

Supporting Information

Arenethiolate-catalyzed C_{aryl}-F bond activation: Synthesis of oxindoles

Shengyun Liu,^a Mingying Li,^b Wei Xiao*^b and Jie Wu*^{bcd}

^a School of Chemistry and Chemical Engineering, Key Laboratory of Surface & Interface Science of Polymer Materials of Zhejiang Province, Zhejiang Sci-Tech University, Hangzhou 310018, People's Republic of China.

^b School of Pharmaceutical and Chemical Engineering & Institute for Advanced Studies, Taizhou University, Taizhou 318000, China.

^c State Key Laboratory of Organometallic Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai 200032, China.

^d School of Chemistry and Chemical Engineering, Henan Normal University, Xinxiang 453007, China.

Corresponding author: xiaowei1992@tzc.edu.cn; jie_wu@fudan.edu.cn

Contents

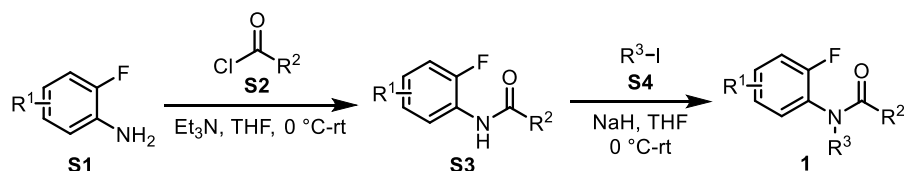
1. General information	2
2. General procedure for the synthesis of substrates 1 and 3.....	3
3. General procedure for the arenethiolate-catalyzed C-F bond activation to access oxindoles.....	5
4. Condition optimization for the arenethiolate-catalyzed C-F bond activation to access oxindoles.....	6
5. TEMPO trapping experiments.....	7
6. UV-vis absorption spectra.....	7
7. Reference	8
8. Characterization of all products.....	8
9. NMR spectra of compounds	18

1. General information

All glassware was thoroughly oven-dried. Chemicals and solvents were either purchased from commercial suppliers or purified by standard techniques. Thin-layer chromatography plates were visualized by exposure to ultraviolet light and/or staining with phosphomolybdic acid followed by heating on a hot plate. Flash chromatography was carried out using silica gel (200–300 mesh). ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker AM-400 (400 MHz). The spectra were recorded in deuteriochloroform (CDCl_3) as solvent at room temperature, ^1H and ^{13}C NMR chemical shifts are reported in ppm relative to the residual solvent peak. The residual solvent signals were used as references and the chemical shifts were converted to the TMS scale (CDCl_3 : $\delta_{\text{H}} = 7.26$ ppm, $\delta_{\text{C}} = 77.0$ ppm). Data for ^1H NMR are reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, dd = doublet, br = broad), integration, coupling constant (Hz) and assignment. Data for ^{13}C NMR are reported as chemical shift. Mass spectra were measured on Shimadzu GCMS-QP2010 instrument (EI). Electrospray-ionisation HRMS data were acquired on a Q-TOF mass spectrometer (Waters SYNAPT G2-Si) LC-MS TOF. UV/vis absorption spectra were acquired on a UV-5 spectrophotometer

2. General procedure for the synthesis of substrates 1 and 3

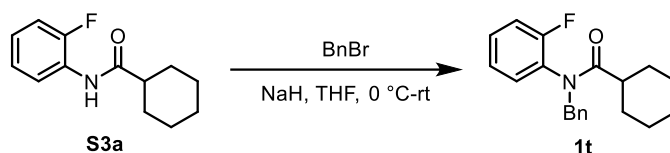
2.1. General procedure for the synthesis of substrates 1a-1s.



To a stirred, cooled (0–5 °C) solution of 2-fluoroaniline **S1** (0.96 mL, 10 mmol) and Et₃N (1.55 mL, 11 mmol) in THF (20 mL), a solution of acyl chloride **S2** (10 mmol) in THF (5 mL) was added dropwise within 10 min. Then the ice bath was removed and the mixture was stirred vigorously for 30 min at room temperature. Then the solid Et₃N·HCl was filtered off and washed with THF (3 x 5 mL) and the resulting organic layers were combined and THF was removed under reduced pressure to yield crude amides **S3**. Recrystallization from hexane/CHCl₃ and drying in vacuum afforded pure compounds **S3**.

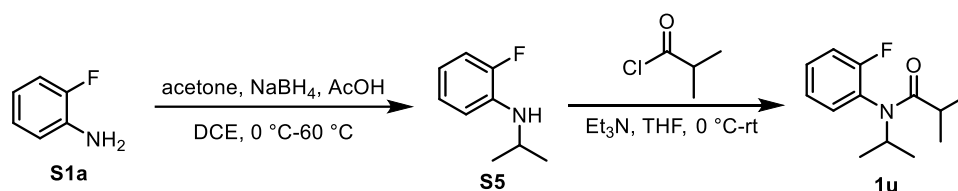
To a stirred suspension of NaH (132.0 mg, 5.5 mmol) in THF (5 mL) at 0 °C, the respective amide **S3** (5 mmol) dissolved in THF (10 mL) was added dropwise within 10 min. The reaction mixture was stirred until the solution became clear (30 min, with hydrogen gas evolved), and the solution of the corresponding alkyl iodide **S4** (6.5 mmol) in THF (5 mL) was added dropwise within 10 min. The solution was warmed up to room temperature. The reaction mixture was monitored by TLC and quenched with water (30 mL). The resulting solution was extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine (1 x 20 mL) and dried over Na₂SO₄. The ethyl acetate was removed under reduced pressure to give the crude product, which was purified by flash chromatography to afford the pure compound **1a-1s**.¹

2.2. General procedure for the synthesis of substrates 1t.



To a stirred suspension of NaH (132.0 mg, 5.5 mmol) in THF (5 mL) at 0 °C, the amide **S3a** (5 mmol) dissolved in THF (10 mL) was added dropwise within 10 min. The reaction mixture was stirred until the solution became clear (30 min, with hydrogen gas evolved), and the solution of BnBr (0.77 mL, 6.5 mmol) in THF (5 mL) was added dropwise within 10 min. The solution was warmed up to room temperature. The reaction mixture was monitored by TLC and quenched with water (30 mL). Then the resulting solution was extracted with ethyl acetate (3 x 20 mL). The combined organic layers were washed with brine (1 x 20 mL) and dried over Na₂SO₄. The ethyl acetate was removed under reduced pressure to give the crude product, which was purified by flash chromatography to afford the pure compound **1t**.

2.3. General procedure for the synthesis of substrates **1u**.

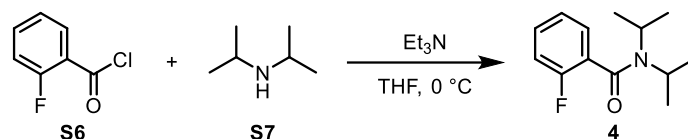


Acetic acid (7.72 mL, 45 mmol) was added to the suspension of NaBH₄ (0.56 g, 15 mmol) in DCE or CH₂Cl₂ at 0 °C dropwise over 1 h and the resulting suspension was stirred at room temperature for 1-18 h. The mixture was added a solution of an aniline derivative **S1** (0.96 mL, 10 mmol), ketone (0.73 mL, 10 mmol), and acetic acid (1.7 mL, 10 mmol) in DCE. The solution was stirred at rt-60 °C for 3-16 h. The mixture was quenched by H₂O, and the organic layer was washed with H₂O (3 times) and all the volatiles were removed in vacuo. The residue was purified by flash chromatography to give the corresponding N-alkylaniline **S5**.²

To a stirred, cooled (0–5 °C) solution of N-alkylaniline **S6** (0.76 g, 5 mmol) and Et₃N (0.55 g, 0.77 mL, 5.5 mmol) in 10 mL of dry THF, a solution of an appropriate acyl chloride (5 mmol) in 5 mL of dry THF was added dropwise within 10 min. Then the ice bath was removed and the mixture was stirred vigorously for 30 min at room temperature. After solid Et₃N·HCl was filtered off and washed with THF (3 x 5 mL),

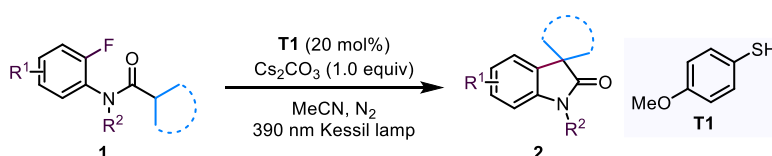
the resulting organic fractions were combined and THF was removed under reduced pressure to yield crude amides **1u**, which was purified by flash chromatography to afford the pure compound **1u**.¹

2.4. General procedure for the synthesis of substrates **4**.



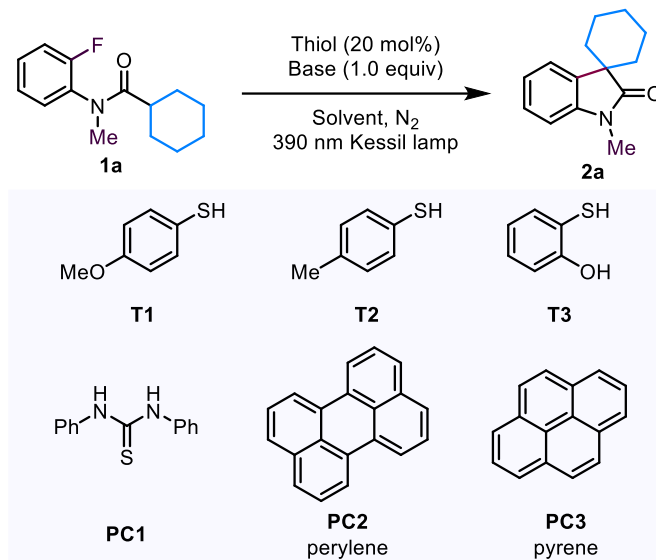
To a stirred, cooled (0–5 °C) solution of diisopropylamine **S7** (0.96 mL, 10 mmol) and Et₃N (1.55 mL, 11 mmol) in THF (20 mL), a solution of acyl chloride **S6** (10 mmol) in THF (5 mL) was added dropwise within 10 min. Then the ice bath was removed and the mixture was stirred vigorously for 30 min at room temperature. After solid Et₃N·HCl was filtered off and washed with THF (3 x 5 mL), the resulting organic layers were combined and THF was removed under reduced pressure to yield crude product, which was purified by flash chromatography to afford the pure product **4**.

3. General procedure for the arenethiolate-catalyzed C-F bond activation to access oxindoles.



All optimization reactions were set up in a glove box under N₂ atmosphere. Substrate **1** (0.2 mmol, 1.0 equiv), 4-methoxybenzenethiol (0.04 mmol, 0.2 equiv), Cs₂CO₃ (0.2 mmol, 1.0 equiv) were added to dry MeCN (2 mL) at room temperature under 390 nm Kessil lamp. The resulting mixture was stirred at rt for 24 h. Upon completion of the reaction, the solvent was removed under reduced pressure and the crude product was purified by column chromatography on silica gel to afford the desired product **2**. The light source is a 40 W Kessil LED photoreaction lighting and the intensity values are the average of PR160L-390 nm. The distance from the light source to the irradiation vessel is about 3 cm.

4. Condition optimization for the arenethiolate-catalyzed C-F bond activation to access oxindoles.^a

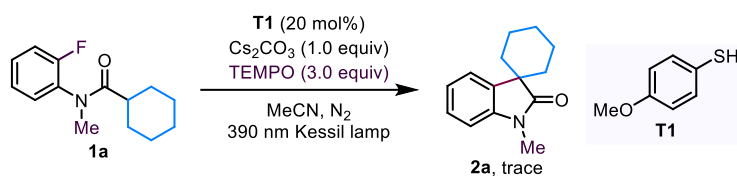


Entry	Photocatalyst	Base	Solvent	Yield (%) ^b
1	T1	K ₂ CO ₃	DMSO	57
2 ^c	T1	K ₂ CO ₃	DMSO	24
3 ^d	T1	K ₂ CO ₃	DMSO	/
4	T2	K ₂ CO ₃	DMSO	50
5	T3	K ₂ CO ₃	DMSO	17
6	T1	KHCO ₃	DMSO	56
7	T1	Cs ₂ CO ₃	DMSO	71
8	T1	Cs ₂ CO ₃	DMF	77
9	T1	Cs ₂ CO ₃	MeCN	82 (77) ^e
10	T1	Cs ₂ CO ₃	THF	/
11	T1	Cs ₂ CO ₃	DCM	/
12	T1	Cs ₂ CO ₃	DCE	/
13	T1	Cs ₂ CO ₃	MeOH	13
14	T1	Cs ₂ CO ₃	EtOH	34
15	Ru(bpy) ₃ Cl ₂	Cs ₂ CO ₃	MeCN	33
16	<i>fac</i> -Ir(ppy) ₃	Cs ₂ CO ₃	MeCN	62

17	PC1	Cs ₂ CO ₃	MeCN	59
18	PC2	Cs ₂ CO ₃	MeCN	30
19	PC3	Cs ₂ CO ₃	MeCN	48

^a Unless otherwise noted, reaction conditions are as follows: **1a** (0.2 mmol), thiol (0.04 mmol), base (0.2 mmol), solvent (2.0 mL), 390 nm Kessil lamp, 24 h, under a N₂ atmosphere; ^b Yields determined by ¹H NMR yield using trimethoxybenzene as an internal standard; ^c Under air; ^d In the dark; ^e isolated yield in parenthesis.

5. TEMPO trapping experiments



Substrate **1a** (0.2 mmol, 1.0 equiv), 4-methoxybenzenethiol (0.04 mmol, 0.2 equiv), Cs₂CO₃ (0.2 mmol, 1.0 equiv) and TEMPO (0.6 mmol, 3.0 equiv) were added to dry MeCN (2 mL) at room temperature under 390 nm Kessil lamp. The resulting mixture was stirred at rt for 24 h. Only trace amount of product **2a** was detected, indicating that the reaction is proceeded through a radical pathway.

6. UV-vis absorption spectra

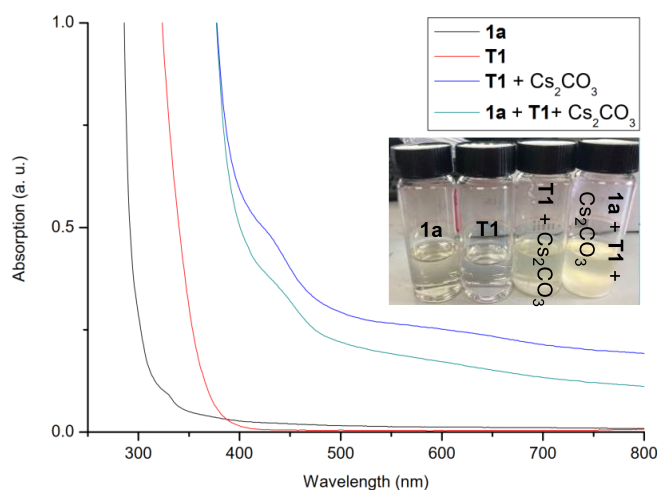


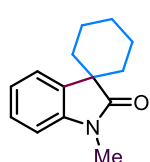
Figure S1. Absolute absorption spectra of 4-methoxybenzenethiol, substrate **1a**, Cs₂CO₃ and their mixtures. The UV/vis spectra of **1a** (0.1 M in MeCN), 4-methoxybenzenethiol (0.02 M in MeCN), Cs₂CO₃ (0.1 M in MeCN).

7. Reference

1. J.-Q. Chen, R. Chang, J.-B. Lin, Y.-C. Luo, P.-F. Xu, Photoredox-induced intramolecular 1,5-H transfer reaction of aryl iodides for the synthesis of spirocyclic γ -lactams. *Org. Lett.*, 2018, **20**, 2395-2398.
2. S. Okumura, T. Komine, E. Shigeki, K. Eemba, Y. Nakao, Site-selective linear alkylation of anilides by cooperative nickel/aluminum catalysis. *Angew. Chem. Int. Ed.*, 2018, **57**, 929-932.

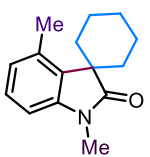
8. Characterization of all products

1'-Methylspiro[cyclohexane-1,3'-indolin]-2'-one (2a)



Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Yellow oil; 33.1 mg, 77% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.45 (d, *J* = 7.4 Hz, 1H), 7.27 (td, *J* = 7.7, 1.2 Hz, 1H), 7.04 (td, *J* = 7.6, 1.1 Hz, 1H), 6.84 (d, *J* = 7.7 Hz, 1H), 3.20 (s, 3H), 2.01–1.89 (m, 2H), 1.89–1.80 (m, 2H), 1.80–1.67 (m, 3H), 1.67–1.60 (m, 1H), 1.60–1.52 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 180.7, 142.8, 135.4, 127.4, 123.9, 121.9, 107.9, 47.5, 33.0, 26.2, 25.2, 21.2.

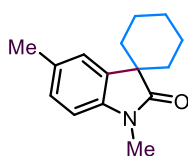
1',4'-Dimethylspiro[cyclohexane-1,3'-indolin]-2'-one (2b)



Purification by flash chromatography (*n*-hexane/ethyl acetate = 15/1); White solid; mp 52–54 °C; 18.8 mg, 41% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.14 (t, *J* = 7.8 Hz, 1H), 6.79 (d, *J* = 7.8 Hz, 1H), 6.64 (d, *J* = 7.7 Hz, 1H), 3.15 (s, 3H), 2.45 (s, 3H), 2.38–2.22 (m, 2H), 2.13 (td, *J* = 13.8, 4.4 Hz, 2H), 1.95–1.83 (m, 1H), 1.69–1.52 (m, 4H), 1.40–1.18 (m, 1H); ¹³C NMR (100

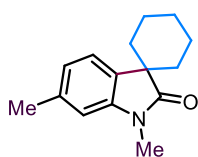
MHz, CDCl₃) δ (ppm) = 180.3, 143.2, 133.8, 131.9, 127.5, 125.5, 105.6, 47.4, 30.0, 25.8, 25.5, 20.5, 18.6; HRMS (ESI) for C₁₅H₂₀NO [M+H]⁺ calcd. 230.1539, found 230.1538

1',5'-Dimethylspiro[cyclohexane-1,3'-indolin]-2'-one (2c)



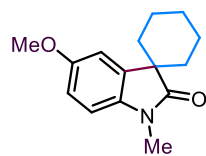
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 35.7 mg, 78% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.27 (s, 1H), 7.07 (d, *J* = 7.8 Hz, 1H), 6.73 (d, *J* = 7.9 Hz, 1H), 3.18 (s, 3H), 2.36 (s, 3H), 2.00–1.88 (m, 2H), 1.88–1.76 (m, 3H), 1.76–1.69 (m, 2H), 1.68–1.60 (m, 1H), 1.59–1.51 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 180.7, 140.5, 135.5, 131.3, 127.6, 124.8, 107.6, 47.5, 33.1, 26.2, 25.2, 21.3, 21.3; HRMS (ESI) for C₁₅H₁₉NO [M+H]⁺ calcd. 230.1539, found 230.1537

1',6'-Dimethylspiro[cyclohexane-1,3'-indolin]-2'-one (2d)



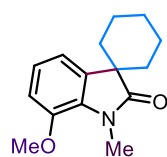
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Yellow oil; 23.0 mg, 50% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.35 (d, *J* = 7.6 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 6.68 (s, 1H), 3.18 (s, 3H), 2.39 (s, 3H), 1.97–1.87 (m, 2H), 1.87–1.79 (m, 2H), 1.79–1.67 (m, 3H), 1.67–1.57 (m, 1H), 1.57–1.49 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 181.0, 143.0, 137.5, 132.5, 123.7, 122.3, 108.9, 47.4, 33.1, 26.1, 25.2, 21.7, 21.3; HRMS (ESI) for C₁₅H₂₀NO [M+H]⁺ calcd. 230.1539, found 230.1538.

5'-Methoxy-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2e)



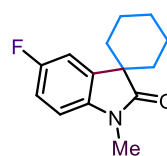
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 39.2 mg, 75% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.08 (d, *J* = 2.4 Hz, 1H), 6.79 (dd, *J* = 8.4, 2.4 Hz, 1H), 6.74 (d, *J* = 8.4 Hz, 1H), 3.81 (s, 3H), 3.18 (s, 3H), 2.00–1.89 (m, 2H), 1.89–1.80 (m, 2H), 1.80–1.67 (m, 3H), 1.67–1.59 (m, 1H), 1.59–1.50 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 180.4, 155.5, 136.8, 136.5, 112.3, 110.8, 107.8, 55.9, 47.8, 33.0, 26.2, 25.1, 21.2.

7'-Methoxy-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2f)



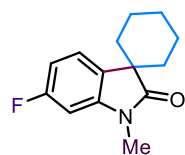
Purification by flash chromatography (*n*-hexane/ethyl acetate = 15/1); Yellow oil; 26.6 mg, 54% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.05 (dd, J = 7.4, 0.8 Hz, 1H), 6.98 (t, J = 7.9 Hz, 1H), 6.85 (dd, J = 8.2, 0.7 Hz, 1H), 3.85 (s, 3H), 3.47 (s, 3H), 2.09–1.90 (m, 2H), 1.86–1.76 (m, 2H), 1.76–1.62 (m, 4H), 1.61–1.50 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 180.9, 145.2, 137.2, 130.7, 122.4, 116.6, 111.5, 56.0, 47.3, 33.3, 29.4, 25.2, 21.1; HRMS (ESI) for $\text{C}_{15}\text{H}_{20}\text{NO}_2$ $[\text{M}+\text{H}]^+$ calcd. 246.1489, found 246.1488.

5'-Fluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2g)



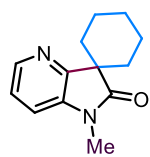
Purification by flash chromatography (*n*-hexane/ethyl acetate = 15/1); Colorless oil; 35.5 mg, 76% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.21 (dd, J = 8.5, 2.6 Hz, 1H), 6.97 (td, J = 8.8, 2.6 Hz, 1H), 6.75 (dd, J = 8.5, 4.3 Hz, 1H), 3.19 (s, 3H), 2.03–1.90 (m, 2H), 1.90–1.79 (m, 2H), 1.77–1.58 (m, 4H), 1.58–1.47 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 180.4, 158.8 (J = 239.3 Hz), 138.8, 136.9 (J = 7.8 Hz), 113.4 (J = 23.3 Hz), 112.2 (J = 25.0 Hz), 108.1 (J = 8.2 Hz), 47.9, 32.9, 26.3, 25.0, 21.1; HRMS (ESI) for $\text{C}_{14}\text{H}_{17}\text{FNO}$ $[\text{M}+\text{H}]^+$ calcd. 234.1289, found 234.1289.

6'-Fluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2h)



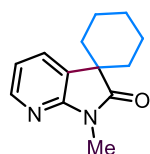
Purification by flash chromatography (*n*-hexane/ethyl acetate = 10/1); Colorless oil; 10.7 mg, 23% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.21 (td, J = 8.1, 5.3 Hz, 1H), 6.72 (t, J = 9.1 Hz, 1H), 6.61 (d, J = 7.7 Hz, 1H), 3.17 (s, 3H), 2.19–2.04 (m, 2H), 2.04–1.93 (m, 2H), 1.85–1.72 (m, 3H), 1.72–1.62 (m, 2H), 1.55–1.40 (m, 1H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 179.8, 158.9 (J = 247.0 Hz), 145.1 (J = 10.2 Hz), 129.2 (J = 9.0 Hz), 120.6 (J = 19.0 Hz), 110.3 (J = 22.4 Hz), 103.9 (J = 2.9 Hz), 47.6, 31.6, 26.3, 25.1, 20.7; HRMS (ESI) for $\text{C}_{14}\text{H}_{17}\text{FNO}$ $[\text{M}+\text{H}]^+$ calcd. 234.1289, found 234.1287.

1'-Methylspiro[cyclohexane-1,3'-pyrrolo[3,2-b]pyridin]-2'(1'H)-one (2i)



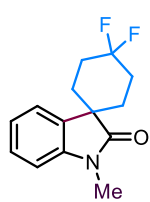
Purification by flash chromatography (*n*-hexane/ethyl acetate = 15/1); White solid; mp 72–73 °C; 33.3 mg, 77% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 8.22 (dd, *J* = 5.1, 1.2 Hz, 1H), 7.16 (dd, *J* = 7.8, 5.1 Hz, 1H), 7.05 (dd, *J* = 7.8, 1.2 Hz, 1H), 3.21 (s, 3H), 2.06–1.85 (m, 4H), 1.83–1.57 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 179.2, 156.2, 142.1, 137.5, 122.0, 113.6, 47.3, 31.7, 25.8, 25.4, 20.7; HRMS (ESI) for C₁₃H₁₇N₂O [M+H]⁺ calcd. 217.1335, found 217.1335.

1'-methylspiro[cyclohexane-1,3'-pyrrolo[2,3-b]pyridin]-2'(1'H)-one (2j)



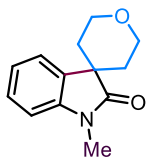
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); White solid; mp 64–66 °C; 32.8 mg, 76% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 8.18 (dd, *J* = 5.3, 1.6 Hz, 1H), 7.70 (dd, *J* = 7.3, 1.4 Hz, 1H), 6.94 (dd, *J* = 7.3, 5.3 Hz, 1H), 3.29 (s, 3H), 2.00–1.82 (m, 4H), 1.82–1.50 (m, 6H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 180.3, 156.3, 146.2, 131.1, 129.5, 117.6, 47.5, 32.5, 25.4, 25.0, 21.4; HRMS (ESI) for C₁₃H₁₇N₂O [M+H]⁺ calcd. 217.1335, found 217.1334.

4,4-Difluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2k)



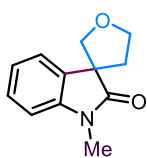
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); White solid; mp 70–71 °C; 39.3 mg, 91% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.30 (td, *J* = 7.7, 1.1 Hz, 1H), 7.25 (d, *J* = 7.7 Hz, 1H), 7.08 (td, *J* = 7.6, 0.8 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 3.21 (s, 3H), 2.74–2.53 (m, 2H), 2.18–2.03 (m, 2H), 2.05–1.85 (m, 4H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 179.2, 142.8, 133.4, 128.2, 123.2 (*J* = 240.2 Hz), 122.6, 122.6, 108.2, 45.1, 30.3 (*J* = 3.2 Hz), 29.2 (*J* = 24.6 Hz), 26.1; HRMS (ESI) for C₁₄H₁₆F₂NO [M+H]⁺ calcd. 252.1194, found 252.1193.

1-Methyl-2',3',5',6'-tetrahydrospiro[indoline-3,4'-pyran]-2-one (2l)



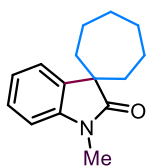
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); White solid; mp 94–95 °C; 28.0 mg, 64% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.38 (d, *J* = 7.4 Hz, 1H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.09 (t, *J* = 7.0 Hz, 1H), 6.86 (d, *J* = 7.7 Hz, 1H), 4.34–4.19 (m, 2H), 3.98–3.88 (m, 2H), 3.21 (s, 3H), 1.86 (t, *J* = 5.4 Hz, 4H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 179.7, 142.8, 134.1, 128.0, 123.1, 122.5, 108.1, 63.0, 44.3, 32.9, 26.1.

1'-Methyl-4,5-dihydro-2H-spiro[furan-3,3'-indolin]-2'-one (2m)



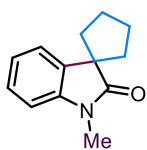
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); White solid; mp 84–85 °C; 22.7 mg, 56% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.35–7.23 (m, 2H), 7.09 (t, *J* = 7.2 Hz, 1H), 6.86 (d, *J* = 7.6 Hz, 1H), 4.28–4.15 (m, 2H), 4.03 (d, *J* = 8.4 Hz, 1H), 3.93 (d, *J* = 8.5 Hz, 1H), 3.24 (s, 3H), 2.58–2.47 (m, 1H), 2.20–2.09 (m, 1H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 178.2, 142.9, 134.0, 128.1, 123.0, 122.6, 108.0, 69.1, 54.3, 38.6, 26.4; HRMS (ESI) for C₁₂H₁₄NO₂ [M+H]⁺ calcd. 204.1019, found 204.1019.

1'-Methylspiro[cycloheptane-1,3'-indolin]-2'-one (2n)



Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 26.2 mg, 57% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.34 (d, *J* = 7.4 Hz, 1H), 7.25 (t, *J* = 7.2 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.82 (d, *J* = 7.7 Hz, 1H), 3.18 (s, 3H), 2.05–1.86 (m, 4H), 1.81–1.62 (m, 8H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 182.2, 142.5, 137.4, 127.4, 122.7, 122.3, 107.8, 50.1, 36.9, 31.3, 26.1, 23.8.

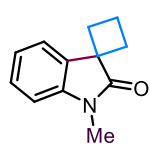
1'-Methylspiro[cyclopentane-1,3'-indolin]-2'-one (2o)



Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); White solid; mp 55–56 °C; 19.3 mg, 48% yield; ¹H NMR (400 MHz, CDCl₃) δ (ppm) = 7.24 (t, *J* = 7.7 Hz, 1H), 7.20 (d, *J* = 7.3 Hz, 1H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.82 (d, *J* = 7.7 Hz, 1H), 3.21 (s, 3H), 2.20–2.02 (m, 4H), 2.02–1.91 (m, 2H), 1.90–1.77 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ (ppm) = 182.0, 142.9,

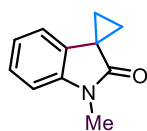
136.9, 127.4, 122.5, 122.2, 107.7, 53.9, 38.3, 26.7, 26.3.

1'-Methylspiro[cyclobutane-1,3'-indolin]-2'-one (2p)



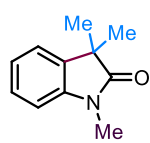
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Yellow oil; 9.3 mg, 25% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.51 (dd, $J = 7.3, 0.6$ Hz, 1H), 7.26 (td, $J = 7.7, 1.2$ Hz, 1H), 7.10 (td, $J = 7.6, 0.9$ Hz, 1H), 6.79 (d, $J = 7.7$ Hz, 1H), 3.19 (s, 3H), 2.74–2.58 (m, 2H), 2.44–2.19 (m, 4H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 180.2, 143.0, 134.4, 127.8, 122.6, 122.2, 107.6, 48.1, 31.3, 26.2, 16.8.

1'-Methylspiro[cyclopropane-1,3'-indolin]-2'-one (2q)



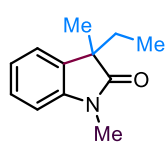
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); White solid; mp 78–79 °C; 28.4 mg, 82% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.26 (t, $J = 7.7$ Hz, 1H), 7.03 (t, $J = 7.5$ Hz, 1H), 6.91 (d, $J = 7.8$ Hz, 1H), 6.84 (d, $J = 7.1$ Hz, 1H), 3.30 (s, 3H), 1.74 (dd, $J = 4.0, 7.6$ Hz, 2H), 1.51 (q, $J = 4.0, 7.6$ Hz, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 177.1, 143.6, 130.9, 126.8, 122.0, 118.3, 108.0, 27.1, 26.6, 19.2.

1,3,3-Trimethylindolin-2-one (2r)



Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Yellow oil; 20.3 mg, 58% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.26 (td, $J = 7.7, 1.1$ Hz, 1H), 7.21 (d, $J = 7.3$ Hz, 1H), 7.06 (td, $J = 7.6, 0.1$ Hz, 1H), 6.85 (d, $J = 7.8$ Hz, 1H), 3.22 (s, 3H), 1.37 (s, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 181.4, 142.6, 135.8, 127.7, 122.5, 122.3, 108.0, 44.2, 26.2, 24.4.

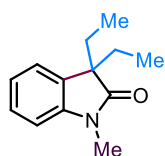
3-Ethyl-1,3-dimethylindolin-2-one (2s)



Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Yellow oil; 24.6 mg, 65% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.27 (t, $J = 7.7$ Hz, 1H), 7.17 (d, $J = 7.2$ Hz, 1H), 7.07 (t, $J = 7.4$ Hz, 1H), 6.84 (d, $J = 7.8$ Hz, 1H), 3.22 (s, 3H), 2.00–1.86 (m, 1H), 1.84–1.71 (m, 1H), 1.35

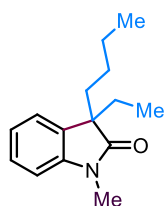
(s, 3H), 0.59 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 180.8, 143.5, 134.0, 127.6, 122.5, 122.4, 107.8, 49.0, 31.5, 26.1, 23.3, 8.9.

3,3-Diethyl-1-methylindolin-2-one (2t)



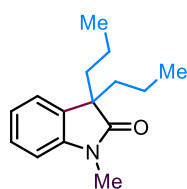
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Yellow oil; 31.5 mg, 77% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 7.27 (t, $J = 7.6$, 1.4 Hz, 1H), 7.14 (d, $J = 6.4$ Hz, 1H), 7.08 (t, $J = 7.4$, 0.8 Hz, 1H), 6.84 (d, $J = 7.7$ Hz, 1H), 3.22 (s, 3H), 1.99–1.86 (m, 2H), 1.85–1.72 (m, 2H), 0.56 (t, $J = 7.4$ Hz, 6H).; ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 180.1, 144.4, 132.0, 127.6, 122.7, 122.3, 107.7, 54.4, 30.6, 25.9, 8.7.

3-Butyl-3-ethyl-1-methylindolin-2-one (2u)



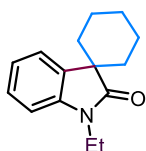
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 33.7 mg, 73% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 7.27 (t, $J = 7.5$, 1.3 Hz, 1H), 7.14 (d, $J = 7.3$, 0.9 Hz, 1H), 7.07 (t, $J = 7.4$, 0.8 Hz, 1H), 6.84 (d, $J = 7.7$ Hz, 1H), 3.22 (s, 3H), 1.97–1.81 (m, 2H), 1.81–1.69 (m, 4H), 1.31–1.06 (m, 1H), 1.02–0.86 (m, 1H), 0.76 (t, $J = 7.3$ Hz, 3H), 0.55 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 180.3, 144.2, 132.4, 127.5, 122.7, 122.4, 107.7, 53.7, 37.6, 31.0, 26.4, 26.0, 22.9, 13.8, 8.6.

1-Ethyl-3,3-dipropylyndolin-2-one (2v)



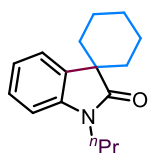
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 25.9 mg, 56% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 7.26 (t, $J = 7.4$ Hz, 1H), 7.14 (d, $J = 7.2$ Hz, 1H), 7.06 (t, $J = 7.4$ Hz, 1H), 6.82 (d, $J = 7.7$ Hz, 1H), 3.20 (s, 3H), 1.92–1.80 (m, 2H), 1.71 (td, $J = 12.5$, 4.0 Hz, 2H), 1.07–0.92 (m, 2H), 0.88–0.79 (m, 2H), 0.76 (t, $J = 6.7$ Hz, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 180.4, 144.0, 132.8, 127.5, 122.6, 122.3, 107.6, 53.3, 40.3, 25.9, 17.5, 14.2.

1'-Ethylspiro[cyclohexane-1,3'-indolin]-2'-one (2w)



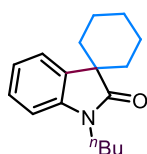
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Yellow oil; 30.9 mg, 67% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.46 (d, $J = 7.4$ Hz, 1H), 7.26 (t, $J = 7.7$ Hz, 1H), 7.03 (t, $J = 7.6, 0.7$ Hz, 1H), 6.87 (d, $J = 7.8$ Hz, 1H), 3.75 (q, $J = 7.2$ Hz, 2H), 2.01–1.89 (m, 2H), 1.88–1.80 (m, 2H), 1.80–1.68 (m, 3H), 1.68–1.60 (m, 1H), 1.59–1.51 (m, 2H), 1.25 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 180.3, 141.9, 135.7, 127.4, 124.1, 121.7, 108.0, 47.3, 34.4, 33.0, 25.2, 21.2, 12.7; HRMS (ESI) for $\text{C}_{15}\text{H}_{20}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 230.1539, found 230.1538.

1'-Propylspiro[cyclohexane-1,3'-indolin]-2'-one (2x)



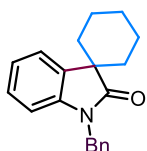
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 37.9 mg, 77% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.46 (d, $J = 7.3$ Hz, 1H), 7.25 (td, $J = 7.7, 1.1$ Hz, 1H), 7.03 (t, $J = 7.6, 0.9$ Hz, 1H), 6.86 (d, $J = 7.8$ Hz, 1H), 3.72–3.61 (m, 2H), 2.00–1.89 (m, 2H), 1.89–1.80 (m, 2H), 1.80–1.66 (m, 5H), 1.66–1.60 (m, 1H), 1.60–1.50 (m, 2H), 0.94 (t, $J = 7.4$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 180.7, 142.3, 135.6, 127.3, 124.0, 121.6, 108.2, 47.3, 41.2, 33.1, 25.2, 21.2, 20.7, 11.3; HRMS (ESI) for $\text{C}_{16}\text{H}_{22}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 244.1696, found 244.1694.

1'-Butylspiro[cyclohexane-1,3'-indolin]-2'-one (2y)



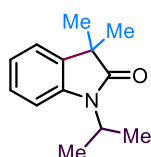
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 37.7 mg, 73% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.46 (d, $J = 7.4$ Hz, 1H), 7.25 (td, $J = 7.7, 1.0$ Hz, 1H), 7.02 (t, $J = 7.5$ Hz, 1H), 6.86 (d, $J = 7.8$ Hz, 1H), 3.70 (t, $J = 7.3$ Hz, 2H), 2.03–1.89 (m, 2H), 1.89–1.80 (m, 2H), 1.80–1.69 (m, 3H), 1.69–1.60 (m, 3H), 1.60–1.51 (m, 2H), 1.43–1.30 (m, 2H), 0.94 (t, $J = 7.4$ Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 180.6, 142.2, 135.6, 127.3, 124.0, 121.6, 108.2, 47.3, 39.5, 33.1, 29.5, 25.2, 21.2, 20.1, 13.8; HRMS (ESI) for $\text{C}_{17}\text{H}_{24}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 258.1852, found 258.1850.

1'-Benzylspiro[cyclohexane-1,3'-indolin]-2'-one (2z)



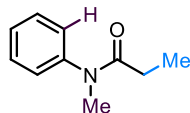
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 29.7 mg, 51% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.46 (d, J = 7.3 Hz, 1H), 7.32–7.19 (m, 5H), 7.14 (t, J = 7.7, 1.0 Hz, 1H), 7.00 (t, J = 7.9 Hz, 1H), 6.71 (d, J = 7.7 Hz, 1H), 4.90 (s, 2H), 2.05–1.86 (m, 4H), 1.84–1.68 (m, 4H), 1.68–1.58 (m, 2H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 180.8, 141.9, 136.2, 135.4, 128.8, 127.5, 127.4, 127.1, 123.9, 122.0, 109.0, 47.5, 43.4, 33.2, 25.3, 21.2; HRMS (ESI) for $\text{C}_{20}\text{H}_{22}\text{NO}$ $[\text{M}+\text{H}]^+$ calcd. 292.1696, found 292.1694.

1-Isopropyl-3,3-dimethylindolin-2-one (2aa)



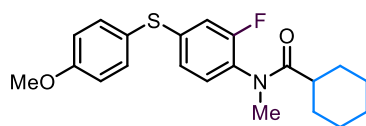
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 30.6 mg, 75% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.24–7.17 (m, 2H), 7.03 (t, J = 7.6 Hz, 2H), 4.73–4.58 (m, 1H), 1.48 (d, J = 7.0 Hz, 6H), 1.35 (s, 6H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 181.1, 141.2, 136.4, 127.3, 122.5, 121.9, 109.9, 43.9, 43.4, 24.5, 19.4.

N-Methyl-*N*-phenylpropionamide (2ab')



Purification by flash chromatography (*n*-hexane/ethyl acetate = 5/1); White solid; mp 50–52 °C; 8.8 mg, 27% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.42 (t, J = 7.5 Hz, 2H), 7.33 (t, J = 7.4 Hz, 1H), 7.19 (d, J = 7.4 Hz, 2H), 3.27 (s, 3H), 2.09 (q, J = 7.5 Hz, 2H), 1.05 (t, J = 7.5 Hz, 3H); $^{13}\text{C NMR}$ (100 MHz, CDCl_3) δ (ppm) = 174.0, 144.3, 129.7, 127.7, 127.3, 37.3, 27.5, 9.7.

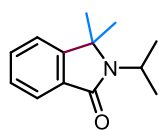
N-(2-Fluoro-4-((4-methoxyphenyl)thio)phenyl)-*N*-methylcyclohexanecarboxamide (2ac')



Purification by flash chromatography (*n*-hexane/ethyl acetate = 3/1); Colorless oil; 13.4 mg, 18% yield; $^1\text{H NMR}$ (400 MHz, CDCl_3) δ (ppm) = 7.50 (d, J = 8.8 Hz, 2H), 7.04 (t, J = 8.1 Hz, 1H), 6.98 (d, J = 8.8 Hz, 2H), 6.88 (dd, J = 10.3, 2.0 Hz, 1H), 6.81 (dd, J = 10.4, 2.1 Hz, 1H), 3.86 (s, 3H), 3.14 (s, 3H), 2.17–2.04 (m, 1H), 1.73–

1.57 (m, 4H), 1.56–1.42 (m, 3H), 1.24–1.10 (m, 1H), 1.09–0.93 (m, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 176.8, 160.8, 158.2 ($J = 251.7$ Hz), 142.3 (d, $J = 7.5$ Hz), 136.9, 129.6, 128.5 ($J = 13.9$ Hz), 122.6 ($J = 3.6$ Hz), 121.4, 115.5, 114.5 ($J = 23.0$ Hz), 55.5, 41.4, 36.6, 29.6, 29.1, 25.6, 25.5, 25.5; HRMS (ESI) for $\text{C}_{21}\text{H}_{25}\text{FNO}_2\text{S}$ $[\text{M}+\text{H}]^+$ calcd. 374.1585, found 374.1585.

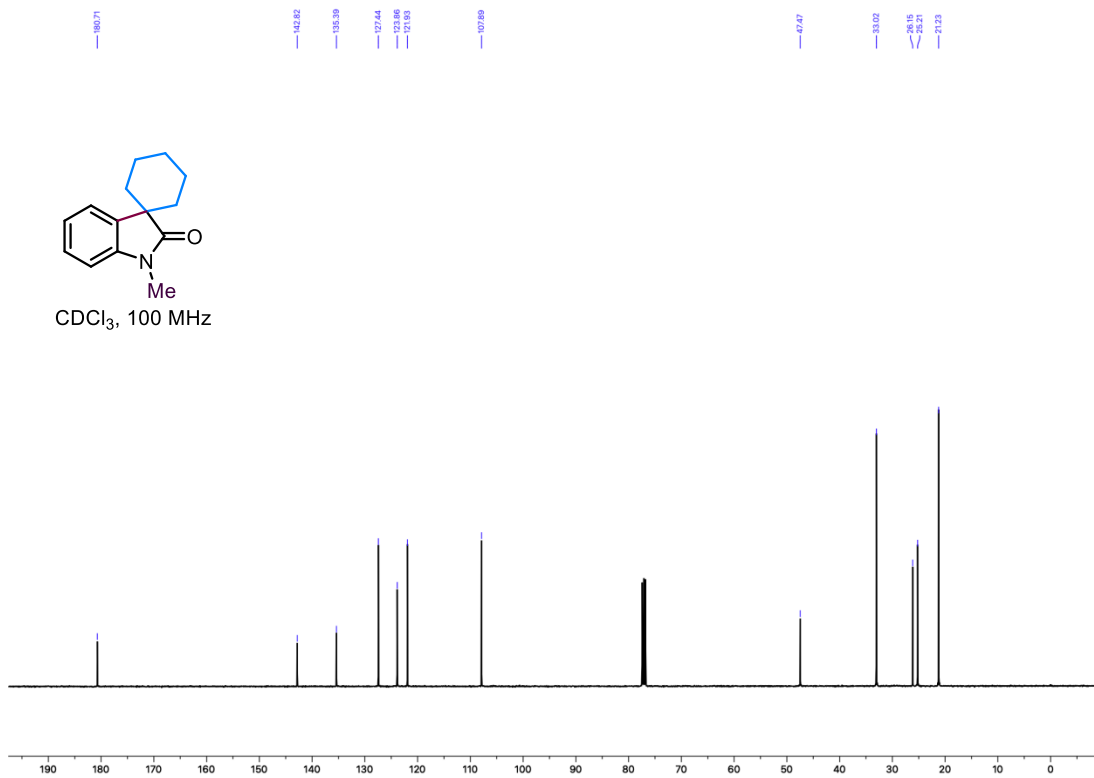
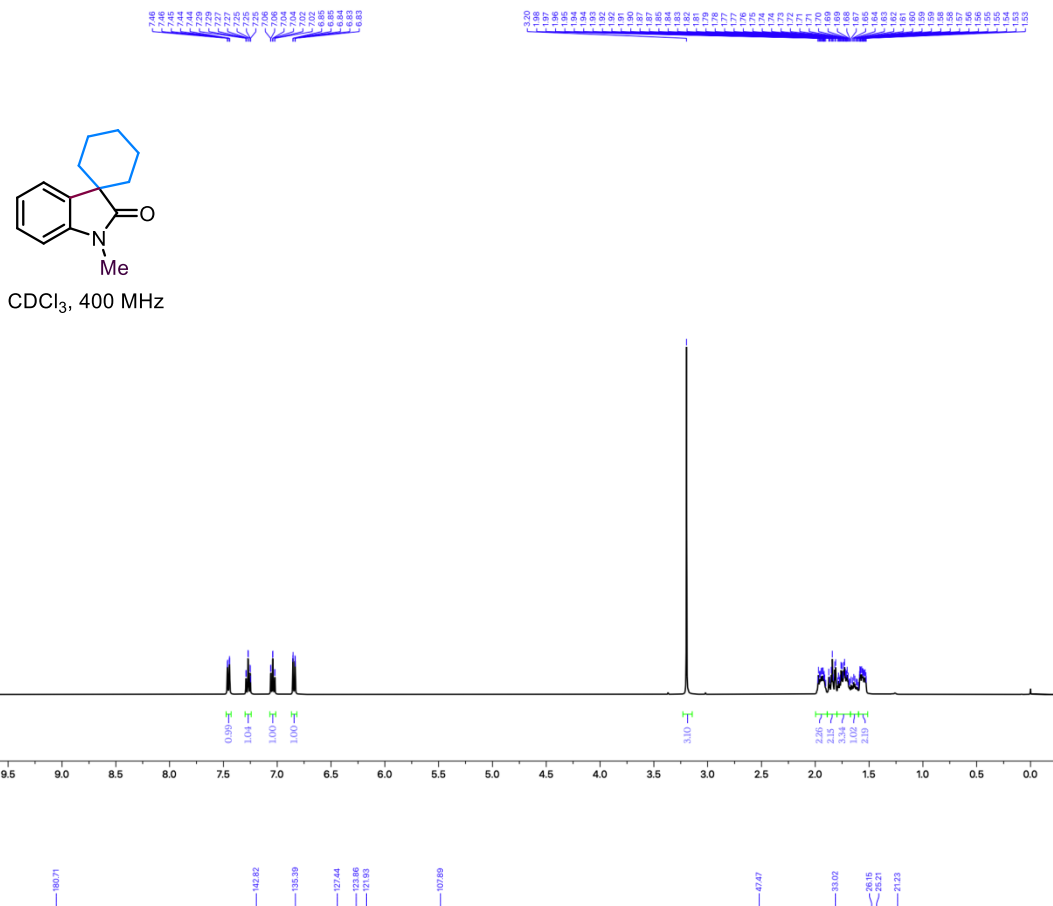
7-Fluoro-2-isopropyl-3,3-dimethylisoindolin-1-one (4)



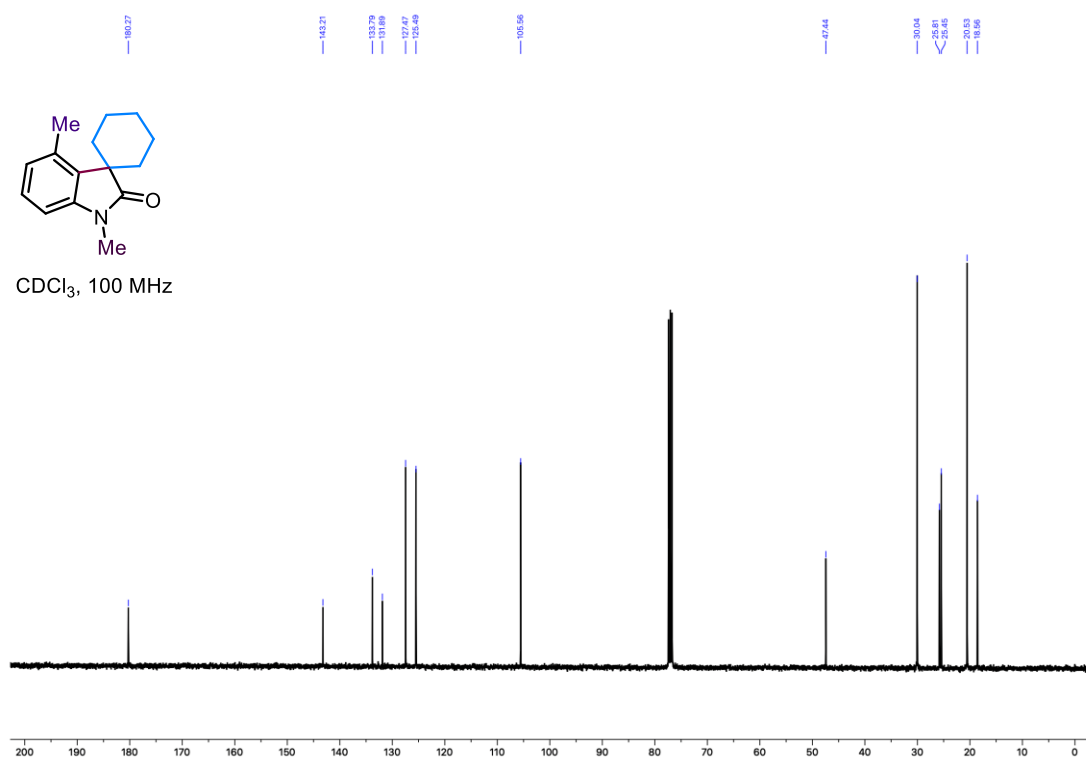
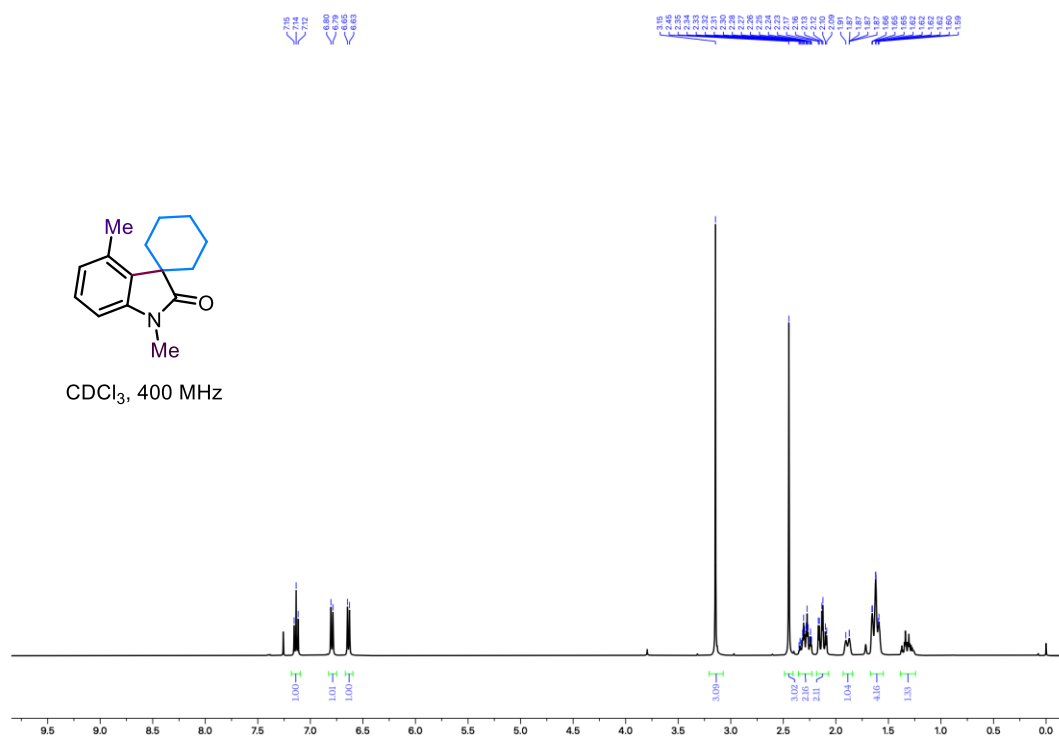
Purification by flash chromatography (*n*-hexane/ethyl acetate = 30/1); Colorless oil; 30.6 mg, 75% yield; ^1H NMR (400 MHz, CDCl_3) δ (ppm) = 7.77 (d, $J = 7.5$ Hz, 1H), 7.51 (td, $J = 7.5, 1.2$ Hz, 1H), 7.40 (t, $J = 7.4$ Hz, 1H), 7.34 (d, $J = 7.5$ Hz, 1H), 3.72–3.58 (m, 1H), 1.56 (d, $J = 6.9$ Hz, 6H), 1.48 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ (ppm) = 167.2, 151.3, 132.0, 131.2, 127.9, 123.2, 120.6, 63.3, 44.6, 25.5, 20.5.

9. NMR spectra of compounds

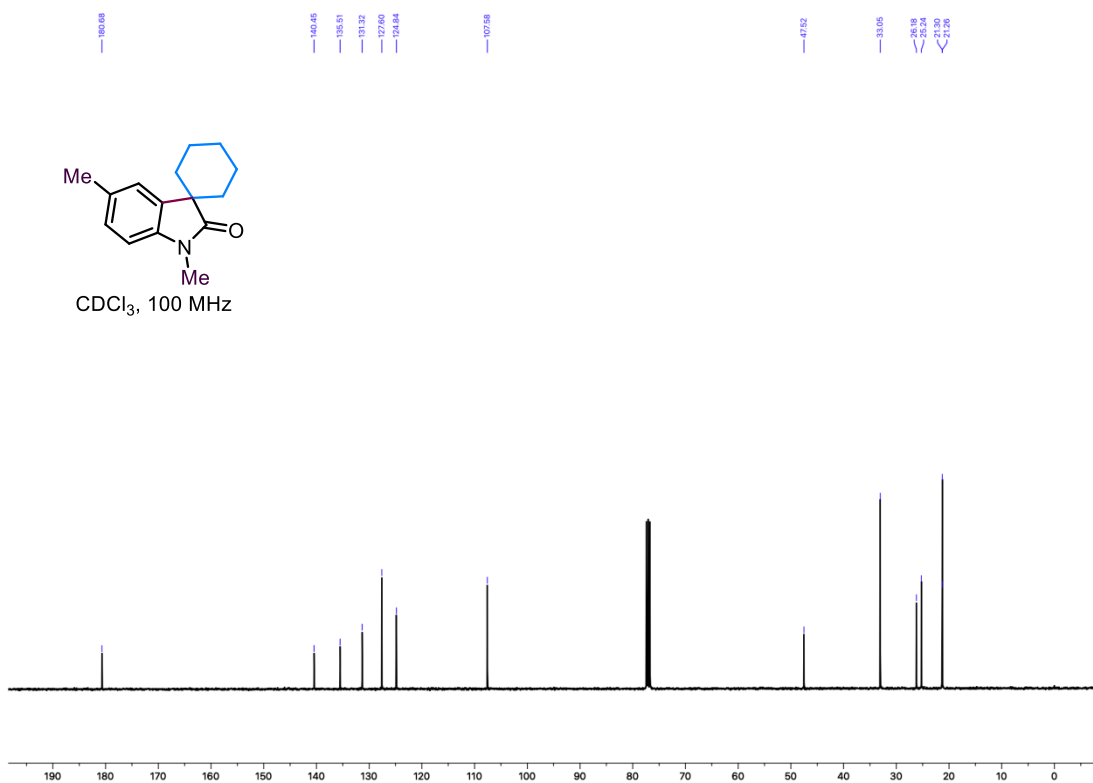
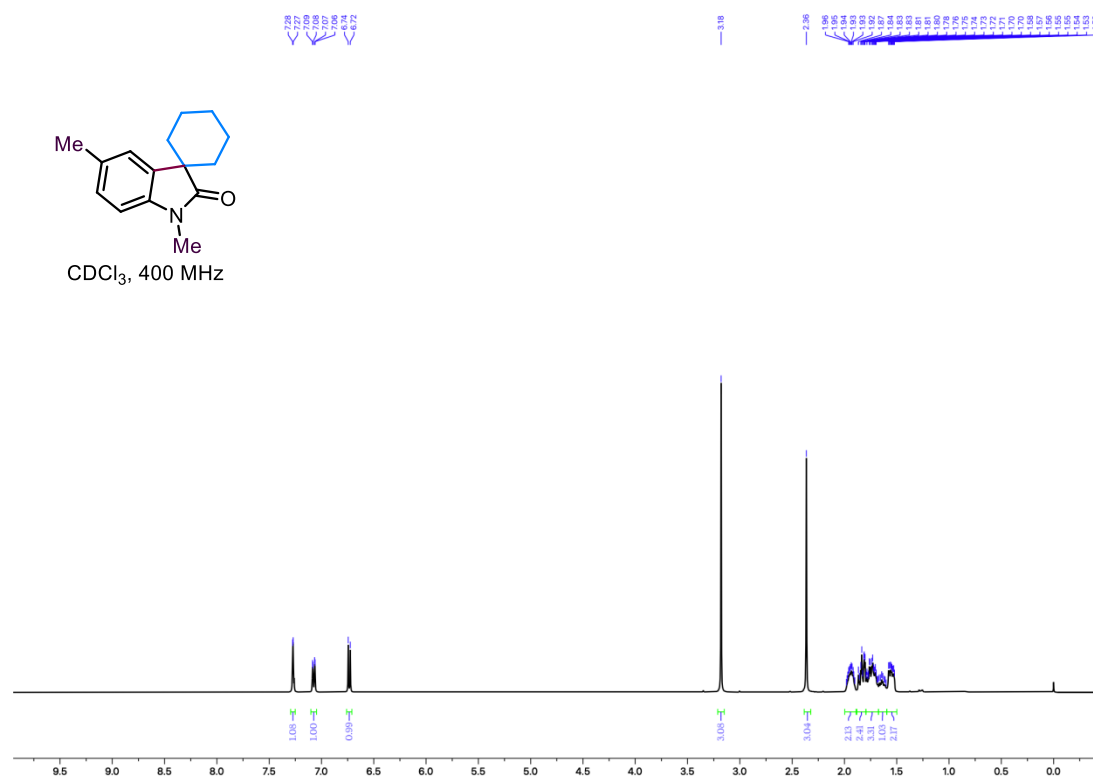
1'-Methylspiro[cyclohexane-1,3'-indolin]-2'-one (2a)



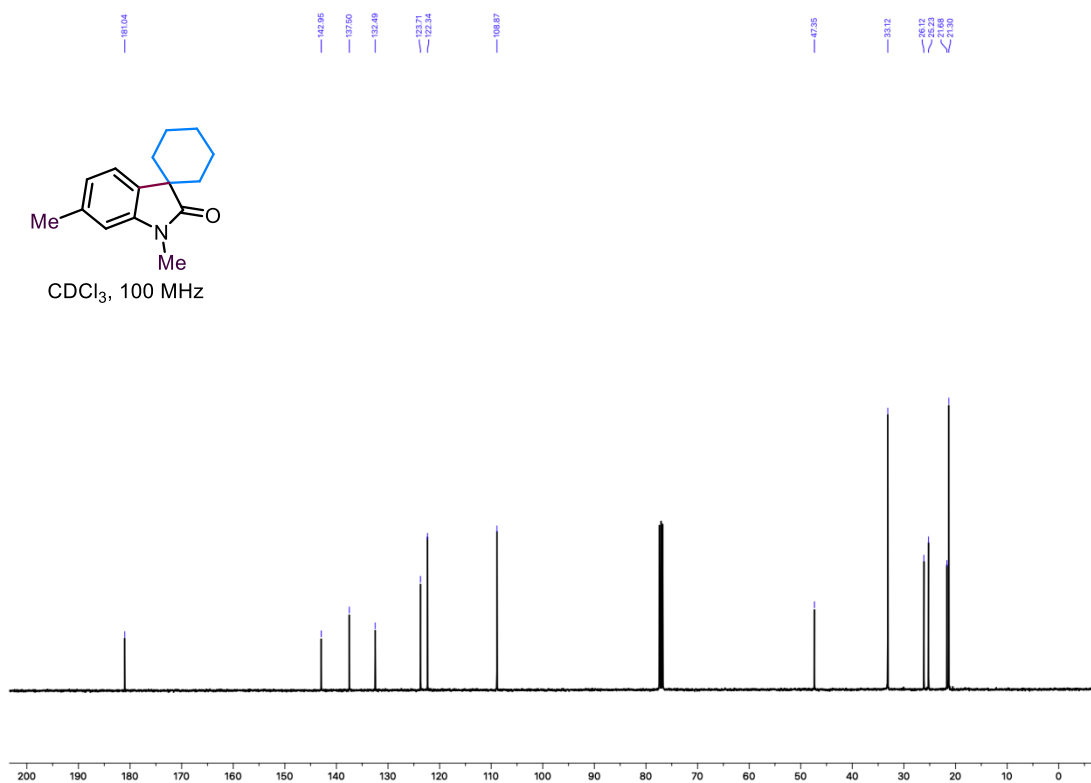
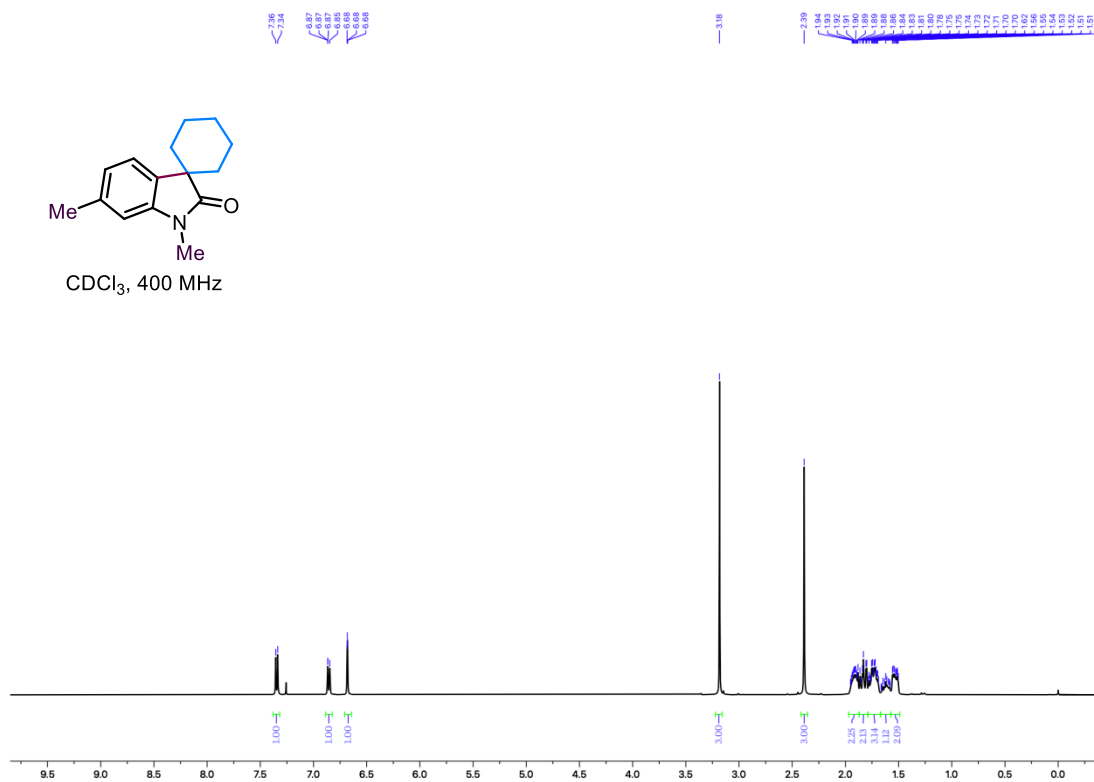
1',4'-Dimethylspiro[cyclohexane-1,3'-indolin]-2'-one (2b)



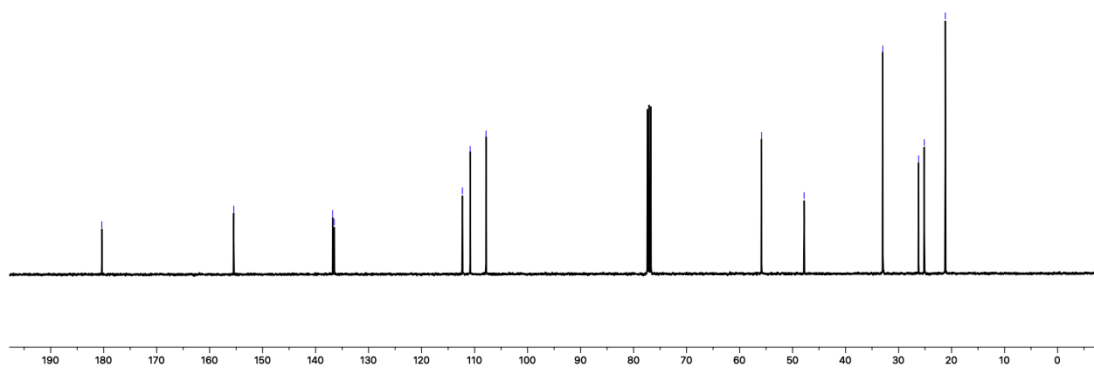
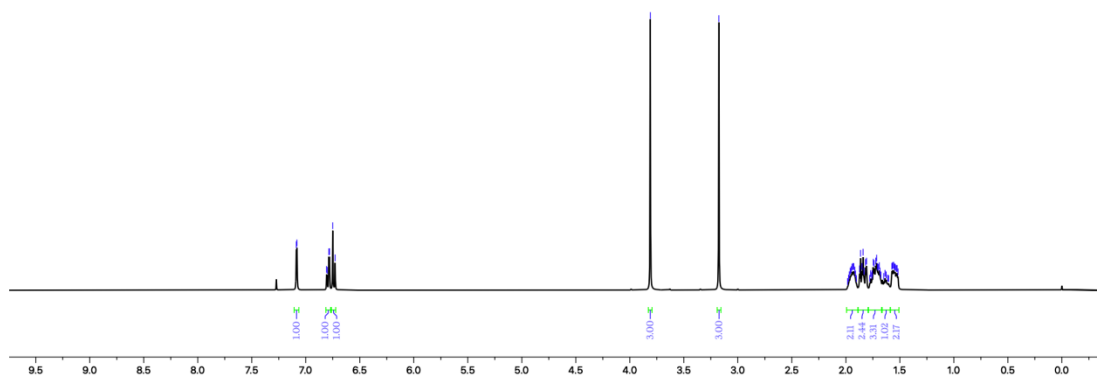
1',5'-Dimethylspiro[cyclohexane-1,3'-indolin]-2'-one (2c)



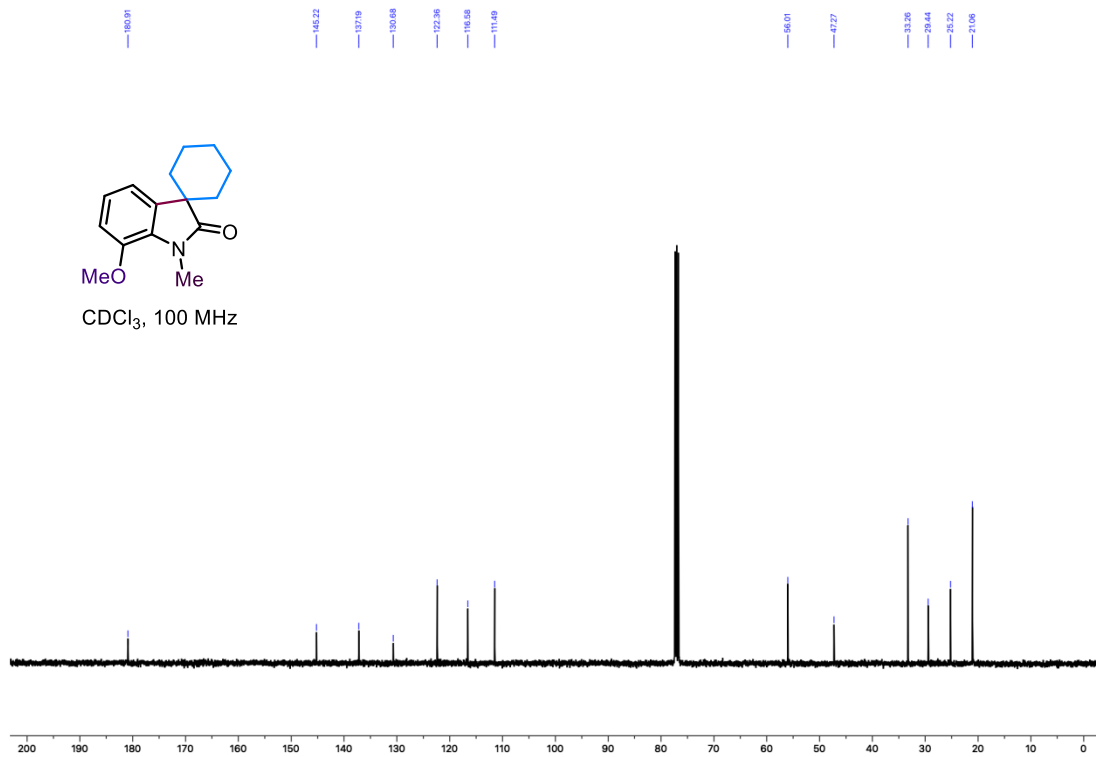
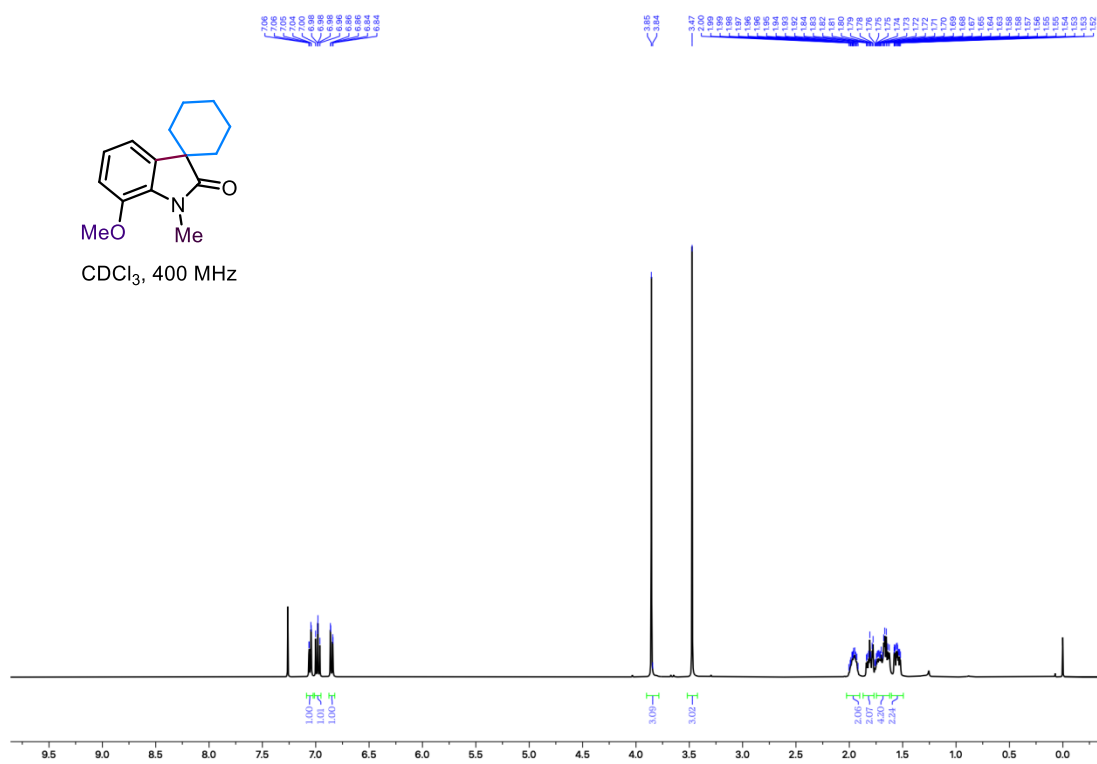
1',6'-Dimethylspiro[cyclohexane-1,3'-indolin]-2'-one (2d)



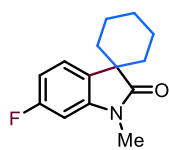
5'-Methoxy-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2e)



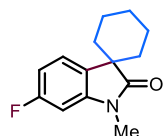
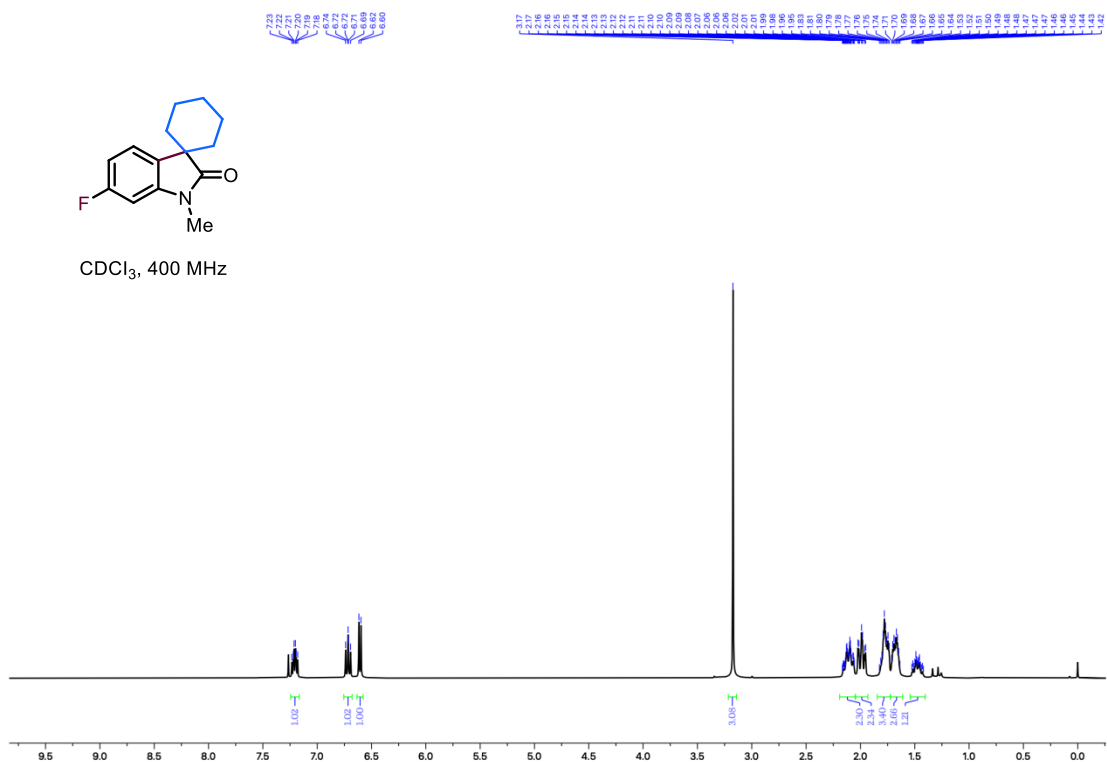
7'-Methoxy-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2f)



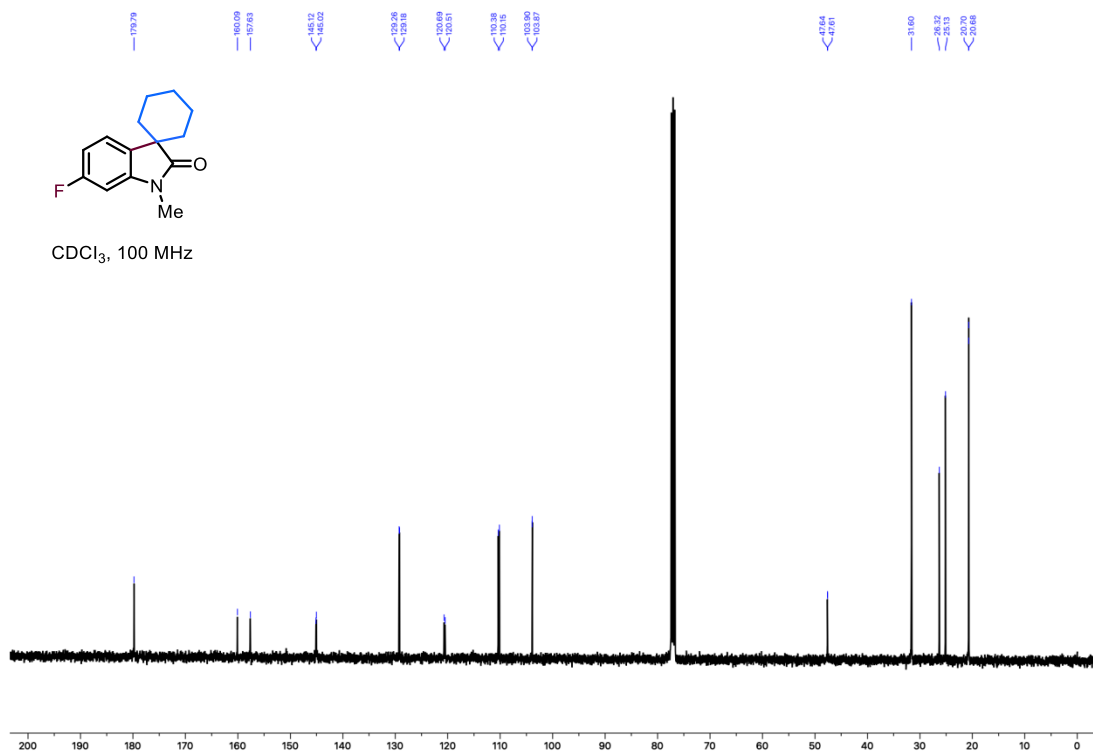
6'-Fluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2g)



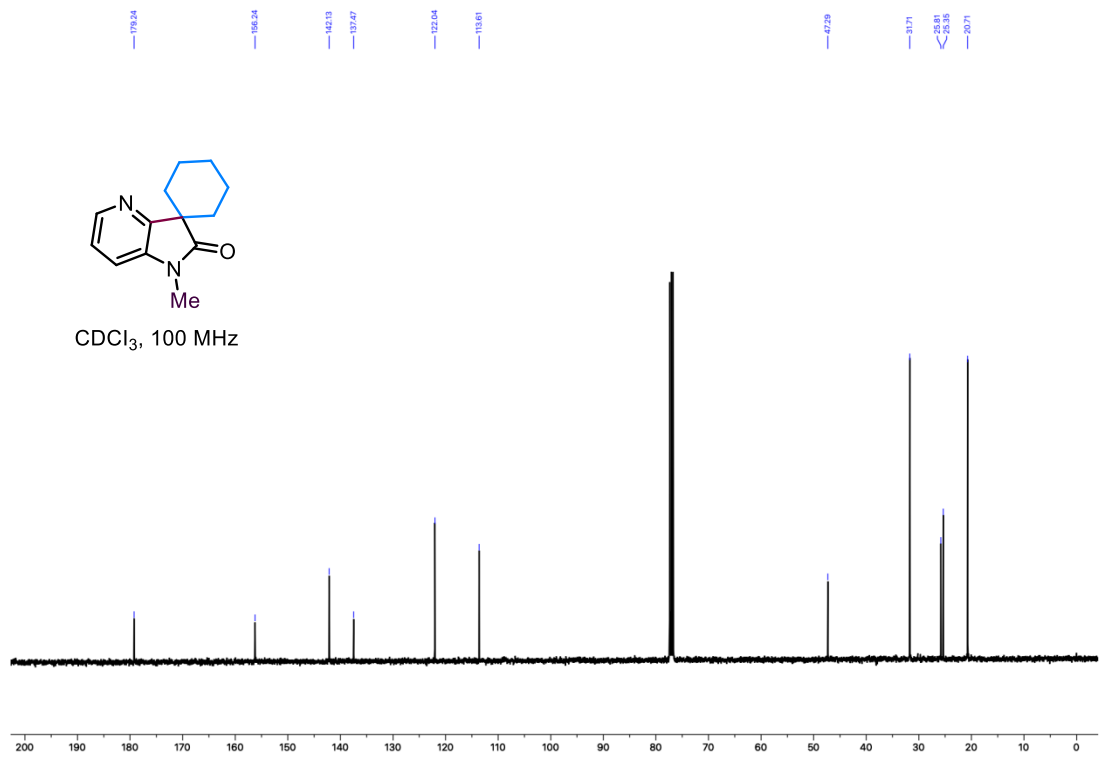
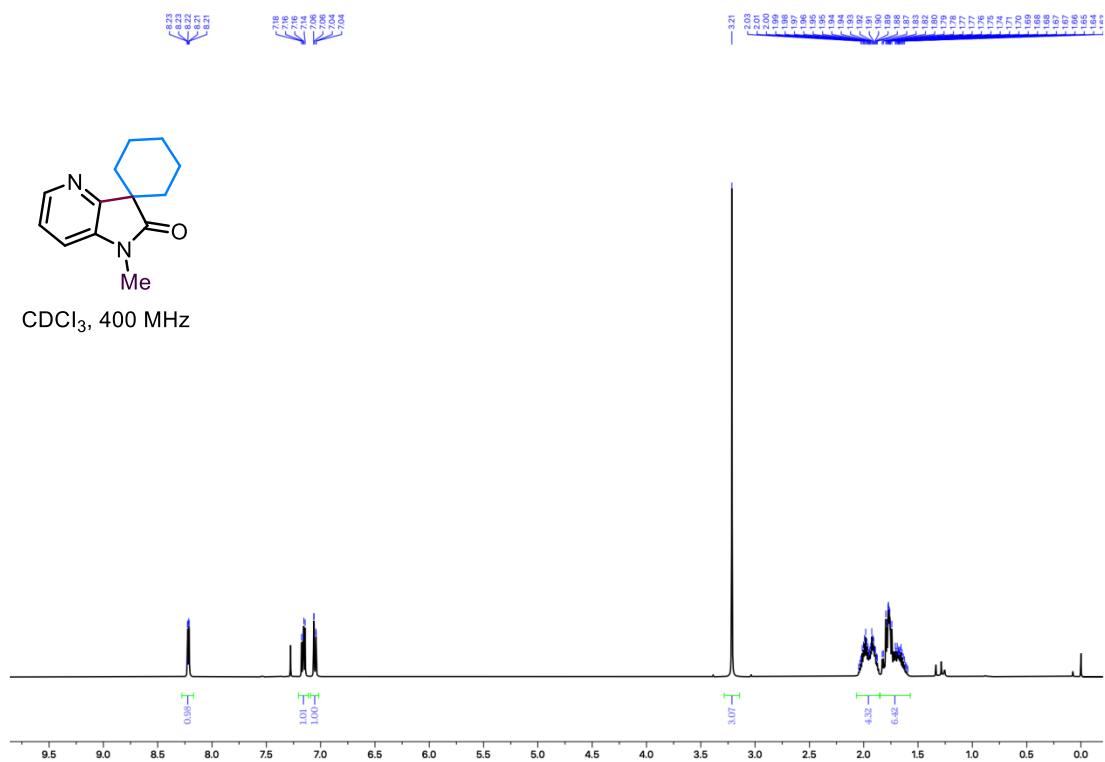
CDCl₃, 400 MHz



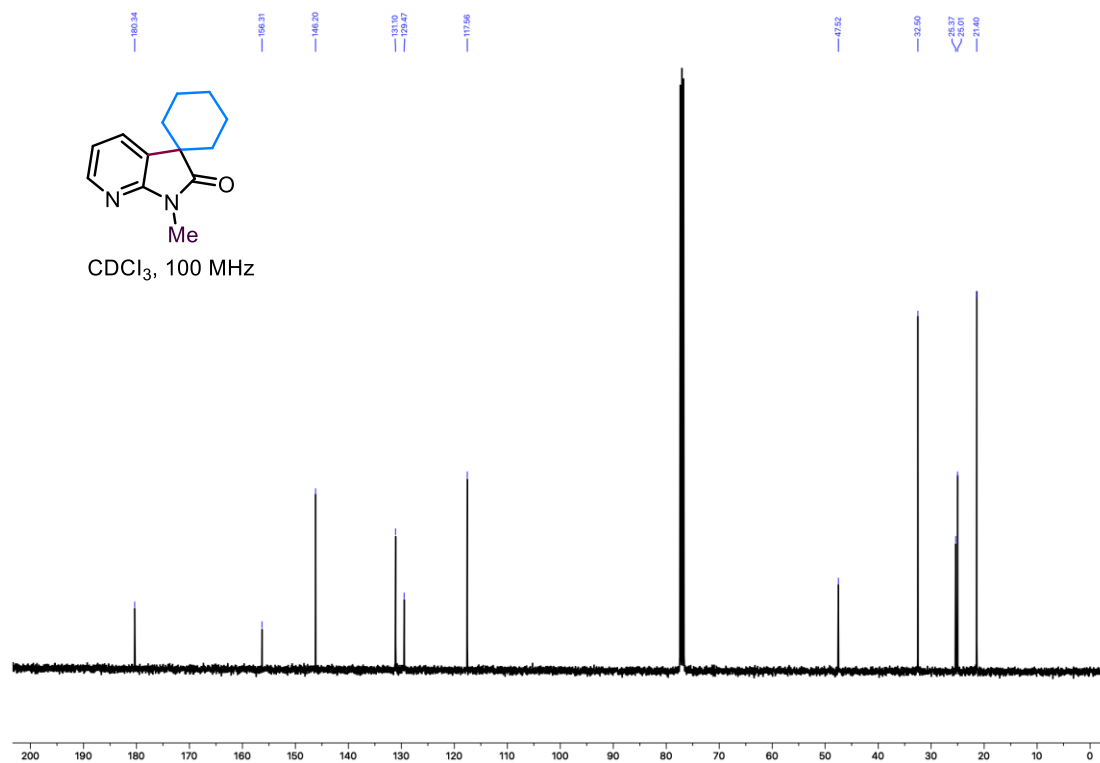
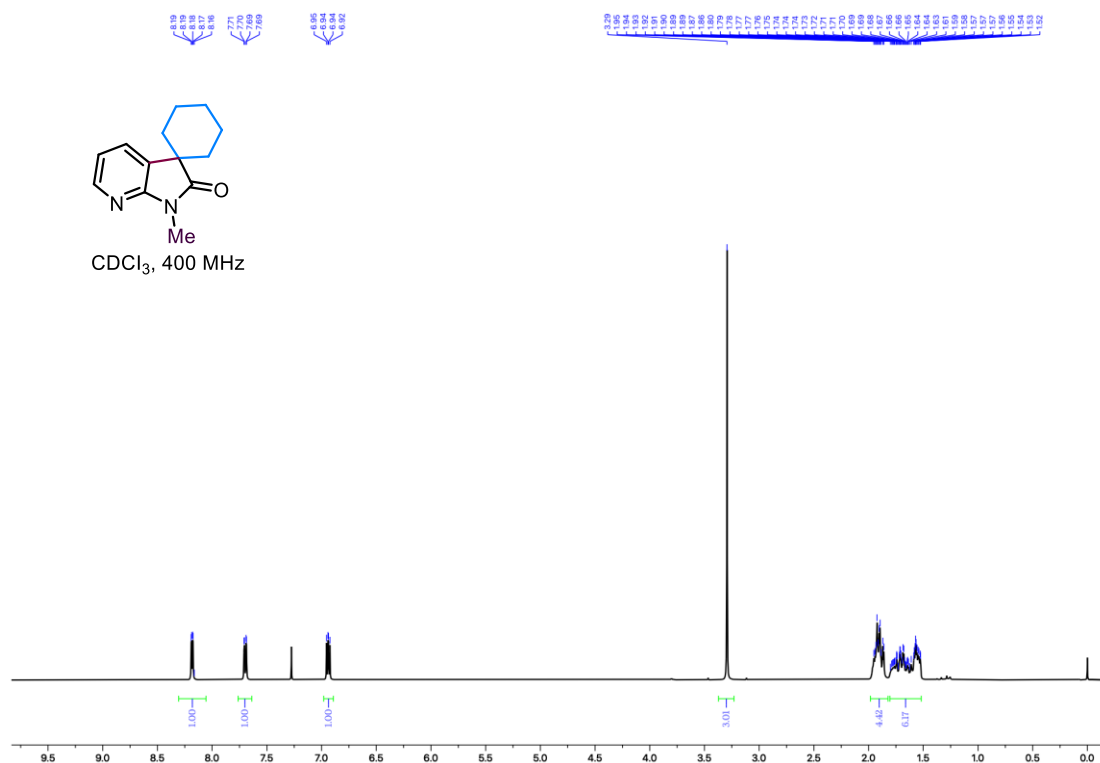
CDCl₃, 100 MHz



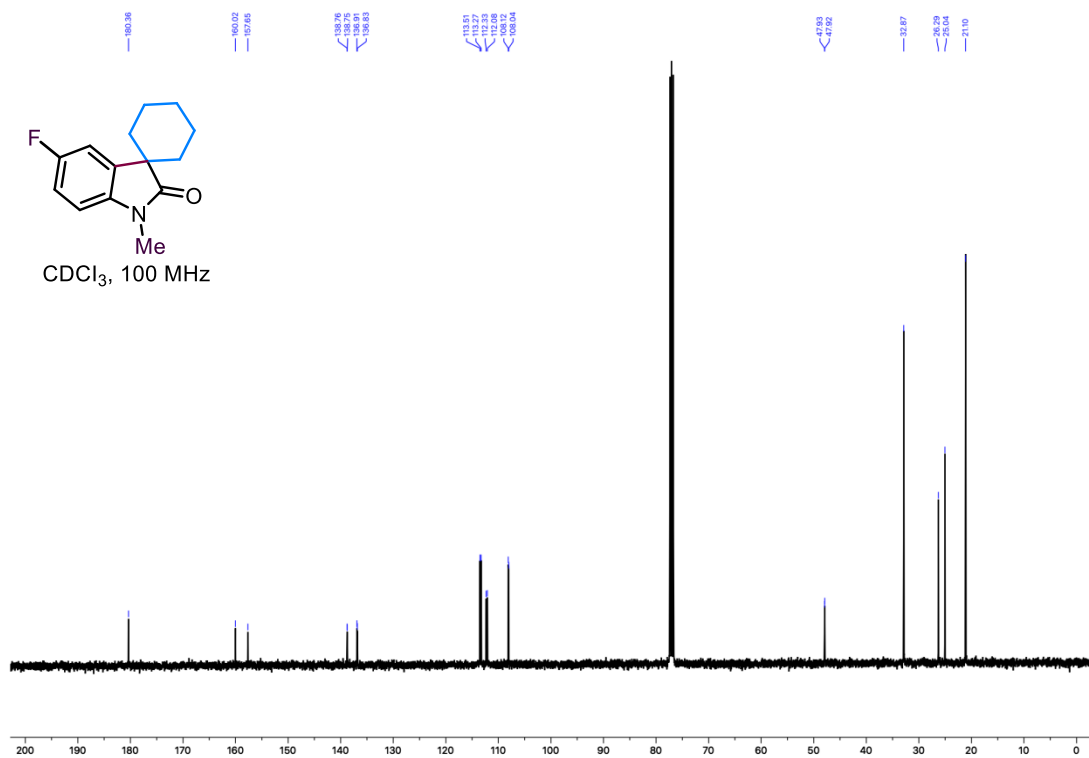
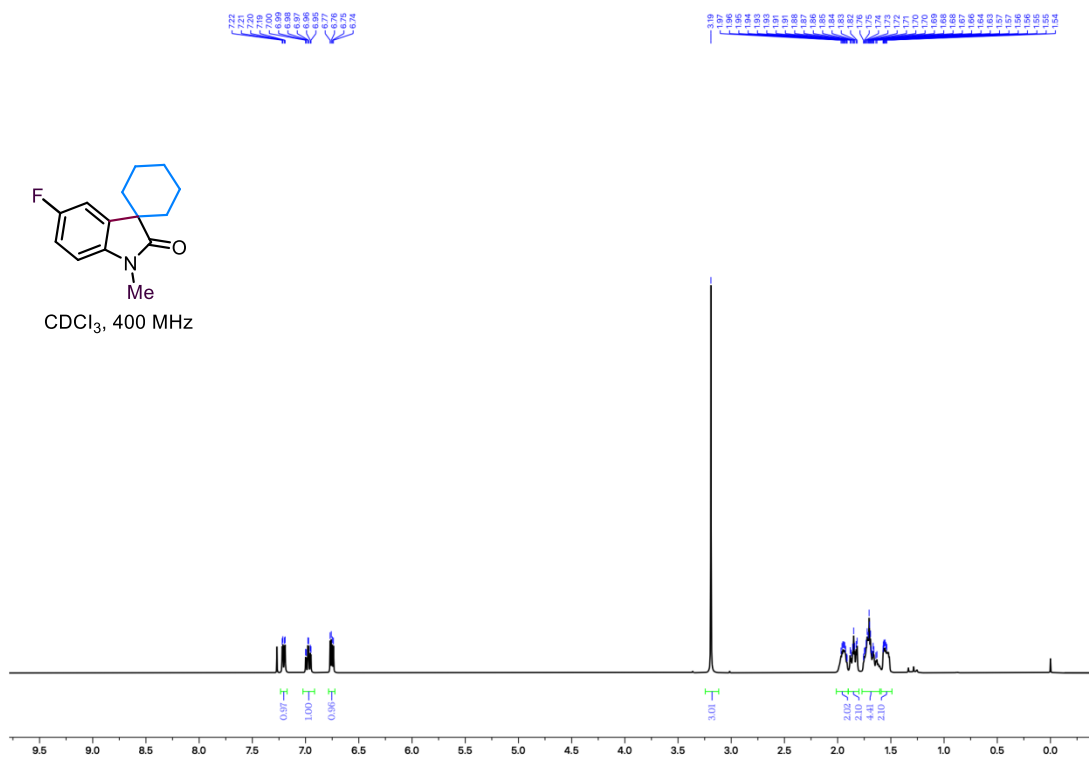
1'-Methylspiro[cyclohexane-1,3'-pyrrolo[3,2-b]pyridin]-2'(1'H)-one (2h)



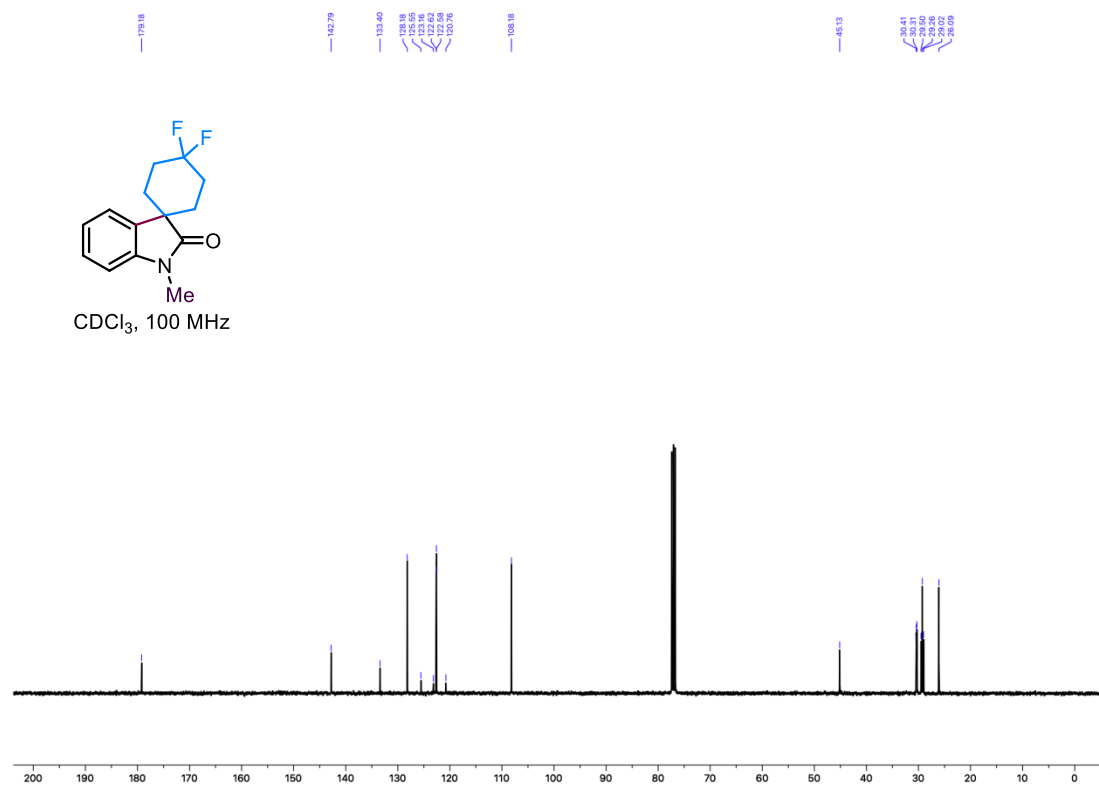
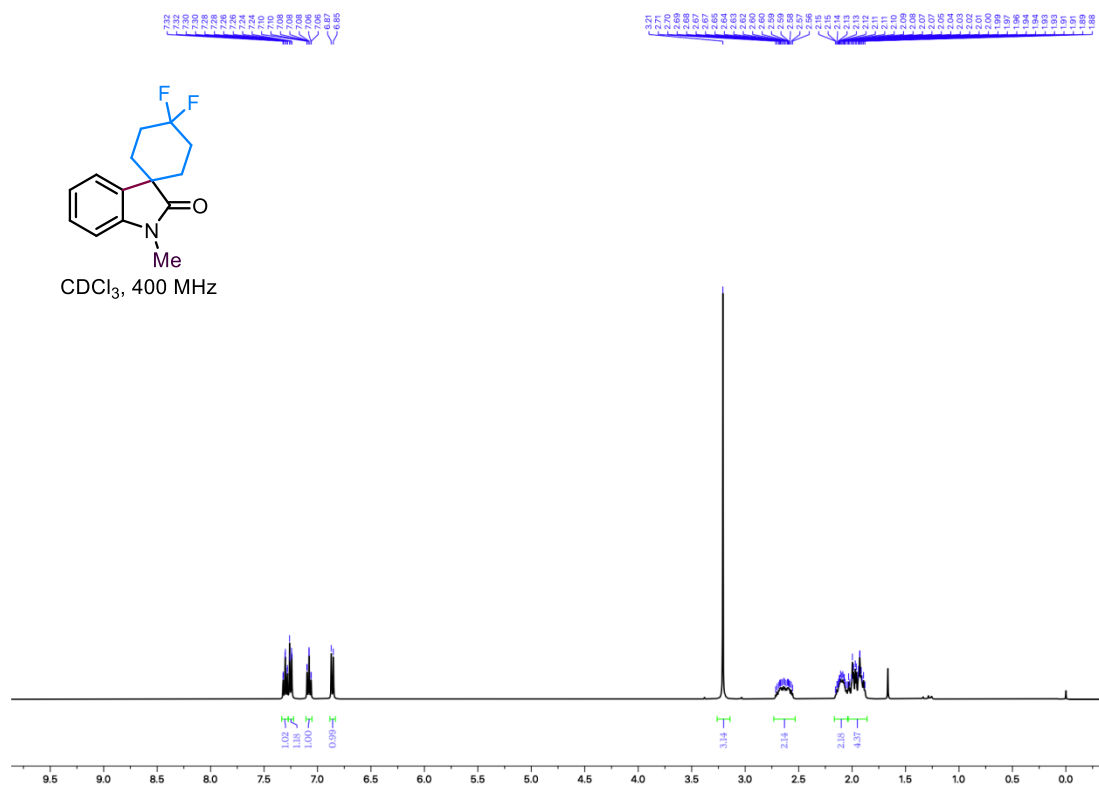
1'-Methylspiro[cyclohexane-1,3'-pyrrolo[2,3-b]pyridin]-2'(1'H)-one (2i)



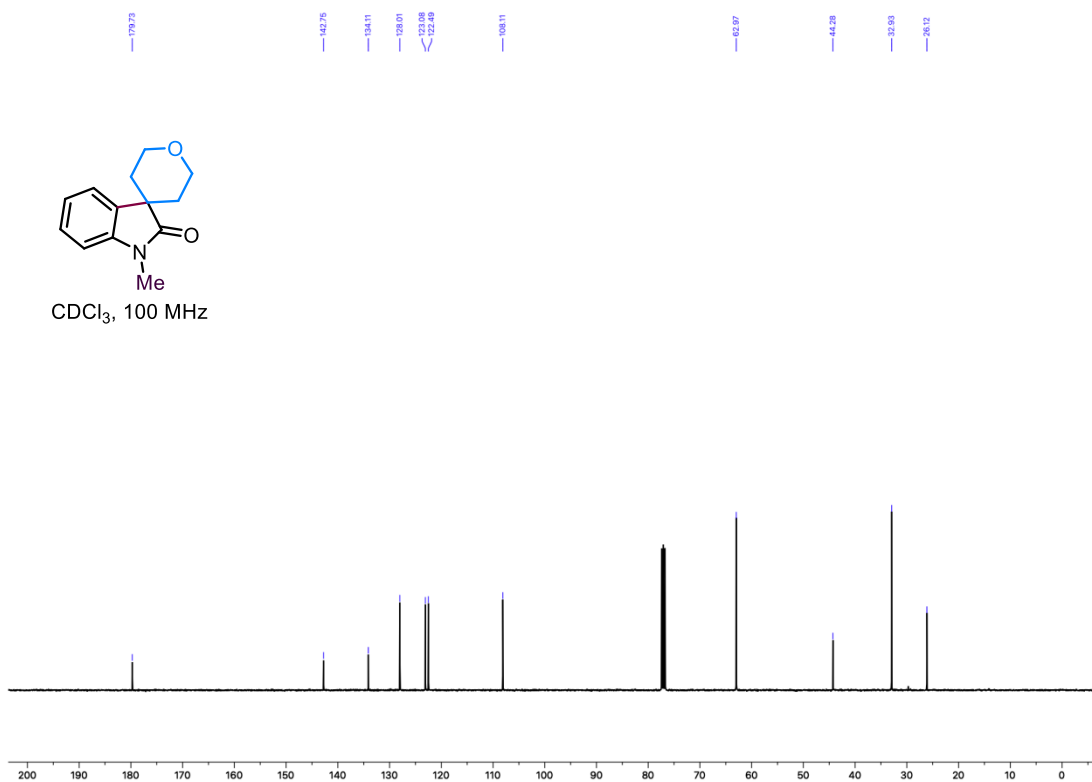
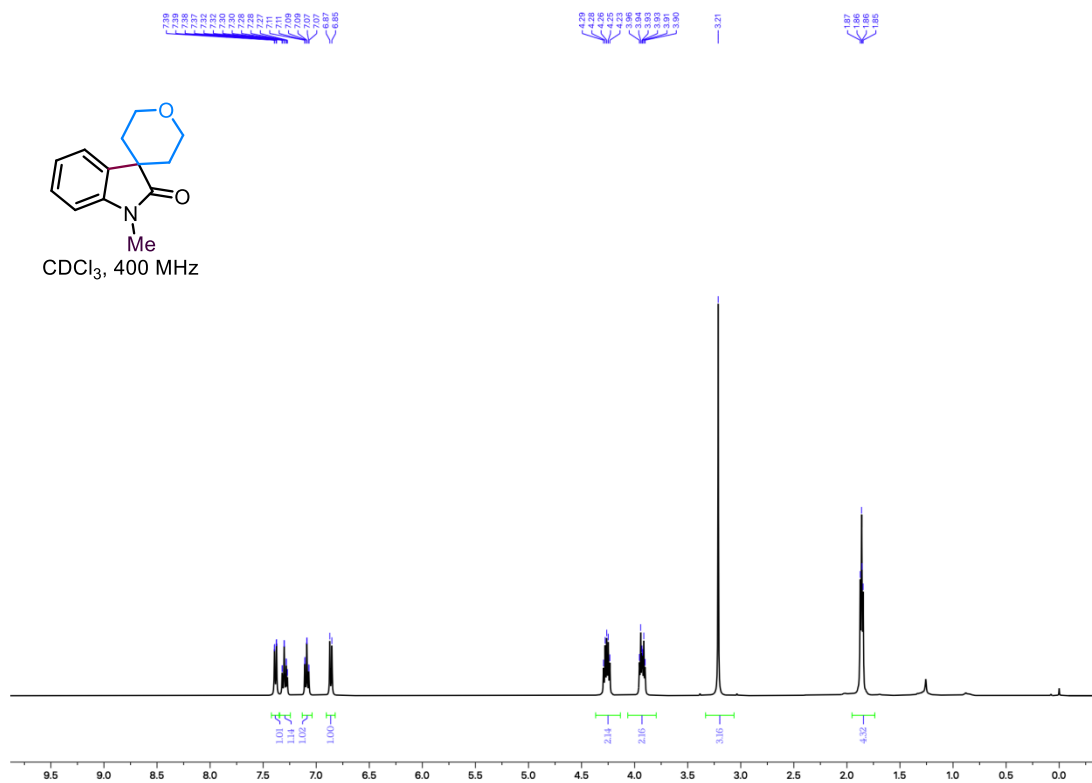
5'-Fluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2j)



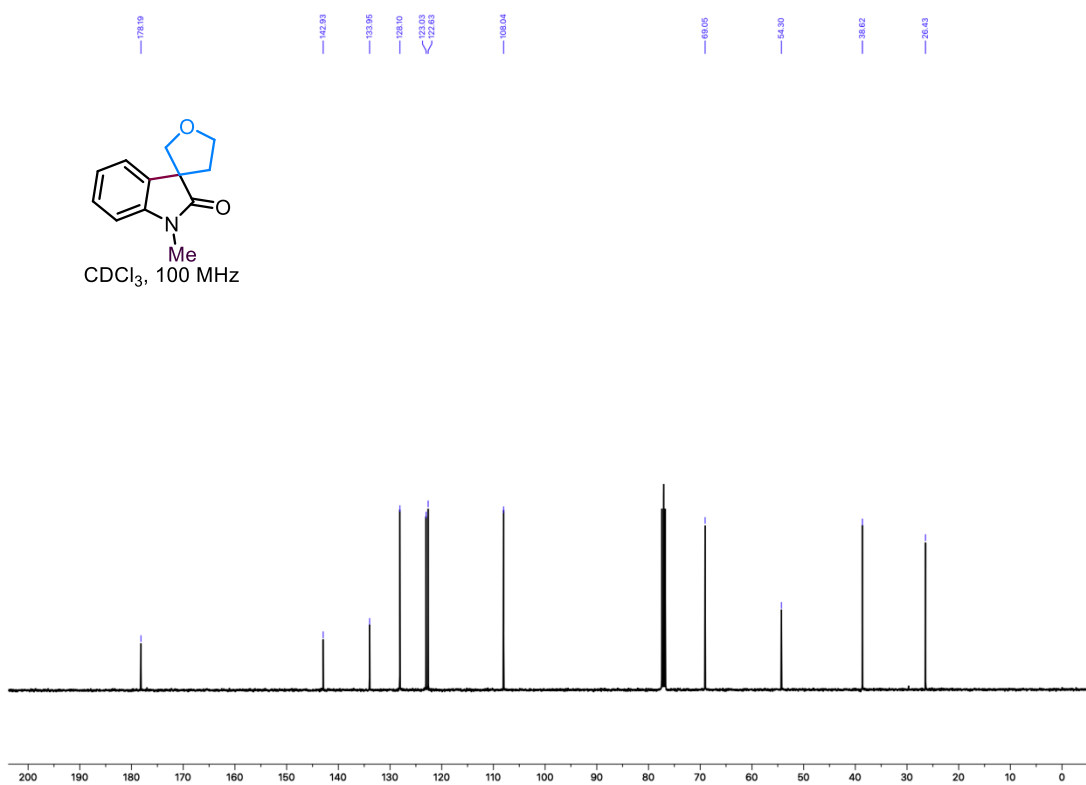
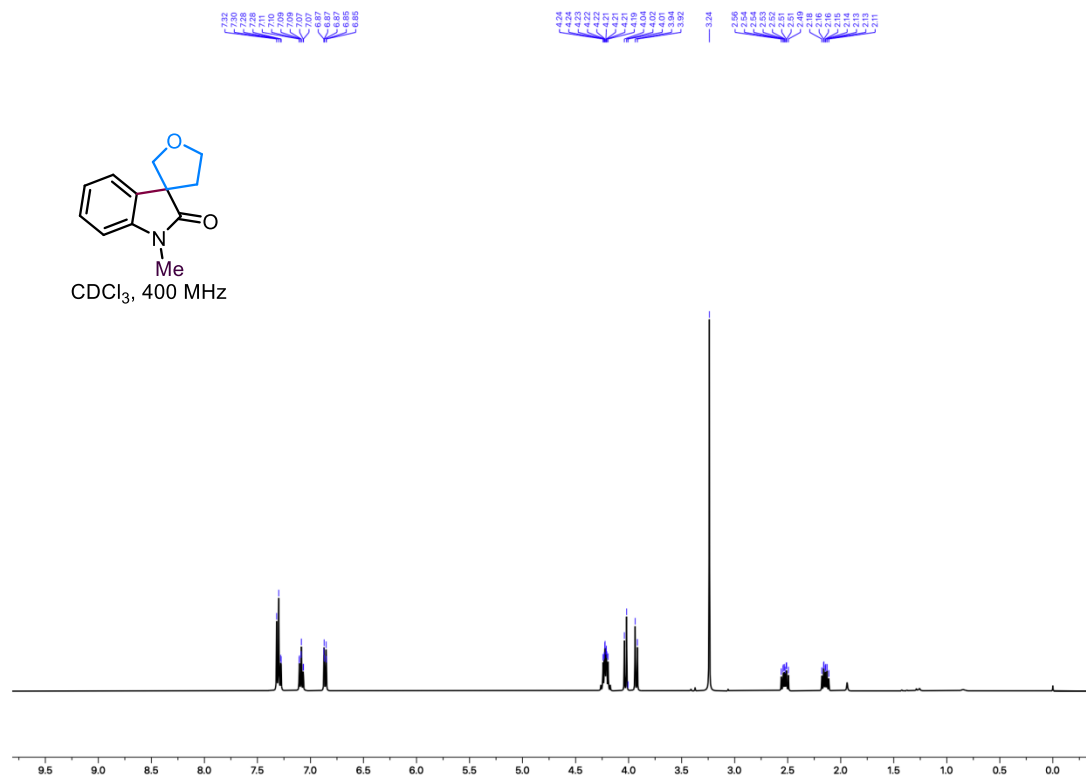
4,4-Difluoro-1'-methylspiro[cyclohexane-1,3'-indolin]-2'-one (2k)



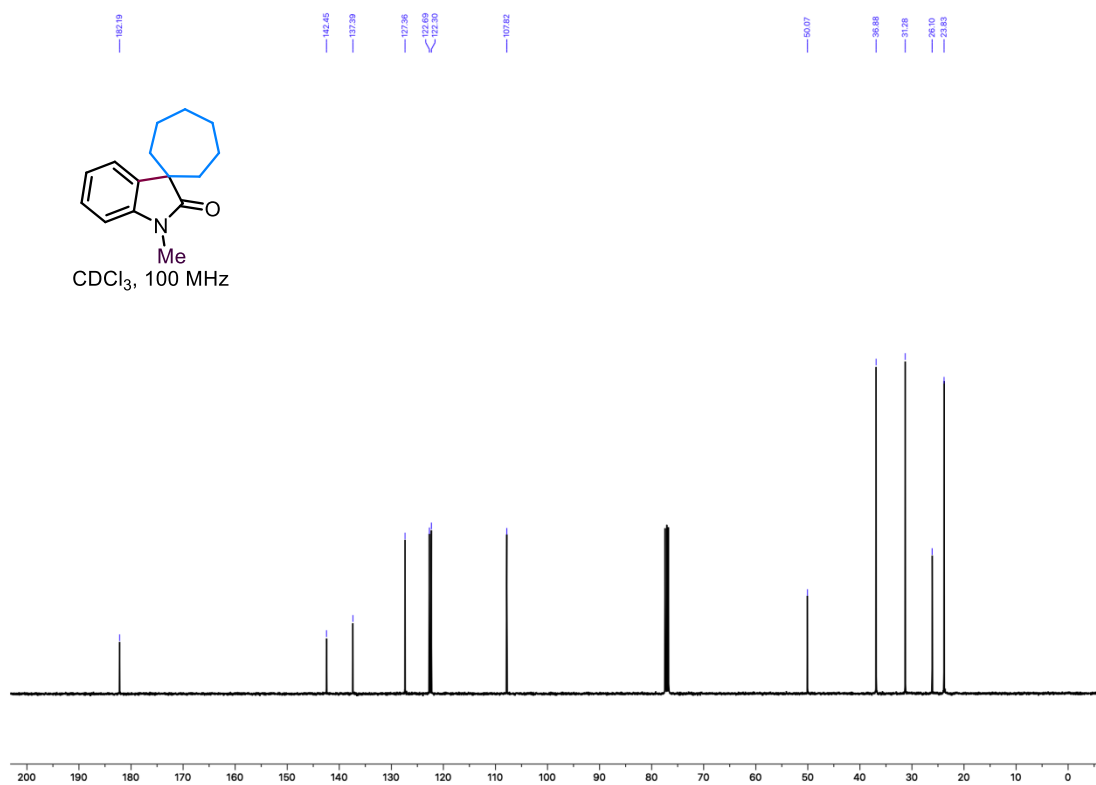
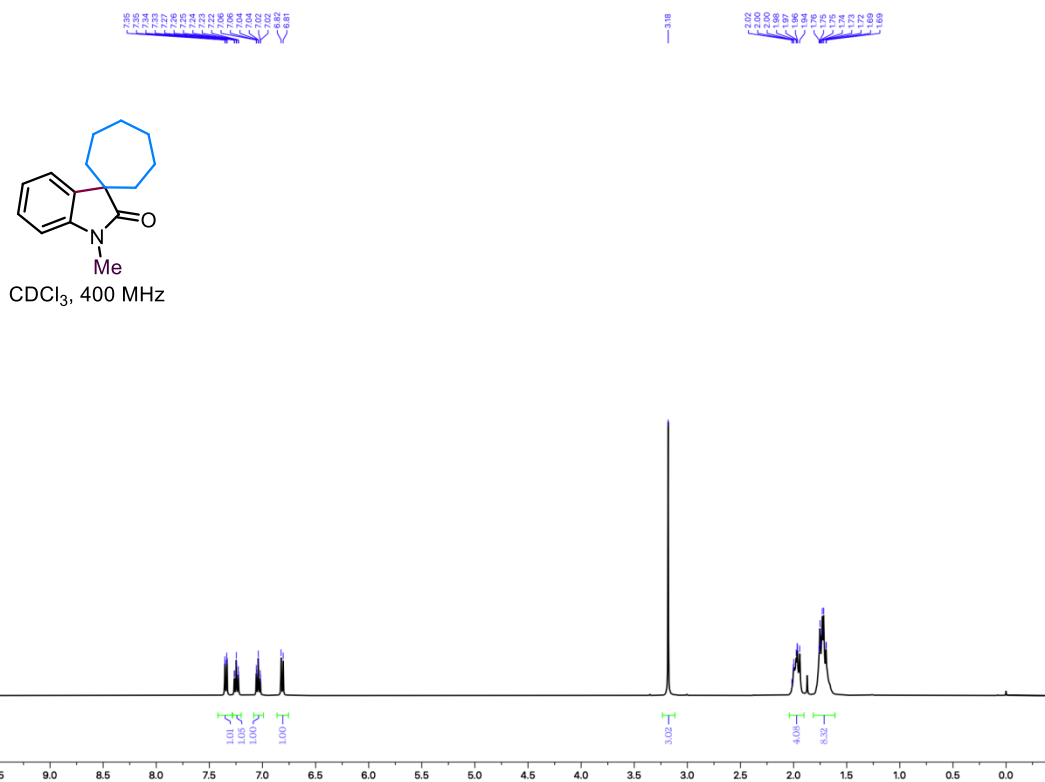
1-Methyl-2',3',5',6'-tetrahydrospiro[indoline-3,4'-pyran]-2-one (2l)



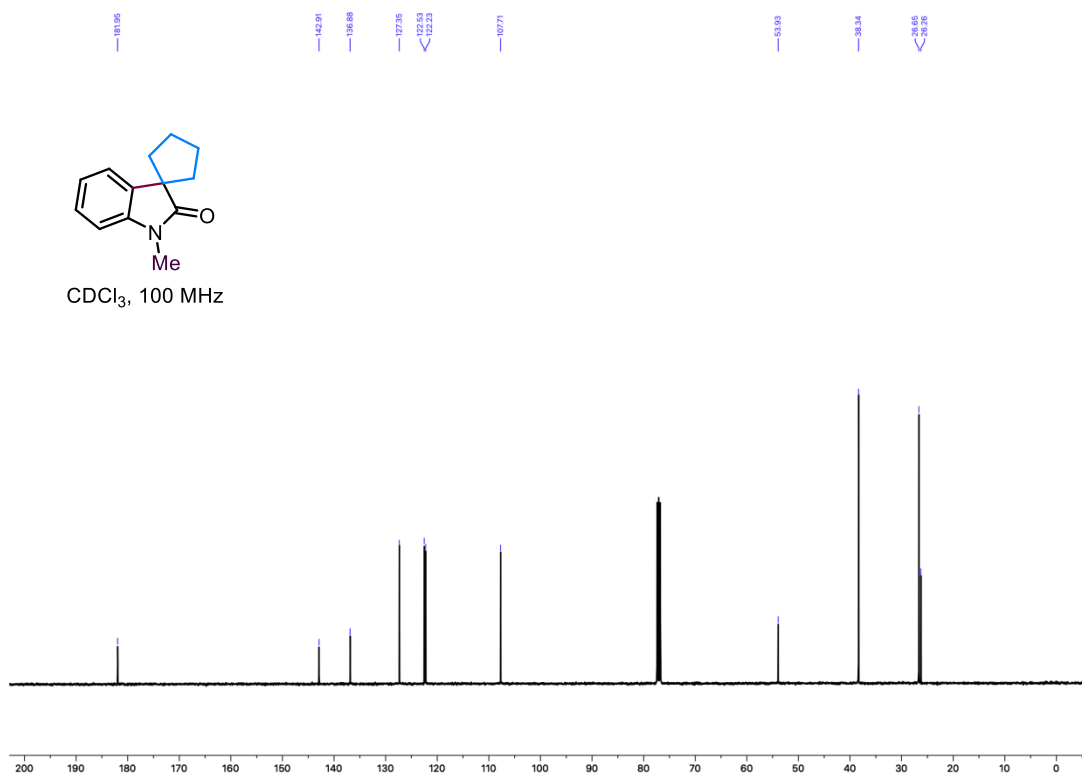
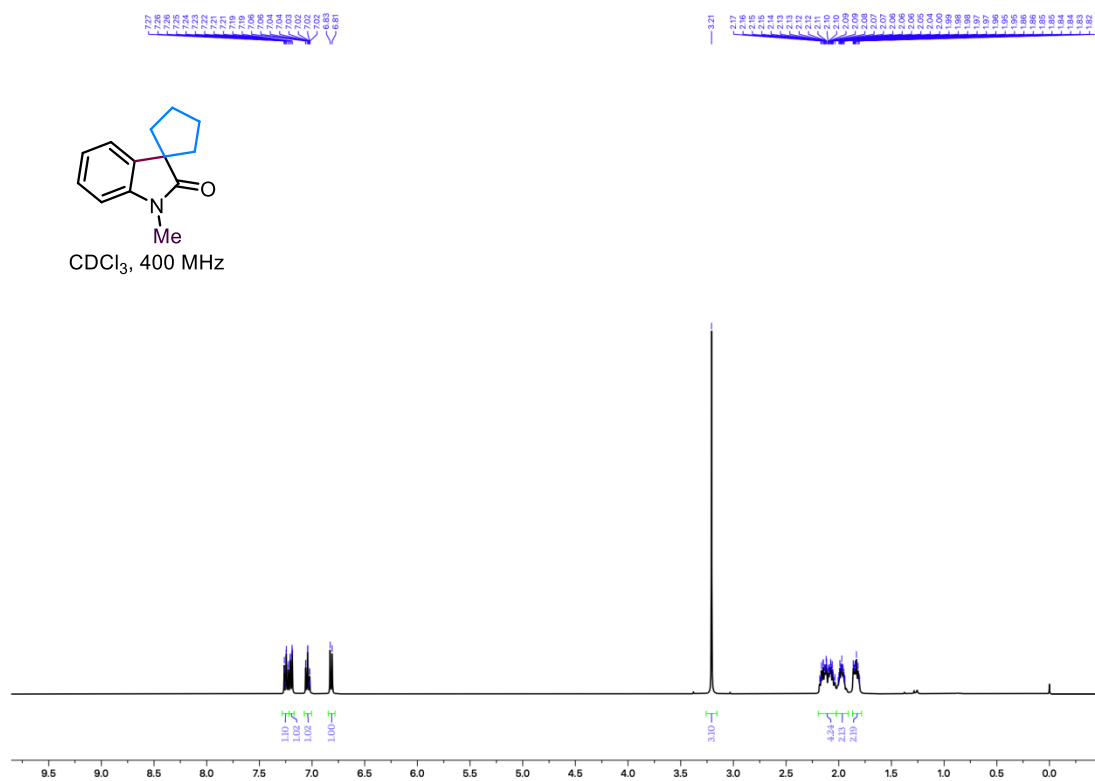
1'-Methyl-4,5-dihydro-2H-spiro[3.3]heptan-2-one (2m)



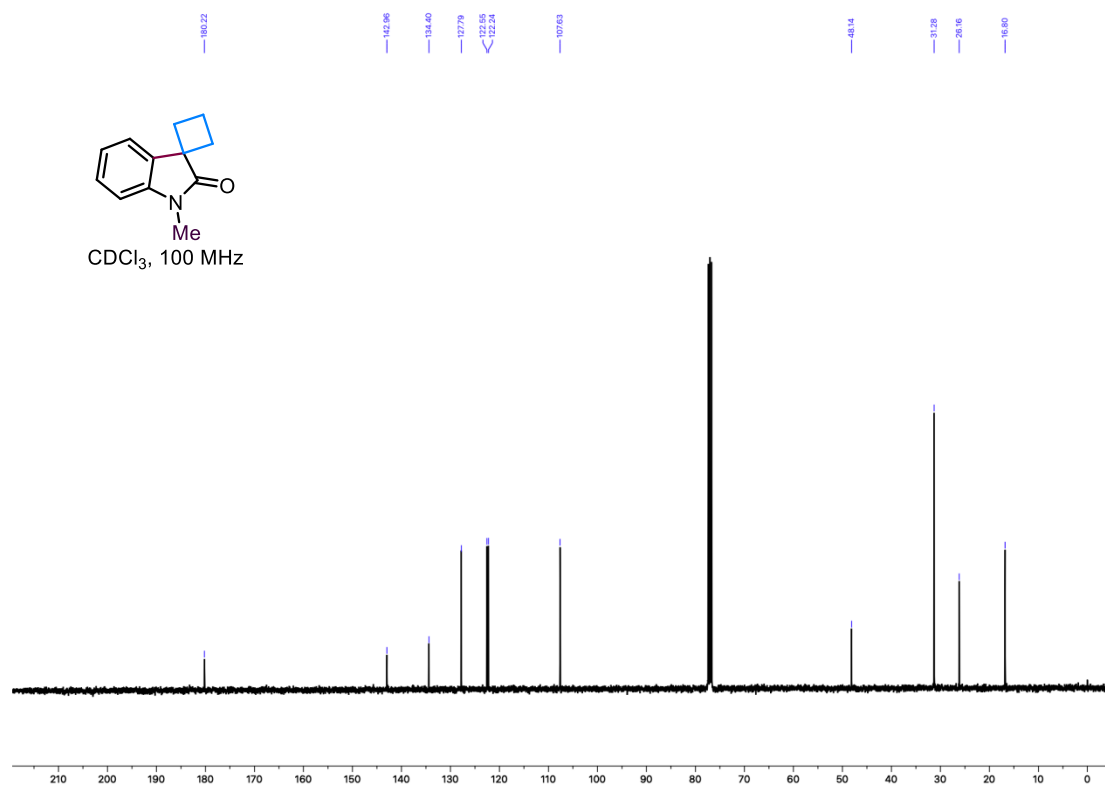
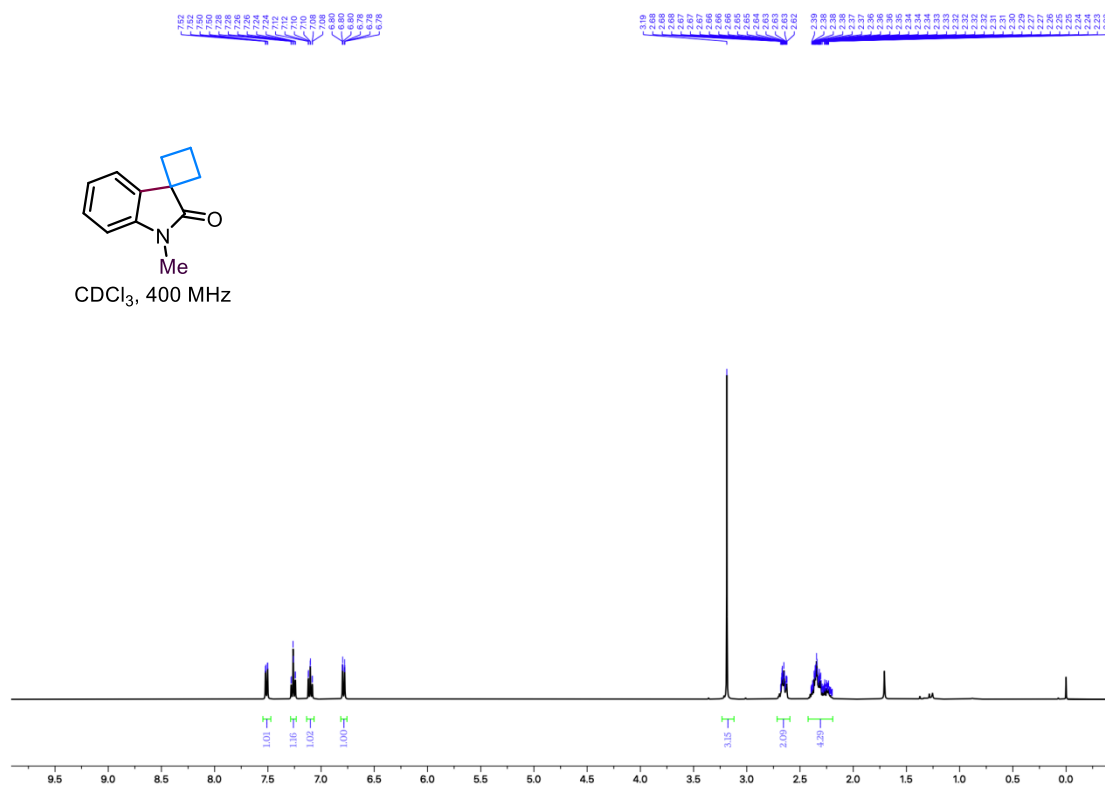
1'-Methylspiro[cycloheptane-1,3'-indolin]-2'-one (2n)



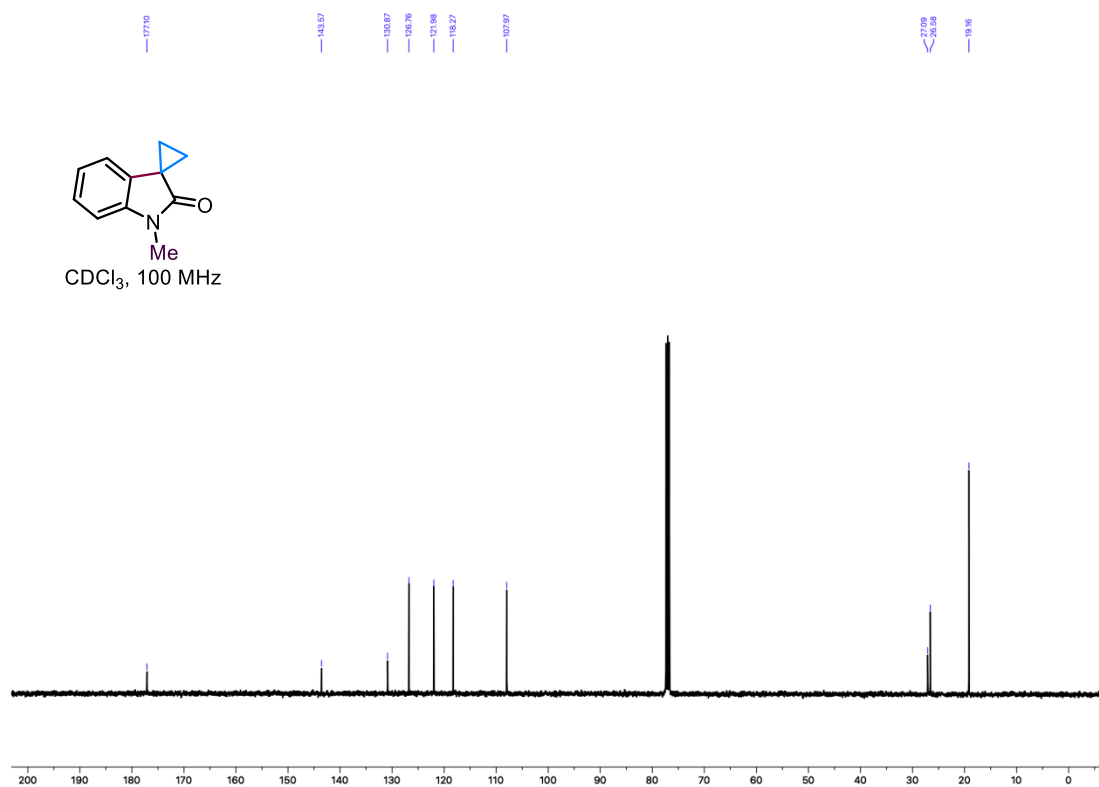
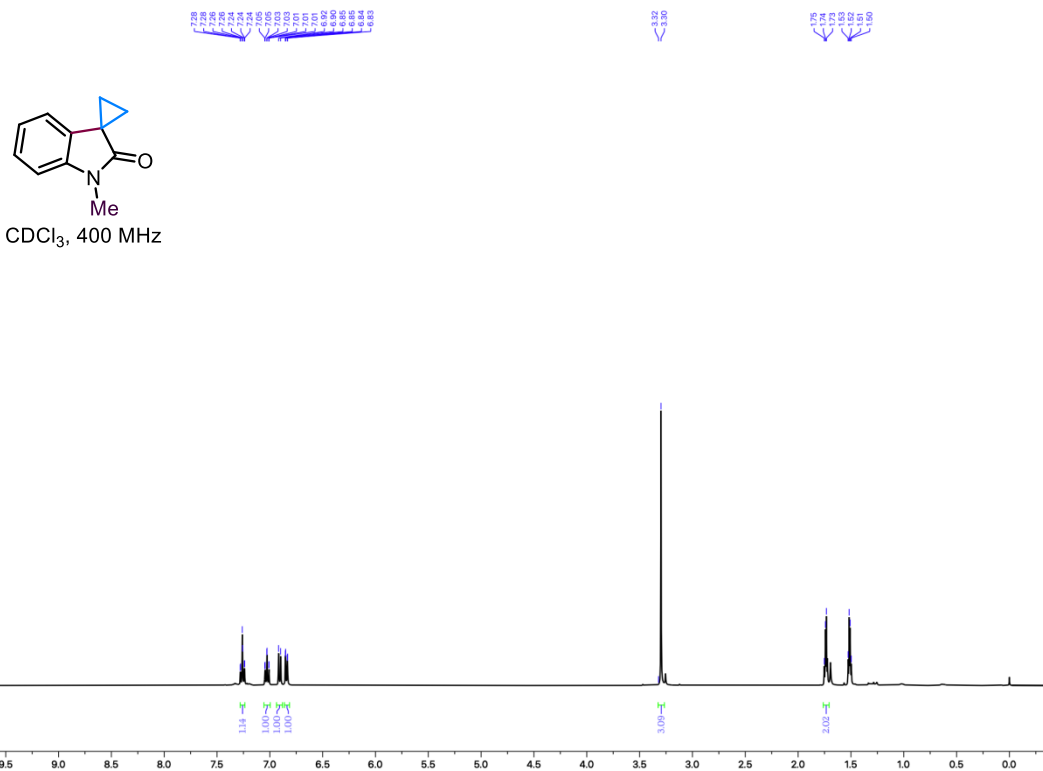
1'-Methylspiro[cyclopentane-1,3'-indolin]-2'-one (2o)



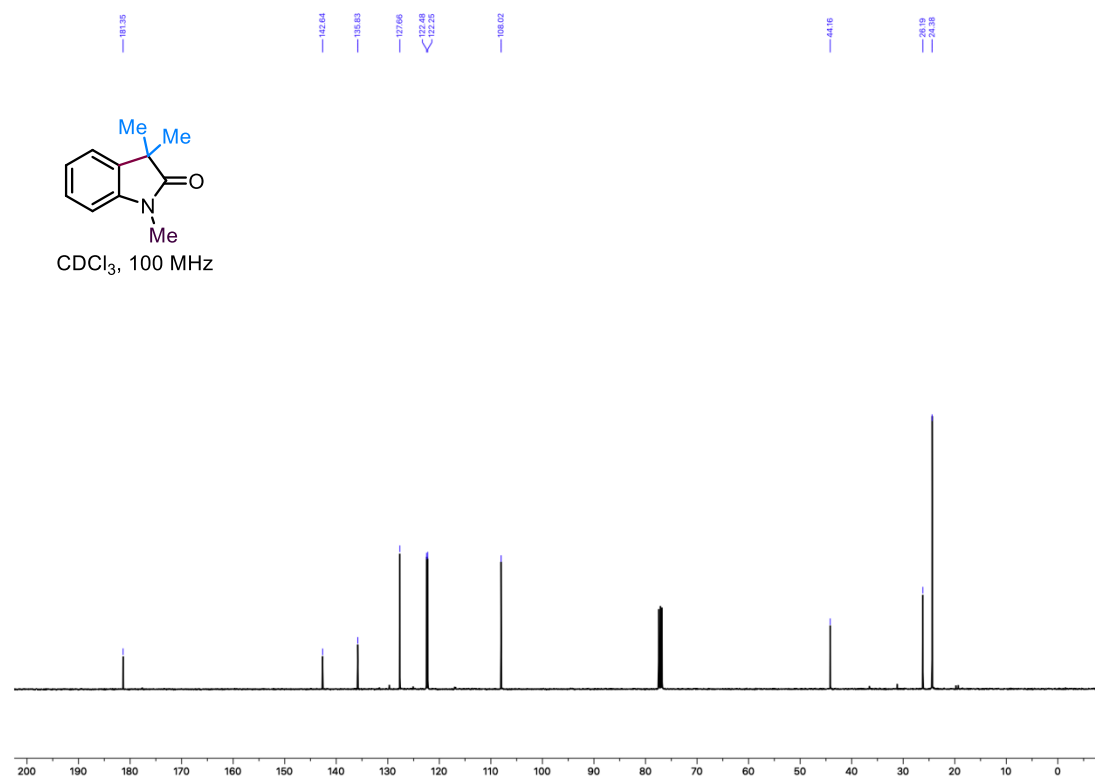
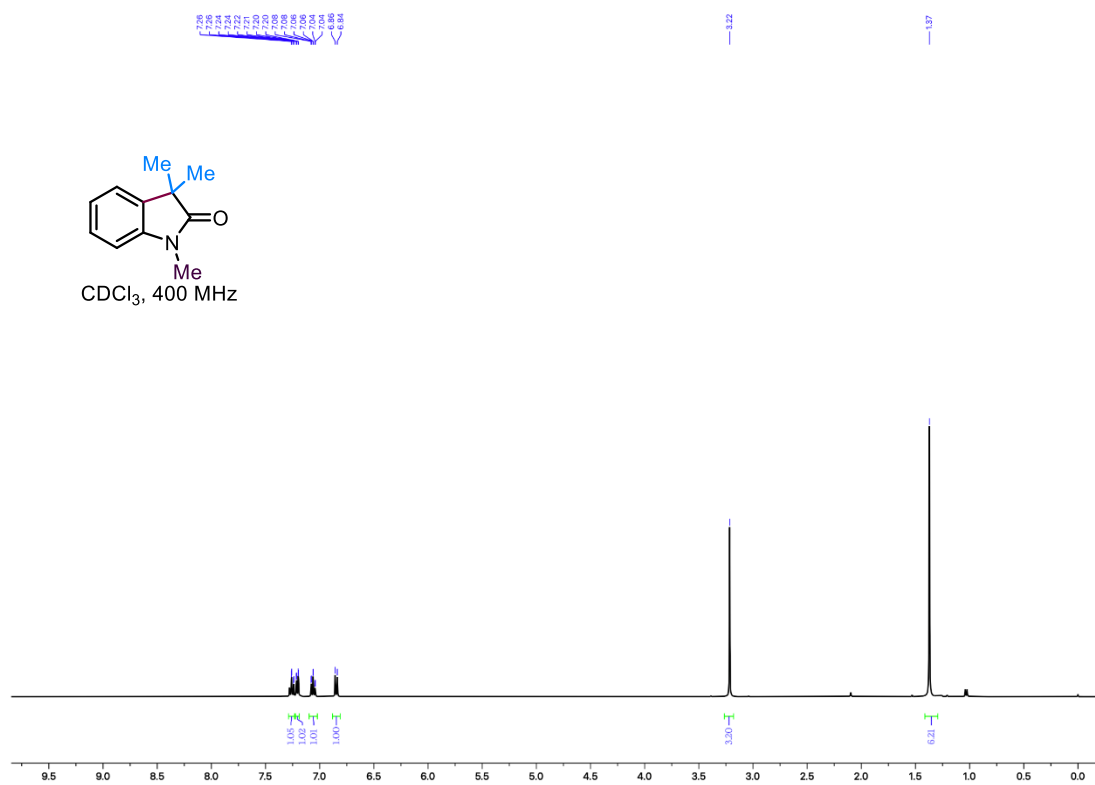
1'-Methylspiro[cyclobutane-1,3'-indolin]-2'-one (2p)



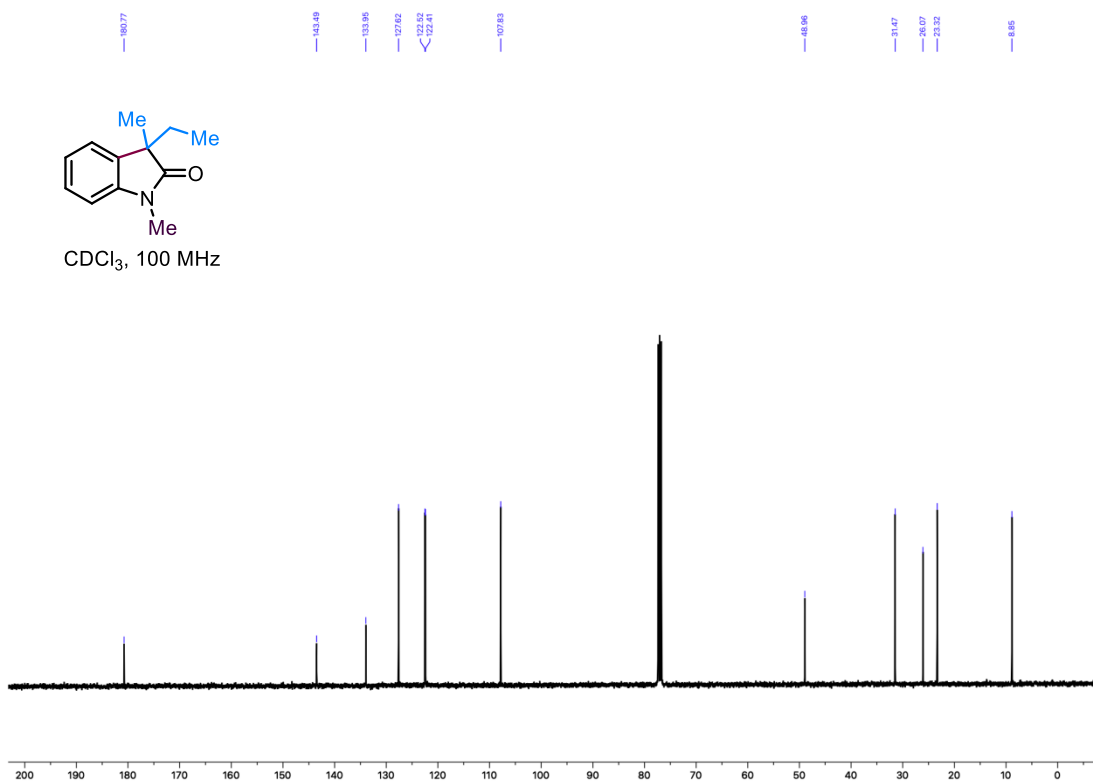
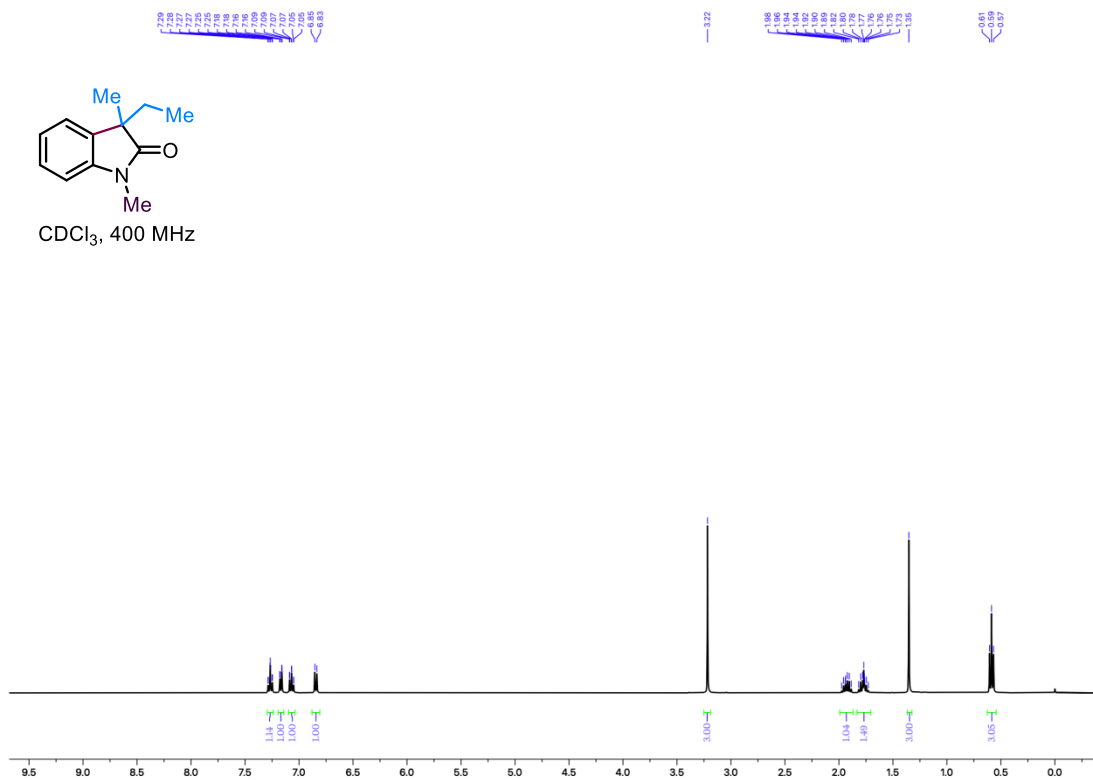
1'-Methylspiro[cyclopropane-1,3'-indolin]-2'-one (2q)



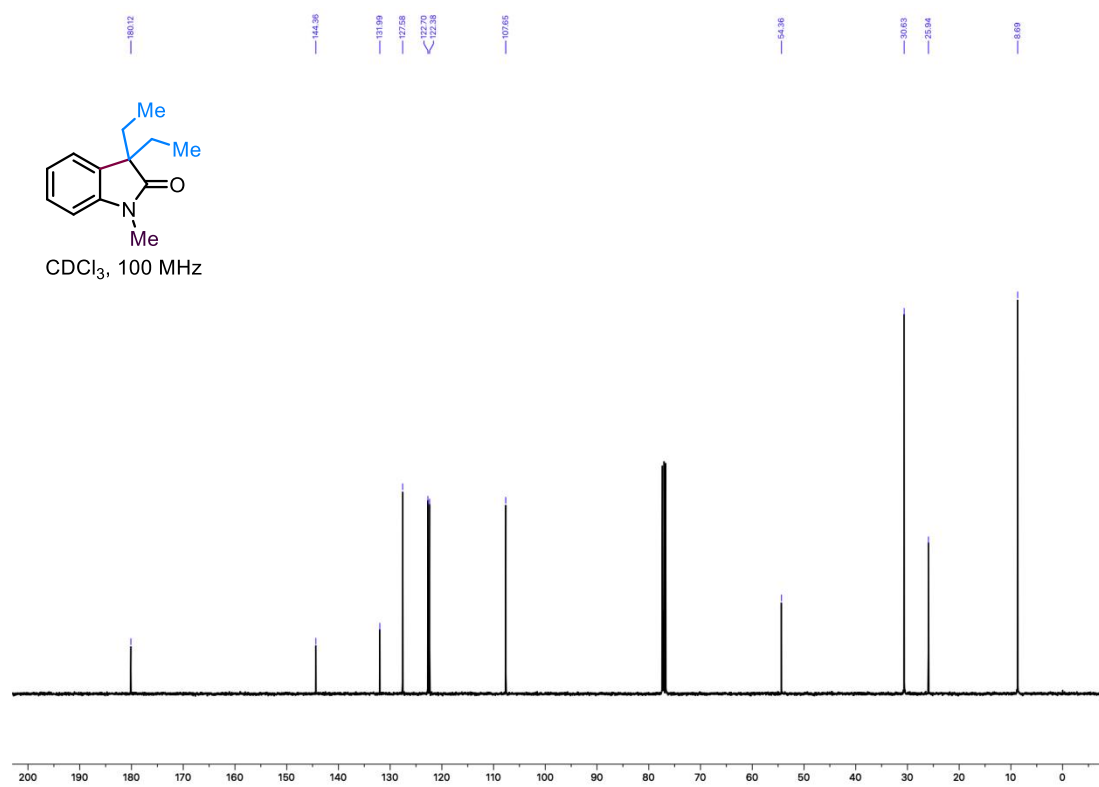
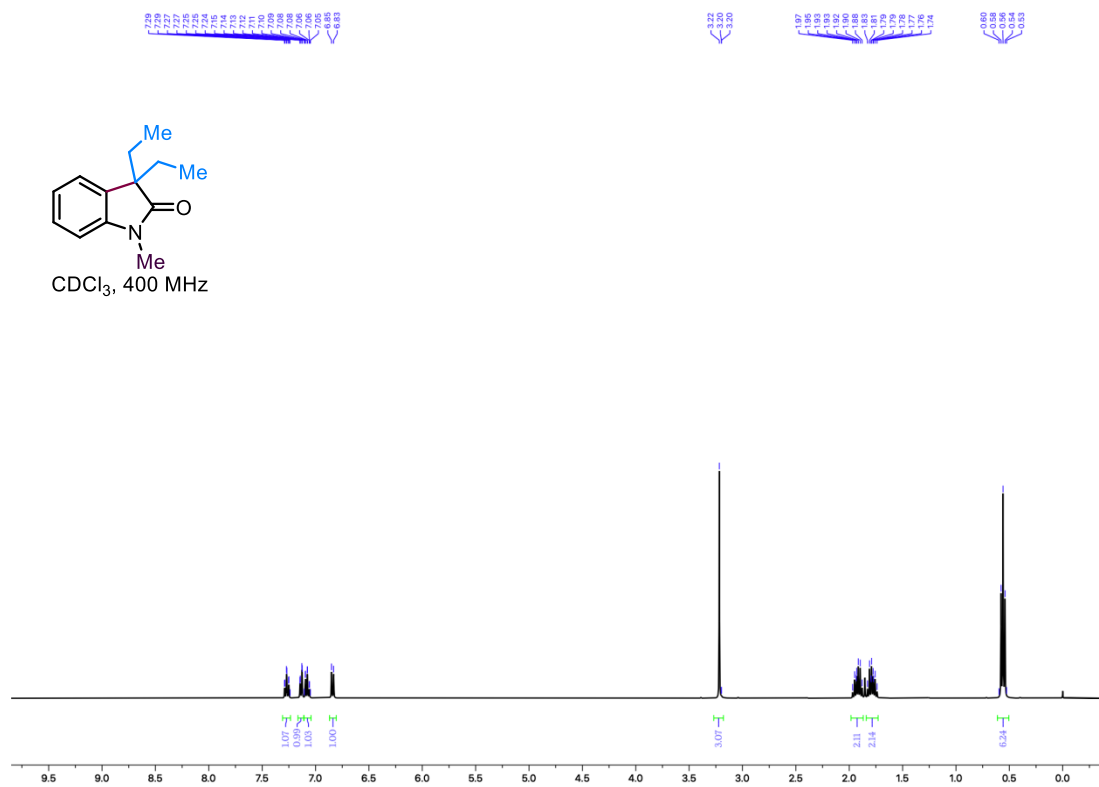
1,3,3-Trimethylindolin-2-one (2r)



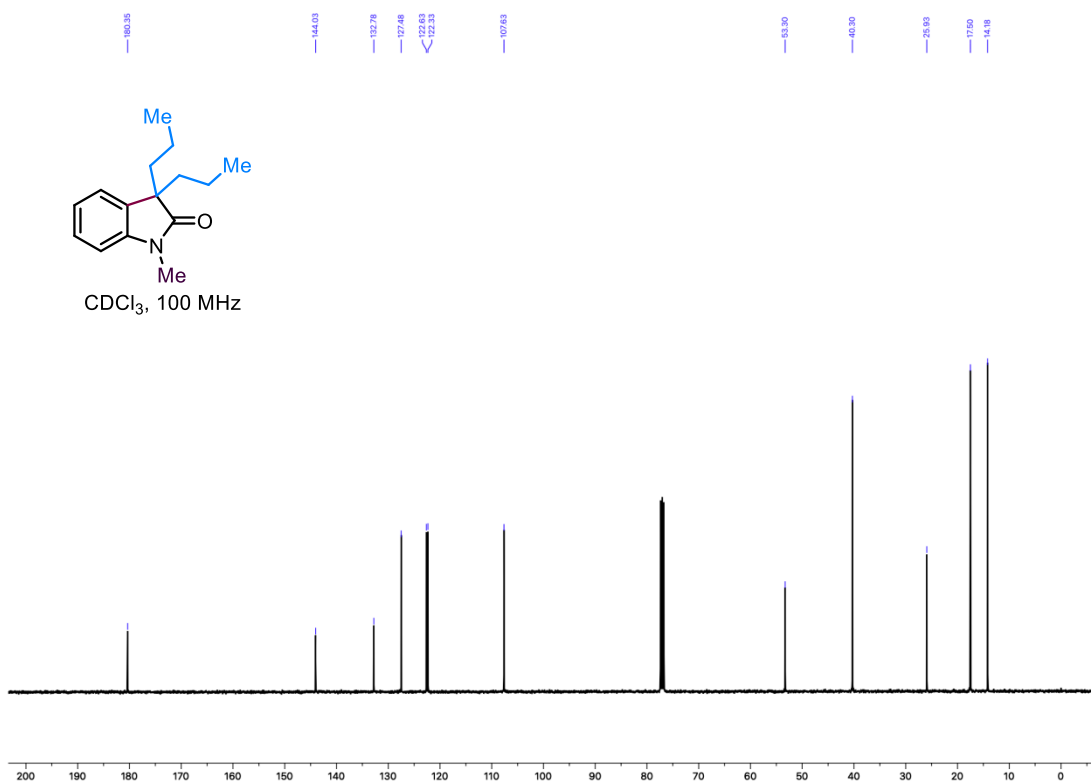
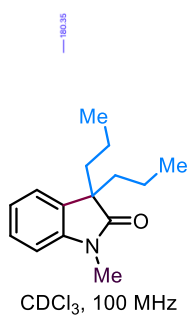
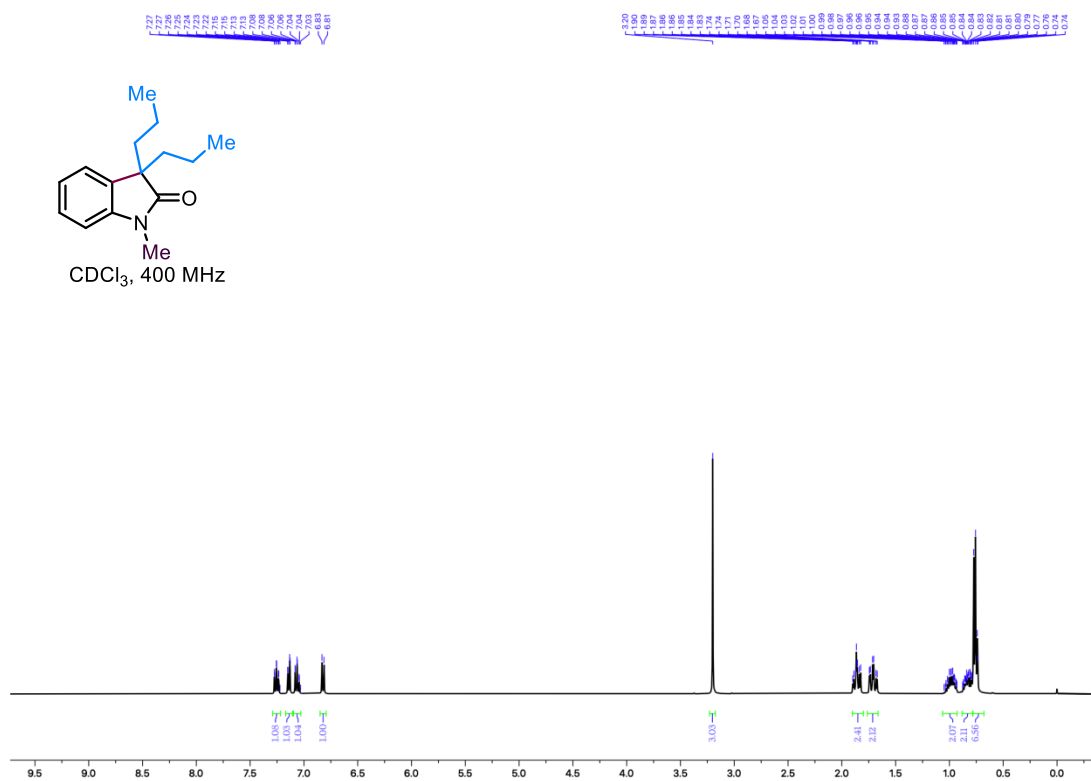
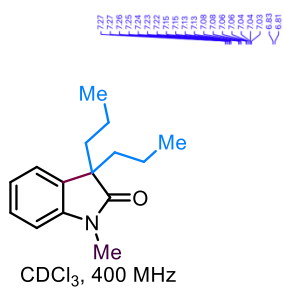
2-Ethyl-1,3-dimethylindolin-2-one (2s)



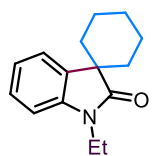
3,3-Diethyl-1-methylindolin-2-one (2t)



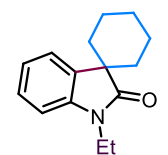
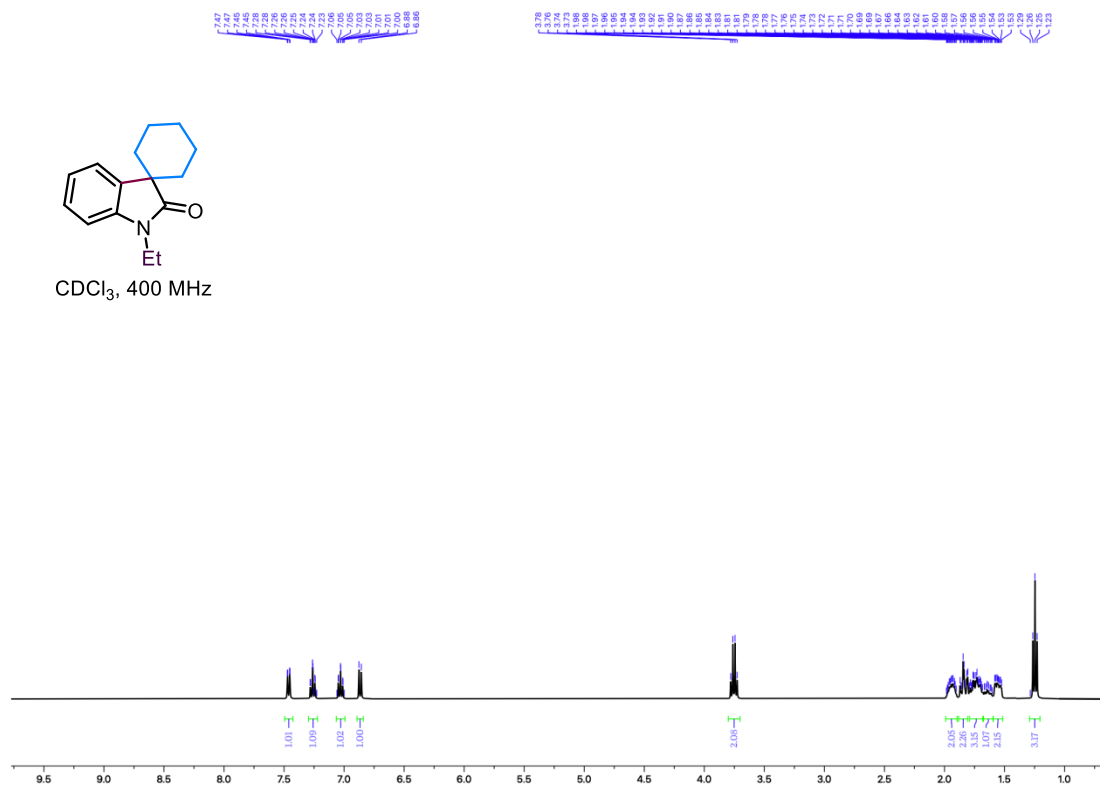
1-Methyl-3,3-dipropylindolin-2-one (2v)



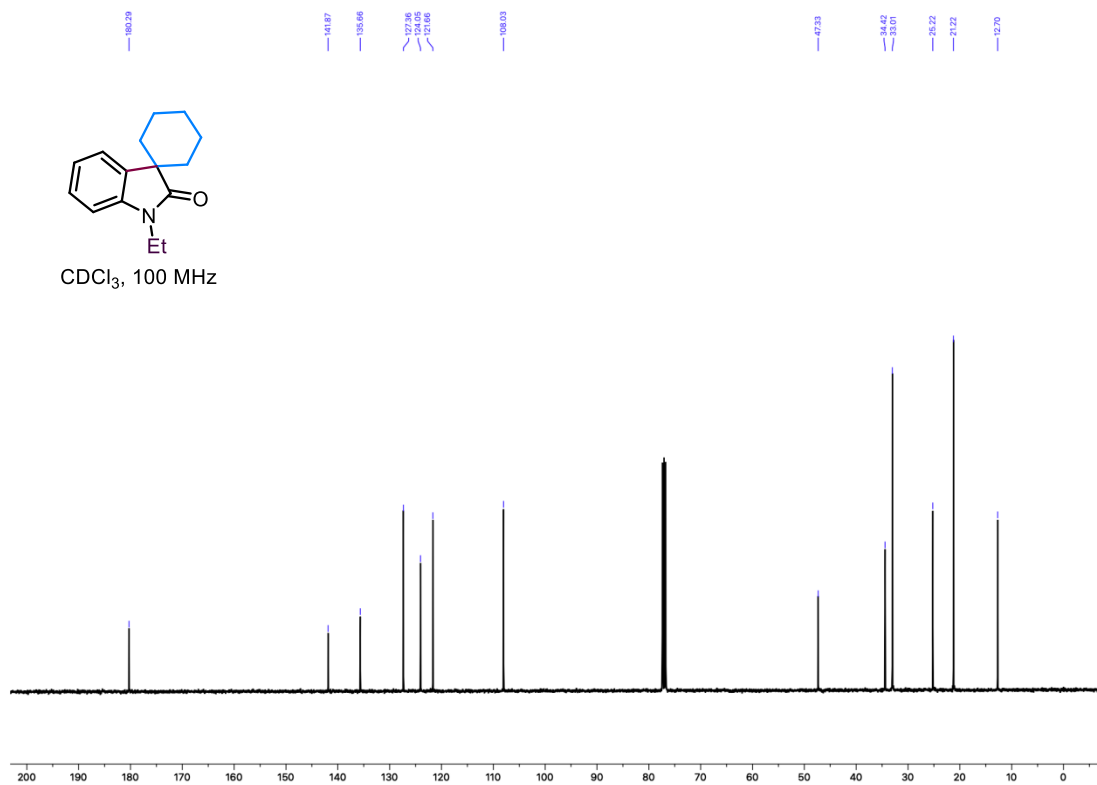
1'-Ethylspiro[cyclohexane-1,3'-indolin]-2'-one (2w)



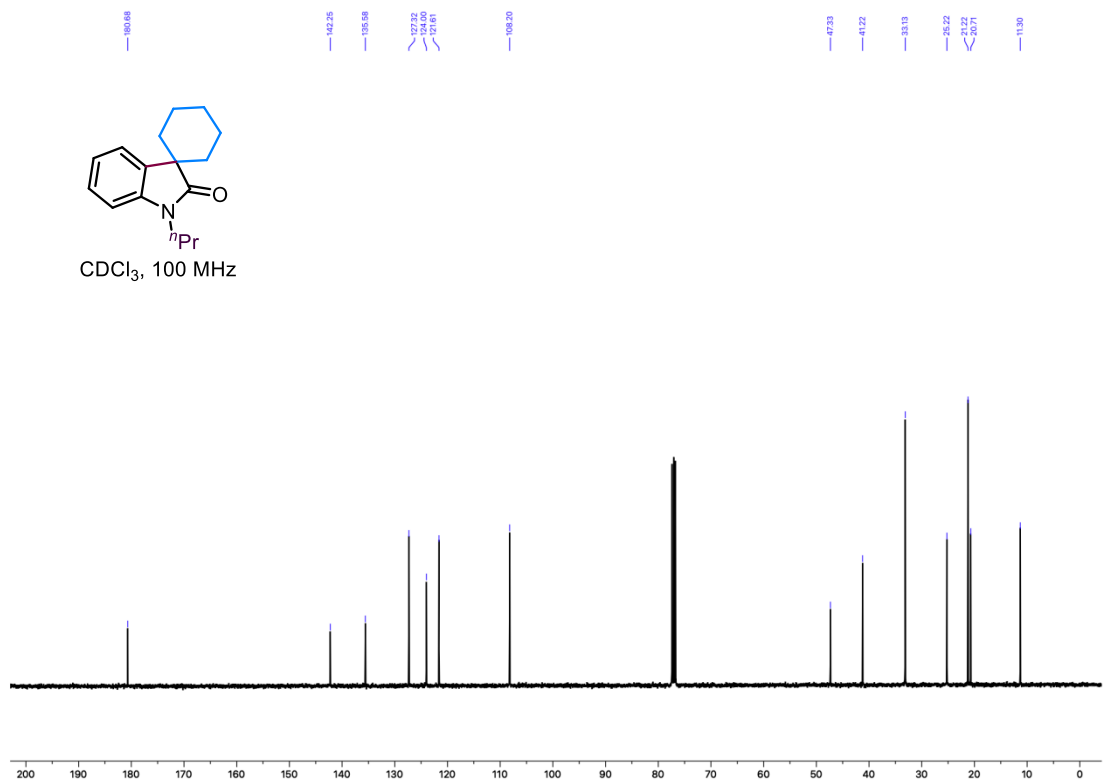
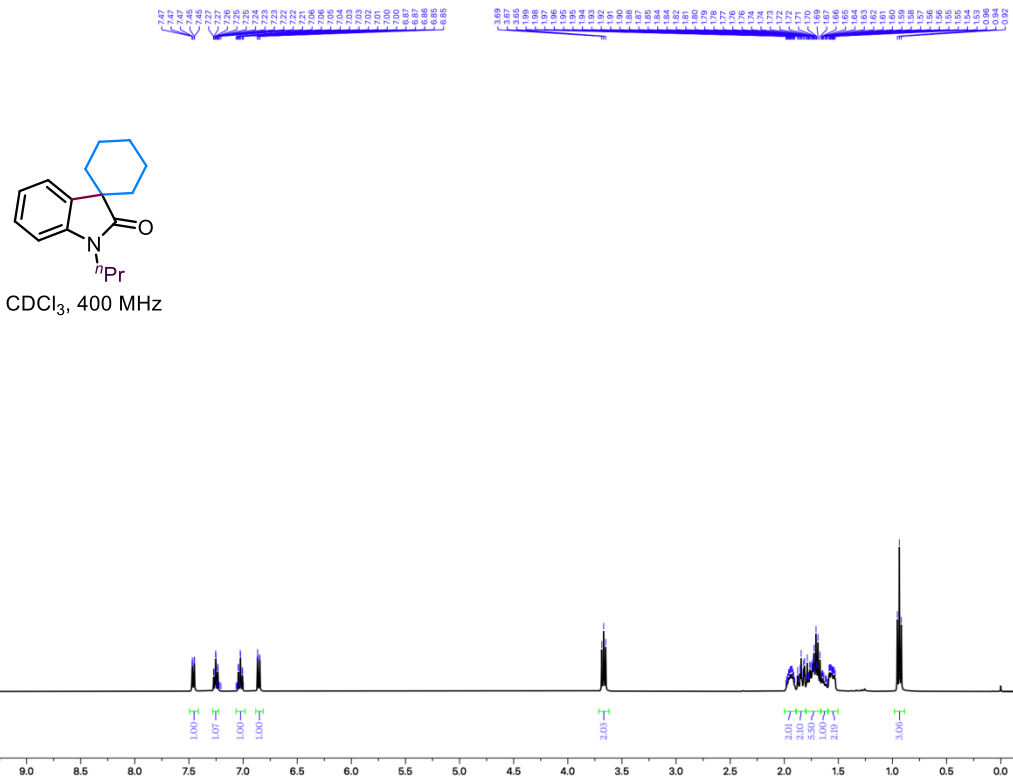
CDCl₃, 400 MHz



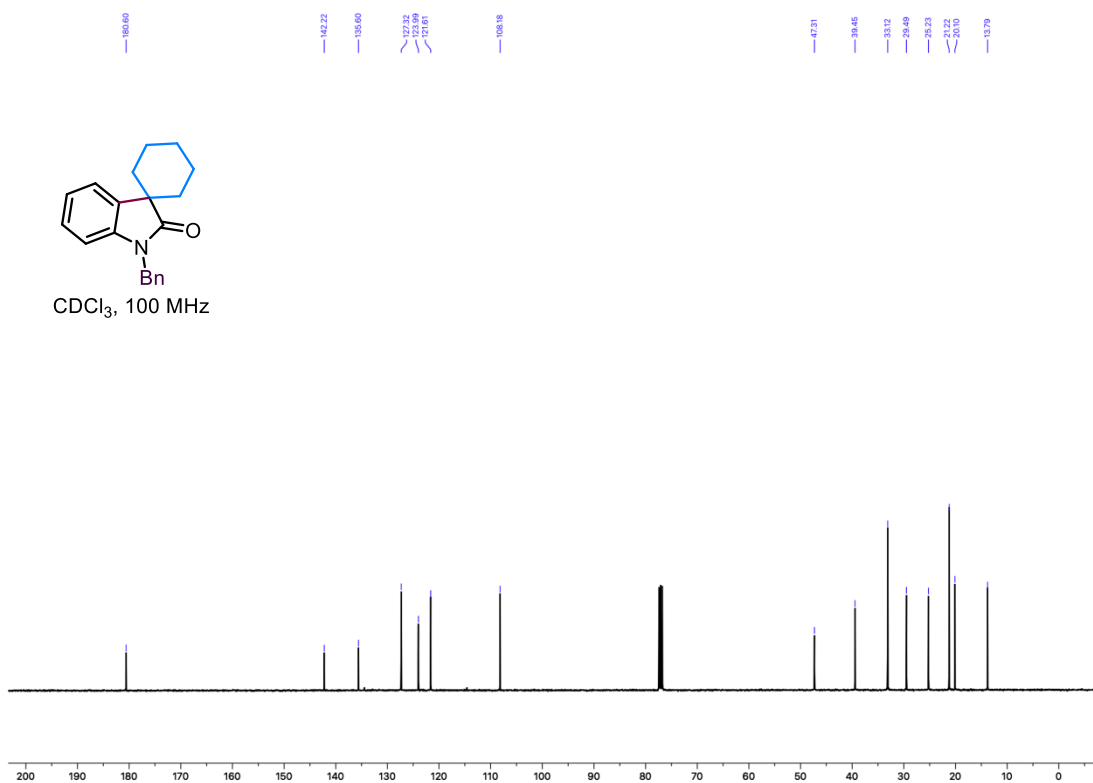
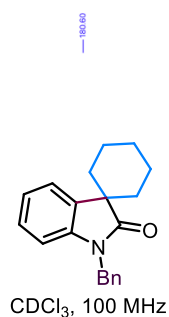
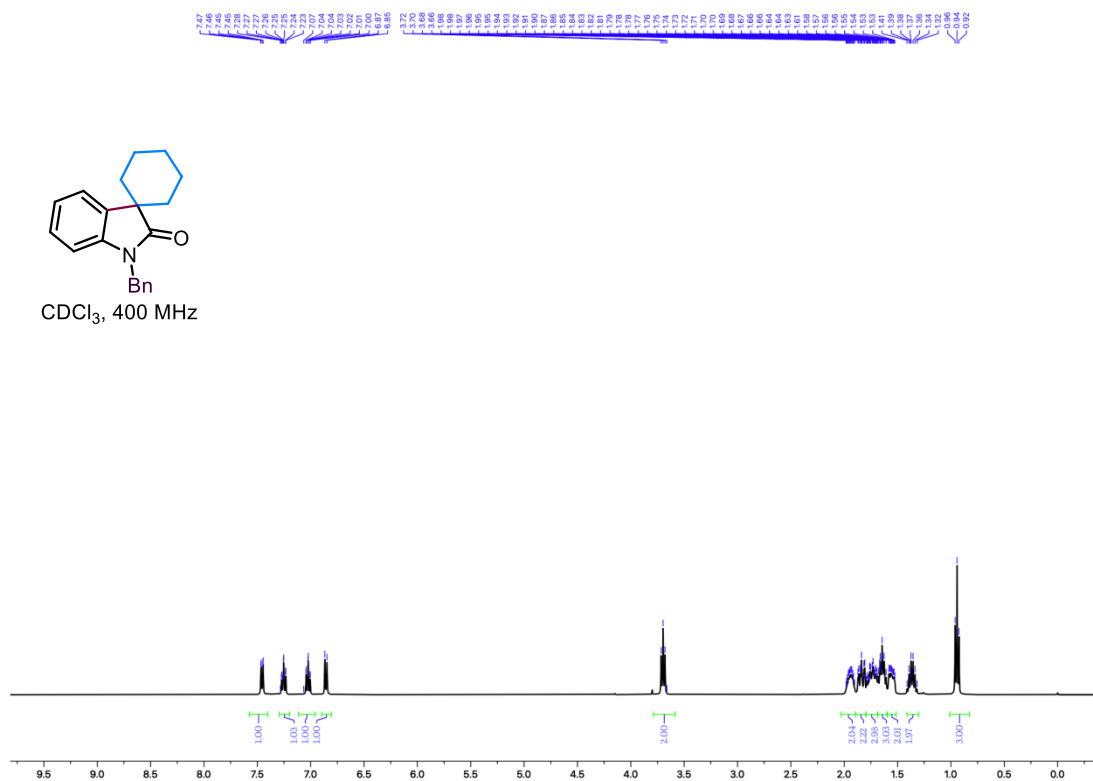
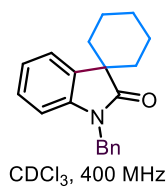
CDCl₃, 100 MHz



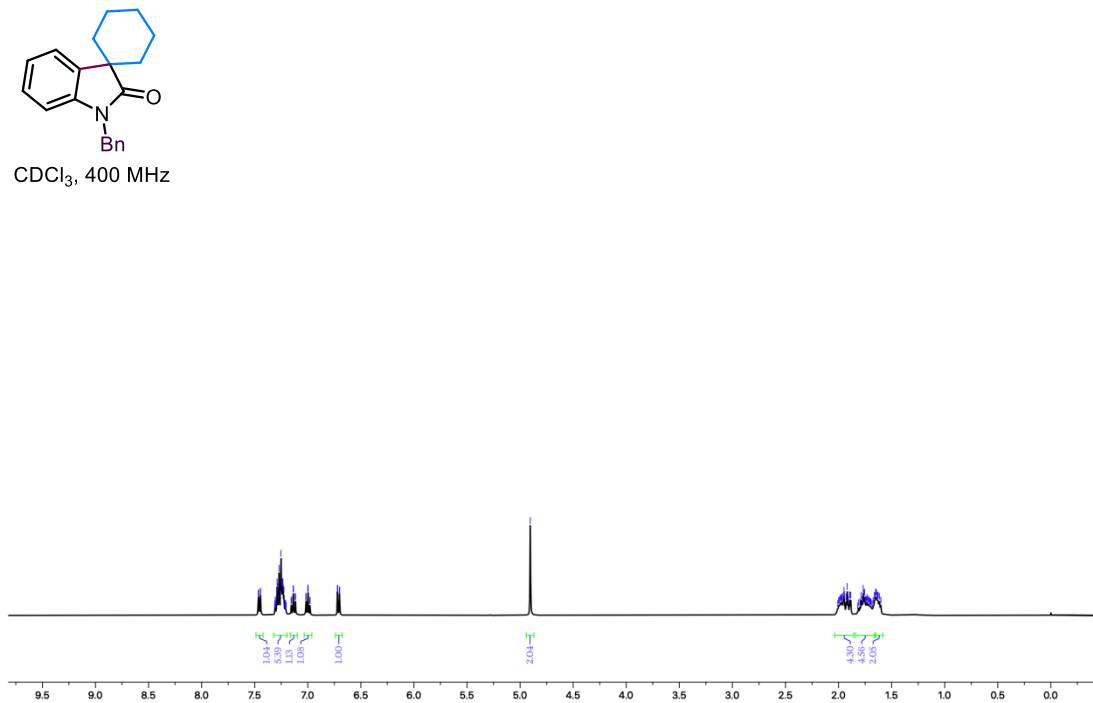
1'-Propylspiro[cyclohexane-1,3'-indolin]-2'-one (2x)



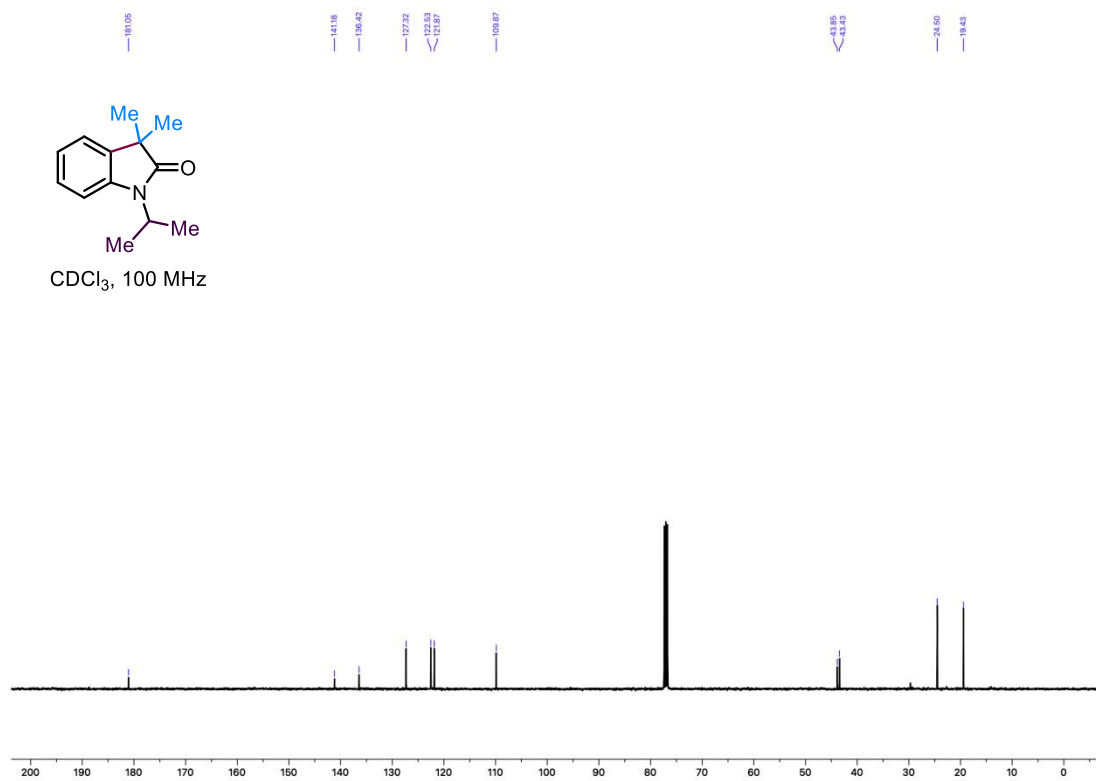
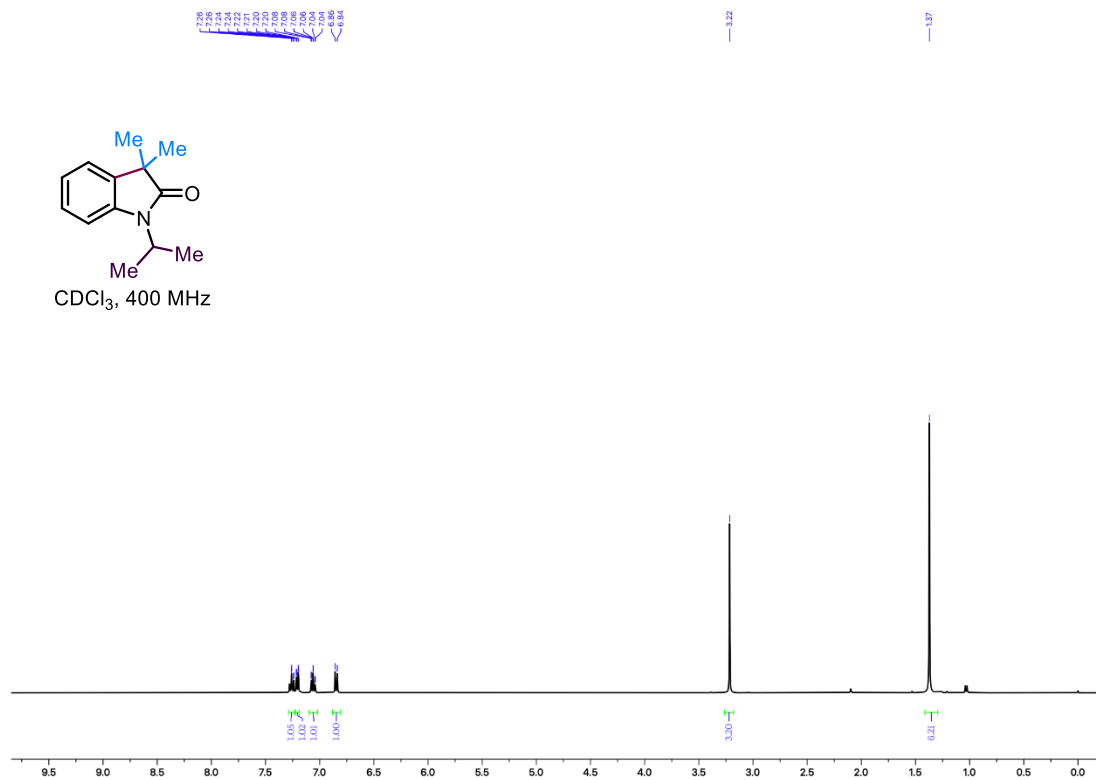
1'-Butylspiro[cyclohexane-1,3'-indolin]-2'-one (2y)



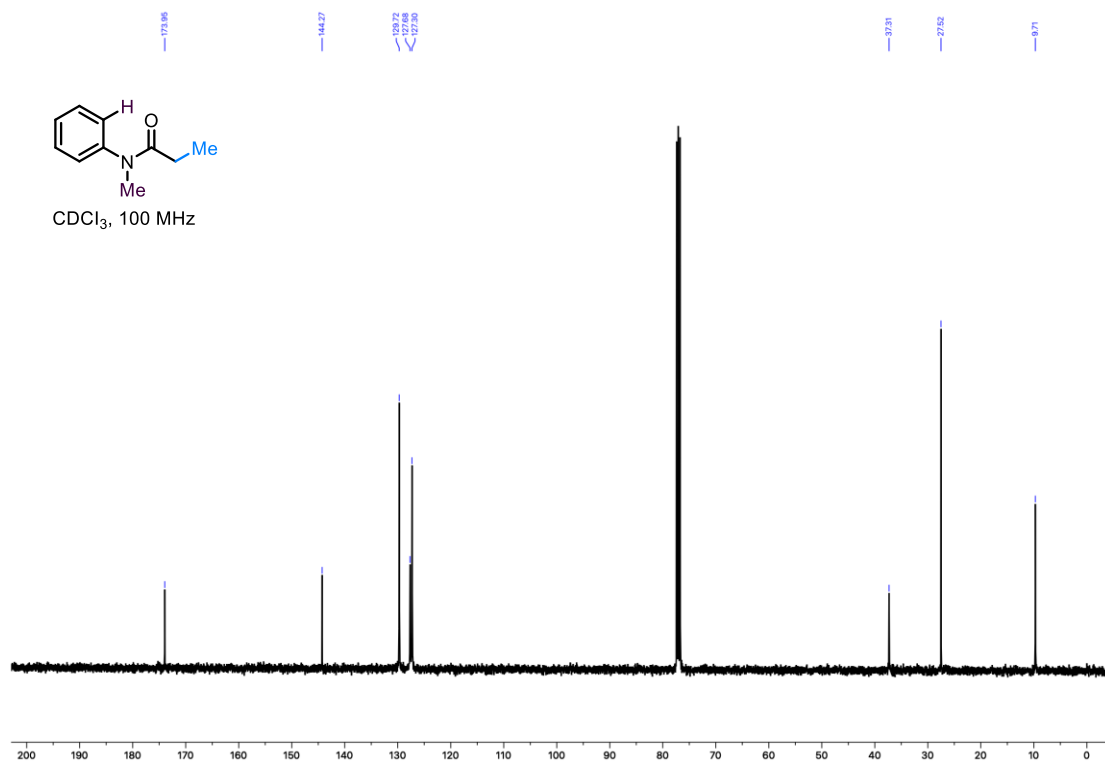
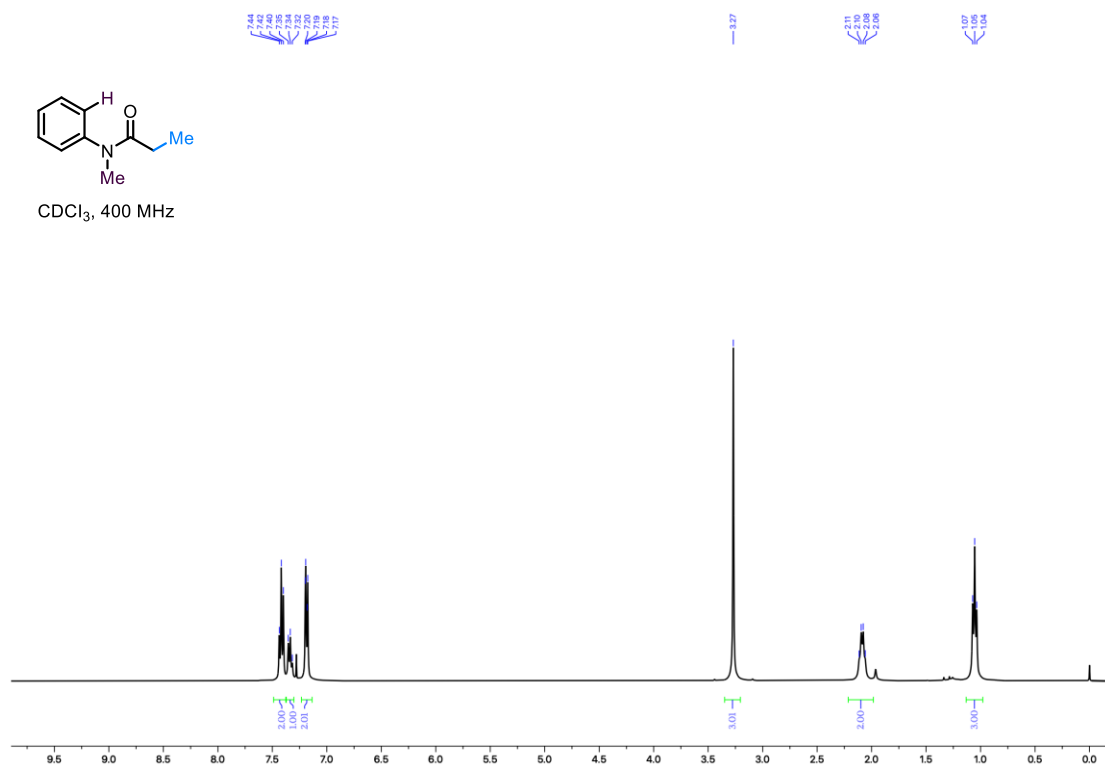
1'-Benzylspiro[cyclohexane-1,3'-indolin]-2'-one (2z)



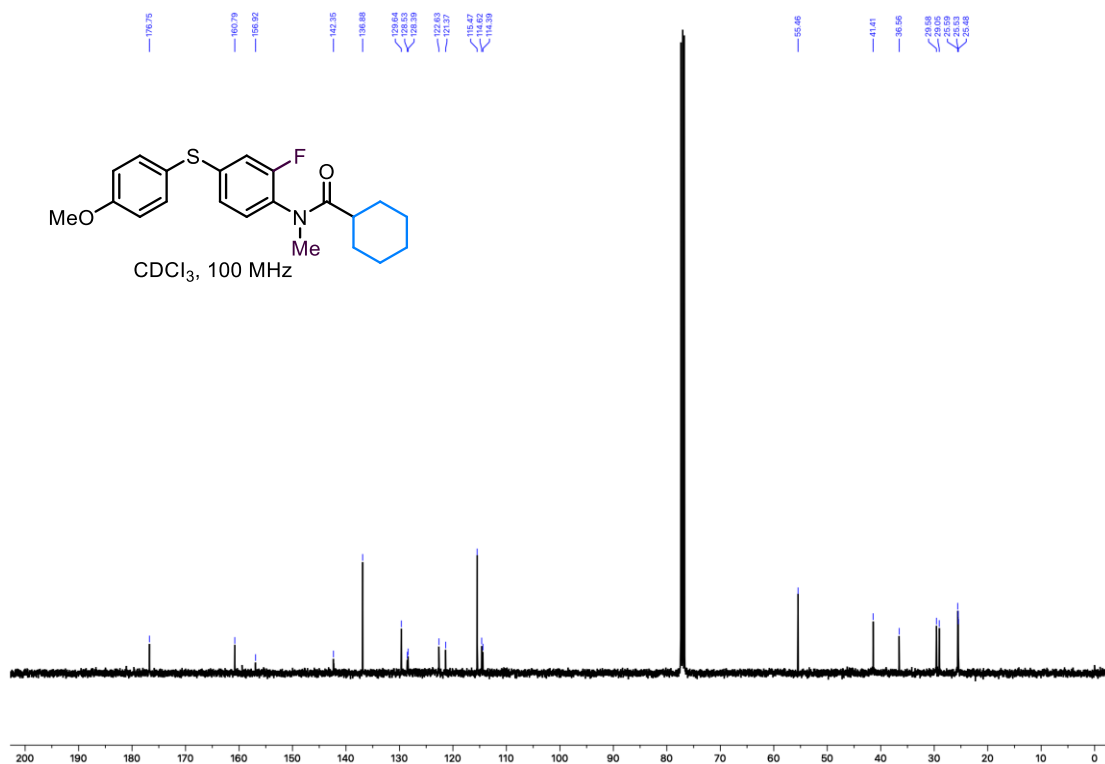
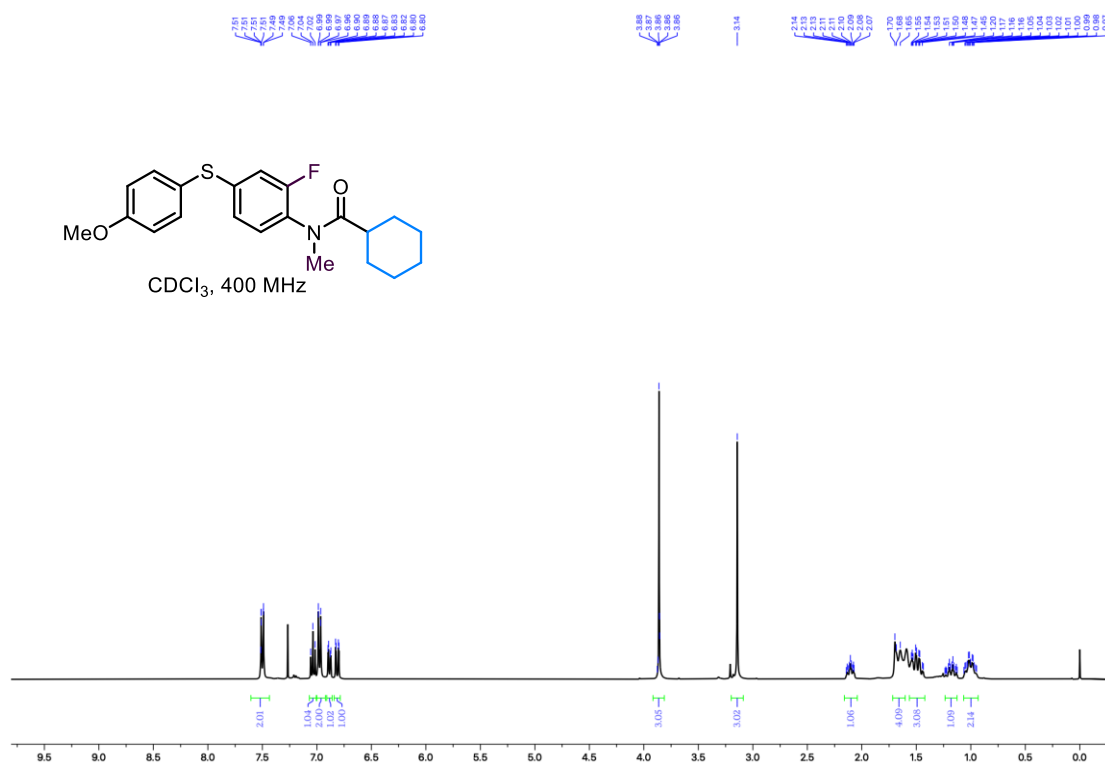
1-Isopropyl-3,3-dimethylindolin-2-one (2aa)



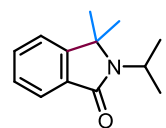
N-Methyl-*N*-phenylpropionamide (2ab')



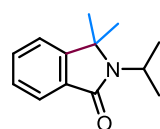
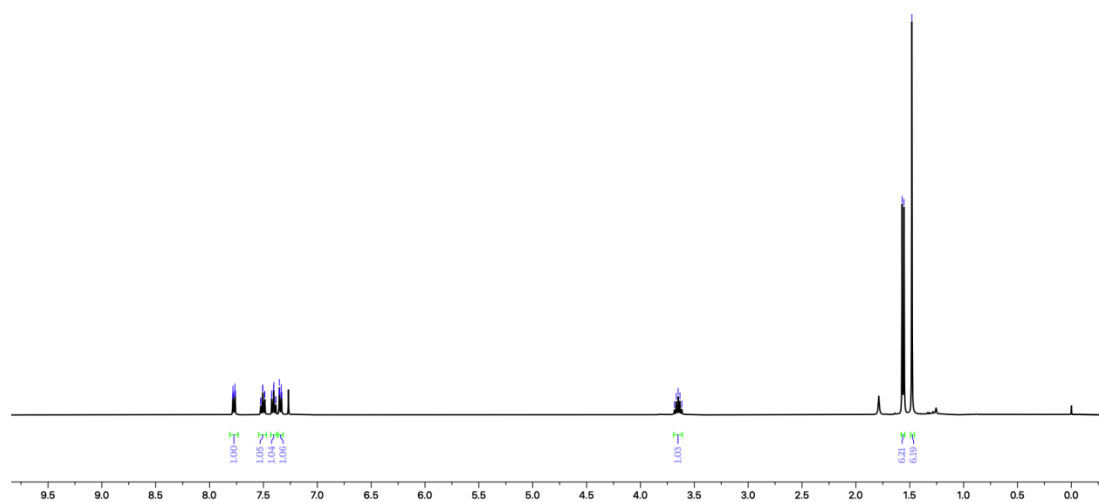
N-(2-Fluoro-4-((4-methoxyphenyl)thio)phenyl)-*N*-methylcyclohexanecarboxamide
(2ac')



7-Fluoro-2-isopropyl-3,3-dimethylisoindolin-1-one (4)



CDCl₃, 400 MHz



CDCl₃, 100 MHz

