Supporting Information

Molecular Iodine Catalyzed C3-Quaternization of Indoles *via* Oxidative Dearomatization: Direct Access to 3,3-di(indolyl)indoline-2-Ones

Neetika Singh, Hari Prasad Kokatla Nagaraju Naddi and Madhu Inapanuri*

Department of Chemistry, National Institute of Technology Warangal, Warangal, Telangana-506004, India

Email: harikokatla@nitw.ac.in

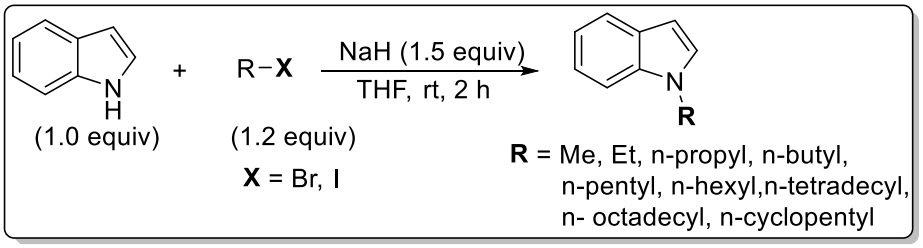
TABLE OF CONTENTS

1. General information

All chemicals and solvents were purchased from Alfa Aesar, Spectrochem, SRL, and Finar used as received. The confirmation of the reactions was monitored using analytical Thin Layer Chromatography (TLC), Merck silica gel G/GF 254 plates, and a UV-Cabinet for visualization of compound spots on the TLC plate. Purification of compounds using column chromatography was performed with the Merck silica gel (100-200 mesh). The melting points of solid compounds were determined by open capillaries using the Stuart SMP30 melting point apparatus and are uncorrected. NMR (^{1}H, ^{13}C and ^{19}F) spectra of all the synthesized compounds were recorded on a Bruker AVANCE HD (400 MHz, 100 MHz, and 376 MHz) spectrometer using $CDCl_3$ and DMSO-d_6 as solvents and TMS as an internal standard. The data of the compounds were recorded as chemical shifts (δ ppm) (multiplicity, coupling constant (Hz), integration). Abbreviations for the multiplicity are as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet. The mass spectrum analysis was recorded in a Bruker micro-TOF MS analyzer.

2. General procedures

2.1 General procedure for the synthesis of *N*-methylated indoles (1j-1t)[1,2]



To a well-stirred solution of indole derivative (10 mmol, 1 equiv) in THF (15 mL) at 0 ºC was added sodium hydride (60% in mineral oil, 15 mmol, 1.5 equiv). The reaction was warmed to room temperature and allowed to stir for 30 min. After 30 min, the reaction flask was cooled again to 0 ºC and alkyl halide (12 mmol, 1.2 equiv) was added dropwise. The reaction mixture was warmed to room temperature and

allowed to stir until the reaction was completed (monitored by TLC) and then cooled to 0 ºC and quenched with saturated aqueous NH_4Cl. The product was extracted with diethyl ether (3 x 20 mL) and dried over anhydrous Na_2SO_4. The organic phase was concentrated in vacuum to obtain the crude mixture, which was further purified by column chromatography (using 10% ethyl acetate/hexane), giving a desired compound up to 99% yield **(1j-1t)**.

2.2 General procedure for the synthesis of 3,3-di(indolyl)indoline-2-ones (2a-2t)

An oven-dried 25 mL reaction flask equipped with a magnetic stirring bar was charged with appropriate indole (1.0 mmol), iodine (10 mol%) in 2 mL of DMSO solvent. The reaction mixture was stirred at 100 °C using an oil bath for the appropriate time (5-9 h), and the progress of the reaction was monitored by TLC using hexane and ethyl acetate as an eluent. After the disappearance of the indole on TLC, the reaction was stopped. To this, sodium thiosulfate was added, and the organic compound was extracted with ethyl acetate (3 x 25 mL). Organic layers were washed with brine and with fresh water. The organic layers were dried on Na_2SO_4 and evaporated to give 3,3-di(indolyl)indoline-2-ones. The crude mixture was purified using column chromatography on silica gel by eluting with ethyl acetate/petroleum ether as a mobile phase.

References

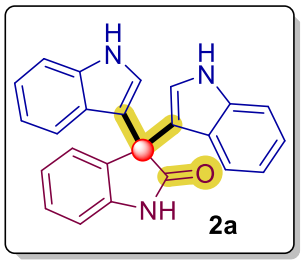
1. Kong, A; Han, X.; Lu, X. *Org. Lett.* 2006, **8**, 1339-1342.
2. Banjare, S. K; Nanda, T.P; Ravikumar, C. *Org. Lett*. 2019, **21**, 8138−8143.
3. K. X. Wu, Y. Z. Xu, L. Cheng, R. S. Wu, P. Z. Liu and D. Z. Xu, *Green Chem.,* 2021, 23, 8448–8452.
4. P. Kamboj, A. Mohapatra, D. Mandal and V. Tyagi, *Org. Biomol. Chem.,* 2024, **22**, 1839–1849.
5. G. Brahmachari and B. Banerjee, *ACS Sustain. Chem. Eng.*, 2014, **2**, 2802–2812

6. S. Kumar, S. K. Rastogi, A. Singh, M. Bharati Ahirwar, M. M. Deshmukh, A. K. Sinha and R. Kumar, *Asian J. Org. Chem.*, 2022, **11**, e202100749

7. W.-B. Qin, Q. Chang, H.-Q. Luo, Y.-H. Bao, Z.-W. Chen and L.-X. Liu, *Current Organic Synthesis*, 2013, ***10***, 495

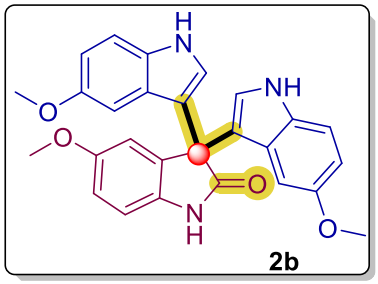
Characterization Data

1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2a)[3,4]



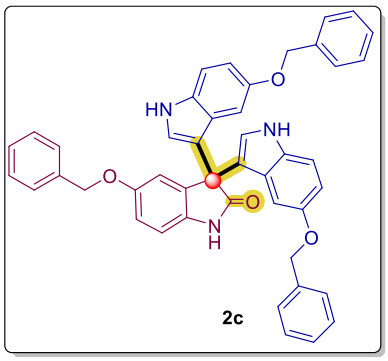
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); white solid; Yield: (234 mg, 84%); mp: 294-295 °C; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.96 (s, 2H), 10.60 (s, 1H), 7.36 (d, J = 8.0 Hz, 2H), 7.24 (d, J = 8.0 Hz, 4H), 7.05 – 6.99 (m, 3H), 6.94 (t, J = 7.2 Hz, 1H), 6.86 (d, J = 2.6 Hz, 2H), 6.81 (t, J = 7.4 Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 179.2, 141.8, 137.4, 135.1, 128.3, 126.2, 125.4, 124.7, 121.9, 121.4, 121.2, 118.7, 114.8, 112.1, 110.0, 53.0; HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_{24}H_{17}N_3NaO$ 386.1264; observed 386.1244.

5,5',5''-trimethoxy-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2b)[4]



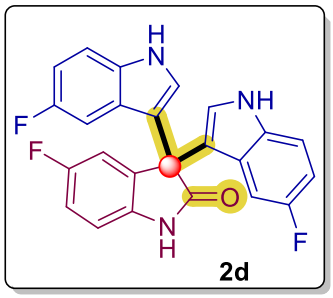
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3(v/v); light pink solid; Yield: (200 mg, 72%); mp: 241-242 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.82 (s, 2H), 10.46 (s, 1H), 7.30 – 7.26 (m, 2H), 6.95 (d, J = 8.2 Hz, 1H), 6.90 (s, 2H), 6.84 (dd, J = 8.4, 2.4 Hz, 1H), 6.79 (d, J = 2.6 Hz, 1H), 6.72 (d, J = 7.2 Hz, 4H), 3.63 (s, 3H), 3.55 (s, 6H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 179.1, 155.1, 152.9, 136.3, 135.3, 132.7, 126.6, 125.7, 114.1, 112.7, 112.6, 112.5, 110.9, 110.3, 103.8, 55.8, 55.6, 53.5. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{27}H_{24}N_3O_4$ 454.1761; observed 454.1723.

5,5',5''-tris(benzyloxy)-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2c)[7]



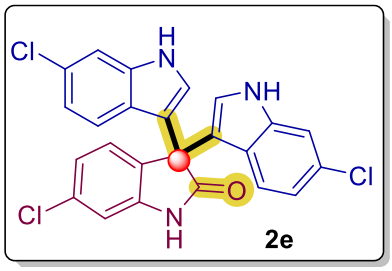
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); Pale yellow solid; Yield: (207 mg, 75%); mp: 228-229 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.82 (s, 2H), 10.45 (s, 1H), 7.30 (dt, J = 14.0, 4.4 Hz, 16H), 6.94 – 6.91 (m, 2H), 6.85 – 6.77 (m, 8H), 4.94 (s, 2H), 4.83 (s, 4H).^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 179.1, 154.1, 152.0, 138.0, 137.9, 137.6, 136.2, 135.4, 132.9, 128.8, 128.8, 128.7, 128.2, 128.2, 128.1, 128.1, 126.6, 125.7, 114.2, 114.1, 113.5, 112.5, 111.7, 110.4, 105.5, 70.4, 70.3, 53.4. HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_{45}H_{35}N_3NaO_4$ 704.2520; observed 704.2506.

5,5',5''-trifluoro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2d)[5]



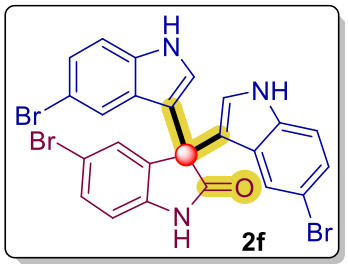
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); Pale brown solid; Yield: (234 mg, 84%); mp: 316-317 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.17 (s, 2H), 10.72 (s, 1H), 7.39 (dd, J = 8.6, 4.6 Hz, 2H), 7.14 – 7.01 (m, 5H), 6.90 (ddt, J = 12.8, 6.0, 2.6 Hz, 4H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 178.9, 159.6 (d, $^{1}J_{C-F}$ = 235.0 Hz), 157.9 (d, $^{1}J_{C-F}$ = 229.0 Hz), 137.9, 136.0 (d, $^{3}J_{C-F}$ = 8.0 Hz), 134.1, 126.9, 126.1 (d, $^{3}J_{C-F}$ = 8.0 Hz), 115.1 (d, $^{2}J_{C-F}$ = 23.0 Hz), 114.1 (d, $^{4}J_{C-F}$ = 4.0 Hz), 113.3 (d, $^{3}J_{C-F}$ = 10.0 Hz), 113.1 (d, $^{2}J_{C-F}$ = 24.0 Hz), 111.1 (d, $^{3}J_{C-F}$ = 8.0 Hz), 110.0 (d, $^{2}J_{C-F}$ = 26.0 Hz), 105.6 (d, $^{2}J_{C-F}$ = 23.0 Hz), 53.2. ^{19}F NMR (376 MHz, DMSO-d_6) δ (ppm): -120.24, -126.10. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{25}H_{15}F_3N_3O$ 418.1162; observed 418.1164.

6,6',6''-trichloro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2e)[7]



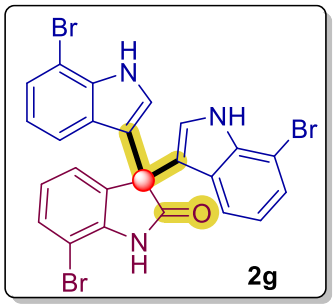
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3(v/v); Pale brown solid; Yield: (216 mg, 78%); mp: 278-279 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.18 (s, 2H), 10.79 (s, 1H), 7.40 (d, J = 11.6 Hz, 2H), 7.18 (td, J = 14.0, 8.4 Hz, 3H), 7.01 (d, J = 13.6 Hz, 2H), 6.93 – 6.82 (m, 4H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 178.7, 143.2, 137.8, 133.3, 132.7, 126.7, 126.4, 125.8, 124.8, 122.2, 121.9, 119.3, 114.3, 111.8, 110.3, 52.4. HRMS (ESI-TOF) m/z: [M+Na]$^{+}$ calcd for $C_{24}H_{14}Cl_3N_3NaO$ 488.0095; observed 488.0054.

5,5',5''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2f)[3,4]



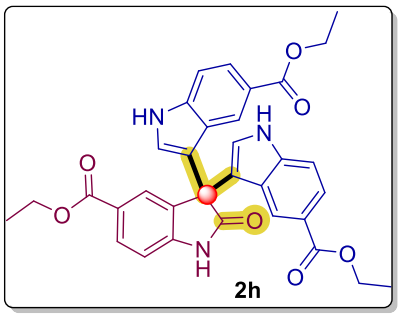
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); off white solid; Yield: (204 mg, 74%); mp: 310-311 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.29 (s, 2H), 10.91 (s, 1H), 7.48 (d, J = 7.8 Hz, 1H), 7.42 – 7.33 (m, 4H), 7.26 (s, 1H), 7.19 (d, J = 8.2 Hz, 2H), 7.05 – 6.93 (m, 3H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 178.5, 141.0, 136.4, 136.2, 131.6, 127.9, 127.5, 126.7, 124.3, 122.9, 114.5, 114.0, 113.5, 112.4, 111.7, 52.8. HRMS (ESI-TOF) m/z: [M+H]$^{+}$ calcd for $C_{24}H_{15}Br_3N_3O$ 597.8760; observed 597.8677.

7,7',7''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2g)



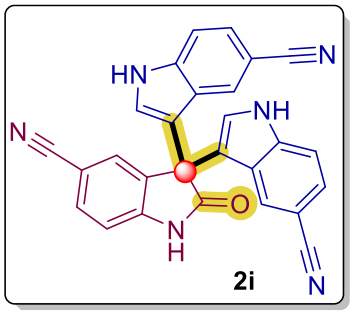
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); Pale brown solid; Yield: (215 mg, 78%); mp: 294-295 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.33 (s, 2H), 11.01 (s, 1H), 7.48 – 7.45 (m, 1H), 7.29 (d, J = 7.4 Hz, 2H), 7.23 (d, J = 7.6 Hz, 3H), 6.94 (d, J = 7.8 Hz, 1H), 6.89 (d, J = 2.6 Hz, 2H), 6.83 (d, J = 7.8 Hz, 2H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 178.4, 141.2, 135.8, 135.6, 131.7, 127.6, 126.0, 124.5, 124.4, 124.0, 120.6, 120.5, 115.4, 104.9, 102.7, 54.1. HRMS (ESI-TOF) m/z: [M+H]$^{+}$ calcd for $C_{24}H_{15}Br_3N_3O$ 597.8760; observed 597.8675.

triethyl 2'-oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarboxylate (2h)



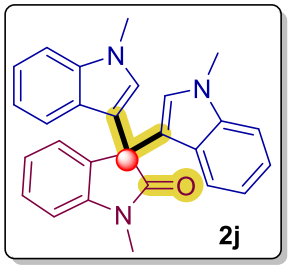
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); White solid; Yield: (155 mg, 56%); mp: 270-271 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.47 (s, 2H), 11.24 (s, 1H), 8.02 – 7.96 (m, 3H), 7.78 (q, J = 1.6 Hz, 1H), 7.70 (dt, J = 8.6, 1.4 Hz, 2H), 7.49 (dd, J = 8.4, 1.8 Hz, 2H), 7.18 (dd, J = 8.2, 2.0 Hz, 1H), 7.07 (d, J = 2.6 Hz, 2H), 4.20 (dq, J = 10.6, 7.0 Hz, 6H), 1.24 (dt, J = 18.2, 7.2 Hz, 9H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 183.8, 171.8, 170.7, 151.2, 144.9, 139.3, 135.9, 131.6, 130.7, 130.2, 128.6, 128.4, 127.7, 125.7, 120.1, 117.0, 115.1, 65.6, 65.2, 57.5, 19.5, 19.3. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{33}H_{30}N_3O_7$ 580.2078; observed 580.2079.

2'-oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarbonitrile (2i)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); Pale yellow solid; Yield: (145 mg, 52%); mp: 272-273 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.74 (d, J = 2.4 Hz, 2H), 11.34 (s, 1H), 7.79 (dd, J = 8.0, 1.6 Hz, 1H), 7.75 (d, J = 1.6 Hz, 1H), 7.59 (dd, J = 2.6, 1.2 Hz, 3H), 7.58 (d, J = 0.6 Hz, 1H), 7.43 (d, J = 1.4 Hz, 1H), 7.41 (d, J = 1.4 Hz, 1H), 7.22 – 7.19 (m, 3H).^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 178.5, 145.9, 139.3, 134.5, 129.0, 127.9, 126.1, 125.4, 124.5, 121.1, 119.8, 114.3, 113.9, 111.5, 104.8, 101.3, 52.3. HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_{27}H_{14}N_6NaO$ 461.1121; observed 461.1122.

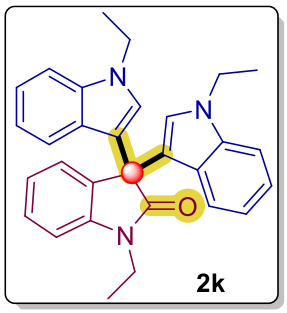
1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2j)[6]



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8:2 (v/v); off White solid; Yield: (262 mg, 94%); mp: 222-223 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.37 (t, J = 8.4 Hz, 3H), 7.33 – 7.29 (m, 1H), 7.18 (dd, J = 11.0, 7.8 Hz, 3H), 7.09 (t, J = 7.6 Hz, 2H), 7.03 (t, J = 7.4 Hz, 1H),

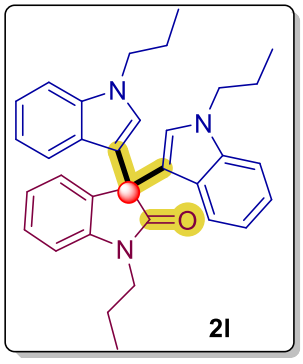
6.89 (s, 2H), 6.85 (t, J = 7.4 Hz, 2H), 3.70 (s, 6H), 3.27 (s, 3H). $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 177.2, 143.1, 137.8, 134.0, 129.0, 128.5, 126.4, 125.0, 122.8, 121.6, 121.3, 119.0, 113.6, 110.3, 109.2, 52.5, 32.8, 26.8. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{27}H_{14}N_3O$ 406.1914 observed 406.1902.

1,1',1''-triethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2k)



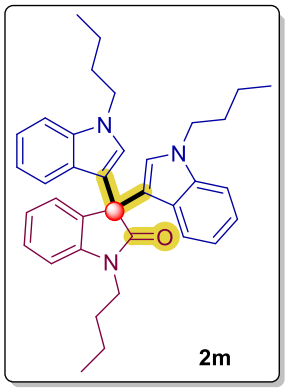
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8:2 (v/v); light brown solid; Yield: (247 mg, 89%); mp: 164-165 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.42 (d, J = 8.2 Hz, 2H), 7.36 – 7.30 (m, 2H), 7.19 (dd, J = 14.2, 7.8 Hz, 3H), 7.07 (t, J = 7.6 Hz, 2H), 7.01 (t, J = 7.4 Hz, 1H), 6.92 (s, 2H), 6.83 (t, J = 7.4 Hz, 2H), 4.13 (q, J = 7.2 Hz, 4H), 3.83 (q, J = 6.8 Hz, 2H), 1.27 (t, J = 6.4 Hz, 9H). $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 176.8, 142.1, 136.8, 134.3, 128.5, 127.4, 126.6, 125.3, 122.6, 121.5, 121.4, 118.8, 113.8, 110.3, 109.3, 52.6, 34.8, 15.9, 13.0. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{30}H_{30}N_3O$ 448.2383; observed 448.2379.

1,1',1''-tripropyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2l)



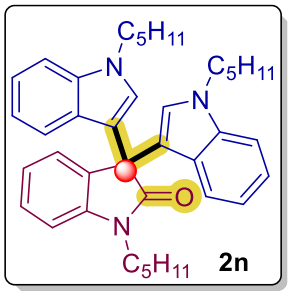
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 9:1 (v/v); White solid; Yield: (246 mg, 89%); mp: 133-134 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.42 (d, J = 8.2 Hz, 2H), 7.32 (p, J = 3.8 Hz, 2H), 7.19 (s, 2H), 7.03 (dt, J = 23.4, 7.4 Hz, 4H), 6.89 (s, 2H), 6.81 (t, J = 7.4 Hz, 2H), 4.04 (t, J = 6.8 Hz, 4H), 3.76 (t, J = 6.8 Hz, 2H), 1.68 (t, J = 8.2 Hz, 6H), 0.87 (t, J = 7.2 Hz, 3H), 0.78 (t, J = 7.2 Hz, 6H). $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 177.2, 142.6, 137.1, 134.2, 128.5, 126.5, 125.2, 122.6, 121.5, 121.5, 118.7, 113.8, 110.4, 109.4, 52.5, 47.4, 41.5, 23.5, 20.8, 11.6, 11.5. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{33}H_{36}N_3O$ 490.2853; observed 490.2787.

1,1',1''-tributyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2m)



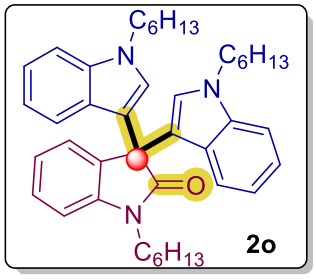
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 9:1 (v/v); White solid; Yield: (248 mg, 90%); mp: 132-133 °C. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.41 (d, J = 8.2 Hz, 2H), 7.34 – 7.29 (m, 2H), 7.18 (t, J = 7.8 Hz, 3H), 7.06 (q, J = 5.4 Hz, 2H), 7.00 (t, J = 7.4 Hz, 1H), 6.87 (s, 2H), 6.80 (t, J = 7.4 Hz, 2H), 4.08 (t, J = 6.8 Hz, 4H), 3.79 (t, J = 7.2 Hz, 2H), 1.67 – 1.60 (m, 6H), 1.30 (q, J = 7.4 Hz, 2H), 1.23 – 1.14 (m, 4H), 0.89 – 0.81 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 177.1, 142.5, 137.1, 134.1, 128.5, 128.0, 126.5, 125.2, 122.6, 121.5, 121.4, 118.7, 113.8, 110.4, 109.4, 52.5, 45.5, 32.3, 29.6, 20.1, 19.9, 14.1, 14.0. HRMS (ESI-TOF) m/z: [M+H]$^+$ calcd for $C_{36}H_{42}N_3O$ 532.3322; observed 532.3289.

1,1',1''-tripentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2n)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 9:1 (v/v); light yellow oil; Yield: (242 mg, 88%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.35 (dd, J = 7.6, 1.2 Hz, 1H), 7.24 – 7.18 (m, 5H), 7.03 (ddd, J = 8.0, 6.8, 1.2 Hz, 2H), 6.93 – 6.88 (m, 2H), 6.83 – 6.80 (m, 2H), 6.79 (s, 2H), 3.94 – 3.89 (m, 4H), 3.76 – 3.72 (m, 2H), 1.69 (p, J = 7.2 Hz, 6H), 1.29 – 1.18 (m, 12H), 0.81 – 0.76 (m, 9H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ (ppm): 177.6, 142.5, 137.0, 134.6, 127.9, 127.8, 126.6, 125.5, 122.4, 121.3, 121.6, 118.8, 113.9, 109.5, 108.4, 52.8, 46.4, 40.4, 29.9, 29.7, 29.2, 29.1, 27.2, 22.4, 22.3, 14.2, 14.0. HRMS (ESI-TOF) m/z: [M+H]$^+$ calcd for $C_{39}H_{48}N_3O$ 574.3792; observed 574.2886.

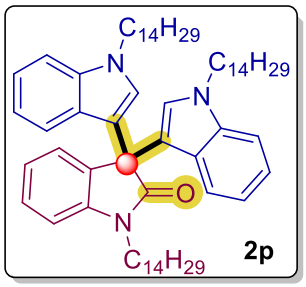
1,1',1''-trihexyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2o)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 9:1 (v/v); brown oil; Yield: (220 mg, 80%). ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.48 (dd, J = 7.6, 1.2 Hz, 1H), 7.44 – 7.31 (m, 5H), 7.17 (ddd, J = 8.2, 6.8, 1.2 Hz, 2H), 7.10 – 7.01 (m, 2H), 6.96 – 6.93 (m, 1H), 6.92 (s, 2H), 6.88

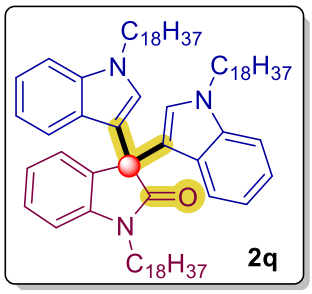
(d, J = 8.2 Hz, 1H), 4.07 – 3.98 (m, 4H), 3.86 (q, J = 6.4 Hz, 2H), 1.87 – 1.77 (m, 6H), 1.48 – 1.28 (m, 22H), 0.91 (dq, J = 5.4, 3.2 Hz, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 177.6, 142.5, 137.0, 134.6, 127.9, 126.6, 125.5, 122.4, 121.6, 121.3, 118.8, 113.9, 109.5, 108.4, 52.8, 46.4, 40.4, 31.6, 31.4, 30.2, 27.5, 26.8,26.7, 22.6, 22.4, 22.3, 14.1. HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_{42}H_{53}N_3NaO$ 638.4081; observed 638.4081.

1,1',1''-tritetradecyl-[3,3':3',3''-terindolin]-2'-one (2p)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 9.5:0.5 (v/v); brown oil; Yield: (192 mg, 70%). ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.46 (dd, J = 7.6, 1.2 Hz, 1H), 7.31 (dd, J = 8.2, 6.8 Hz, 5H), 7.17 – 7.12 (m, 2H), 7.04 – 6.99 (m, 2H), 6.94 – 6.91 (m, 2H), 6.89 (s, 2H), 4.02 (t, J = 7.2 Hz, 4H), 3.85 (t, J = 7.2 Hz, 2H), 1.83 – 1.76 (m, 6H), 1.29 (d, J = 8.2 Hz, 66H), 0.92 (s, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 177.6, 142.5, 137.0, 134.6, 127.8, 127.8, 126.6, 125.5, 122.3, 121.6, 121.2, 118.8, 113.8, 109.4, 108.4, 52.8, 46.4, 40.4, 32.0, 30.2, 29.7, 29.7, 29.6, 29.6, 29.4, 29.3, 27.5, 27.1, 27.0, 22.7, 14.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{66}H_{102}N_3O$ 952.8017; observed 952.7836.

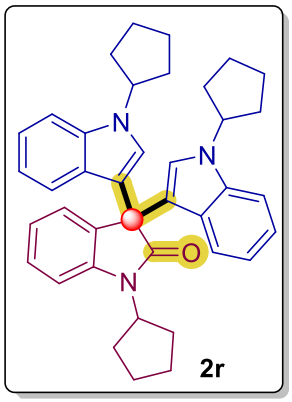
1,1',1''-trioctadecyl-[2,3':3',2''-terindolin]-2'-one (2q)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 9.5:0.5 (v/v); light pink solid; Yield: (191 mg, 70%); mp: 79-80 °C. ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.35 (dd, J = 7.6, 1.2 Hz, 1H), 7.24 – 7.19 (m, 3H), 7.18 (s, 2H), 7.03 (ddd, J = 8.0, 6.8, 1.2 Hz, 2H), 6.92 – 6.88 (m, 2H), 6.83 – 6.79 (m, 2H), 6.78 (s, 2H), 3.95 – 3.87 (m, 4H), 3.74 (t, J = 7.4 Hz, 2H), 1.72 – 1.64 (m, 6H), 1.17 (d, J = 8.8 Hz, 90H), 0.81 (t, J = 6.8 Hz, 9H). $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 177.6, 142.4, 137.0, 127.8, 127.8, 126.6, 125.5, 122.3, 121.6, 121.2, 118.8, 113.8, 109.4, 108.4, 52.8, 46.4, 40.4, 32.0,

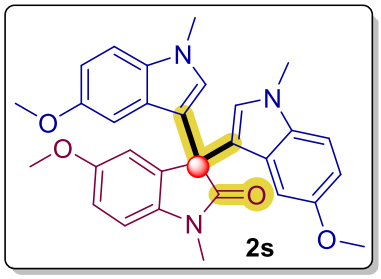
30.2, 29.7, 29.7, 29.6, 29.6, 29.4, 29.3, 27.5, 27.1, 27.0, 22.7, 14.2. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{78}H_{126}N_3O$ 1120.9895; observed 1120.9967.

1,1',1''-tricyclopentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2r)



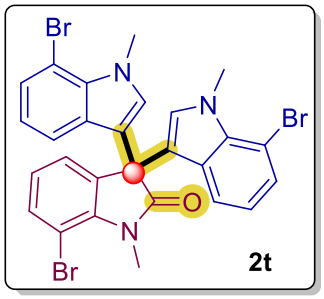
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8.5:1.5 (v/v); light yellow solid; Yield: (232 mg, 84%); mp: 198-199 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.31 (dd, J = 7.4, 1.2 Hz, 1H), 7.26 (dd, J = 8.4, 0.8 Hz, 2H), 7.19 (dd, J = 7.8, 1.2 Hz, 1H), 7.16 – 7.12 (m, 2H), 7.05 – 6.98 (m, 3H), 6.89 (s, 2H), 6.86 (dd, J = 7.4, 0.8 Hz, 1H), 6.81 (td, J = 7.4, 0.8 Hz, 2H), 4.87 (p, J = 8.8 Hz, 1H), 4.63 (p, J = 7.2 Hz, 2H), 2.18 – 2.02 (m, 6H), 1.94 – 1.86 (m, 4H), 1.82 – 1.72 (m, 4H), 1.71 – 1.57 (m, 10H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ (ppm): 177.8, 141.4, 137.2, 135.0, 127.4, 126.8, 125.7, 124.8, 122.1, 121.4, 121.1, 118.8, 114.0, 109.9, 109.7, 57.1, 52.9, 52.5, 32.4, 32.3, 27.6, 25.3, 23.9. HRMS (ESI-TOF) m/z: $[M+Na]^+$ calcd for $C_{39}H_{41}N_3NaO$ 590.3142; observed 590.3143.

5,5',5''-trimethoxy-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2s)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8:2(v/v); pale green solid; Yield: (244 mg, 88%); mp: 238-239 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.06 (d, J = 8.8 Hz, 2H), 6.95 (d, J = 2.4 Hz, 1H), 6.82 – 6.73 (m, 6H), 6.67 (d, J = 2.4 Hz, 2H), 3.62 (s, 3H), 3.58 (s, 6H), 3.55 (s, 6H), 3.24 (s, 3H). ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ (ppm): 177.6, 156.0, 153.5, 136.7, 135.5, 133.2, 129.4, 126.7, 112.9, 112.9, 112.2, 111.8, 109.9, 108.2, 103.4, 55.8, 55.7, 53.1, 33.0, 26.7. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{30}H_{30}N_3O_4$ 496.2231; observed 496.2230.

7,7',7''-tribromo-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2t)



Purified by column chromatography eluting with petroleum ether/ethyl acetate = 8:2 (v/v); Yellow solid; Yield: (209 mg, 76%); mp: 260-261 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.36 (dd, J = 8.2, 1.2 Hz, 1H), 7.21 – 7.15 (m, 6H), 6.78 (dd, J = 8.2, 7.4 Hz, 1H), 6.67 (t, J = 7.8 Hz, 2H), 6.58 (s, 2H), 3.98 (s, 6H), 3.61 (s, 3H).^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ (ppm): 177.6, 140.2, 136.3, 134.1, 134.0, 131.6, 129.3, 127.0, 124.2, 123.9, 120.7, 120.5, 113.0, 104.0, 102.6, 52.0, 37.1, 30.4. HRMS (ESI-TOF) m/z: $[M+H]^+$ calcd for $C_{27}H_{21}Br_3N_3O$ 639.9229; observed 639.9267.

DEPT-135 NMR study (100 MHz, DMSO-d_6) spectrum of 1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2j)

The DEPT-135 spectrum of compound **2j** was recorded to confirm the nature of the carbon signals observed in the decoupled ^{13}C NMR spectrum. Quaternary carbons at δ 52.5 ppm, which do not carry directly bonded protons, were absent from the DEPT-135 spectrum but were clearly visible in the ^{13}C{^{1}H} spectrum, thereby allowing their distinction from protonated carbons. Importantly, the carbonyl carbon resonance at δ 177.2 ppm, observed in the ^{13}C spectrum, disappeared completely in the DEPT-135 experiment, consistent with its quaternary nature. This analysis corroborates the proposed molecular framework of compound **2j**, ensuring that all carbon resonances, including the quaternary carbonyl centre, were identified and consistent with the expected substitution pattern of the structure (Figure S1).

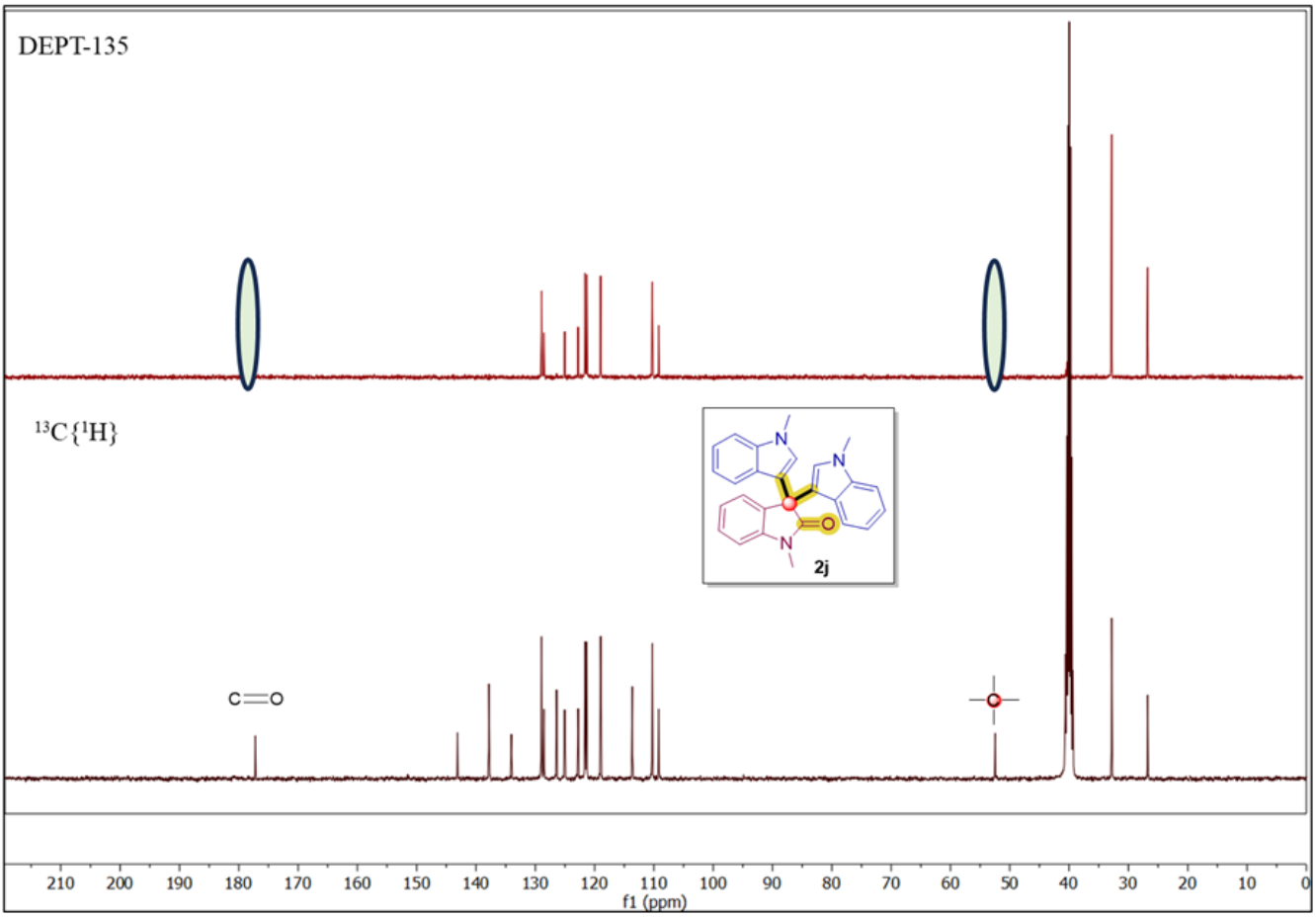


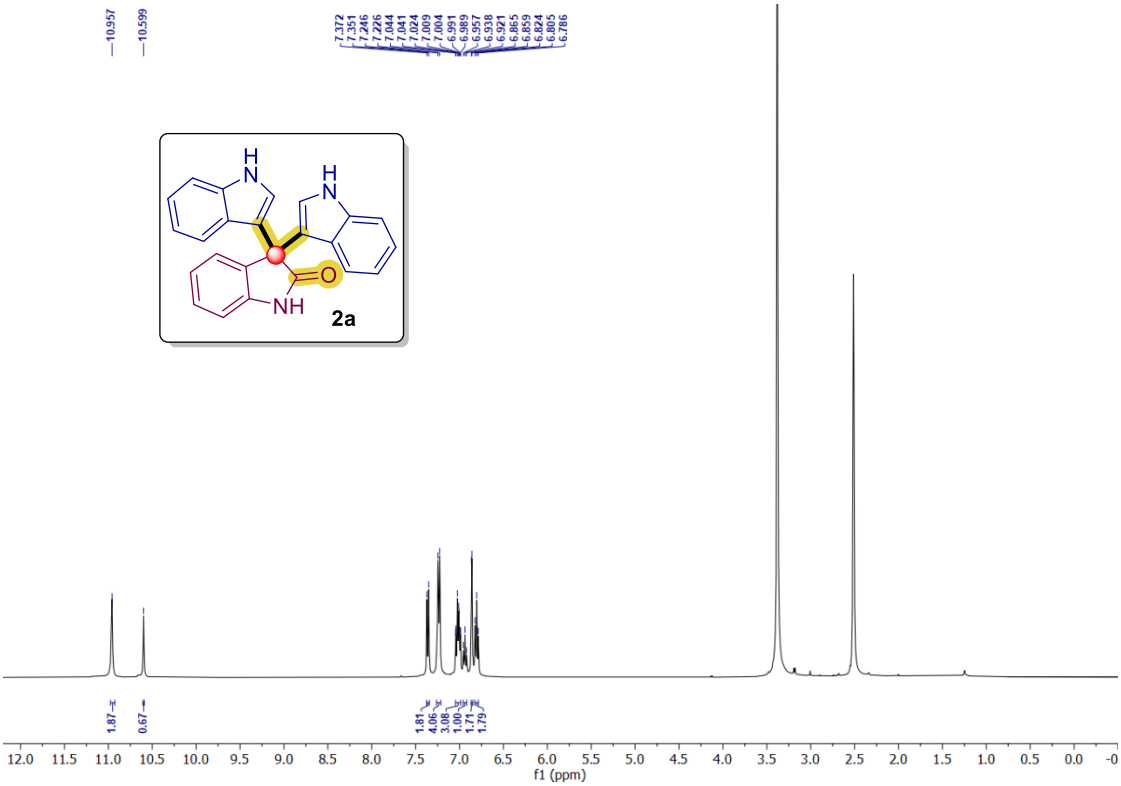
Figure S1: DEPT-135 NMR study (100 MHz, DMSO-d_6) spectrum of 1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2j)

Table S1: Crystallographic Data and Structure Refinement of Compound (**2j**).

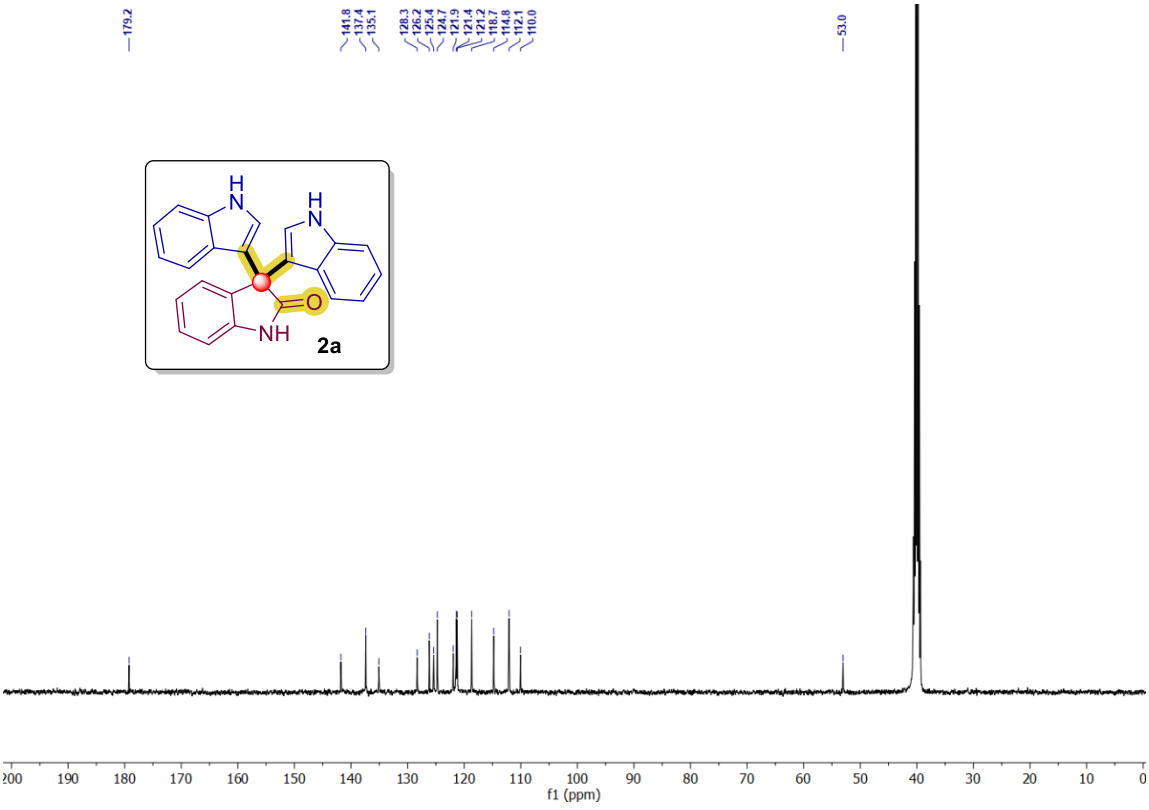
Identification Code	**Compound 2j (EXTER108_0m_a)**
Empirical formula	$C_{27}H_{23}N_3O$
Formula weight	405.48
Temperature/K	297.0
Crystal system	Monoclinic
Radiation	MoKα (λ = 0.71073)

Space group	$P2_1/c$
a/Å	11.2233(18)
b/Å	21.237(3)
c/Å	9.1008(12)
α/°	90
β/°	101.530(6)
γ/°	90
Volume/Å^3	2125.4(5)
Z	4
ρ_{calc} g/cm^3	1.267
μ/mm^{-1}	0.078
F(000)	856.0
Crystal size/mm^3	0.121 x 0.111 x 0.201
2Θ range for data collection/°	3.704 to 55.246
Index ranges	$-14 \leq h \leq 14$, $-27 \leq k \leq 27$, $-11 \leq l \leq 11$
Reflections collected	33877
Independent reflections	4913 [R_{int} = 0.0595, R_{sigma} = 0.0402]
Data/restraints/parameters	4913/0/283
Goodness-of-fit on F^2	1.062
Final R indexes [I>=2σ (I)]	R_1 = 0.0553, wR_2 = 0.1531
Final R indexes [all data]	R_1 = 0.0714, wR_2 = 0.1624
Largest diff. peak/hole / e Å^{-3}	0.27/-0.22
CCDC	2490627

^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2a)

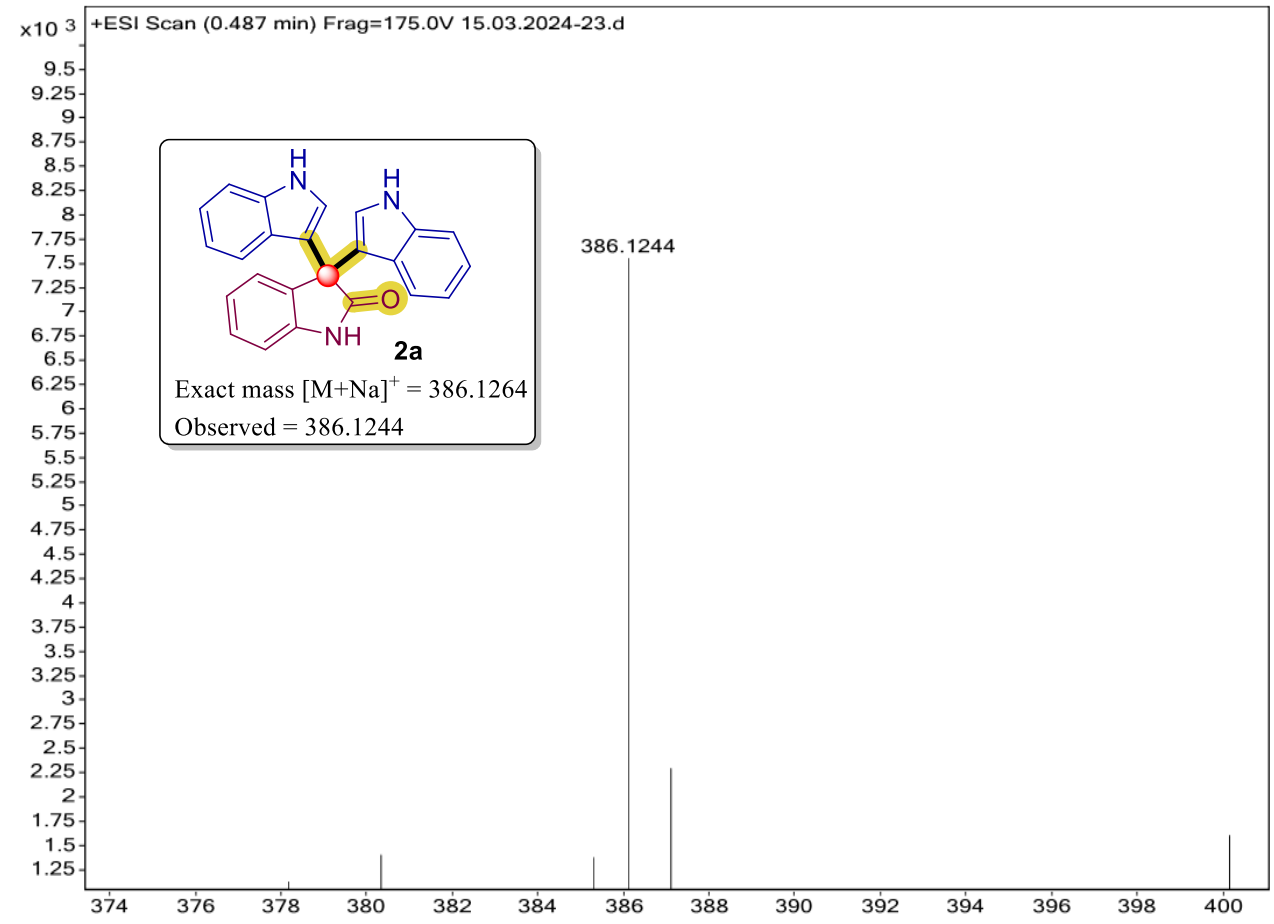


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2a)

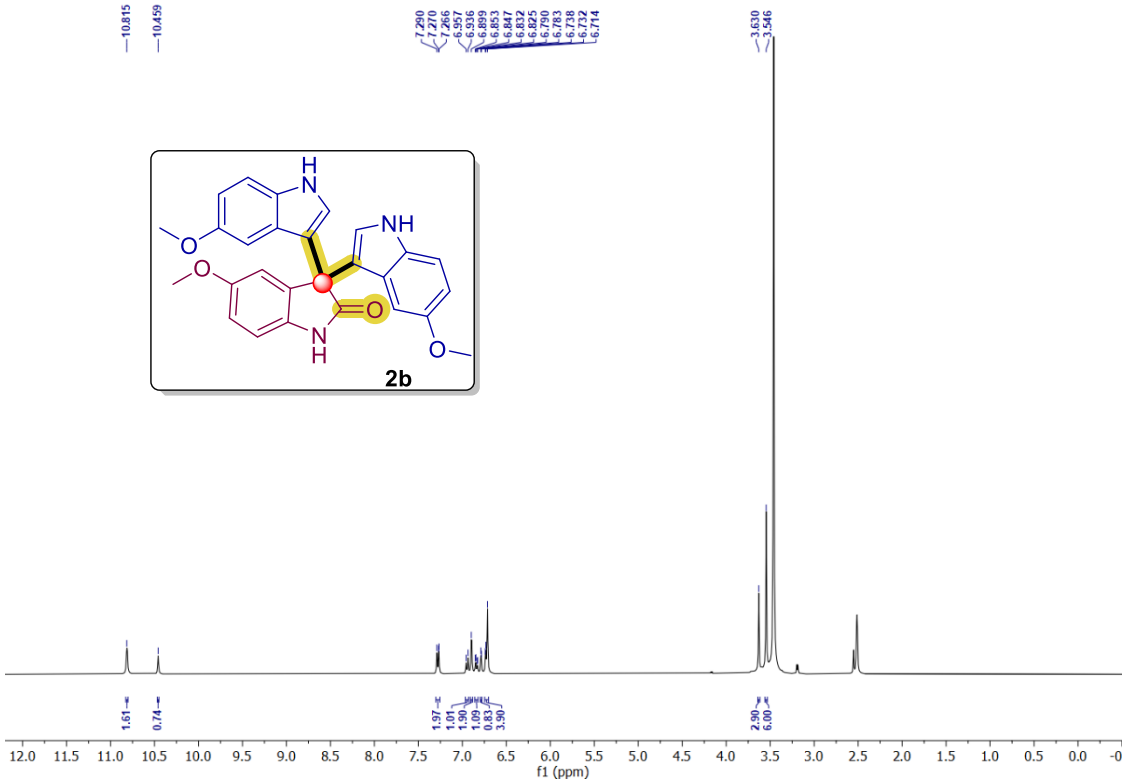


HRMS spectrum of 1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2a)

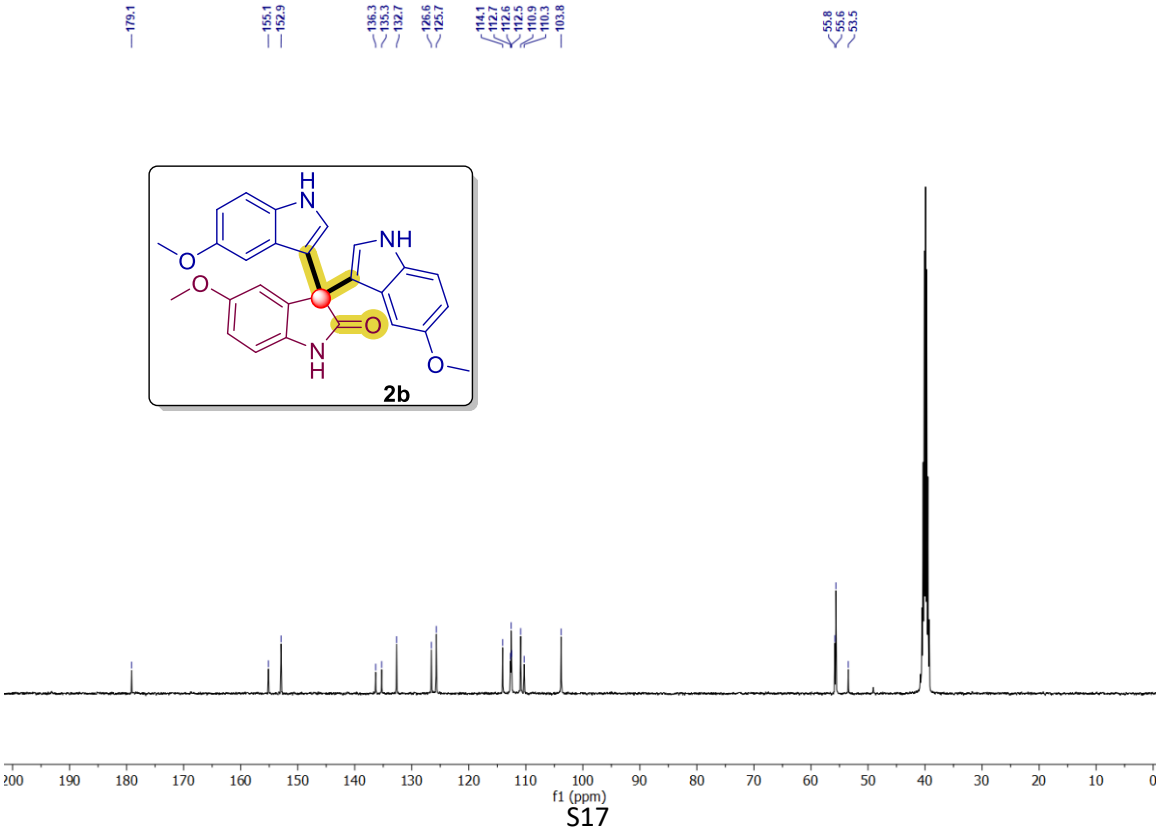
Sample Name	exp-5	**Position**	P1-C5	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-23.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:28:33



^{1}H NMR (400 MHz, DMSO-*d*$_6$) spectrum of 5,5',5''-trimethoxy-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2b)

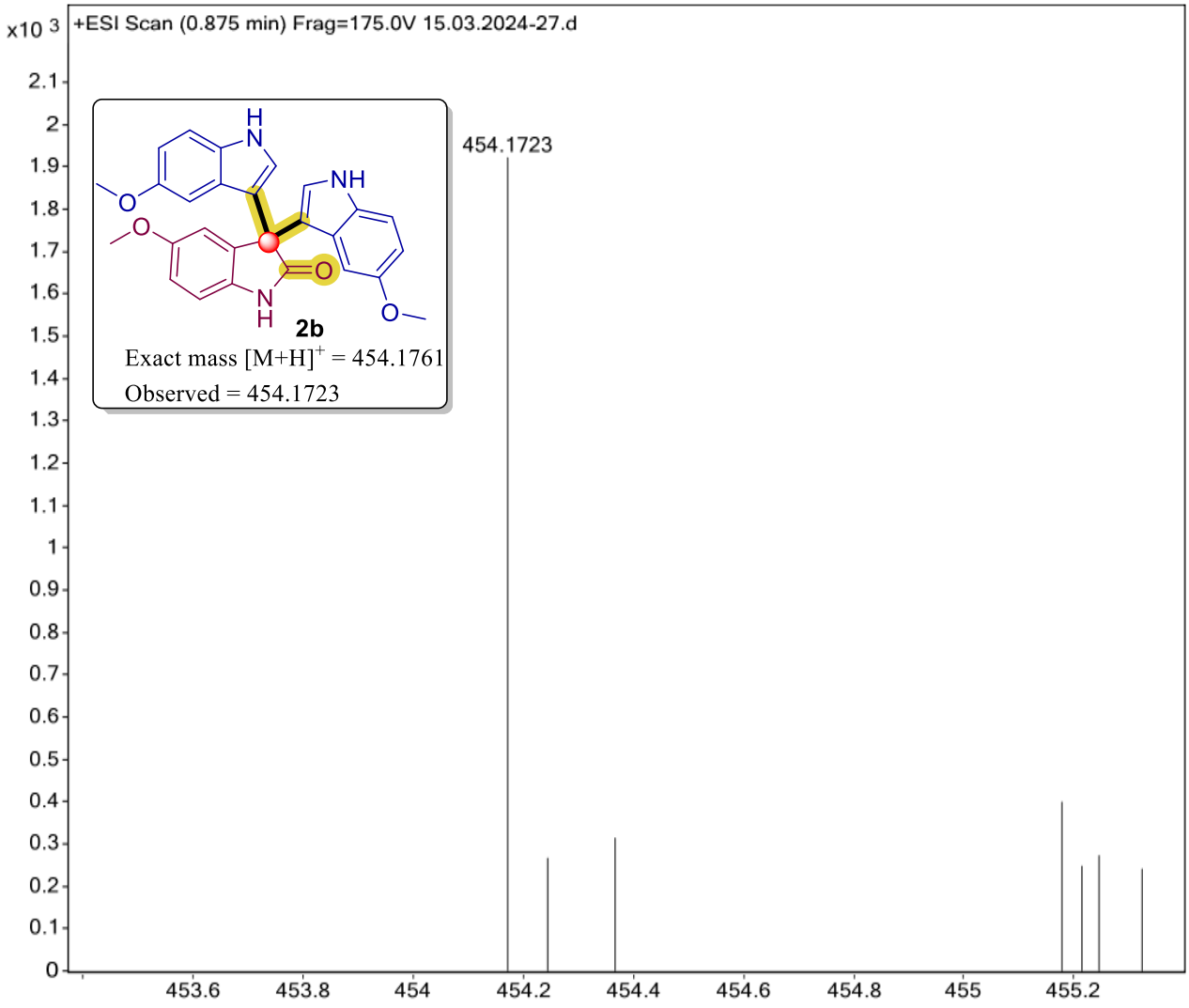


^{13}C{^{1}H} NMR (100 MHz, DMSO-*d*$_6$) spectrum of 5,5',5''-trimethoxy-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2b)

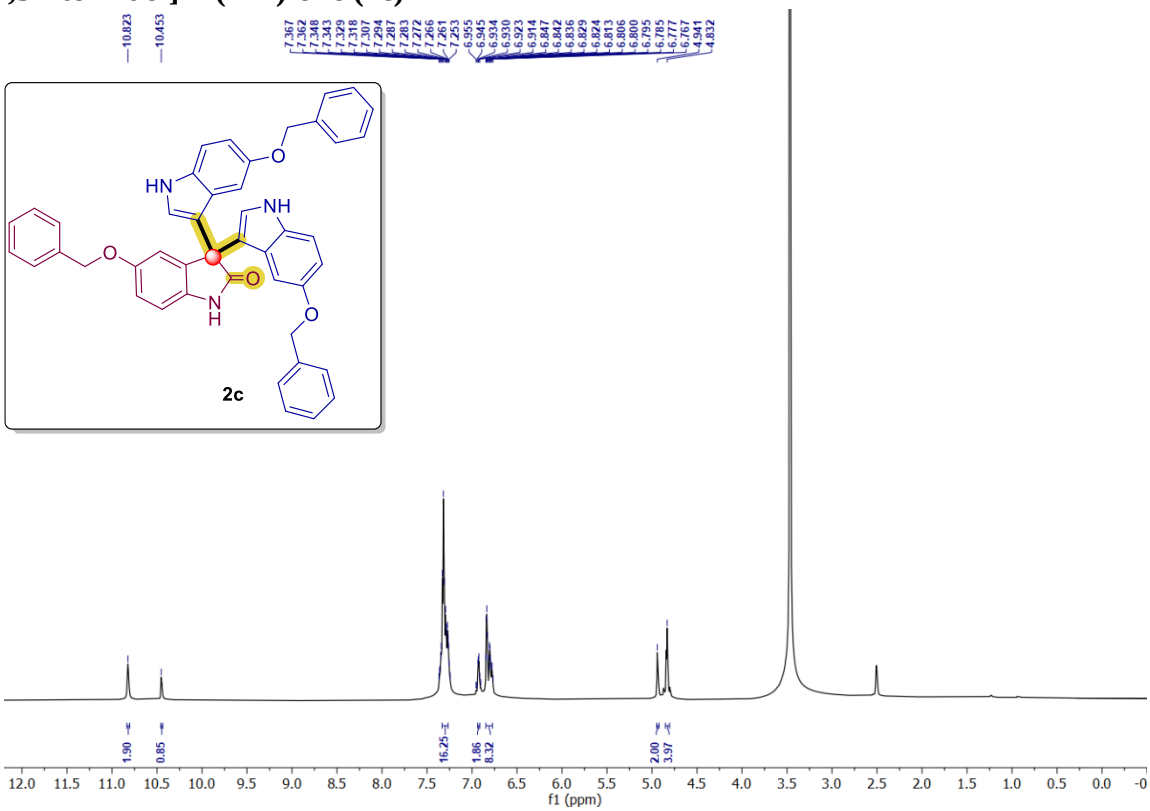


HRMS spectrum of 5,5',5''-trimethoxy-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2b)

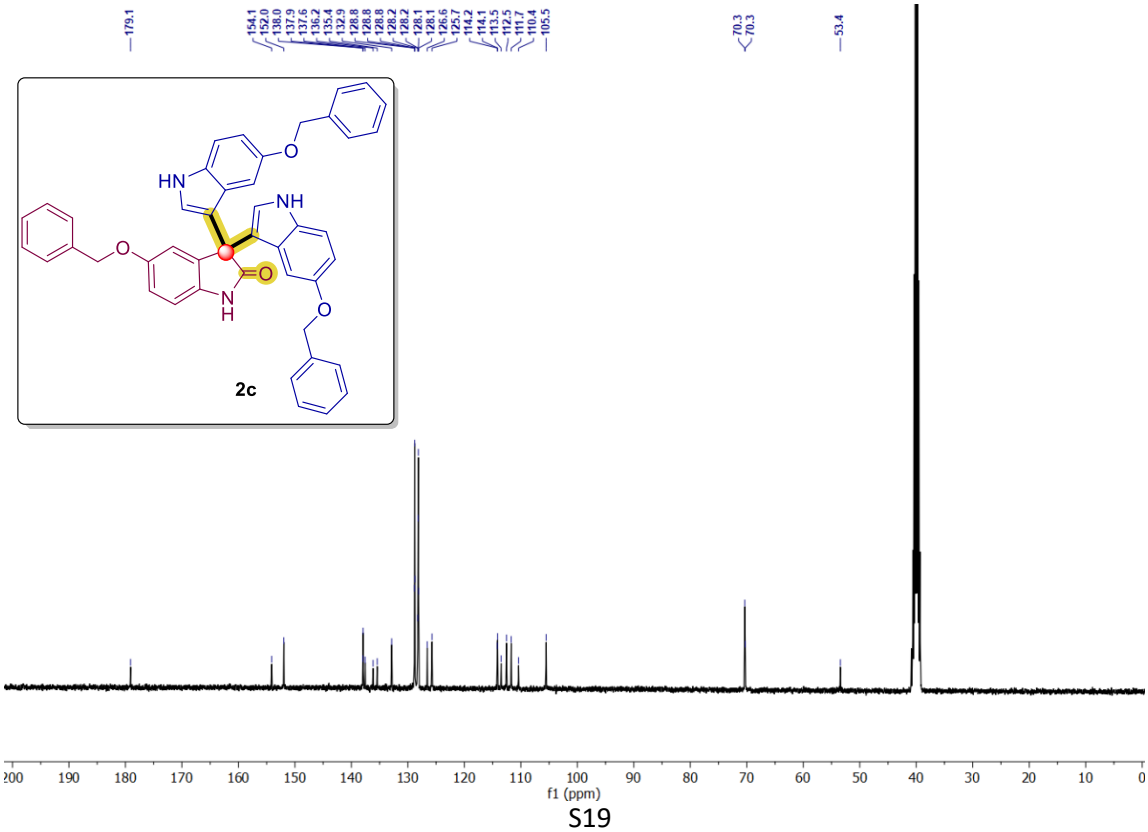
Sample Name	exp-9	**Position**	P1-C9	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-27.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:40:35



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5,5',5''-tris(benzyloxy)-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2c)

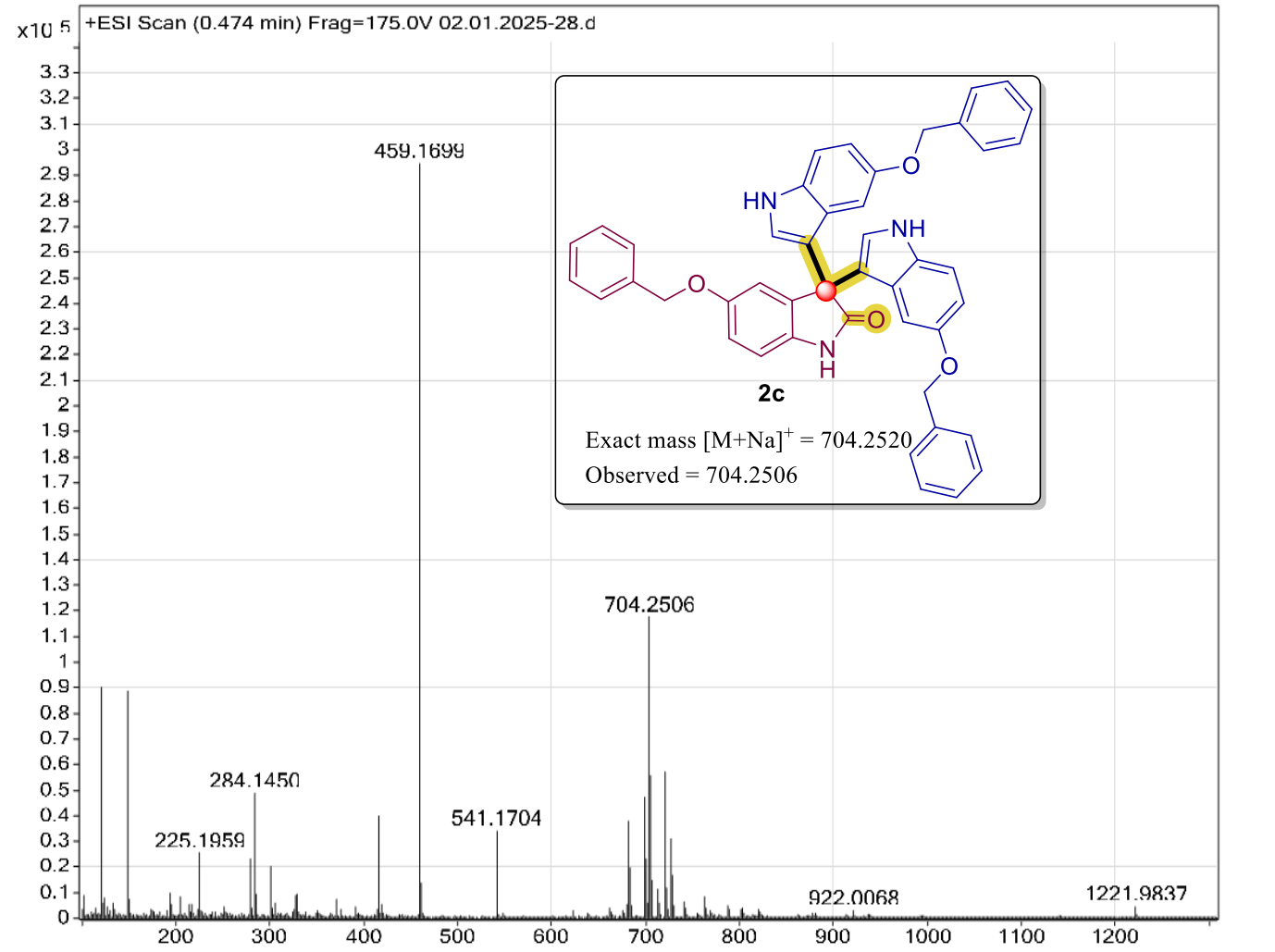


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5,5',5''-tris(benzyloxy)-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2c)

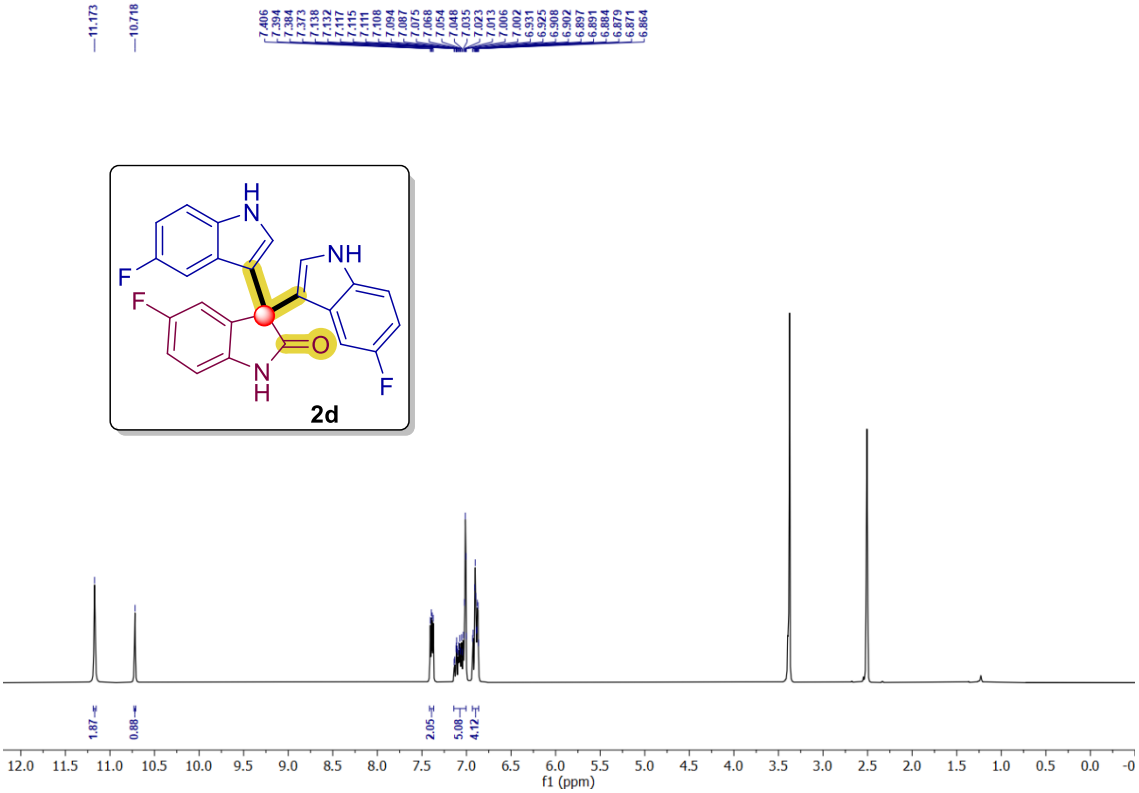


HRMS spectrum of 5,5',5''-tris(benzyloxy)-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2c)

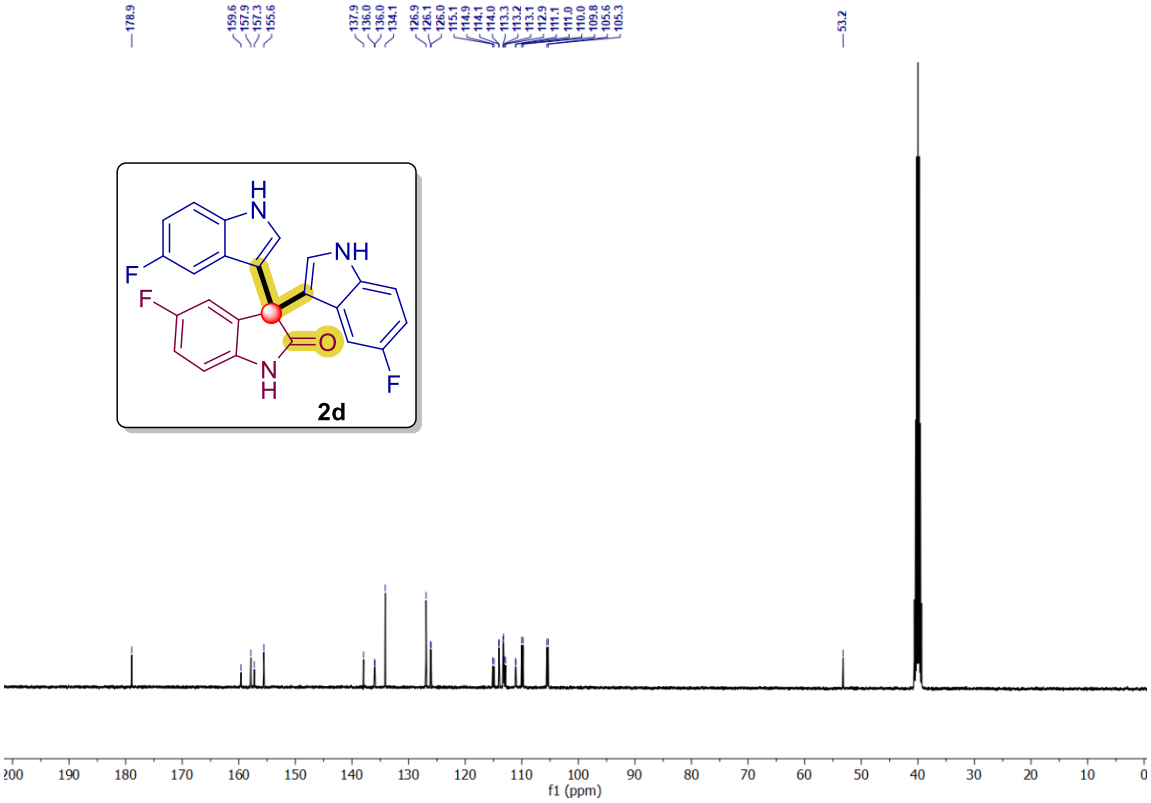
Sample Name	khp-ns-obn	**Position**	P1-D1	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	02.01.2025-28.d	**ACQ Method**	M60 W40.m	**Comment**		**Acquired Time**	03-01-2025 15:03:11



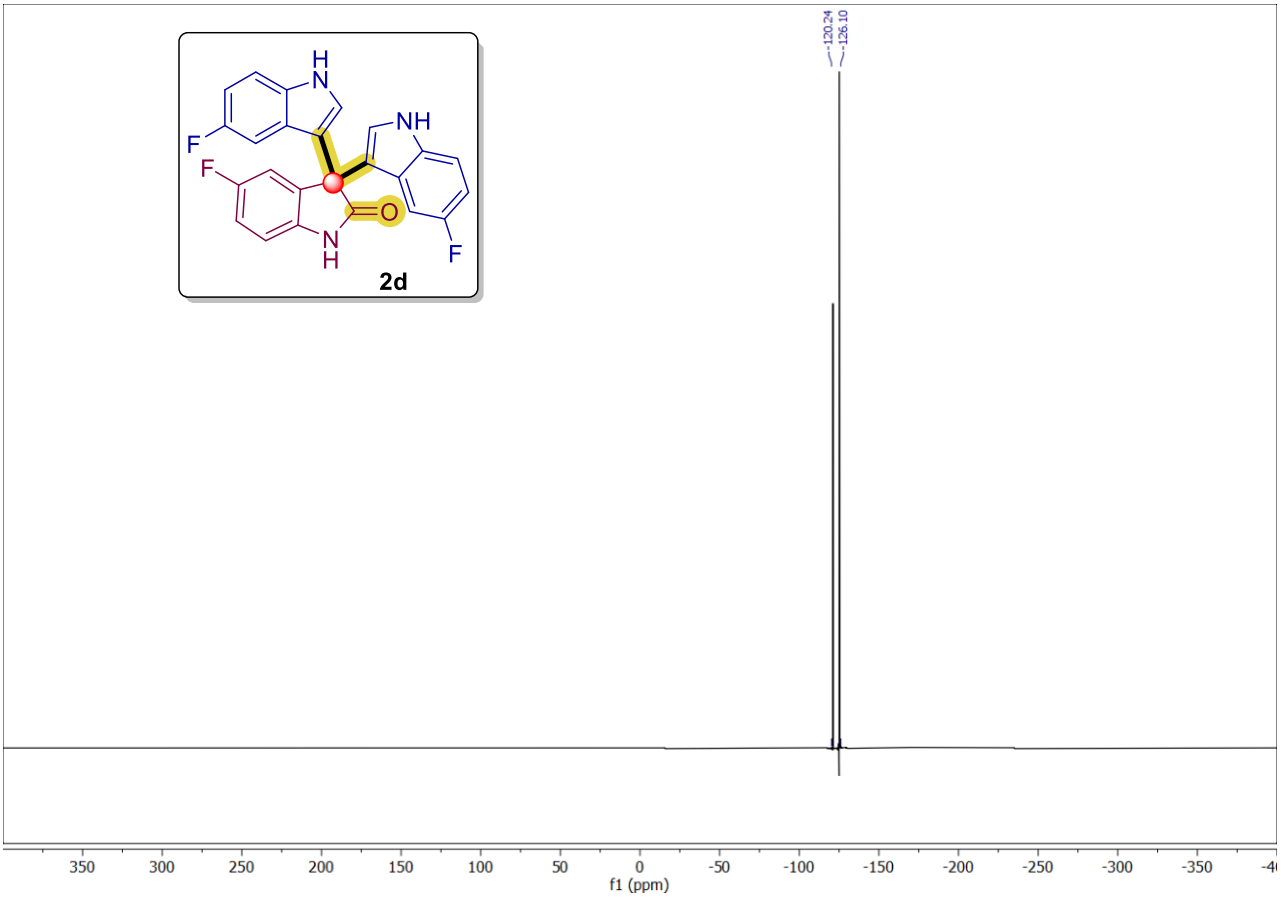
^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5,5',5''-trifluoro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2d)



^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5,5',5''-trifluoro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2d)

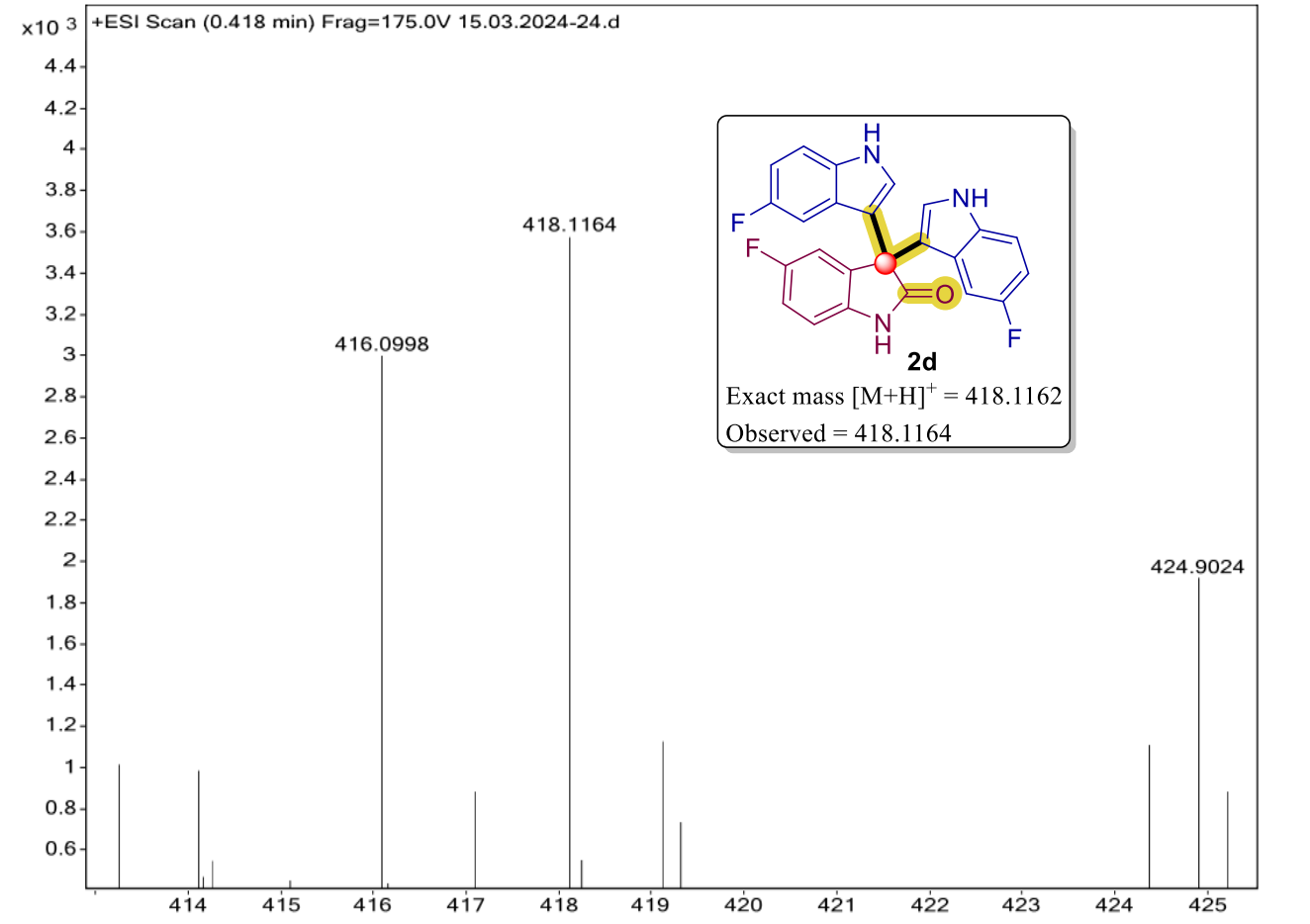


^{19}F NMR (376 MHz, DMSO-d_6) spectrum of 5,5',5''-trifluoro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2d)

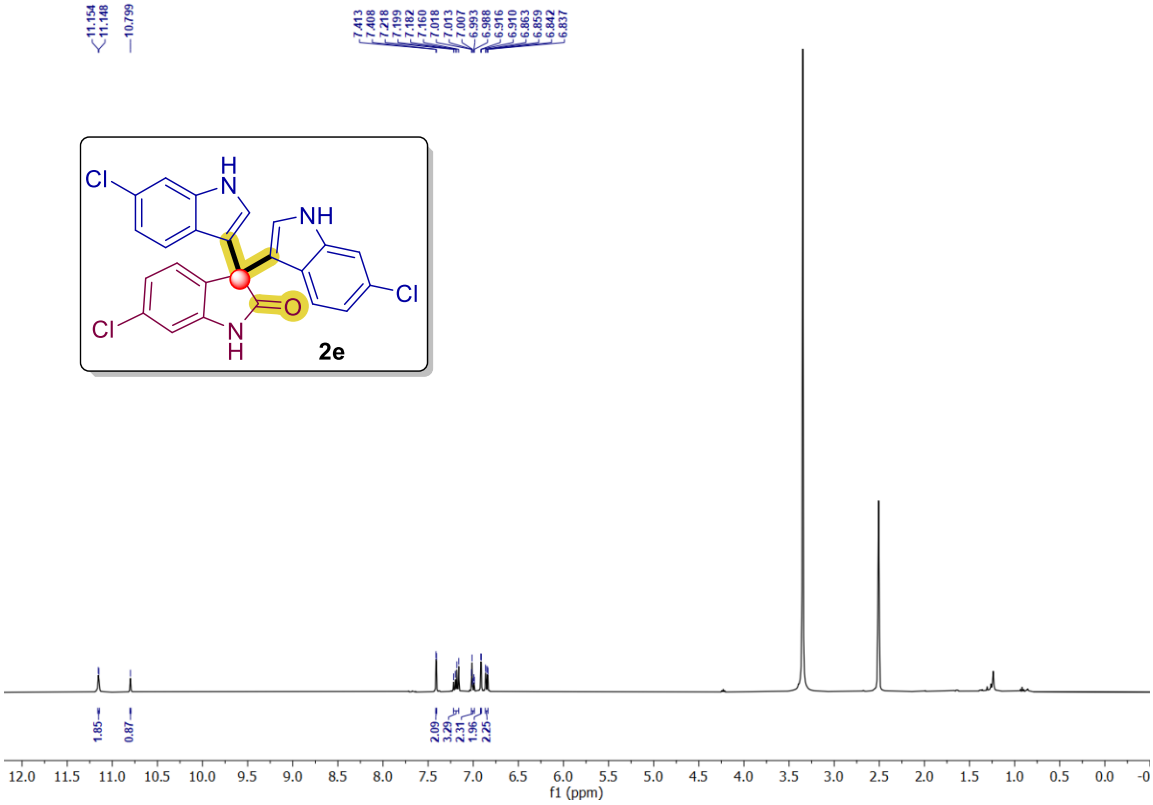


HRMS spectrum of 5,5',5''-trifluoro-1*H*,1''*H*-[3,3':3',3''-terindol]-2' (1'*H*)-one (2d)

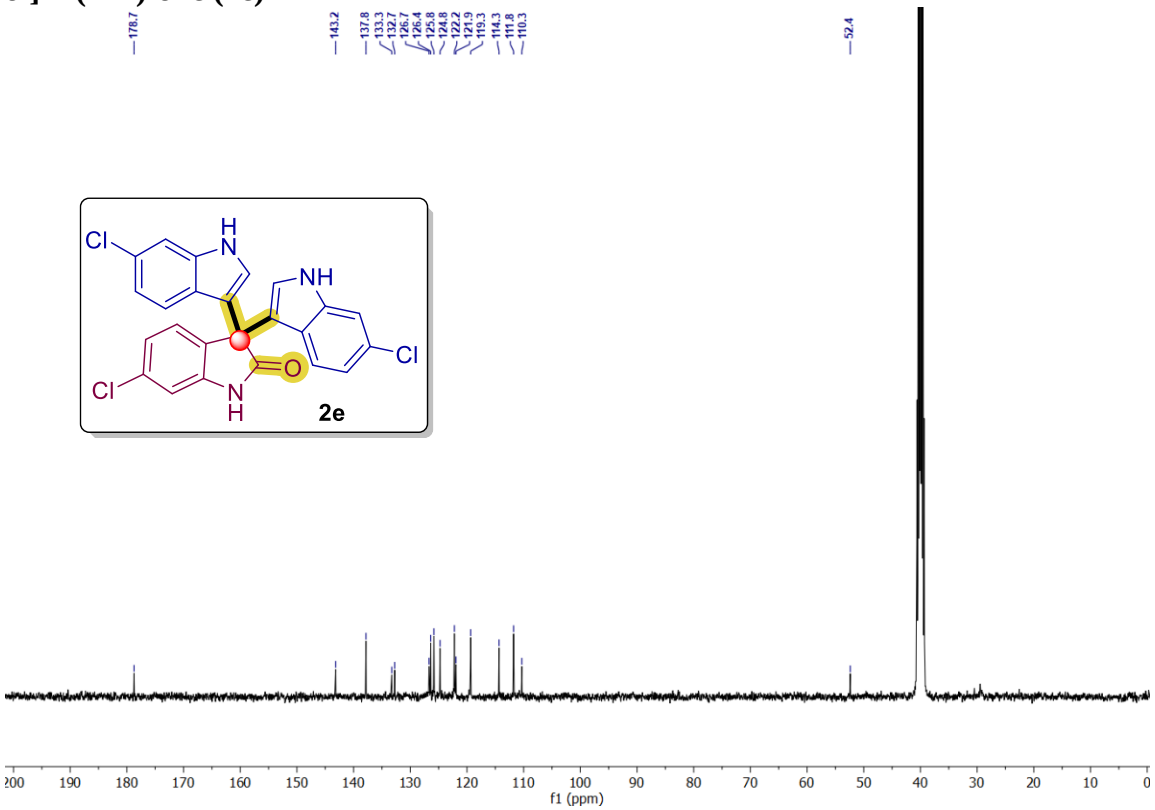
Sample Name	exp-6	**Position**	P1-C6	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-24.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 13:20:15



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 6,6',6''-trichloro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2e)

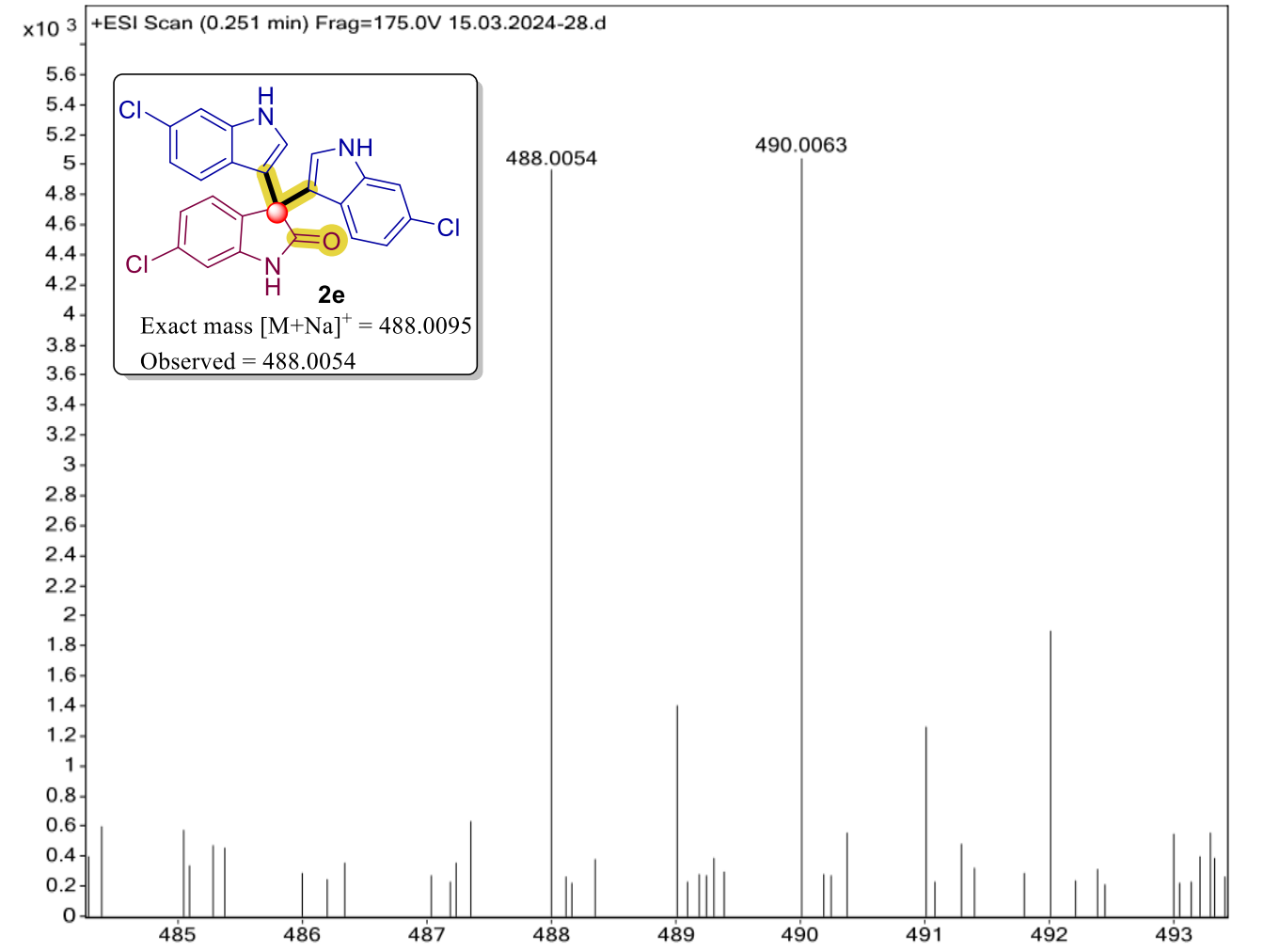


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 6,6',6''-trichloro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2e)

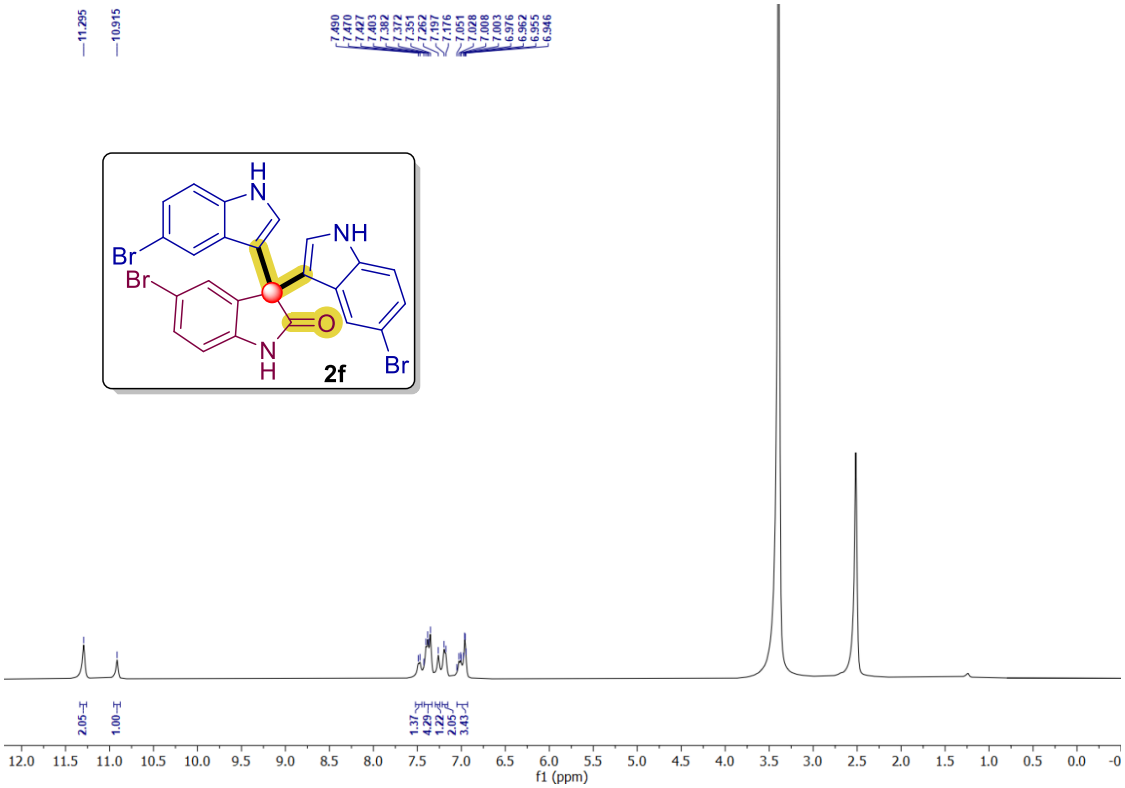


HRMS spectrum of 6,6',6''-trichloro-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2e)

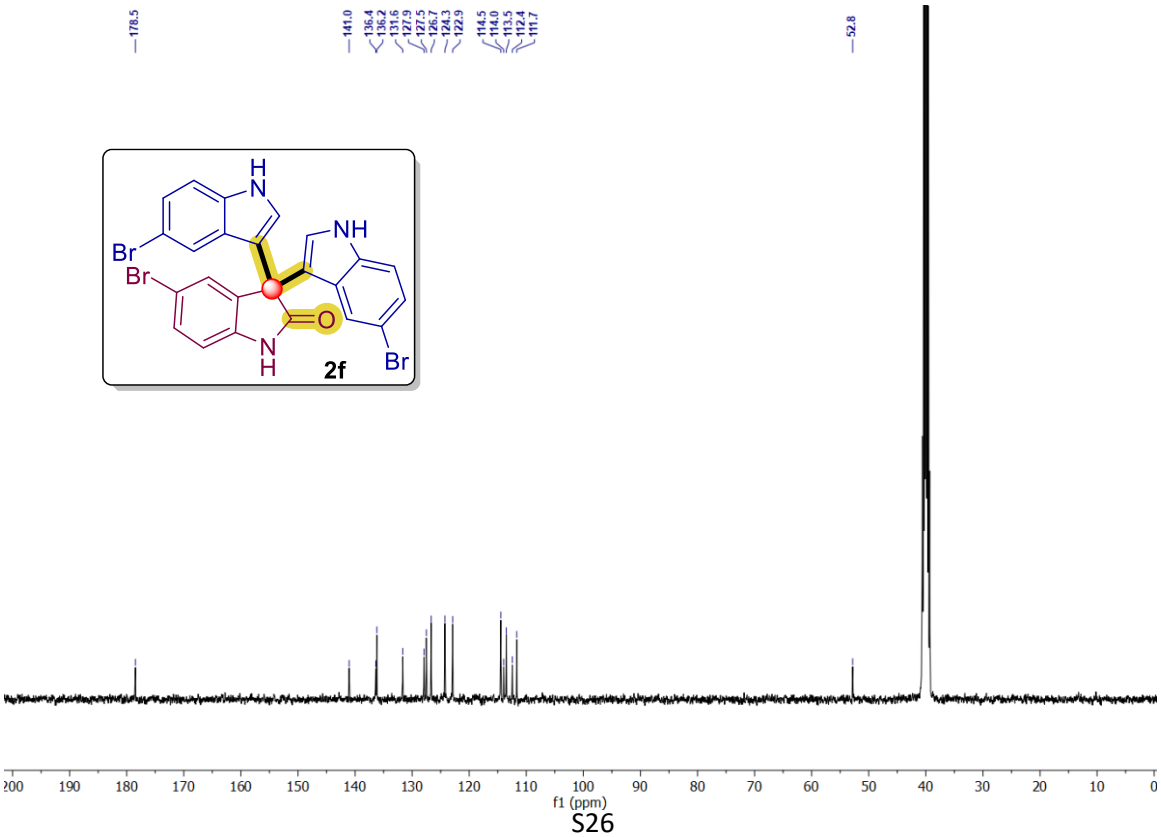
Sample Name	exp-10	**Position**	P1-D1	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-28.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:44:31



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5,5',5''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2f)

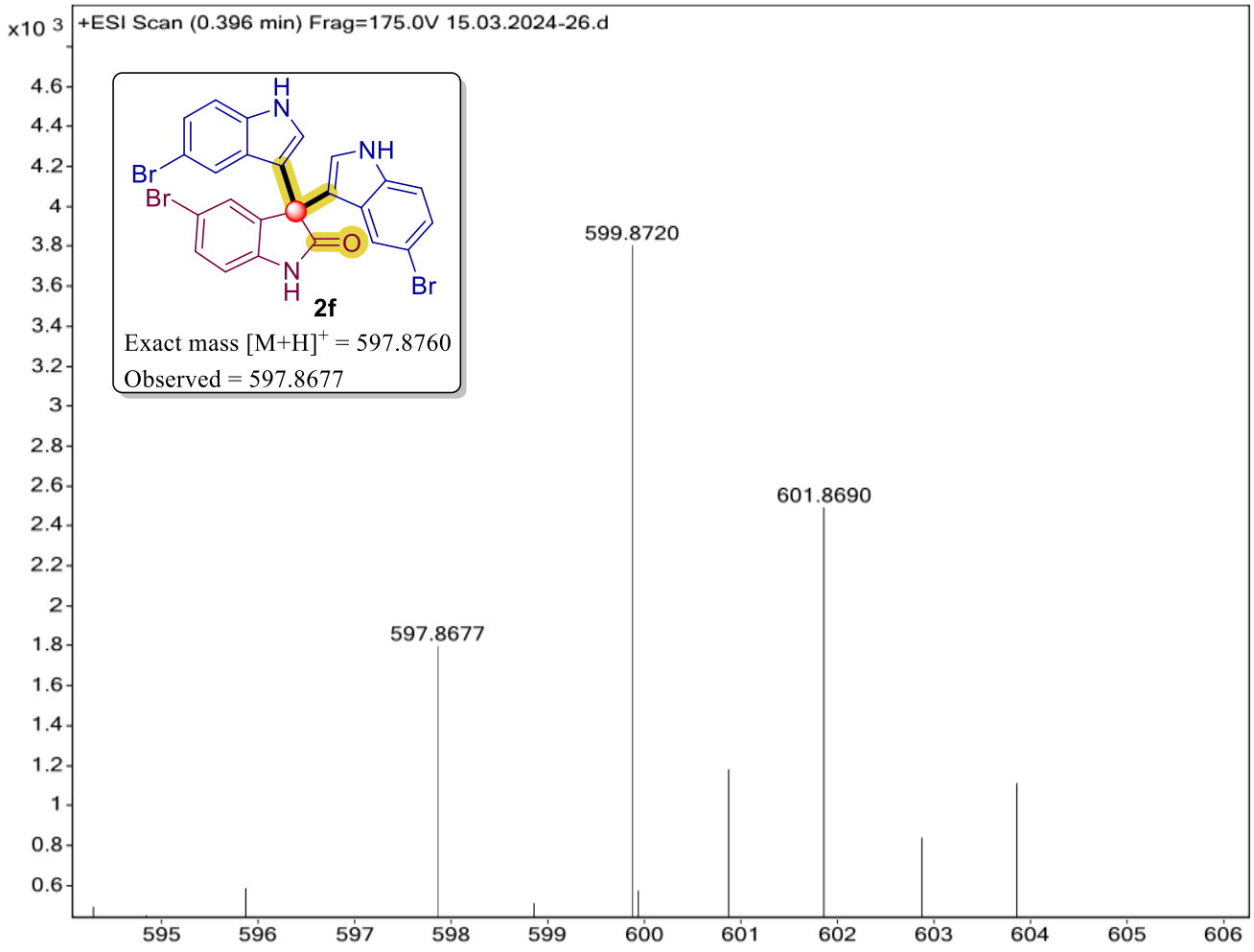


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5,5',5''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2f)

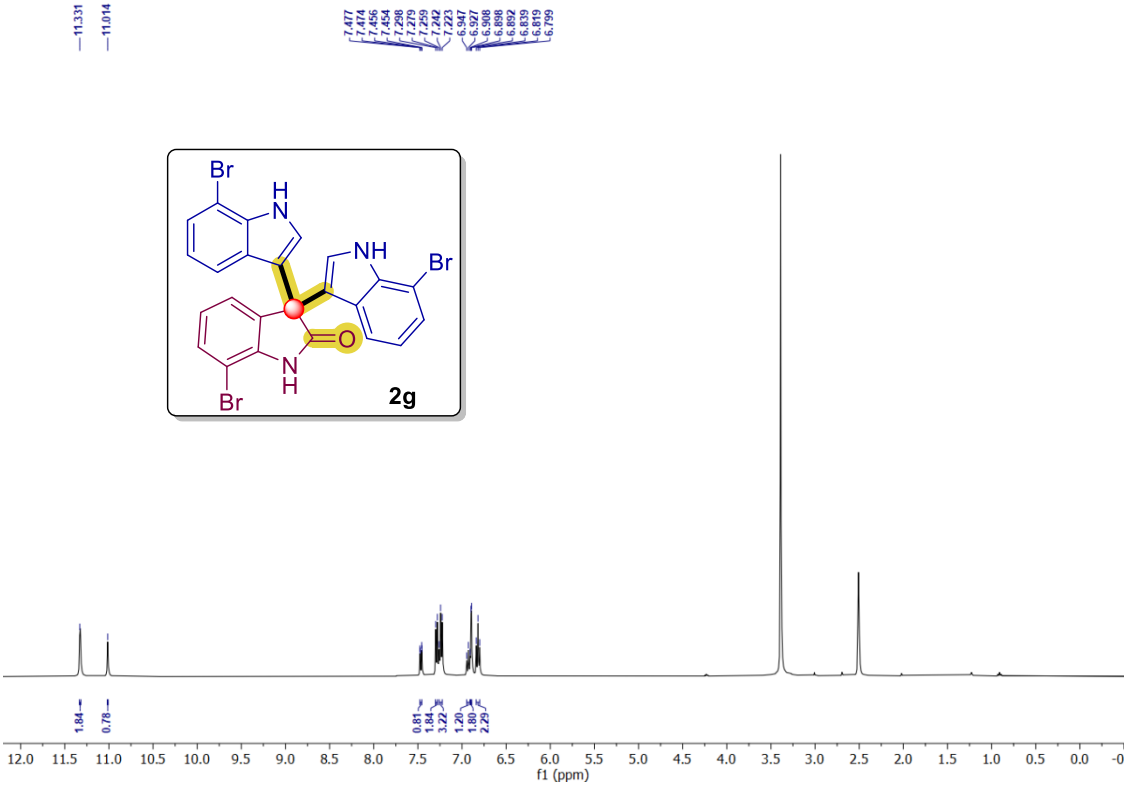


HRMS spectrum of 5,5',5''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2f)

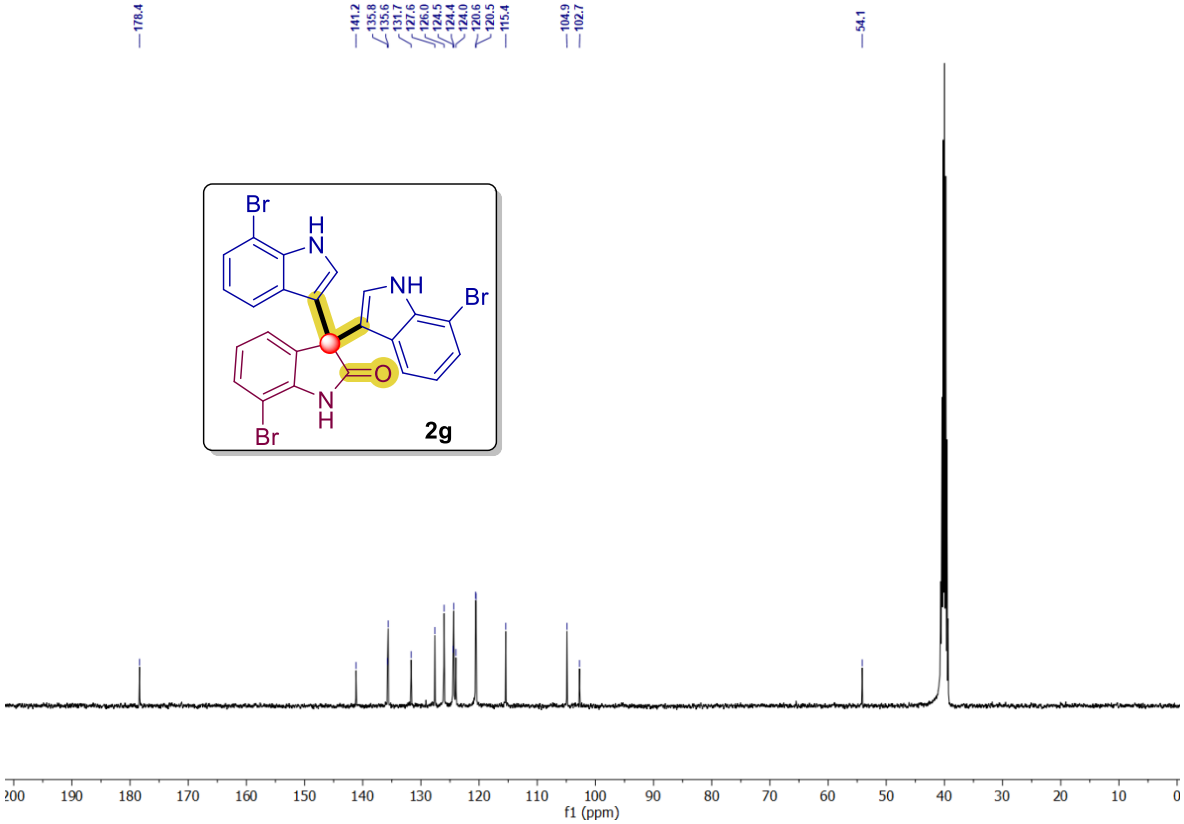
Sample Name	exp-8	**Position**	P1-C8	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-26.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:36:36



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 7,7',7''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2g)

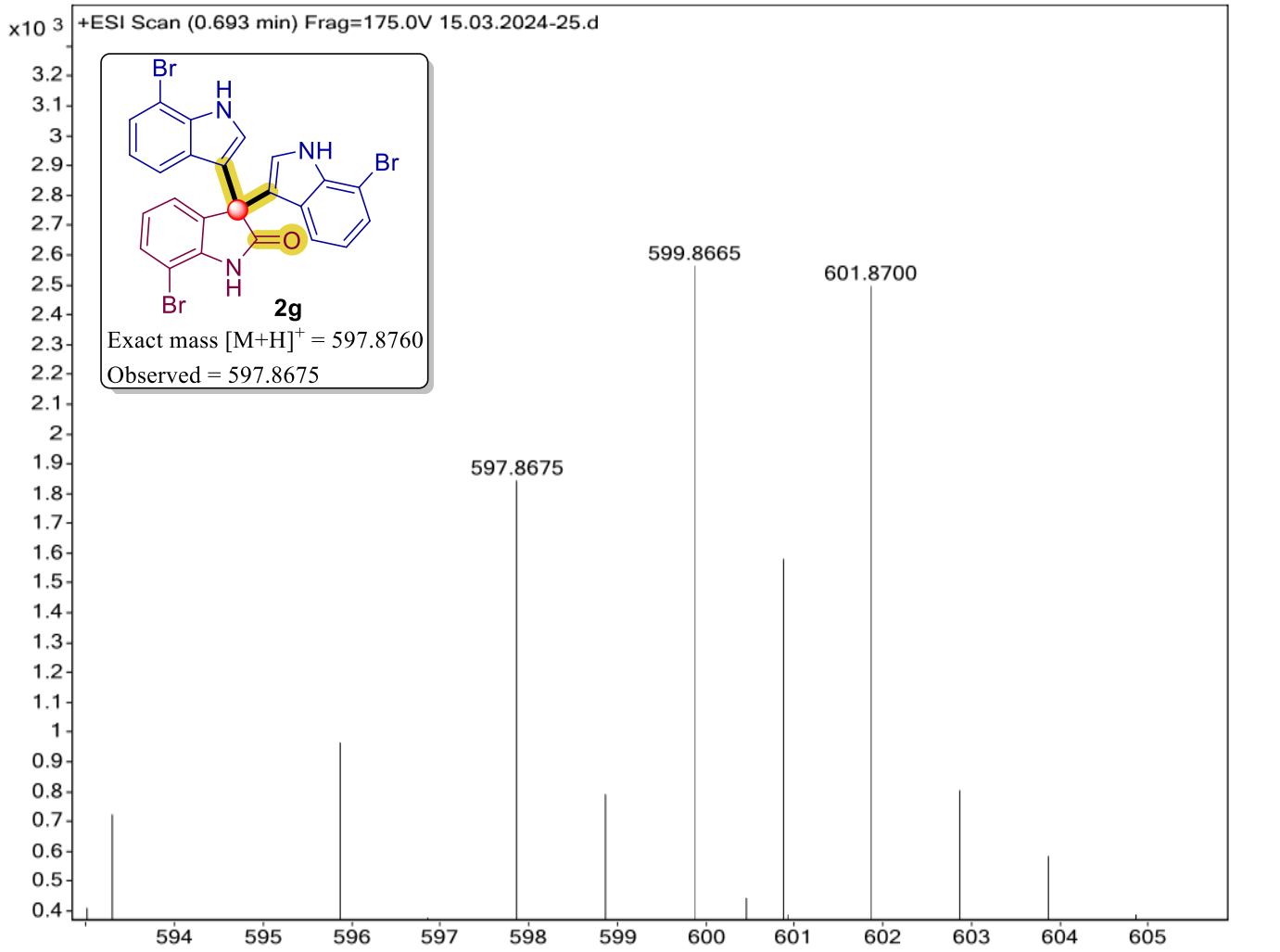


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 7,7',7''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2g)

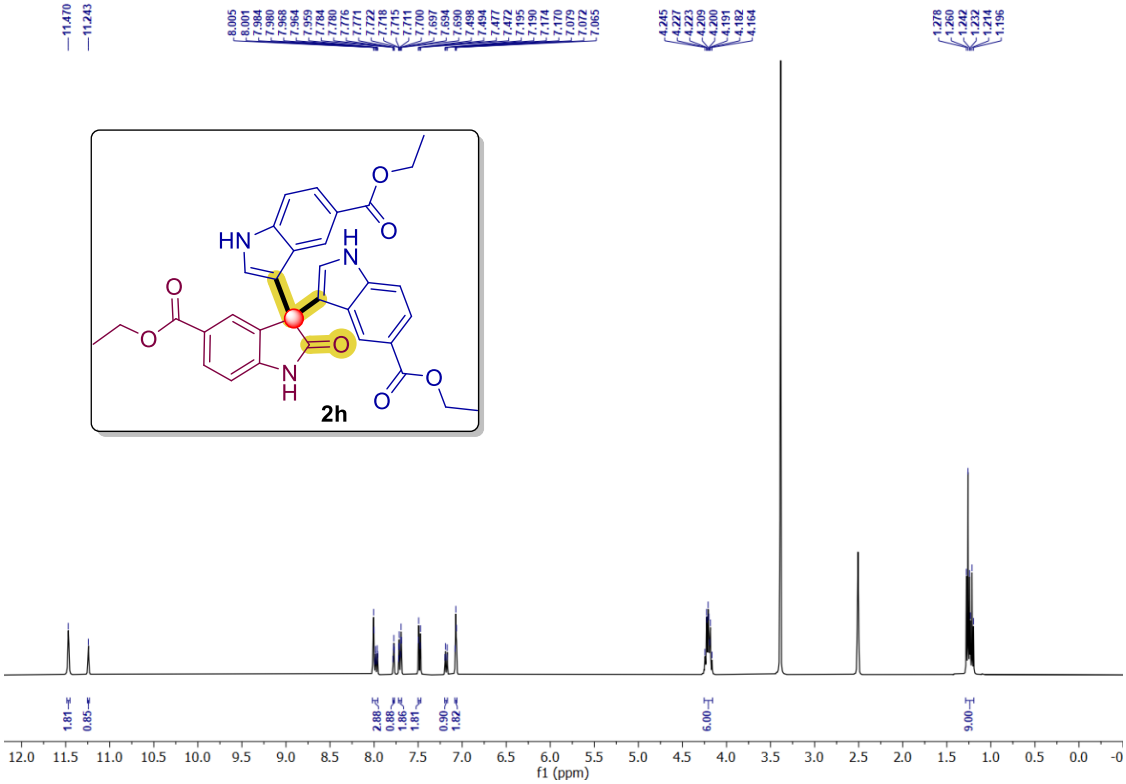


HRMS spectrum of 7,7',7''-tribromo-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2g)

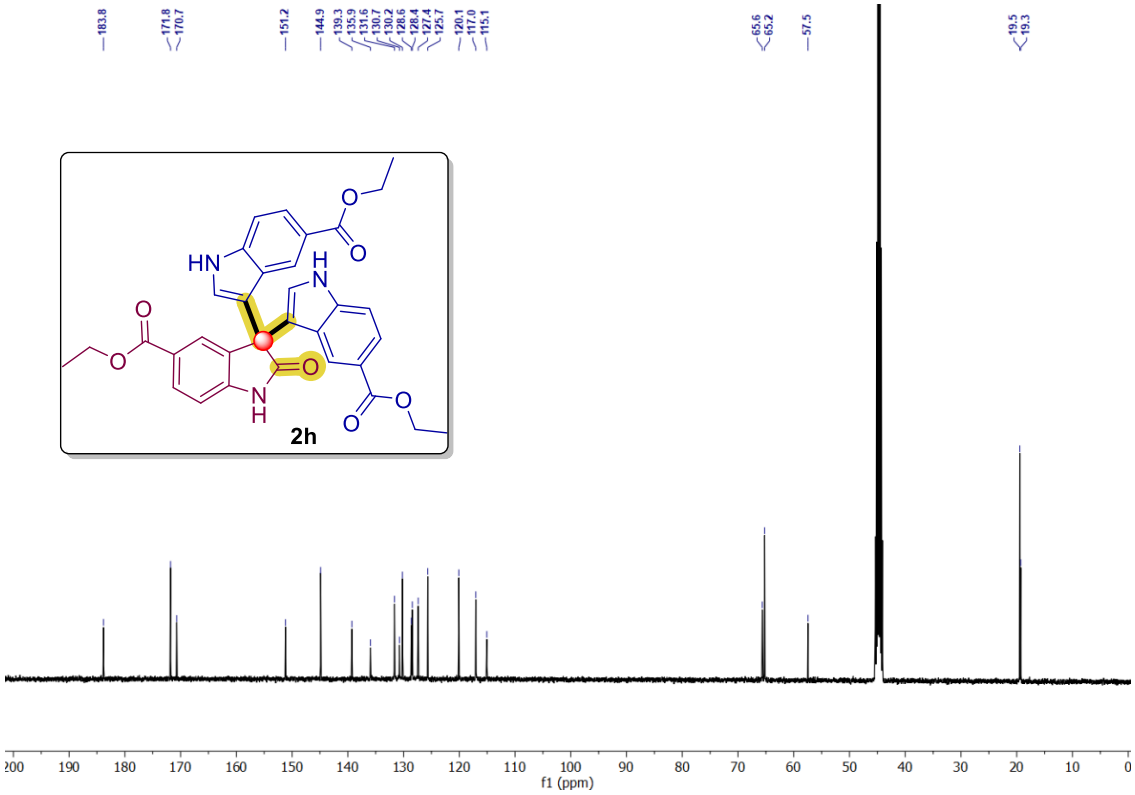
Sample Name	exp-7	**Position**	P1-C7	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-25.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:32:36



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of triethyl 2'-oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarboxylate (2h)

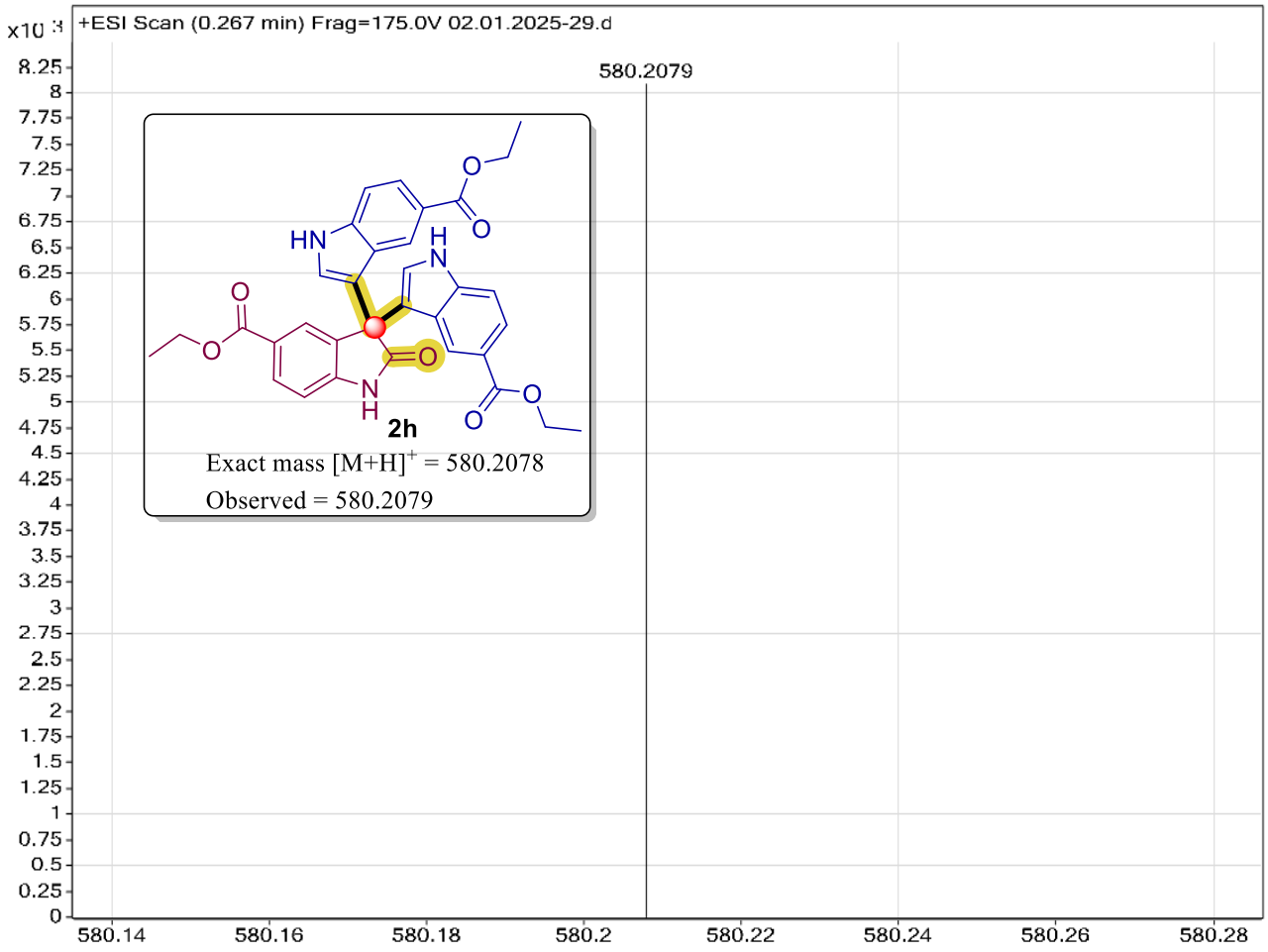


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of triethyl 2'-oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarboxylate (2h)

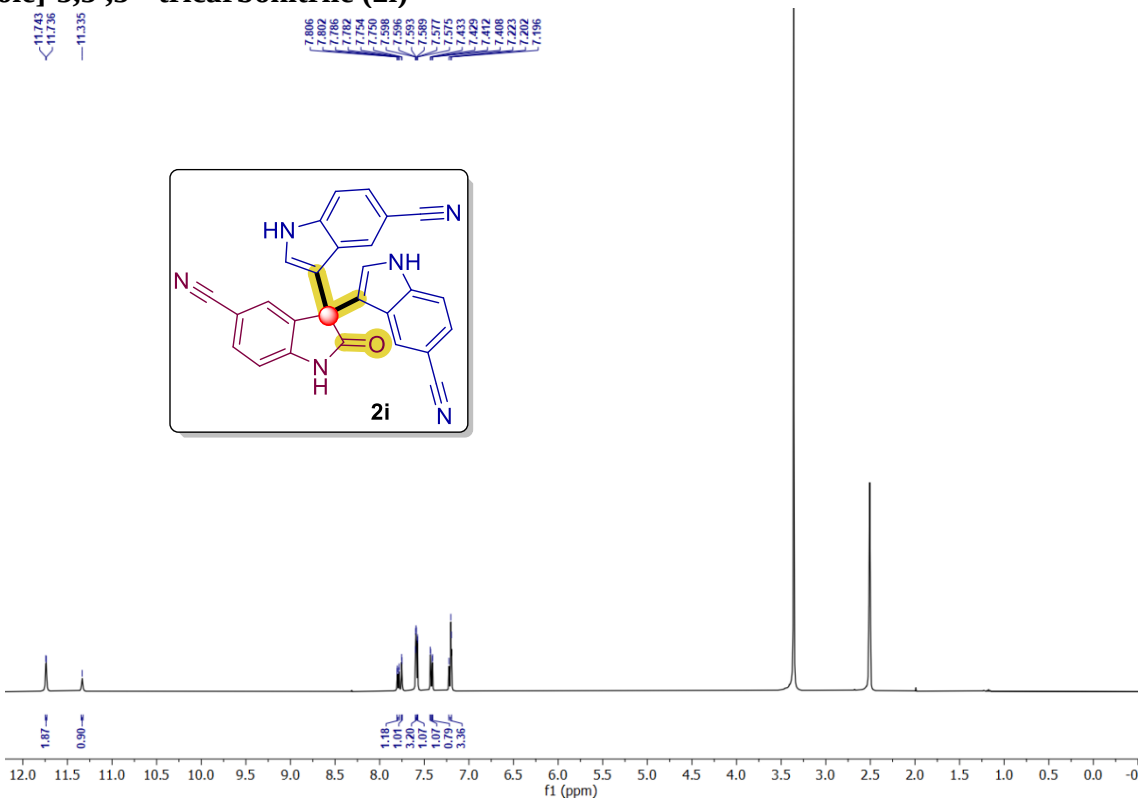


HRMS spectrum of triethyl 2'-oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarboxylate (2h)

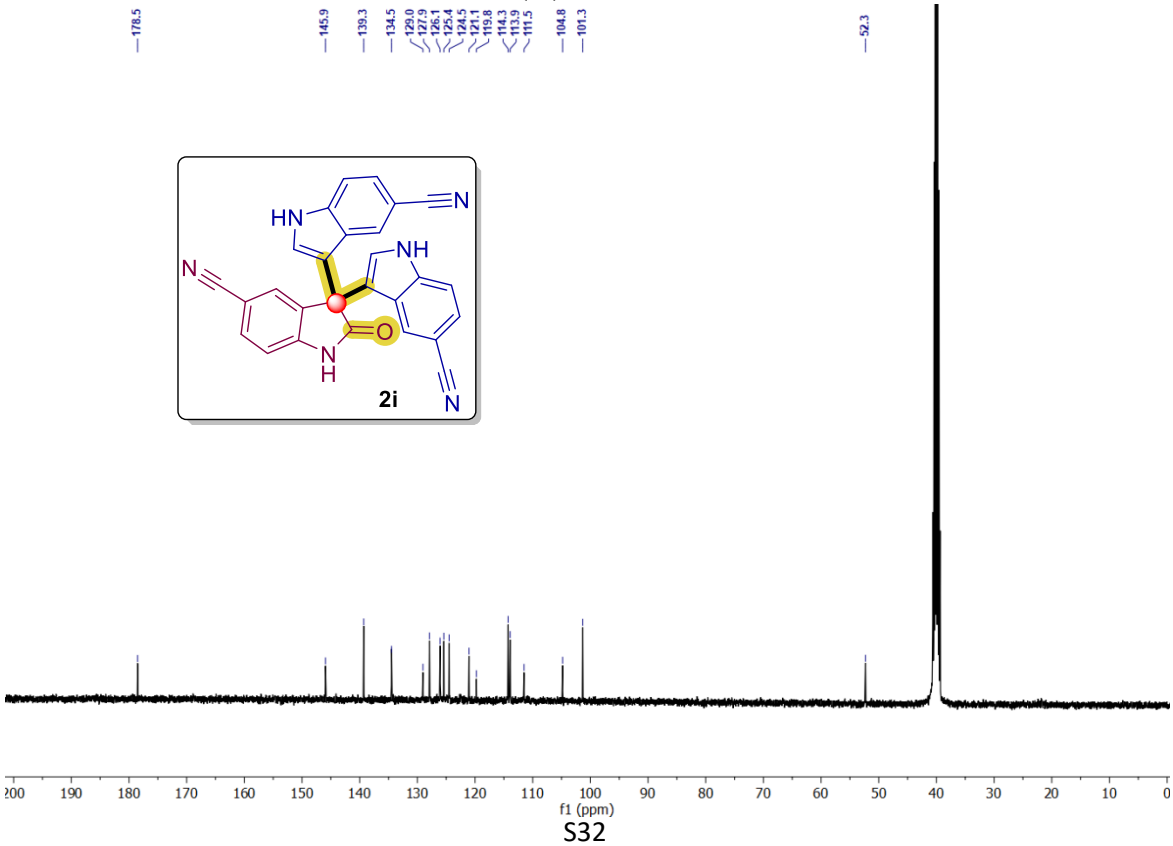
Sample Name	khp-ns-obcorbxy	**Position**	P1-D2	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	02.01.2025-29.d	**ACQ Method**	M60 W40.m	**Comment**		**Acquired Time**	03-01-2025 15:07:16



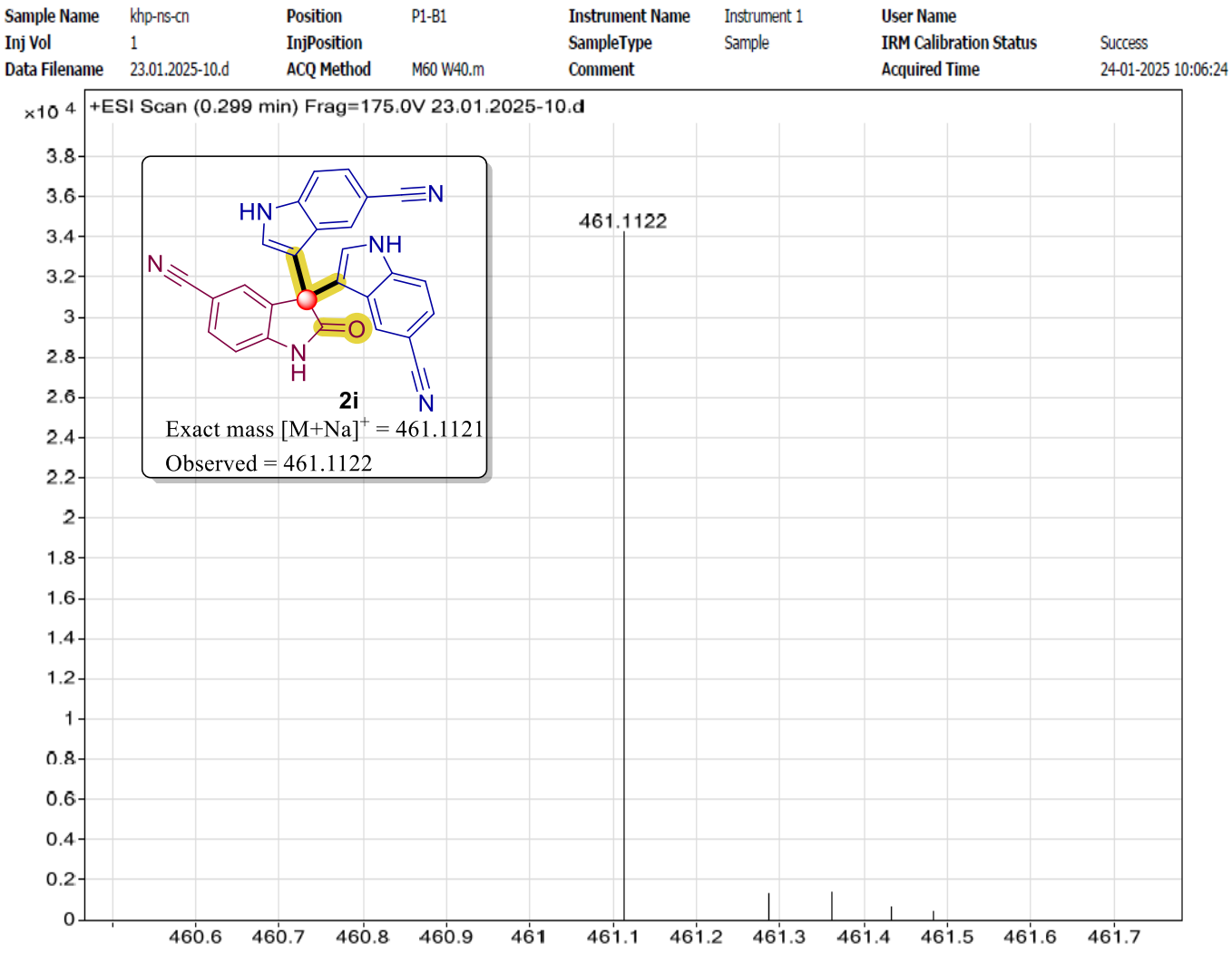
^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 2'-oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarbonitrile (2i)



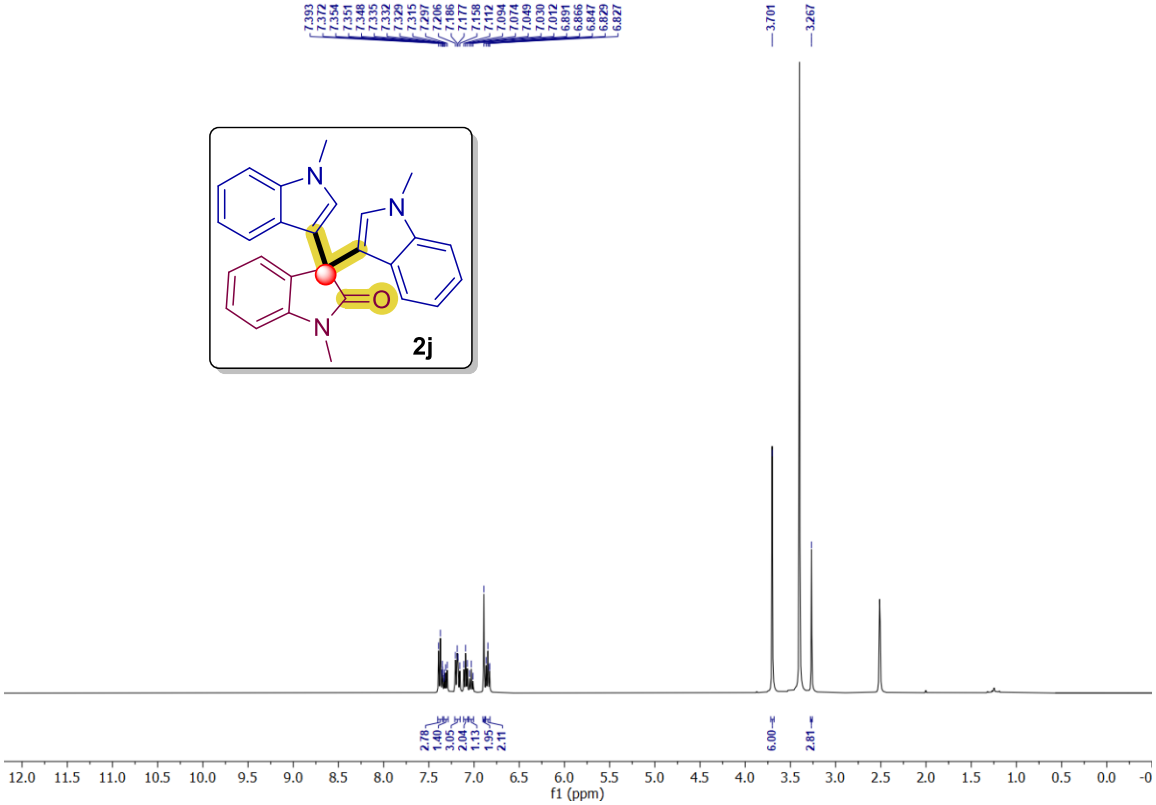
^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarbonitrile (2i)



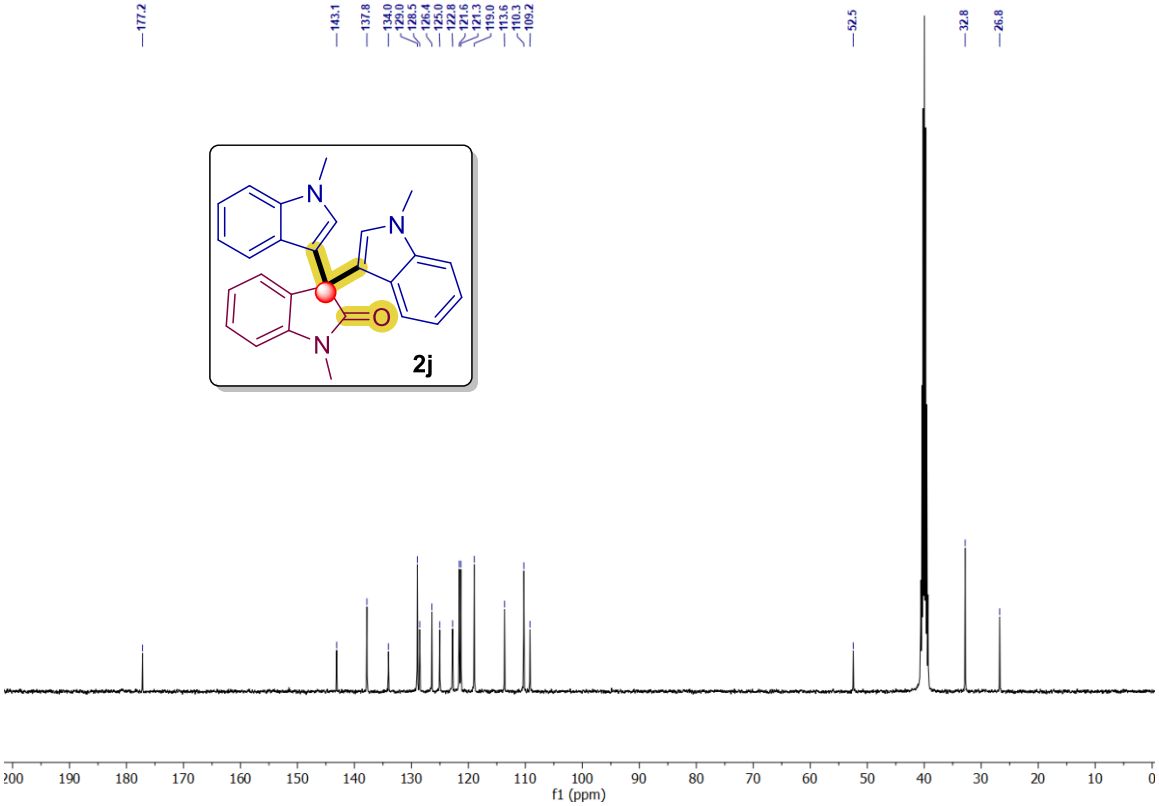
HRMS spectrum of oxo-1',2'-dihydro-1*H*,1''*H*-[3,3':3',3''-terindole]-5,5',5''-tricarbonitrile (2i)



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2j)

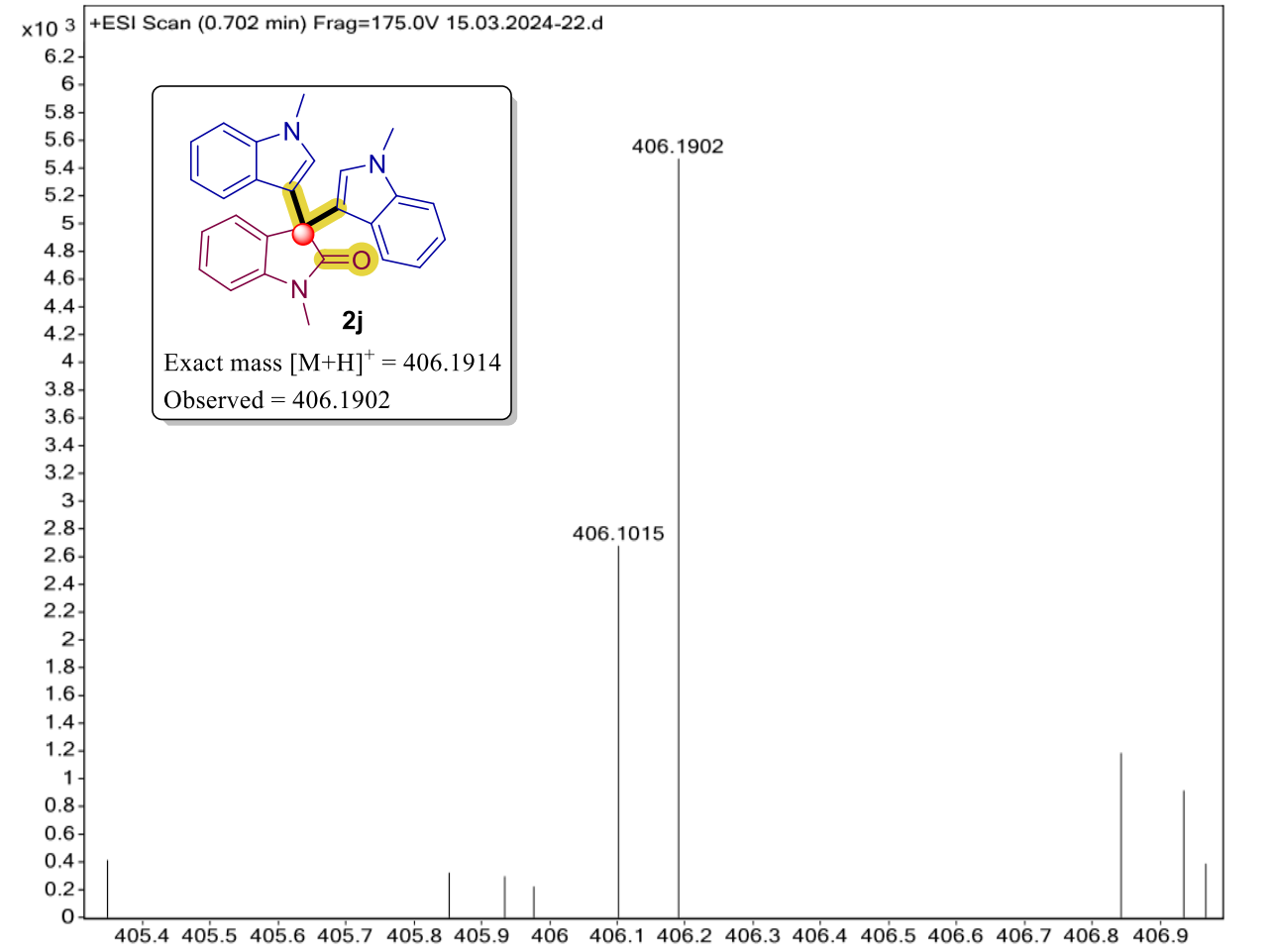


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2j)

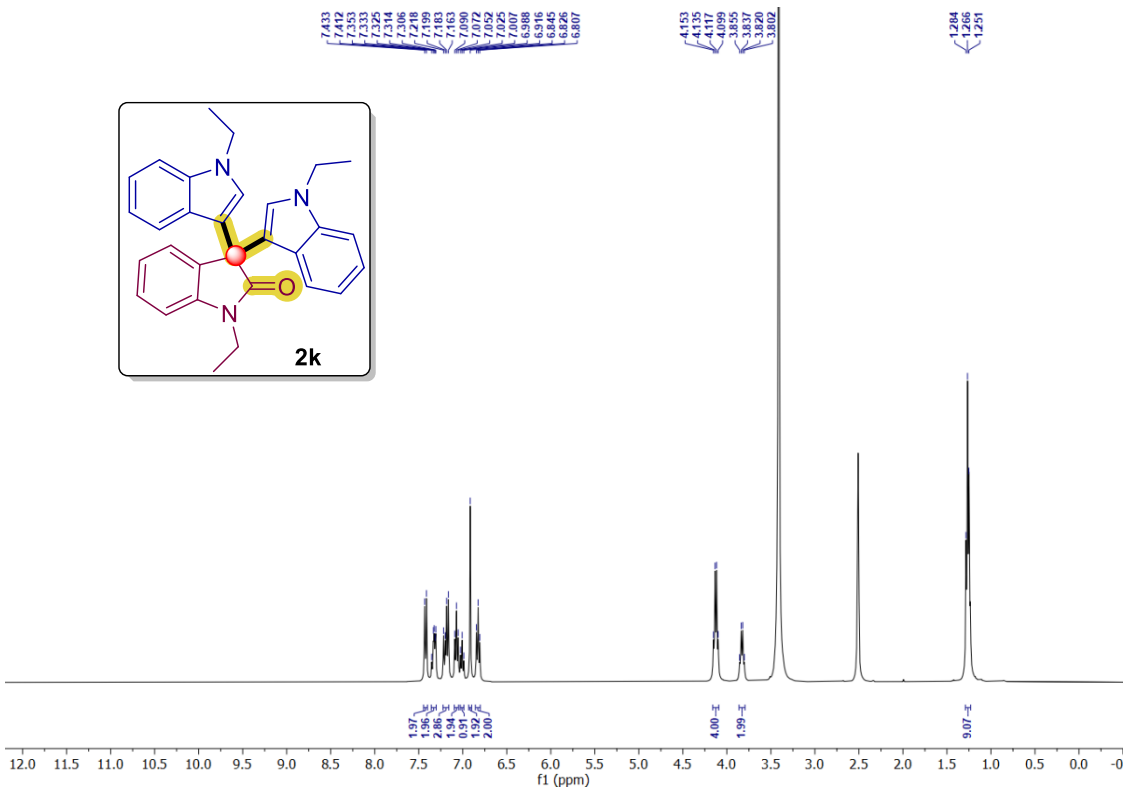


HRMS spectrum of 1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2j)

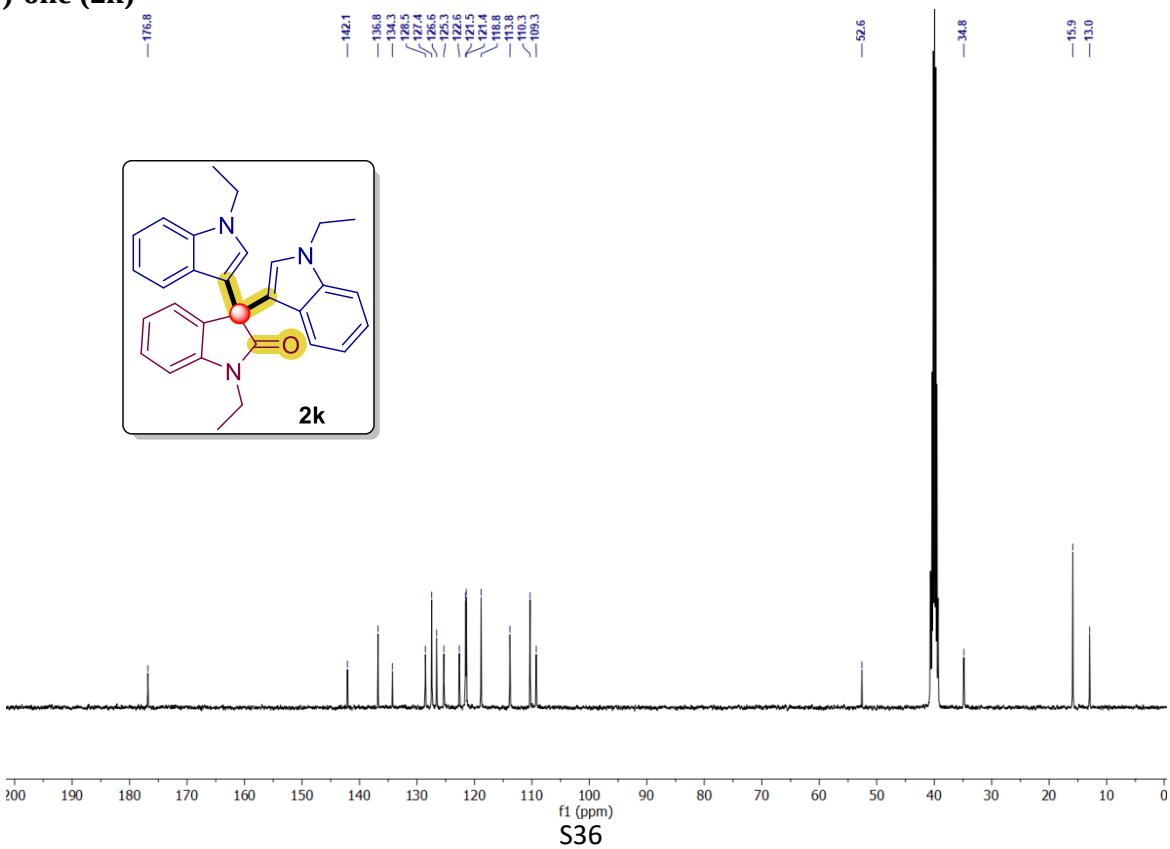
Sample Name	exp-4	**Position**	P1-C4	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-22.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:24:30



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1,1',1''-triethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2k)

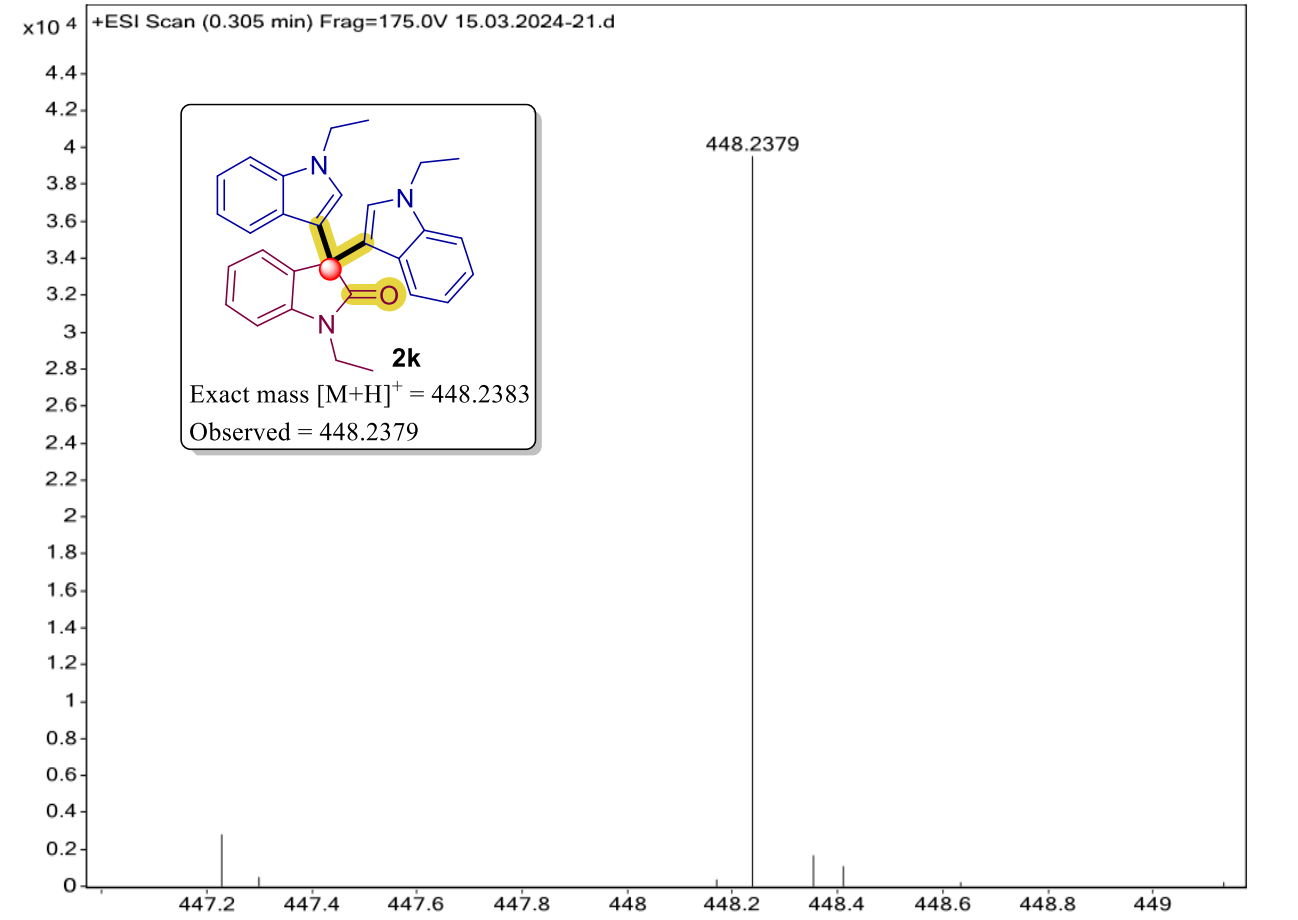


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1,1',1''-triethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2k)

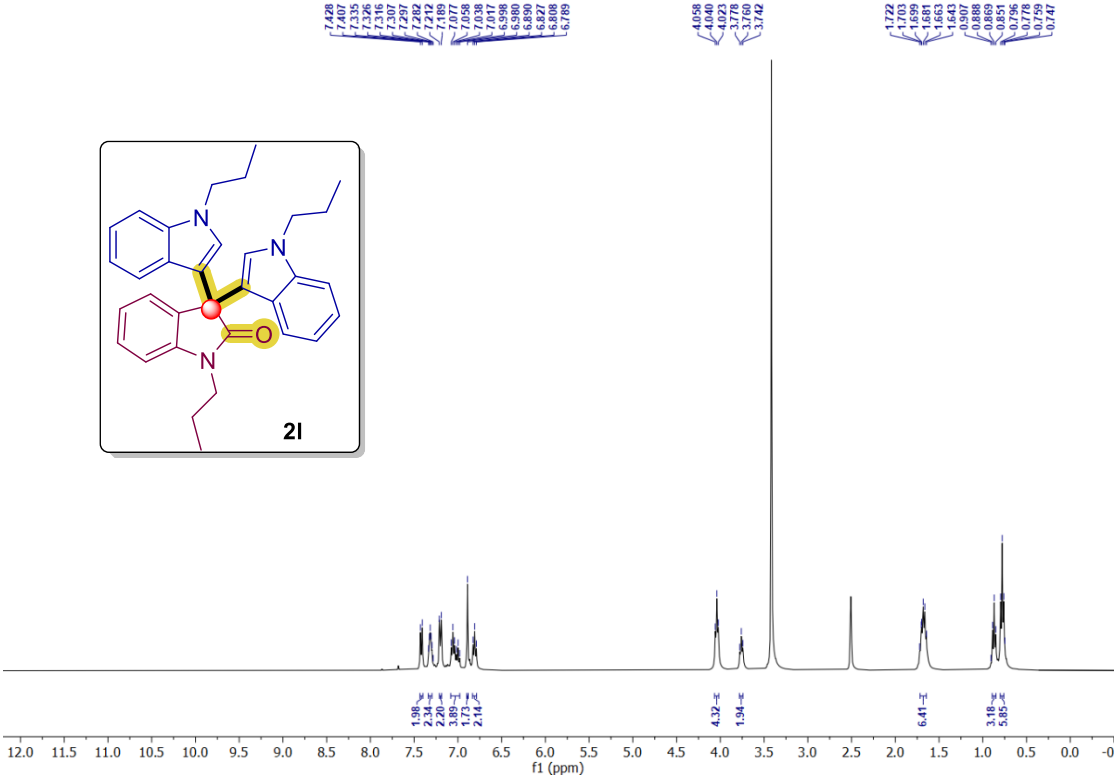


HRMS spectrum of 1,1',1''-triethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2k)

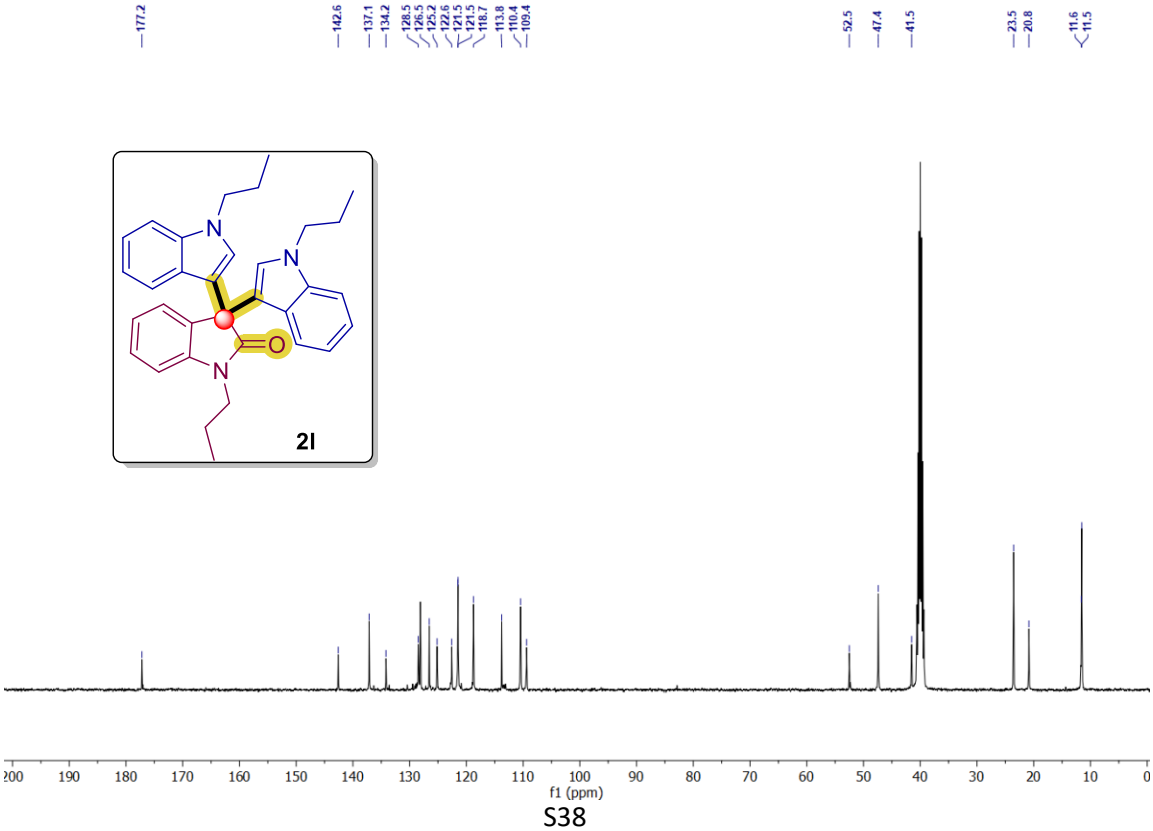
Sample Name	exp-3	**Position**	P1-C3	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-21.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:20:31



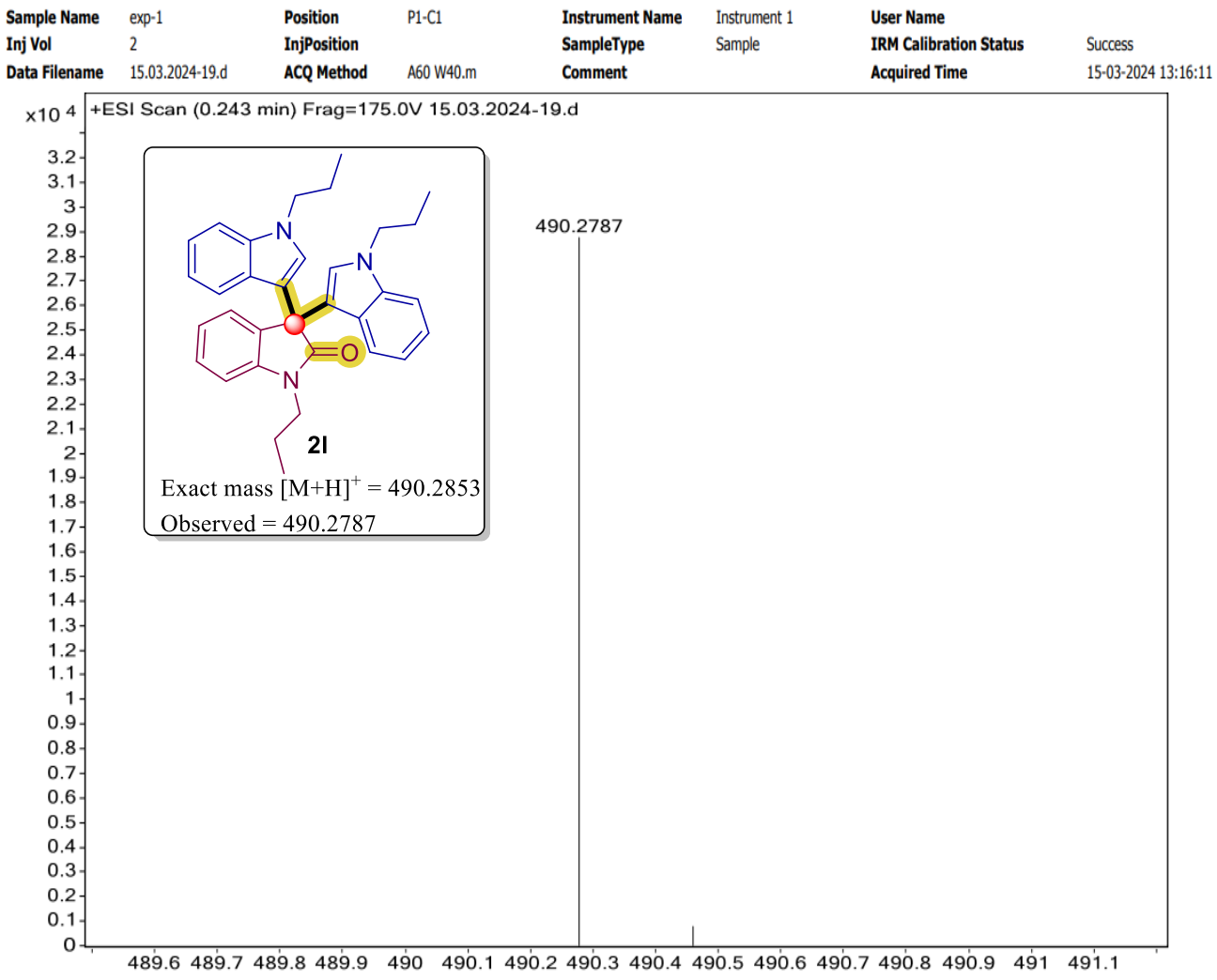
^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1,1',1''-tripropyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2l)



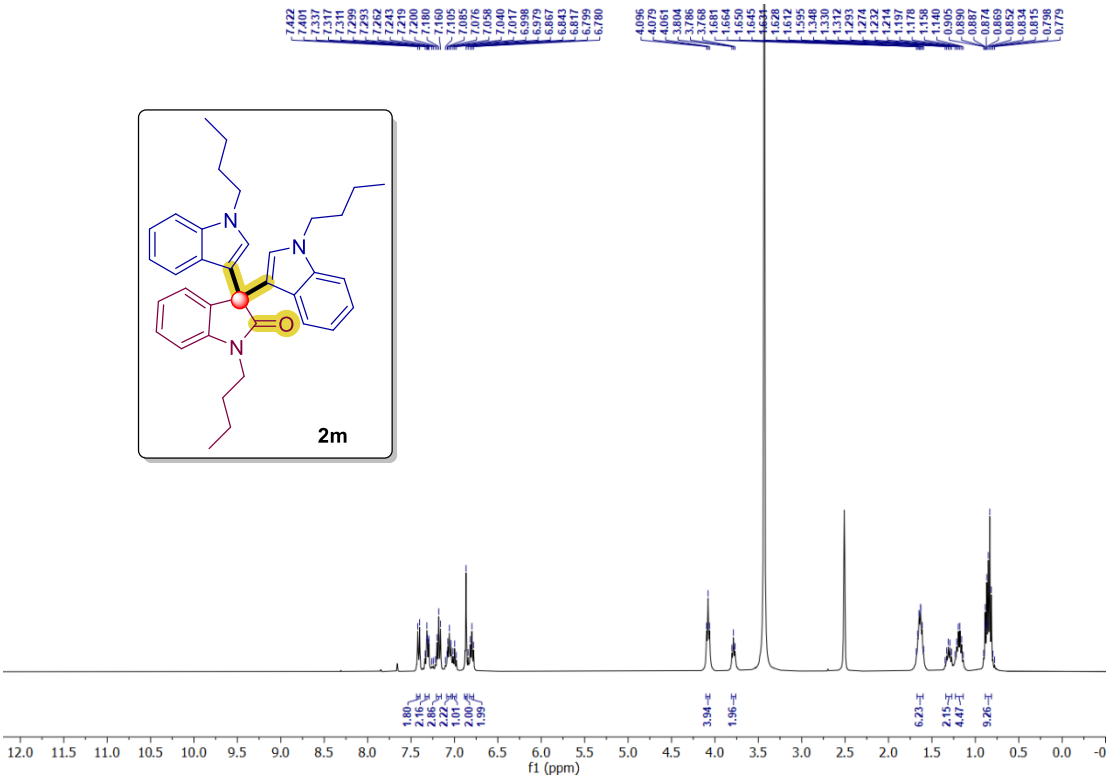
^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1,1',1''-tripropyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2l)



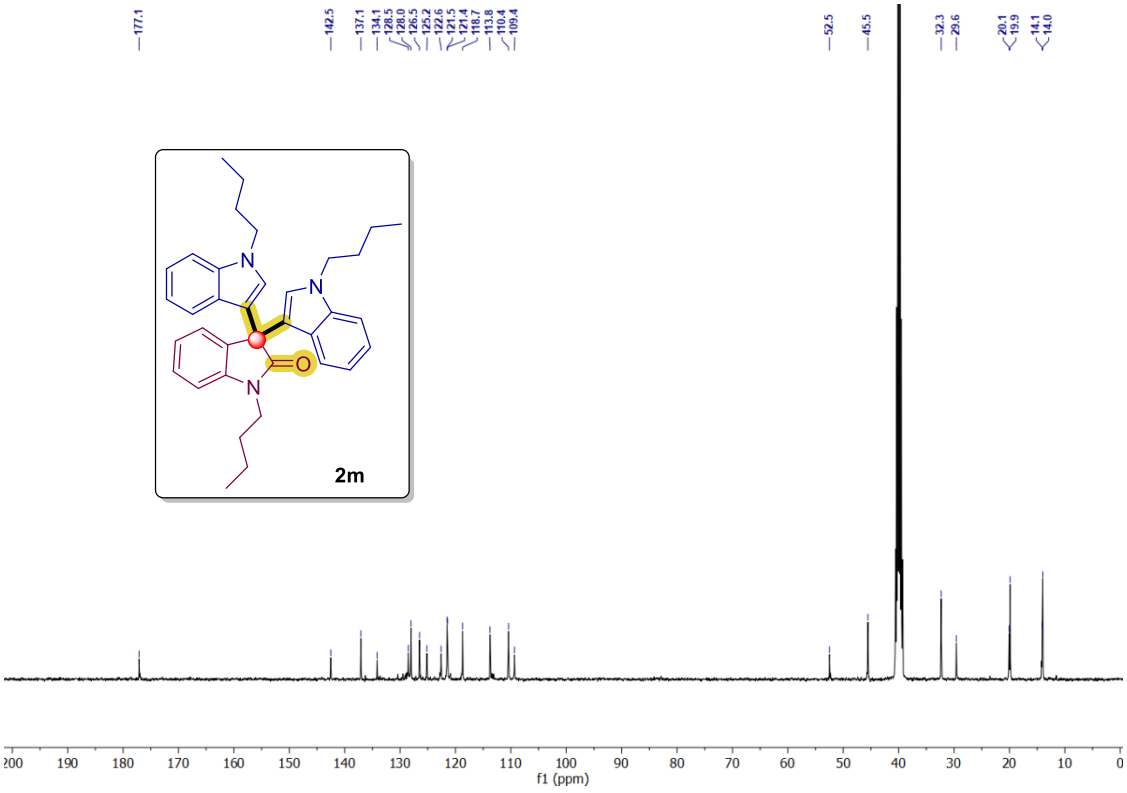
HRMS spectrum of 1,1',1''-tripropyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2l)



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1,1',1''-tributyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2m)

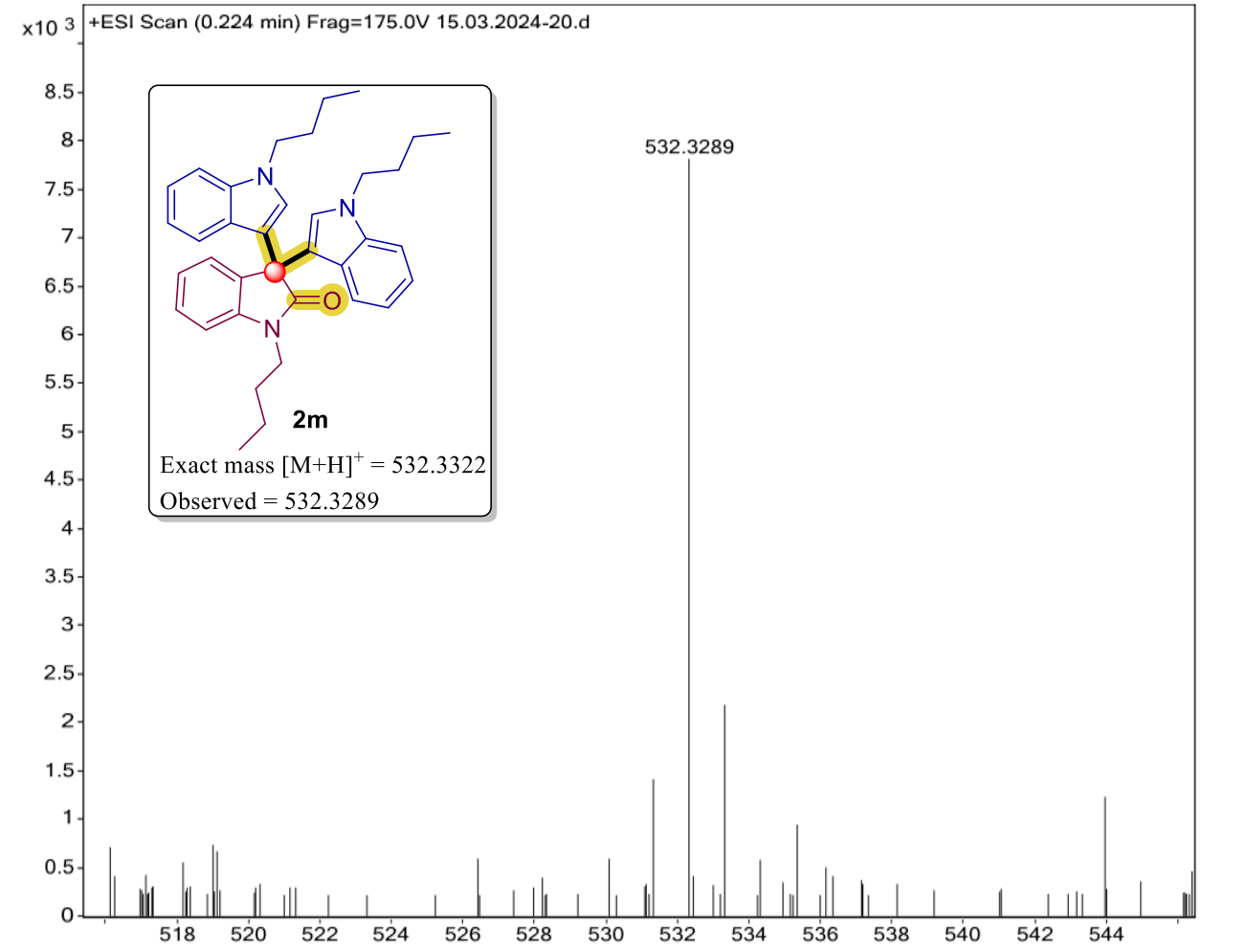


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1,1',1''-tributyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2m)

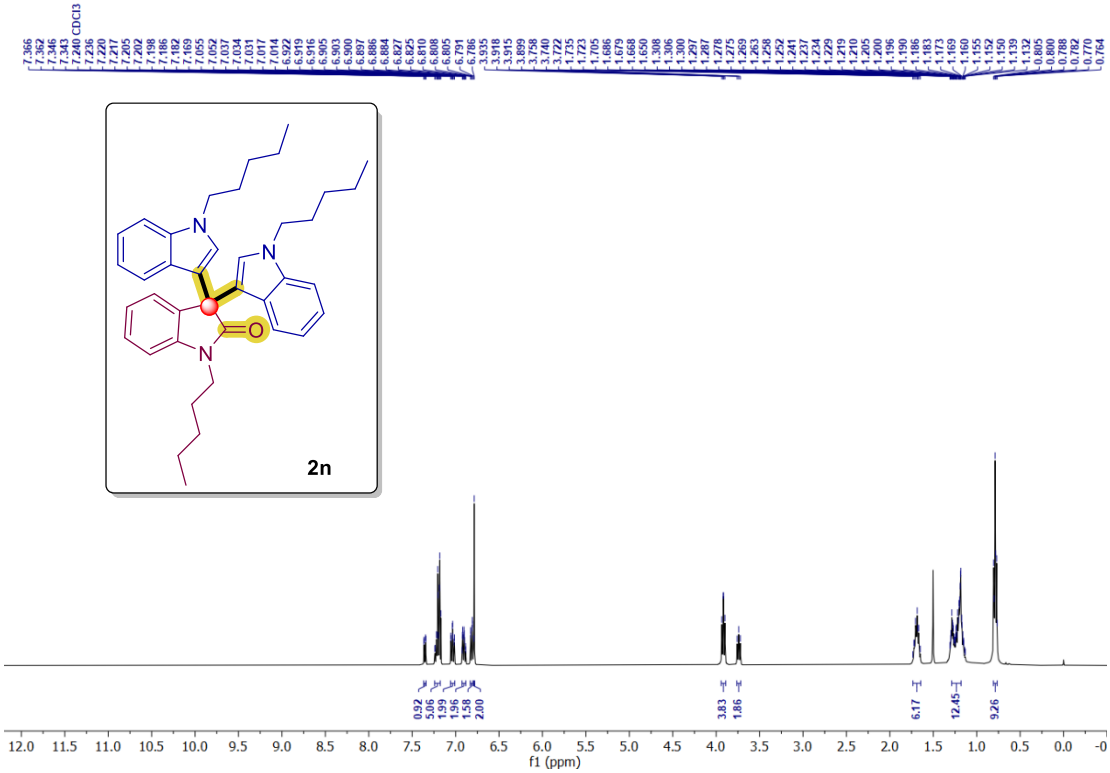


HRMS spectrum of 1,1',1''-tributyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2m)

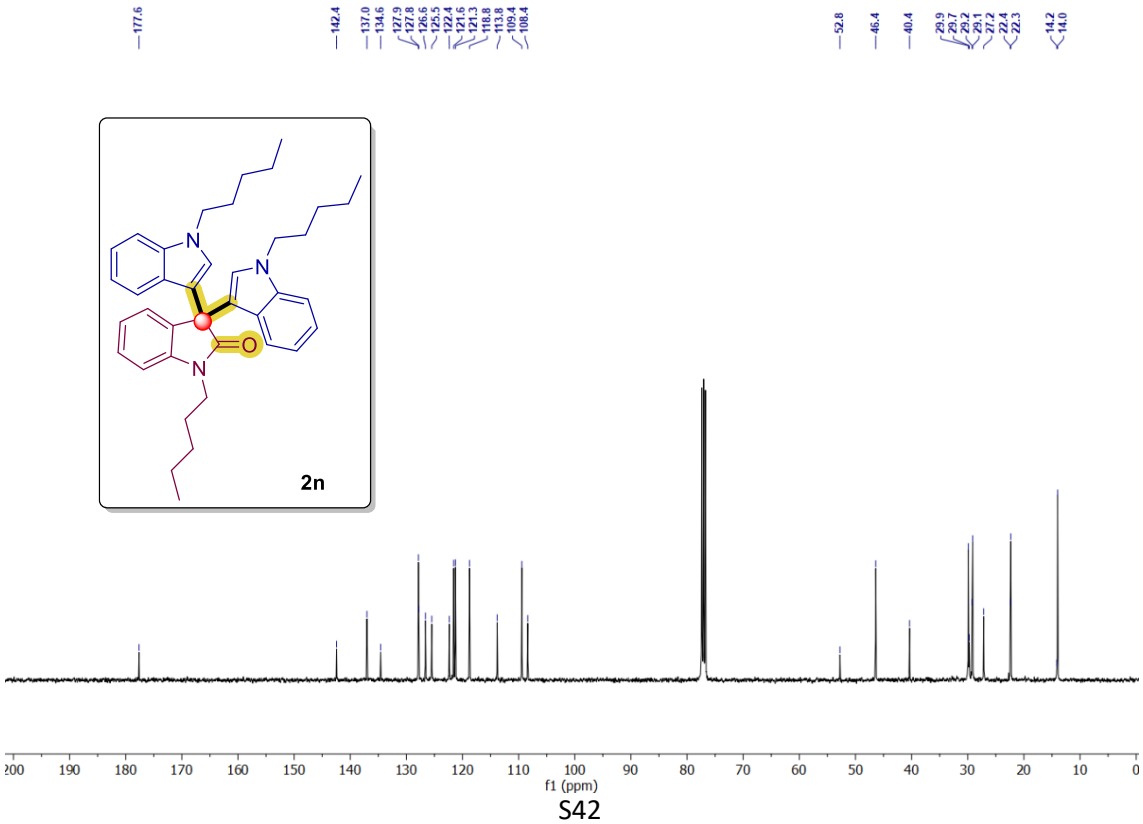
Sample Name	exp-2	**Position**	P1-C2	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	2	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	15.03.2024-20.d	**ACQ Method**	A60 W40.m	**Comment**		**Acquired Time**	15-03-2024 14:16:33



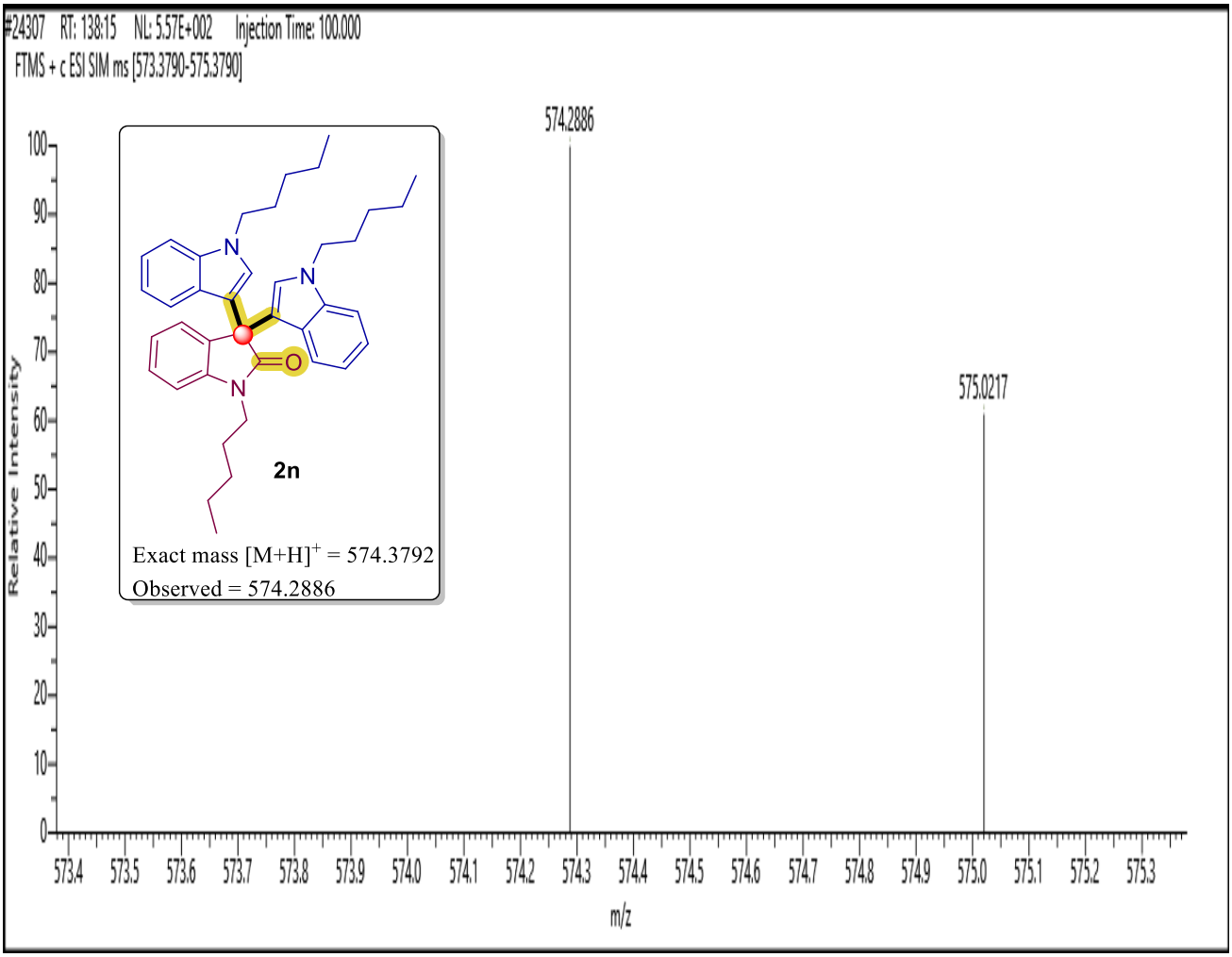
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1,1',1''-tripentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2n)



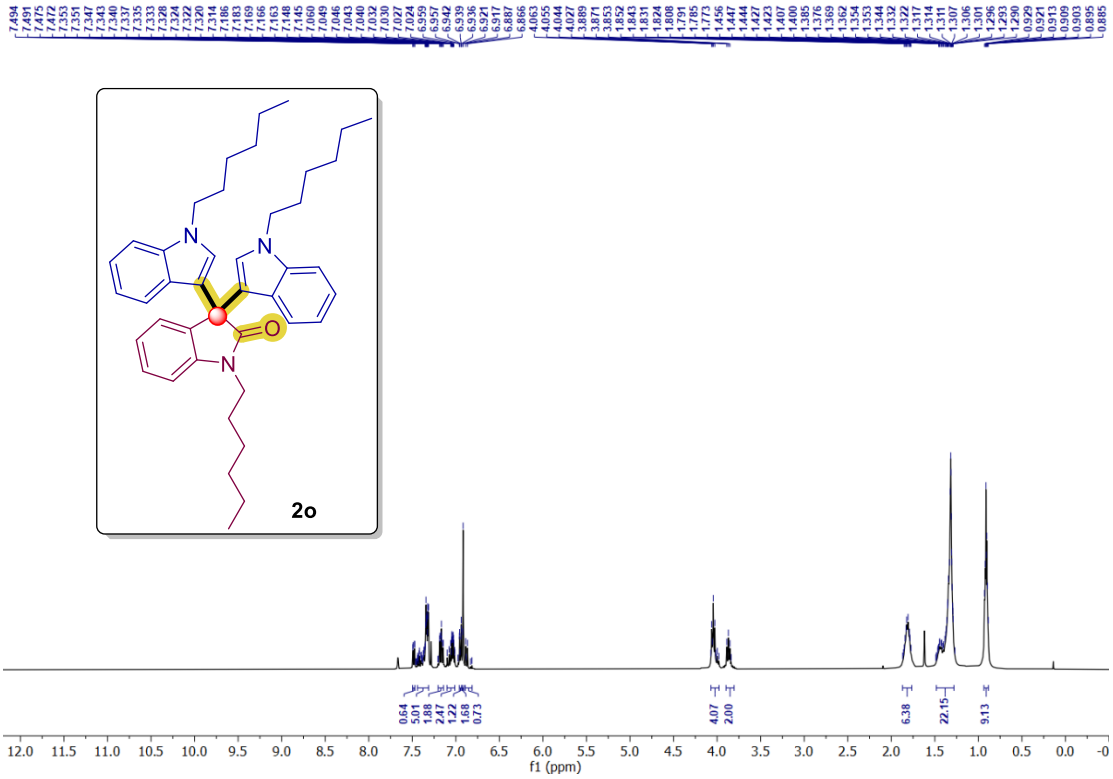
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 1,1',1''-tripentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2n)



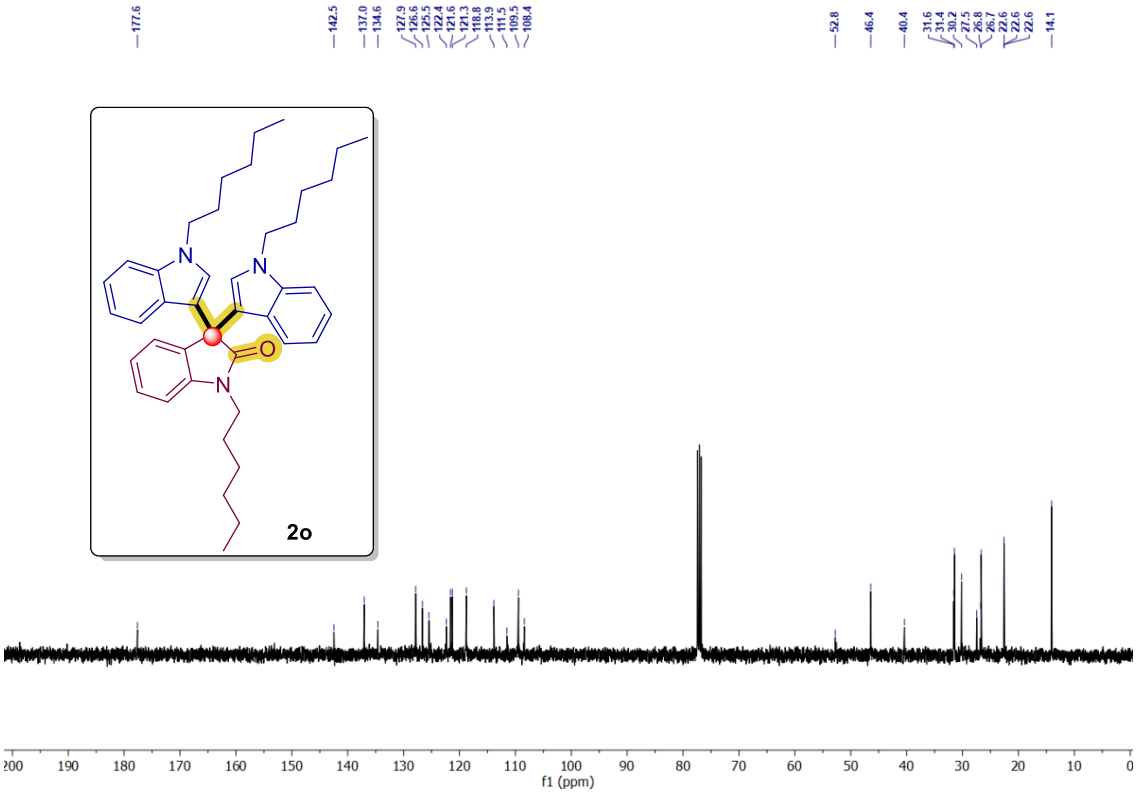
HRMS spectrum of 1,1',1''-tripentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2n)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1,1',1''-trihexyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2o)

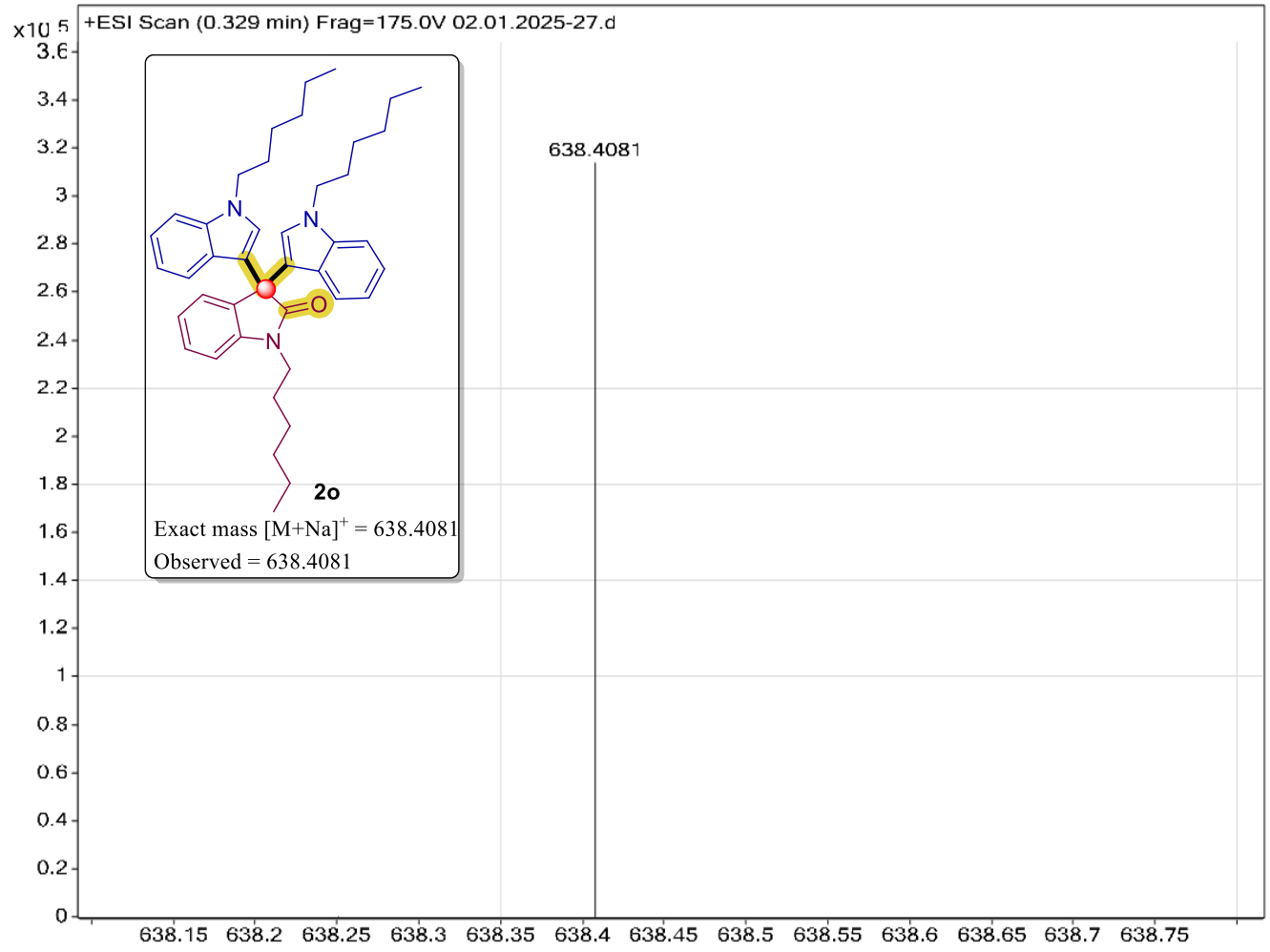


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 1,1',1''-trihexyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2o)

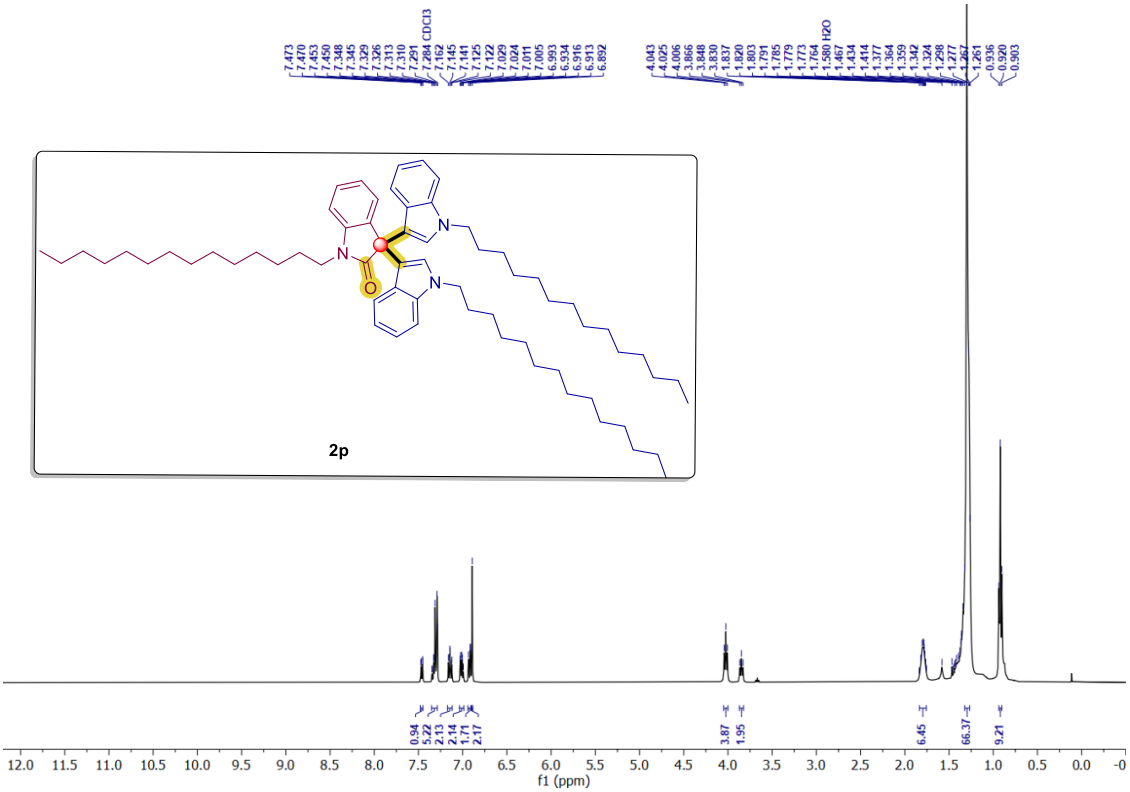


HRMS spectrum of 1,1',1''-trihexyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2o)

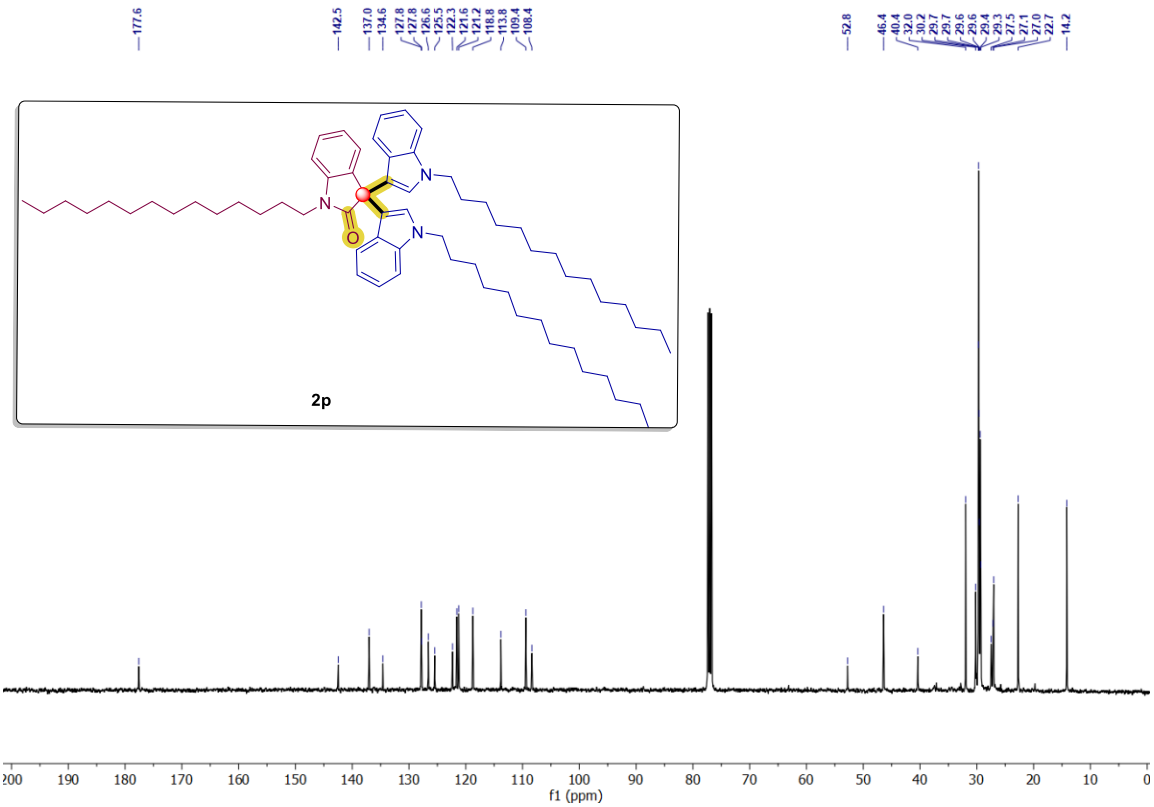
Sample Name	khp-ns-c6	**Position**	P1-C9	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	02.01.2025-27.d	**ACQ Method**	M60 W40.m	**Comment**		**Acquired Time**	03-01-2025 14:59:09



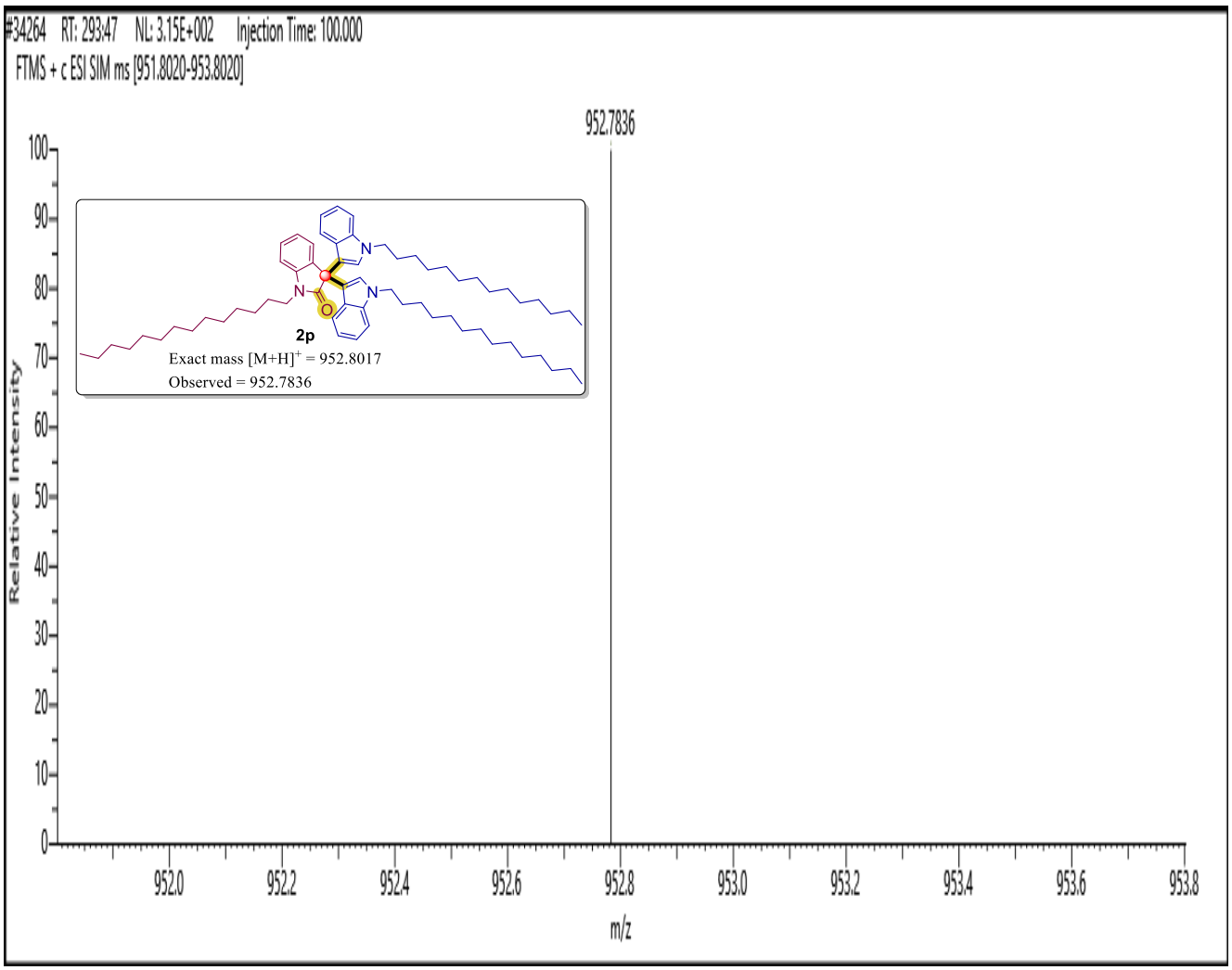
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1,1',1''-tritetradecyl-[3,3':3',3''-terindolin]-2'-one (2p)



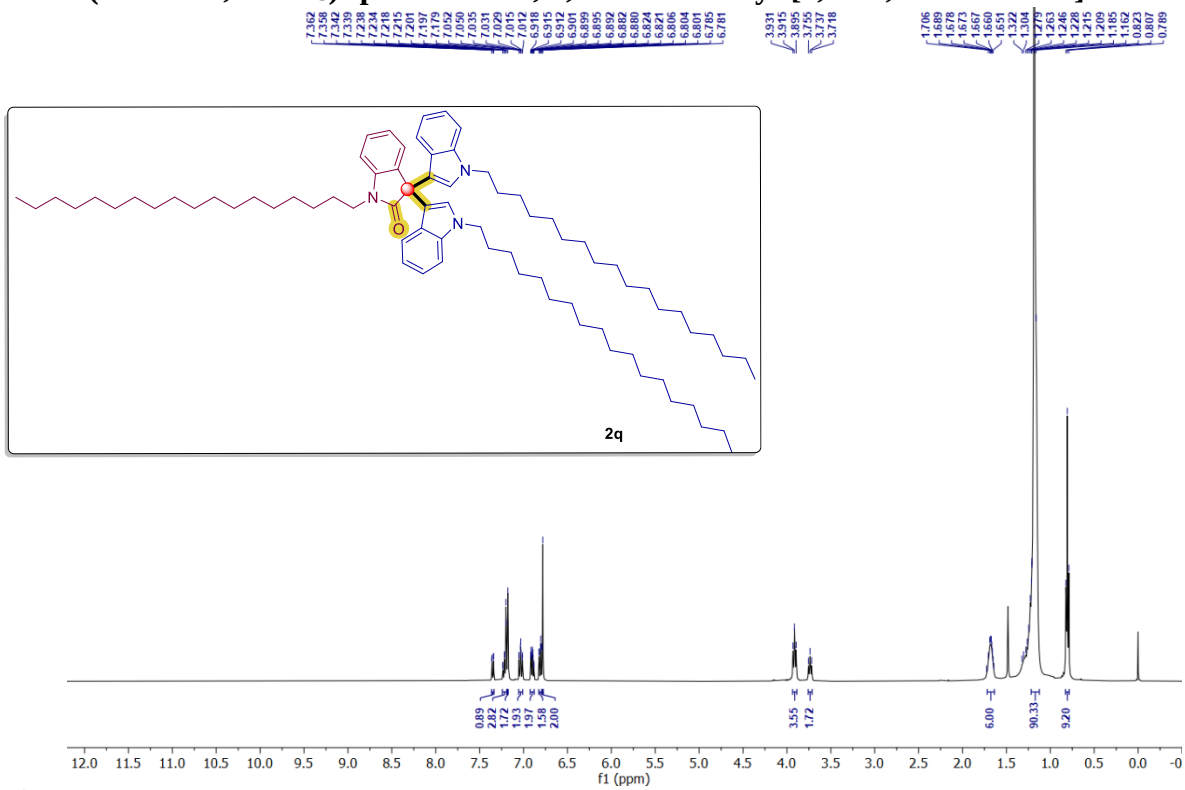
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 1,1',1''-tritetradecyl-[3,3':3',3''-terindolin]-2'-one (2p)



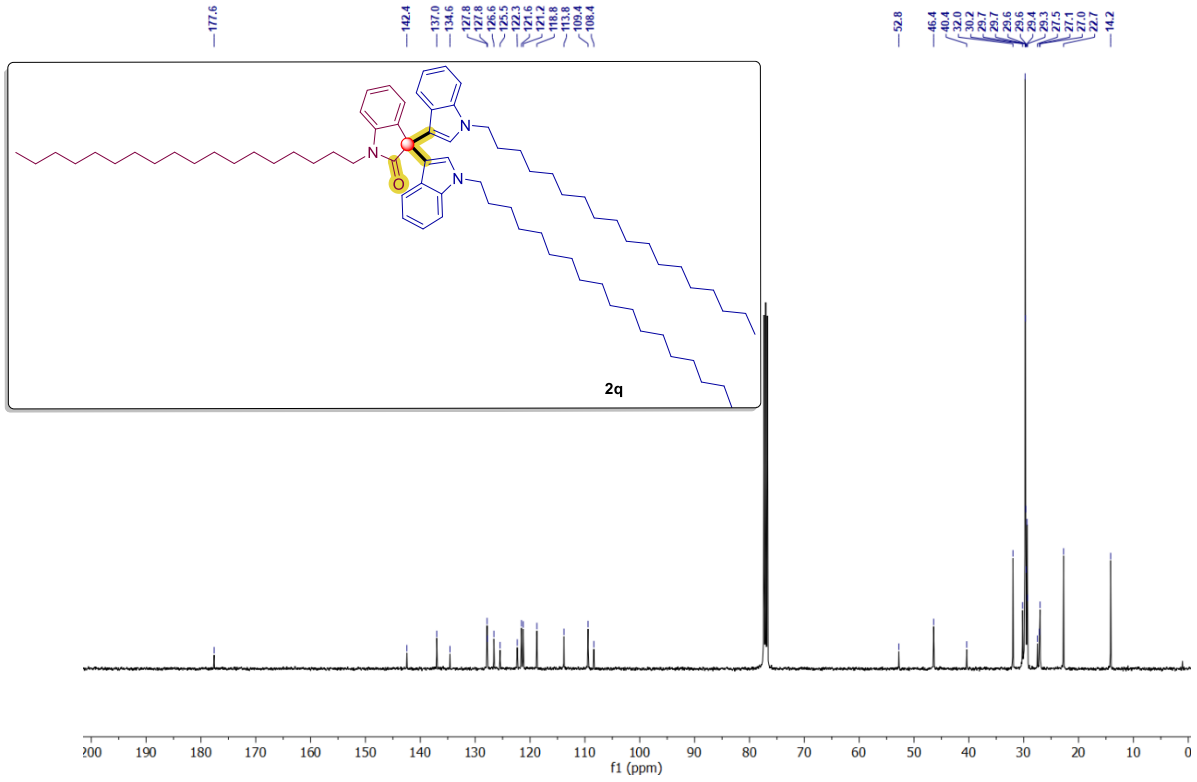
HRMS spectrum of 1,1',1''-tritetradecyl-[3,3':3',3''-terindolin]-2'-one (2p)



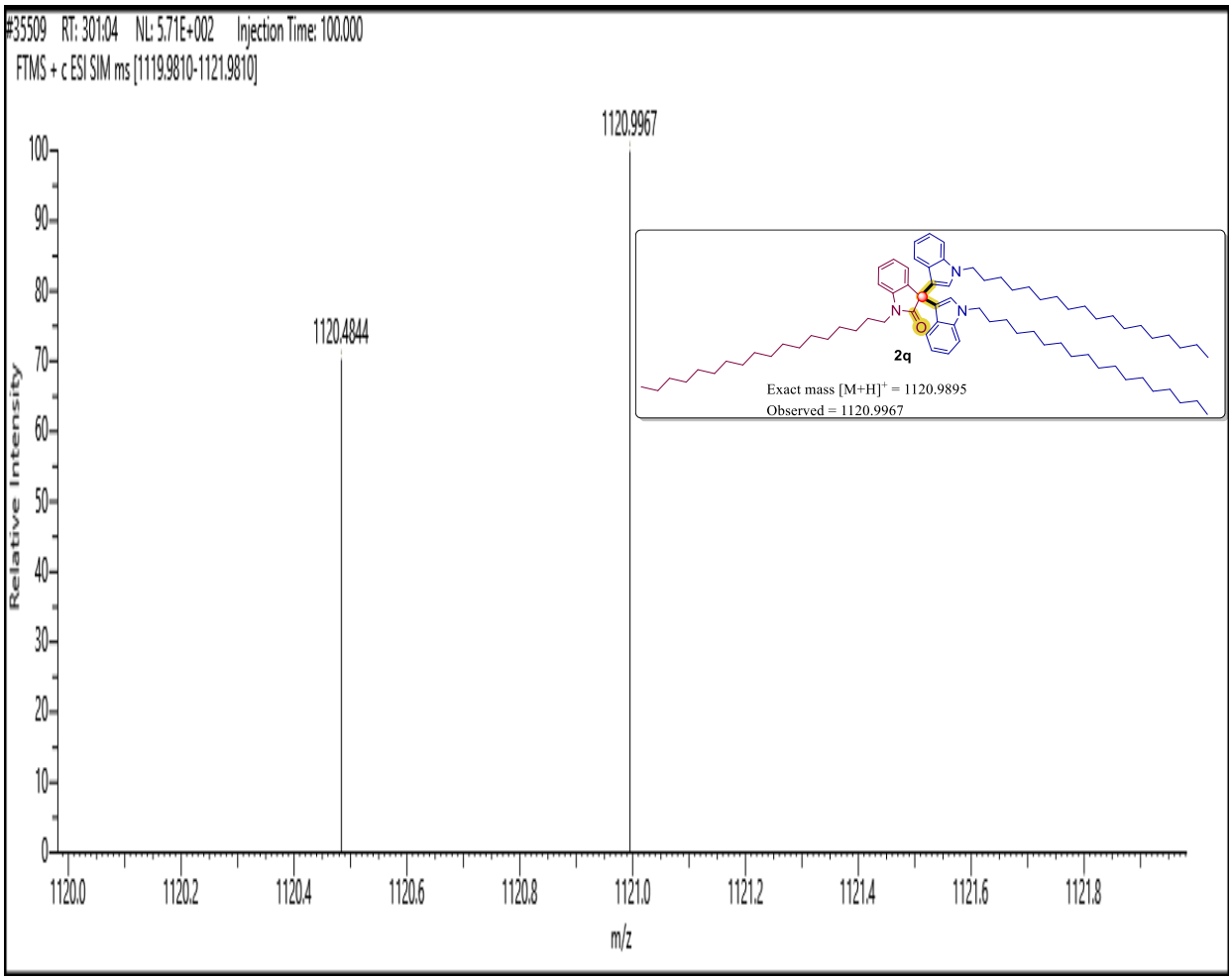
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1,1',1''-trioctadecyl-[2,3':3',2''-terindolin]-2'-one (2q)



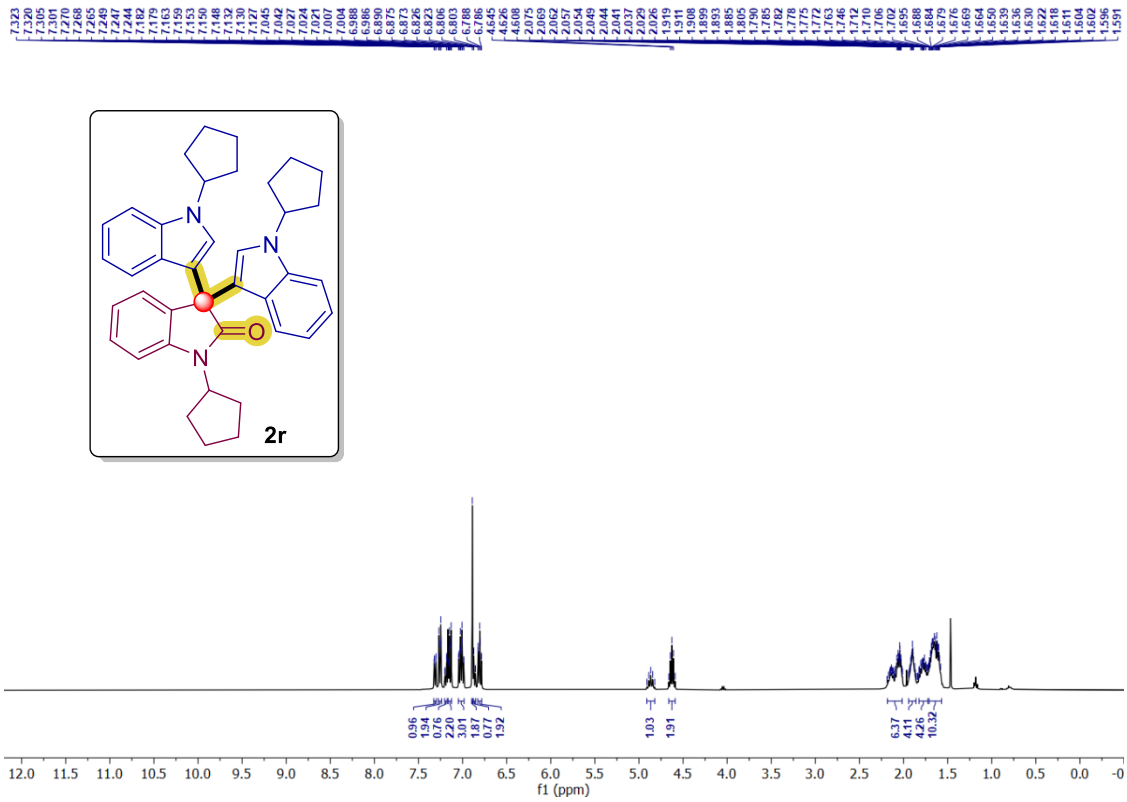
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 1,1',1''-trioctadecyl-[2,3':3',2''-terindolin]-2'-one (2q)



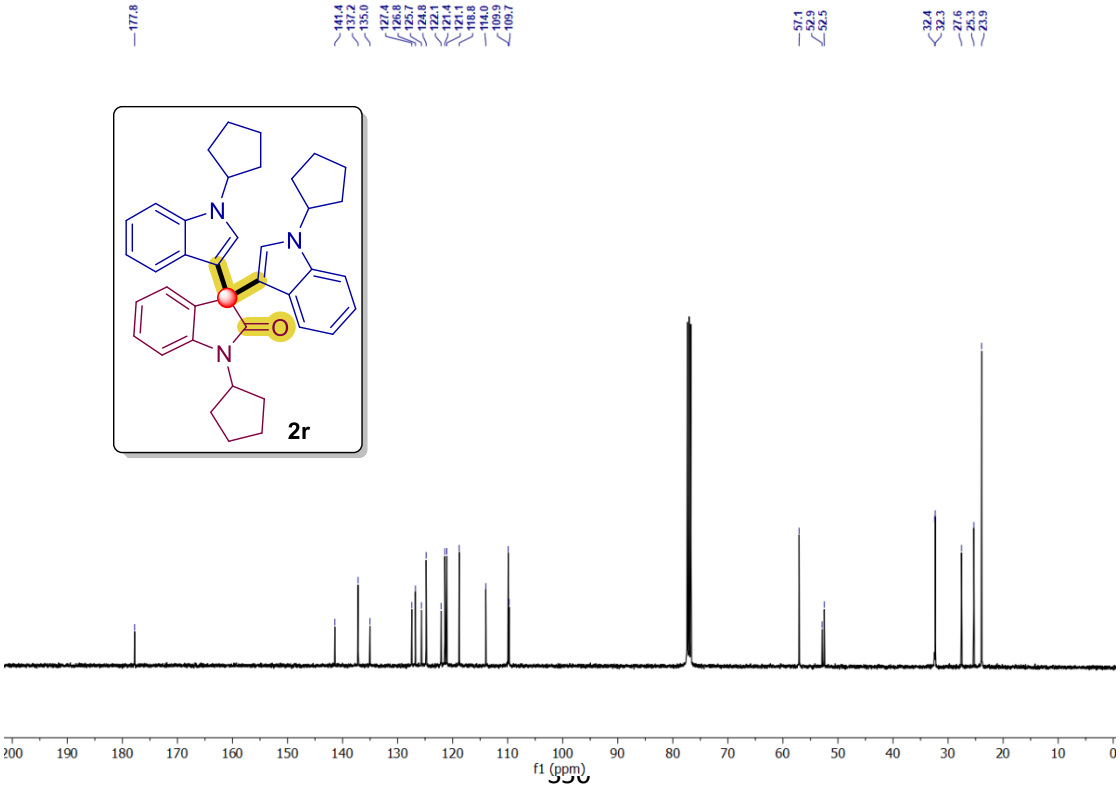
HRMS spectrum of 1,1',1''-trioctadecyl-[2,3':3',2''-terindolin]-2'-one (2q)



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1,1',1''-tricyclopentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2r)

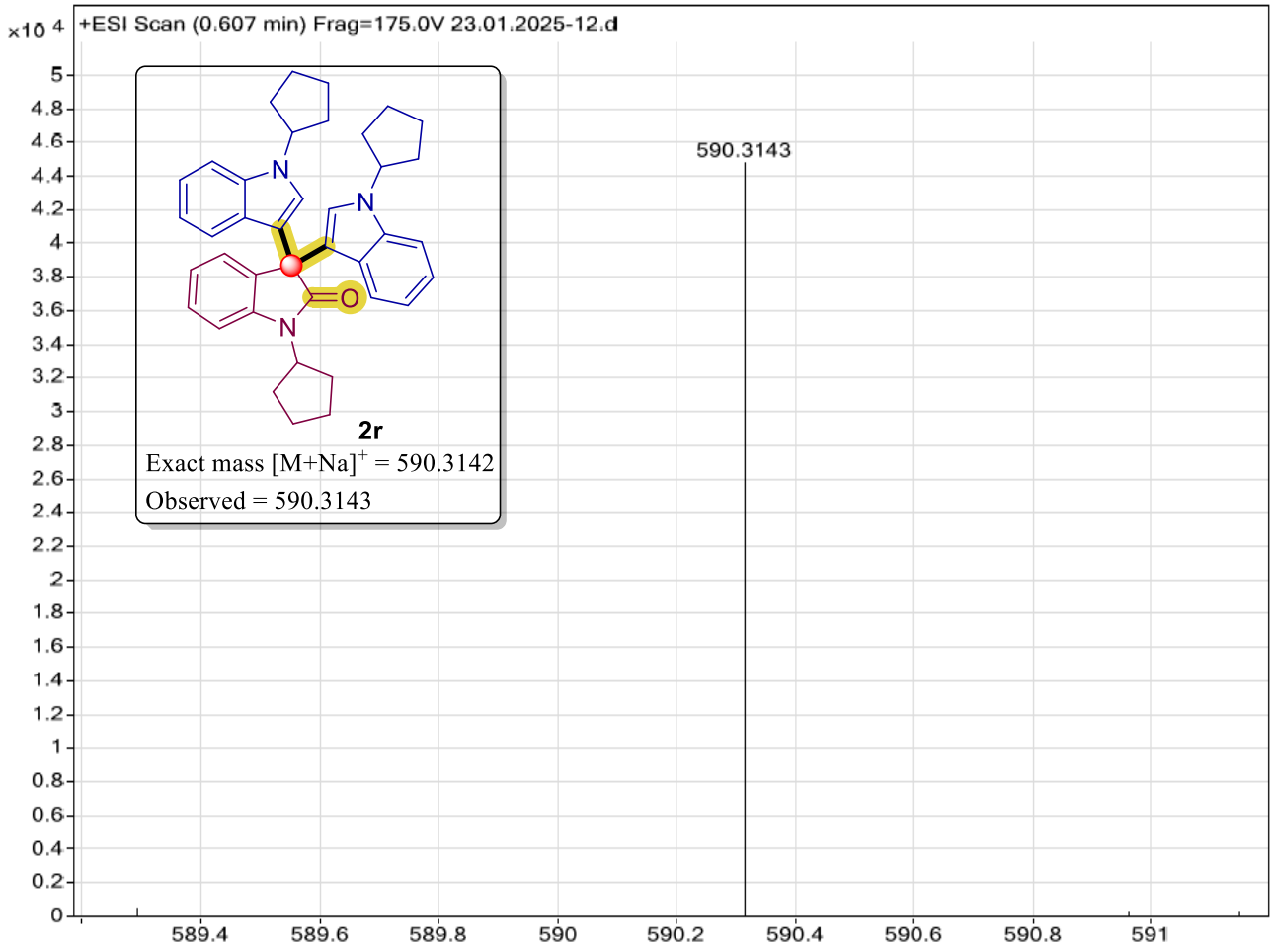


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 1,1',1''-tricyclopentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2r)

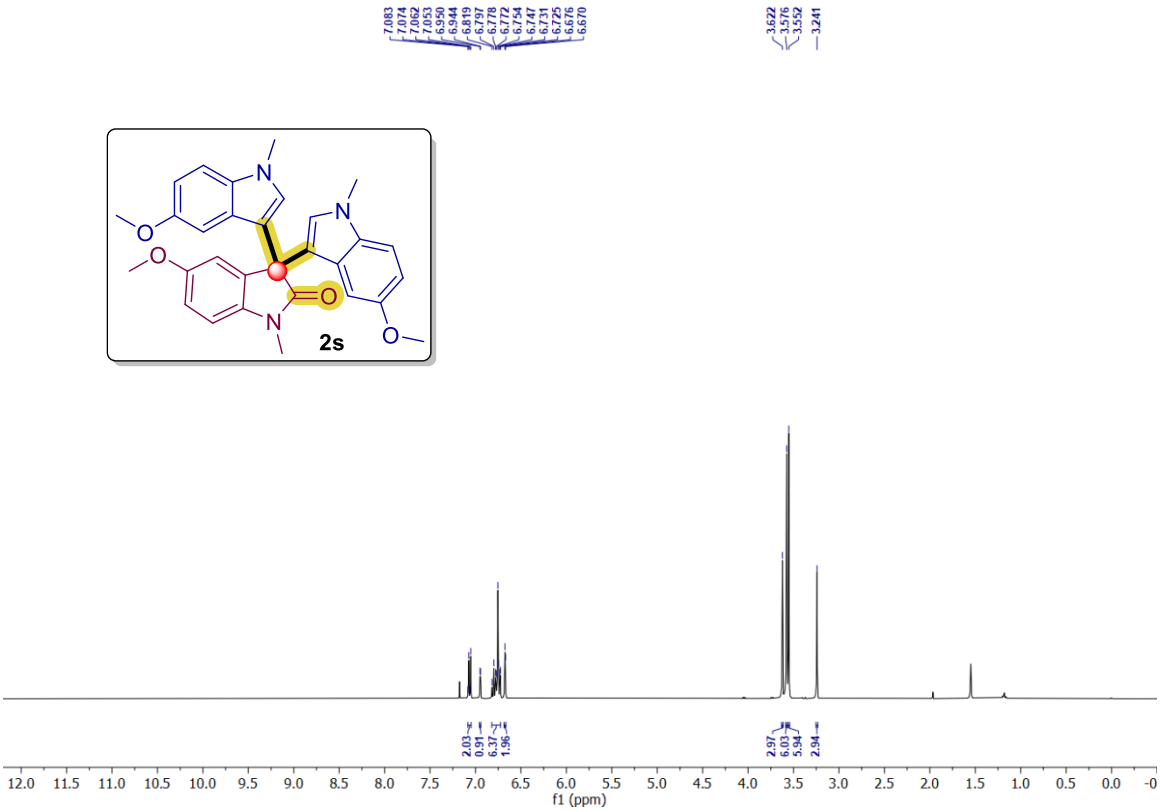


HRMS spectrum of 1,1',1''-tricyclopentyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2r)

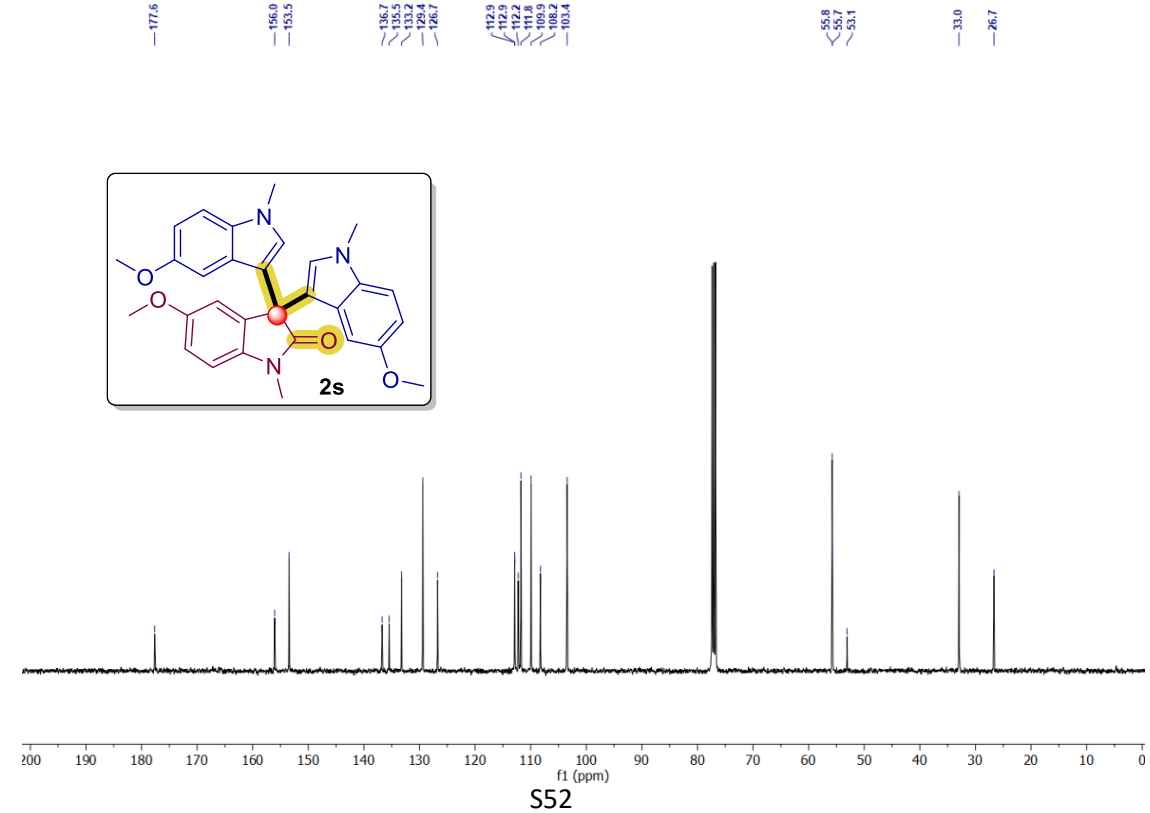
Sample Name	khp-ns-n-cy	Position	P1-B3	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	23.01.2025-12.d	ACQ Method	M60 W40.m	Comment		Acquired Time	24-01-2025 10:14:21



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5,5',5''-trimethoxy-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2s)

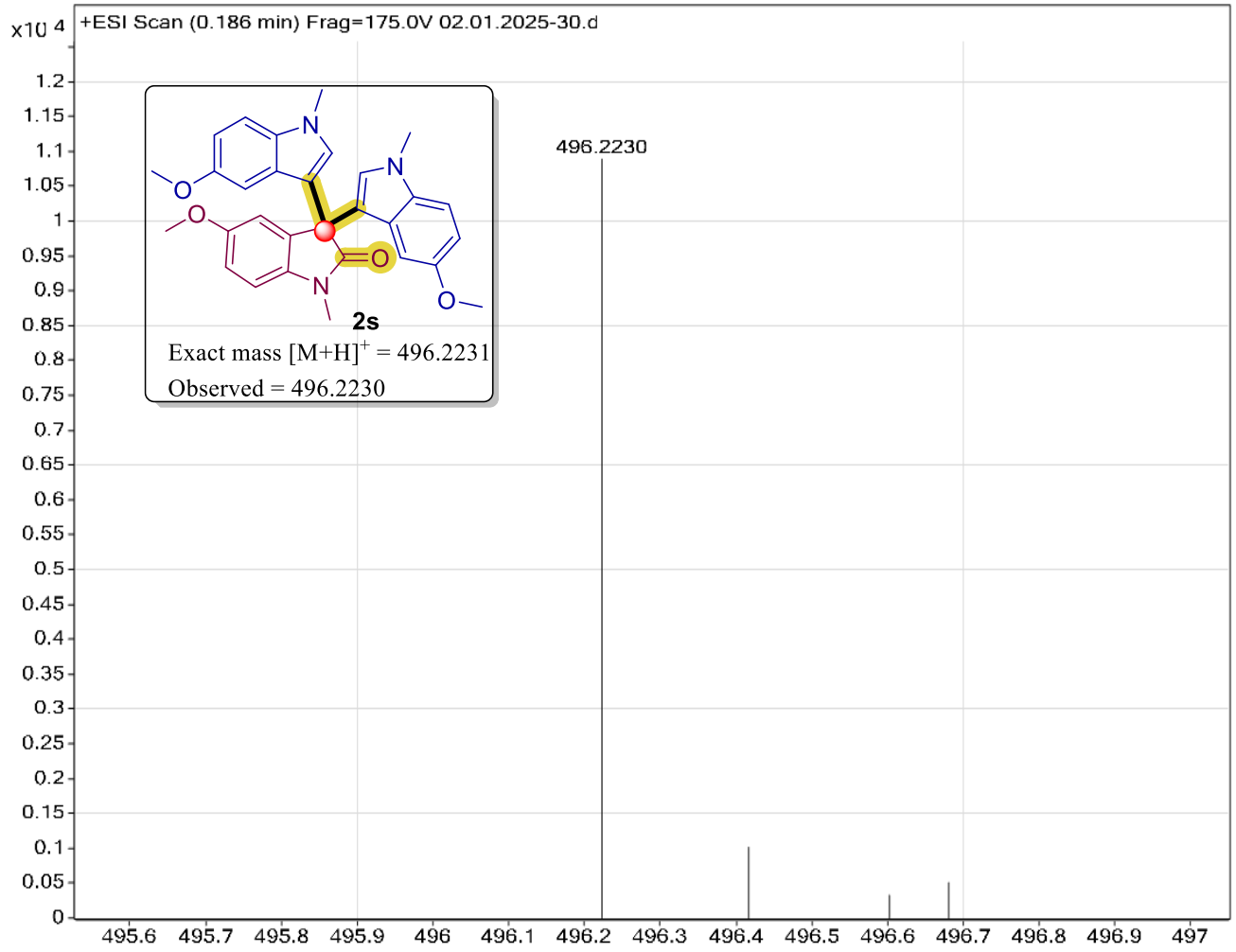


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 5,5',5''-trimethoxy-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2s)

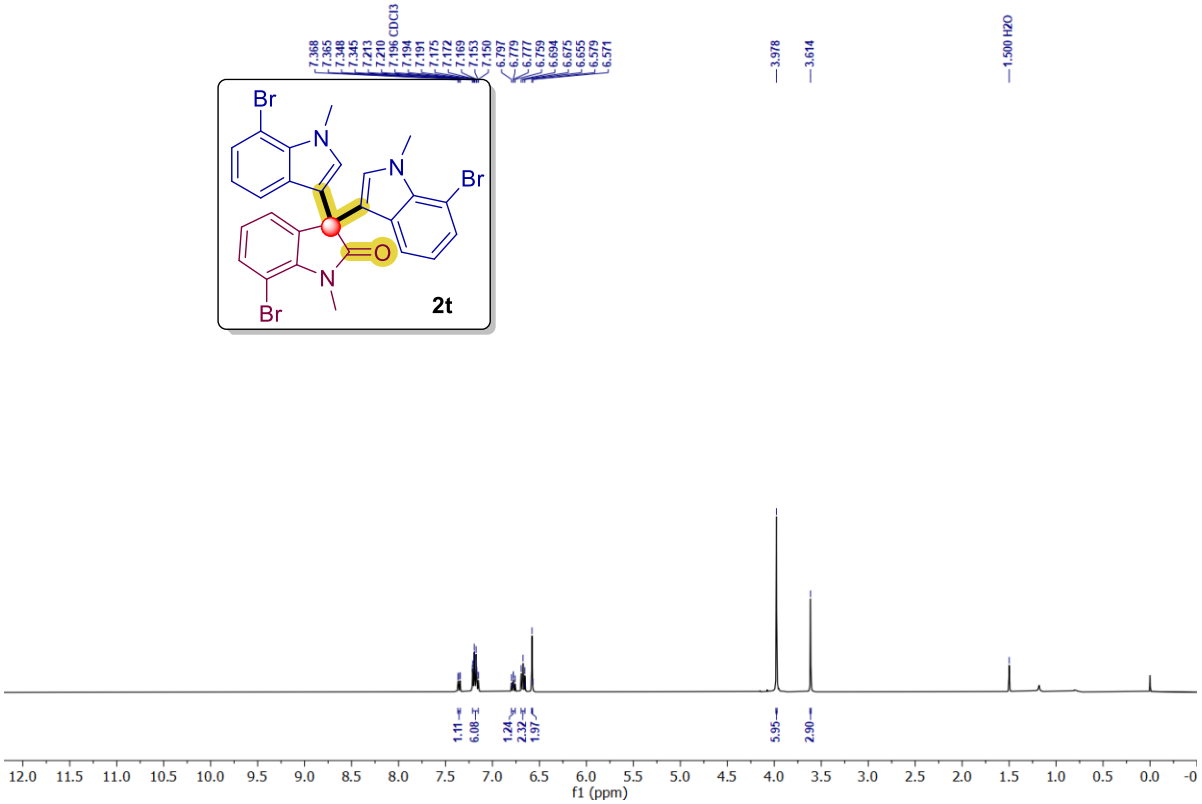


HRMS spectrum of 5,5',5''-trimethoxy-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2s)

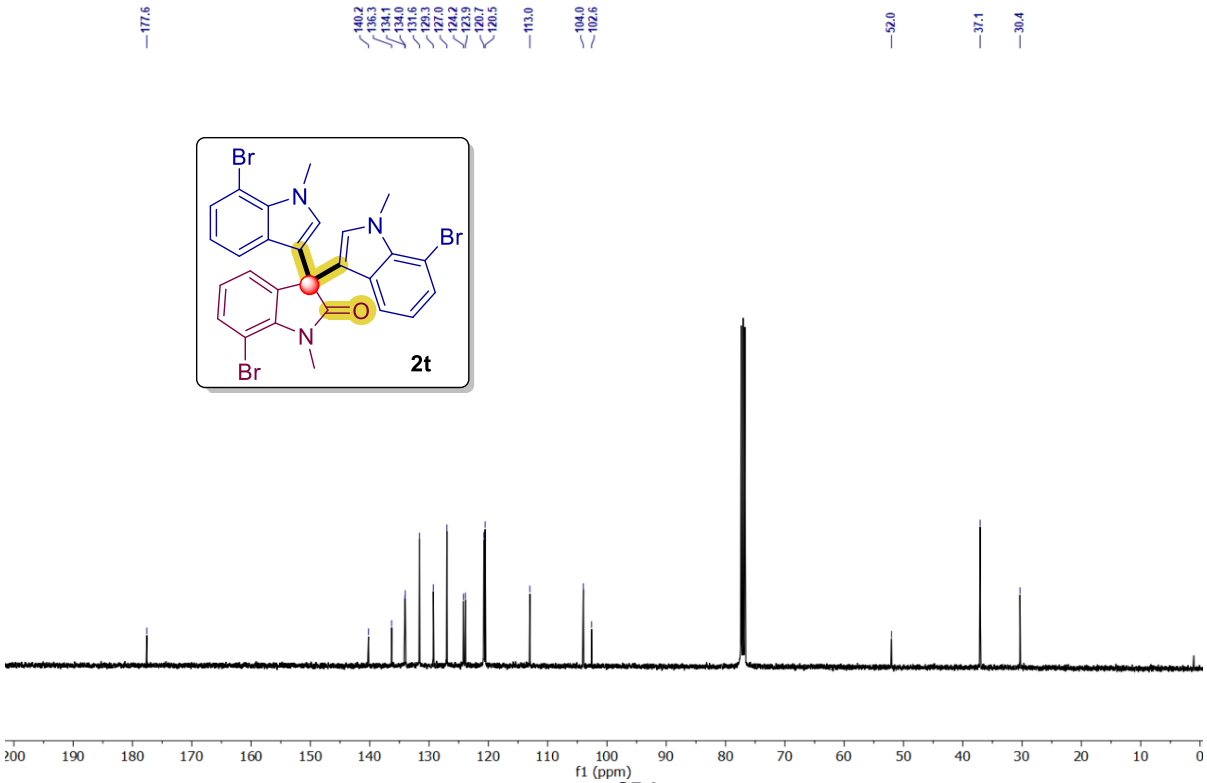
Sample Name	khp-ns-5-ome	**Position**	P1-D3	**Instrument Name**	Instrument 1	**User Name**	
Inj Vol	1	**InjPosition**		**SampleType**	Sample	**IRM Calibration Status**	Success
Data Filename	02.01.2025-30.d	**ACQ Method**	M60 W40.m	**Comment**		**Acquired Time**	03-01-2025 15:11:21



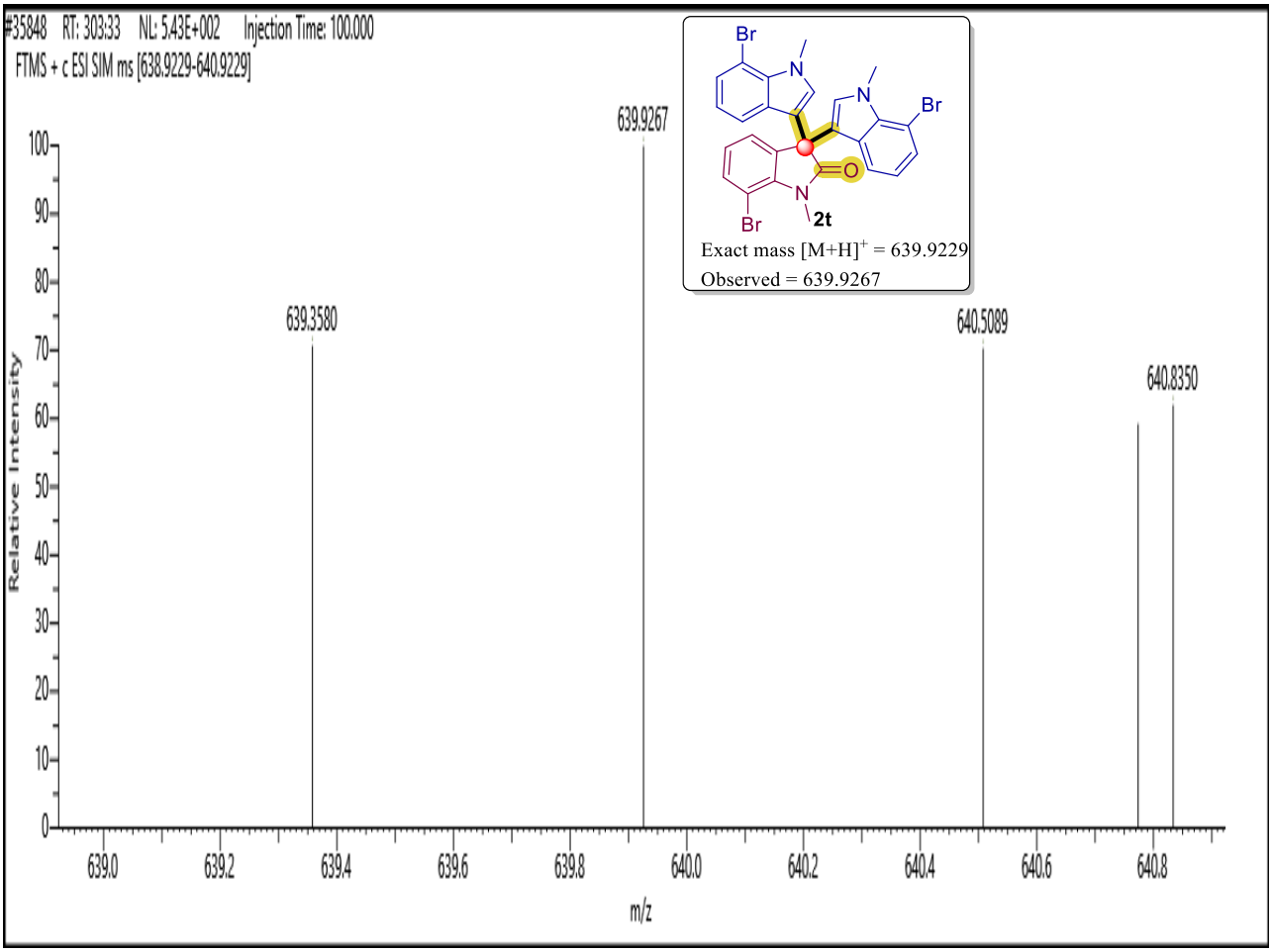
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 7,7',7''-tribromo-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2t)



^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 7,7',7''-tribromo-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2t)

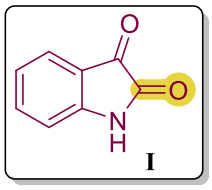


HRMS spectrum of 7,7'',7''-tribromo-1,1',1''-trimethyl-1*H*,1''*H*-[3,3':3',3''-terindol]-2'(1'*H*)-one (2t)



Characterization data of 1*H*-Indole-2,3-dione (I)

Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); orange

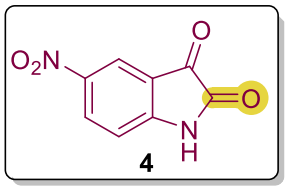


I

solid; Yield: (59 mg, 40%); mp: 197-198 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.03 (s.1H), 7.60 (tt, J = 7.6, 1.6 Hz, 1H), 7.52-7.48 (m,1H), 7.07 (tt, J = 7.4,1.8 Hz, 1H), 6.94 – 6.89 (m, 1H).^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 184.8, 159.8, 151.2, 138.8, 125.2, 123.3, 118.3, 112.7.

Characterization data of 5-nitroindoline-2,3-dione (4)

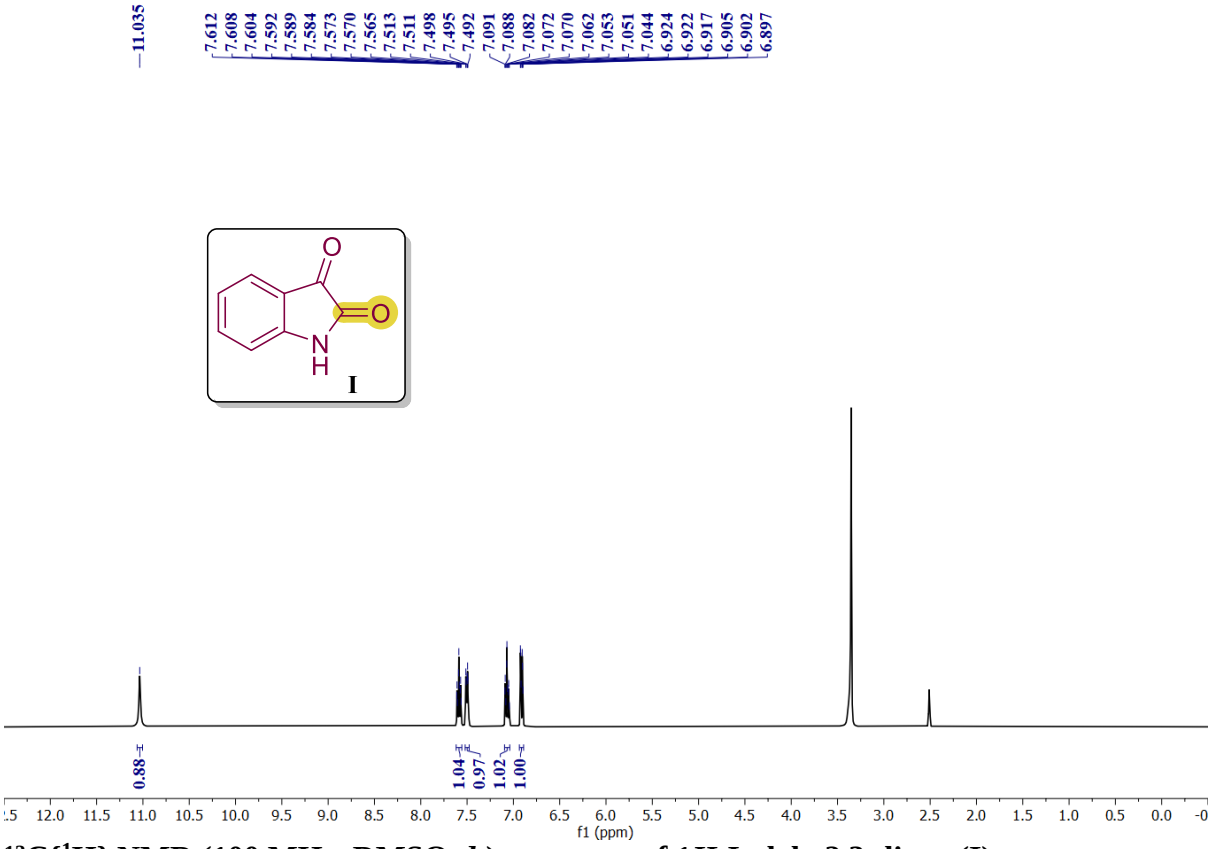
Purified by column chromatography eluting with petroleum ether/ethyl acetate = 7:3 (v/v); yellow



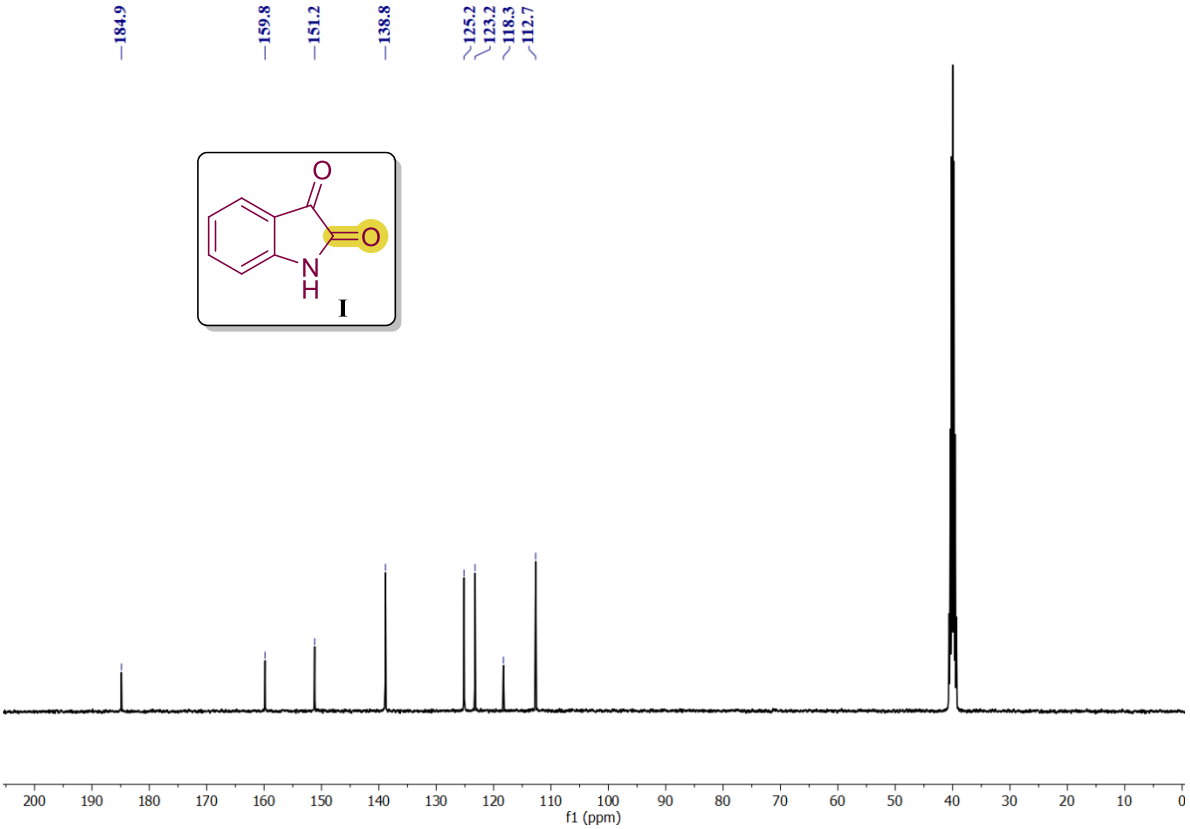
4

solid; Yield: (109 mg, 62%); mp: 253-254 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 11.32 (s.1H), 7.78 (dd, J = 8.2, 1.2 Hz, 1H), 7.51 (dd, J = 7.2, 1.0 Hz, 1H), 7.02 (t, J = 7.6, Hz, 1H). ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 184.1, 160.2, 149.9, 140.8, 124.6, 124.1, 120.6, 105.1.

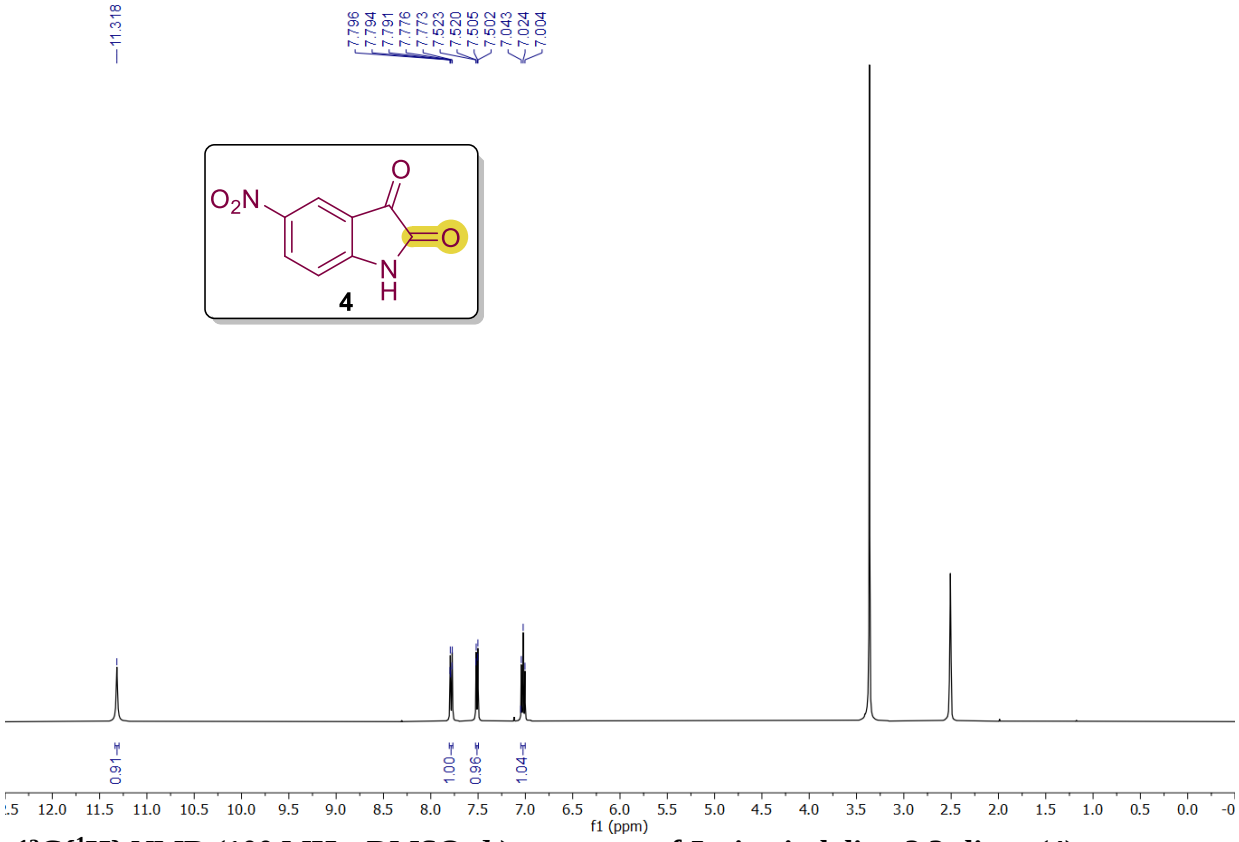
^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1*H*-Indole-2,3-dione (I)



^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1*H*-Indole-2,3-dione (I)



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5-nitroindoline-2,3-dione (4)



^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5-nitroindoline-2,3-dione (4)