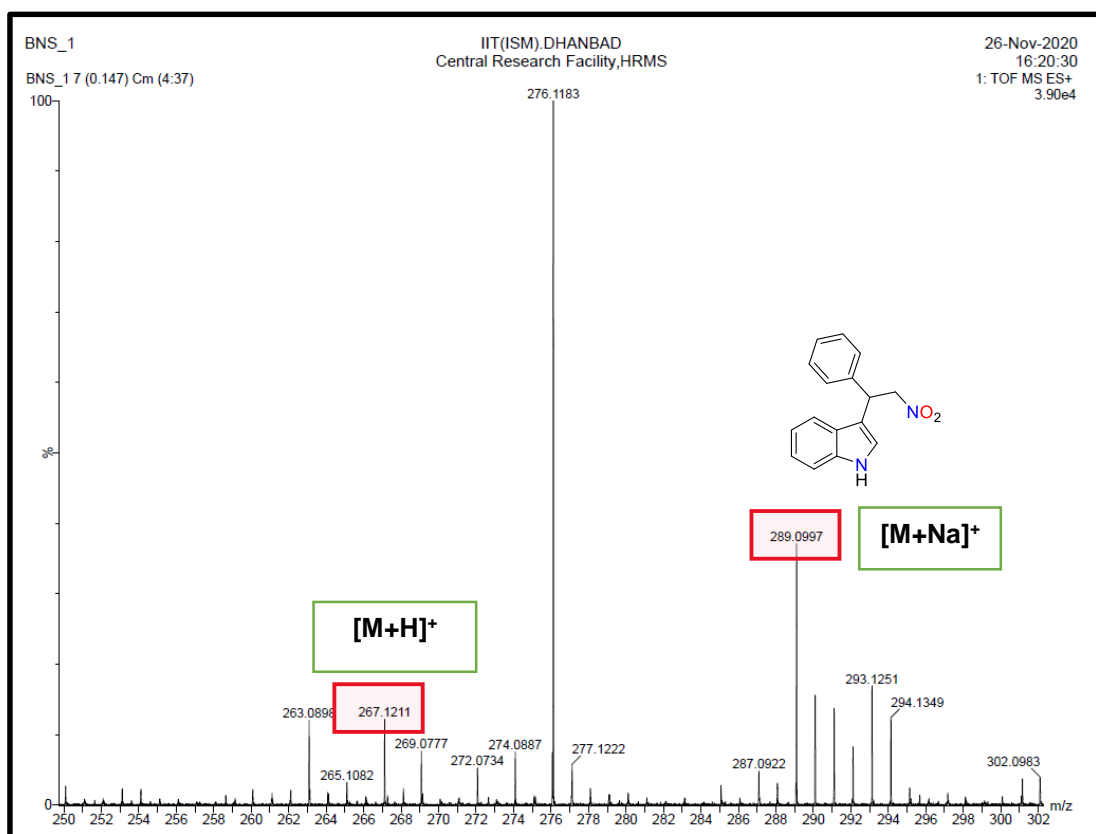


Figure S70: HRMS (ESI) spectrum of **3a**.

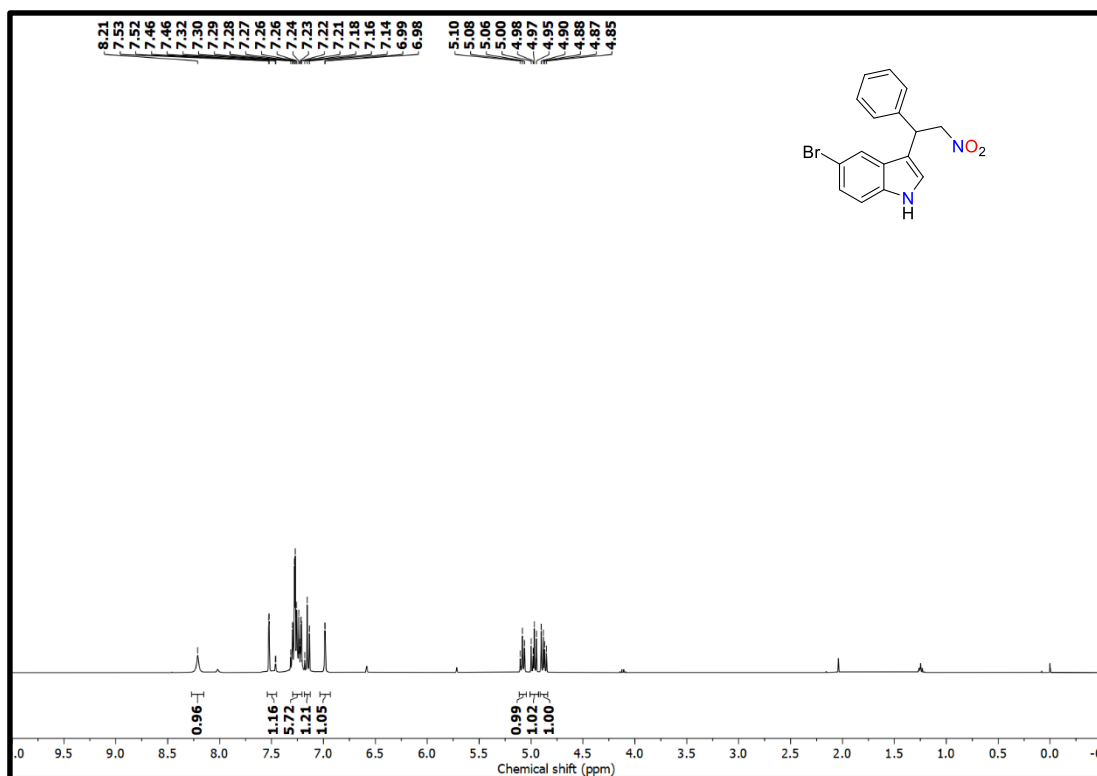


Figure S71: ¹H NMR spectrum of **3b** recorded in CDCl₃.

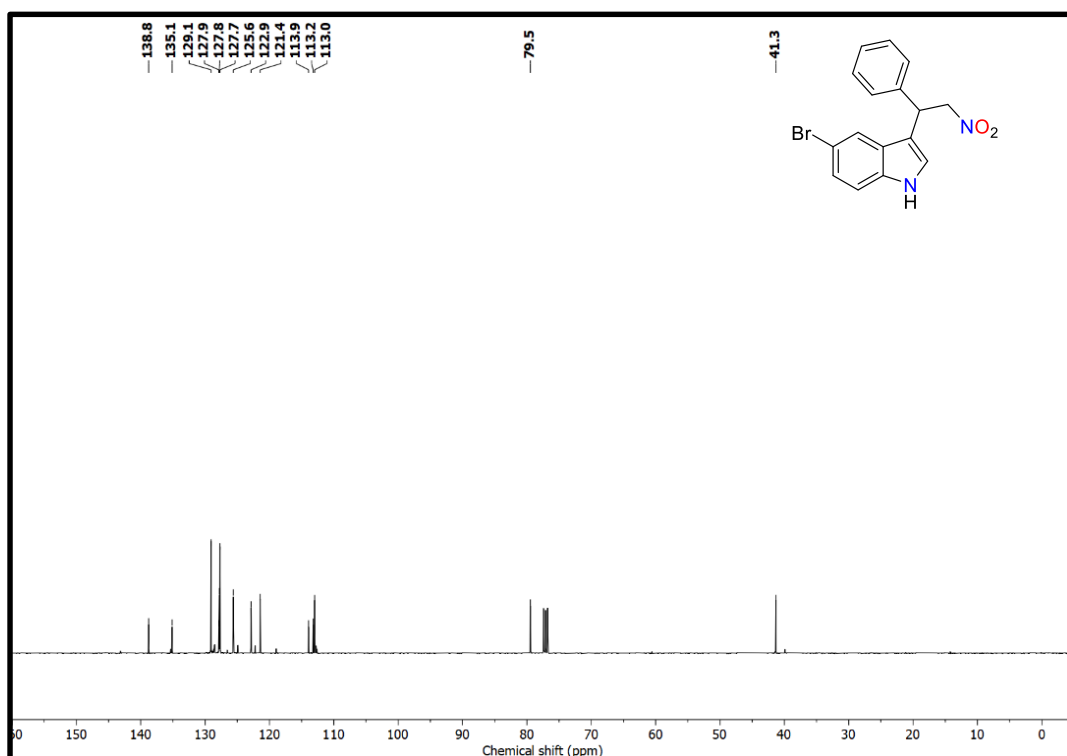


Figure S72: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3b** recorded in CDCl_3 .

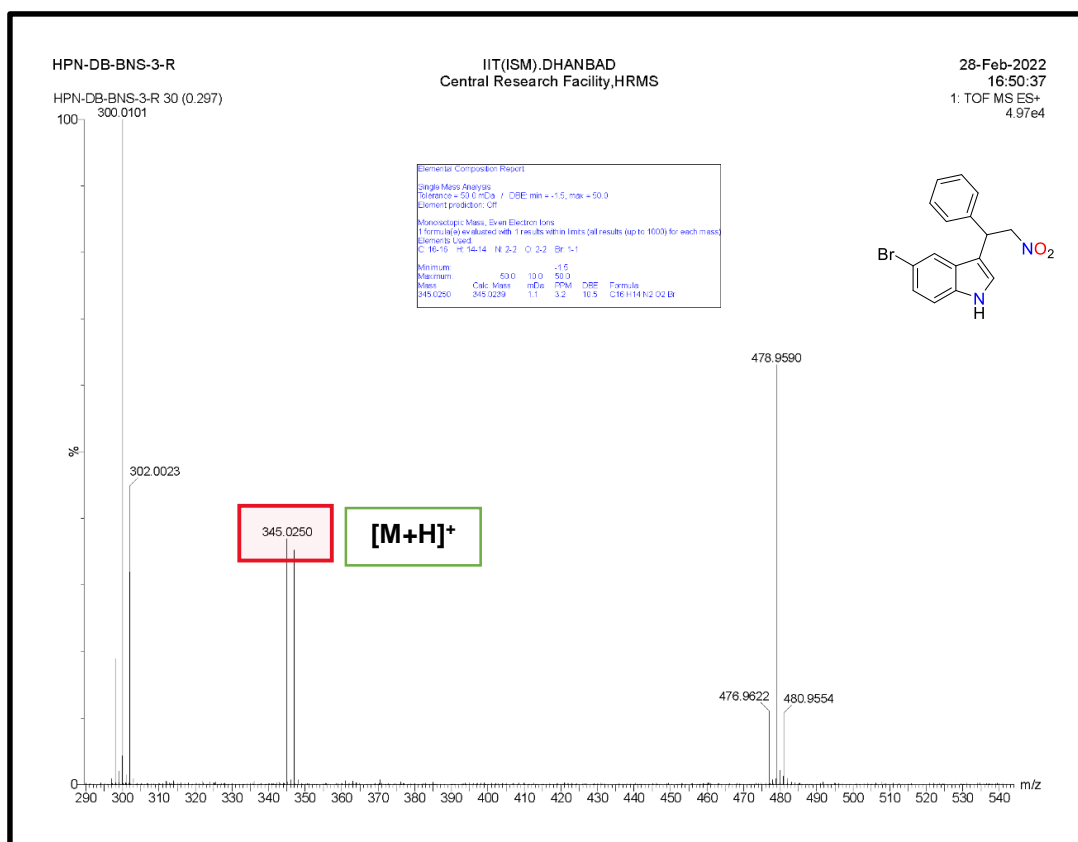


Figure S73: HRMS (ESI) spectrum of **3b**.

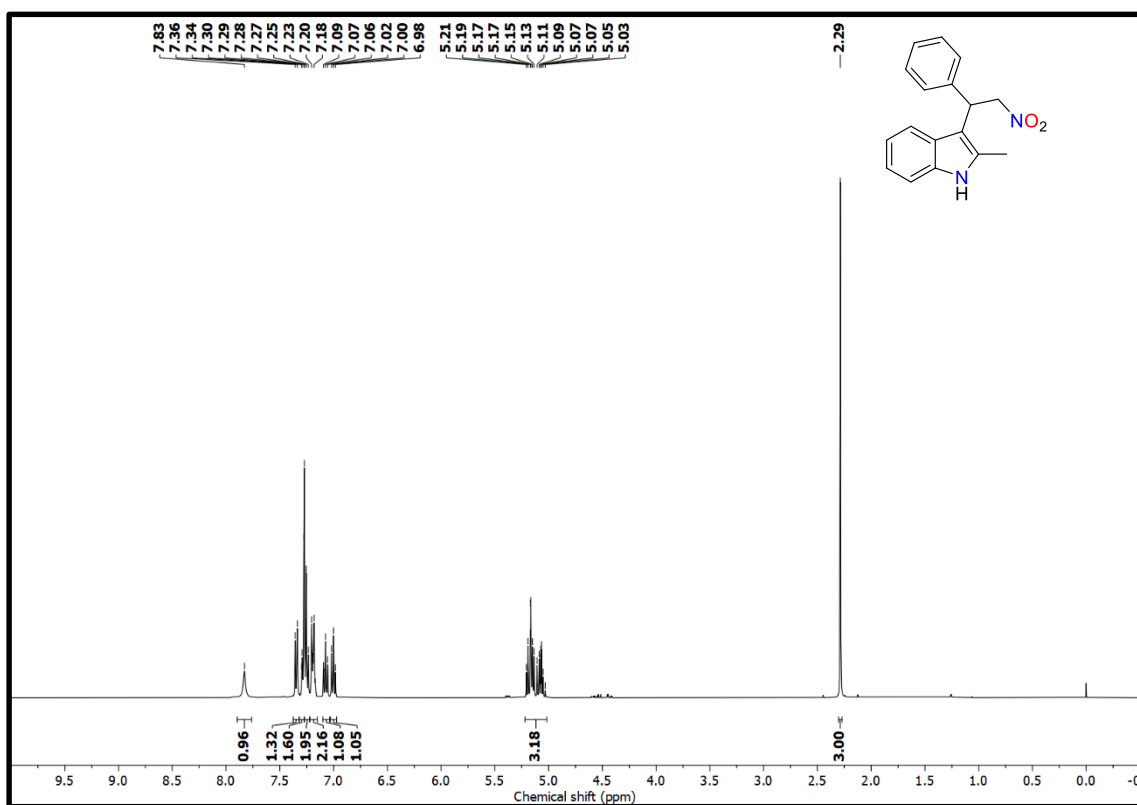


Figure S74: ¹H NMR spectrum of **3c** recorded in CDCl₃.

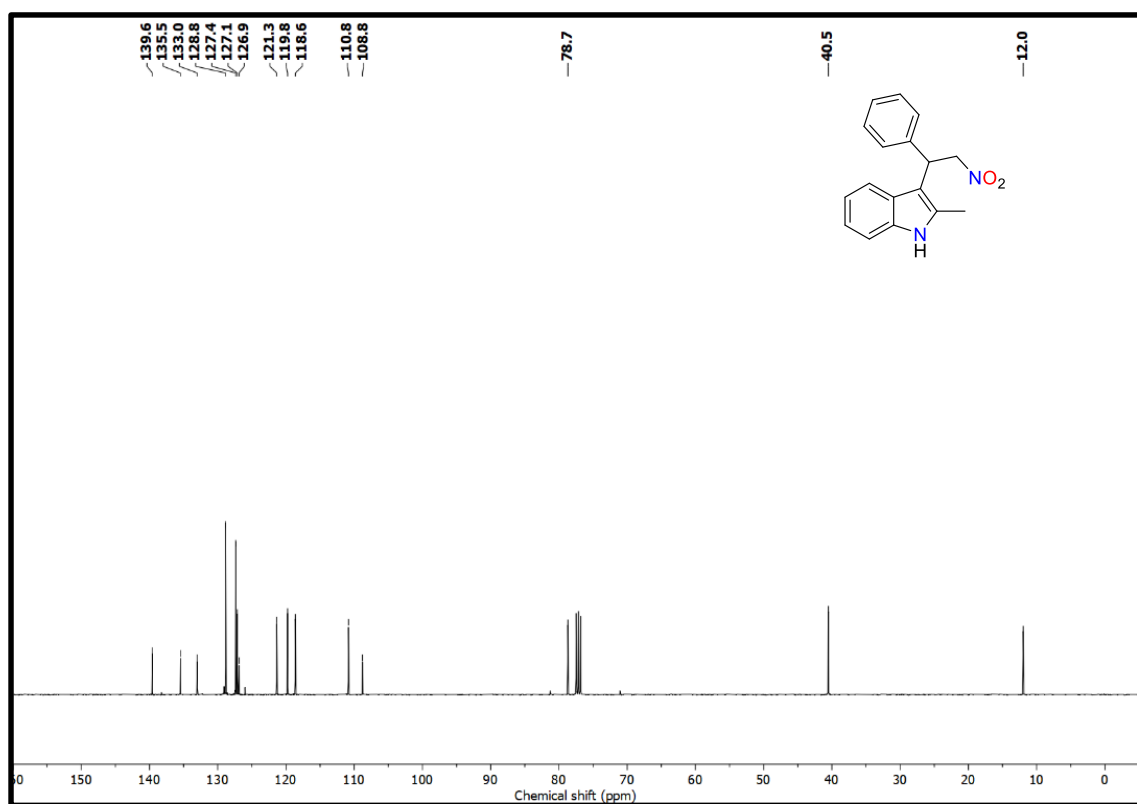


Figure S75: ¹³C{¹H} NMR spectrum of **3c** recorded in CDCl₃.

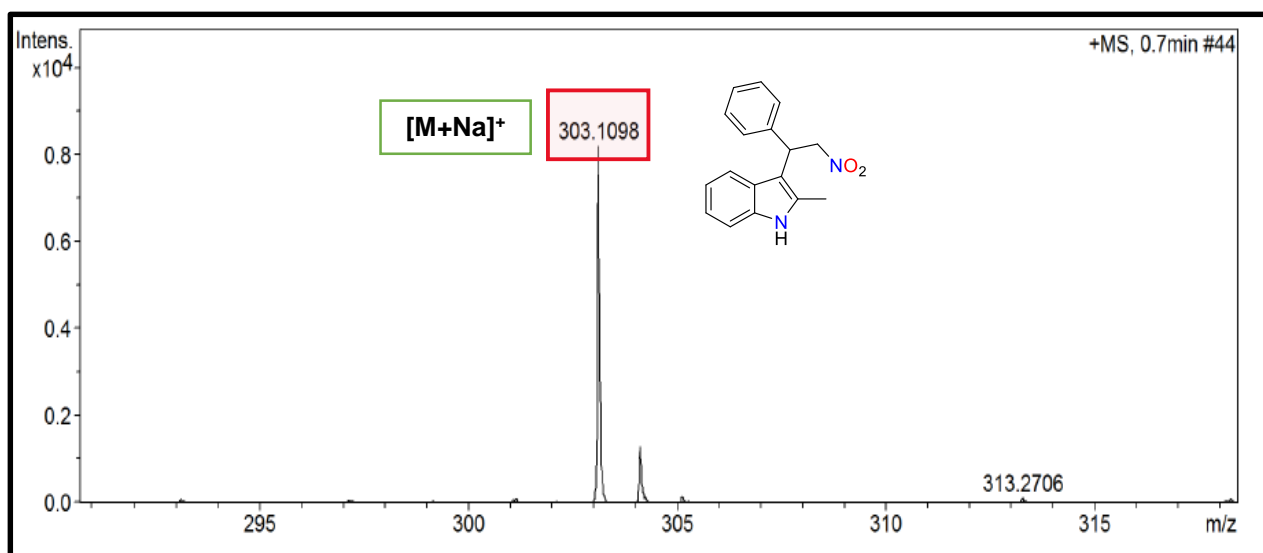


Figure S76: HRMS (ESI) spectrum of **3c**.

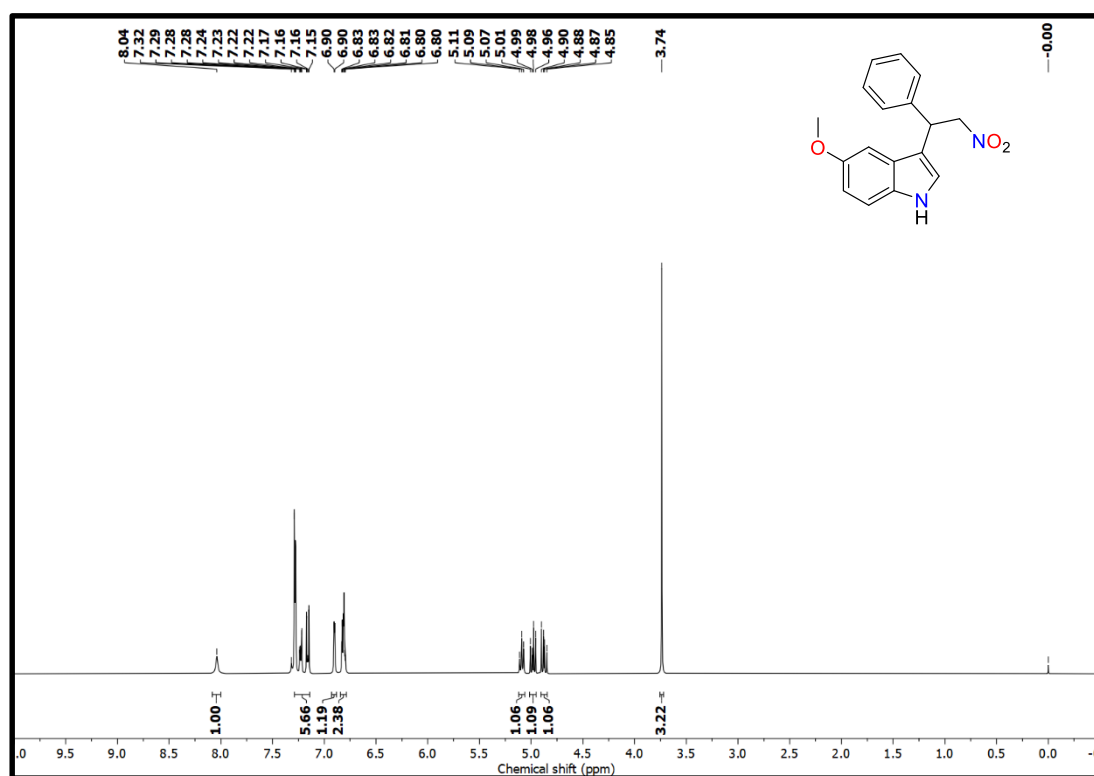


Figure S77: ^1H NMR spectrum of **3d** recorded in CDCl_3 .

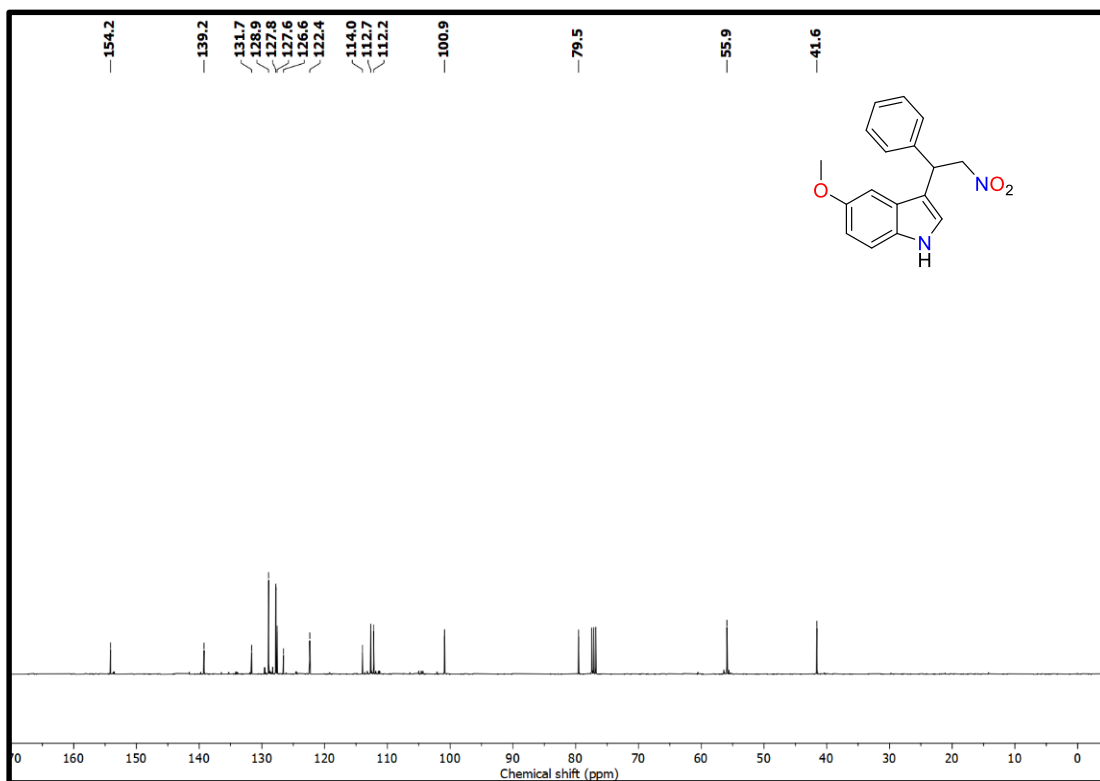


Figure S78: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3d** recorded in CDCl_3 .

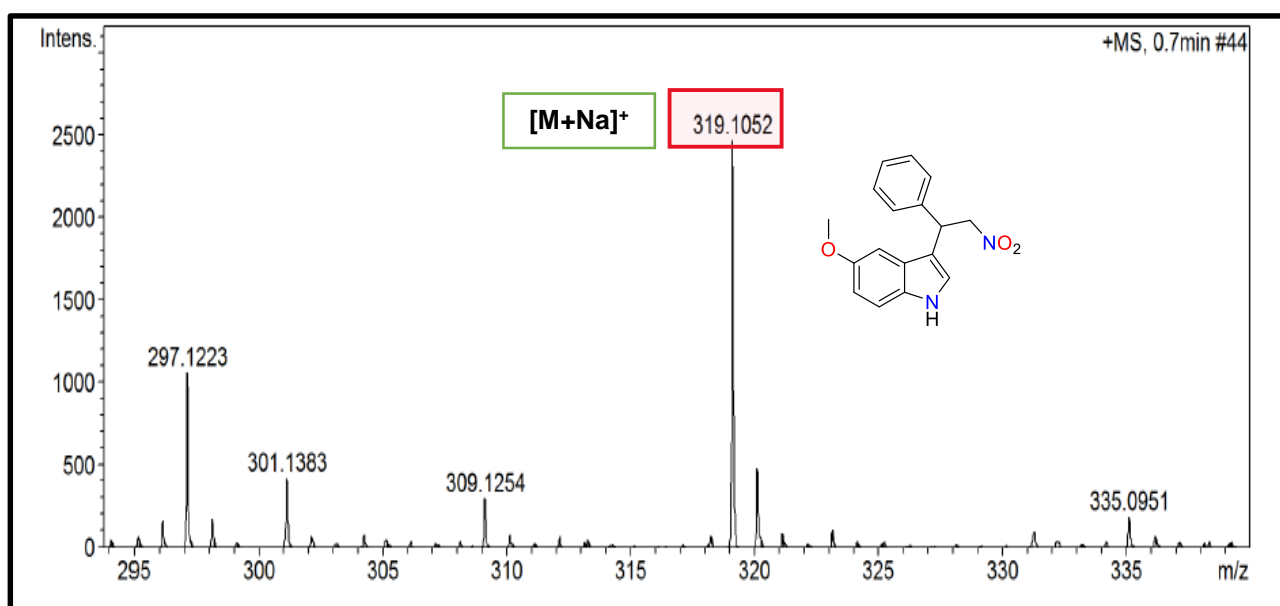


Figure S79: HRMS (ESI) spectrum of **3d**.

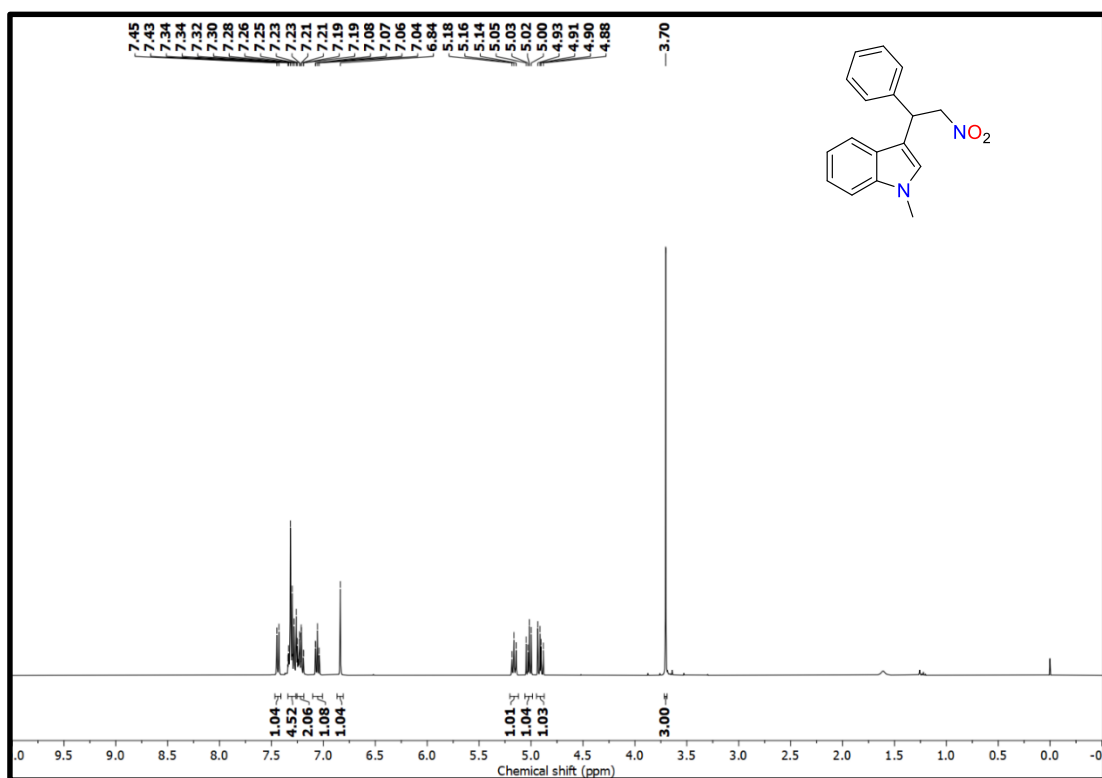


Figure S80: ^1H NMR spectrum of **3e** recorded in CDCl_3 .

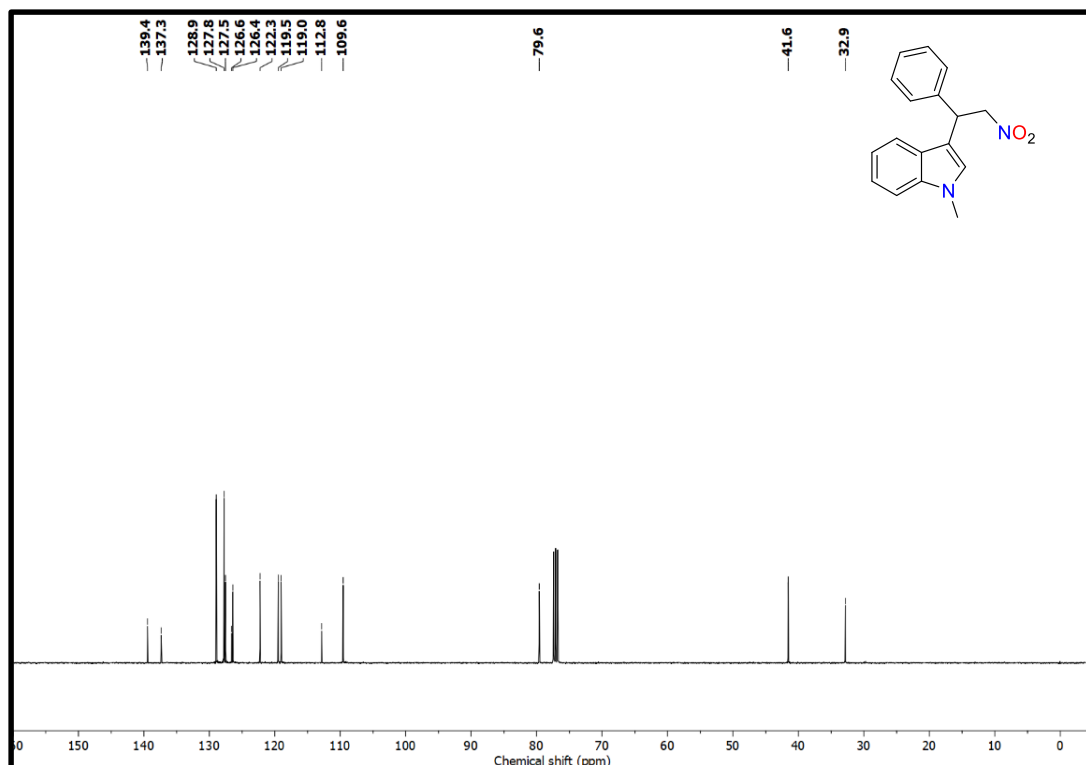


Figure S81: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3e** recorded in CDCl_3 .

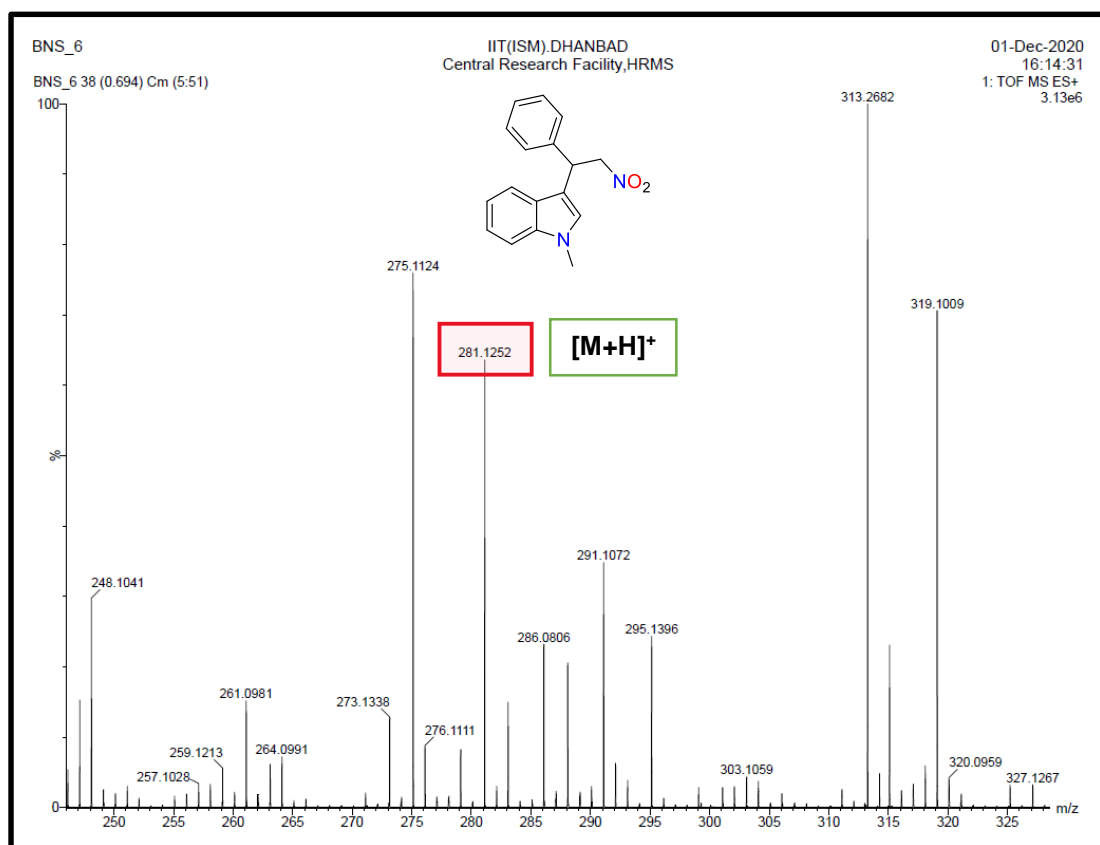


Figure S82: HRMS (ESI) spectrum of **3e**.

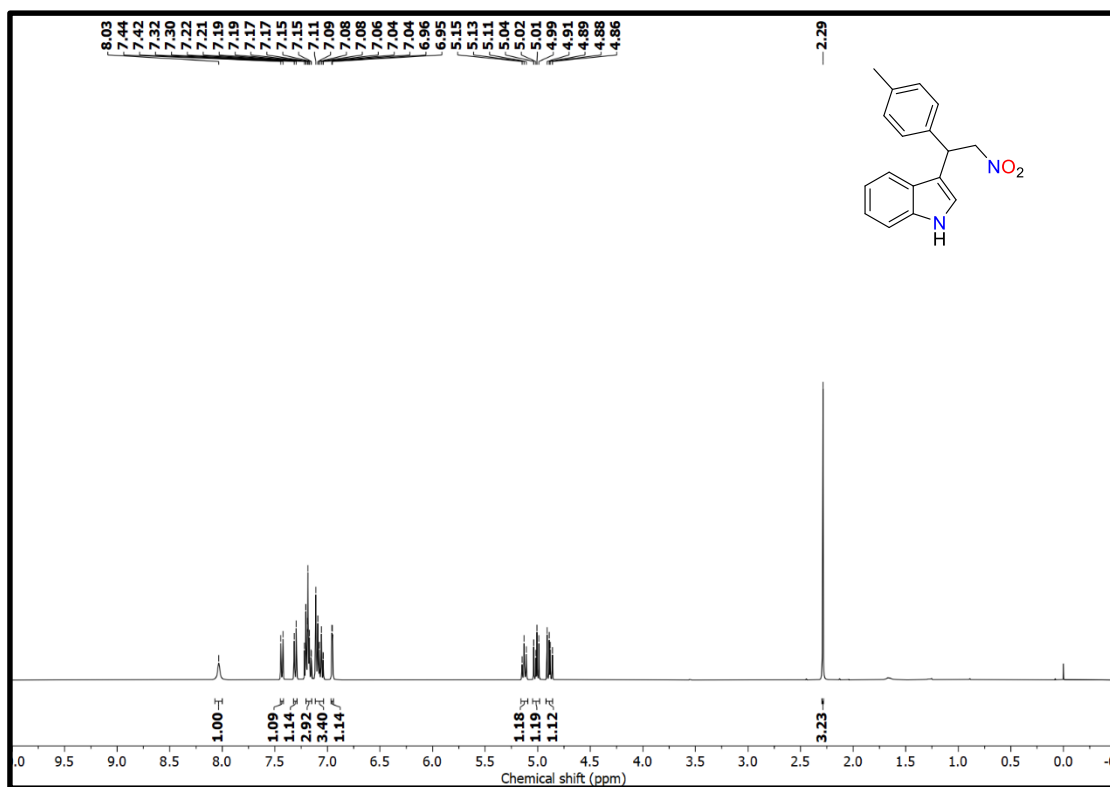


Figure S83: ¹H NMR spectrum of **3f** recorded in CDCl₃.

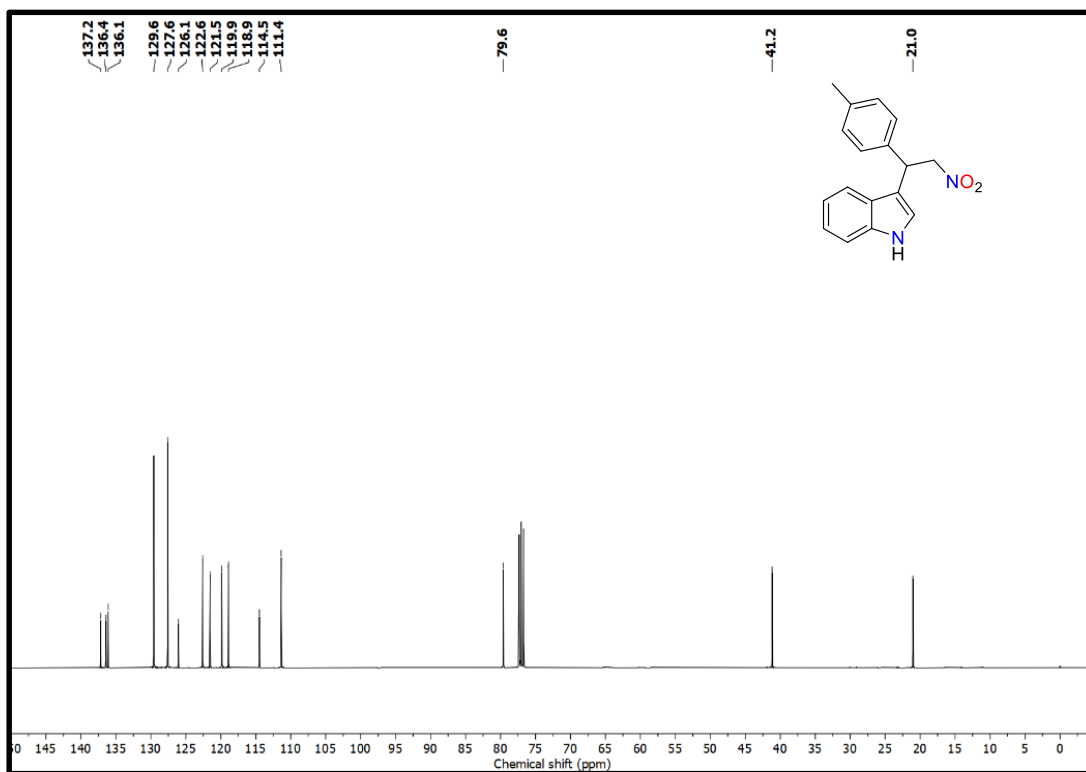


Figure S84: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3f** recorded in CDCl_3 .

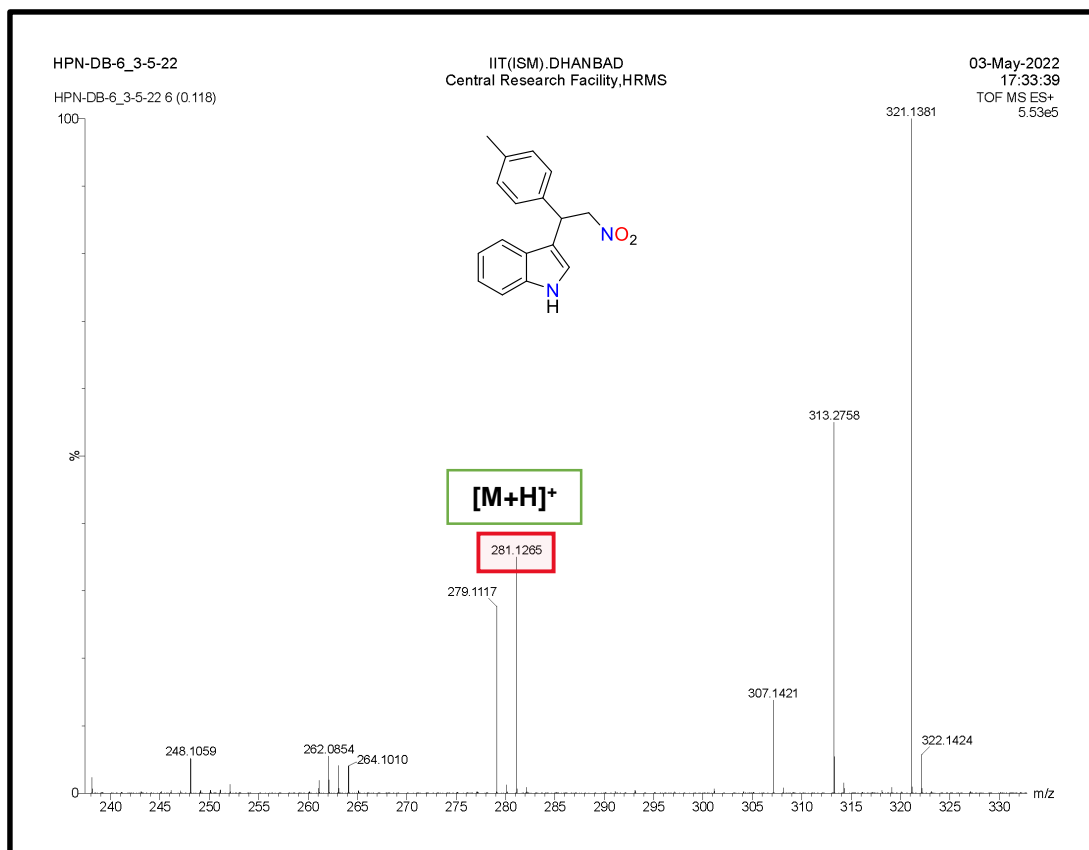


Figure S85: HRMS (ESI) spectrum of **3f**.

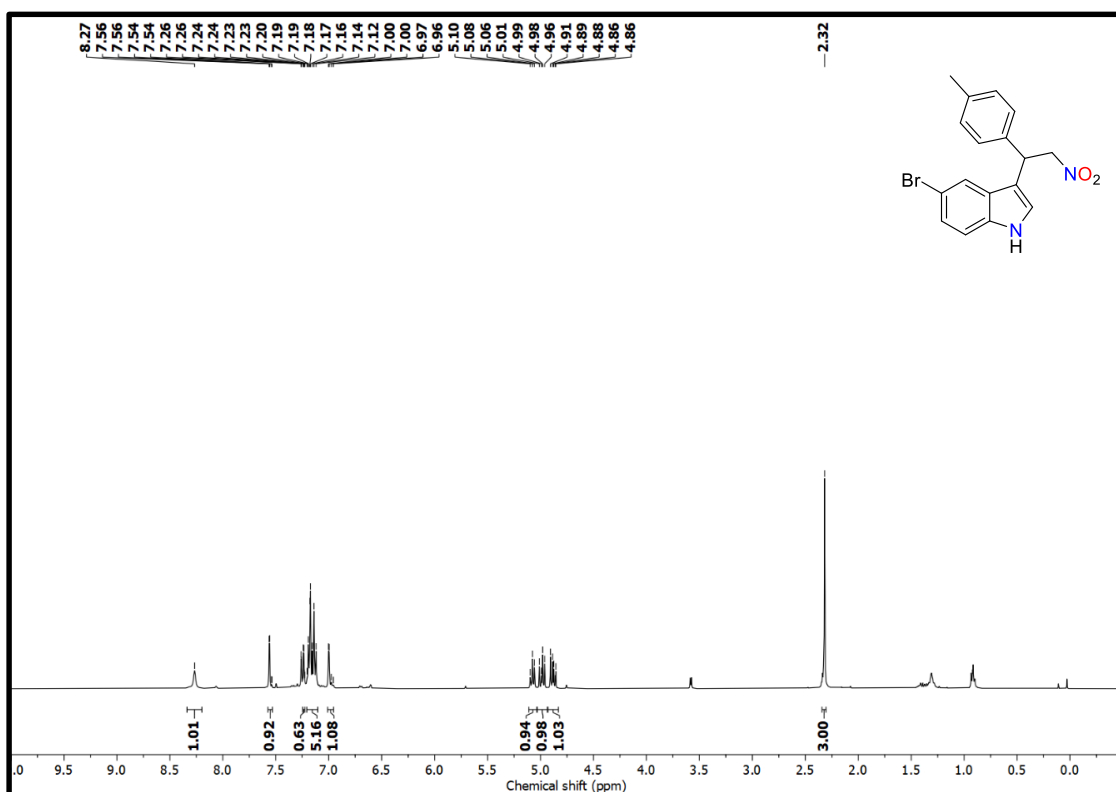


Figure S86: ^1H NMR spectrum of **3g** recorded in CDCl_3 .

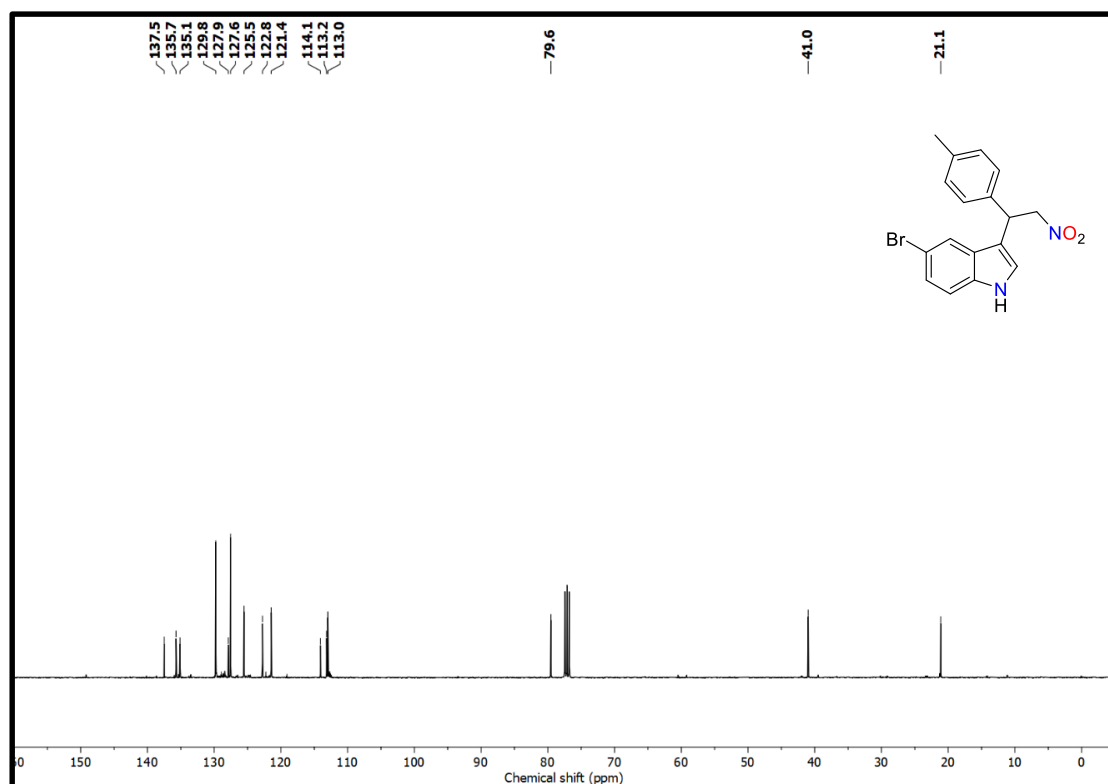


Figure S87: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3g** recorded in CDCl_3 .

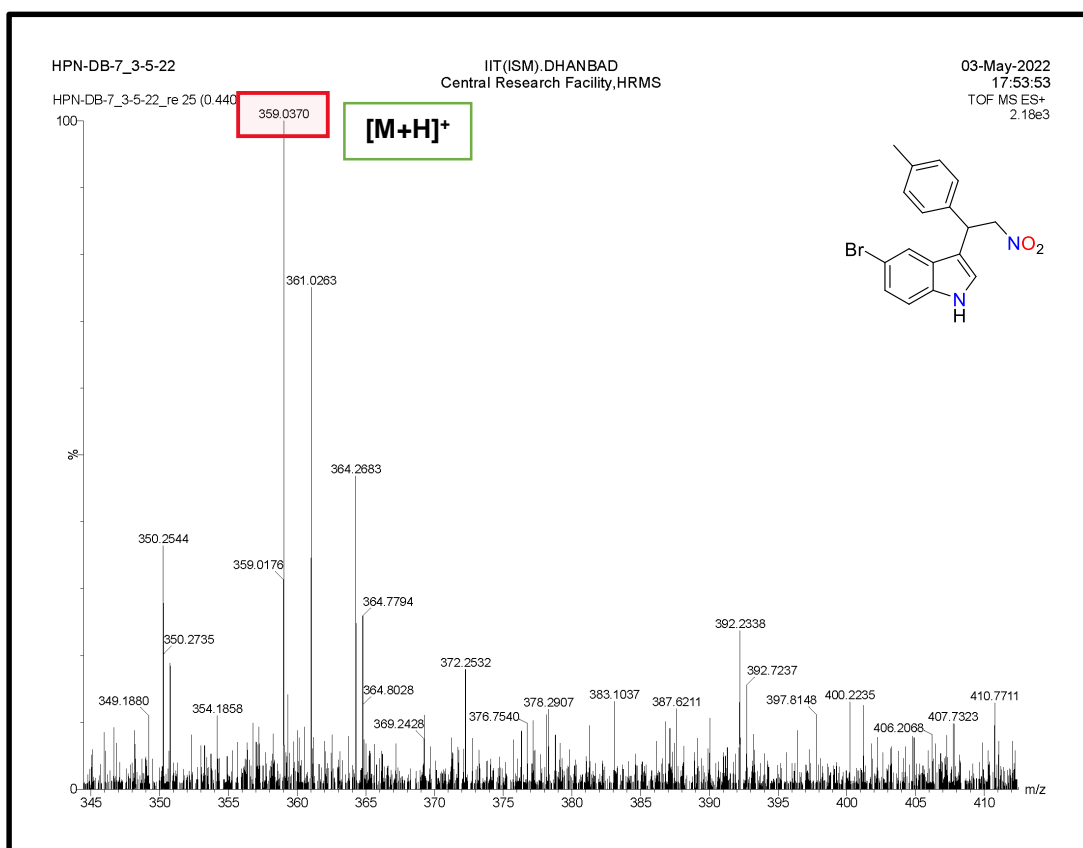


Figure S88: HRMS (ESI) spectrum of **3g**.

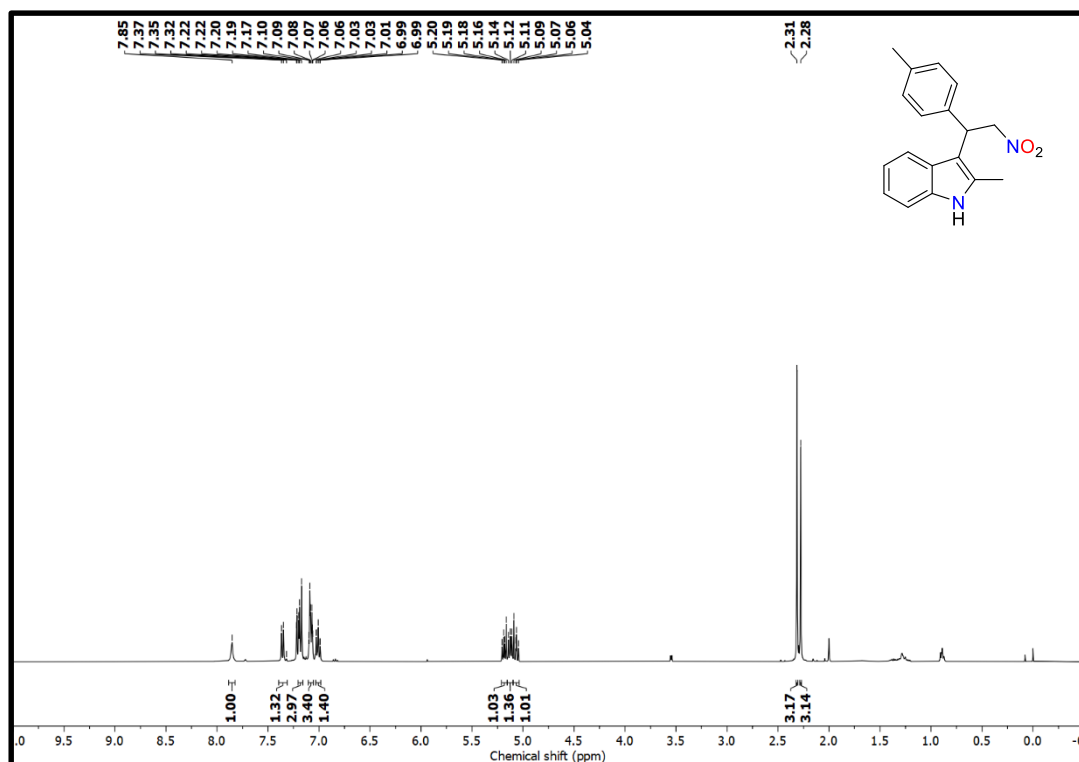


Figure S89: ¹H NMR spectrum of **3h** recorded in CDCl₃.

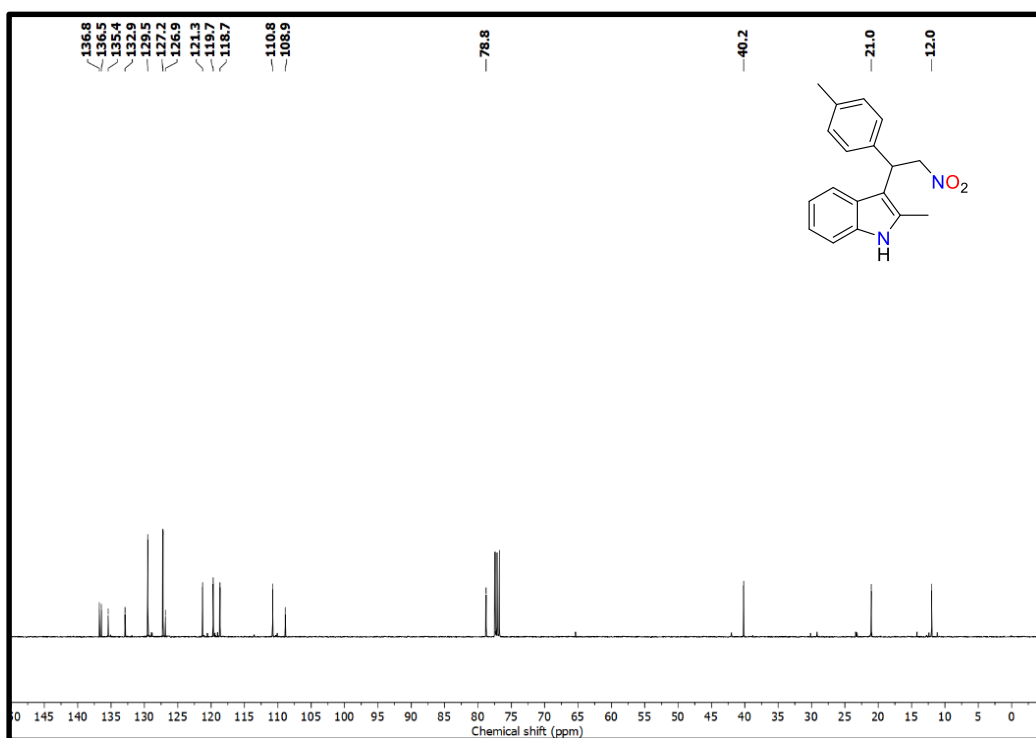


Figure S90: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3h** recorded in CDCl_3 .

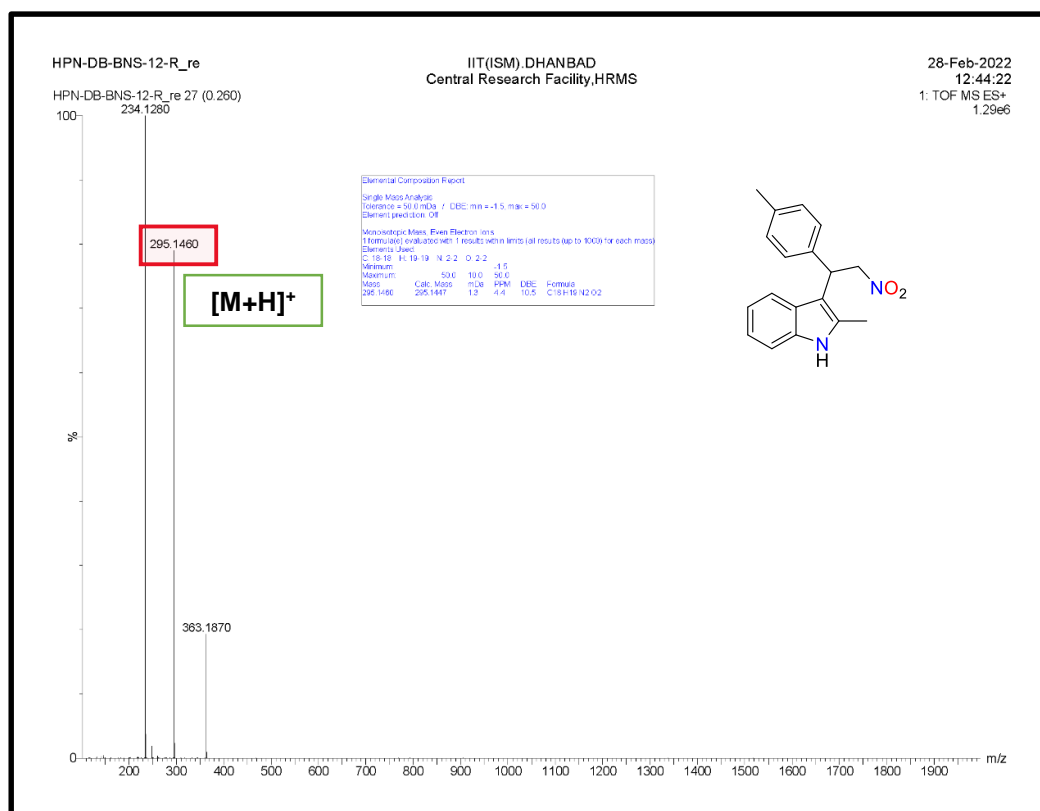


Figure S91: HRMS (ESI) spectrum of **3h**.

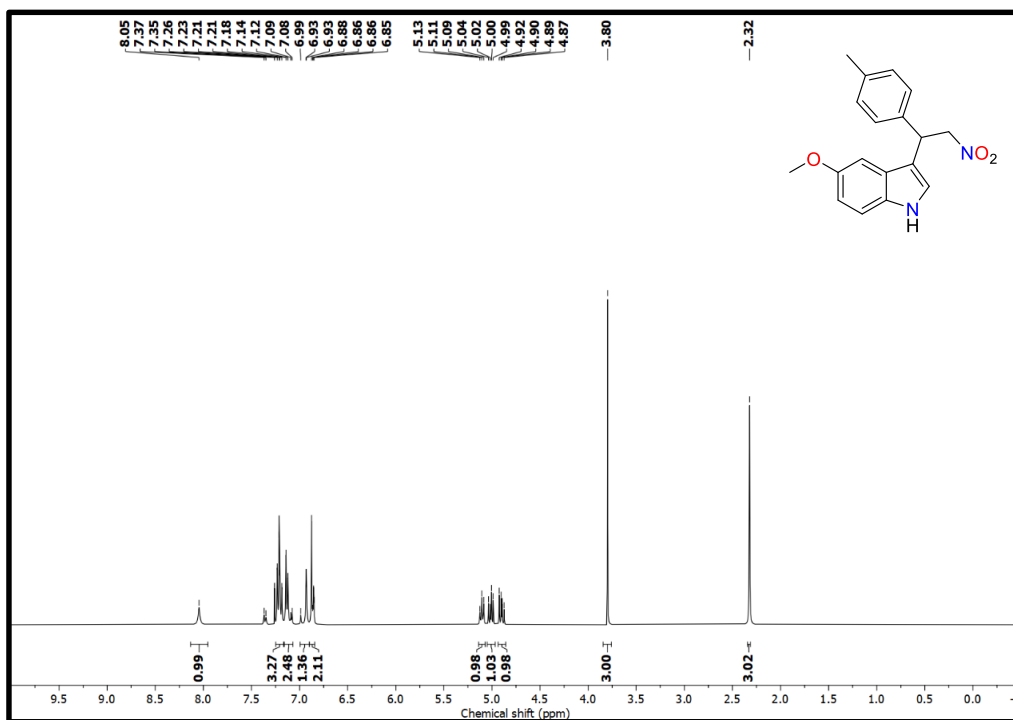


Figure S92: ^1H NMR spectrum of **3i** recorded in CDCl_3 .

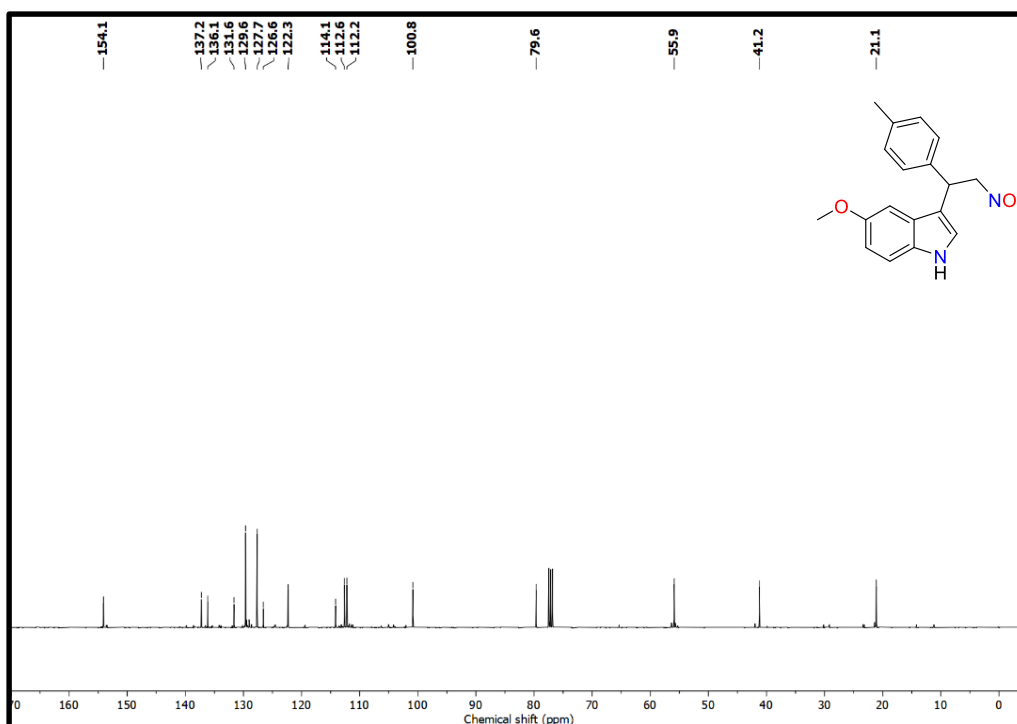


Figure S93: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3i** recorded in CDCl_3 .

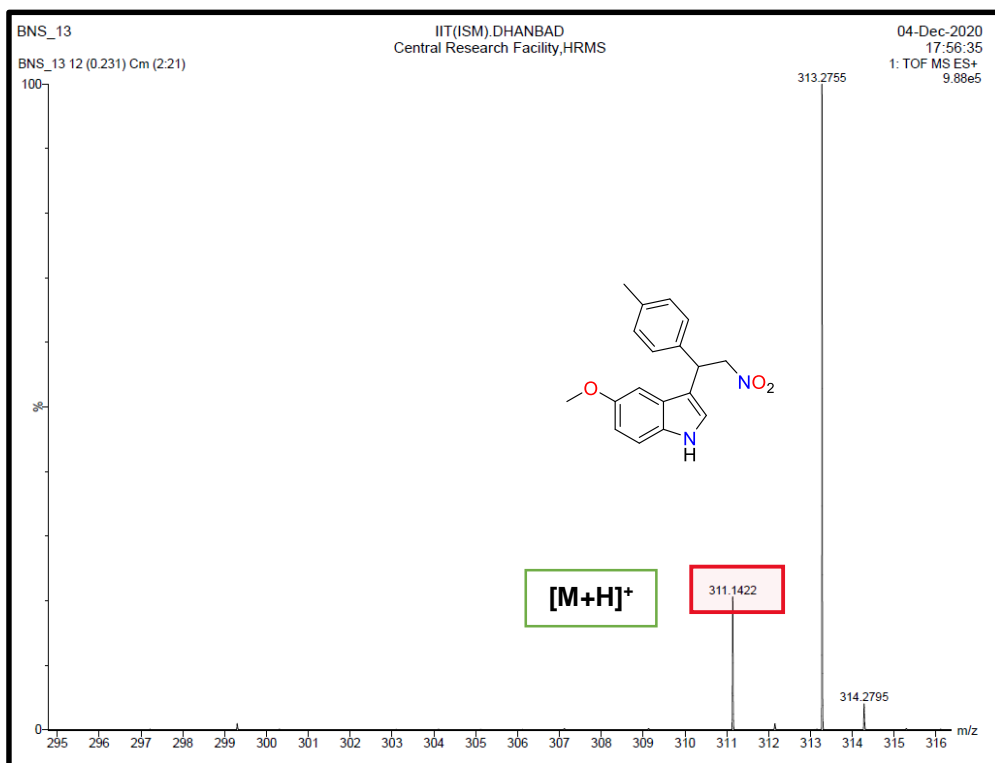


Figure S94: HRMS (ESI) spectrum of **3i**.

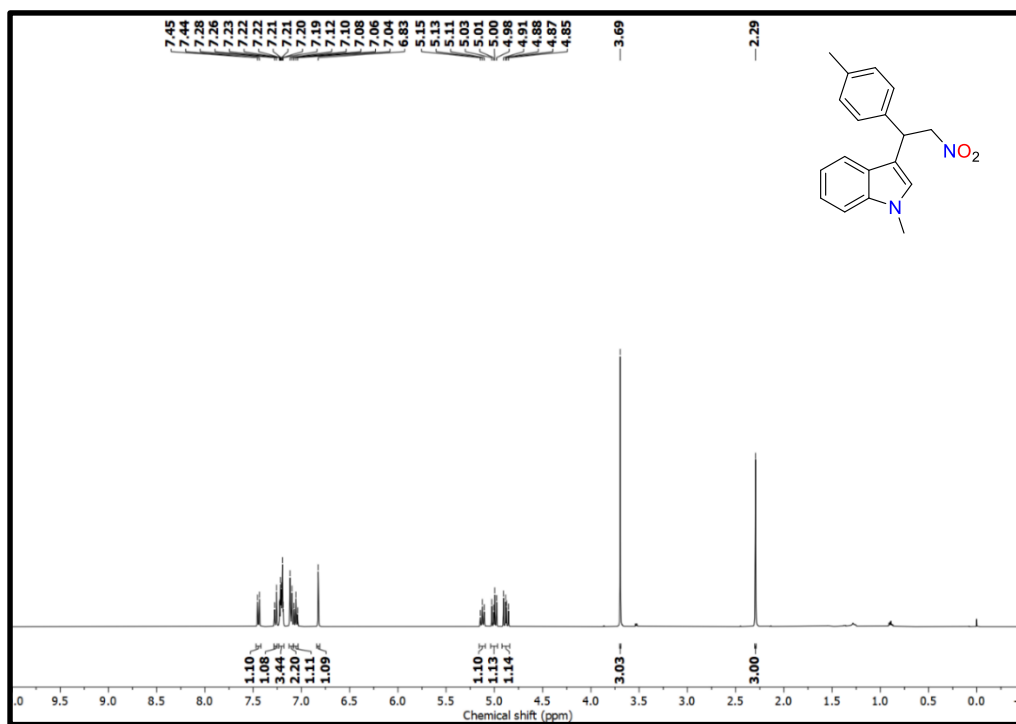


Figure S95: ^1H NMR spectrum of **3j** recorded in CDCl_3 .

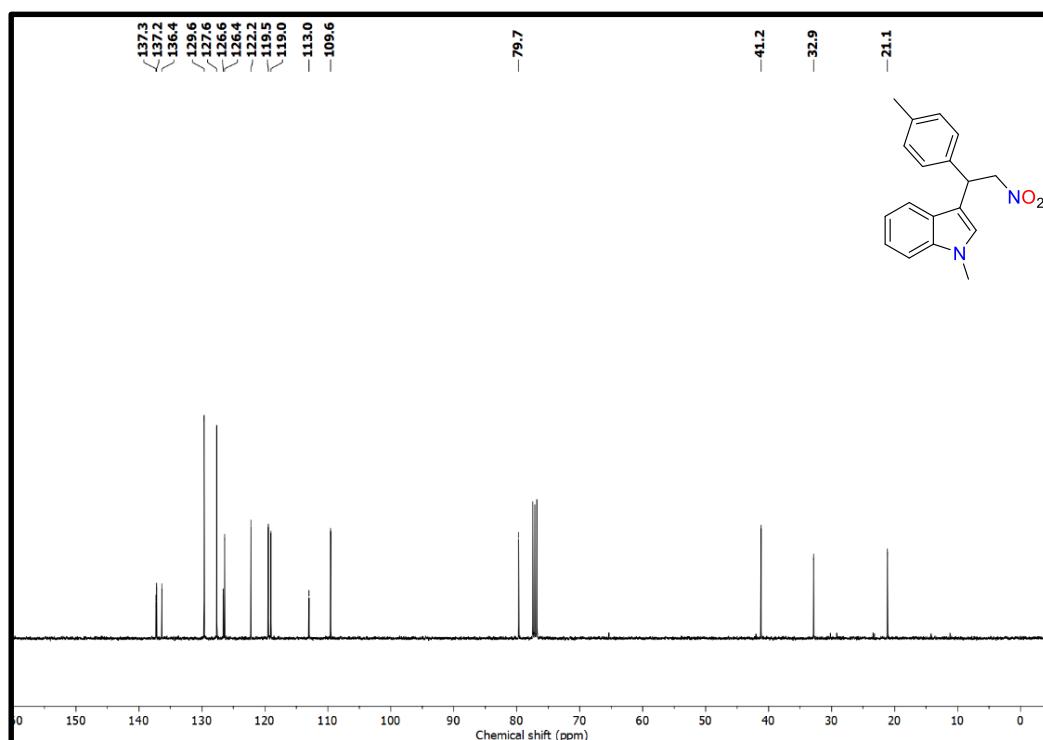


Figure S96: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3j** recorded in CDCl_3 .

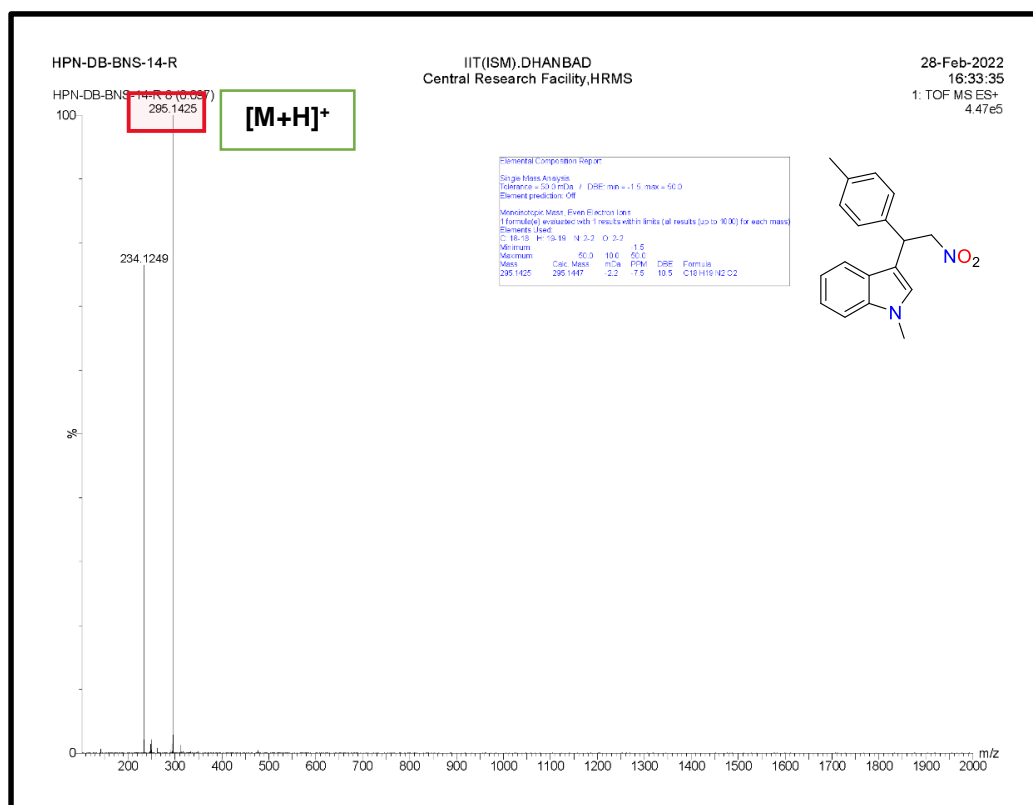


Figure S97: HRMS (ESI) spectrum of **3j**.

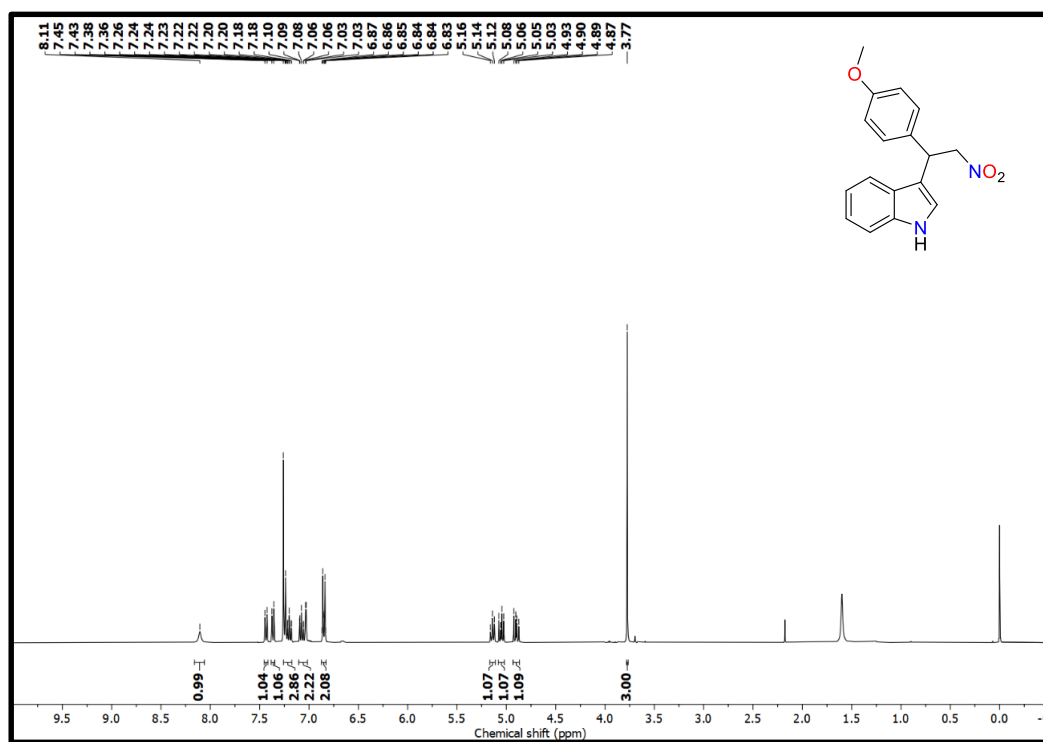


Figure S98: ^1H NMR spectrum of **3k** recorded in CDCl_3 .

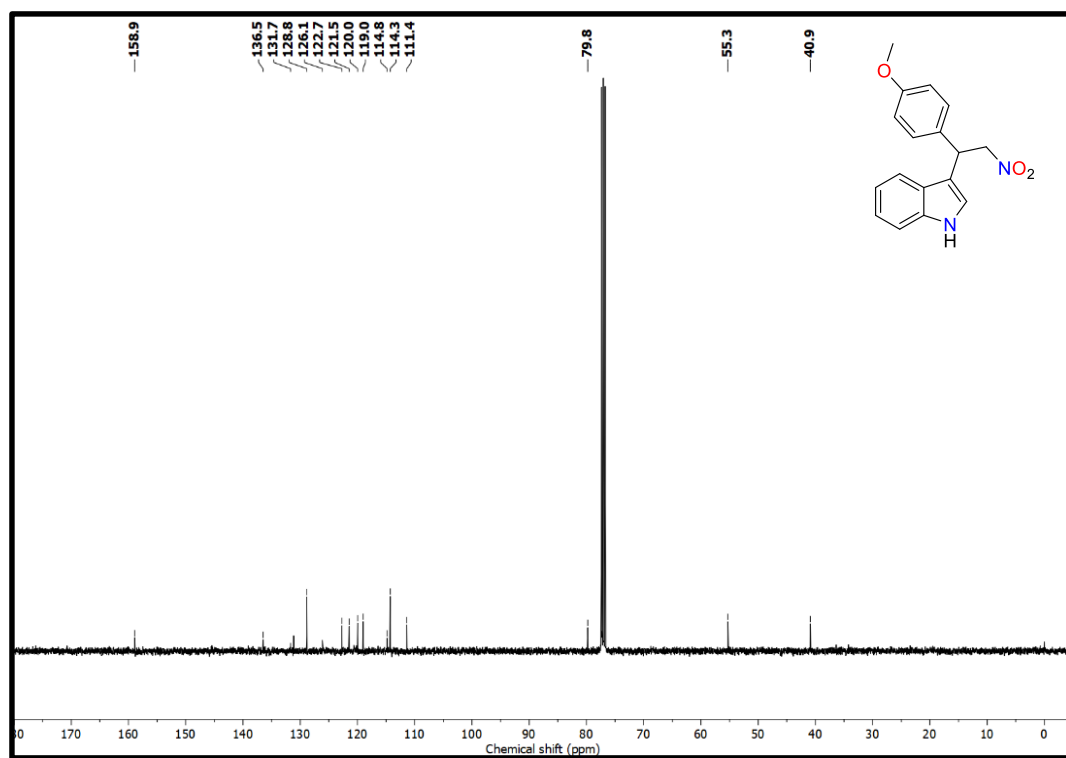


Figure S99: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3k** recorded in CDCl_3 .

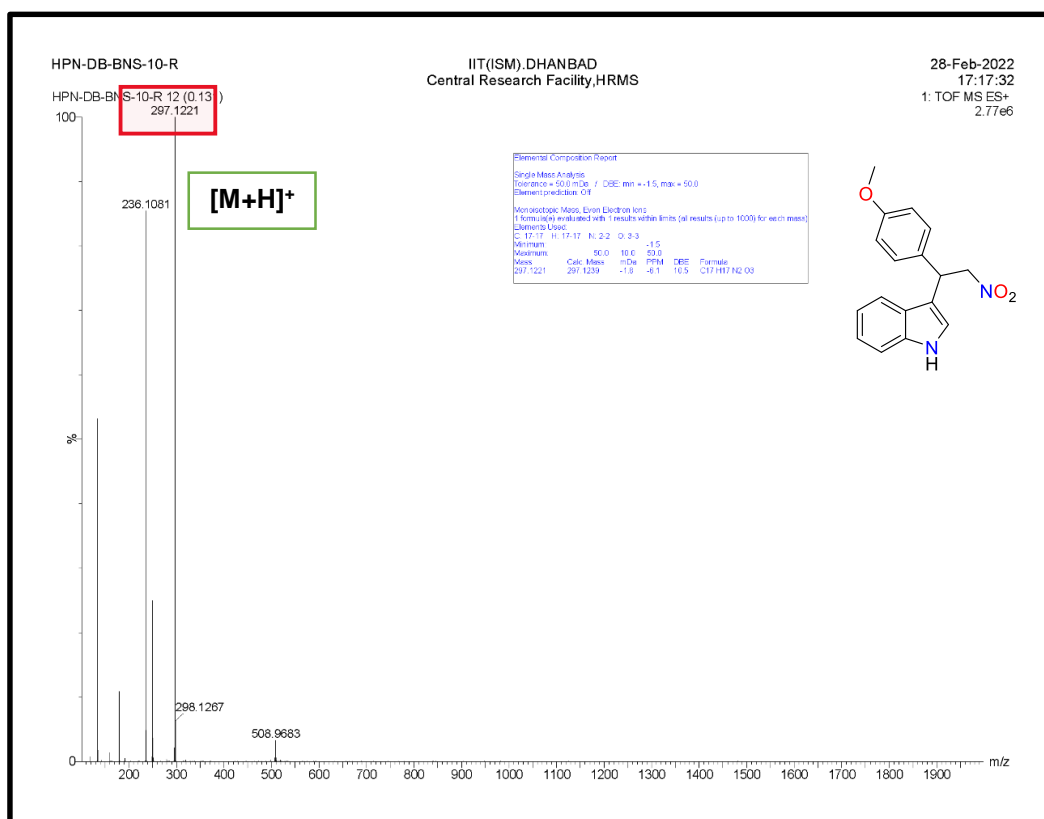


Figure S100: HRMS (ESI) spectrum of 3k.

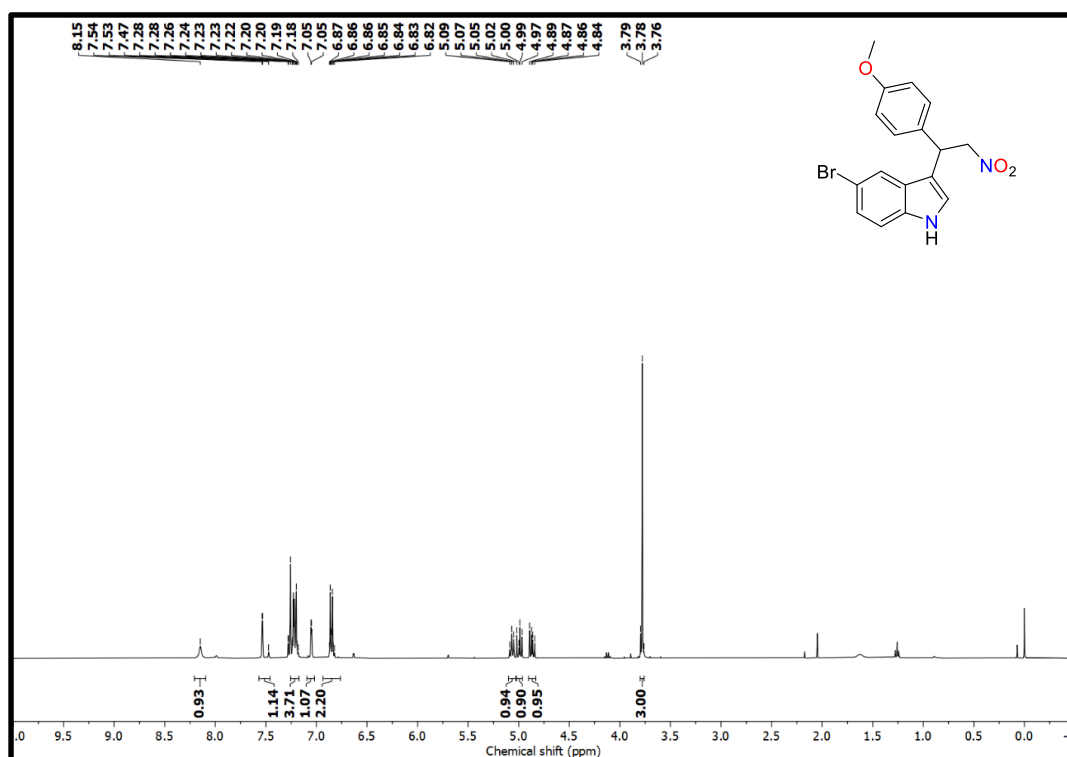


Figure S101: ¹H NMR spectrum of 3l recorded in CDCl₃.

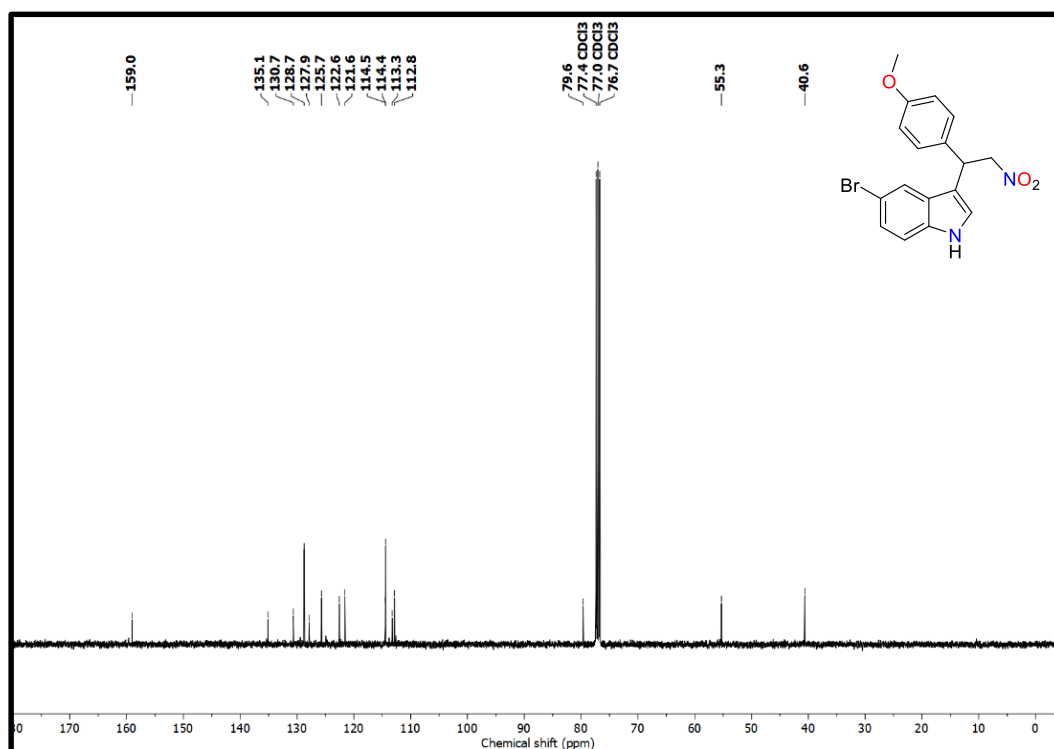


Figure S102: ¹³C{¹H} NMR spectrum of **3I** recorded in CDCl₃.

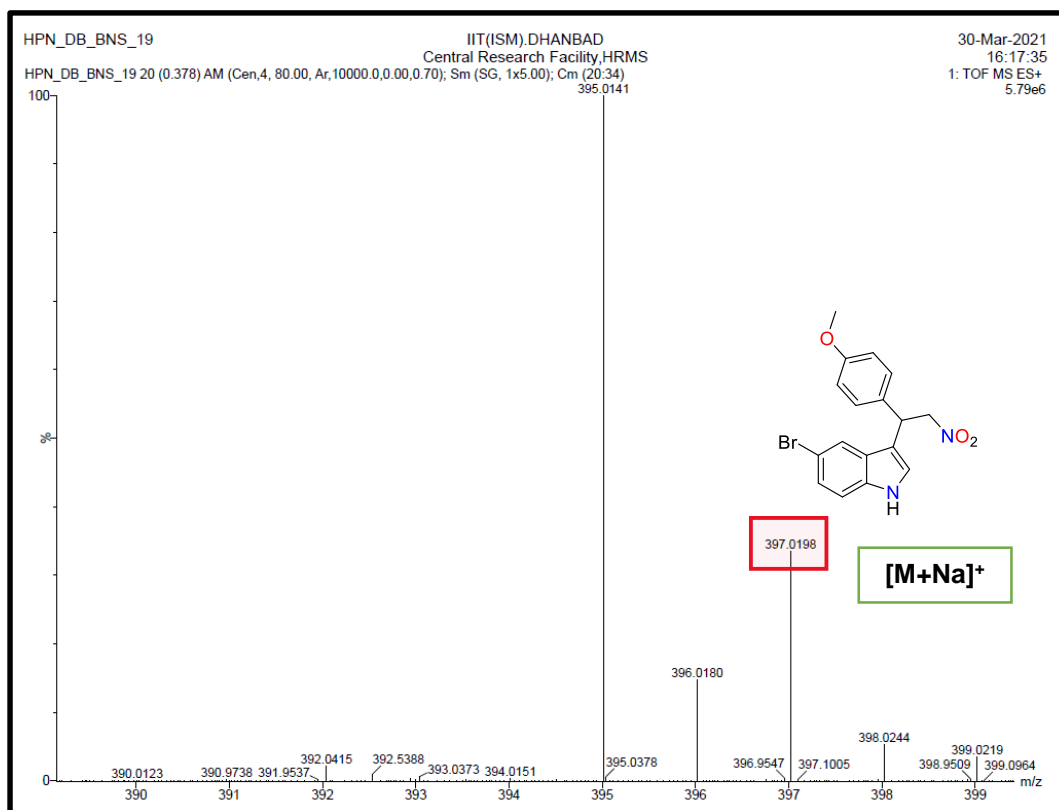


Figure S103: HRMS (ESI) spectrum of **3I**.

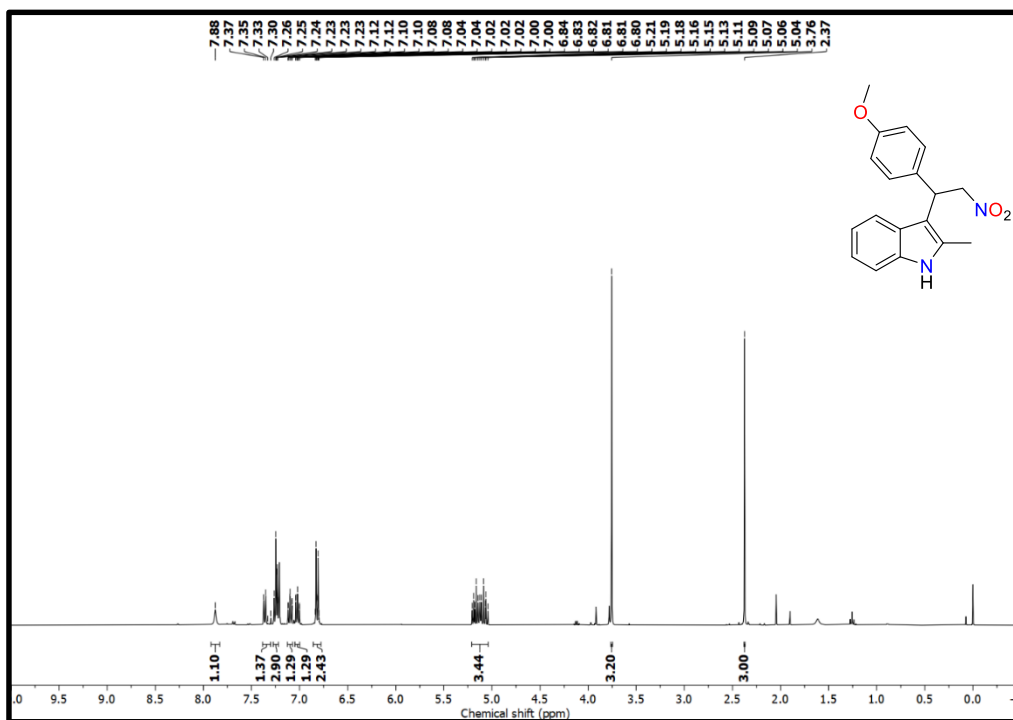


Figure S104: ^1H NMR spectrum of **3m** recorded in CDCl_3 .

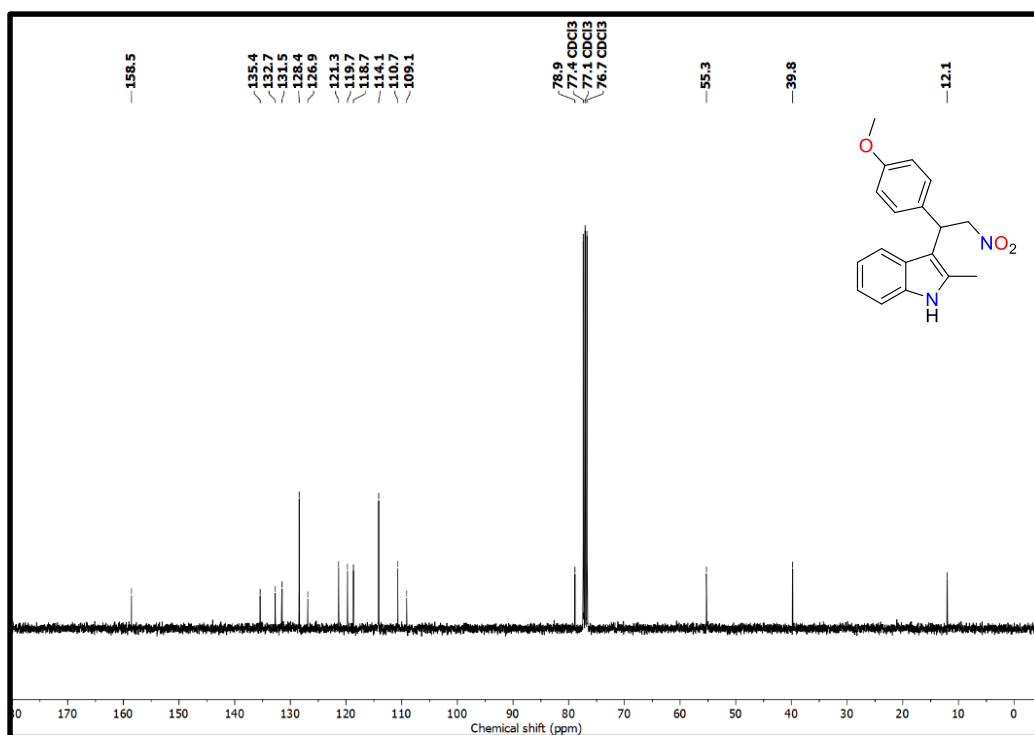


Figure S105: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3m** recorded in CDCl_3 .

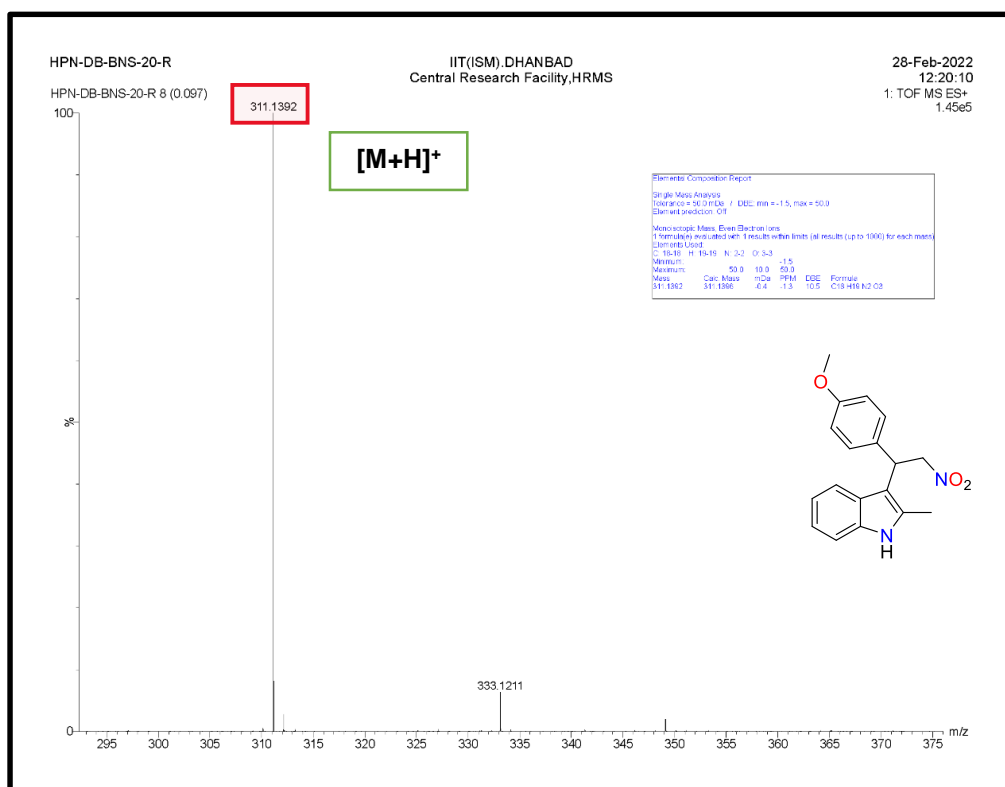


Figure S106: HRMS (ESI) spectrum of **3m**.

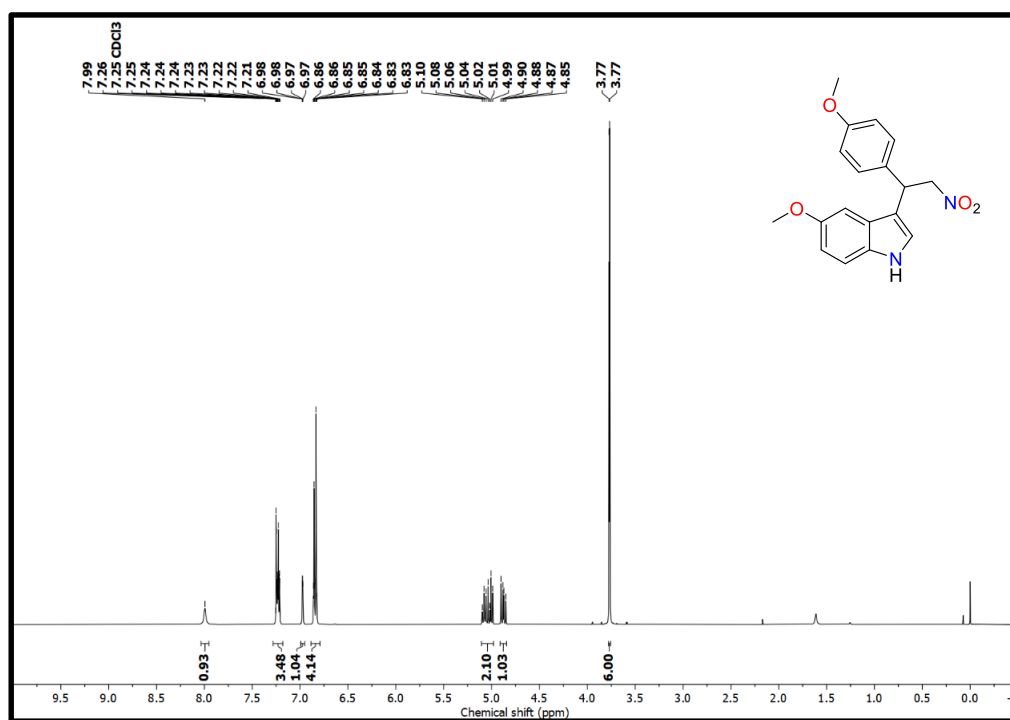


Figure S107: ¹H NMR spectrum of **3n** recorded in CDCl₃.

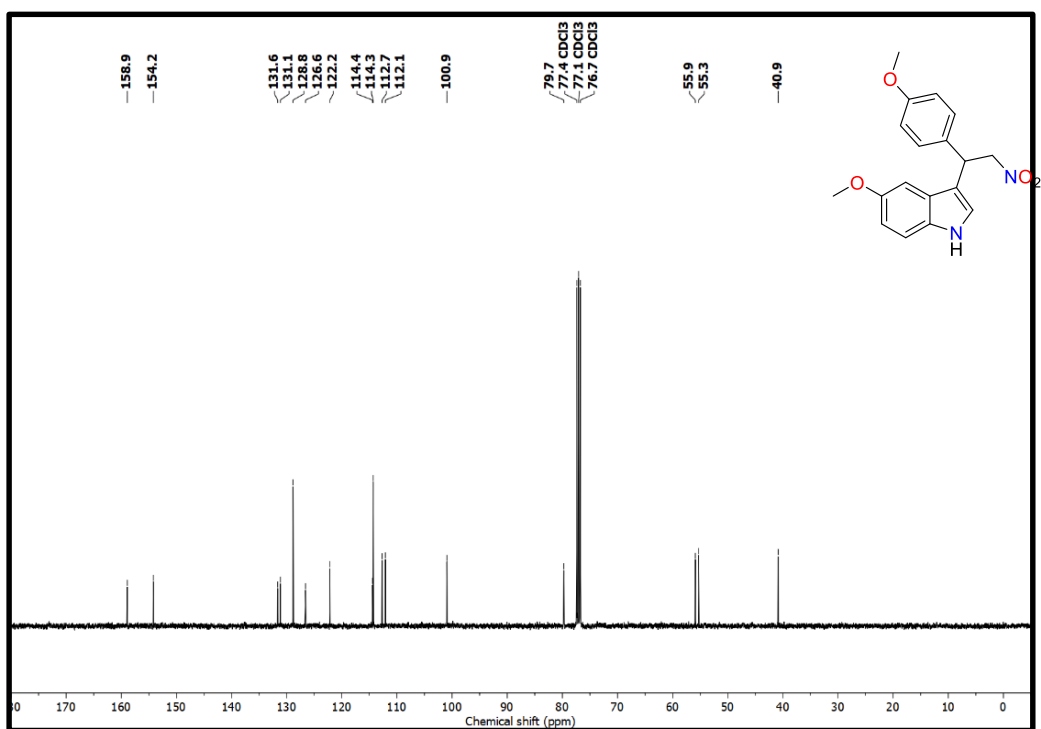


Figure S108: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3n** recorded in CDCl_3 .

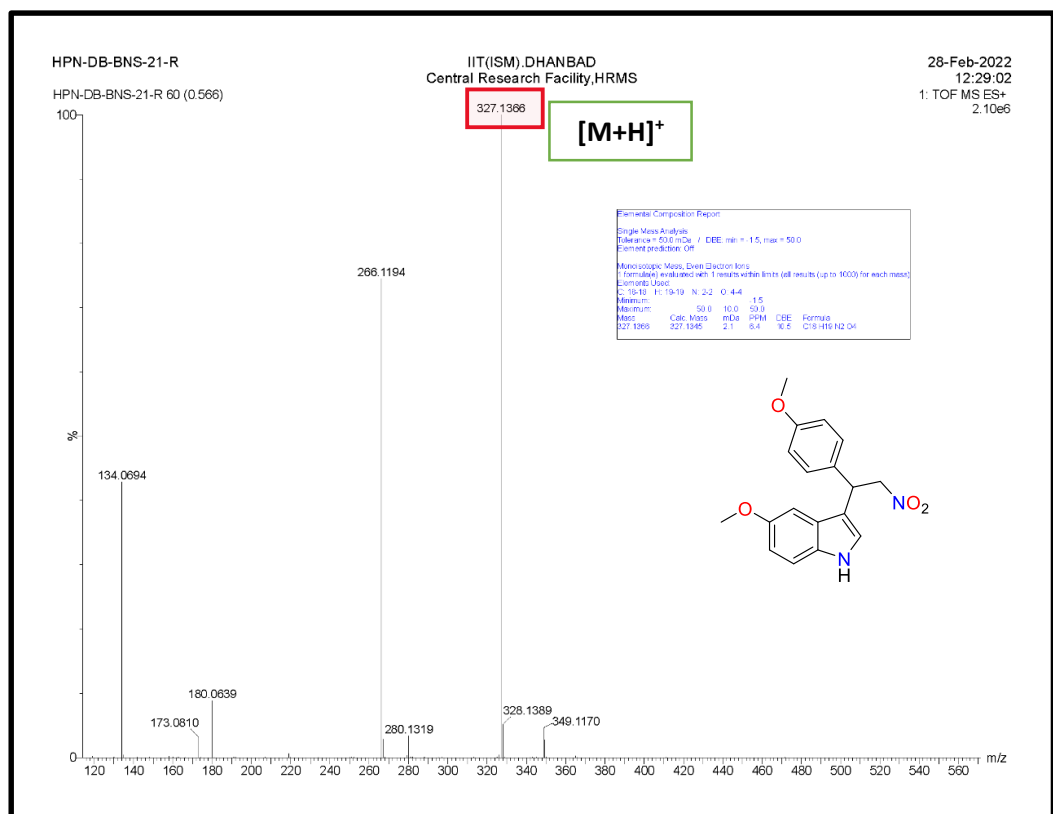


Figure S109: HRMS (ESI) spectrum of **3n**.

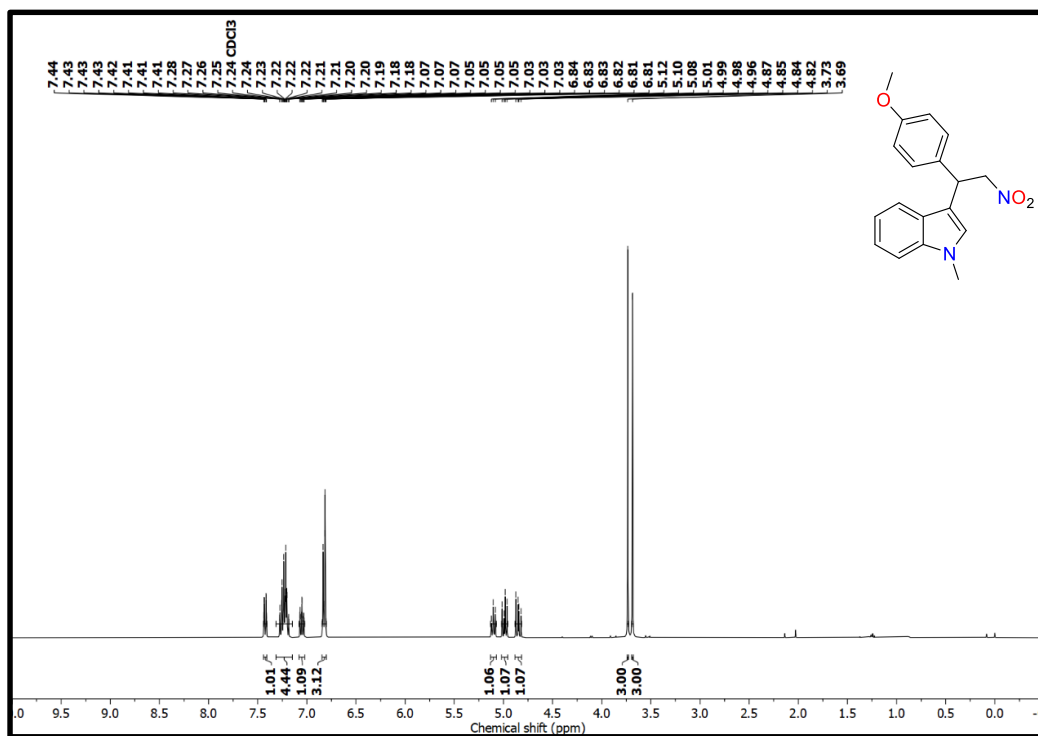


Figure S110: ^1H NMR spectrum of **3o** recorded in CDCl_3 .

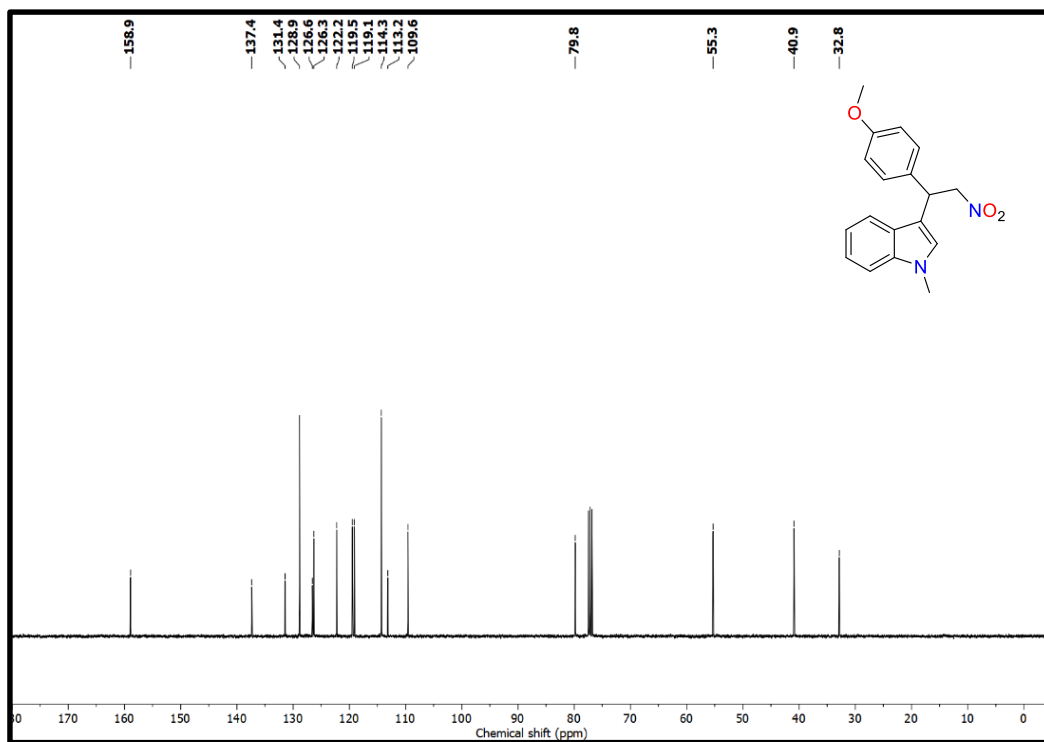


Figure S111: $^{13}\text{C}\{^1\text{H}\}$ NMR spectrum of **3o** recorded in CDCl_3 .

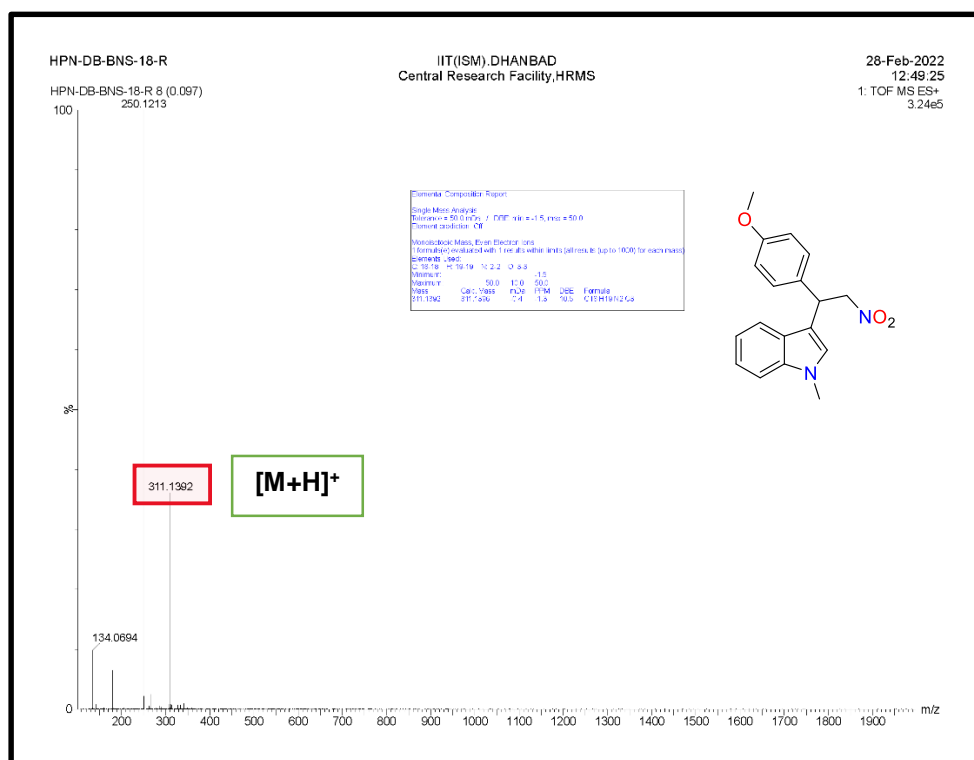


Figure S112: HRMS (ESI) spectrum of 30.

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