Electrochemical bromocyclization enables 3, 5-diversification of heterocyclic indolines

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Supporting Information

Content

1. General Information

All solvents and reagents were obtained from commercial sources and were purified according to standard procedures before use (unless stated otherwise). Column chromatography was performed on silica gel (Qingdao, 300 - 400 mesh) using the indicated eluents. ^{1}H and ^{13}C NMR data were collected on a Varian Mercury 400 MHz or Agilent Mercury 600 MHz NMR spectrometer at room temperature using chloroform-*d* or DMSO-d_6 as a solvent and TMS as an internal standard, and chemical shift (δ) was expressed in parts per million (ppm). ^{1}H and ^{13}C NMR spectra were internally referenced to the proton (^{1}H) of the internal TMS signal at 0.00 ppm or the solvent residue of DMSO-d_6 at 2.50 ppm and the residual carbon nuclei (^{13}C) of the solvent at 77.0 or 39.5 ppm, respectively. The following abbreviations were used in expressing the multiplicity: s = singlet, brs = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet. High resolution mass spectra (HRMS-ESI) were recorded on a Bruker ESI-QTOF mass spectrometer. The course of the reactions was monitored by thin-layer chromatography (TLC). Melting points are measured in WRX-4 melting point apparatus purchased from Shanghai YICE Instrumental Company. The NaBr was obtained commercially and used directly without further purification.

2. Optimization of the reaction conditions

2.1 Electrolysis general informations

Electrochemical reactions were performed with ElectraSyn 2.0 package (IKA) using the constant current or constant voltage modes. The reactions were conducted in a 10 mL vial for 4 mL of solvent with a stir bar and a carbon graphite-SK-50 (5.0 × 0.8 × 0.2 cm) working electrode (anode and cathode) with a distance of 0.6 cm between the two electrodes.

2.2 Conditions screening [a]

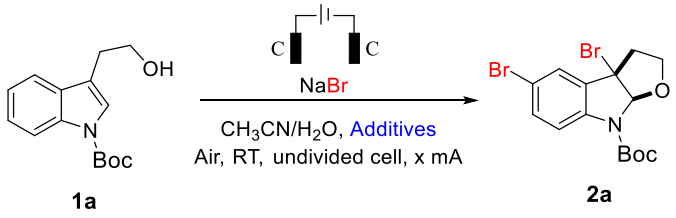


Table S1

Entry	NaBr (x equiv.)	Additives	CCE (mA)	H_2O (μL)	Time (h)	**2a** (%)
1	2	-	5	100	12	31
2	2.5	-	5	0	12	35
3	3	-	5	100	12	35
4	4	-	5	0	12	40
5	2.5	CH_3CO_2H (10 μL)	5	100	12	66
6	2.5	CH_3CO_2H (20 μL)	5	100	12	70
7	2.5	CH_3CO_2H (30 μL)	5	100	12	71
8	2.5	CH_3CO_2H (50 μL)	5	100	12	59
9	2.5	CH_3CO_2H (30 μL)	5	30	12	78
10	2.5	CH_3CO_2H (30 μL)	5	50	9	75
11	2.5	CH_3CO_2H (30 μL)	5	80	9	61
12	2.5	CH_3CO_2H (30 μL)	10	30	4	85
13	2.5	CH_3CO_2H (20 μL)	10	30	3	85
14	2.5	CH_3CO_2H (20 μL)	10	-	12	0
15	2.5	-	10	30	3	17
16	2.5	CH_3CO_2H (20 μL)	-	30	12	0
17	2.5	$PhCO_2H$ (40 μL)	10	30	3	84
18	2.5	EtOH (20 μL)	10	30	3	24
19	2.5	HFIP (37 μL)	10	30	3	0
20	2.5	CH_3SO_3H (23 μL)	10	30	3	67
21	2.5	$C_2H_5CO_2H$ (26 μL)	10	30	3	84
22	2.5	CH_3CO_2Na (28.7 mg)	10	30	3	trace
23	2.5	$CH_3CO_2NH_4$ (27.0 mg)	10	30	3	trace

[a] Conditions: undivided cell, carbon-cloth anode and cathode, **1a** (0.2 mmol), NaBr, CH_3CN (4 mL), H_2O (30 μL), Additives (CH_3CO_2H, 20 μL = 0.35 mmol), 3 – 12 h (13.6 – 37.6 F mol^{-1}), RT, under air. Yields are isolated by column chromatography. CCE = constant current electrolysis.

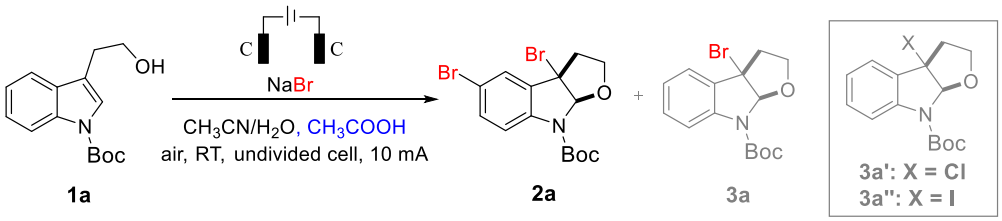


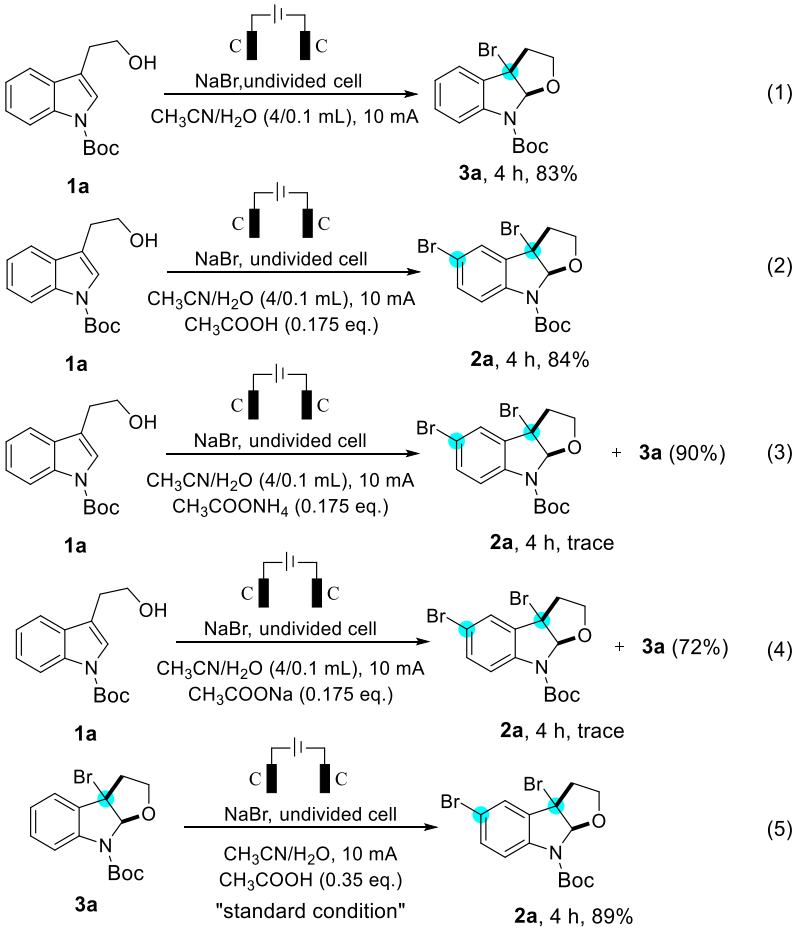
Table S2

Entry	Derivation from standard condtions	**2a** (%)	**3a** (%)
1	None	85	0
2	Without CH_3CO_2H	17	79
3	Without H_2O	0	89
4	EtOH instead of CH_3CO_2H	24	72
5	HFIP instead of CH_3CO_2H	0	38
6	CH_3SO_3H instead of CH_3CO_2H	67	0
7	$C_2H_5CO_2H$ instead of CH_3CO_2H	84	0
8	CH_3CO_2Na instead of CH_3CO_2H	trace	72
9	$CH_3CO_2NH_4$ instead of CH_3CO_2H	trace	90
10	CF_3CO_2H instead of CH_3CO_2H	0	78
11	$PhCO_2H$ instead of CH_3CO_2H	84	0
12	30 uL CH_3CO_2H	85	0
13	No electric current	0	0
14	NaCl instead of NaBr	0	trace (**3a′**)
15	NaI instead of NaBr	0	49 (**3a′′**)
16	TBAB instead of NaBr	0	53
17	Pt electrode instead of C electrode	0	0

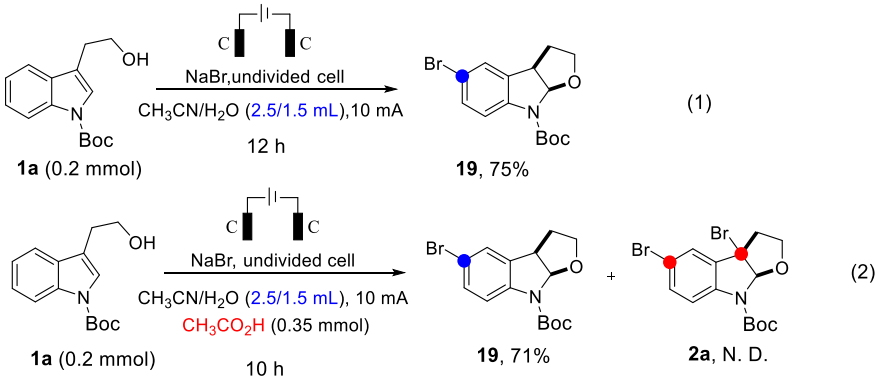
[a] Conditions: undivided cell, carbon-cloth anode and cathode, **1a** (0.2 mmol), NaBr (0.5 mmol), CH_3CN (4 mL), additive H_2O (30 μL), CH_3COOH (20 μL = 0.35 mmol), CCE = 10.0 mA, 3 –12 h (13.6–37.6 F mol^{-1}), RT, under air. Yields are based on product isolated by column chromatography. CCE = constant current electrolysis.

3. Control experiments

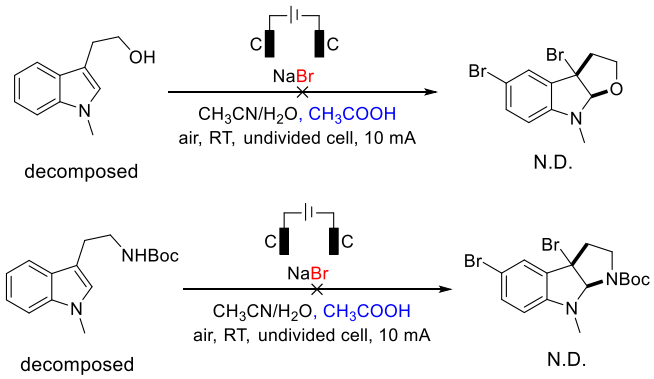
Several control experiments were conducted to investigate the functions of the protonic acid. In our previous work, a 4/0.1 (*v*/*v*) mixture of acetonitrile and water enabled highly selective 3-bromocyclization (**3a**: 83% yield), and a trace amount of 3,5-dibromoindoline **2a** was not observed (eq. 1). When CH_3COOH was added under such conditions, the 3,5-dibromoindoline **2a** was obtained as the final product in 84% yield (eq. 2). These results indicate that CH_3COOH plays a key role in the 5-bromination process. When CH_3COONa or CH_3COONH_4 was used as the additive instead of CH_3COOH, the dibromination reaction was significantly restrained, which suggests that the hydrogen ion (H^+) plays an important role in promotion of the bromination reactions (eqs. 2 and 3). When the 3-bromoindoline **3a** was employed under the optimal conditions, the 3, 5-dibromoindoline **2a** was successfully obtained in high yield (eq. 5), which indicates that a cascade electrophilic C3a-bromocyclization and C5a-bromination process may be responsible for this reaction.



In our previous work,[1] a 2.5/1.5 (*v*/*v*) mixture of acetonitrile and water enabled highly selective 5-bromocyclization to form the 5-bromoindoline **19**, and none of 3,5-dibromoindoline **2a** was observed (eq. 1). The mechanism of the formation of 5-bromoindoline has been verified through a key reductive hydrodebromination of 3a-bromofuranindoline at the cathode.[1] On the basis of the previous conditions (eq. 1), when CH_3COOH was added into this condition, the 5-bromoindoline **19** was obtained in 71% yield after 10 h and none of 3, 5-dibromoindoline **2a** was detected. These results suggested that the reductive hydrodebromination of 3a-bromoindoline at the cathode was mainly controlled by the use of more amount of water and the electrophilic 5-bromoniation process was accelerated by CH_3COOH.



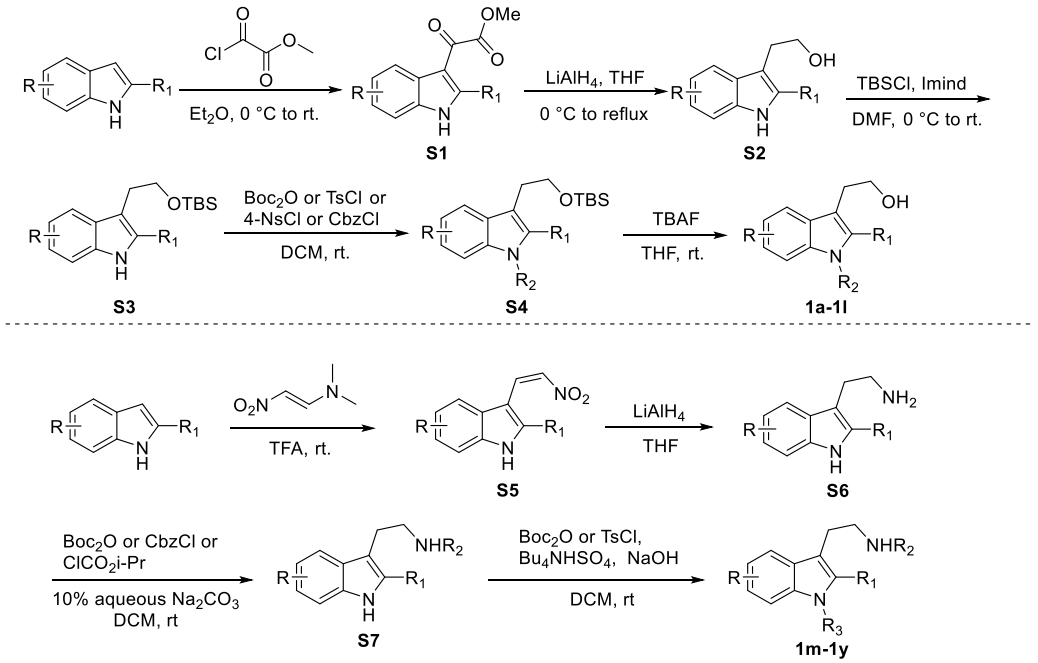
When the N-methyl indole substrates were employed in the standard conditions, none of desired dibromoindoline product was obtained and the substrates decomposed. We assumed that the electron-rich of indole was unstable under the current electronic oxidative conditions, an electron-withdrawing group is necessary for the reaction.



4. Experimental procedures and characterizations

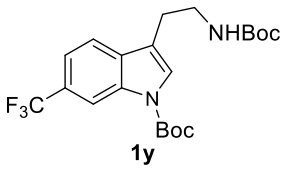
4.1 Preparation of tryptamine/tryptophol substrates

All of the different tryptamine/tryptophol substrates were prepared according to the published procedures[1].



The physical datas for new substrate compounds were provided below:

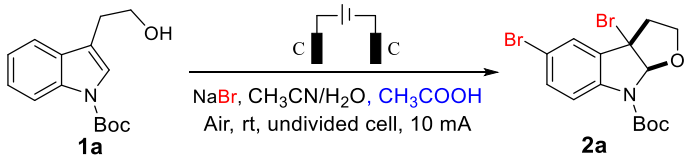
tert-butyl 3-(2-((tert-butoxycarbonyl)amino)ethyl)-6-(trifluoromethyl)-1*H*-indole -1-carboxylate (1y)



1y

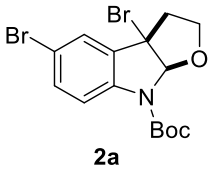
Light yellow oil, 64% yield;**^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.47 (s, 1H), 7.63 (d, J = 8.2 Hz, 1H), 7.55 (s, 1H), 7.49 (d, J = 8.0 Hz, 1H), 4.66 (brs, 1H), 3.47 – 3.44 (m, 2H), 2.92 (t, J = 7.0 Hz, 2H), 1.68 (s, 9H), 1.44 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 155.9, 149.2, 134.7, 132.9, 126.5 (q, $^{2}J_{C\text{-}F}$ = 31.4 Hz), 125.5, 124.8 (q, $^{1}J_{C\text{-}F}$ = 271.6 Hz), 119.3, 119.2 (q, $^{3}J_{C\text{-}F}$ = 3.0 Hz), 117.6, 112.8 (q, $^{3}J_{C\text{-}F}$ = 4.4 Hz), 84.4, 79.4, 40.2, 28.3, 28.1, 25.5 ppm. **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -61.0 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{21}H_{27}O_4N_2F_3Na$ 451.1815; Found 451.1807.

4.2 General Procedure for cyclic 3a, 5a-dibromoindolines



A mixture of **1a** (52.26 mg, 0.2 mmol), NaBr (51.4 mg, 0.5 mmol) and CH_3CN (4 mL), H_2O (30 μL), CH_3CO_2H (20 μL) were added in a 10 mL vial, then the vial was covered with the electrode holder at room temperature, the electrolysis was carried out using a constant current of 10.0 mA a carbon graphite anode and carbon graphite cathode (1.4 × 0.8 × 0.2 cm submerged in solution) with stirring. After completion as detected by TLC. The reaction mixture was diluted with EA. The organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 10/1 to give compound **2a** (71.3 mg, 85% yield).

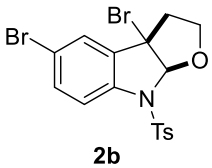
tert-butyl (3a*S*,8a*R*)-3a,5-dibromo-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (2a)



2a

Light yellow oil, 4 h, 71.3 mg, 85% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.73 (brs, 1H), 7.51 (s, 1H), 7.38 (d, *J* = 8.2 Hz, 1H), 6.15 (brs, 1H), 4.01 (t, *J* = 8.2 Hz, 1H), 3.52 – 3.48 (m, 1H), 2.90 – 2.85 (m, 1H), 2.78 – 2.75 (m, 1H), 1.59 (s, 9H) ppm.

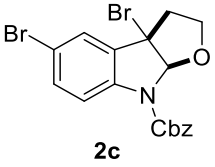
(3a*S*,8a*R*)-3a,5-dibromo-8-tosyl-3,3a,8,8a-tetrahydro-2*H*-furo[2,3-*b*]indole (2b)



2b

White solid, 4 h, 44.6 mg, 47% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.2 Hz, 2H), 7.45 (d, *J* = 1.8 Hz, 1H), 7.39 – 7.35 (m, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 6.21 (s, 1H), 4.03 – 4.00 (m, 1H), 3.46 – 3.42 (m, 1H), 2.84 – 2.79 (m, 1H), 2.71 – 2.68 (m, 1H), 2.38 (s, 3H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 144.7, 139.6, 135.2, 134.5, 133.5, 129.8, 128.3, 127.3, 117.2, 115.7, 103.5, 68.0, 60.1, 44.5, 21.6 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{15}O_3NBr_2NaS$ 493.9031; Found 493.9027.

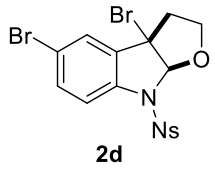
benzyl (3a*S*,8a*R*)-3a,5-dibromo-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (2c)



2c

Colorless oil, 5 h , 44.5 mg, 49% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.53 (d, *J* = 2.0 Hz, 1H), 7.50 – 7.28 (m, 6H), 6.26 (s, 1H), 5.41 – 5.24 (m, 2H), 4.04 (t, *J* = 8.2 Hz, 1H), 3.54 – 3.50 (m, 1H), 2.92 – 2.85 (m, 1H), 2.79 – 2.76 (m, 1H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 152.1, 140.5, 135.5, 133.5, 129.0, 128.6, 128.4, 127.9, 127.1, 116.4, 116.3, 100.8, 67.9, 67.8, 60.4, 44.8 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{18}H_{15}O_3NBr_2Na$ 473.9311; Found 473.9307.

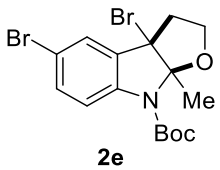
(3a*S*,8a*R*)-3a,5-dibromo-8-((4-nitrophenyl)sulfonyl)-3,3a,8,8a-tetrahydro-2*H*-furo[2,3-*b*]indole (2d)



2d

Light yellow oil, 4 h, 53.9 mg, 50% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.33 (d, *J* = 8.8 Hz, 2H), 8.11 (d, *J* = 9.0 Hz, 2H), 7.50 (d, *J* = 1.8 Hz, 1H), 7.44 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.39 (d, *J* = 8.6 Hz, 1H), 6.20 (s, 1H), 4.04 – 4.01 (m, 1H), 3.41 – 4.37 (m, 1H), 2.86 – 2.81 (m, 1H), 2.75 – 2.72 (m, 1H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 133.5, 128.7, 128.5, 128.4, 128.2, 127.9, 116.4, 116.3, 100.8, 67.9, 67.8, 44.8, 1.0 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{12}O_5N_2Br_2NaS$ 524.8726; Found 524.8707.

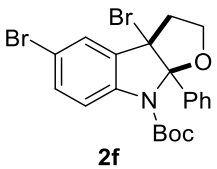
tert-butyl (3a*S*,8a*R*)-3a,5-dibromo-8a-methyl-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]-indole-8-carboxylate (2e)



2e

Light yellow oil, 4 h, 71.0 mg, 82% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.63 (d, *J* = 8.6 Hz, 1H), 7.54 (d, *J* = 2.2 Hz, 1H), 7.33 (dd, *J* = 8.8, 2.1 Hz, 1H), 4.00 – 3.89 (m, 1H), 3.46 – 3.42 (m, 1H), 2.86 – 2.81 (m, 1H), 2.77 – 2.73 (m, 1H), 2.08 (s, 3H), 1.59 (s, 9H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 151.6, 141.0, 133.4, 133.1, 128.0, 116.8, 115.3, 104.2, 82.5, 70.0, 66.2, 46.2, 28.3, 26.3 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{16}H_{19}O_3NBr_2Na$ 453.9624; Found 453.9624.

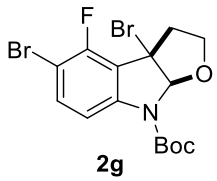
tert-butyl (3a*S*,8a*R*)-3a,5-dibromo-8a-phenyl-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*] indole-8-carboxylate (2f)



2f

Yellow solid, 4 h, 90.9 mg, 92% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.8 Hz, 1H), 7.63 (s, 1H), 7.54 (d, *J* = 2.0 Hz, 1H), 7.48 – 7.30 (m, 4H), 7.15 (s, 1H), 4.26 – 4.15 (m, 1H), 3.70 – 3.66 (m, 1H), 2.92 – 2.87 (m, 1H), 2.82 – 2.76 (m, 1H), 1.14 (s, 9H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 151.5, 141.6, 141.5, 133.2, 133.1, 128.27, 128.2, 127.7, 127.4, 126.8, 125.3, 116.2, 115.5, 107.0, 82.0, 71.0, 66.8, 46.8, 27.6 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{21}O_3NBr_2Na$ 515.9780; Found 515.9776.

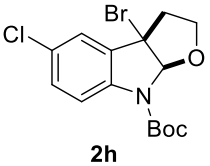
tert-butyl (3a*S*,8a*R*)-3a,5-dibromo-4-fluoro-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]-indole-8-carboxylate (2g)



2g

Light yellow oil, 4 h, 61.1 mg, 70% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.58 (brs, 1H), 7.47 (t, *J* = 7.8 Hz, 1H), 6.15 (brs, 1H), 4.05 (t, *J* = 8.4 Hz, 1H), 3.59 – 3.54 (m, 1H), 3.00 – 2.97 (m, 1H), 2.87 – 2.82 (m, 1H), 1.59 (s, 9H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 154.9 (d, $^{1}J_{C\text{-}F}$ = 250.0 Hz), 151.2, 143.3, 135.4, 119.4, 111.9 (d, $^{3}J_{C\text{-}F}$ = 4.5 Hz), 102.2 (d, $^{2}J_{C\text{-}F}$ = 17.5 Hz), 101.7, 82.8, 68.1, 57.6, 42.8, 28.2 ppm; **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -110.8 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{16}O_3NBr_2FNa$ 457.9373; Found 457.9370.

tert-butyl (3a*S*,8a*R*)-3a-bromo-5-chloro-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]-indole-8-carboxylate (2h)

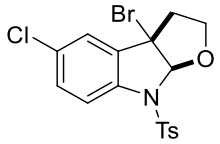


2h

Colorless oil, 5 h, 56.2 mg, 75% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.78 (brs, 1H), 7.37 (d, *J* = 2.2 Hz, 1H), 7.24 (d, *J* = 8.6 Hz, 1H), 6.16 (brs, 1H), 4.01 (t, *J* = 8.3 Hz, 1H), 3.68 – 3.39 (m, 1H), 2.91 – 2.85 (m, 1H), 2.78 – 2.75 (m, 1H), 1.59 (s, 9H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 151.5, 140.5, 133.3, 130.4, 128.6, 124.8, 116.0, 101.0, 82.4, 67.7, 60.6, 44.8, 28.2 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for

$C_{15}H_{17}O_3NBrClNa$ 395.9972; Found 395.9972.

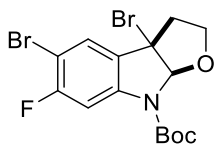
(3a*S*,8a*R*)-3a-bromo-5-chloro-8-tosyl-3,3a,8,8a-tetrahydro-2*H*-furo[2,3-*b*]indole (2i)



2i

White solid, 5 h, 61.7 mg, 72% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.77 (d, *J* = 8.4 Hz, 2H), 7.42 (d, *J* = 8.7 Hz, 1H), 7.30 (d, *J* = 2.2 Hz, 1H), 7.27 (d, *J* = 8.2 Hz, 2H), 7.24 (dd, *J* = 8.6, 2.0 Hz, 1H), 6.21 (s, 1H), 4.04 – 4.01 (m, 1H), 3.47 – 3.42 (m, 1H), 2.85 – 2.80 (m, 1H), 2.71 – 2.68 (m, 1H), 2.38 (s, 3H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 144.7, 139.1, 135.2, 134.2, 130.7, 129.9, 129.8, 127.3, 125.3, 115.3, 103.5, 67.9, 60.2, 44.4, 21.6 ppm; **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{15}O_3NBrClNaS$ 449.9537; Found 449.9532.

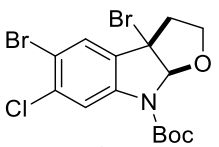
tert-butyl (3a*S*,8a*R*)-3a,5-dibromo-6-fluoro-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]-indole-8-carboxylate (2j)



2j

Light yellow oil, 8 h, 57.7 mg, 66% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.66 (brs, 1H), 7.28 (d, *J* = 6.8 Hz, 1H), 6.16 (brs, 1H), 4.00 (t, *J* = 8.2 Hz, 1H), 3.90 (t, *J* = 7.6 Hz, 1H), 3.48 – 3.43 (m, 1H), 2.34 – 2.27 (m, 1H), 2.06 (dd, *J* = 12.2, 4.6 Hz, 1H), 1.59 (s, 9H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 158.8 (d, $^1J_{C\text{-}F}$ = 241.5 Hz), 151.9, 128.1, 103.5 (d, $^2J_{C\text{-}F}$ = 30.0 Hz), 101.2 (d, $^2J_{C\text{-}F}$ = 22.5 Hz), 94.0, 82.1, 66.4, 44.3, 33.8, 28.3 ppm. **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -106.7 ppm; **HRMS (ESI)** m/z: $[M + Na]^+$ Calcd for $C_{15}H_{16}O_3NBr_2FNa$ 457.9373; Found 457.9363.

tert-butyl (3a*S*,8a*R*)-3a,5-dibromo-6-chloro-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]-indole-8-carboxylate (2k)

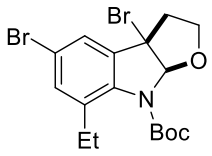


2k

Light yellow oil, 7 h, 55.5 mg, 61% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 8.02 (brs, 1H), 7.61 (s, 1H), 6.15 (brs, 1H), 4.02 (t, *J* = 8.2 Hz, 1H), 3.52 – 3.48 (m, 1H), 2.89 – 2.84 (m, 1H), 2.76 – 2.73 (m, 1H), 1.59 (s, 9H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 151.2, 141.7, 136.4, 132.0, 129.3, 116.6, 115.8, 101.2, 82.8, 67.8, 59.7, 44.8, 28.2;

HRMS (ESI) m/z: [M + Na]$^+$ Calcd for $C_{15}H_{16}O_3NBr_2ClNa$ 473.9078; Found 473.9069.

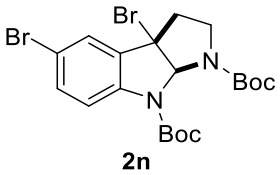
tert-butyl 3a,5-dibromo-7-ethyl-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (2l)



2l

Colorless oil, 4 h, 68.5 mg, 77% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.38 (d, *J* = 2.0 Hz, 1H), 7.32 (d, *J* = 2.0 Hz, 1H), 6.15 (s, 1H), 3.99 – 3.93 (m, 1H), 3.46 – 3.42 (m, 1H), 2.89 – 2.81 (m, 1H), 2.78 – 2.68 (m, 3H), 1.56 (s, 9H), 1.18 (t, *J* = 7.6 Hz, 3H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 152.3, 139.0, 136.6, 136.4, 133.5, 124.6, 118.4, 102.9, 82.5, 67.8, 60.3, 43.2, 28.1, 25.7, 13.2 ppm; **HRMS** (ESI) m/z: [M + Na]+ Calcd for $C_{17}H_{21}O_3NBr_2Na$ 467.9780; Found 467.9760.

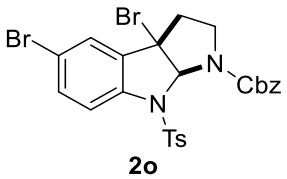
di-tert-butyl (3a*S*,8a*S*)-3a,5-dibromo-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (2n)



2n

Light yellow oil, 4 h, 85.7 mg, 83% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.50 – 7.47 (m, 2H), 7.39 (dd, *J* = 8.6, 2.2 Hz, 1H), 6.43 (s, 1H), 3.78 – 3.75 (m, 1H), 2.85 – 2.80 (m, 1H), 2.75 – 2.67 (m, 2H), 1.58 (s, 9H), 1.48 (s, 9H) ppm.

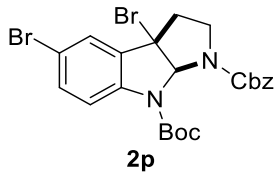
benzyl (3a*S*,8a*S*)-3a,5-dibromo-8-tosyl-3,3a,8,8a-tetrahydropyrrolo[2,3-*b*]indole-1(2*H*)-carboxylate (2o)



2o

Colorless oil, 6 h, 77.1 mg, 64% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.73 – 7.28 (m, 10H), 7.17 (d, *J* = 7.3 Hz, 2H), 6.26 (brs, 1H), 5.30 – 5.19 (m, 2H), 3.85 – 3.81 (m, 1H), 2.86 (s, 1H), 2.70 – 2.59 (m, 2H), 2.34 (s, 3H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.7, 144.6, 140.3, 136.0, 135.5, 134.9, 133.7, 129.6, 128.5, 128.3, 127.8, 127.4, 119.3, 118.4, 87.0, 67.8, 60.6, 45.9, 42.3, 21.6 ppm. **HRMS** (ESI) m/z: [M + Na]$^+$ Calcd for $C_{25}H_{22}O_4N_2Br_2NaS$ 626.9559; Found 626.9552.

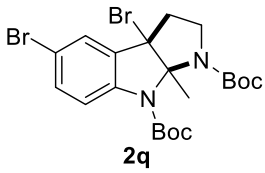
1-benzyl 8-(tert-butyl) (3a*S*,8a*S*)-3a,5-dibromo-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*] indole-1,8-dicarboxylate (2p)



2p

Colorless oil, 8 h, 79.8 mg, 72% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.53 (brs, 1H), 7.47 (d, *J* = 2.0 Hz, 1H), 7.44 – 7.26 (m, 6H), 6.44 (s, 1H), 5.20 – 5.14 (m, 2H), 3.84 – 3.80 (m, 1H), 2.94– 2.89 (m, 1H), 2.79 – 2.70 (m, 2H), 1.53 (s, 9H) ppm; **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.8, 151.7, 141.0, 136.3, 134.4, 133.4, 128.4, 128.1, 128.0, 126.8, 118.8, 116.3, 84.3, 82.6, 67.3, 60.8, 46.3, 41.1, 28.1 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{24}O_4N_2Br_2Na$ 572.9995; Found 572.9978.

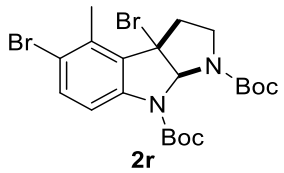
di-tert-butyl (3a*S*,8a*S*)-3a,5-dibromo-8a-methyl-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (2q)



2q

Yellow oil, 24 h, 78.4 mg, 74% yield; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.60 (brs, 1H), 7.46 (d, *J* = 2.2 Hz, 1H), 7.36 (dd, *J* = 8.8, 2.2 Hz, 1H), 3.44 (t, *J* = 9.6 Hz, 1H), 2.95 – 2.90 (m, 1H), 2.81 – 2.77 (m, 1H), 2.66 – 2.60 (m, 1H), 2.12 (s, 3H), 1.59 (s, 9H), 1.41 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 141.3, 134.1, 132.9, 126.0, 119.9, 115.4, 88.5, 82.1, 80.5, 69.2, 45.7, 35.9, 28.4, 28.3, 24.0 ppm. **HRMS** (ESI**)** m/z: $[M + Na]^+$ Calcd for $C_{21}H_{28}O_4N_2Br_2Na$ 553.0308; Found 553.0289.

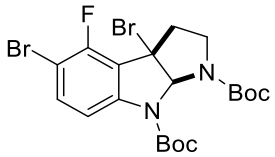
di-tert-butyl (3a*S*,8a*S*)-3a,5-dibromo-4-methyl-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (2r)



2r

Colorless foam oil, 4 h, 76.6 mg, 72% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.48 (d, *J* = 8.7 Hz, 1H), 7.40 (brs, 1H), 6.50 (s, 1H), 3.77 – 3.75 (m, 1H), 2.95 (dd, *J* = 12.4, 4.8 Hz, 1H), 2.81 (td, *J* = 12.0, 4.8 Hz, 1H), 2.68 – 2.62 (m, 1H), 2.51 (s, 3H), 1.57 (s, 9H), 1.49 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.4, 151.8, 141.9, 134.7, 134.1, 130.8, 120.3, 116.0, 84.9, 82.4, 80.8, 63.0, 46.1, 40.8, 28.3, 28.2, 19.2 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{28}O_4N_2Br_2Na$ 553.0308; Found 553.0289.

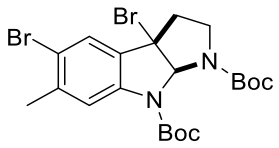
di-tert-butyl (3a*S*,8a*S*)-3a,5-dibromo-4-fluoro-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*] indole-1,8-dicarboxylate (2s)



2s

Yellow foam oil, 6 h, 77.4 mg, 72% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.48 – 7.45 (m, 1H), 7.36 (brs, 1H), 6.44 (s, 1H), 3.82 – 3.79 (m, 1H), 3.03 – 3.00 (m, 1H), 2.92 – 2.88 (m, 1H), 2.67 – 2.61 (m, 1H), 1.58 (s, 1H), 1.48 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 154.7 (d, $^{1}J_{C\text{-}F}$ = 250.5 Hz), 153.2, 151.5, 143.5 (d, $^{3}J_{C\text{-}F}$ = 4.5 Hz), 135.2, 132.1 (d, $^{2}J_{C\text{-}F}$ = 9.0 Hz), 120.4, 113.7, 102.7, 85.0, 82.8, 81.0, 58.8, 46.5, 40.2, 28.3, 28.1 ppm. **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -111.2 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{20}H_{25}O_4N_2Br_2NaF$ 557.0057; Found 557.0053.

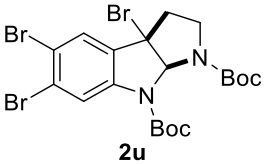
di-tert-butyl (3a*S*,8a*S*)-3a,5-dibromo-6-methyl-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*] indole-1,8-dicarboxylate (2t)



2t

White solid, 5 h,72.3mg, 68% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.56 (br, 1H), 7.49 (s, 1H), 6.41 (s, 1H), 3.76 – 3.73 (m, 1H), 2.85 – 2.80 (m, 1H), 2.74 – 2.65 (m, 2H), 2.39 (s, 3H), 1.58 (s, 9H), 1.48 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.2, 152.0, 141.5, 140.2, 132.1, 127.3, 119.2, 118.8, 84.4, 82.4, 80.8, 61.3, 46.2, 41.8, 28.3, 28.2, 23.6 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{28}O_4N_2Br_2Na$ 553.0308; Found 553.0303.

di-tert-butyl (3a*S*,8a*S*)-3a,5,6-tribromo-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (2u)

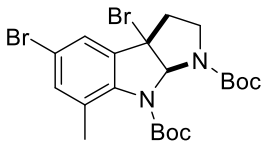


2u

White foam oil, 10 h, 70.4 mg, 61% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.97 (brs, 1H), 7.57 (s, 1H), 6.42 (s, 1H), 3.80 – 3.76 (m, 1H), 2.86 – 2.82 (m, 1H), 2.73 – 2.65 (m, 2H), 1.59 (s, 9H), 1.48 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*)

δ 153.1, 151.5, 142.0, 133.7, 128.4, 126.5, 121.9, 118.5, 84.5, 83.0, 81.0, 60.4, 46.2, 41.9, 28.3, 28.1 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{20}H_{25}O_4N_2Br_3Na$ 616.9257; Found 616.9242.

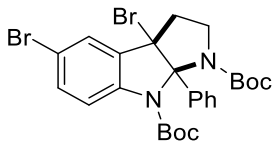
di-tert-butyl (3a*S*,8a*S*)-3a,5-dibromo-7-methyl-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*] indole-1,8-dicarboxylate (2v)



2v

White foam oil, 8 h, 72.8 mg, 68% yield; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.31 (d, *J* = 2.0 Hz, 1H), 7.28 (d, *J* = 2.0 Hz, 1H), 6.22 (brs, 1H), 3.58 – 3.55 (m, 1H), 2.82 – 2.74 (m, 2H), 2.72 – 2.66 (m, 1H), 2.28 (s, 3H), 1.53 (s, 9H), 1.49 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.3, 140.9, 137.2, 135.0, 133.0, 123.4, 118.6, 86.2, 82.3, 80.7, 61.1, 45.5, 37.8, 28.5, 28.1, 19.2 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{28}O_4N_2Br_2Na$ 553.0308; Found 553.0303.

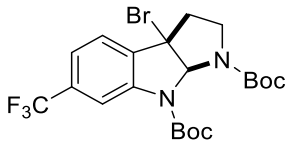
di-tert-butyl (3a*S*,8a*S*)-3a,5-dibromo-8a-phenyl-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (2w)



2w

White foam oil, 10 h, 80.0 mg, 67% yield; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.8 Hz, 1H), 7.66 (d, *J* = 6.1 Hz, 1H), 7.48 (d, *J* = 2.1 Hz, 1H), 7.41 (dd, *J* = 8.8, 2.0 Hz, 1H), 7.35 – 7.32 (m, 3H), 7.15 (d, *J* = 6.6 Hz, 1H), 3.71 – 3.62 (m, 1H), 3.25 – 3.20 (m, 1H), 2.77 – 2.74 (m, 1H), 2.42 – 2.36 (m, 1H), 1.36 (s, 9H), 1.24 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 151.8, 151.6, 141.9, 135.8, 133.6, 133.0, 132.8, 128.1, 126.8, 126.1, 125.5, 125.4, 119.5, 115.1, 92.5, 81.6, 80.2, 71.9, 47.1, 35.4, 28.2, 27.5 ppm. **HRMS** (ESI) m/z: [M + Na]+ Calcd for $C_{26}H_{30}O_4N_2Br_2Na$ 615.0464; Found 615.0449.

di-tert-butyl (3a*S*,8a*S*)-3a-bromo-6-(trifluoromethyl)-2,3,3a,8a-tetrahydro-pyrrolo[2,3-*b*]indole-1,8-dicarboxylate (3y)

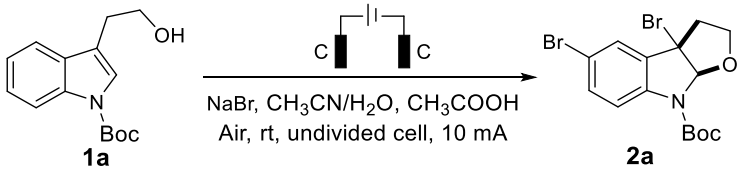


3y

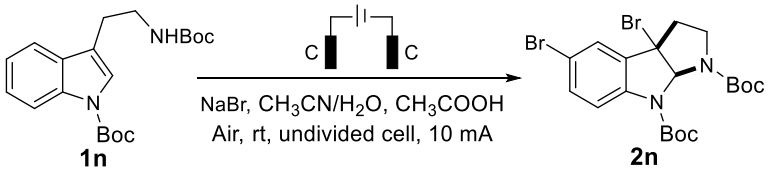
White foam oil, 3 h, 37.6 mg, 37% yield; **^{1}H NMR** (600 MHz, Chloroform-*d*) δ 7.92 (brs, 1H), 7.47 (d, *J* = 8.0 Hz,

1H), 7.36 (d, *J* = 8.0 Hz, 1H), 6.48 (s, 1H), 3.78 (dd, *J* = 10.8, 7.2 Hz, 1H), 2.84 – 2.73 (m, 3H), 1.60 (s, 9H), 1.49 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*)) δ 153.2, 151.6, 142.3, 136.2, 132.4 (q, $^{2}J_{C\text{-}F}$ = 33.0 Hz), 124.3, 123.8 (q, $^{1}J_{C\text{-}F}$ = 271.6 Hz), 120.9, 114.4, 84.2, 82.8, 81.0, 60.7, 46.1, 41.5, 28.3, 28.1 ppm. **^{19}F NMR** (565 MHz, Chloroform-*d*) δ -62.7 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{26}O_4N_2F_3BrNa$ 529.0920; Found 529.0911.

5. Scale-up experiments

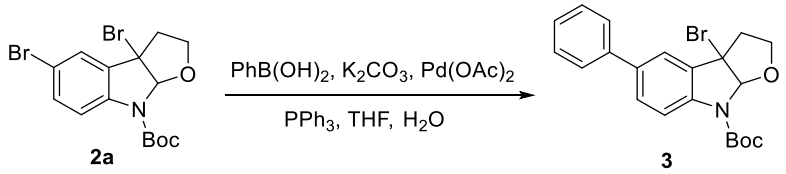


A mixture of **1a** (261.32 mg, 1.0 mmol), NaBr (257.23 mg, 2.5 mmol) and CH_3CN (8 mL), H_2O (150 μL), CH_3CO_2H (100 μL) were added in a 10 mL vial, then the vial was covered with the electrode holder at room temperature, the electrolysis was carried out using a constant current of 10.0 mA a carbon graphite anode and carbon graphite cathode (1.4 × 0.8 × 0.2 cm submerged in solution) with stirring. After completion as detected by TLC. The reaction mixture was diluted with EA. The organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 10/1 to give compound **2a** (295.0 mg, 70% yield).



A mixture of **1a** (360.45 mg, 1.0 mmol), NaBr (257.23 mg, 2.5 mmol) and CH_3CN (8 mL), H_2O (150 μL), CH_3CO_2H (100 μL) were added in a 10 mL vial, then the vial was covered with the electrode holder at room temperature, the electrolysis was carried out using a constant current of 10.0 mA a carbon graphite anode and carbon graphite cathode (1.4 × 0.8 × 0.2 cm submerged in solution) with stirring. After completion as detected by TLC. The reaction mixture was diluted with EA. The organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 10/1 to give compound **2a** (379.8 mg, 73% yield).

6. Synthetic transformations and applications

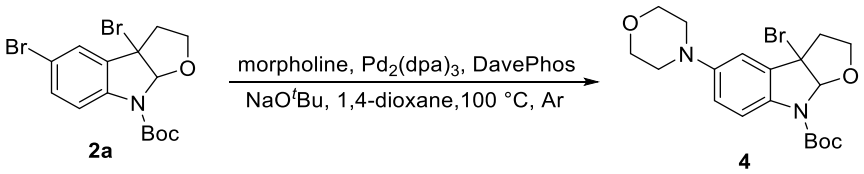


2a **3**

To a solution of **2a** (83.82 mg, 0.2 mmol) in THF (4 mL) were sequentially added $PhB(OH)_2$ (48.78 mg, 0.4 mmol), K_2CO_3 (30.41 mg, 0.22 mmol), $Pd(OAc)_2$ (13.46 mg, 0.06 mmol), PPh_3 (15.72 mg, 0.06 mmol) and H_2O (54 μL, 3.0 mmol) under argon atmosphere, the mixture was stirred at room temperature, the reaction was completed after for 12 h. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, the crude product was purified by flash column chromatography petroleum ether/ethyl acetate to afford the product **3** as colorless oil (60.2 mg, 72% yield).

tert-butyl (3a*S*,8a*R*)-3a-bromo-5-phenyl-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*] indole-8-carboxylate (3)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (brs, 1H), 7.63 (s, 1H), 7.57 (d, *J* = 7.6 Hz, 2H), 7.53 (d, *J* = 8.2 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 1H), 6.22 (brs, 1H), 4.03 (t, *J* = 8.4 Hz, 1H), 3.57 – 3.53 (m, 1H), 2.96 – 2.91 (m, 1H), 2.87 – 2.84 (m, 1H), 1.62 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 140.2, 137.0, 129.4, 128.8, 127.2, 126.8, 115.1, 101.1, 67.8, 45.1, 28.3 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{21}H_{22}O_3NBrNa$ 438.0675; Found 438.0672.



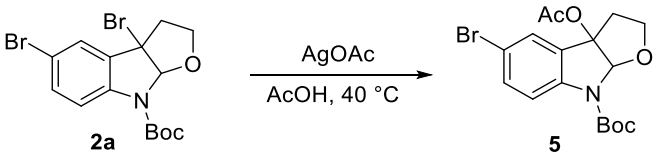
2a **4**

A mixture of **2a** (83.82 mg, 0.2 mmol), DavePhos (11.8 mg, 0.03 mmol), $Pd_2(dba)_3$ (18.3 mg, 0.02 mmol), NaO^tBu (38.4 mg, 0.4 mmol) and morpholine (52.4 μL, 0.6 mmol) were dissolved in 1,4-dioxane (2 mL) under argon atmosphere, then the mixture was stirred at 100 ℃ and the reaction was completed as monitored by

TLC. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 2/1 to give **4** as a yellow oil (25.0 mg, 29% yield).

tert-butyl 3a-bromo-5-morpholino-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (4)

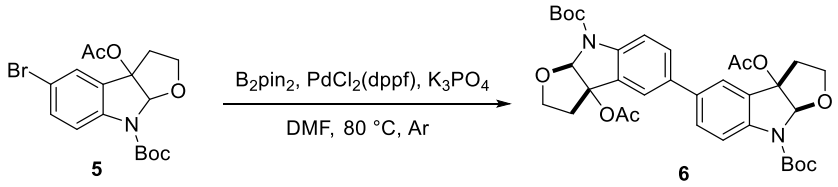
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.71 (brs, 1H), 6.80 (s, 2H), 6.16 (br, 1H), 3.98 (t, *J* = 8.0 Hz, 1H), 3.91 – 3.82 (m, 4H), 3.48 – 3.44 (m, 1H), 3.13 – 3.06 (m, 4H), 2.37 – 2.24 (m, 1H), 2.10 – 2.08 (m, 1H), 1.58 (s, 9H) ppm.**^{13}C NMR** (150 MHz, Chloroform-*d*) δ 147.7, 136.7, 132.1, 128.4, 116.0, 114.8, 112.8, 93.5, 81.0, 66.9, 66.3, 50.5, 45.3, 33.7, 28.4 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{19}H_{26}O_4N_2Br$ 425.1070; Found 425.1073.



To a solution of **2a** (83.82 mg, 0.2 mmol) and AgOAc (36.72 mg, 0.22 mmol) in AcOH was heated to 40 ℃ in the dark, the reaction was completed as monitored by TLC, after 4 h. The reaction was extracted with EA, the organic phase was washed with brine and dried over Na_2SO_4, purified by flash column chromatography to afford the product **5** as a white foam oil (66.3 mg, 83%yield).

tert-butyl 3a-acetoxy-5-bromo-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (5)

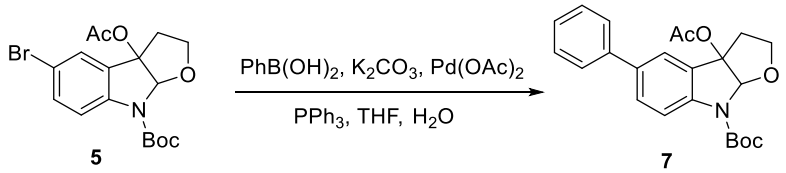
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.77 (brs, 1H), 7.61 (d, *J* = 2.0 Hz, 1H), 7.41 (d, *J* = 8.0 Hz, 1H), 6.06 (brs, 1H), 4.10 (t, *J* = 8.8 Hz, 1H), 3.57 – 3.53 (m, 1H), 2.68 –2.57 (m, 2H), 2.06 (s, 3H), 1.58 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 169.8, 151.8, 140.3, 133.5, 130.5, 128.1, 116.3, 115.3, 96.7, 90.7, 82.1, 67.0, 39.7, 28.3, 21.3 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{17}H_{20}O_5NBrNa$ 420.0417; Found 420.0402.



A mixture of **5** (39.83 mg, 0.1 mmol), K_3PO_4 (63.68 mg, 0.3 mmol), $PdCl_2$(dppf) (7.26 mg, 0.01 mmol), and B_2Pin_2 (27.93 mg, 0.11 mmol) were dissolved in DMF (3 mL) under argon atmosphere, the mixture was heated to 80 ℃ and the reaction was monitored by TLC. The mixture was extracted with EA, the organic phase was washed with brine, dried over Na_2SO_4, purified by flash column chromatography petroleum ether/ethyl acetate= 4/1 to give compound **6** as a colorless oil (29.5 mg, 46% yield).

di-tert-butyl (3a*S*,3'a*S*,8a*R*,8'a*R*)-3a,3'a-diacetoxy-2,2',3,3a,3',3'a,8a,8'a octahy-dro-8*H*,8'*H*-[5,5'-bifuro[2,3-*b*]indole]-8,8'-dicarboxylate (6)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.92 (brs, 2H), 7.67 (s, 2H), 7.52 (d, *J* = 8.0 Hz, 2H), 6.12 (brs, 2H), 4.12 (t, *J* = 8.4 Hz, 2H), 3.63 – 3.58 (m, 2H), 2.78 – 2.73 (m, 2H), 2.68 – 2.63 (m, 2H), 2.07 (s, 3H), 2.06 (s, 3H), 1.61 (s, 18H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 169.8, 152.0, 143.4, 138.9, 135.6, 129.2, 123.2, 115.0, 96.8, 91.3, 81.7, 67.0, 39.7, 28.3, 21.4 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{34}H_{40}O_{10}N_2Na$ 659.2575; Found 659.2574.

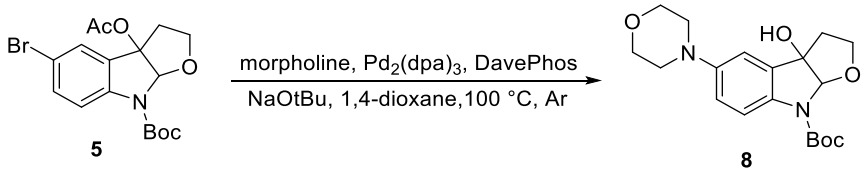


To a solution of **5** (79.65 mg, 0.2 mmol) in THF (4 mL) were sequentially added $PhB(OH)_2$ (48.78 mg, 0.4 mmol), K_2CO_3 (30.41 mg, 0.22 mmol), $Pd(OAc)_2$ (13.46 mg, 0.06 mmol), PPh_3 (15.72 mg, 0.06 mmol) and H_2O (54 μL, 3.0 mmol) under argon atmosphere, the mixture was stirred at room temperature, the reaction was completed after for 12 h. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over

Na_2SO_4 and concentrated under reduced pressure, the crude product was purified by silica gel column chromatography on silica gel to afford the product **7** as a colorless oil (44.3 mg, 56% yield).

tert-butyl 3a-acetoxy-5-phenyl-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (7)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.94 (brs, 1H), 7.75 (s, 1H), 7.61 – 7.51 (m, 3H), 7.42 (t, *J* = 7.7 Hz, 2H), 7.31 (t, *J* = 7.4 Hz, 1H), 6.12 (brs, 1H), 4.12 (t, *J* = 8.5 Hz, 1H), 3.63 – 3.58 (m, 1H), 2.78 – 2.75 (m, 1H), 2.67 – 2.62 (m, 1H), 2.05 (s, 3H), 1.61 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 169.8, 151.9, 143.5, 140.4, 136.3, 129.5, 128.7, 127.0, 126.7, 123.7, 114.9, 96.8, 91.3, 81.7, 67.1, 39.6, 28.3, 21.3 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{23}H_{25}O_5NNa$ 418.1625; Found 418.1614.

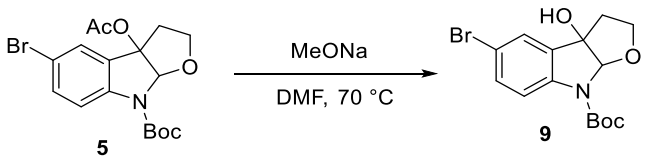


A mixture of **5** (79.65 mg, 0.2 mmol), DavePhos (11.8 mg, 0.03 mmol), $Pd_2(dba)_3$ (18.3 mg, 0.02 mmol), NaO^{t}Bu (38.4 mg, 0.4 mmol) and morpholine (52.4 μL, 0.6 mmol) were dissolved in 1,4-dioxane (2 mL) under argon atmosphere, then the mixture was stirred at 100 ℃ and the reaction was completed as monitored by TLC. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 2/1 to give compound **8** as a yellow oil (50.0 mg, 69% yield).

tert-butyl 3a-hydroxy-5-morpholino-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (8)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (brs, 1H), 6.93 (s, 1H), 6.87 (d, *J* = 8.8 Hz, 1H), 5.70 (s, 1H), 4.03 (t, *J* = 8.4 Hz, 1H), 3.84 – 3.78 (m, 4H), 3.56 – 3.50 (m, 1H), 3.47 (s, 1H), 3.06 – 2.98 (m, 4H), 2.51 – 2.46 (m, 1H), 2.34 – 2.32 (m, 1H), 1.56 (s, 9H) ppm. **^{13}C NMR** (150 MHz, $CDCl_3$) δ 152.3, 147.8, 136.8, 132.4, 118.7, 115.4, 111.7, 98.6, 86.7, 81.4, 67.7, 66.7, 50.4, 40.4, 28.3 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd

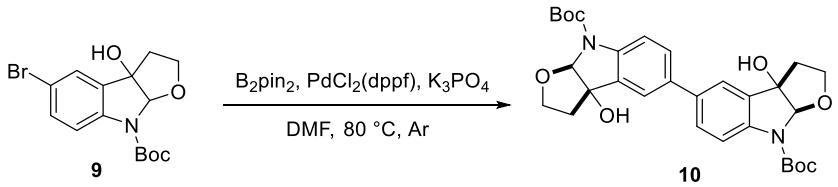
for $C_{19}H_{26}O_5N_2Na$ 385.1733; Found 385.1728.



To a solution of **5** (79.65 mg, 0.2 mmol) in dry DMF (4 mL) and was added MeONa in MeOH (4.0 M, 0.8 mL), the reaction was heated to 70 ℃ under argon atmosphere, the reaction was completed as monitored by TLC, after 1 h. The reaction was extracted with EA, the organic phase was washed with brine and dried over Na_2SO_4, purified by column chromatography on silica gel to afford the product **9** as a white foam oil (55.8 mg, 78% yield).

tert-butyl 5-bromo-3a-hydroxy-2,3,3a,8a-tetrahydro-8*H*-furo[2,3-*b*]indole-8-carboxylate (9)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.67 (brs, 1H), 7.50 (s, 1H), 7.40 (d, *J* = 8.6 Hz, 1H), 5.73 (s, 1H), 4.04 (t, *J* = 8.4 Hz, 1H), 3.56 – 3.49 (m, 1H), 2.98 (s, 1H), 2.52 – 2.46 (m, 1H), 2.35 – 2.32 (m, 1H), 1.56 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 152.1, 142.0, 133.7, 133.2, 127.1, 116.4, 115.5, 98.5, 86.3, 82.2, 67.7, 40.7, 28.3 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{15}H_{18}O_4NBrNa$ 378.0311; Found 378.0308.

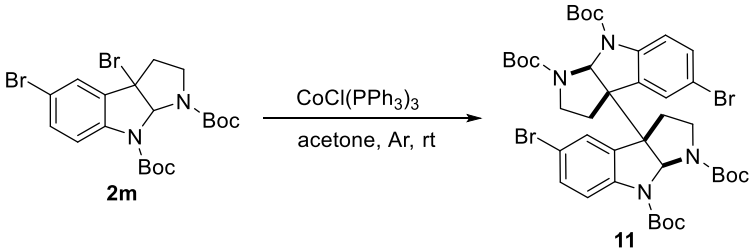


A mixture of **9** (71.24 mg, 0.2 mmol), K_3PO_4 (127.4 mg, 0.6 mmol), $PdCl_2$(dppf) (14.52 mg, 0.02 mmol), and B_2Pin_2 (55.86 mg, 0.22 mmol) were dissolved in DMF (3 mL) under argon atmosphere, the mixture was heated to 80 ℃ and the reaction was monitored by TLC. The mixture was extracted with EA, the organic phase was washed with brine, dried over Na_2SO_4, purified by flash column chromatography petroleum ether/ethyl acetate= 3/1 to give compound **10** as a colorless oil (45.6 mg, 41% yield).

di-tert-butyl (3a*S*,3'a*S*,8a*R*,8'a*R*)-3a,3'a-dihydroxy-2,2',3,3a,3',3'a,8a,8'a

octahy-dro-8*H*,8'*H*-[5,5'-bifuro[2,3-*b*]indole]-8,8'-dicarboxylate (10)

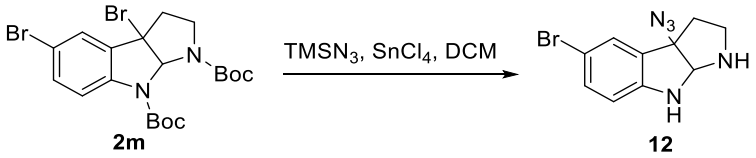
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (brs, 2H), 7.59 (d, *J* = 9.2 Hz, 2H), 7.50 (d, *J* = 8.4 Hz, 2H), 5.81 (brs, 2H), 4.10 – 4.08 (m, 2H), 3.61 – 3.56 (m, 2H), 2.63 (br, 2H), 2.59 – 2.53 (m, 2H), 2.45 – 2.40 (m, 2H), 1.60 (s, 18H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.4, 151.5, 142.2, 133.4, 132.0, 126.1, 117.2, 115.1, 82.5, 80.6, 78.8, 60.5, 45.1, 28.4, 28.3 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{30}H_{36}O_8N_2Na$ 575.2364; Found 575.2345.



Under an argon atmosphere, freshly prepared $CoCl(PPh_3)_3$ (105.75 mg, 0.12 mmol) was added quickly to a degassed (nitrogen bubbling for 10 min) solution of **2m** (51.82 mg, 0.1 mmol) in dry acetone (1.0 mL). After 15 min, the reaction mixture was diluted with DCM and quenched with saturated NH_4Cl solution. The organic layer was separated and the aqueous layer was extracted with DCM. the organic phase was washed with brine, dried over Na_2SO_4, purified by flash column chromatography petroleum ether/ethyl acetate= 10/1 to give **11** as a Yellow oil (25.6 mg, 29% yield).

tetra-tert-butyl (3a*R*,3'a*R*,8a*S*,8'a*S*)-5,5'-dibromo-2,2',3,3'-tetrahydro-1*H*,1'*H*-[3a,3'a-bipyrrolo[2,3-*b*]indole]-1,1',8,8'(8a*H*,8'a*H*)-tetracarboxylate (11)

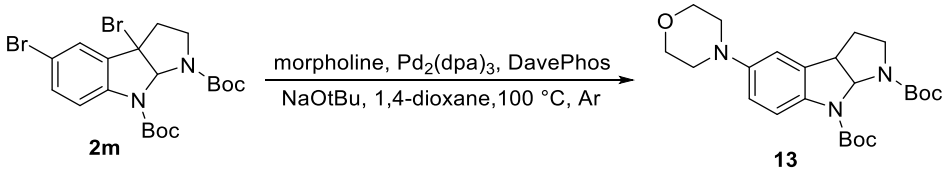
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.49 (dd, *J* = 8.0, 1.2 Hz, 2H), 7.32 (dd, *J* = 7.6, 1.2 Hz, 2H), 7.07 (t, *J* = 7.8 Hz, 2H), 6.19 (s, 2H), 3.59 – 3.53 (m, 2H), 2.81 – 2.74 (m, 4H), 2.74 – 2.66 (m, 2H), 1.54 (s, 18H), 1.51 (s, 18H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.4, 151.5, 142.2, 133.4, 132.0, 126.1, 117.2, 115.1, 82.5, 80.6, 78.8, 60.5, 45.1, 34.7, 28.4, 28.3 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{40}H_{52}N_4Br_2O_8Na$ 897.2044; Found 897.2028.



Under an argon atmosphere, $SnCl_4$ (11.3 μL, 1.0 mmol) was added to a solution of bromide **2m** (51.82 mg, 0.1 mmol) and trimethylsilylazide (0.26 mL, 1.0 mmol) in dry DCM (1.0 mL) by syringe at room temperature. After stirring at room temperature for 12h, the reaction mixture was cooled to 0 ℃ and quenched by saturated $NaHCO_3$ solution. The reaction mixture was diluted with water and extracted with DCM. The combined organic layers were dried over anhydrous Na_2SO_4, the solvent was concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 10/1 to give **12** as a yellow solid (9.9 mg, 35% yield).

3a-azido-5-bromo-1,2,3,3a,8,8a-hexahydropyrrolo[2,3-*b*]indole (12)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.33 (d, *J* = 2.0 Hz, 1H), 7.25 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.52 (d, *J* = 8.6 Hz, 1H), 5.14 (s, 1H), 3.17 – 3.14 (m, 1H), 2.86 – 2.81 (m, 1H), 2.26 – 2.23 (m, 1H), 2.18 – 2.13 (m, 1H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 149.2, 133.1, 129.1, 127.2, 111.2, 110.7, 84.5, 79.0, 45.8, 40.7 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{10}H_{11}N_5Br$ 280.0192; Found 280.0194.

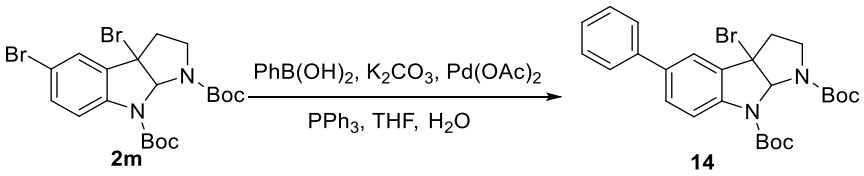


A mixture of **2m** (51.82 mg, 0.1 mmol), DavePhos (11.8 mg, 0.03 mmol), $Pd_2(dba)_3$ (18.3 mg, 0.02 mmol), NaO^tBu (38.4 mg, 0.4 mmol) and morpholine (52.4 μL, 0.6 mmol) were dissolved in 1,4-dioxane (2 mL) under argon atmosphere, then the mixture was stirred at 100 ℃ and the reaction was completed as monitored by TLC. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 10/1 to give compound **13** as a yellow oil (22.4 mg, 50% yield).

di-tert-butyl

5-morpholino-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarb-onxylate (13)

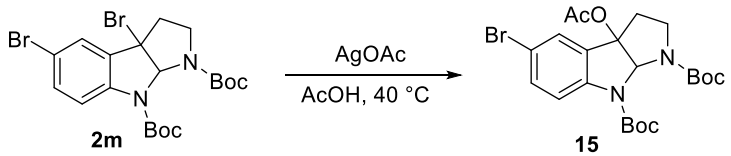
^{1}H NMR (600 MHz, Chloroform-*d*) δ 7.50 (br, 1H), 6.76 (d, *J* = 8.4 Hz, 2H), 6.41 (s, 1H), 3.95 (t, *J* = 7.3 Hz, 1H), 3.89 – 3.84 (m, 4H), 3.81 – 3.77 (m, 1H), 3.18 – 3.04 (m, 4H), 2.87 – 2.82 (m, 1H), 2.13 – 1.97 (m, 1H), 1.56 (s, 9H), 1.49 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 154.0, 152.6, 147.9, 136.7, 133.1, 128.4, 115.8, 112.4, 81.1, 80.0, 76.2, 66.9, 50.5, 45.4, 44.9, 29.7, 28.4 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{24}H_{35}O_3N_5Na$ 468.2469; Found 468.2474.



To a solution of **2m** (103.65 mg, 0.2 mmol) in THF (4 mL) were sequentially added $PhB(OH)_2$ (48.78 mg, 0.4 mmol), K_2CO_3 (30.41 mg, 0.22 mmol), $Pd(OAc)_2$ (13.46 mg, 0.06 mmol), PPh_3 (15.72 mg, 0.06 mmol) and H_2O (54 μL, 3.0 mmol) under argon atmosphere, the mixture was stirred at room temperature, the reaction was completed after for 12 h. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, the crude product was purified by silica gel column chromatography on silica gel to afford the product **14** as colorless oil (81.4 mg, 79% yield).

di-tert-butyl 3a-bromo-5-phenyl-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (14)

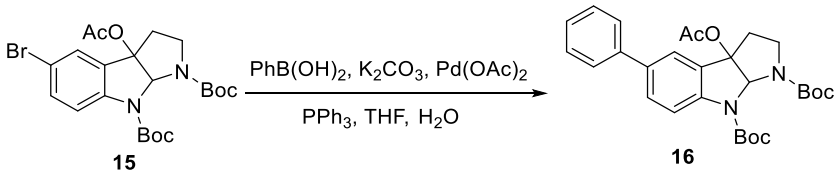
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (brs, 1H), 7.58 – 7.56 (m, 3H), 7.53 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.34 (t, *J* = 7.4 Hz, 1H), 6.48 (s, 1H), 3.79 – 3.75 (m, 1H), 2.89 – 2.83 (m, 2H), 2.79 – 2.71 (m, 1H), 1.60 (s, 9H), 1.50 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 153.4, 152.1, 141.4, 140.3, 137.4, 133.2, 129.3, 128.8, 127.3, 126.8, 122.4, 117.6, 84.2, 82.2, 80.8, 62.2, 46.2, 42.1, 28.4, 28.3 ppm. **HRMS** (ESI) m/z: $[M + Na]^+$ Calcd for $C_{26}H_{31}O_4N_2BrNa$ 537.1359; Found 537.1346.



To a solution of **2m** (103.65 mg, 0.2 mmol) and AgOAc (36.72 mg, 0.22 mmol) in AcOH(20 mL) was heated to 40 ℃ in the dark, the reaction was completed as monitored by TLC, after 4 h. The reaction was extracted with EA, the organic phase was washed with brine and dried over Na_2SO_4, purified by column chromatography on silica gel to afford the product **15** as a white foam oil (81.7 mg, 82% yield)

di-tert-butyl 3a-acetoxy-5-bromo-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (15)

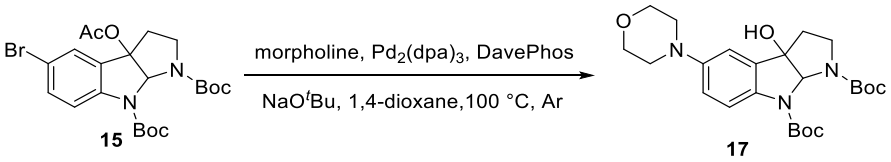
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 – 7.50 (m, 1H), 7.43 (dd, *J* = 8.6, 2.0 Hz, 1H), 6.42 (s, 1H), 3.95 – 3.92 (m, 1H), 2.85 – 2.80 (m, 1H), 2.62 – 2.56 (m, 1H), 2.43 – 2.37 (m, 1H), 2.05 (s, 3H), 1.59 (s, 9H), 1.51 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 169.5, 153.7, 152.0, 143.7, 133.4, 130.9, 127.7, 117.9, 115.4, 90.5, 82.1, 80.6, 80.1, 77.2, 77.0, 76.8, 44.9, 28.3, 28.3, 21.4 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{22}H_{29}O_6N_2BrNa$ 519.1101; Found 519.1086.



To a solution of **15** (99.48 mg, 0.2 mmol) in THF (2 mL) were sequentially added $PhB(OH)_2$ (48.8 mg, 0.4 mmol), K_2CO_3 (30.41 mg, 0.22 mmol), $Pd(OAc)_2$ (13.5 mg, 0.06 mmol), PPh_3 (15.7 mg, 0.06 mmol) and H_2O (54 μL, 3.0 mmol) under argon atmosphere, the mixture was stirred at room temperature, the reaction was completed after for 12 h. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, the crude product was purified by silica gel column chromatography on silica gel to afford the product **16** as a colorless oil (53.8 mg, 54% yield).

di-tert-butyl 3a-acetoxy-5-phenyl-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (16)

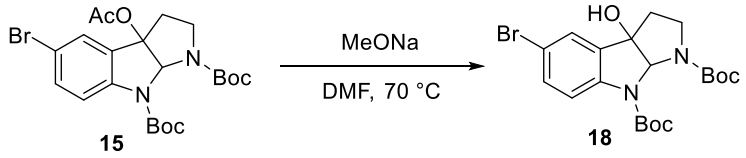
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 – 7.64 (m, 2H), 7.57 – 7.49 (m, 3H), 7.42 (t, *J* = 7.8 Hz, 2H), 7.31 (t, *J* = 7.3 Hz, 1H), 6.45 (s, 1H), 3.94 – 3.91 (m, 1H), 2.89 – 2.85 (m, 1H), 2.71 – 2.68 (m, 1H), 2.47 – 2.42 (m, 1H), 2.02 (s, 3H), 1.60 (s, 9H), 1.51 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 169.6, 153.8, 152.3, 143.9, 140.4, 136.5, 129.5, 129.4, 128.7, 127.0, 123.4, 120.1, 115.3, 91.0, 81.9, 80.1, 62.9, 45.1, 28.4, 28.3, 26.9, 21.4 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{28}H_{34}O_6N_2Na$ 517.2309; Found 517.2299.



A mixture of **15** (99.48 mg, 0.2 mmol), DavePhos (11.8 mg, 0.03 mmol), $Pd_2(dba)_3$ (18.3 mg, 0.02 mmol), NaO^tBu (38.4 mg, 0.4 mmol) and morpholine (52.4 μL, 0.6 mmol) were dissolved in 1,4-dioxane (2 mL) under argon atmosphere, then the mixture was stirred at 100 ℃ and the reaction was completed as monitored by TLC. The solution was washed with water and the aqueous layer was extracted with DCM, the organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure, purified by flash column chromatography petroleum ether/ethyl acetate= 2/1 to give compound **17** as a yellow oil (36.0 mg, 40% yield).

di-tert-butyl 3a-hydroxy-5-morpholino-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (17)

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (brs, 1H), 6.93 (s, 1H), 6.87 (dd, *J* = 8.8, 2.6 Hz, 1H), 6.01 (s, 1H), 3.84 – 3.80 (m, 5H), 3.35 (br, 1H), 3.09 – 3.03 (m, 4H), 2.83 – 2.79 (m, 1H), 2.32 – 2.25 (m, 2H), 1.54 (s, 9H), 1.47 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 154.0, 152.6, 147.9, 136.7, 133.1, 118.4, 117.0, 111.3, 86.4, 81.8, 81.4, 80.2, 77.2, 77.0, 76.8, 66.7, 50.4, 45.5, 38.6, 28.3 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{24}H_{35}O_6N_3Na$ 484.2418; Found 484.2398.



To a solution of **15** (99.48 mg, 0.2 mmol) in dry DMF (4 mL) was added MeONa in MeOH (4.0 M, 0.8 mL), the reaction was heated to 70 ℃ under argon atmosphere, the reaction was completed as monitored by TLC, after 1 h. The reaction was extracted with EA, the organic phase was washed with brine and dried over Na_2SO_4, purified by column chromatography on silica gel to afford the product **18** as a white foam oil (65.6 mg, 83% yield)

di-tert-butyl 5-bromo-3a-hydroxy-2,3,3a,8a-tetrahydropyrrolo[2,3-*b*]indole-1,8-dicarboxylate (18)

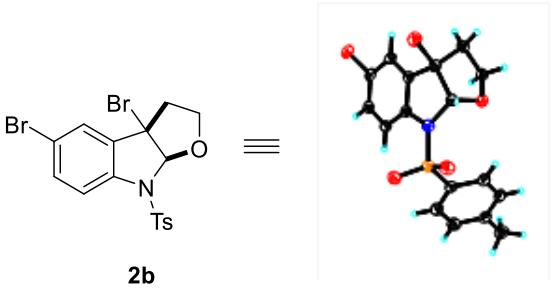
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (brs, 1H), 7.47 (d, *J* = 2.1 Hz, 1H), 7.39 (dd, *J* = 8.7, 2.1 Hz, 1H), 6.04 (s, 1H), 3.85 – 3.82 (m, 1H), 2.84 – 2.80 (m, 1H), 2.34 – 2.22 (m, 2H), 1.53 (s, 9H), 1.46 (s, 9H) ppm. **^{13}C NMR** (150 MHz, Chloroform-*d*) δ 154.0, 152.3, 142.1, 134.4, 133.0, 126.7, 117.8, 115.6, 86.0, 82.2, 81.9, 80.5, 45.6, 38.9, 28.3, 28.3 ppm. **HRMS** (ESI) *m/z*: $[M + Na]^+$ Calcd for $C_{20}H_{27}O_5N_2BrNa$ 477.0996; Found 477.0981.

7. X-ray Dates

Single-crystal X-ray diffraction measurements were carried out on an Agilent Gemini E diffractometer using graphite monochromated Mo radiation (λ = 0.71073 Å, 50kV 40mA). The crystal data was collected on a Agilent Gemini E diffractometer (Mo, 50kV 40mA) and reducted by CrysAlisPro (Rigaku). The structures were solved by direct methods using SHELXS-97. Refinements were performed with SHELXL-2013 using fullmatrix least-squares calculations on F2, with anisotropic displacement parameters for all the nonhydrogen atoms.

The single crystals of compound **2b** suitable for X-ray diffraction analysis were obtained by slow evaporation of a mixed THF/Petroleum ether solution of **2b** at room temperature.

Molecular structure of **2b**, ellipsoids shown at 30% probability.



2b

CCDC: 2288080

Crystal data and structure refinement for CCDC: 2288080.

Identification code global

Bond precision: C-C = 0.0055 A Wavelength=1.54178

Cell: a=8.1543(2) b=10.4250(2) c=12.7925(3) alpha=66.467(1) beta=77.140(1) gamma=80.707(1)

Temperature: 150 K Calculated Reported

Volume 968.81(4) 968.81(4)

Space group P -1 P -1

Hall group -P 1 -P 1

Moiety formula C17 H15 Br2 N O3 S [+ solvent] C34 H30 Br4 N2 O6 S2

Sum formula C17 H15 Br2 N O3 S [+ solvent] C34 H30 Br4 N2 O6 S2

Mr 473.16 946.36

Dx, g cm-3 1.622 1.622

Z 2 1

Mu (mm-1) 6.439 6.439

F000 468.0 468.0

F000’ 466.91

h, k, lmax 9,12,15 9,12,15

Nref 3565 3541

Tmin, Tmax 0.524, 0.637 0.190,0.660

Tmin’ 0.387

Correction method= # Reported T Limits: Tmin=0.190 Tmax=0.660 AbsCorr = MULTI-SCAN Data completeness= 0.993

Theta (max)= 68.320

8. References

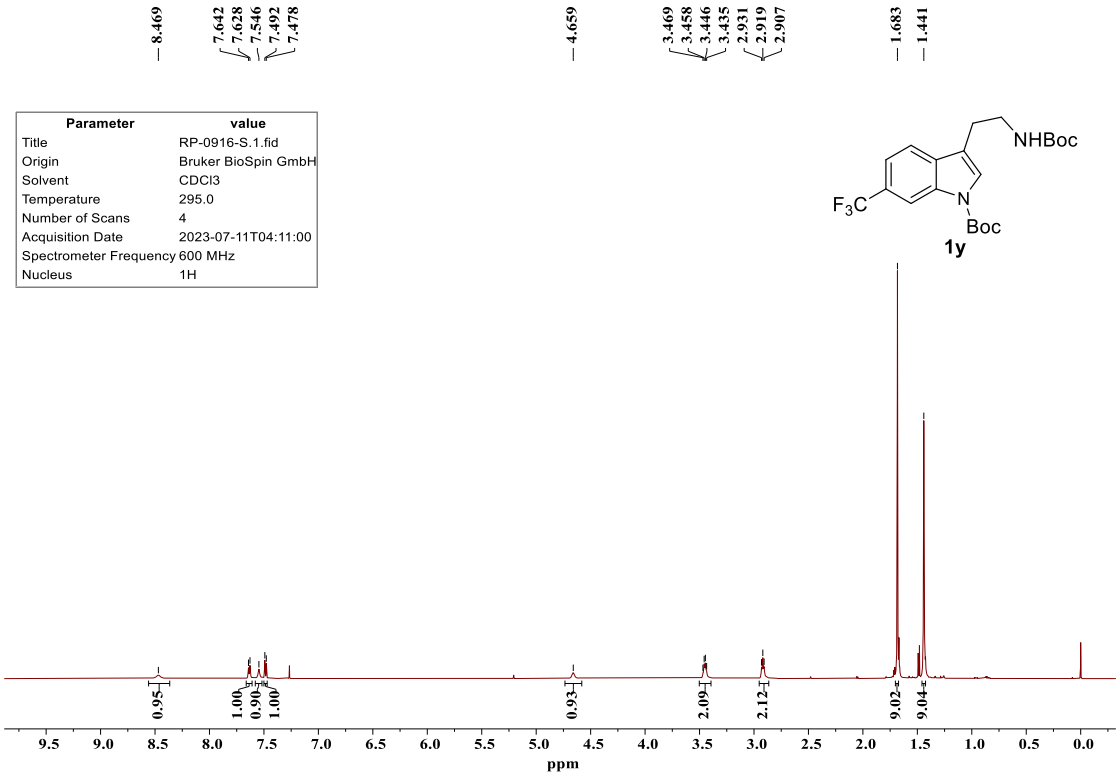
[1] Y.-A. Wu, R.-A. Wang, S.-Y. Jiang, T.-B. Jiang, J.-R. Song, J. Shi, W. Wu, W.-D. Pan and H. Ren, *Green Chem.* 2022, **24**, 6720–6726.

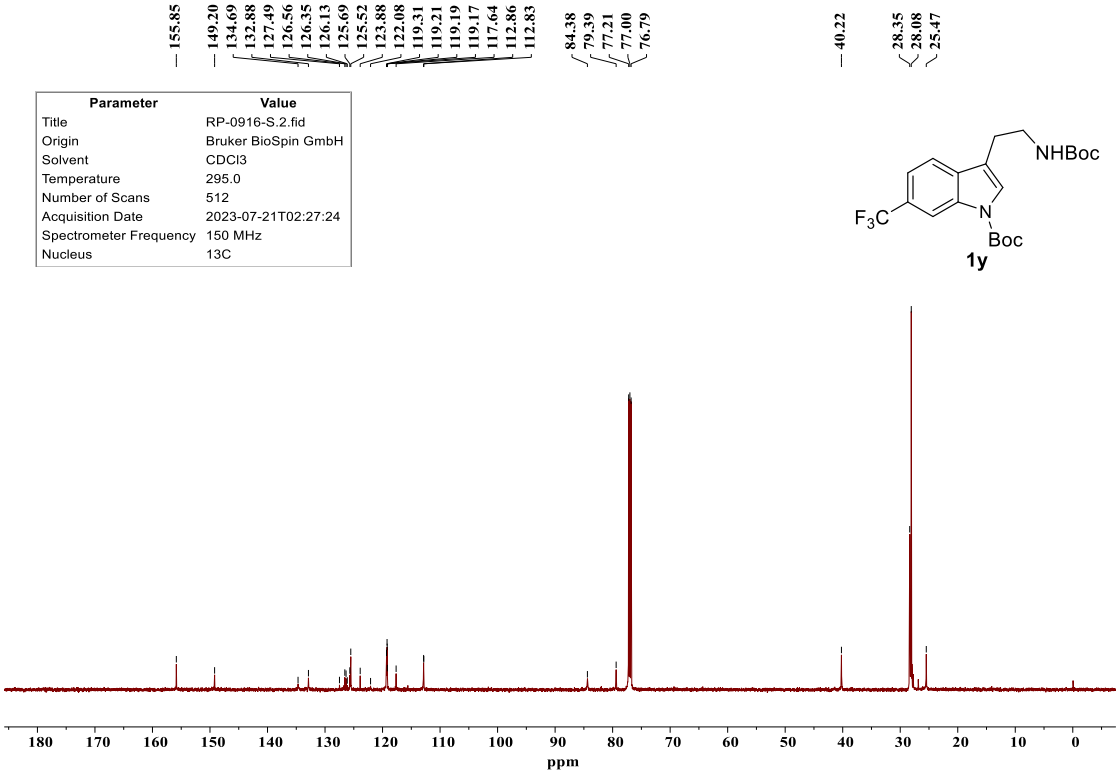
[2] T.-T. Sun, K. Liu, S.-X. Zhang, C.-R. Wang, C.-Z. Yao and J. Yu, *Synlett.* 2021, **32**, 701–707.

[3] A.-E. Blom, J.-Y. Su, L.-M. Repka, S.-E. Reisman and D. A. Dougherty, *ACS Medicinal Chem. Lett.* 2020, **11**, 2204–2211.

[4] W. Xie, G. Jiang, H. Liu, J. Hu, X. Pan, H. Zhang, X.-L. Wang, Y.-S. Lai and D. Ma, *Angew. Chem. Int. Ed.* 2013, **52**, 12924–12927.

9. ^{1}H NMR, ^{13}C NMR and ^{19}F NMR spectra of compounds





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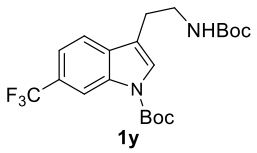

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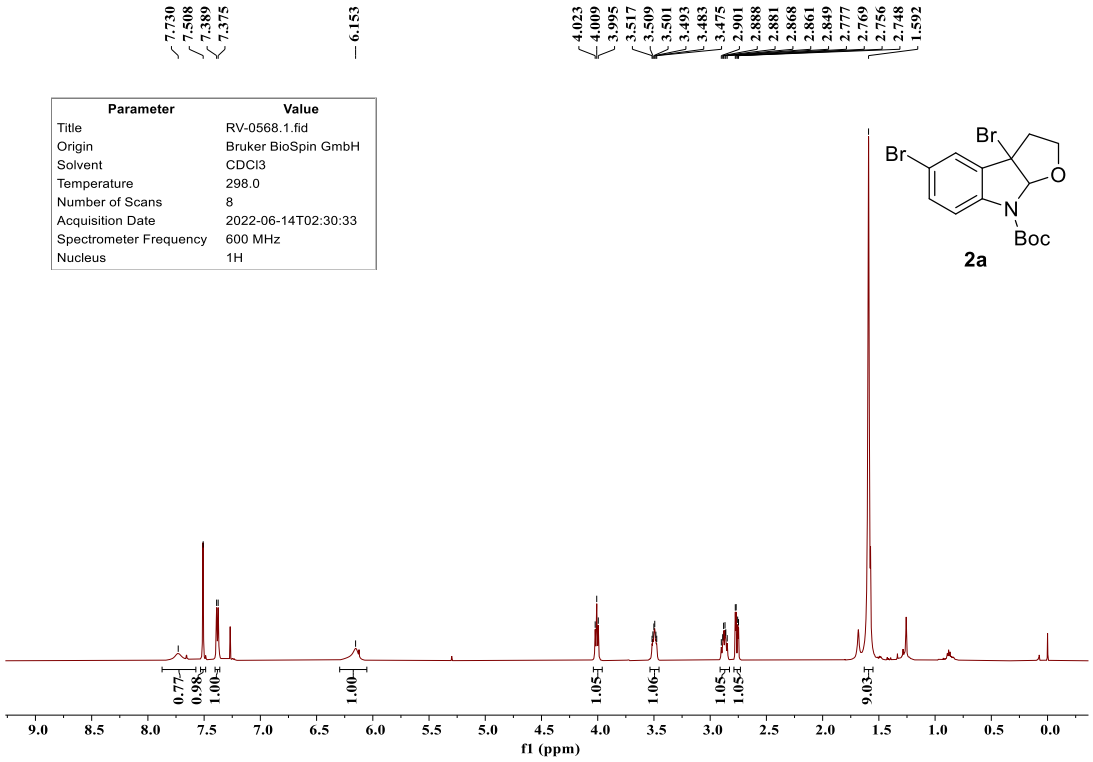
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Parameter
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2022-06-14T02:30:33
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Br
Br
O
N
Boc
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1.05
1.05
9.03
9.0
8.5
8.0
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7.0
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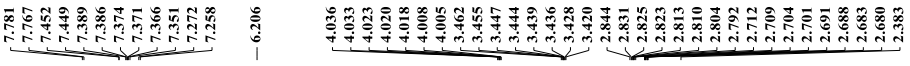
f1 (ppm)



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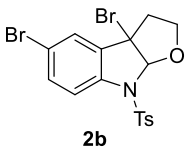
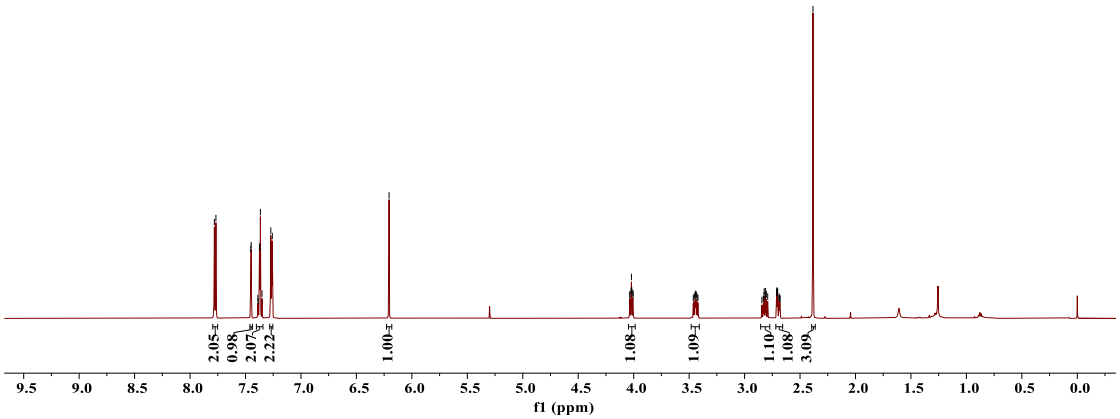
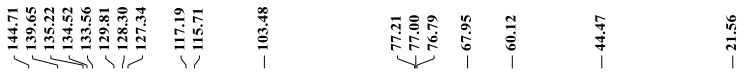


2b





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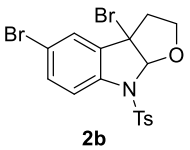

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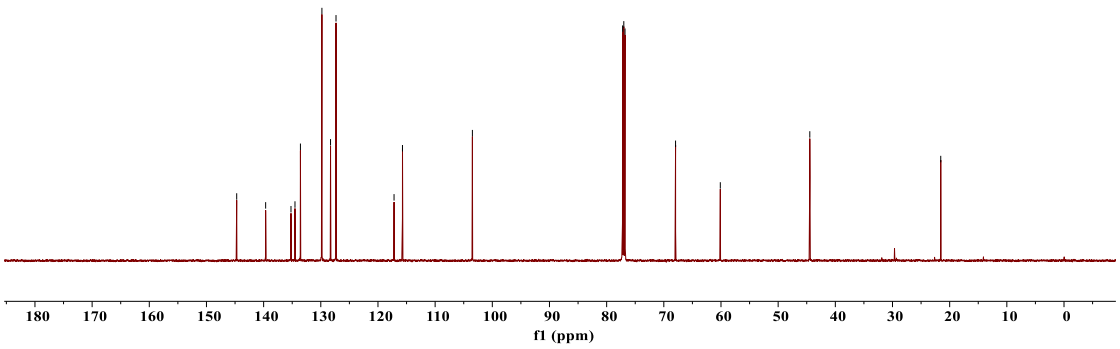

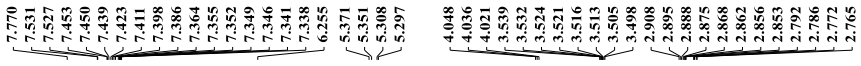

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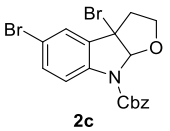

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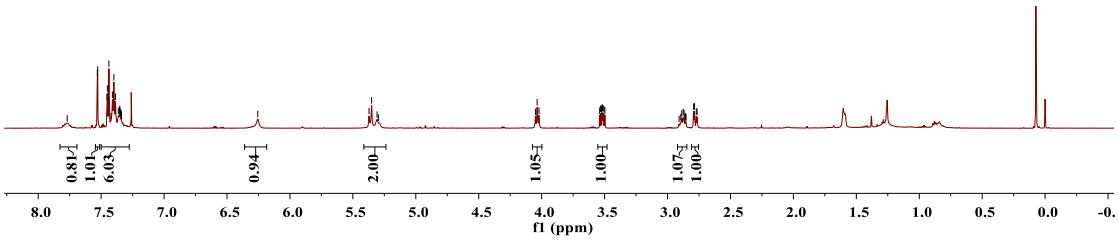

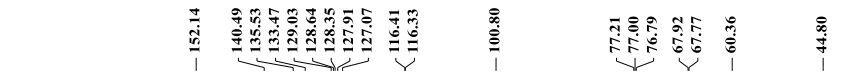

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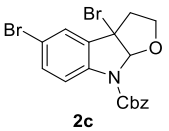

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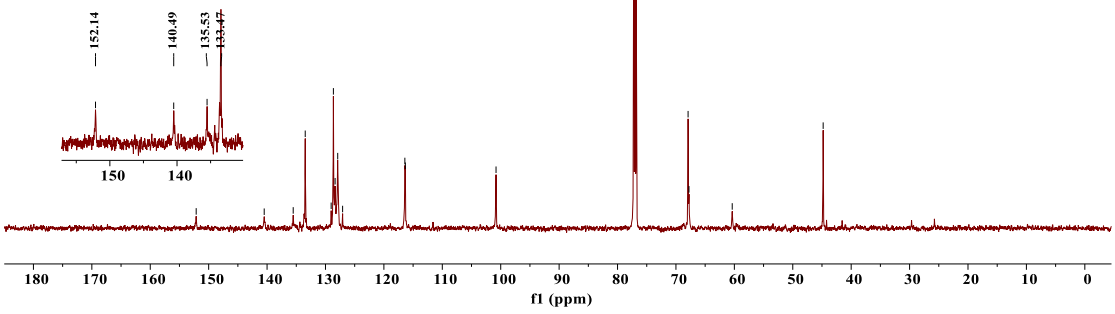

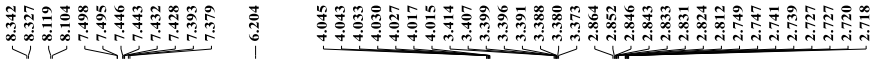

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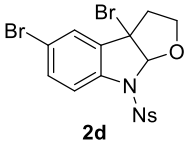

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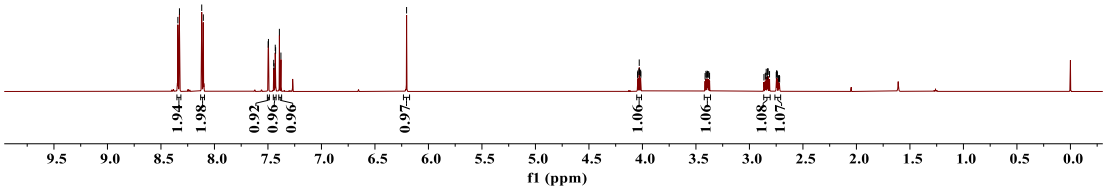

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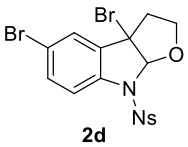
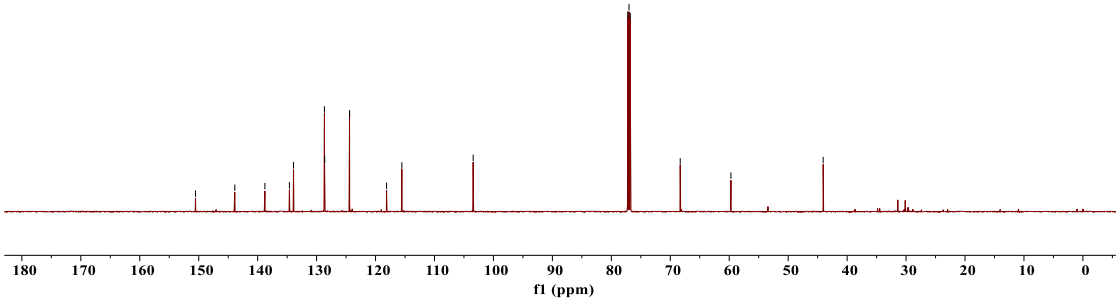
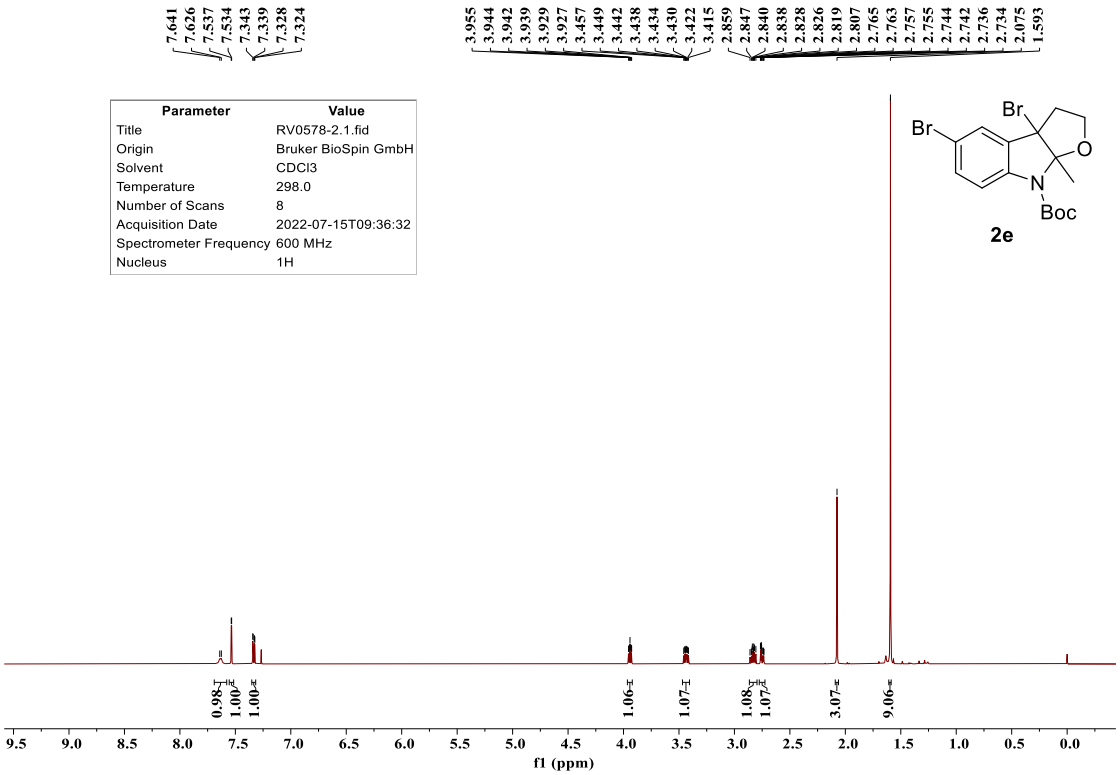


2d

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7.626
7.537
7.534
7.343
7.339
7.328
7.324
3.955
3.944
3.942
3.939
3.929
3.927
3.457
3.449
3.442
3.438
3.434
3.430
3.422
3.415
2.859
2.847
2.840
2.838
2.828
2.826
2.819
2.807
2.765
2.763
2.757
2.755
2.744
2.742
2.736
2.734
2.075
1.593
Parameter Value
Title RV0578-2.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-07-15T09:36:32
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
O
N
Boc
2e
0.98
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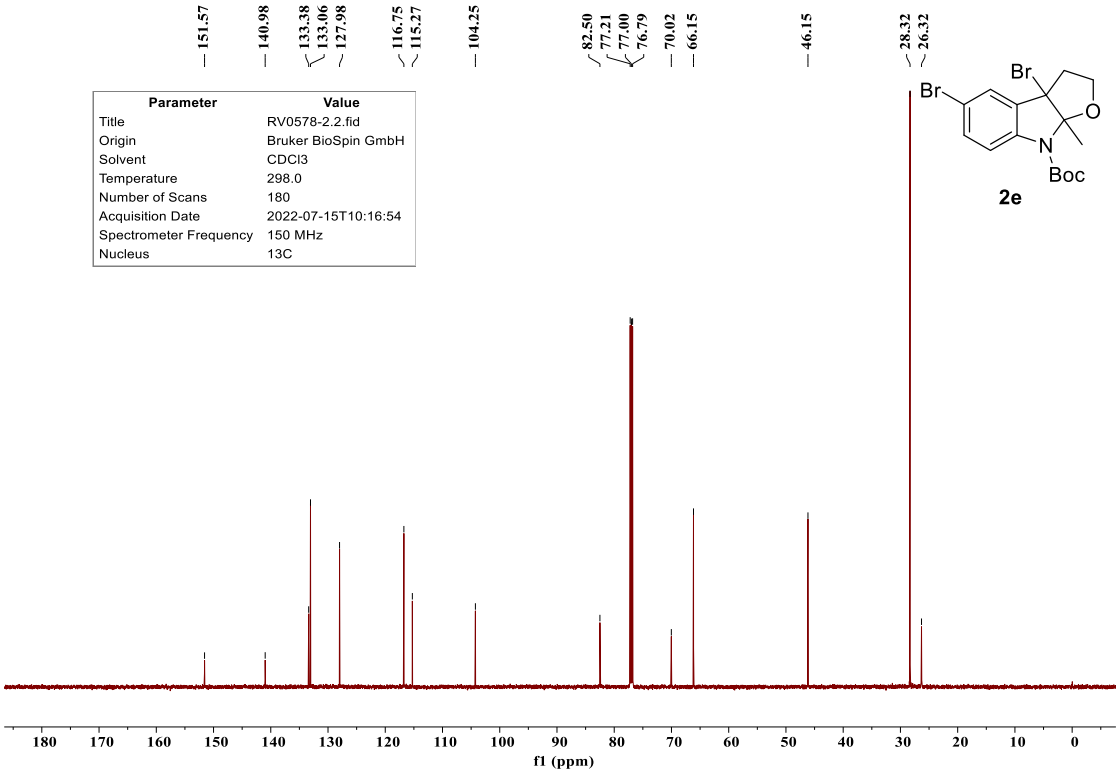
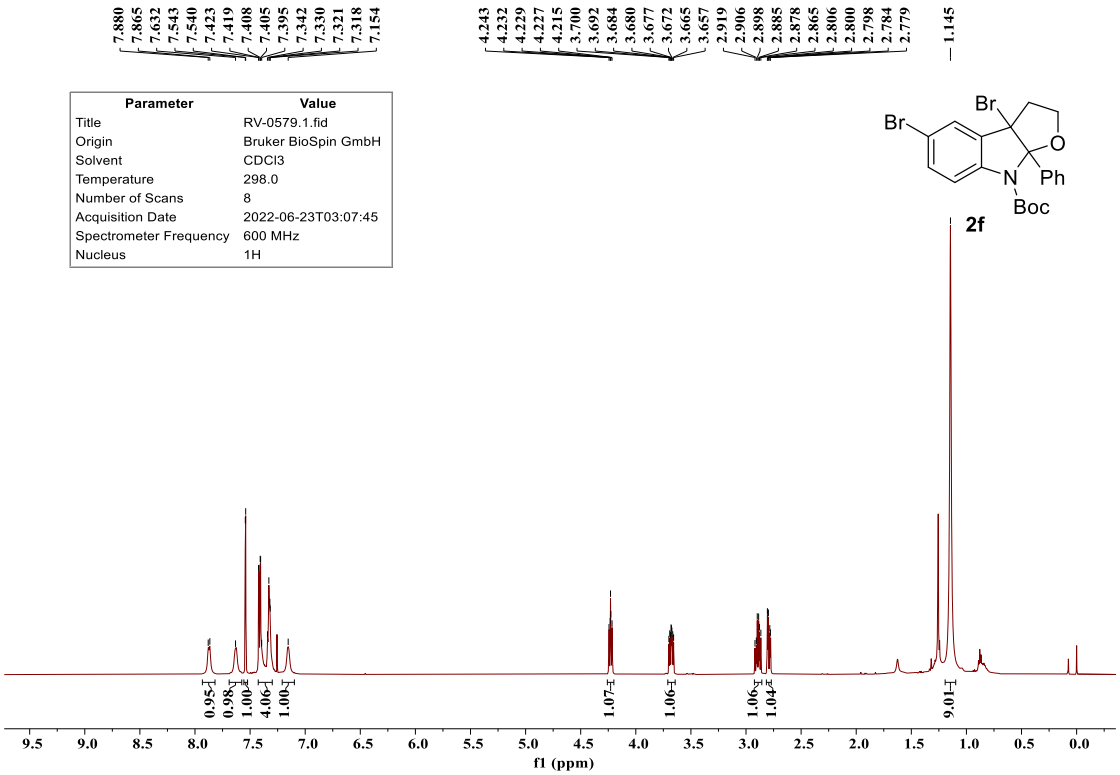
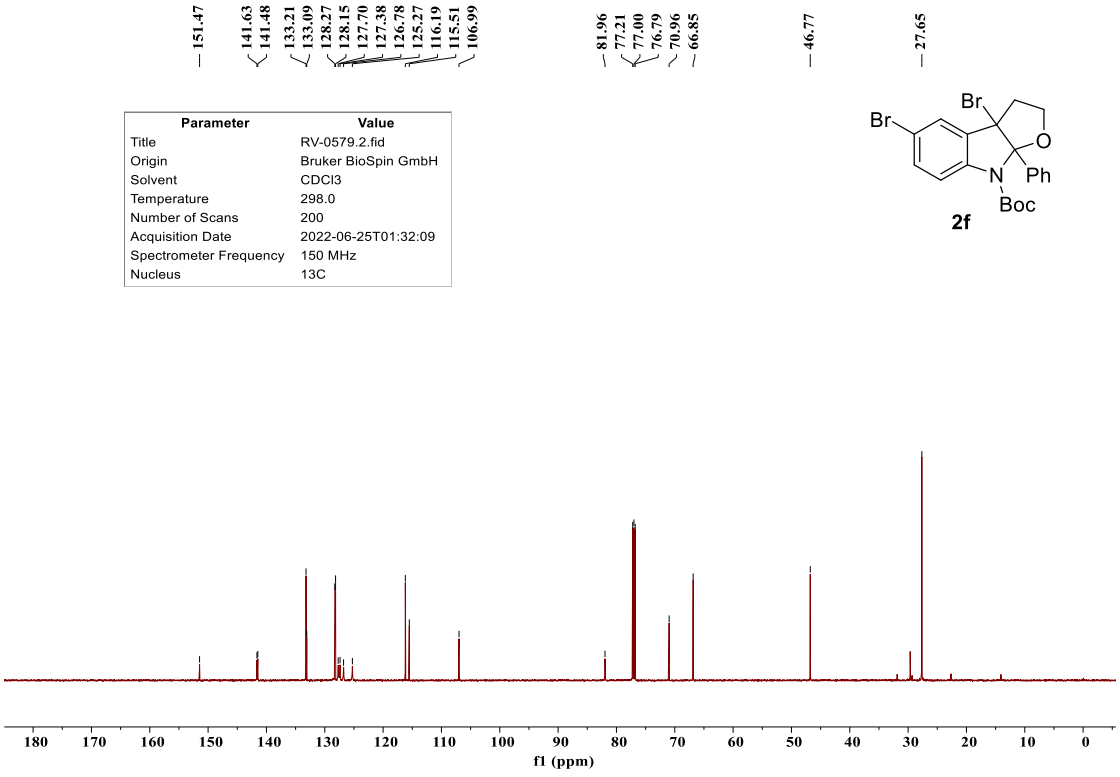
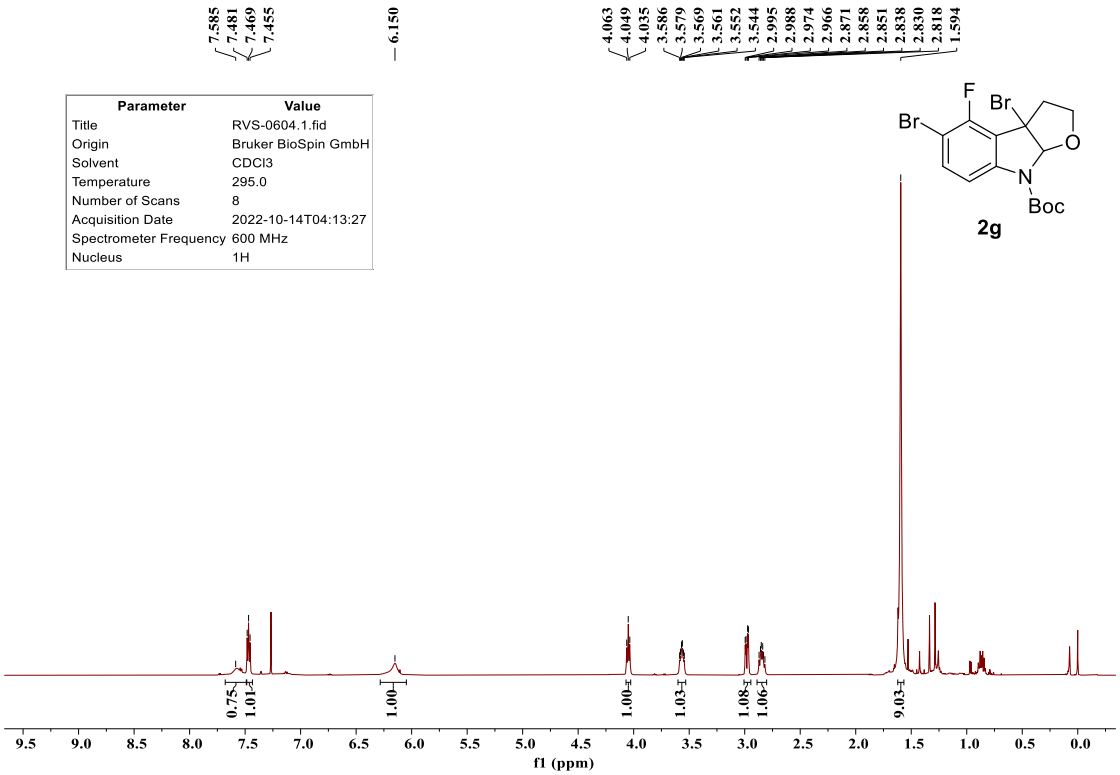
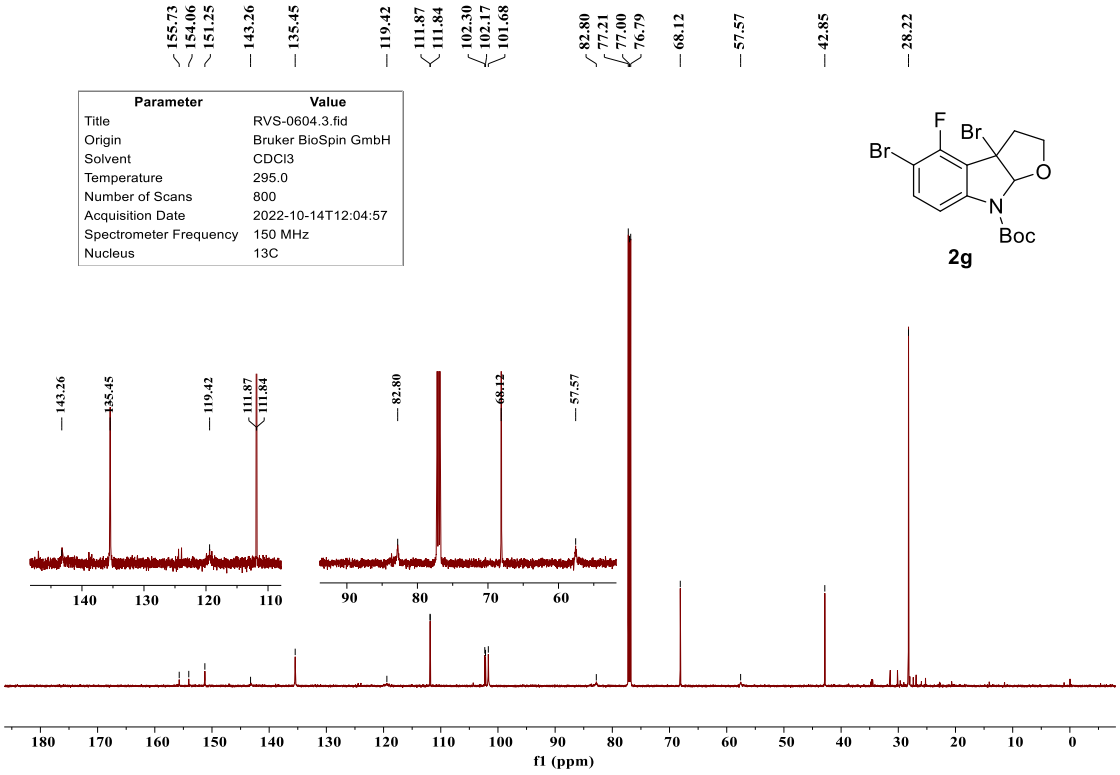
1.08
1.07
3.07
9.06
f1 (ppm)


151.57
140.98
133.38
133.06
127.98
116.75
115.27
104.25
82.50
77.21
77.00
76.79
70.02
66.15
46.15
28.32
26.32
Parameter Value
Title RV0578-2.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 180
Acquisition Date 2022-07-15T10:16:54
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
O
N
Boc
2e
f1 (ppm)

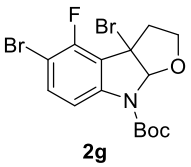

7.880
7.865
7.632
7.543
7.540
7.423
7.419
7.408
7.405
7.395
7.342
7.330
7.321
7.318
7.154
4.243
4.232
4.229
4.227
4.215
3.700
3.692
3.684
3.680
3.677
3.672
3.665
3.657
2.919
2.906
2.898
2.885
2.878
2.865
2.806
2.800
2.798
2.784
2.779
1.145
Parameter Value
Title RV-0579.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-06-23T03:07:45
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
O
N
Ph
Boc
2f
0.95
0.98
1.00
4.06
1.00
1.07
1.06
1.06
1.04
9.01
f1 (ppm)


151.47
141.63
141.48
133.21
133.09
128.27
128.15
127.70
127.38
126.78
125.27
116.19
115.51
106.99
81.96
77.21
77.00
76.79
70.96
66.85
46.77
27.65
Parameter Value
Title RV-0579.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 200
Acquisition Date 2022-06-25T01:32:09
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
O
N
Ph
Boc
2f
f1 (ppm)

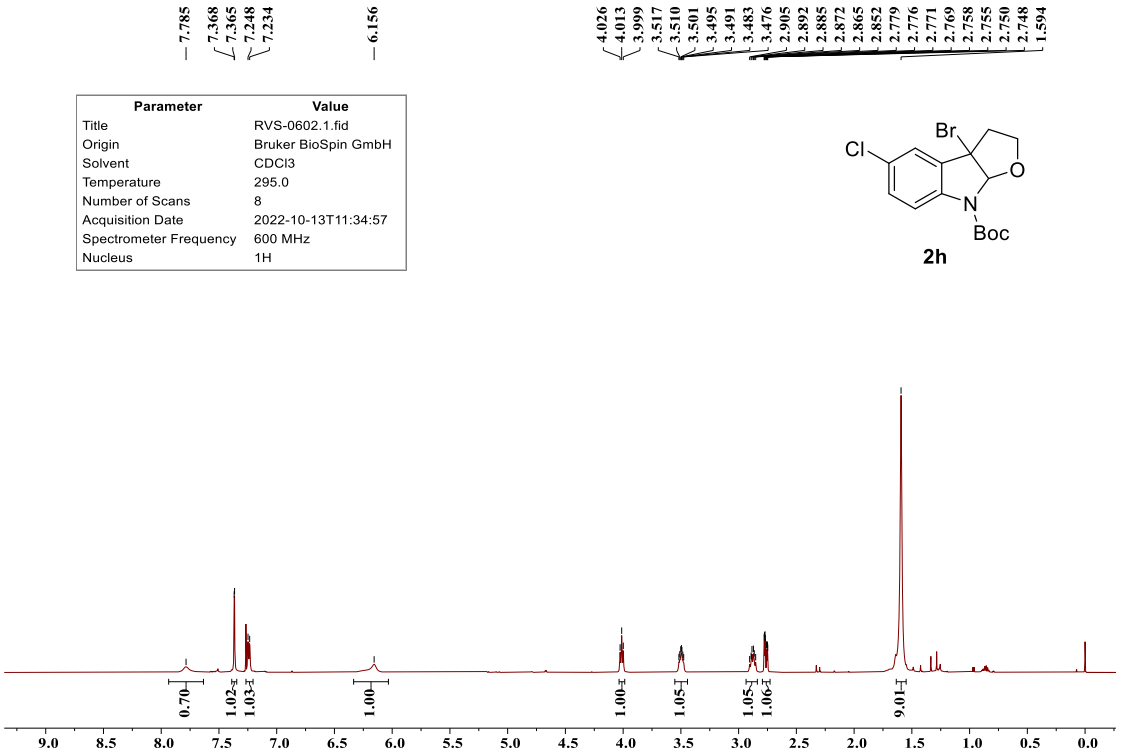

7.585
7.481
7.469
7.455
6.150
4.063
4.049
4.035
3.586
3.579
3.569
3.561
3.552
3.544
2.995
2.988
2.974
2.966
2.871
2.858
2.851
2.838
2.830
2.818
1.594
Parameter Value
Title RVS-0604.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 8
Acquisition Date 2022-10-14T04:13:27
Spectrometer Frequency 600 MHz
Nucleus 1H
F
Br
Br
O
N
Boc
2g
0.75
1.01
1.00
1.00
1.03
1.08
1.06
9.03
f1 (ppm)


155.73
154.06
151.25
143.26
135.45
119.42
111.87
111.84
102.30
102.17
101.68
82.80
77.21
77.00
76.79
68.12
57.57
42.85
28.22
Parameter Value
Title RVS-0604.3.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 800
Acquisition Date 2022-10-14T12:04:57
Spectrometer Frequency 150 MHz
Nucleus 13C
F
Br
Br
O
N
Boc
2g
f1 (ppm)

Parameter	Value
Title	RVS-0604.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.2
Number of Scans	16
Acquisition Date	2022-10-14T11:25:42
Spectrometer Frequency	565 MHz
Nucleus	19F



2g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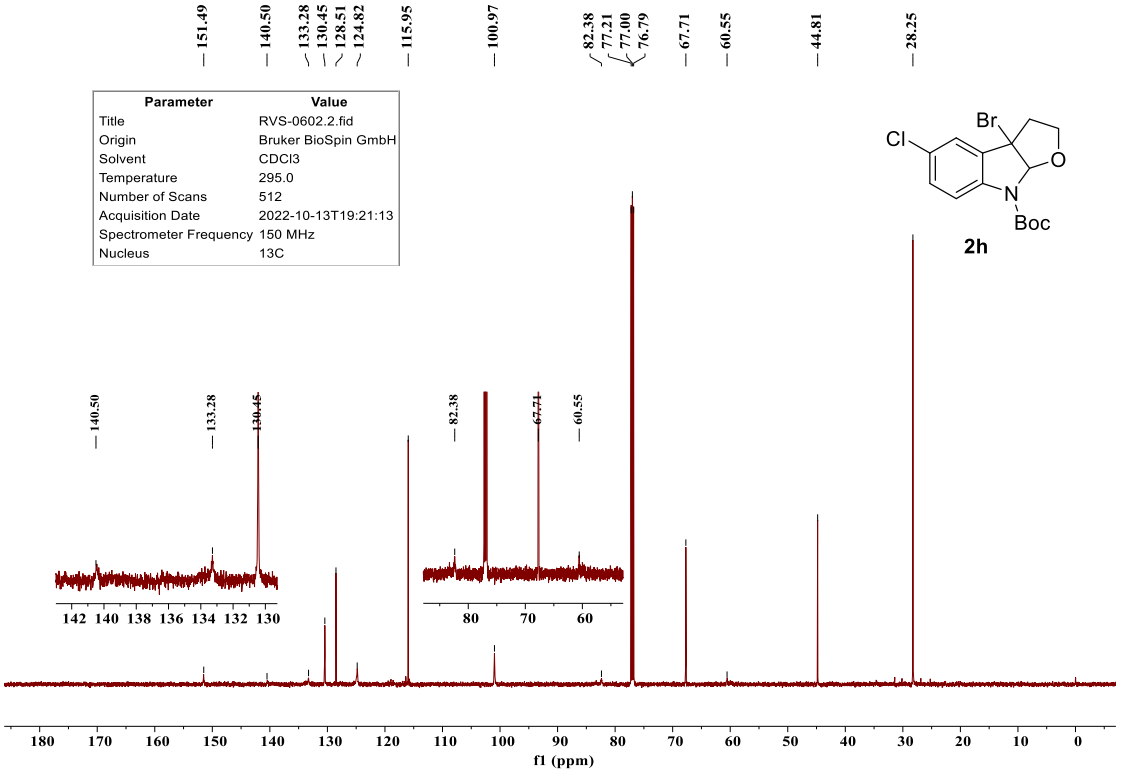
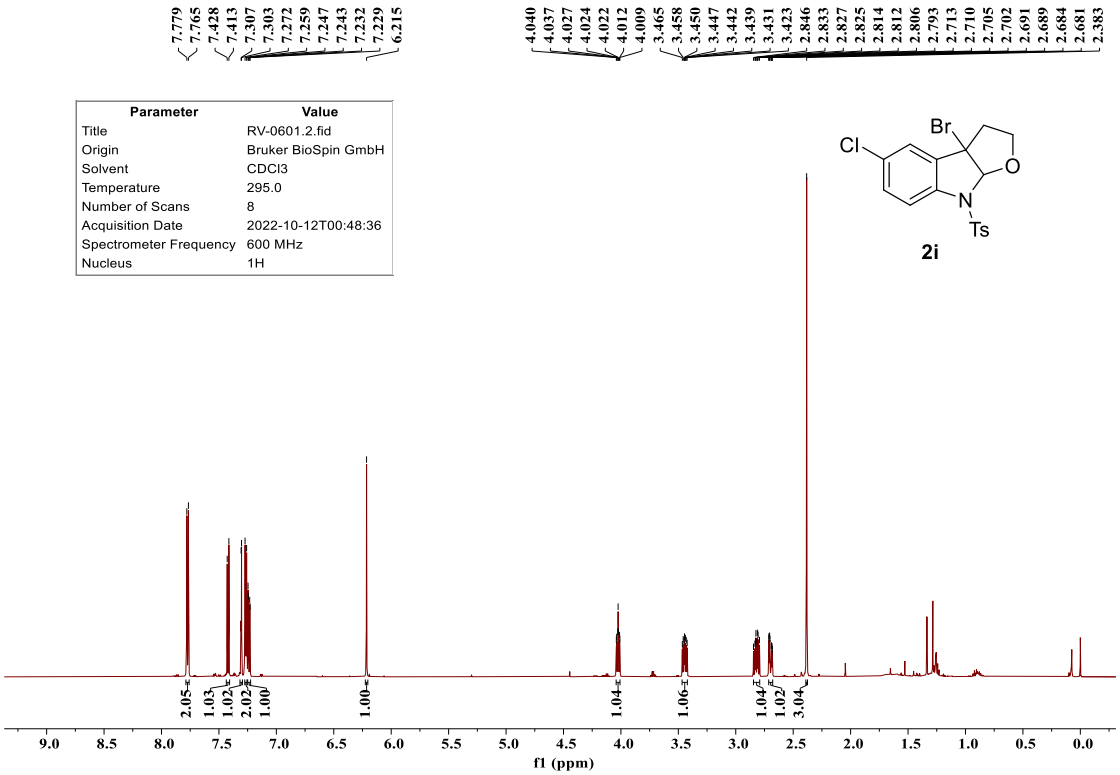
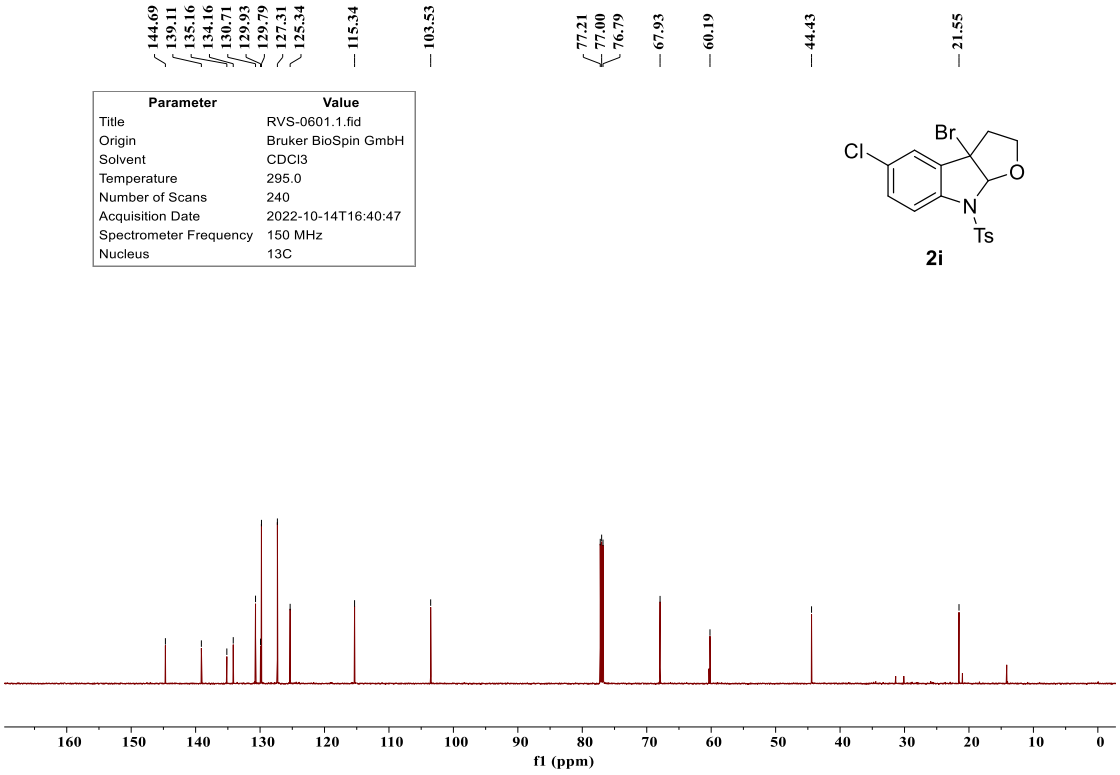
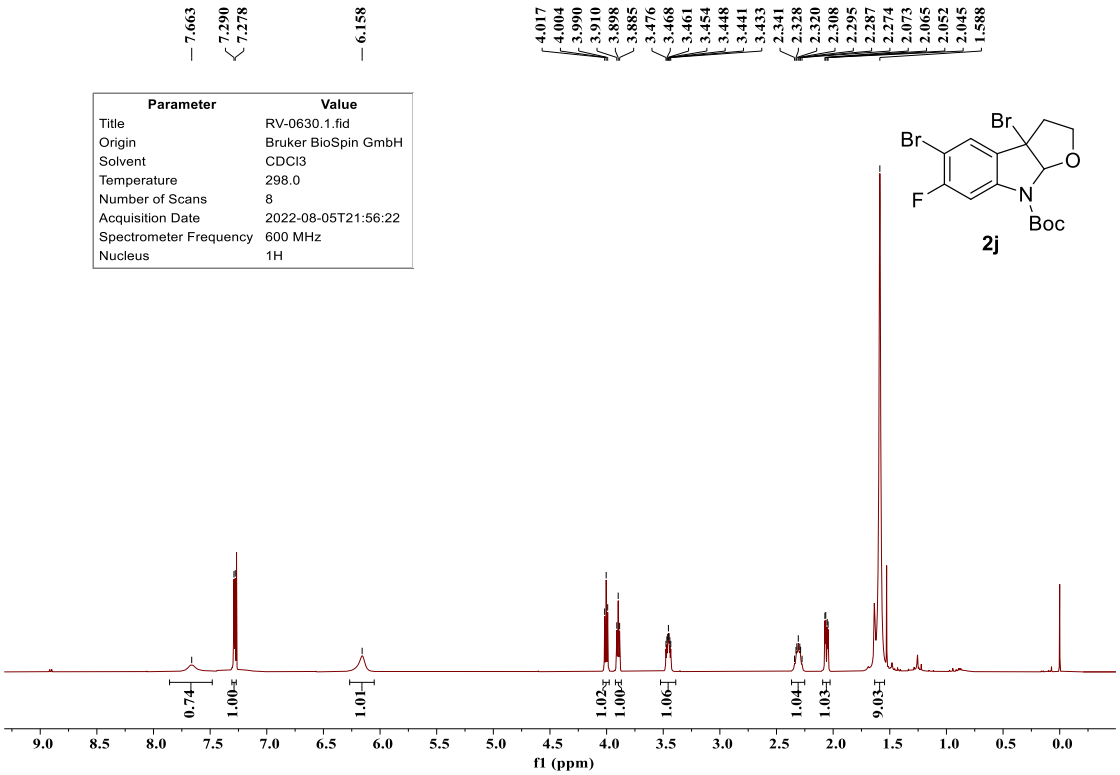
7.368
7.365
7.248
7.234
6.156
4.026
4.013
3.999
3.517
3.510
3.501
3.495
3.491
3.483
3.476
2.905
2.892
2.885
2.872
2.865
2.852
2.779
2.776
2.771
2.769
2.758
2.755
2.750
2.748
1.594
Parameter Value
Title RVS-0602.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 8
Acquisition Date 2022-10-13T11:34:57
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Cl
O
N
Boc
2h
0.70
1.02
1.03
1.00
1.00
1.05
1.05
1.06
9.01
f1 (ppm)


151.49
140.50
133.28
130.45
128.51
124.82
115.95
100.97
82.38
77.21
77.00
76.79
67.71
60.55
44.81
28.25
Parameter Value
Title RVS-0602.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 512
Acquisition Date 2022-10-13T19:21:13
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Cl
O
N
Boc
2h
f1 (ppm)


7.779
7.765
7.428
7.413
7.307
7.303
7.272
7.259
7.247
7.243
7.232
7.229
6.215
4.040
4.037
4.027
4.024
4.022
4.012
4.009
3.465
3.458
3.450
3.447
3.442
3.439
3.431
3.423
2.846
2.833
2.827
2.825
2.814
2.812
2.806
2.793
2.713
2.710
2.705
2.702
2.691
2.689
2.684
2.681
2.383
Parameter Value
Title RV-0601.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 8
Acquisition Date 2022-10-12T00:48:36
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Cl
O
N
Ts
2i
2.05
1.03
1.02
2.02
1.00
1.00
1.04
1.06
1.04
1.02
3.04
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


144.69
139.11
135.16
134.16
130.71
129.93
129.79
127.31
125.34
115.34
103.53
77.21
77.00
76.79
67.93
60.19
44.43
21.55
Parameter Value
Title RVS-0601.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 240
Acquisition Date 2022-10-14T16:40:47
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Cl
O
N
Ts
2i
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.663
7.290
7.278
6.158
4.017
4.004
3.990
3.910
3.898
3.885
3.476
3.468
3.461
3.454
3.448
3.441
3.433
2.341
2.328
2.320
2.308
2.295
2.287
2.274
2.073
2.065
2.052
2.045
1.588
Parameter Value
Title RV-0630.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-08-05T21:56:22
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
O
F
N
Boc
2j
0.74
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1.01
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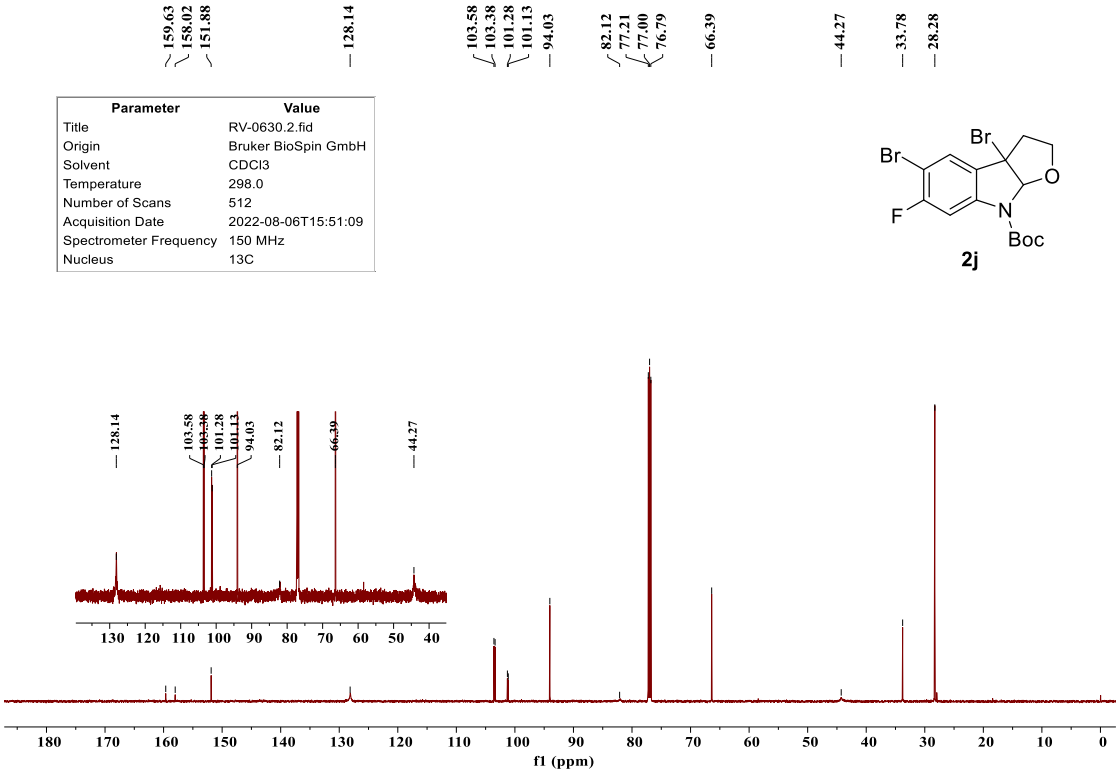
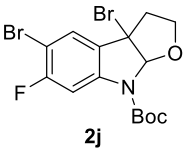
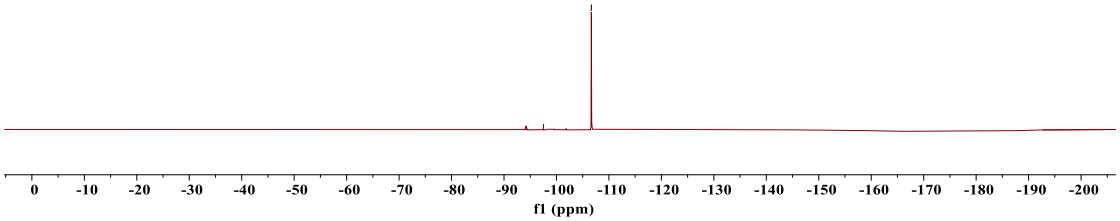
1.06
1.04
1.03
9.03
f1 (ppm)

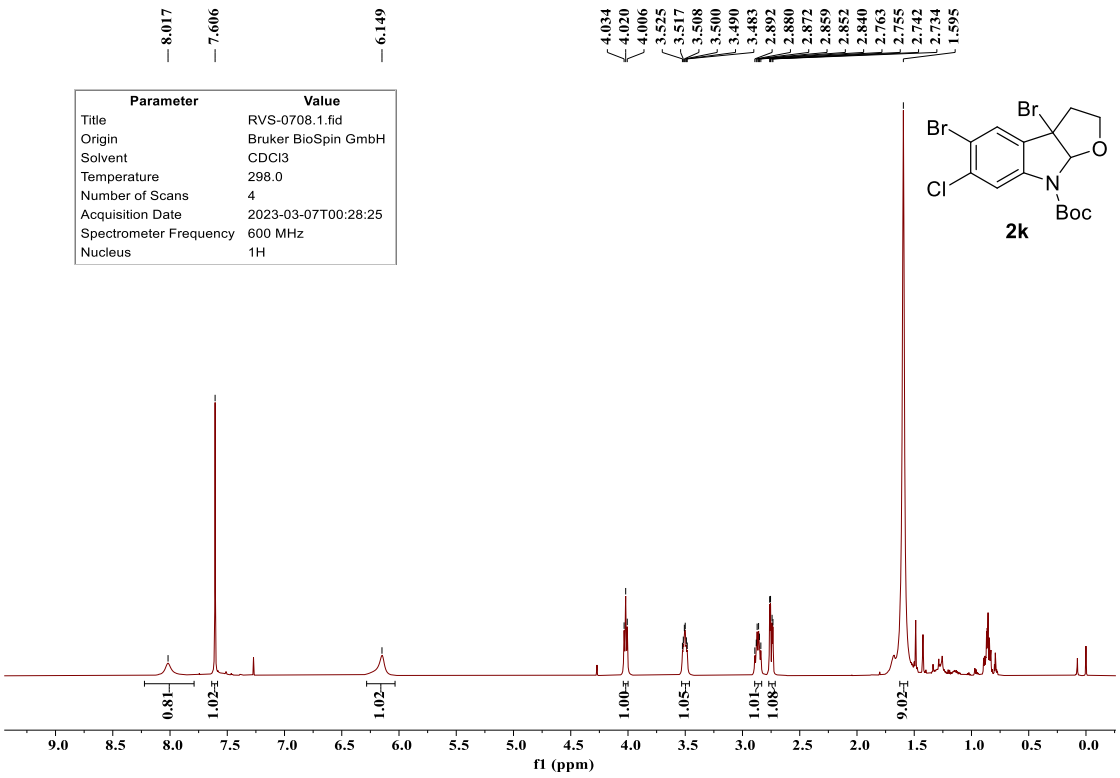

159.63
158.02
151.88
128.14
103.58
103.38
101.28
101.13
94.03
82.12
77.21
77.00
76.79
66.39
44.27
33.78
28.28
Parameter Value
Title RV-0630.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 512
Acquisition Date 2022-08-06T15:51:09
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
O
F
N
Boc
2j
f1 (ppm)

Parameter	Value
Title	RV-0630.3.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	16
Acquisition Date	2022-08-06T15:52:35
Spectrometer Frequency	565 MHz
Nucleus	19F



2j

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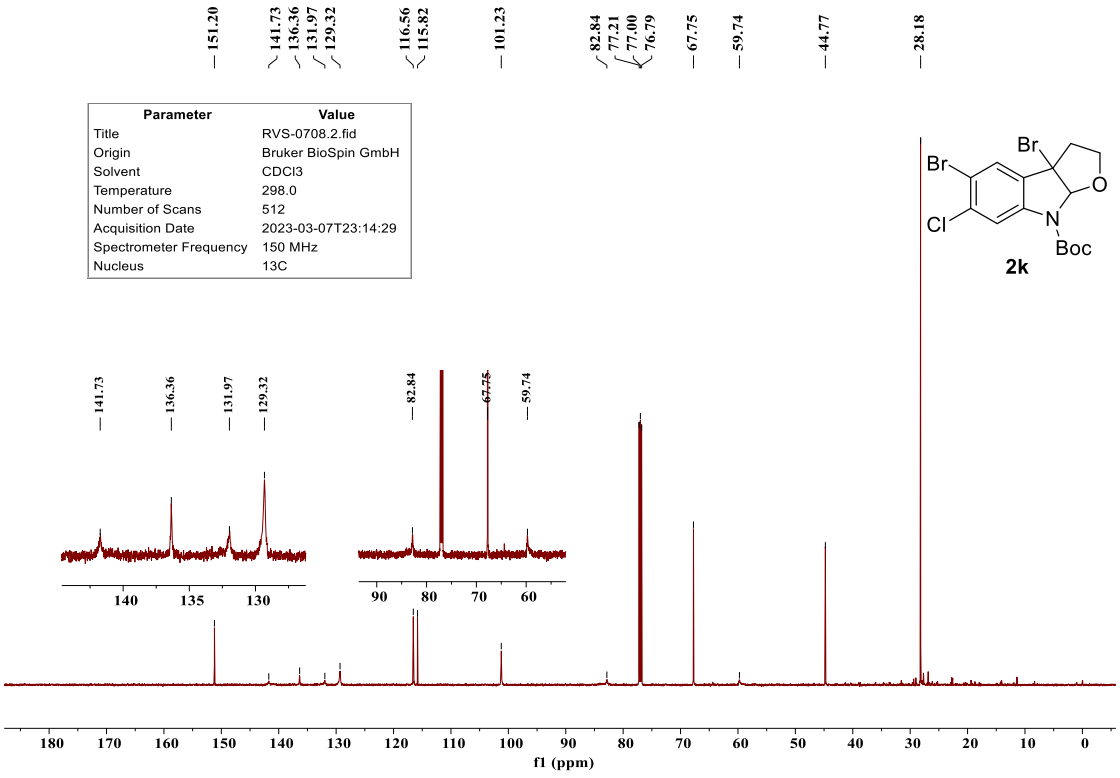
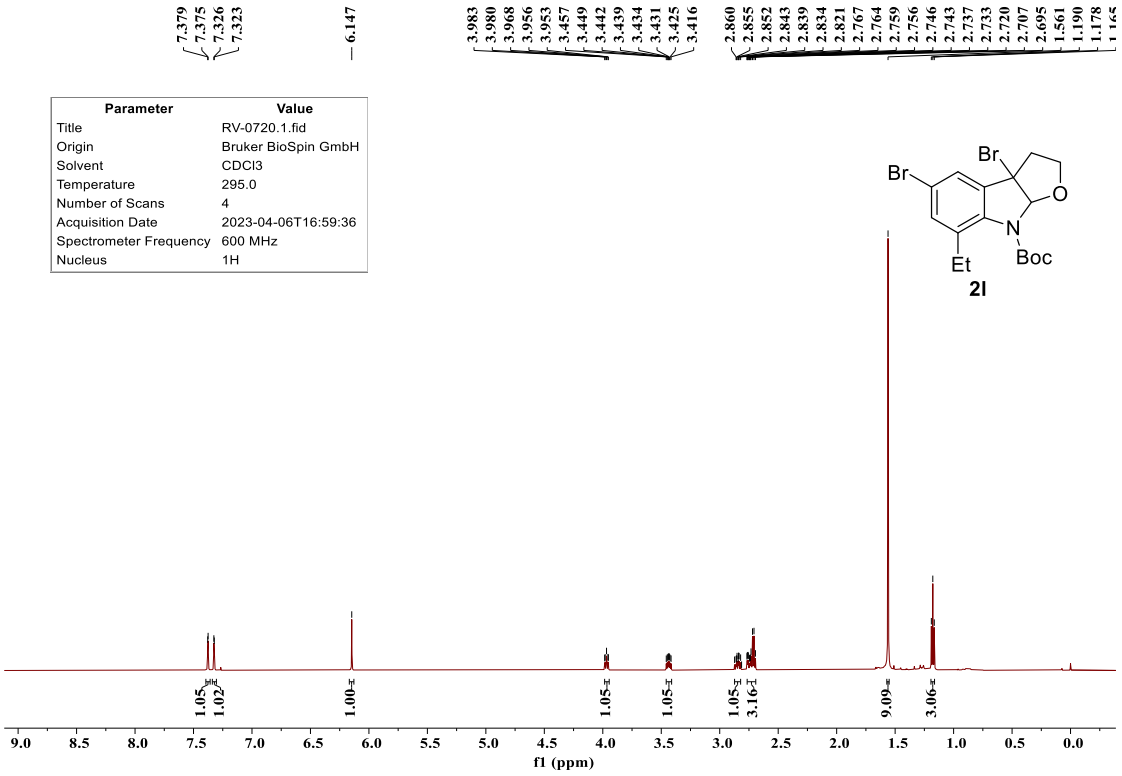
6.149
4.034
4.020
4.006
3.525
3.517
3.508
3.500
3.490
3.483
2.892
2.880
2.872
2.859
2.852
2.840
2.763
2.755
2.742
2.734
1.595
Parameter Value
Title RVS-0708.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 4
Acquisition Date 2023-03-07T00:28:25
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
Cl
O
N
Boc
2k
0.81
1.02
1.02
1.00
1.05
1.01
1.08
9.02
f1 (ppm)


151.20
141.73
136.36
131.97
129.32
116.56
115.82
101.23
82.84
77.21
77.00
76.79
67.75
59.74
44.77
28.18
Parameter Value
Title RVS-0708.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 512
Acquisition Date 2023-03-07T23:14:29
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
Cl
O
N
Boc
2k
f1 (ppm)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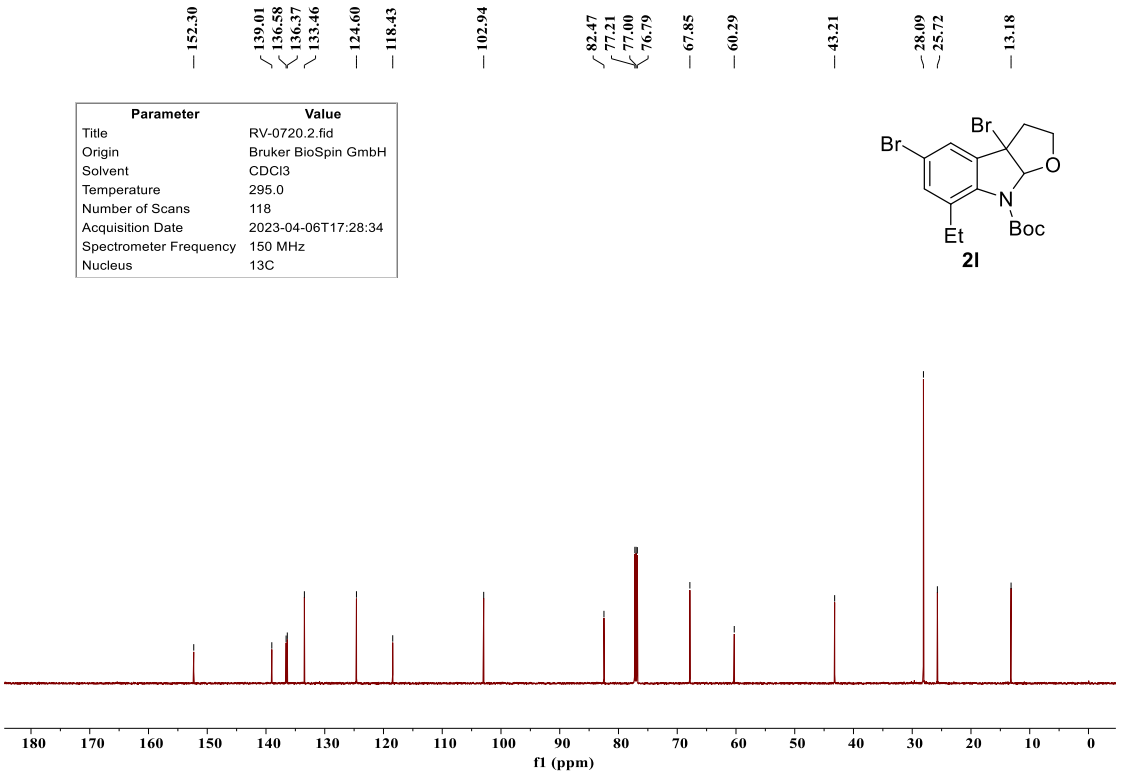
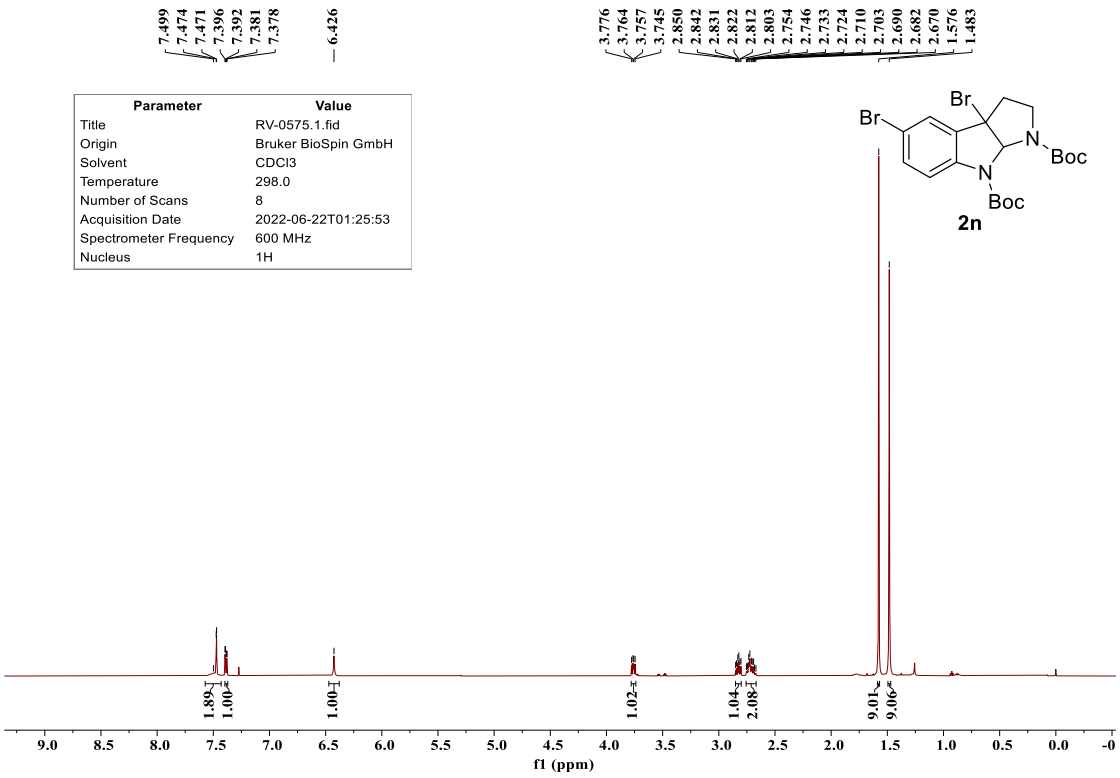
7.326
7.323
6.147
3.983
3.980
3.968
3.956
3.953
3.457
3.449
3.442
3.439
3.434
3.431
3.425
3.416
2.860
2.855
2.852
2.843
2.839
2.834
2.821
2.767
2.764
2.759
2.756
2.746
2.743
2.737
2.733
2.720
2.707
2.695
1.561
1.190
1.178
Parameter Value
Title RV-0720.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-04-06T16:59:36
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
O
N
Et
Boc
2l
1.05
1.02
1.00
1.05
1.05
1.05
3.16
9.09
3.06
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


152.30
139.01
136.58
136.37
133.46
124.60
118.43
102.94
82.47
77.21
77.00
76.79
67.85
60.29
43.21
28.09
25.72
13.18
Parameter Value
Title RV-0720.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 118
Acquisition Date 2023-04-06T17:28:34
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
O
N
Et
Boc
2l
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)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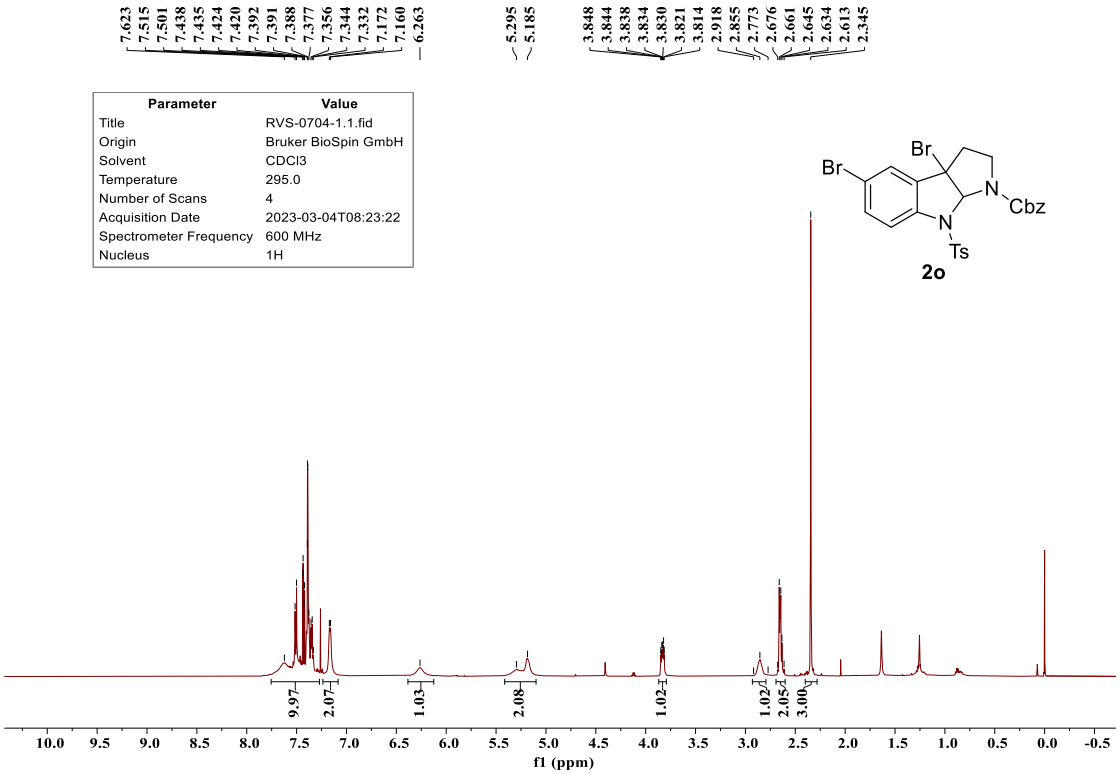
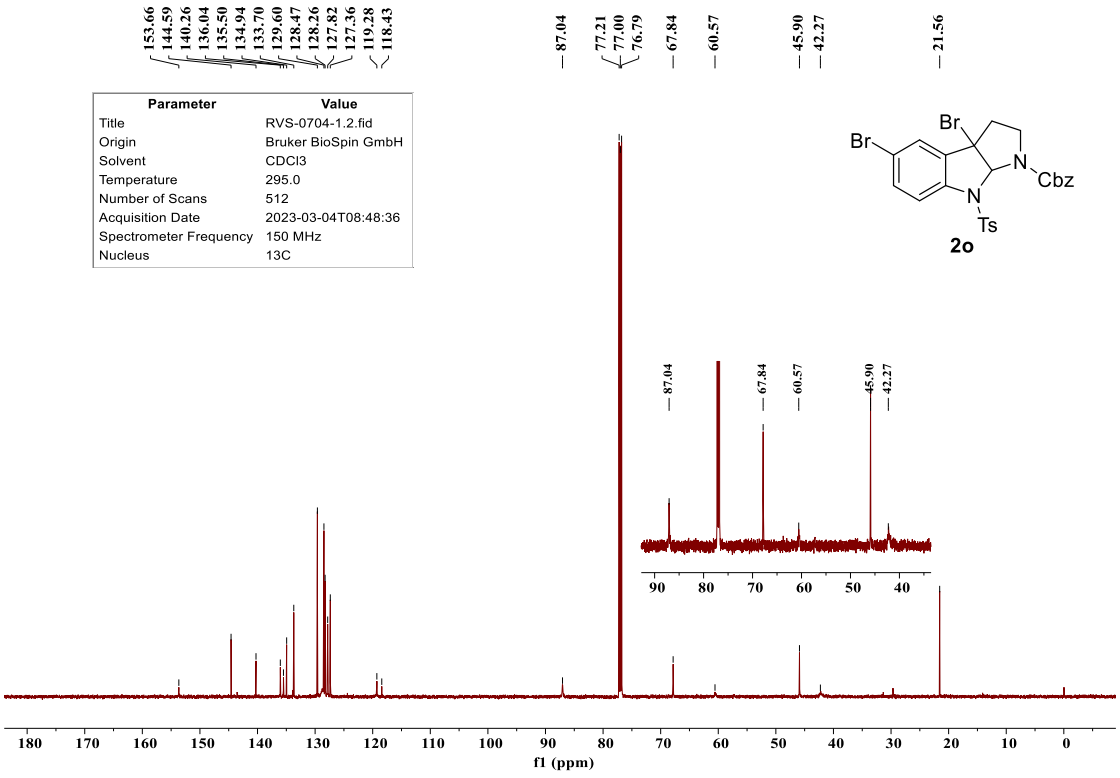
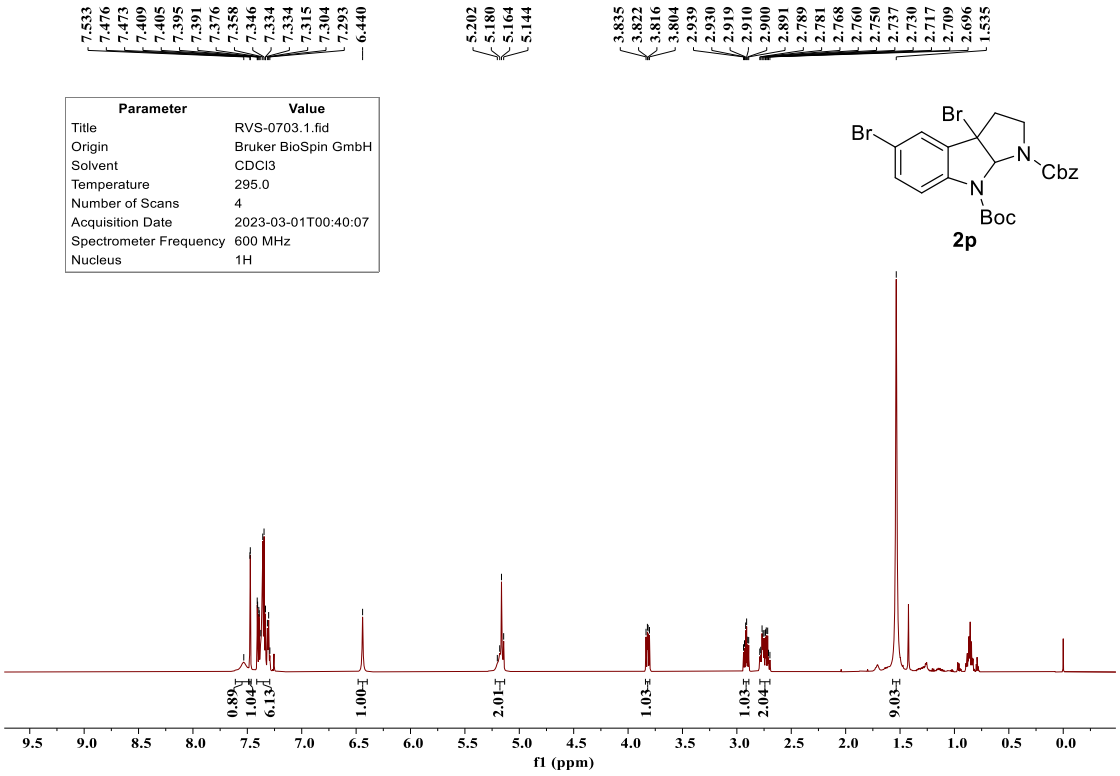
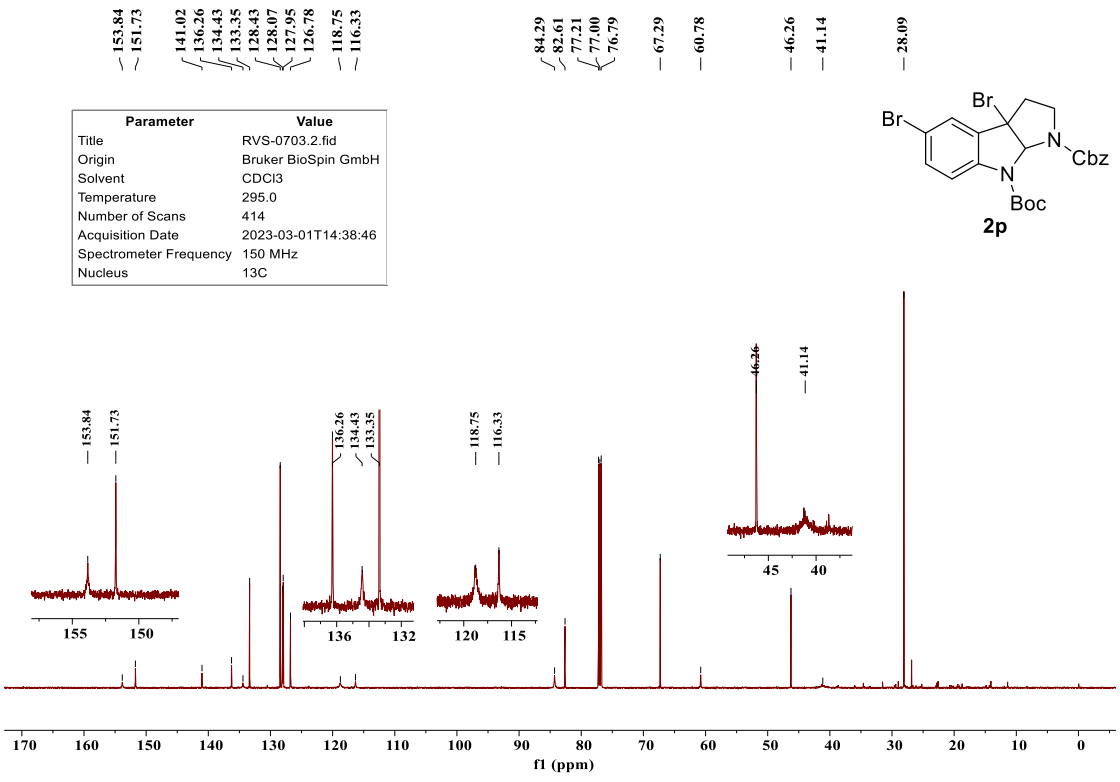
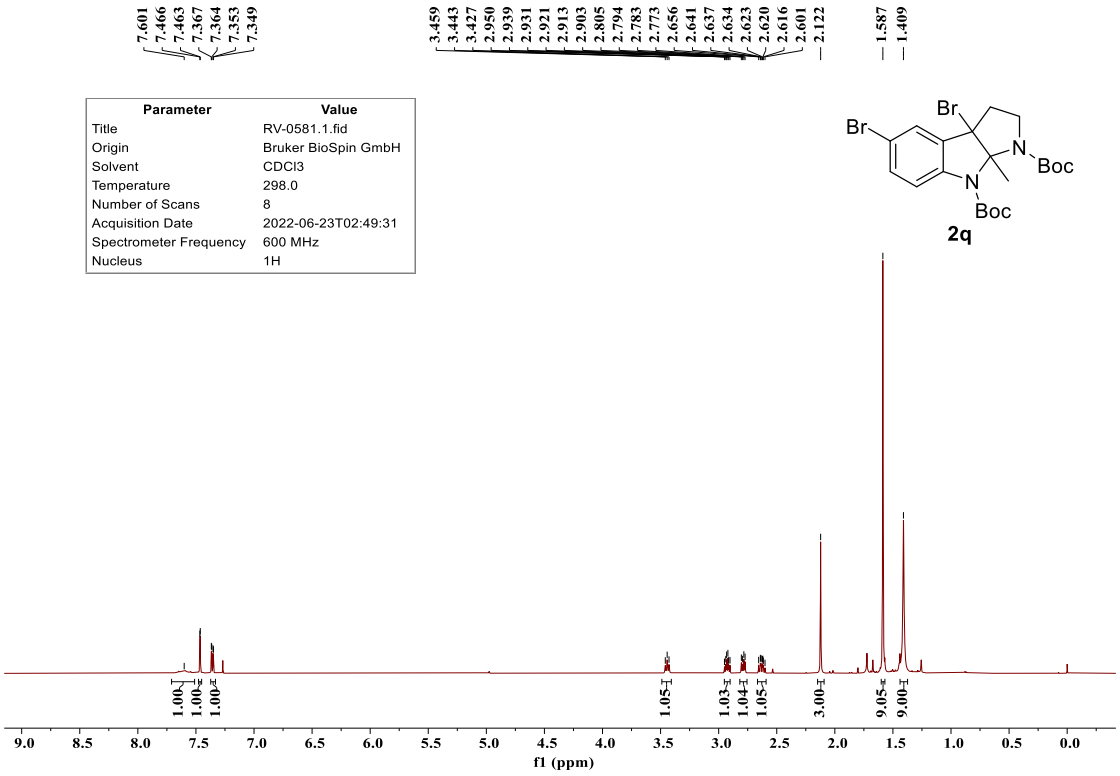
7.396
7.392
7.381
7.378
6.426
3.776
3.764
3.757
3.745
2.850
2.842
2.831
2.822
2.812
2.803
2.754
2.746
2.733
2.724
2.710
2.703
2.690
2.682
2.670
1.576
1.483
Parameter
Value
Title
RV-0575.1.fid
Origin
Bruker BioSpin GmbH
Solvent
CDCl3
Temperature
298.0
Number of Scans
8
Acquisition Date
2022-06-22T01:25:53
Spectrometer Frequency
600 MHz
Nucleus
1H
Br
Br
N
Boc
N
Boc
2n
1.89
1.00
1.00
1.02
1.04
2.08
9.01
9.06
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0
f1 (ppm)

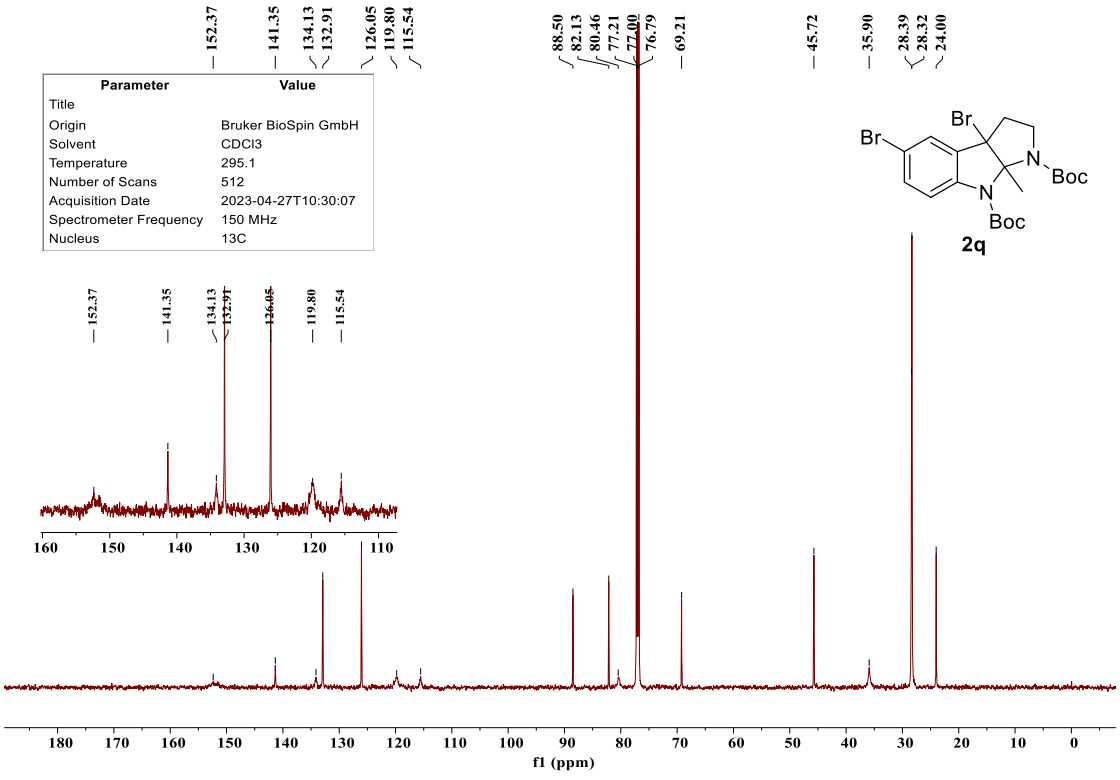

7.623
7.515
7.501
7.438
7.435
7.424
7.420
7.392
7.391
7.388
7.377
7.356
7.344
7.332
7.172
7.160
6.263
5.295
5.185
3.848
3.844
3.838
3.834
3.830
3.821
3.814
2.918
2.855
2.773
2.676
2.661
2.645
2.634
2.613
2.345
Parameter Value
Title RVS-0704-1.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-03-04T08:23:22
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
N
Cbz
N
Ts
2o
9.97
2.07
1.03
2.08
1.02
1.02
2.05
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

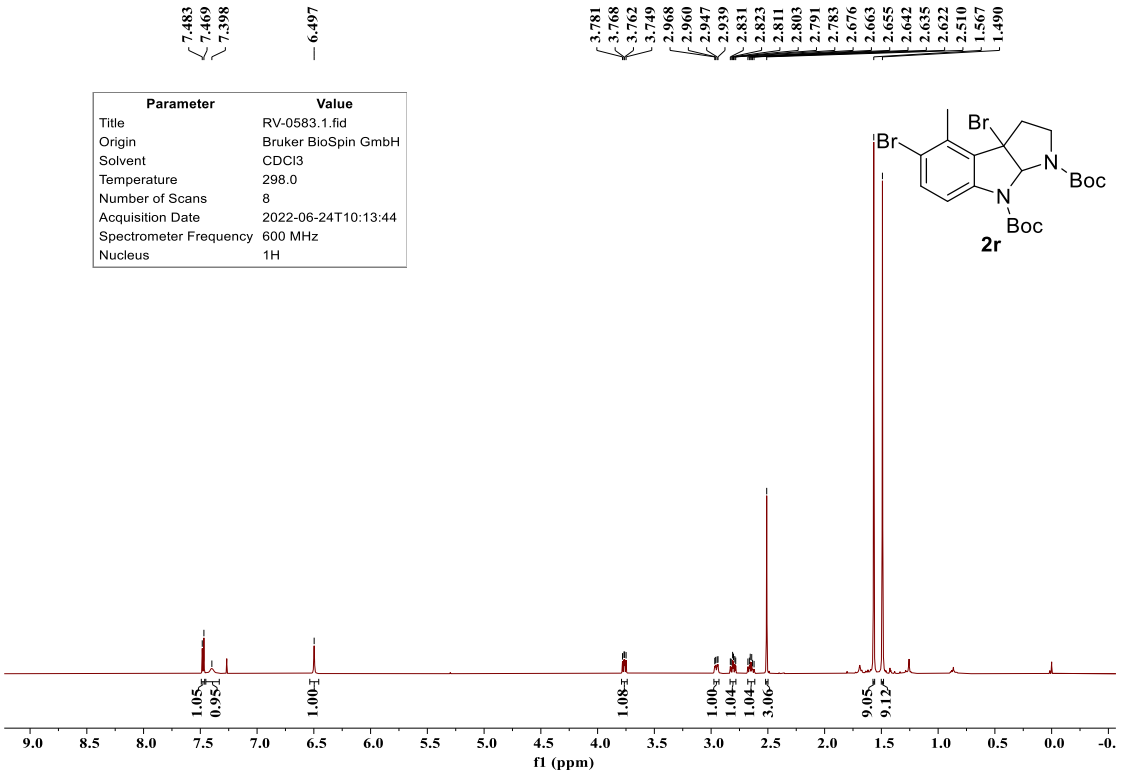

153.66
144.59
140.26
136.04
135.50
134.94
133.70
129.60
128.47
128.26
127.82
127.36
119.28
118.43
87.04
77.21
77.00
76.79
67.84
60.57
45.90
42.27
21.56
Parameter Value
Title RVS-0704-1.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 512
Acquisition Date 2023-03-04T08:48:36
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
N
Cbz
N
Ts
2o
87.04
67.84
60.57
45.90
42.27
90 80 70 60 50 40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

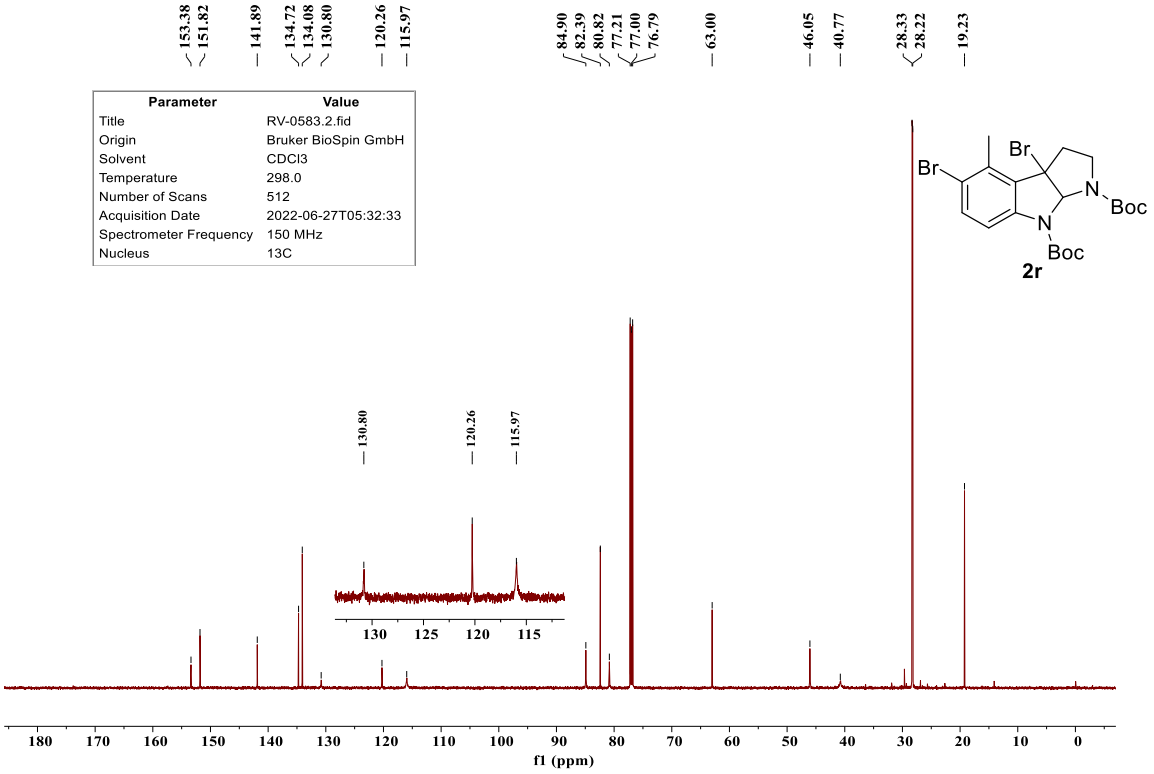

7.533
7.476
7.473
7.409
7.405
7.395
7.391
7.376
7.358
7.346
7.334
7.334
7.315
7.304
7.293
6.440
5.202
5.180
5.164
5.144
3.835
3.822
3.816
3.804
2.939
2.930
2.919
2.910
2.900
2.891
2.789
2.781
2.768
2.760
2.750
2.737
2.730
2.717
2.709
2.696
1.535
Parameter Value
Title RVS-0703.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-03-01T00:40:07
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
N
Cbz
N
Boc
2p
0.89
1.04
6.13
1.00
2.01
1.03
1.03
2.04
9.03
f1 (ppm)

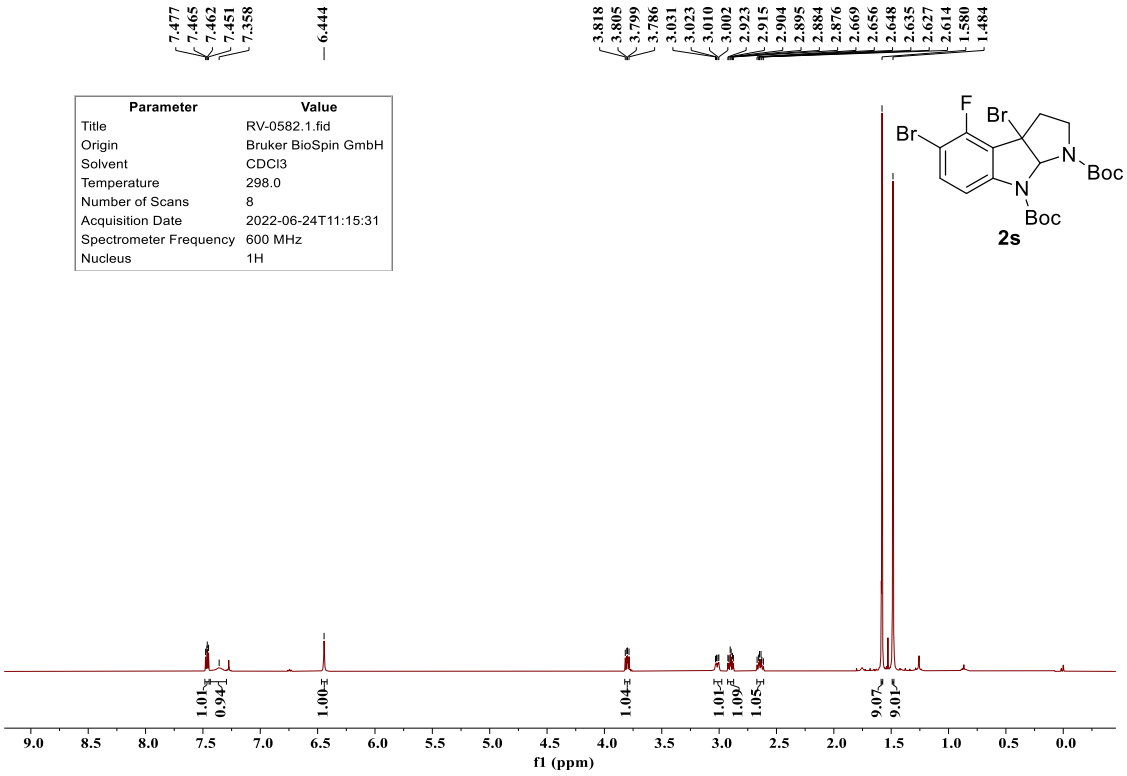

153.84
151.73
141.02
136.26
134.43
133.35
128.43
128.07
127.95
126.78
118.75
116.33
84.29
82.61
77.21
77.00
76.79
67.29
60.78
46.26
41.14
28.09
Parameter Value
Title RVS-0703.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 414
Acquisition Date 2023-03-01T14:38:46
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
N
Cbz
N
Boc
2p
f1 (ppm)

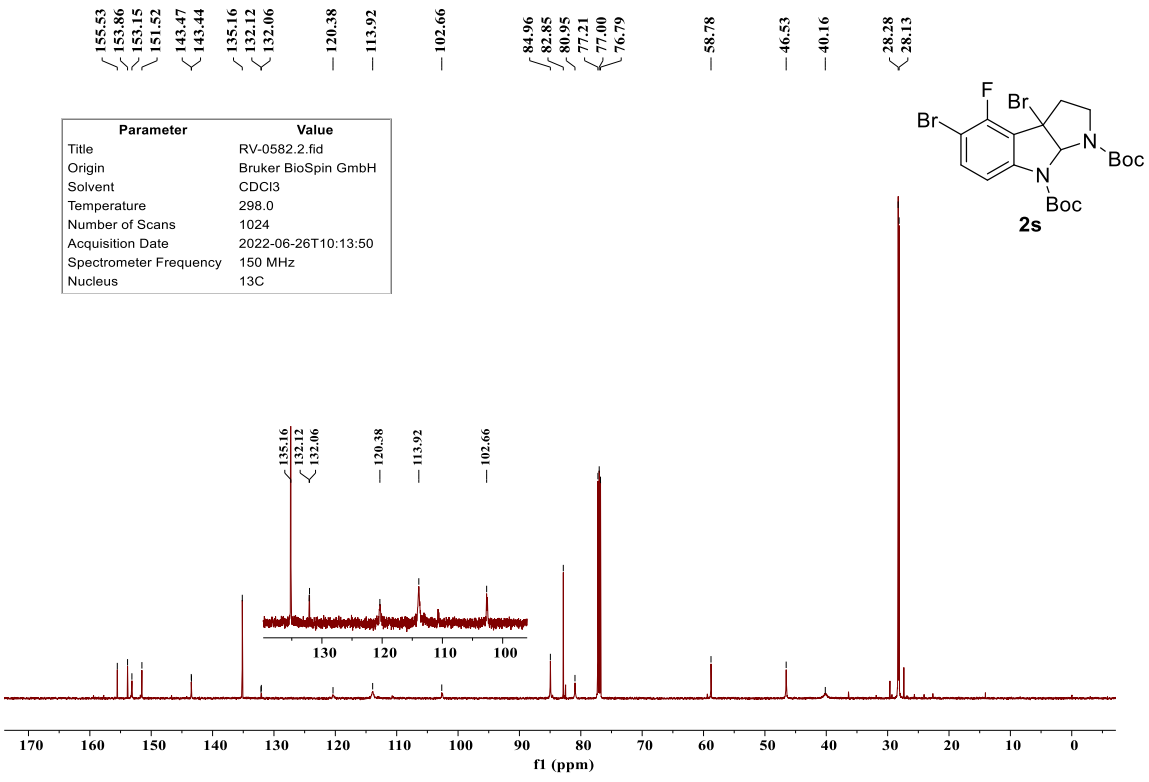

7.601
7.466
7.463
7.367
7.364
7.353
7.349
3.459
3.443
3.427
2.950
2.939
2.931
2.921
2.913
2.903
2.805
2.794
2.783
2.773
2.656
2.641
2.637
2.634
2.623
2.620
2.616
2.601
2.122
1.587
1.409
Parameter Value
Title RV-0581.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-06-23T02:49:31
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
N
Boc
2q
1.00
1.00
1.00
1.05
1.03
1.04
1.05
3.00
9.05
9.00
f1 (ppm)


152.37
141.35
134.13
132.91
126.05
119.80
115.54
88.50
82.13
80.46
77.21
77.00
76.79
69.21
45.72
35.90
28.39
28.32
24.00
Parameter Value
Title
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.1
Number of Scans 512
Acquisition Date 2023-04-27T10:30:07
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
N
Boc
2q
f1 (ppm)

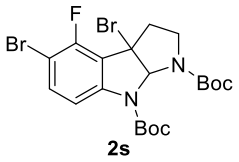

7.483
7.469
7.398
6.497
3.781
3.768
3.762
3.749
2.968
2.960
2.947
2.939
2.831
2.823
2.811
2.803
2.791
2.783
2.676
2.663
2.655
2.642
2.635
2.622
2.510
1.567
1.490
Parameter Value
Title RV-0583.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-06-24T10:13:44
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
N
Boc
N
Boc
2r
1.05
0.95
1.00
1.08
1.00
1.04
1.04
3.06
9.05
9.12
f1 (ppm)


153.38
151.82
141.89
134.72
134.08
130.80
120.26
115.97
84.90
82.39
80.82
77.21
77.00
76.79
63.00
46.05
40.77
28.33
28.22
19.23
Parameter Value
Title RV-0583.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 512
Acquisition Date 2022-06-27T05:32:33
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
N
Boc
N
Boc
2r
130.80
120.26
115.97
f1 (ppm)

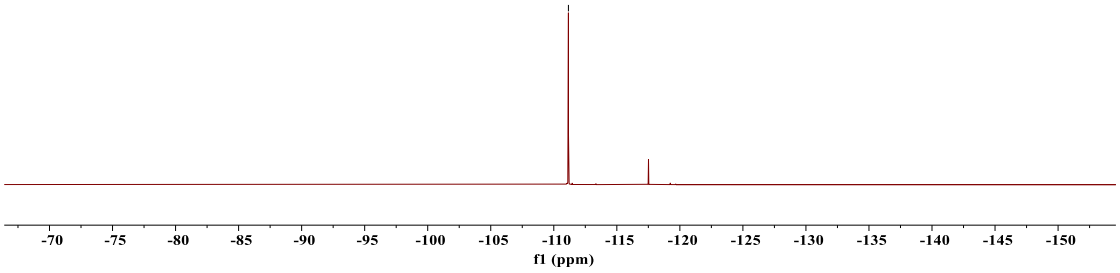

7.477
7.465
7.462
7.451
7.358
6.444
3.818
3.805
3.799
3.786
3.031
3.023
3.010
3.002
2.923
2.915
2.904
2.895
2.884
2.876
2.669
2.656
2.648
2.635
2.627
2.614
1.580
1.484
Parameter Value
Title RV-0582.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-06-24T11:15:31
Spectrometer Frequency 600 MHz
Nucleus 1H
F
Br
Br
N
Boc
N
Boc
2s
1.01
0.94
1.00
1.04
1.01
1.09
1.05
9.07
9.01
f1 (ppm)

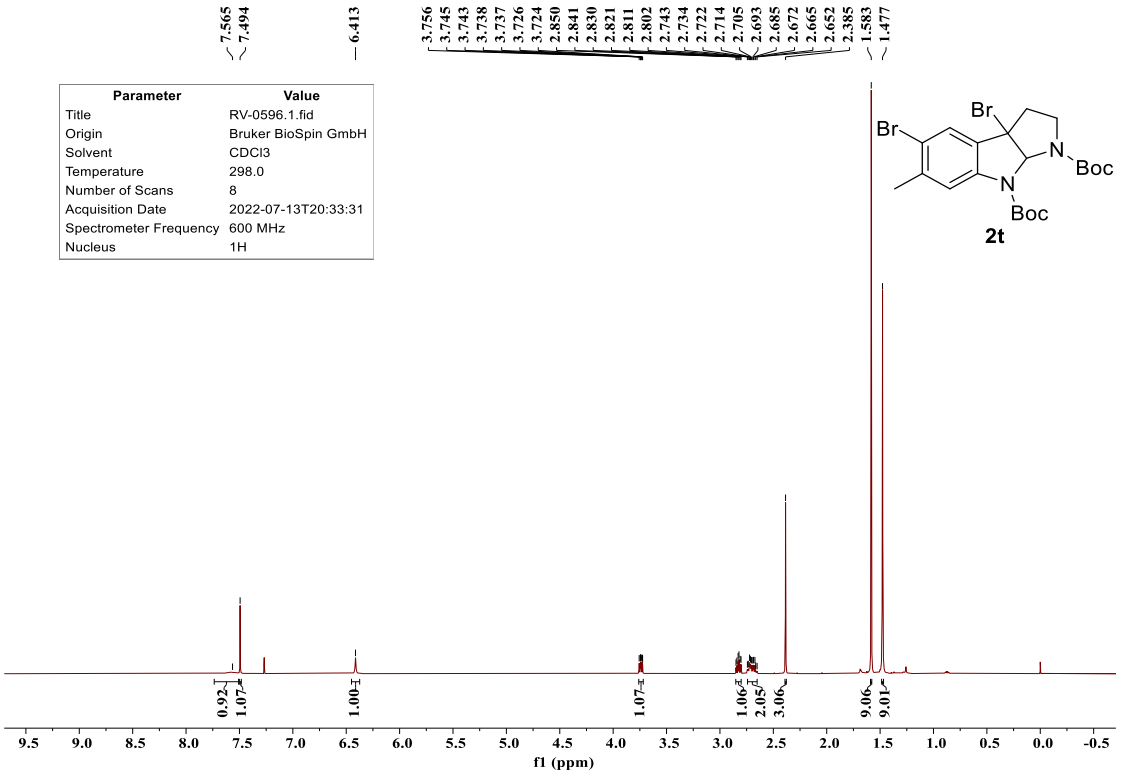

155.53
153.86
153.15
151.52
143.47
143.44
135.16
132.12
132.06
120.38
113.92
102.66
84.96
82.85
80.95
77.21
77.00
76.79
58.78
46.53
40.16
28.28
28.13
Parameter Value
Title RV-0582.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 1024
Acquisition Date 2022-06-26T10:13:50
Spectrometer Frequency 150 MHz
Nucleus 13C
F
Br
Br
N
Boc
N
Boc
2s
f1 (ppm)

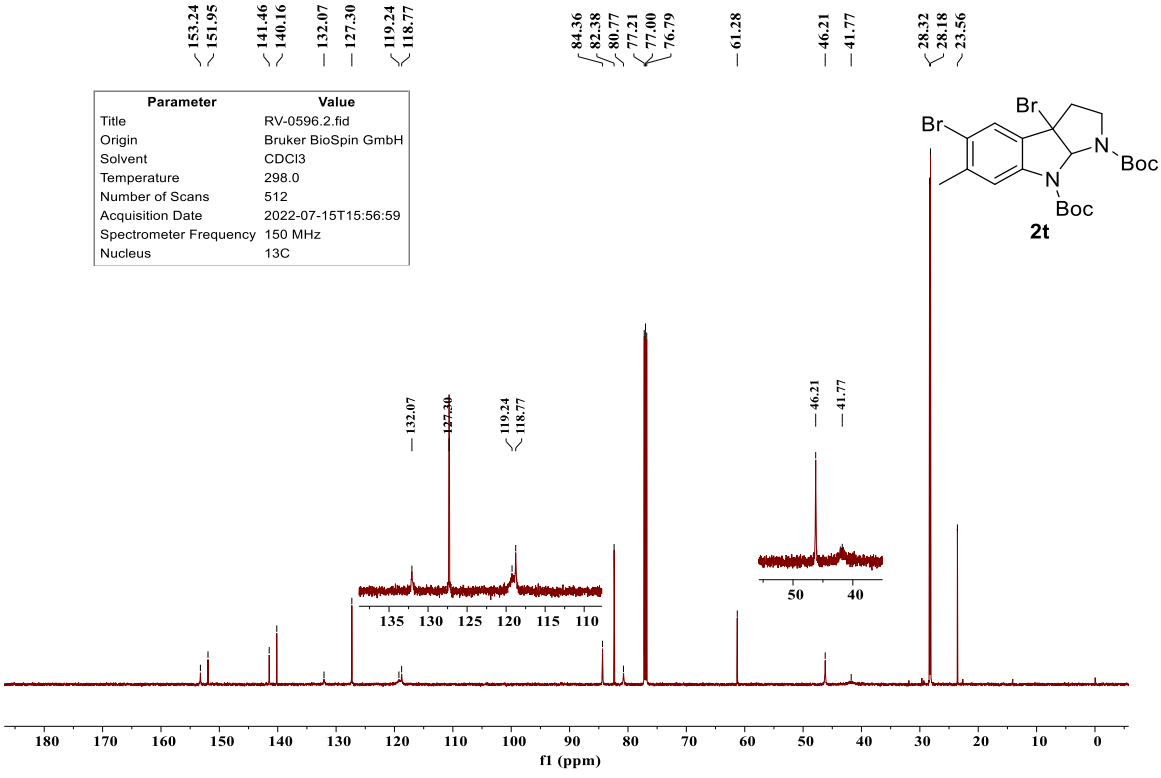
Parameter	Value
Title	RV-0582.3.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	16
Acquisition Date	2022-06-26T10:15:23
Spectrometer Frequency	565 MHz
Nucleus	19F

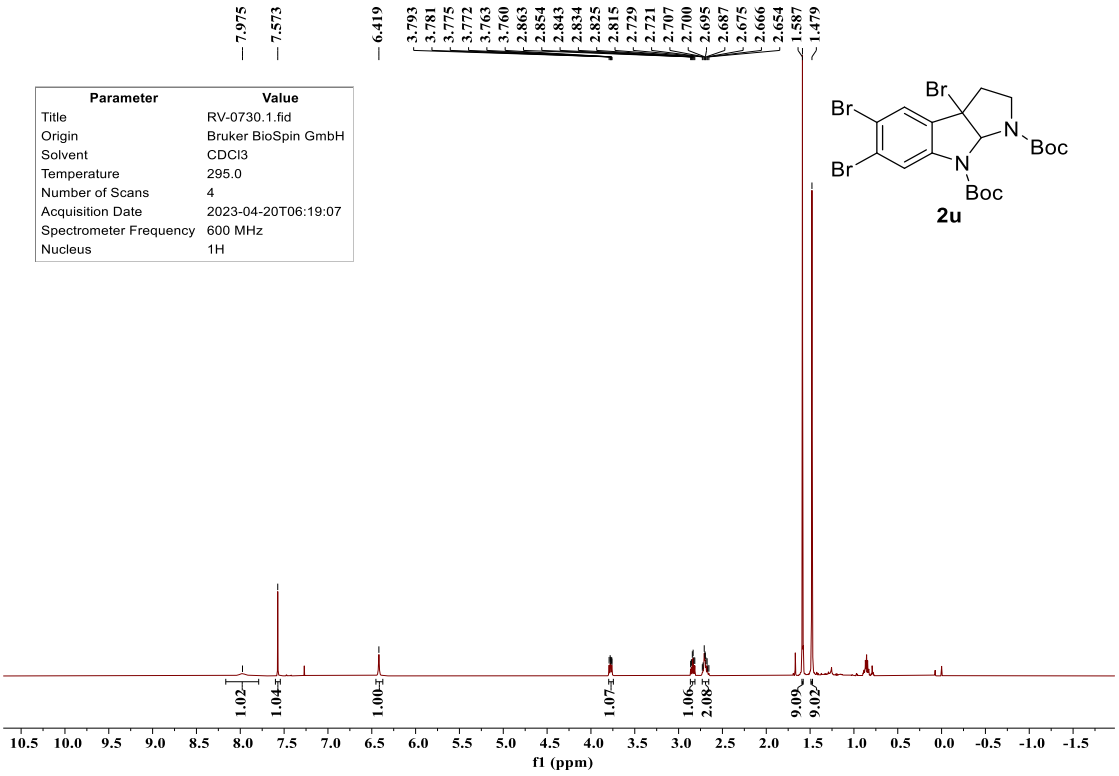


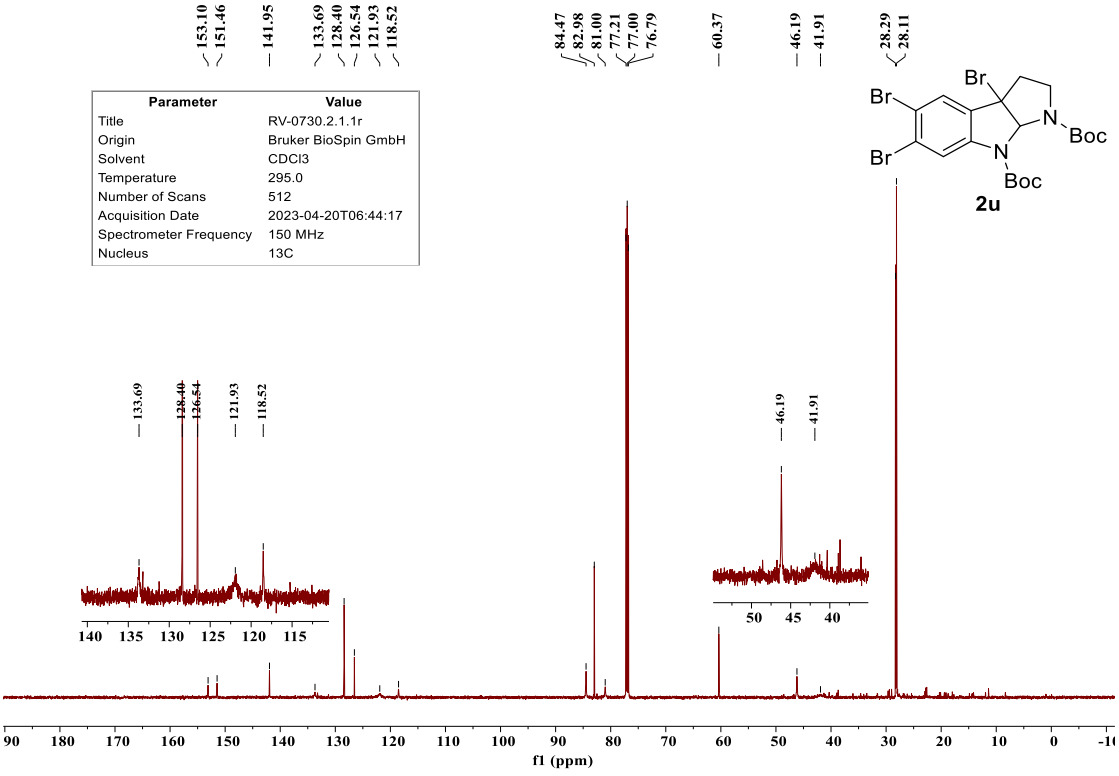
2s

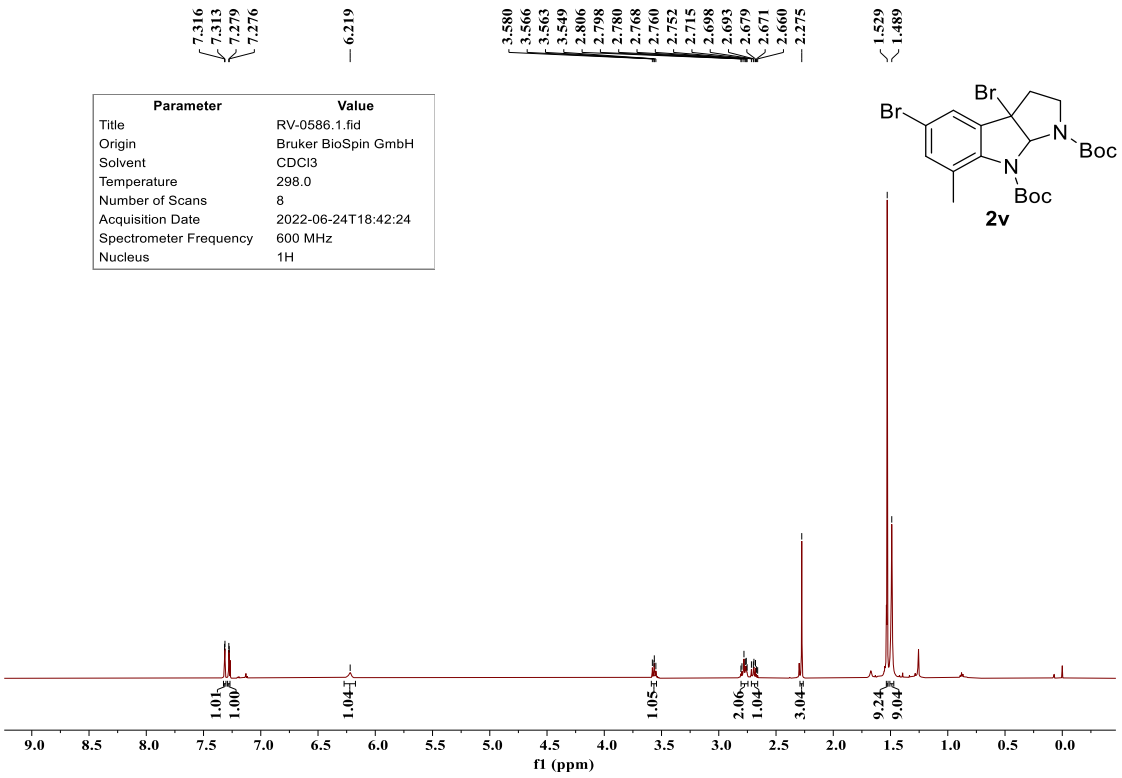


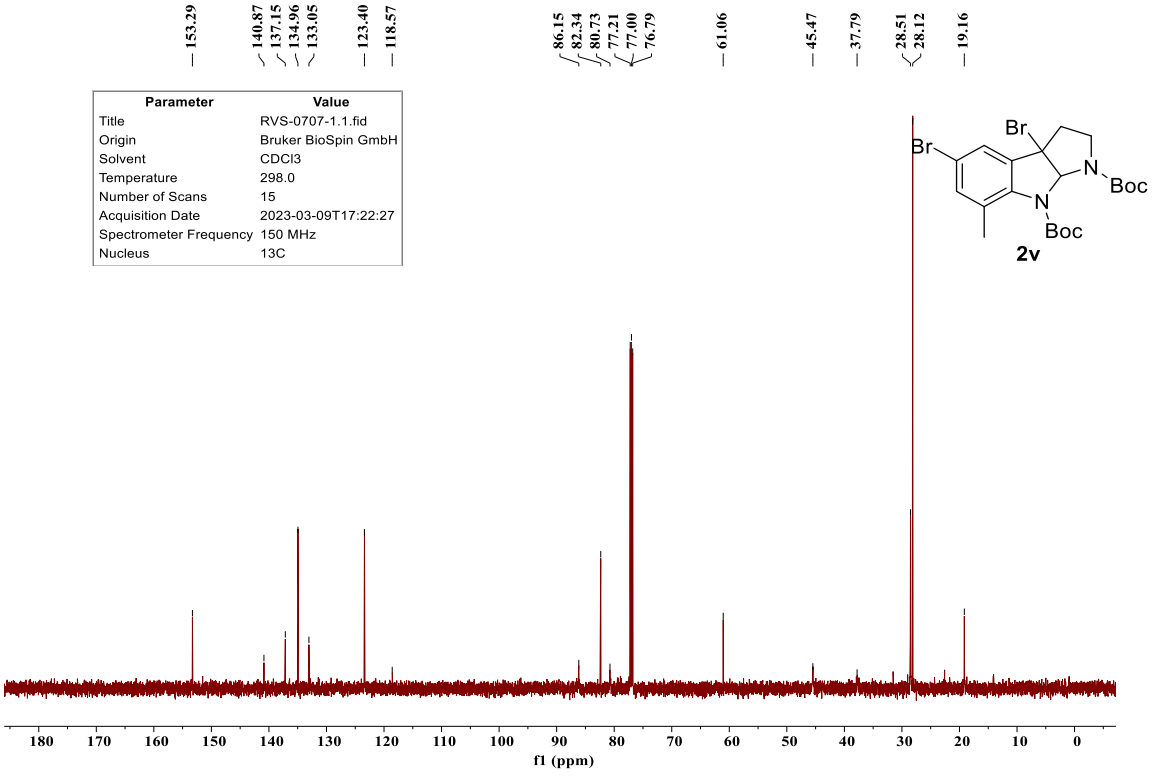

7.565
7.494
6.413
3.756
3.745
3.743
3.738
3.737
3.726
3.724
2.850
2.841
2.830
2.821
2.811
2.802
2.743
2.734
2.722
2.714
2.705
2.693
2.685
2.672
2.665
2.652
2.385
1.583
1.477
Parameter Value
Title RV-0596.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-07-13T20:33:31
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
N
Boc
N
Boc
2t
0.92
1.07
1.00
1.07
1.06
2.05
3.06
9.06
9.01
f1 (ppm)

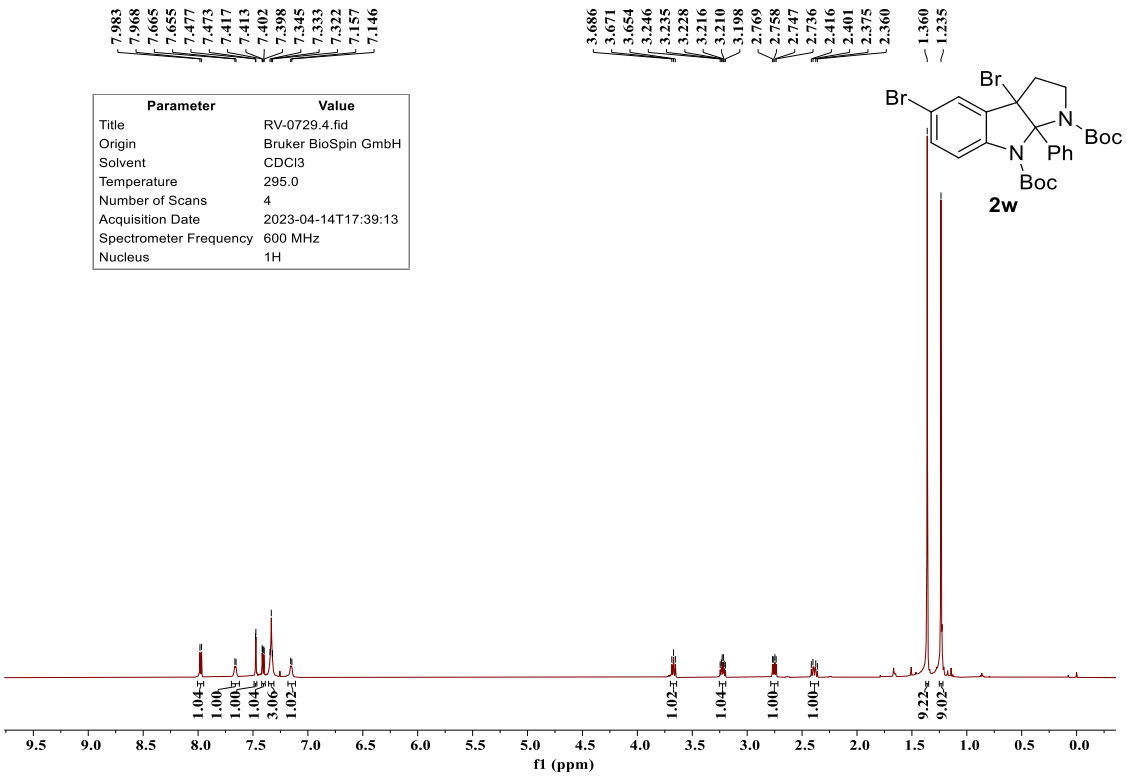

153.24
151.95
141.46
140.16
132.07
127.30
119.24
118.77
84.36
82.38
80.77
77.21
77.00
76.79
61.28
46.21
41.77
28.32
28.18
23.56
Parameter Value
Title RV-0596.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 512
Acquisition Date 2022-07-15T15:56:59
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
N
Boc
N
Boc
2t
f1 (ppm)


7.975
7.573
6.419
3.793
3.781
3.775
3.772
3.763
3.760
2.863
2.854
2.843
2.834
2.825
2.815
2.729
2.721
2.707
2.700
2.695
2.687
2.675
2.666
2.654
1.587
1.479
Parameter Value
Title RV-0730.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-04-20T06:19:07
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
N
Boc
N
Boc
2u
1.02
1.04
1.00
1.07
1.06
2.08
9.09
9.02
f1 (ppm)


153.10
151.46
141.95
133.69
128.40
126.54
121.93
118.52
84.47
82.98
81.00
77.21
77.00
76.79
60.37
46.19
41.91
28.29
28.11
Parameter Value
Title RV-0730.2.1.1r
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 512
Acquisition Date 2023-04-20T06:44:17
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
N
Boc
N
Boc
2u
f1 (ppm)


7.316
7.313
7.279
7.276
6.219
3.580
3.566
3.563
3.549
2.806
2.798
2.780
2.768
2.760
2.752
2.715
2.698
2.693
2.679
2.671
2.660
2.275
1.529
1.489
Parameter Value
Title RV-0586.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-06-24T18:42:24
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
N
Boc
N
Boc
2v
1.01
1.00
1.04
1.05
2.06
1.04
3.04
9.24
9.04
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


153.29
140.87
137.15
134.96
133.05
123.40
118.57
86.15
82.34
80.73
77.21
77.00
76.79
61.06
45.47
37.79
28.51
28.12
19.16
Parameter Value
Title RVS-0707-1.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 15
Acquisition Date 2023-03-09T17:22:27
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
N
Boc
N
Boc
2v
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.983
7.968
7.665
7.655
7.477
7.473
7.417
7.413
7.402
7.398
7.345
7.333
7.322
7.157
7.146
3.686
3.671
3.654
3.246
3.235
3.228
3.216
3.210
3.198
2.769
2.758
2.747
2.736
2.416
2.401
2.375
2.360
1.360
1.235
Parameter Value
Title RV-0729.4.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-04-14T17:39:13
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
Br
N
Boc
N
Ph
Boc
2w
1.04
1.00
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3.06
1.02
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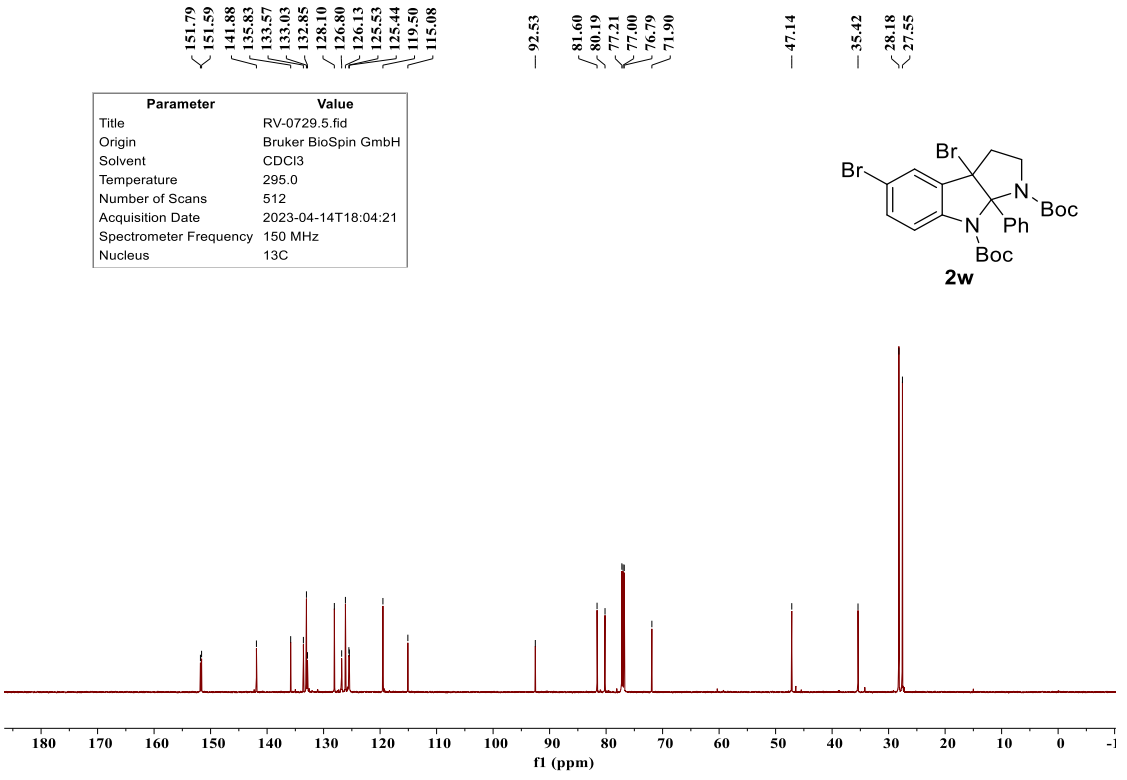
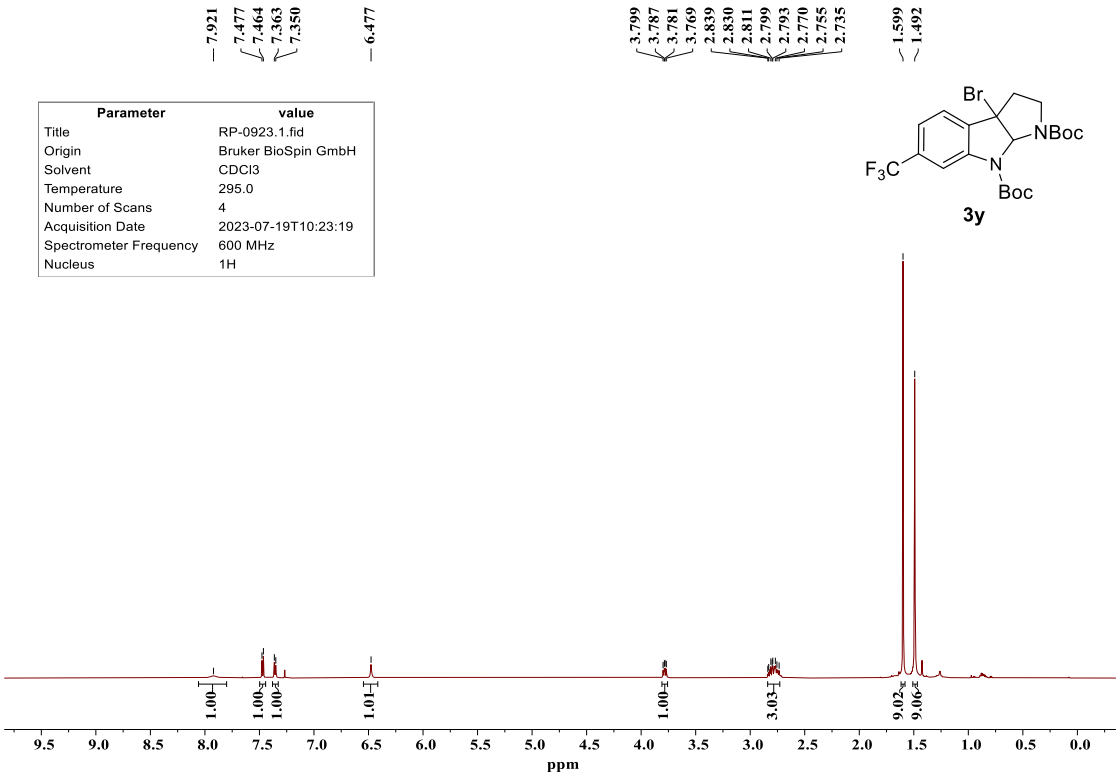
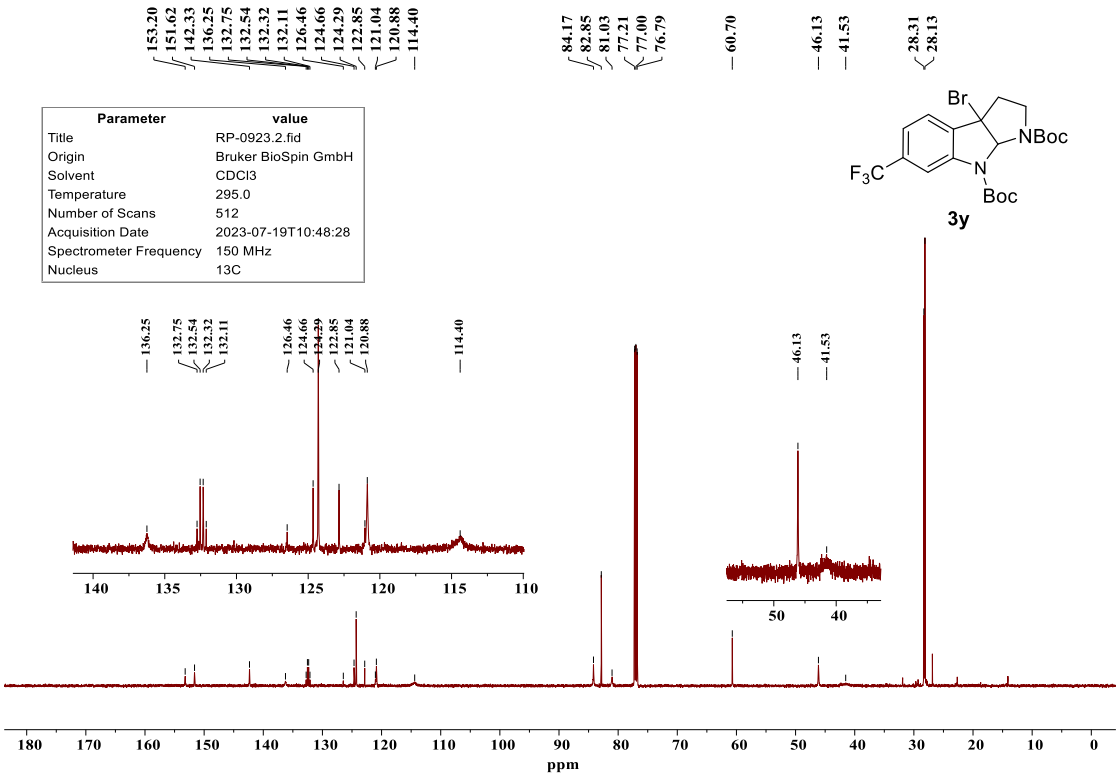
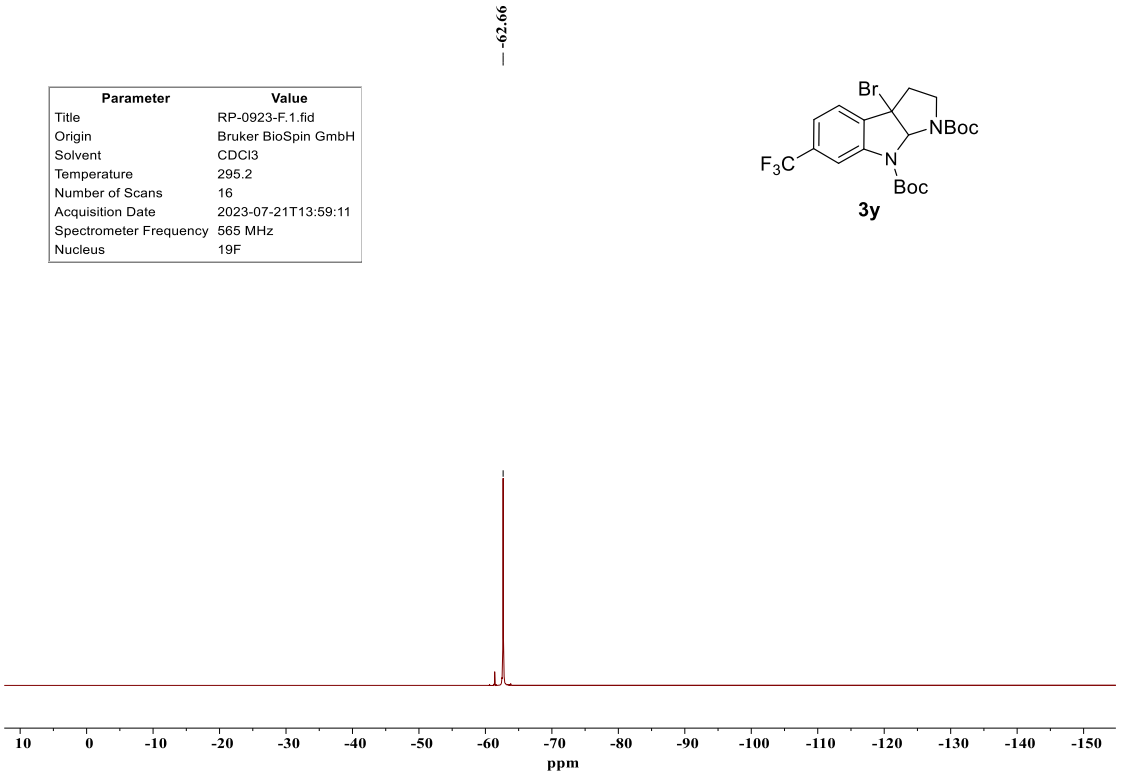
1.04
1.00
1.00
9.22
9.02
f1 (ppm)


151.79
151.59
141.88
135.83
133.57
133.03
132.85
128.10
126.80
126.13
125.53
125.44
119.50
115.08
92.53
81.60
80.19
77.21
77.00
76.79
71.90
47.14
35.42
28.18
27.55
Parameter Value
Title RV-0729.5.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 512
Acquisition Date 2023-04-14T18:04:21
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
Br
N
Boc
N
Ph
Boc
2w
f1 (ppm)

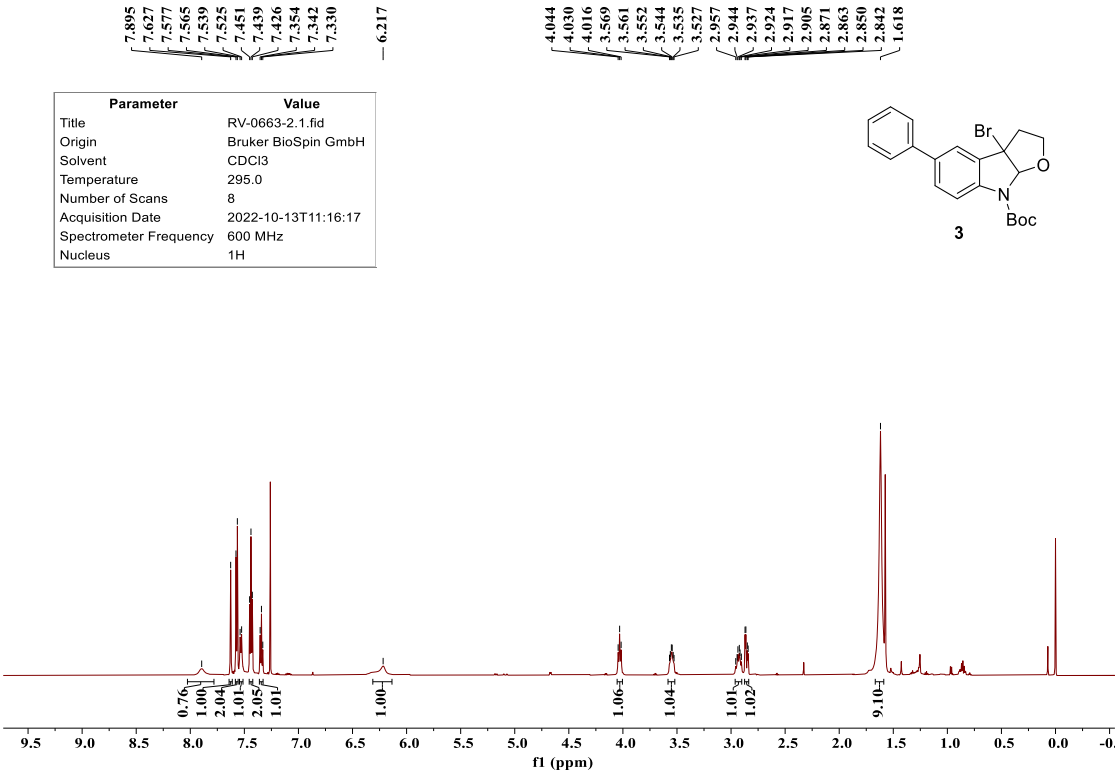

7.921
7.477
7.464
7.363
7.350
6.477
3.799
3.787
3.781
3.769
2.839
2.830
2.811
2.799
2.793
2.770
2.755
2.735
1.599
1.492
Parameter value
Title RP-0923.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-07-19T10:23:19
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
NBoc
F3C
N
Boc
3y
1.00
1.00
1.00
1.01
1.00
3.03
9.02
9.06
ppm

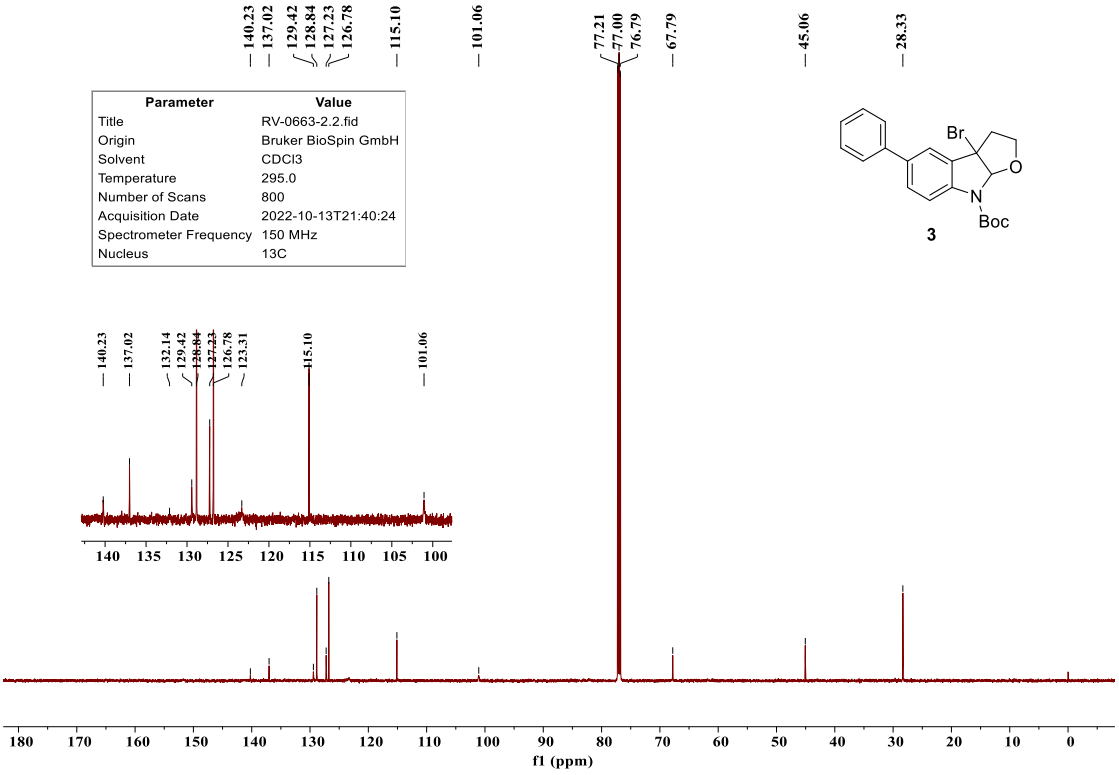

153.20
151.62
142.33
136.25
132.75
132.54
132.32
132.11
126.46
124.66
124.29
122.85
121.04
120.88
114.40
84.17
82.85
81.03
77.21
77.00
76.79
60.70
46.13
41.53
28.31
28.13
Parameter value
Title RP-0923.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 512
Acquisition Date 2023-07-19T10:48:28
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
NBoc
F3C
N
Boc
3y
ppm

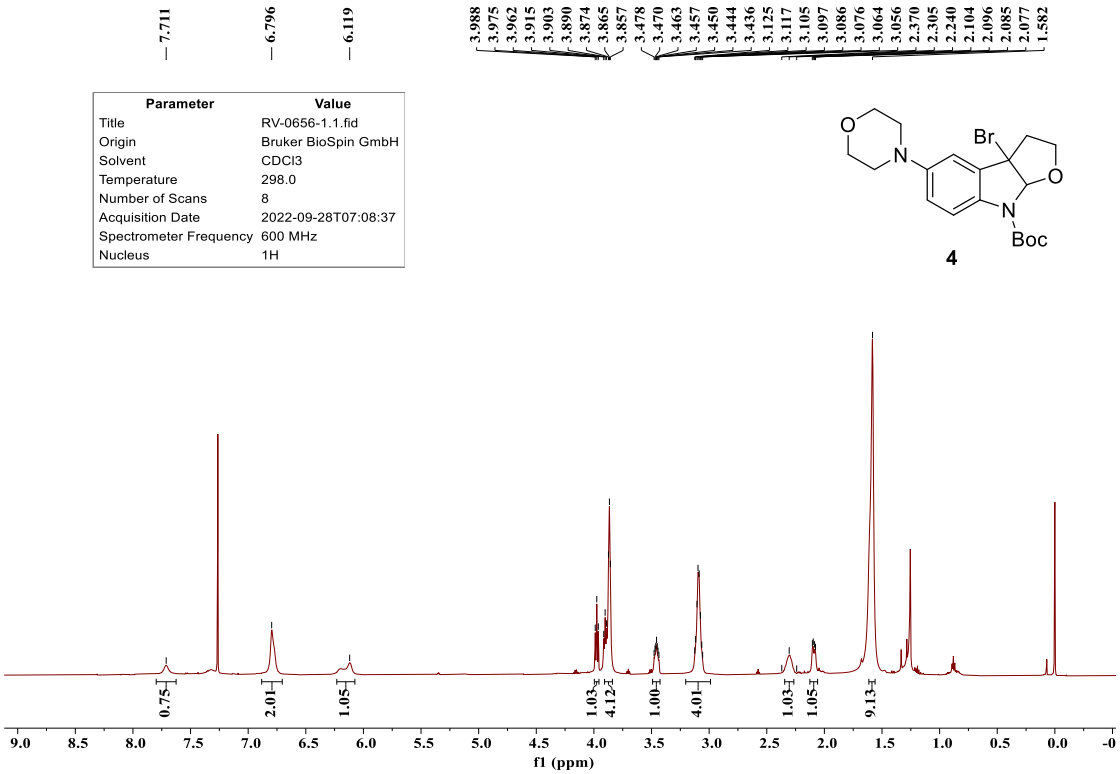
Parameter	Value
Title	RP-0923-F.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	295.2
Number of Scans	16
Acquisition Date	2023-07-21T13:59:11
Spectrometer Frequency	565 MHz
Nucleus	19F

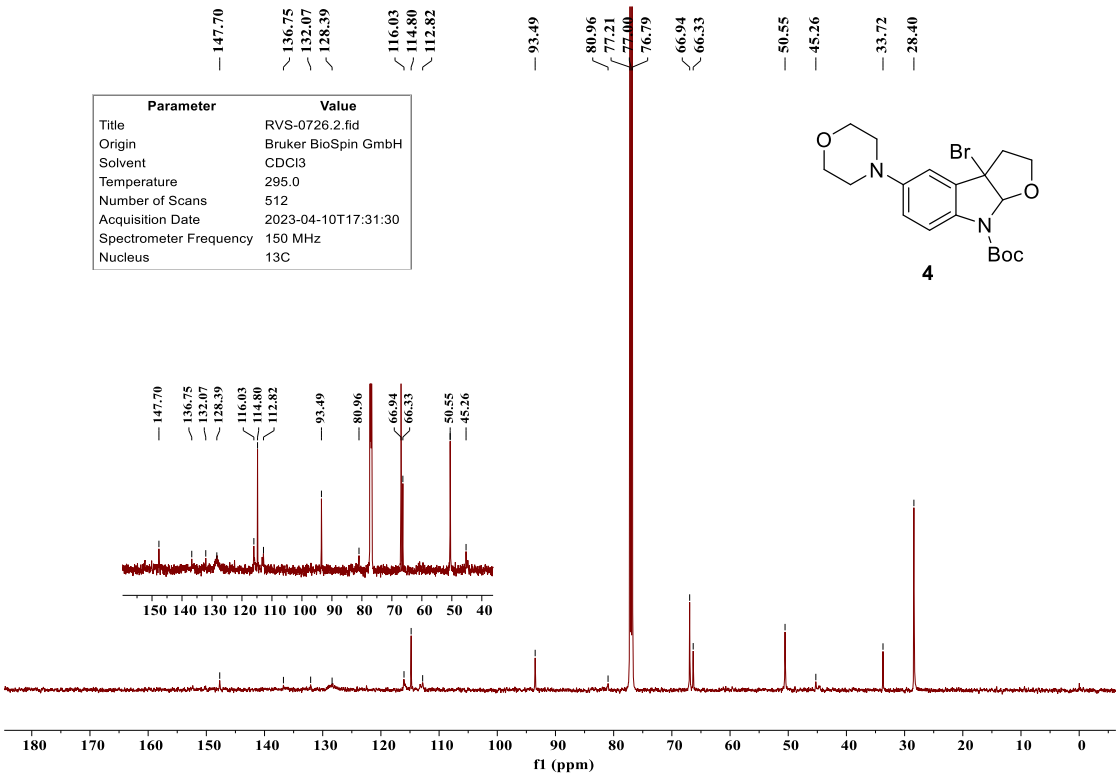


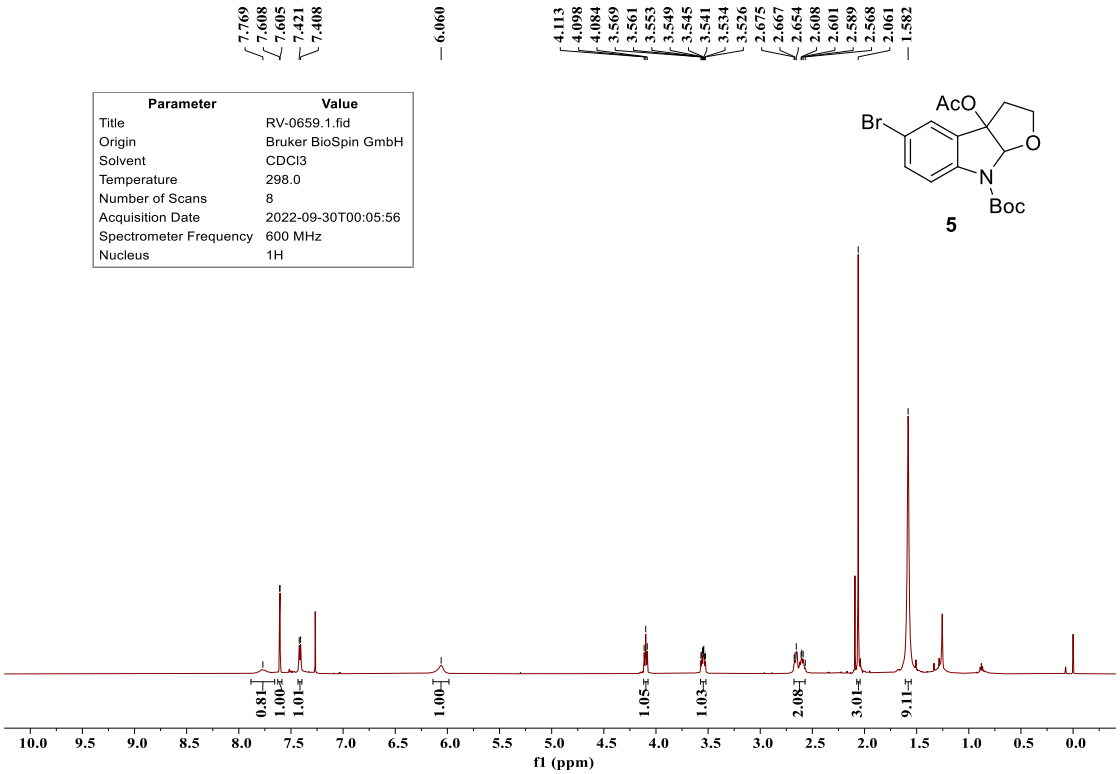
3y

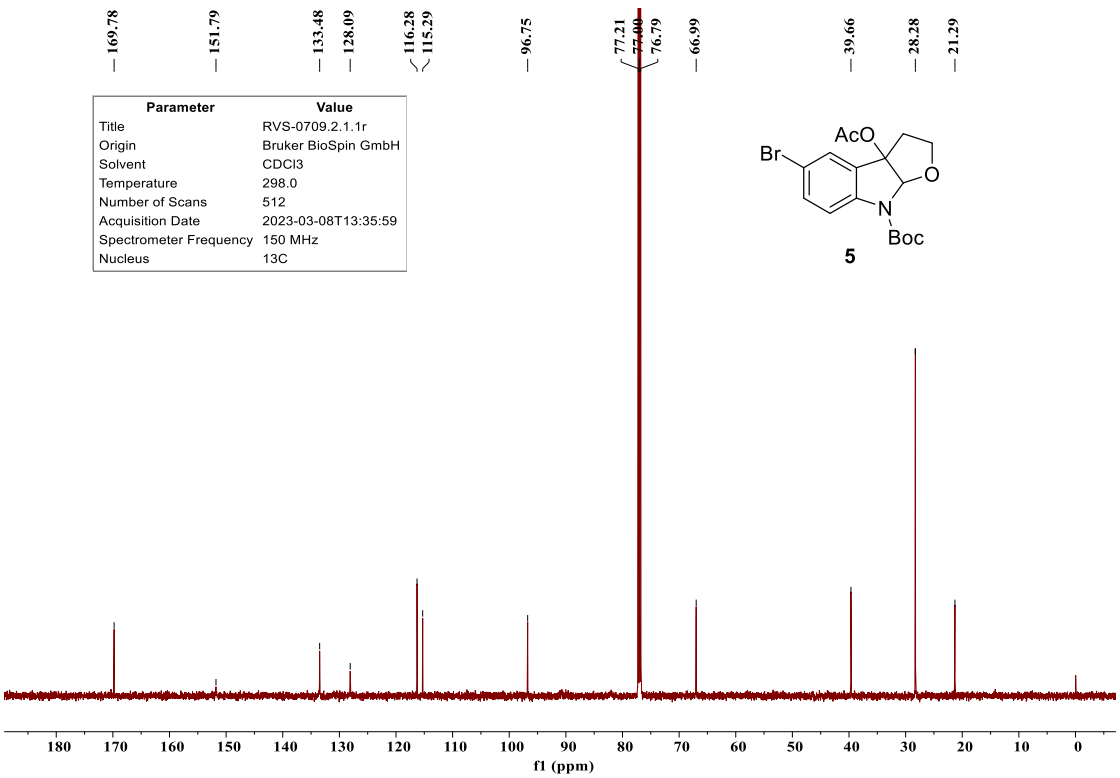

7.895
7.627
7.577
7.565
7.539
7.525
7.451
7.439
7.426
7.354
7.342
7.330
6.217
4.044
4.030
4.016
3.569
3.561
3.552
3.544
3.535
3.527
2.957
2.944
2.937
2.924
2.917
2.905
2.871
2.863
2.850
2.842
1.618
Parameter Value
Title RV-0663-2.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 8
Acquisition Date 2022-10-13T11:16:17
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
O
N
Boc
3
0.76
1.00
2.04
1.01
2.05
1.01
1.00
1.06
1.04
1.01
1.02
9.10
f1 (ppm)

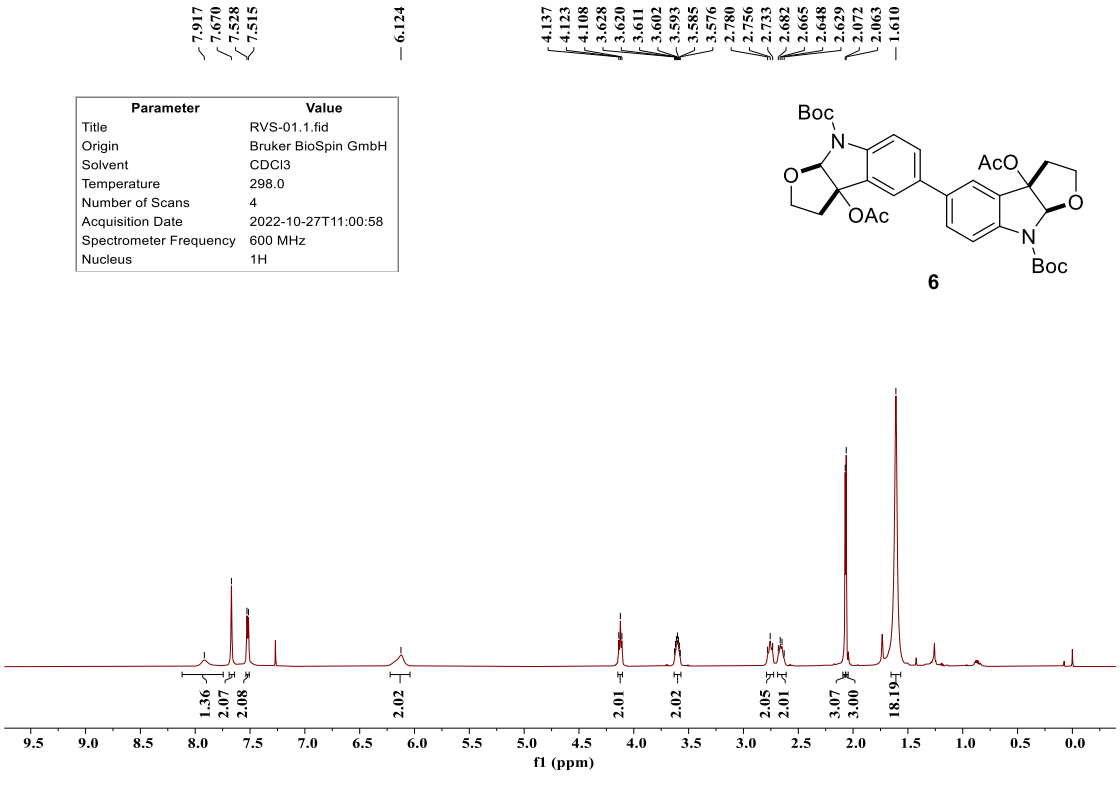

140.23
137.02
129.42
128.84
127.23
126.78
115.10
101.06
77.21
77.00
76.79
67.79
45.06
28.33
Parameter Value
Title RV-0663-2.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 800
Acquisition Date 2022-10-13T21:40:24
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
O
N
Boc
3
140.23
137.02
132.14
129.42
128.84
127.23
126.78
123.31
115.10
101.06
f1 (ppm)

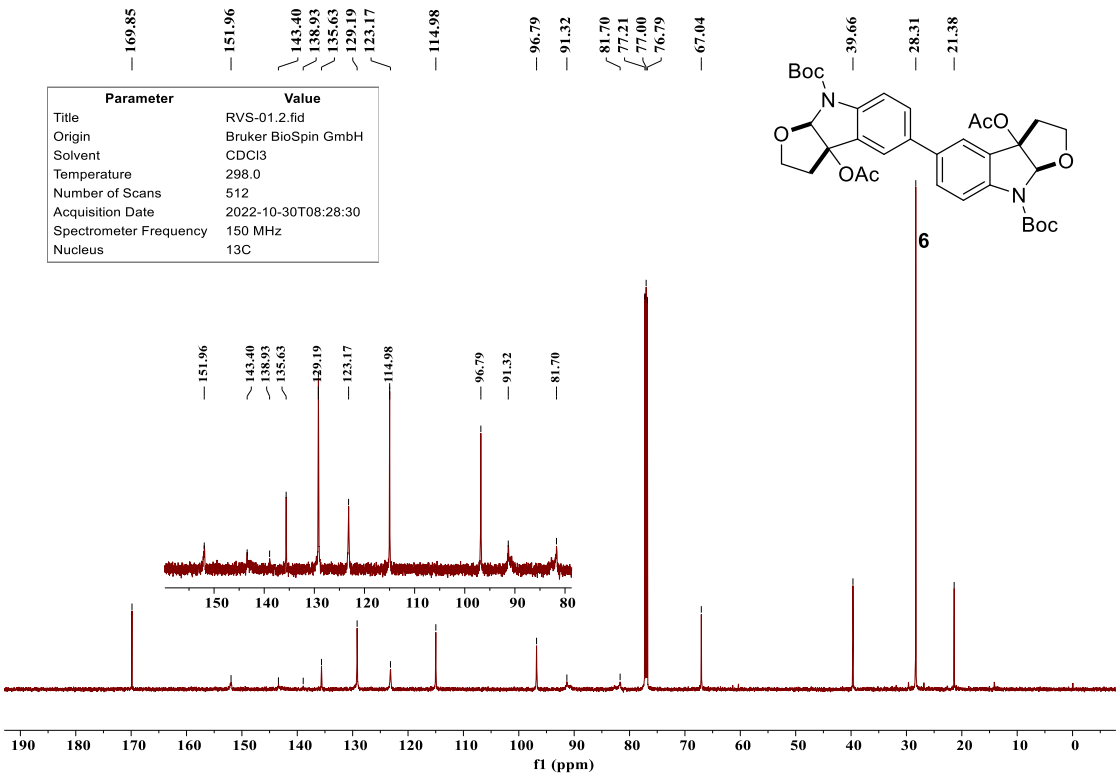

7.711
6.796
6.119
3.988
3.975
3.962
3.915
3.903
3.890
3.874
3.865
3.857
3.478
3.470
3.463
3.457
3.450
3.444
3.436
3.125
3.117
3.105
3.097
3.086
3.076
3.064
3.056
2.370
2.305
2.240
2.104
2.096
2.085
2.077
1.582
Parameter Value
Title RV-0656-1.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-09-28T07:08:37
Spectrometer Frequency 600 MHz
Nucleus 1H
O
N
Br
O
N
Boc
4
0.75
2.01
1.05
1.03
4.12
1.00
4.01
1.03
1.05
9.13
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

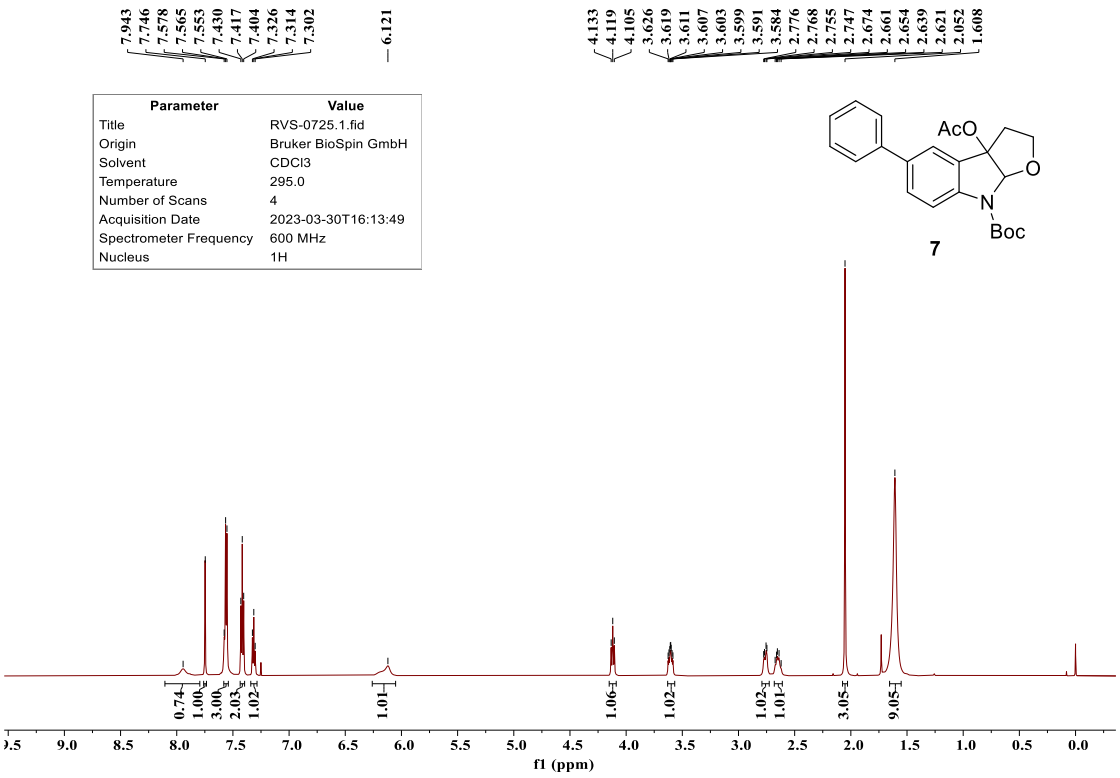

147.70
136.75
132.07
128.39
116.03
114.80
112.82
93.49
80.96
77.21
77.00
76.79
66.94
66.33
50.55
45.26
33.72
28.40
Parameter Value
Title RVS-0726.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 512
Acquisition Date 2023-04-10T17:31:30
Spectrometer Frequency 150 MHz
Nucleus 13C
O
N
Br
O
N
Boc
4
150 140 130 120 110 100 90 80 70 60 50 40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

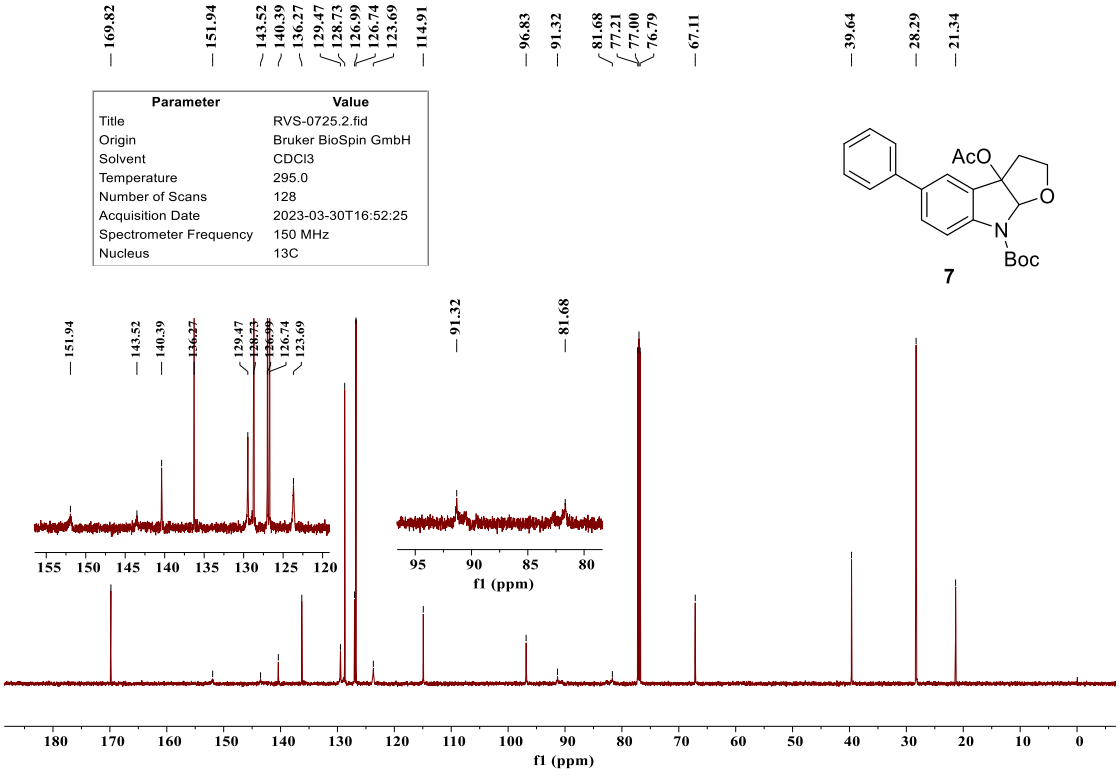

7.769
7.608
7.605
7.421
7.408
6.060
4.113
4.098
4.084
3.569
3.561
3.553
3.549
3.545
3.541
3.534
3.526
2.675
2.667
2.654
2.608
2.601
2.589
2.568
2.061
1.582
Parameter Value
Title RV-0659.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 8
Acquisition Date 2022-09-30T00:05:56
Spectrometer Frequency 600 MHz
Nucleus 1H
AcO
Br
O
N
Boc
5
0.81
1.00
1.01
1.00
1.05
1.03
2.08
3.01
9.11
f1 (ppm)

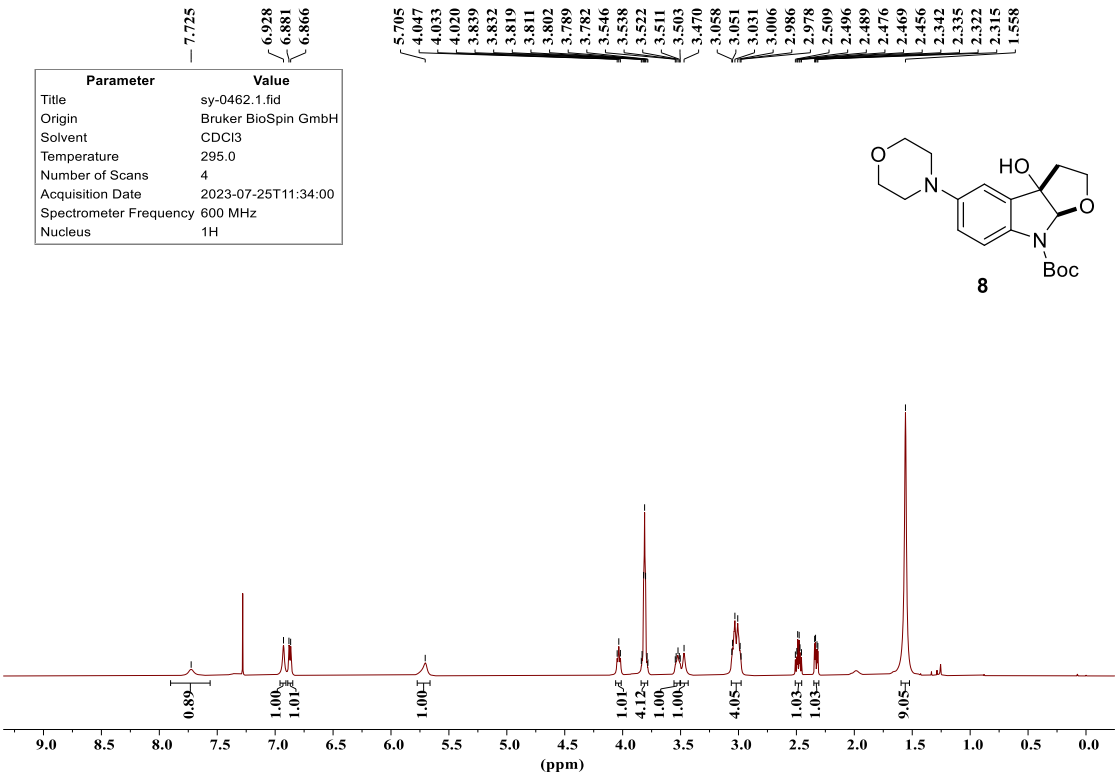

169.78
151.79
133.48
128.09
116.28
115.29
96.75
77.21
77.00
76.79
66.99
39.66
28.28
21.29
Parameter Value
Title RVS-0709.2.1.1r
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 512
Acquisition Date 2023-03-08T13:35:59
Spectrometer Frequency 150 MHz
Nucleus 13C
AcO
Br
O
N
Boc
5
f1 (ppm)

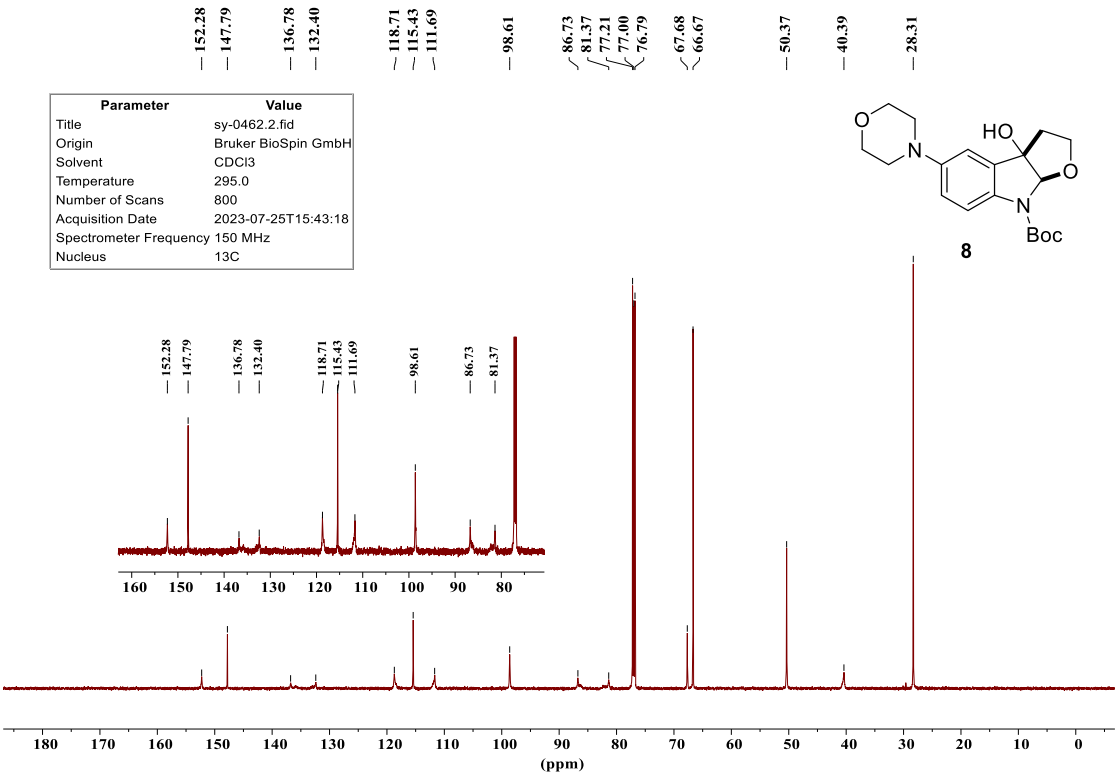

7.917
7.670
7.528
7.515
6.124
4.137
4.123
4.108
3.628
3.620
3.611
3.602
3.593
3.585
3.576
2.780
2.756
2.733
2.682
2.665
2.648
2.629
2.072
2.063
1.610
Parameter Value
Title RVS-01.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 4
Acquisition Date 2022-10-27T11:00:58
Spectrometer Frequency 600 MHz
Nucleus 1H
Boc
N
O
OAc
AcO
O
N
Boc
6
1.36
2.07
2.08
2.02
2.01
2.02
2.05
2.01
3.07
3.00
18.19
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

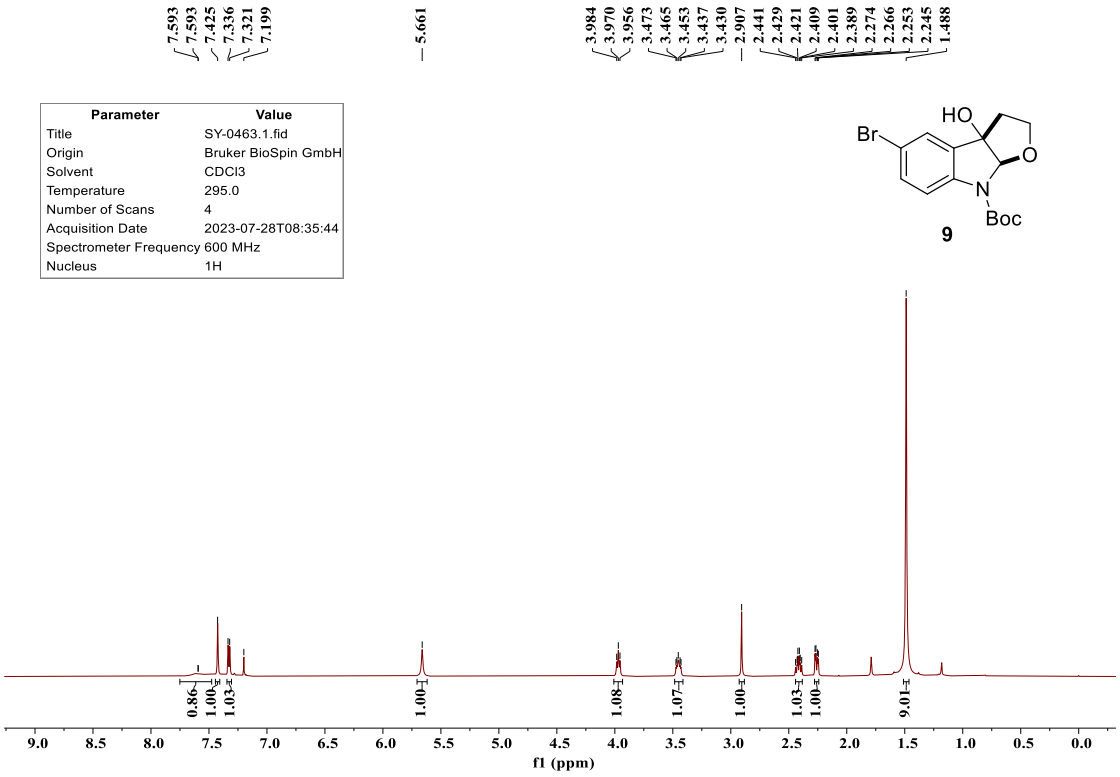

169.85
151.96
143.40
138.93
135.63
129.19
123.17
114.98
96.79
91.32
81.70
77.21
77.00
76.79
67.04
39.66
28.31
21.38
Parameter Value
Title RVS-01.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 512
Acquisition Date 2022-10-30T08:28:30
Spectrometer Frequency 150 MHz
Nucleus 13C
Boc
N
O
OAc
AcO
O
N
Boc
6
151.96
143.40
138.93
135.63
129.19
123.17
114.98
96.79
91.32
81.70
150 140 130 120 110 100 90 80
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

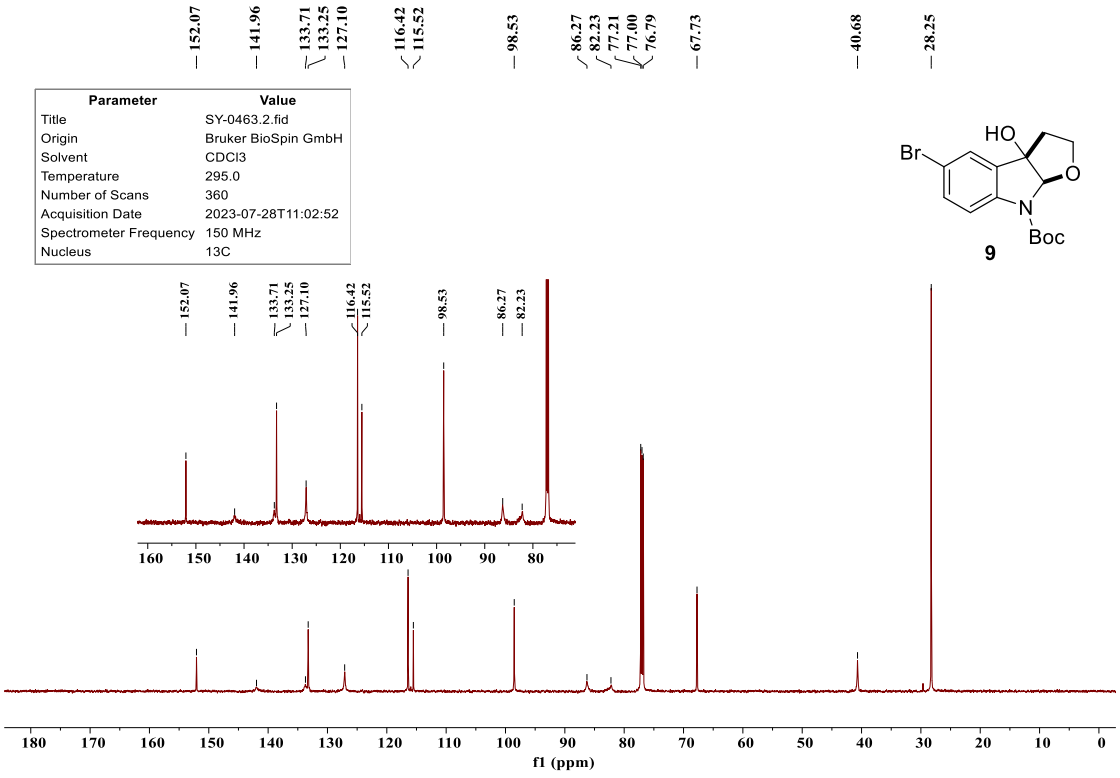

7.943
7.746
7.578
7.565
7.553
7.430
7.417
7.404
7.326
7.314
7.302
6.121
4.133
4.119
4.105
3.626
3.619
3.611
3.607
3.603
3.599
3.591
3.584
2.776
2.768
2.755
2.747
2.674
2.661
2.654
2.639
2.621
2.052
1.608
Parameter Value
Title RVS-0725.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-03-30T16:13:49
Spectrometer Frequency 600 MHz
Nucleus 1H
AcO
O
N
Boc
7
0.74
1.00
3.00
2.03
1.02
1.01
1.06
1.02
1.02
1.01
3.05
9.05
f1 (ppm)

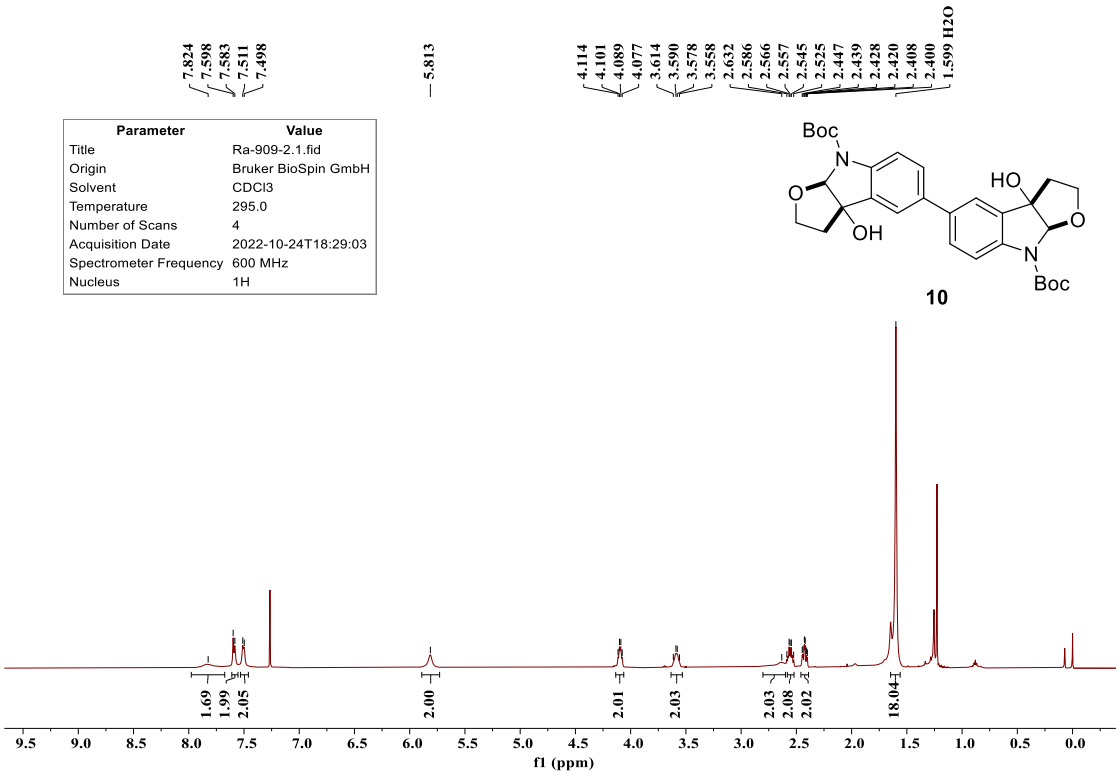

169.82
151.94
143.52
140.39
136.27
129.47
128.73
126.99
126.74
123.69
114.91
96.83
91.32
81.68
77.21
77.00
76.79
67.11
39.64
28.29
21.34
Parameter Value
Title RVS-0725.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 128
Acquisition Date 2023-03-30T16:52:25
Spectrometer Frequency 150 MHz
Nucleus 13C
AcO
O
N
Boc
7
f1 (ppm)

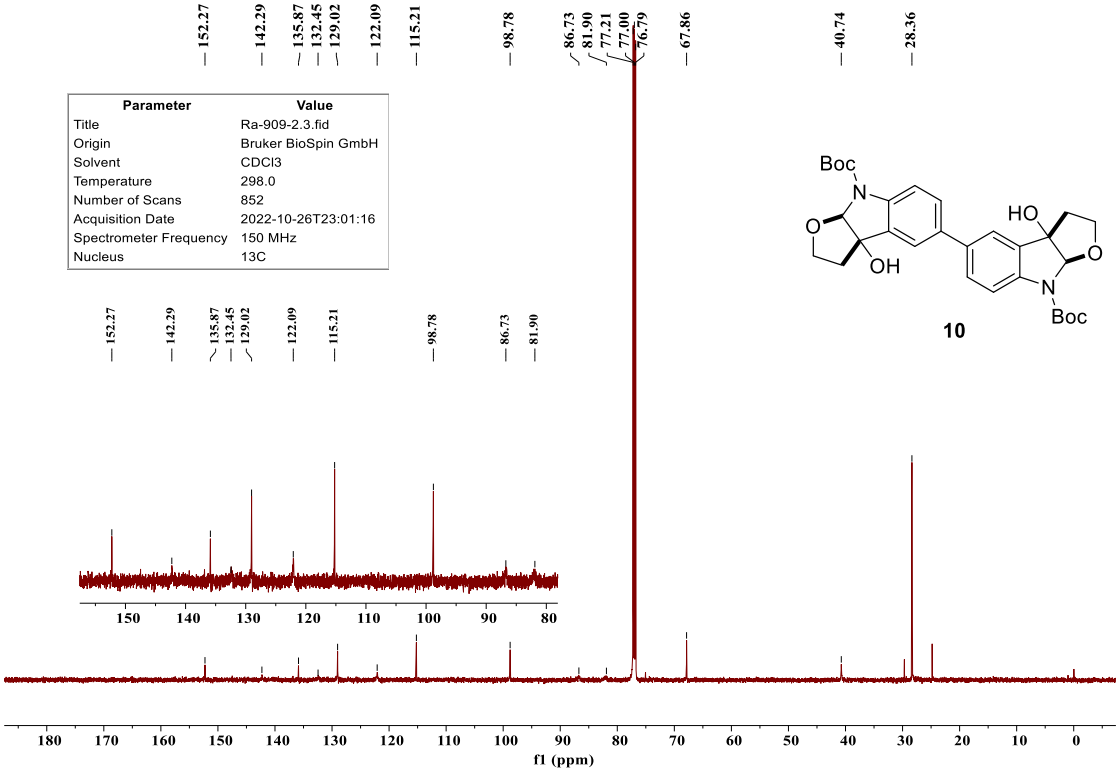

7.725
6.928
6.881
6.866
5.705
4.047
4.033
4.020
3.839
3.832
3.819
3.811
3.802
3.789
3.782
3.546
3.538
3.522
3.511
3.503
3.470
3.058
3.051
3.031
3.006
2.986
2.978
2.509
2.496
2.489
2.476
2.469
2.456
2.342
2.335
2.322
2.315
1.558
Parameter Value
Title sy-0462.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-07-25T11:34:00
Spectrometer Frequency 600 MHz
Nucleus 1H
O
N
HO
O
N
Boc
8
0.89
1.00
1.01
1.00
1.01
4.12
1.00
1.00
4.05
1.03
1.03
9.05
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

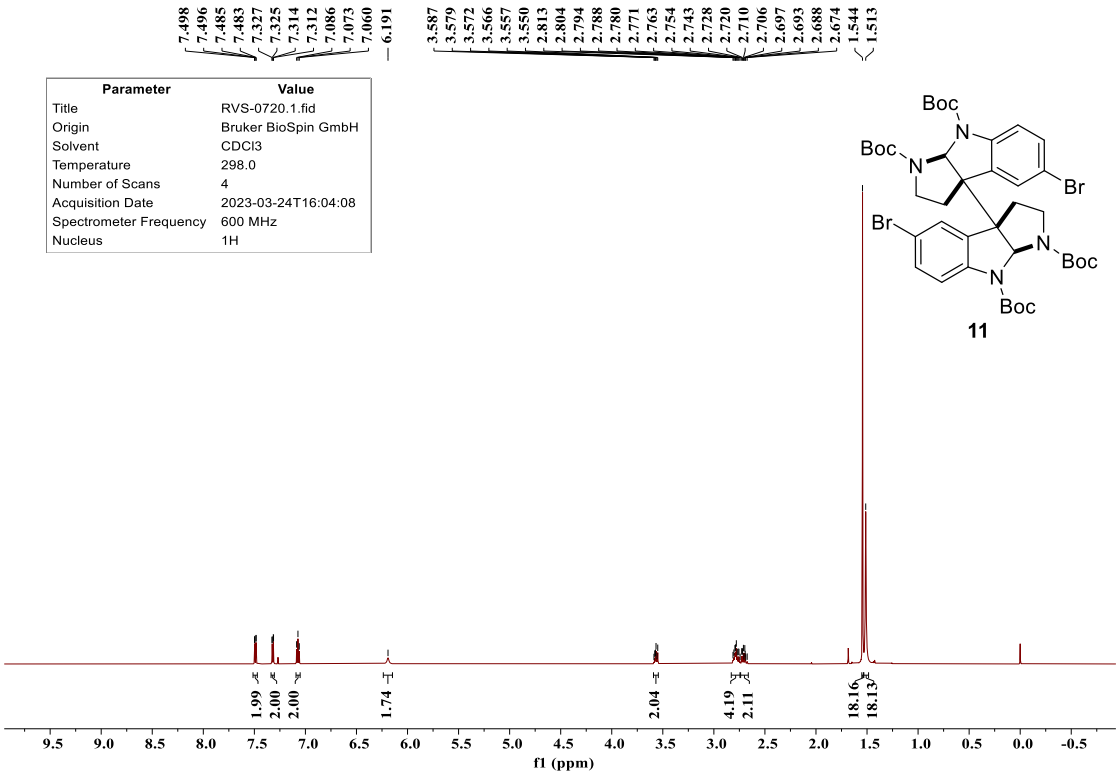

152.28
147.79
136.78
132.40
118.71
115.43
111.69
98.61
86.73
81.37
77.21
77.00
76.79
67.68
66.67
50.37
40.39
28.31
Parameter Value
Title sy-0462.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 800
Acquisition Date 2023-07-25T15:43:18
Spectrometer Frequency 150 MHz
Nucleus 13C
O
N
HO
O
N
Boc
8
160 150 140 130 120 110 100 90 80
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

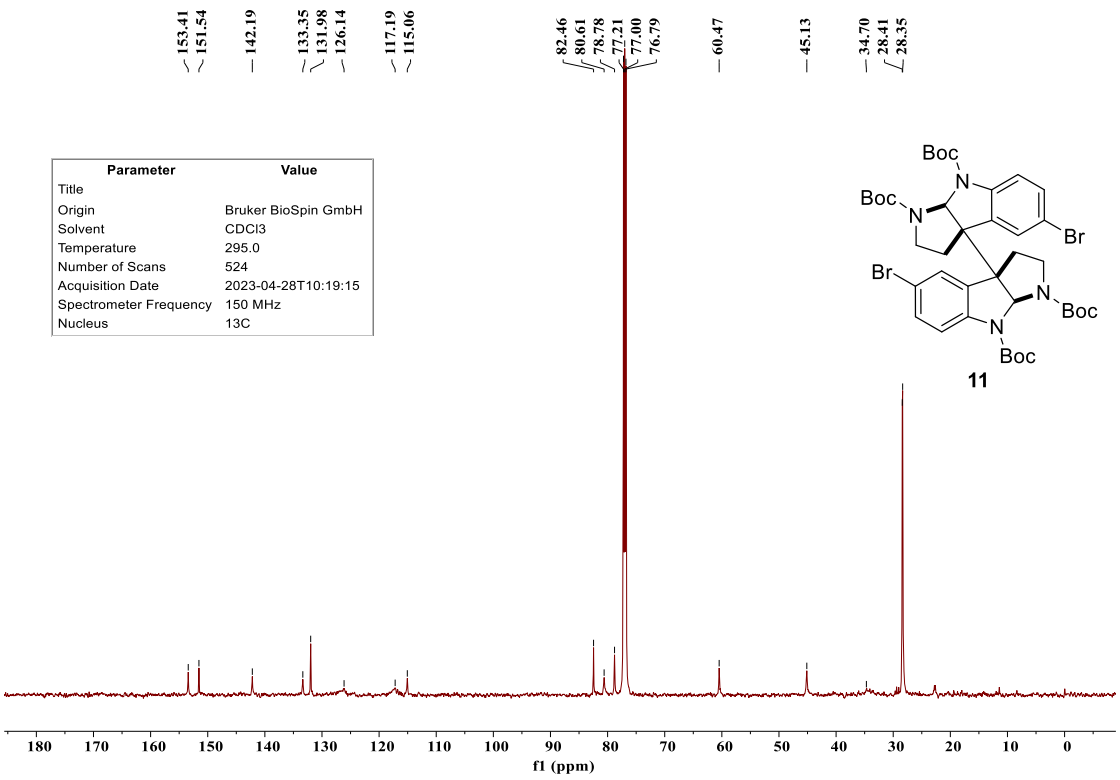

7.593
7.593
7.425
7.336
7.321
7.199
5.661
3.984
3.970
3.956
3.473
3.465
3.453
3.437
3.430
2.907
2.441
2.429
2.421
2.409
2.401
2.389
2.274
2.266
2.253
2.245
1.488
Parameter Value
Title SY-0463.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-07-28T08:35:44
Spectrometer Frequency 600 MHz
Nucleus 1H
HO
Br
O
N
Boc
9
0.86
1.00
1.03
1.00
1.08
1.07
1.00
1.03
1.00
9.01
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

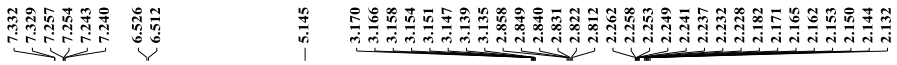

152.07
141.96
133.71
133.25
127.10
116.42
115.52
98.53
86.27
82.23
77.21
77.00
76.79
67.73
40.68
28.25
Parameter Value
Title SY-0463.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 360
Acquisition Date 2023-07-28T11:02:52
Spectrometer Frequency 150 MHz
Nucleus 13C
HO
Br
O
N
Boc
9
160 150 140 130 120 110 100 90 80
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.824
7.598
7.583
7.511
7.498
5.813
4.114
4.101
4.089
4.077
3.614
3.590
3.578
3.558
2.632
2.586
2.566
2.557
2.545
2.525
2.447
2.439
2.428
2.420
2.408
2.400
1.599 H2O
Parameter Value
Title Ra-909-2.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2022-10-24T18:29:03
Spectrometer Frequency 600 MHz
Nucleus 1H
Boc
N
O
OH
HO
O
N
Boc
10
1.69
1.99
2.05
2.00
2.01
2.03
2.03
2.08
2.02
18.04
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

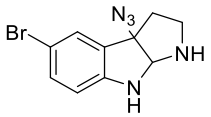

152.27
142.29
135.87
132.45
129.02
122.09
115.21
98.78
86.73
81.90
77.21
77.00
76.79
67.86
40.74
28.36
Parameter Value
Title Ra-909-2.3.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 852
Acquisition Date 2022-10-26T23:01:16
Spectrometer Frequency 150 MHz
Nucleus 13C
Boc
N
O
OH
HO
O
N
Boc
10
152.27
142.29
135.87
132.45
129.02
122.09
115.21
98.78
86.73
81.90
150 140 130 120 110 100 90 80
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.498
7.496
7.485
7.483
7.327
7.325
7.314
7.312
7.086
7.073
7.060
6.191
3.587
3.579
3.572
3.566
3.557
3.550
2.813
2.804
2.794
2.788
2.780
2.771
2.763
2.754
2.743
2.728
2.720
2.710
2.706
2.697
2.693
2.688
2.674
1.544
1.513
Parameter Value
Title RVS-0720.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 4
Acquisition Date 2023-03-24T16:04:08
Spectrometer Frequency 600 MHz
Nucleus 1H
Boc
N
Br
11
1.99
2.00
2.00
1.74
2.04
4.19
2.11
18.16
18.13
f1 (ppm)

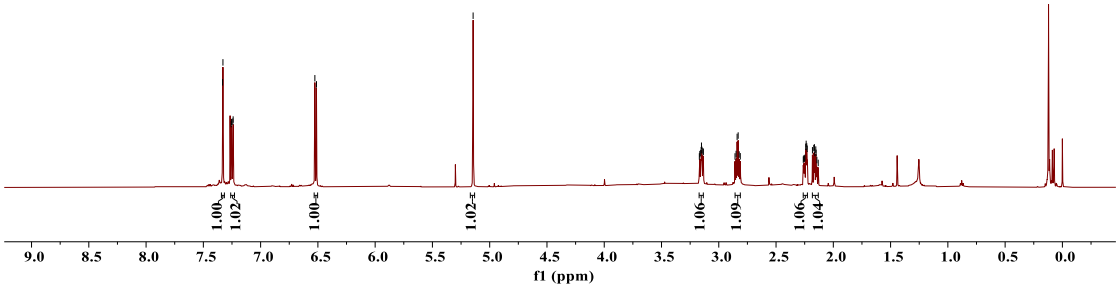

153.41
151.54
142.19
133.35
131.98
126.14
117.19
115.06
82.46
80.61
78.78
77.21
77.00
76.79
60.47
45.13
34.70
28.41
28.35
Parameter Value
Title
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 524
Acquisition Date 2023-04-28T10:19:15
Spectrometer Frequency 150 MHz
Nucleus 13C
Boc
N
Br
11
f1 (ppm)

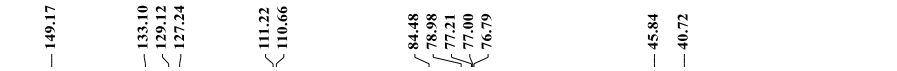


Parameter	Value
Title	RV-0694.2.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	4
Acquisition Date	2023-02-07T19:23:11
Spectrometer Frequency	600 MHz
Nucleus	1H

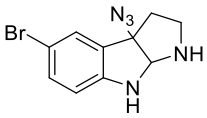


12

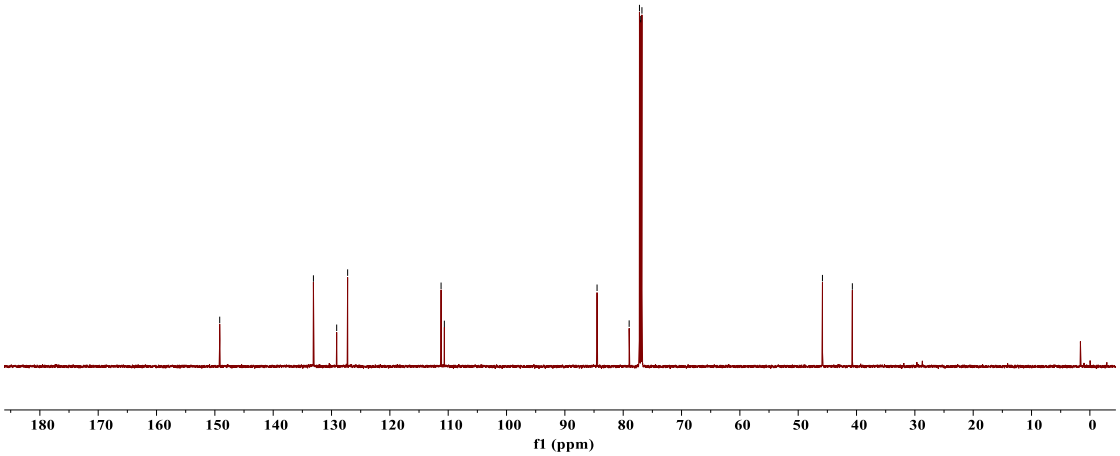


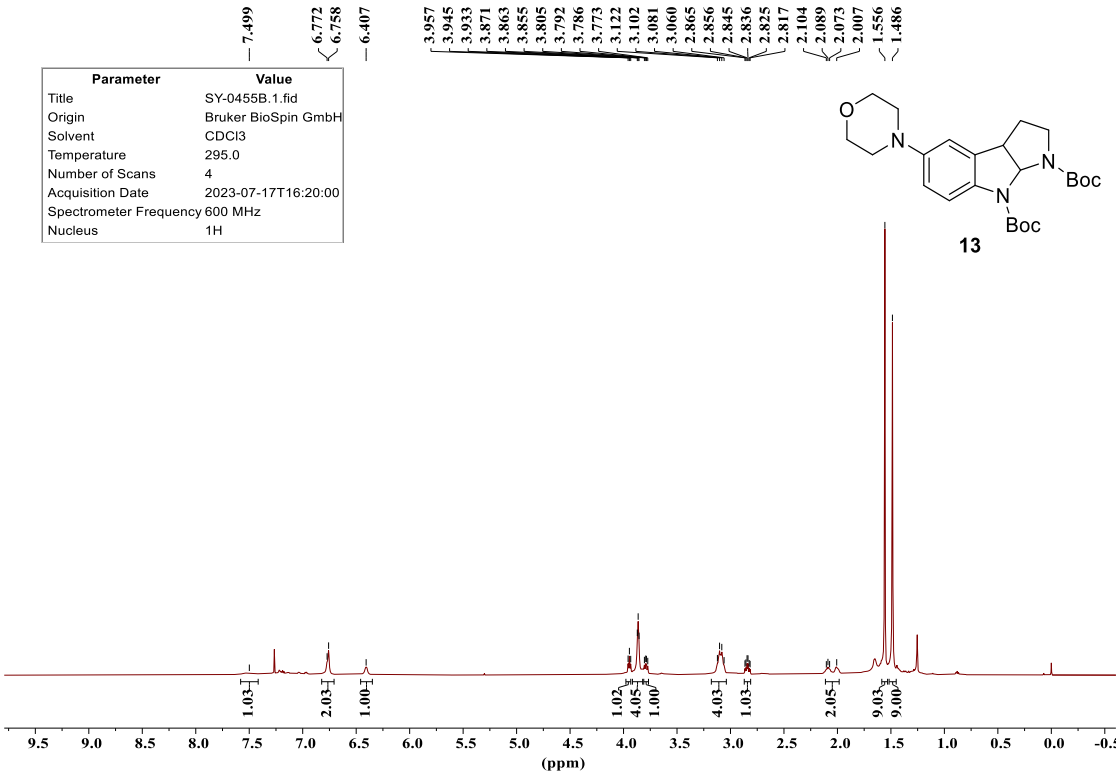


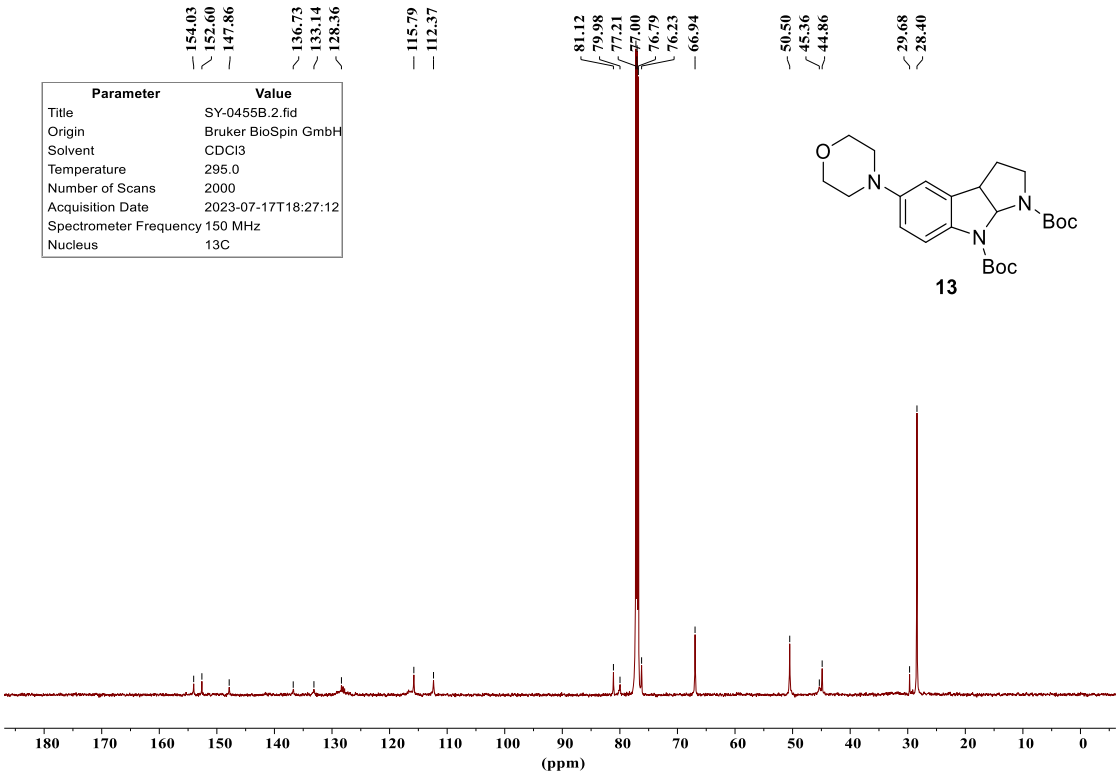
Parameter	Value
Title	RV-0694-1.1.fid
Origin	Bruker BioSpin GmbH
Solvent	CDCl3
Temperature	298.0
Number of Scans	512
Acquisition Date	2023-02-09T07:28:45
Spectrometer Frequency	150 MHz
Nucleus	13C

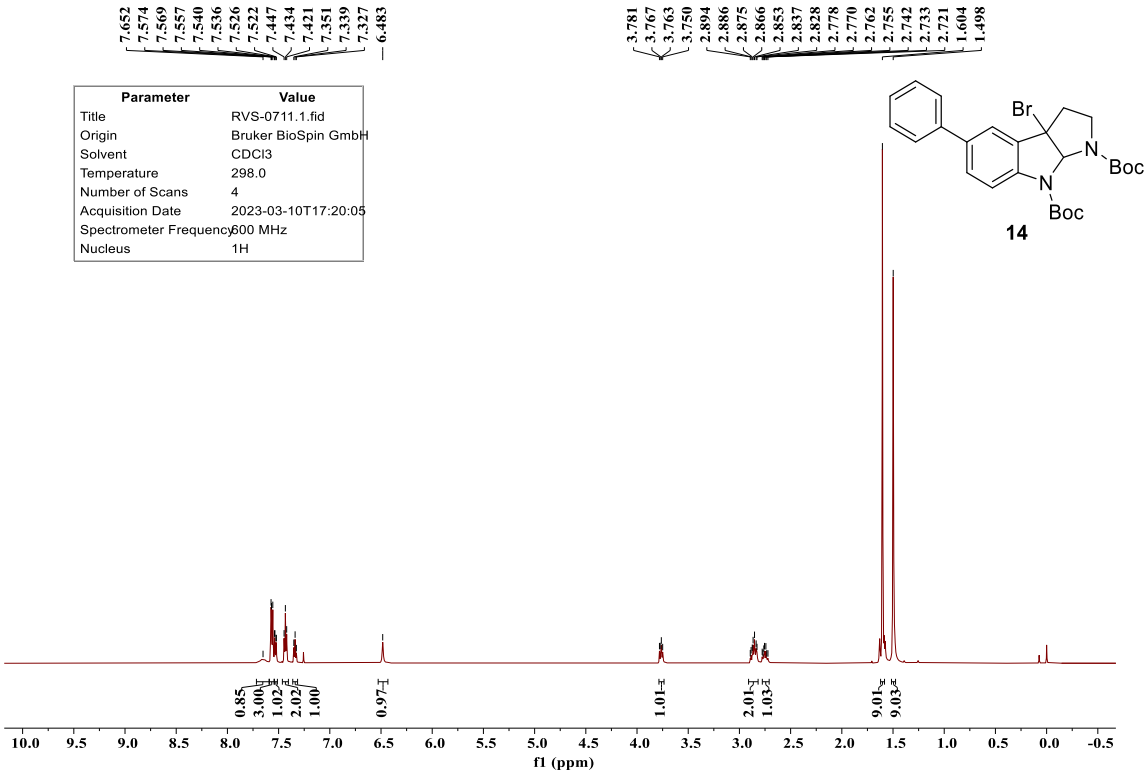


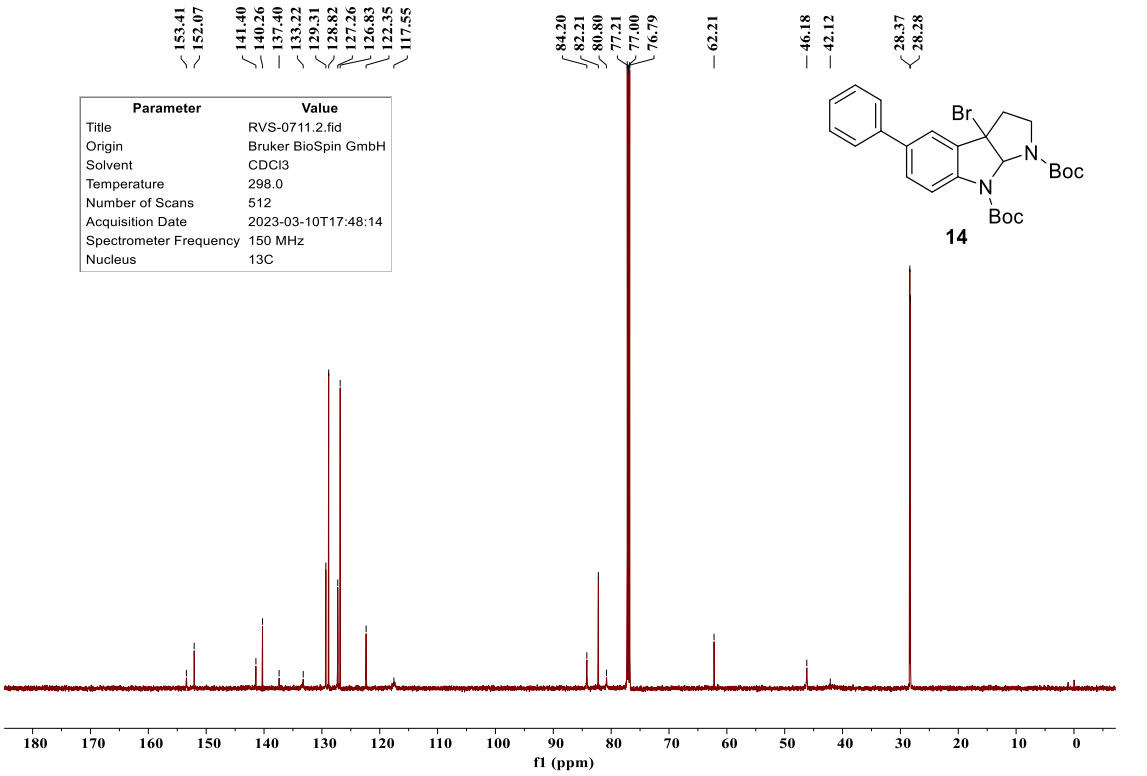
12

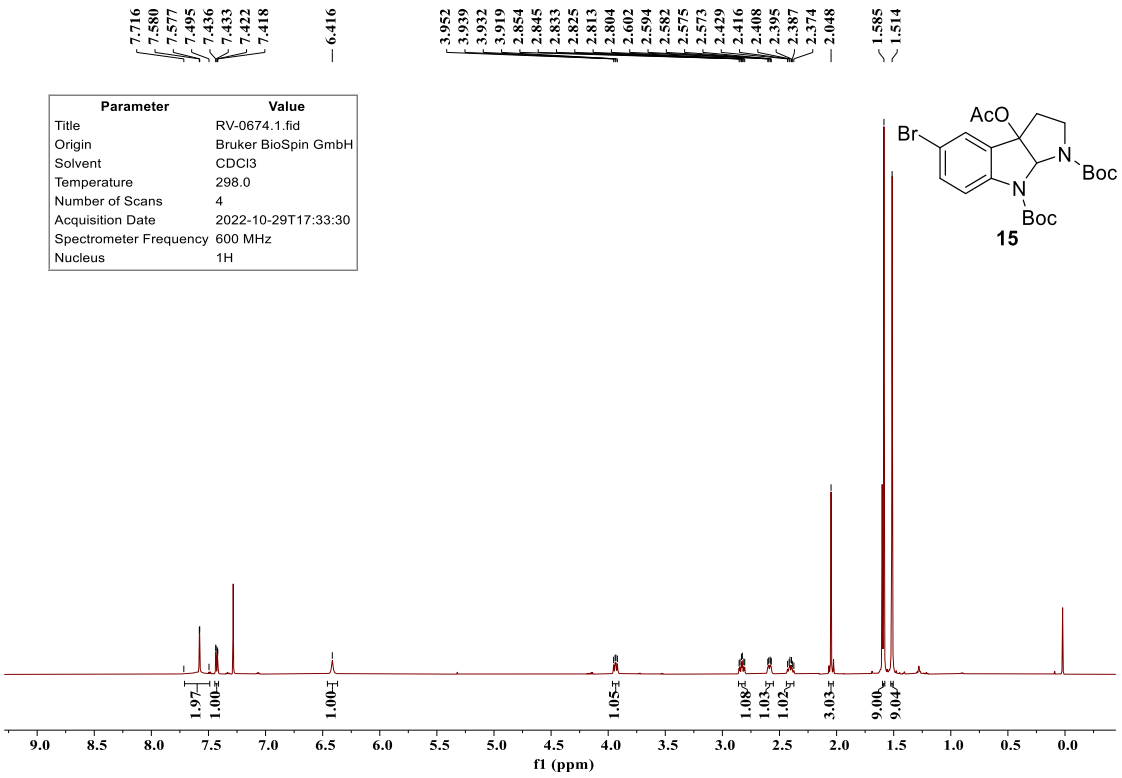


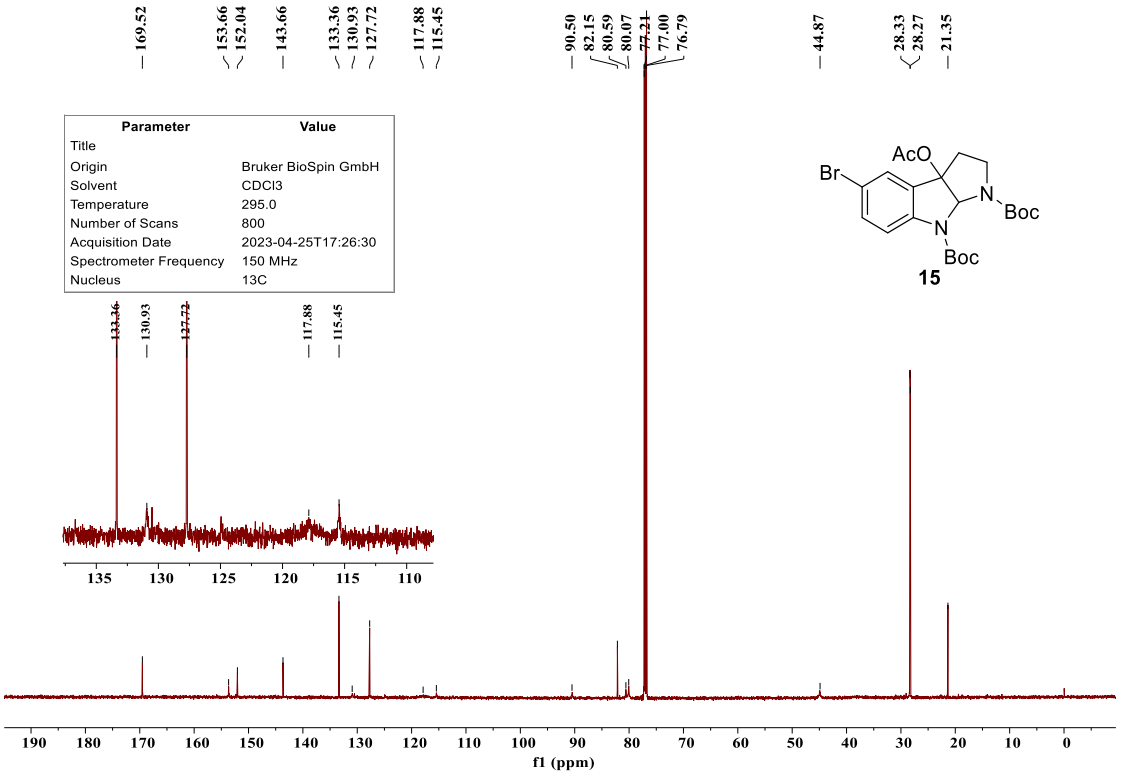

7.499
6.772
6.758
6.407
3.957
3.945
3.933
3.871
3.863
3.855
3.805
3.792
3.786
3.773
3.122
3.102
3.081
3.060
2.865
2.856
2.845
2.836
2.825
2.817
2.104
2.089
2.073
2.007
1.556
1.486
Parameter Value
Title SY-0455B.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-07-17T16:20:00
Spectrometer Frequency 600 MHz
Nucleus 1H
O
N
N
Boc
N
Boc
13
1.03
2.03
1.00
1.02
4.05
1.00
4.03
1.03
2.05
9.03
9.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
(ppm)

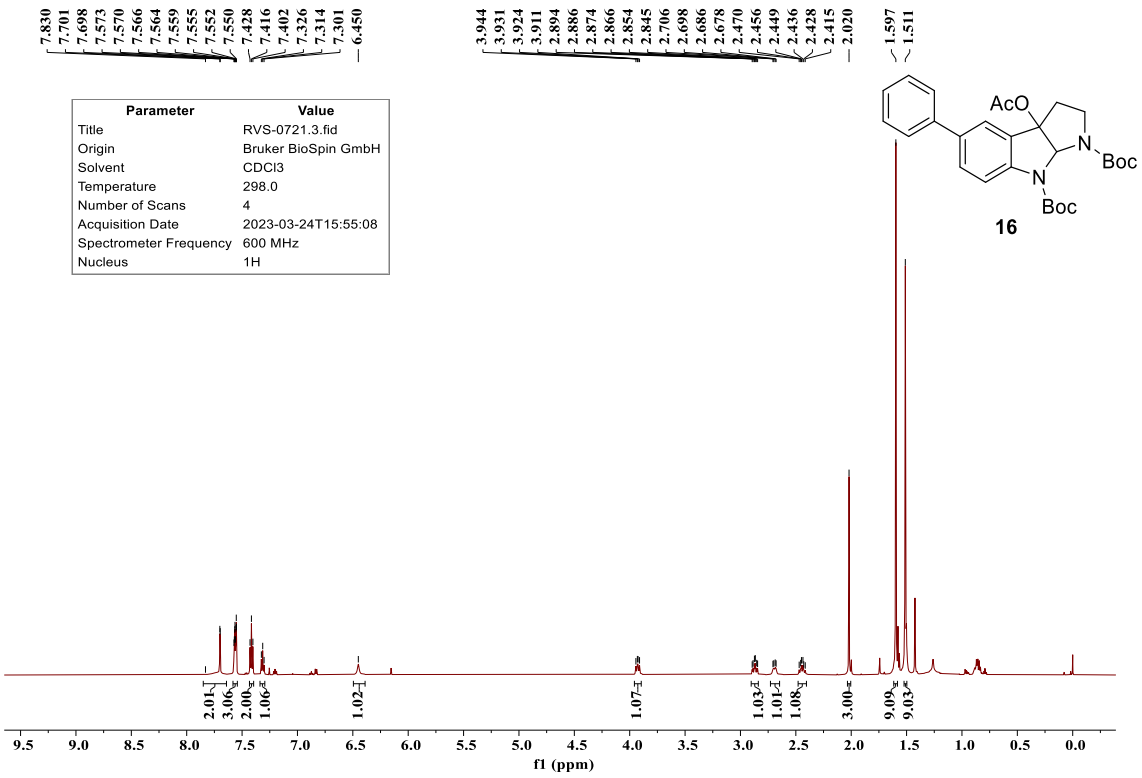

154.03
152.60
147.86
136.73
133.14
128.36
115.79
112.37
81.12
79.98
77.21
77.00
76.79
76.23
66.94
50.50
45.36
44.86
29.68
28.40
Parameter Value
Title SY-0455B.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 2000
Acquisition Date 2023-07-17T18:27:12
Spectrometer Frequency 150 MHz
Nucleus 13C
O
N
N
Boc
N
Boc
13
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
(ppm)

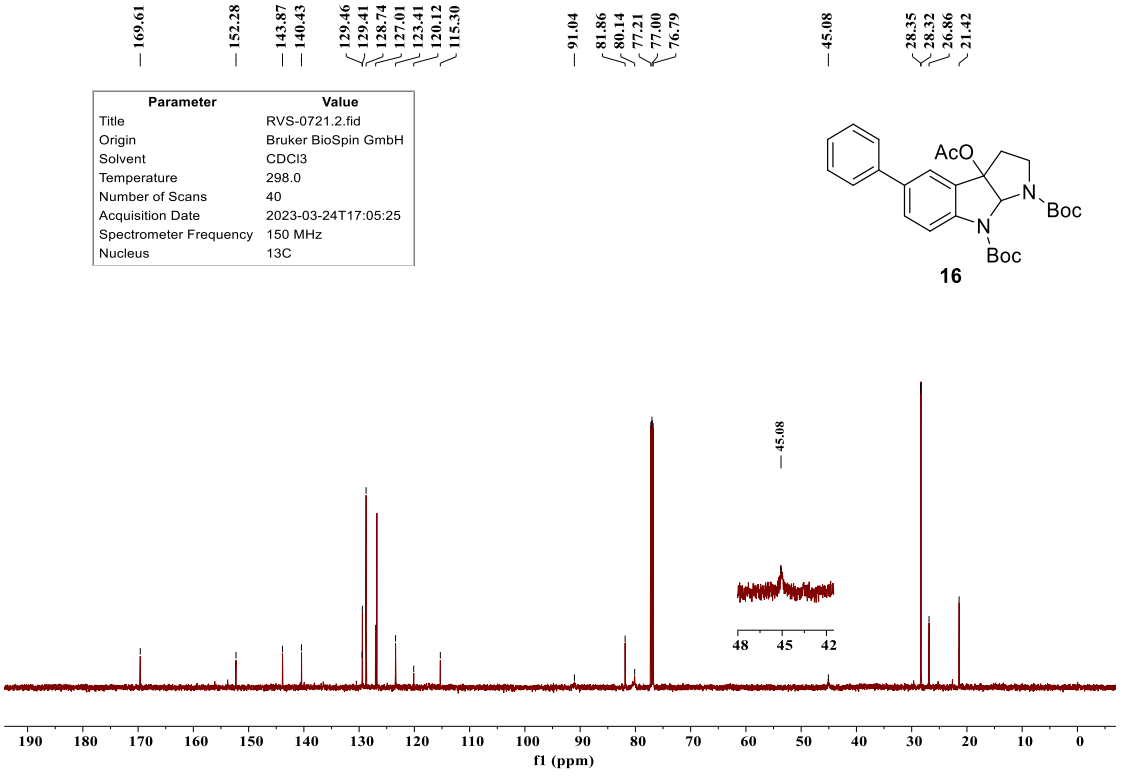

7.652
7.574
7.569
7.557
7.540
7.536
7.526
7.522
7.447
7.434
7.421
7.351
7.339
7.327
6.483
3.781
3.767
3.763
3.750
2.894
2.886
2.875
2.866
2.853
2.837
2.828
2.778
2.770
2.762
2.755
2.742
2.733
2.721
1.604
1.498
Parameter Value
Title RVS-0711.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 4
Acquisition Date 2023-03-10T17:20:05
Spectrometer Frequency 600 MHz
Nucleus 1H
Br
N
Boc
N
Boc
14
0.85
3.00
1.02
2.02
1.00
0.97
1.01
2.01
1.03
9.01
9.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

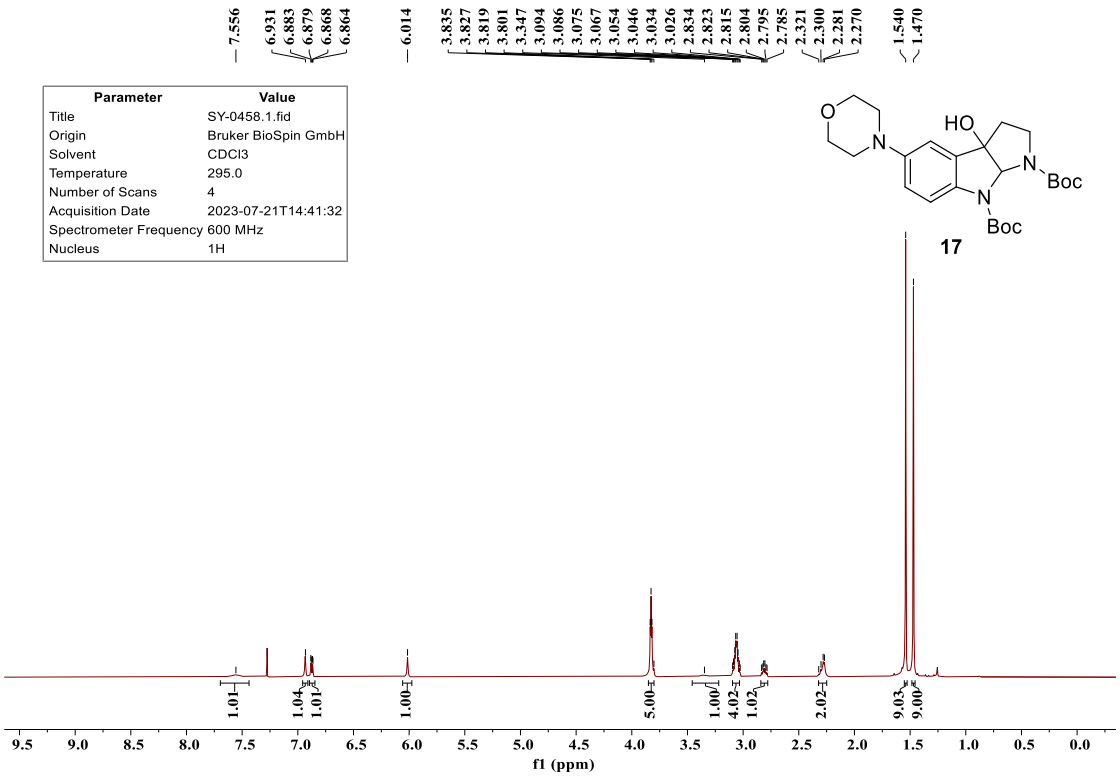

153.41
152.07
141.40
140.26
137.40
133.22
129.31
128.82
127.26
126.83
122.35
117.55
84.20
82.21
80.80
77.21
77.00
76.79
62.21
46.18
42.12
28.37
28.28
Parameter Value
Title RVS-0711.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 512
Acquisition Date 2023-03-10T17:48:14
Spectrometer Frequency 150 MHz
Nucleus 13C
Br
N
Boc
N
Boc
14
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

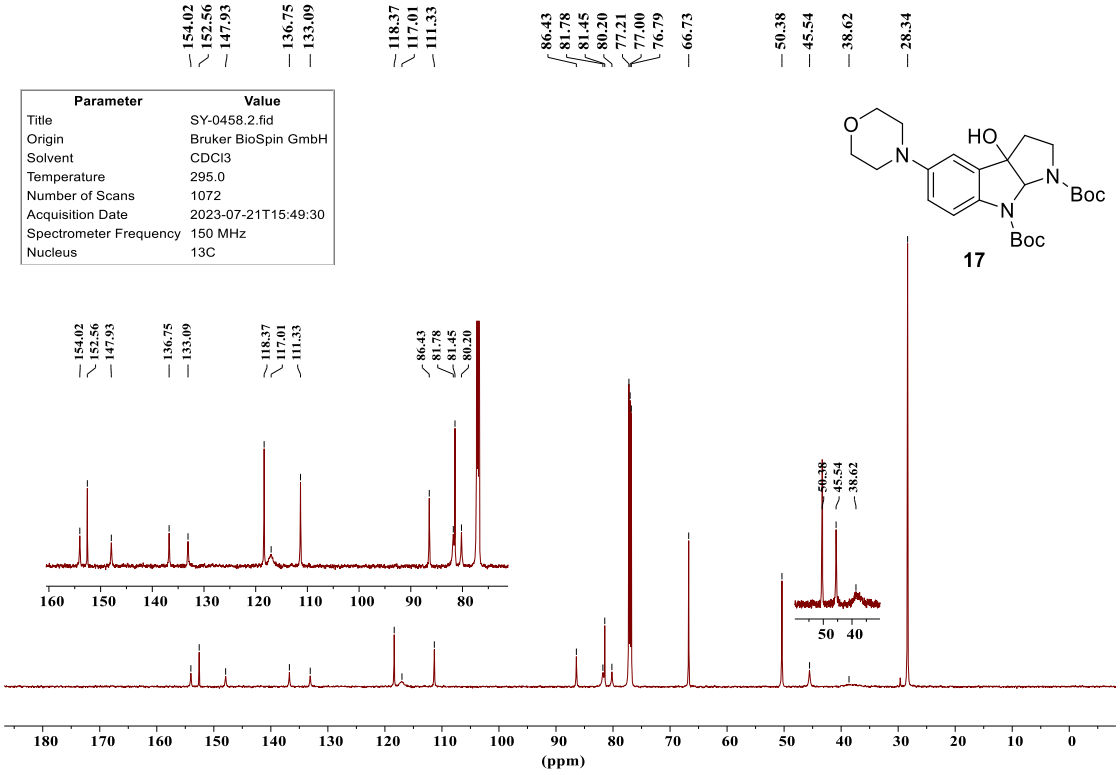

7.716
7.580
7.577
7.495
7.436
7.433
7.422
7.418
6.416
3.952
3.939
3.932
3.919
2.854
2.845
2.833
2.825
2.813
2.804
2.602
2.594
2.582
2.575
2.573
2.429
2.416
2.408
2.395
2.387
2.374
2.048
1.585
1.514
Parameter Value
Title RV-0674.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 4
Acquisition Date 2022-10-29T17:33:30
Spectrometer Frequency 600 MHz
Nucleus 1H
AcO
Br
N
Boc
N
Boc
15
1.97
1.00
1.00
1.05
1.08
1.03
1.02
3.03
9.00
9.04
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

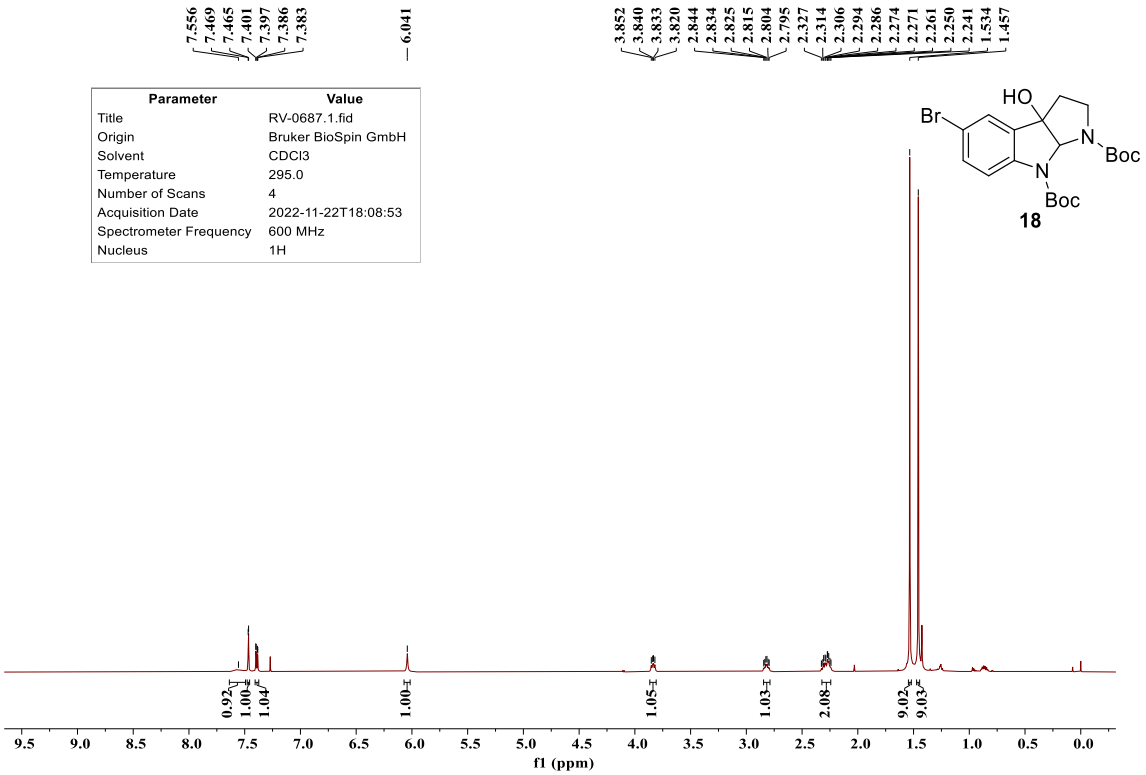

169.52
153.66
152.04
143.66
133.36
130.93
127.72
117.88
115.45
90.50
82.15
80.59
80.07
77.21
77.00
76.79
44.87
28.33
28.27
21.35
Parameter Value
Title
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 800
Acquisition Date 2023-04-25T17:26:30
Spectrometer Frequency 150 MHz
Nucleus 13C
AcO
Br
N
Boc
N
Boc
15
135 130 125 120 115 110
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

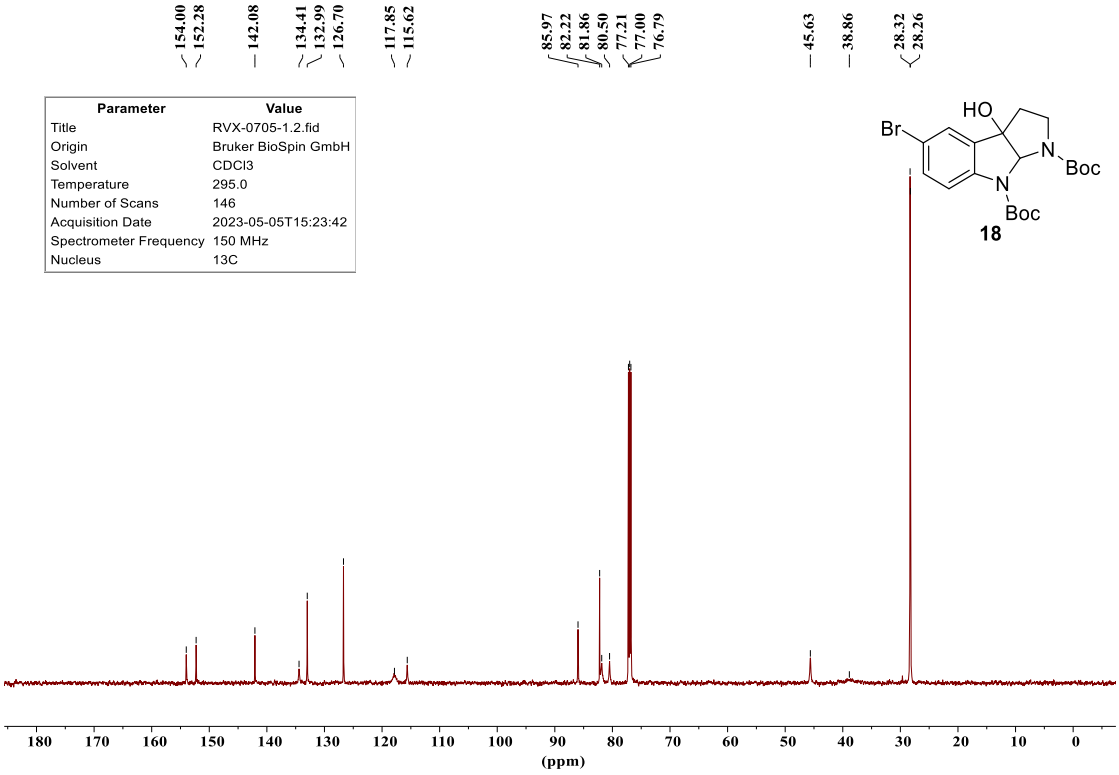

7.830
7.701
7.698
7.573
7.570
7.566
7.564
7.559
7.555
7.552
7.550
7.428
7.416
7.402
7.326
7.314
7.301
6.450
3.944
3.931
3.924
3.911
2.894
2.886
2.874
2.866
2.854
2.845
2.706
2.698
2.686
2.678
2.470
2.456
2.449
2.436
2.428
2.415
2.020
1.597
1.511
Parameter Value
Title RVS-0721.3.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 4
Acquisition Date 2023-03-24T15:55:08
Spectrometer Frequency 600 MHz
Nucleus 1H
AcO
N
Boc
N
Boc
16
2.01
3.06
2.00
1.06
1.02
1.07
1.03
1.01
1.08
3.00
9.09
9.03
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


169.61
152.28
143.87
140.43
129.46
129.41
128.74
127.01
123.41
120.12
115.30
91.04
81.86
80.14
77.21
77.00
76.79
45.08
28.35
28.32
26.86
21.42
Parameter Value
Title RVS-0721.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 298.0
Number of Scans 40
Acquisition Date 2023-03-24T17:05:25
Spectrometer Frequency 150 MHz
Nucleus 13C
AcO
N
Boc
N
Boc
16
45.08
48 45 42
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


7.556
6.931
6.883
6.879
6.868
6.864
6.014
3.835
3.827
3.819
3.801
3.347
3.094
3.086
3.075
3.067
3.054
3.046
3.034
3.026
2.834
2.823
2.815
2.804
2.795
2.785
2.321
2.300
2.281
2.270
1.540
1.470
Parameter Value
Title SY-0458.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2023-07-21T14:41:32
Spectrometer Frequency 600 MHz
Nucleus 1H
HO
N
Boc
Boc
17
1.01
1.04
1.01
1.00
5.00
1.00
4.02
1.02
2.02
9.03
9.00
f1 (ppm)


154.02
152.56
147.93
136.75
133.09
118.37
117.01
111.33
86.43
81.78
81.45
80.20
77.21
77.00
76.79
66.73
50.38
45.54
38.62
28.34
Parameter Value
Title SY-0458.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 1072
Acquisition Date 2023-07-21T15:49:30
Spectrometer Frequency 150 MHz
Nucleus 13C
HO
N
Boc
Boc
17
(ppm)


7.556
7.469
7.465
7.401
7.397
7.386
7.383
6.041
3.852
3.840
3.833
3.820
2.844
2.834
2.825
2.815
2.804
2.795
2.327
2.314
2.306
2.294
2.286
2.274
2.271
2.261
2.250
2.241
1.534
1.457
Parameter Value
Title RV-0687.1.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 4
Acquisition Date 2022-11-22T18:08:53
Spectrometer Frequency 600 MHz
Nucleus 1H
HO
Br
N
Boc
N
Boc
18
0.92
1.00
1.04
1.00
1.05
1.03
2.08
9.02
9.03
f1 (ppm)


154.00
152.28
142.08
134.41
132.99
126.70
117.85
115.62
85.97
82.22
81.86
80.50
77.21
77.00
76.79
45.63
38.86
28.32
28.26
Parameter Value
Title RVX-0705-1.2.fid
Origin Bruker BioSpin GmbH
Solvent CDCl3
Temperature 295.0
Number of Scans 146
Acquisition Date 2023-05-05T15:23:42
Spectrometer Frequency 150 MHz
Nucleus 13C
HO
Br
N
Boc
N
Boc
18
(ppm)