Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Palladium-Catalyzed Aerobic Oxidative Carbonylation of Alkynes with Amines: A General Access to Substituted Maleimides

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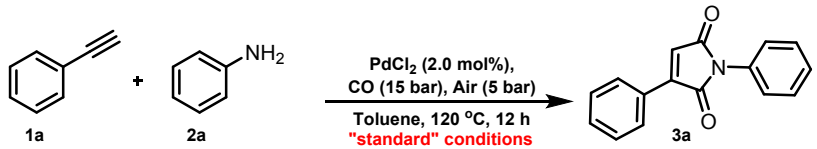
General Considerations

All commercial reagents were ordered from Alfa Aesar, Aldrich, TCI or Strem. Unless otherwise statement, commercial reagents were used without purification. Air- and moisture-sensitive syntheses were performed under argon atmosphere in heating gun vacuum dried glassware. Analytical data of literature known compounds were in accord with reported data. NMR spectra were recorded on Bruker Avance 300 (300 MHz) NMR spectrometers. Multiplets were assigned as s (singlet), d (doublet), t (triplet), dd (doublet of doublet), m (multiplet) and br. s (broad singlet). All measurements were carried out at room temperature unless otherwise stated. Electron impact (EI) mass spectra were recorded on AMD 402 mass spectrometer (70 eV). High resolution mass spectra (HRMS) were recorded on Agilent 6210 Time-of-Flight LC/MS (Agilent) with electrospray ionization (ESI). The data are given as mass units per charge (m/z) and intensities of signals are given in brackets. For GC analyses, HP 6890 chromatograph with a 29 m HP5 column was used.

Experimental sections

Preparation of Polysubstituted Maleimides

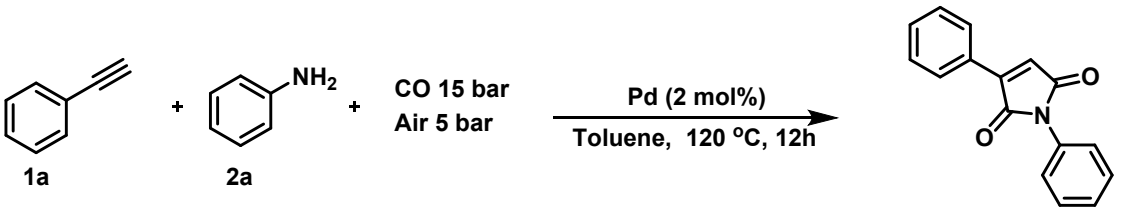


A 4 mL screw-cap vial was charged with Palladium salts (2.0 mol%)), toluene (2.0 mL), amines (1.0 mmol), alkyne (1.5 mmol) and a stirring bar. The vial was closed by PTFE/white rubber septum (Wheaton 13 mm Septa) and phenolic cap and connected with atmosphere with a needle. Then, the vial was fixed in an alloy plate and put into Paar 4560 series autoclave (300 mL). At room temperature, the autoclave is flushed with air (5 bar) and carbon monoxide (15 bar), total pressure to 20 bar.

The reaction was heated under specified temperature (usually 120 °C) for 12 hours. Afterwards, the autoclave was cooled to room temperature and the pressure was carefully released. Isooctane (0.5 mmol) was added into the reaction as internal standard. A sample of the mixture was analyzed by gas chromatography. Pure product could be obtained by column chromatography on silica gel (eluent: pentane/ethyl acetate = 20:1).

Optimization of reaction conditions.

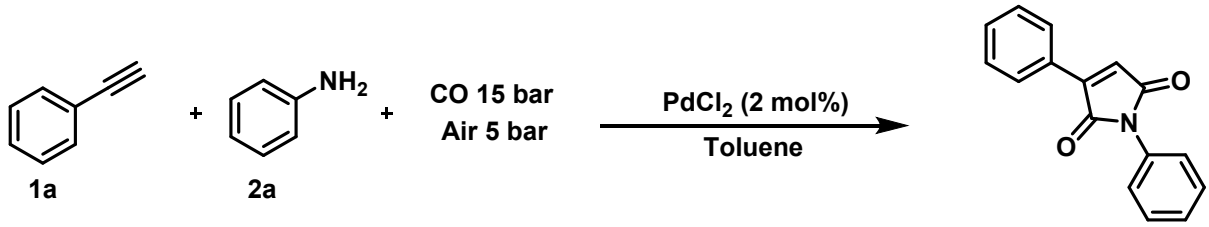
The effect of palladium precursors.



Entry	Add in reaction	Conversions [%]	Yield [%]	Notes
1	$Pd(acac)_2$	>99	trace	Ph—≡—≡—Ph by product yield 95%
2	$Pd(acac)_2$, Xantphos, LiI, H_2O	>99	90	
3	$Pd(acac)_2$, LiI, H_2O	>99	91	
4	$PdCl_2$	>99	92	
5	PdI_2	>99	92	

Reaction conditions:1a (0.75 mmol), 2a (0.5 mmol), Pd (2.0 mol%), ligand (2.0 mol%), CO (15 bar), air (5 bar), toluene (2.0 mL), H_2O (0.1 mL), 120 °C, 12 h. Yields were determined by GC analysis using isooctane as the internal standard.

The effect of temperature.

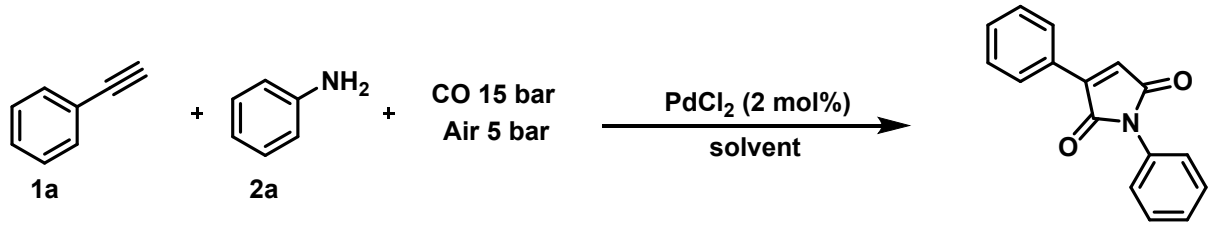


1a 2a

Entry	Temperature [°C]	Conversions [%]	Yield [%]
1	125	>99	92
2	120	>99	92
3	100	>99	77
4	80	77	59
5	60	34	42

Reaction conditions:1a (1.5 mmol), 2a (1.0 mmol), $PdCl_2$ (2 mol%), CO (15 bar), air (5 bar), toluene (2.0 mL), 12 h. Yields were determined by GC analysis using isooctane as the internal standard.

The effect of solvent.



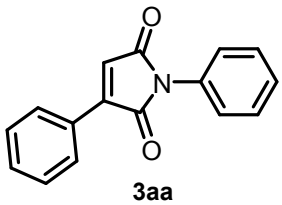
1a 2a

Entry	Solvent	Conversions [%]	Yield [%]
1	THF	>99	81
2	toluene	>99	82
3	Anisole	>99	69
4	MeCN	>99	42
5	Diglyme	>99	78
6	DMF	>99	26
7	1,4-dioxane	>99	77

In order to better investigate the difference between different solvents, we reduced the reaction temperature to 100 ^{o}C.

Reaction conditions:1a (1.5 mmol), 2a (1.0 mmol), $PdCl_2$ (2 mol%), CO (15 bar), air (5 bar), solvent (2.0 mL), 100 ^{o}C, 12 h. Yields were determined by GC analysis using isooctane as the internal standard.

Characterization of products



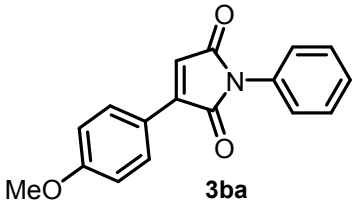
3aa

1,3-diphenyl-1H-Pyrrole-2,5-dione:[1]

229.1 mg, 92% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300MHz , $CDCl_3$) δ 7.89-7.85 (m, 2H), 7.40-7.34 (m, 5H), 7.30-7.25 (m, 3H), 6.75 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.37, 169.10, 143.62, 131.47, 131.31, 129.06, 128.98, 128.76, 128.54, 127.82, 126.18, 124.01.



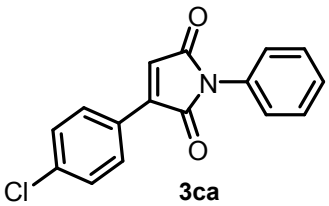
3ba

3-(4-methoxyphenyl)-1-phenyl- 1H-Pyrrole-2,5-dione[2]:

206.5 mg, 74% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz , $CDCl_3$) δ 7.92-7.80 (m, 2H), 7.42-7.32(m, 2H), 7.31-7.24 (m, 3H), 6.90-6.84 (m, 2H), 6.61 (s, 1H), 3.74 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.73, 169.43, 162.14, 143.04, 131.61, 130.58, 128.99, 127.68, 126.19, 121.20, 120.95, 114.51, 77.48, 77.06, 76.64, 55.39.



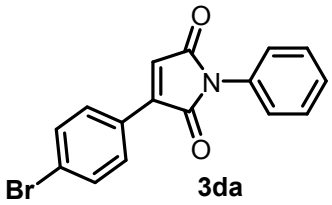
3ca

3-(4-chlorophenyl)-1-phenyl- 1H-Pyrrole-2,5-dione[3]:

240.6 mg, 85% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz , $CDCl_3$) δ 7.84-7.78 (m, 2H), 7.37-7.26 (m, 6H), 6.74 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 169.15, 168.80, 142.34, 137.64, 131.33, 130.00, 129.30, 129.08, 127.91, 126.94, 126.14, 124.13.



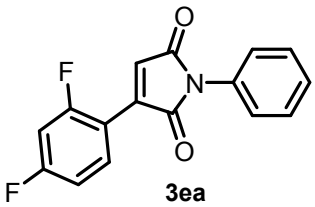
3da

3-(4-bromophenyl)-1-phenyl- 1H-Pyrrole-2,5-dione[4]:

240.6 mg, 83% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz , $CDCl_3$) δ 7.90-7.83 (m, 2H), 7.65-7.59 (m, 2H), 7.52-7.45 (m, 2H), 7.41-7.35 (m, 3H), 6.88 (s, 1H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 169.15, 168.85, 142.56, 132.36, 131.36, 130.20, 129.14, 127.99, 127.41, 126.28, 126.20, 124.25.



3ea

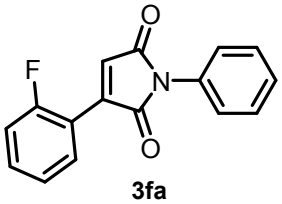
3-(2,4-difluorophenyl)-1-phenyl- 1H-Pyrrole-2,5-dione:

239.4 mg, 84% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz , $CDCl_3$) δ 8.42-8.18 (m, 1H), 7.45-7.35 (m, 2H), 7.32-7.26 (m, 3H), 6.97 (d, J = 2.8 Hz, 1H), 6.95-6.83 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 169.31, 169.19, 166.11, 165.94, 164.56, 164.40, 162.71, 162.55, 161.14, 160.98, 132.72, 132.67, 132.58, 132.54, 131.28, 129.11, 127.98, 127.66, 127.63, 127.47, 127.44, 126.20, 112.28, 112.05, 112.00, 105.28, 104.94, 104.60.

HRMS (ESI) calculated for $C_{16}H_9F_2NO_2(M+H)^+$: 286.0680; found: 286.0668.



3fa

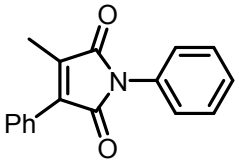
3-(2-fluorophenyl)-1H-Pyrrole-2,5-dione,

232.3 mg, 87% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz , $CDCl_3$) δ 8.20 (td, J = 7.7, 1.7 Hz, 1H), 7.40-7.23 (m, 6H), 7.20-7.05 (m, 2H), 6.99 (d, J = 2.6 Hz, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.29, 162.08 (d, *J* = 255.5 Hz), 136.98, 132.58 (d, J = 9.1 Hz), 131.34, 131.22, 129.06, 128.30 (d, *J* = 14.0 Hz), 127.88, 126.19, 124.57 (d, *J* = 3.7 Hz), 116.91 (d, *J* = 11.0 Hz), 116.17 (d, *J* = 22.0 Hz).

HRMS (ESI) calculated for $C_{16}H_{10}FNO_2$ $(M+H)^+$: 268.0774; found: 268.0758.



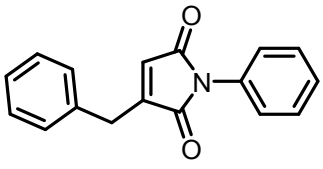
3ga

3-methyl-1,4-diphenyl-1H-Pyrrole-2,5-dione[5]:

168.3 mg, 63% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.60-7.50 (m, 2H), 7.45-7.30 (m, 7H), 7.30-7.20 (m, 1H), 2.20 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.74, 169.74, 137.17, 136.79, 131.85, 129.77, 129.64, 129.03, 128.81, 128.62, 127.60, 125.98, 10.15.



3ha

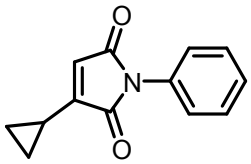
1-phenyl-3-phenylmethyl- 1H-Pyrrole-2,5-dione:

228.8 mg, 87% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz , $CDCl_3$) δ 7.40-7.14 (m, , 10H), 6.16 (t, J = 1.9 Hz, 1H), 3.73 (d, *J* = 1.9 Hz, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.08, 169.34, 149.23, 135.66, 131.54, 129.10, 129.05, 129.00, 127.70, 127.45, 127.30, 125.87, 32.00.

HRMS (ESI) calculated for $C_{17}H1_3NO_2$ $(M+H)^+$: 264.1025; found: 264.1016.



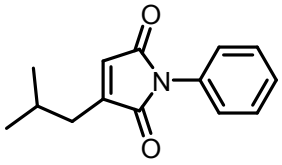
3ia

3-cyclopropyl-1-phenyl-1H-Pyrrole-2,5-dione[6],

193.8 mg, 91% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz , $CDCl_3$) δ 7.40-7.30 (m, 2H), 7.28-7.22(m, 3H), 6.02(s, 1H), 1.90-1.82 (m, 1H), 1.15-1.10 (m,2H), 1.00-0.94 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.64, 169.16, 153.44, 131.54, 128.94, 127.54, 125.92, 120.65, 12.00, 8.38.



3ja

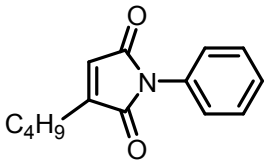
***3-(2-methylpropyl)- 1-phenyl-1H-Pyrrole-2,5-dione*:**

210.7 mg, 92% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz , $CDCl_3$) δ 7.40-7.33 (m, 2H), 7.29-7.20 (m, 3H), 6.34 (t, J = 1.5 Hz, 1H), 2.32 (dd, J = 7.0, 1.5 Hz, 2H), 2.05-1.80 (m, 1H), 0.92 (d, J = 6.7 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.50, 169.65, 148.90, 131.63, 128.98, 127.57, 127.20, 125.84, 34.30, 27.18, 22.43.

HRMS (ESI) calculated for $C_{14}H_{15}NO_2$ $(M+H)^+$: 230.1181; found: 230.1173.



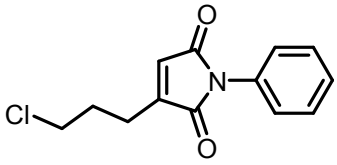
3ka

3-butyl- 1-phenyl-1H-Pyrrole-2,5-dione[7]:

208.4 mg, 91% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz , $CDCl_3$) δ 7.50-7.42 (m,2H), 7.38-7.30 (m, 3H), 6.43 (t, J = 1.8 Hz, 1H), 2.57-2.50 (m, 2H), 1.70-1.55 (m, 2H), 1.50-1.40 (m, 2H), 0.97 (t, J = 7.3 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.49, 169.80, 150.41, 131.67, 129.10, 127.70, 126.37, 125.97, 29.22, 25.27, 22.40, 13.80.



3la

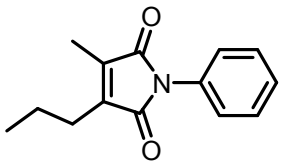
3-(3-chloro-propyl)- 1-phenyl-1H-Pyrrole-2,5-dione:

201.7 mg, 81% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz , $CDCl_3$) δ 7.50-7.40 (m, 2H), 7.40-7.29 (m, 3H), 6.48-6.45 (m, 1H), 3.61 (t, *J* = 6.3 Hz, 2H), 2.73-2.63 (m, 2H), 2.18-2.06 (m, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.02, 169.25, 148.37, 131.46, 129.04, 127.72, 125.85, 77.48, 77.06, 76.64, 43.78, 29.72, 22.99.

HRMS (ESI) calculated for $C_{13}H_{12}ClNO_2$ $(M+H)^+$: 250.0635; found: 250.0626.



3ma

3-methyl- 1-phenyl -4-propyl -1H-Pyrrole-2,5-dione:

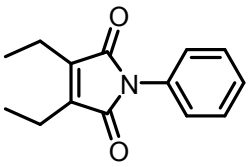
187.8 mg, 82% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.40-7.15(m, 5H), 2.45-2.30 (m, 2H), 1.98 (s, 3H), 1.60-1.50 (m, 2H),

0.91 (t, J = 7.4 Hz, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 171.01, 170.71, 141.10, 137.49, 131.99, 128.96, 127.36, 125.75, 25.74, 21.62, 14.06, 8.92.

HRMS (ESI) calculated for $C_{14}H_{15}NO_2$ $(M+H)^+$: 230.1181; found: 230.1178.



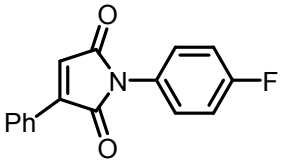
3na

3,4-diethyl-1-phenyl-1H-Pyrrole-2,5-dione[5]:

162.6 mg, 71% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.48-7.40 (m, 2H), 7.40-7.30 (m, 3H), 2.51 (q, J = 7.6 Hz, 4H), 1.21 (t, J = 7.6 Hz, 6H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.71, 141.94, 131.98, 128.97, 127.36, 125.78, 17.18, 13.38.



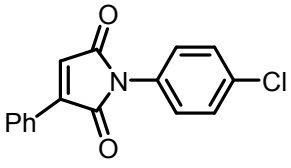
3ab

1-(4-fluorophenyl)-3-phenyl-1H-Pyrrole-2,5-dione[8]:

243.0 mg, 91% yield, , yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00-7.95 (m, 2H), 7.52-7.46 (m,3H), 7.41-7.35 (m, 2H), 7.20-7.13 (m, 2H), 6.87 (s, 1H), 3.05 (t, *J* = 7.0 Hz, 2H)

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.34, 169.03, 161.75 (d, *J* = 247.8 Hz), 143.77, 131.45, 129.05, 128.77, 128.46, 128.04 (d, *J* = 8.7 Hz), 127.39 (d, *J* = 3.2 Hz), 123.95, 116.08 (d, J = 22.9 Hz).



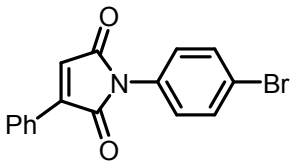
3ac

1-(4-Chlorophenyl)-3-phenyl-1H-Pyrrole-2,5-dione[9]:

237.7 mg, 84% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00-7.93 (m, 2H), 7.52-7.40 (m, 5H), 7.42-7.33 (m, 2H), 6.87 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ169.12, 168.82, 143.88, 133.53, 131.53, 130.03, 129.29, 129.08, 128.81, 128.41, 127.29, 124.02.



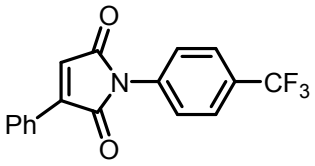
3ad

1-(4-bromophenyl)-3-phenyl-1H-Pyrrole-2,5-dione[9]:

265.7 mg, 81% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00-7.93 (m, 2H), 7.63-7.55 (M, 2H), 7.53-7.45 (m, 3H), 7.35-7.30 (m, 2H), 6.87 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 169.05, 168.74, 143.87, 132.26, 131.54, 130.58, 129.09, 128.82, 128.40, 127.56, 124.04, 121.51.



3ae

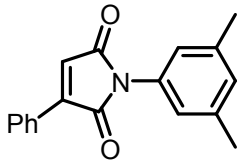
1-(4-(trifluoromethyl)phenyl)-3-phenyl-1H-Pyrrole-2,5-dione:

301.2 mg, 95% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00-7.95 (m, 2H), 7.75 (d, J = 8.4 Hz, 2H), 7.61 (d, J = 8.9 Hz, 2H), 7.55-7.45 (m, 3H), 6.90 (s, 1H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 168.91, 168.56, 144.03, 134.74, 131.67, 129.53 (d, J = 32.9 Hz), 128.99 (d, J = 28.8 Hz), 128.29, 126.21 (q, *J* = 3.7 Hz), 125.93, 124.09, 123.82 (CF_3, d, J = 272.2 Hz), 119.76.

HRMS (ESI) calculated for $C_{17}H_{10}F_3NO_2$ $(M+H)^+$: 318.0742; found: 318.0738.



3af

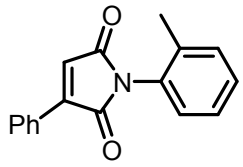
1-(3,5-dimethylphenyl)-3-phenyl-1H-Pyrrole-2,5-dione:

252.1 mg, 91% yield , yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00-7.95 (m, 2H), 7.52-7.45 (m, 3H), 7.02 (d, J = 11.0 Hz, 1H), 6.85 (s, 1H), 2.38 (s, 6H).

^{13}C NMR (100 MHz, $CDCl_3$) 169.65, 169.38, 143.61, 138.91, 131.31, 131.21, 129.89, 129.02, 128.81, 128.69, 124.21, 124.06, 21.34.

HRMS (ESI) calculated for $C_{18}H_{15}NO_2$ $(M+H)^+$: 278.1181; found: 278.1175.



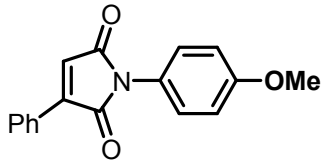
3ag

1-(2-methylphenyl)-3-phenyl-1H-Pyrrole-2,5-dione:

205.1 mg, 78% yield , yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.90-7.86 (m, 2H), 7.42-7.34 (m, 4H), 7.25-7.18 (m, 3H), 7.08-7.05 (d, 1H), 6.78 (s, 1H), 2.10 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) 169.48, 169.24, 143.82, 136.53, 132.59, 131.30, 131.10, 130.32, 129.30, 129.23, 128.97, 128.73, 126.81, 124.06, 17.97.



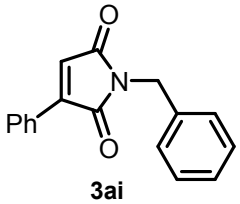
3ah

1-(4-methoxyphenyl)-3-phenyl-1H-Pyrrole-2,5-dione:

198.1 mg, 71% yield , yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93-7.87 (m, 2H), 7.47-7.37 (m, 3H), 7.23-7.16 (m, 2H), 6.95-6.90 (M, 2H), 6.79 (s, 1H), 3.77 (s, 3H).

^{13}C NMR (100 MHz, $CDCl_3$) 169.75, 169.54, 159.15, 143.75, 131.34, 129.05, 128.80, 128.69, 127.76, 124.12, 124.01, 114.51, 55.55.



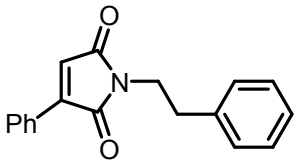
3ai

3-phenyl-1-(phenylmethyl)-1H-Pyrrole-2,5-dione[10]:

239.3 mg, 91% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85-7.78 (m, 2H), 7.40-7.33 (m, 5H), 7.33-7.15 (m, 3H), 6.63 (s, 1H), 4.64 (s, 2H).

^{13}C NMR (100 MHz, $CDCl_3$) δ 170.34, 169.98, 143.79, 136.37, 131.13, 128.91, 128.70, 128.68, 128.60, 128.48, 127.82, 123.87, 41.57.



3aj

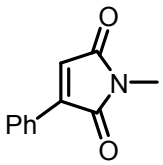
3-phenyl-1-(2-phenylethyl)- 1H-Pyrrole-2,5-dione:

241.0 mg, 87% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.85-7.75 (m, 2H), 7.40-7.34 (m, 3H), 7.24-7.18 (m, 2H), 7.17-7.12 (m, 3H), 6.60 (s, 1H), 3.80-3.70 (m, 2H), 2.90-2.83 (m, 2H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.45, 170.16, 143.68, 138.00, 131.09, 128.93, 128.86, 128.76, 128.59, 128.57, 126.65, 123.84, 39.29, 34.62.

HRMS (ESI) calculated for $C_{18}H_{15}NO_2$ $(M+H)^+$: 278.1181; found: 278.1176.



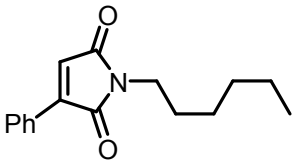
3ak

1-methyl- 3-phenyl-1H-Pyrrole-2,5-dione[10]:

142.1 mg, 76% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 7.95-7.90 (m, 2H), 7.50-7.40 (m, 3H), 6.73 (s, 1H), 3.07 (s, 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.75, 170.45, 143.94, 131.10, 128.94, 128.76, 128.57, 123.91, 23.88.



3al

1-pentyl- 3-phenyl-1H-Pyrrole-2,5-dione:

215.9 mg, 84% yield, yellow solid, 1 mmol scale substrate.

^{1}H NMR (300 MHz, $CDCl_3$) δ 8.00-7.90 (m, 2H), 7.48-7.44 (m, 3H), 6.71 (s, 1H), 3.60-3.54 (m, 2H), 1.64-1.60 (m, 2H), 1.29-1.33 (m, 6H), 0.85-0.92 (m , 3H).

^{13}C NMR (75 MHz, $CDCl_3$) δ 170.76, 170.51, 143.63, 132.51, 131.04, 128.93, 128.60, 123.87, 38.10, 31.37, 28.57, 26.49, 22.54, 14.04.

References

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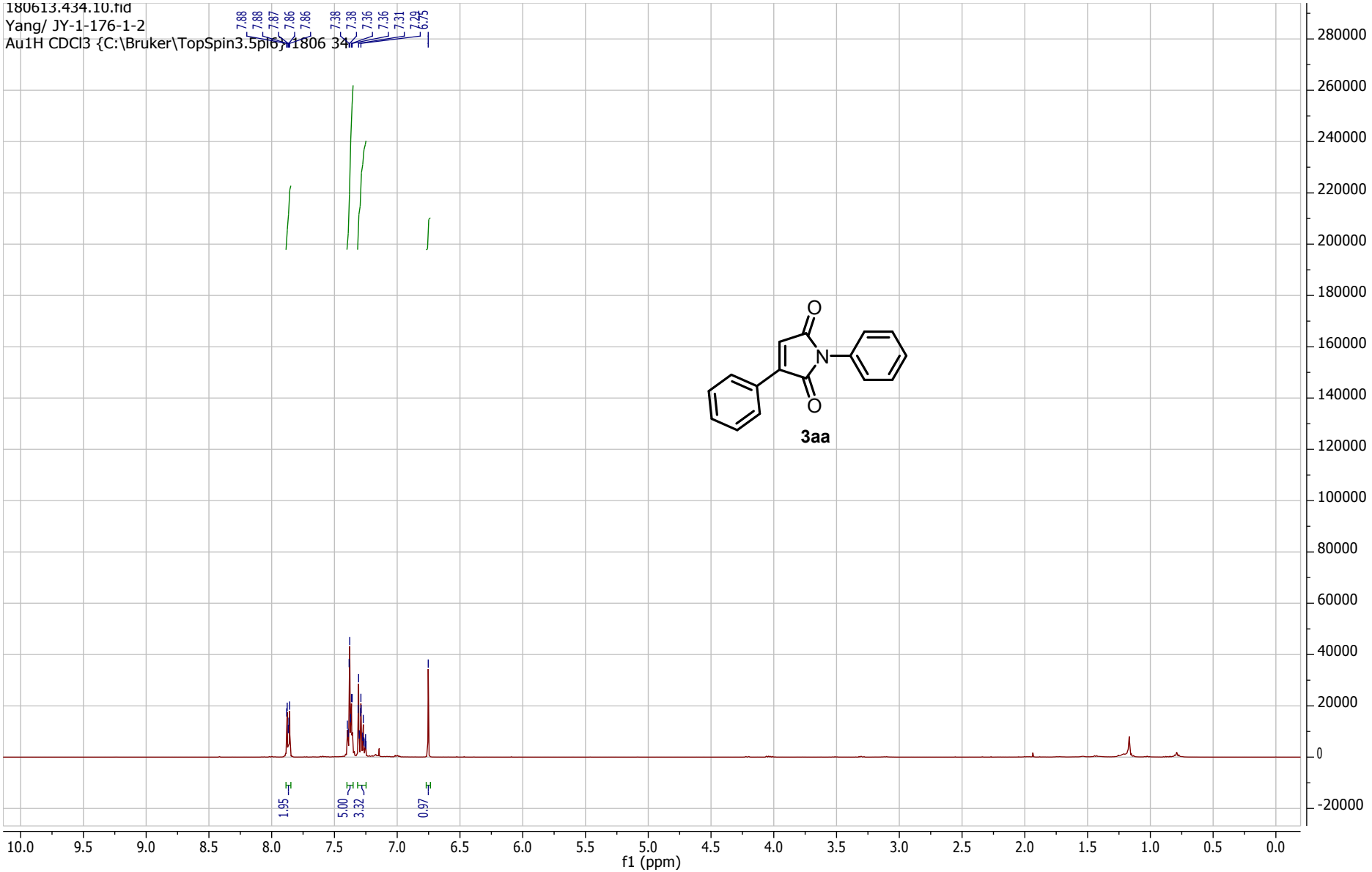
NMR spectra of products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7.38
7.36
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7.29
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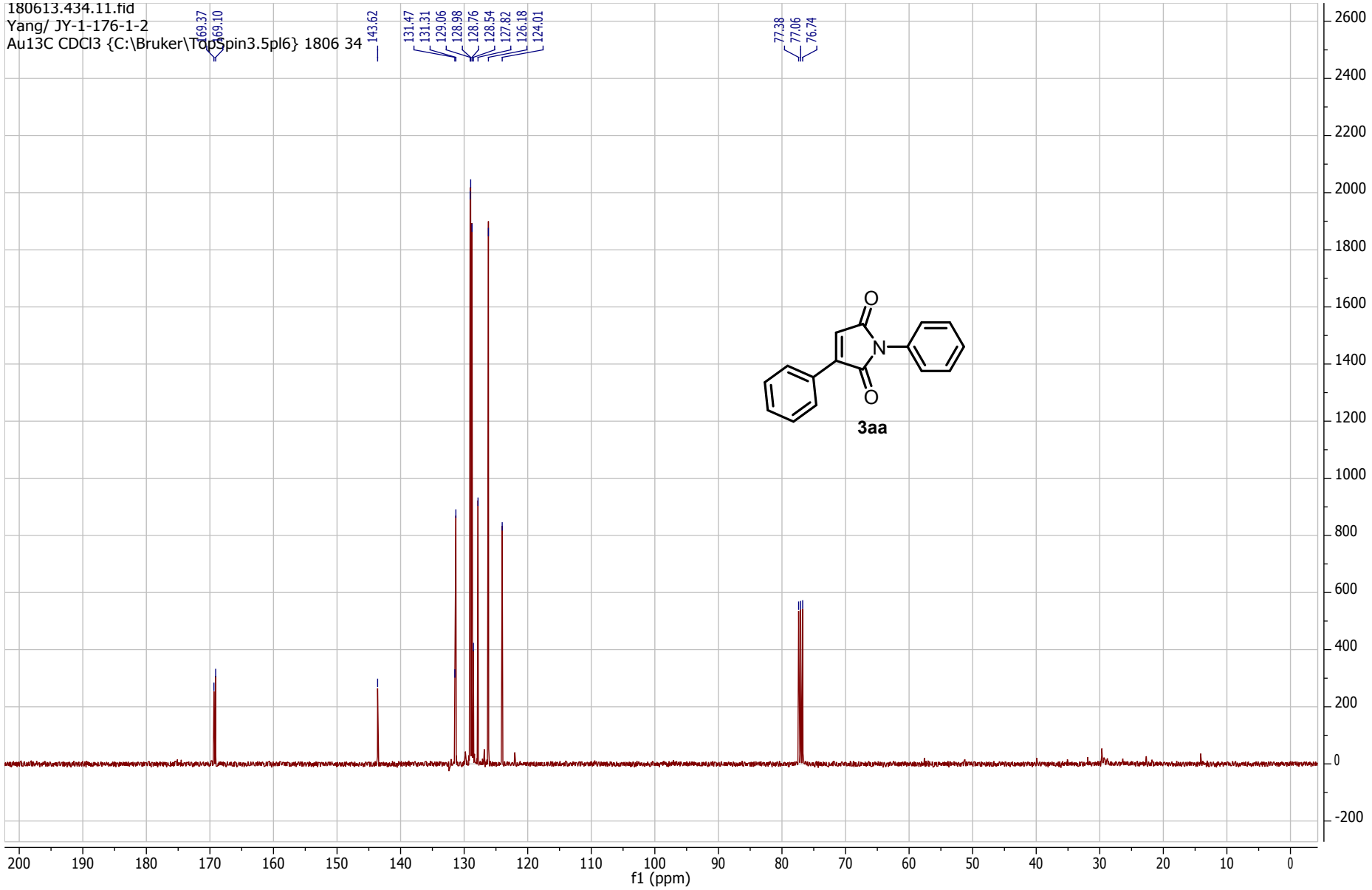
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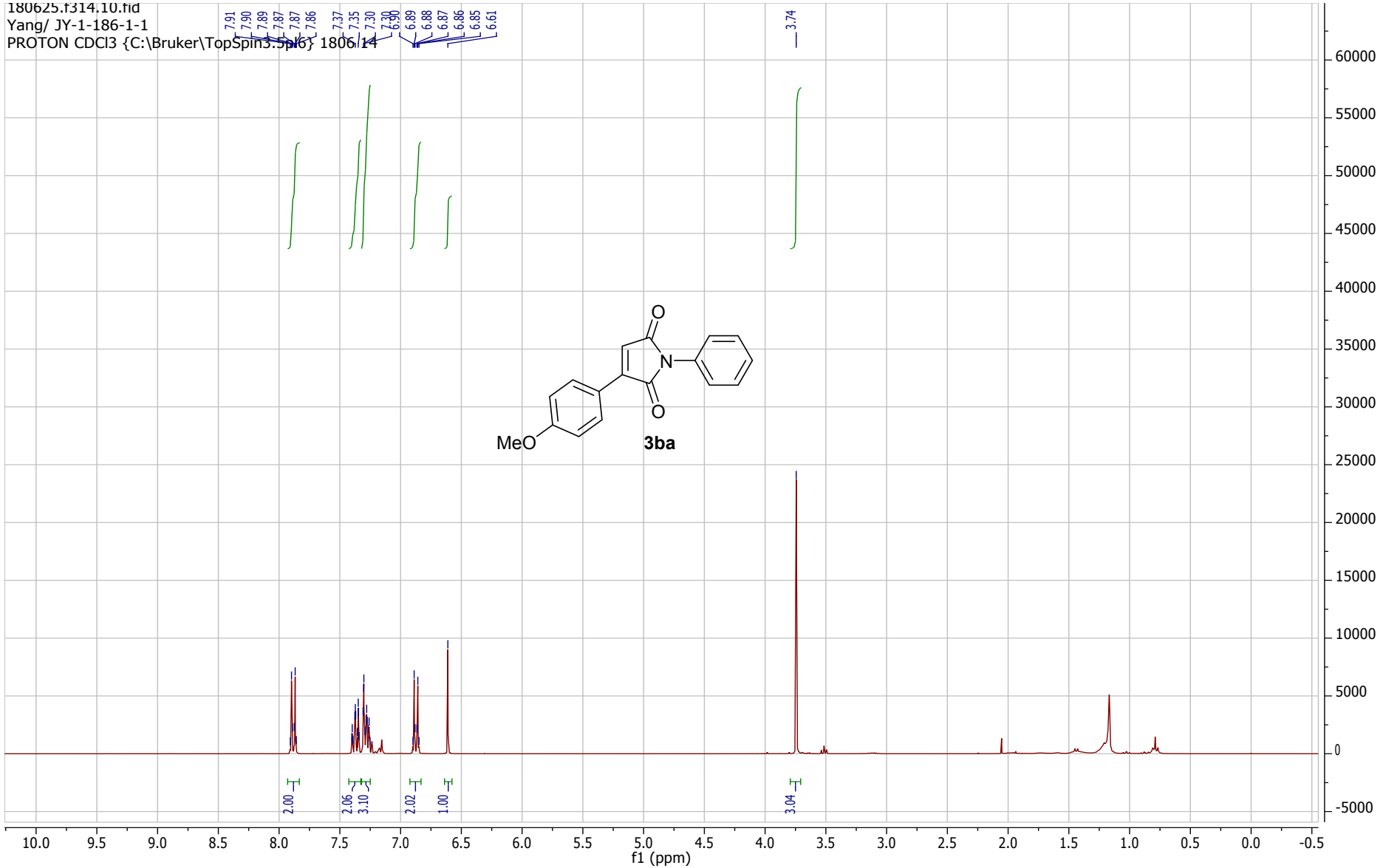
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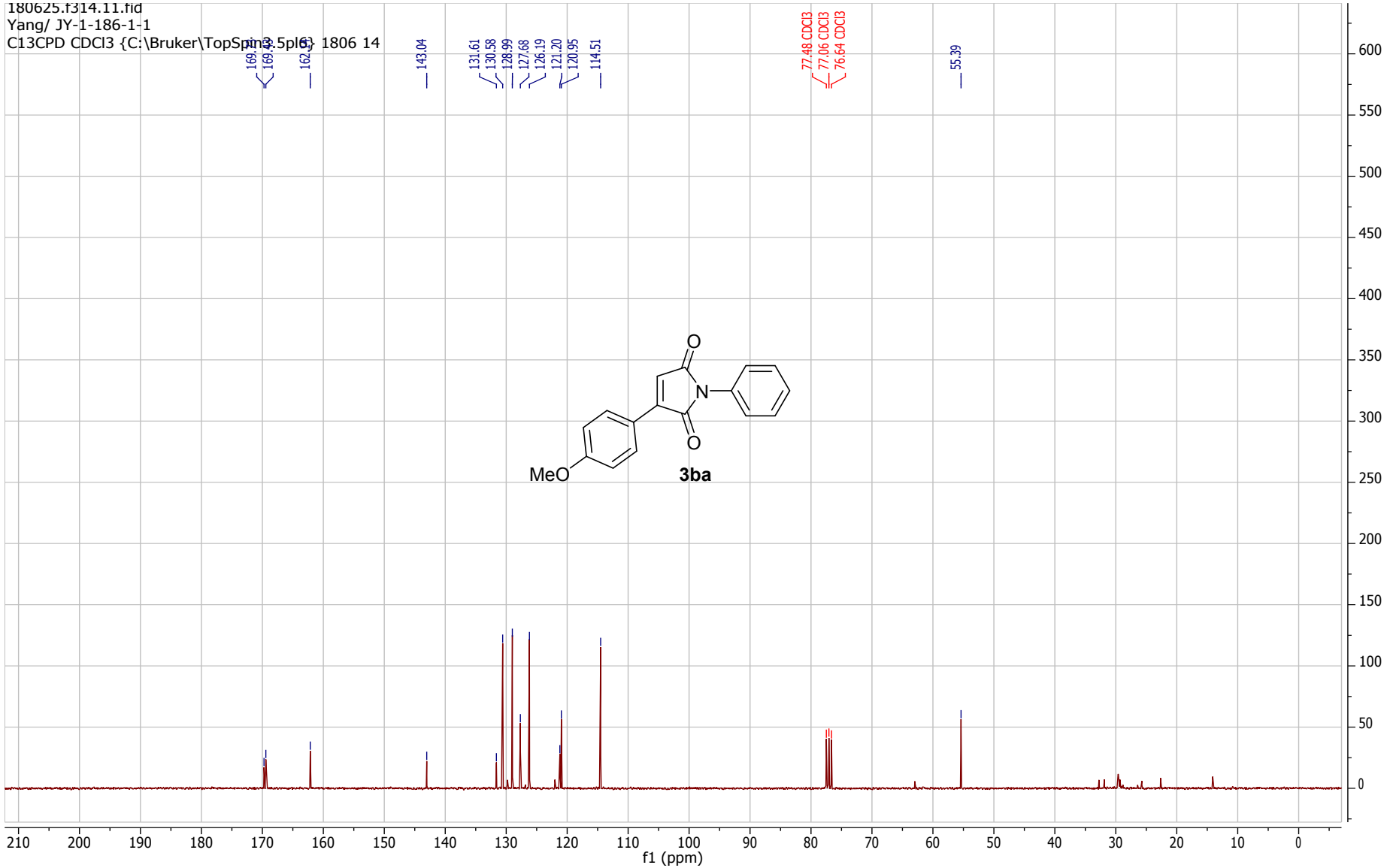
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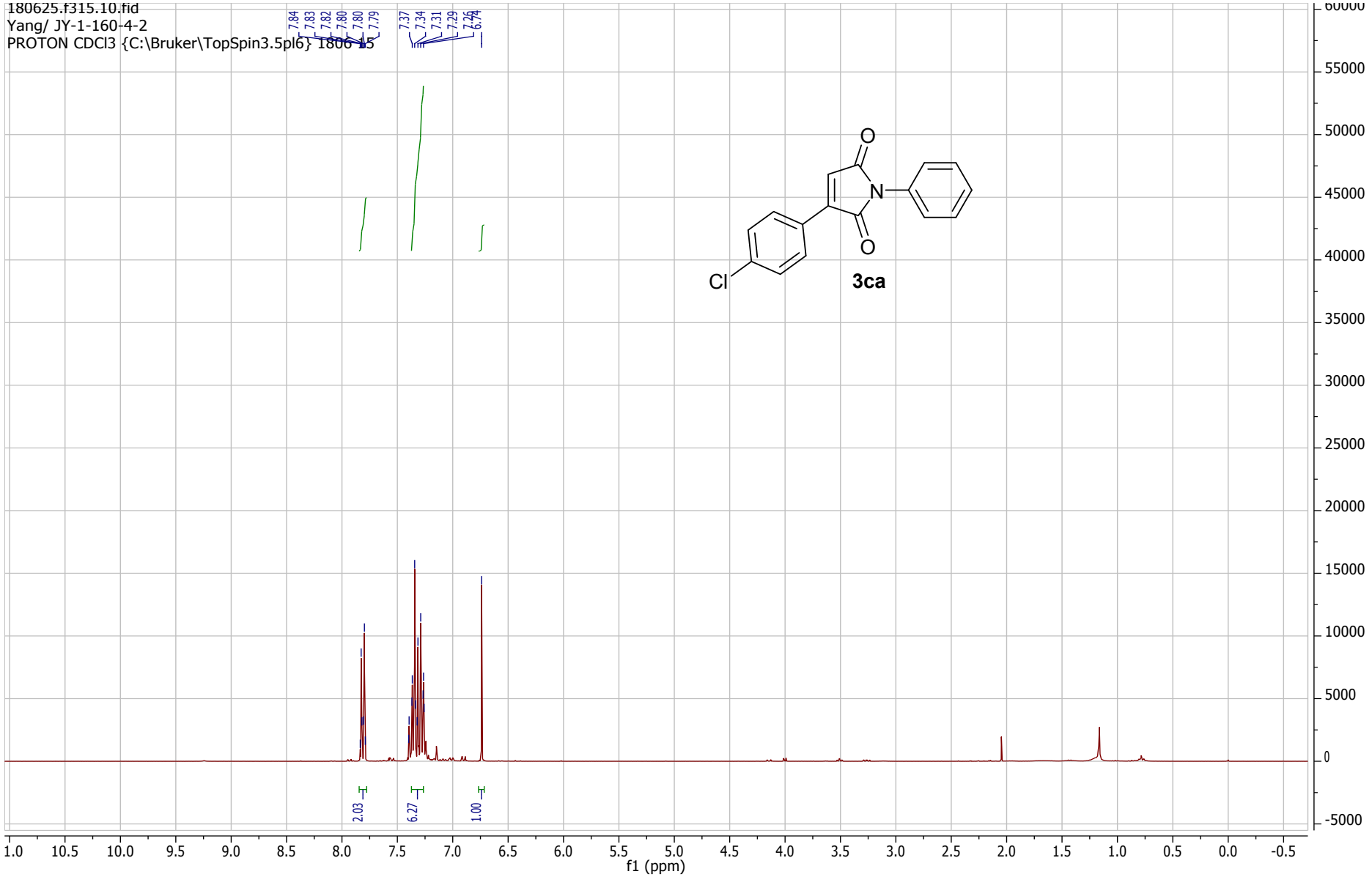
f1 (ppm)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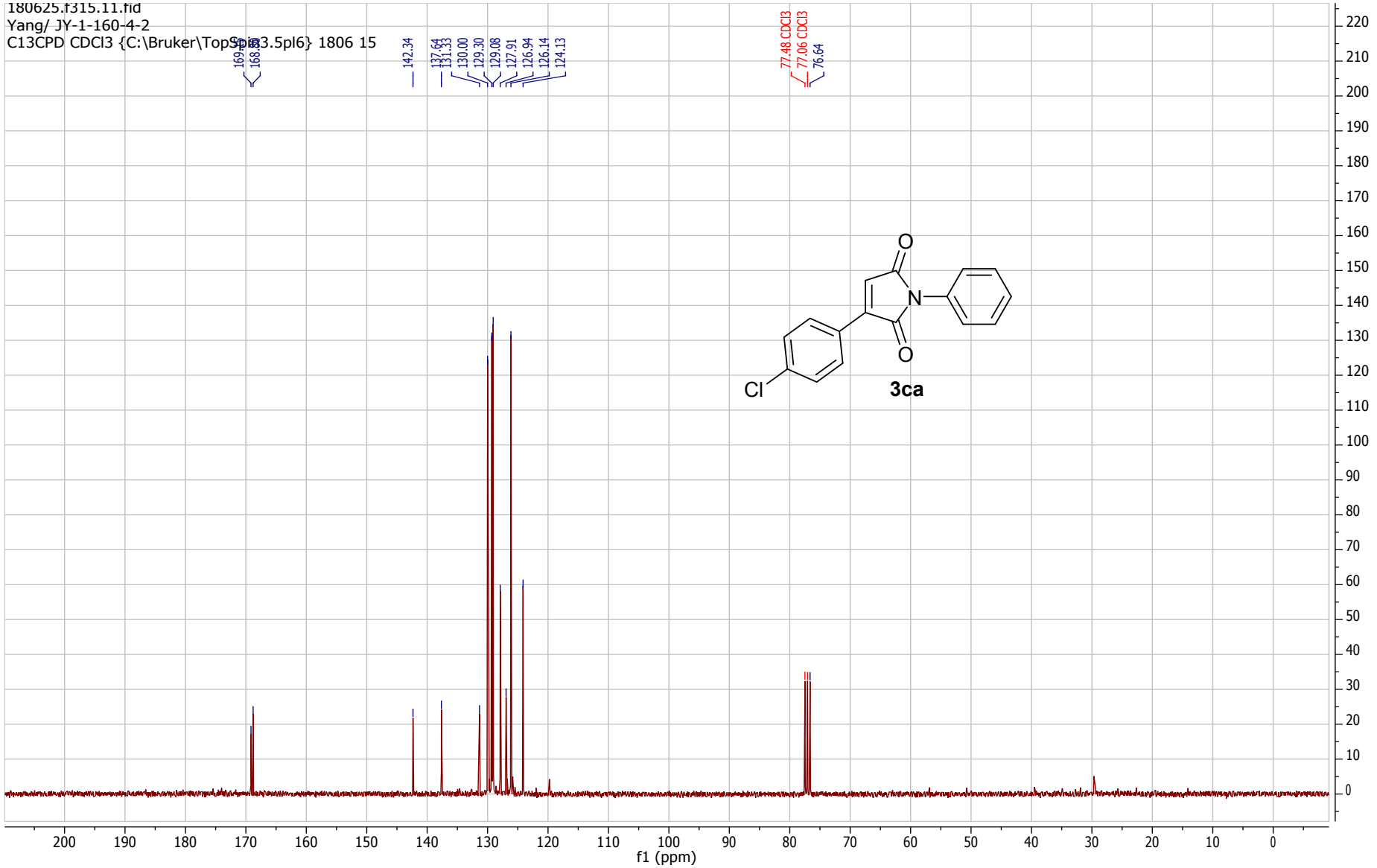
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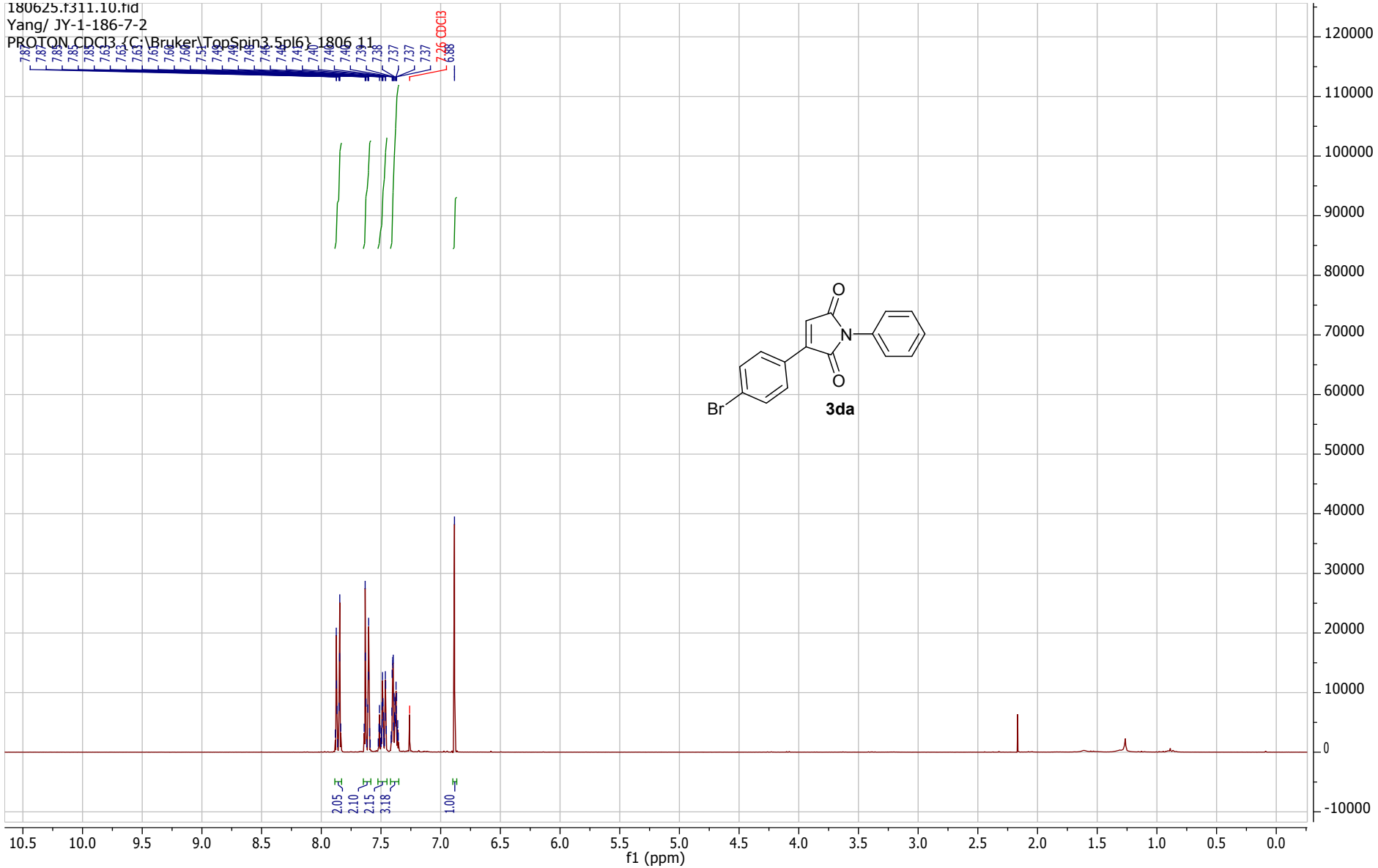
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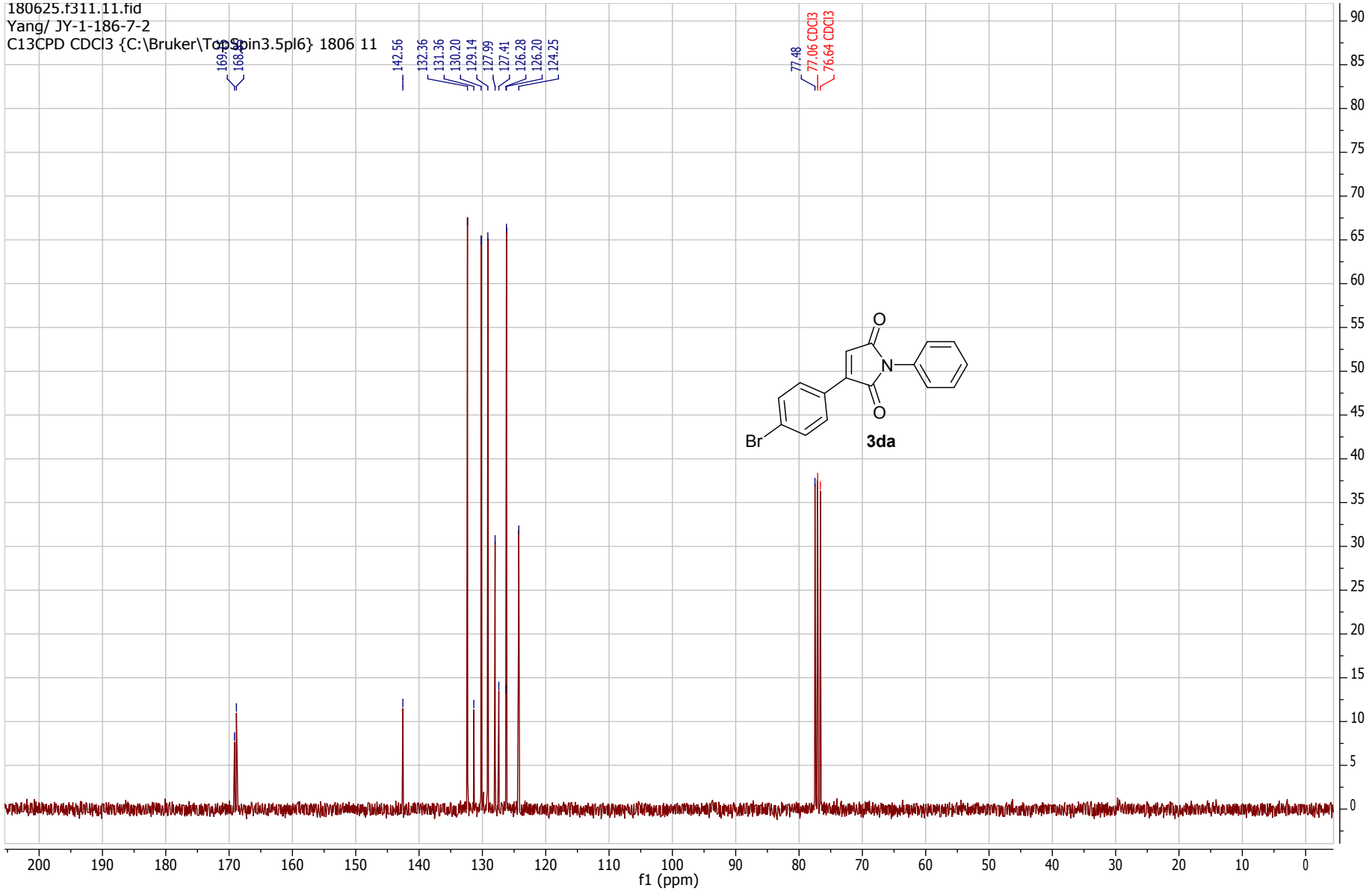
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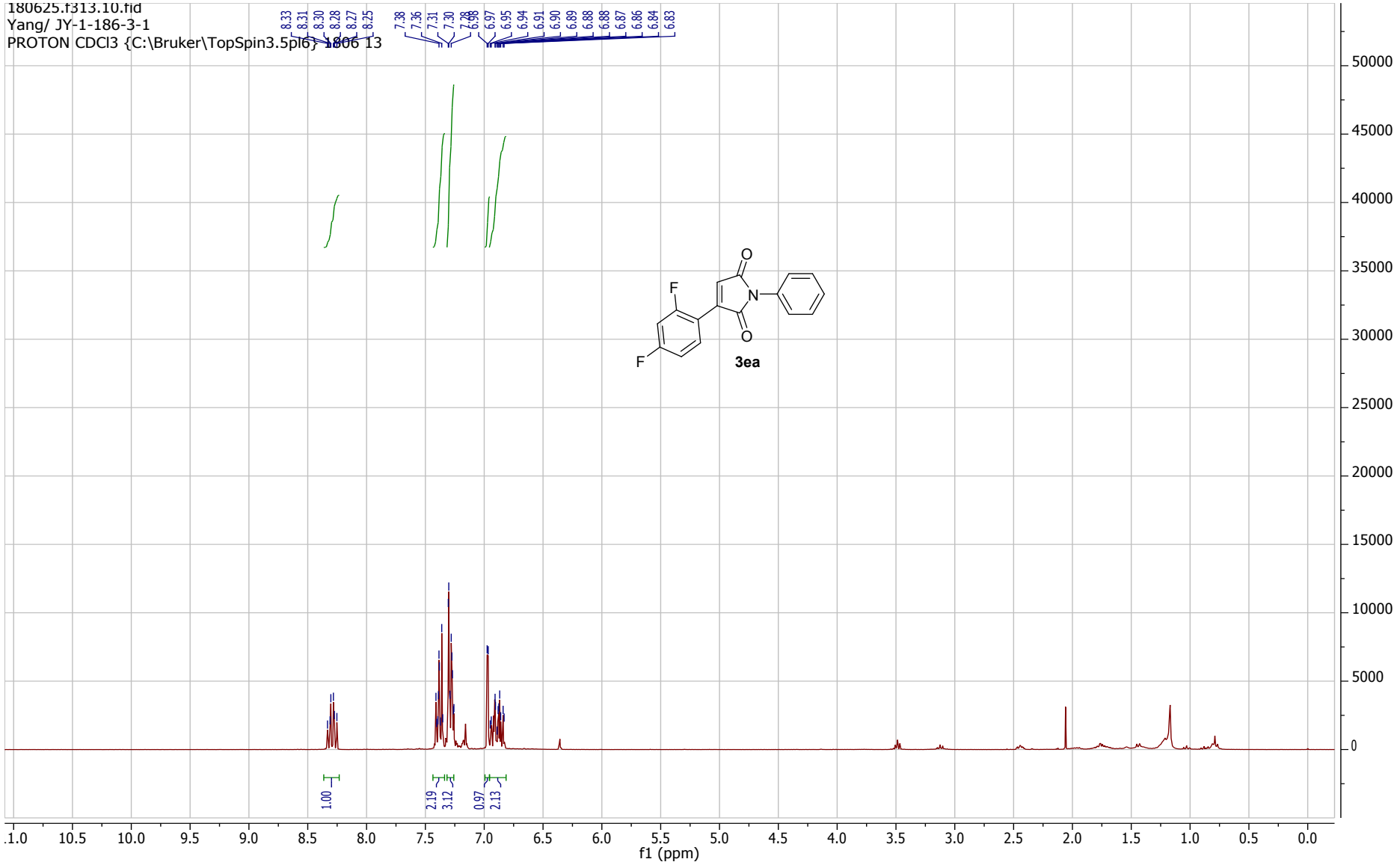
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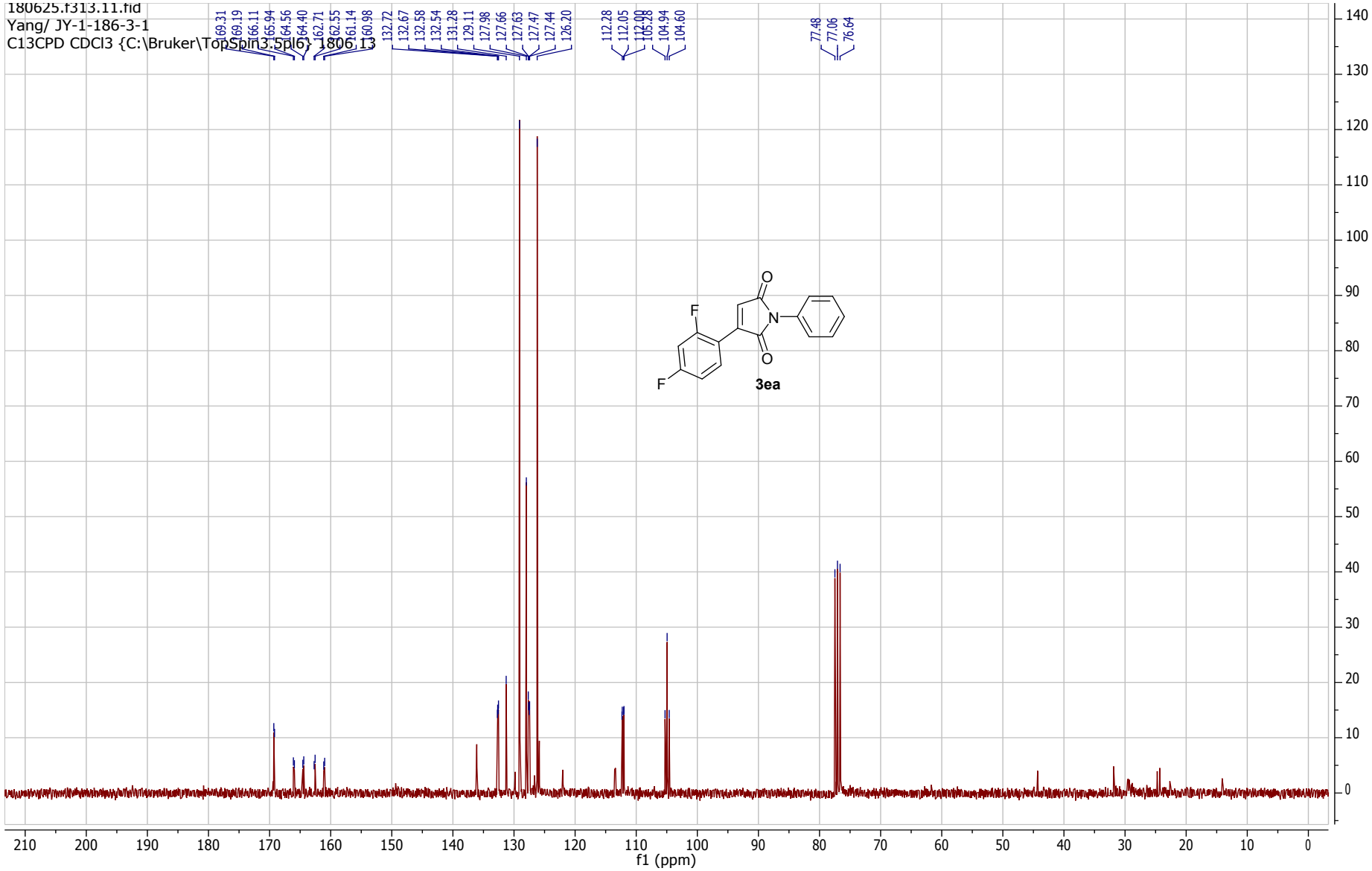
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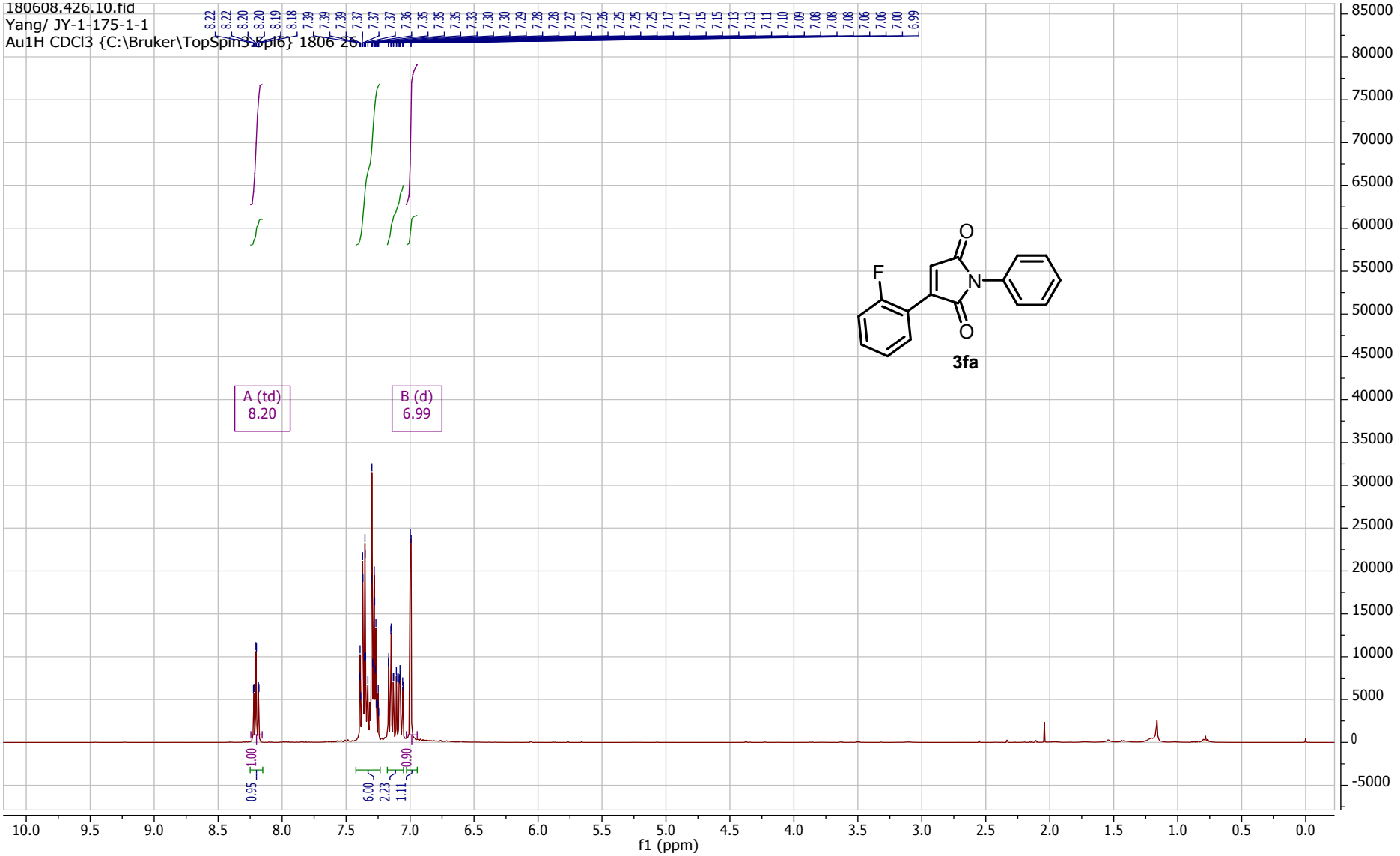
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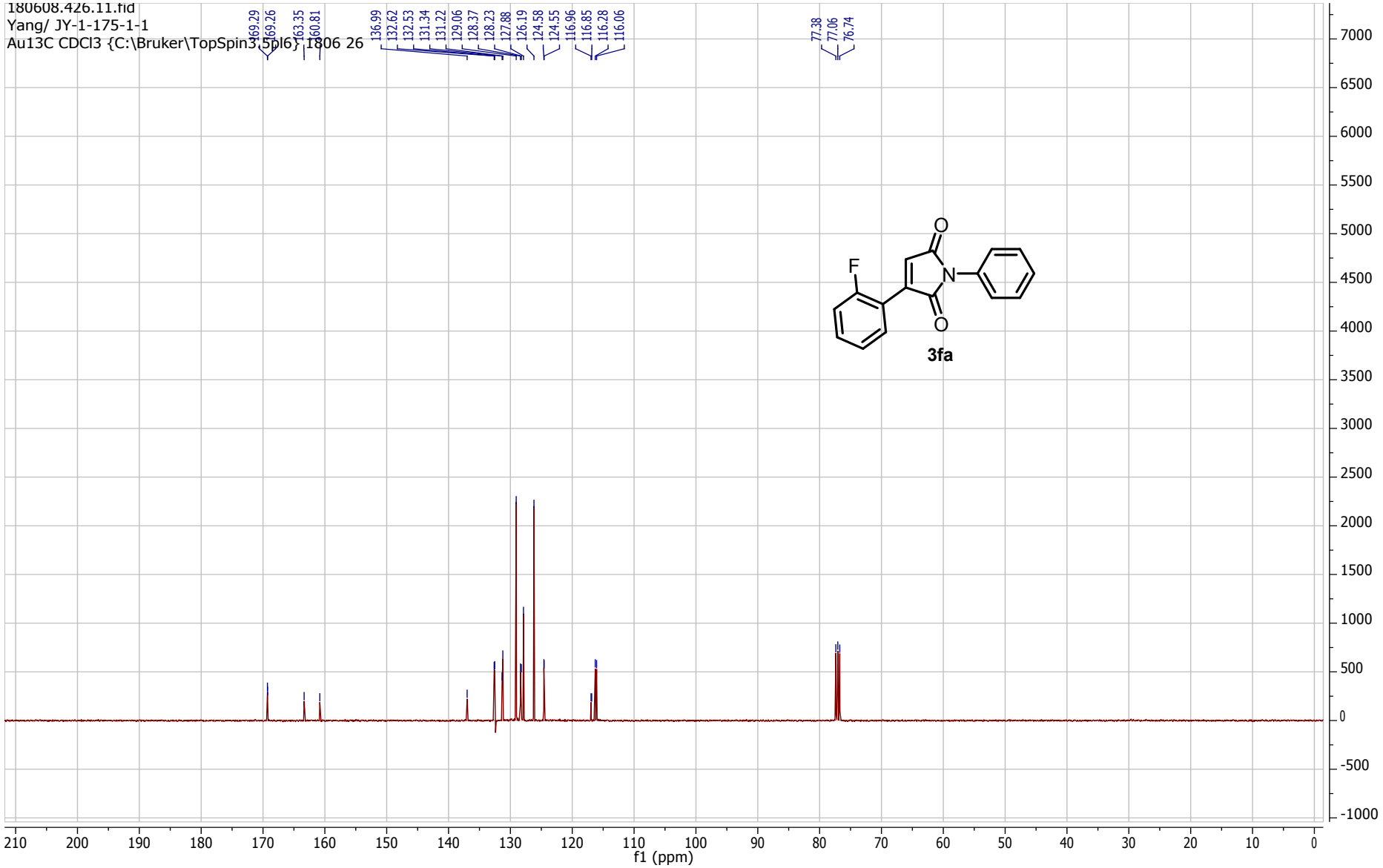
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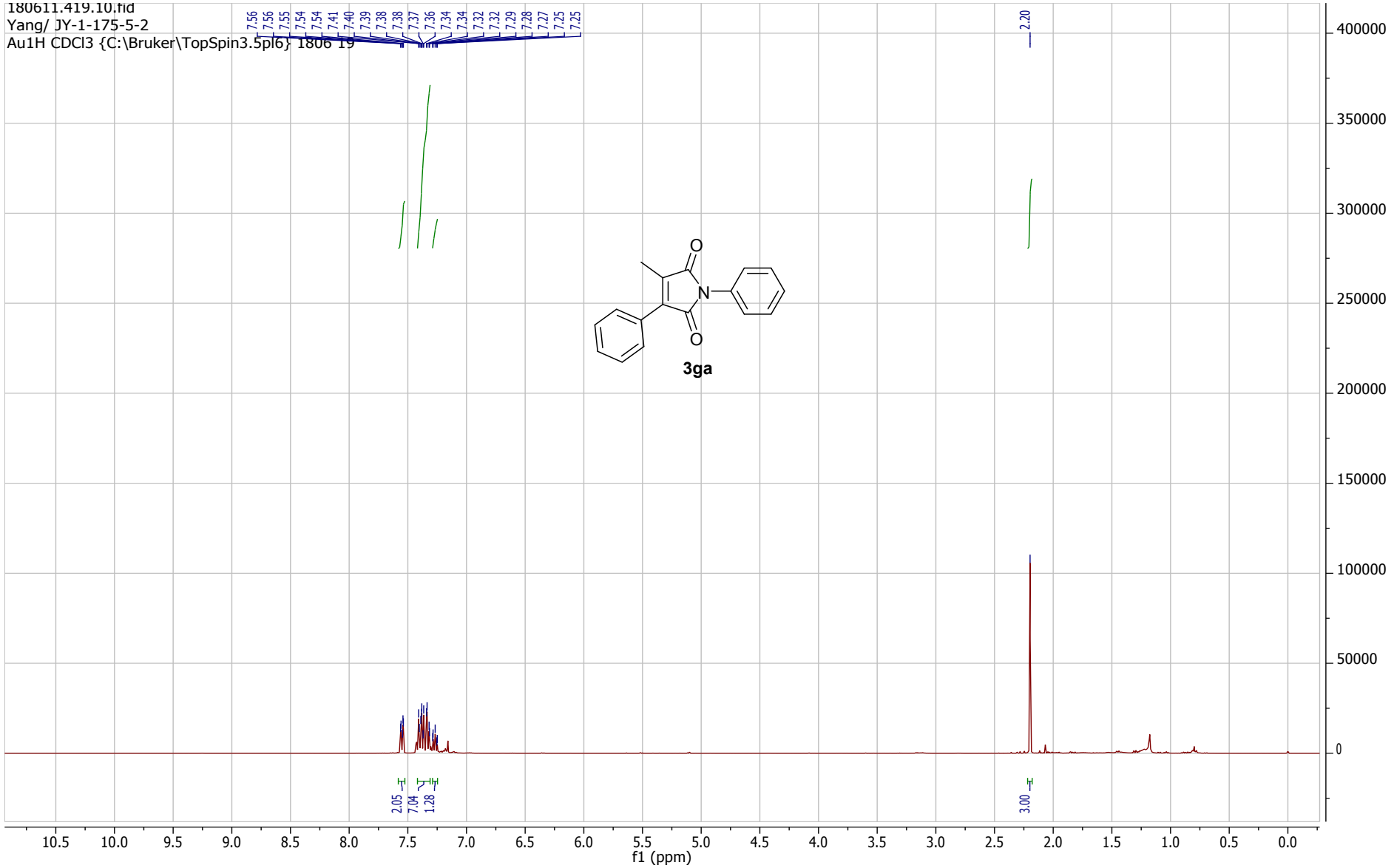
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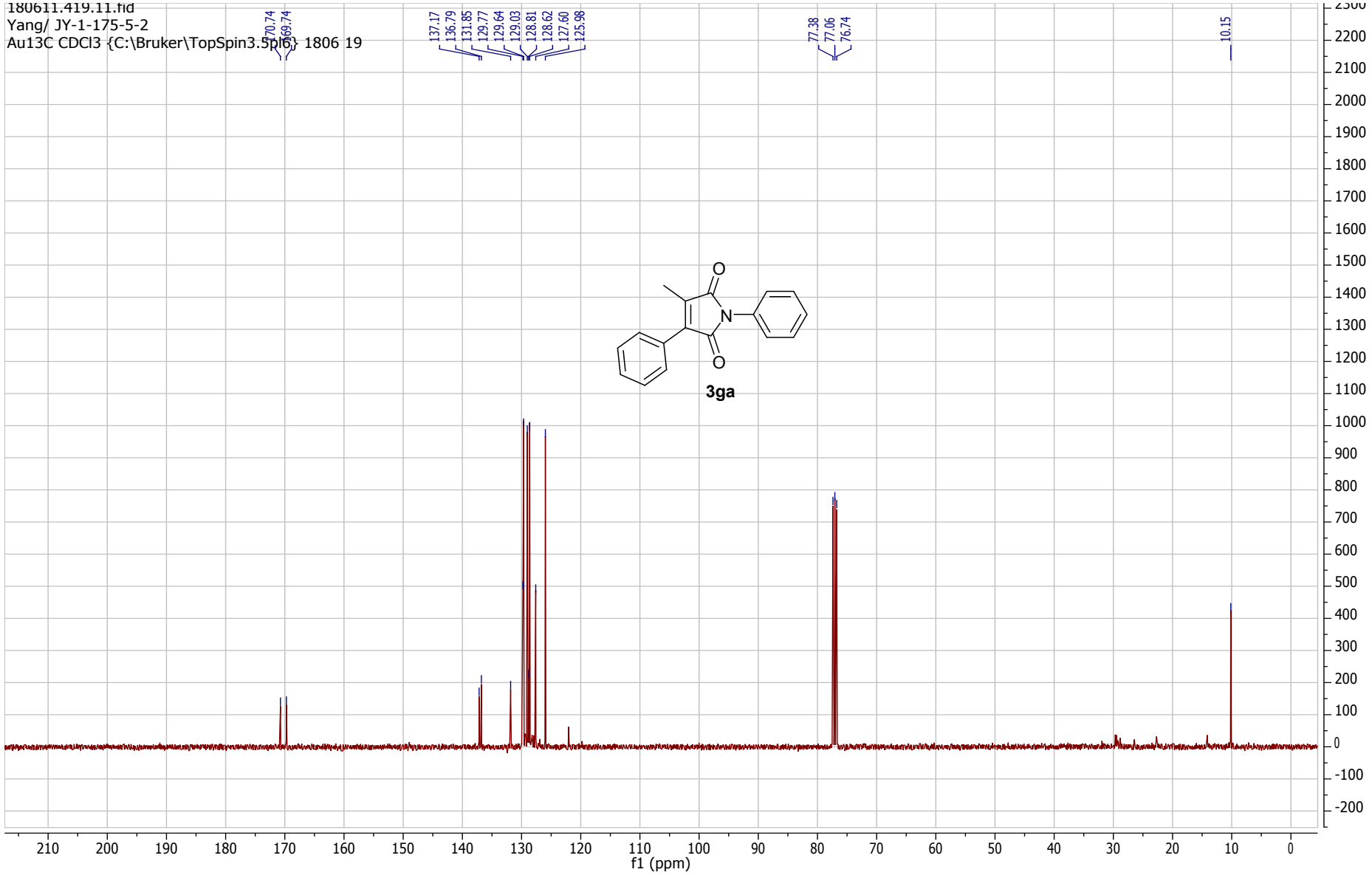
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N
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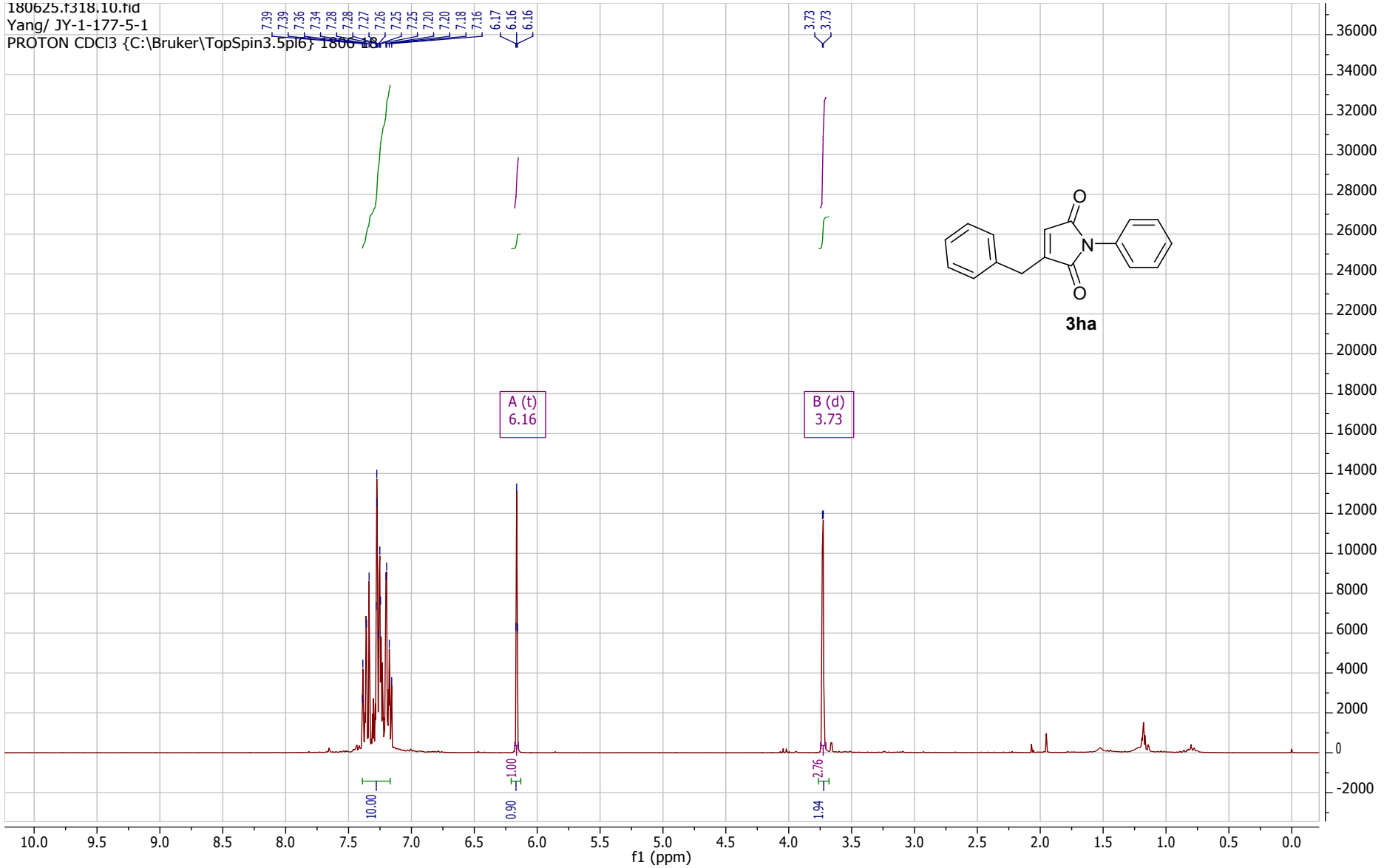
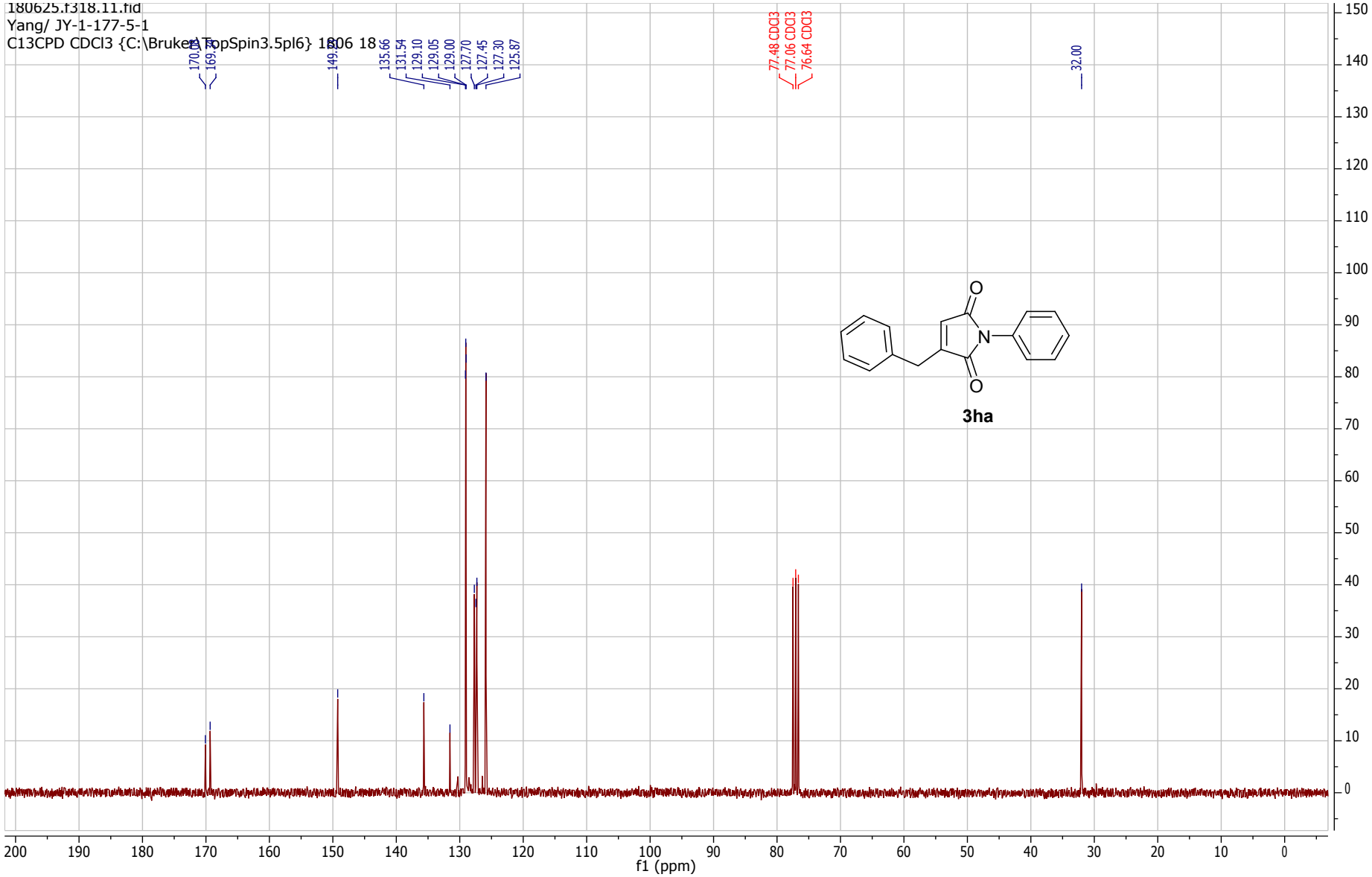
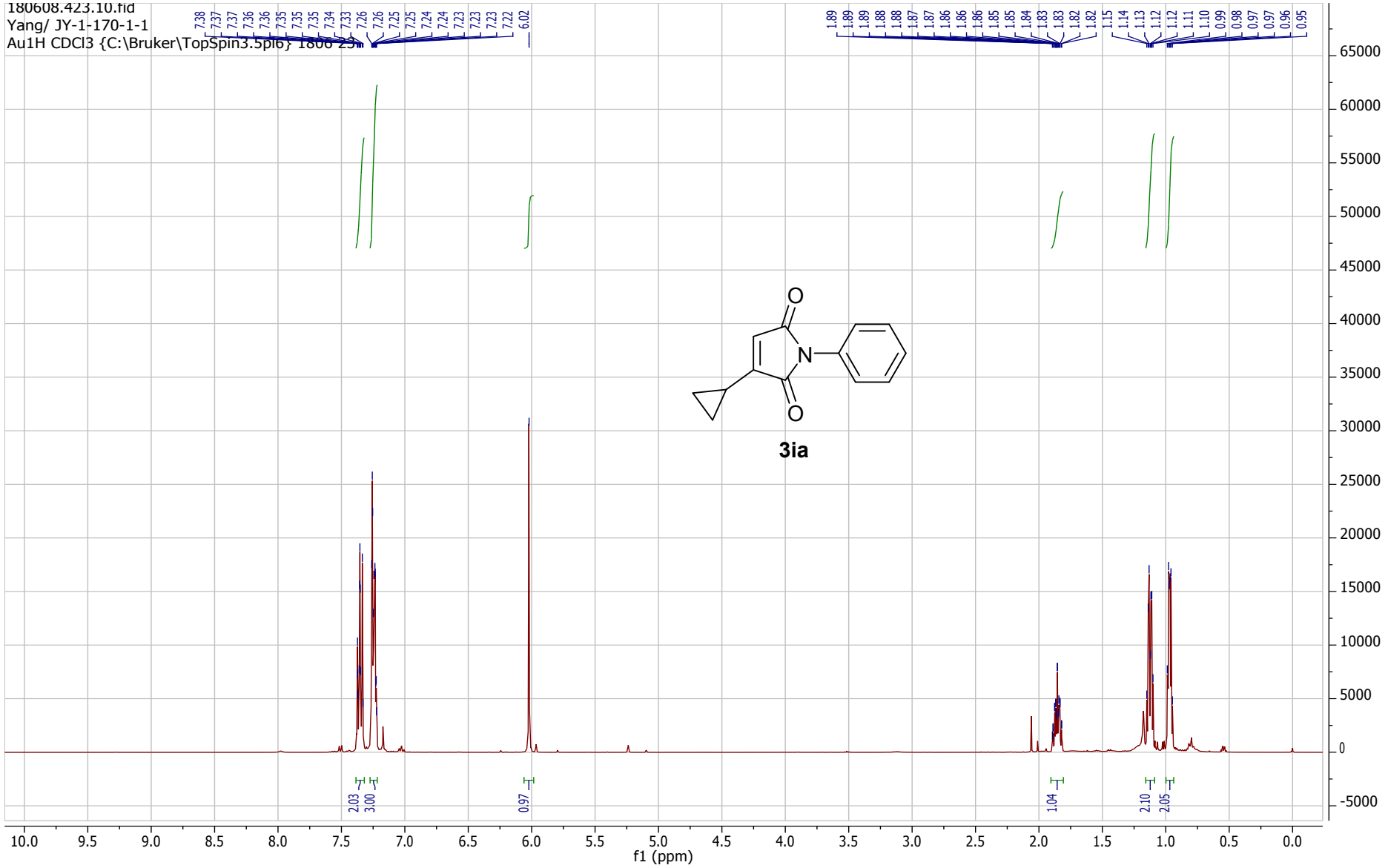
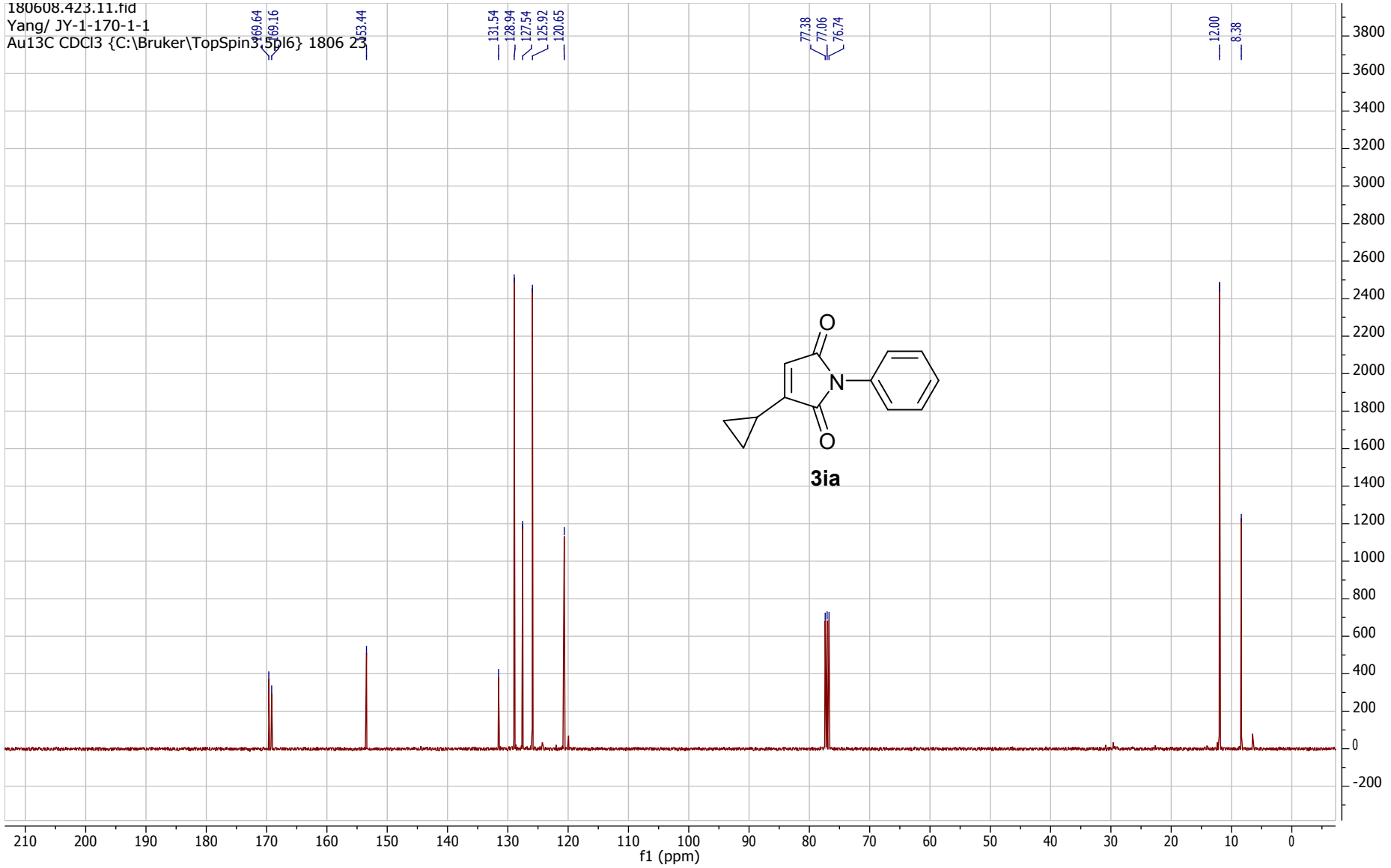
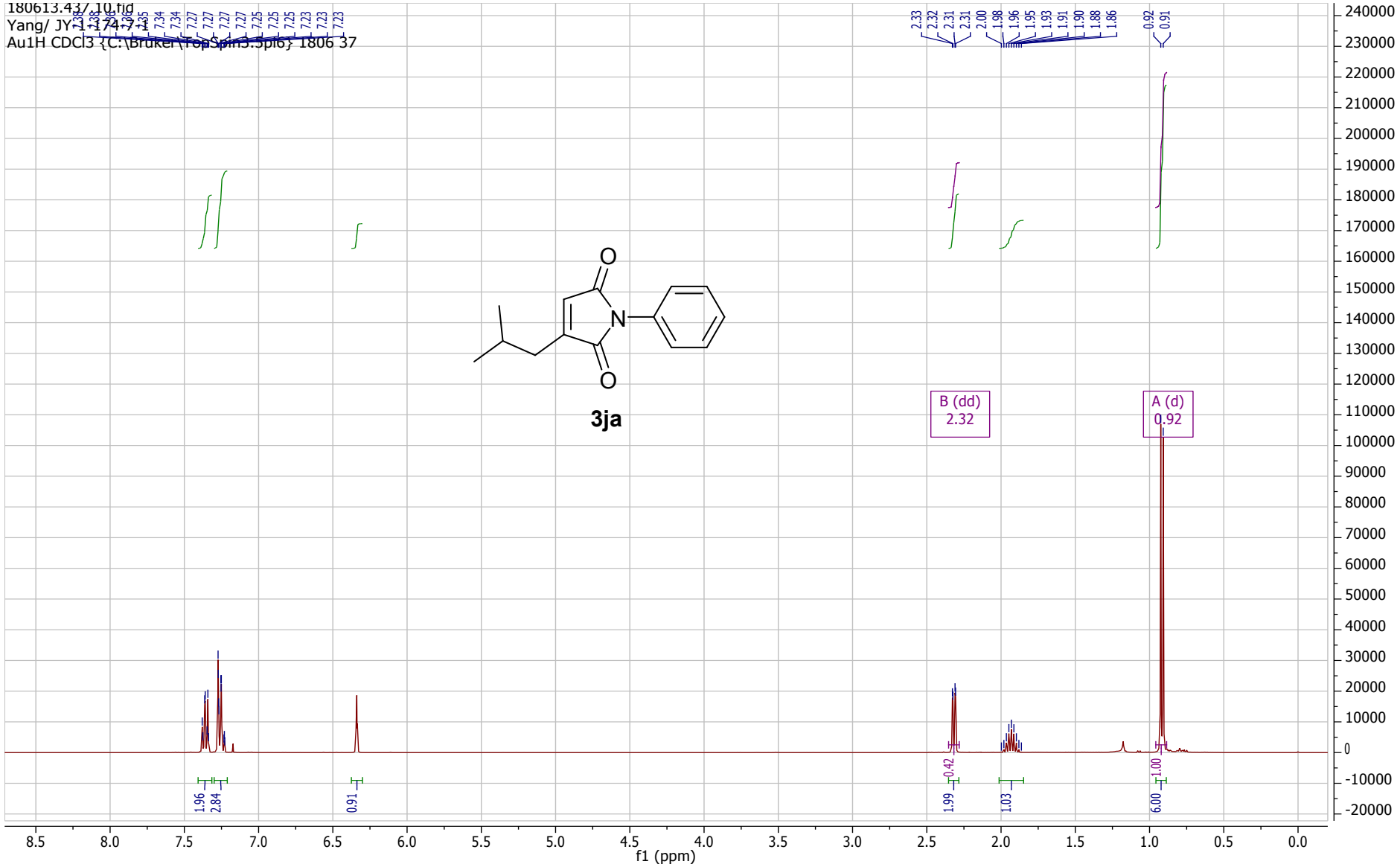
3ga
f1 (ppm)

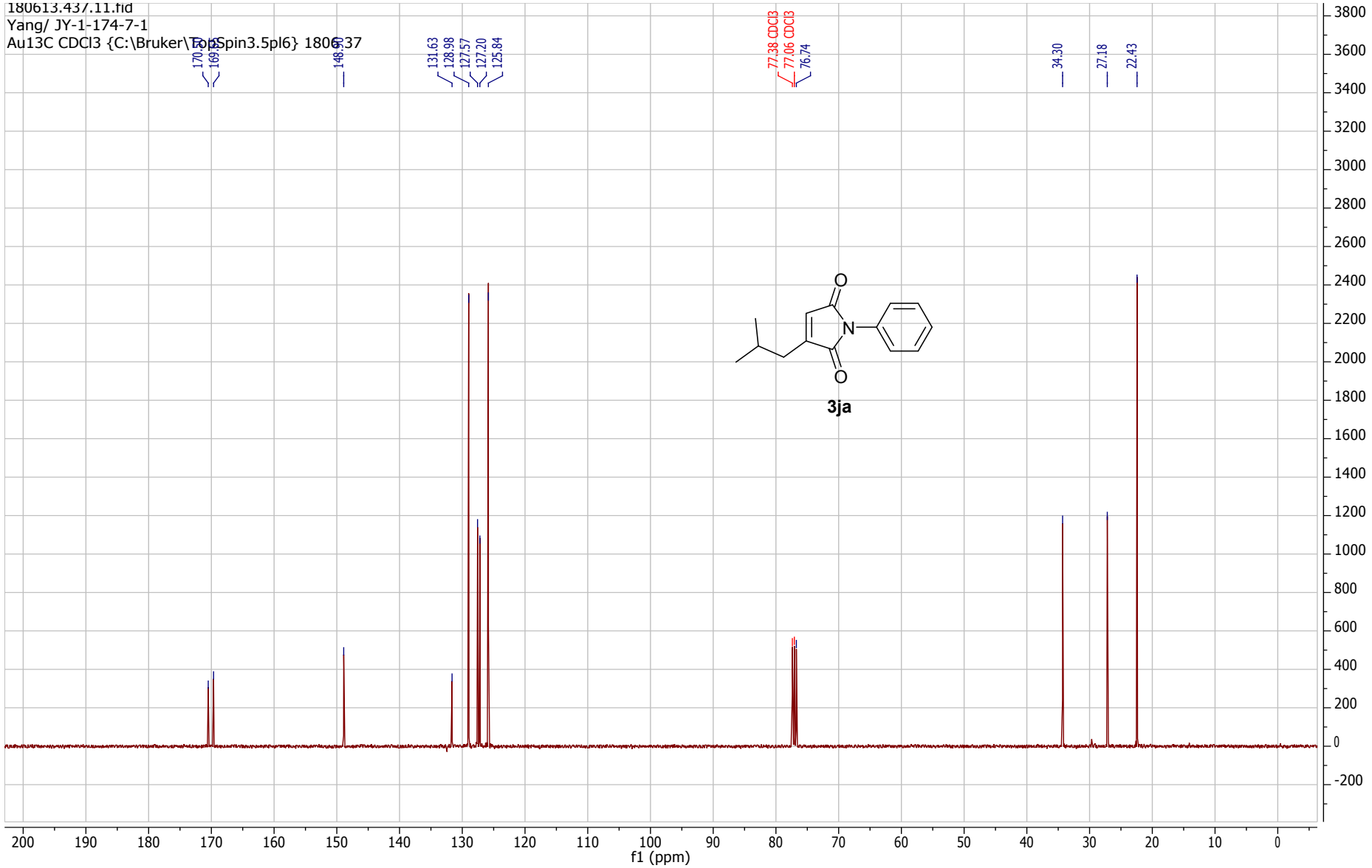

180625.f318.10.fid
Yang/ JY-1-177-5-1
PROTON CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806
7.39 7.39 7.36 7.34 7.28 7.28 7.27 7.26 7.25 7.25 7.20 7.20 7.18 7.16
6.17 6.16 6.16
3.73 3.73
A (t)
6.16
B (d)
3.73
10.00
1.00
0.90
2.76
1.94
f1 (ppm)
3ha

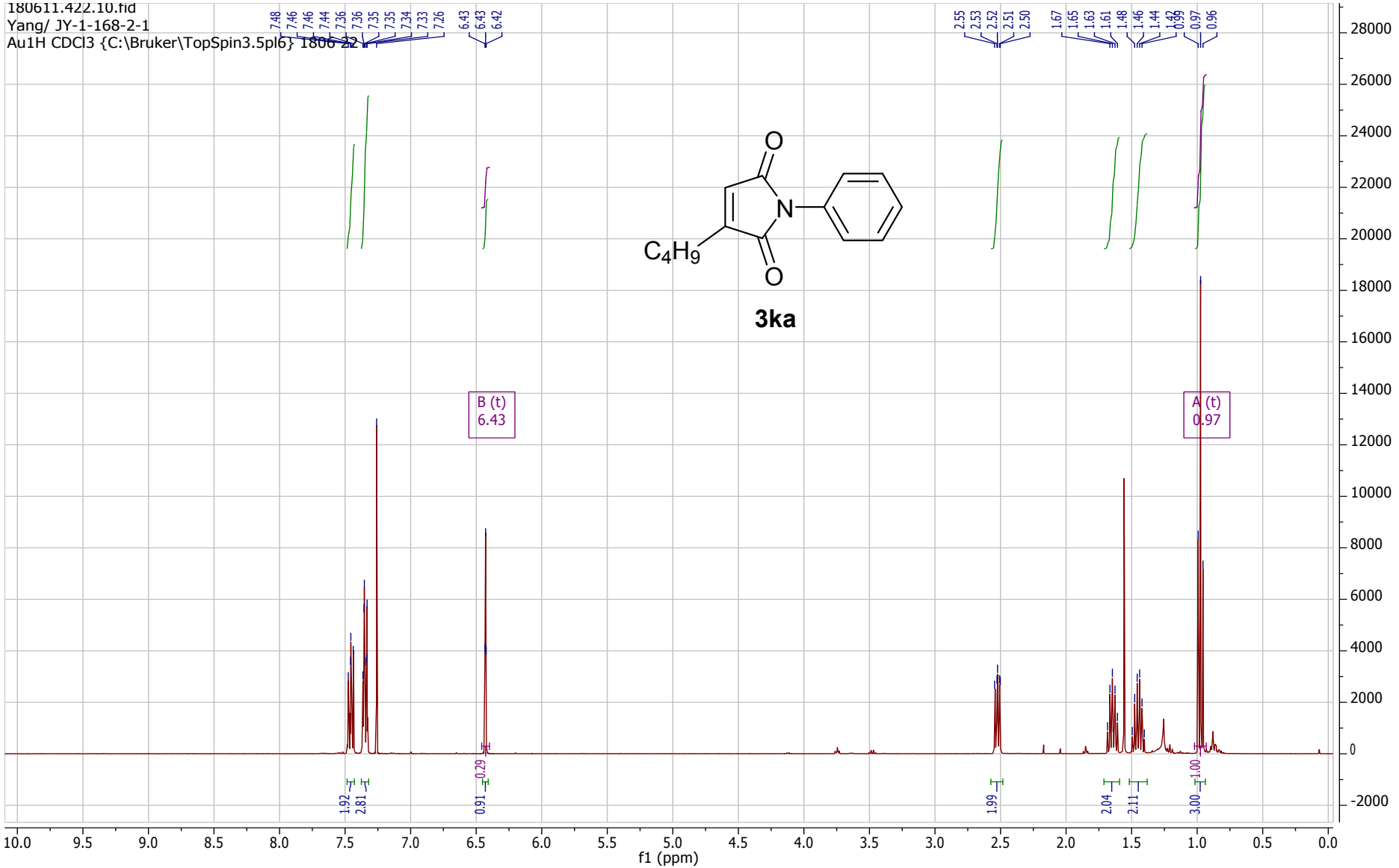

180625.f318.11.fid
Yang/ JY-1-177-5-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 18
170.43
169.74
149.33
135.66
131.54
129.10
129.05
129.00
127.70
127.45
127.30
125.87
77.48 CDCl3
77.06 CDCl3
76.64 CDCl3
32.00
O
N
O
3ha
f1 (ppm)

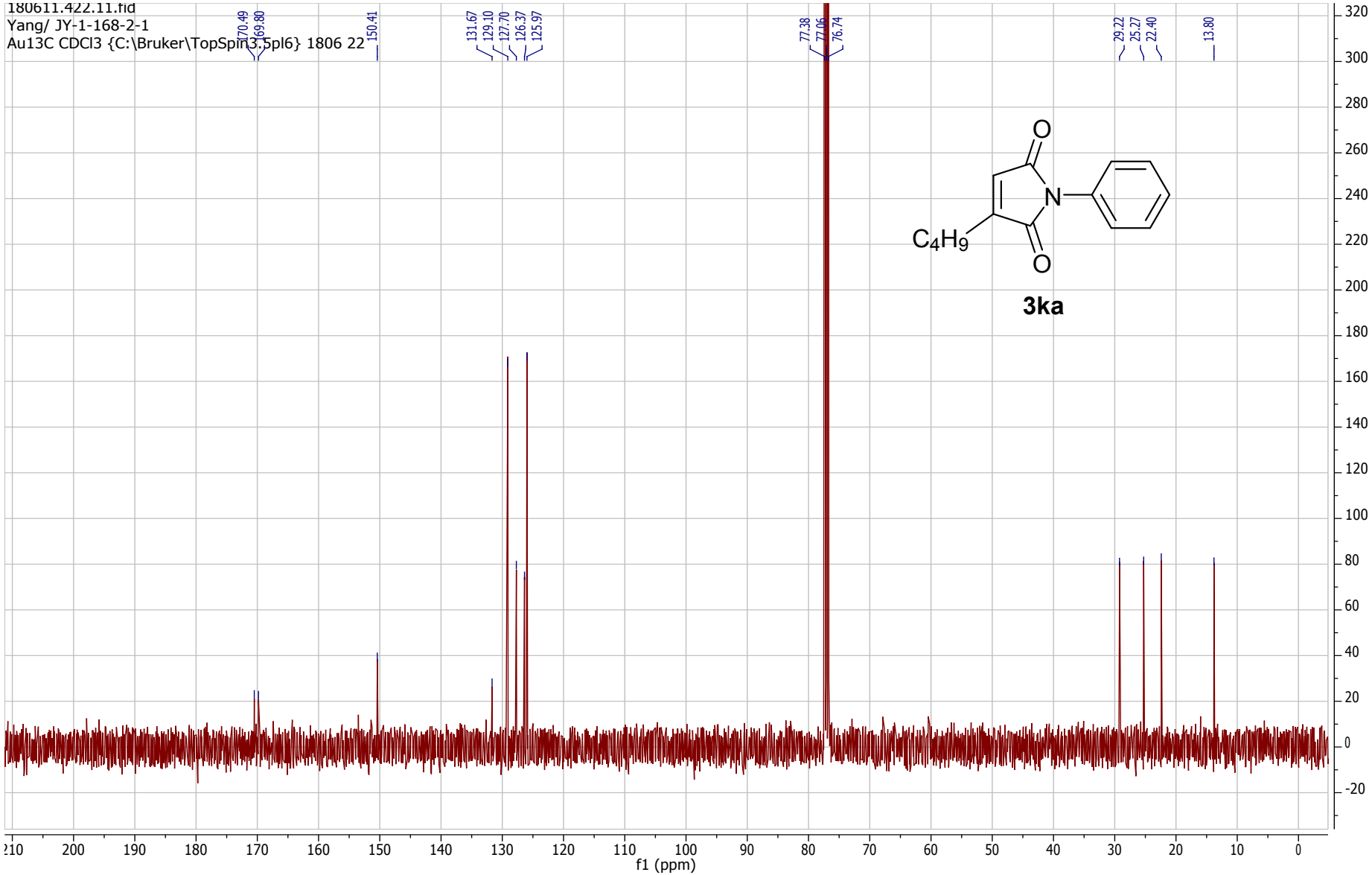

180608.423.10.fid
Yang/ JY-1-170-1-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 23
O
N
O
3ia
2.03
3.00
0.97
1.04
2.10
2.05
f1 (ppm)

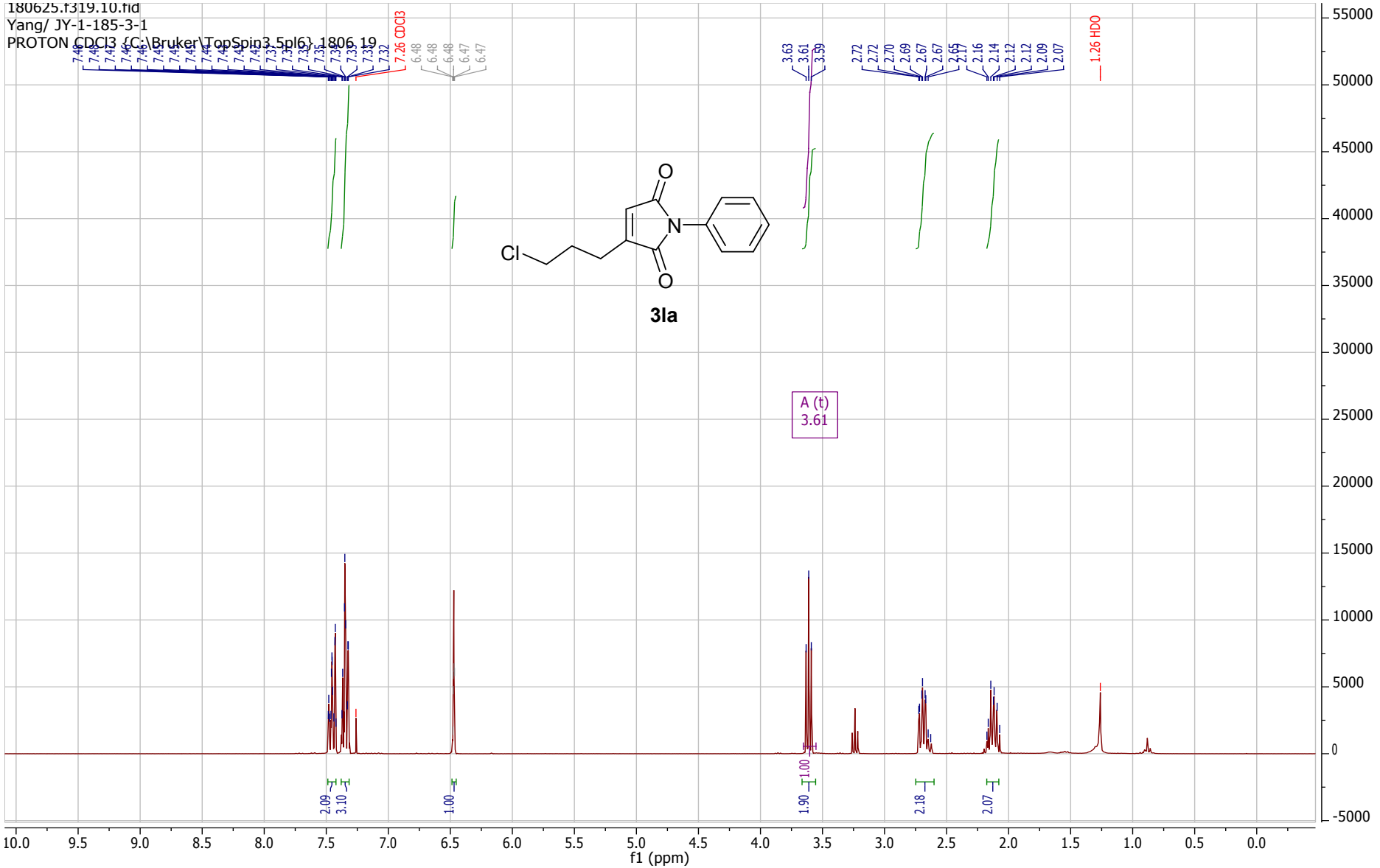

180608.423.11.fid
Yang/ JY-1-170-1-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 23
169.64
169.16
153.44
131.54
128.94
127.54
125.92
120.65
77.38
77.06
76.74
12.00
8.38
O
N
O
3ia
f1 (ppm)

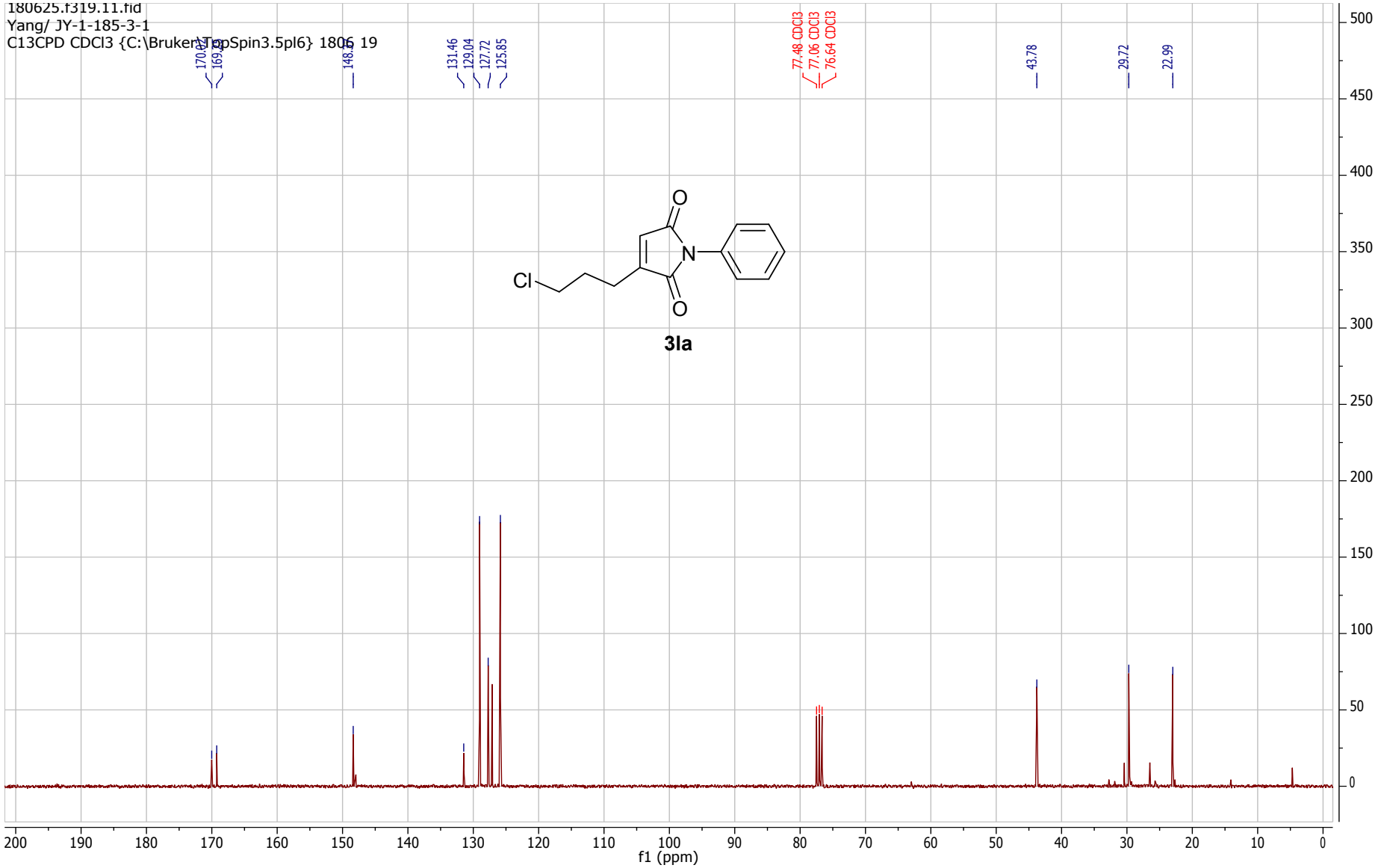

180613.43/ 10.fid
Yang/ JY-1-174-7-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 37
O
N
O
3ja
B (dd)
2.32
A (d)
0.92
f1 (ppm)

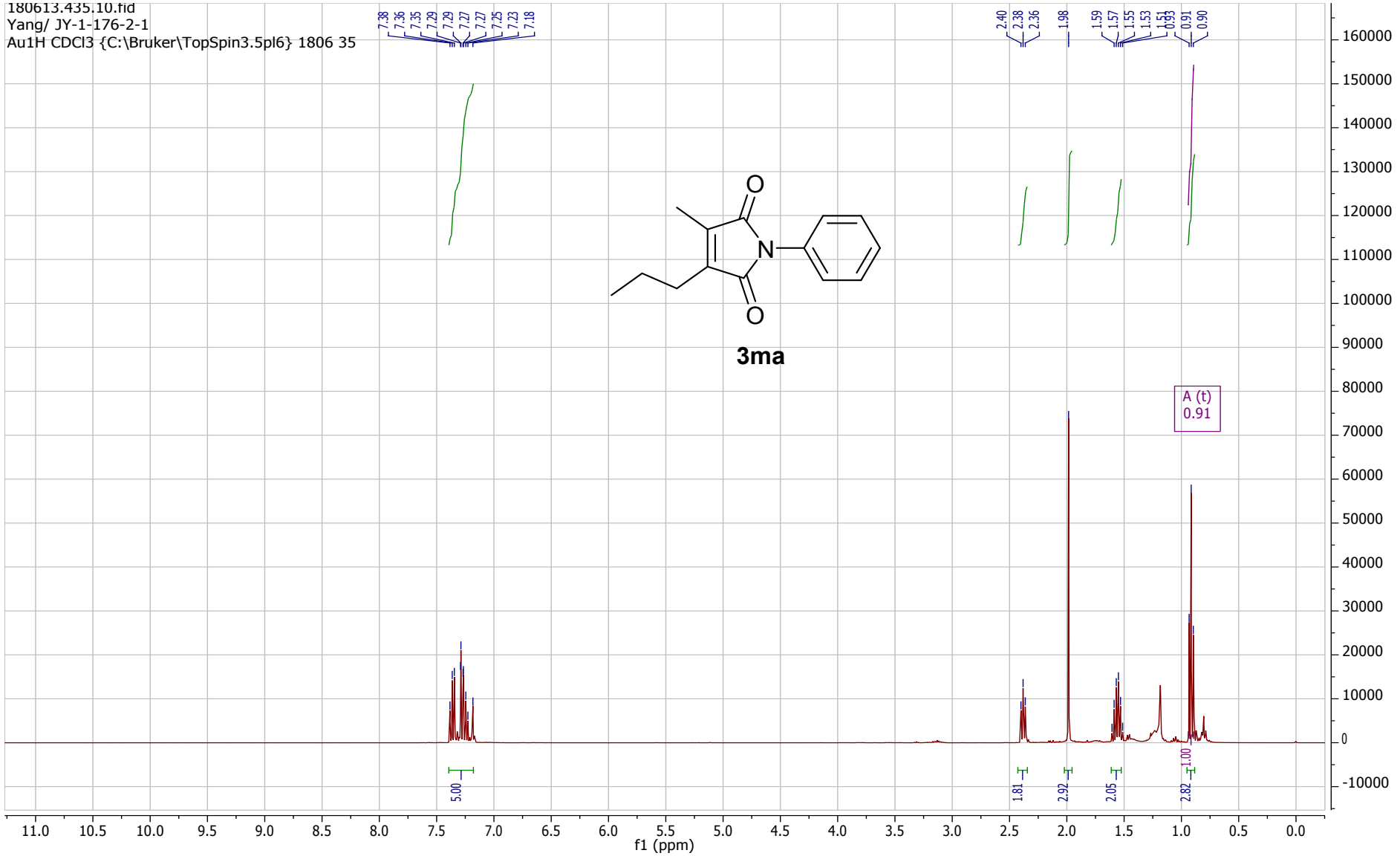

180613.437.11.fid
Yang/ JY-1-174-7-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 37
170.45
169.13
148.90
131.63
128.98
127.57
127.20
125.84
77.38 CDCl3
77.06 CDCl3
76.74
34.30
27.18
22.43
O
N
O
3ja
f1 (ppm)

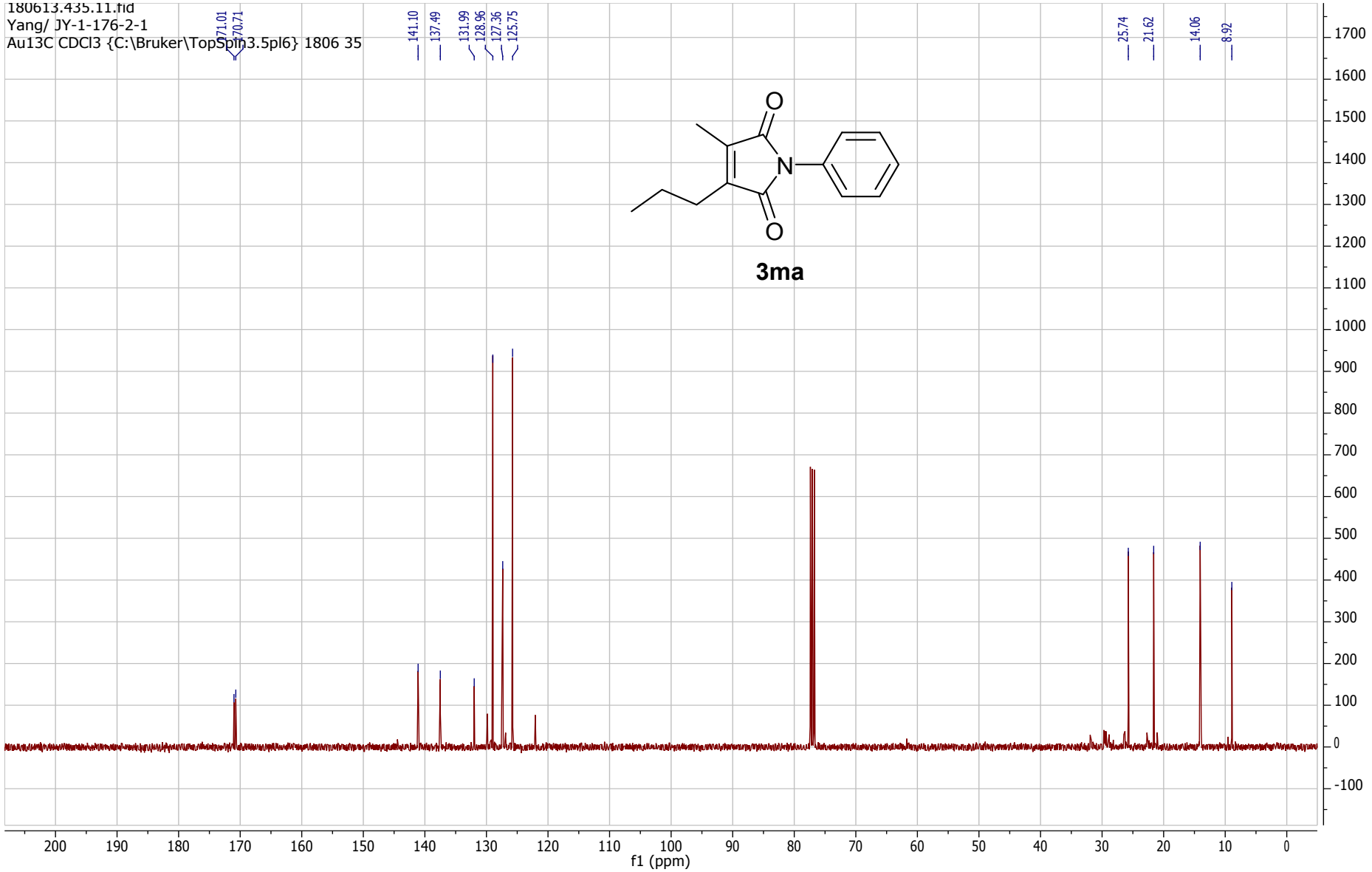

180611.422.10.fid
Yang/ JY-1-168-2-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 22
7.48 7.46 7.46 7.44 7.36 7.36 7.35 7.35 7.34 7.33 7.26 6.43 6.43 6.42
2.55 2.53 2.52 2.51 2.50 1.67 1.65 1.63 1.61 1.48 1.46 1.44 0.99 0.97 0.96
O
N
O
C4H9
3ka
B (t)
6.43
A (t)
0.97
1.92 2.81 0.29 0.91 1.99 2.04 2.11 3.00 1.00
f1 (ppm)

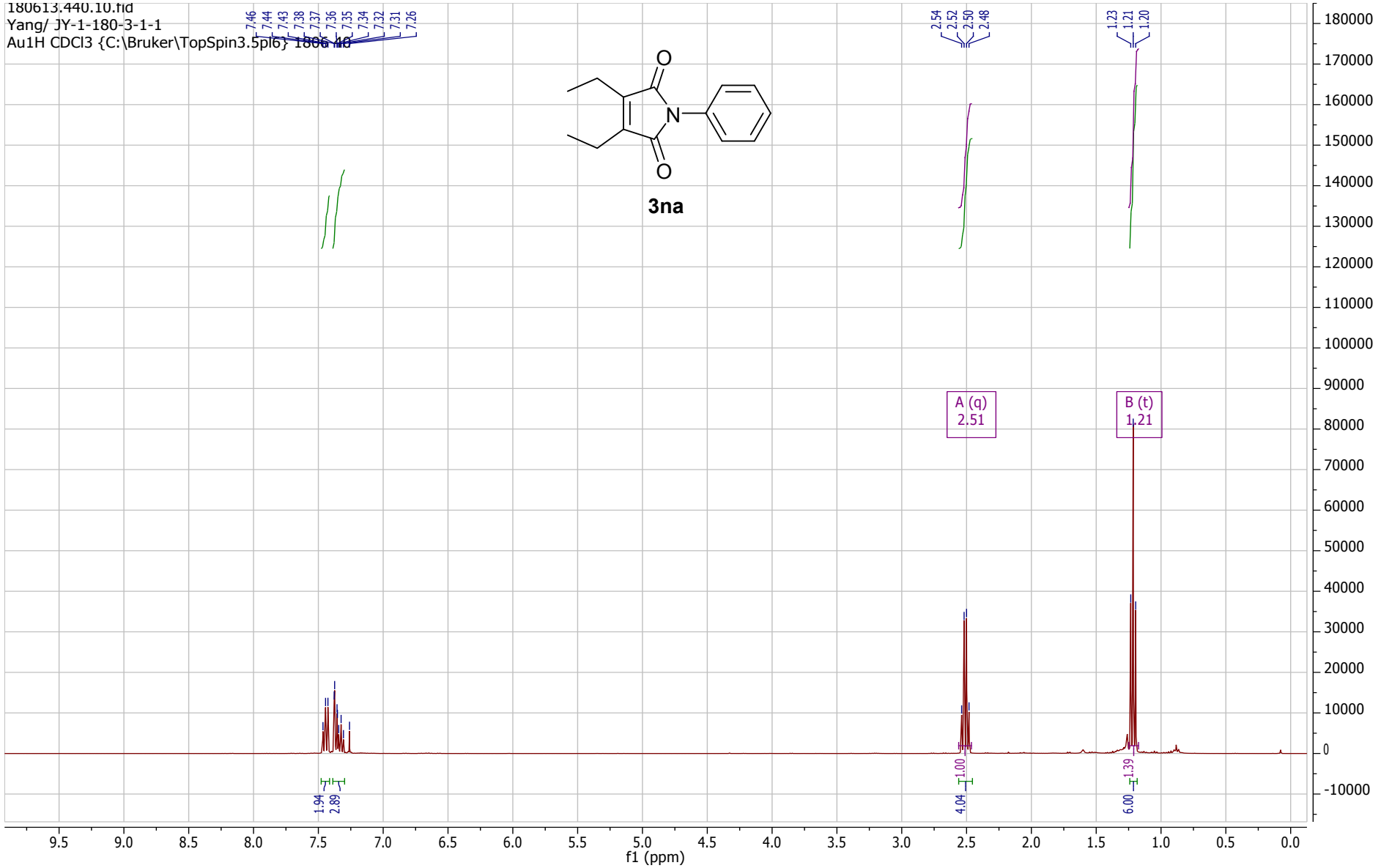

180611.422.11.fid
Yang/ JY-1-168-2-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 22
170.49
169.80
150.41
131.67
129.10
127.70
126.37
125.97
77.38
77.06
76.74
29.22
25.27
22.40
13.80
O
N
O
C4H9
3ka
f1 (ppm)

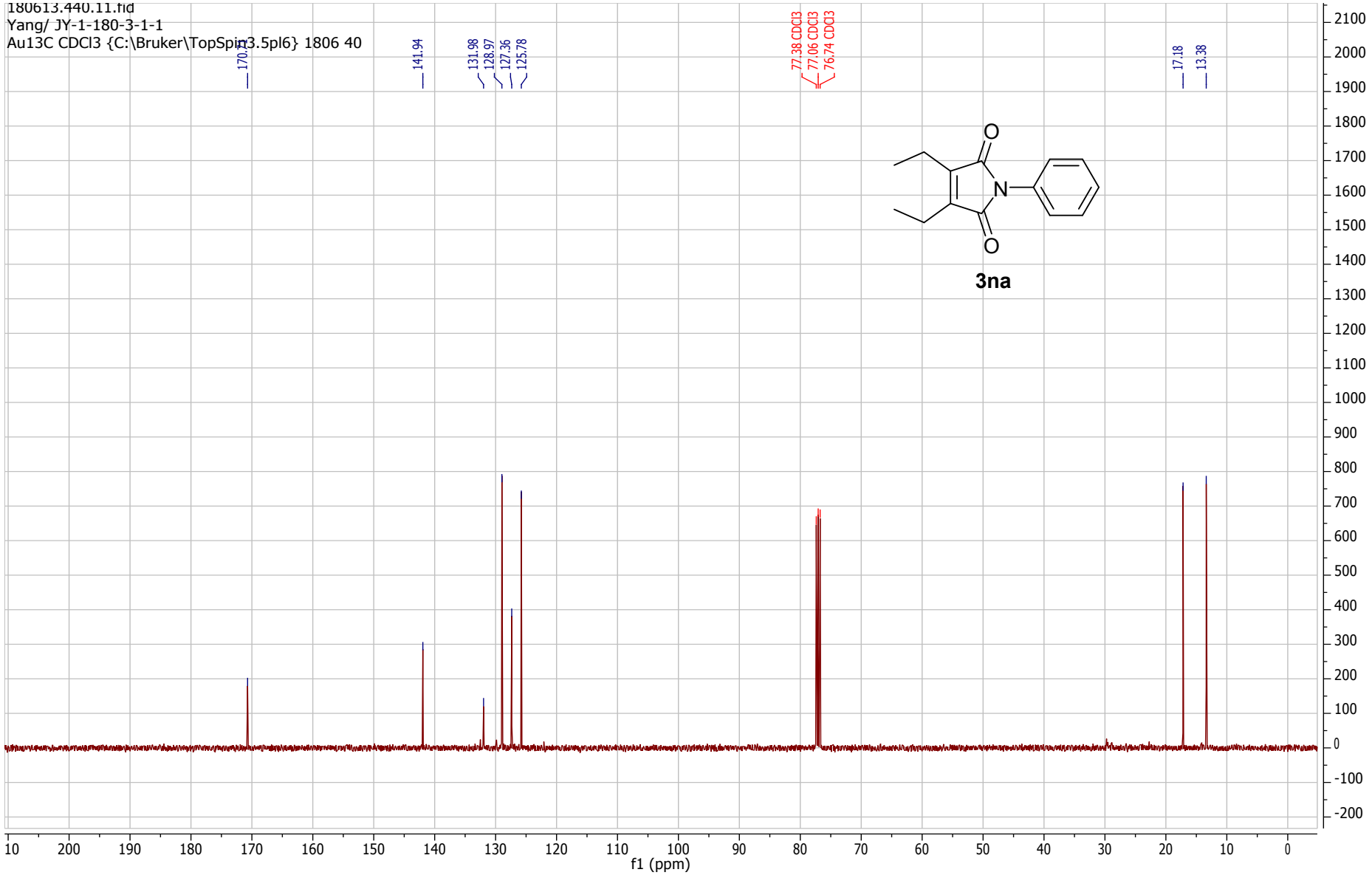

180625.f319.10.fid
Yang/ JY-1-185-3-1
PROTON CDCl3 (C:\Bruker\TopSpin3.5pl6) 1806 19
7.26 CDCl3
1.26 HDO
3.63
3.61
3.59
2.72
2.72
2.70
2.69
2.67
2.67
2.65
2.16
2.14
2.12
2.12
2.09
2.07
6.48
6.48
6.48
6.47
6.47
O
N
O
Cl
3la
A (t)
3.61
2.09
3.10
1.00
1.90
1.00
2.18
2.07
f1 (ppm)

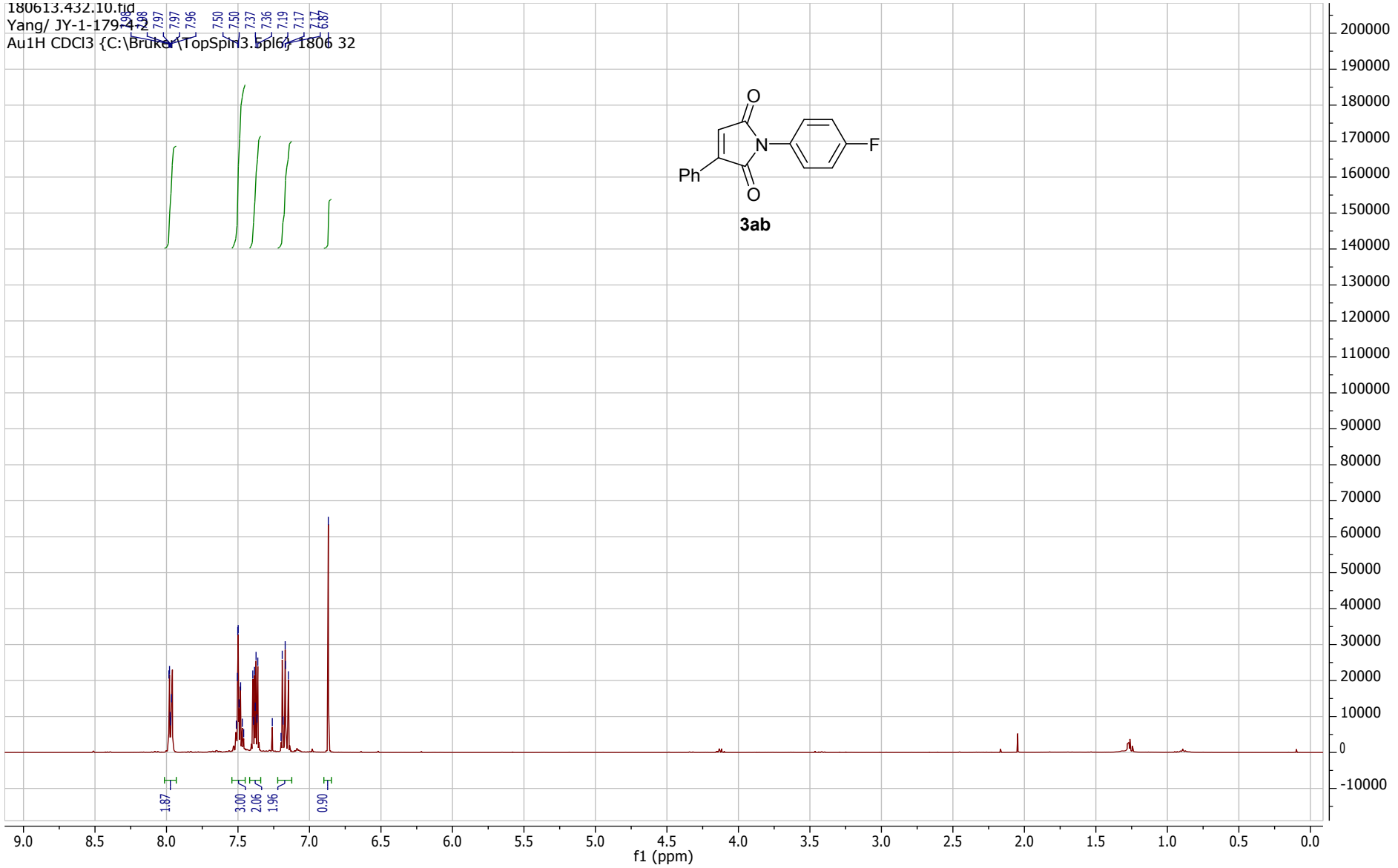

180625.f319.11.fid
Yang/ JY-1-185-3-1
C13CPD CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 19
170.02
169.90
148.15
131.46
129.04
127.72
125.85
77.48 CDCl3
77.06 CDCl3
76.64 CDCl3
43.78
29.72
22.99
O
N
Cl
O
3la
f1 (ppm)

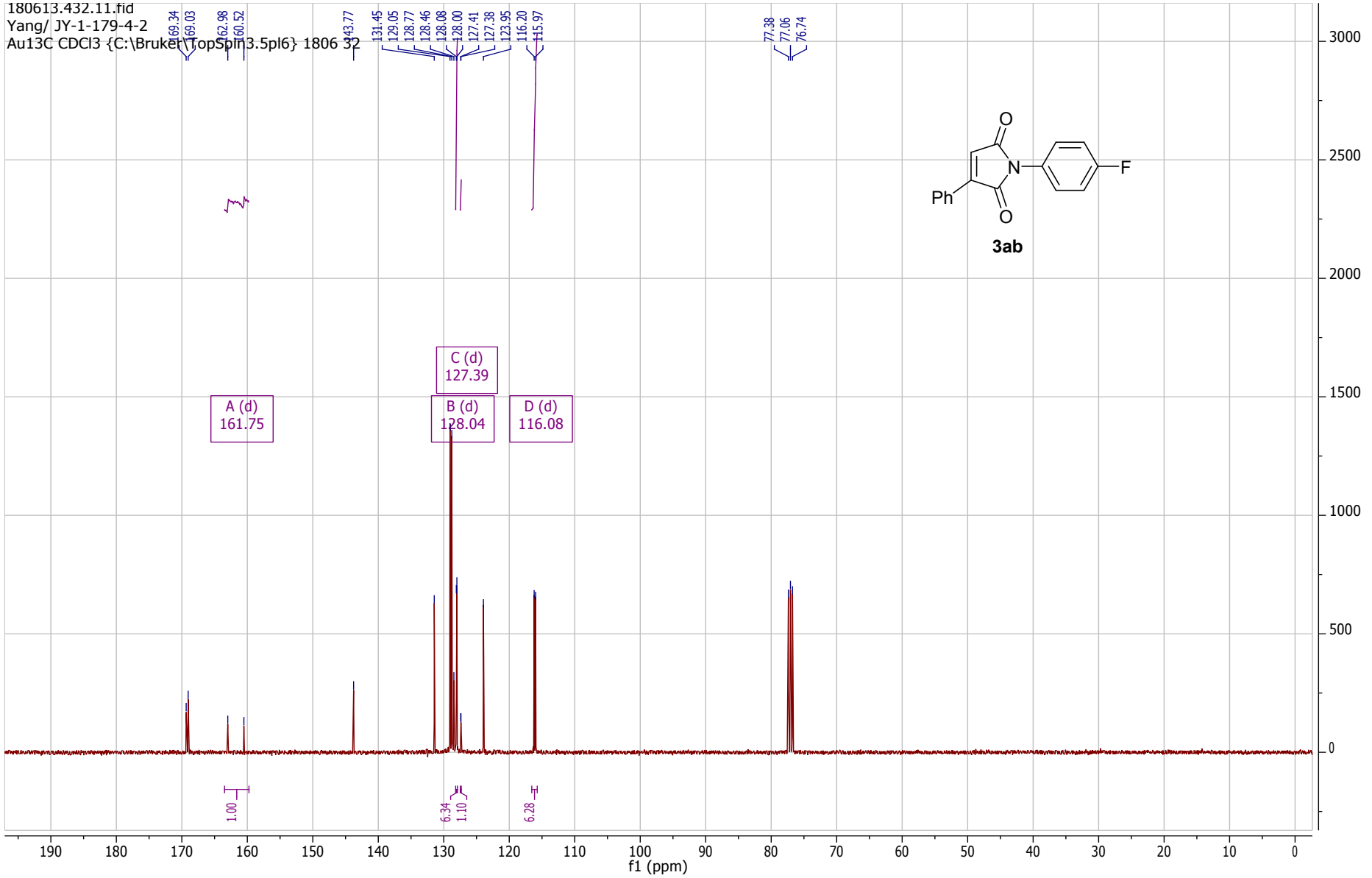

180613.435.10.fid
Yang/ JY-1-176-2-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 35
7.38
7.36
7.35
7.29
7.29
7.27
7.27
7.25
7.23
7.18
2.40
2.38
2.36
1.98
1.59
1.57
1.55
1.53
1.51
0.93
0.91
0.90
O
N
O
3ma
A (t)
0.91
5.00
1.81
2.92
2.05
2.82
1.00
f1 (ppm)

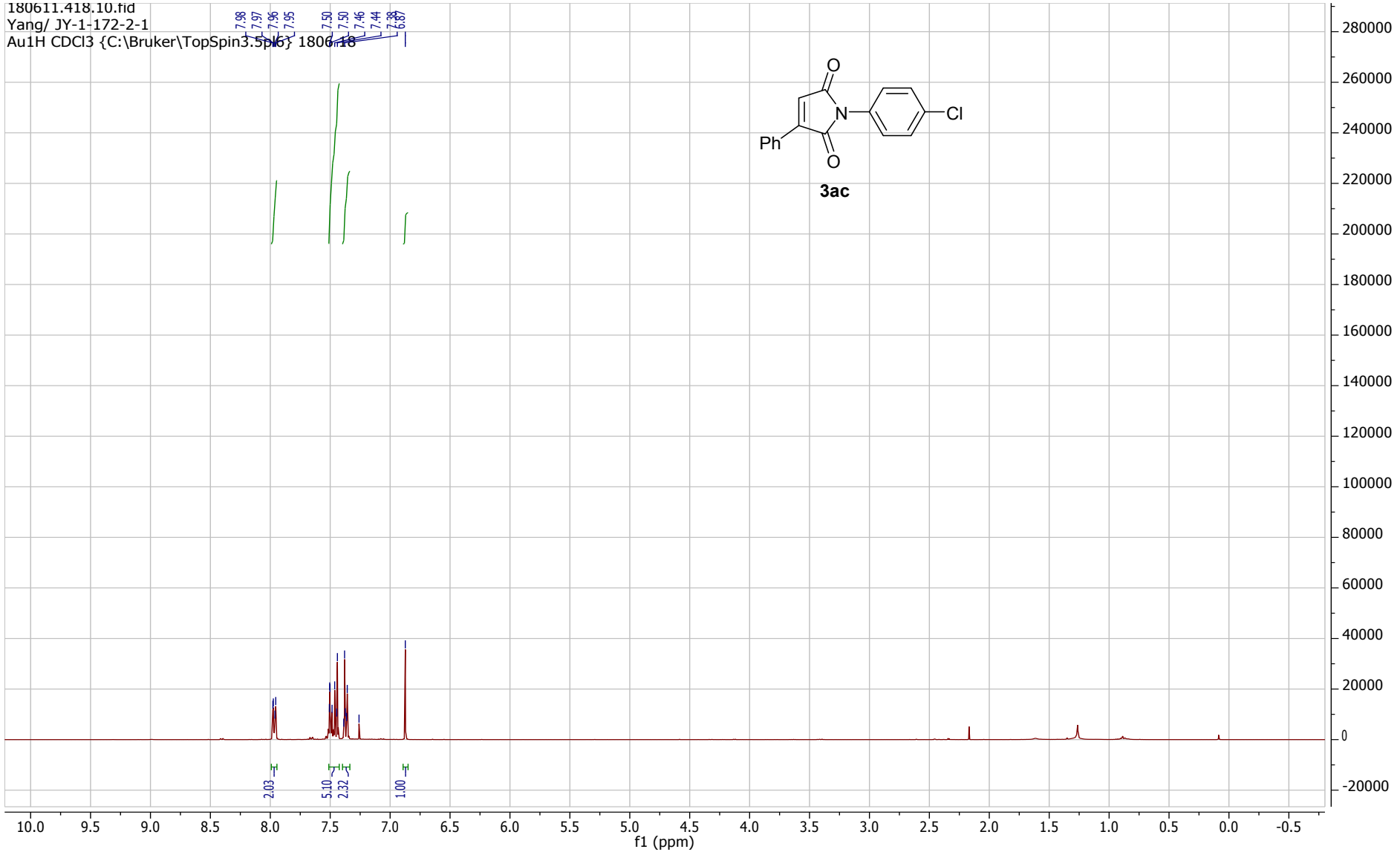

180613.435.11.fid
Yang/ JY-1-176-2-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 35
171.01
170.71
141.10
137.49
131.99
128.96
127.36
125.75
25.74
21.62
14.06
8.92
O
N
O
3ma
f1 (ppm)

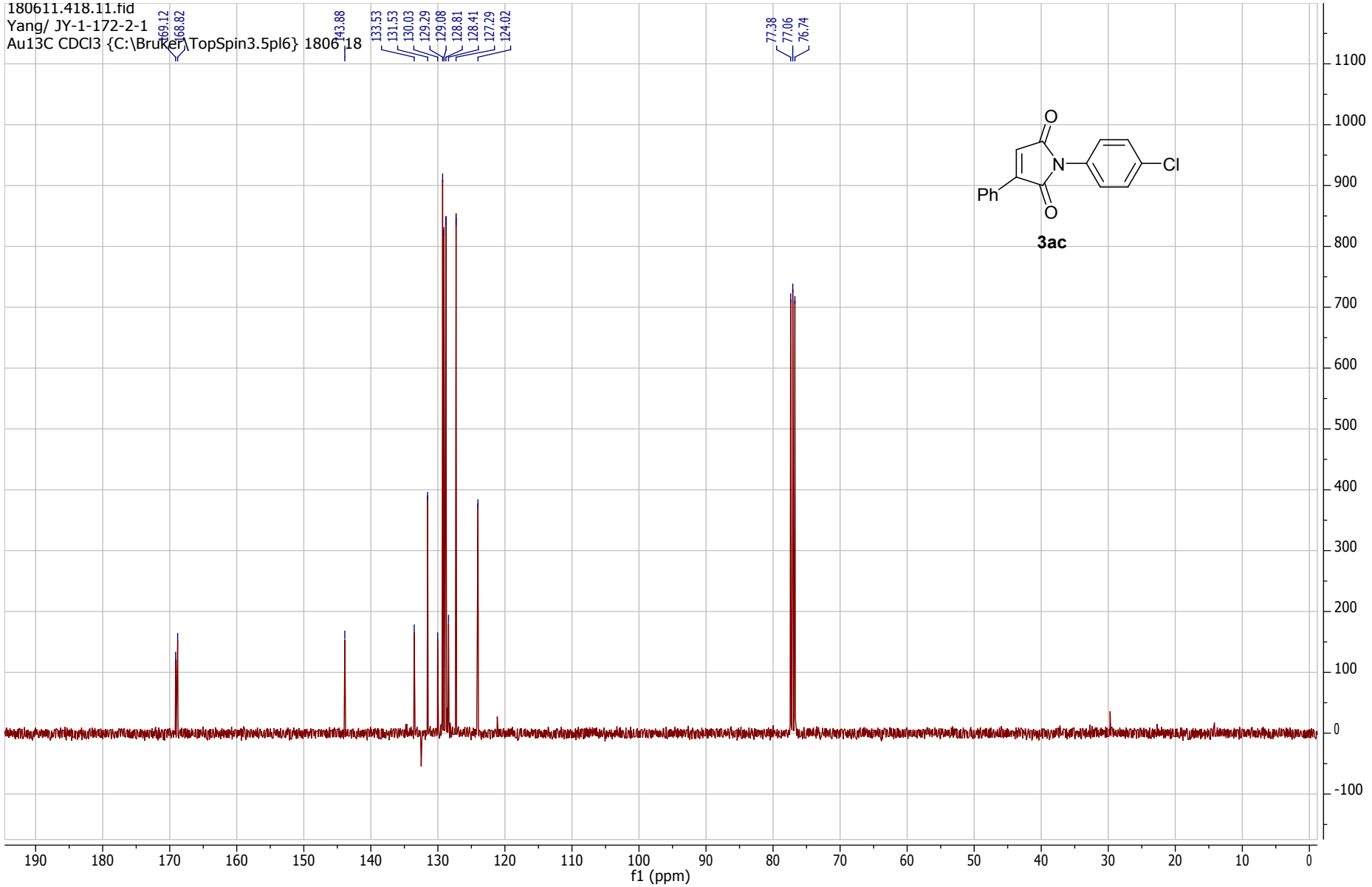

180613.440.10.fid
Yang/ JY-1-180-3-1-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 40
7.46
7.44
7.43
7.38
7.37
7.36
7.35
7.34
7.32
7.31
7.26
2.54
2.52
2.50
2.48
1.23
1.21
1.20
O
N
O
3na
A (q)
2.51
B (t)
1.21
1.94
2.89
1.00
4.04
1.39
6.00
f1 (ppm)


180613.440.11.fid
Yang/ JY-1-180-3-1-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 40
170.17
141.94
131.98
128.97
127.36
125.78
77.38 CDCl3
77.06 CDCl3
76.74 CDCl3
17.18
13.38
O
N
O
3na
f1 (ppm)

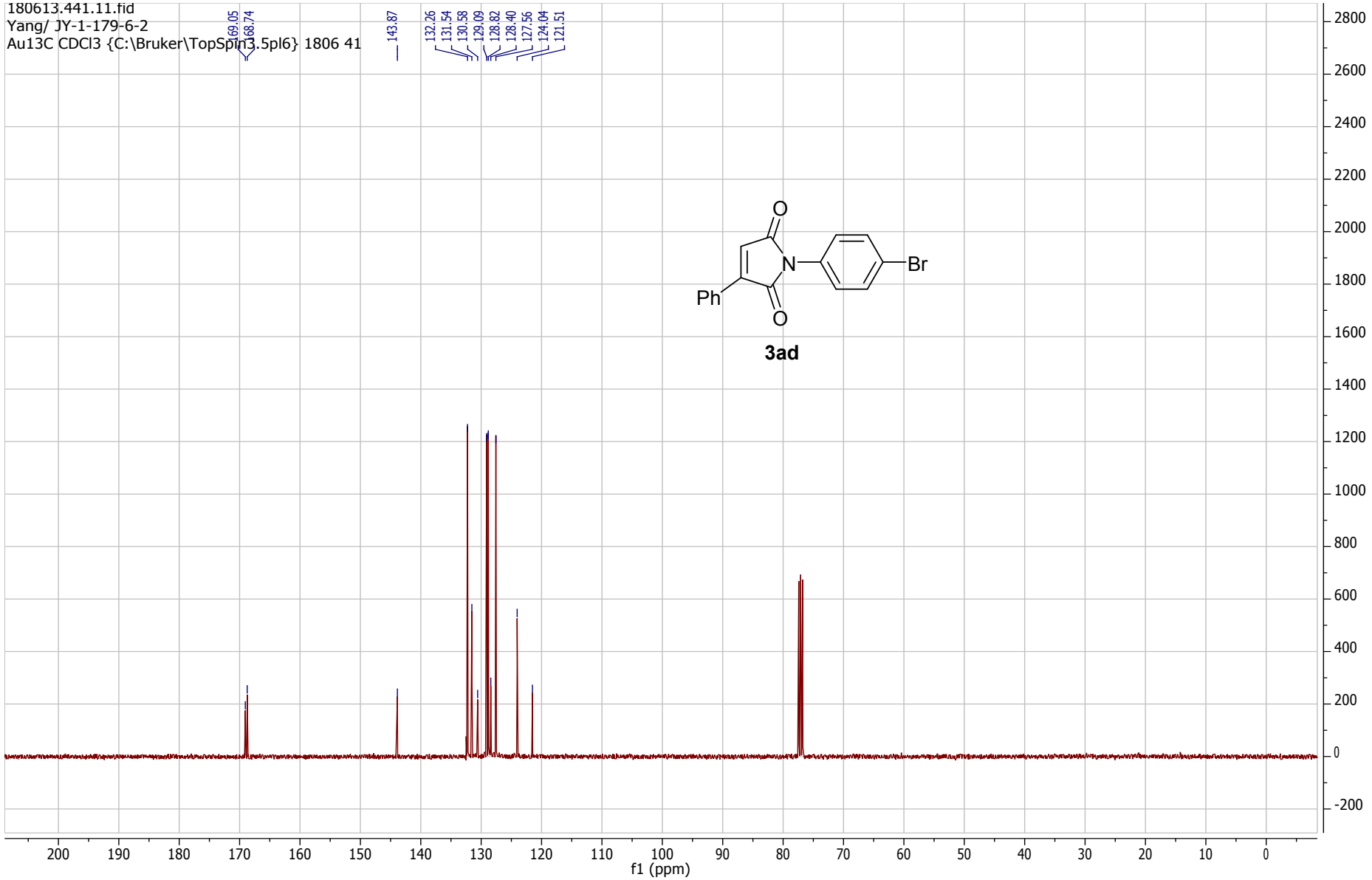

180613.432.10.fid
Yang/ JY-1-179-4-2
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 32
O
N
F
Ph
O
3ab
1.87
3.00
2.06
1.96
0.90
f1 (ppm)

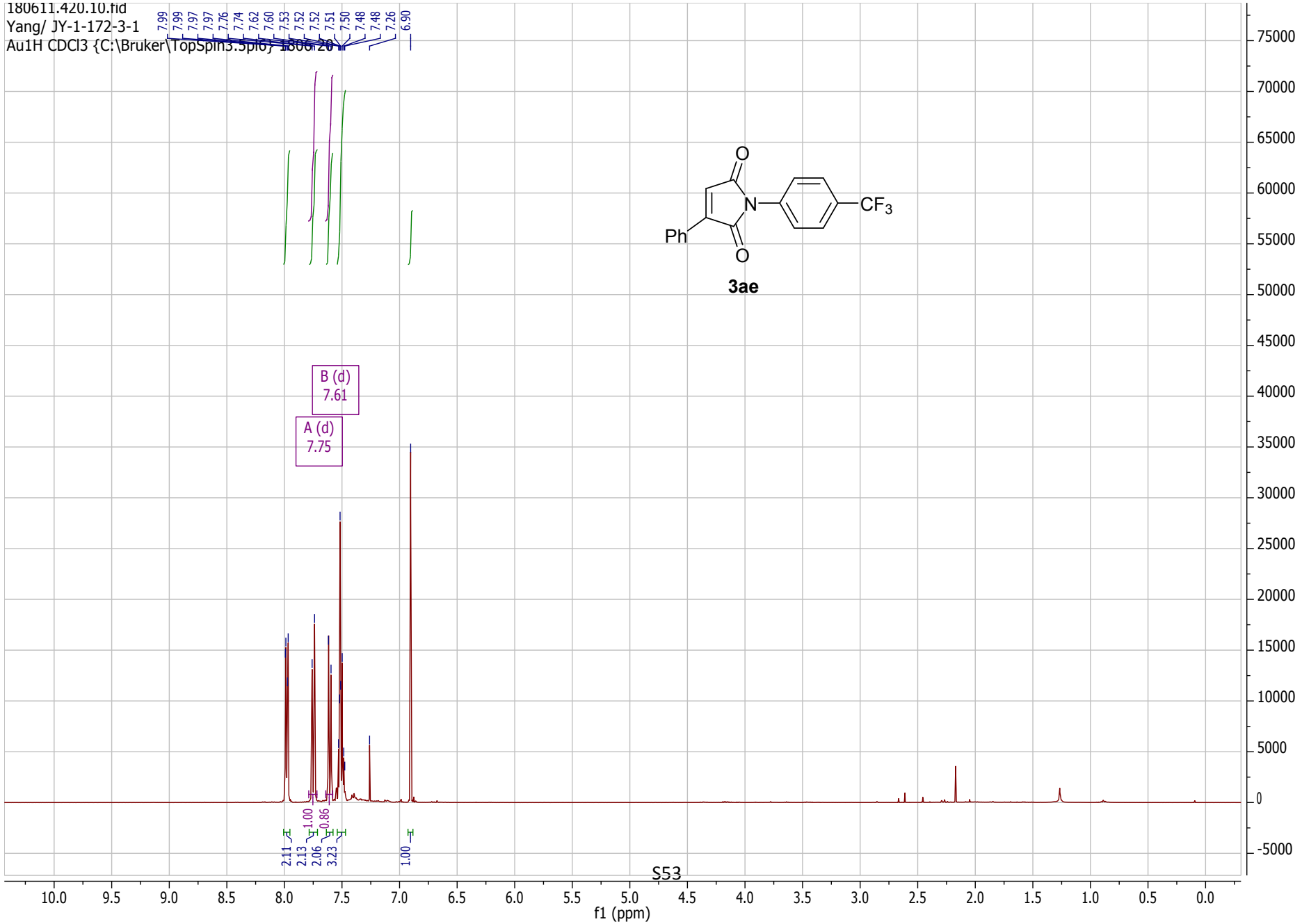

180613.432.11.fid
Yang/ JY-1-179-4-2
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 32
69.34
69.03
62.98
60.52
43.77
131.45
129.05
128.77
128.46
128.08
128.00
127.41
127.38
123.95
116.20
115.97
77.38
77.06
76.74
A (d)
161.75
C (d)
127.39
B (d)
128.04
D (d)
116.08
1.00
6.34
1.10
6.28
O
N
F
Ph
O
3ab
f1 (ppm)

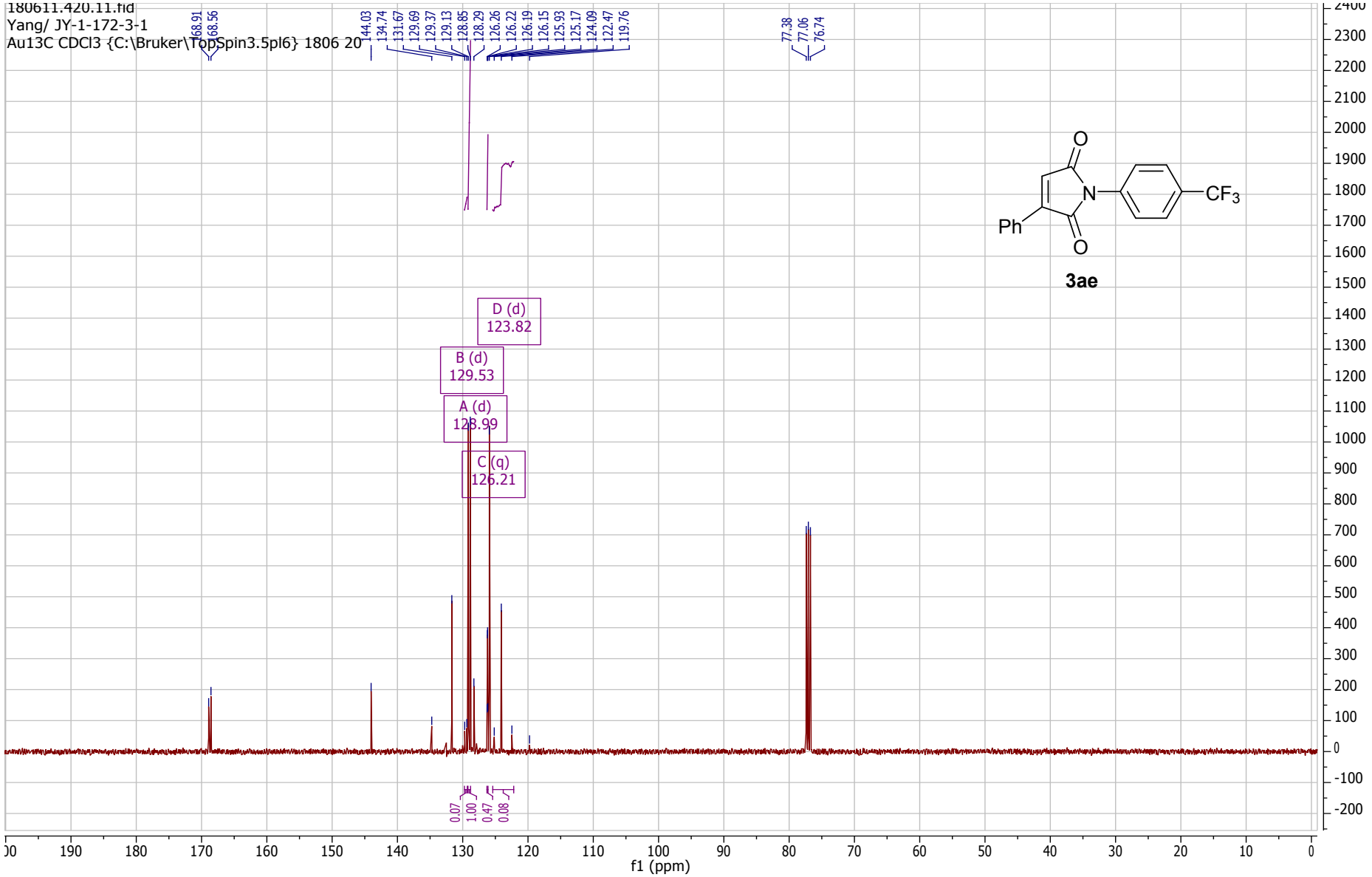

180611.418.10.fid
Yang/ JY-1-172-2-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806-18
7.98
7.97
7.96
7.95
7.50
7.50
7.46
7.44
7.38
6.87
O
N
Cl
Ph
O
3ac
2.03
5.10
2.32
1.00
f1 (ppm)

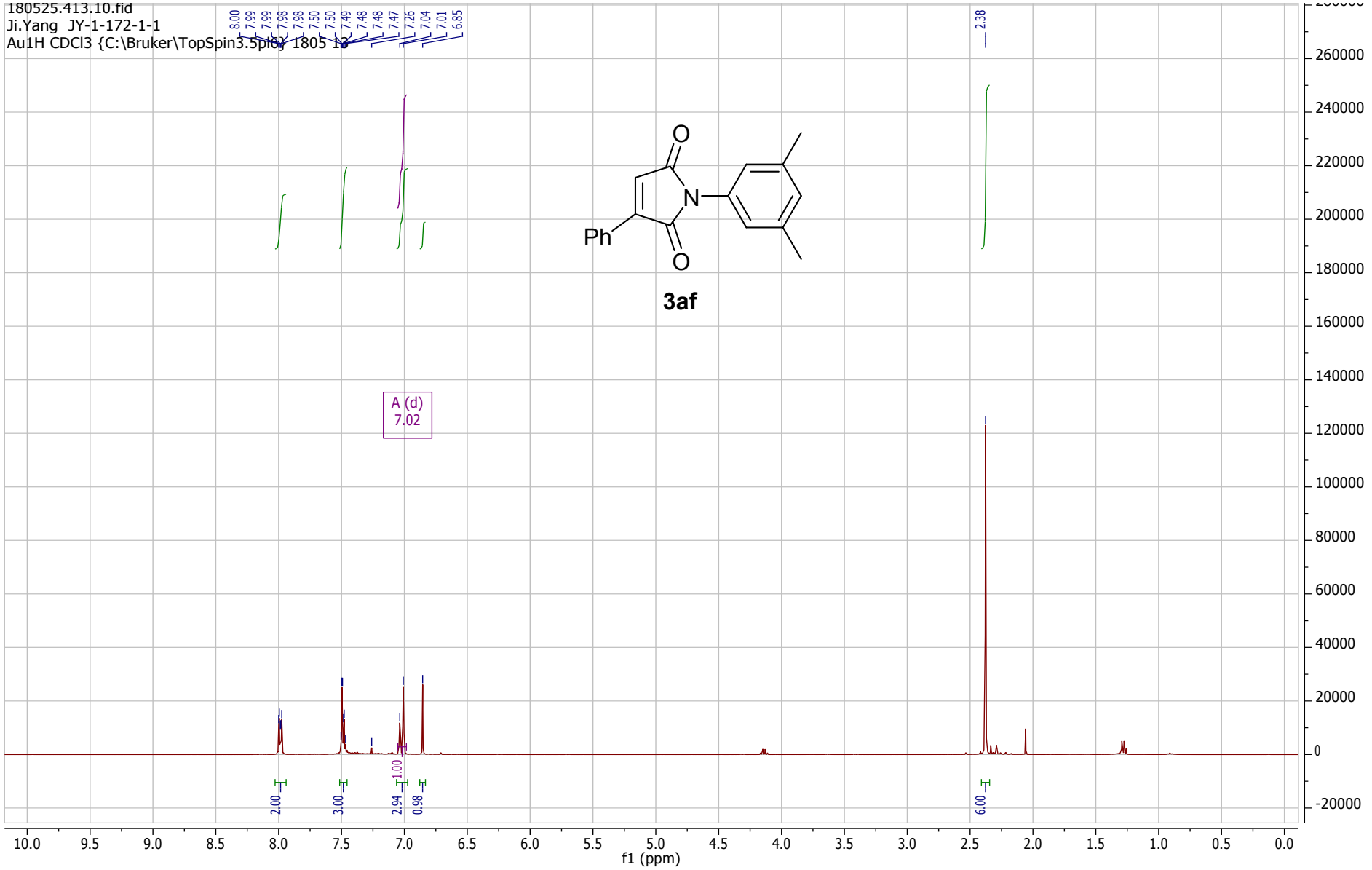

180611.418.11.fid
Yang/ JY-1-172-2-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 18
169.12
168.82
143.88
133.53
131.53
130.03
129.29
129.08
128.81
128.41
127.29
124.02
77.38
77.06
76.74
O
N
Cl
Ph
O
3ac
f1 (ppm)

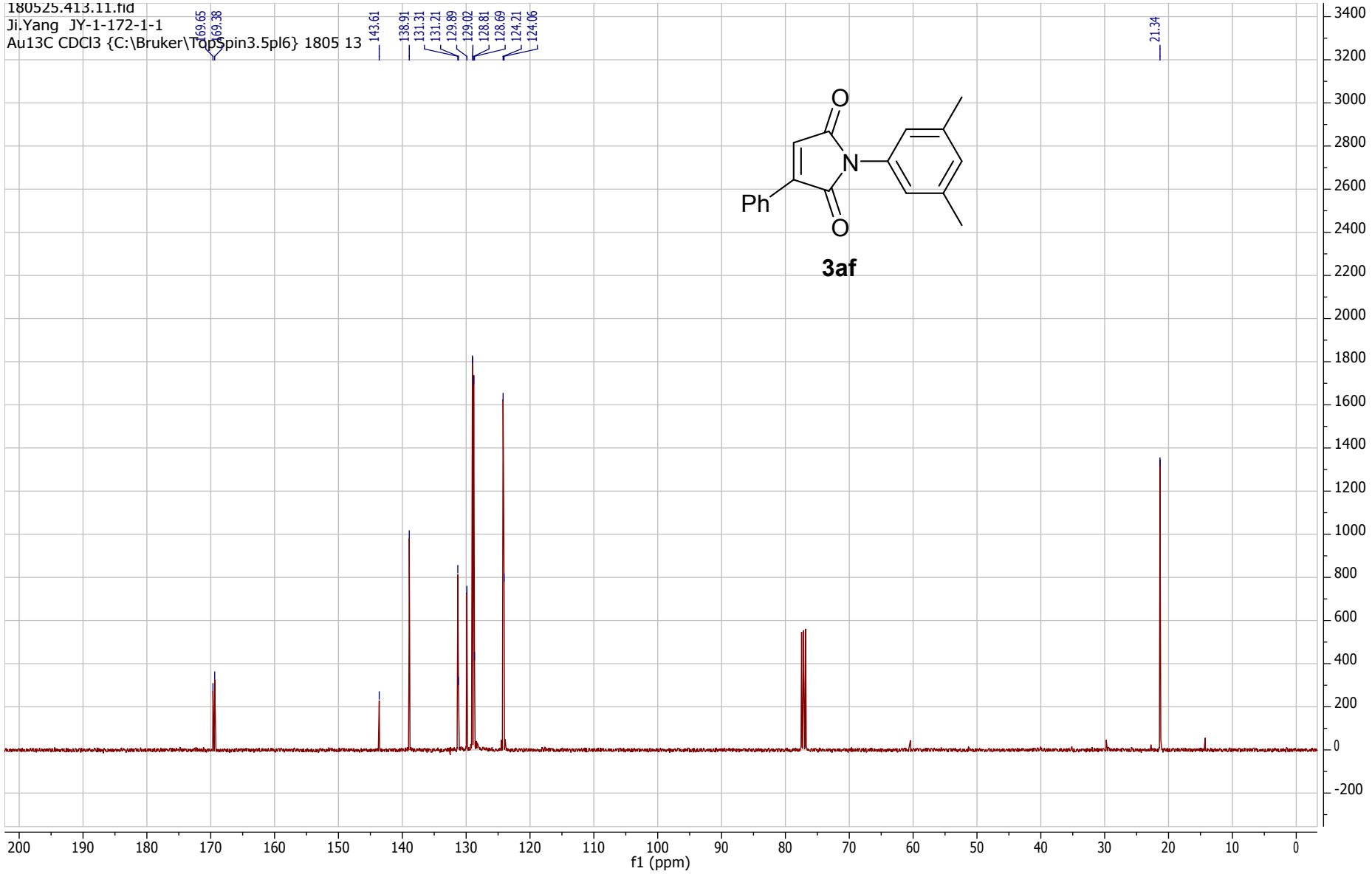

180613.441.10.fid
Yang/ JY-1-179-6-2
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 r1
7.98
7.97
7.97
7.96
7.95
7.61
7.59
7.50
7.48
7.32
7.30
6.87
O
N
Br
Ph
O
3ad
2.03
2.05
3.15
2.16
1.00
f1 (ppm)

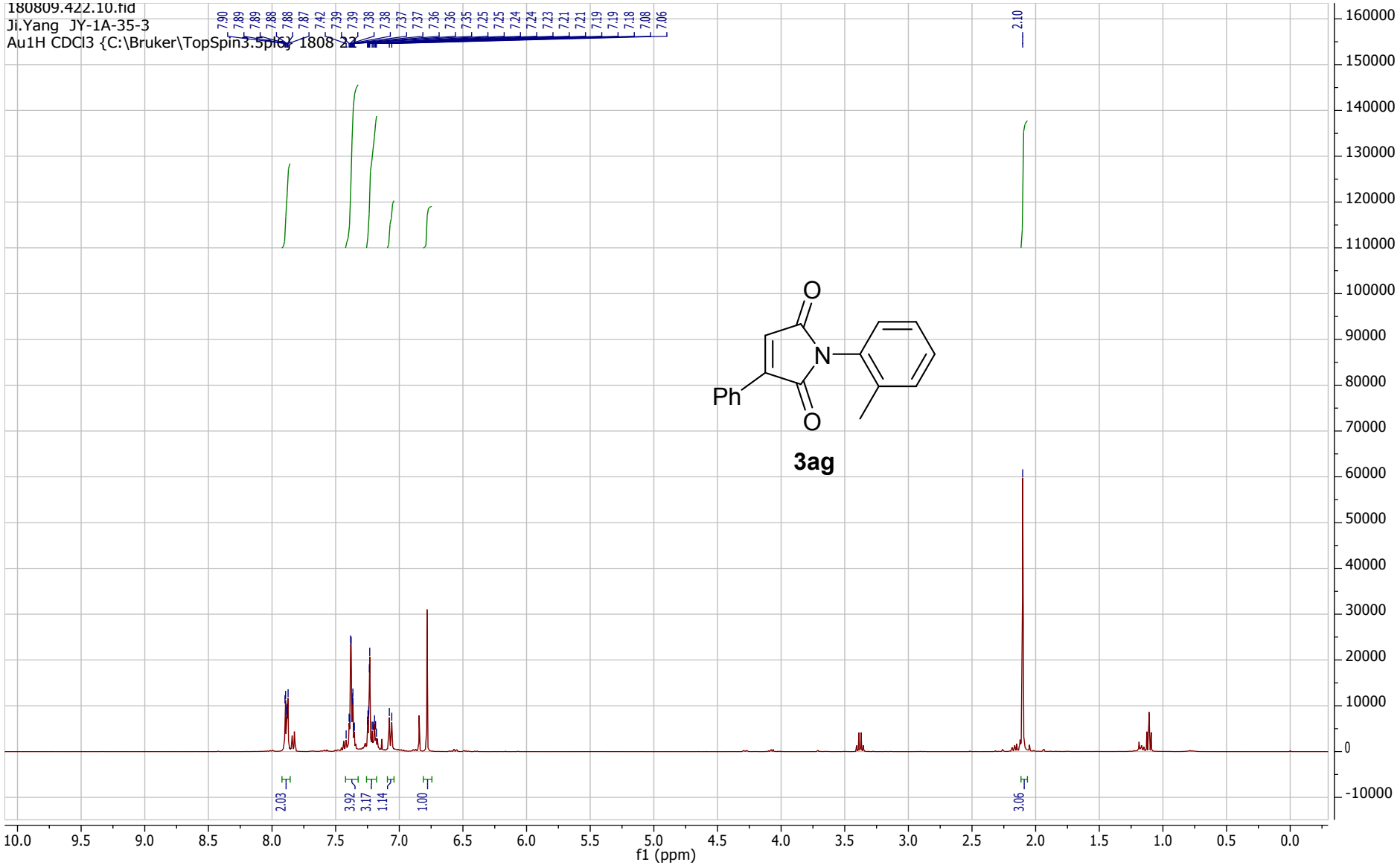

180613.441.11.fid
Yang/ JY-1-179-6-2
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 41
169.05
168.74
143.87
132.26
131.54
130.58
129.09
128.82
128.40
127.56
124.04
121.51
O
N
Br
Ph
O
3ad
f1 (ppm)

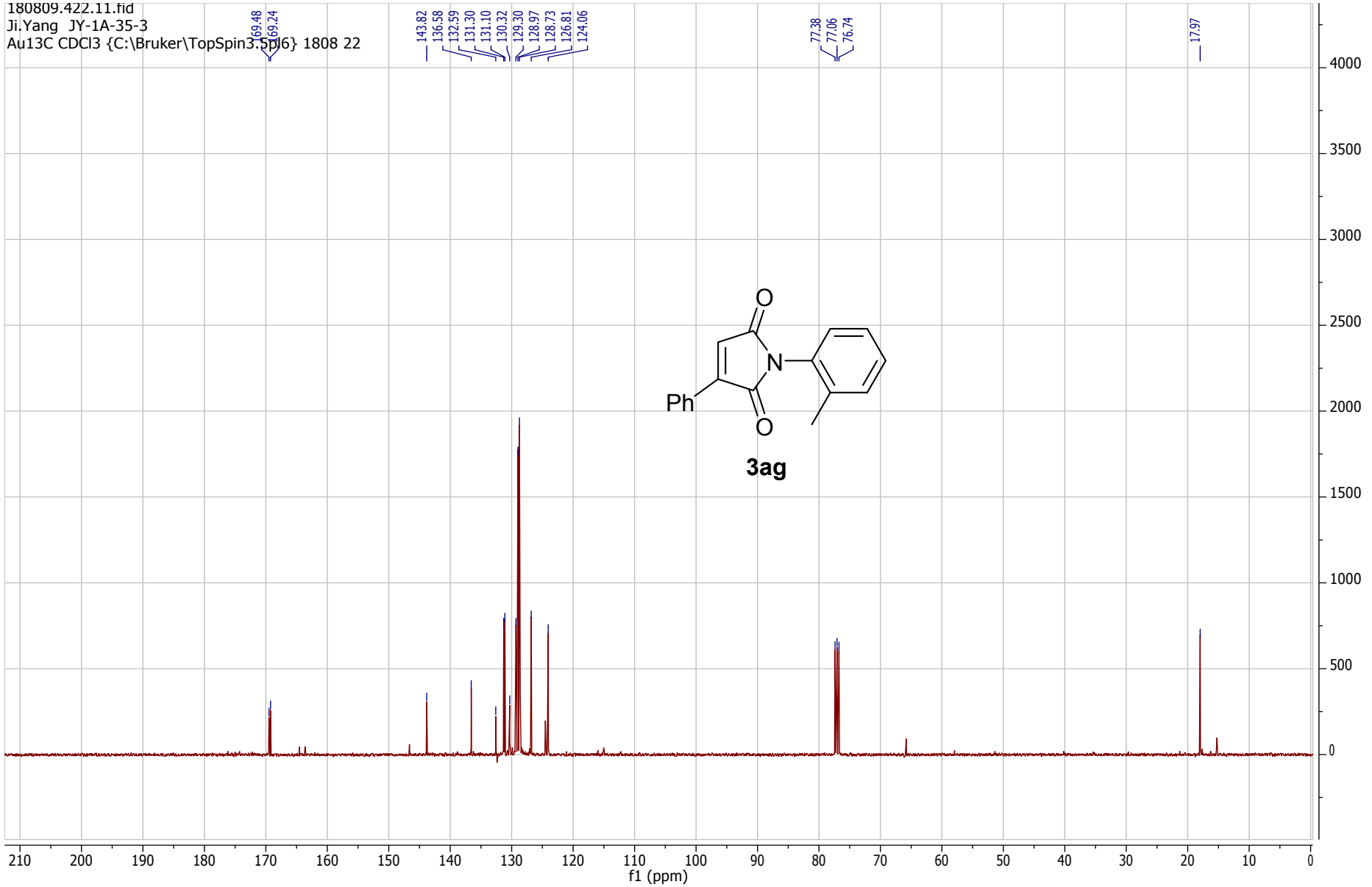

180611.420.10.fid
Yang/ JY-1-172-3-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 600/20
7.99
7.99
7.97
7.97
7.76
7.74
7.62
7.60
7.53
7.52
7.52
7.51
7.50
7.48
7.48
7.26
6.90
O
N
CF3
Ph
O
3ae
B (d)
7.61
A (d)
7.75
2.11
2.13
1.00
2.06
0.86
3.23
1.00
S53
f1 (ppm)

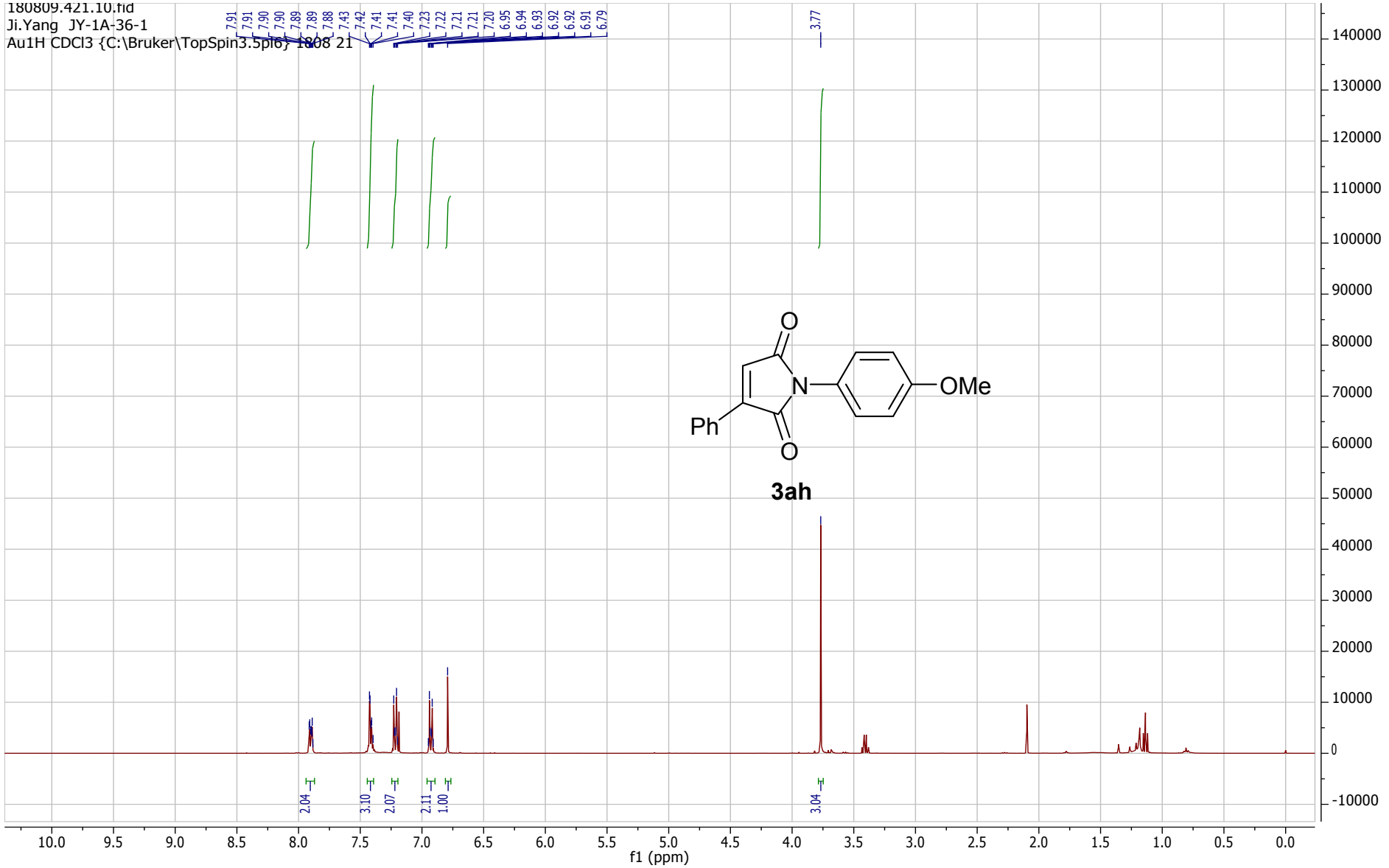

180611.420.11.fid
Yang/ JY-1-172-3-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 20
168.91
168.56
144.03
134.74
131.67
129.69
129.37
129.13
128.85
128.29
126.26
126.22
126.19
126.15
125.93
125.17
124.09
122.47
119.76
77.38
77.06
76.74
D (d)
123.82
B (d)
129.53
A (d)
128.99
C (q)
126.21
0.07
1.00
0.47
0.08
O
N
CF3
Ph
O
3ae
f1 (ppm)

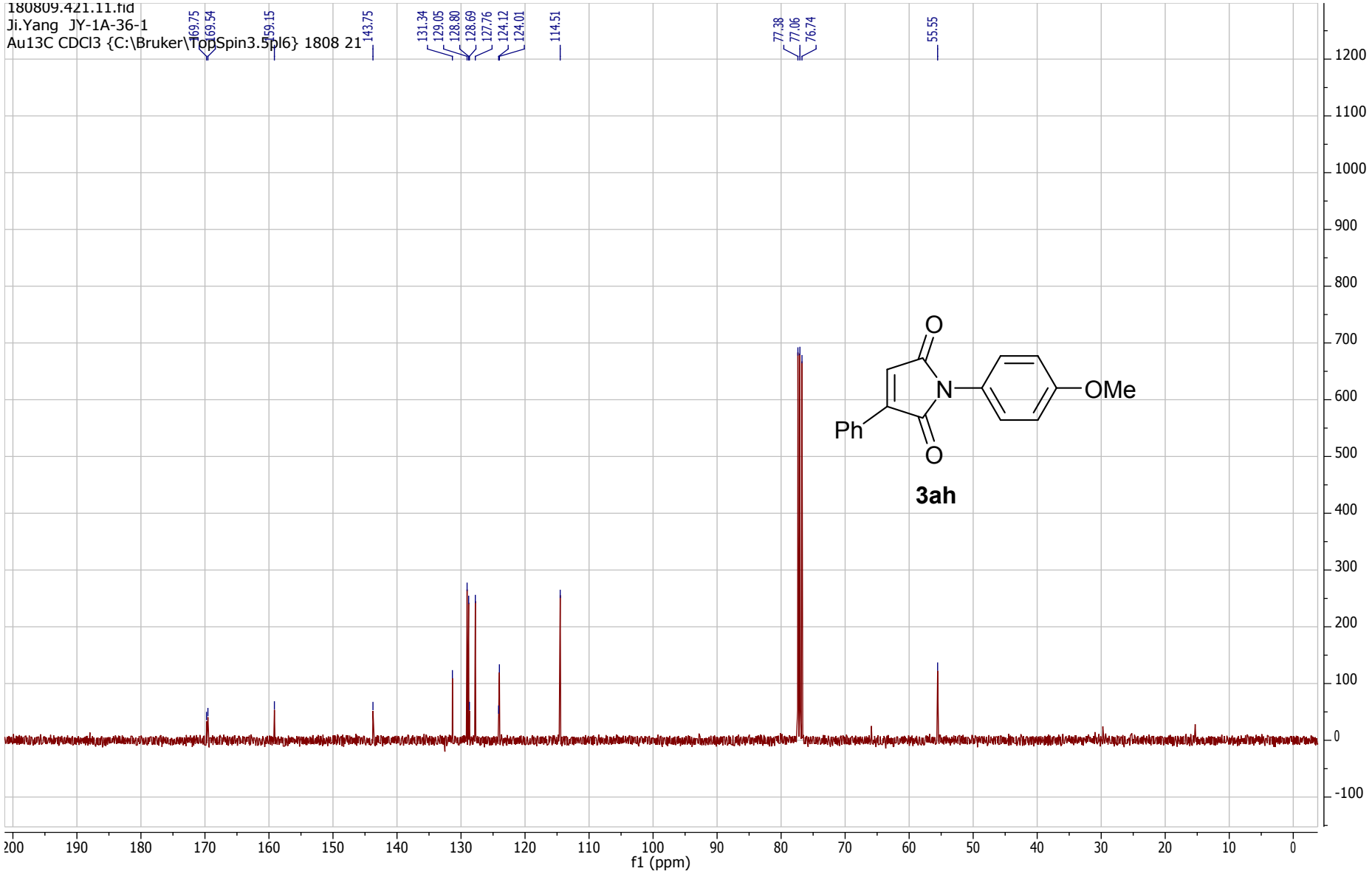

180525.413.10.fid
Ji.Yang JY-1-172-1-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1805 1
8.00
7.99
7.99
7.98
7.98
7.50
7.50
7.49
7.48
7.48
7.47
7.26
7.04
7.01
6.85
2.38
O
N
Ph
O
3af
A (d)
7.02
2.00
3.00
2.94
1.00
0.98
6.00
f1 (ppm)


180525.413.11.fid
Ji.Yang JY-1-172-1-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1805 13
169.65
169.38
143.61
138.91
131.31
131.21
129.89
129.02
128.81
128.69
124.21
124.06
21.34
O
N
Ph
O
3af
f1 (ppm)


180809.422.10.fid
Ji.Yang JY-1A-35-3
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1808 22
7.90 7.89 7.89 7.88 7.88 7.87 7.42 7.39 7.39 7.38 7.38 7.37 7.37 7.36 7.36 7.35 7.25 7.25 7.24 7.24 7.23 7.21 7.21 7.19 7.19 7.18 7.08 7.06
2.10
O
N
Ph
O
3ag
2.03
3.92
3.17
1.14
1.00
3.06
f1 (ppm)


180809.422.11.fid
Ji.Yang JY-1A-35-3
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1808 22
169.48
169.24
143.82
136.58
132.59
131.30
131.10
130.32
129.30
128.97
128.73
126.81
124.06
77.38
77.06
76.74
17.97
O
N
Ph
O
3ag
f1 (ppm)


180809.421.10.fid
Ji.Yang JY-1A-36-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1808 21
7.91
7.91
7.90
7.90
7.89
7.89
7.88
7.43
7.42
7.41
7.41
7.40
7.23
7.22
7.21
7.21
7.20
6.95
6.94
6.93
6.92
6.92
6.91
6.79
3.77
O
N
OMe
Ph
O
3ab
2.04
3.10
2.07
2.11
1.00
3.04
f1 (ppm)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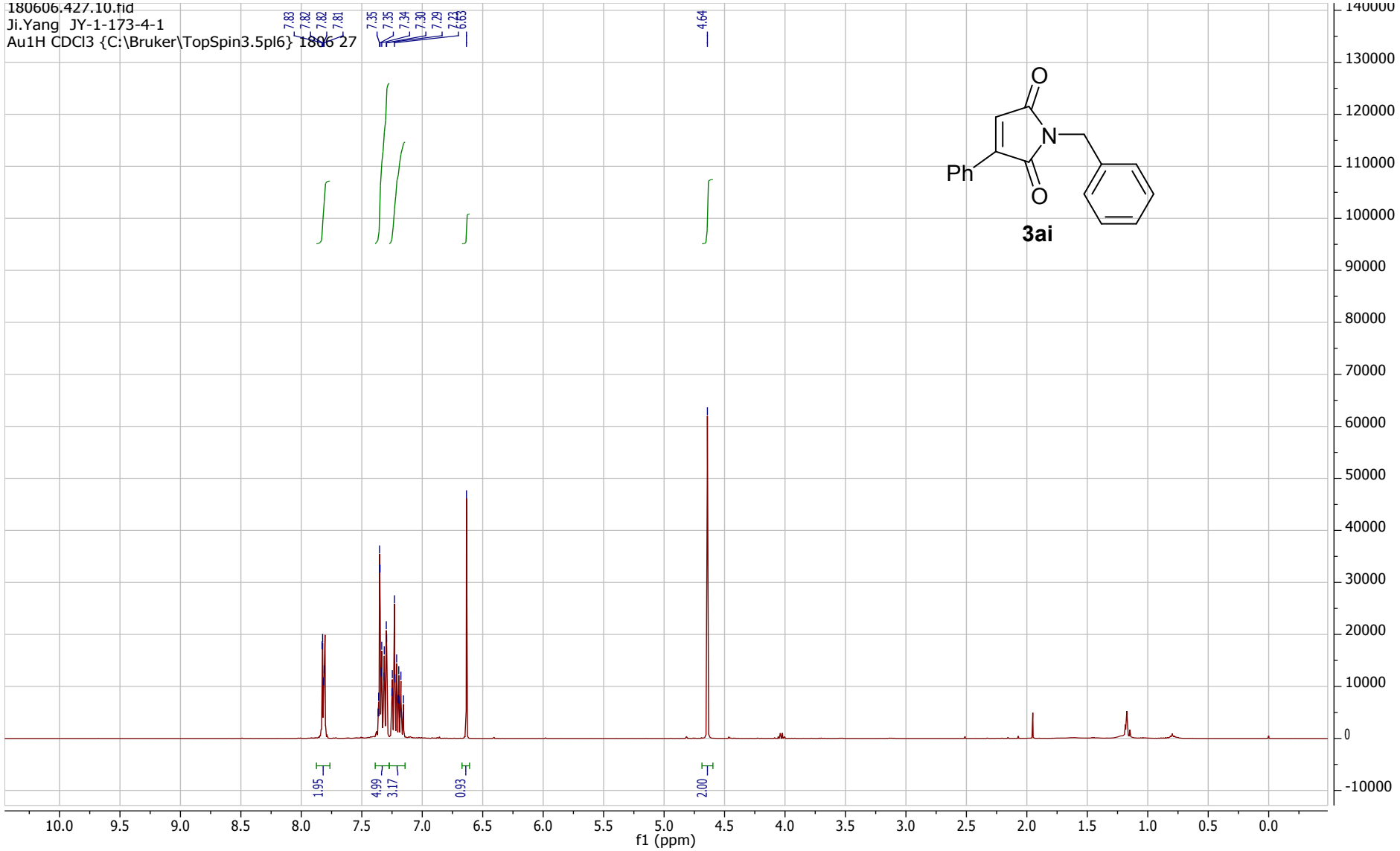
Ji.Yang JY-1A-36-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1808 21
169.75
169.54
159.15
143.75
131.34
129.05
128.80
128.69
127.76
124.12
124.01
114.51
77.38
77.06
76.74
55.55
O
N
OMe
Ph
O
3ah
f1 (ppm)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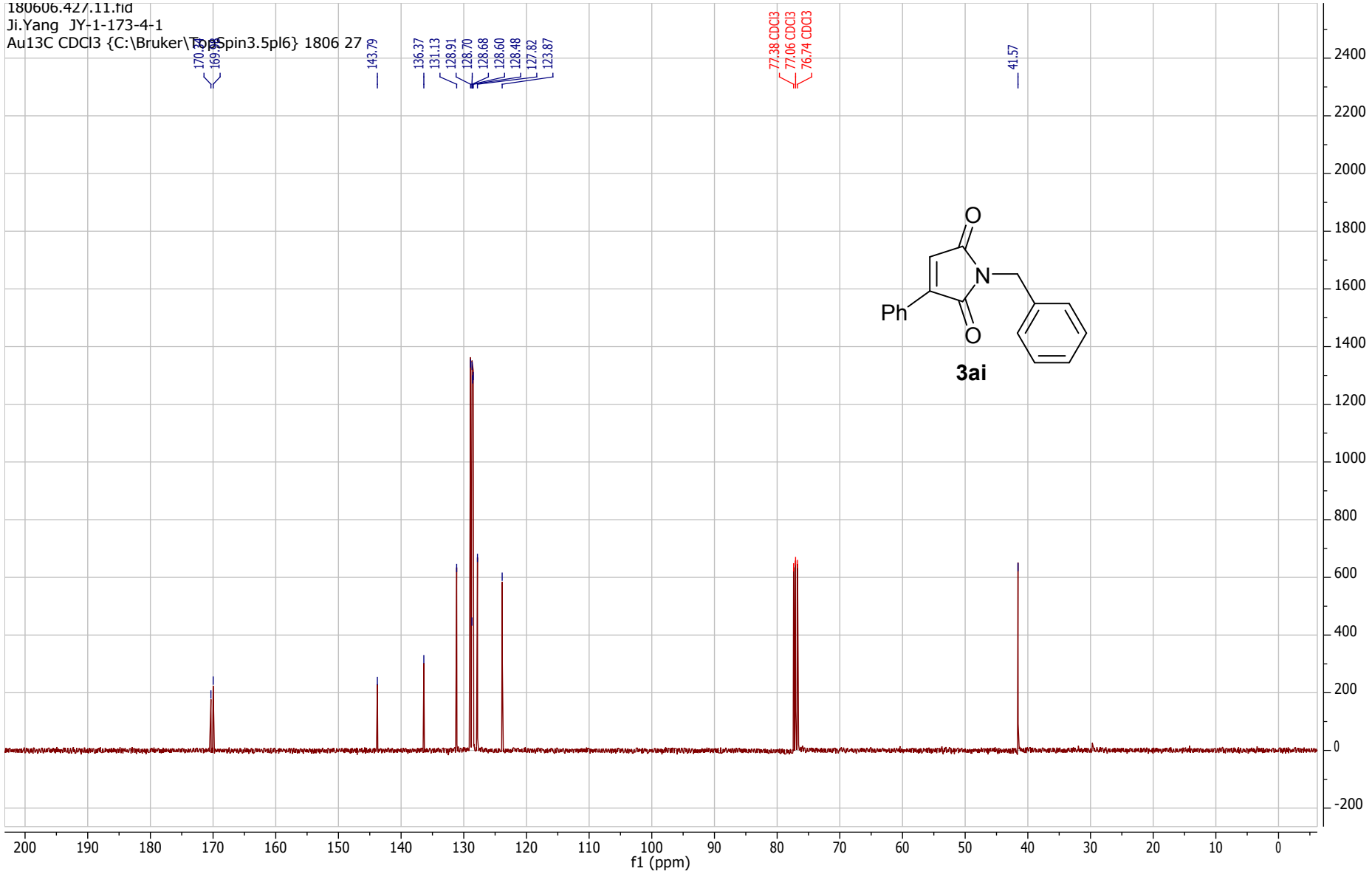
Ji.Yang JY-1-173-4-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 27
7.83
7.82
7.82
7.81
7.35
7.35
7.34
7.30
7.29
7.23
6.63
4.64
O
N
Ph
O
3ai
1.95
4.99
3.17
0.93
2.00
f1 (ppm)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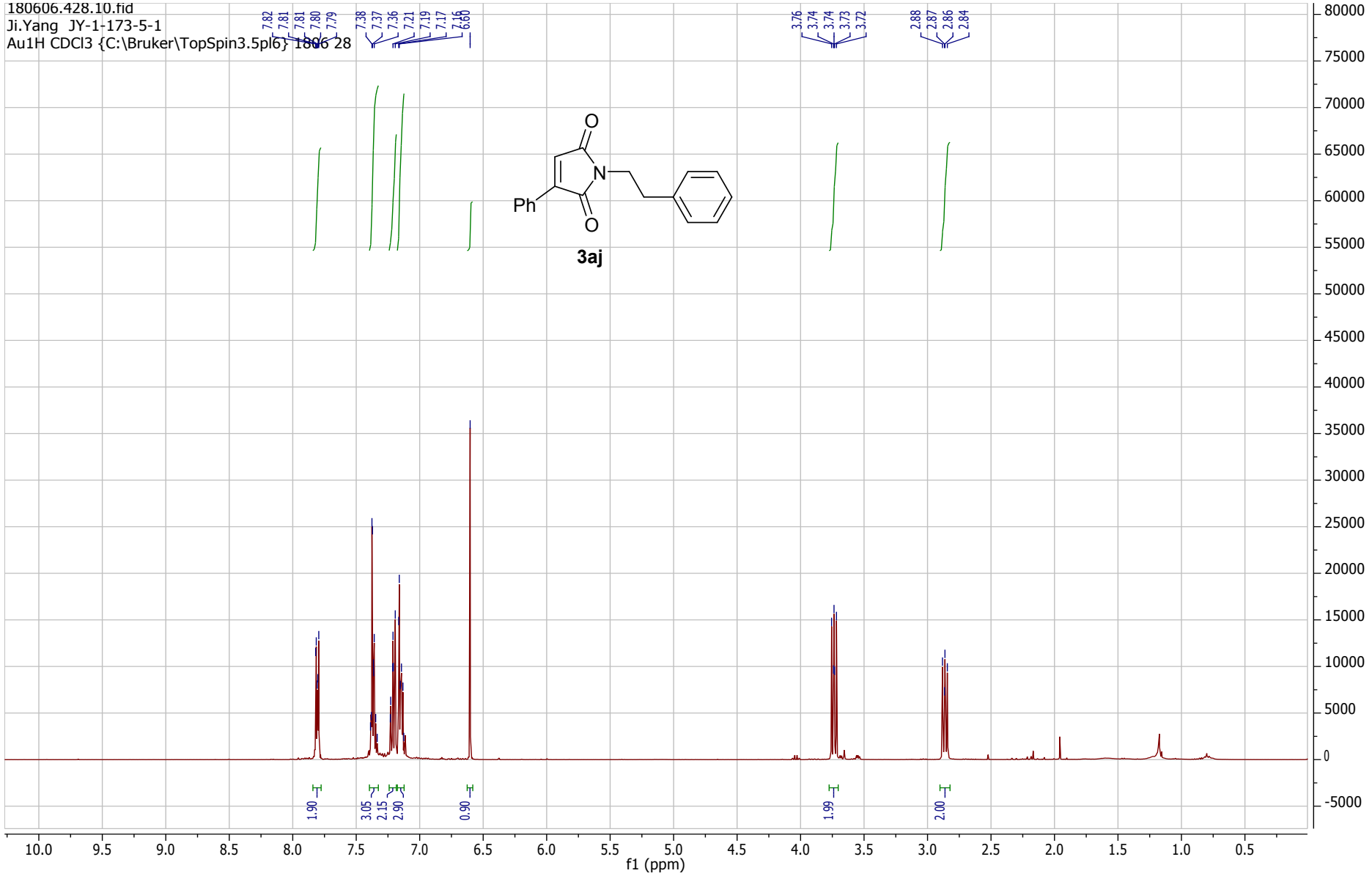
Ji.Yang JY-1-173-4-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 27
170.23
169.83
143.79
136.37
131.13
128.91
128.70
128.68
128.60
128.48
127.82
123.87
77.38 CDCl3
77.06 CDCl3
76.74 CDCl3
41.57
O
N
Ph
O
3ai
f1 (ppm)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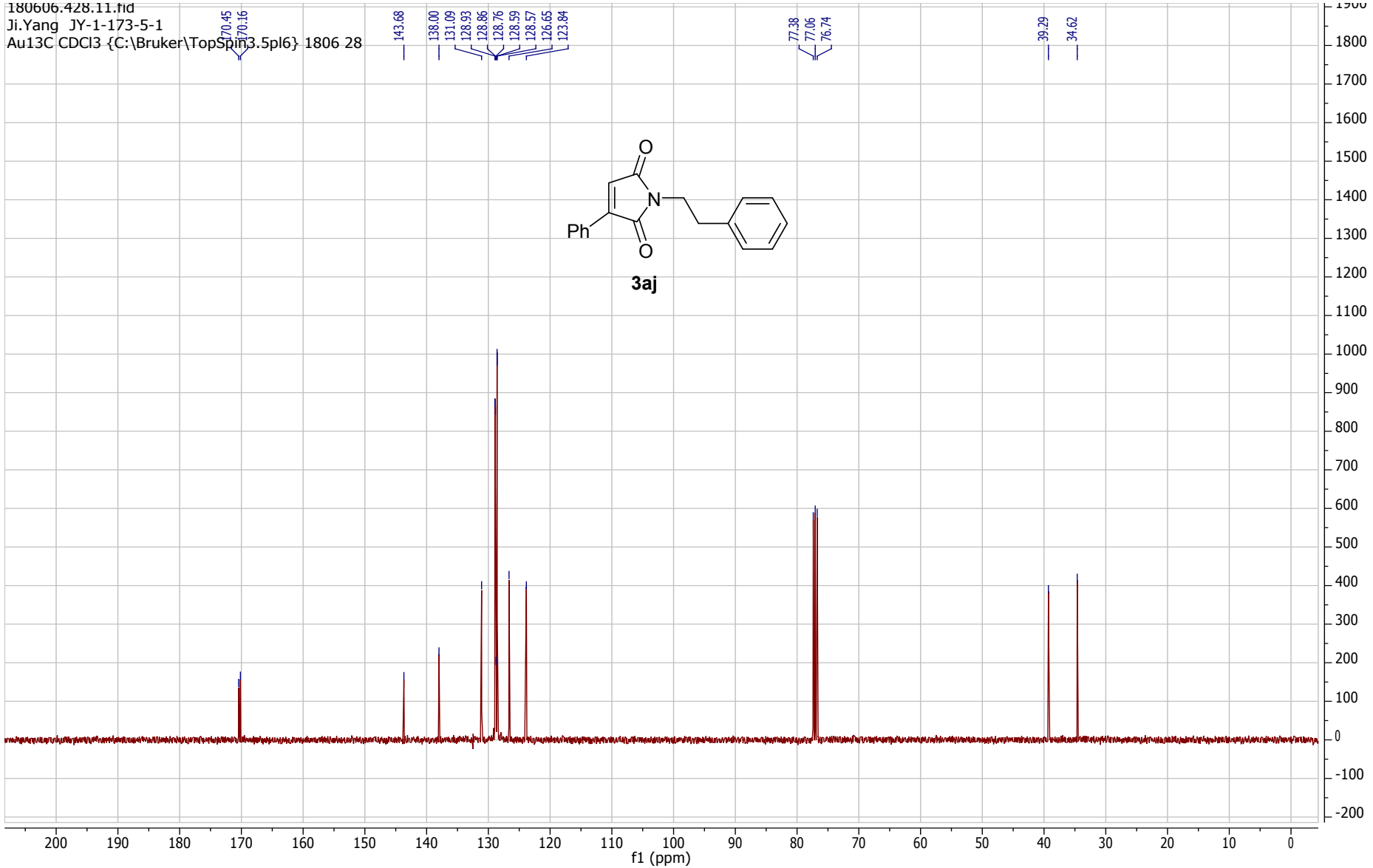
Ji.Yang JY-1-173-5-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 28
7.82 7.81 7.81 7.80 7.79 7.38 7.37 7.36 7.21 7.19 7.17 7.16 6.60
3.76 3.74 3.74 3.73 3.72
2.88 2.87 2.86 2.84
O
N
Ph
O
3aj
1.90 3.05 2.15 2.90 0.90 1.99 2.00
f1 (ppm)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Ji.Yang JY-1-173-5-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 28
170.45
170.16
143.68
138.00
131.09
128.93
128.86
128.76
128.59
128.57
126.65
123.84
77.38
77.06
76.74
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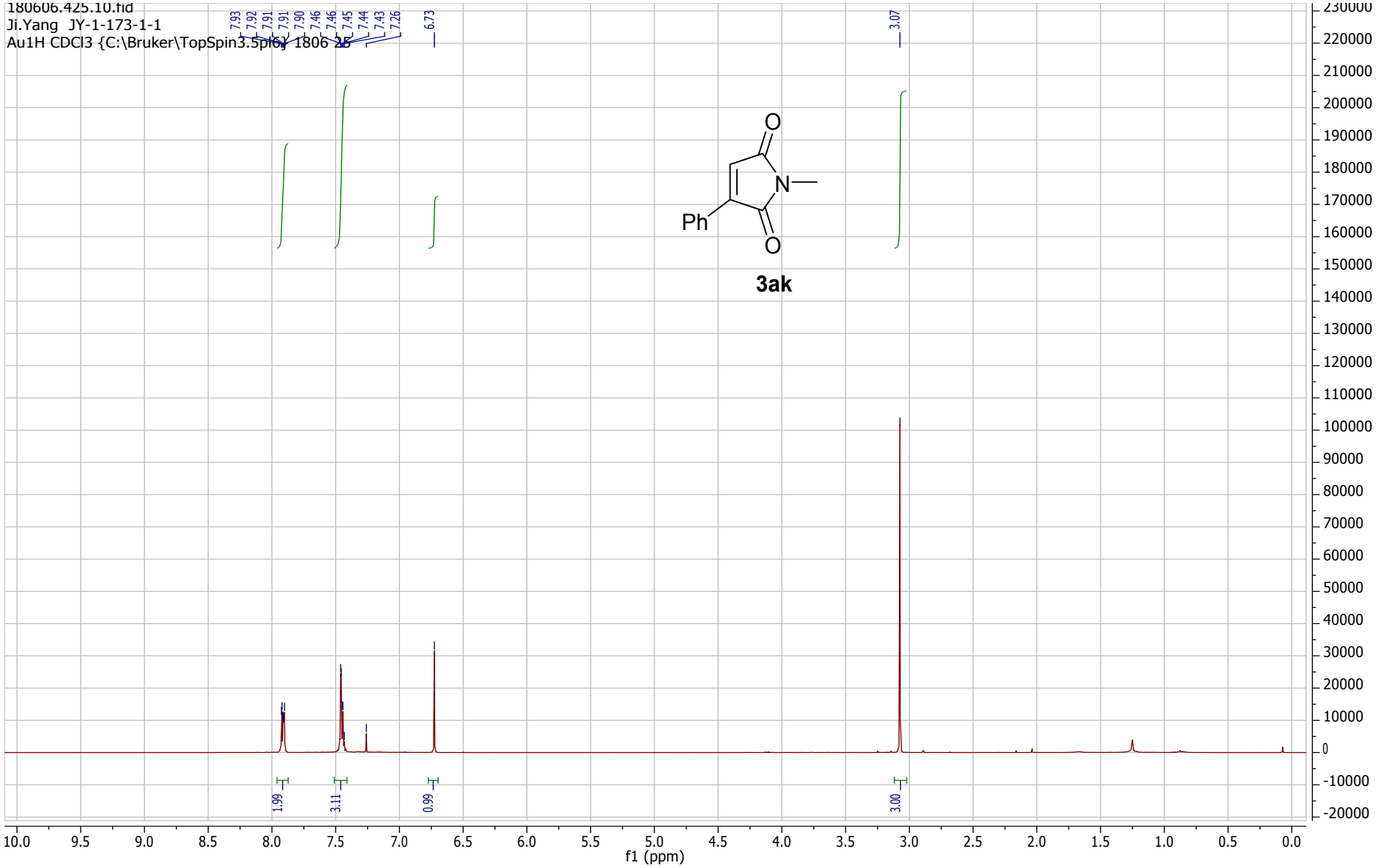
34.62
O
N
Ph
O
3aj
f1 (ppm)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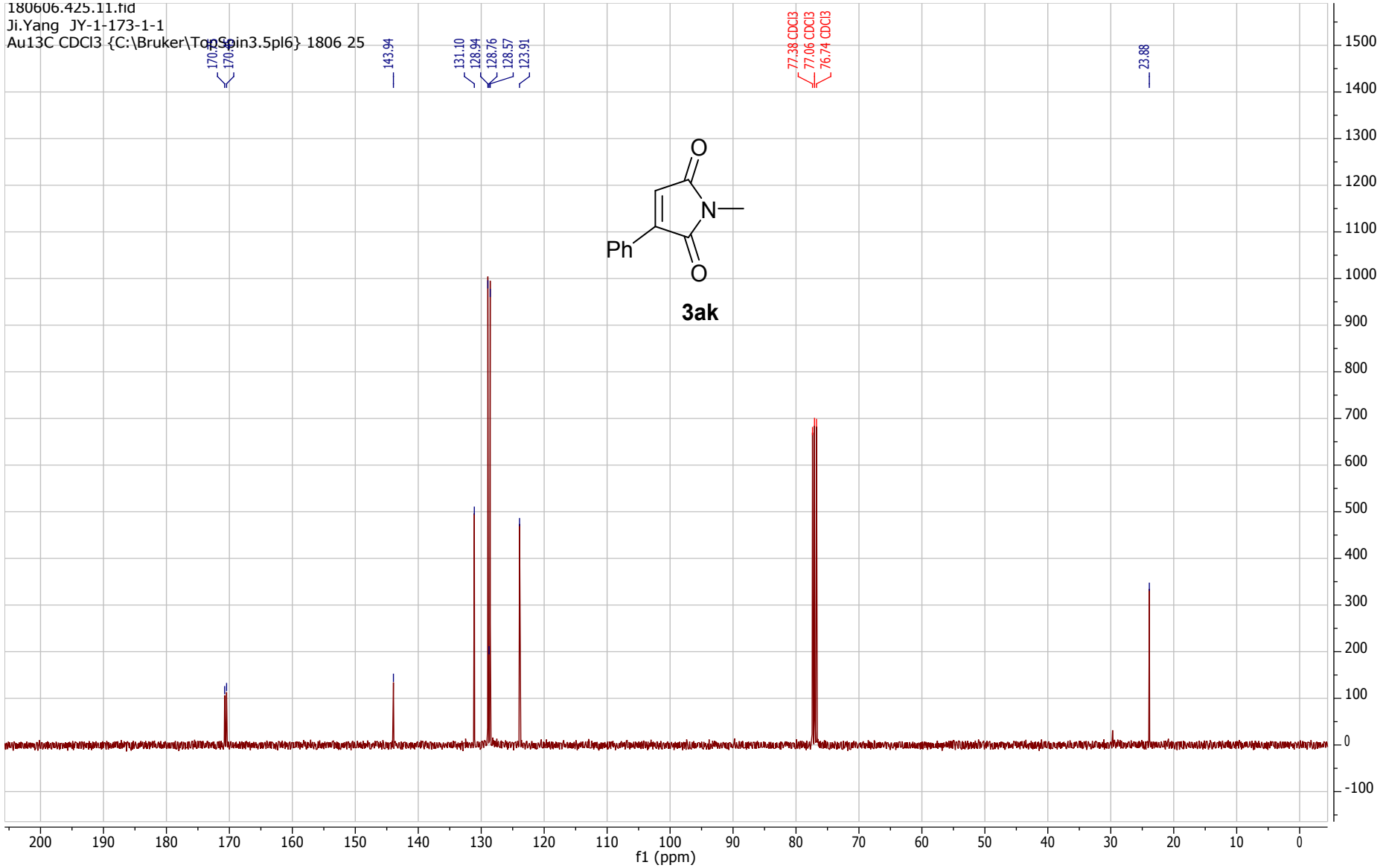
Ji.Yang JY-1-173-1-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 25
7.93
7.92
7.91
7.91
7.90
7.46
7.46
7.45
7.44
7.43
7.26
6.73
3.07
O
N
Ph
O
3ak
1.99
3.11
0.99
3.00
f1 (ppm)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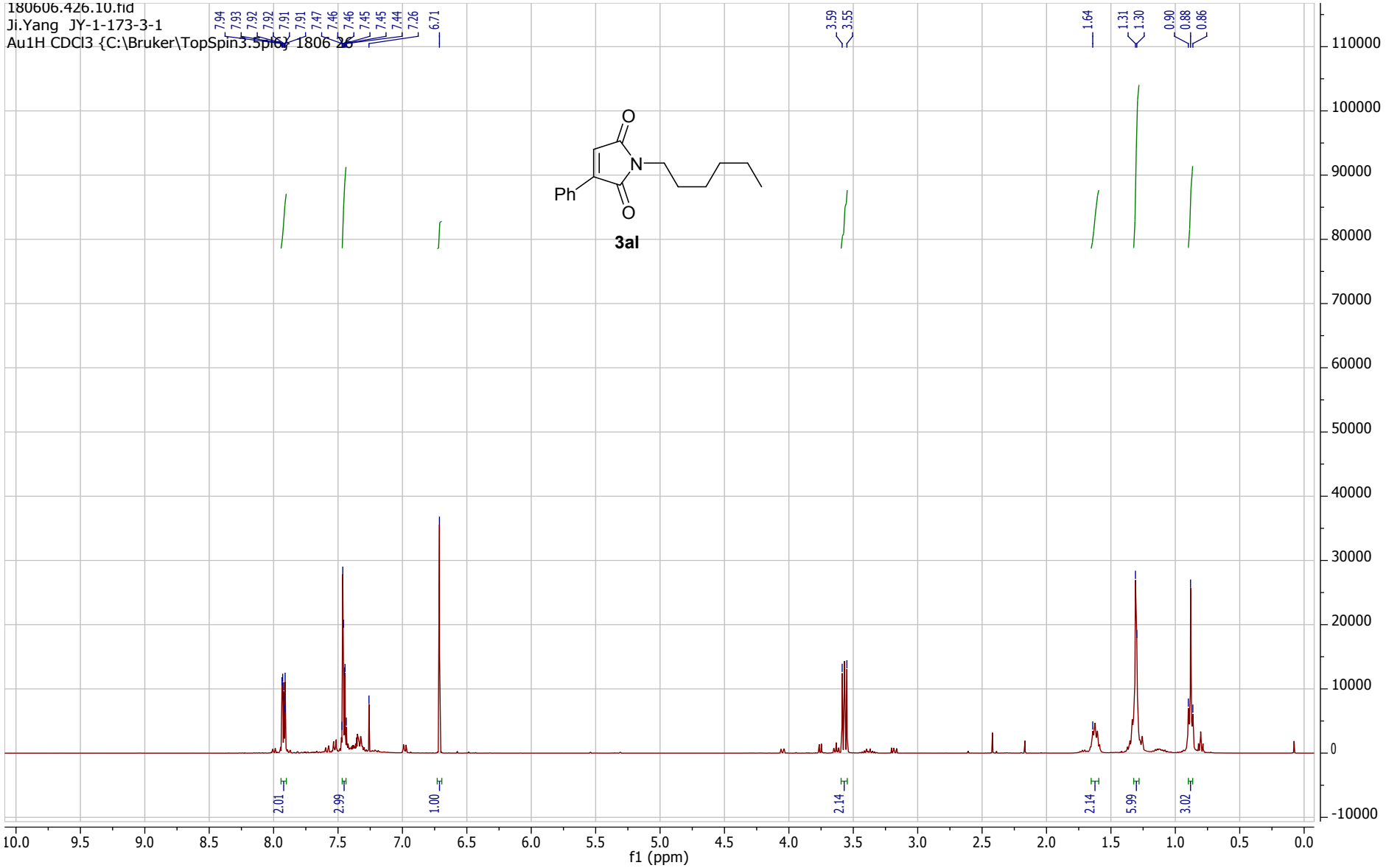
Ji.Yang JY-1-173-1-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 25
170.47
170.41
143.94
131.10
128.94
128.76
128.57
123.91
77.38 CDCl3
77.06 CDCl3
76.74 CDCl3
23.88
O
N
Ph
O
3ak
f1 (ppm)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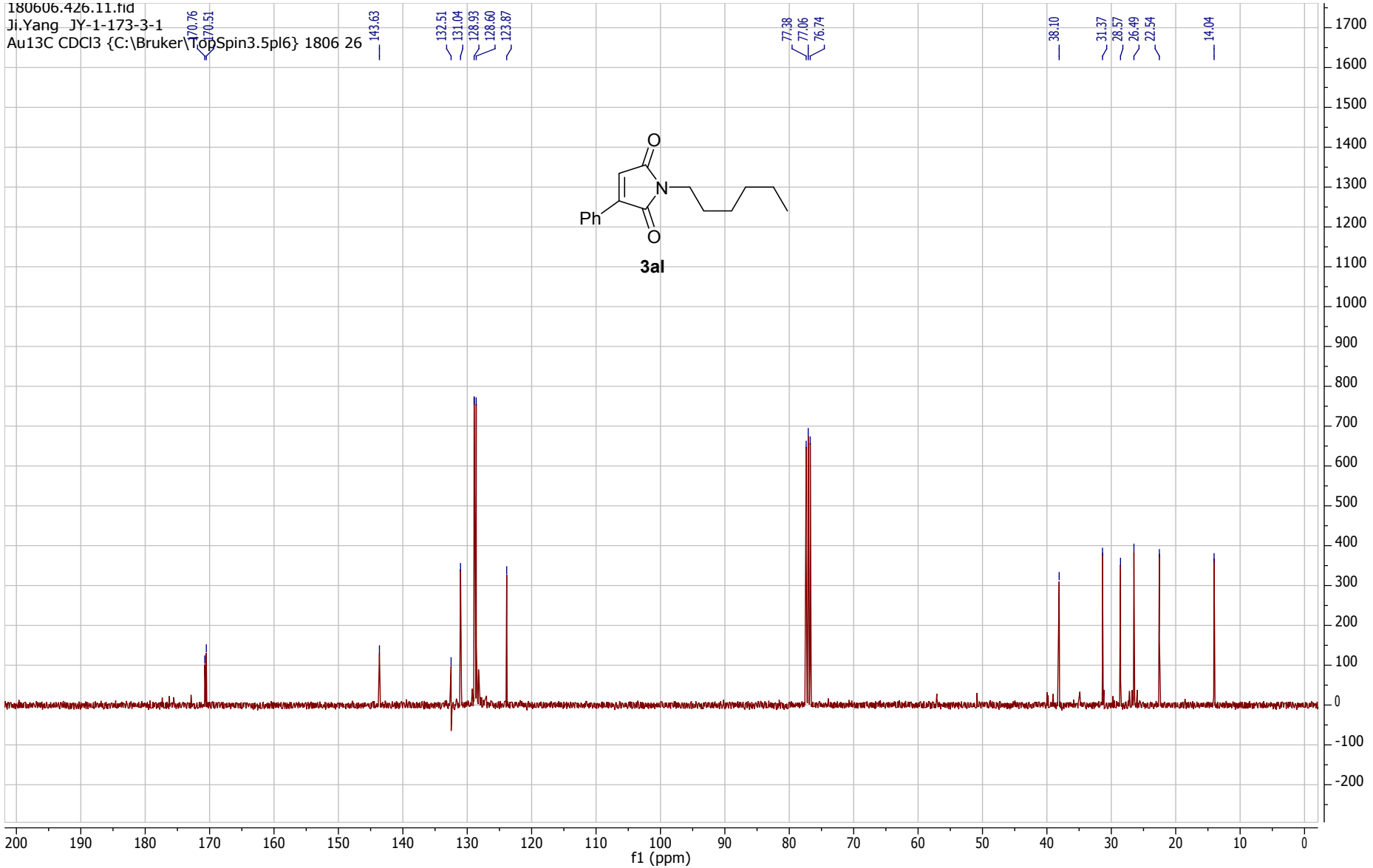
Ji.Yang JY-1-173-3-1
Au1H CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 26
7.94
7.93
7.92
7.92
7.91
7.91
7.47
7.46
7.46
7.45
7.45
7.44
7.26
6.71
3.59
3.55
1.64
1.31
1.30
0.90
0.88
0.86
O
N
Ph
O
3al
2.01
2.99
1.00
2.14
2.14
5.99
3.02
f1 (ppm)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Ji.Yang JY-1-173-3-1
Au13C CDCl3 {C:\Bruker\TopSpin3.5pl6} 1806 26
170.76
170.51
143.63
132.51
131.04
128.93
128.60
123.87
77.38
77.06
76.74
38.10
31.37
28.57
26.49
22.54
14.04
O
N
Ph
O
3al
f1 (ppm)