

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(5H)-ones**

Rong Huang,<sup>a</sup> Wenbo Wang,<sup>a</sup> Kui Lu<sup>b</sup> and Xia Zhao<sup>\*a</sup>

*<sup>a</sup>College of Chemistry, Tianjin Key Laboratory of Structure and Performance for  
Functional Molecules, Key laboratory of Inorganic-organic Hybrid Functional  
Material Chemistry, Ministry of Education, Tianjin Normal University, Tianjin,  
China, 300387*

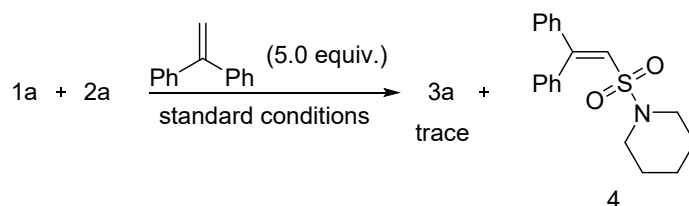
*<sup>b</sup>China International Science and Technology Cooperation Base of Food  
Nutrition/Safety and Medicinal Chemistry, College of Biotechnology, Tianjin  
University of Science & Technology, Tianjin, China, 300457*

\*E-mail: [hxyzhx@mail.tjnu.edu.cn](mailto:hxyzhx@mail.tjnu.edu.cn);

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## 1. The radical trapping experiments



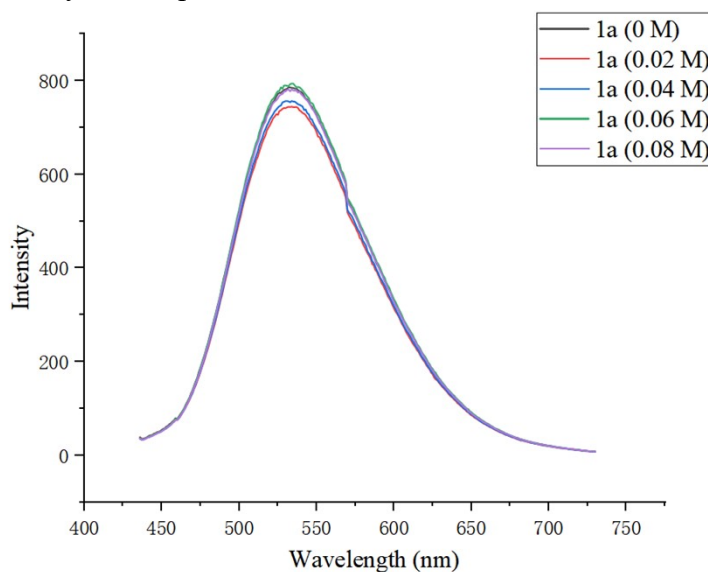
Add **1a** (0.20 mmol), **2a** (0.46 mmol), 1,1-stilbene (1.2 mmol, 4.0 equiv.) to the sealed tube and dry DMAc (1.0 mL). The reaction mixture was evacuated and with pure N<sub>2</sub> for three times and stirred under the irradiation of 36 W blue LEDs at room temperature for 1 h. After irradiation, 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<sub>2</sub>SO<sub>4</sub> 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**: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): 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.

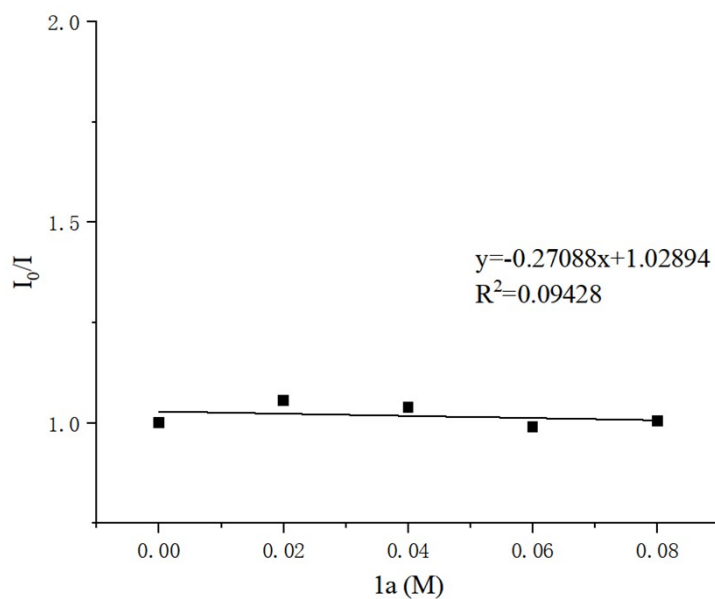
**For Experiment 1:** Constant photocatalyst; Varied 2-methyl-1-(2-phenyl-1H-benzo[d]imidazol-1-yl)prop-2-en-1-one.

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.



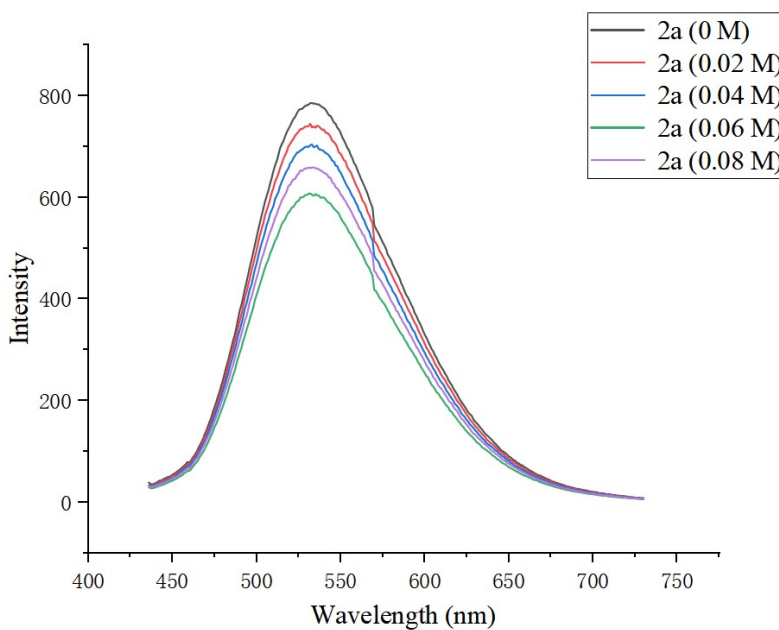
Supplementary figure 1: Fluorescence spectrum of PC + **1a**.

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



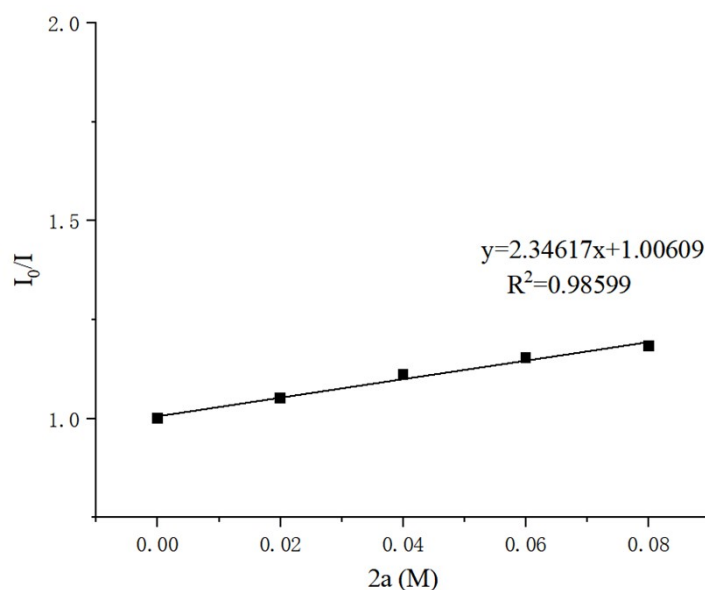
**Supplementary figure 2:** Stern-Volmer Luminescence Quenching Analysis of PC + **1a**.

**For Experiment 2:** Constant photocatalyst; Varied piperidine-1-sulfonyl chloride. Add 30  $\mu\text{L}$  4-CzIPN-Br solution and 0  $\mu\text{L}$ , 60  $\mu\text{L}$ , 120  $\mu\text{L}$ , 180  $\mu\text{L}$ , 240  $\mu\text{L}$  **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.



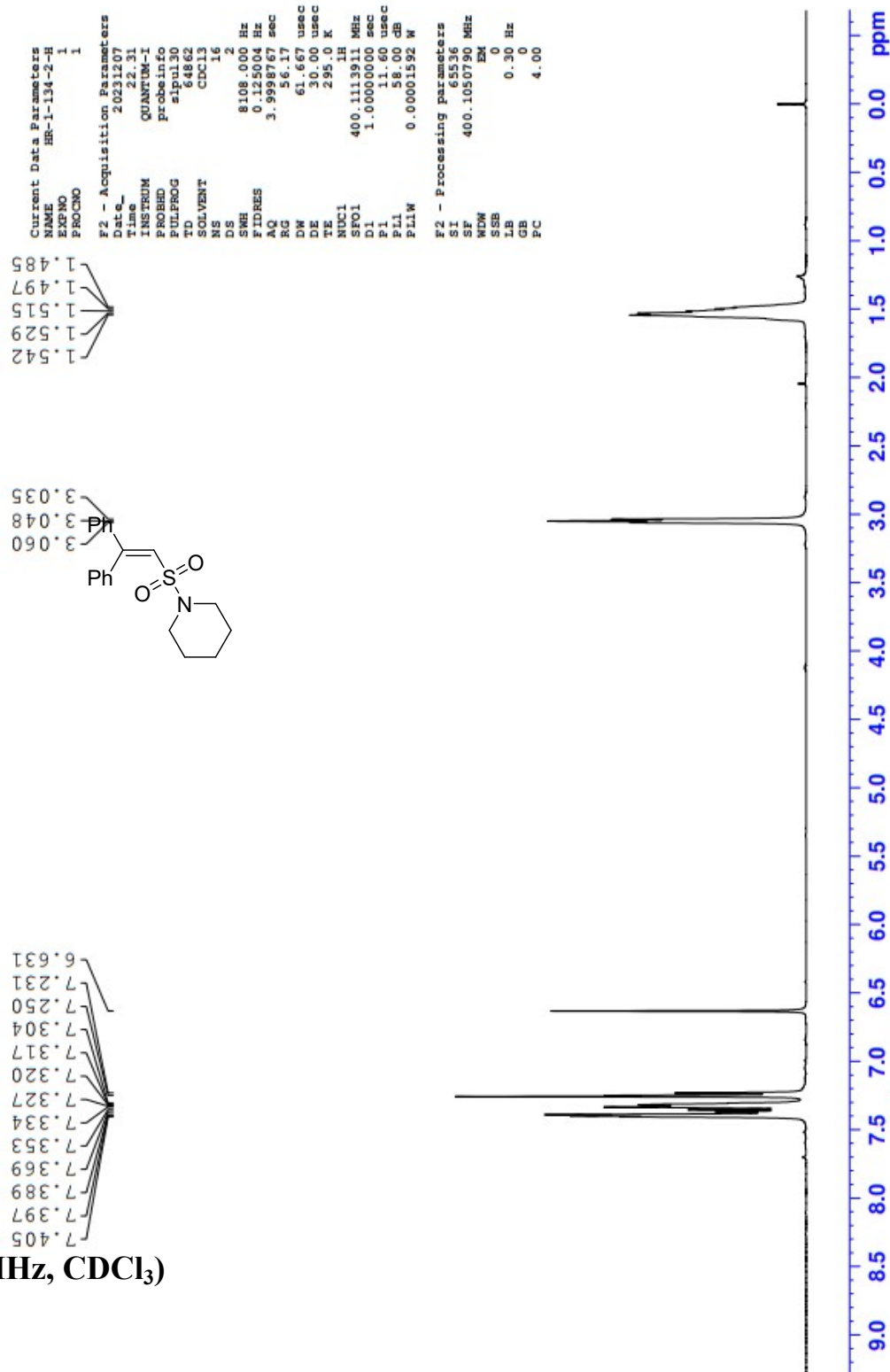
**Supplementary figure 4:** Stern-Volmer Luminescence Quenching Analysis of PC + 2a.

### 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 °C 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  $\text{Na}_2\text{SO}_4$  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. $^1\text{H}$ NMR, $^{13}\text{C}$

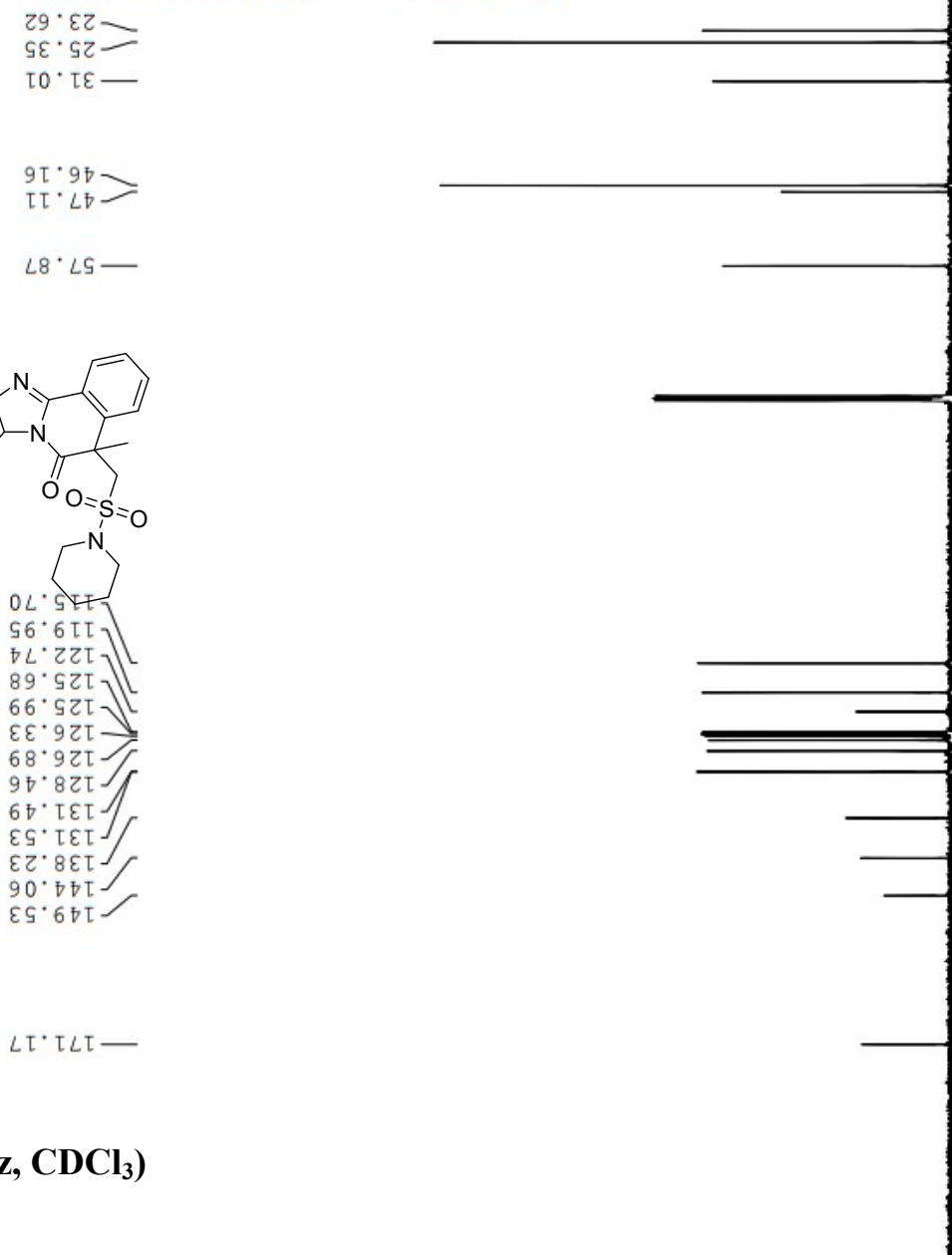
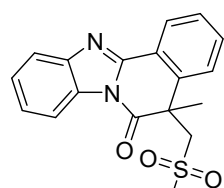
## 4 $^1\text{H}$ NMR (400



## 3a $^1\text{H}$ NMR (400 MHz, $\text{CDCl}_3$ )

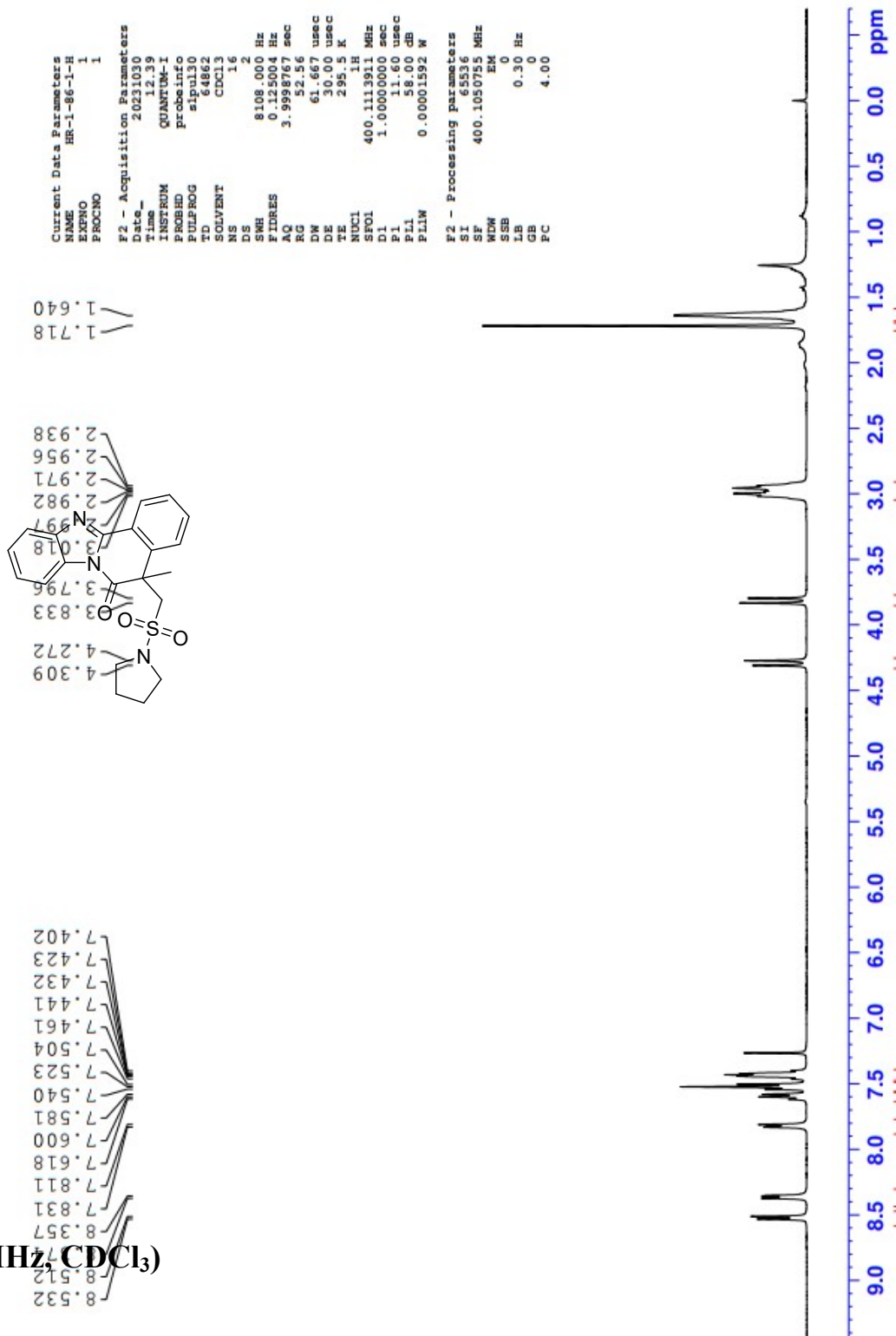


**3b <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**

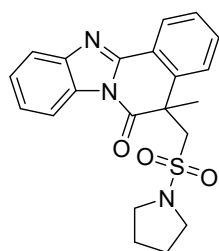




**3b <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



# 3c <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



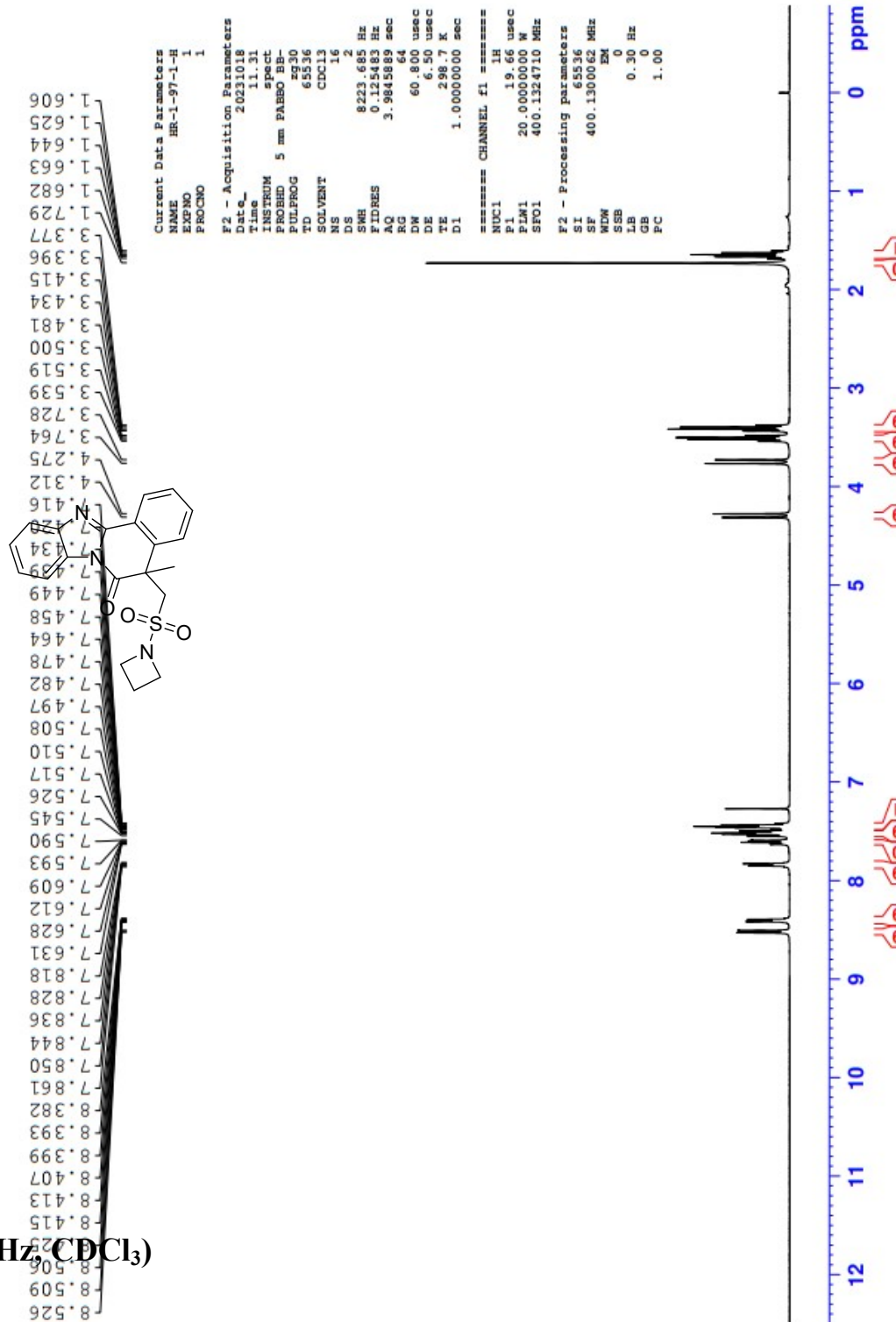
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122.74  
119.93  
115.66

58.78  
47.17  
47.01

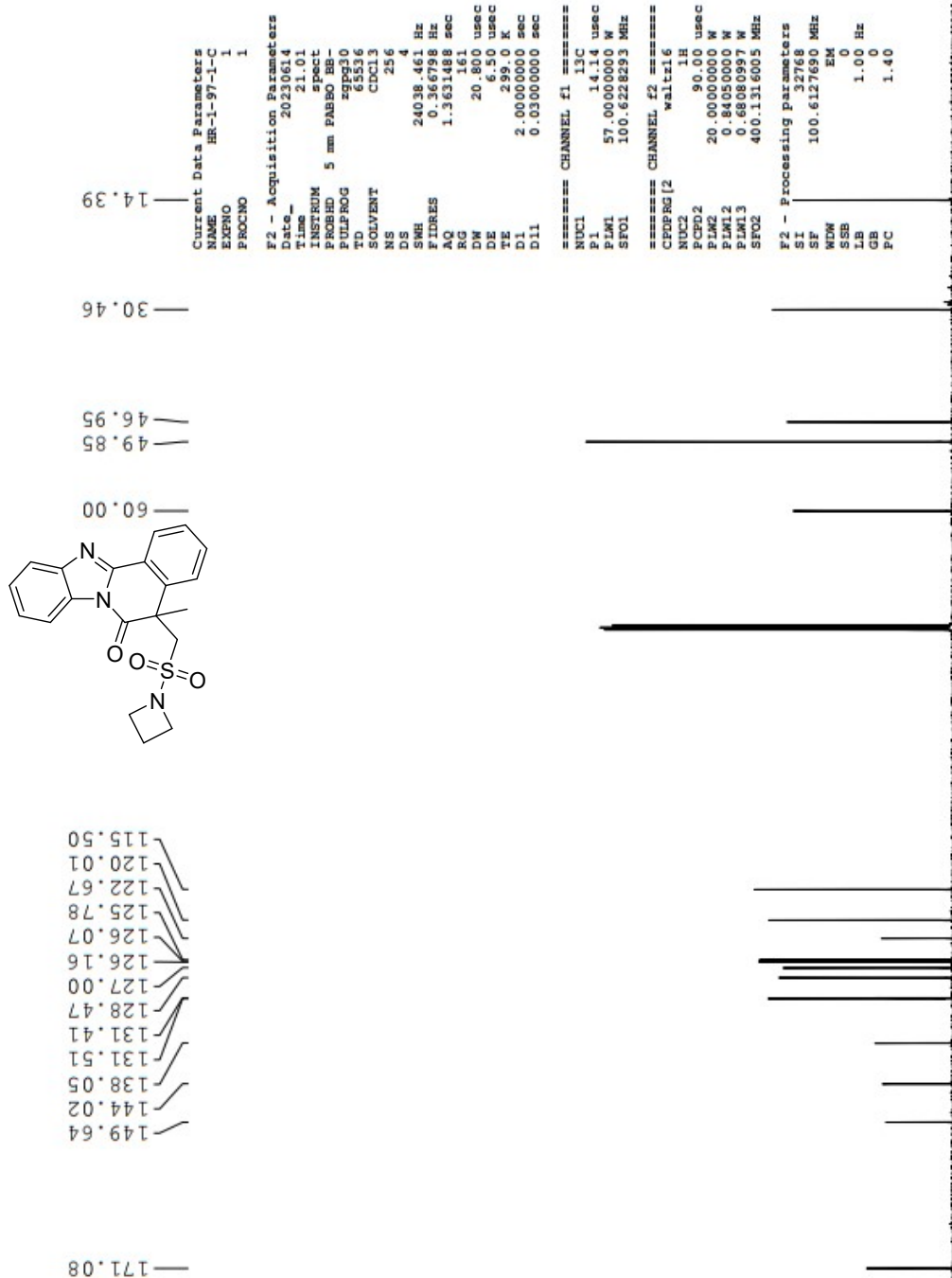
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25.51

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FIDRES 0.366798 Hz  
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DE 6.50 usec  
TE 298.7 K  
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PL1 57.0000000 W  
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GB 0  
FC 1.40

3c <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



3d <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



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RG 32  
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TE 298.2 K  
D1 1.0000000 sec

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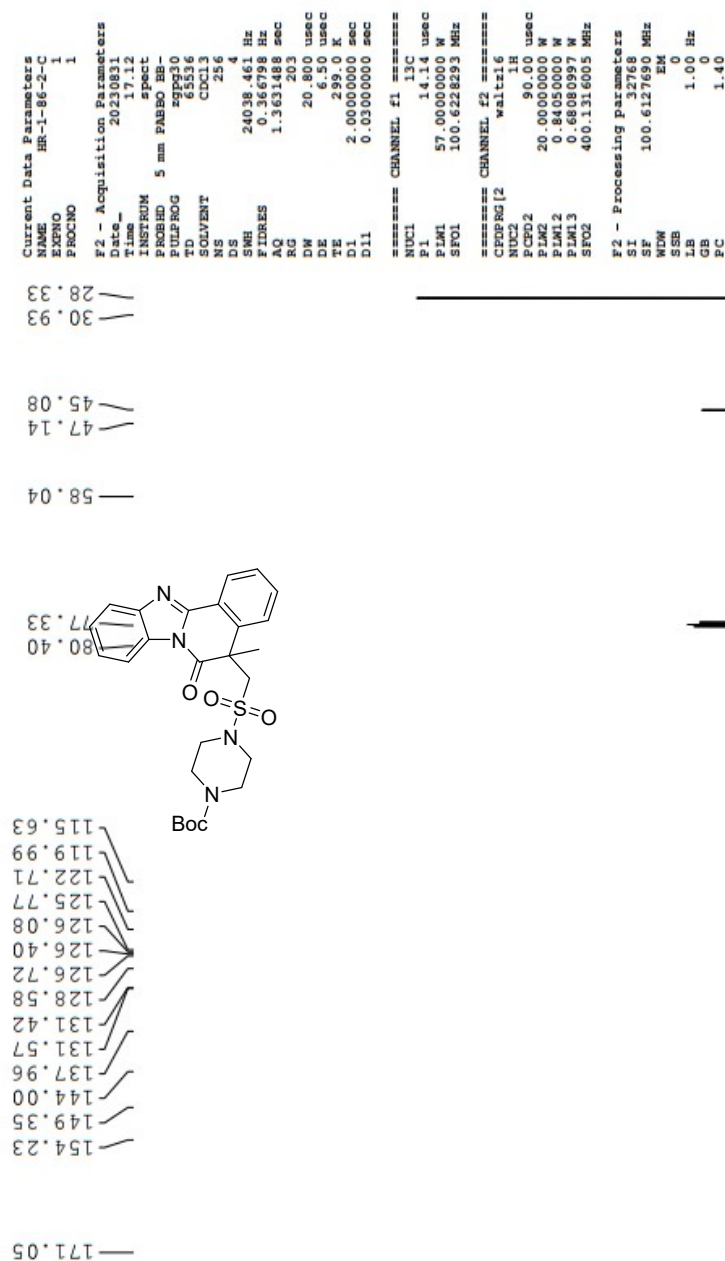
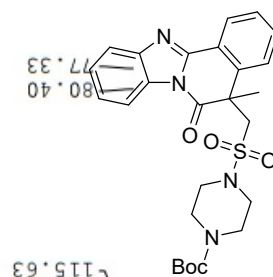
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2.941  
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4.308  
4.308  
4.272  
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7.436  
7.445  
7.465  
7.485  
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7.619  
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8.535

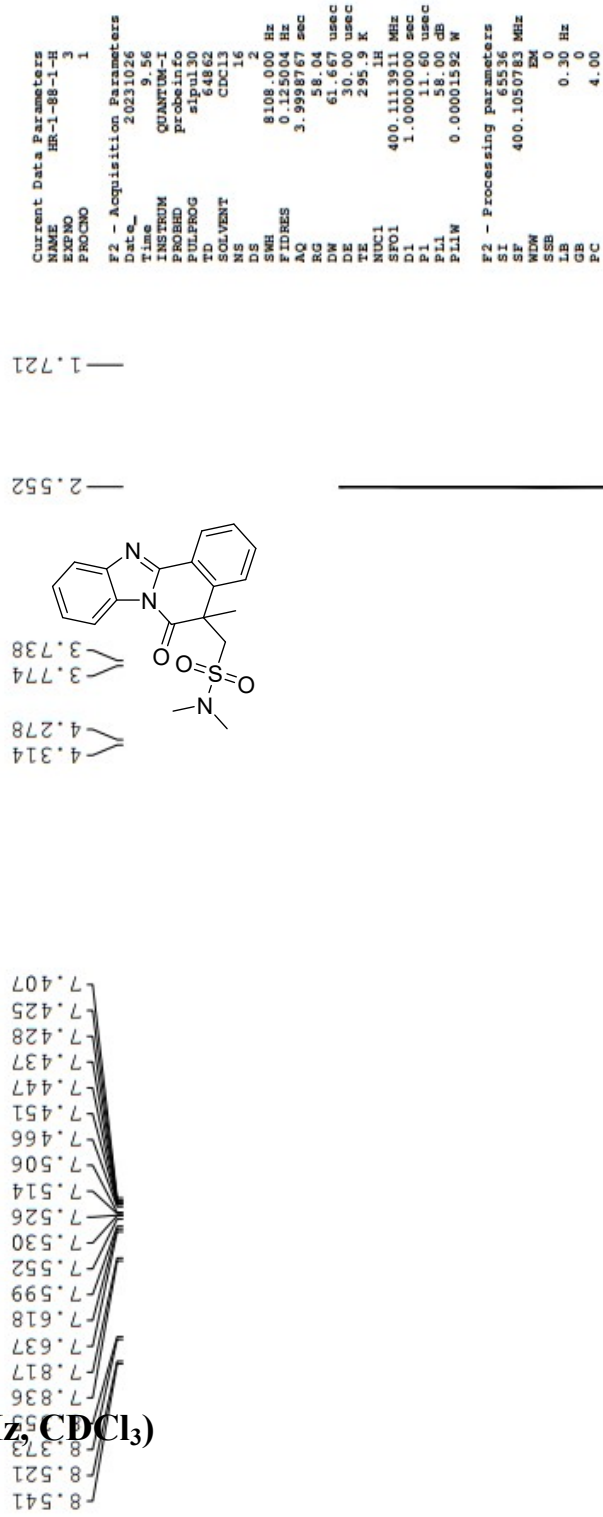
Chemical structure of a complex molecule, likely a derivative of a natural product, showing a central carbon atom bonded to a phenyl ring, a piperidine ring, and a sulfonamide group. The structure is labeled with various atoms and bonds, including a Boc group and a sulfonamide group.

1H NMR (400 MHz, CDCl<sub>3</sub>)

3e <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

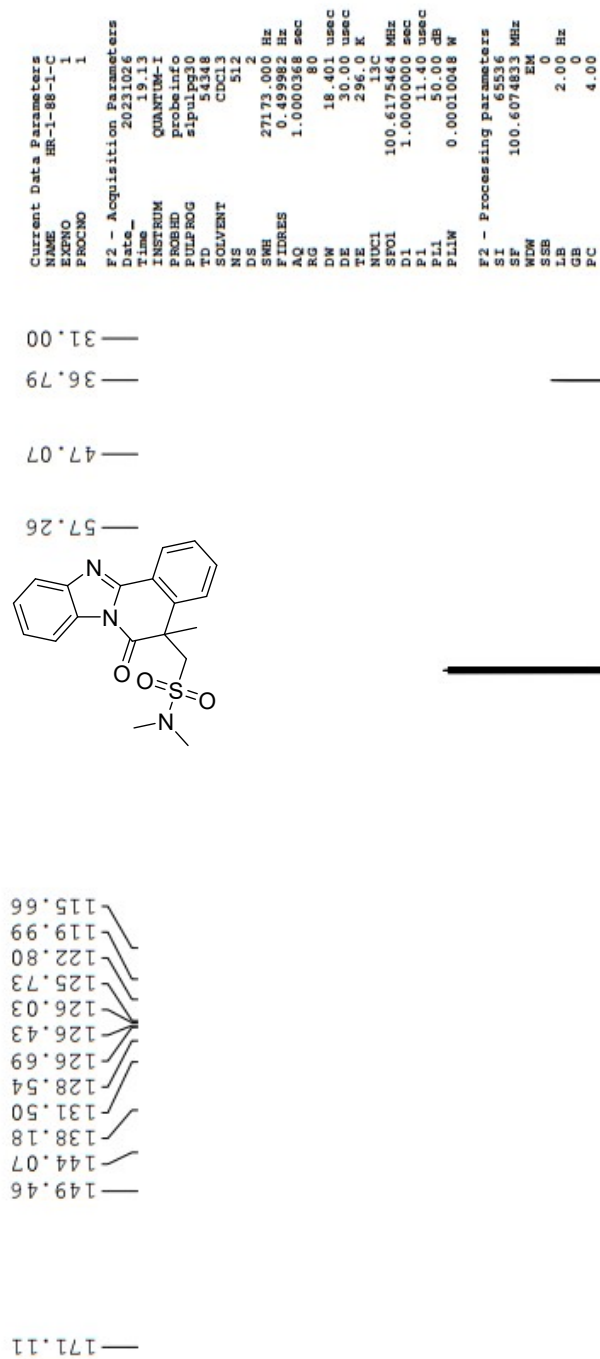


**3e  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )**



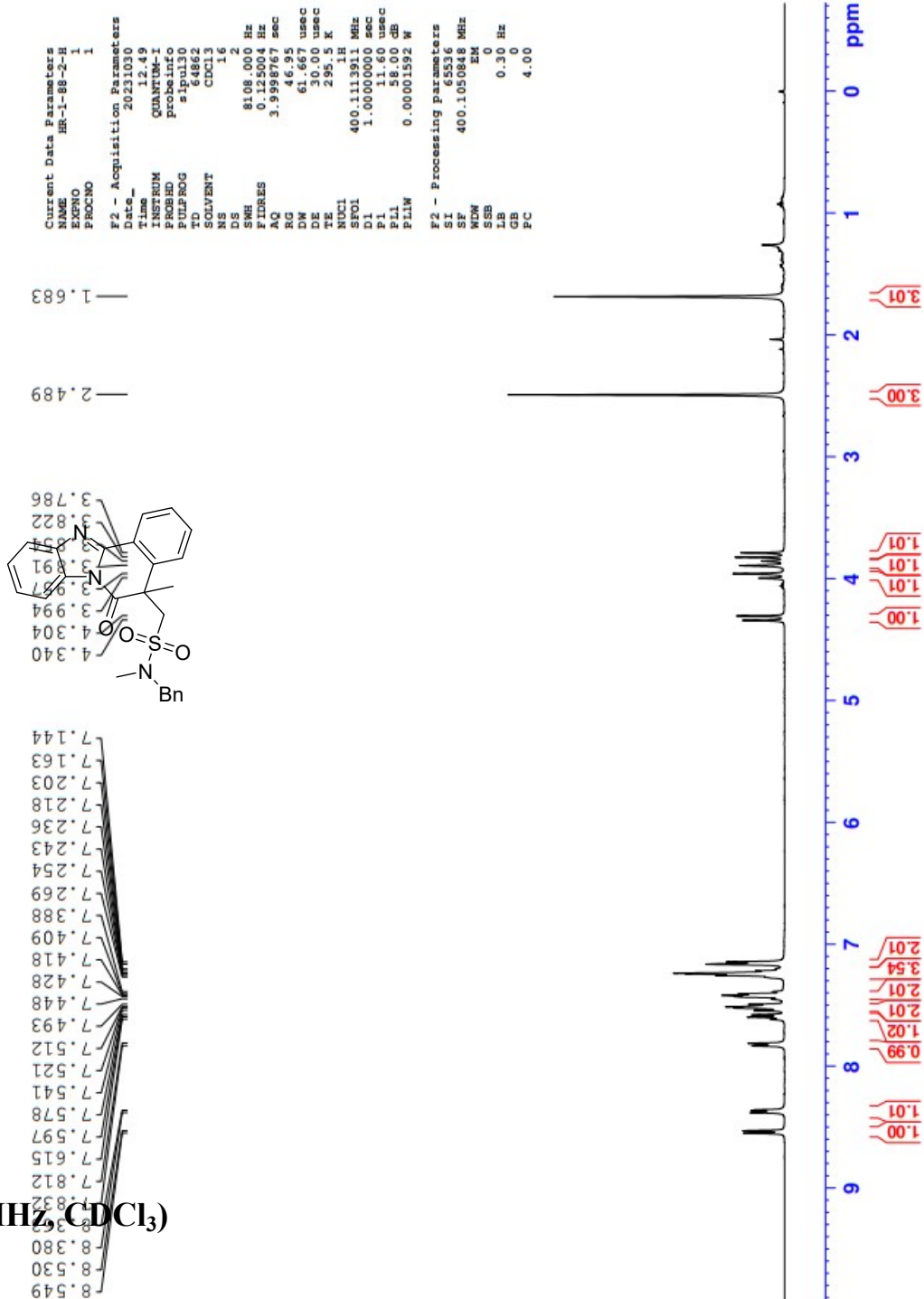


3f <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

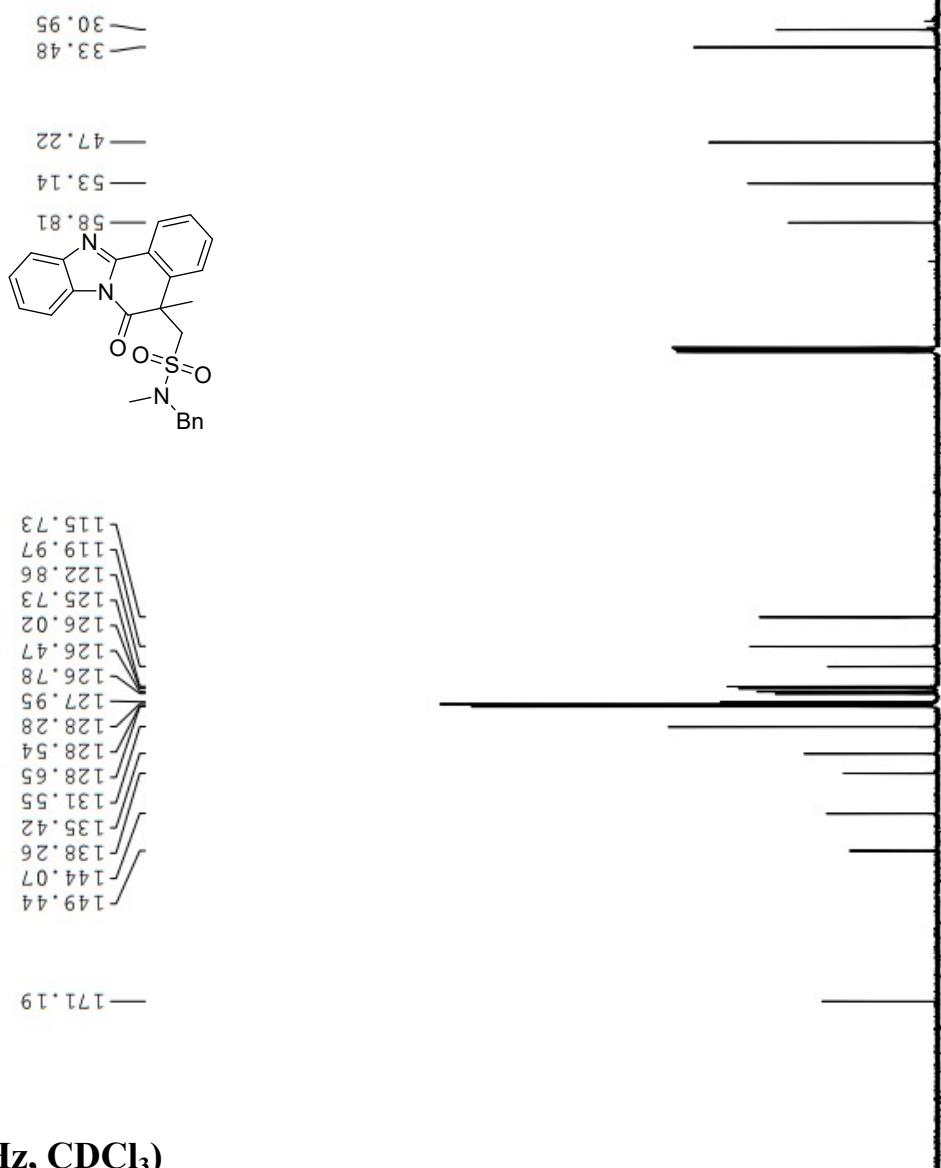




3f <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



3g <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



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PROCNO    1

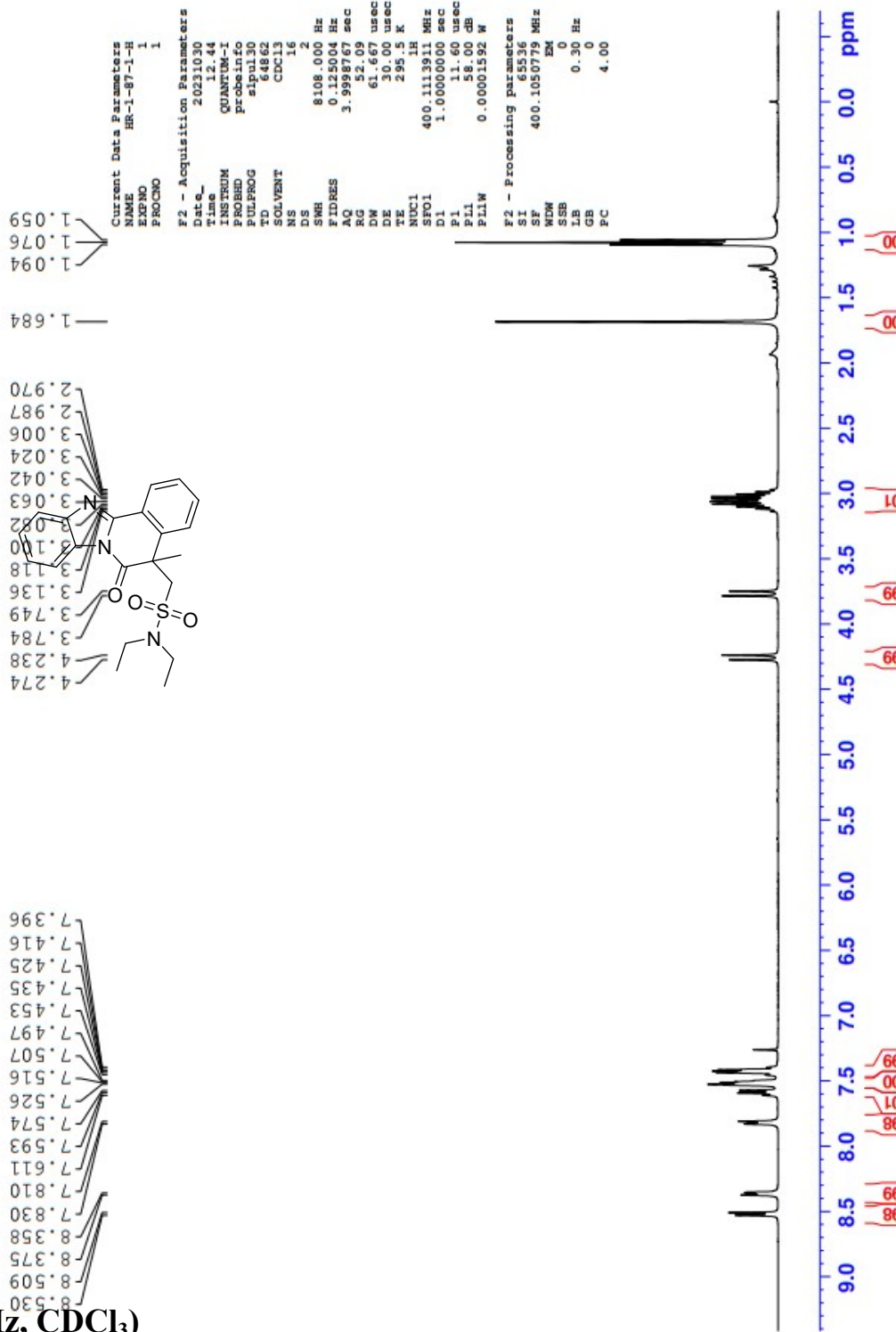
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FIDRES     0.366798 Hz
AQ          1.3631488 sec
RG          203
DM          20.800 usec
DE          6.50 usec
TE          299.6 K
D1          2.0000000 sec
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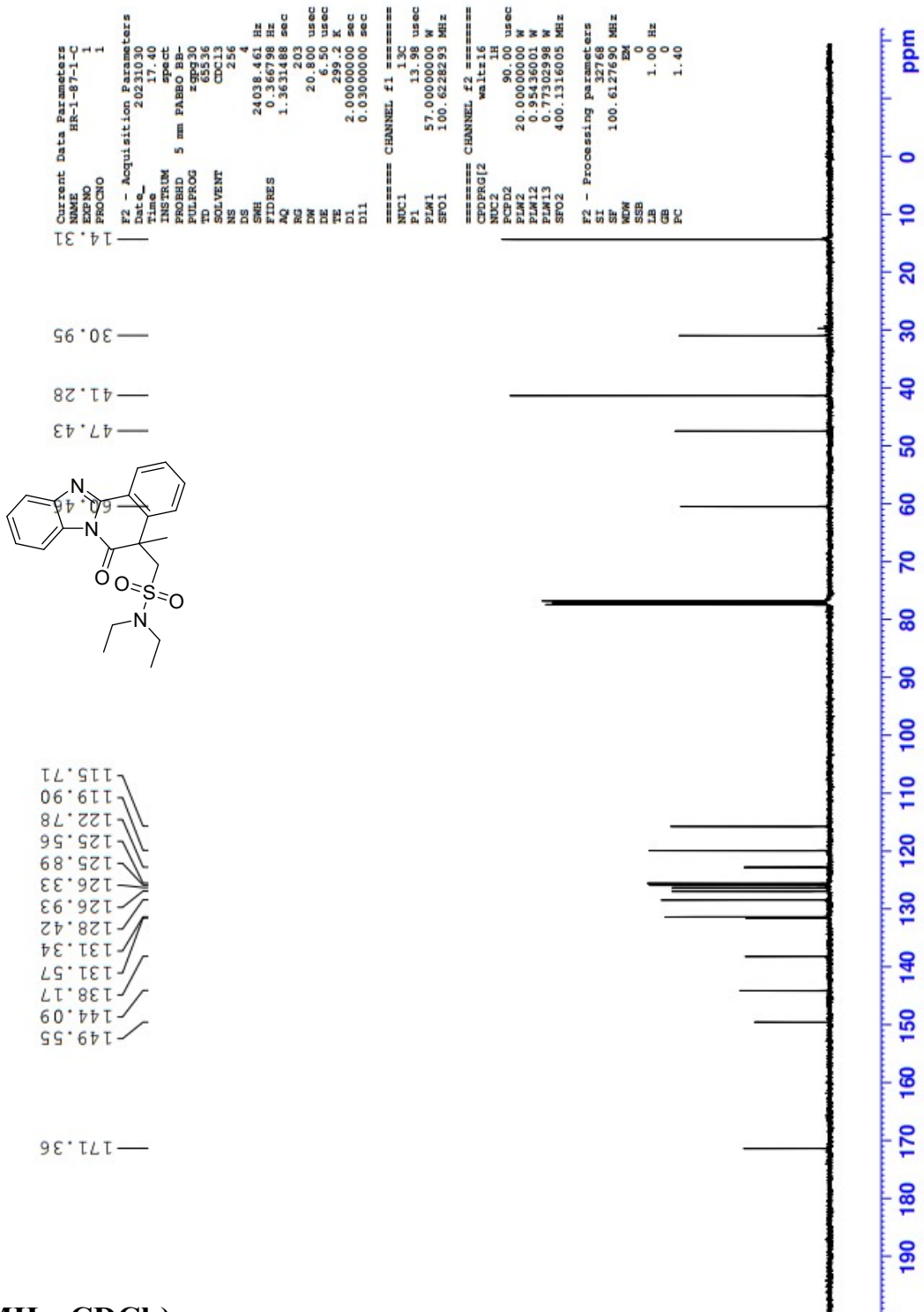
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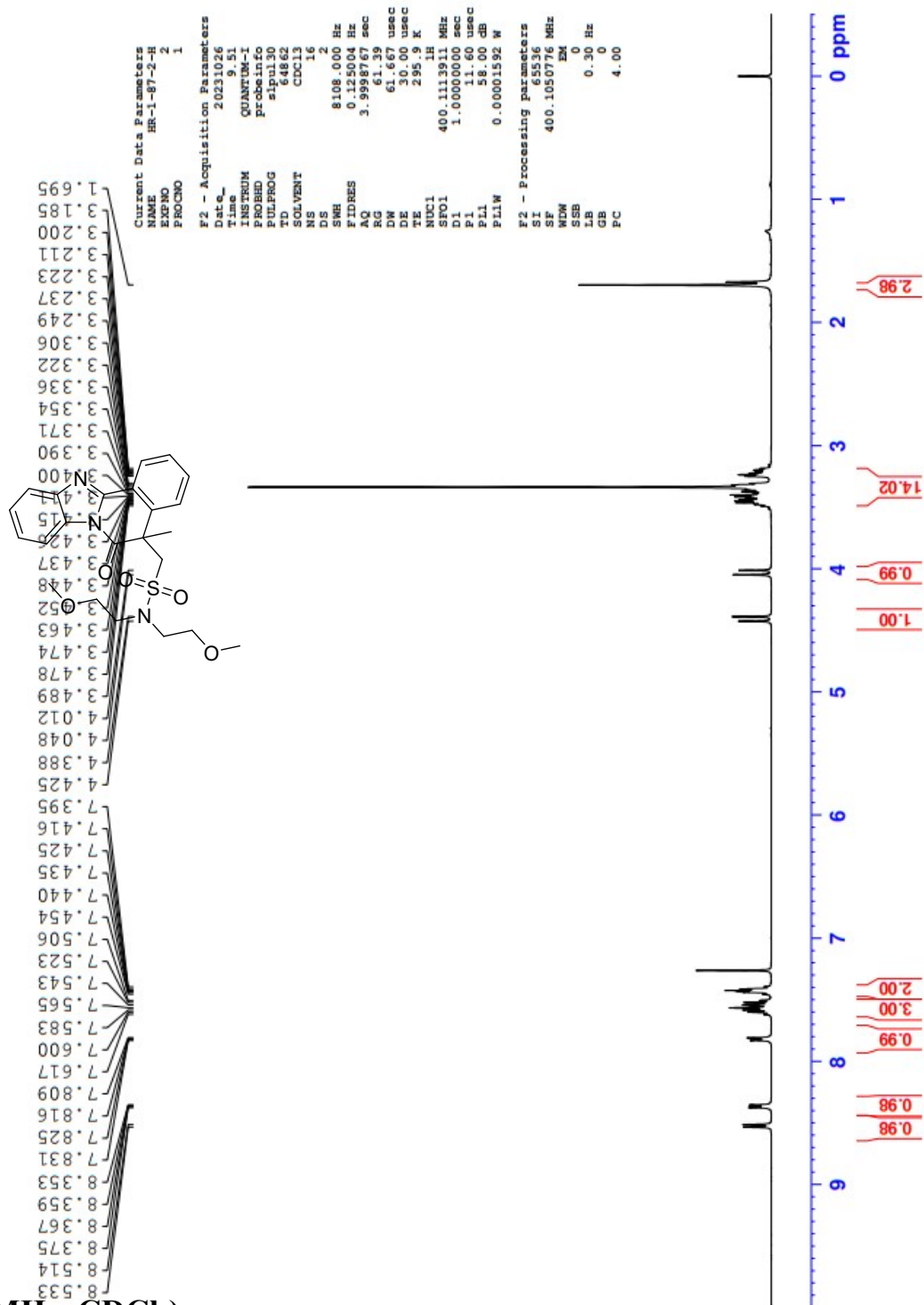
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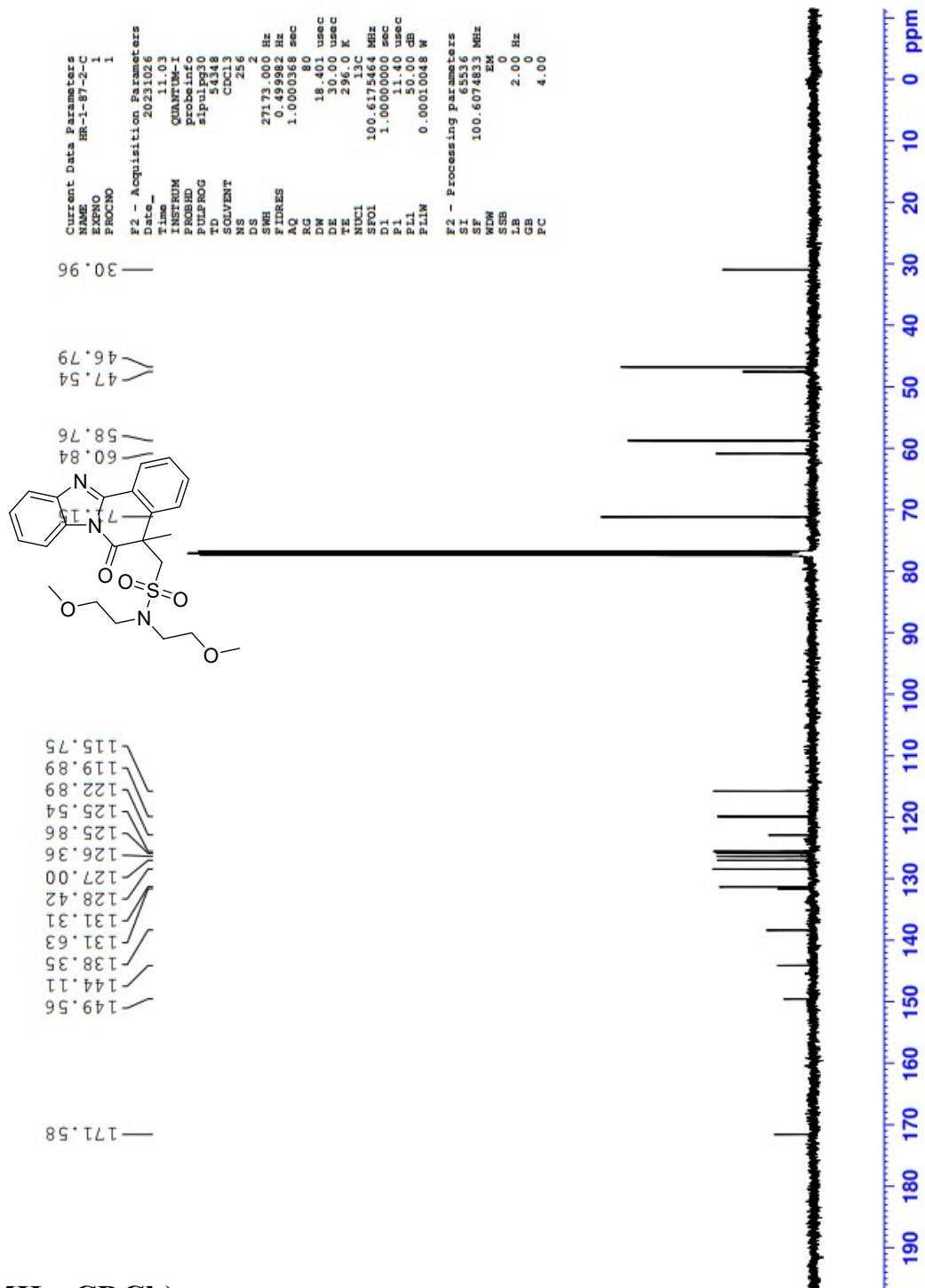
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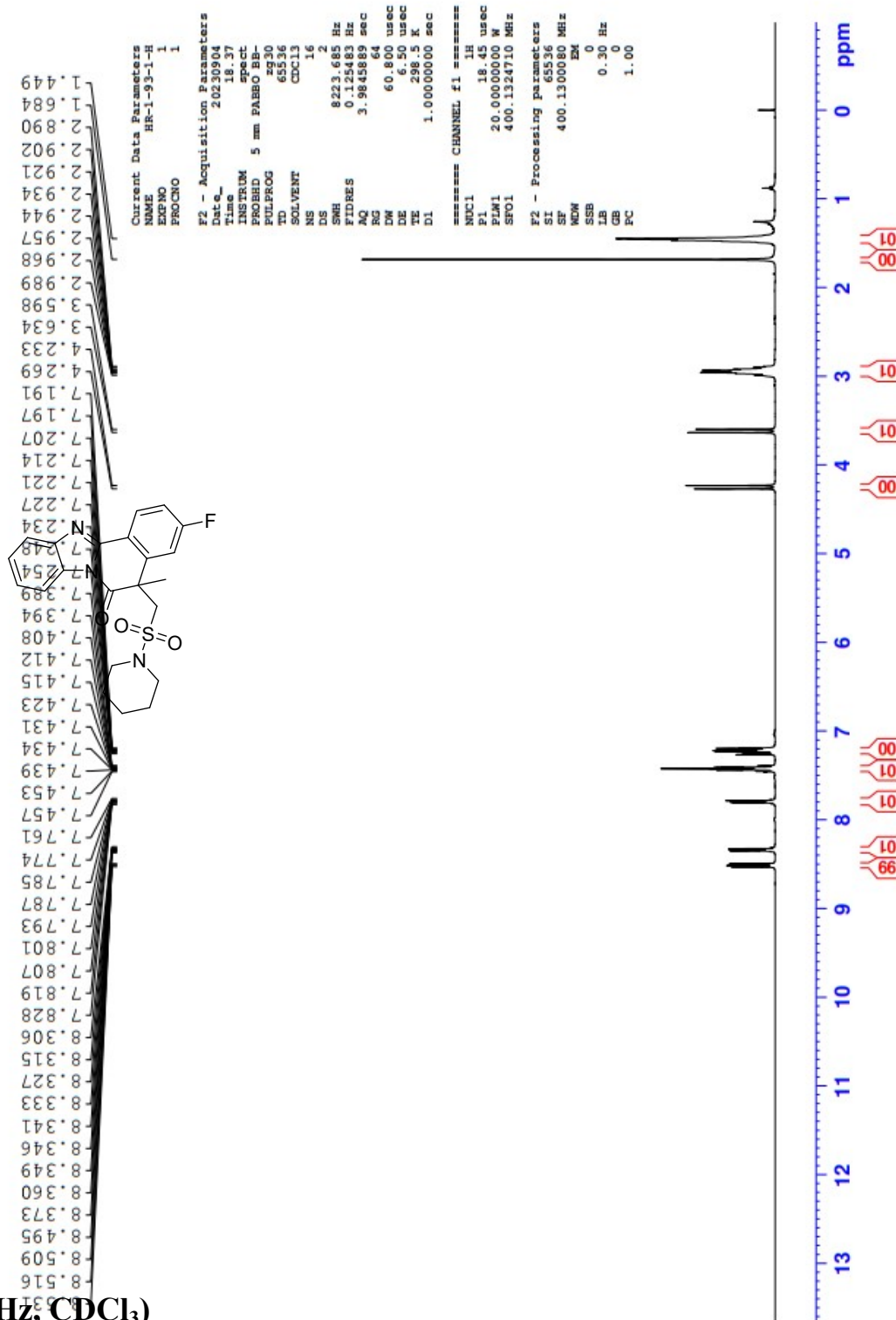
3h <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



3i <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

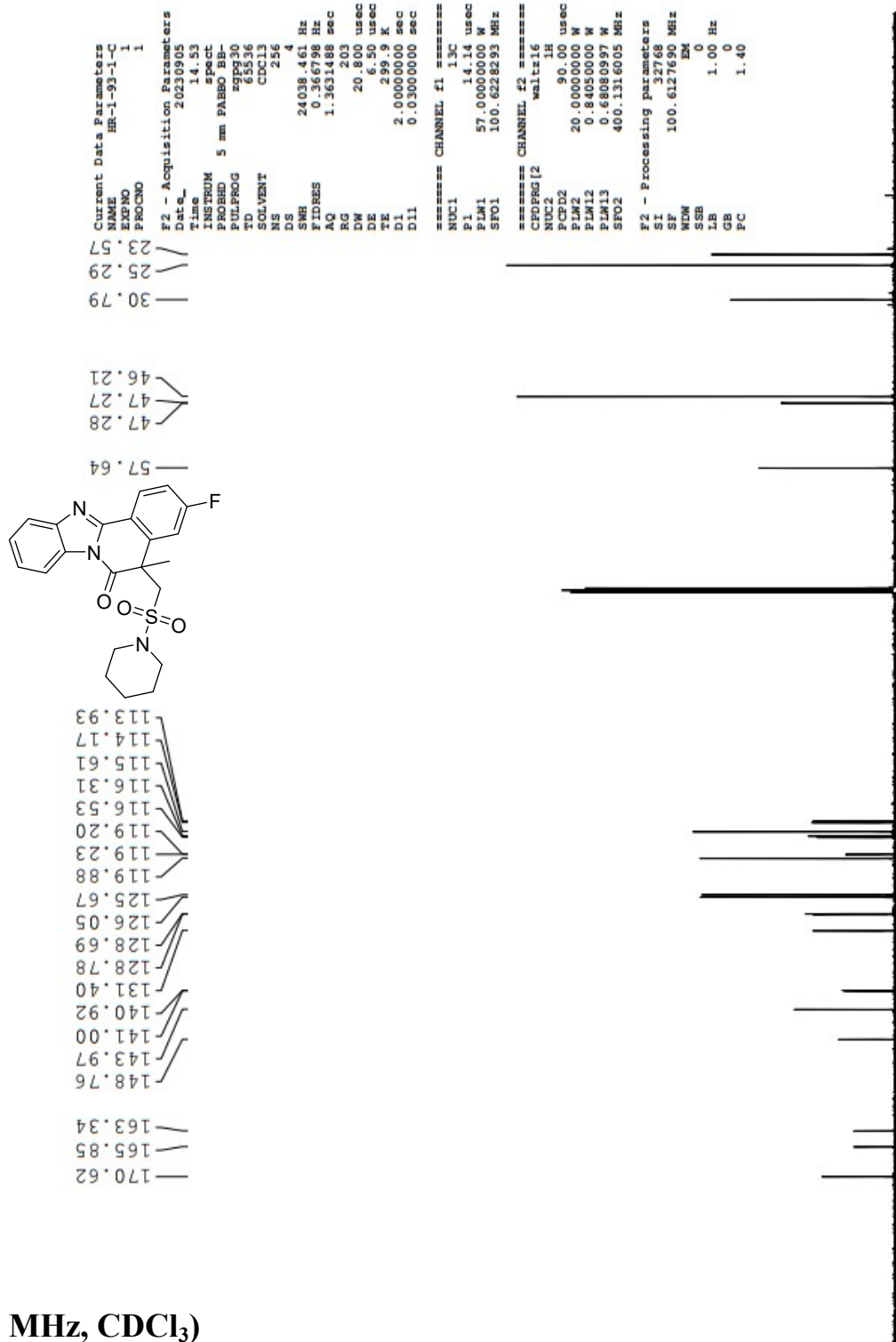


3i <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



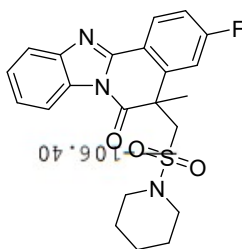


3i <sup>19</sup>F NMR (376 MHz, CDCl<sub>3</sub>)





# 3j <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



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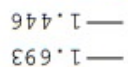
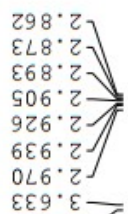
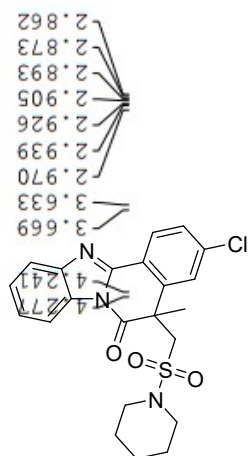
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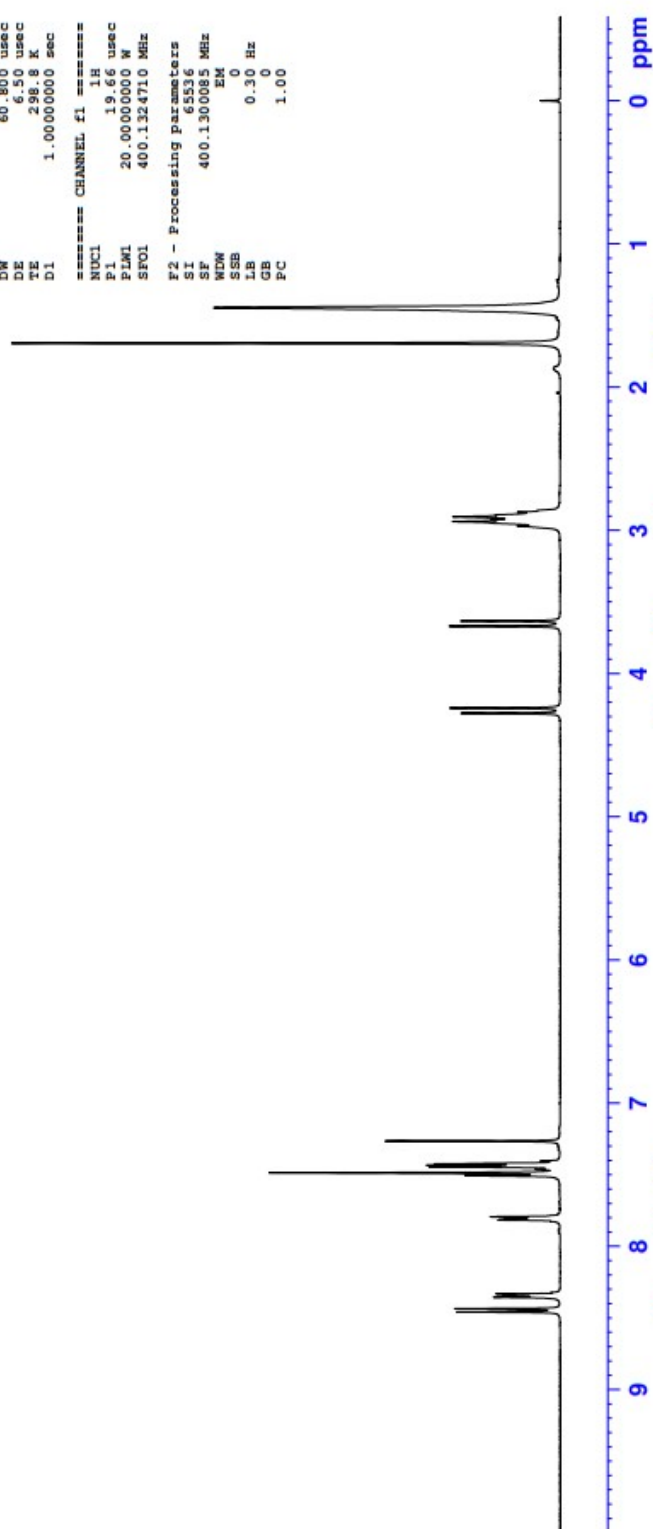
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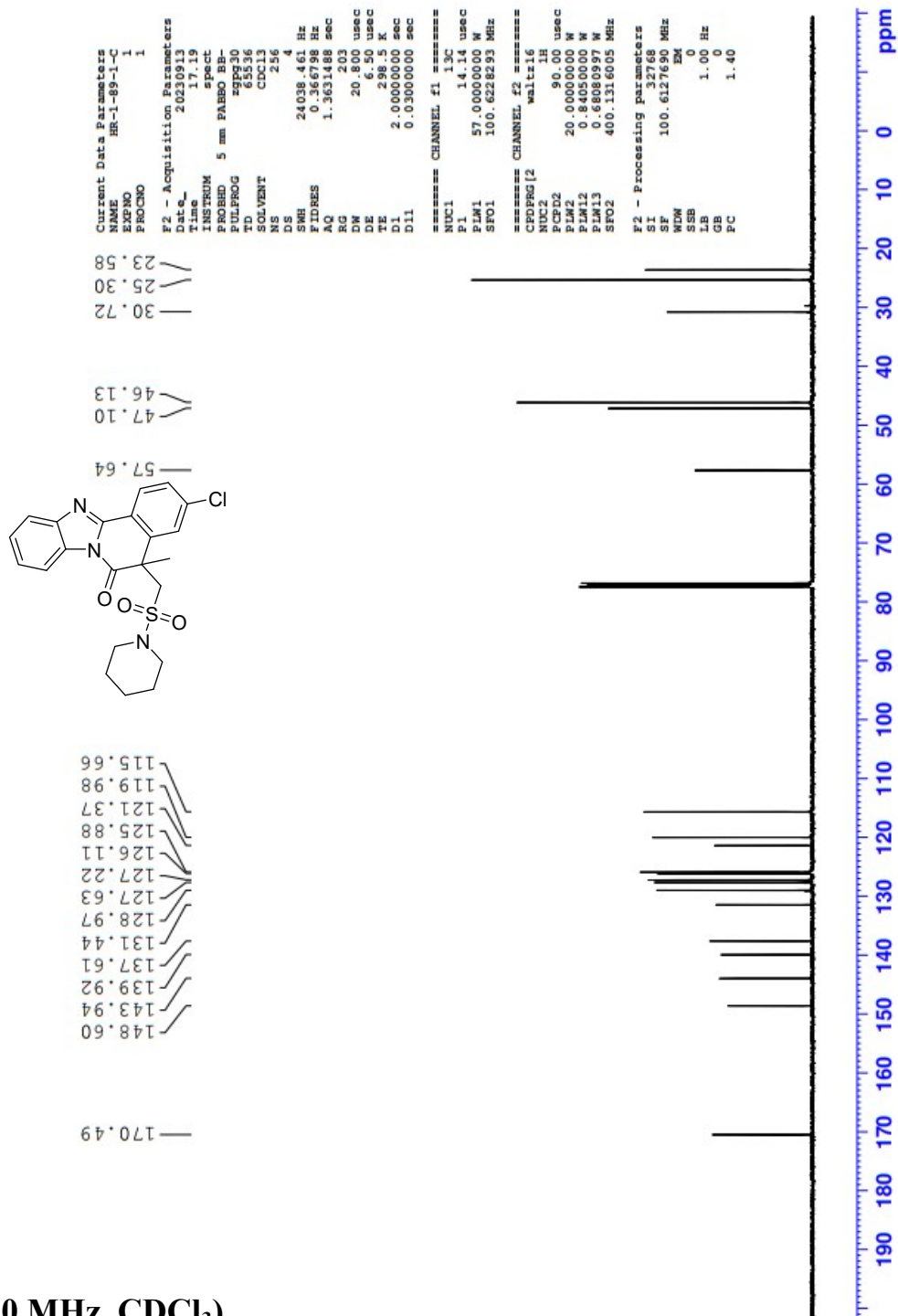
3j <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



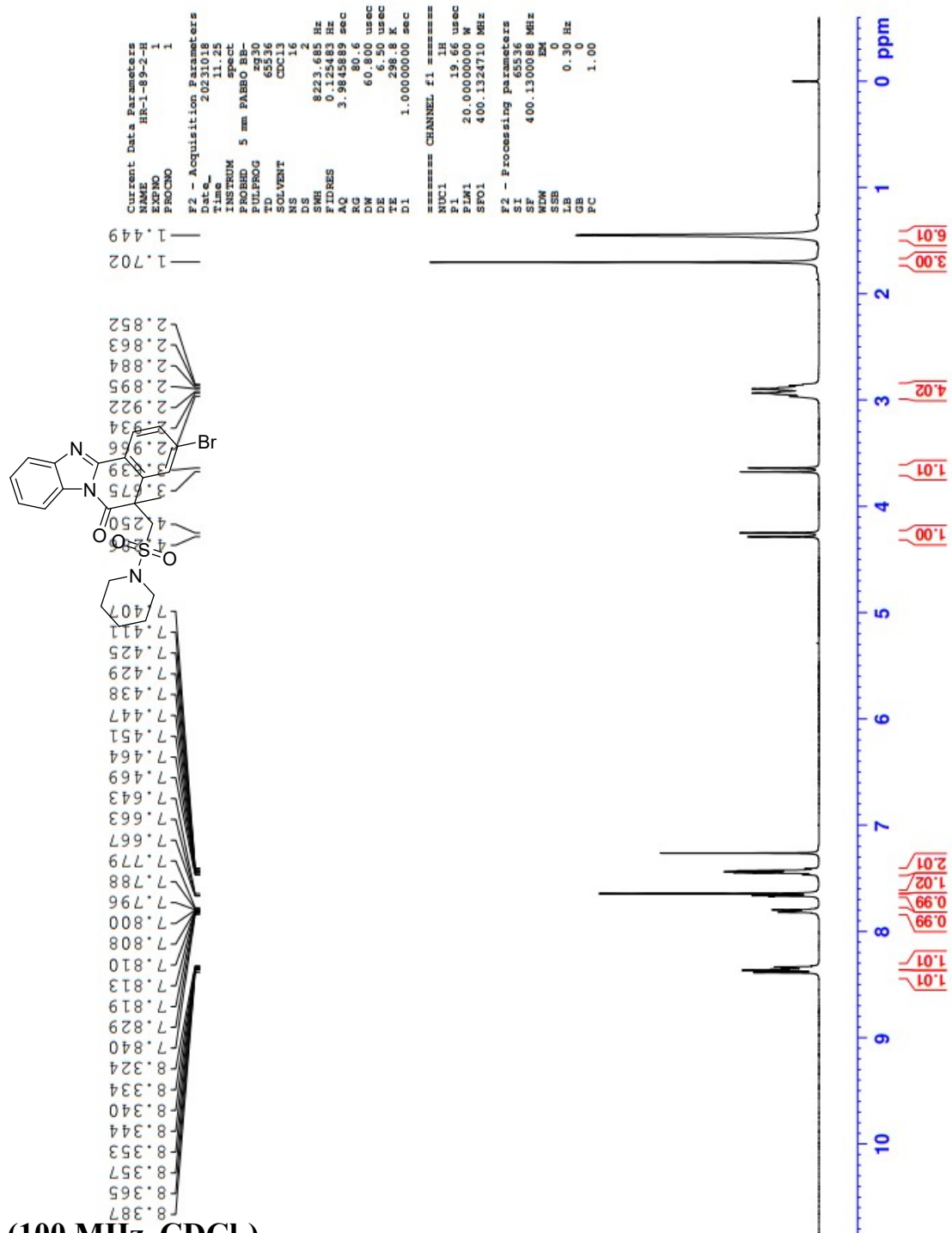
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RG 84  
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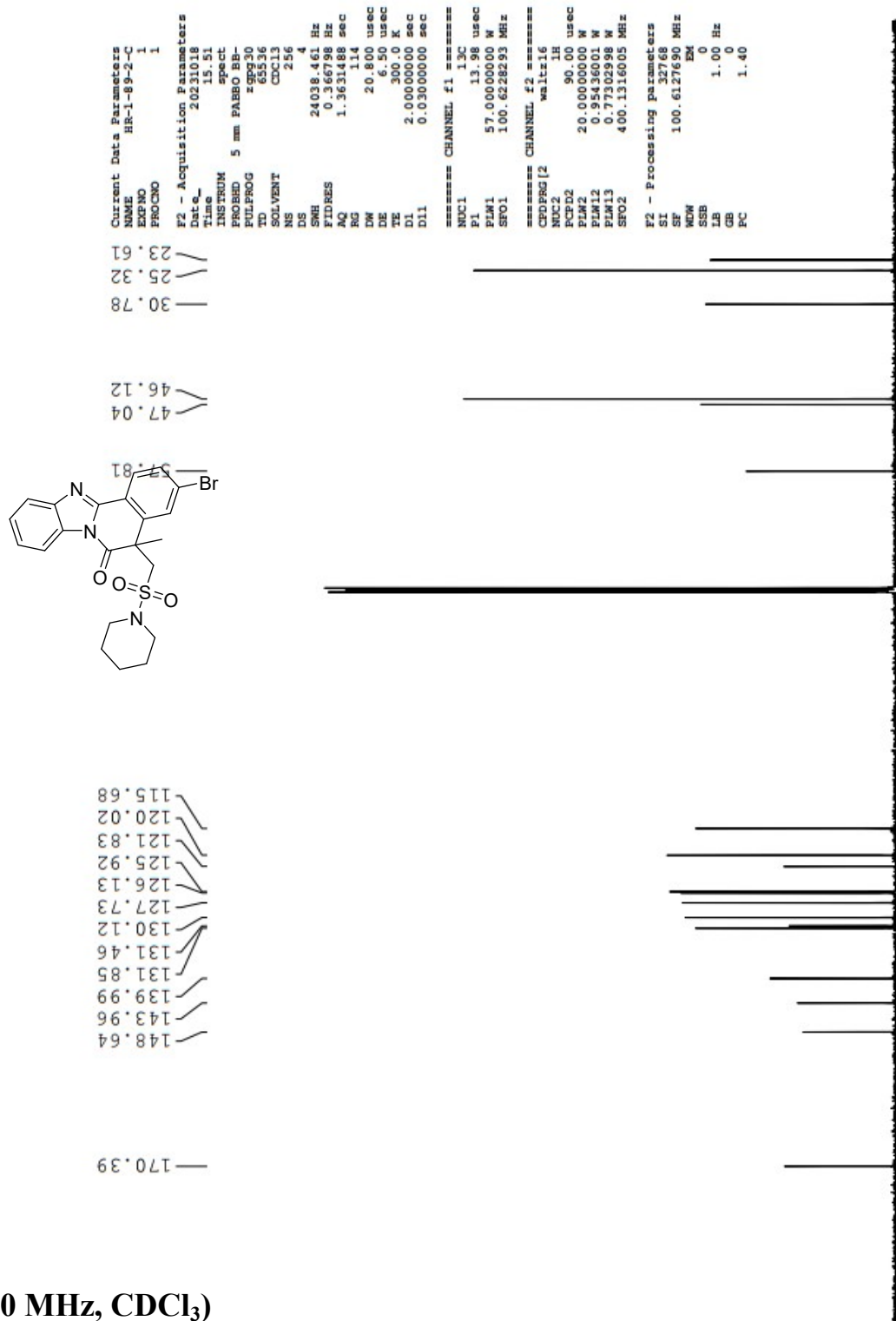
# 3k <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



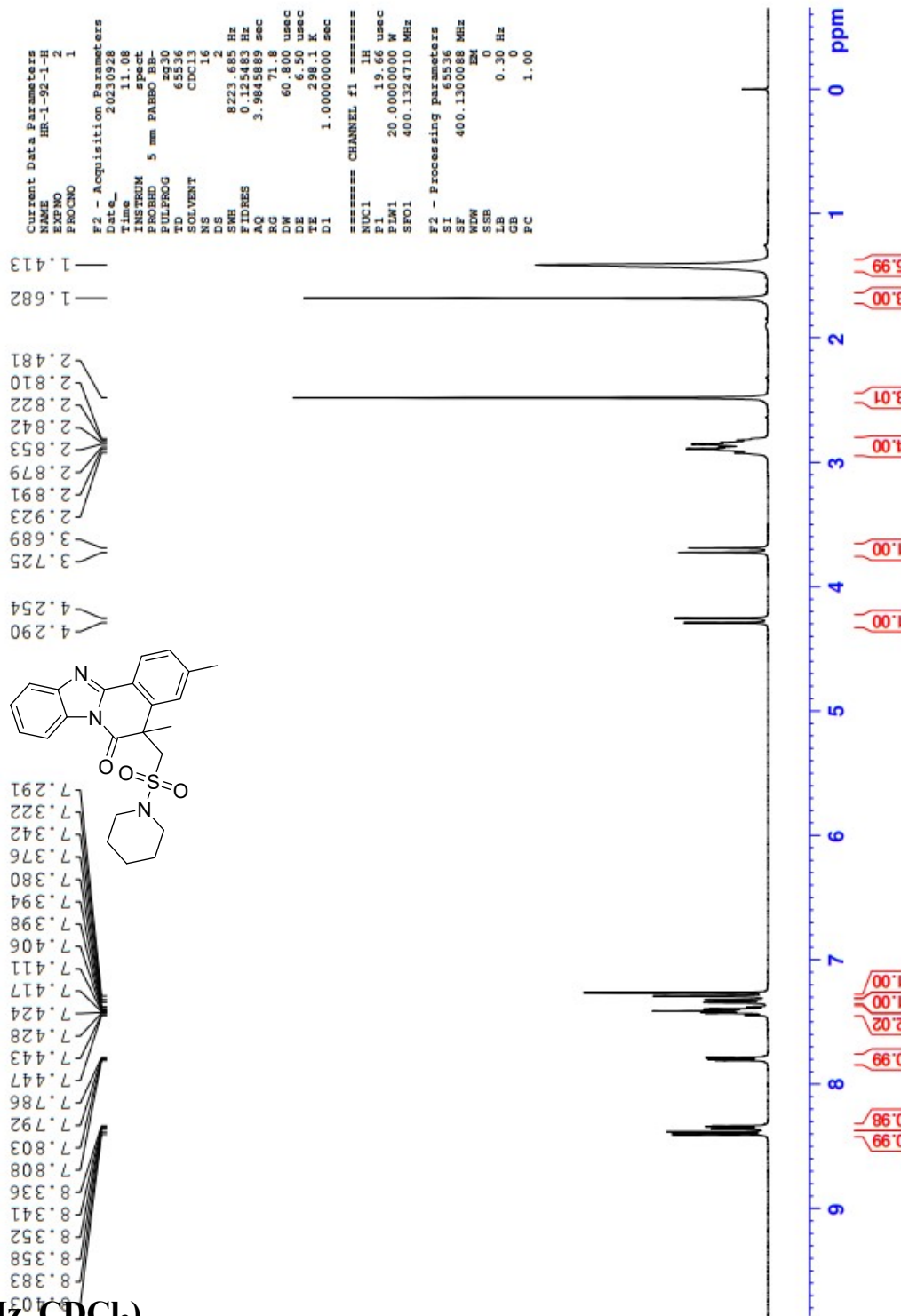
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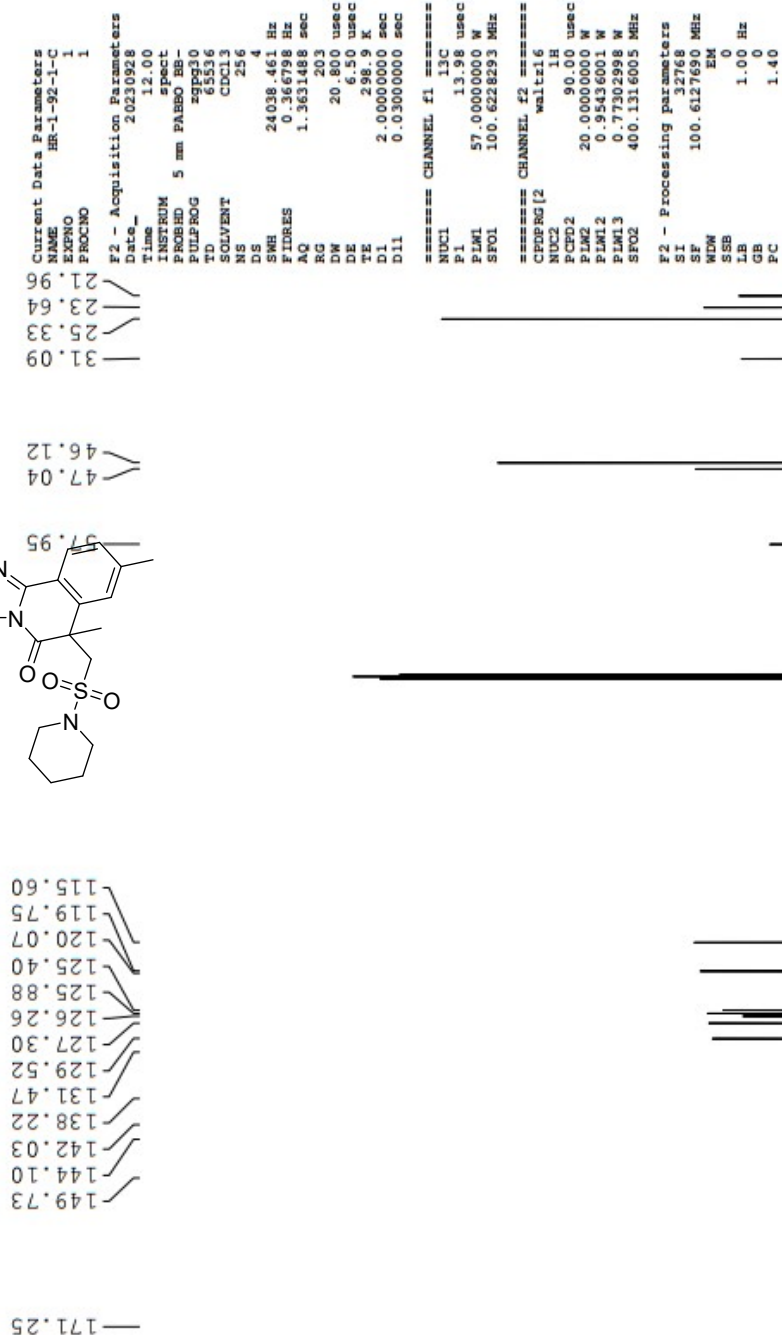
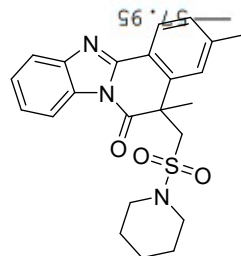
# 31 <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



**$^{31}\text{P}$   $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )**



# 3m <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





100 MHz (CDCl<sub>3</sub>)

CC1=CC=C(C=C1)N2C(=O)C3CC4C(C(=O)N3CC4)C2

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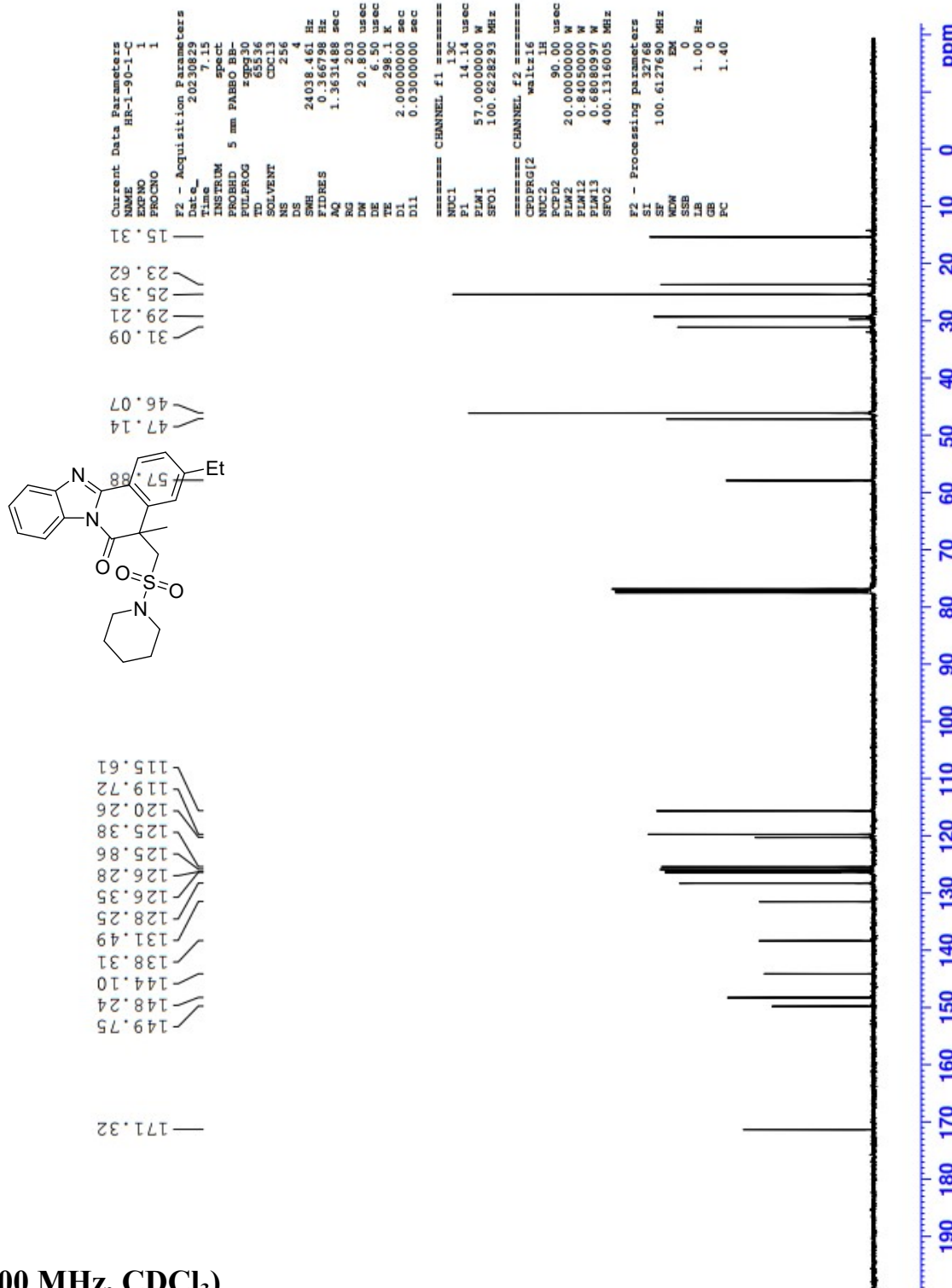
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2.791  
2.805  
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7.304  
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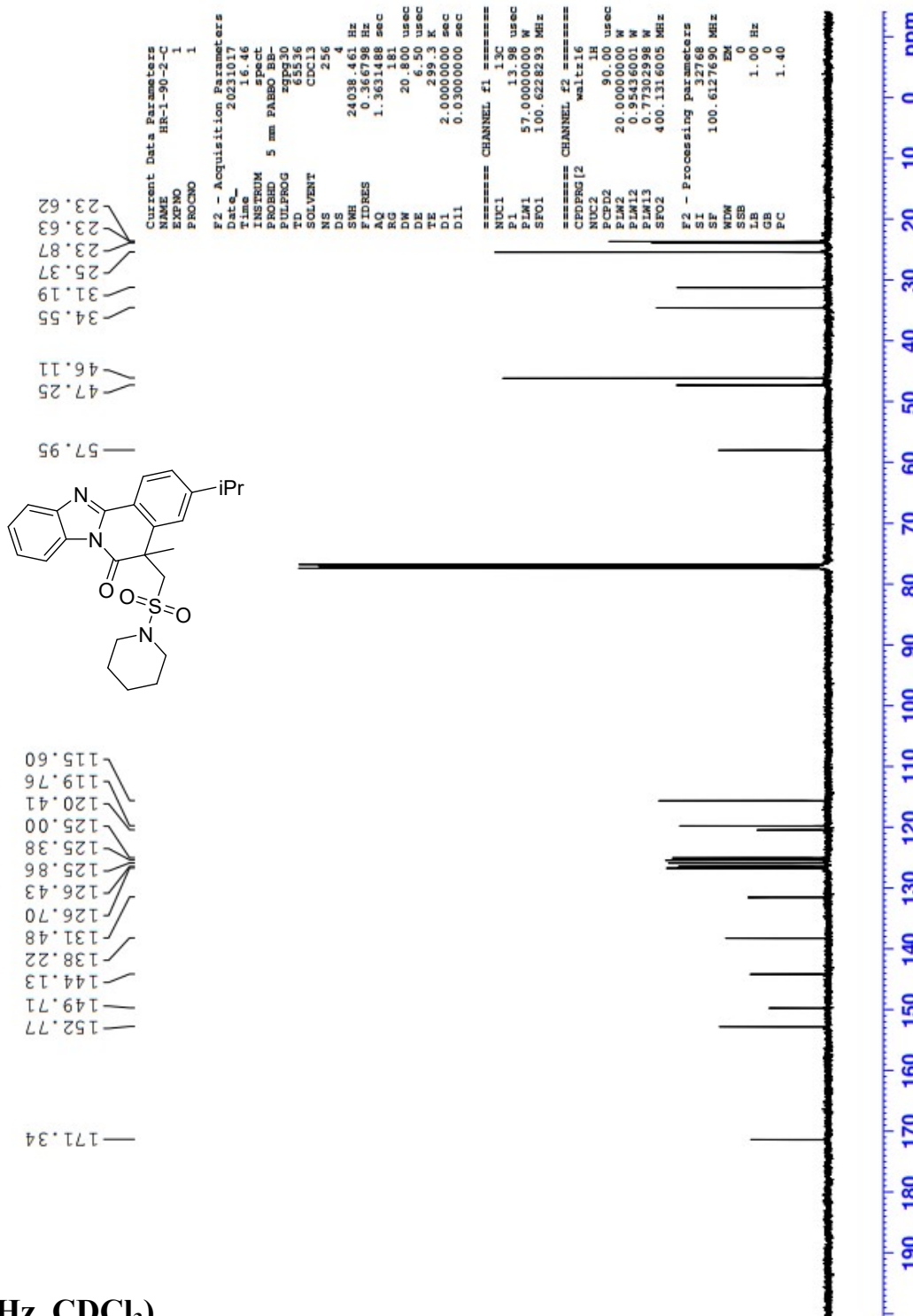


# 3n <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

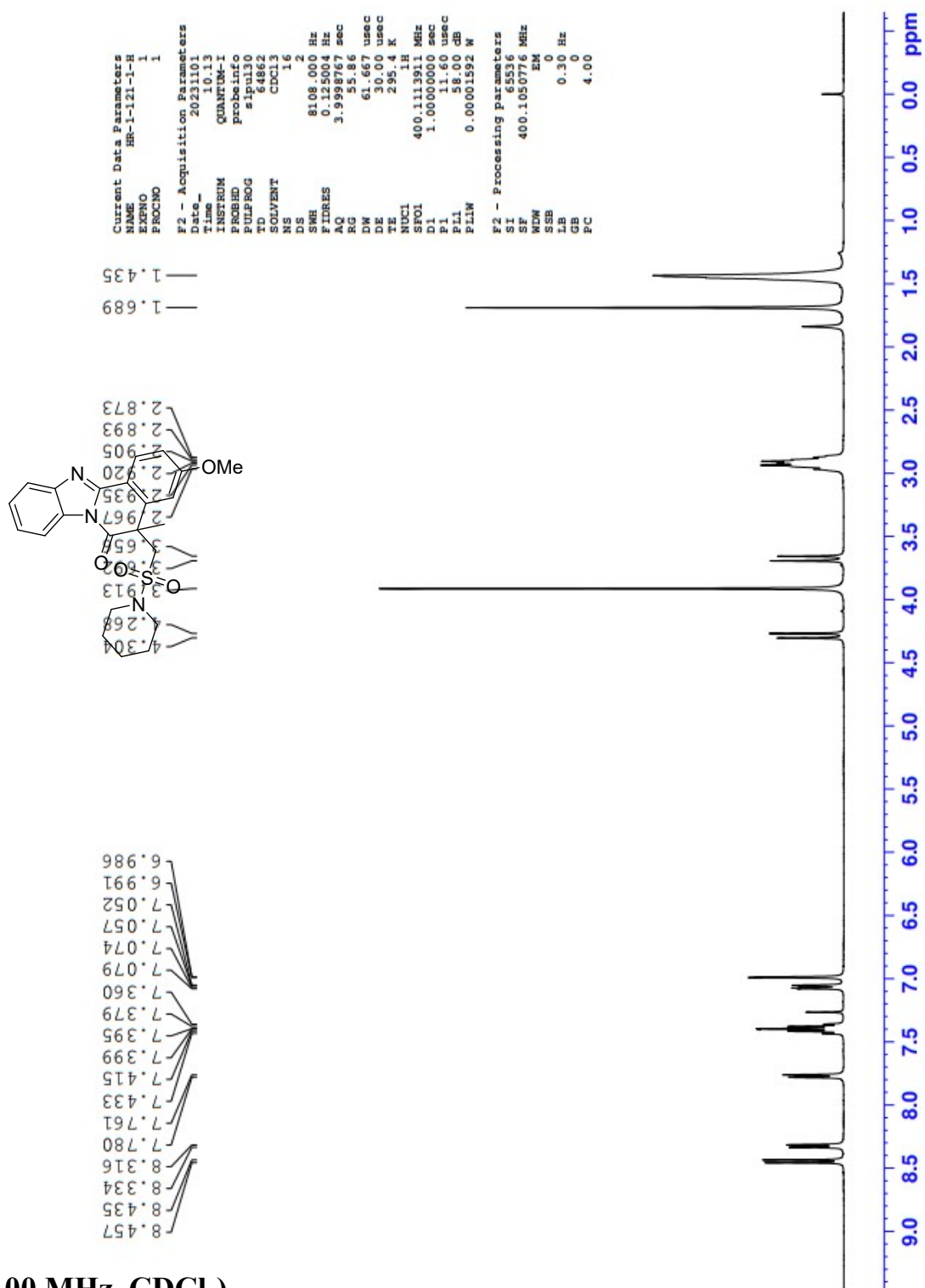




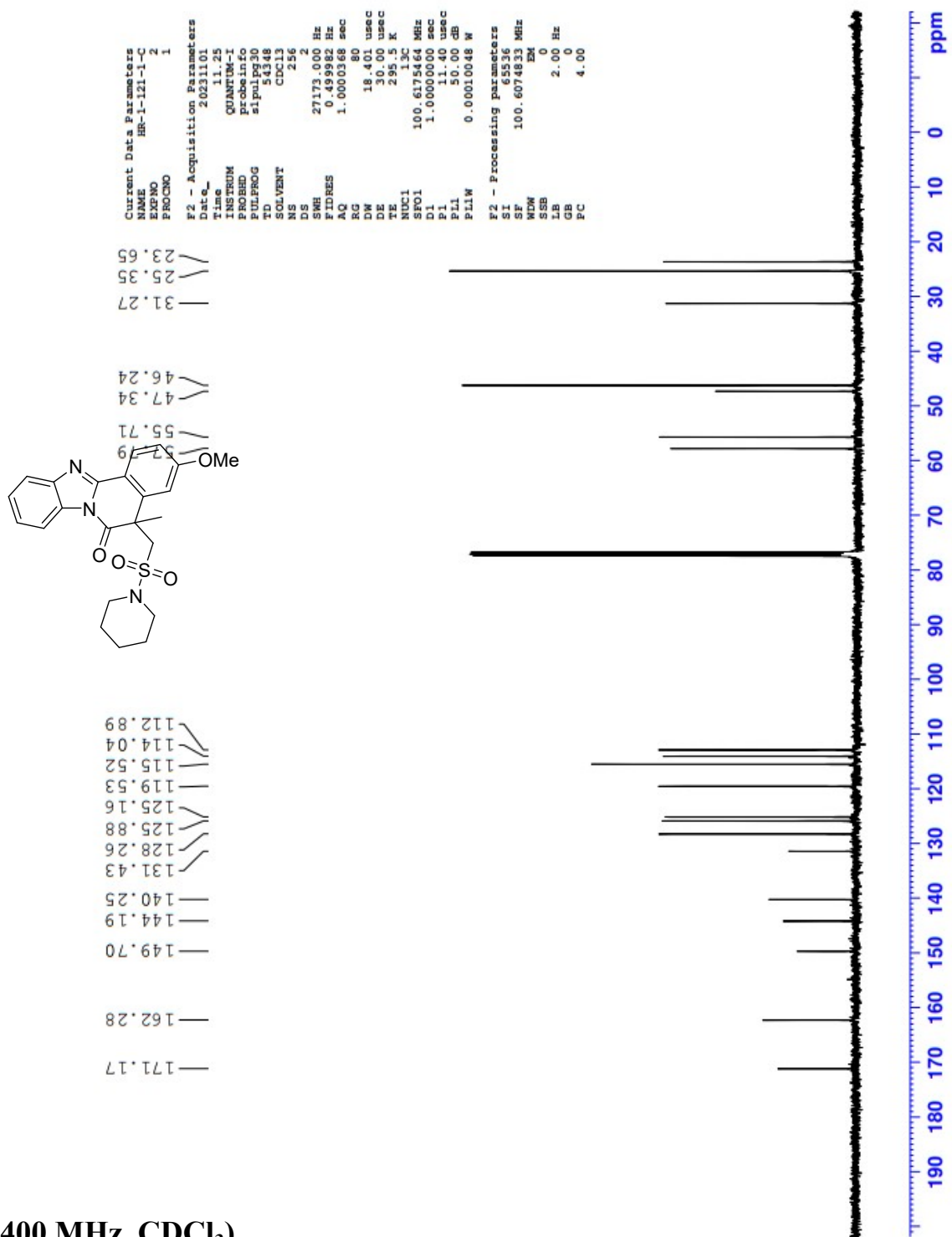
3o <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

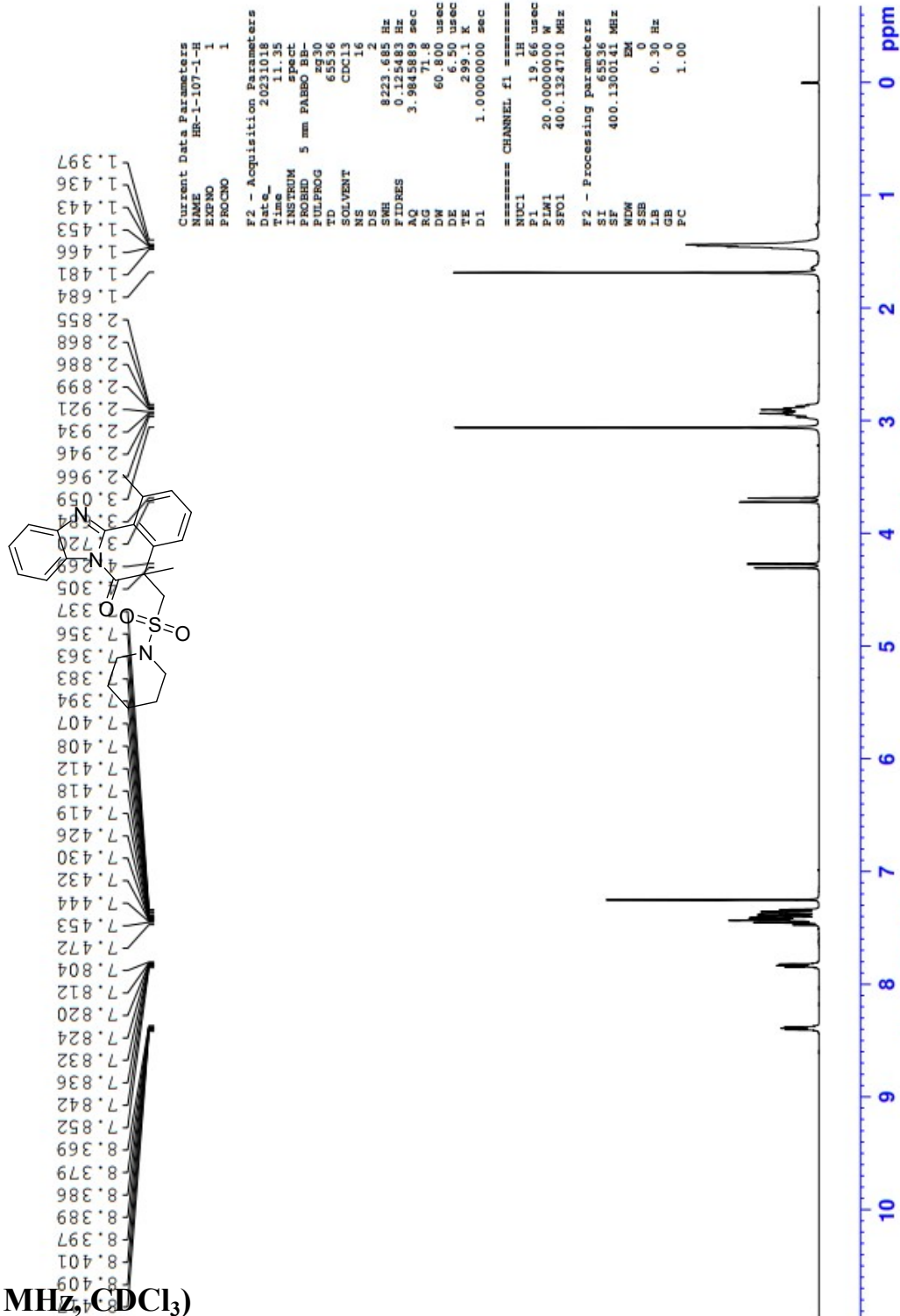


**3o**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



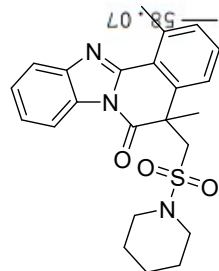
# 3p <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)







3q <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

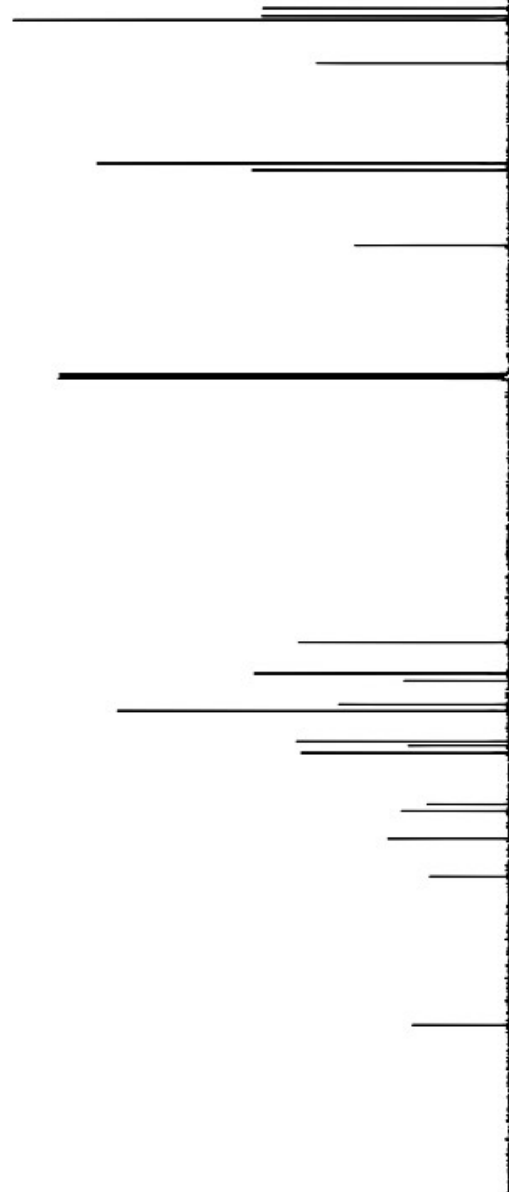


171.29  
149.73  
144.20  
140.20  
139.22  
131.77  
130.68  
130.12  
125.67  
124.74  
121.30  
120.23  
115.72

58.07  
47.15  
46.17

31.63  
25.36  
24.79  
23.65

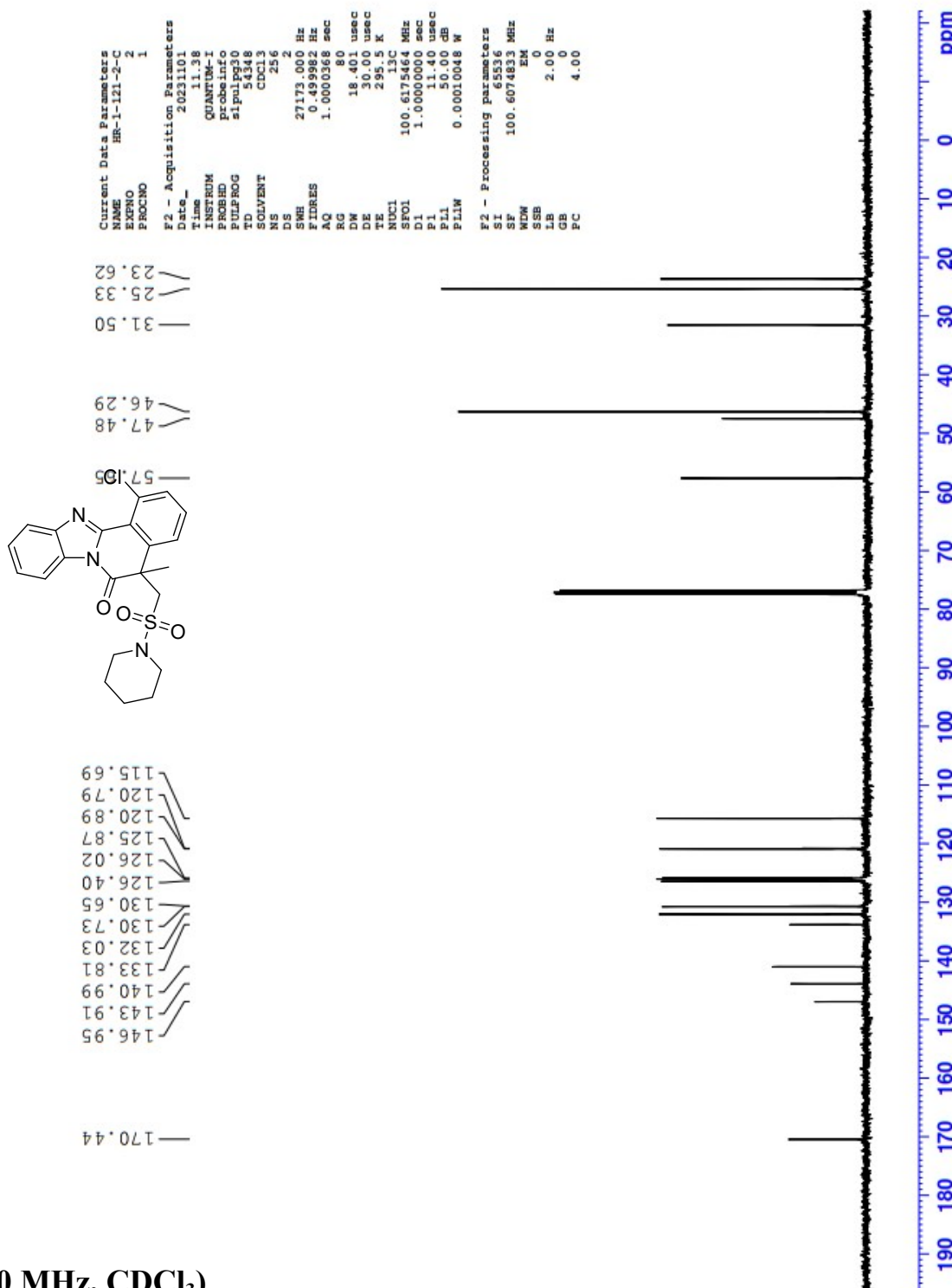
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PROCNO 1  
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Time 16.11  
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PROBHD 5 mm PABBO BBO  
PULPROG zgpg30  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 24038.461 Hz  
FIDRES 0.366798 Hz  
AQ 1.3631488 sec  
RG 181  
DW 20.800 usec  
DE 6.50 usec  
TE 299.9 K  
D1 2.00000000 sec  
D11 0.03000000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
P1 13.98 usec  
PLW1 57.00000000 W  
SFO1 100.6228293 MHz  
===== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 90.00 usec  
PLW2 20.00000000 W  
PLW12 0.95436001 W  
PLW13 0.77302998 W  
SFO2 400.1316005 MHz  
F2 - Processing parameters  
SI 32768  
SF 100.6127690 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



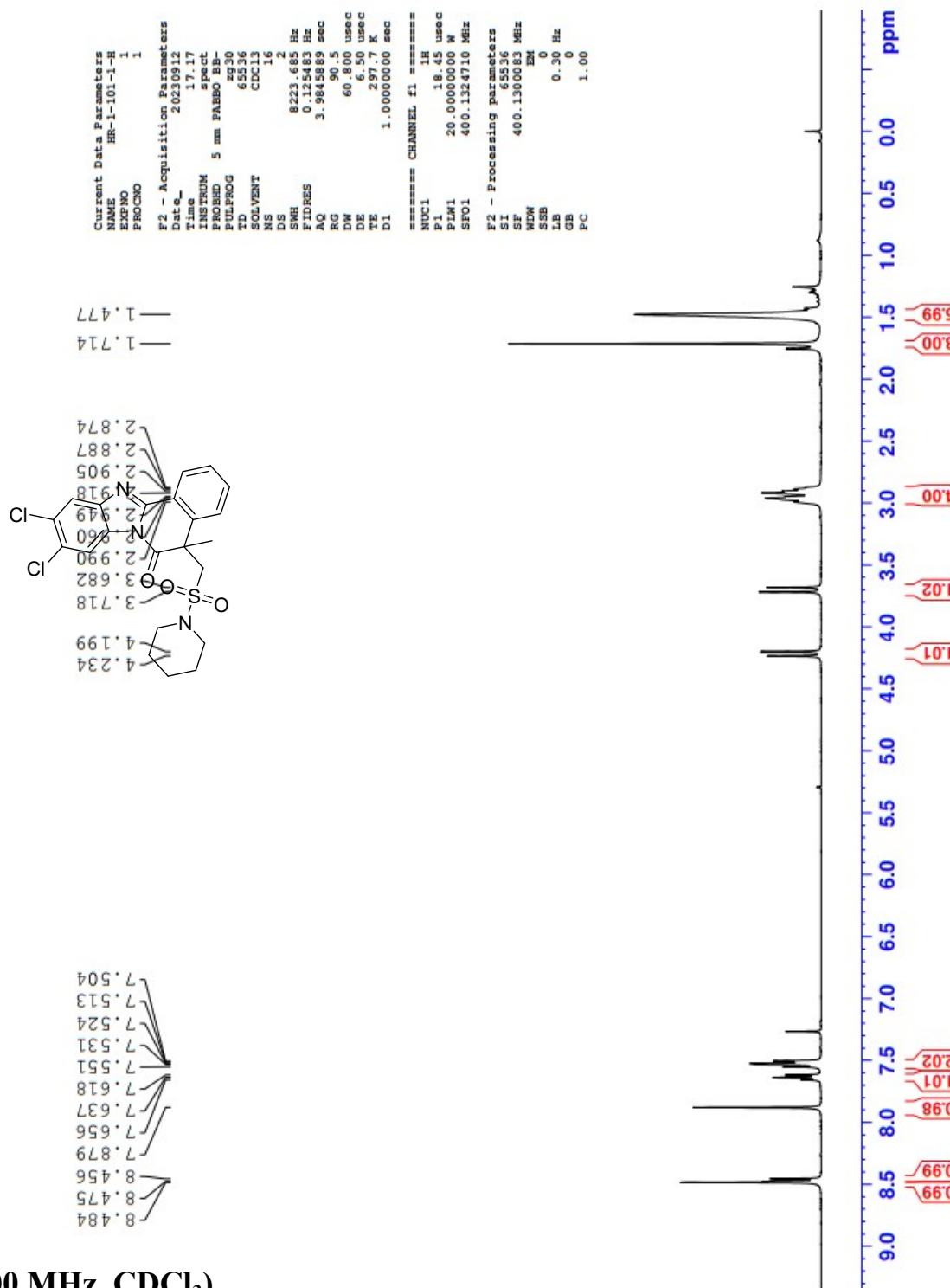




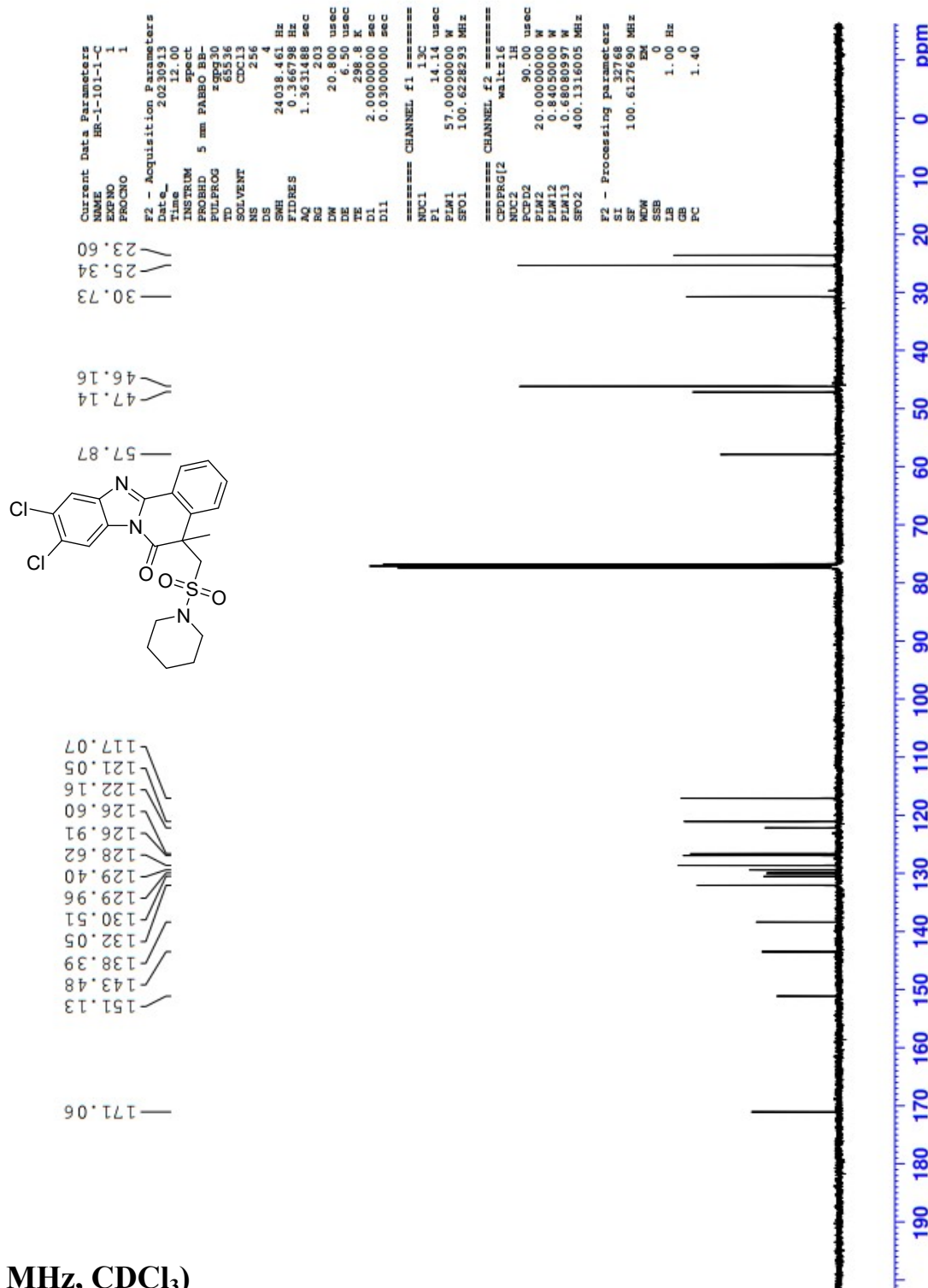
**3r <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)**



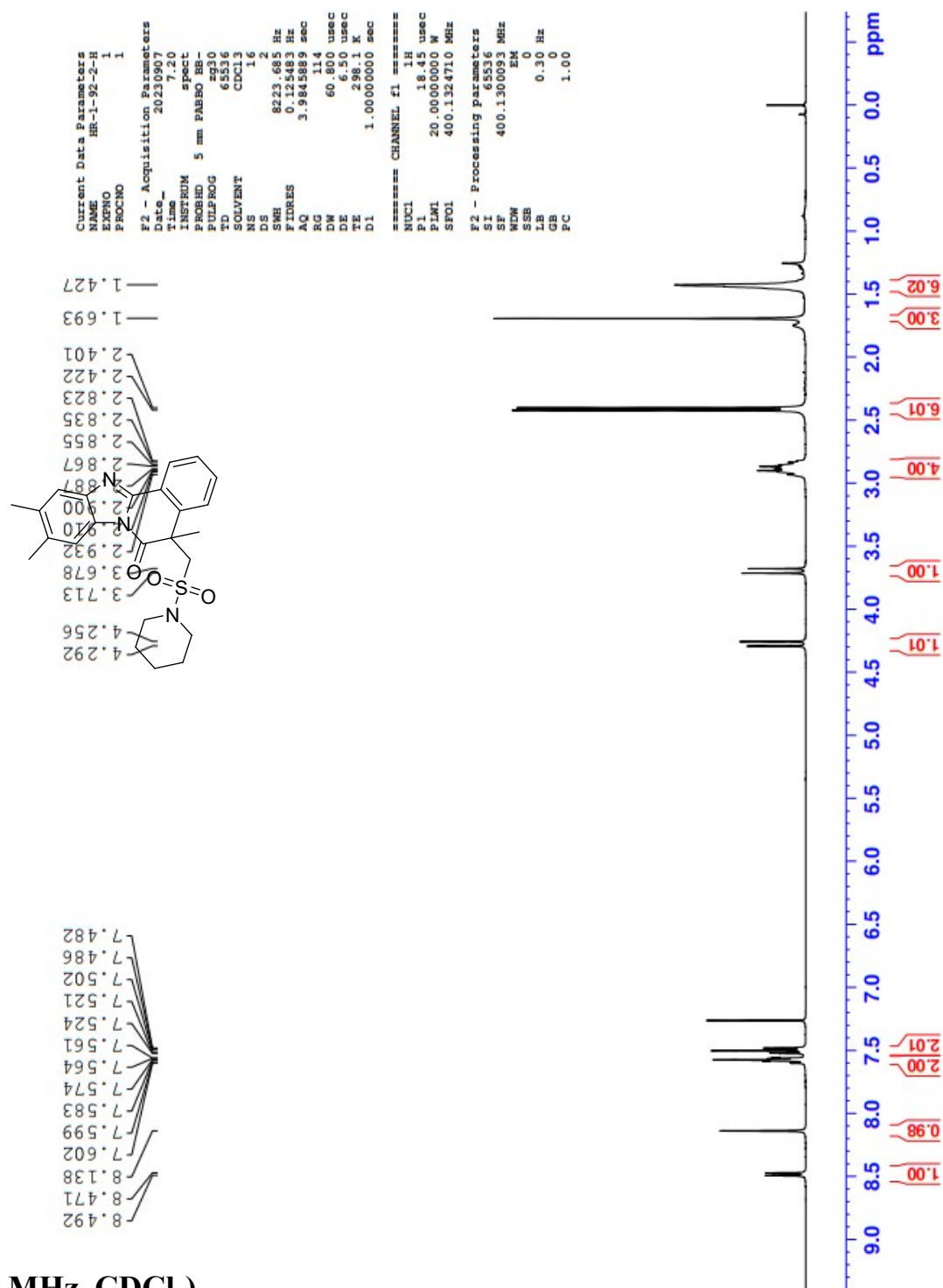
**3r <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**



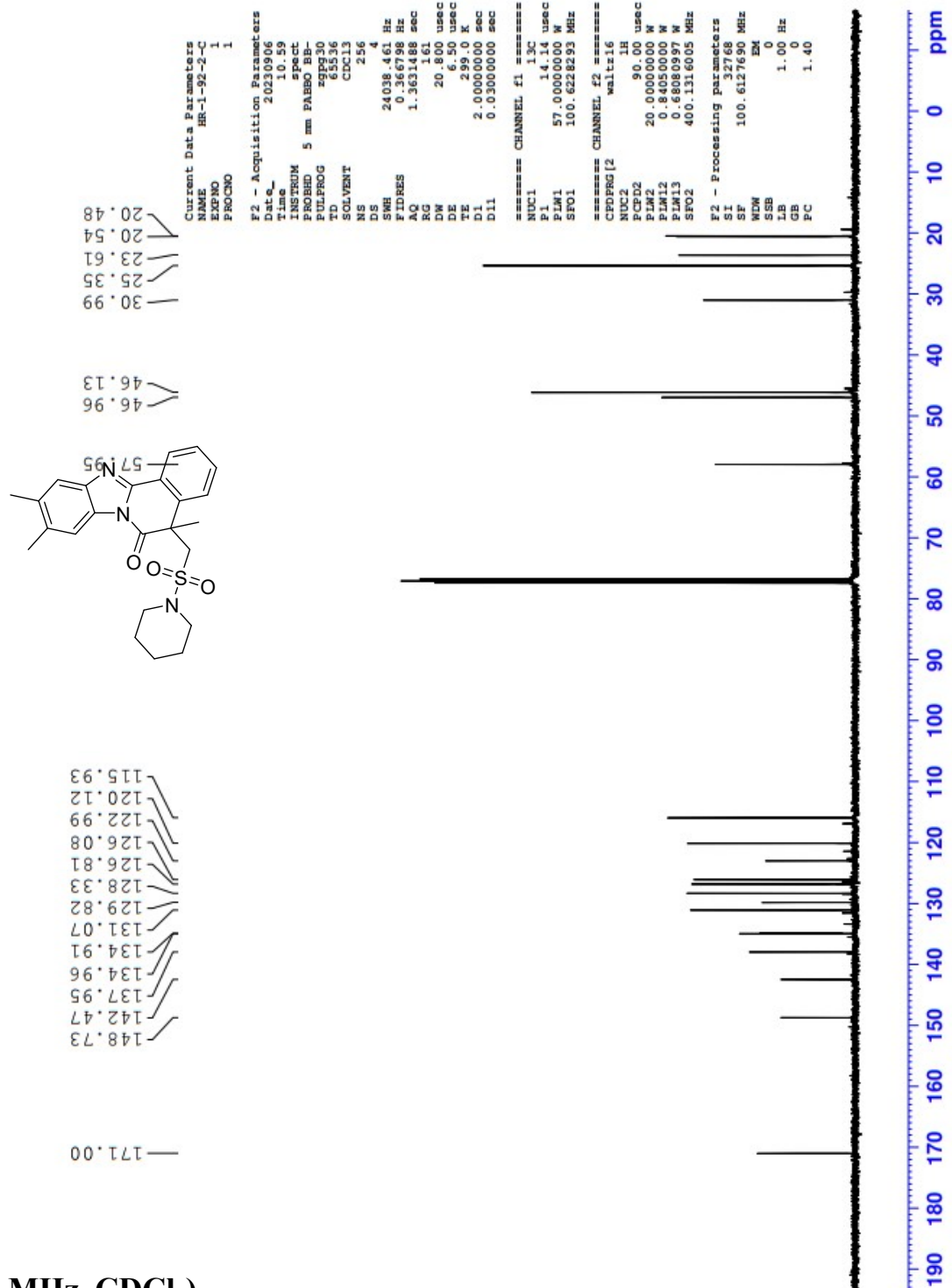
3s <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



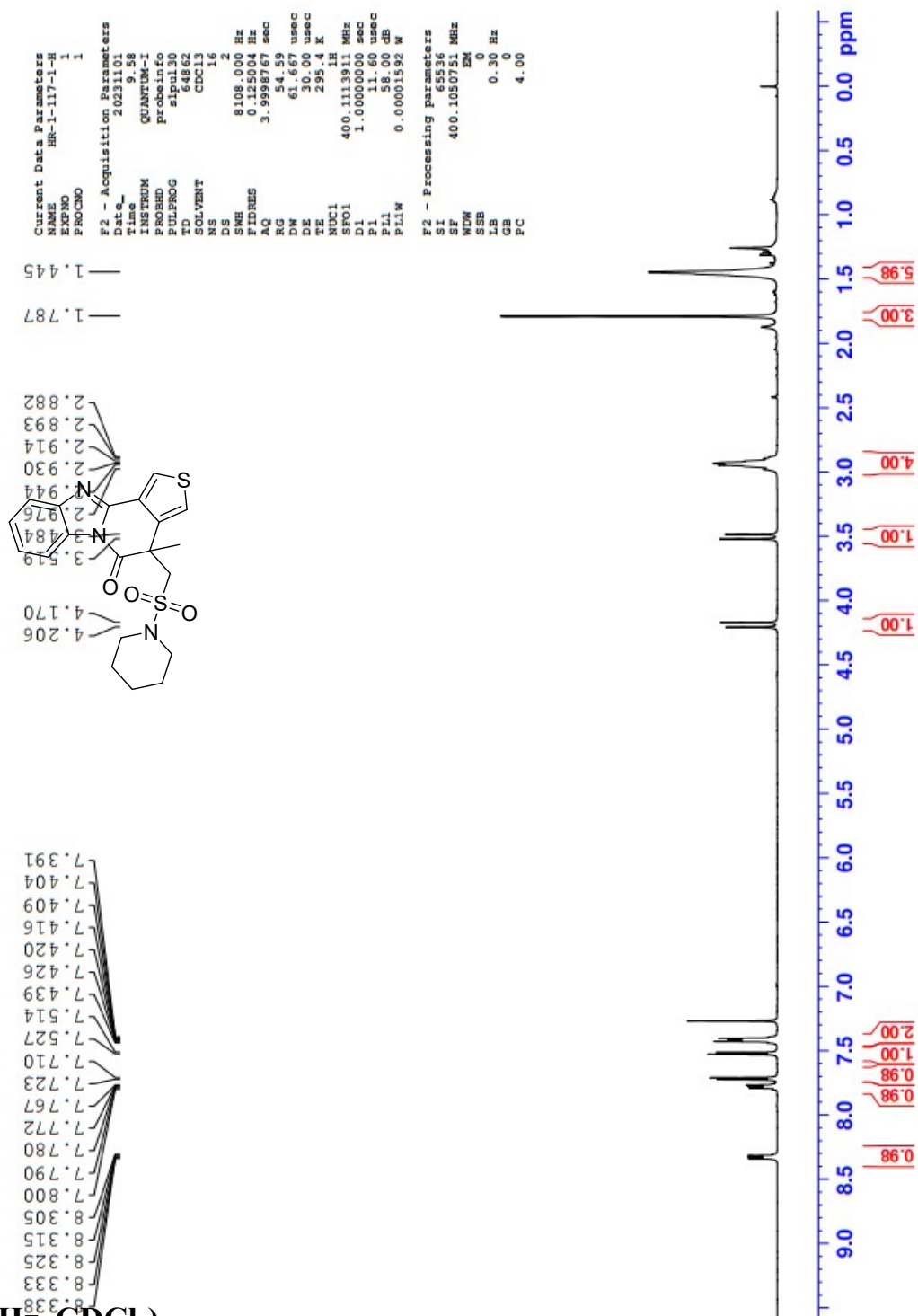
3s <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



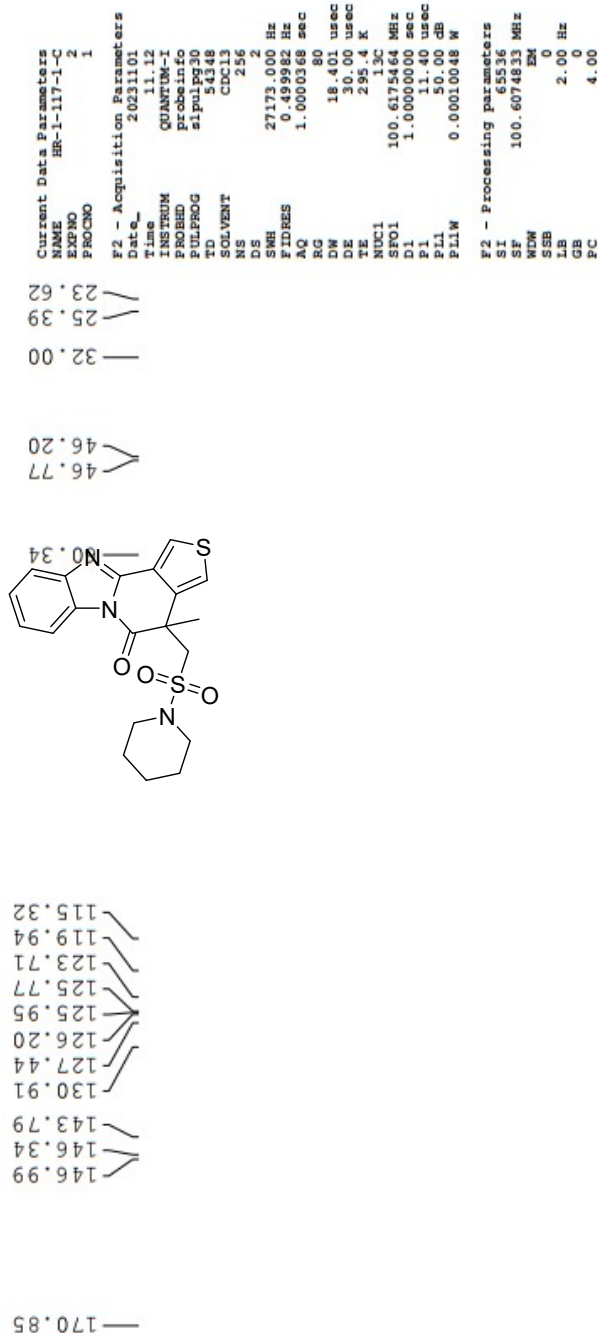
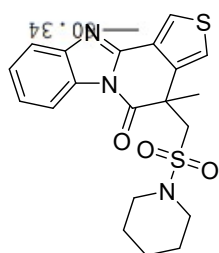
3t <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



3t <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

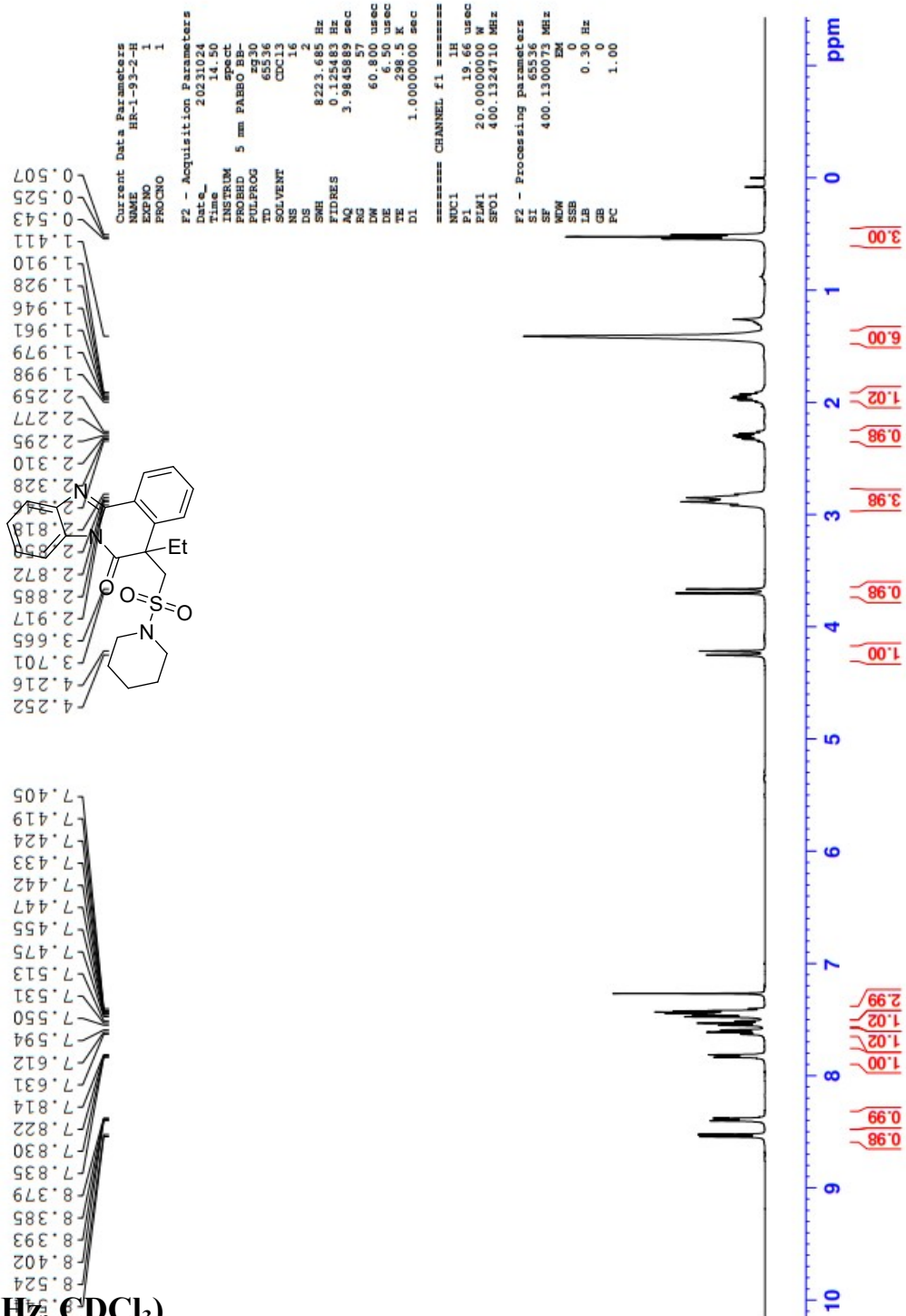


3u <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



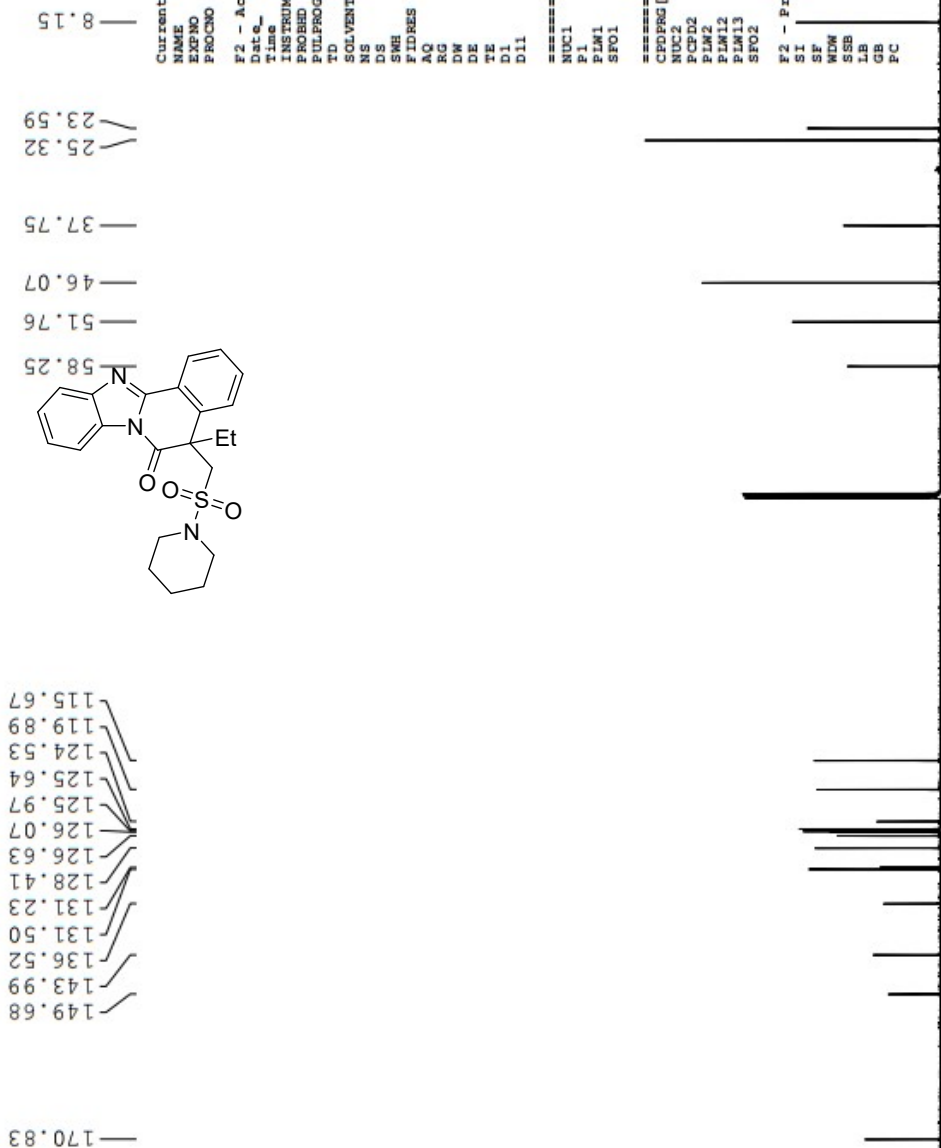


**3u <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**





# 3v <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



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 PROCNO 1  
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 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 256  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.366798 Hz  
 AQ 1.3631488 sec  
 RG 128  
 DW 20.800 usec  
 DE 6.50 usec  
 TE 298.8 K  
 D1 2.00000000 sec  
 D11 0.03000000 sec  
 ===== CHANNEL f1 =====  
 NUC1 13C  
 P1 13.98 usec  
 PLW1 57.0000000 W  
 SFO1 100.6228293 MHz  
 ===== CHANNEL f2 =====  
 CPDPRG2 waltz16  
 NUC2 1H  
 P2 90.00 usec  
 PLW2 20.0000000 W  
 PLM2 0.95436001 W  
 PLM13 0.77302998 W  
 SFO2 400.1316005 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 100.6127690 MHz  
 WDW EM  
 SSB 0  
 LB 1.00 Hz  
 GB 0  
 PC 1.40

Current Data Parameters  
 HR-1-97-2-1  
 EXNO 1  
 PROCNO 1

F2 - Acquisition Parameters  
 Date\_ 20231109  
 Time 10.39  
 INSTRUM QUANTUM-I  
 PROBED probeinfo  
 PULPROG zgpg30  
 TD 64862  
 SOLVENT CDCl3  
 NS 16  
 DS 8108.000 Hz  
 SH 0.125004 Hz  
 FIDRES 3.9938767 sec  
 AQ 50.88  
 RG 61.667 usec  
 DW 30.00 usec  
 DE 295.3 K  
 TE 1H  
 NUC1 400.1113911 MHz  
 SFO1 1.00000000 sec  
 D1 11.60 usec  
 F1 56.00 dB  
 Fll 0.0001592 W  
 Fllw

F2 - Processing parameters  
 SI 400.1050822 MHz  
 SF 400.1050822 MHz  
 EM 0  
 SSB 0.30 Hz  
 LB 0.0  
 GB 0.0  
 PC 4.00

1.453

2.894  
2.925  
2.969  
3.000  
3.101  
3.133  
3.169  
3.433  
3.872  
3.908  
4.457  
4.491

6.425  
6.445  
6.736  
6.754  
6.772  
6.850  
6.867  
6.886  
7.338  
7.358  
7.367  
7.371  
7.378  
7.396  
7.400  
7.473  
7.490  
7.509  
7.509  
7.599  
7.620  
7.643  
7.663  
8.273  
8.293  
8.327

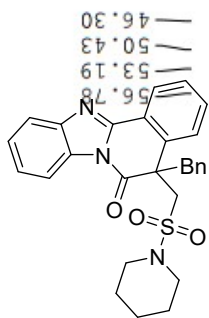
1.98  
1.99  
0.99  
2.02  
1.02  
3.02  
0.98  
0.99

4.00  
1.00  
1.00  
1.00  
1.00  
1.00

6.02

0.0 ppm

1H, CDCl<sub>3</sub>)



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

170.23

25.40  
23.65

Current Data Parameters  
NAME HR-1-97-2-C  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20231109  
Time 13.38  
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PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 256  
DS 4  
SWH 27173.000 Hz  
FIDRES 0.49982 Hz  
AQ 1.0000368 sec  
RG 80  
RG 18.401 usec  
DE 30.00 usec  
TE 295.5 K  
NDC1 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

