Aluminum-Catalyzed Cross Selective C3–N1' Coupling Reactions of *N*-Methoxyindoles with Indoles

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SUPPORTING INFORMATION

Contents

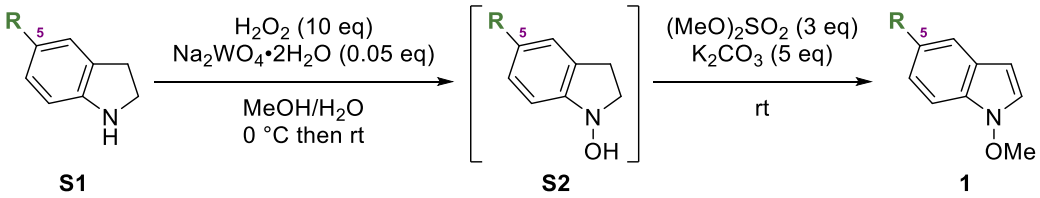
1. General Experimental

High-resolution MS spectra were recorded with a Brucker micrOTOF mass spectrometers (ESI-TOF-MS). The NMR experiments were performed with JEOL JNM-ECZ600R (^{1}H NMR: 600 MHz, ^{13}C NMR: 151 MHz) spectrometer, Varian 600-MR ASW (^{1}H NMR: 600 MHz, ^{13}C NMR: 151 MHz) spectrometer and Varian 400-MR ASW (^{1}H NMR: 400 MHz, ^{13}C NMR: 100 MHz) spectrometer, and chemical shifts are expressed in ppm (δ) using residual undeuterated solvent as an internal reference ($CDCl_3$, ^{1}H NMR: δ 7.25, ^{13}C NMR: δ 77.1). The following abbreviations were used to explain NMR peak multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, sep = septet, m = multiplet, dd = doublet of doublets, ddd = doublet of doublet of doublets, br = broad; coupling constants in Hz; integration. Reactions were monitored by thin layer chromatography (TLC) carried out on a silica gel plates (60F-254) and visualized under UV illumination at 254 or 365 nm depending on the compounds. Flash column chromatography was performed on silica gel (WAKO Gel 75–150 mesh, WAKO Co., Ltd.).

2. Experimental Procedure

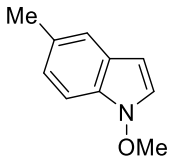
2-1. Preparation of NMeOINs

The *N*-MeOINs **1** were prepared by reported method.[S1] All substrates were used as received from commercial suppliers (Sigma-Aldrich, Kanto Chemical, TCI and Wako) and all reagents were weighed and handled in air at room temperature. Analytical data are in accordance with the literature values.[S2]



A solution with the indoline (2 mmol) and Na_2WO_4•$2H_2O$ (0.1 mmol, 0.05 eq) in MeOH (6 mL) and H_2O (0.6 mL) was cooled to 0 °C. 30% H_2O_2 (2.24 mL, 20 mmol) was added dropwise. The mixture was stirred for 5–10 minutes at room temperature. Then, $(MeO)_2SO_2$ (6 mmol, 3 eq) and K_2CO_3 (10 mmol, 5 eq) was added to the reaction mixture and stirred until the complete disappearance of **S2** indicated by TLC. After H_2O (20 mL) was added to the mixture, the whole was extracted with AcOEt (3 x 20 mL), washed with brine (20 mL). The combined organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/5) to give **1**.

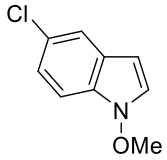
5-Methyl-1-methoxyindole (1b)



1b

167 mg, 52% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.43 (d, J = 6.0 Hz, 1H), 7.40–7.37 (m, 1H), 7.25 (d, J = 3.0 Hz, 1H), 7.14–7.11 (m, 1H), 6.31 (d, J = 3.0 Hz, 1H), 4.09 (s, 3H), 2.49 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 130.4, 129.3, 124.7, 124.1, 123.2, 120.9, 108.1, 97.6, 65.8, 21.5; HRMS (ESI) *m/z*: 162.0920 (Calcd for $C_{10}H_{12}NO$ $[M+H]^+$: 162.0919).

5-Chloro-1-methoxyindole (1c)

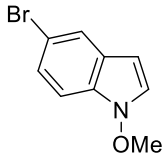


1c

184 mg, 51% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.56 (d, J = 2.0 Hz, 1H), 7.37 (d, J = 8.4 Hz, 1H), 7.29 (d, J = 3.6 Hz, 1H), 7.20 (dd, J = 8.4, 2.0 Hz, 1H), 6.31 (d, J = 3.6 Hz, 1H), 4.08 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 130.2, 125.6, 125.1, 124.2, 122.6, 120.5, 109.3, 97.6, 65.9; HRMS (ESI) *m/z*: 182.0373, 184.0344 (Calcd

for C_9H_9ClNO $[M+H]^+$: 182.0373, 184.0343).

5-Bromo-1-methoxyindole (1d)

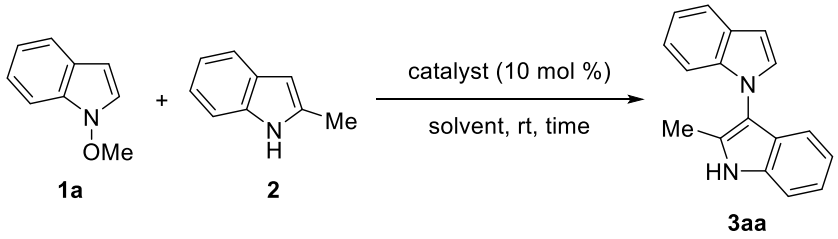


1d

230 mg, 51% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.71 (s, 1H), 7.31–7.31 (m, 2H), 7.25–7.25 (m, 1H), 6.29 (d, J = 3.6 Hz, 1H), 4.07 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ:130.5, 125.9, 125.3, 124.1, 123.7, 113.2, 109.7, 97.6, 66.2; HRMS (ESI) *m/z*: 225.9867, 227.9847 (Calcd for C_9H_9rNO $[M+H^+$: 225.9868, 227.9847).

2-2. Optimization of reaction conditions

■Optimization of reaction conditions (Table 1)

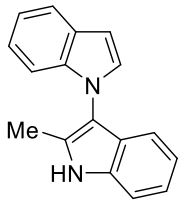


run	catalyst	solvent	time (h)	yield (%) of **3aa**[a]
1	$In(OTf)_3$	MeCN	1.5	72
2	$InF_3•3H_2O$	MeCN	1.5	14
3	$InBr_3$	MeCN	1.5	18
4	$InCl_3•4H_2O$	MeCN	1.5	8
5	$Ga(OTf)_3$	MeCN	1.5	79
6	$La(OTf)_3$	MeCN	1.5	31
7	$Bi(OTf)_3$	MeCN	1.5	60
8	AgOTf	MeCN	1.5	15
9	$Yb(OTf)_3$	MeCN	1.5	0
10	$Cu(OTf)_2$	MeCN	1.5	72
11	$Zn(OTf)_2$	MeCN	1.5	7
12	**$Al(OTf)_3$**	**MeCN**	**1.5**	**83 (87)**[b]
13	$AlCl_3$	MeCN	1.5	23
14	$Al(O\text{-}iPr)_3$	MeCN	1.5	9
15	$Al(OTf)_3$	PhCl	1.5	69
16	$Al(OTf)_3$	1,4-dioxane	1.5	43
17	$Al(OTf)_3$	$CHCl_3$	1.5	71
18	TfOH	MeCN	1.5	54
19	---	MeCN	24	nr

[a] NMR yields. [b] Isolated yields.

To a solution of **1a** (0.1 mmol) and **2a** (0.1 mmol, 1 eq) in solvent (1 mL, 0.1 M) was added catalyst (0.1 mmol, 10 mol %) at room temperature. The mixture was stirred for 1.5 h. After H_2O (20 mL) was added to the mixture, the whole was extracted with $CHCl_3$ (3 x 20 mL), washed with brine (20 mL). The combined organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo*. The yields were determined by ^1H NMR spectrum using 1,3,5-trimethoxybenzene (0.1 mmol, 1 eq) as internal standard material. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/5) to give **3aa**.

2'-Methyl-1,3'-bisindole (3aa)

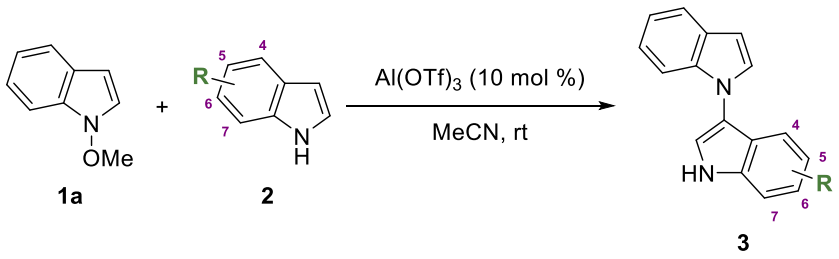


3aa

Run 12: 21.4 mg, 87% yield. colorless solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.91 (br s, 1H), 7.77–7.75 (m, 1H), 7.36 (d, *J* = 8.4 Hz, 1H), 7.28 (d, *J* = 3.6 Hz, 1H), 7.26–7.17 (m, 4H), 7.15–7.13 (m, 1H), 7.09 (ddd, *J* = 8.4, 7.2, 1.2 Hz, 1H), 6.74 (d, *J* = 3.6 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.8, 133.8, 130.7, 130.0, 128.5, 125.5, 122.2, 121.9, 120.9, 120.4, 119.9, 117.7, 113.7, 111.0, 110.9, 102.5, 11.3; HRMS (ESI) *m/z*: 247.1235 (Calcd for $C_{17}H_{15}N_2$ $[M+H]^+$: 247.1235).

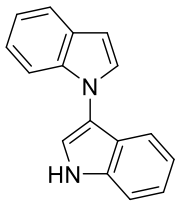
2-3. Substrate scope

■General procedure for the reaction of 1a with 2 (Scheme 2: indoles)



To a solution of **1a** (1 mmol) and **2** (1 mmol, 1 eq) in MeCN (10 mL, 0.1 M) was added $Al(OTf)_3$ (0.1 mmol, 10 mol %) at room temperature. The mixture was stirred until the complete disappearance of starting material indicated by TLC. After H_2O (20 mL) was added to the mixture, the whole was extracted with AcOEt (3 x 20 mL), washed with brine (20 mL). The combined organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/5) to give **3**.

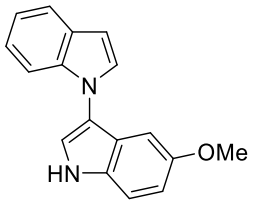
1,3'-bisindole (3ab)



3ab

105 mg, 45% yield. colorless solid; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.17 (br s, 1H), 7.73–7.71 (m, 1H), 7.49–7.45 (m, 2H), 7.37–7.28 (m, 4H), 7.21–7.13 (m, 3H), 6.71–6.70 (m, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.5, 134.7, 129.6, 128.6, 123.7, 123.1, 122.0, 120.9, 120.5, 120.0, 119.2, 118.6, 117.6, 111.7, 110.9, 102.5; HRMS (ESI) *m/z*: 233.1079 (Calcd for $C_{16}H_{13}N_2$ $[M+H]^+$: 233.1079).

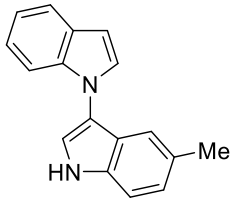
5'-Methoxy-1,3'-bisindole (3ac)



3ac

143 mg, 55% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.04 (br s, 1H), 7.82–7.78 (m, 1H), 7.41–7.37 (m, 2H), 7.32 (d, J = 8.8 Hz, 1H), 7.29 (d, J = 2.4 Hz, 1H), 7.27–7.23 (m, 2H), 6.99 (dd, J = 8.8, 2.4 Hz, 1H), 6.89 (d, J = 2.4 Hz, 1H), 6.78 (dd, J = 3.2, 0.8 Hz, 1H), 3.76 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 154.7, 137.5, 129.8, 129.7, 128.5, 124.2, 122.0, 120.9, 120.2, 120.0, 117.2, 114.0, 112.7, 110.9, 102.4, 99.5, 55.8; HRMS (ESI) *m/z*: 263.1185 (Calcd for $C_{17}H_{15}N_2O$ $[M+H]^+$: 263.1184).

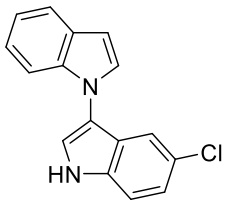
5’-Methyl-1,3’-bisindole (3ad)



3ad

166 mg, 67% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.95–7.93 (m, 1H), 7.76 (br s, 1H), 7.52–7.50 (m, 2H), 7.41–7.36 (m, 3H), 7.33 (d, *J* = 8.4 Hz, 1H), 7.25 (dd, *J* = 8.4, 2.4 Hz, 1H), 7.21 (d, *J* = 2.4 Hz, 1H), 6.91 (dd, *J* = 3.2, 0.8 Hz, 1H), 2.53 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.7, 133.2, 130.1, 130.0, 128.7, 124.9, 124.1, 122.2, 121.1, 120.2, 119.7, 118.0, 116.9, 111.6, 111.1, 102.5, 21.6; HRMS (ESI) *m/z*: 247.1234 (Calcd for $C_{17}H_{15}N_2$ $[M+H]^+$: 247.1235).

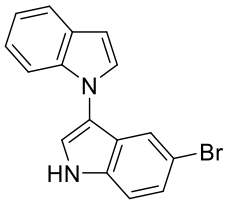
5’-Chloro-1,3’-bisindole (3ae)



3ae

55.0 mg, 21% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 8.16 (br s, 1H), 7.75–7.72 (m, 1H), 7.43 (d, *J* = 1.8 Hz, 1H), 7.36–7.29 (m, 4H), 7.25–7.18 (m, 3H), 6.72 (dd, *J* = 3.0, 0.6 Hz, 1H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.5, 133.2, 129.5, 128.7, 126.5, 124.9, 123.8, 122.3, 121.1, 120.8, 120.2, 119.1, 117.3, 112.9, 110.7, 102.9; HRMS (ESI) *m/z*: 267.0690, 269.0666 (Calcd for $C_{16}H_{12}ClN_2$ $[M+Na]^+$: 267.0689, 269.0660).

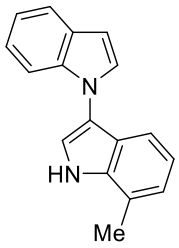
5’-Bromo-1,3’-bisindole (3af)



3af

61.1 mg, 20% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07 (br s, 1H), 7.78–7.75 (m, 1H), 7.60–7.60 (m, 1H), 7.37 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.33–7.30 (m, 2H), 7.28–7.21 (m, 4H), 6.75 (dd, *J* = 3.2, 0.8 Hz, 1H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.4, 133.3, 129.5, 128.6, 126.2, 125.4, 122.2, 121.0, 121.0, 120.6, 120.2, 116.9, 113.9, 113.2, 110.6, 102.9; HRMS (ESI) *m/z*: 311.0183, 313.0164 (Calcd for $C_{16}H_{12}BrN_2$ $[M+Na]^+$: 311.0184, 313.0163).

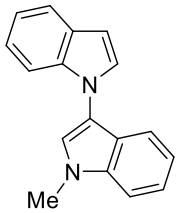
7'-Methyl-1,3'-bisindole (3ag)



3ag

108 mg, 44% yield. colorless solid; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 8.17 (br s, 1H), 7.74 (d, J = 5.2 Hz, 1H), 7.42–7.35 (m, 4H), 7.21–7.17 (m, 2H), 7.14–7.08 (m, 2H), 6.72 (d, J = 3.0 Hz, 1H), 2.59 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.6, 134.4, 129.7, 128.6, 123.7, 123.4, 122.0, 120.9, 120.8, 120.0, 119.0, 118.1, 116.5, 111.0, 102.5, 16.4; HRMS (ESI) *m/z*: 247.1234 (Calcd for $C_{17}H_{15}N_2$ $[M+H]^+$: 247.1235).

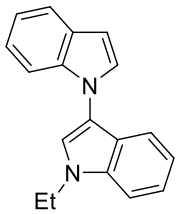
1'-Methyl-1,3'-bisindole (3ah)



3ah

157 mg, 64% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.73–7.71 (m, 1H), 7.49–7.45 (m, 2H), 7.37–7.29 (m, 4H), 7.21–7.13 (m, 3H), 6.71–6.70 (m, 1H), 3.89 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.5, 135.6, 129.7, 128.5, 124.1, 123.7, 122.7, 121.9, 120.8, 120.0, 119.9, 118.7, 116.0, 110.8, 109.7, 102.3, 33.0; HRMS (ESI) *m/z*: 247.1235 (Calcd for $C_{17}H_{15}N_2$ $[M+H]^+$: 247.1235).

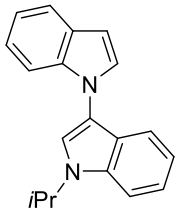
1'-Ethyl-1,3'-bisindole (3ai)



3ai

146 mg, 56% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.75–7.72 (m, 1H), 7.50–7.46 (m, 2H), 7.39–7.36 (m, 2H), 7.35–7.31 (m, 2H), 7.22–7.13 (m, 3H), 6.71 (dd, J = 3.2, 0.8 Hz, 1H), 4.27 (q, J = 7.2 Hz, 2H), 1.57 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 137.5, 134.6, 129.7, 128.5, 124.2, 122.5, 121.9, 121.8, 120.8, 119.9, 119.9, 118.8, 116.1, 110.8, 109.7, 102.3, 41.2, 15.5; HRMS (ESI) *m/z*: 261.1392 (Calcd for $C_{18}H_{17}N_2$ $[M+H]^+$: 261.1392).

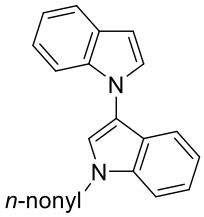
1'-Isopropyl-1,3'-bisindole (3aj)



3aj

227 mg, 83% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.73–7.71 (m, 1H), 7.50–7.47 (m, 2H), 7.42 (s, 1H), 7.37 (d, *J* = 3.0 Hz, 1H), 7.35 (d, *J* = 7.2 Hz, 1H), 7.31 (t, *J* = 7.2 Hz, 1H), 7.20–7.13 (m, 3H), 6.70 (d, *J* = 3.0 Hz, 1H), 4.80 (sep, *J* = 6.0 Hz, 1H), 1.61 (d, *J* = 6.0 Hz, 6H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.8, 134.6, 129.9, 128.8, 124.4, 122.5, 122.0, 121.0, 120.1, 120.0, 119.0, 116.5, 111.0, 110.1, 102.5, 47.5, 23.0; HRMS (ESI) *m/z*: 275.1548 (Calcd for $C_{19}H_{19}N_2$ $[M+H]^+$: 275.1548).

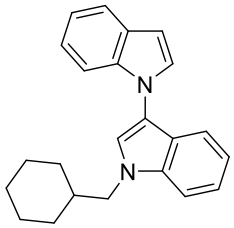
1'-*n*-Nonyl-1,3'-bisindole (3ak)



3ak

243 mg, 68% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.74 (d, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.8 Hz, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.38–7.31 (m, 4H), 7.22–7.19 (m, 2H), 7.15 (t, *J* = 7.8 Hz, 1H), 6.72 (d, *J* = 3.0 Hz, 1H), 4.21 (t, *J* = 7.2 Hz, 2H), 1.96–1.91 (m, 2H), 1.42–1.30 (m, 12H), 0.92–0.90 (m, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.6, 135.0, 129.8, 128.6, 124.3, 122.8, 122.6, 121.9, 120.9, 120.0, 118.9, 116.0, 110.9, 110.0, 102.4, 46.7, 31.9, 30.3, 29.6, 29.3, 27.1, 22.8, 14.2; HRMS (ESI) *m/z*: 359.2486 (Calcd for $C_{25}H_{31}N_2$ $[M+H]^+$: 359.2487).

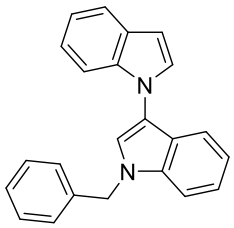
1'-(Cyclohexylmethyl)-1,3'-bisindole (3al)



3al

225 mg, 69% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.72–7.71 (m, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.45 (d, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 3.6 Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.31 (d, *J* = 7.8 Hz, 1H), 7.30 (s, 1H), 7.20–7.12 (m, 3H), 6.70 (d, *J* = 3.6 Hz, 1H), 4.20 (t, *J* = 7.2 Hz, 2H), 1.95–1.90 (m, 2H), 1.42–1.27 (m, 6H), 0.90–0.88 (m, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.7, 135.3, 129.8, 128.7, 124.2, 123.6, 122.5, 122.0, 120.9, 120.0, 119.9, 118.9, 115.9, 111.0, 110.3, 102.4, 53.3, 38.9, 31.2, 26.4, 25.9; HRMS (ESI) *m/z*: 329.2017 (Calcd for $C_{23}H_{25}N_2$ $[M+H]^+$: 329.2018).

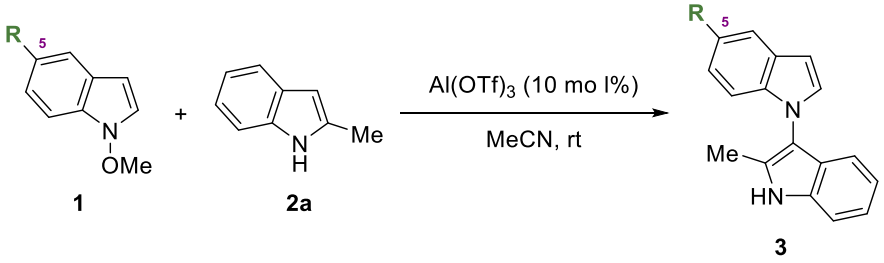
1’-Benzyl-1,3’-bisindole (3am)



3am

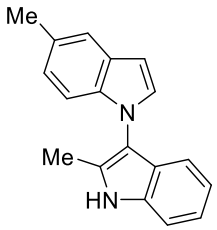
173 mg, 54% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.74–7.72 (m, 1H), 7.52 (d, *J* = 8.4 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 7.39–7.28 (m, 7H), 7.23 (d, *J* = 7.2 Hz, 2H), 7.21–7.15 (m, 3H), 6.71 (d, *J* = 3.0 Hz, 1H), 5.40 (s, 2H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.6, 137.0, 135.4, 129.7, 129.1, 128.7, 128.1, 127.1, 124.4, 123.1, 123.0, 122.0, 120.9, 120.3, 120.0, 119.0, 116.8, 110.9, 110.3, 102.5, 50.4; HRMS (ESI) *m/z*: 323.1546 (Calcd for $C_{23}H_{19}N_2$ $[M+H]^+$: 323.1548).

■General procedure for the reaction of 1 with 2a (Scheme 2: NMeOINs)



To a solution of **1** (0.3 mmol) and **2a** (0.3 mmol, 1 eq) in MeCN (3 mL, 0.1 M) was added $Al(OTf)_3$ (0.03 mmol, 10 mol %) at room temperature. The mixture was stirred until the complete disappearance of starting material indicated by TLC. After H_2O (20 mL) was added to the mixture, the whole was extracted with AcOEt (3 x 20 mL), washed with brine (20 mL). The combined organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/5) to give **3**.

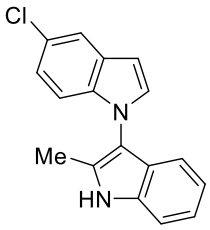
2',5-Dimethyl-1,3'-bisindole (3ba)



3ba

33.6 mg, 43% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.90 (br s, 1H), 7.56–7.56 (m, 1H)), 7.36 (dd, *J* = 8.4, 0.8 Hz, 1H), 7.28–7.21 (m, 2H), 7.12–7.05 (m, 3H), 6.67 (d, *J* = 2.8 Hz, 1H), 2.53 (s, 3H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 136.3, 133.8, 130.6, 130.1, 129.1, 128.7, 125.6, 123.5, 122.1, 120.6, 120.4, 117.7, 113.8, 110.9, 110.7, 102.0, 21.6, 11.2; HRMS (ESI) *m/z*: 261.1392 (Calcd for $C_{18}H_{17}N_2$ $[M+H]^+$: 261.1392).

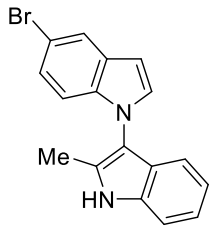
5-Chloro-2'-methyl-1,3'-bisindole (3ca)



3ca

72.9 mg, 87% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.95 (br s, 1H), 7.72 (d, *J* = 1.8 Hz, 1H), 7.36 (d, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 3.0 Hz, 1H), 7.25–7.22 (m, 2H), 7.14–7.10 (m, 2H), 7.04 (d, *J* = 9.0 Hz, 1H), 6.67 (dd, *J* = 3.6, 0.6 Hz, 1H), 2.28 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 136.3, 133.8, 131.3, 130.8, 129.5, 125.6, 125.4, 122.4, 122.3, 120.6, 120.3, 117.5, 113.3, 112.0, 111.0, 102.2, 11.2; HRMS (ESI) *m/z*: 281.0845, 283.0815 (Calcd for $C_{17}H_{14}ClN_2$ $[M+H]^+$: 281.0846, 283.0816).

5-Bromo-2'-methyl-1,3'-bisindole (3da)

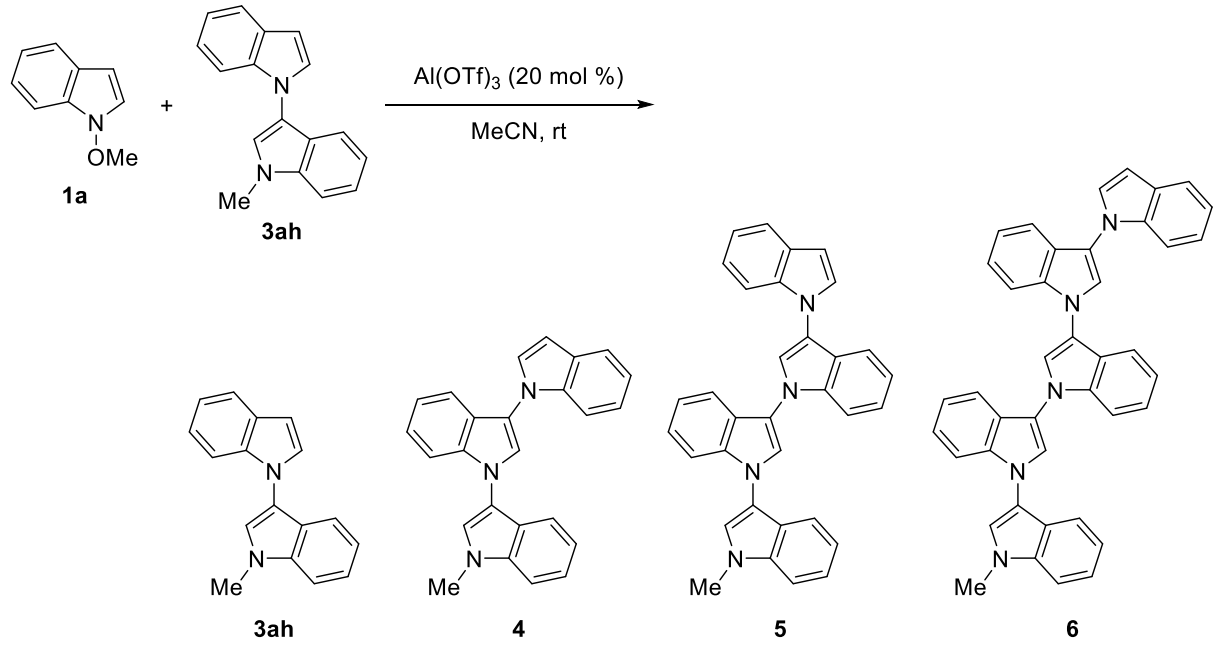


3da

86.9 mg, 89% yield. colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.98 (br s, 1H), 7.89 (d, *J* = 2.0 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.28–7.22 (m, 4H), 7.11 (ddd, *J* = 8.0, 6.8, 1.2 Hz, 1H), 7.01 (d, *J* = 8.8 Hz, 1H), 6.68 (d, *J* = 3.2 Hz, 1H), 2.29 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 136.5, 133.8, 131.2, 130.7, 130.1, 125.3, 124.8, 123.3, 122.3, 120.6, 117.4, 113.2, 112.5, 111.0, 102.1, 11.2; HRMS (ESI) *m/z*: 325.0342, 327.0320 (Calcd for $C_{17}H_{14}BrN_2$ $[M+H]^+$: 325.0340, 327.0320).

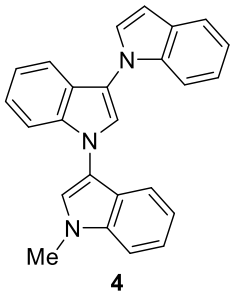
2-4. Oligomerization

■Oligomerization (Scheme 3)



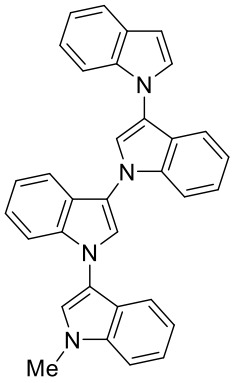
To a solution of **1a** (53.0 mg, 0.36 mmol) and **3ah** (73.9 mg, 0.3 mmol) in MeCN (3 mL, 0.1 M) was added $Al(OTf)_3$ (28.5 mg, 0.06 mmol) under reflux. The mixture was stirred until the complete disappearance of starting material indicated by TLC. After H_2O (10 mL) was added to the mixture, the whole was extracted with AcOEt (3 x 10 mL), washed with brine (10 mL). The combined organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/5) and PTLC (acetone/hexane = 1/5) to give **3ah** (24.4 mg, 33% yield), **4** (16.3 mg, 15% yield), **5** (7.2 mg, 5% yield) and **6** (1.0 mg, 1% yield).

1”-Methyl-1,3’:1’,3”-terindole (4)



16.3 mg, 15% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.73 (dd, *J* = 7.8, 1.2 Hz, 1H), 7.57–7.53 (m, 3H), 7.48–7.42 (m, 4H), 7.37–7.34 (m, 2H), 7.26 (ddd, *J* = 7.8, 6.6, 1.2 Hz, 1H), 7.22–7.16 (m, 4H), 6.73 (dd, *J* = 3.0, 1.2 Hz, 1H), 3.92 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ:137.5, 136.6, 135.7, 129.7, 128.7, 124.5, 124.2, 124.1, 124.1, 123.1, 123.0, 122.1, 120.9, 120.6, 120.4, 120.0, 118.8, 118.6, 117.5, 115.3, 111.5, 111.0, 109.9, 102.6, 33.3; HRMS (ESI) *m/z*: 362.1658 (Calcd for $C_{25}H_{20}N_3$ $[M+H]^+$: 362.1657).

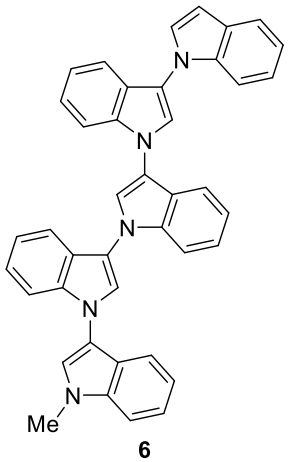
1'''-Methyl-1,3':1',3'':1'',3'''-quaterindole (5)



5

7.2 mg, 5% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.73 (d, *J* = 7.8 Hz, 1H), 7.66 (s, 1H), 7.63 (s, 1H), 7.62 (d, *J* = 7.8 Hz, 1H), 7.58–7.53 (m, 3H), 7.50–7.44 (m, 4H), 7.37–7.34 (m, 2H), 7.31–7.27 (m, 2H), 7.24–7.16 (m, 5H), 6.73 (dd, *J* = 3.0, 0.6 Hz, 1H), 3.93 (s, 3H); ^{13}C NMR (151 MHz, $CDCl_3$) δ: 137.5, 136.6, 136.5, 135.7, 129.7, 128.7, 124.7, 124.3, 124.1, 124.1, 123.4, 123.2, 123.1, 122.1, 120.9, 120.7, 120.4, 120.1, 118.8, 118.6, 117.6, 116.7, 115.2, 111.6, 111.5, 111.0, 110.0, 102.6, 33.3; HRMS (ESI) *m/z*: 477.2075 (Calcd for $C_{33}H_{25}N_4$ $[M+H]^+$: 477.2079).

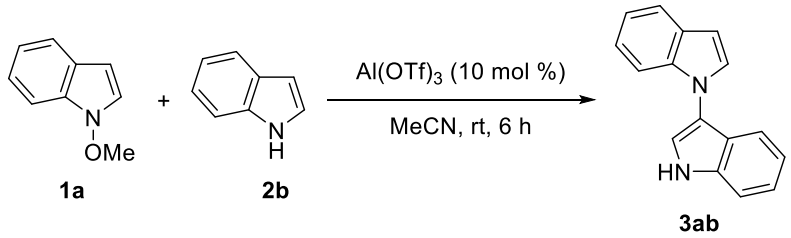
1''''-Methyl-1,3':1',3'':1'',3''':1''':3''''-quinquindole (6)



6

1.0 mg, 1% yield. colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ: 7.76 (s, 1H), 7.74 (d, *J* = 7.8 Hz, 1H), 7.69 (s, 1H), 7.66–7.64 (m, 3H), 7.60–7.59 (m, 3H), 7.56–7.51 (m, 2H), 7.49–7.46 (m, 3H), 7.39–7.29 (m, 6H), 7.24–7.17 (m, 5H), 6.74 (d, *J* = 3.0 Hz, 1H), 3.95 (s, 3H); HRMS (ESI) *m/z*: 592.2505 (Calcd for $C_{41}H_{30}N_5$ $[M+H]^+$: 592.2501).

■Gram-scale synthesis

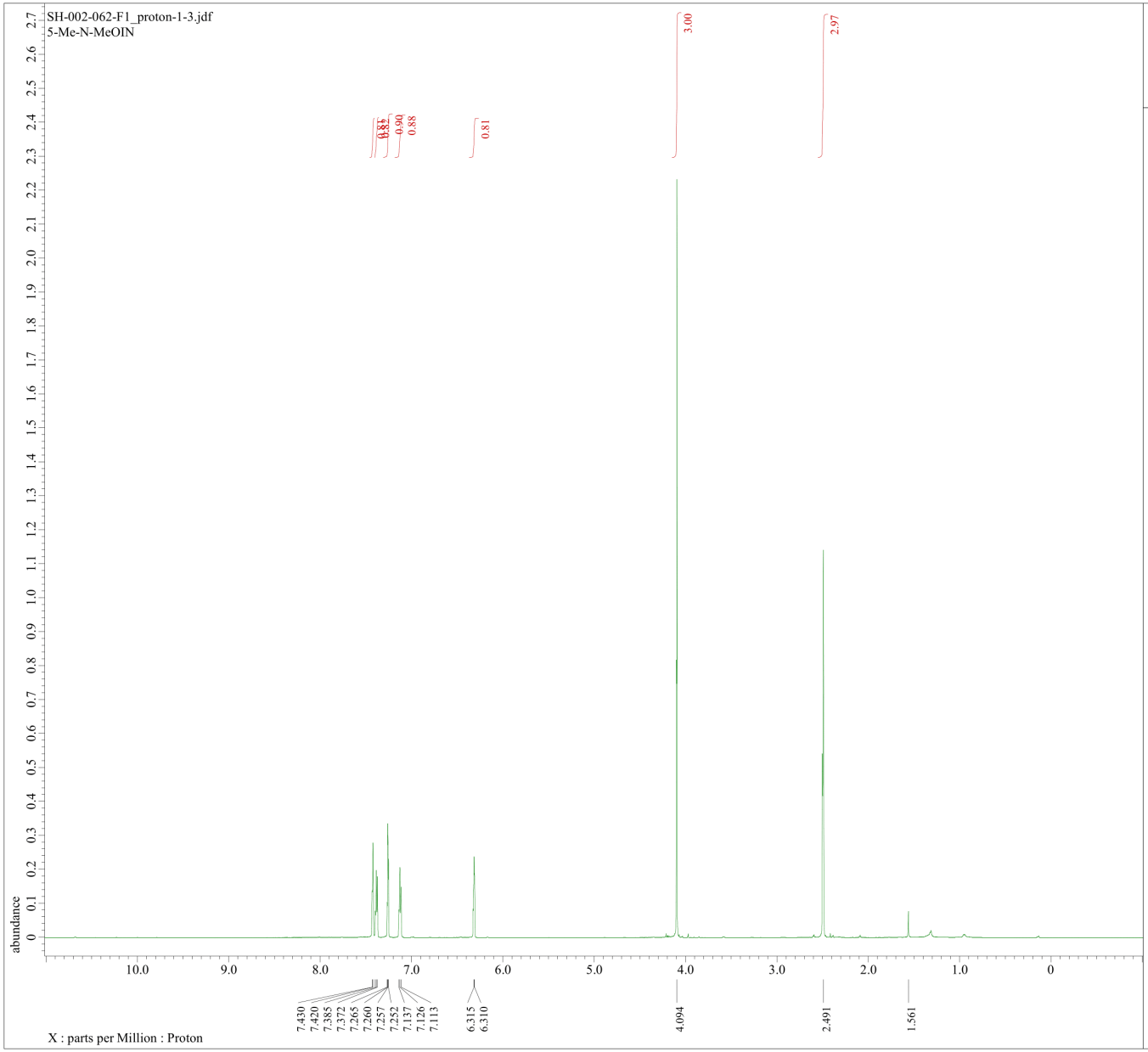


To a solution of **1a** (1.47 g, 10 mmol) and **2b** (1.17 g, 10 mmol) in MeCN (100 mL, 0.1 M) was added $Al(OTf)_3$ (474 mg, 1.0 mmol) at room temperature. The mixture was stirred for 6 h. After H_2O (200 mL) was added to the mixture, the whole was extracted with $CHCl_3$ (3 x 200 mL), washed with brine (200 mL). The combined organic layer was dried over Na_2SO_4, filtered and concentrated *in vacuo*. The residue was purified by silica gel column chromatography (AcOEt/hexane = 1/20–1/5) to give **3aa** (884 mg, 38%).

4. Supplementary References

[S1] Synthesis of 1a: Kawasaki, T.; Kodama, A.; Nishida, T.; Shimizu, K.; Somei, M. *Heterocycles* **1991**, *32*, 221–227.

[S2] 1a: Vo, Q. V.; Trenerry, C.; Rochfort, S.; Wadespm, J.; Leyton, C.; Hughes, A. B. *Bioorg. Med. Chem.* **2014**, *22*, 856–864.



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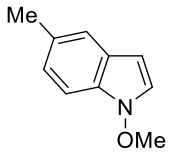
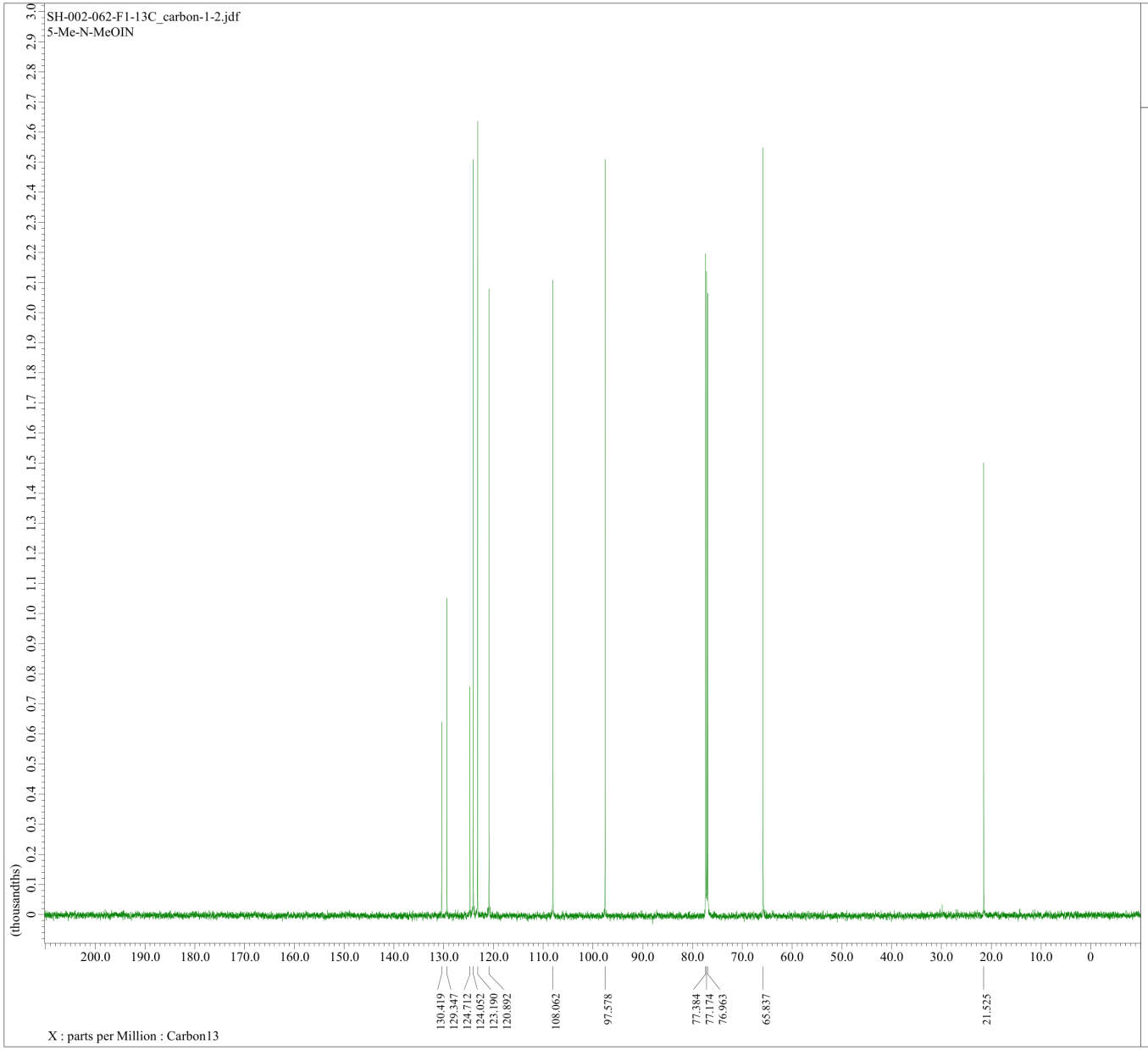


1b



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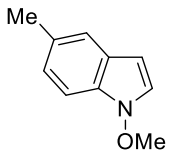

1b

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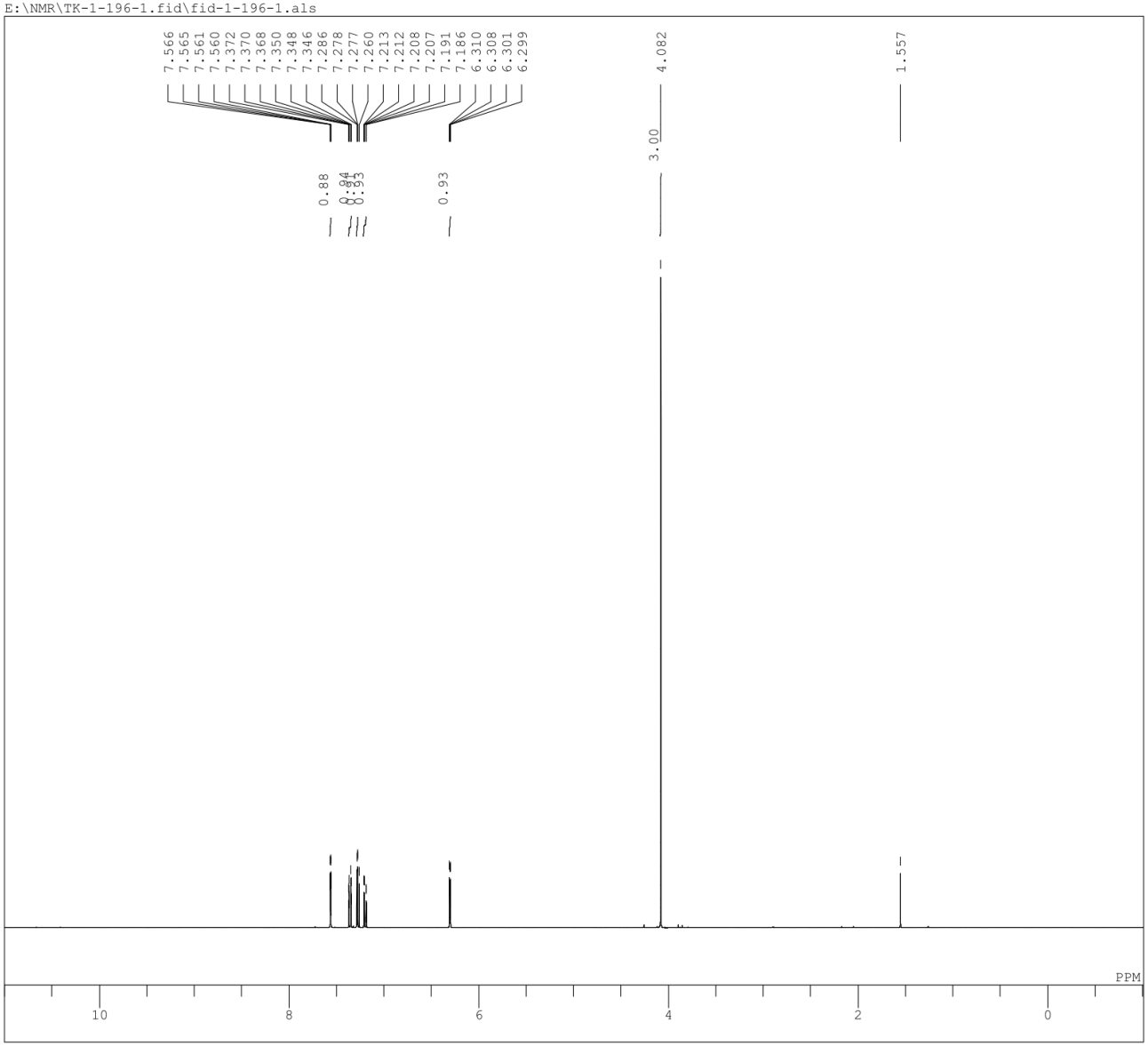


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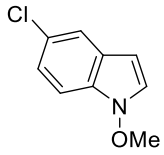

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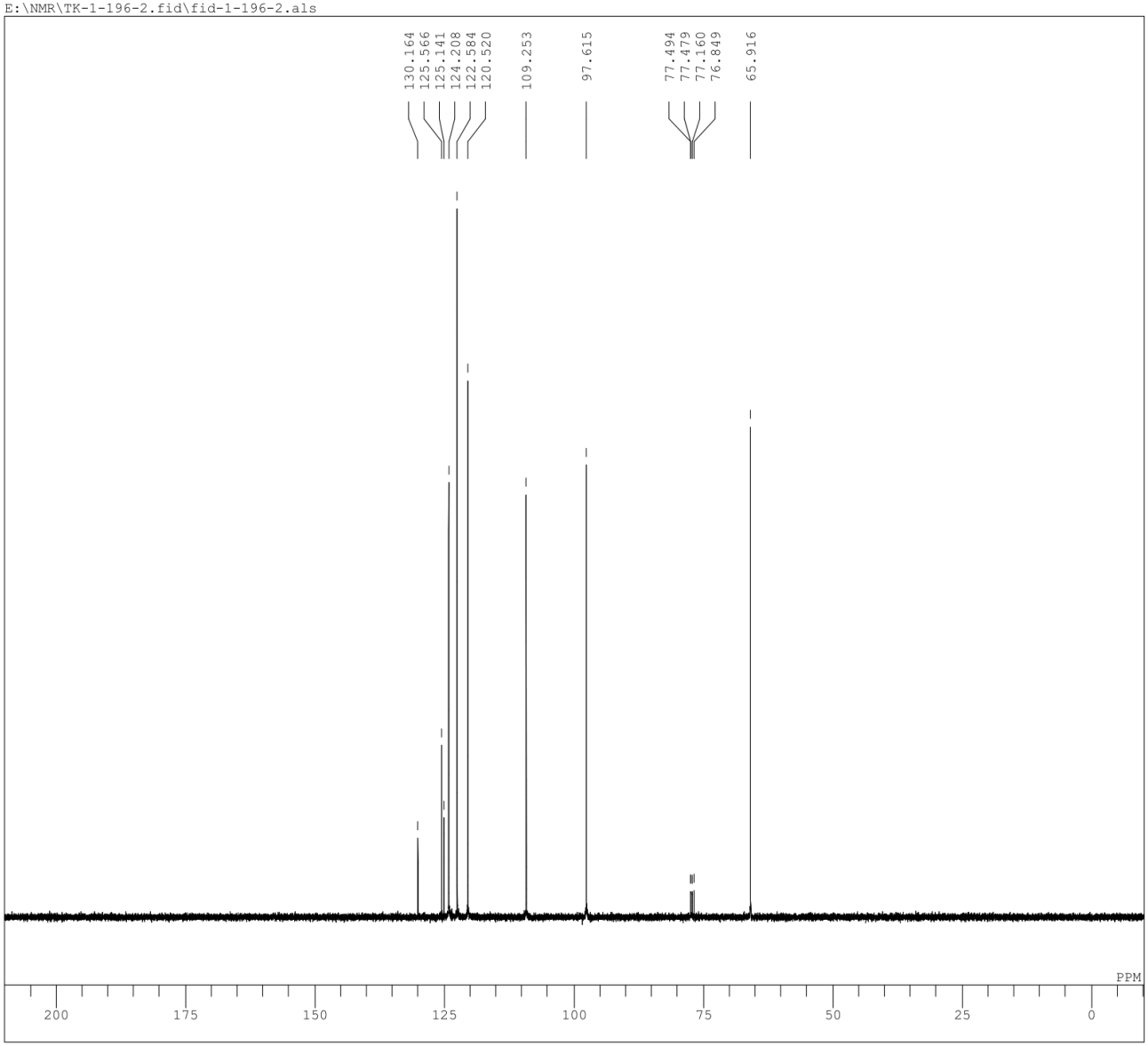


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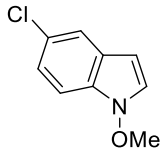

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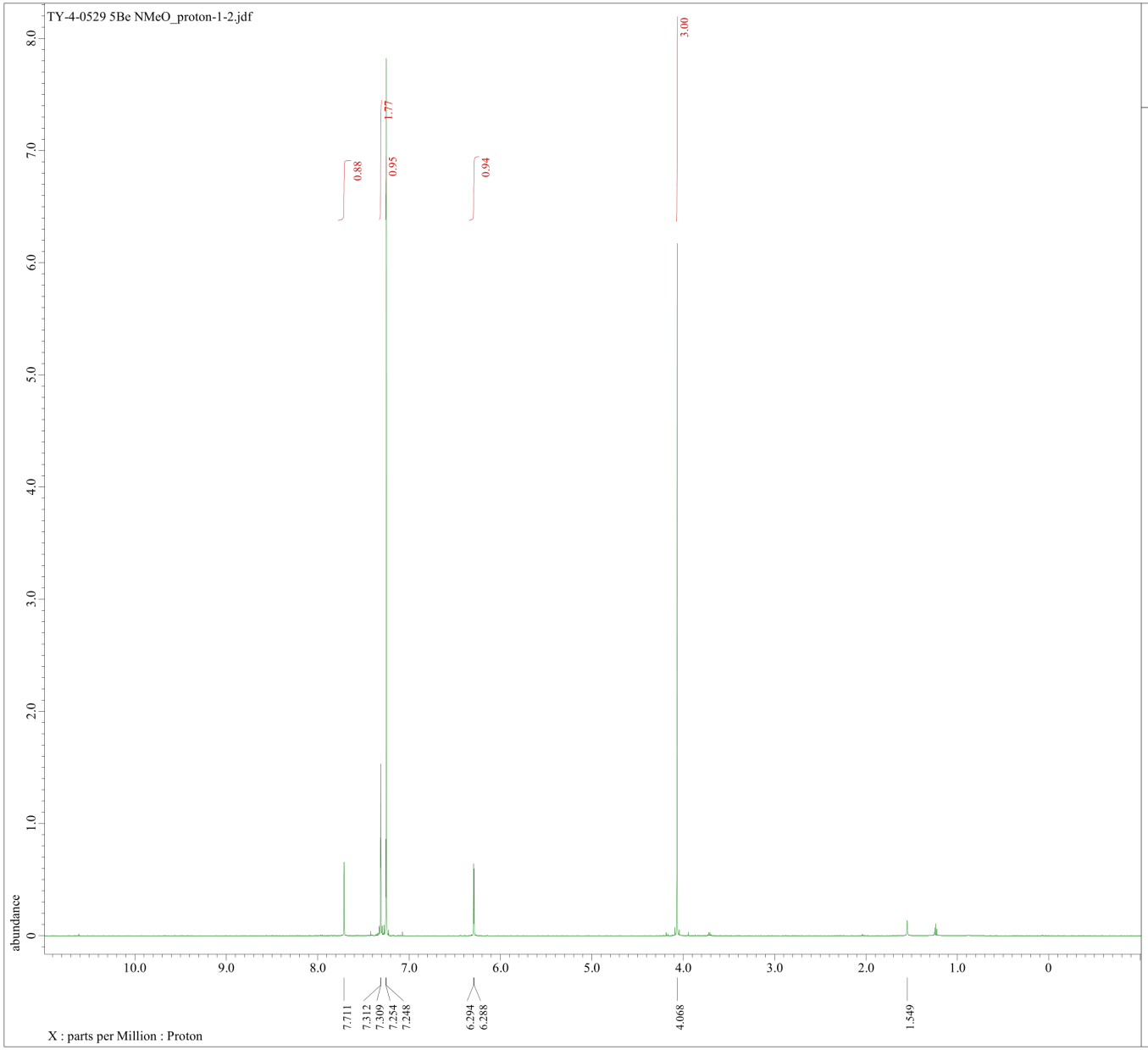

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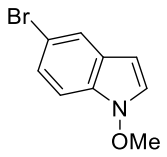
Sample_Id             = TY-4-0529 5Be NMeO
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 29-MAY-2022 13:49:26
Revision_Time         = 29-MAY-2022 13:52:31

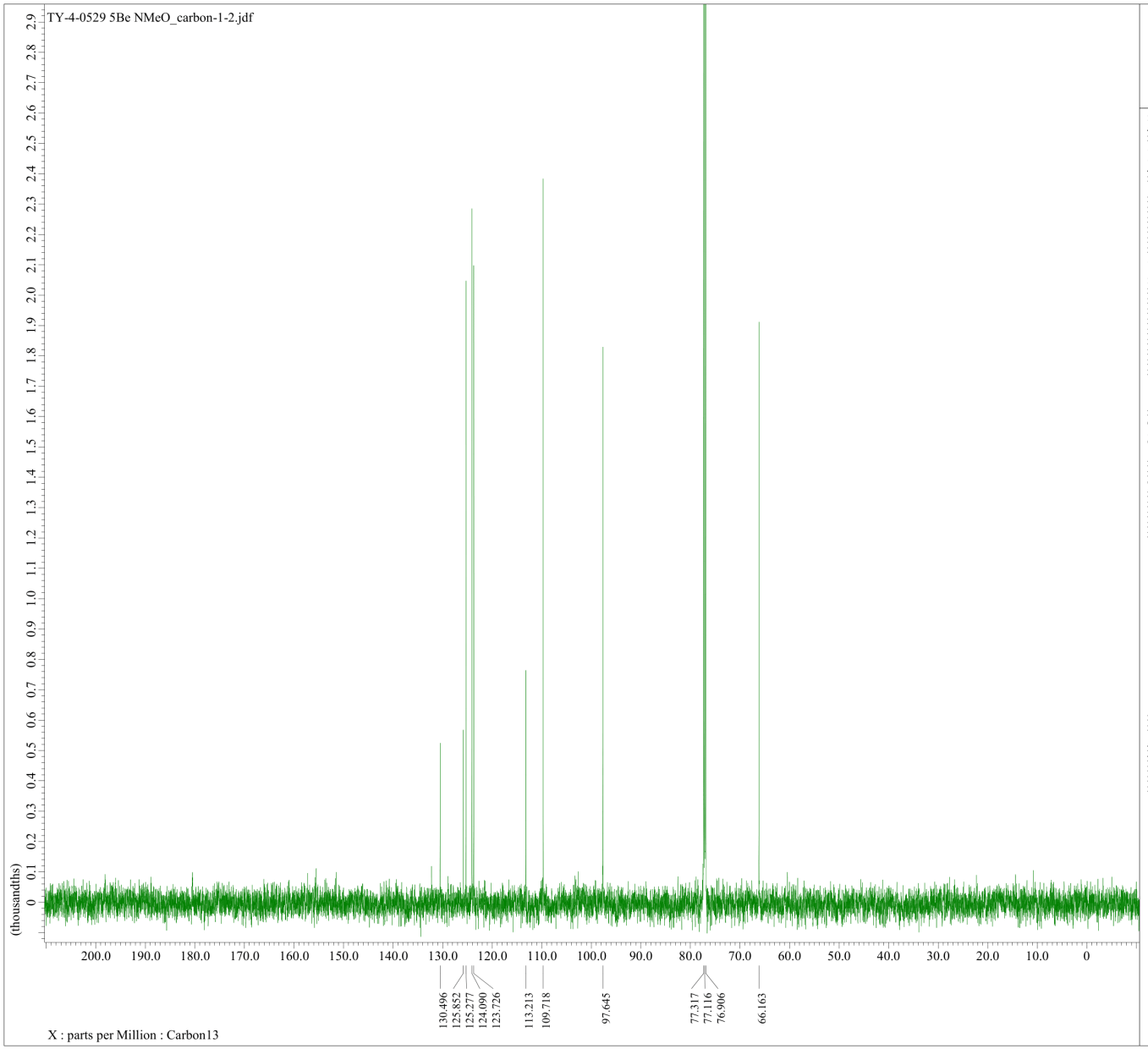
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 4
Total_Scans           = 4

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 20.9[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = (0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```



1d



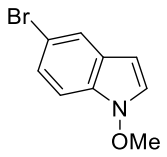
JEOL

```
Filename                = TY-4-0529 5Be NMeO
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = TY-4-0529 5Be NMeO
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 29-MAY-2022 13:51:
Revision_Time           = 29-MAY-2022 13:55:

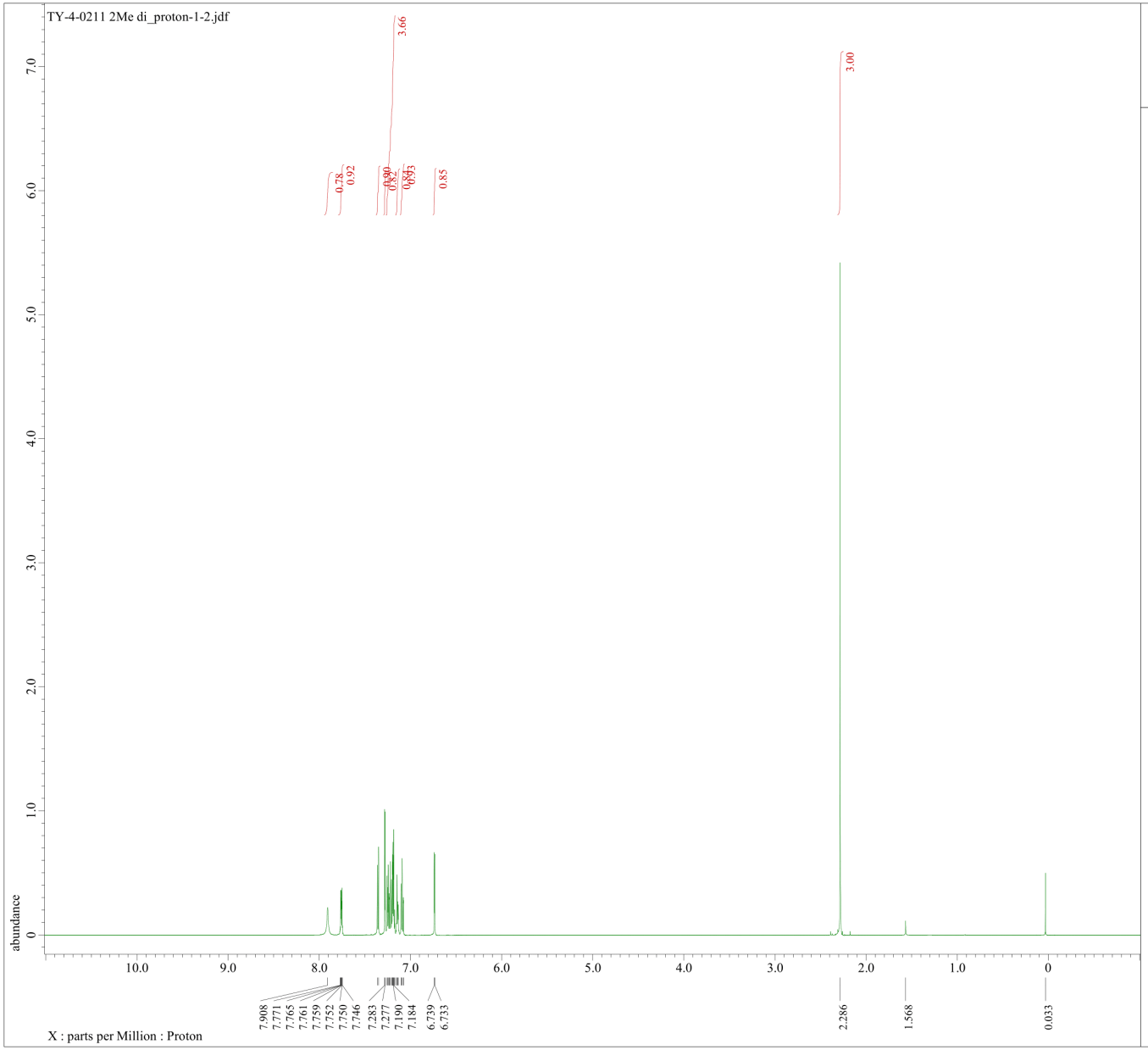
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Scans                   = 176
Total_Scans             = 176

Relaxation_Delay        = 1[s]
Recvr_Gain              = 36
Temp_Get                = 20.9[dC]
X_90_Width              = 11.8[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 9.6[dB]
X_Pulse                 = 3.93333333[us]
Irr_Atn_Dec             = 27.986[dB]
Irr_Atn_Dec_Calc        = 27.986[dB]
Irr_Atn_Dec_Default_Calc = 27.986[dB]
Irr_Atn_Noe             = 27.986[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Templ        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 1[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 1.69206016[s]
```



1d



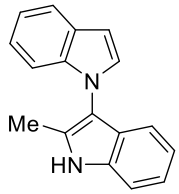
JEOL

```
Filename                = TY-4-0211 2Me di_prot
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = TY-4-0211 2Me di
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-FEB-2023 11:40:52
Revision_Time           = 11-FEB-2023 14:20:08

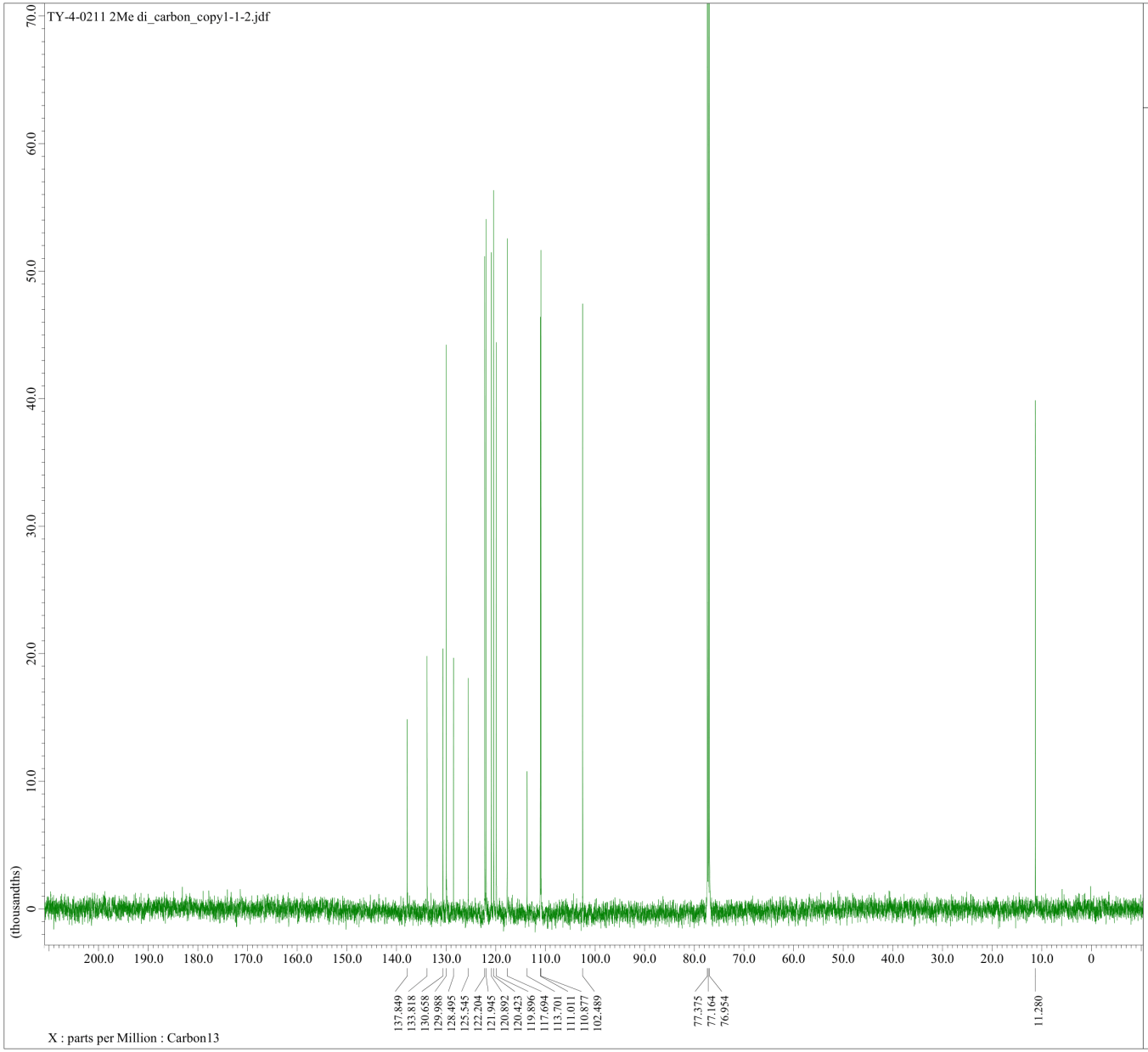
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (600[M
X_Acq_Duration          = 2.18103808[s]
X_Domain                = Proton
X_Freq                  = 600.1723046[MHz]
X_Offset                = 5[ppm]
X_Points                = 32768
X_Prescans              = 1
X_Resolution            = 0.45849727[Hz]
X_Sweep                 = 15.02403846[kHz]
X_Sweep_Clipped         = 12.01923077[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 600.1723046[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 5[us]
Clipped                 = FALSE
Scans                   = 4
Total_Scans             = 4

Relaxation_Delay        = 1[s]
Recvr_Gain              = 36
Temp_Get                = 20.4[dC]
X_90_Width              = 9.9[us]
X_Acq_Time              = 2.18103808[s]
X_Angle                 = 45[deg]
X_Atn                   = 8.1[dB]
X_Pulse                 = 4.95[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 100
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = (0, 90, 270, 180, 180
Presat_Time             = 1[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 3.18103808[s]
```



3aa



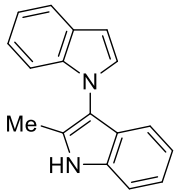
JEOL

```
Filename                = TY-4-0211 2Me di_c
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = TY-4-0211 2Me di
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-FEB-2023 11:42:
Revision_Time           = 11-FEB-2023 14:17:

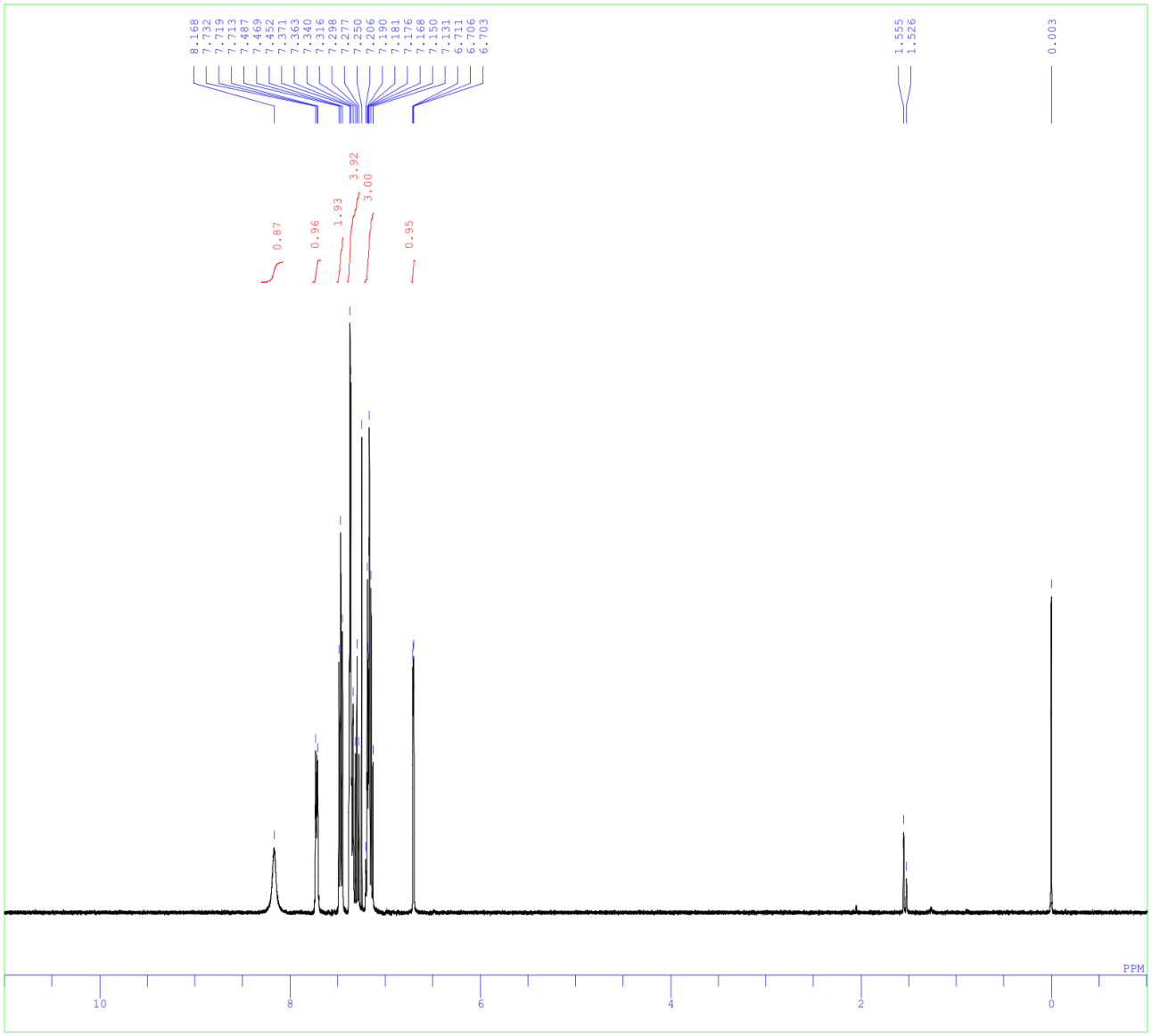
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 15[us]
Clipped                 = FALSE
Incomplete_Copy         = TRUE
Scans                   = 16
Total_Scans             = 16

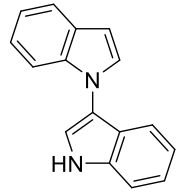
Relaxation_Delay        = 1[s]
Recvr_Gain              = 56
Temp_Get                = 20.4[dC]
X_90_Width              = 8.1[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 11[dB]
X_Pulse                 = 2.7[us]
Irr_Atn_Dec             = 25.803[dB]
Irr_Atn_Dec_Calc        = 25.803[dB]
Irr_Atn_Dec_Default_Calc = 25.803[dB]
Irr_Atn_Noe             = 25.803[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Temp1        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 1[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 1.69206016[s]
```



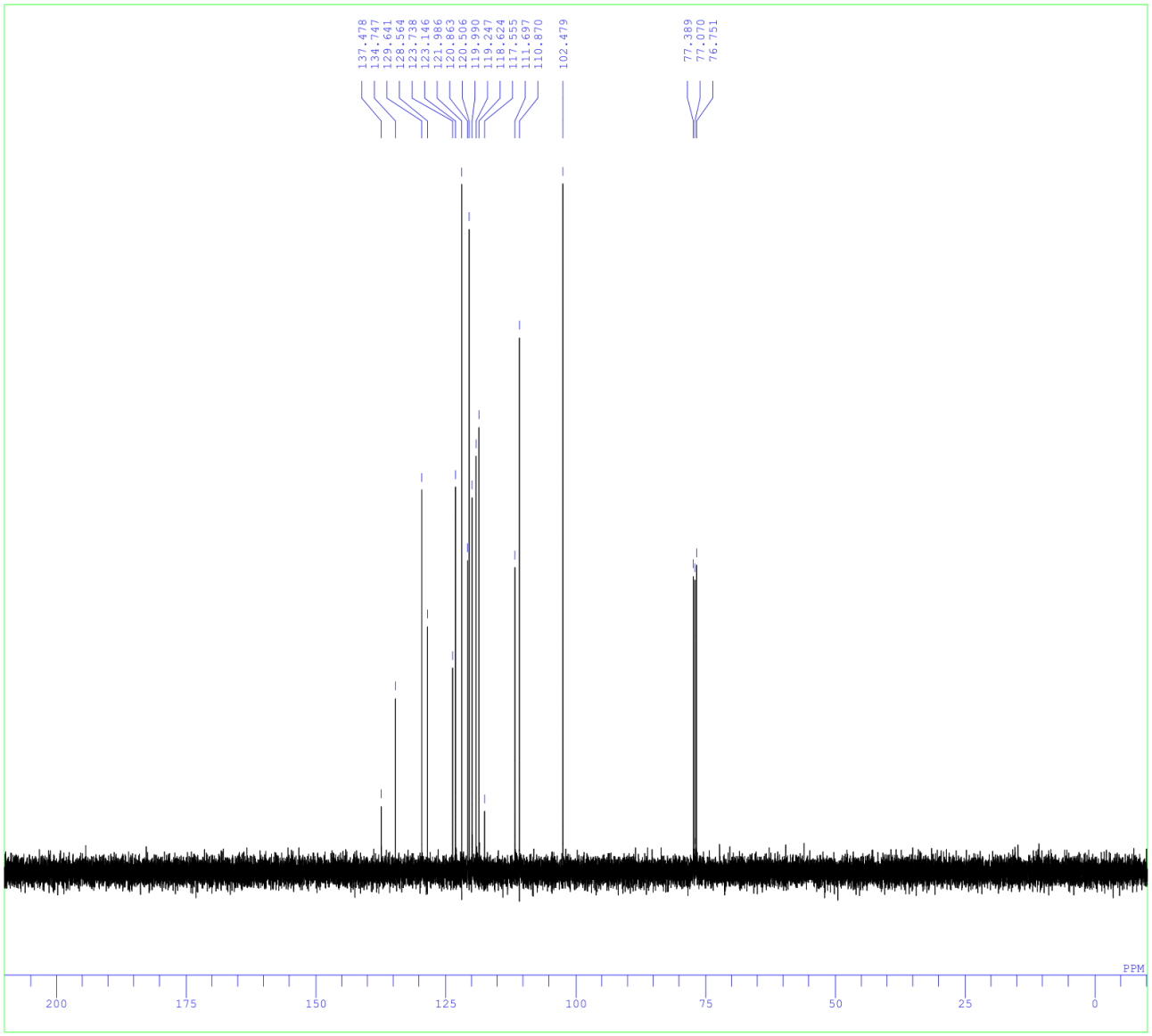
3aa



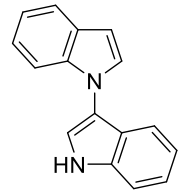

```
DFILE   fid
COMNT
DATIM   2023-02-06 18:13:51
OBNUC   H1
EXMOD   s2pul
OBFRQ         399.91 MHz
OBSET           1.99 KHz
OBFIN           2.00 Hz
POINT          32768
FREQU        6410.26 Hz
SCANS                4
ACQTM          3.5000 sec
PD             1.0000 sec
PW1              7.15 usec
IRNUC
CTEMP           37.0 c
SLVNT   cdcl3
EXREF           7.25 ppm
BF              0.00 Hz
RGAIN             48
```



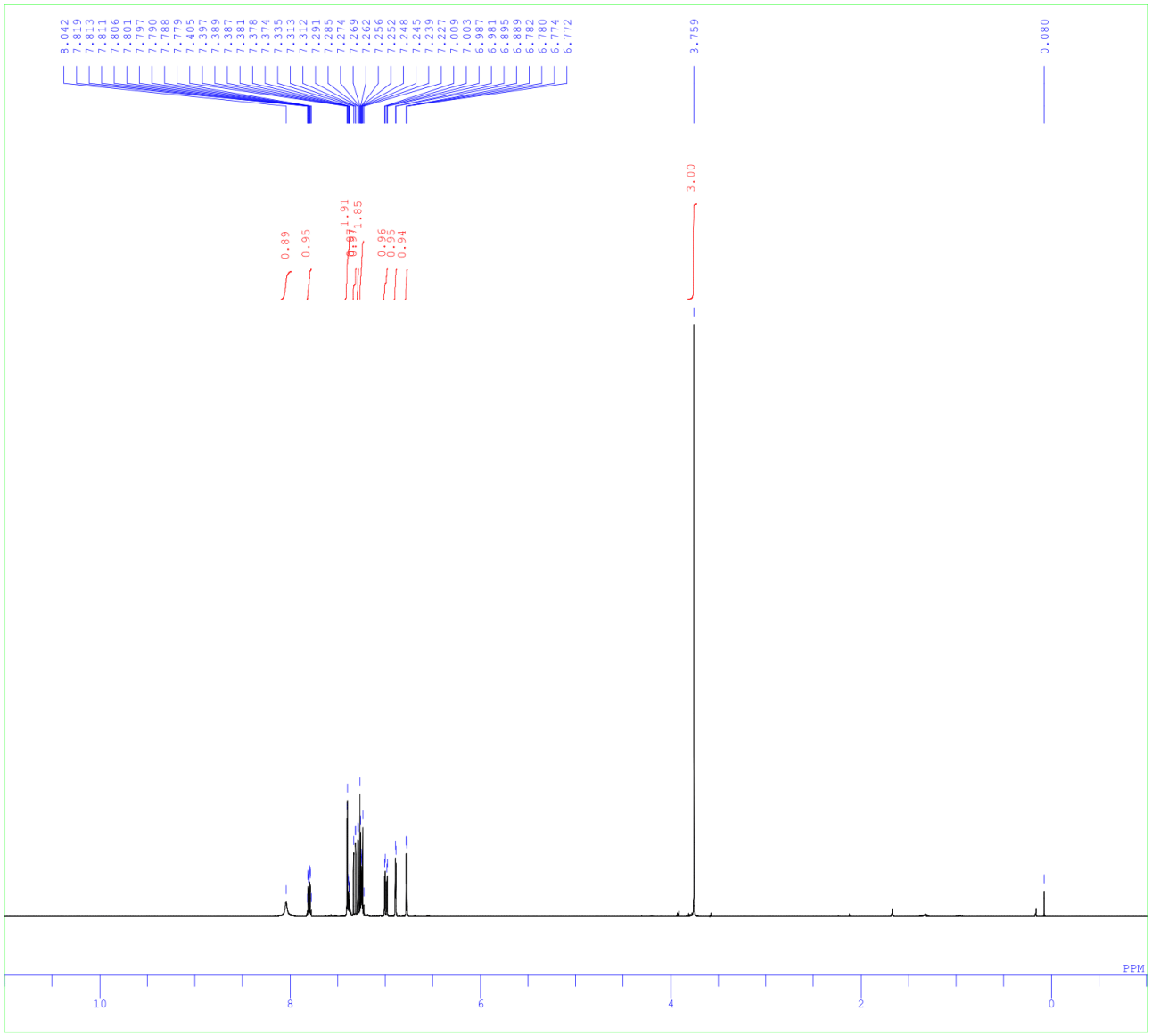
3ab



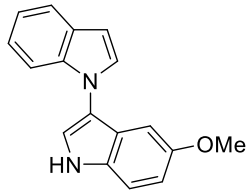

```
DFILE   fid
COMNT   YN-I-53-Crude DMSO-d6
DATIM   2023-02-10 12:25:42
OBNUC   C13
EXMOD   s2pul
OBFRQ         100.56 MHz
OBSET           8.40 KHz
OBFIN           8.30 Hz
POINT          32768
FREQU       25000.00 Hz
SCANS             32
ACQTM          1.3107 sec
PD             1.6893 sec
PW1            5.95 usec
IRNUC
CTEMP          37.0 c
SLVNT   cdcl3
EXREF          0.00 ppm
BF             0.00 Hz
RGAIN            48
```



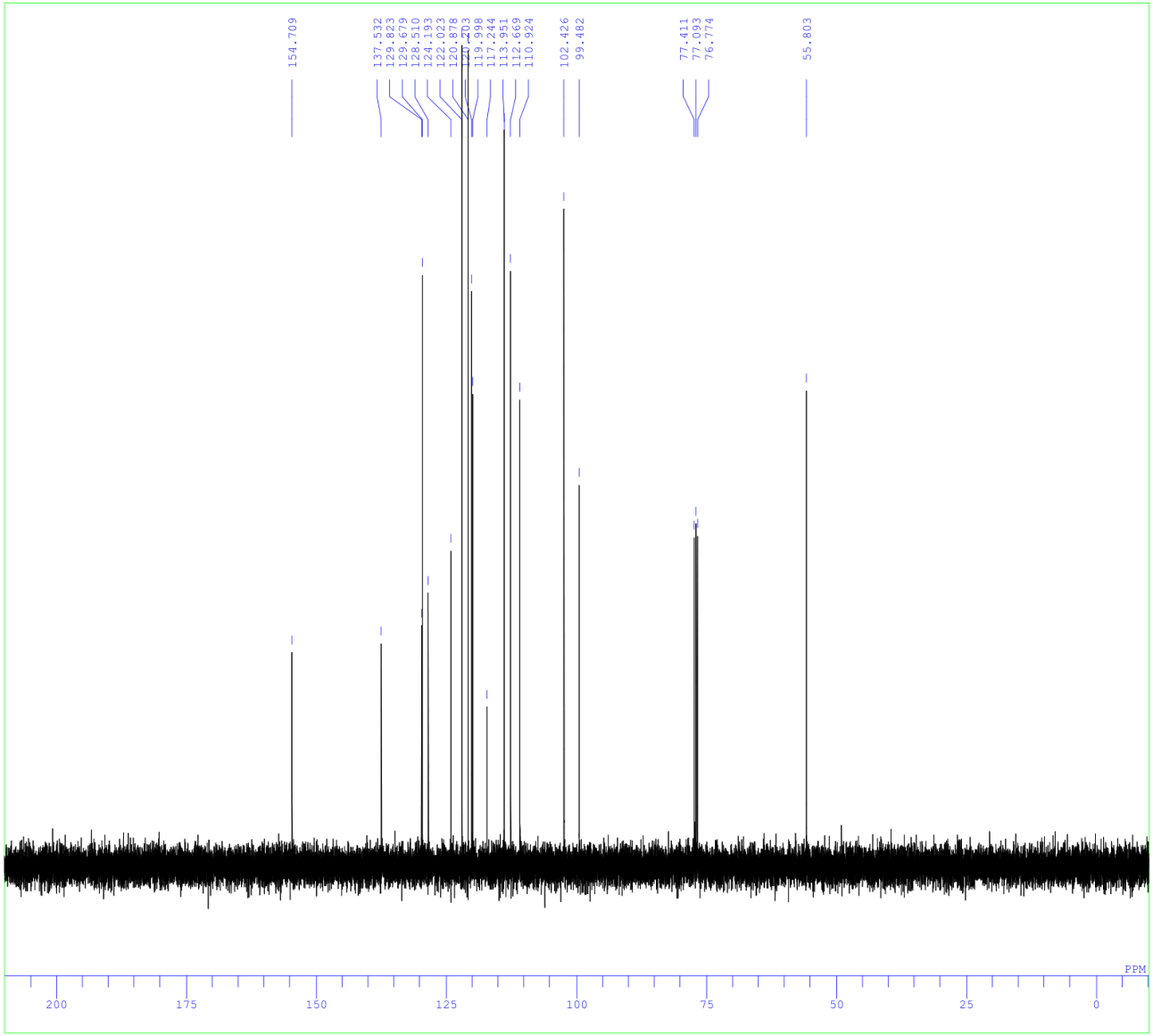
3ab



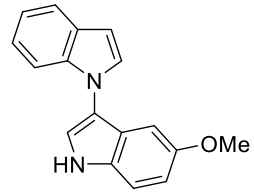

```
DFILE   fid
COMNT
DATIM   2023-02-06 22:18:08
OBNUC   H1
EXMOD   s2pul
OBFRQ         399.91 MHz
OBSET           1.99 KHz
OBFIN           2.00 Hz
POINT          32768
FREQU        6410.26 Hz
SCANS                4
ACQTM          3.5000 sec
PD             1.0000 sec
PW1              7.15 usec
IRNUC
CTEMP           37.0 c
SLVNT   cdcl3
EXREF           0.00 ppm
BF              0.00 Hz
RGAIN             36
```



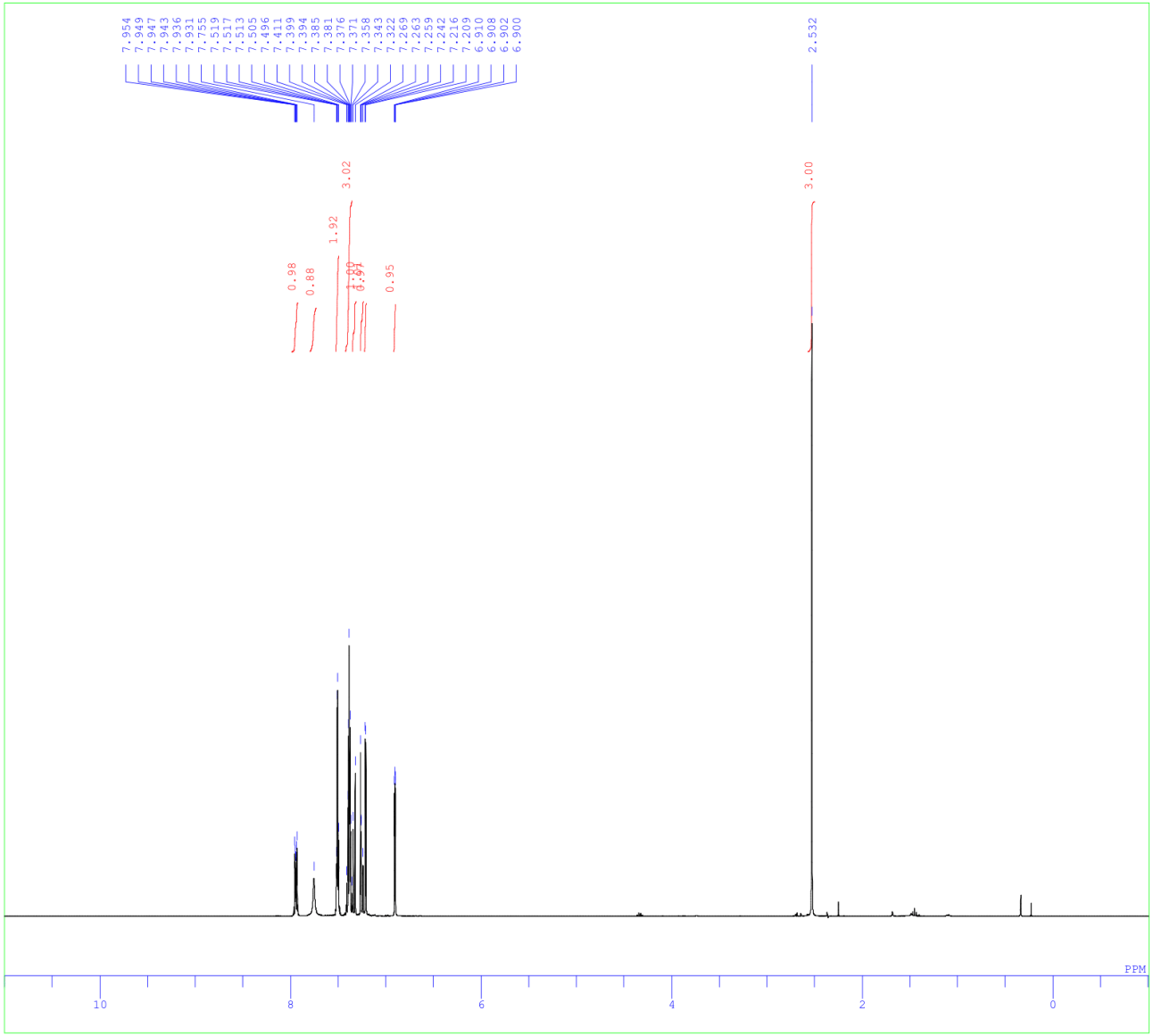
3ac



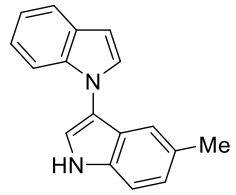

```
DFILE   fid
COMNT   20230207KND16-26
DATIM   2023-02-07 11:02:22
OBNUC   C13
EXMOD   s2pul
OBFRQ         100.56 MHz
OBSET           8.40 KHz
OBFIN           8.30 Hz
POINT          32768
FREQU       25000.00 Hz
SCANS             32
ACQTM          1.3107 sec
PD             1.6893 sec
PW1            5.95 usec
IRNUC
CTEMP          37.0 c
SLVNT   cdcl3
EXREF          0.00 ppm
BF             0.00 Hz
RGAIN            48
```



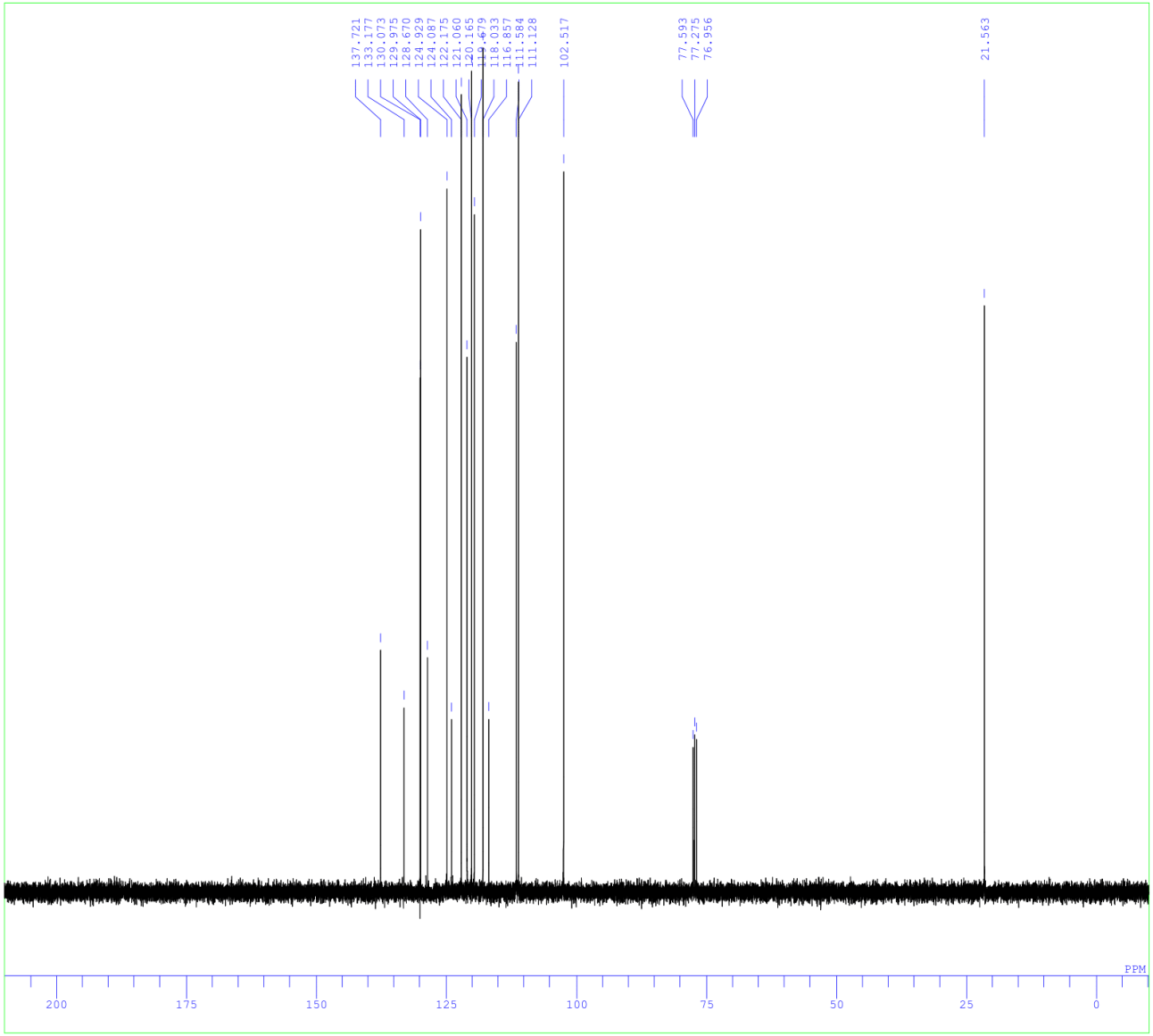
3ac



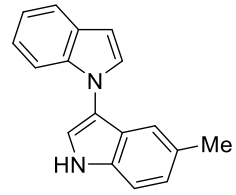

```
DFILE   5Me dimer 1H.als
COMNT
DATIM   2023-02-06 22:22:28
OBNUC   H1
EXMOD   s2pul
OBFRQ         399.91 MHz
OBSET           1.99 KHz
OBFIN           2.00 Hz
POINT          32768
FREQU        6410.26 Hz
SCANS                4
ACQTM          3.5000 sec
PD             1.0000 sec
PW1              7.15 usec
IRNUC
CTEMP            37.0 c
SLVNT   cdcl3
EXREF            0.00 ppm
BF               0.00 Hz
RGAIN              24
```



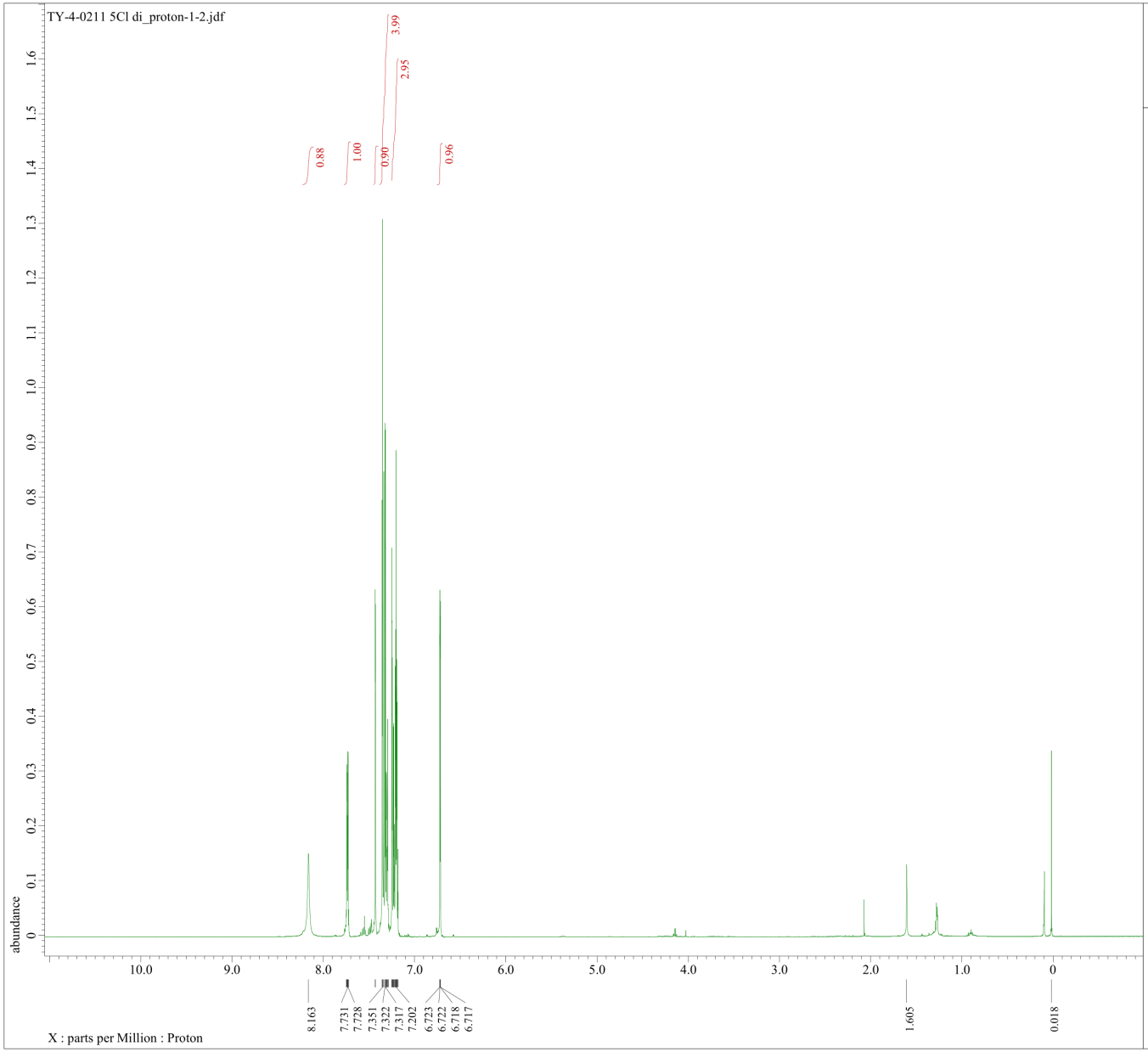
3ad




```
DFILE   5Me dimer 13C.als
COMNT   20230207KND16-26
DATIM   2023-02-07 11:11:06
OBNUC   C13
EXMOD   s2pul
OBFRQ         100.56 MHz
OBSET           8.40 KHz
OBFIN           8.30 Hz
POINT          32768
FREQU       25000.00 Hz
SCANS             32
ACQTM          1.3107 sec
PD             1.6893 sec
PW1            5.95 usec
IRNUC
CTEMP          37.0 c
SLVNT   cdcl3
EXREF          0.00 ppm
BF             0.00 Hz
RGAIN            48
```



3ad



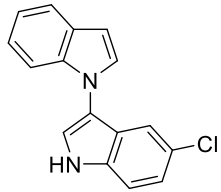
JEOL

```
Filename              = TY-4-0211 5Cl di_prot
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-4-0211 5Cl di
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 11-FEB-2023 11:52:34
Revision_Time         = 11-FEB-2023 14:23:34

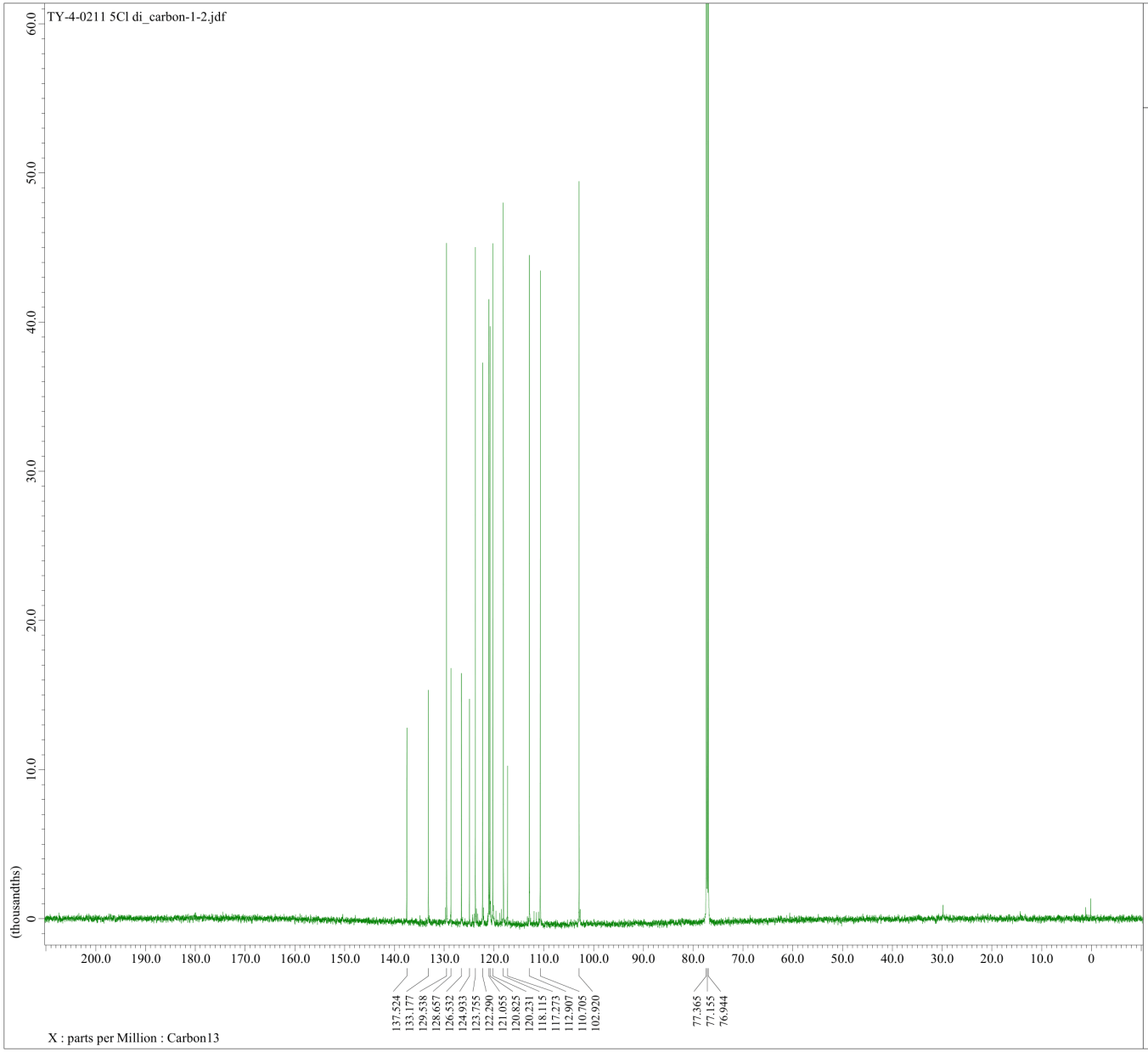
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 5[us]
Clipped               = FALSE
Scans                 = 4
Total_Scans           = 4

Relaxation_Delay      = 1[s]
Recvr_Gain            = 36
Temp_Get              = 20.4[dC]
X_90_Width            = 9.9[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 4.95[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = (0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```

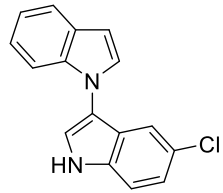


3ae

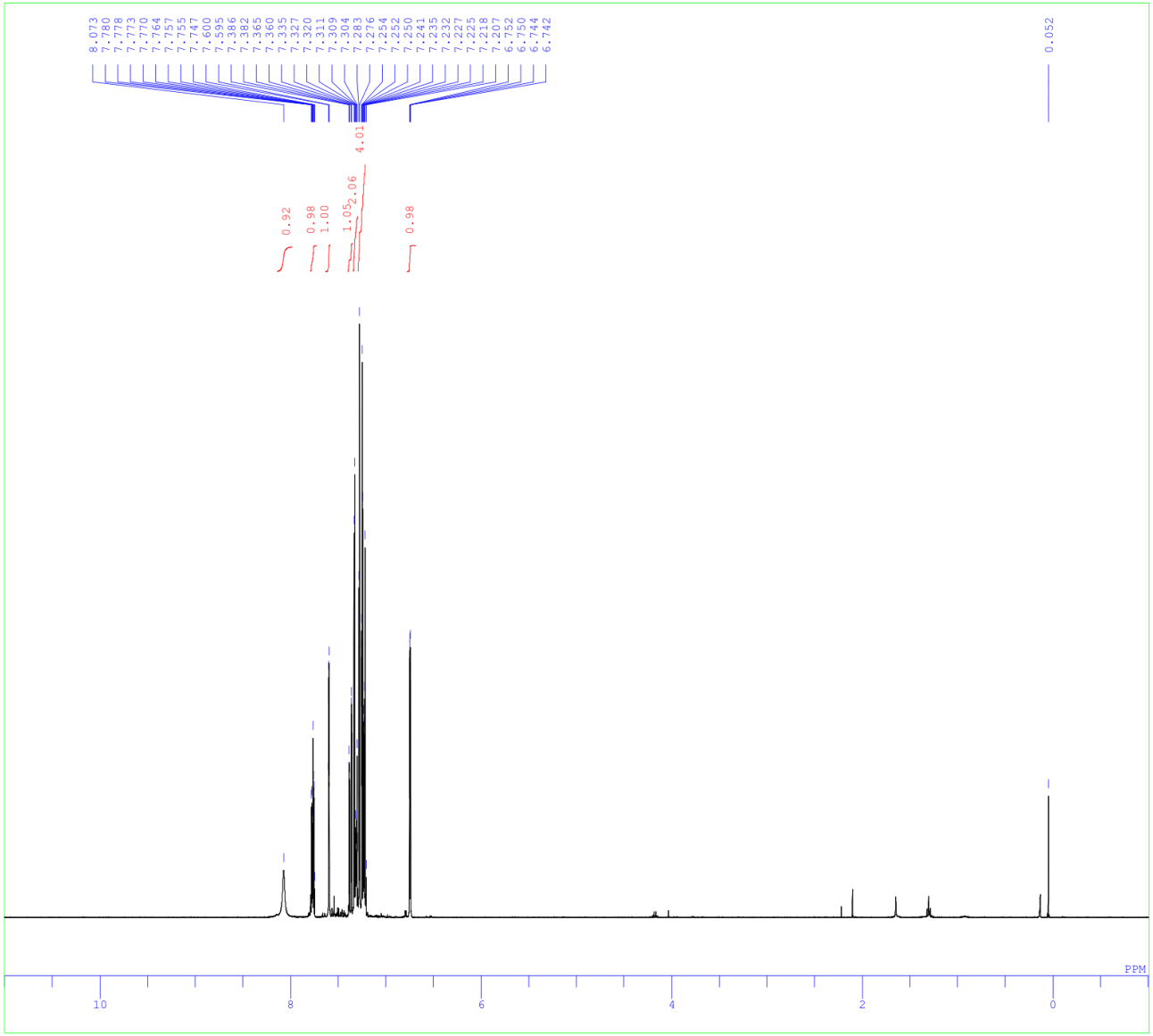


JEOL

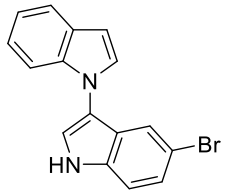
Filename	= TY-4-0211 5Cl di_c
Author	= delta
Experiment	= carbon.jxp
Sample_Id	= TY-4-0211 5Cl di
Solvent	= CHLOROFORM-D
Actual_Start_Time	= 11-FEB-2023 11:54:
Revision_Time	= 11-FEB-2023 14:25:
Data_Format	= 1D COMPLEX
Dim_Size	= 26214
X_Domain	= Carbon13
Dim_Title	= Carbon13
Dim_Units	= [ppm]
Dimensions	= X
Spectrometer	= JNM-ECZ600R/S3
Field_Strength	= 14.09636928[T] (60
X_Acq_Duration	= 0.69206016[s]
X_Domain	= Carbon13
X_Freq	= 150.91343039[MHz]
X_Offset	= 100[ppm]
X_Points	= 32768
X_Prescans	= 4
X_Resolution	= 1.44496109[Hz]
X_Sweep	= 47.34848485[kHz]
X_Sweep_Clipped	= 37.87878788[kHz]
Irr_Domain	= Proton
Irr_Freq	= 600.1723046[MHz]
Irr_Offset	= 5[ppm]
Blanking	= 15[us]
Clipped	= FALSE
Scans	= 256
Total_Scans	= 256
Relaxation_Delay	= 1[s]
Recvr_Gain	= 56
Temp_Get	= 20.5[dC]
X_90_Width	= 8.1[us]
X_Acq_Time	= 0.69206016[s]
X_Angle	= 30[deg]
X_Atn	= 11[dB]
X_Pulse	= 2.7[us]
Irr_Atn_Dec	= 25.803[dB]
Irr_Atn_Dec_Calc	= 25.803[dB]
Irr_Atn_Dec_Default_Calc	= 25.803[dB]
Irr_Atn_Noe	= 25.803[dB]
Irr_Dec_Bandwidth_Hz	= 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm	= 12.05794078[ppm]
Irr_Dec_Freq	= 600.1723046[MHz]
Irr_Dec_Merit_Factor	= 2.2
Irr_Decoupling	= TRUE
Irr_Noe	= TRUE
Irr_Noise	= WALTZ
Irr_Offset_Default	= 5[ppm]
Irr_Pwidth	= 76[us]
Irr_Pwidth_Default	= 76[us]
Irr_Pwidth_Default_Calc	= 76[us]
Irr_Pwidth_Templ	= 76[us]
Irr_Wurst	= FALSE
Decimation_Rate	= 0
Experiment_Path	= c:\Program Files\J
Initial_Wait	= 1[s]
Noe_Time	= 1[s]
Noe_Time_Flag	= FALSE
Relaxation_Delay_Calc	= 0[s]
Relaxation_Delay_Temp	= 1[s]
Repetition_Time	= 1.69206016[s]



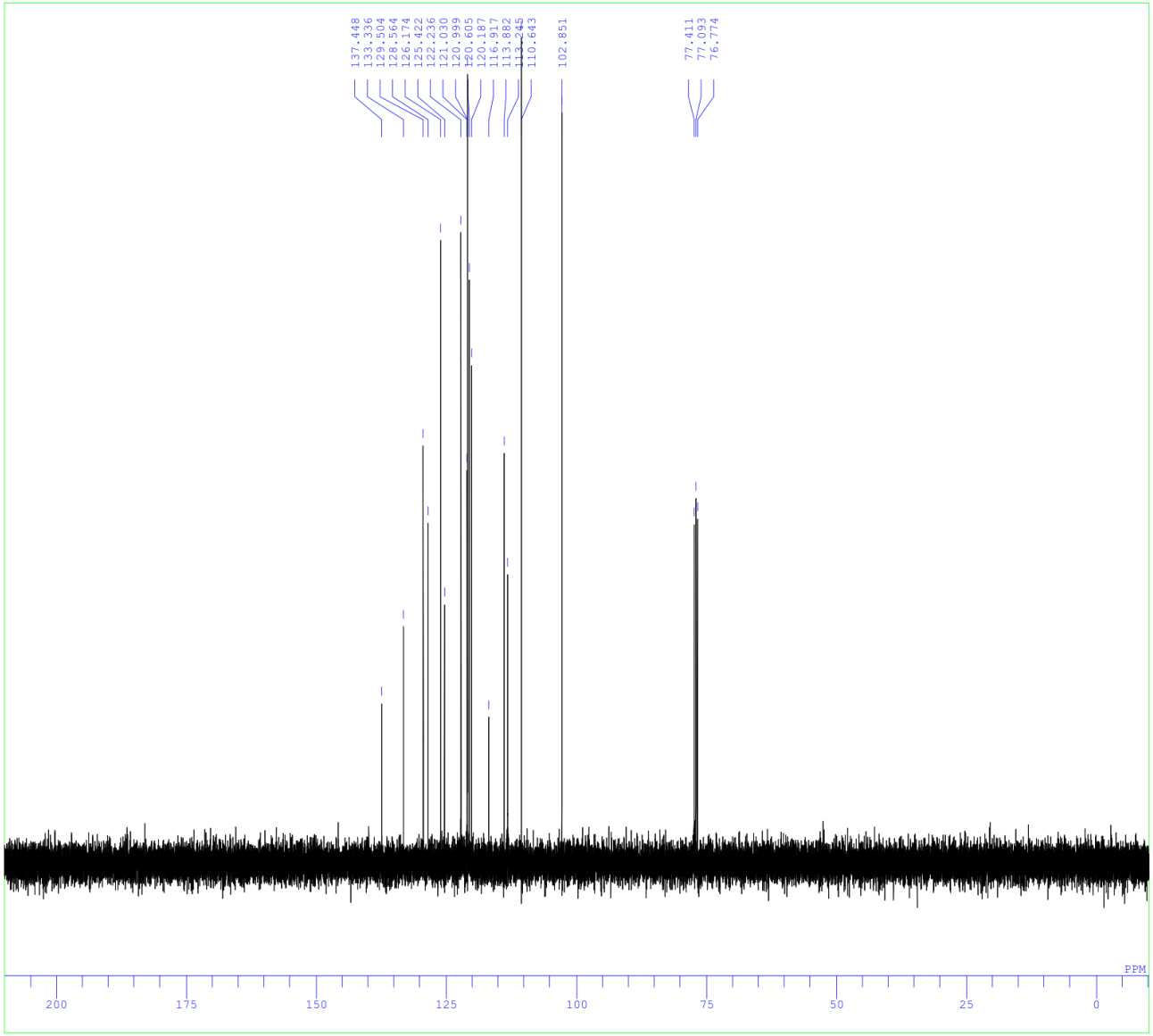
3ae



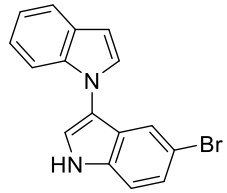

```
DFILE   fid
COMNT
DATIM   2023-02-06 18:28:44
OBNUC   H1
EXMOD   s2pul
OBFRQ         399.91 MHz
OBSET           1.99 KHz
OBFIN           2.00 Hz
POINT          32768
FREQU        6410.26 Hz
SCANS                4
ACQTM          3.5000 sec
PD             1.0000 sec
PW1              7.15 usec
IRNUC
CTEMP            37.0 c
SLVNT   cdcl3
EXREF            7.25 ppm
BF               0.00 Hz
RGAIN              36
```



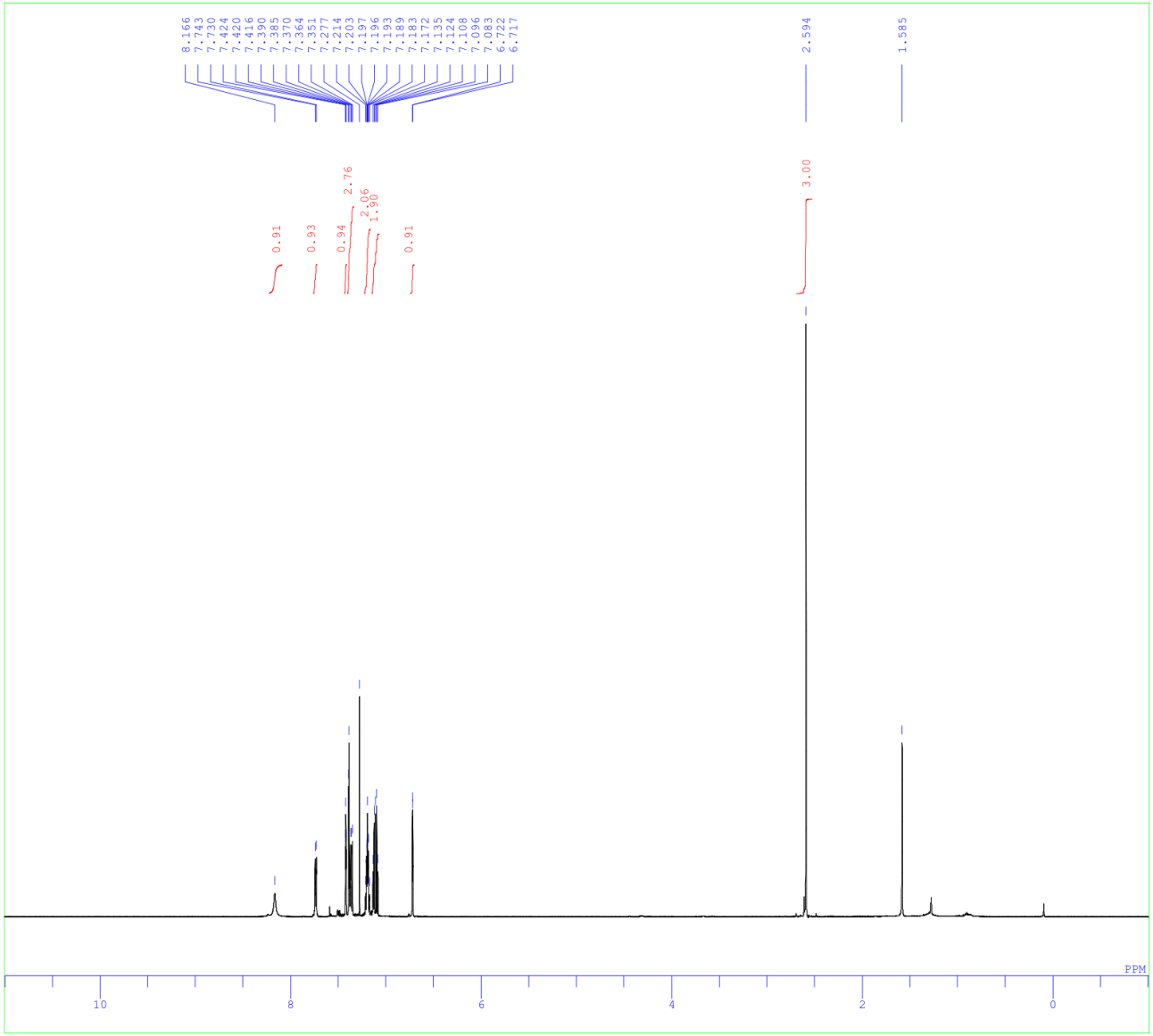
3af



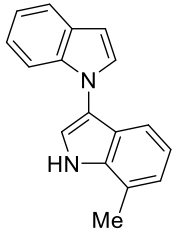

```
DFILE   fid
COMNT   20230207KND16-26
DATIM   2023-02-07 10:55:11
OBNUC   C13
EXMOD   s2pul
OBFRQ         100.56 MHz
OBSET           8.40 KHz
OBFIN           8.30 Hz
POINT          32768
FREQU       25000.00 Hz
SCANS             32
ACQTM          1.3107 sec
PD             1.6893 sec
PW1            5.95 usec
IRNUC
CTEMP          37.0 c
SLVNT   cdcl3
EXREF          0.00 ppm
BF             0.00 Hz
RGAIN            48
```



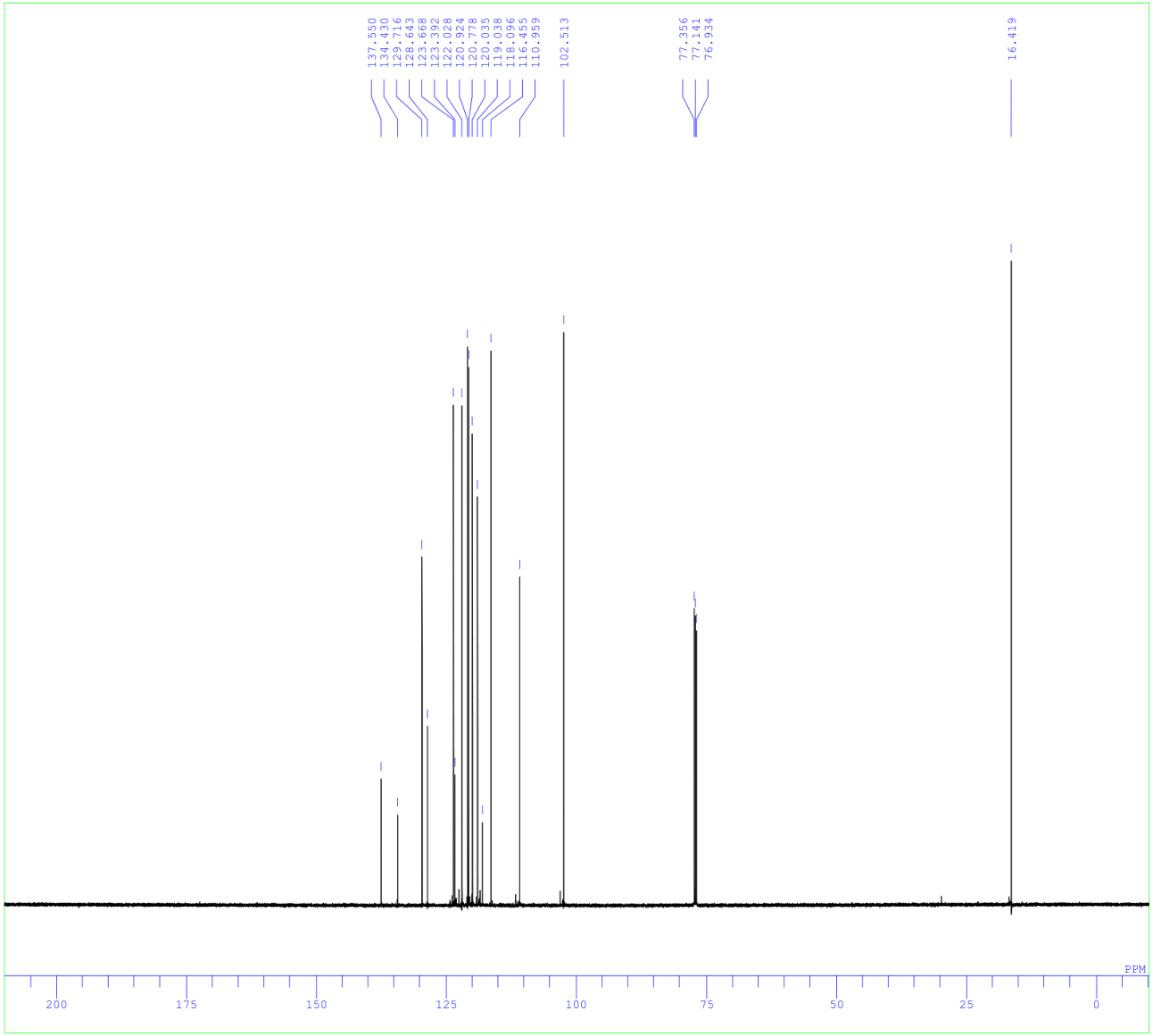
3af



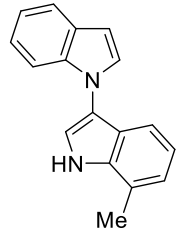

```
DFILE   7Me dimer 1H.als
COMNT   TIT/1803-B
DATIM   2021-08-17 13:44:41
OBNUC   H1
EXMOD   s2pul
OBFRQ         599.76 MHz
OBSET           5.06 KHz
OBFIN           0.60 Hz
POINT          32768
FREQU        9615.38 Hz
SCANS             32
ACQTM          3.4079 sec
PD             1.0000 sec
PW1              5.40 usec
IRNUC
CTEMP            20.0 c
SLVNT   cdcl3
EXREF            0.00 ppm
BF               0.00 Hz
RGAIN              42
```



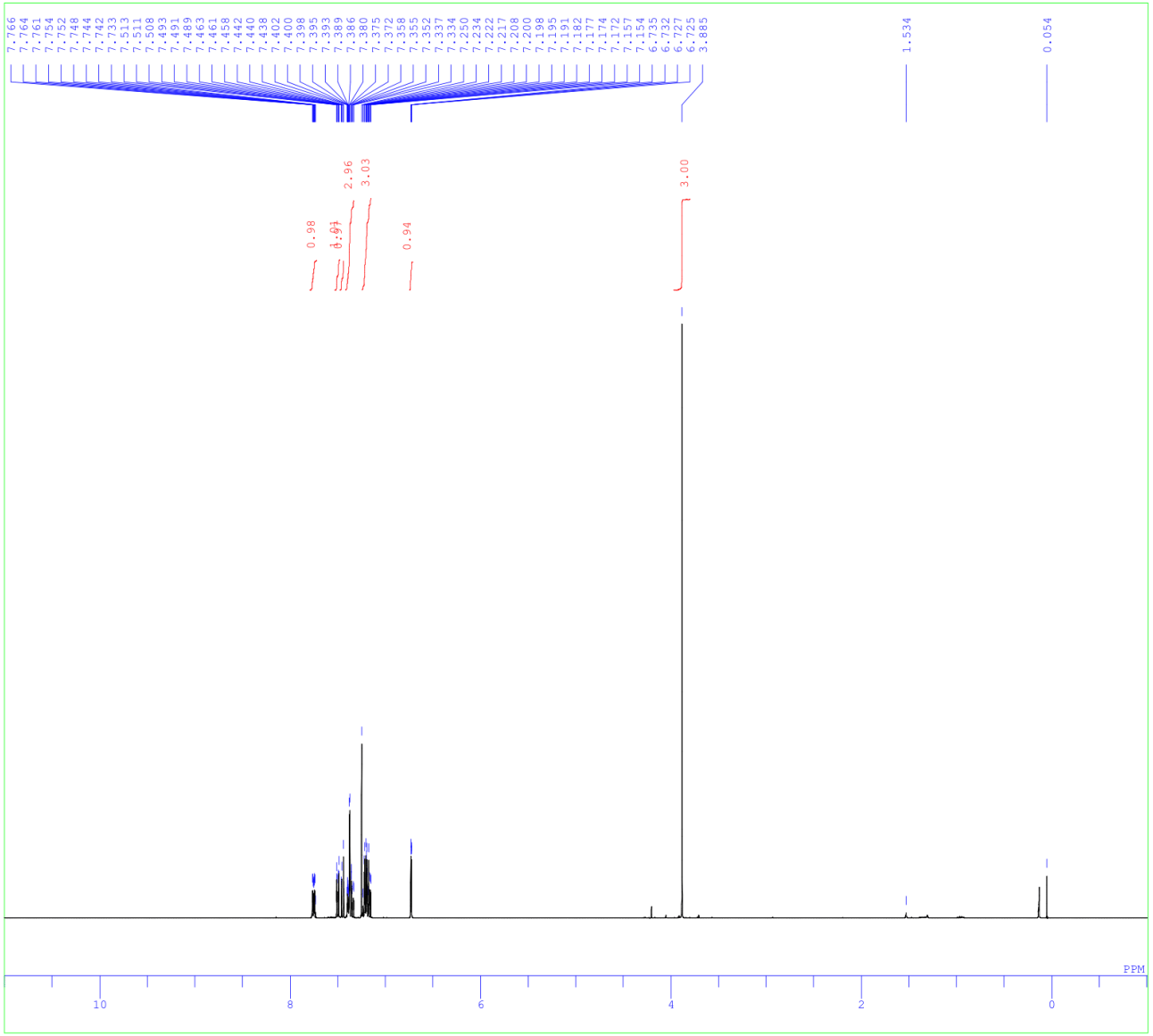
3ag



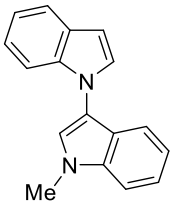

```
DFILE   7Me 13C.als
COMNT
DATIM   2021-08-15 15:54:34
OBNUC   C13
EXMOD   s2pul
OBFRQ         150.82 MHz
OBSET           6.72 KHz
OBFIN           8.70 Hz
POINT          32768
FREQU       37878.79 Hz
SCANS             64
ACQTM          0.8651 sec
PD             2.1349 sec
PW1            6.05 usec
IRNUC
CTEMP          30.0 c
SLVNT   cdcl3
EXREF          0.00 ppm
BF             0.00 Hz
RGAIN            60
```



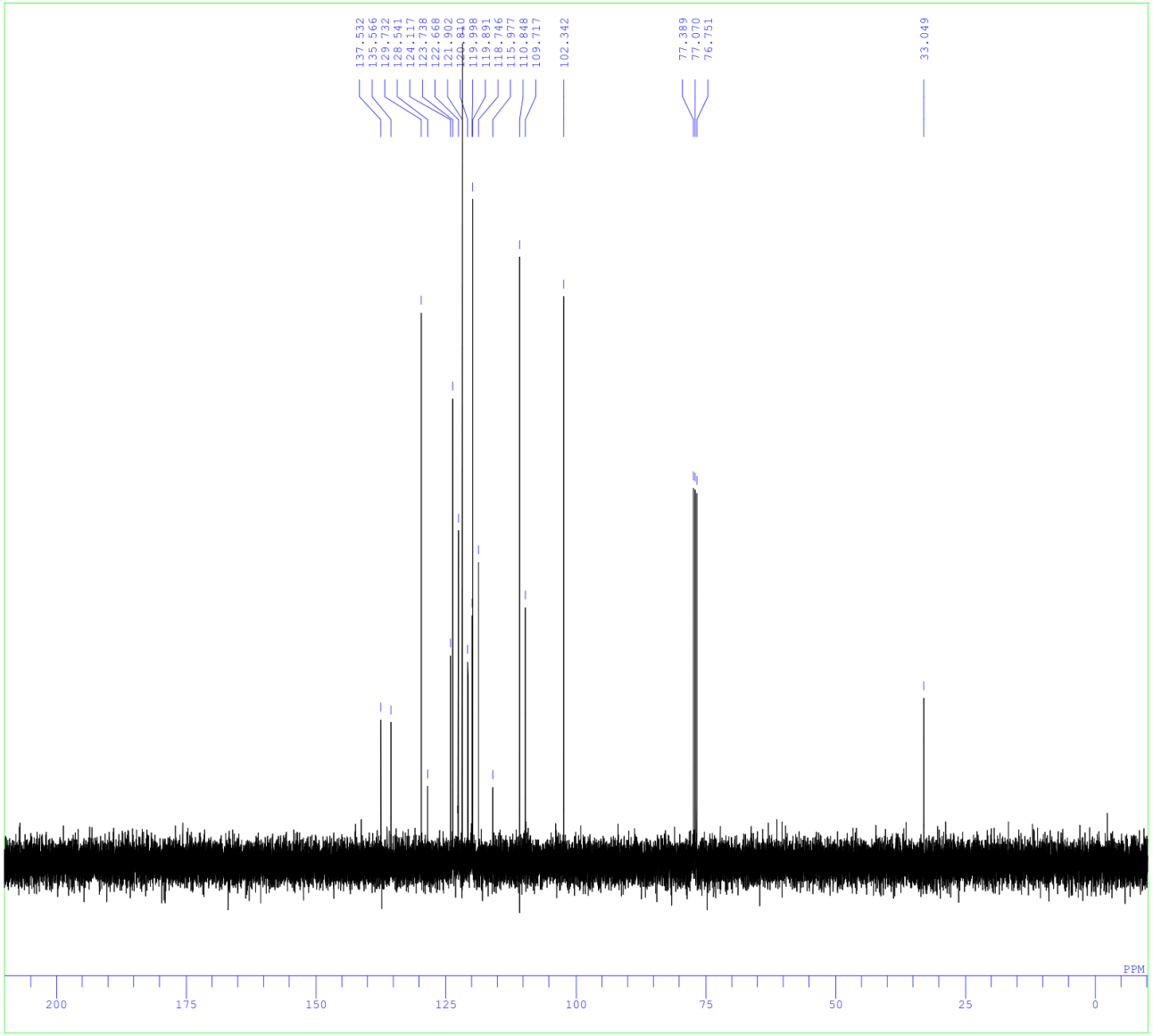
3ag



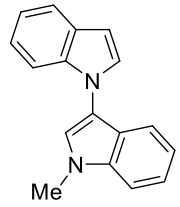

```
DFILE   fid
COMNT
DATIM   2023-02-06 18:18:48
OBNUC   H1
EXMOD   s2pul
OBFRQ         399.91 MHz
OBSET           1.99 KHz
OBFIN           2.00 Hz
POINT          32768
FREQU        6410.26 Hz
SCANS                4
ACQTM          3.5000 sec
PD             1.0000 sec
PW1              7.15 usec
IRNUC
CTEMP            37.0 c
SLVNT   cdcl3
EXREF            7.25 ppm
BF               0.00 Hz
RGAIN              36
```



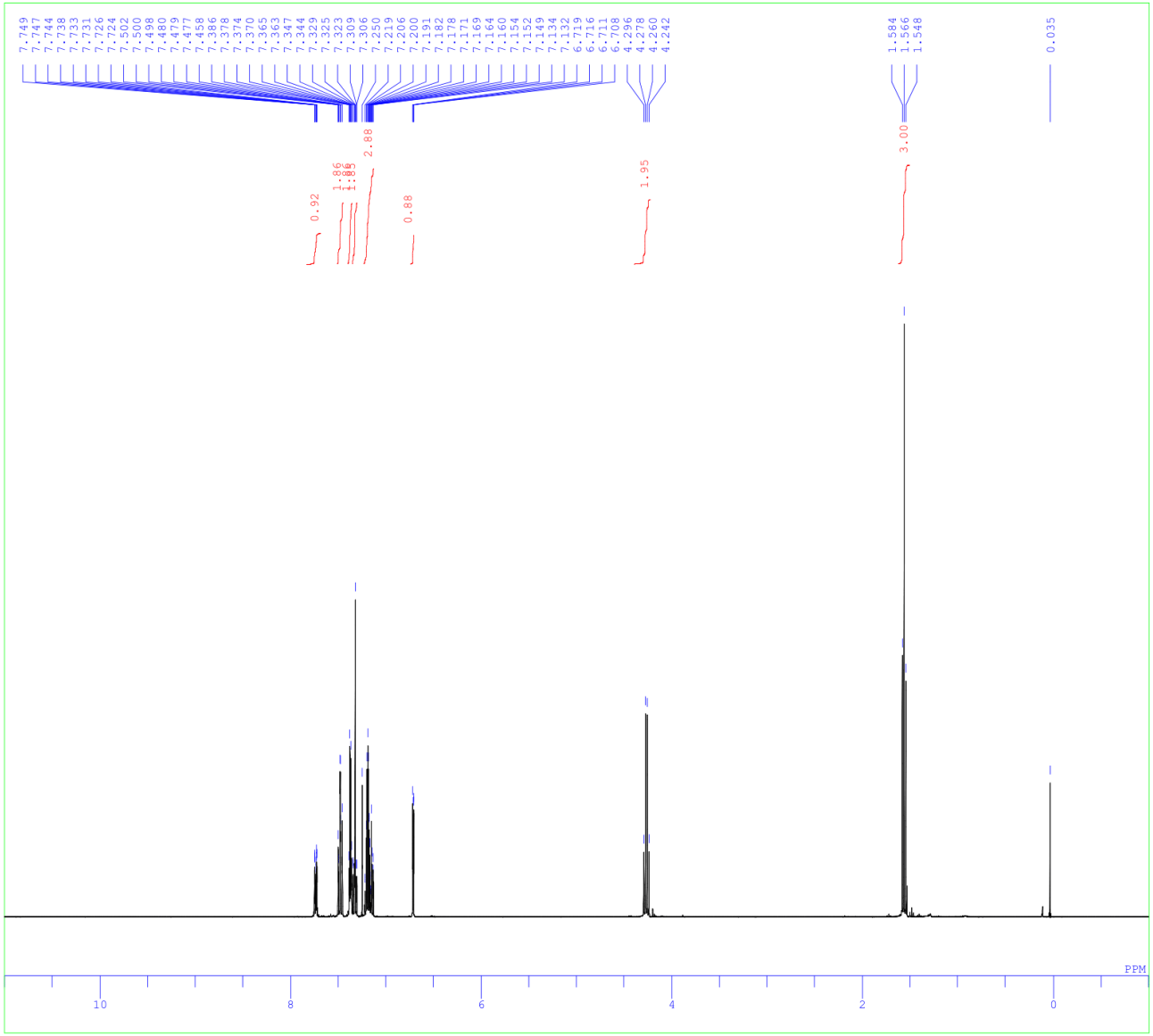
3ah



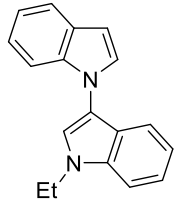

```
DFILE   fid
COMNT   20230207KND16-26
DATIM   2023-02-07 10:38:53
OBNUC   C13
EXMOD   s2pul
OBFRQ        100.56 MHz
OBSET          8.40 KHz
OBFIN          8.30 Hz
POINT         32768
FREQU      25000.00 Hz
SCANS            32
ACQTM          1.3107 sec
PD             1.6893 sec
PW1            5.95 usec
IRNUC
CTEMP          37.0 c
SLVNT   cdcl3
EXREF          0.00 ppm
BF             0.00 Hz
RGAIN            48
```



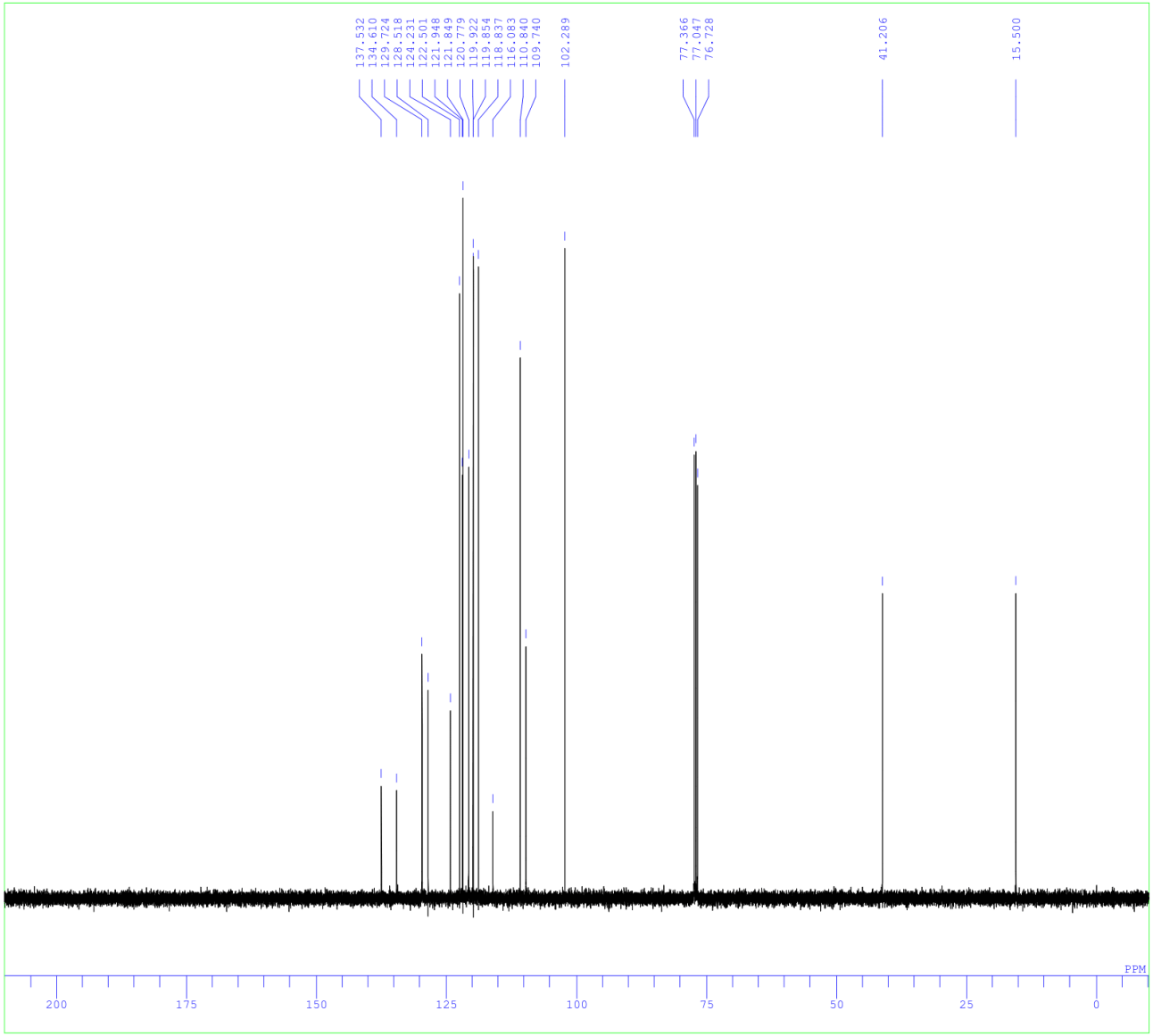
3ah



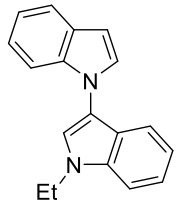

```
DFILE   NEt dimer 1H.als
COMNT
DATIM   2023-02-06 18:22:01
OBNUC   H1
EXMOD   s2pul
OBFRQ         399.91 MHz
OBSET           1.99 KHz
OBFIN           2.00 Hz
POINT          32768
FREQU        6410.26 Hz
SCANS                4
ACQTM          3.5000 sec
PD             1.0000 sec
PW1              7.15 usec
IRNUC
CTEMP            37.0 c
SLVNT   cdcl3
EXREF            7.25 ppm
BF               0.00 Hz
RGAIN              36
```



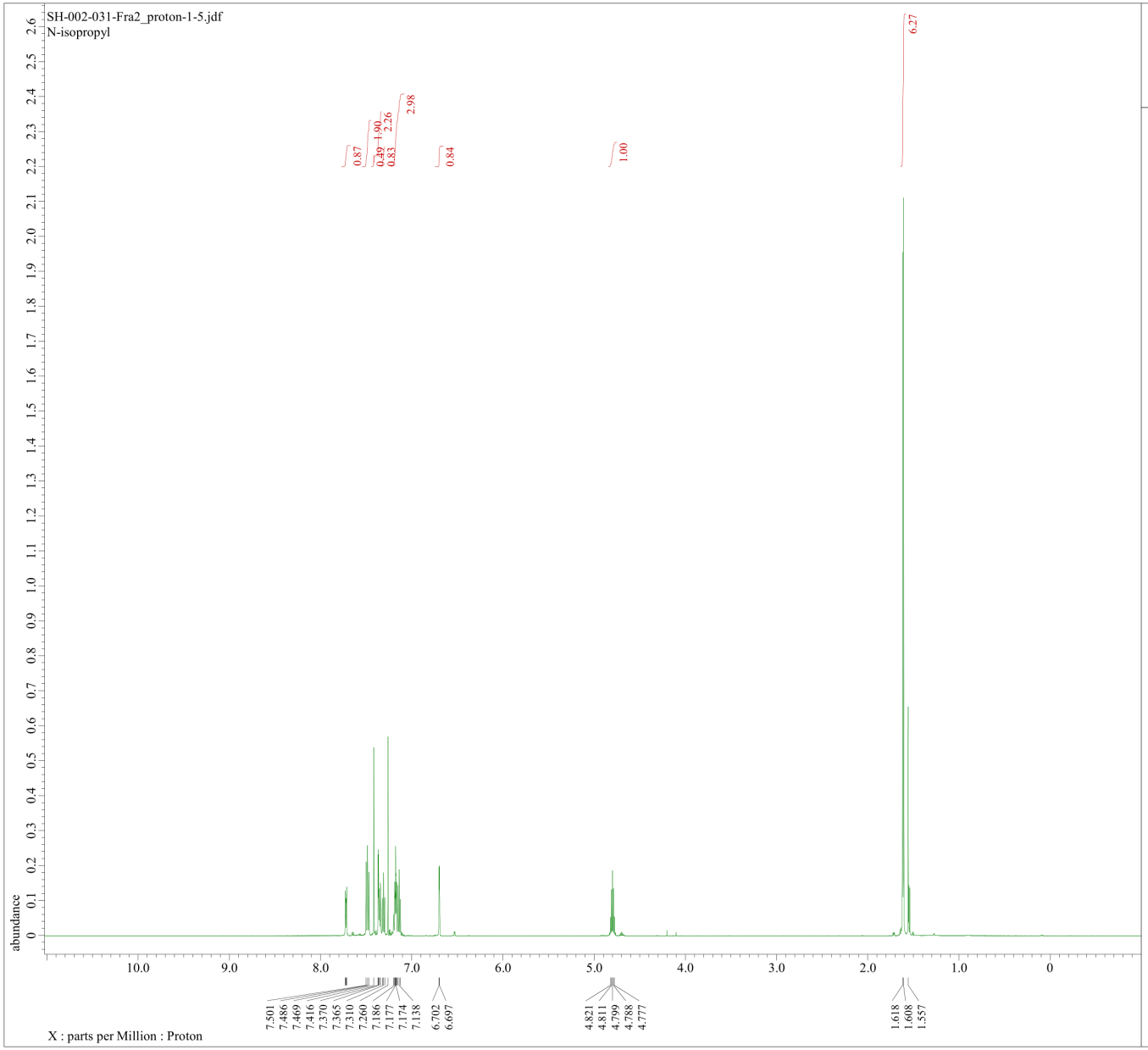
3ai




```
DFILE   fid
COMNT   YN-I-53-Crude DMSO-d6
DATIM   2023-02-10 12:32:18
OBNUC   C13
EXMOD   s2pul
OBFRQ         100.56 MHz
OBSET           8.40 KHz
OBFIN           8.30 Hz
POINT          32768
FREQU       25000.00 Hz
SCANS             32
ACQTM          1.3107 sec
PD             1.6893 sec
PW1            5.95 usec
IRNUC
CTEMP          37.0 c
SLVNT   cdcl3
EXREF          0.00 ppm
BF             0.00 Hz
RGAIN            48
```



3ai



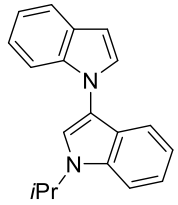
JEOL

```
Filename              = SH-002-031-Fra2_proto
Author                = delta
Experiment            = proton.jxp
Sample_Id             = SH-002-031-Fra2
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 21-AUG-2021 13:14:01
Revision_Time         =  9-FEB-2023 23:00:38

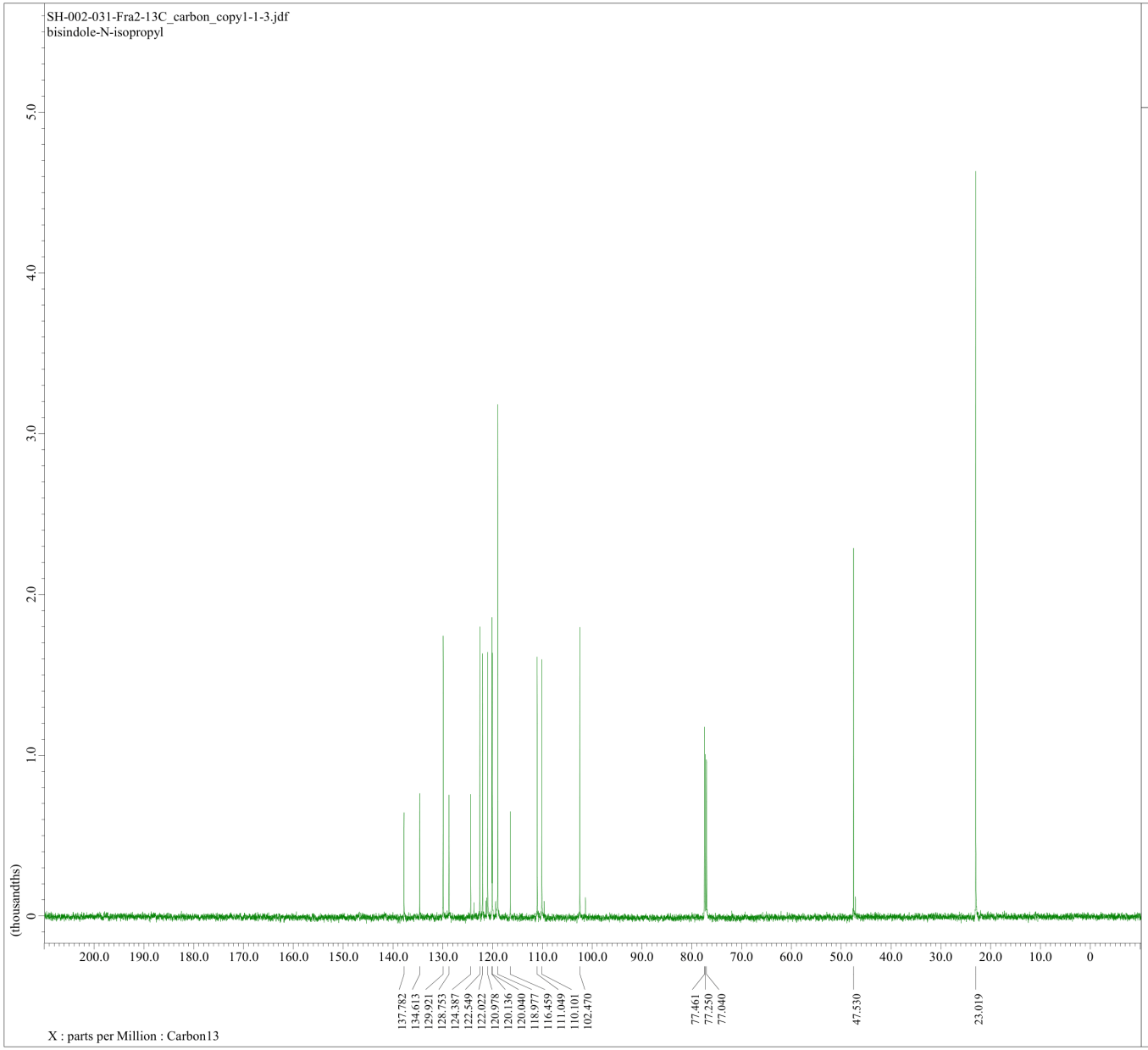
Comment               = N-isopropyl
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 16
Total_Scans           = 16

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 21[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```



3aj

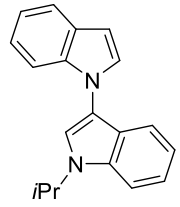



```
Filename                = SH-002-031-Fra2-13
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = SH-002-031-Fra2-13
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 21-AUG-2021 13:33:
Revision_Time           =  9-FEB-2023 23:01:

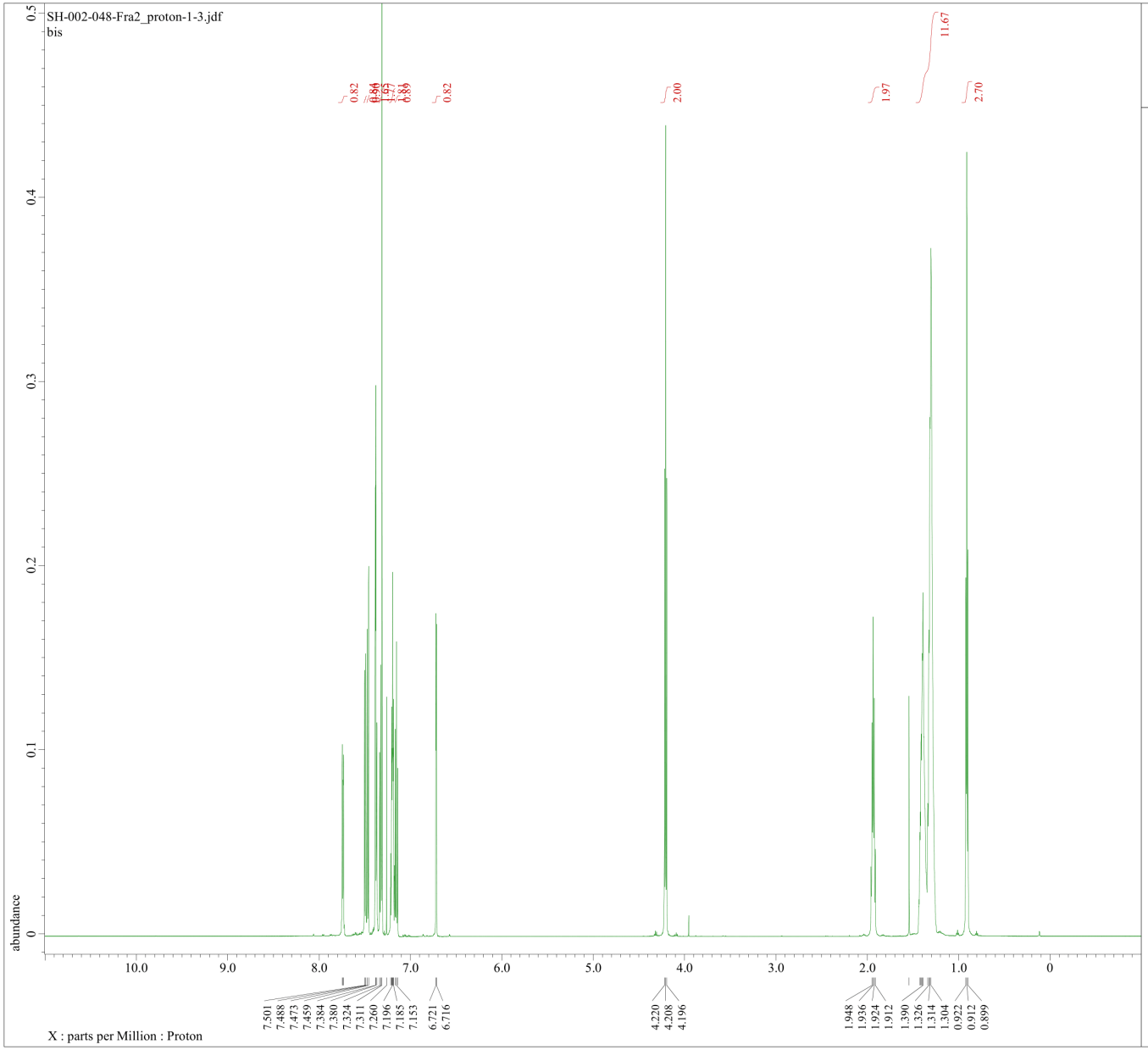
Comment                 = bisindole-N-isopro
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Incomplete_Copy         = TRUE
Scans                   = 149
Total_Scans             = 149

Relaxation_Delay        = 1[s]
Recvr_Gain              = 26
Temp_Get                = 21.1[dC]
X_90_Width              = 11.8[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 9.6[dB]
X_Pulse                 = 3.93333333[us]
Irr_Atn_Dec             = 27.986[dB]
Irr_Atn_Dec_Calc        = 27.986[dB]
Irr_Atn_Dec_Default_Calc = 27.986[dB]
Irr_Atn_Noe             = 27.986[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Templ        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 1[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 1.69206016[s]
```



3aj



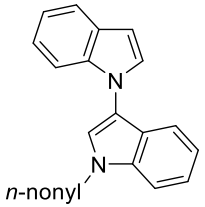
JEOL

```
Filename              = SH-002-048-Fra2_proto
Author                = delta
Experiment            = proton.jxp
Sample_Id             = SH-002-048-Fra2
Solvent               = CHLOROFORM-D
Actual_Start_Time     =  1-SEP-2021 20:28:33
Revision_Time         =  9-FEB-2023 23:12:20

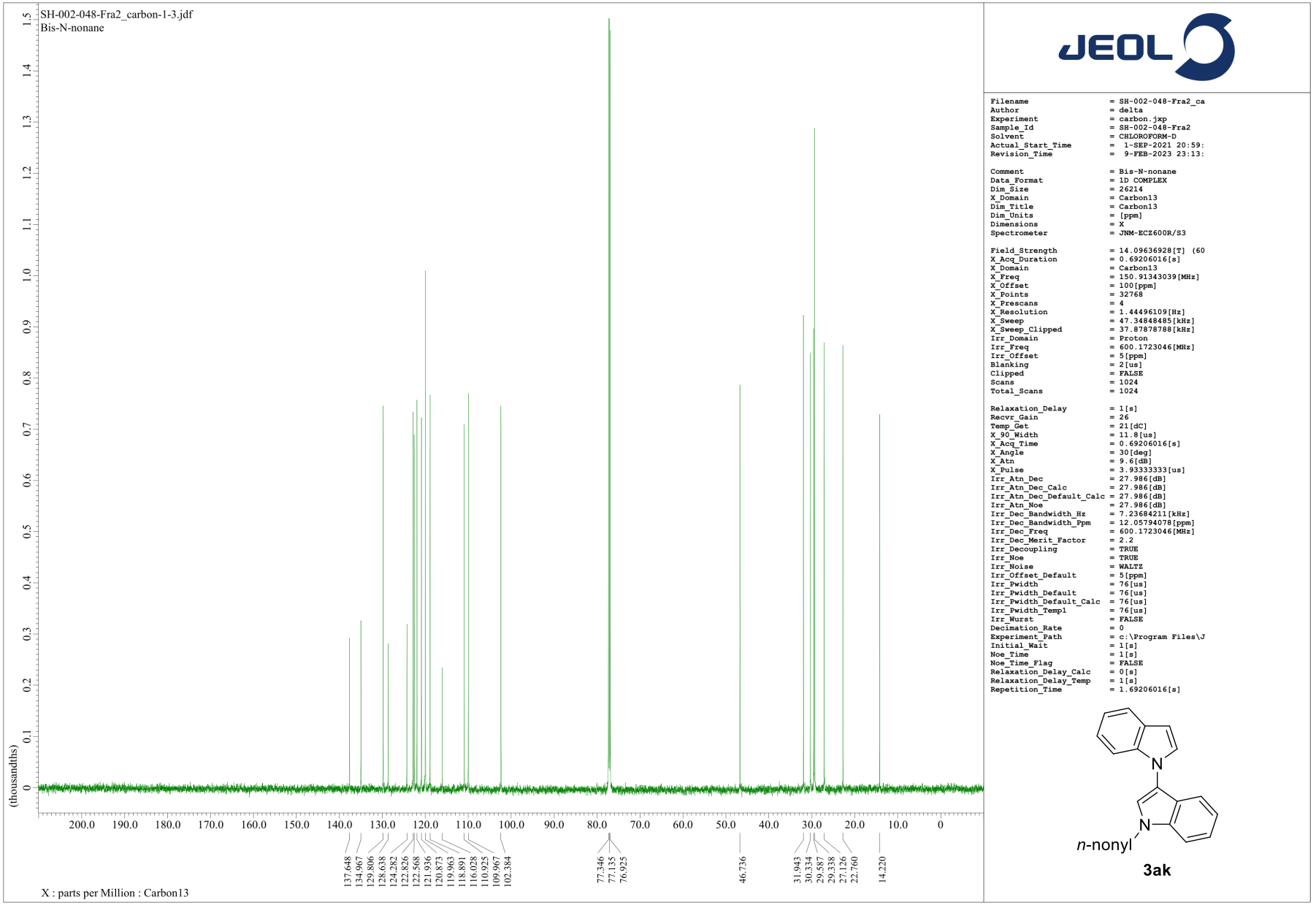
Comment               = bis
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

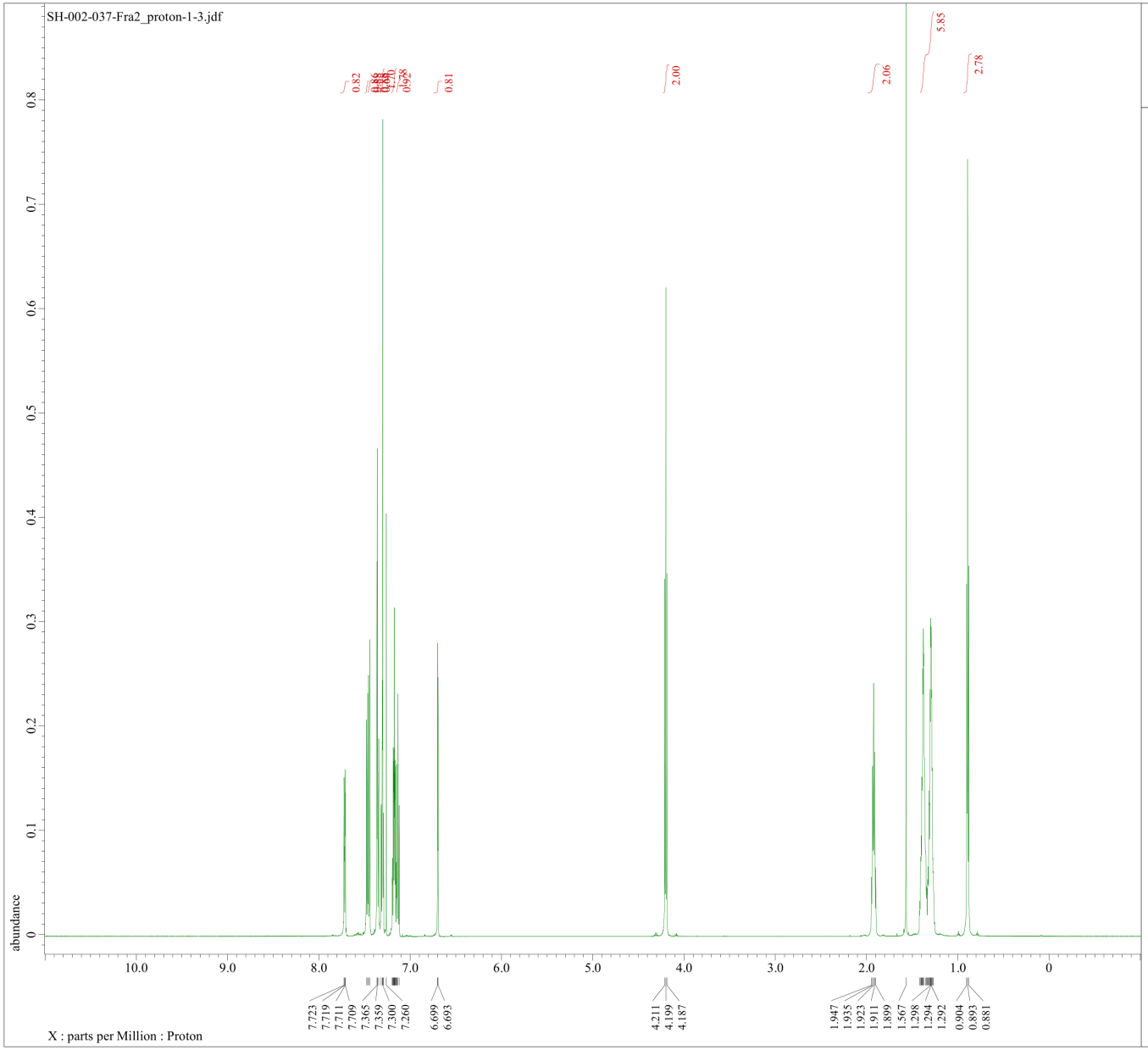
Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 16
Total_Scans           = 16

Relaxation_Delay      = 1[s]
Recvr_Gain            = 36
Temp_Get              = 21[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```



3ak


SH-002-048-Fra2_carbon-1-3.jdf
Bis-N-nonane
(thousandths)
X : parts per Million : Carbon13
137.648
134.967
129.806
128.638
124.282
122.826
122.568
121.936
120.873
119.963
118.891
116.028
110.925
109.967
102.384
77.346
77.135
76.925
46.736
31.943
30.334
29.587
29.338
27.126
22.760
14.220
Filename = SH-002-048-Fra2_ca
Author = delta
Experiment = carbon.jxp
Sample_Id = SH-002-048-Fra2
Solvent = CHLOROFORM-D
Actual_Start_Time = 1-SEP-2021 20:59:
Revision_Time = 9-FEB-2023 23:13:
Comment = Bis-N-nonane
Data_Format = 1D COMPLEX
Dim_Size = 26214
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ600R/S3
Field_Strength = 14.09636928[T] (60
X_Acq_Duration = 0.69206016[s]
X_Domain = Carbon13
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.44496109[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Blanking = 2[us]
Clipped = FALSE
Scans = 1024
Total_Scans = 1024
Relaxation_Delay = 1[s]
Recvr_Gain = 26
Temp_Get = 21[dC]
X_90_Width = 11.8[us]
X_Acq_Time = 0.69206016[s]
X_Angle = 30[deg]
X_Atn = 9.6[dB]
X_Pulse = 3.93333333[us]
Irr_Atn_Dec = 27.986[dB]
Irr_Atn_Dec_Calc = 27.986[dB]
Irr_Atn_Dec_Default_Calc = 27.986[dB]
Irr_Atn_Noe = 27.986[dB]
Irr_Dec_Bandwidth_Hz = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm = 12.05794078[ppm]
Irr_Dec_Freq = 600.1723046[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 76[us]
Irr_Pwidth_Default = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Temp1 = 76[us]
Irr_Wurst = FALSE
Decimation_Rate = 0
Experiment_Path = c:\Program Files\J
Initial_Wait = 1[s]
Noe_Time = 1[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time = 1.69206016[s]
N
N
n-nonyl
3ak



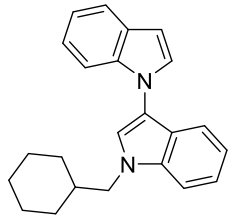
JEOL

```
Filename              = SH-002-037-Fra2_proto
Author                = delta
Experiment            = proton.jxp
Sample_Id             = SH-002-037-Fra2
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 28-AUG-2021 21:06:26
Revision_Time         =  9-FEB-2023 23:06:49

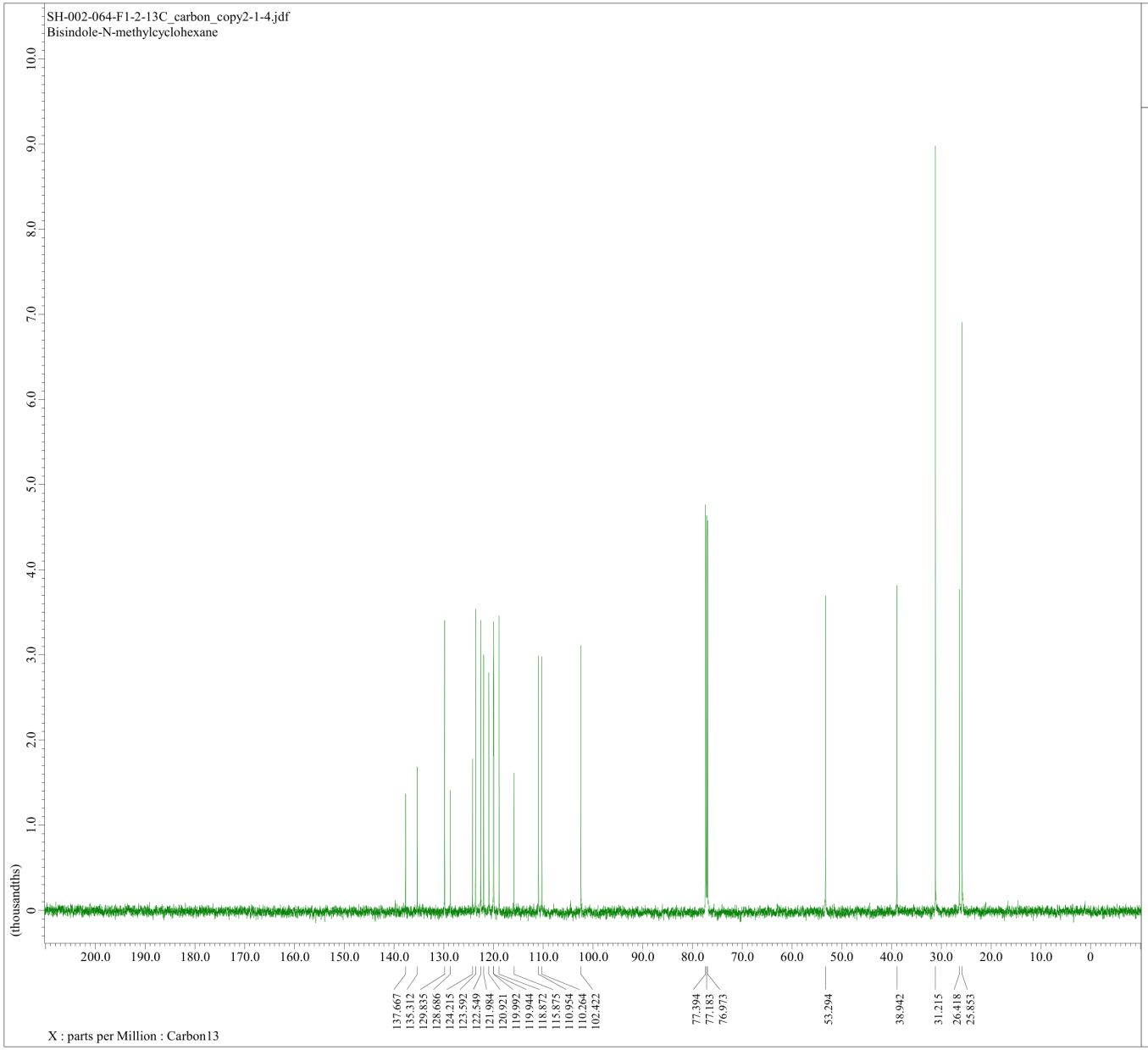
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 16
Total_Scans           = 16

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 20.9[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = (0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```



3al



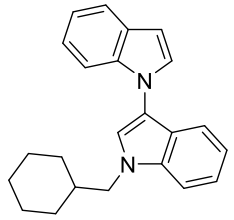
JEOL

```
Filename                = SH-002-064-F1-2-13
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = SH-002-064-F1-2-13
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 14-SEP-2021 12:56:
Revision_Time           =  9-FEB-2023 23:09:

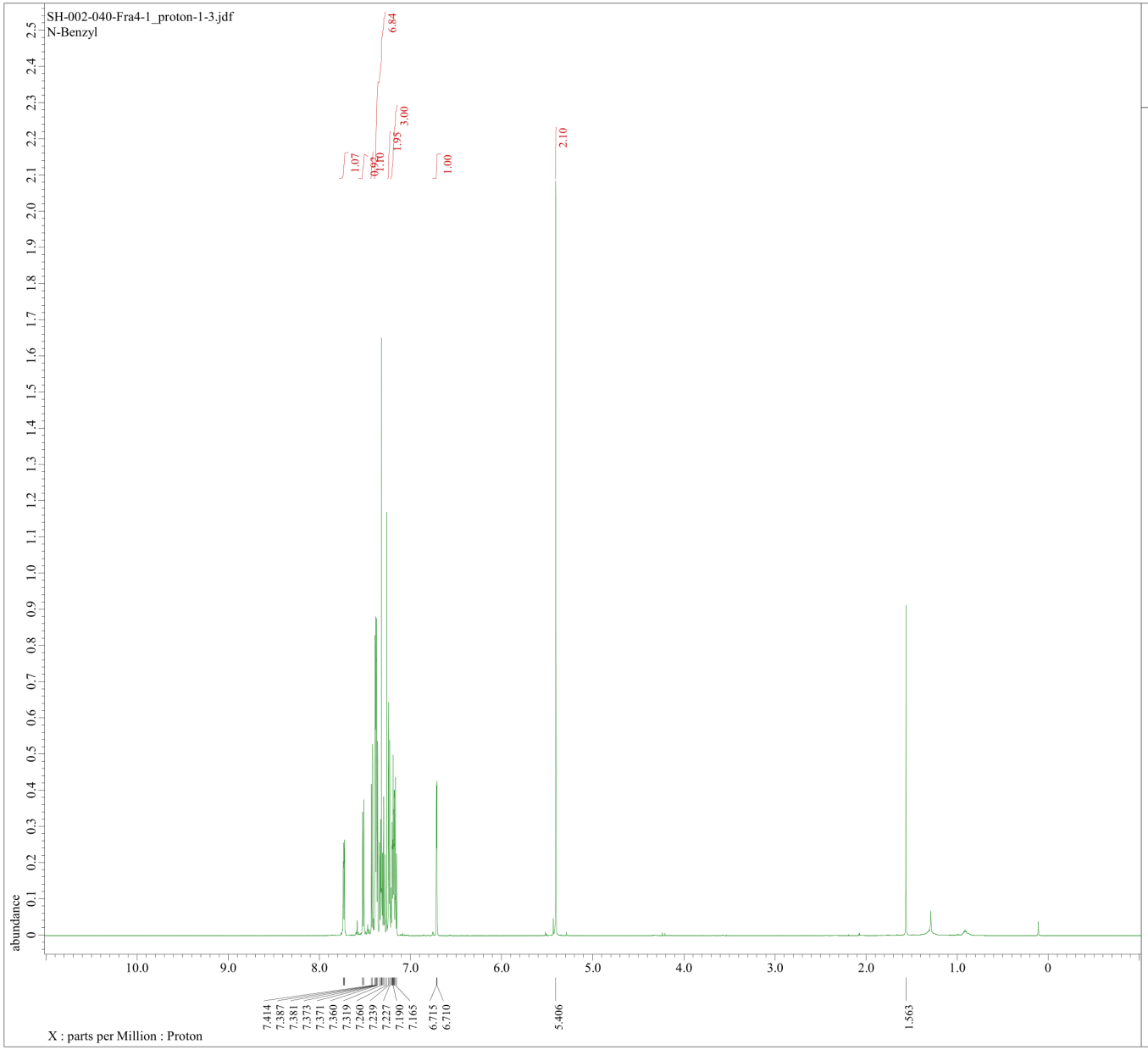
Comment                 = Bisindole-N-methyl
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Incomplete_Copy         = TRUE
Scans                   = 132
Total_Scans             = 132

Relaxation_Delay        = 2[s]
Recvr_Gain              = 36
Temp_Get                = 21.2[dC]
X_90_Width              = 11.8[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 9.6[dB]
X_Pulse                 = 3.93333333[us]
Irr_Atn_Dec             = 27.986[dB]
Irr_Atn_Dec_Calc        = 27.986[dB]
Irr_Atn_Dec_Default_Calc = 27.986[dB]
Irr_Atn_Noe             = 27.986[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Templ        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 2[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 2[s]
Repetition_Time         = 2.69206016[s]
```



3al

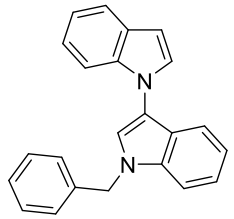



```
Filename              = SH-002-040-Fra4-1_pro
Author                = delta
Experiment            = proton.jxp
Sample_Id             = SH-002-040-Fra4-1
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 25-AUG-2021 21:51:49
Revision_Time         =  9-FEB-2023 22:56:51

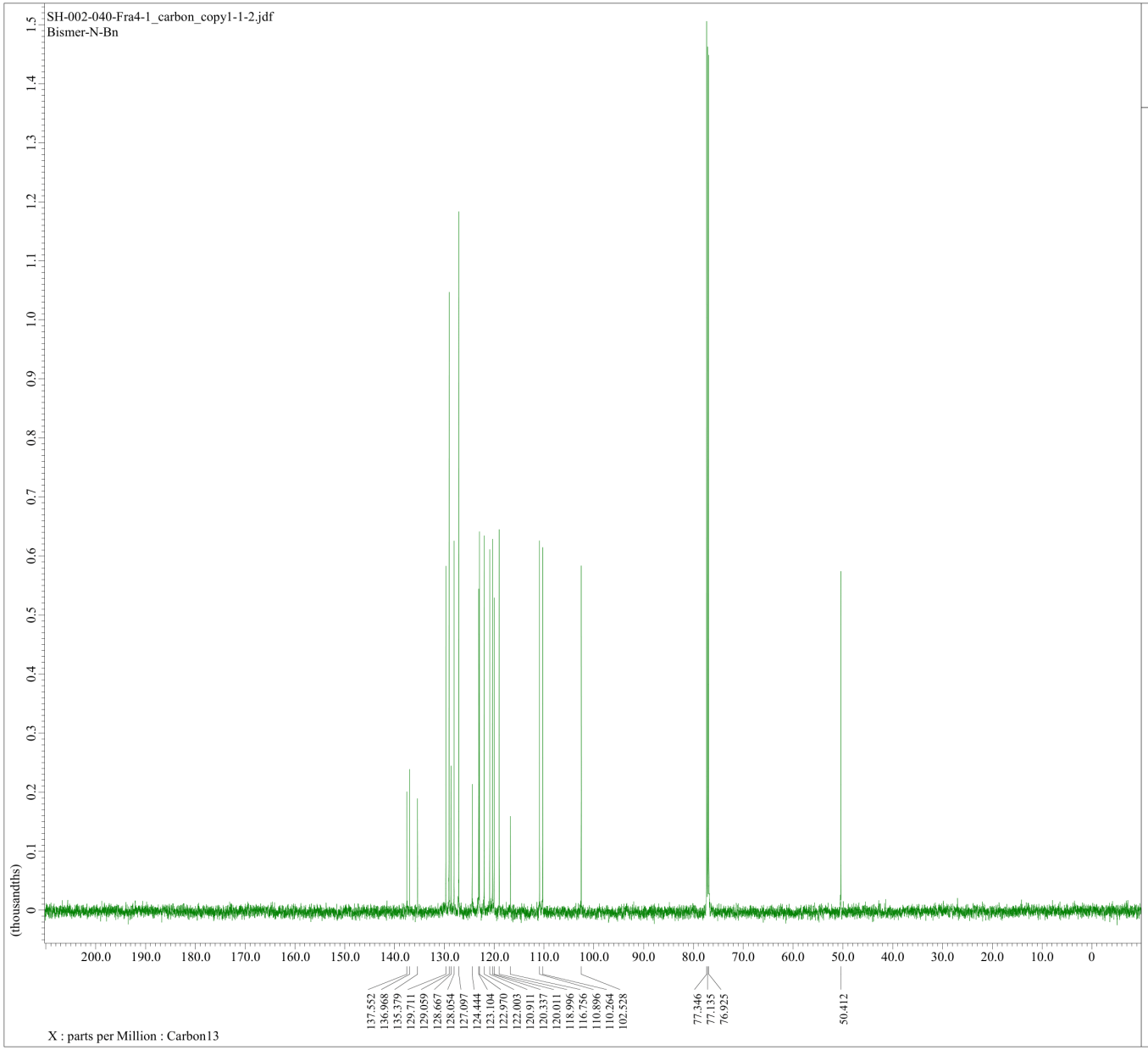
Comment               = N-Benzyl
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 2[us]
Clipped               = FALSE
Scans                 = 16
Total_Scans           = 16

Relaxation_Delay      = 1[s]
Recvr_Gain            = 46
Temp_Get              = 20.8[dC]
X_90_Width            = 7.7[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 3.85[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = {0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```



3am



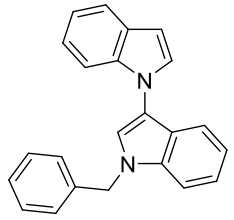
JEOL

```
Filename                = SH-002-040-Fra4-1_
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = SH-002-040-Fra4-1
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 25-AUG-2021 22:16:
Revision_Time           =  9-FEB-2023 22:59:

Comment                 = Bismer-N-Bn
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

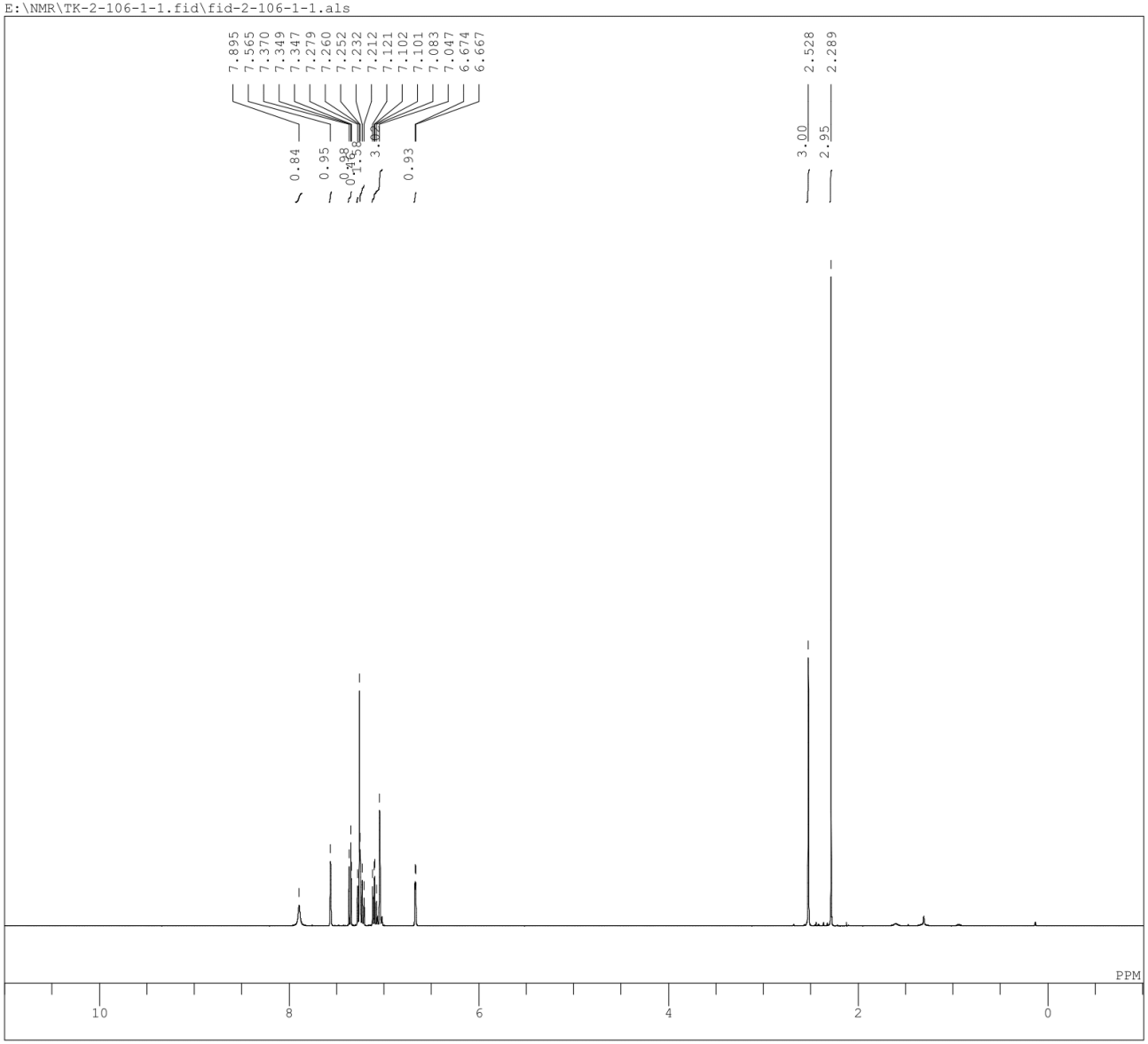
Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 2[us]
Clipped                 = FALSE
Incomplete_Copy         = TRUE
Scans                   = 527
Total_Scans             = 527

Relaxation_Delay        = 1[s]
Recvr_Gain              = 26
Temp_Get                = 21.1[dC]
X_90_Width              = 11.8[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 9.6[dB]
X_Pulse                 = 3.93333333[us]
Irr_Atn_Dec             = 27.986[dB]
Irr_Atn_Dec_Calc        = 27.986[dB]
Irr_Atn_Dec_Default_Calc = 27.986[dB]
Irr_Atn_Noe             = 27.986[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Templ        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 1[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 1.69206016[s]
```

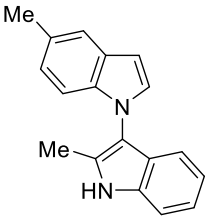


3am

E:\NMR\TK-2-106-1-1.fid\fid-2-106-1-1.als

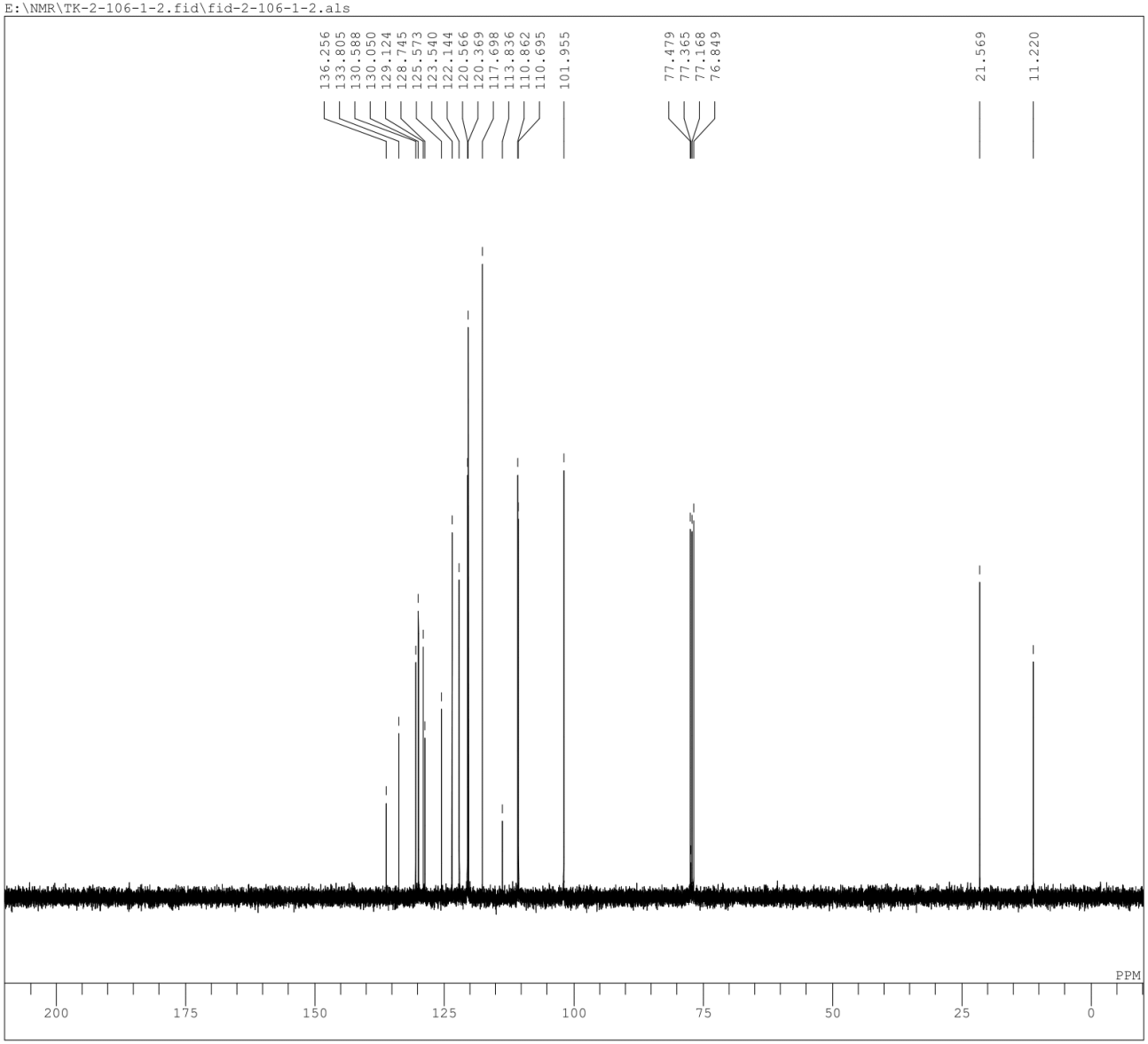



```
DFILE   fid-2-106-1-1.als
COMNT   TK-2-106-1-1(5-methyl-2-methyl-indoledimer)1H
DATIM   2023-02-12 23:02:23
OBNUC   H1
EXMOD   s2pul
OBFRQ         399.91 MHz
OBSET           1.99 KHz
OBFIN           2.00 Hz
POINT          32768
FREQU        6410.26 Hz
SCANS             32
ACQTM          3.5000 sec
PD             1.5000 sec
PW1              7.15 usec
IRNUC
CTEMP            37.0 c
SLVNT   cdcl3
EXREF            7.26 ppm
BF               0.10 Hz
RGAIN              36
```

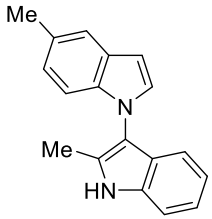


3ba

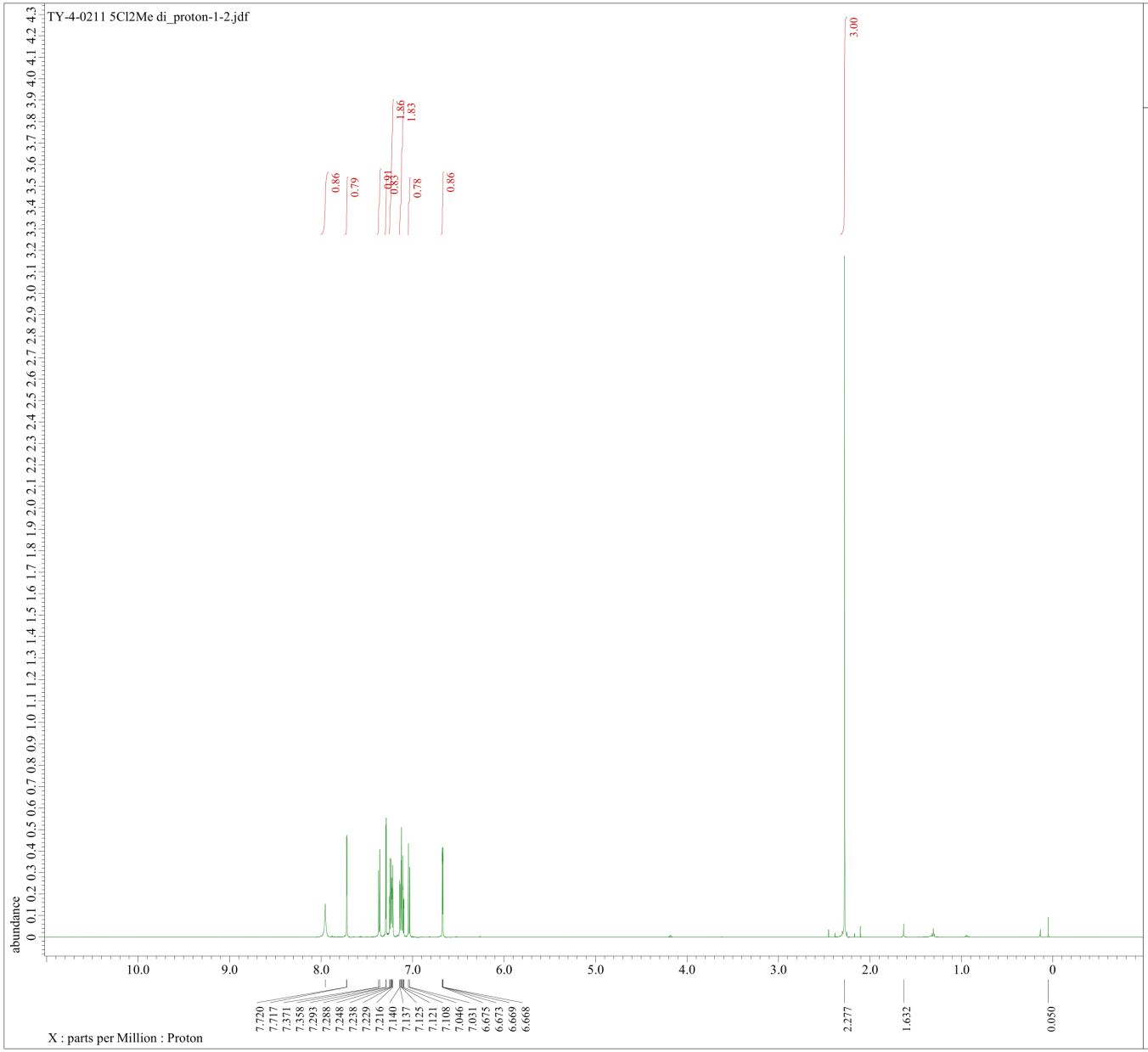
E:\NMR\TK-2-106-1-2.fid\fid-2-106-1-2.als




```
DFILE   fid-2-106-1-2.als
COMNT   TK-2-106-1-2(5-methyl-2-methyl-indoledimer)13
DATIM   2023-02-12 22:49:01
OBNUC   C13
EXMOD   s2pul
OBFRQ         100.56 MHz
OBSET           8.40 KHz
OBFIN           8.30 Hz
POINT          32768
FREQU       25000.00 Hz
SCANS             64
ACQTM          1.3107 sec
PD             1.6893 sec
PW1              5.95 usec
IRNUC
CTEMP           37.0 c
SLVNT   cdcl3
EXREF           77.16 ppm
BF               0.10 Hz
RGAIN              48
```



3ba



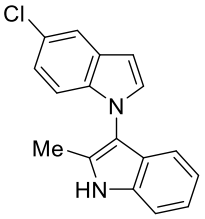
JEOL

```
Filename              = TY-4-0211 5Cl2Me di_p
Author                = delta
Experiment            = proton.jxp
Sample_Id             = TY-4-0211 5Cl2Me di
Solvent               = CHLOROFORM-D
Actual_Start_Time     = 11-FEB-2023 12:09:16
Revision_Time         = 11-FEB-2023 14:28:53

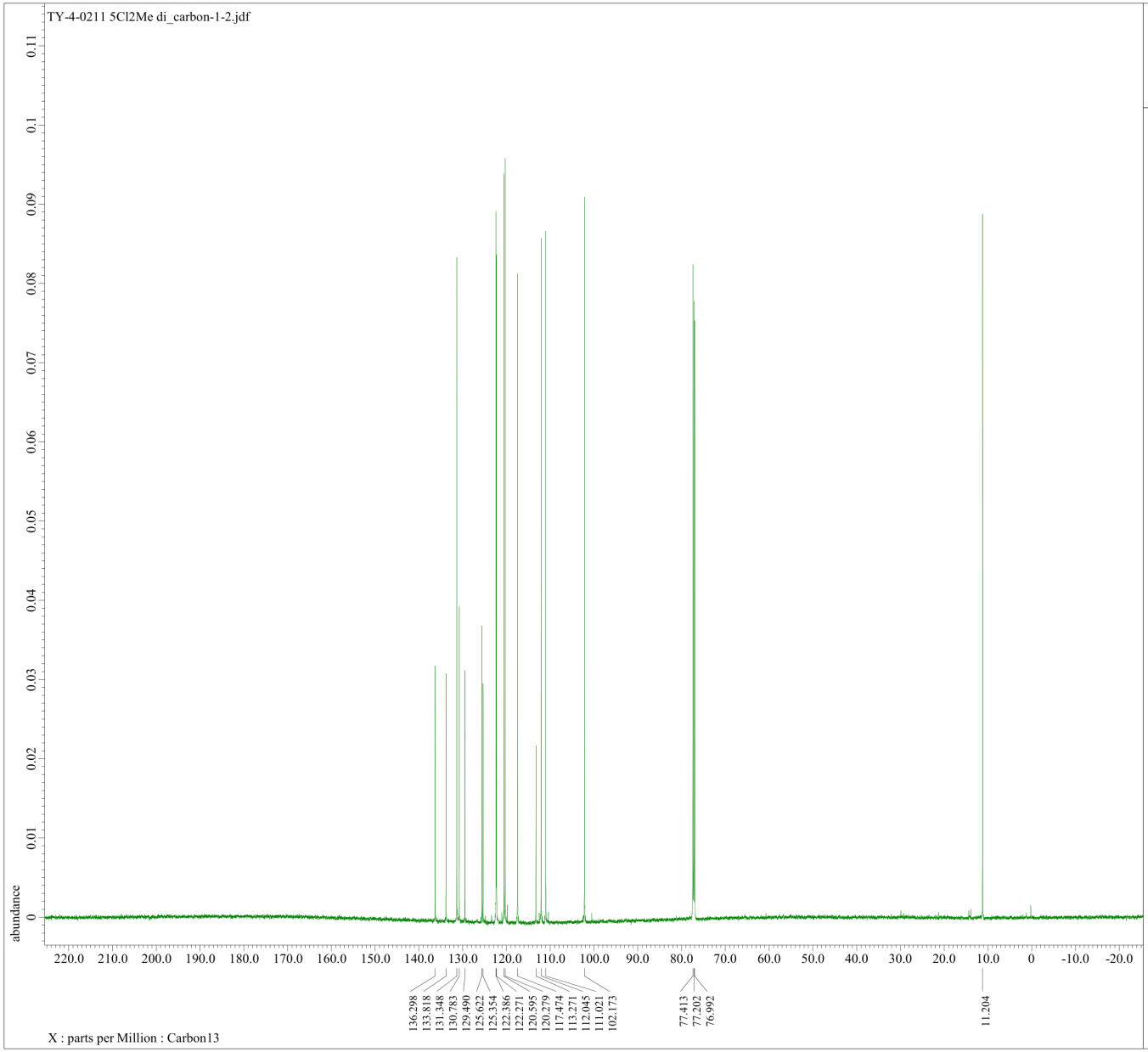
Data_Format           = 1D COMPLEX
Dim_Size              = 26214
X_Domain              = Proton
Dim_Title             = Proton
Dim_Units             = [ppm]
Dimensions            = X
Spectrometer          = JNM-ECZ600R/S3

Field_Strength        = 14.09636928[T] (600[M
X_Acq_Duration        = 2.18103808[s]
X_Domain              = Proton
X_Freq                = 600.1723046[MHz]
X_Offset              = 5[ppm]
X_Points              = 32768
X_Prescans            = 1
X_Resolution          = 0.45849727[Hz]
X_Sweep               = 15.02403846[kHz]
X_Sweep_Clipped       = 12.01923077[kHz]
Irr_Domain            = Proton
Irr_Freq              = 600.1723046[MHz]
Irr_Offset            = 5[ppm]
Tri_Domain            = Proton
Tri_Freq              = 600.1723046[MHz]
Tri_Offset            = 5[ppm]
Blanking              = 5[us]
Clipped               = FALSE
Scans                 = 4
Total_Scans           = 4

Relaxation_Delay      = 1[s]
Recvr_Gain            = 26
Temp_Get              = 20.5[dC]
X_90_Width            = 9.9[us]
X_Acq_Time            = 2.18103808[s]
X_Angle               = 45[deg]
X_Atn                 = 8.1[dB]
X_Pulse               = 4.95[us]
Irr_Mode              = Off
Tri_Mode              = Off
Dante_Loop            = 100
Dante_Presat          = FALSE
Decimation_Rate       = 0
Experiment_Path       = c:\Program Files\JEOL
Initial_Wait          = 1[s]
Phase                 = (0, 90, 270, 180, 180
Presat_Time           = 1[s]
Presat_Time_Flag      = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time       = 3.18103808[s]
```



3ca

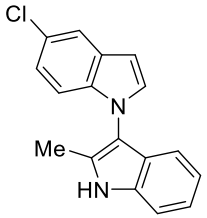



```
Filename                = TY-4-0211 5Cl2Me d
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = TY-4-0211 5Cl2Me d
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-FEB-2023 12:11:
Revision_Time           = 11-FEB-2023 14:26:

Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

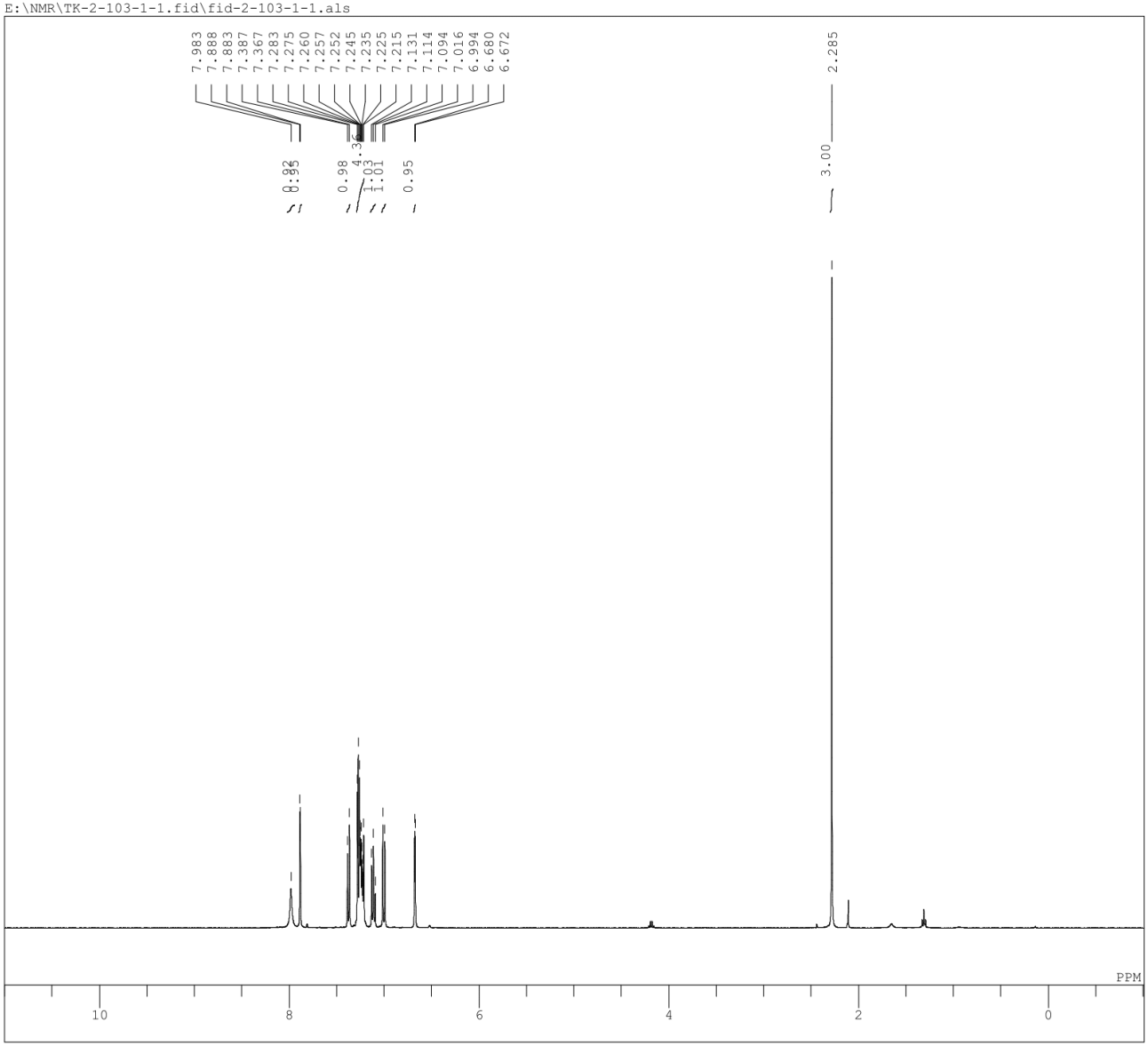
Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 15[us]
Clipped                 = FALSE
Scans                   = 256
Total_Scans             = 256

Relaxation_Delay        = 1[s]
Recvr_Gain              = 56
Temp_Get                = 20.4[dC]
X_90_Width              = 8.1[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 11[dB]
X_Pulse                 = 2.7[us]
Irr_Atn_Dec             = 25.803[dB]
Irr_Atn_Dec_Calc        = 25.803[dB]
Irr_Atn_Dec_Default_Calc = 25.803[dB]
Irr_Atn_Noe             = 25.803[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Templ        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 1[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 1.69206016[s]
```

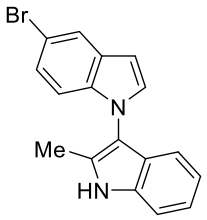


3ca

E:\NMR\TK-2-103-1-1.fid\fid-2-103-1-1.als

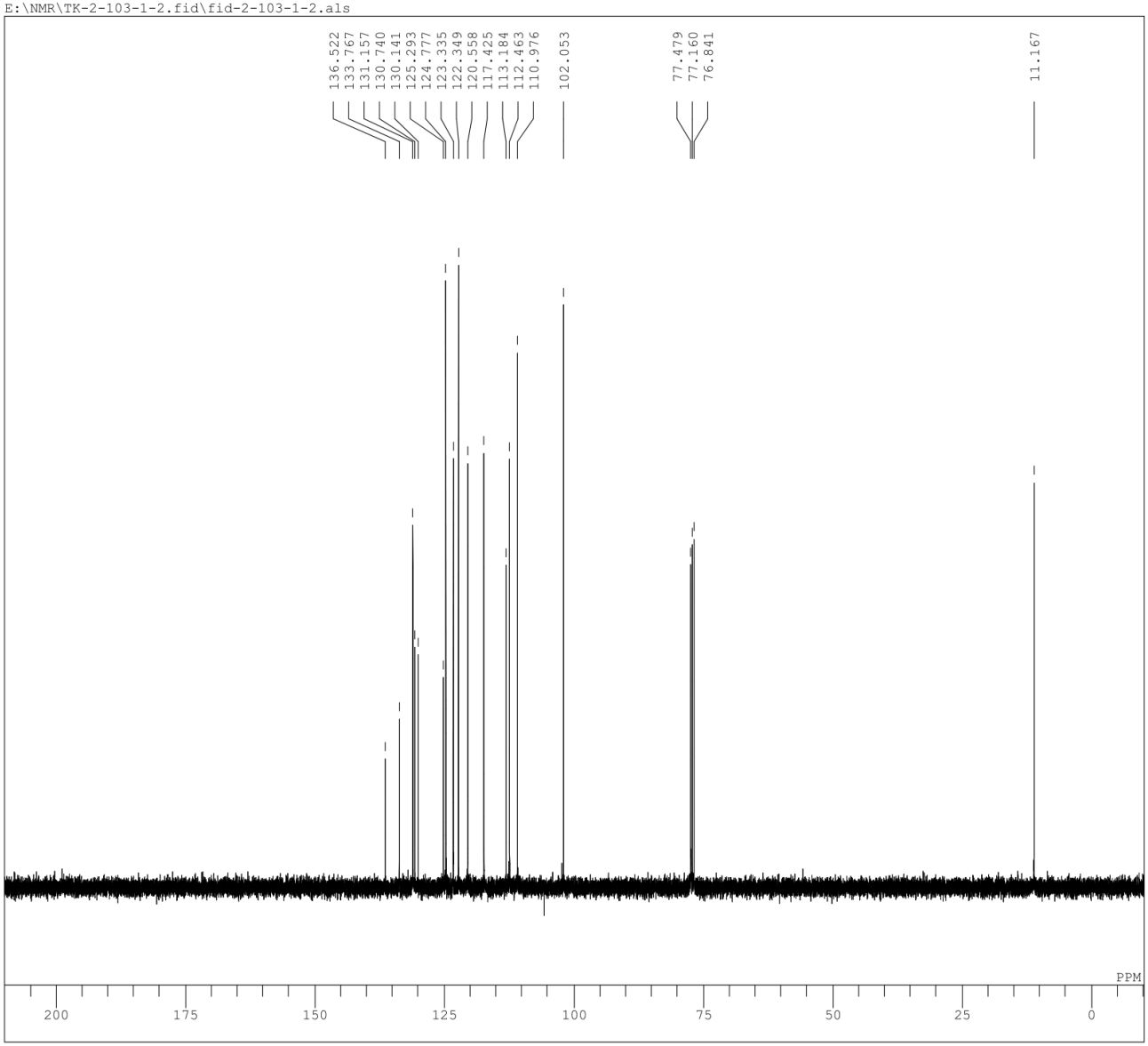



```
DFILE  fid-2-103-1-1.als
COMNT  TK-2-103-1-1(5-Br-2'-Me-indoledimer)1H
DATIM  2023-02-10 22:45:03
OBNUC  H1
EXMOD  s2pul
OBFRQ        399.91 MHz
OBSET          1.99 KHz
OBFIN          2.00 Hz
POINT         32768
FREQU       6410.26 Hz
SCANS            32
ACQTM        3.5000 sec
PD           1.5000 sec
PW1            7.15 usec
IRNUC
CTEMP          37.0 c
SLVNT  cdcl3
EXREF          7.26 ppm
BF             0.10 Hz
RGAIN            34
```

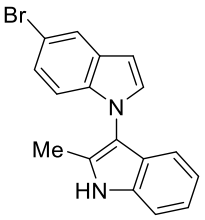


3da

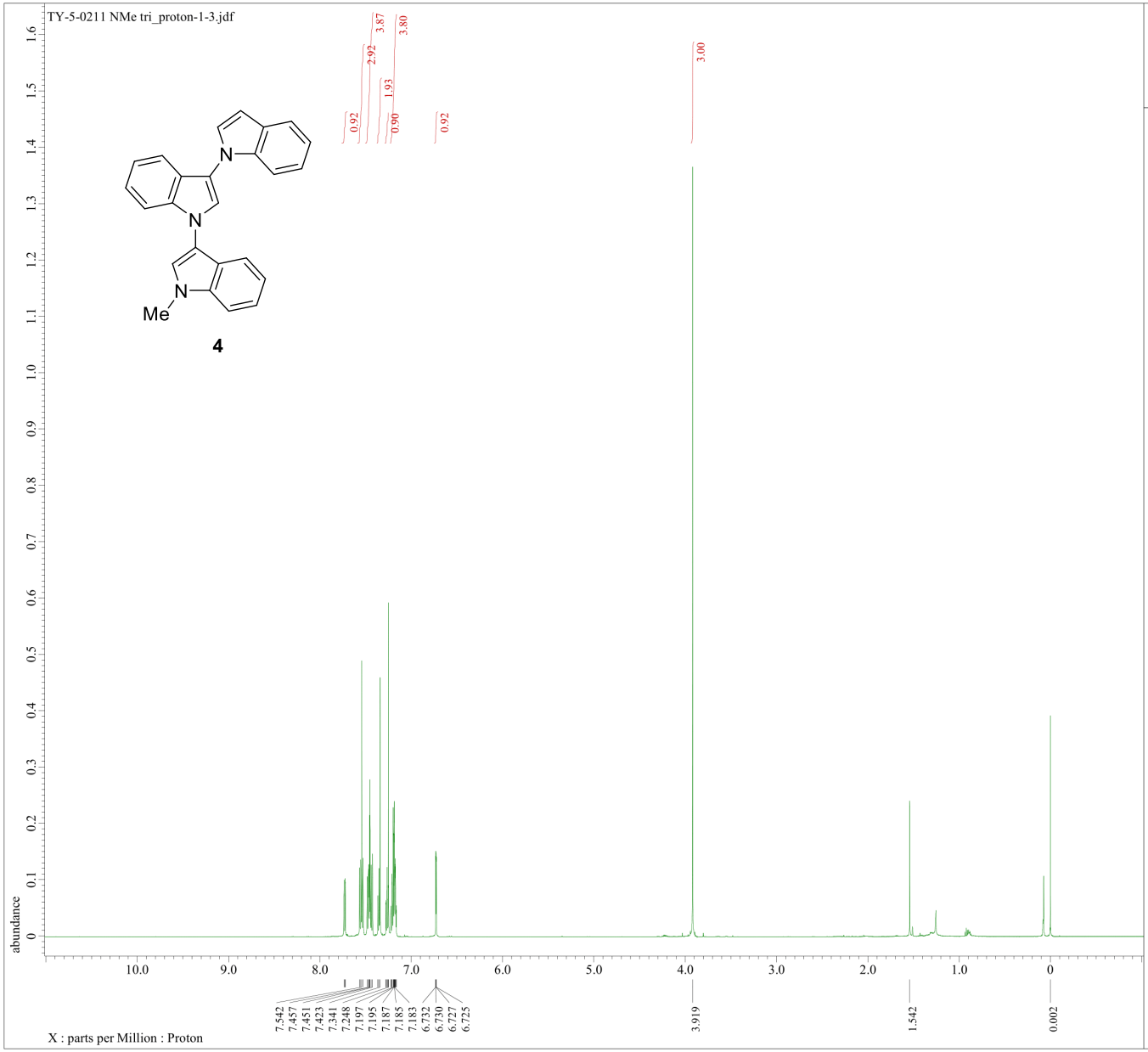
E:\NMR\TK-2-103-1-2.fid\fid-2-103-1-2.als




```
DFILE  fid-2-103-1-2.als
COMNT  TK-2-103-1-1(5-Br-2'-Me-indoledimer)13C
DATIM  2023-02-10 22:29:39
OBNUC  C13
EXMOD  s2pul
OBFRQ        100.56 MHz
OBSET          8.40 KHz
OBFIN          8.30 Hz
POINT         32768
FREQU      25000.00 Hz
SCANS            64
ACQTM         1.3107 sec
PD            1.6893 sec
PW1             5.95 usec
IRNUC
CTEMP           37.0 c
SLVNT  cdcl3
EXREF          77.16 ppm
BF              0.10 Hz
RGAIN             54
```



3da

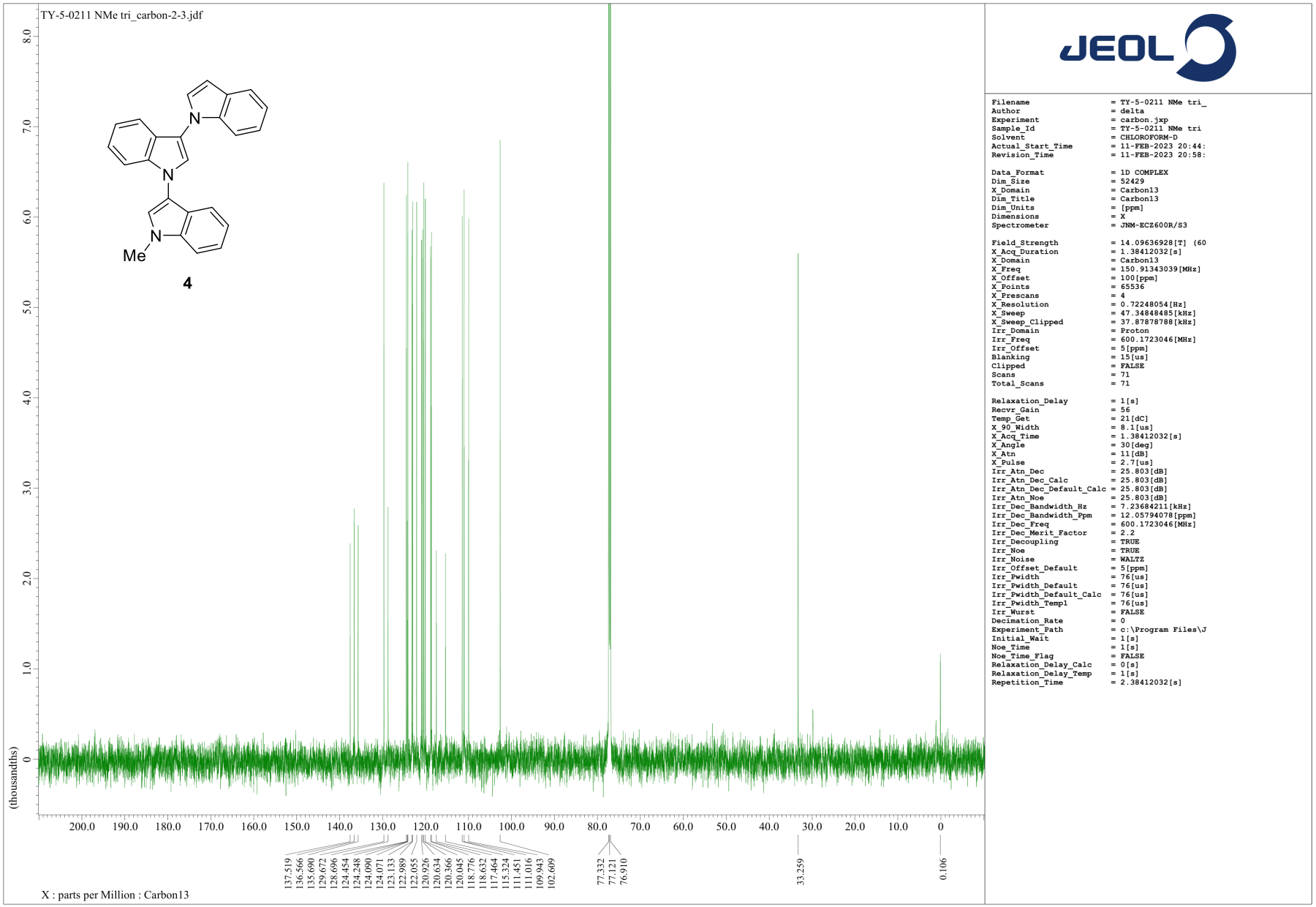


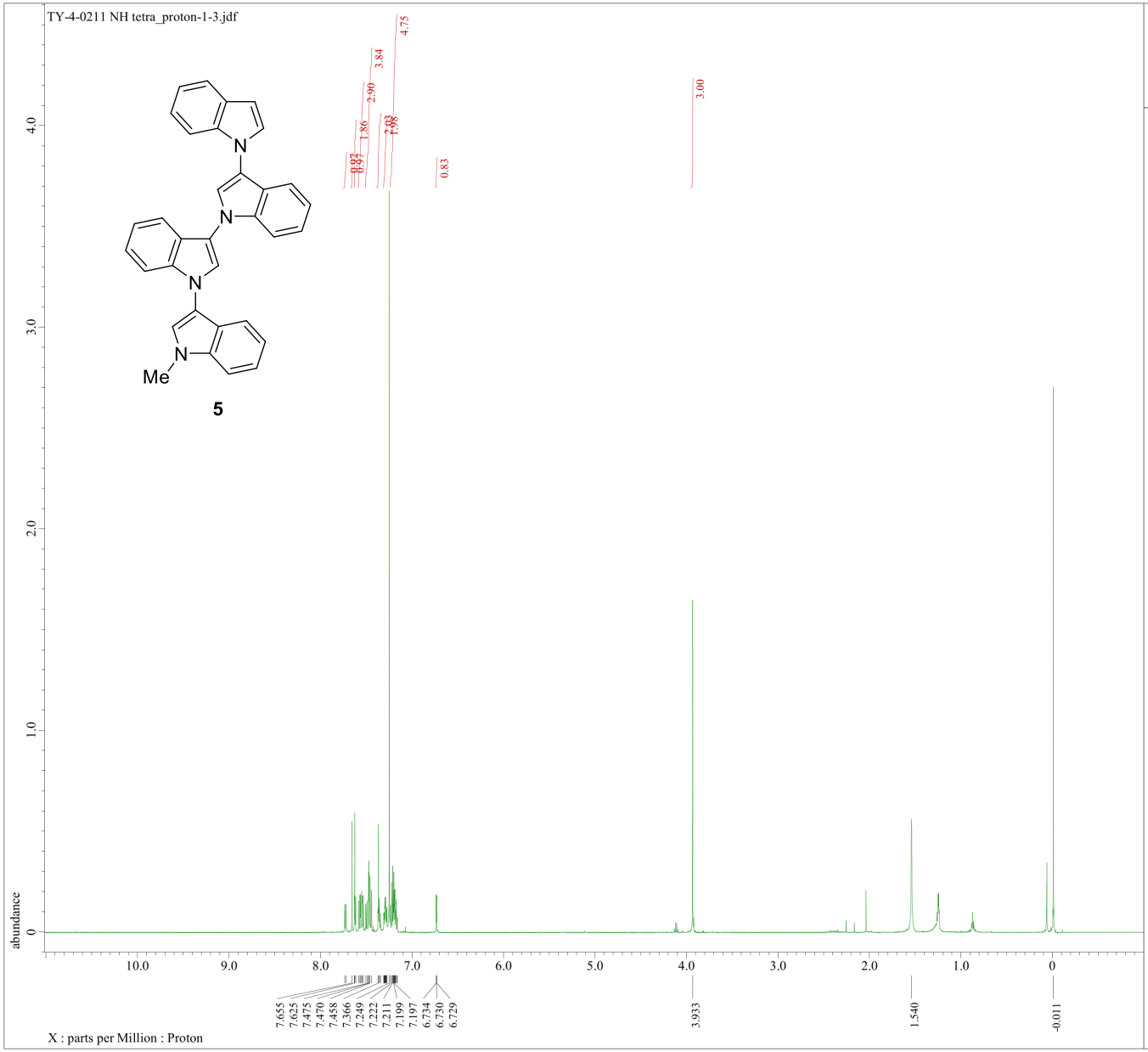

```
Filename                = TY-5-0211 NMe tri_pro
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = TY-5-0211 NMe tri
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-FEB-2023 20:33:26
Revision_Time           = 11-FEB-2023 20:55:49

Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (600[M
X_Acq_Duration          = 2.18103808[s]
X_Domain                = Proton
X_Freq                  = 600.1723046[MHz]
X_Offset                = 5[ppm]
X_Points                = 32768
X_Prescans              = 1
X_Resolution            = 0.45849727[Hz]
X_Sweep                 = 15.02403846[kHz]
X_Sweep_Clipped         = 12.01923077[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 600.1723046[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 5[us]
Clipped                 = FALSE
Scans                   = 4
Total_Scans             = 4

Relaxation_Delay        = 1[s]
Recvr_Gain              = 36
Temp_Get                = 20.8[dC]
X_90_Width              = 9.9[us]
X_Acq_Time              = 2.18103808[s]
X_Angle                 = 45[deg]
X_Atn                   = 8.1[dB]
X_Pulse                 = 4.95[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 100
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = (0, 90, 270, 180, 180
Presat_Time             = 1[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 3.18103808[s]
```


TY-5-0211 NMe tri_carbon-2-3.jdf
N
N
N
Me
4
(thousandths)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
200.0 190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
137.519
136.566
135.690
129.672
128.696
124.454
124.248
124.090
124.071
123.133
122.989
122.055
120.926
120.634
120.366
120.045
118.776
118.632
117.464
115.324
111.451
111.016
109.943
102.609
77.332
77.121
76.910
33.259
0.106
X : parts per Million : Carbon13
JEOL
Filename = TY-5-0211 NMe tri_
Author = delta
Experiment = carbon.jxp
Sample_Id = TY-5-0211 NMe tri
Solvent = CHLOROFORM-D
Actual_Start_Time = 11-FEB-2023 20:44:
Revision_Time = 11-FEB-2023 20:58:
Data_Format = 1D COMPLEX
Dim_Size = 52429
X_Domain = Carbon13
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Spectrometer = JNM-ECZ600R/S3
Field_Strength = 14.09636928[T] (60
X_Acq_Duration = 1.38412032[s]
X_Domain = Carbon13
X_Freq = 150.91343039[MHz]
X_Offset = 100[ppm]
X_Points = 65536
X_Prescans = 4
X_Resolution = 0.72248054[Hz]
X_Sweep = 47.34848485[kHz]
X_Sweep_Clipped = 37.87878788[kHz]
Irr_Domain = Proton
Irr_Freq = 600.1723046[MHz]
Irr_Offset = 5[ppm]
Blanking = 15[us]
Clipped = FALSE
Scans = 71
Total_Scans = 71
Relaxation_Delay = 1[s]
Recvr_Gain = 56
Temp_Get = 21[dC]
X_90_Width = 8.1[us]
X_Acq_Time = 1.38412032[s]
X_Angle = 30[deg]
X_Atn = 11[dB]
X_Pulse = 2.7[us]
Irr_Atn_Dec = 25.803[dB]
Irr_Atn_Dec_Calc = 25.803[dB]
Irr_Atn_Dec_Default_Calc = 25.803[dB]
Irr_Atn_Noe = 25.803[dB]
Irr_Dec_Bandwidth_Hz = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm = 12.05794078[ppm]
Irr_Dec_Freq = 600.1723046[MHz]
Irr_Dec_Merit_Factor = 2.2
Irr_Decoupling = TRUE
Irr_Noe = TRUE
Irr_Noise = WALTZ
Irr_Offset_Default = 5[ppm]
Irr_Pwidth = 76[us]
Irr_Pwidth_Default = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Templ = 76[us]
Irr_Wurst = FALSE
Decimation_Rate = 0
Experiment_Path = c:\Program Files\J
Initial_Wait = 1[s]
Noe_Time = 1[s]
Noe_Time_Flag = FALSE
Relaxation_Delay_Calc = 0[s]
Relaxation_Delay_Temp = 1[s]
Repetition_Time = 2.38412032[s]



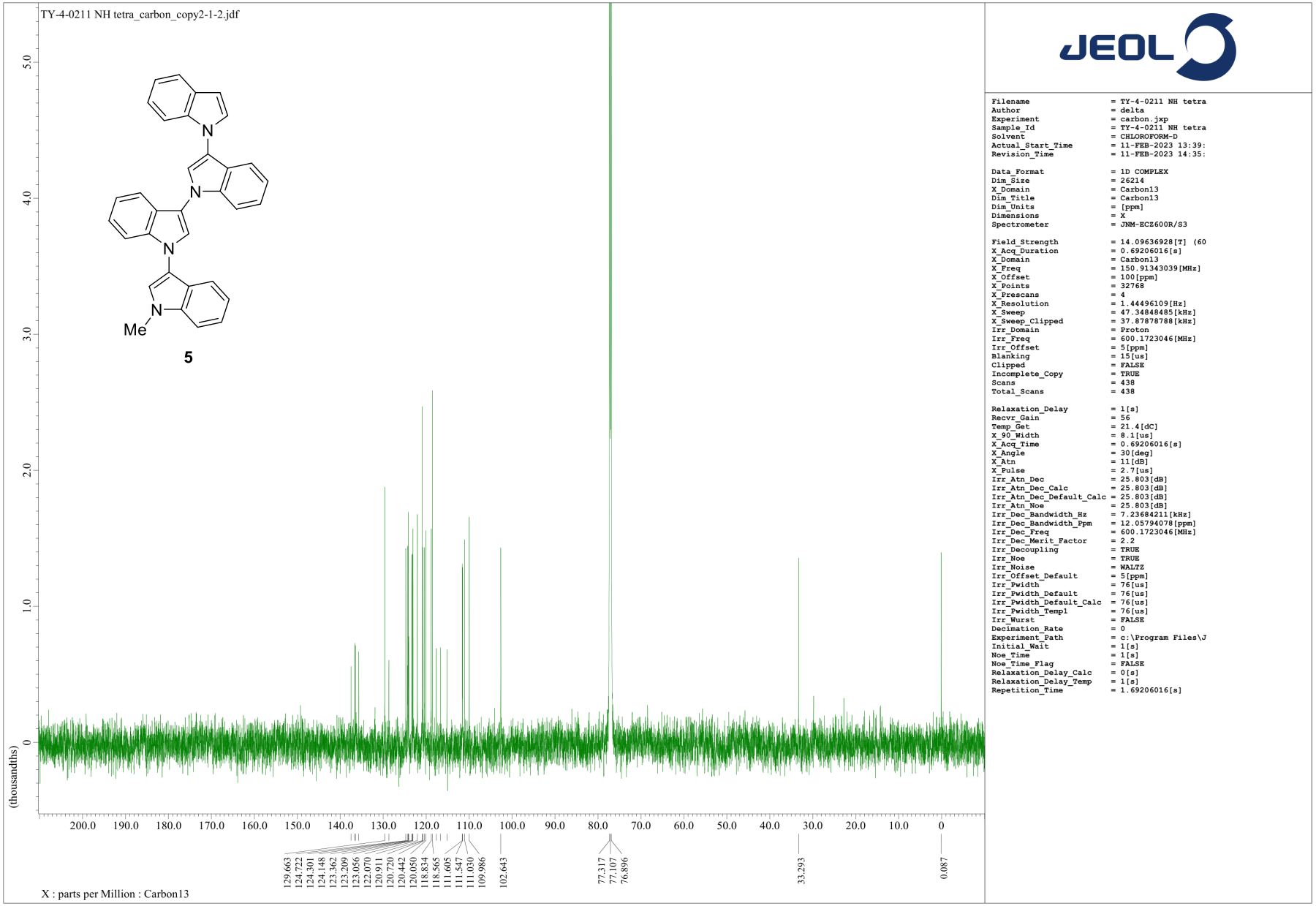
JEOL

```
Filename                = TY-4-0211 NH tetra_pr
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = TY-4-0211 NH tetra
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-FEB-2023 13:37:51
Revision_Time           = 11-FEB-2023 14:41:03

Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (600[M
X_Acq_Duration          = 2.18103808[s]
X_Domain                = Proton
X_Freq                  = 600.1723046[MHz]
X_Offset                = 5[ppm]
X_Points                = 32768
X_Prescans              = 1
X_Resolution            = 0.45849727[Hz]
X_Sweep                 = 15.02403846[kHz]
X_Sweep_Clipped         = 12.01923077[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 600.1723046[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 5[us]
Clipped                 = FALSE
Scans                   = 4
Total_Scans             = 4

Relaxation_Delay        = 1[s]
Recvr_Gain              = 56
Temp_Get                = 21.2[dC]
X_90_Width              = 9.9[us]
X_Acq_Time              = 2.18103808[s]
X_Angle                 = 45[deg]
X_Atn                   = 8.1[dB]
X_Pulse                 = 4.95[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 100
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = (0, 90, 270, 180, 180
Presat_Time             = 1[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 3.18103808[s]
```

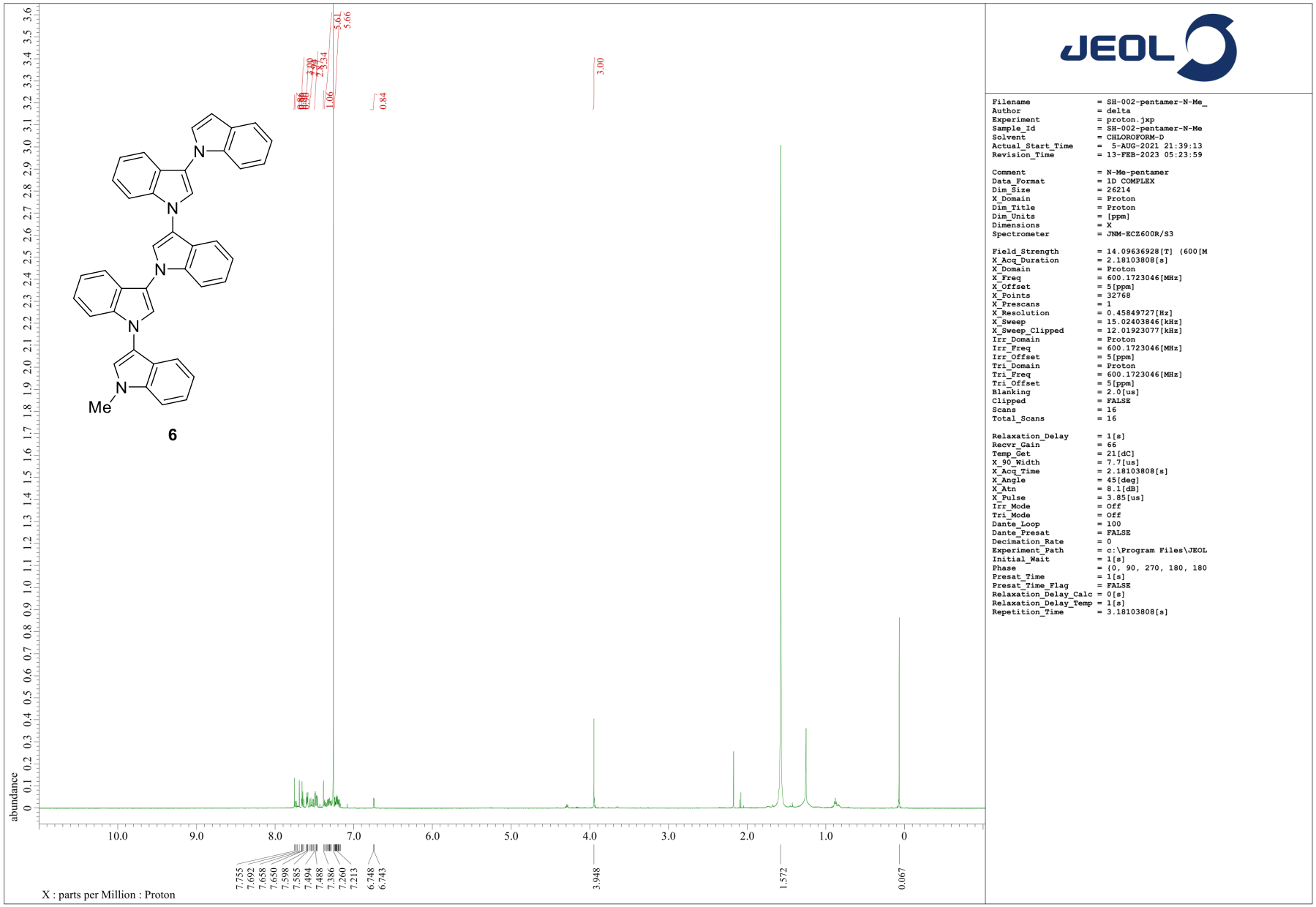



```
Filename                = TY-4-0211 NH tetra
Author                  = delta
Experiment              = carbon.jxp
Sample_Id               = TY-4-0211 NH tetra
Solvent                 = CHLOROFORM-D
Actual_Start_Time       = 11-FEB-2023 13:39:
Revision_Time           = 11-FEB-2023 14:35:

Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Carbon13
Dim_Title               = Carbon13
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (60
X_Acq_Duration          = 0.69206016[s]
X_Domain                = Carbon13
X_Freq                  = 150.91343039[MHz]
X_Offset                = 100[ppm]
X_Points                = 32768
X_Prescans              = 4
X_Resolution            = 1.44496109[Hz]
X_Sweep                 = 47.34848485[kHz]
X_Sweep_Clipped         = 37.87878788[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Blanking                = 15[us]
Clipped                 = FALSE
Incomplete_Copy         = TRUE
Scans                   = 438
Total_Scans             = 438

Relaxation_Delay        = 1[s]
Recvr_Gain              = 56
Temp_Get                = 21.4[dC]
X_90_Width              = 8.1[us]
X_Acq_Time              = 0.69206016[s]
X_Angle                 = 30[deg]
X_Atn                   = 11[dB]
X_Pulse                 = 2.7[us]
Irr_Atn_Dec             = 25.803[dB]
Irr_Atn_Dec_Calc        = 25.803[dB]
Irr_Atn_Dec_Default_Calc = 25.803[dB]
Irr_Atn_Noe             = 25.803[dB]
Irr_Dec_Bandwidth_Hz    = 7.23684211[kHz]
Irr_Dec_Bandwidth_Ppm   = 12.05794078[ppm]
Irr_Dec_Freq            = 600.1723046[MHz]
Irr_Dec_Merit_Factor    = 2.2
Irr_Decoupling          = TRUE
Irr_Noe                 = TRUE
Irr_Noise               = WALTZ
Irr_Offset_Default      = 5[ppm]
Irr_Pwidth              = 76[us]
Irr_Pwidth_Default      = 76[us]
Irr_Pwidth_Default_Calc = 76[us]
Irr_Pwidth_Temp1        = 76[us]
Irr_Wurst               = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\J
Initial_Wait            = 1[s]
Noe_Time                = 1[s]
Noe_Time_Flag           = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 1.69206016[s]
```




```
Filename                = SH-002-pentamer-N-Me_
Author                  = delta
Experiment              = proton.jxp
Sample_Id               = SH-002-pentamer-N-Me
Solvent                 = CHLOROFORM-D
Actual_Start_Time       =  5-AUG-2021 21:39:13
Revision_Time           = 13-FEB-2023 05:23:59

Comment                 = N-Me-pentamer
Data_Format             = 1D COMPLEX
Dim_Size                = 26214
X_Domain                = Proton
Dim_Title               = Proton
Dim_Units               = [ppm]
Dimensions              = X
Spectrometer            = JNM-ECZ600R/S3

Field_Strength          = 14.09636928[T] (600[M
X_Acq_Duration          = 2.18103808[s]
X_Domain                = Proton
X_Freq                  = 600.1723046[MHz]
X_Offset                = 5[ppm]
X_Points                = 32768
X_Prescans              = 1
X_Resolution            = 0.45849727[Hz]
X_Sweep                 = 15.02403846[kHz]
X_Sweep_Clipped         = 12.01923077[kHz]
Irr_Domain              = Proton
Irr_Freq                = 600.1723046[MHz]
Irr_Offset              = 5[ppm]
Tri_Domain              = Proton
Tri_Freq                = 600.1723046[MHz]
Tri_Offset              = 5[ppm]
Blanking                = 2.0[us]
Clipped                 = FALSE
Scans                   = 16
Total_Scans             = 16

Relaxation_Delay        = 1[s]
Recvr_Gain              = 66
Temp_Get                = 21[dC]
X_90_Width              = 7.7[us]
X_Acq_Time              = 2.18103808[s]
X_Angle                 = 45[deg]
X_Atn                   = 8.1[dB]
X_Pulse                 = 3.85[us]
Irr_Mode                = Off
Tri_Mode                = Off
Dante_Loop              = 100
Dante_Presat            = FALSE
Decimation_Rate         = 0
Experiment_Path         = c:\Program Files\JEOL
Initial_Wait            = 1[s]
Phase                   = {0, 90, 270, 180, 180
Presat_Time             = 1[s]
Presat_Time_Flag        = FALSE
Relaxation_Delay_Calc   = 0[s]
Relaxation_Delay_Temp   = 1[s]
Repetition_Time         = 3.18103808[s]
```