Supporting information for

Visible-Light-Induced Cascade Radical Cyclization to Access Sulfamoylated Indole[2, 1-a]isoquinoline Benzo[4,5]imidazo[2,1-*a*]isoquinolin-6(5*H*)-ones

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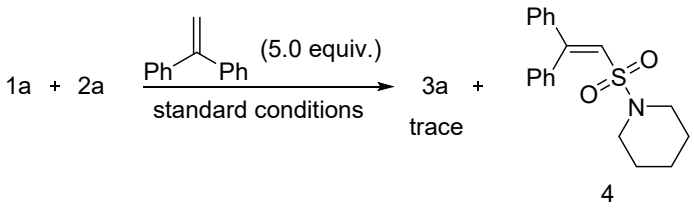
1. The radical trapping experiments



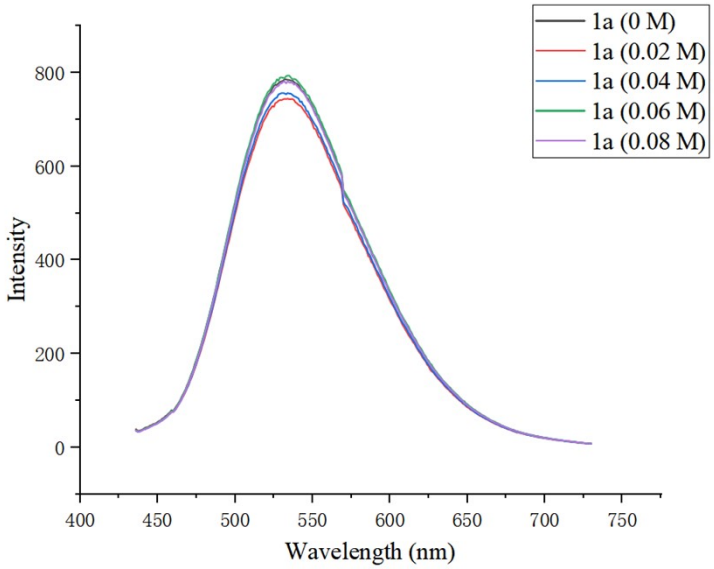
Add **1a** (0.20 mmol), **2a** (0.46 mmol), 1,1-stilbene (1.2 mmol, 4.0 equiv.) to the sealed tube and dry DMAc (1.0 mL) , The reaction mixture was evacuated and with pure N_2 for three times and stirred under the irradiation of 36 W blue LEDs at room temperature for 1 h. After irradiation, Then, the mixture was diluted with water (10 mL) and extracted with ethyl acetate (5 mL × 3). The combined organic phase was washed with brine, dried over Na_2SO_4 and concentrated under reduced pressure to give a residue which was purified by silica gel column chromatography to afford the compound **3a** (trace) and compound **4** (27 mg, 8% yield). Characteristic data for compound **4**: : ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.41-7.30 (m, 8H), 7.24 (d, J = 7.6 Hz, 2H), 6.63 (s, 1H), 3.05 (t, J = 4.6 Hz, 4H), 1.54-1.49 (m, 6H).

2. Stern-Volmer Luminescence Quenching Analysis

Emission intensities were recorded using an Agilent Cary Eclipse Fluorescence Spectrophotometer. First, the emission intensity of 1a solutions was observed at 532 nm. The solutions were irradiated at 426 nm (Maximum absorption wavelength of 1a) and fluorescence was measured from 436 nm to 730 nm. The solution of 4-CzIPN-Br (1 mM, 10 mL), 1a (1 M, 10 mL) and 2a (1 M, 10 mL) were prepared in air.

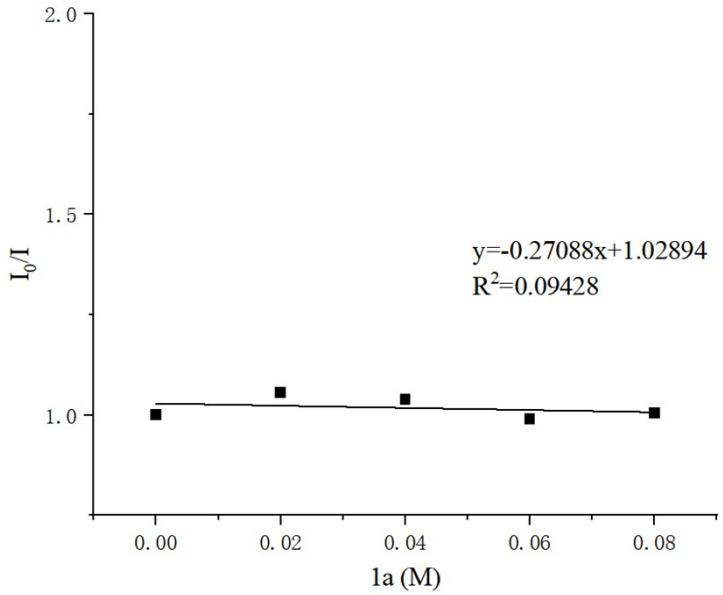
For Experiment 1: Constant photocatalyst; Varied 2-methyl-1-(2-phenyl-1H-benzo[d]imidazol-1-yl)prop-2-en-1-one.

Add 30 μL 4-CzIPN-Br solution and 0 μL, 60 μL, 120 μL, 180 μL, 240 μL **1a** solution respectively in the quartz cuvette, then diluted the solution to 3 mL.



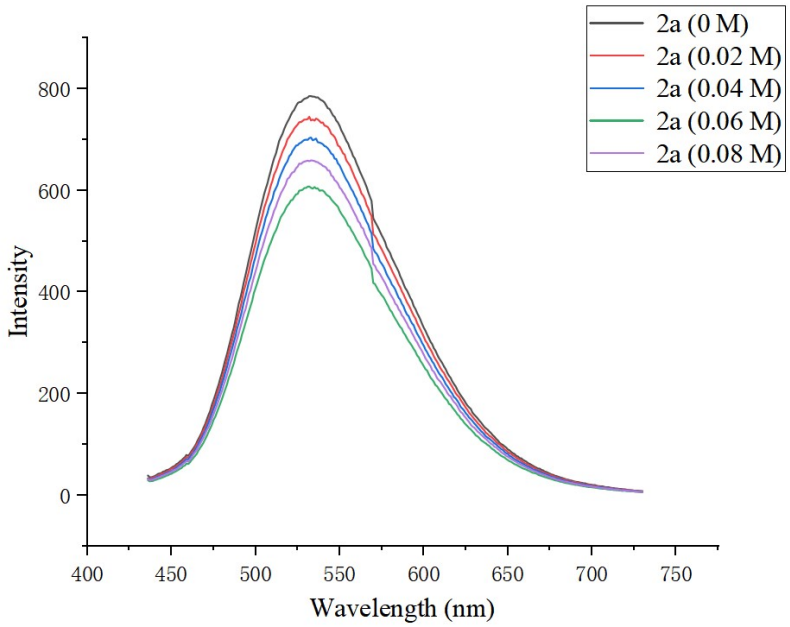
Supplementary figure 1: Fluorescence spectrum of PC + 1a.

Comment: 1a does not react with the excited photocatalyst.



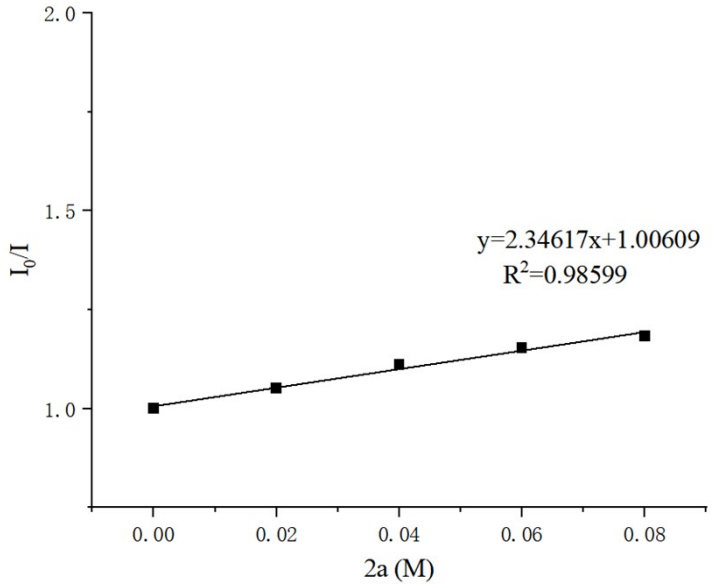
Supplementary figure 2: Stern-Volmer Luminescence Quenching Analysis of PC + 1a.

For Experiment 2: Constant photocatalyst; Varied piperidine-1-sulfonyl chloride. Add 30 μL 4-CzIPN-Br solution and 0 μL, 60 μL, 120 μL, 180 μL, 240 μL 1a solution respectively in the quartz cuvette, then diluted the solution to 3 mL.



Supplementary figure 3: Fluorescence spectrum of PC + 2a.

Comment: Only **2a** could quench the photocatalyst.



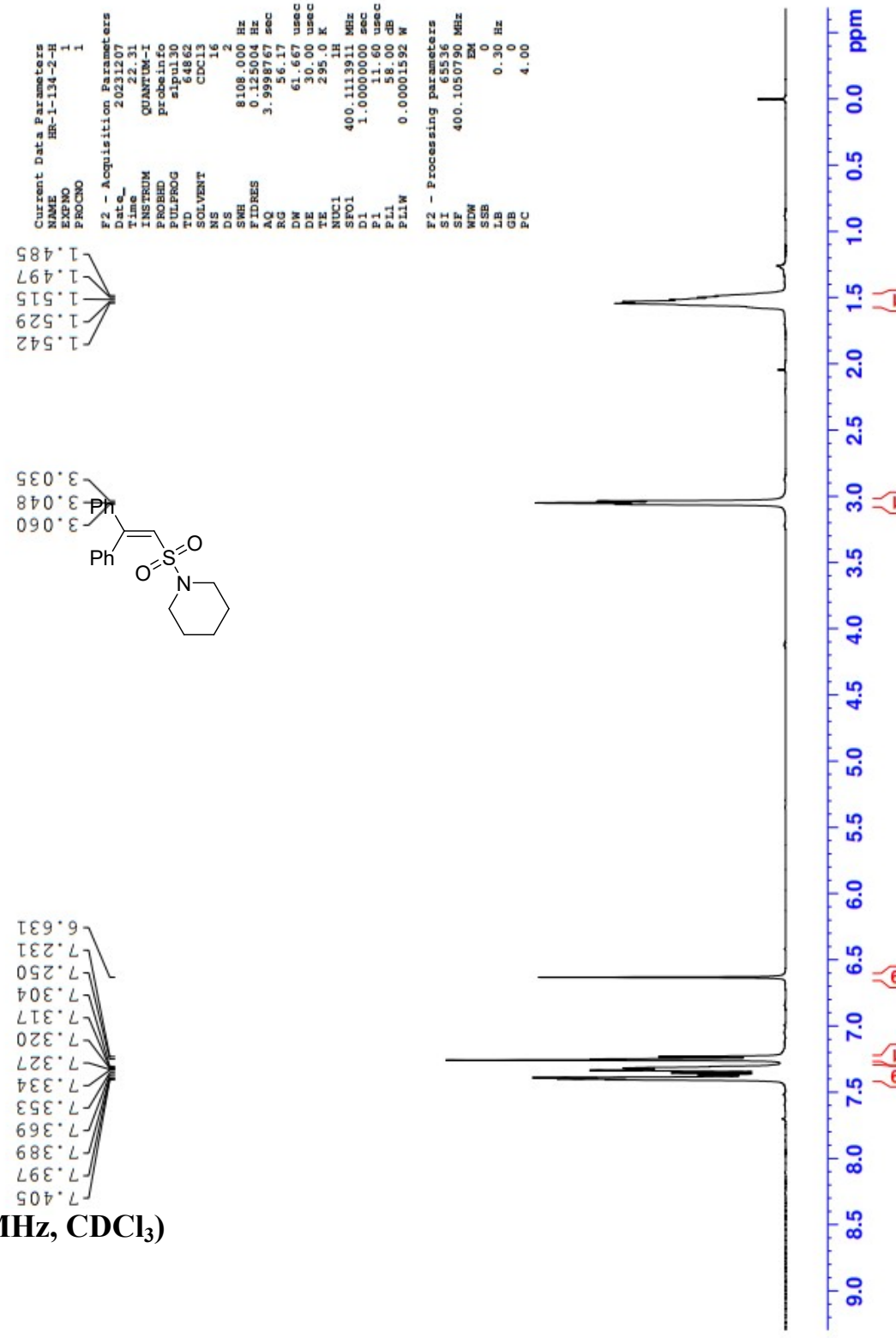
Supplementary figure 4: Stern-Volmer Luminescence Quenching Analysis of PC + 2a.

3. Larger-Scale Experiments

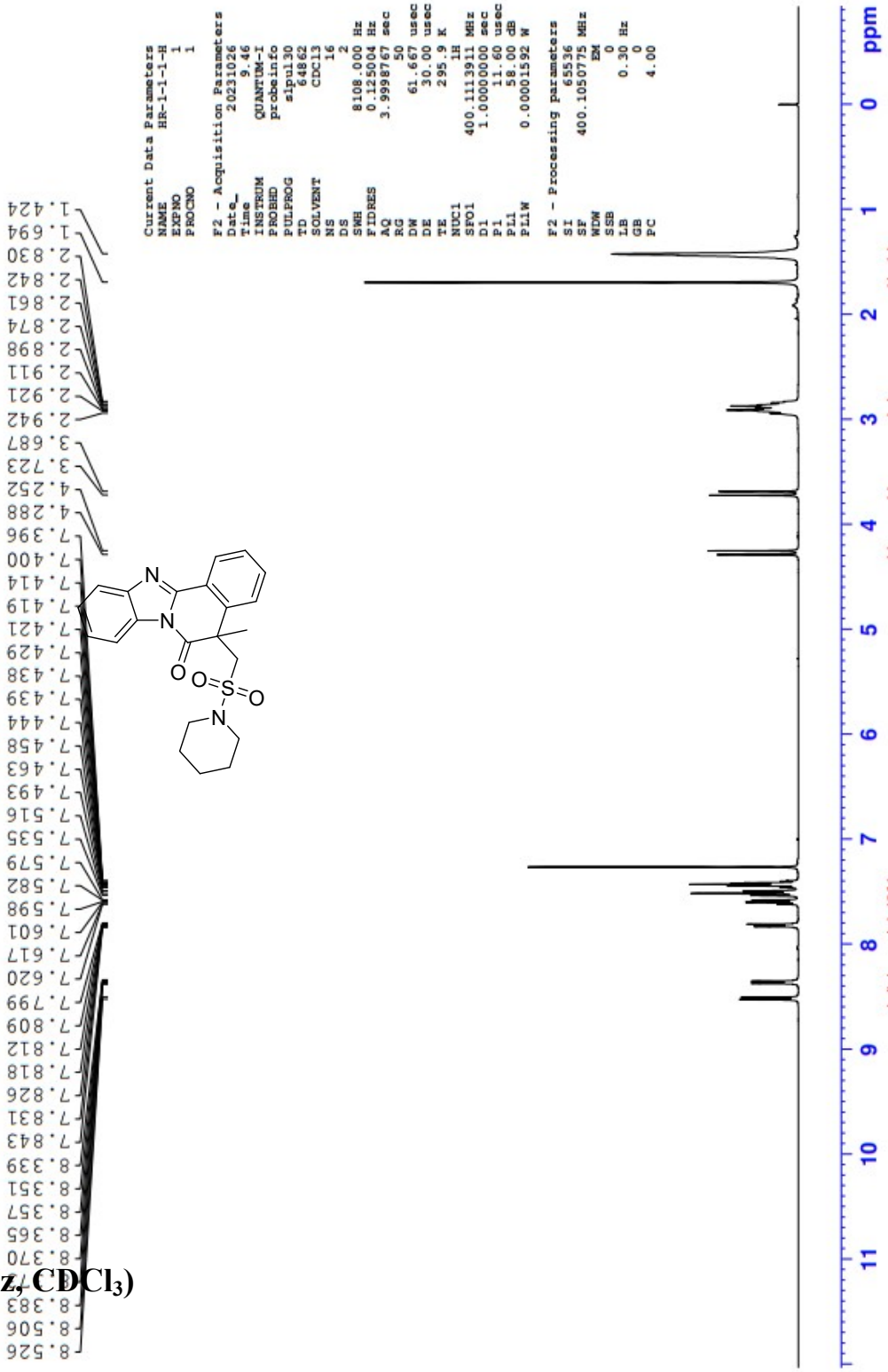
A frame-dried 100 mL Schlenk flask with a magnetic stirring bar was charged with 2-methyl-1-(2-phenyl-1H-benzo[d]imidazol-1-yl)prop-2-en-1-one (1a) (1.05 g, 4.0 mmol), 4-CzIPN-Br (28.4 mg, 0.02 mmol) and dry DMA (20.0 mL) was added via syringe under nitrogen. Then piperidine-1-sulfonyl chloride (2a) (1.69 g, 9.2 mmol) was added and the reaction mixture was stirred under irradiation by a 36 W blue LEDs at 25 ℃ for 2.5 hours under nitrogen. The reaction mixture diluted with water (200 mL) and extracted with ethyl acetate (100 mL) for three times. The combined organic phase was dried over anhydrous Na_2SO_4 and concentrated to give a residue which was purified by silica gel chromatography, eluting with petroleum ether/ethyl acetate, to give compound 4a as a light yellow solid (1.60 g, 98%).

4. ^{1}H NMR, ^{13}C

4 ^{1}H NMR (400

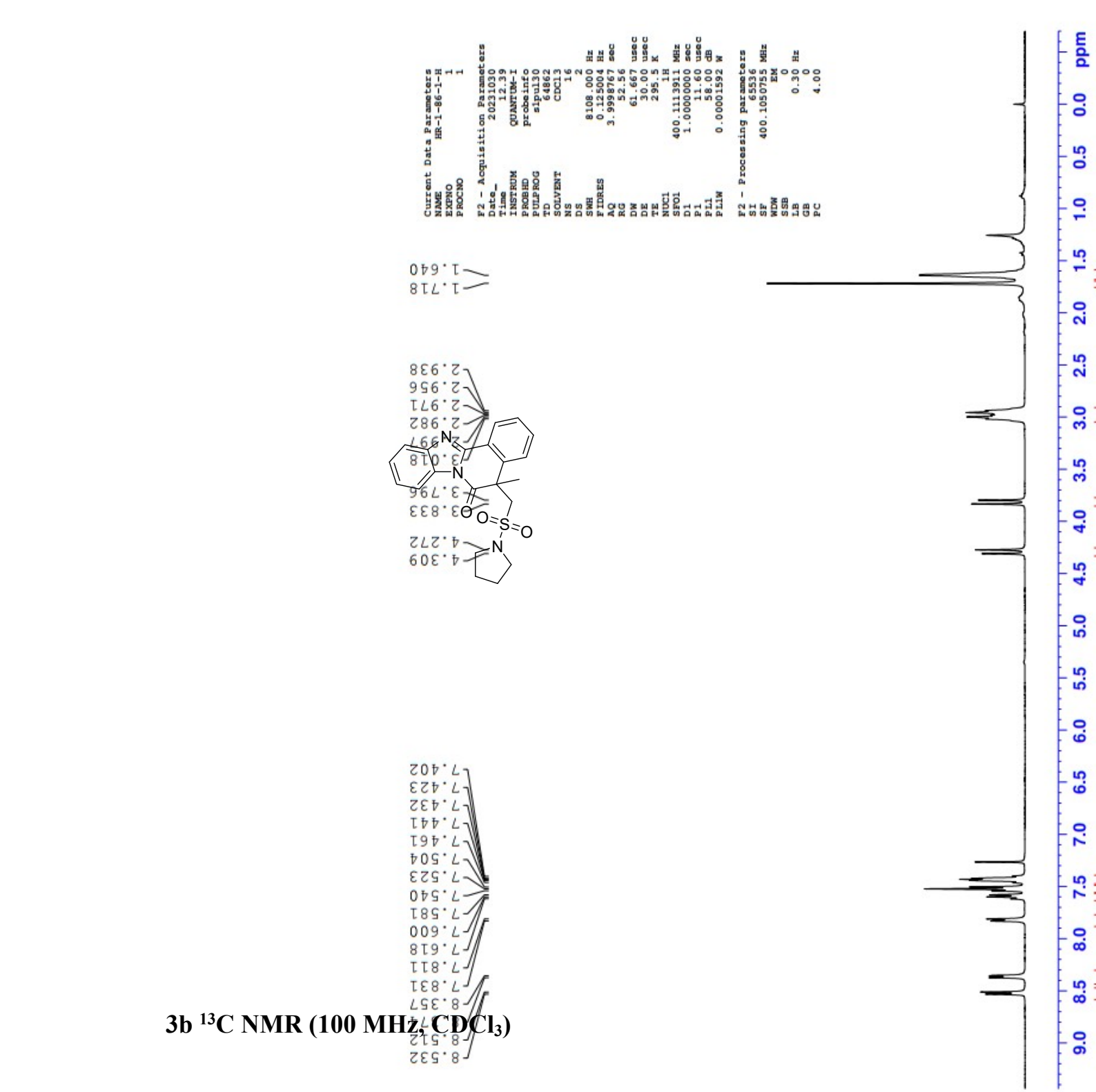


3a ^{1}H NMR (400 MHz, $CDCl_3$)

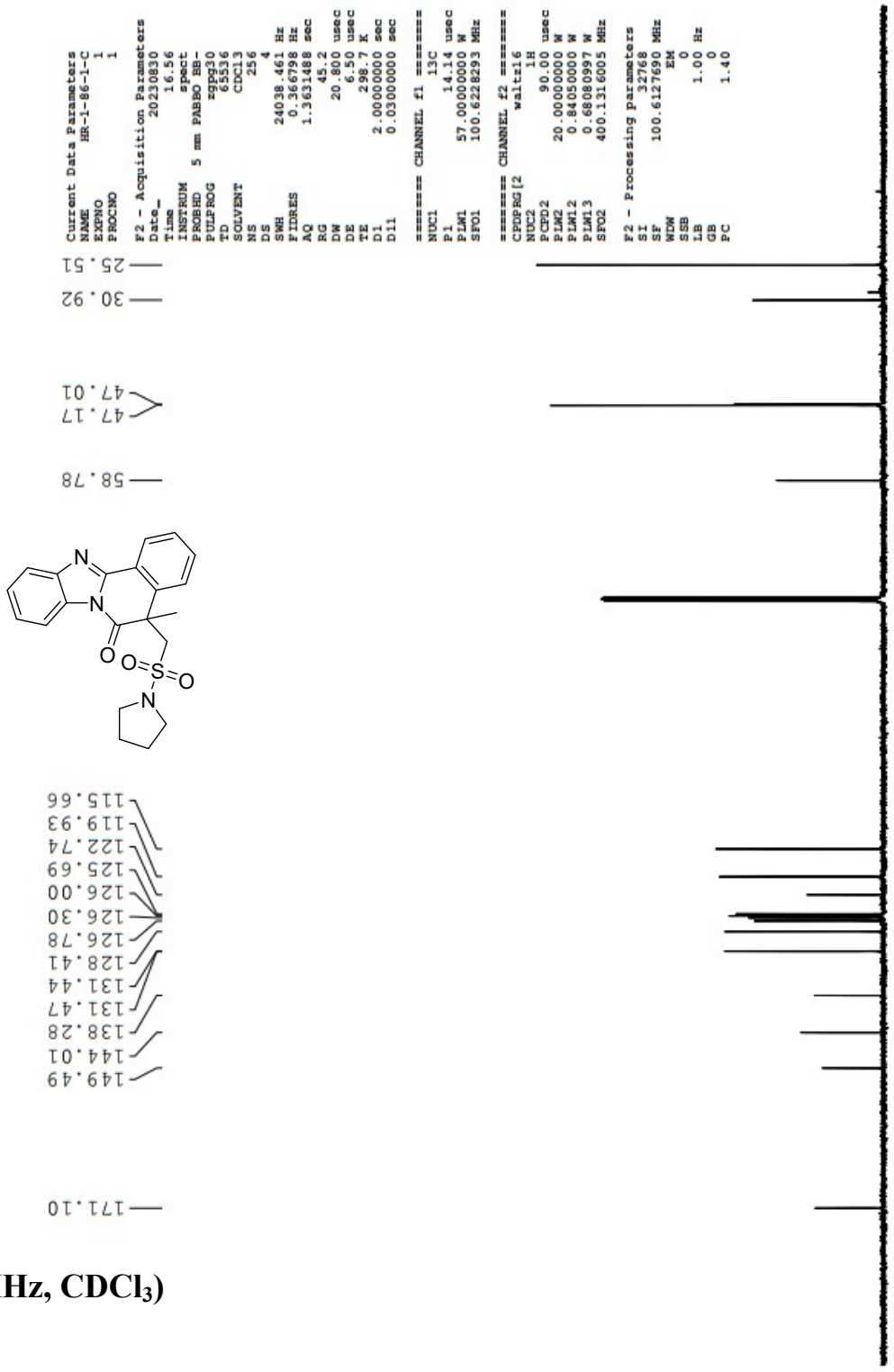


3a ^{13}C NMR (100 MHz, $CDCl_3$)

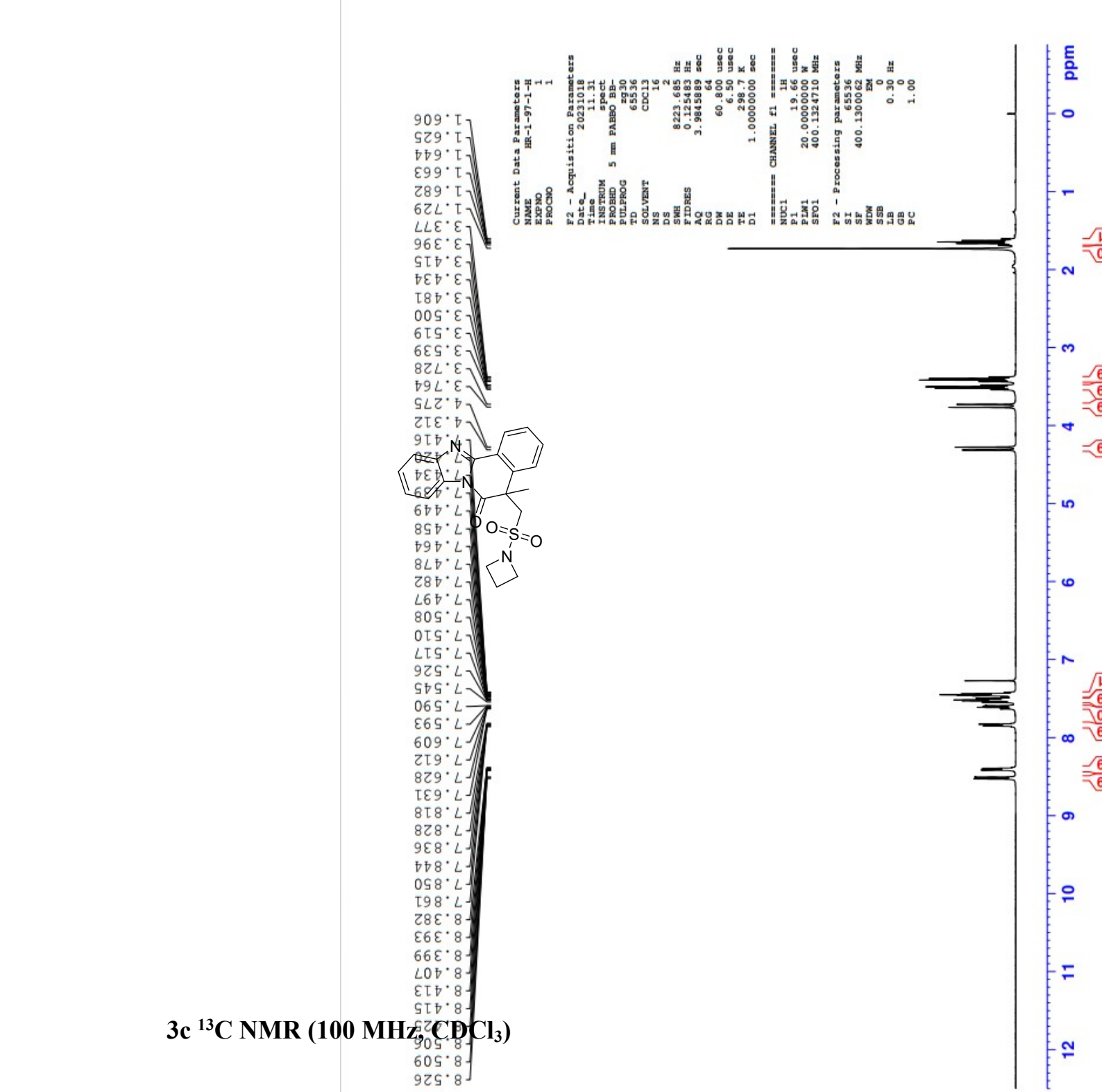
3b ^{1}H NMR (400 MHz, $CDCl_3$)



3b ^{13}C NMR (100 MHz, $CDCl_3$)



3c ^{1}H NMR (400 MHz, $CDCl_3$)



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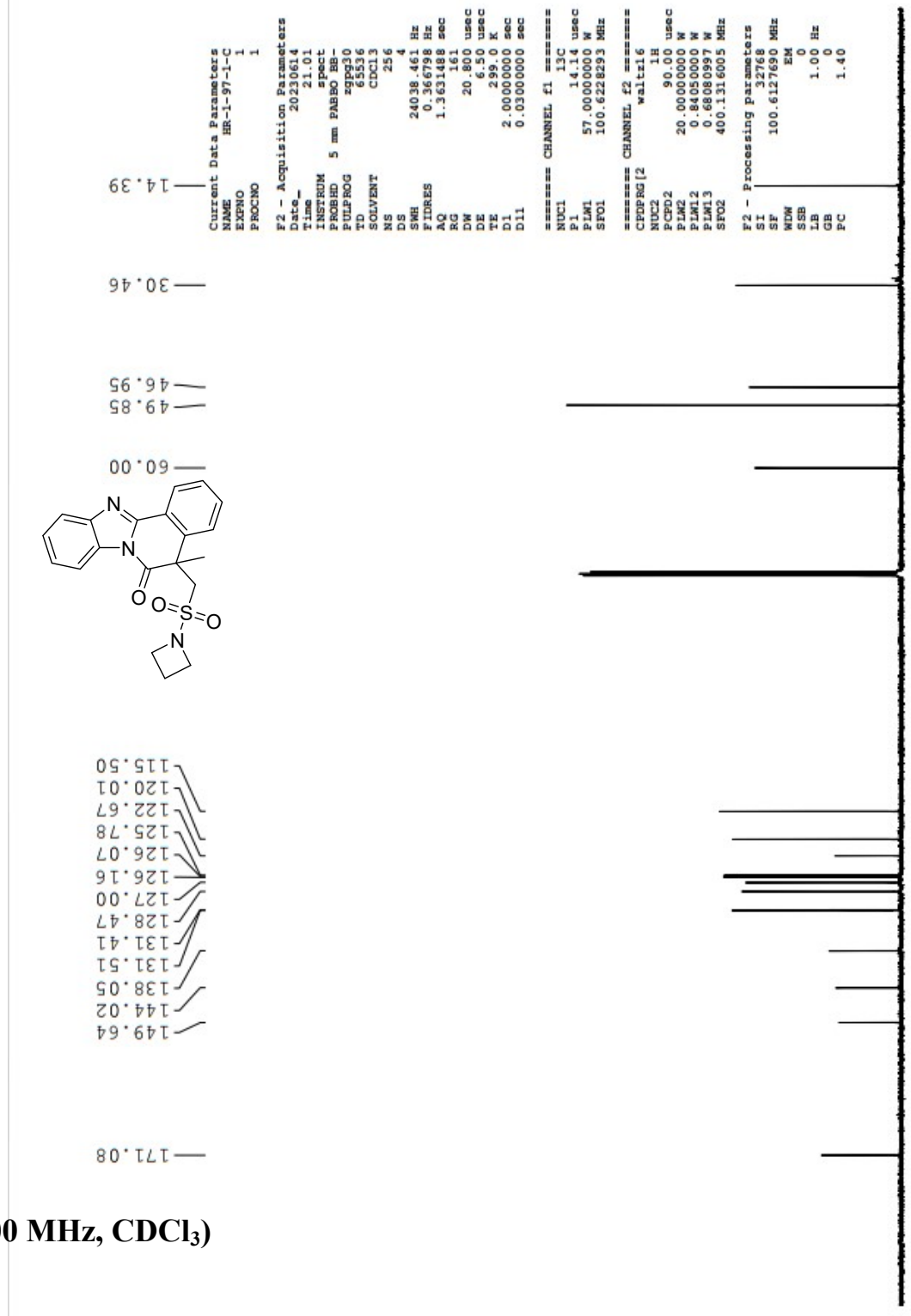

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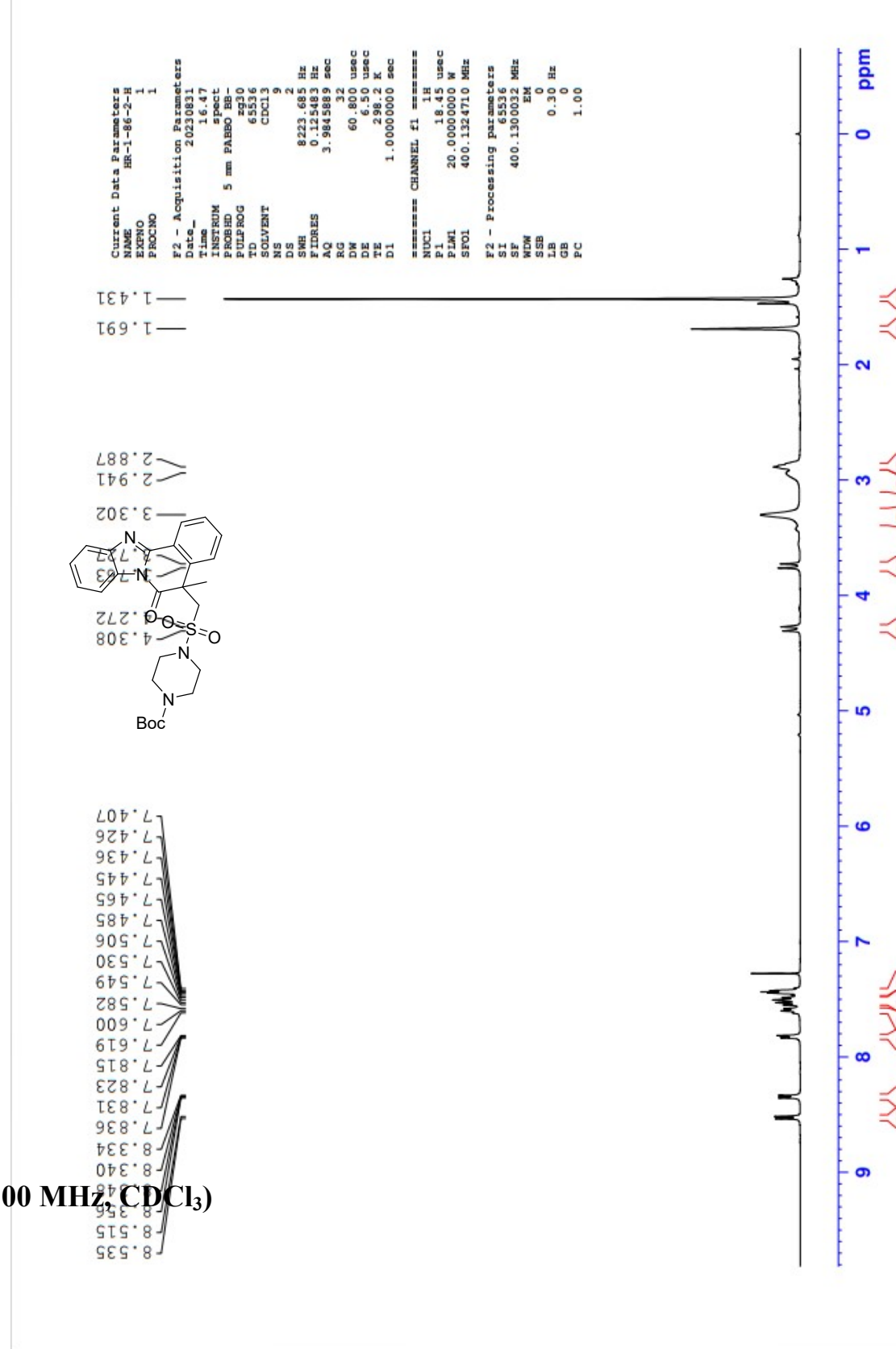

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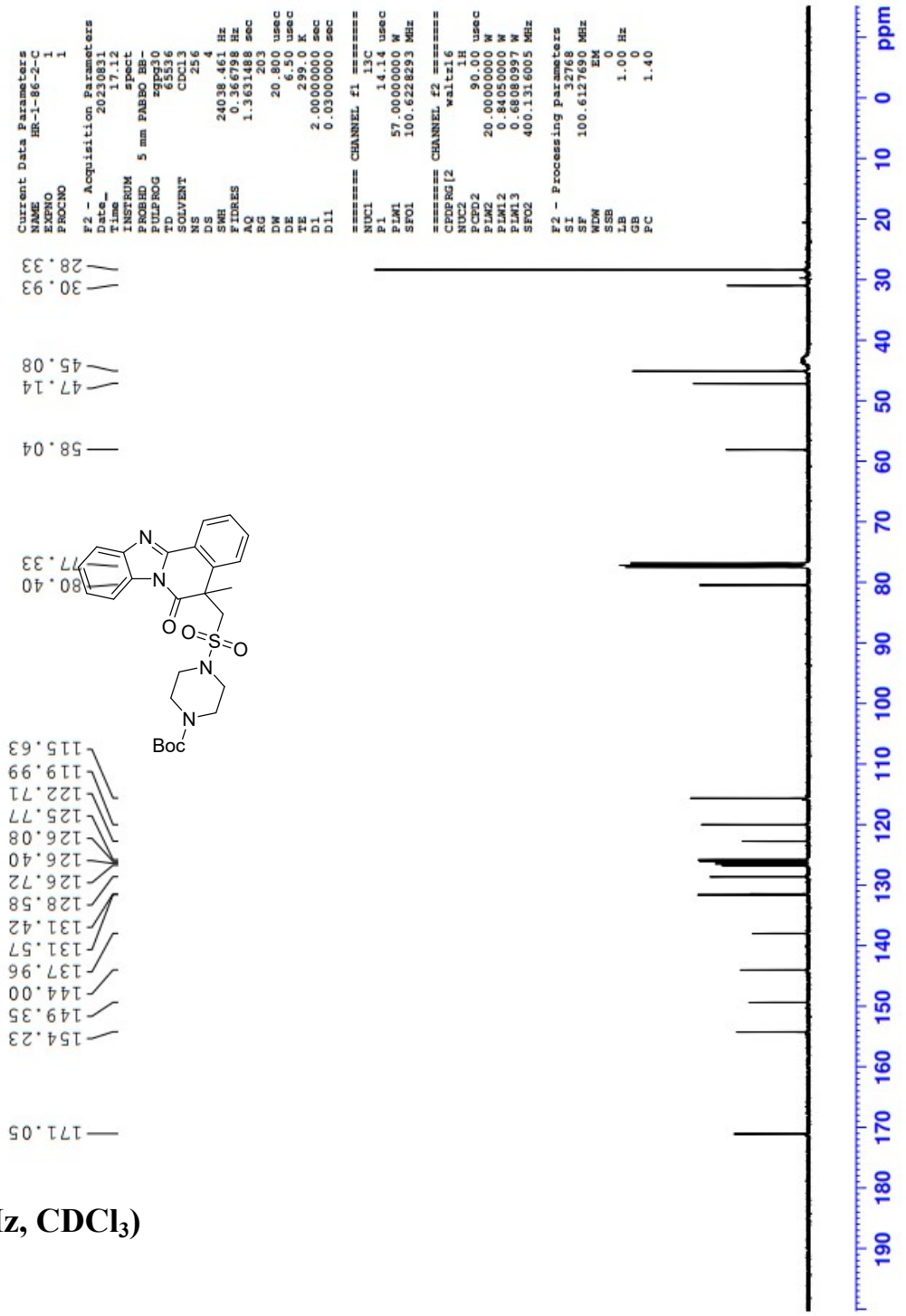

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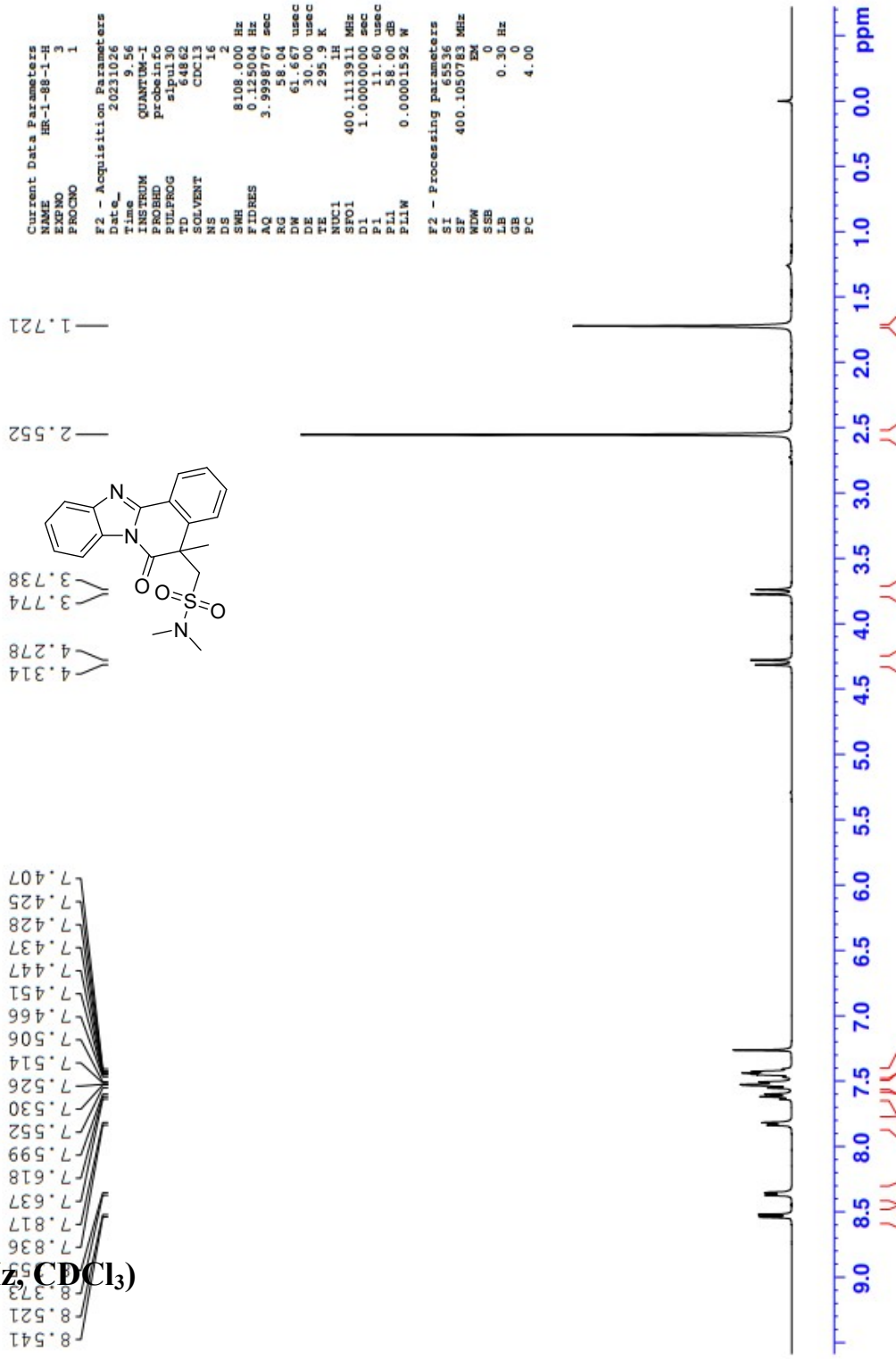

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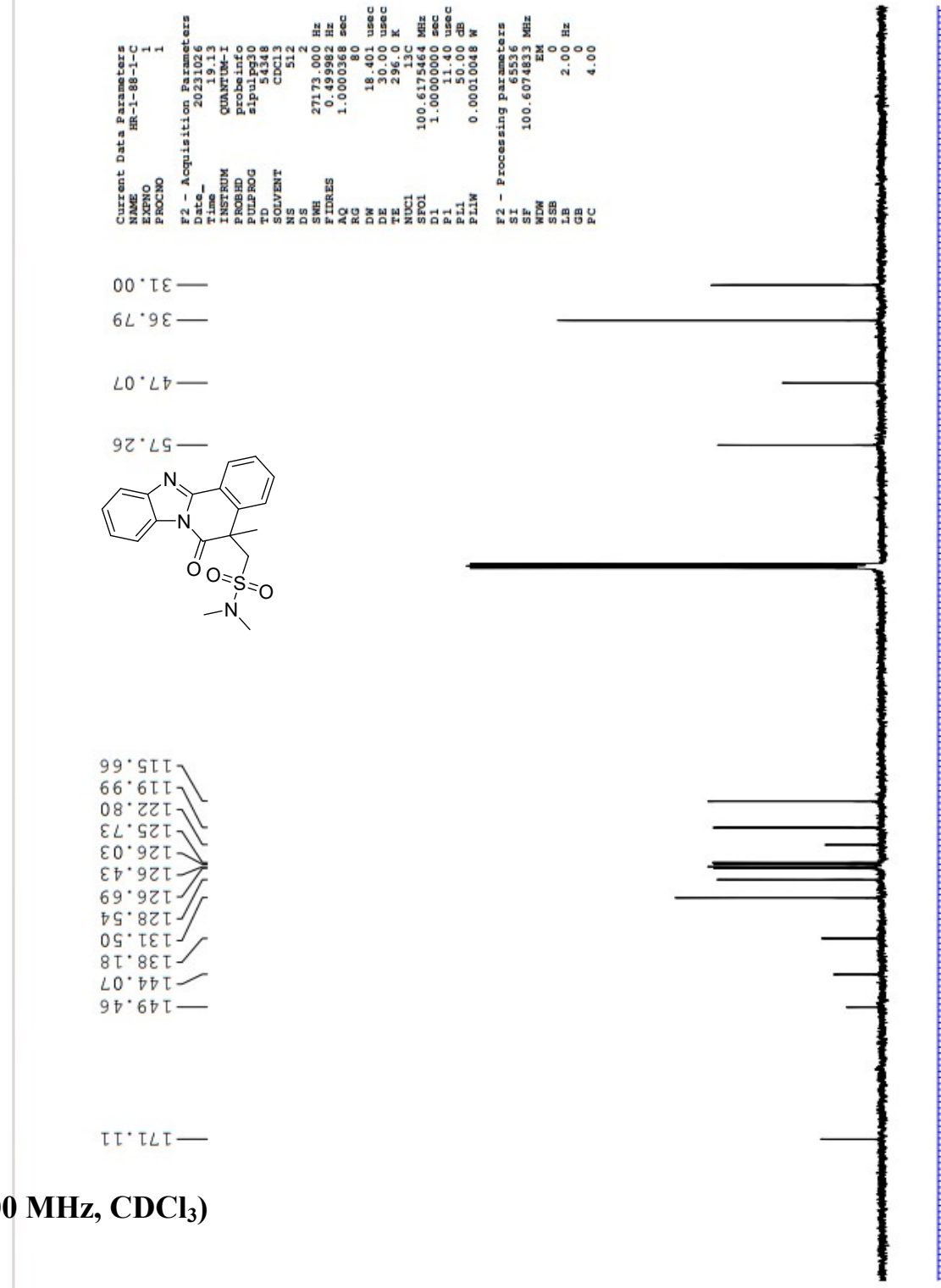

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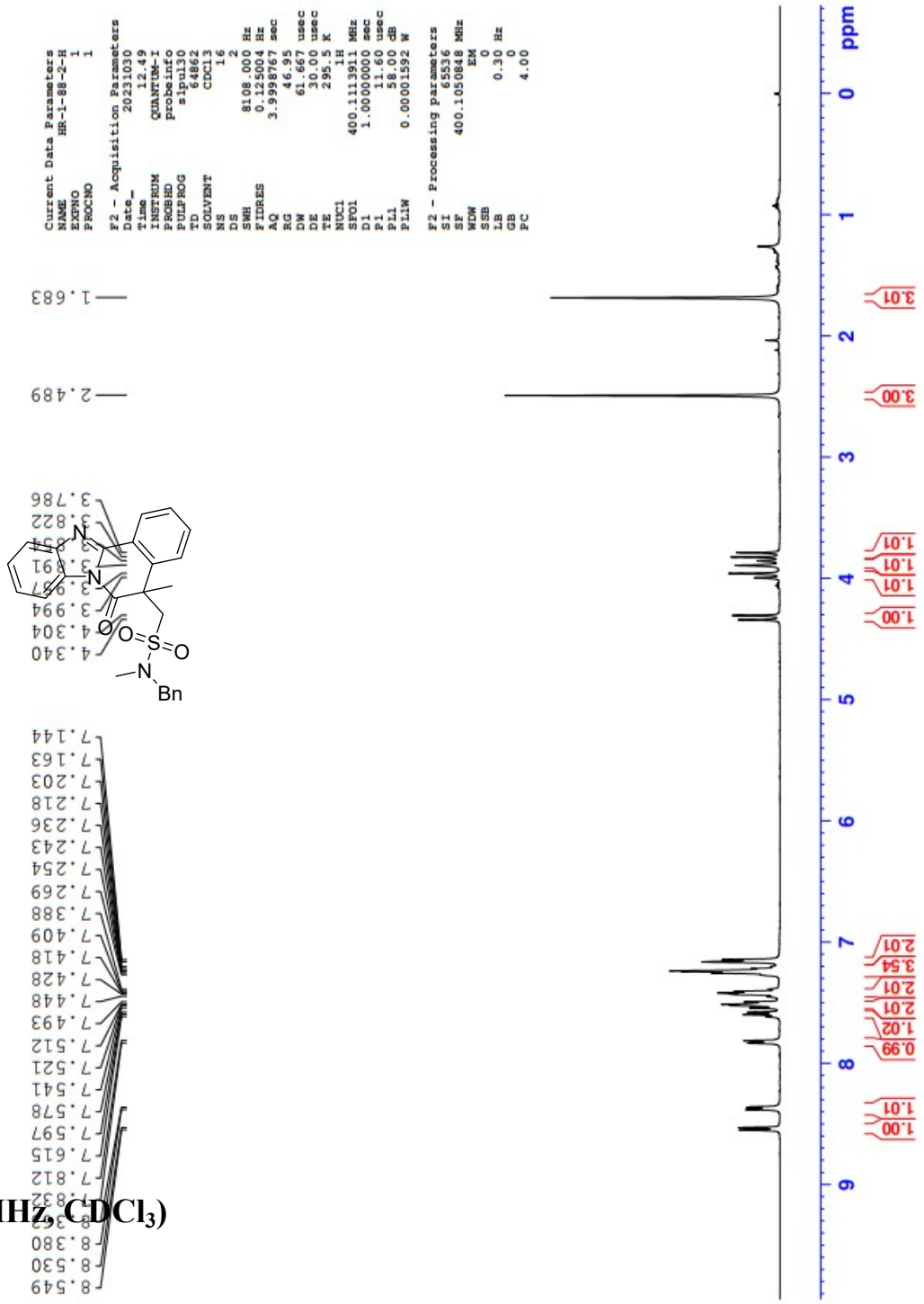

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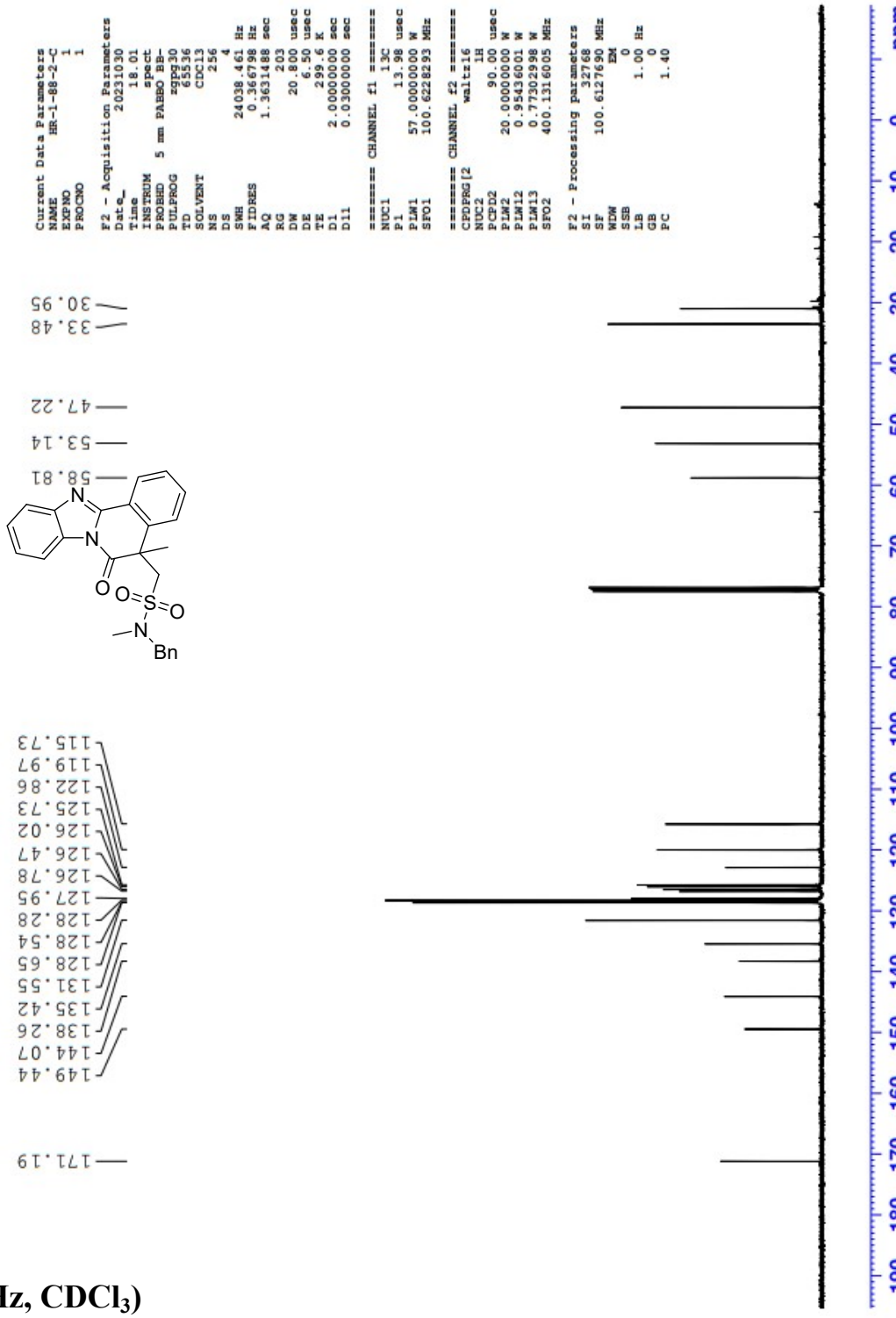
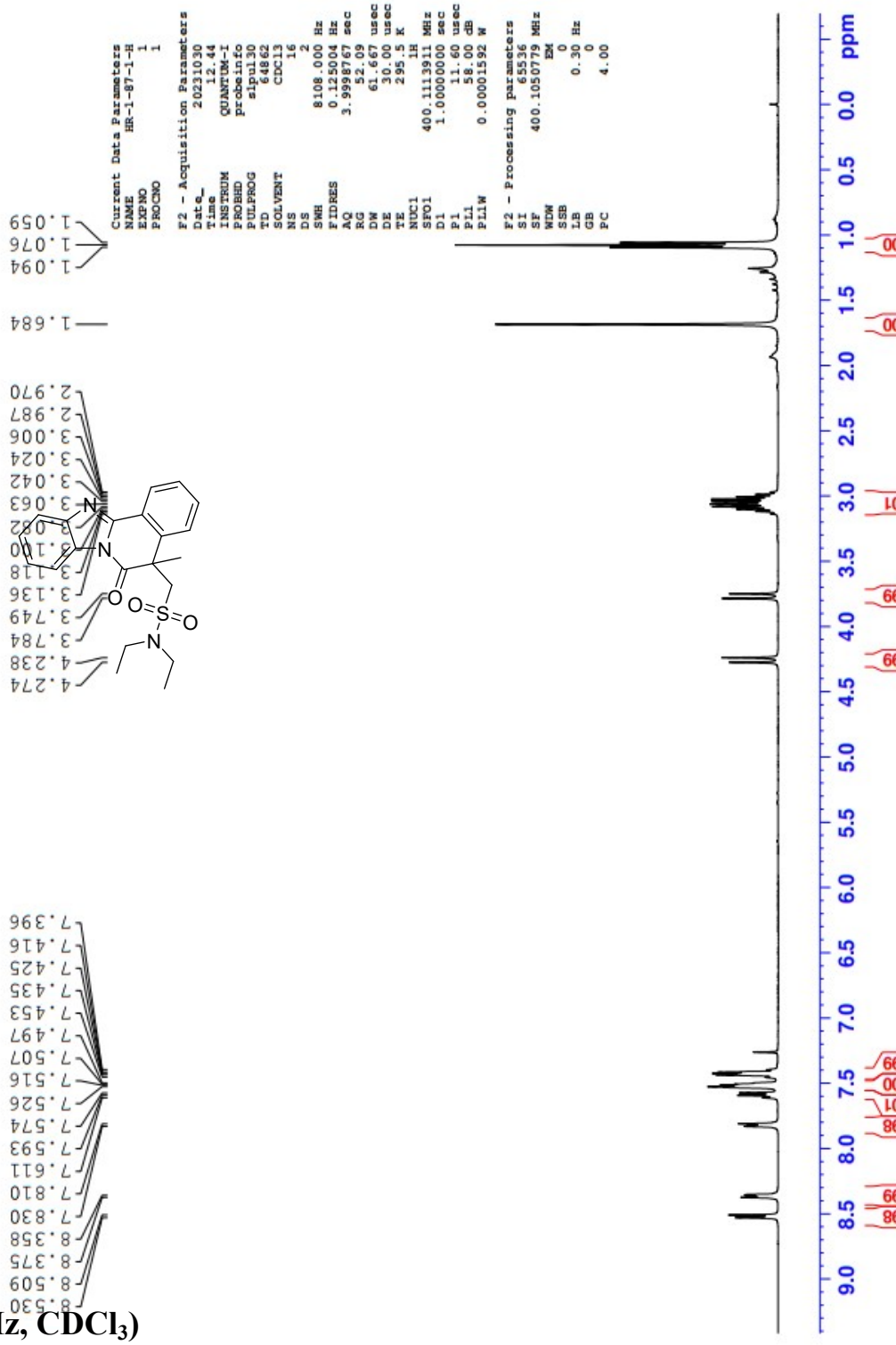
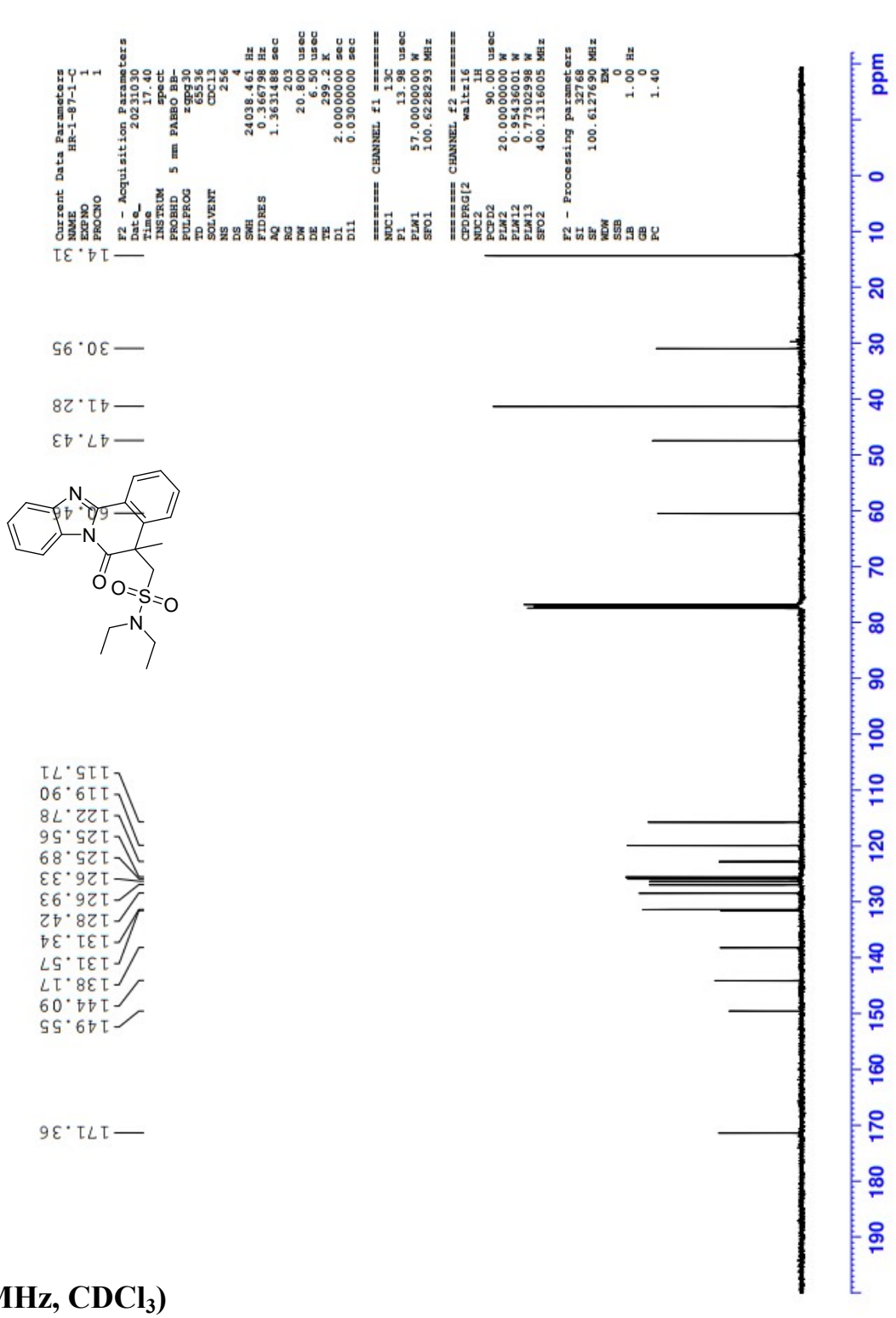


3g ^{1}H NMR (400 MHz, $CDCl_3$)



3g ^{13}C NMR (100 MHz, $CDCl_3$)



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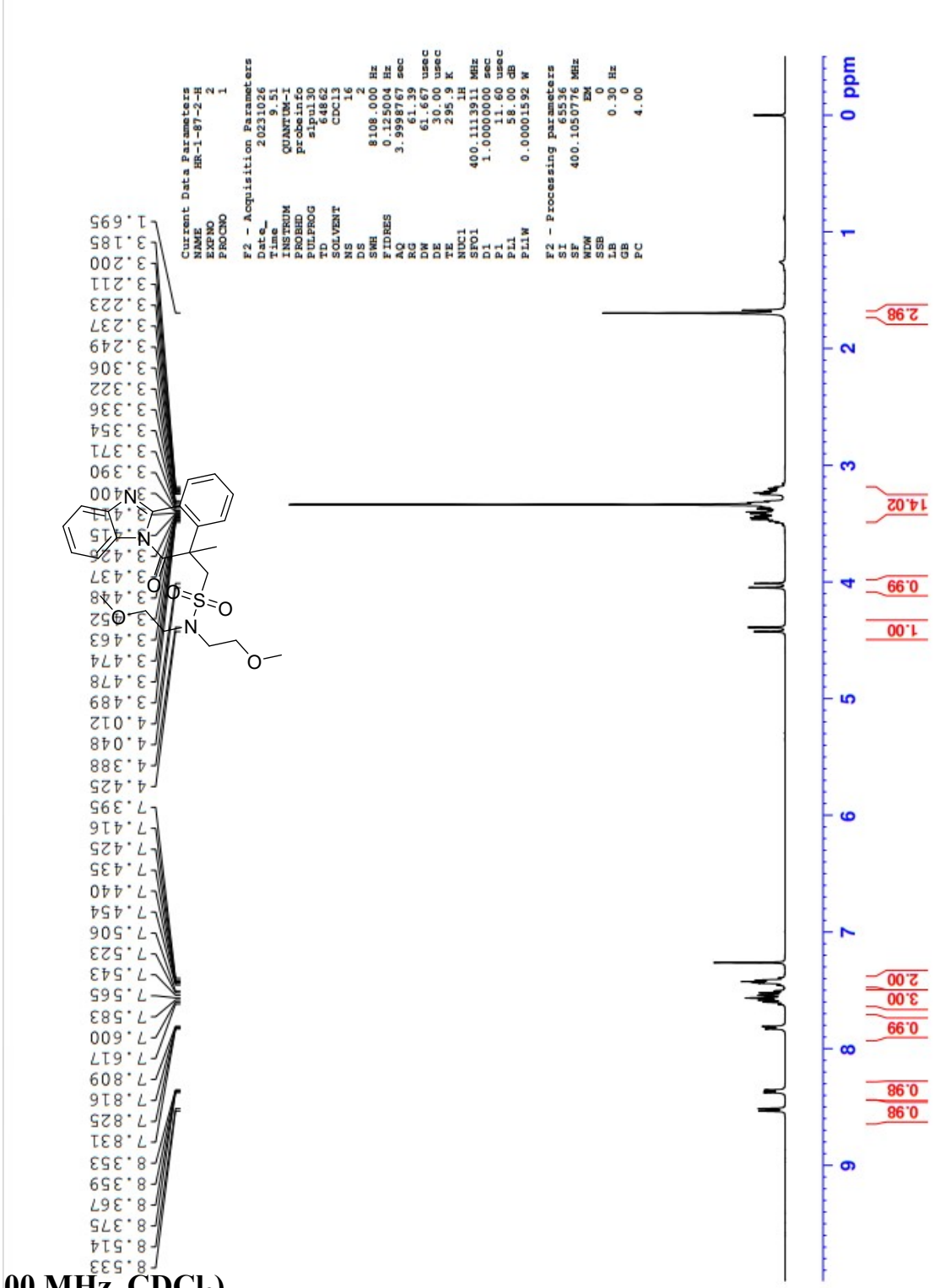
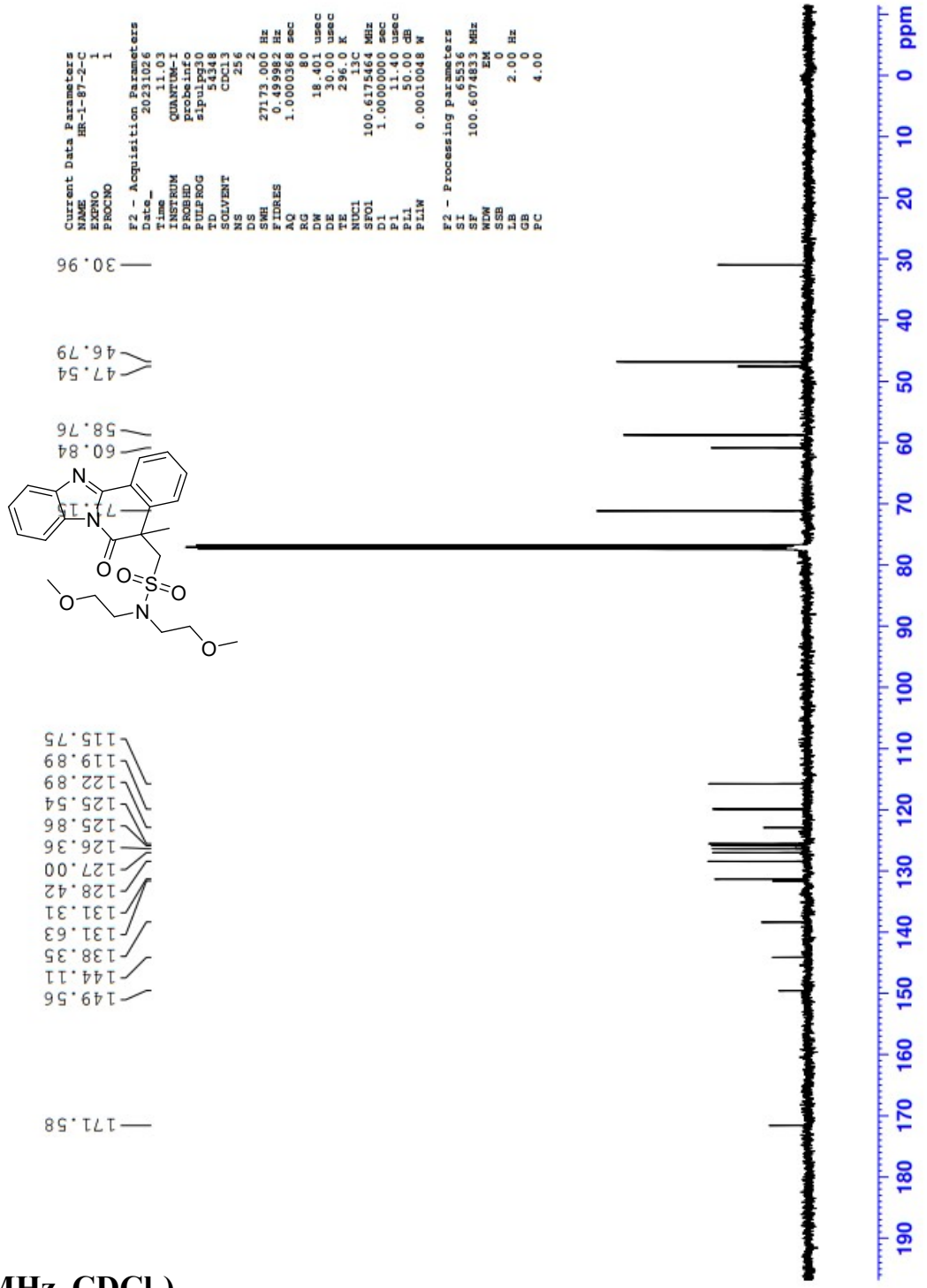
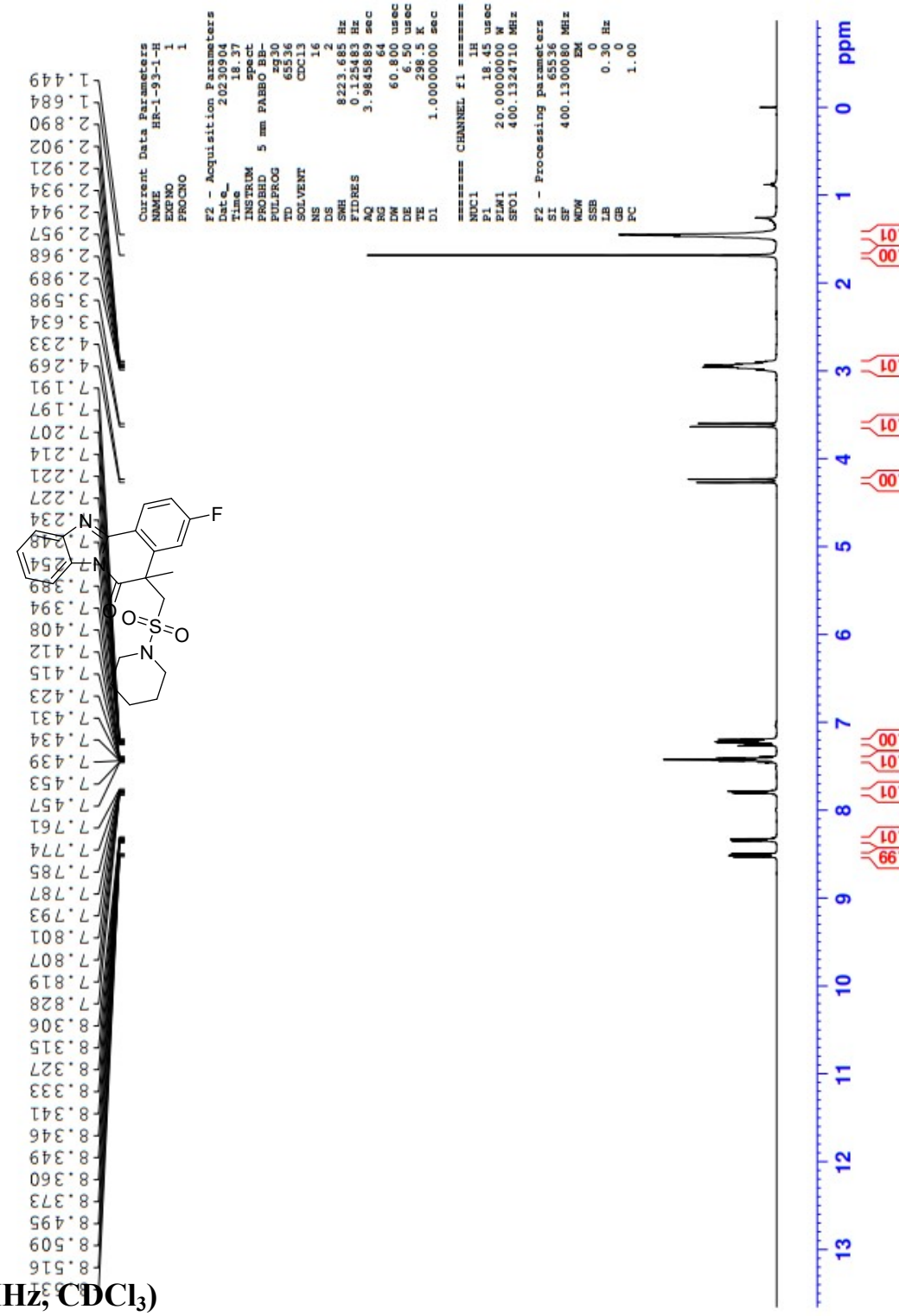


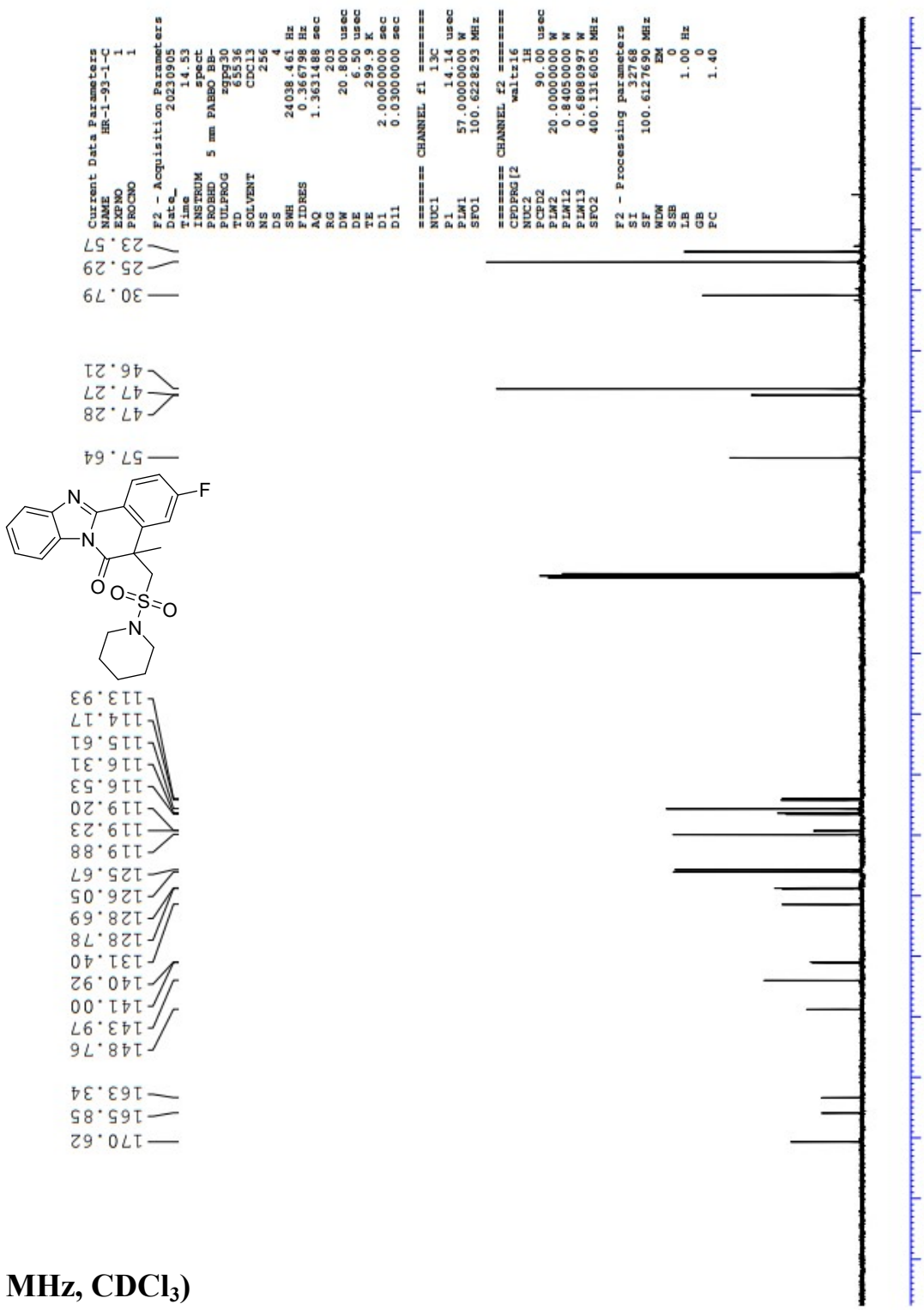
3h ^{13}C NMR (100 MHz, $CDCl_3$)



3i ^{1}H NMR (400 MHz, $CDCl_3$)



3i ^{13}C NMR (100 MHz, $CDCl_3$)



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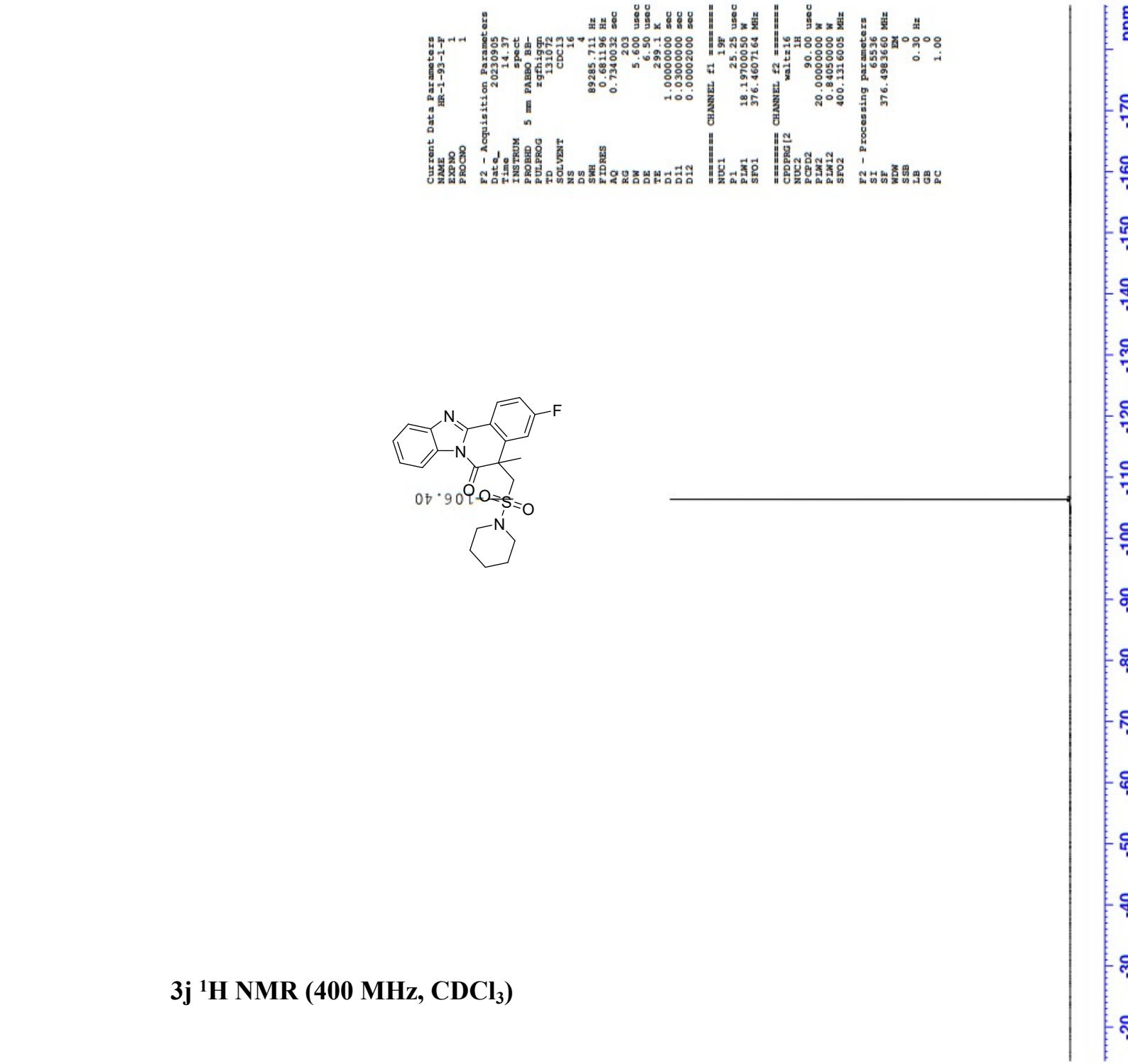
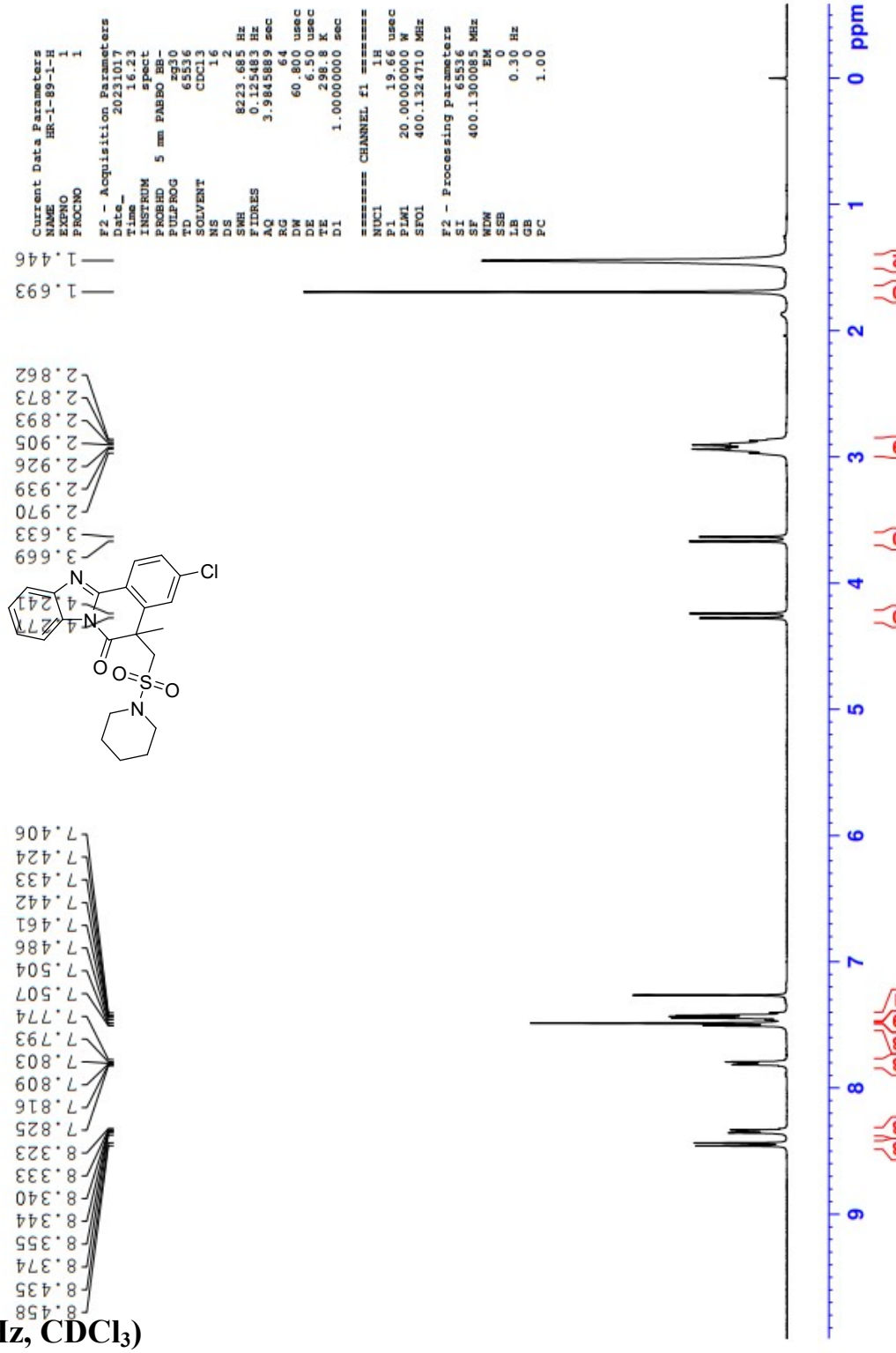
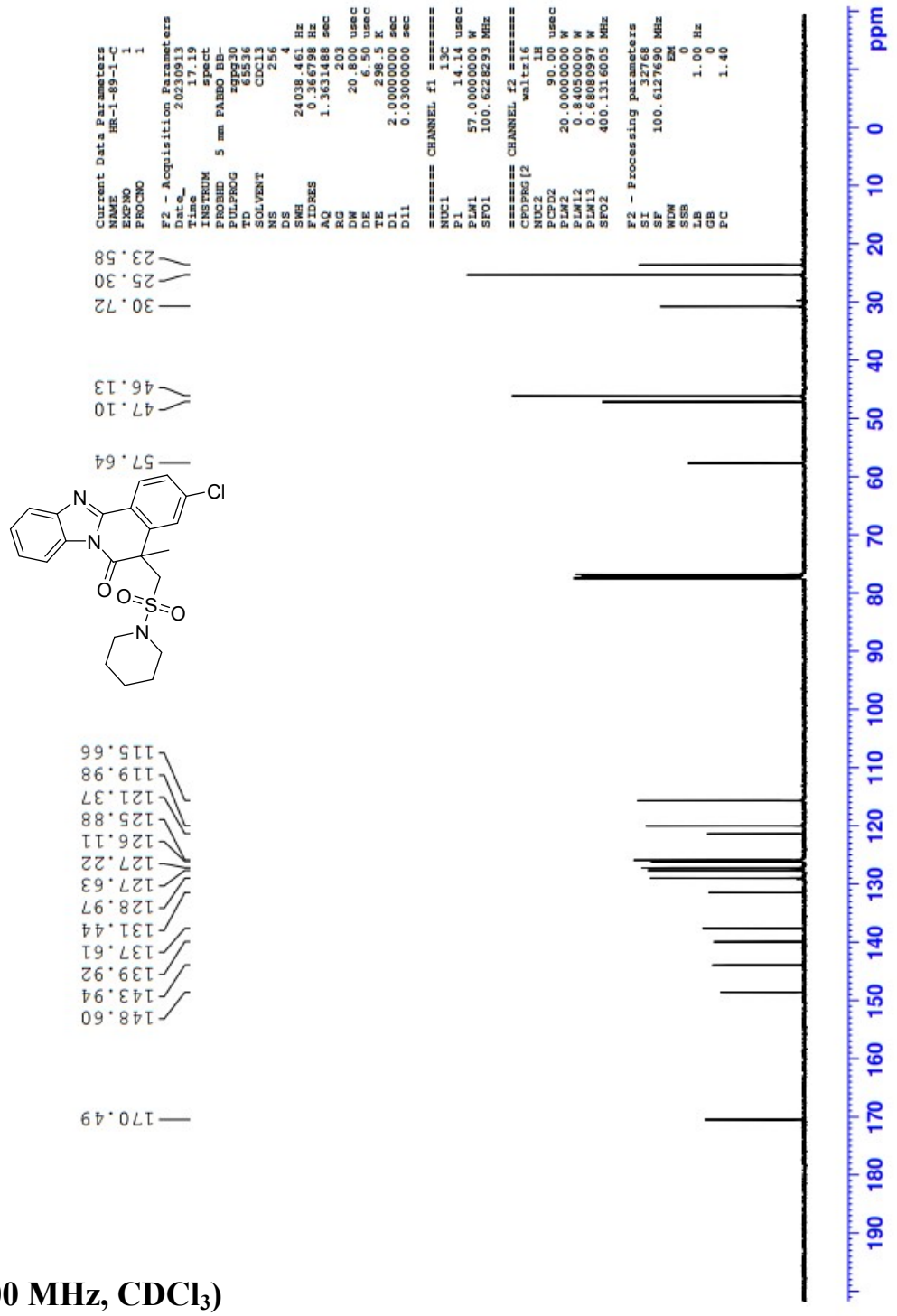


3j ^{1}H NMR (400 MHz, $CDCl_3$)



3j ^{13}C NMR (100 MHz, $CDCl_3$)



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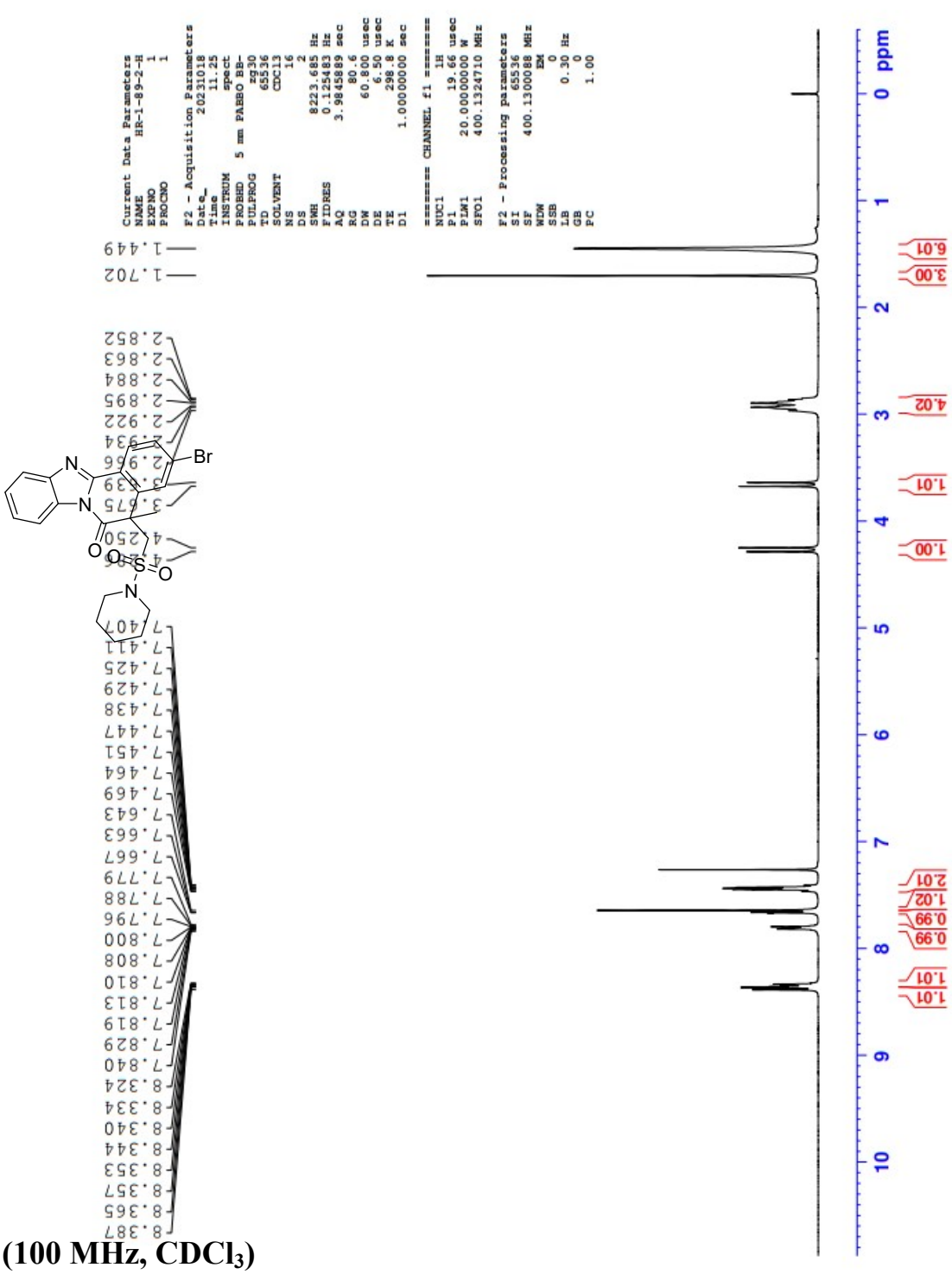

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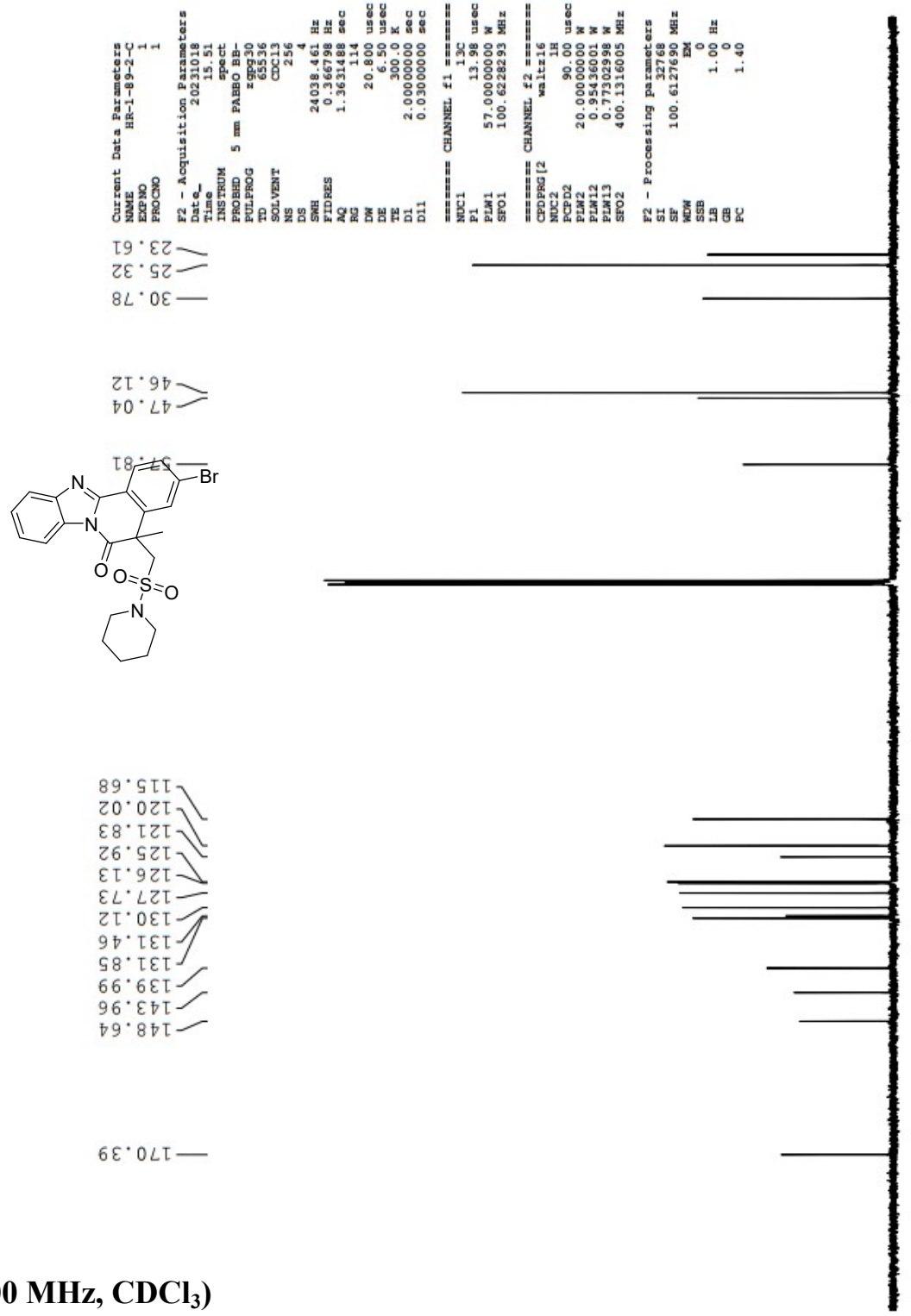

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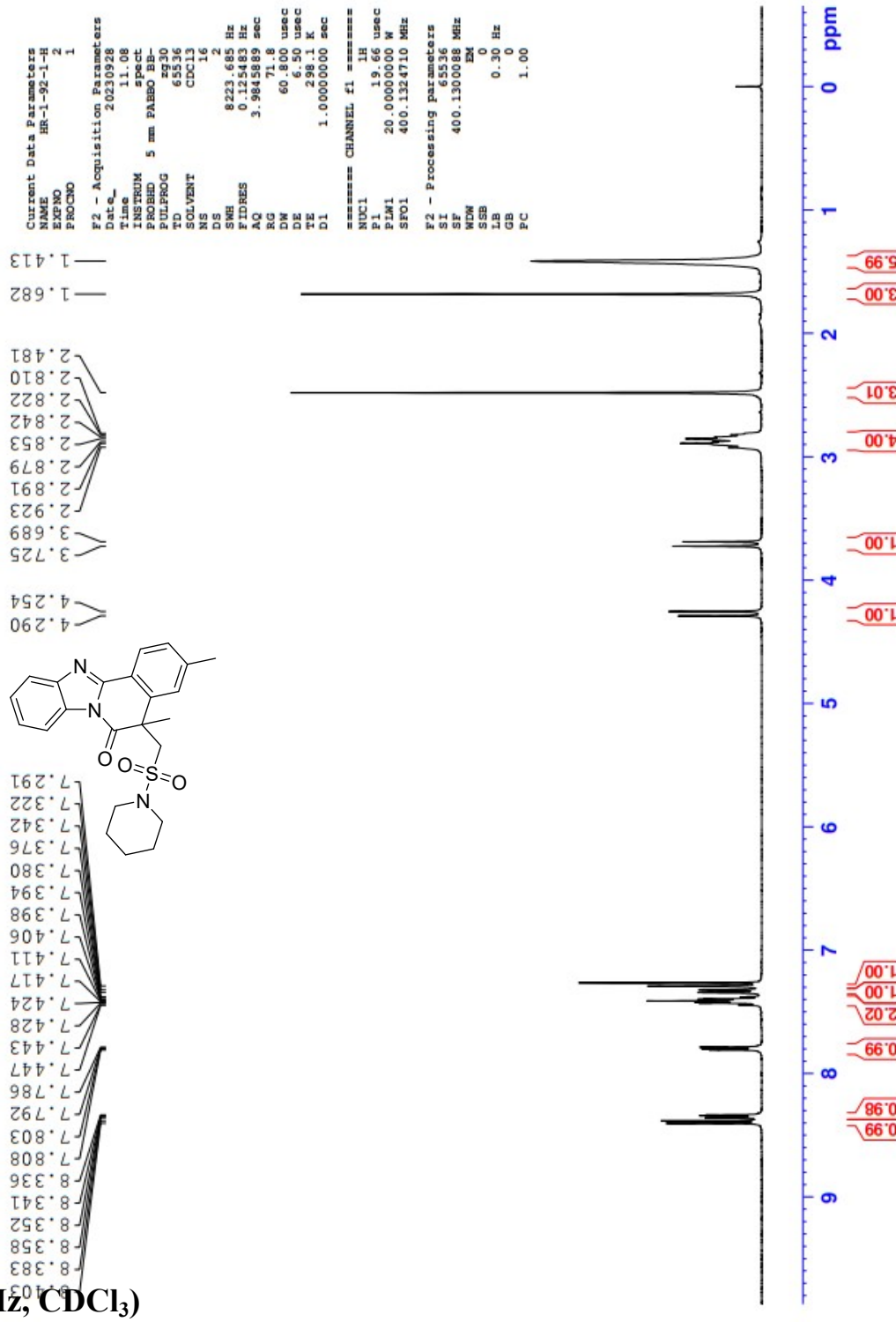

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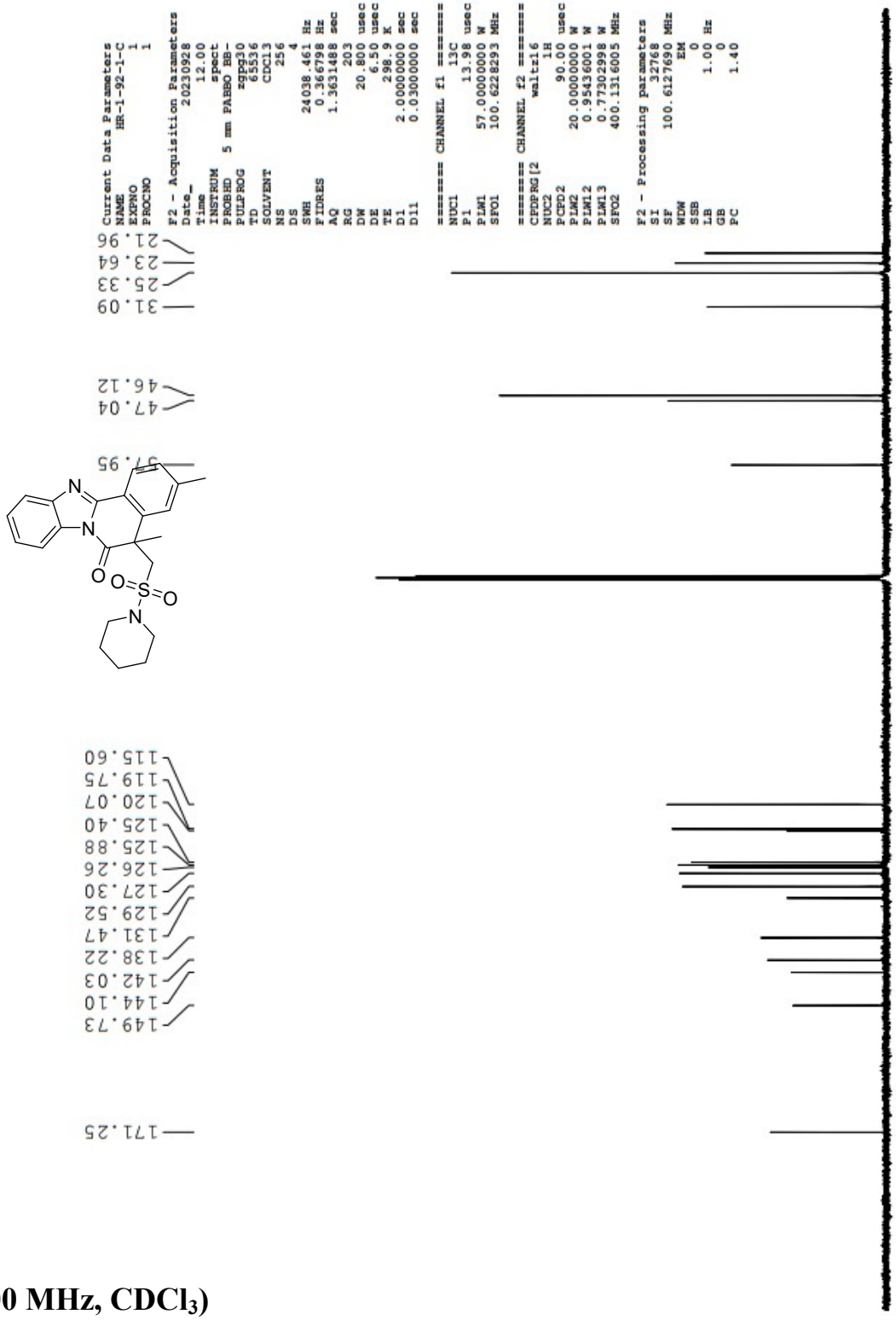

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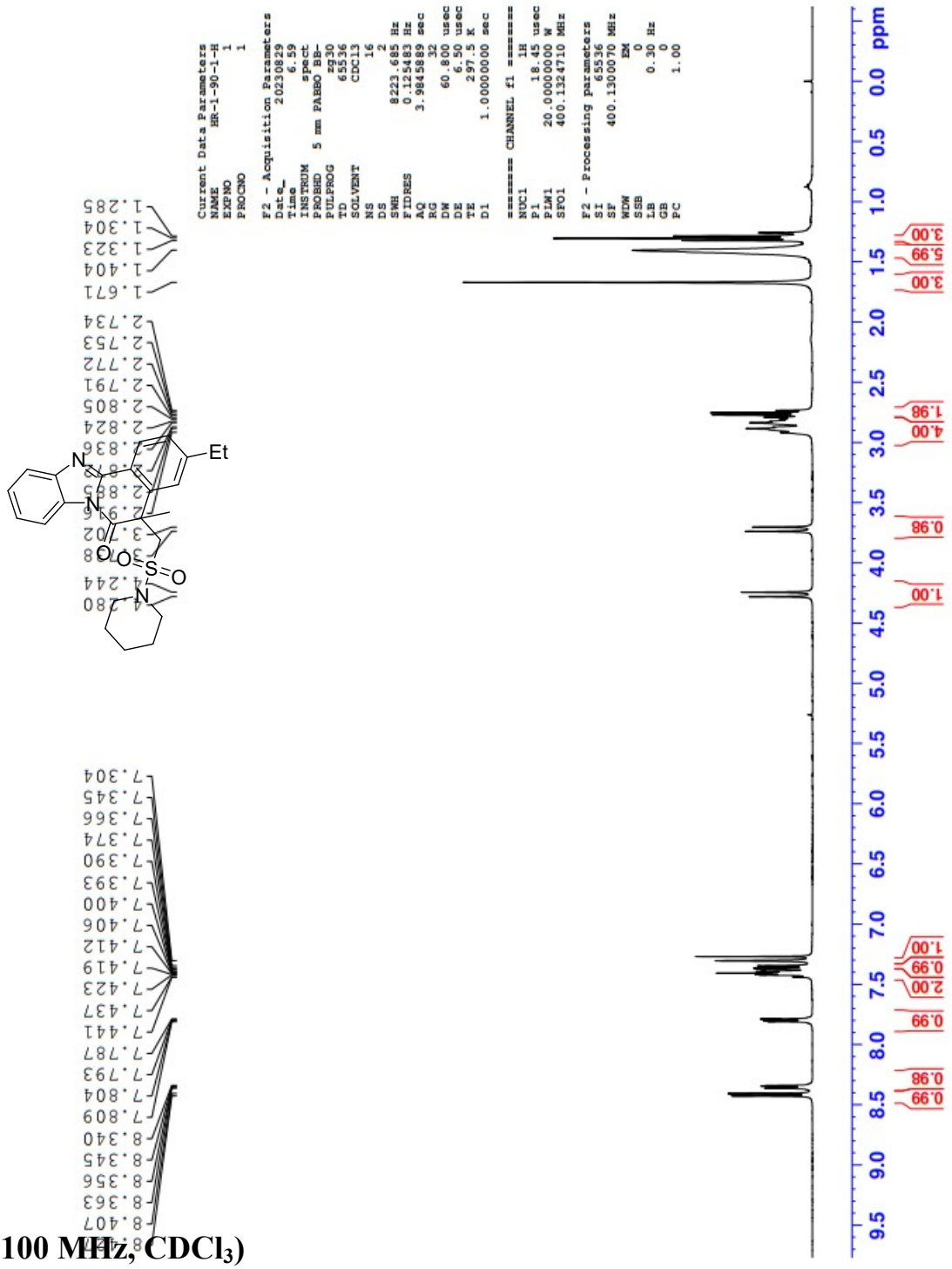

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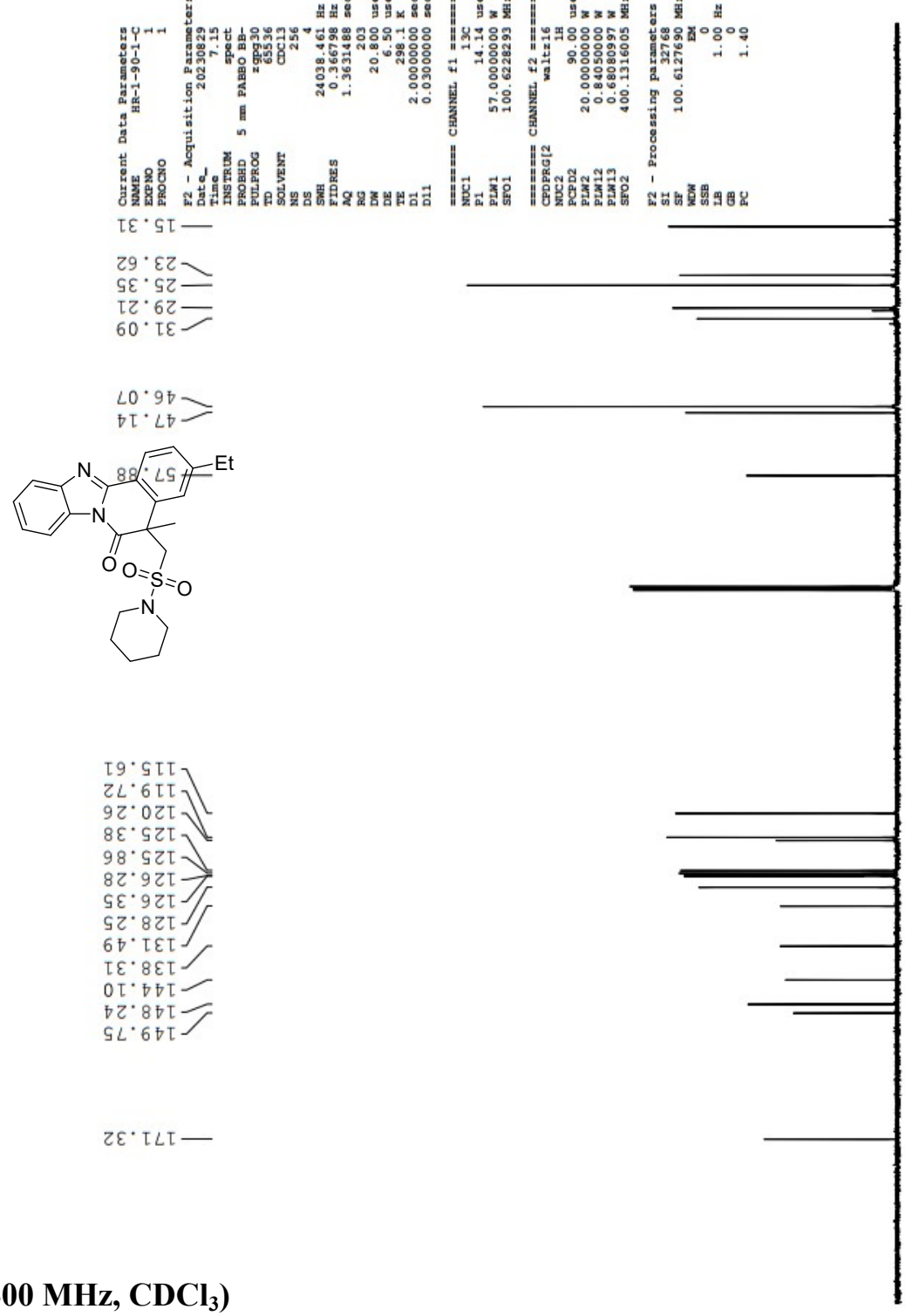

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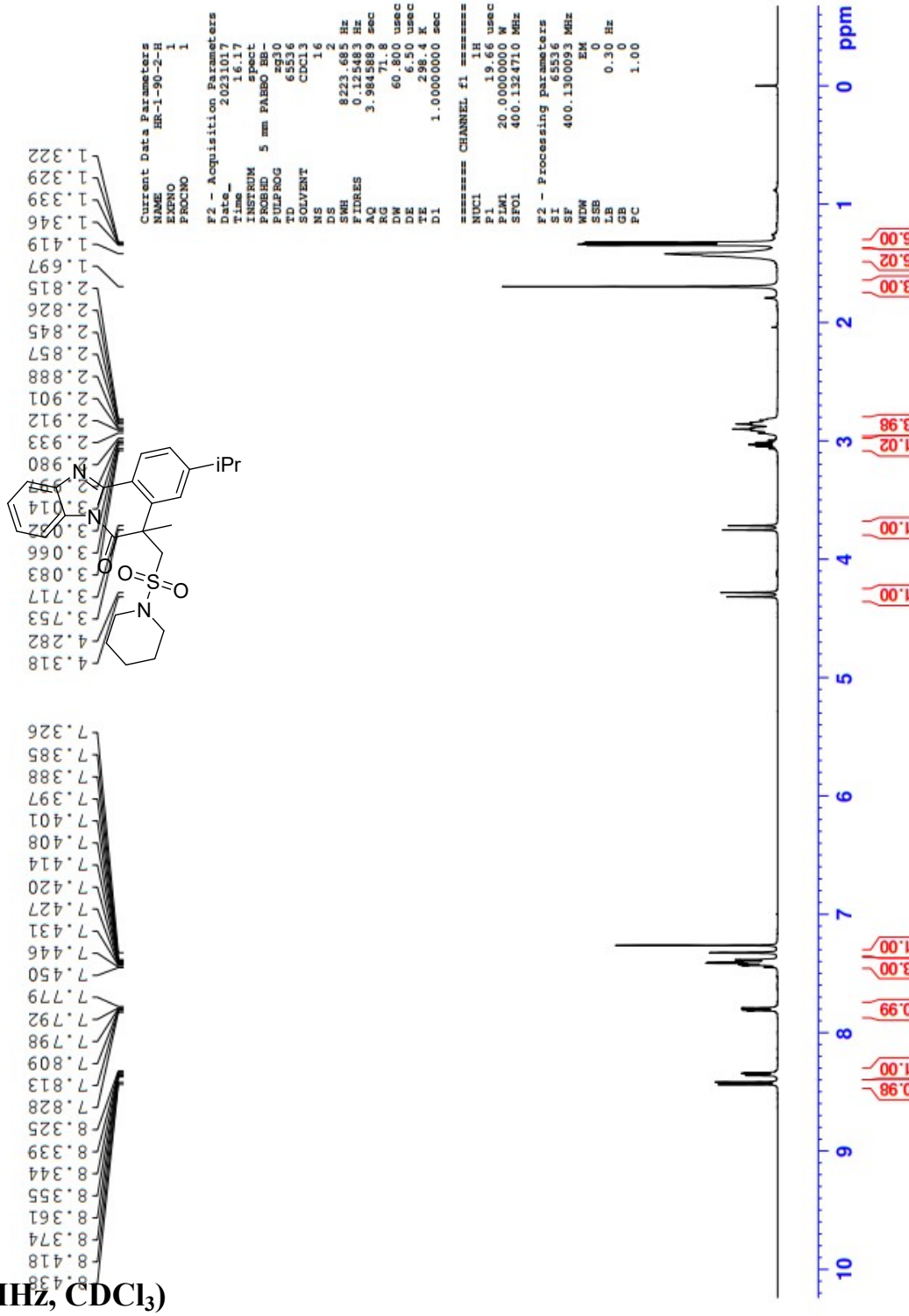

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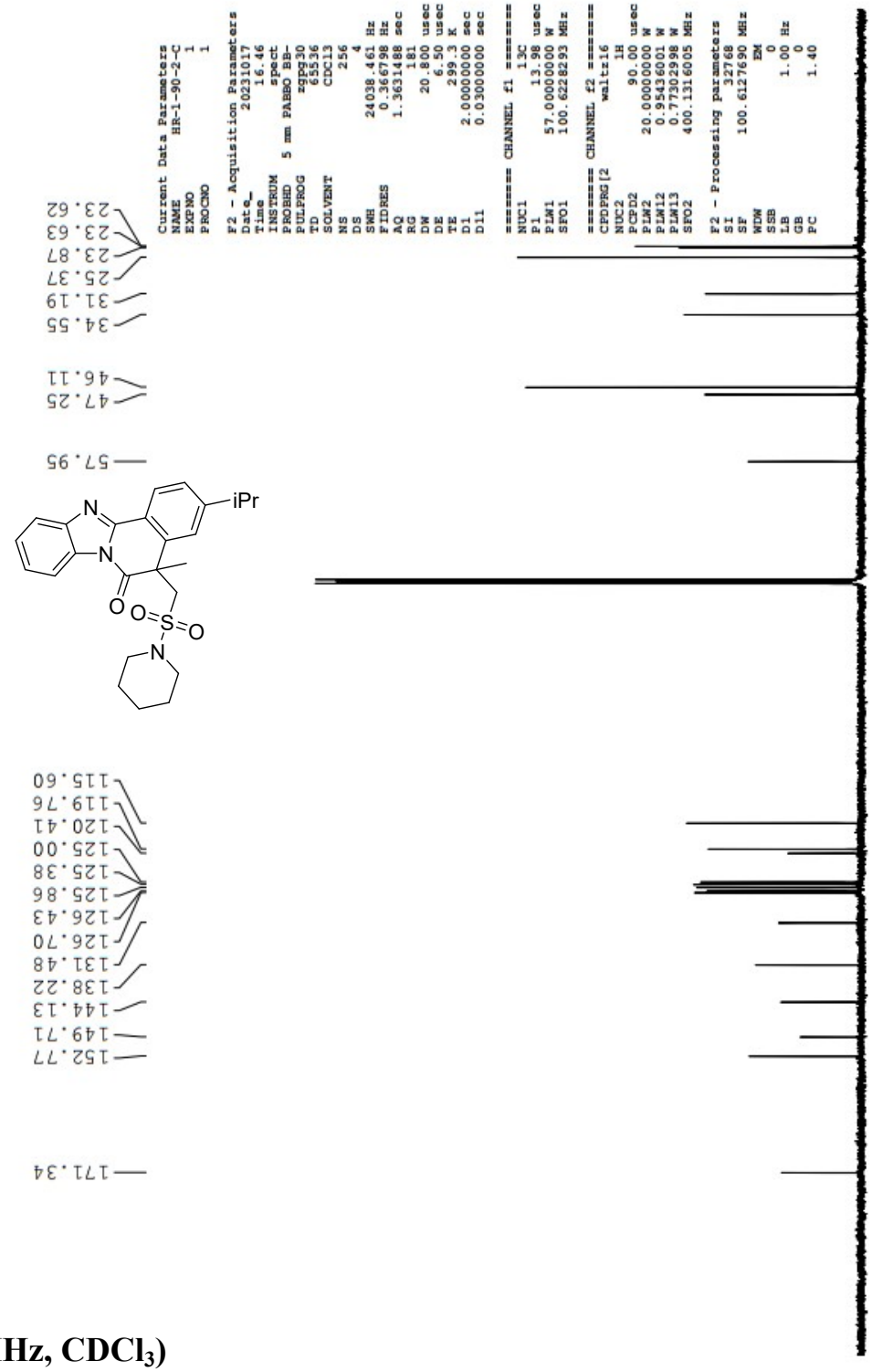

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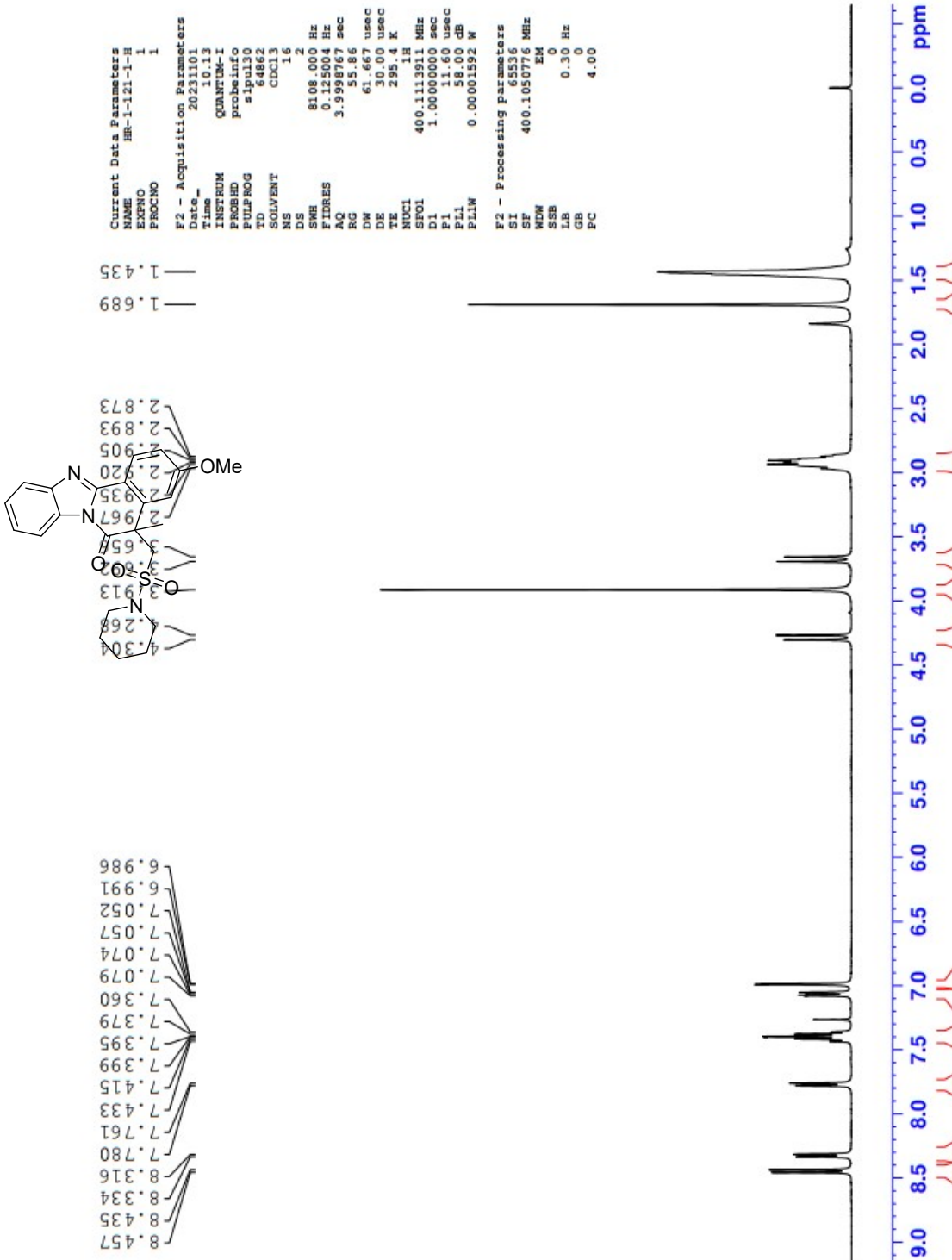

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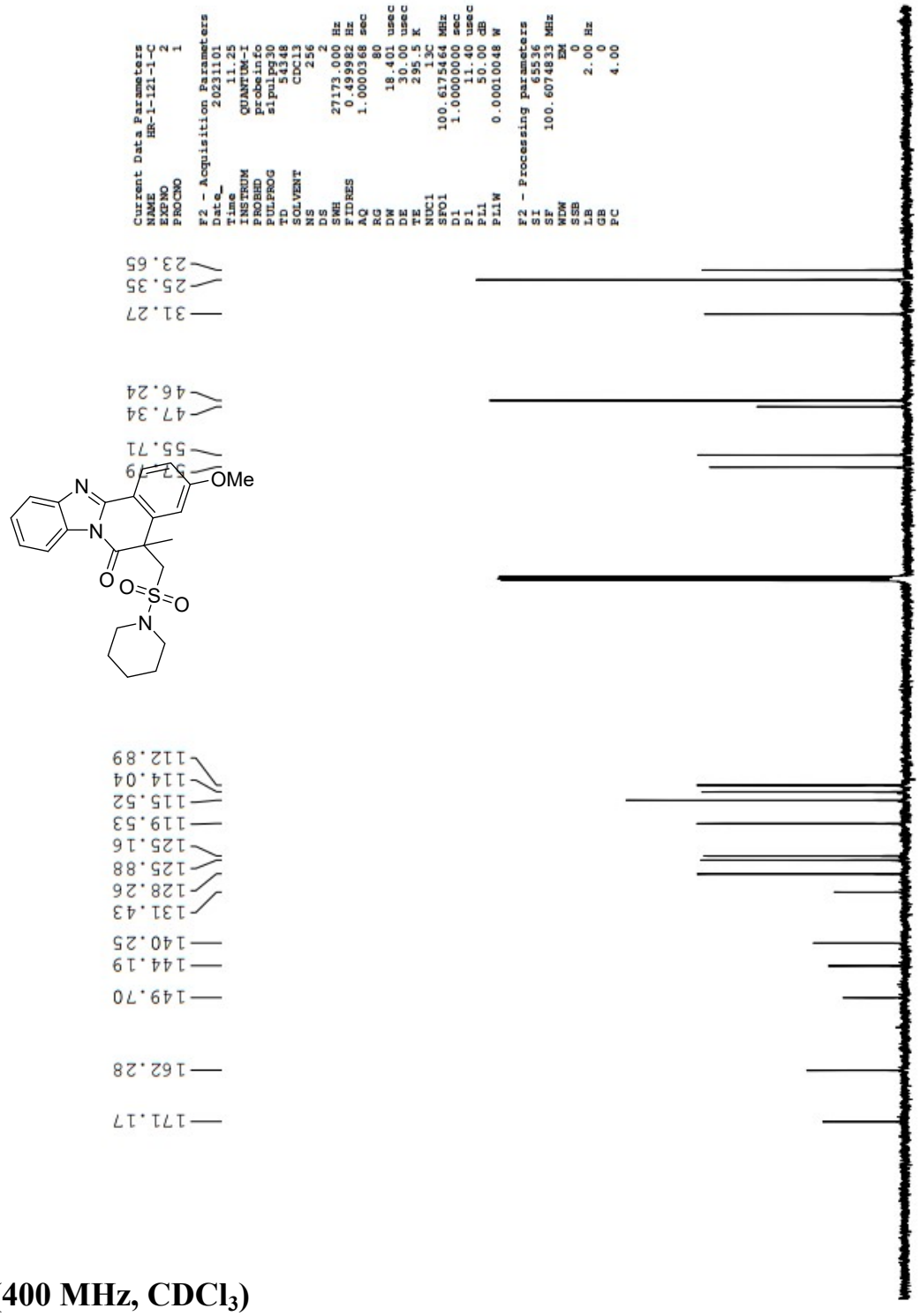

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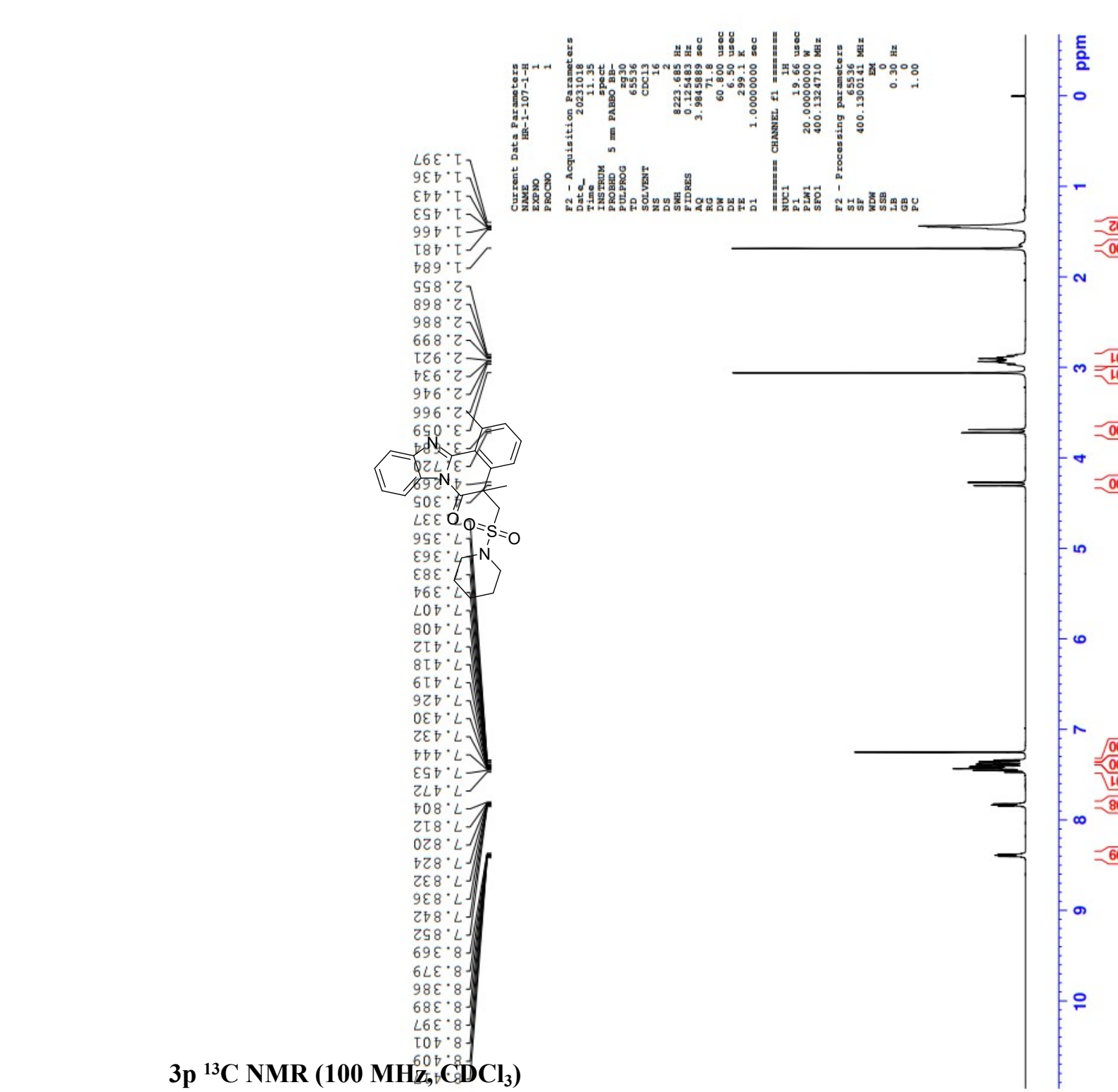

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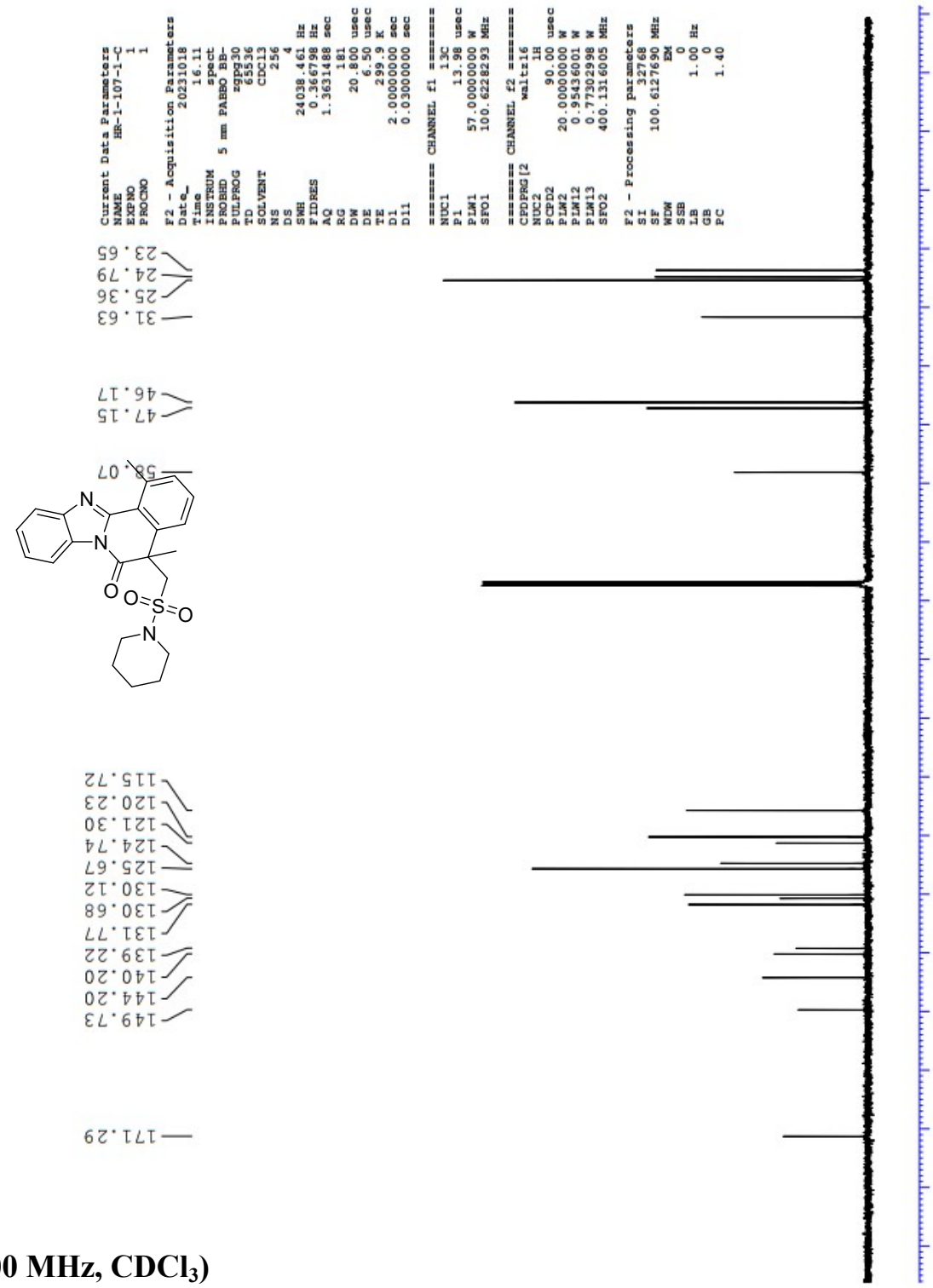

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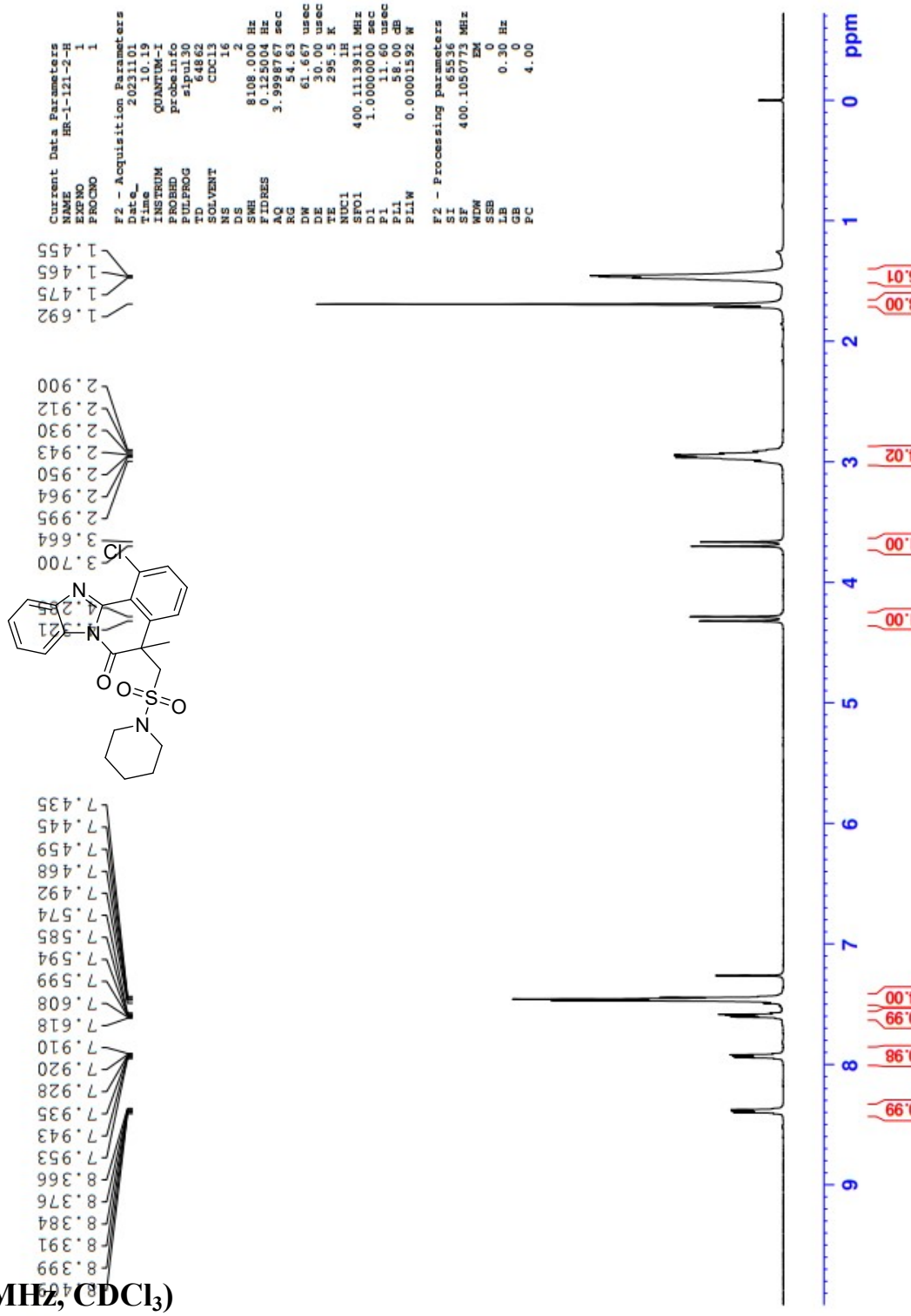
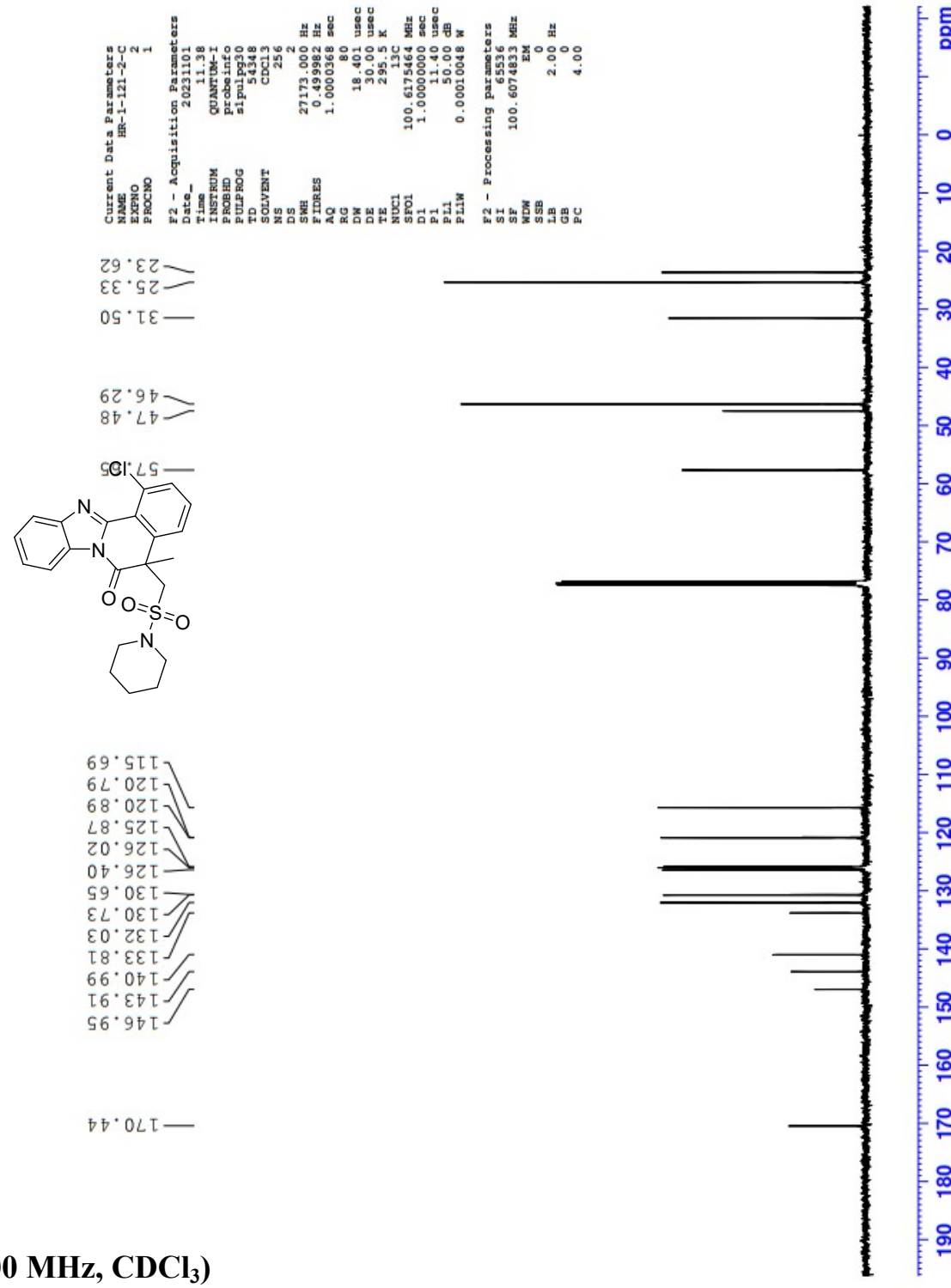


3q ^{13}C NMR (100 MHz, $CDCl_3$)



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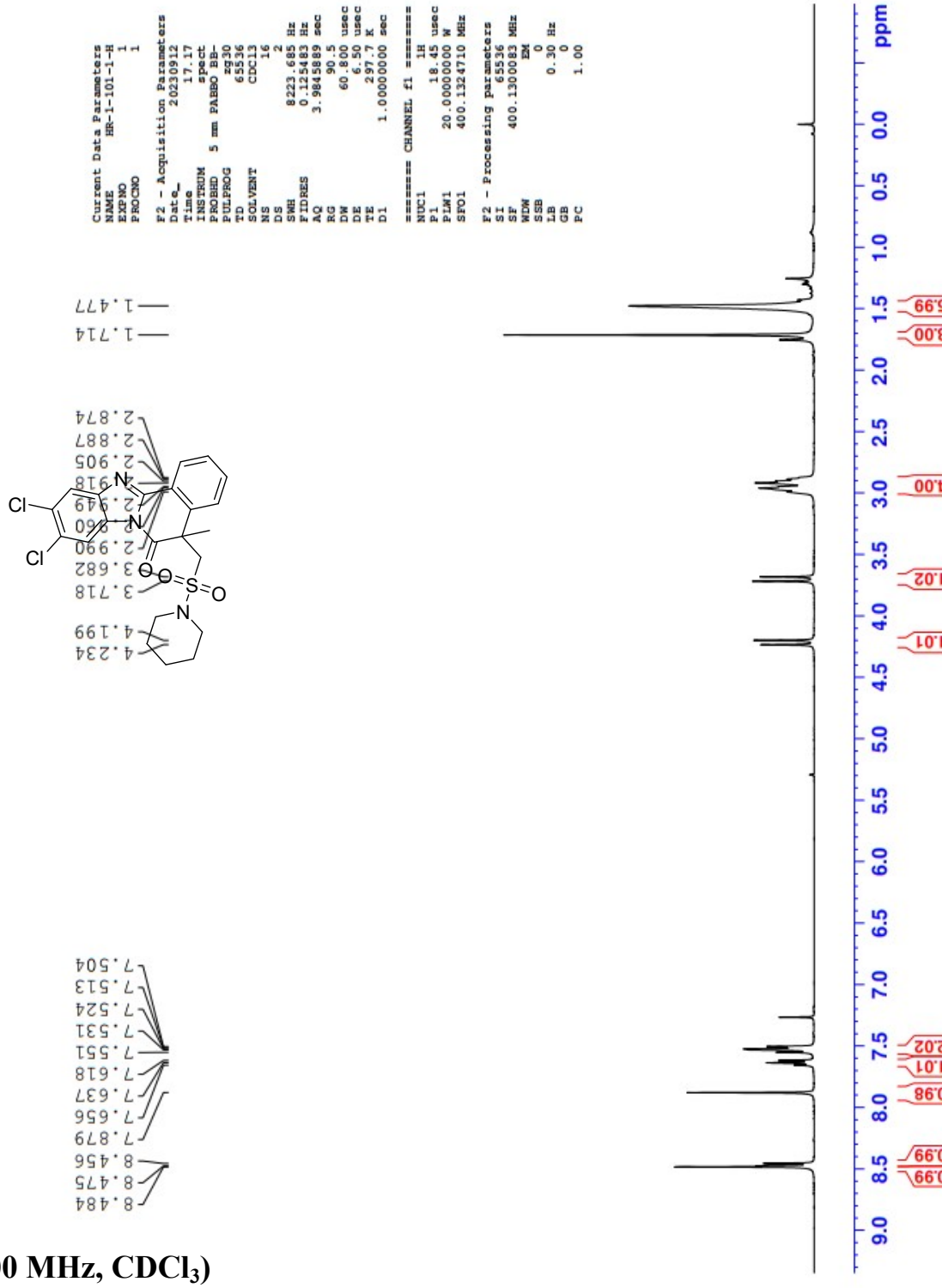

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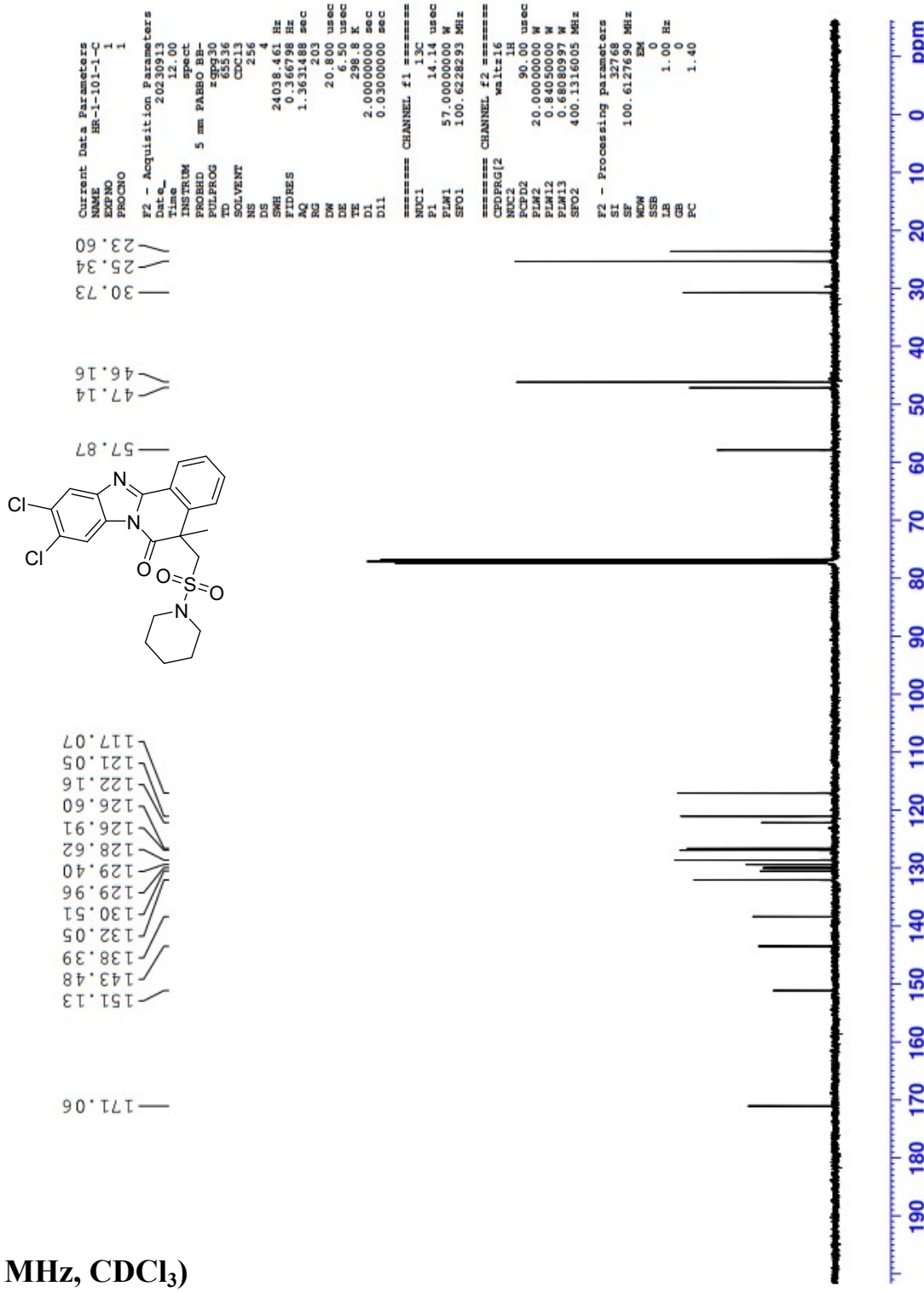

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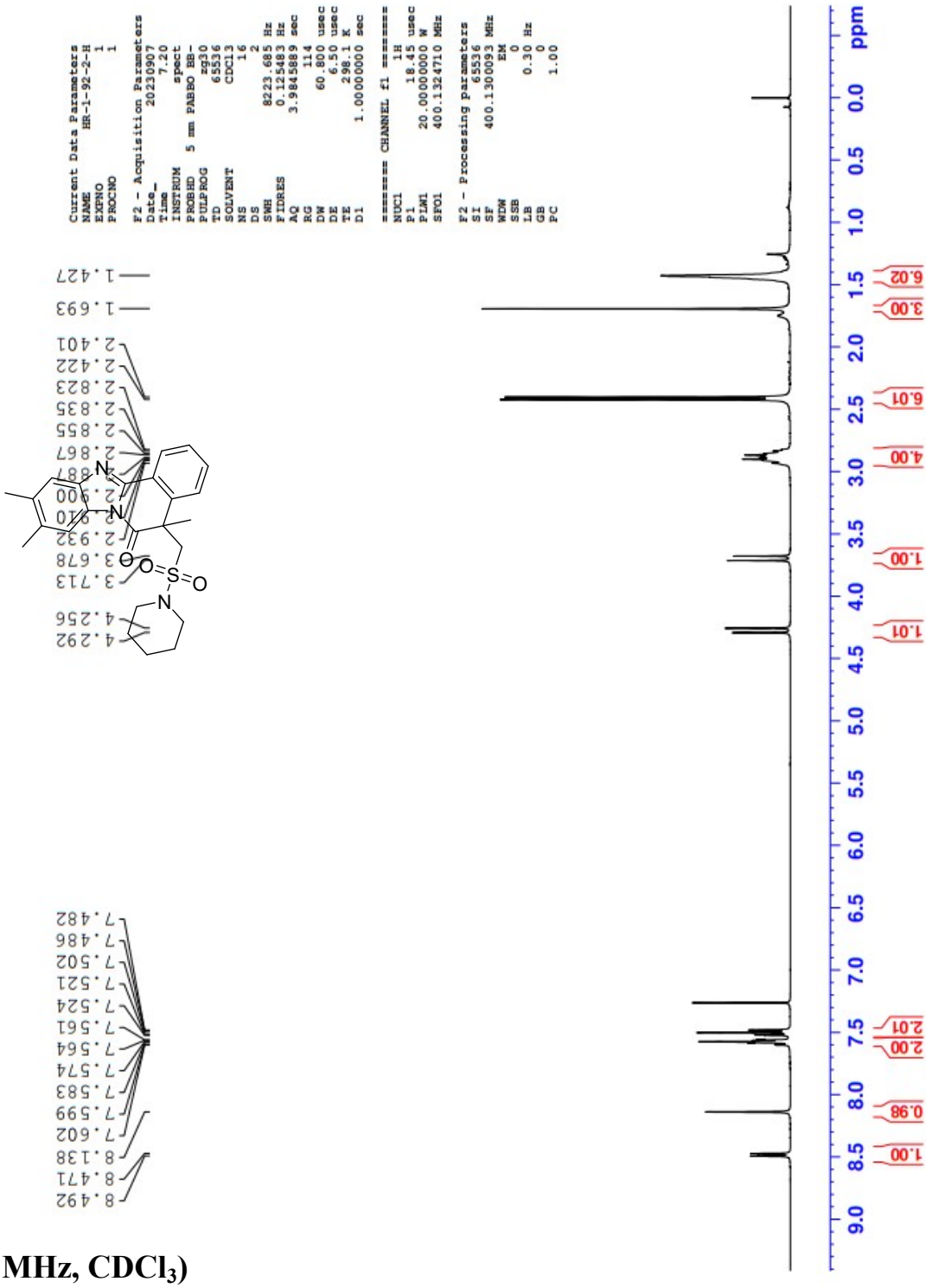

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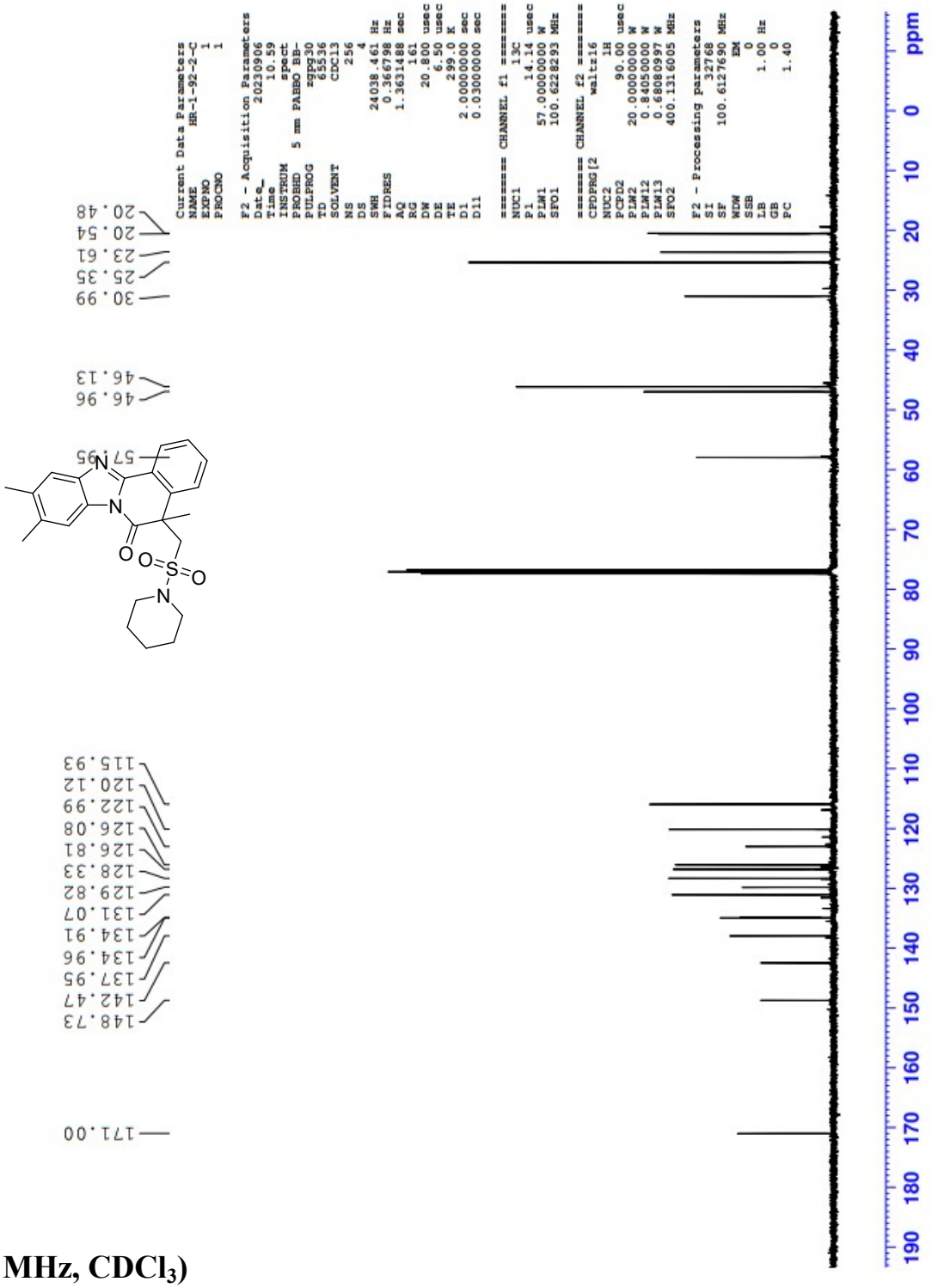

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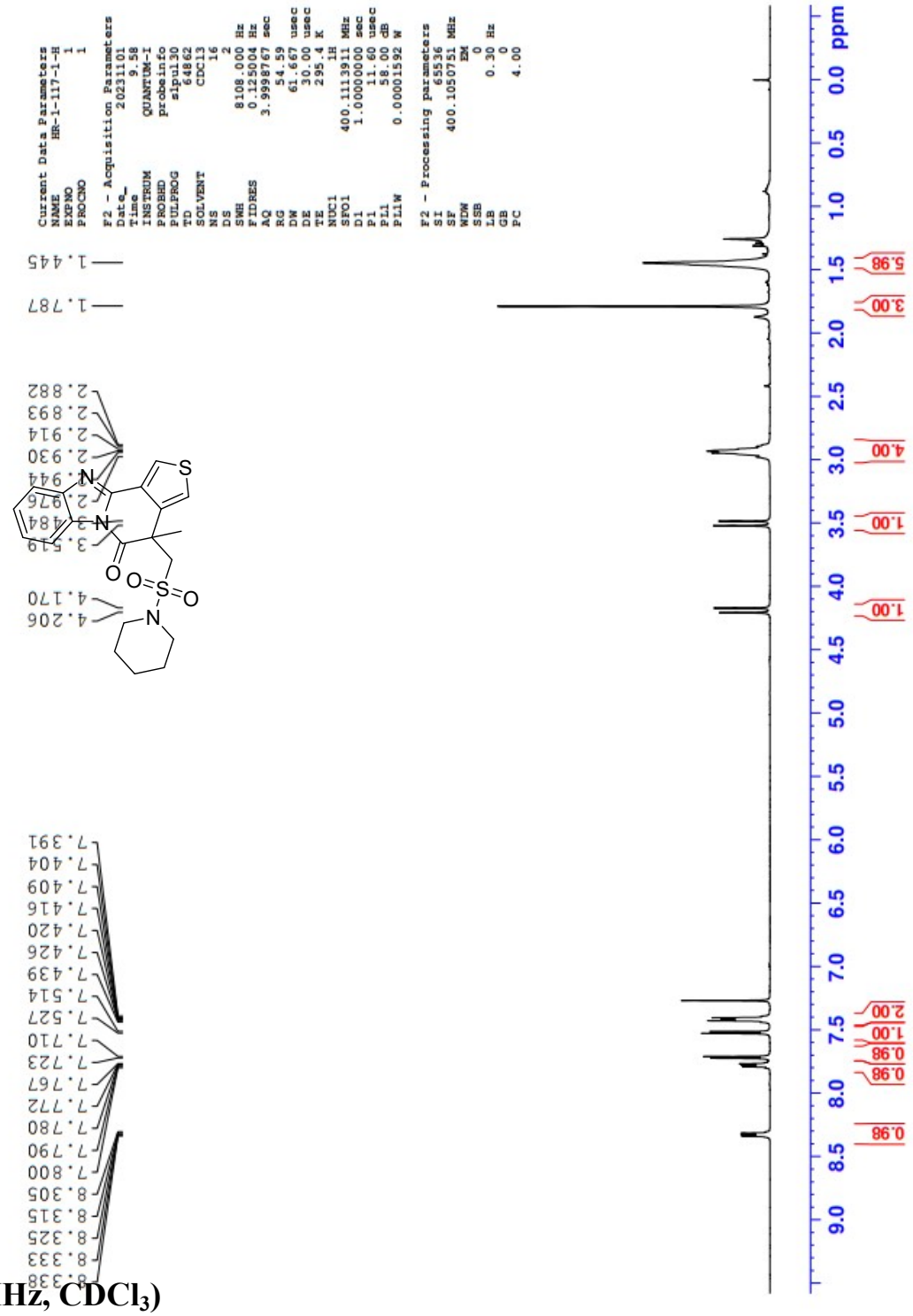

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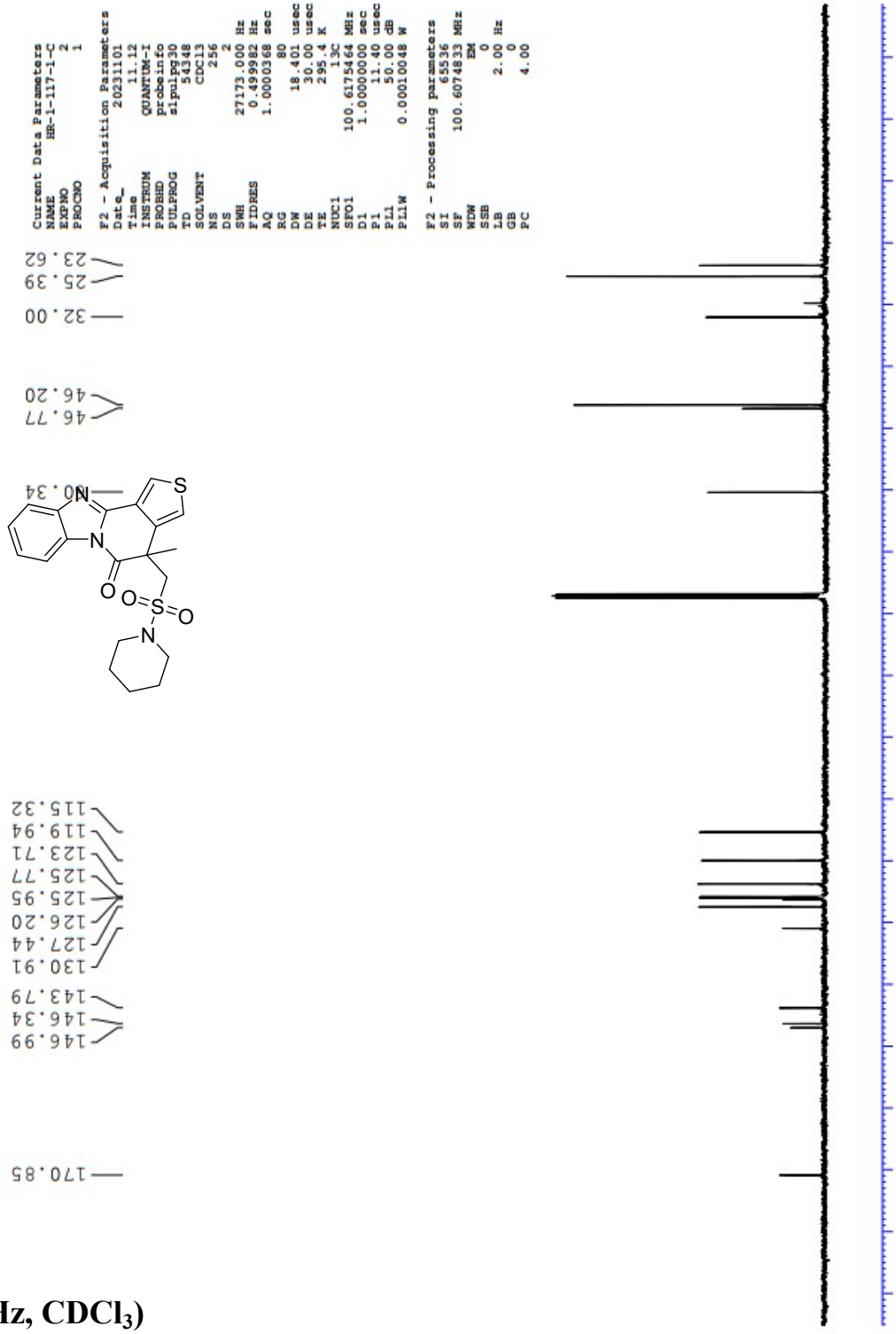

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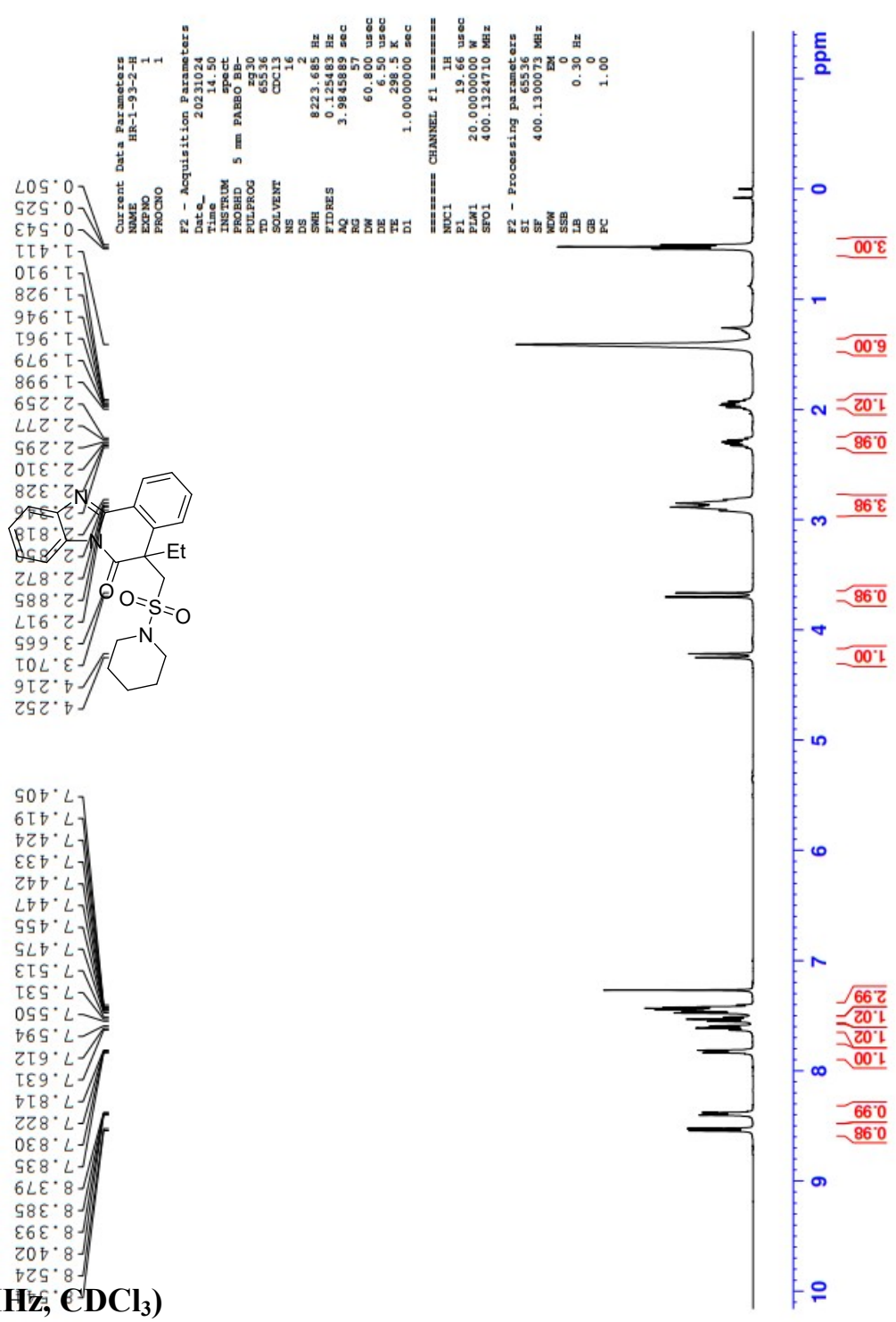
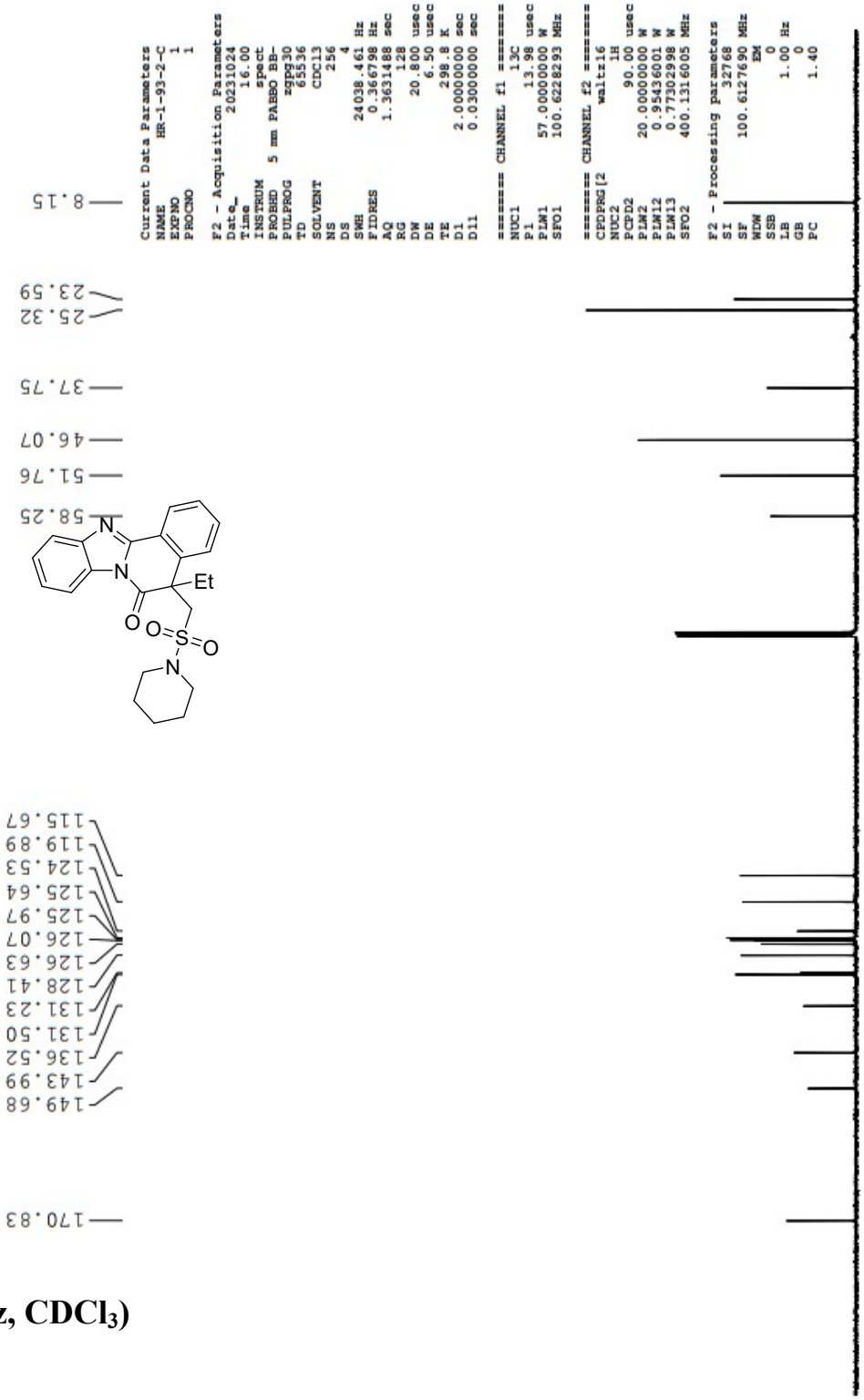
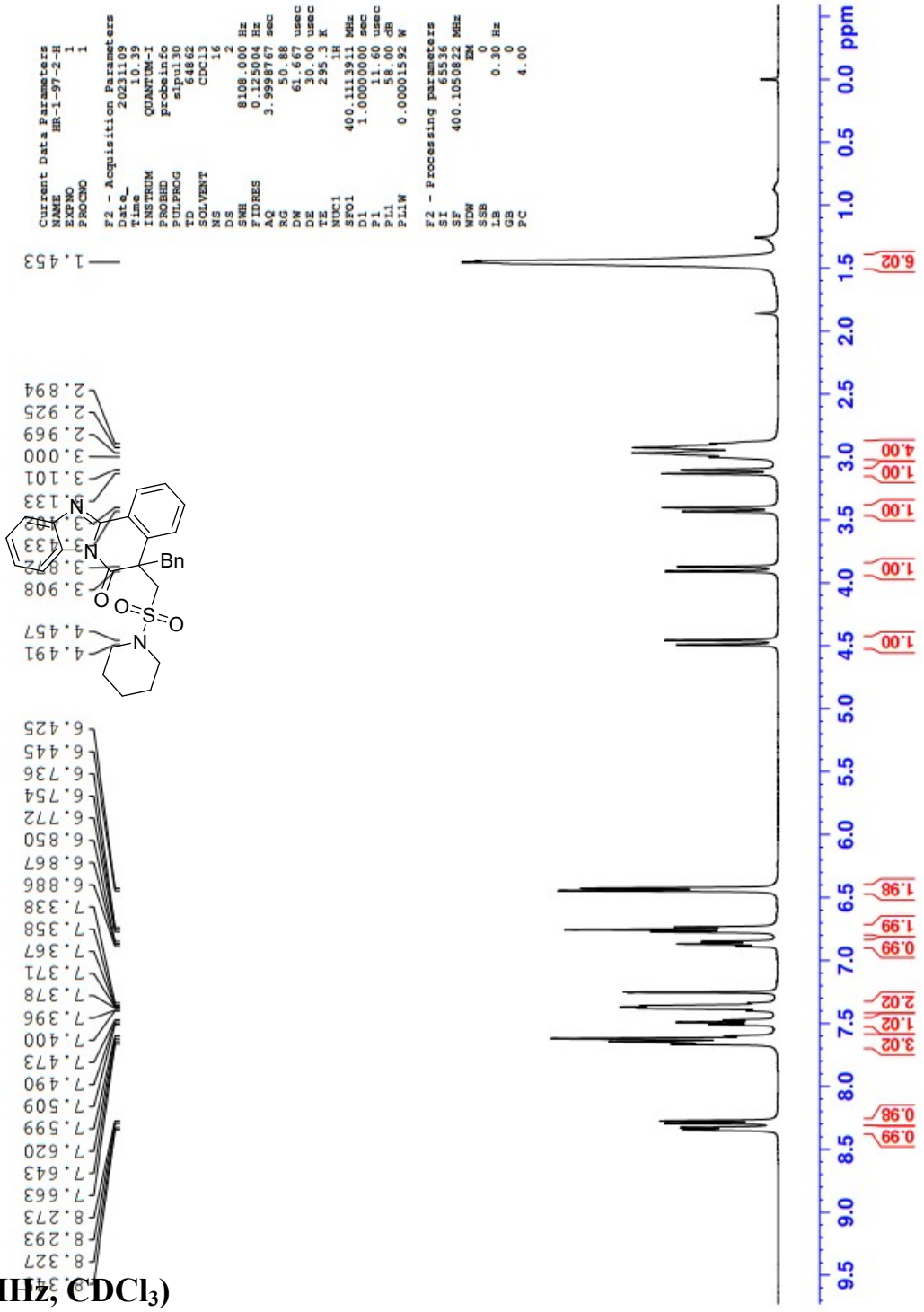


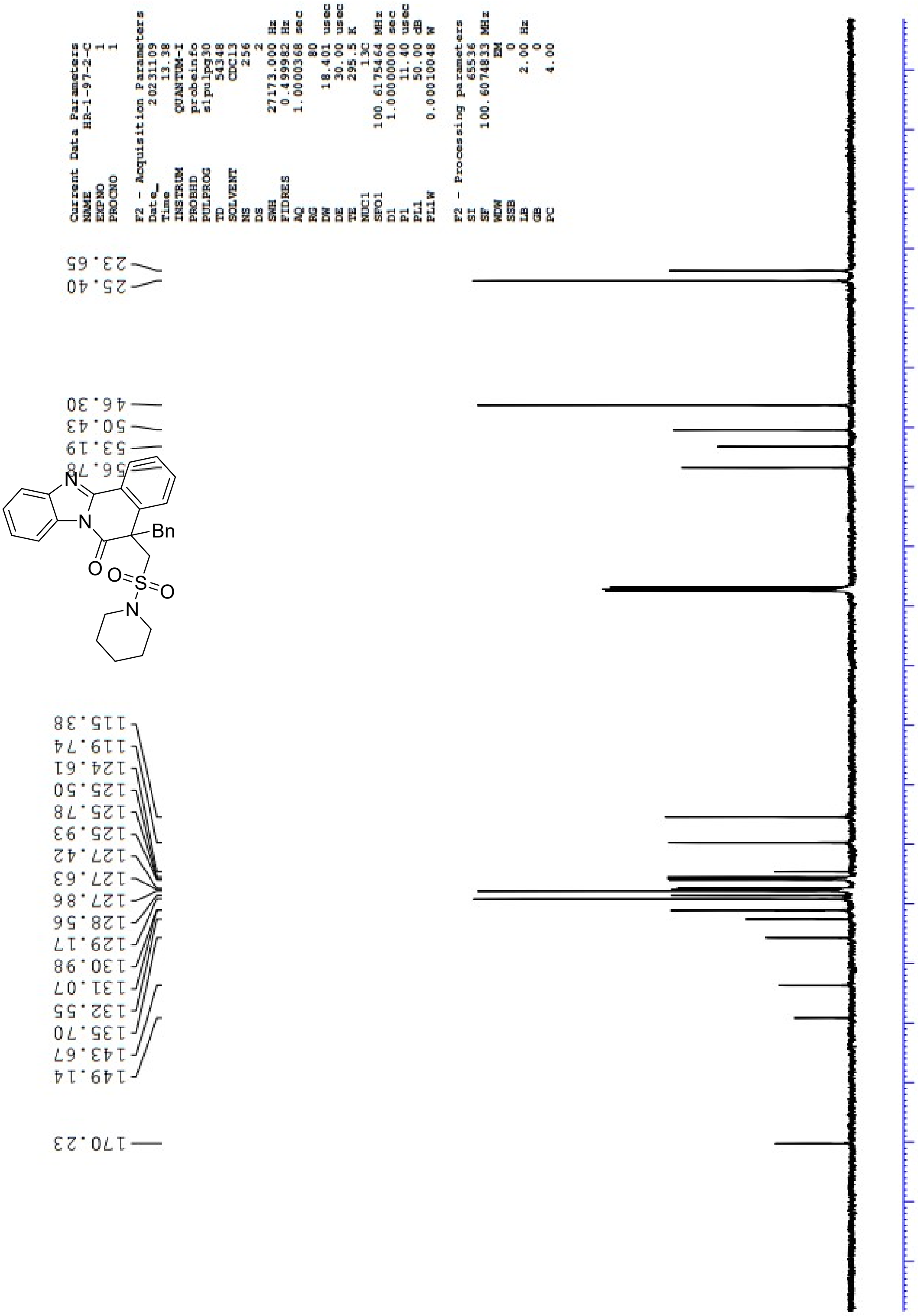
3u ^{13}C NMR (100 MHz, $CDCl_3$)



3v ^{1}H NMR (400 MHz, $CDCl_3$)



3v ^{13}C NMR (100 MHz, $CDCl_3$)


Current Data Parameters
NAME HR-1-97-2-C
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20231109
Time 13.38
INSTRUM QUANTUM-I
PROBHD probeinfo
PULPROG s1pulpg30
TD 54348
SOLVENT CDCl3
NS 256
DS 2
SWH 27173.000 Hz
FIDRES 0.499982 Hz
AQ 1.0000368 sec
RG 80
DW 18.401 usec
DE 30.00 usec
TE 295.5 K
NUC1 13C
SFO1 100.6175464 MHz
D1 1.00000000 sec
P1 11.40 usec
PL1 50.00 dB
PL1W 0.00010048 W
F2 - Processing parameters
SI 65536
SF 100.6074833 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 4.00
170.23
149.14
143.67
135.70
132.55
131.07
130.98
129.17
128.56
127.86
127.63
127.42
125.93
125.78
125.50
124.61
119.74
115.38
56.78
53.19
50.43
46.30
25.40
23.65
N
N
O
Bn
O
S
O
N