

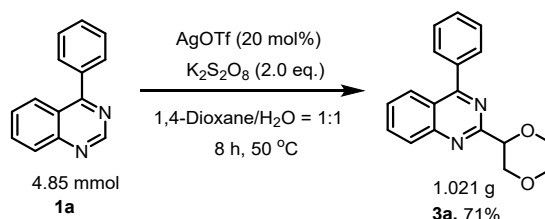
**Supporting Information**  
**Ag-Catalyzed Cross-Dehydrogenative-Coupling for the Synthesis of**  
**1,4-Dioxan-2-yl Substituted Quinazoline Hybrids in Aqueous Media**

Xixian Liu, Dayu Xie, Qin Yang, Zhibin Song · Yang Fu\* and Yiyuan Peng\*

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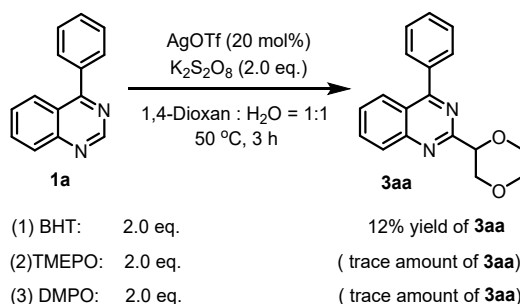
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## I. Gram-scale synthesis of 3aa



A mixture of **1a** (1.006 g, 4.85 mmol), AgOTf (0.249 g, 20 mol%), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.108 g, 0.4 mmol) in an oven-dried 100 mL Schlenk flask was added the 1,4-Dioxan/H<sub>2</sub>O (24 mL/24 mL). The mixture was stirred at 50 °C until the reaction was complete. The reaction was then quenched with saturated NaCl solution (50 mL), while the aqueous phase was extracted three more times with EtOAc (50\*3 mL). The organic phase was combined, dried over Na<sub>2</sub>SO<sub>4</sub>, the filtrate was concentrated and the residue was purified by column chromatography on silica gel to provide the desired product **3a** in 71% yield.

## II. HPLC-HRMS of the radical intermediates

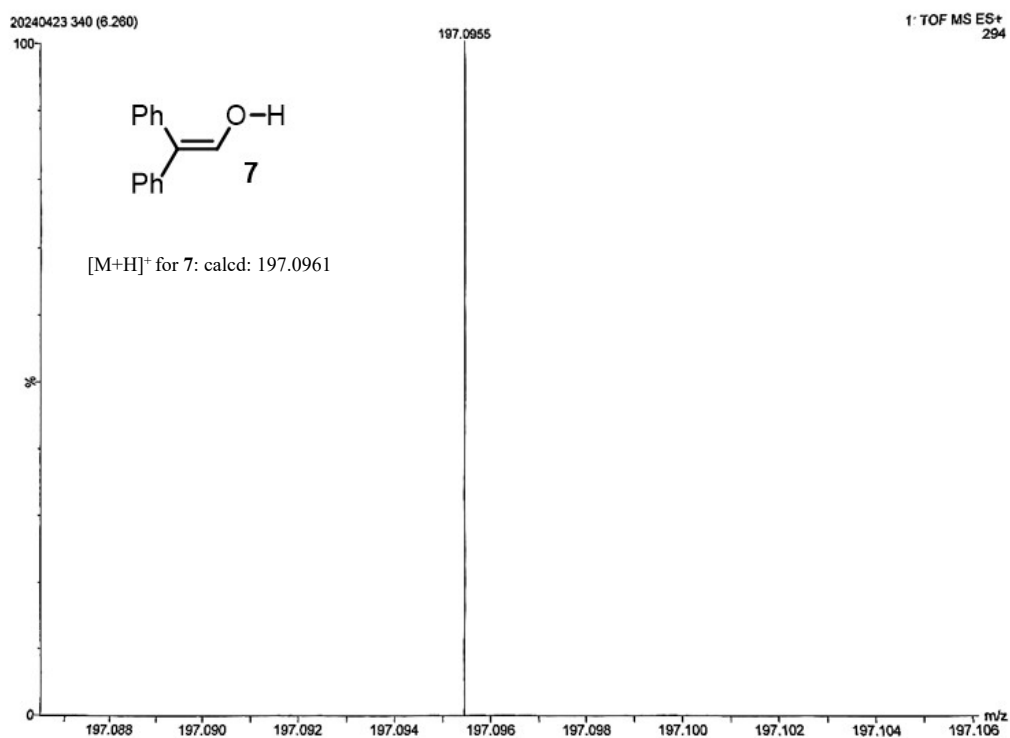
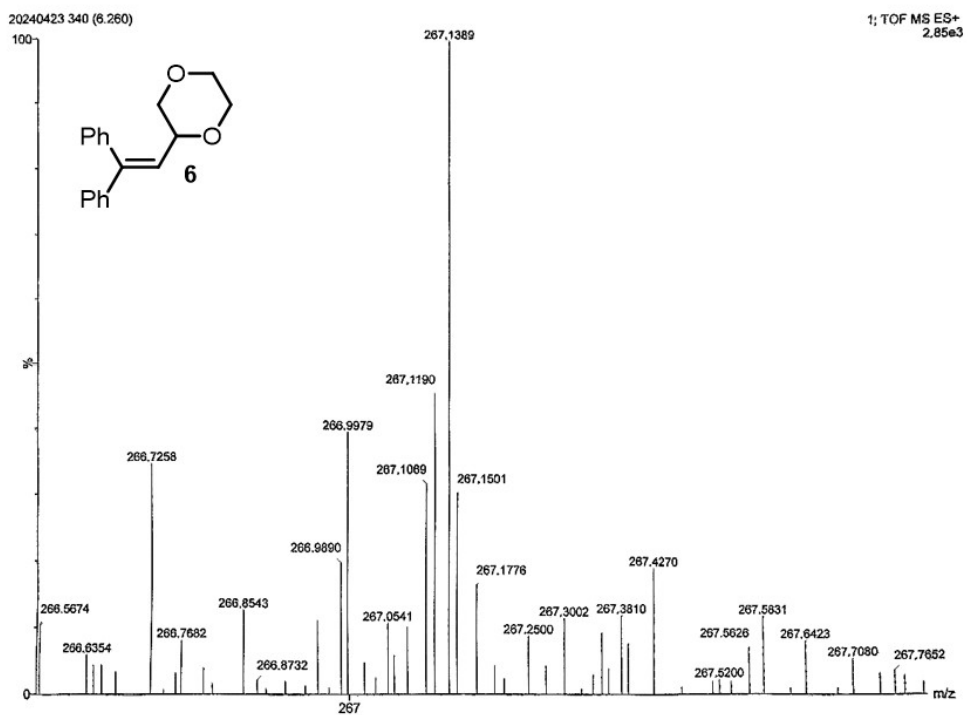


A mixture of **1a** (0.2 mmol, 1.0 equiv.), AgOTf (0.04 mmol, 20 mol%), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.4 mmol, 2.0 equiv.) and BHT (0.4 mmol, 2.0 eq.) in a 25 mL test tube was added the 1,4-Dioxan/H<sub>2</sub>O (1 mL/ 1 mL). The mixture was stirred at 50 °C for 24h. The reaction solution was detected by HPLC-HRMS. The reaction was then quenched with saturated NaCl solution (10 mL), while the aqueous phase was extracted three more times with EtOAc (10\*3 mL). The organic phase was combined, dried over Na<sub>2</sub>SO<sub>4</sub>, the filtrate was concentrated and the residue was purified by column chromatography on silica gel to provide the desired product **3aa** in 12% yield.

A mixture of **1a** (0.2 mmol, 1.0 equiv.), AgOTf (0.04 mmol, 20 mol%), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.4 mmol, 2.0 equiv.) and TEMPO (0.4 mmol, 2.0 eq.) in a 25 mL test tube was added the 1,4-Dioxan/H<sub>2</sub>O (1 mL/1 mL). The mixture was stirred at 50 °C for 24h, No product was detected by TLC. The reaction solution was detected by HPLC-HRMS.

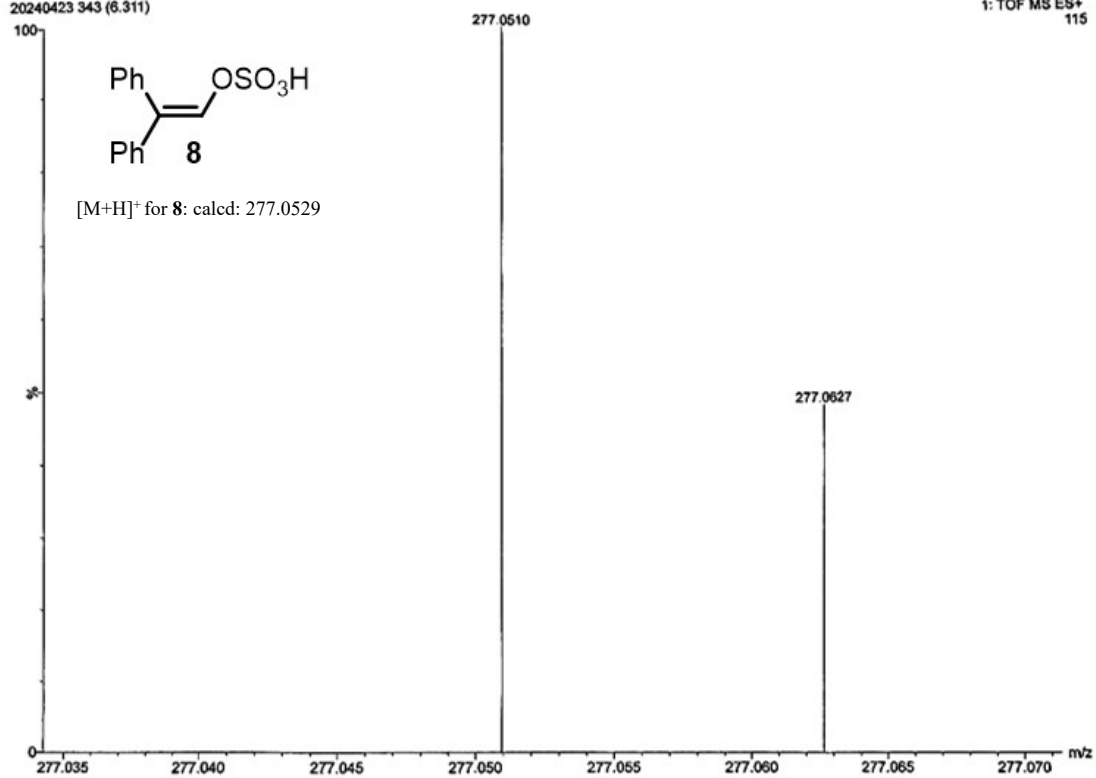
A mixture of **1a** (0.2 mmol, 1.0 equiv.), AgOTf (0.04 mmol, 20 mol%), K<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.4 mmol, 2.0 equiv.) and DMPO (0.4 mmol, 2.0 eq.) in a 25 mL test tube was added the 1,4-Dioxan/H<sub>2</sub>O (1 mL/ 1 mL). The mixture was stirred at 50 °C for 24h, No product was detected by TLC. The reaction

solution was detected by HPLC-HRMS.



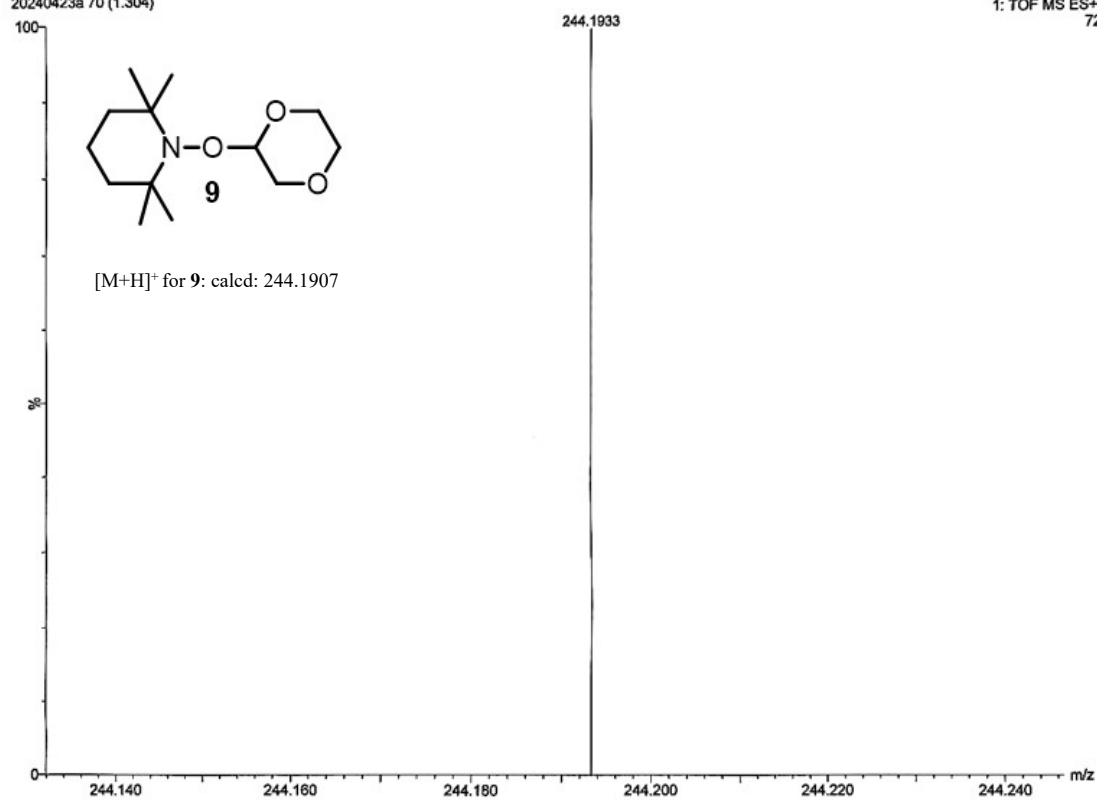
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1: TOF MS ES+  
115



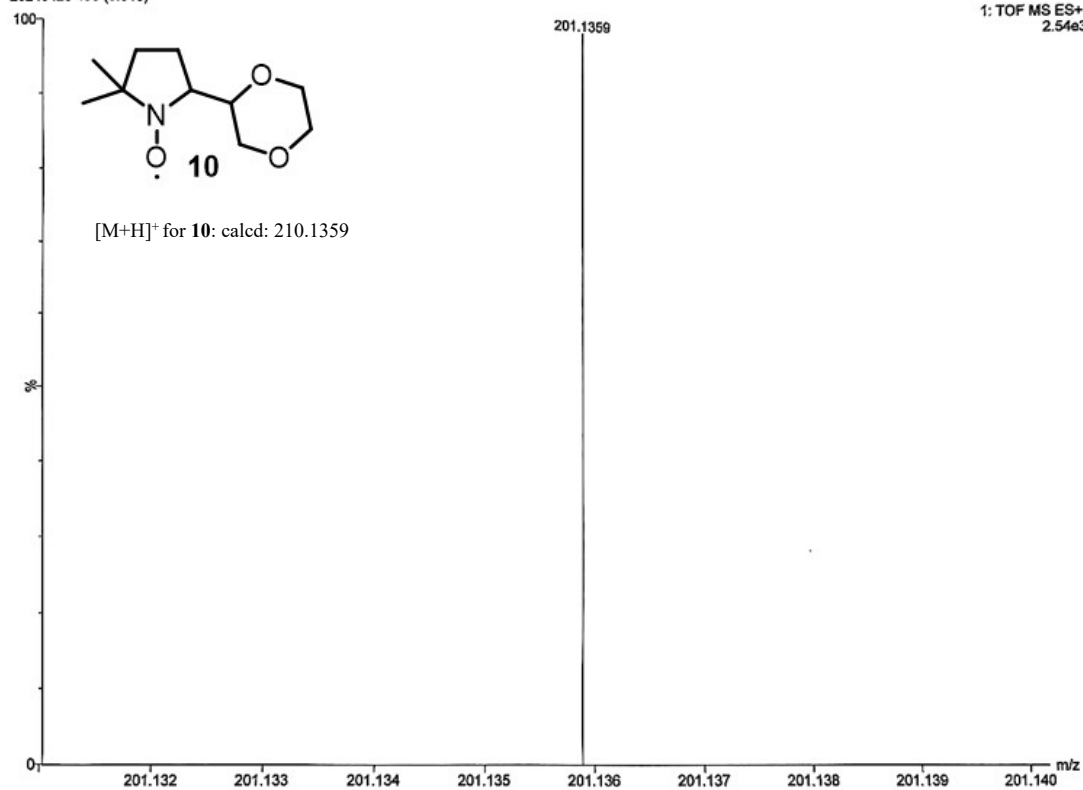
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72



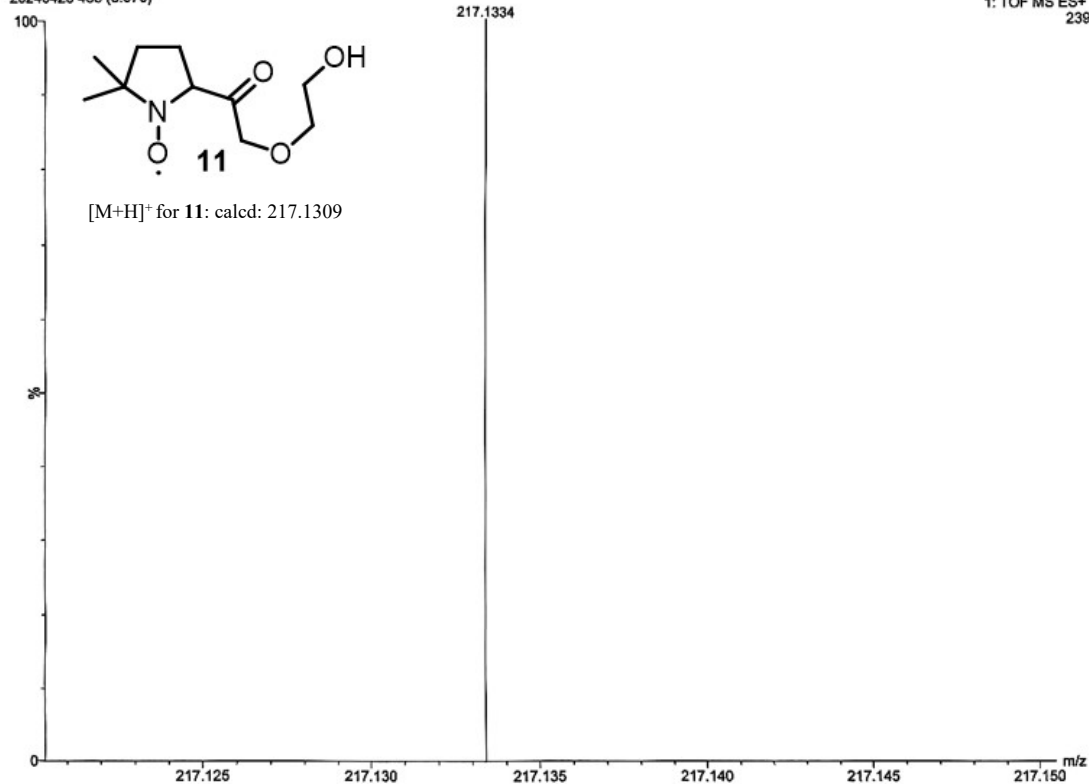
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1: TOF MS ES+  
2.54e3



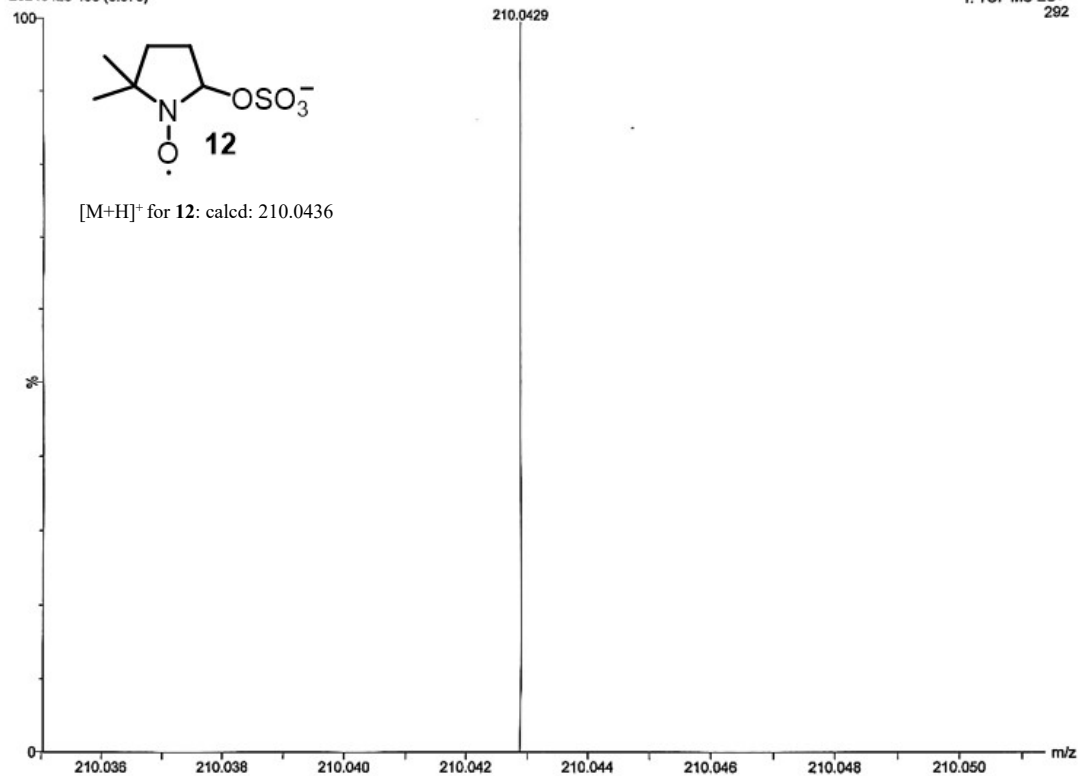
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239



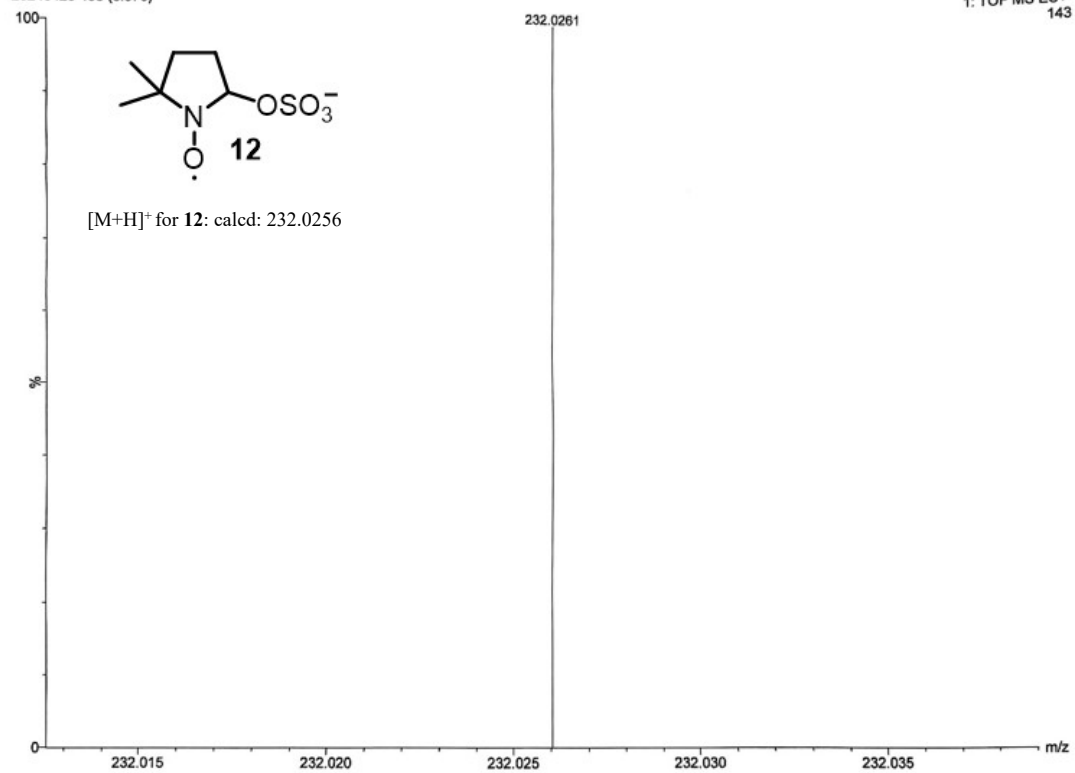
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1: TOF MS ES+  
292

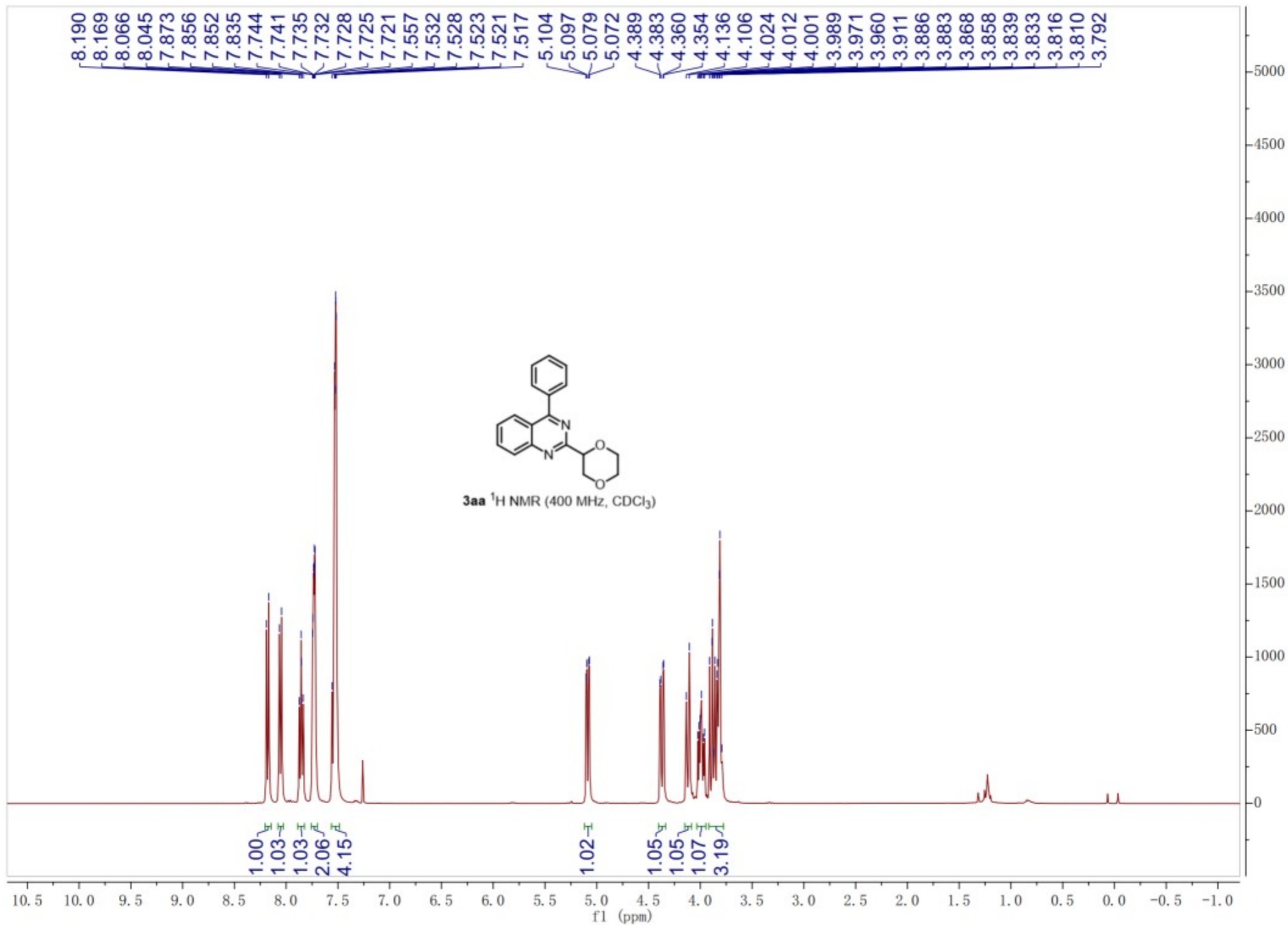


20240423 488 (8.970)

1: TOF MS ES+  
143



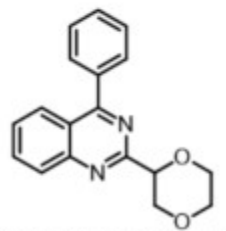
### **III. Copies of NMR Spectra**



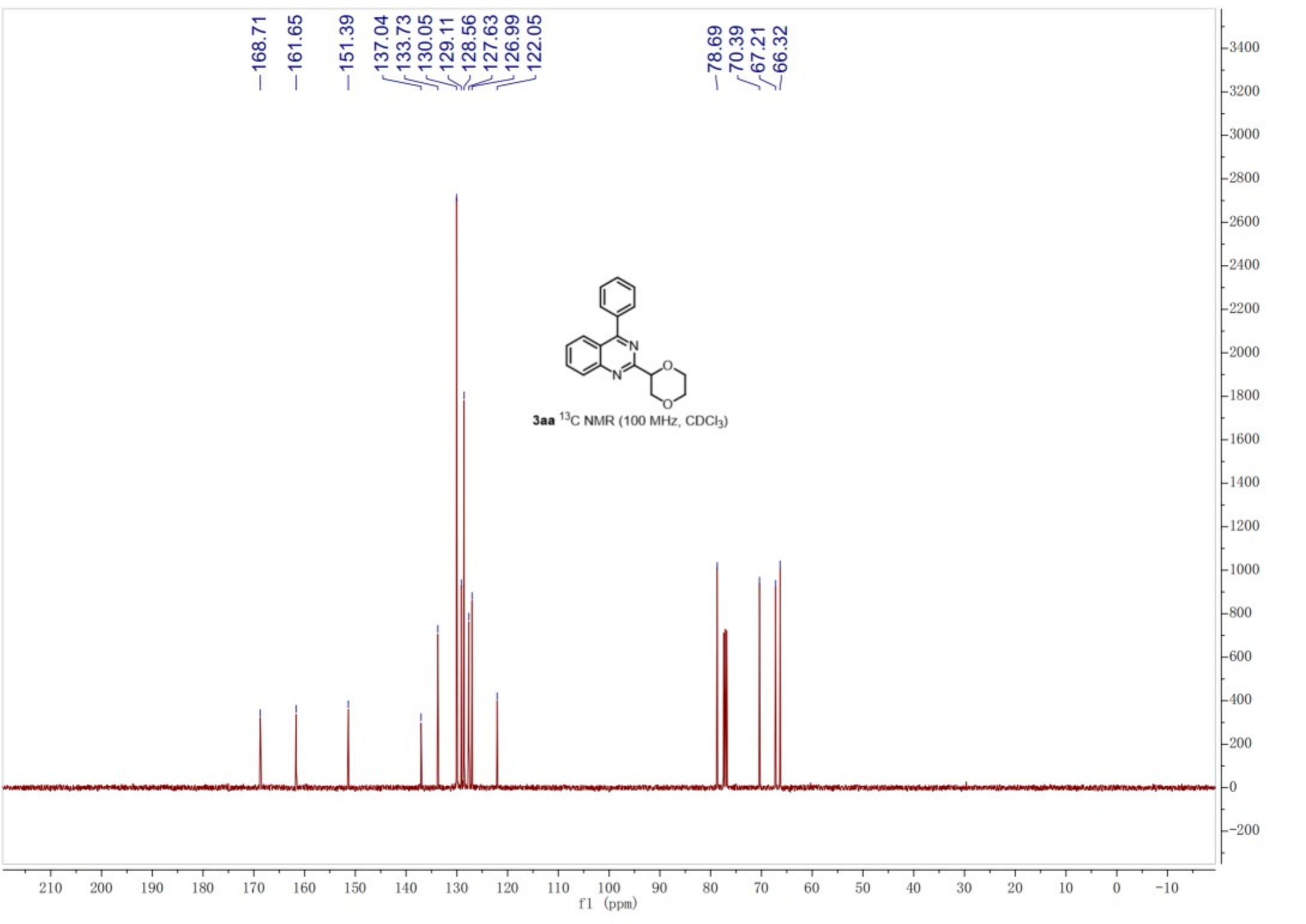


— 168.71  
— 161.65  
— 151.39  
— 137.04  
— 133.73  
— 130.05  
— 129.11  
— 128.56  
— 127.63  
— 126.99  
— 122.05

— 78.69  
— 70.39  
— 67.21  
— 66.32

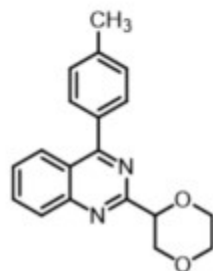


**3aa** <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



8.208  
8.187  
8.124  
8.103  
7.896  
7.875  
7.857  
7.681  
7.661  
7.584  
7.564  
7.545  
7.372  
7.352

5.120  
5.113  
5.095  
5.088  
4.420  
4.414  
4.391  
4.385  
4.164  
4.135  
4.037  
4.026  
4.014  
3.996  
3.985  
3.904  
3.893  
3.878  
3.875  
3.864  
3.857  
3.849  
3.836  
3.819  
2.460



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

1.00  
1.01  
1.01  
2.02  
1.03  
2.01

1.00

1.00  
1.03  
1.06  
3.02

2.96

10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0

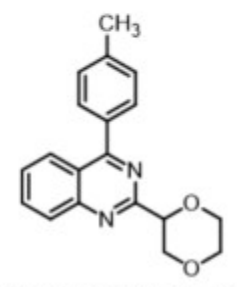
f1 (ppm)

5500  
5000  
4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0  
-500

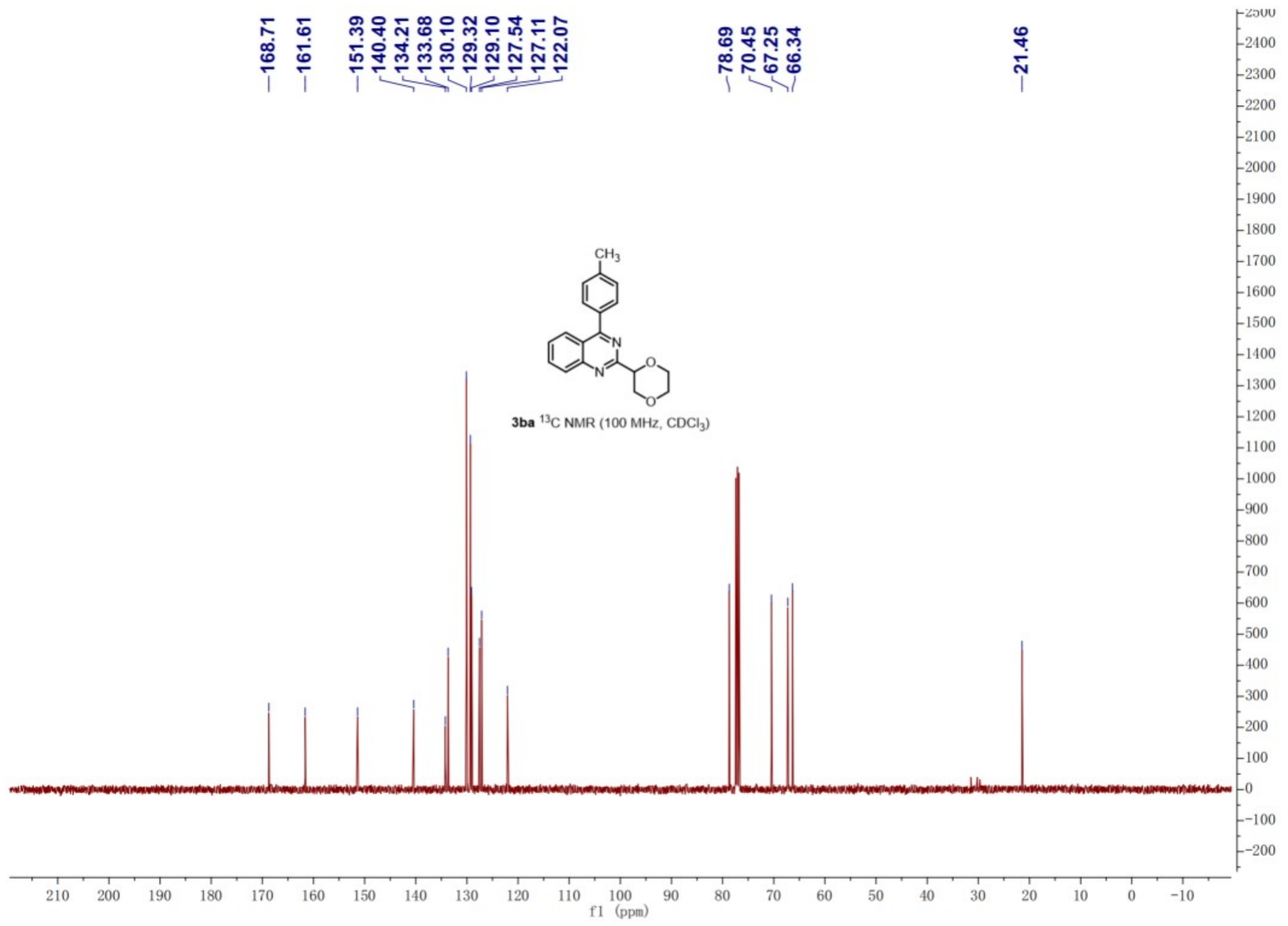
—168.71  
—161.61  
—151.39  
—140.40  
—134.21  
—133.68  
—130.10  
—129.32  
—129.10  
—127.54  
—127.11  
—122.07

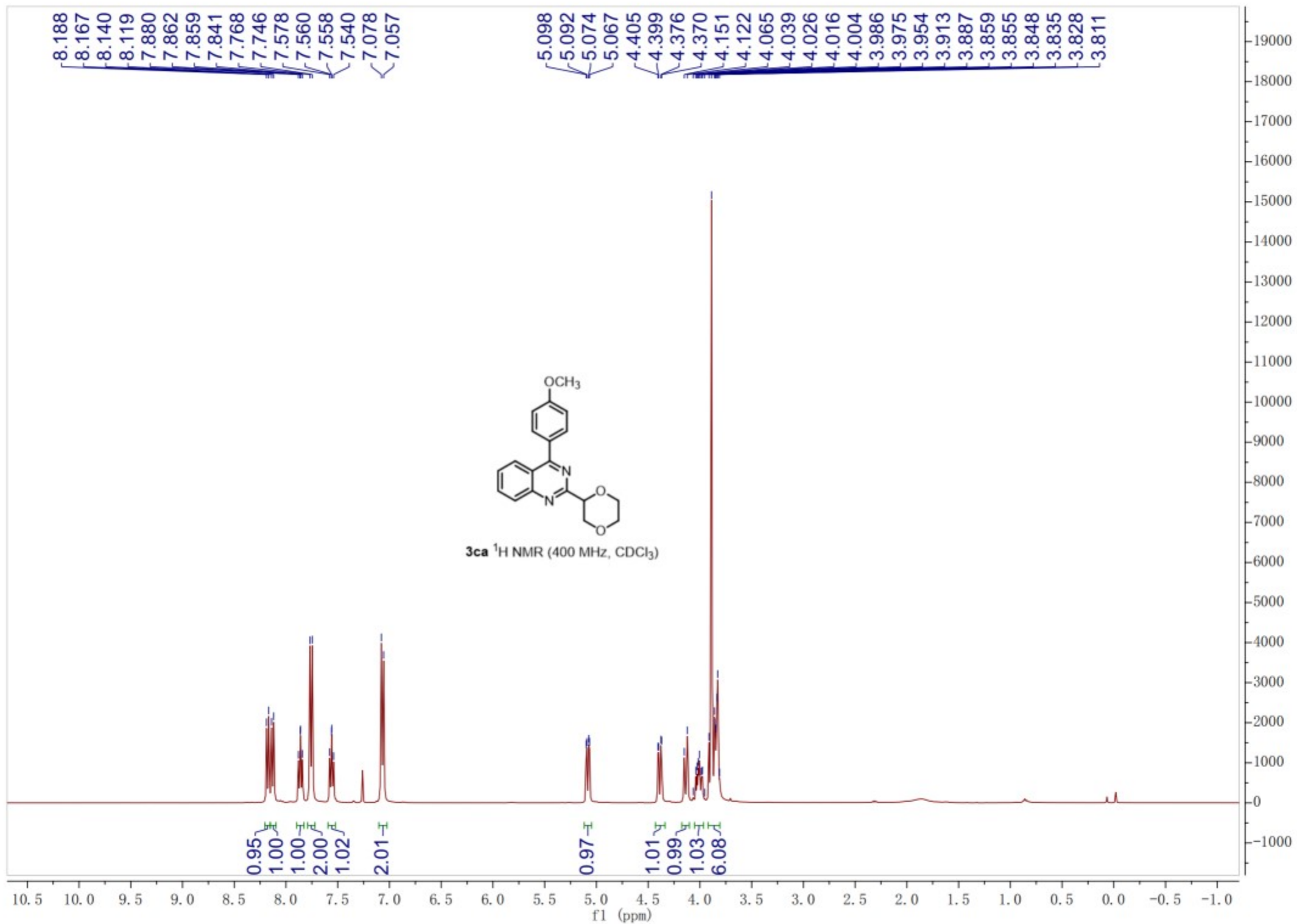
—78.69  
—70.45  
—67.25  
—66.34

—21.46



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

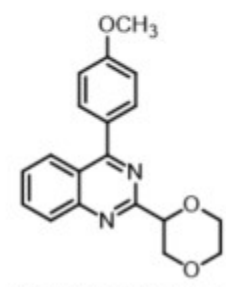




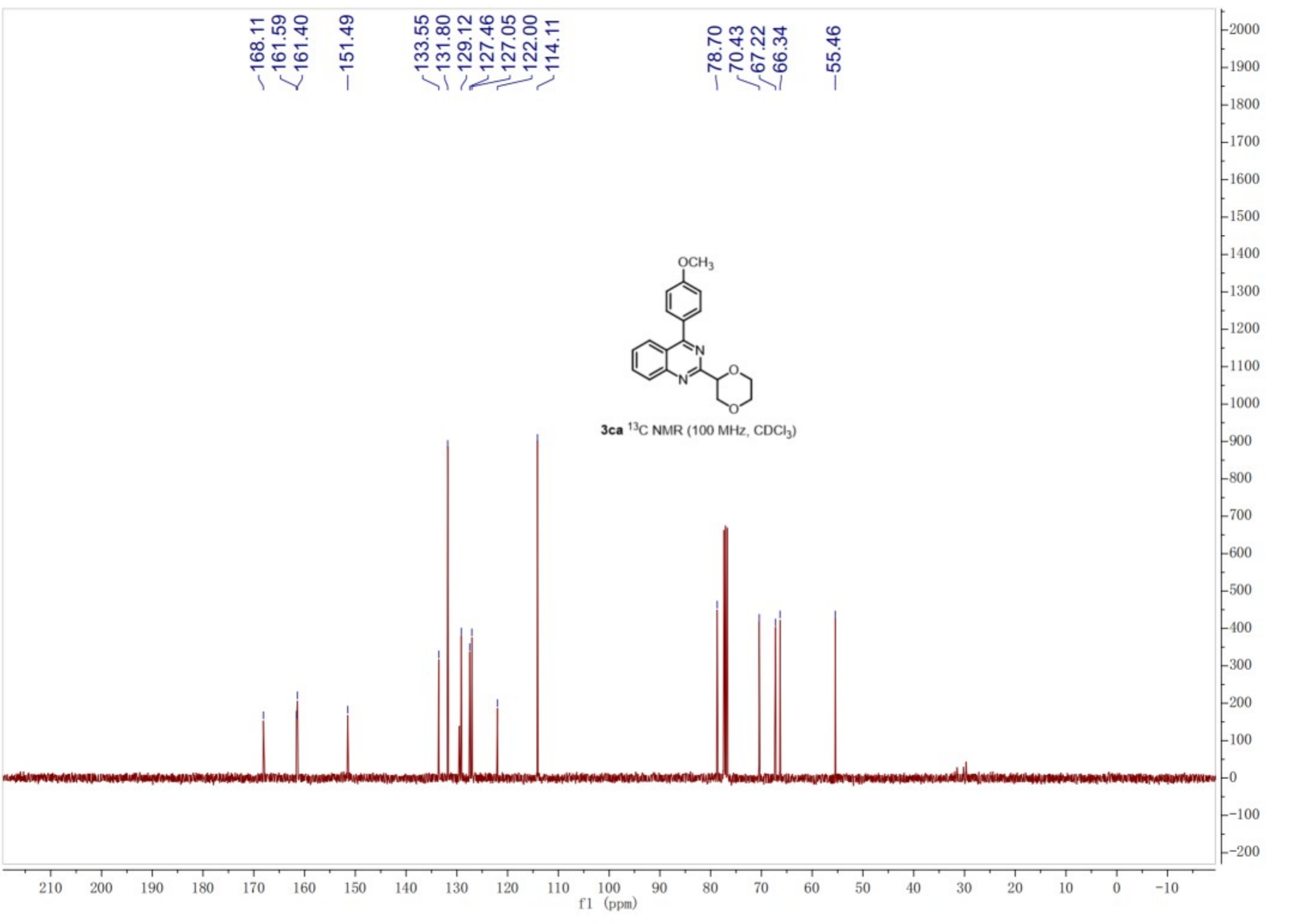
168.11  
161.59  
161.40  
151.49

133.55  
131.80  
129.12  
127.46  
127.05  
122.00  
114.11

78.70  
70.43  
67.22  
66.34  
55.46

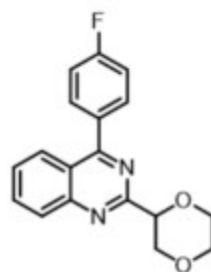


3ca <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



8.209  
8.188  
8.061  
8.040  
7.914  
7.895  
7.877  
7.788  
7.774  
7.767  
7.753  
7.607  
7.589  
7.569  
7.269  
7.247  
7.226

5.103  
5.096  
5.078  
5.072  
4.389  
4.383  
4.361  
4.354  
4.151  
4.122  
4.041  
4.027  
4.020  
4.007  
3.999  
3.990  
3.977  
3.960  
3.909  
3.884  
3.856  
3.846  
3.832  
3.829  
3.816



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

1.00  
1.04  
1.05  
2.03  
1.02  
2.09

1.01

1.05  
1.04  
1.15  
0.97  
2.06

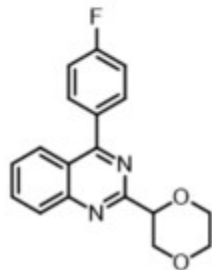
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0  
f1 (ppm)

5000  
4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0

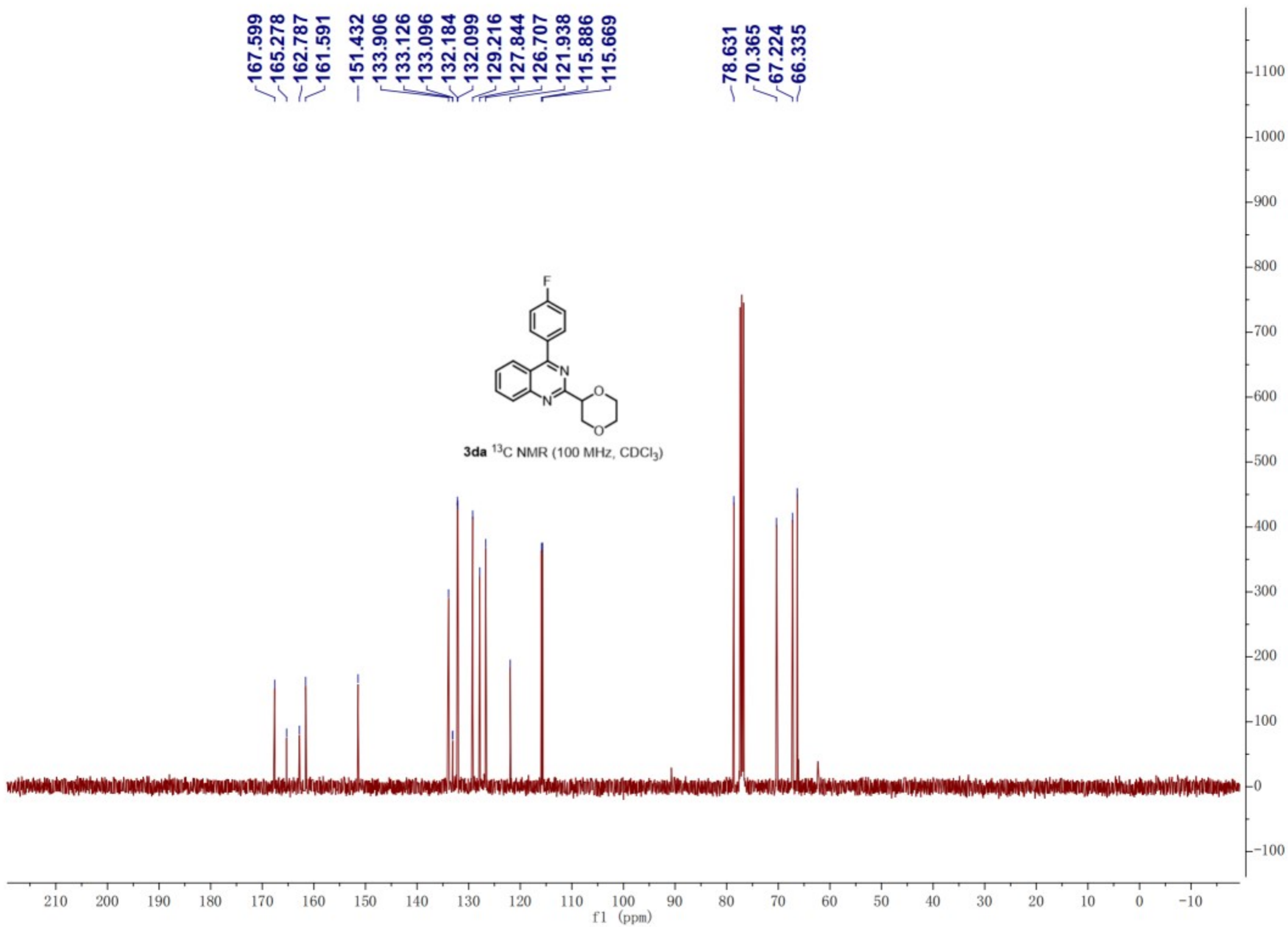


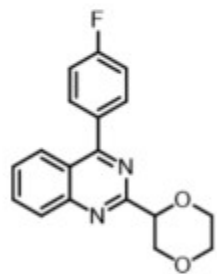
167.599  
165.278  
162.787  
161.591  
151.432  
133.906  
133.126  
133.096  
132.184  
132.099  
129.216  
127.844  
126.707  
121.938  
115.886  
115.669

78.631  
70.365  
67.224  
66.335

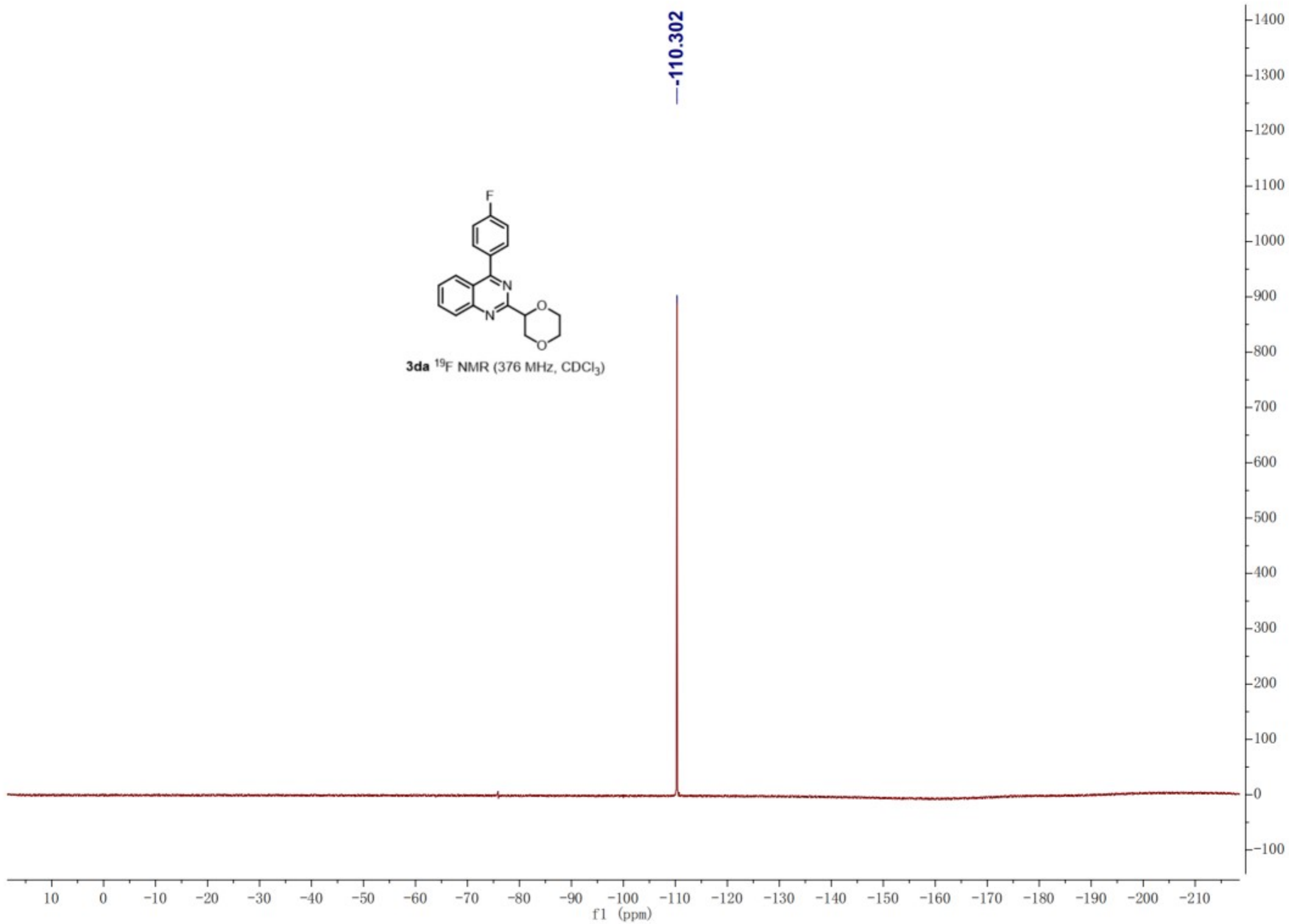


**3da**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )





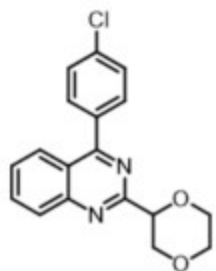
**3da**  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )





8.215  
8.194  
8.048  
8.027  
7.920  
7.899  
7.881  
7.723  
7.702  
7.610  
7.590  
7.571  
7.544  
7.523

5.104  
5.097  
5.079  
5.073  
4.390  
4.383  
4.361  
4.355  
4.155  
4.126  
4.042  
4.027  
4.022  
4.007  
3.999  
3.991  
3.978  
3.901  
3.876  
3.854  
3.848  
3.833  
3.829



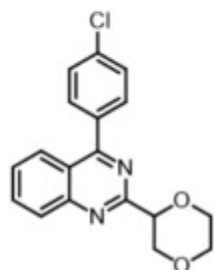
**3ea** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

1.00  
1.01  
1.03  
2.02  
1.03  
2.02

1.01  
1.02  
1.03  
1.05  
3.10

10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0  
f1 (ppm)

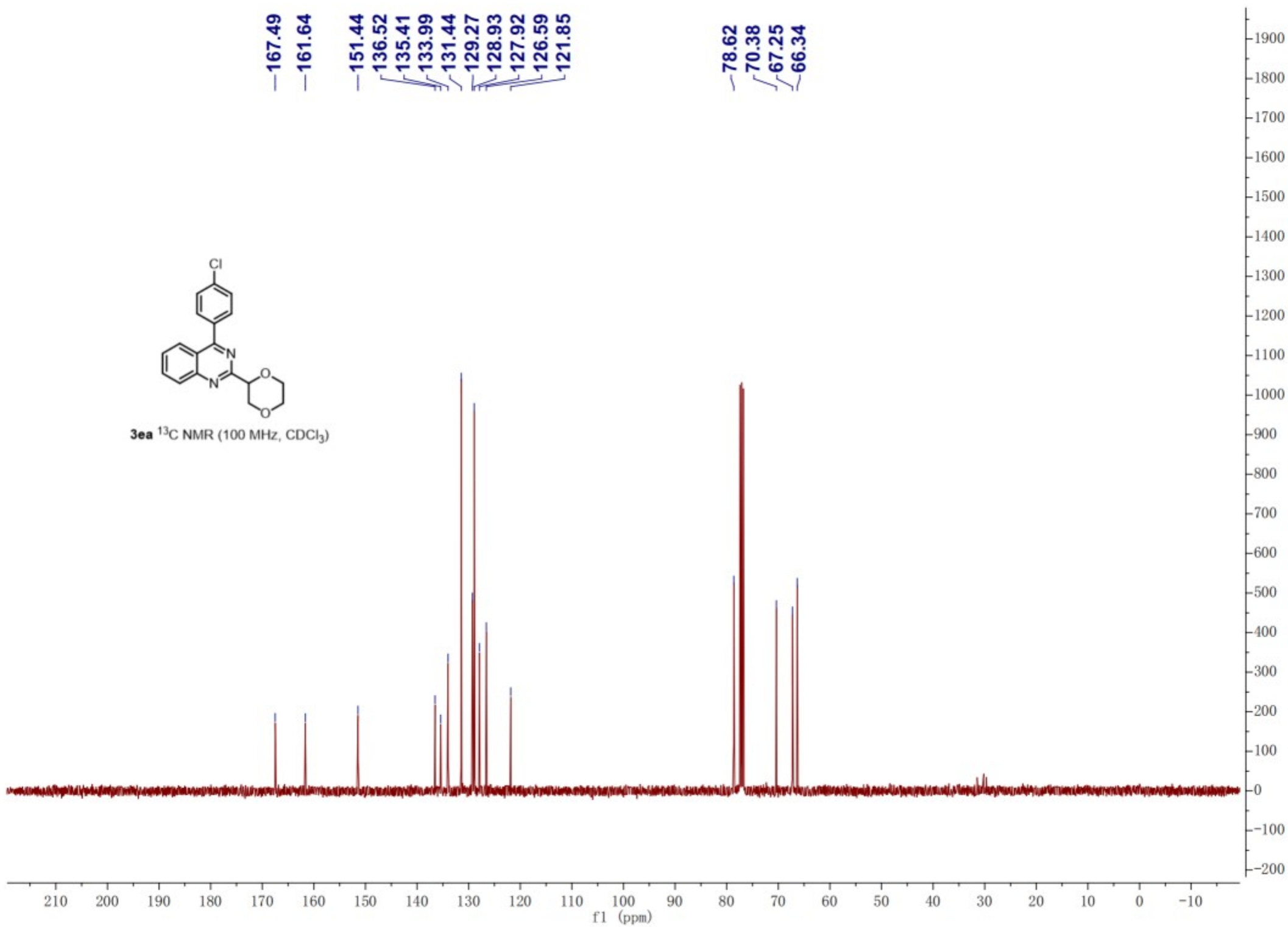
5000  
4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0

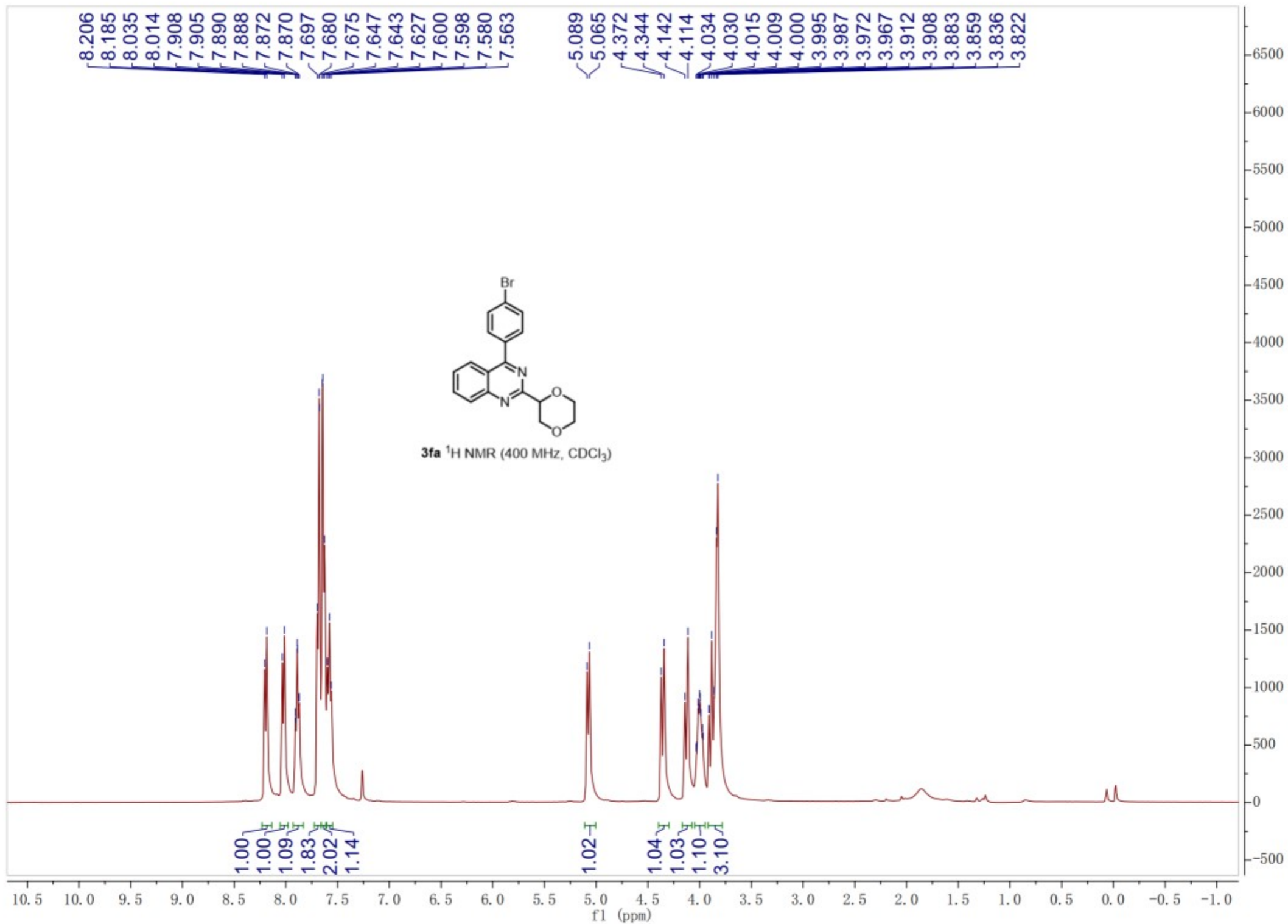


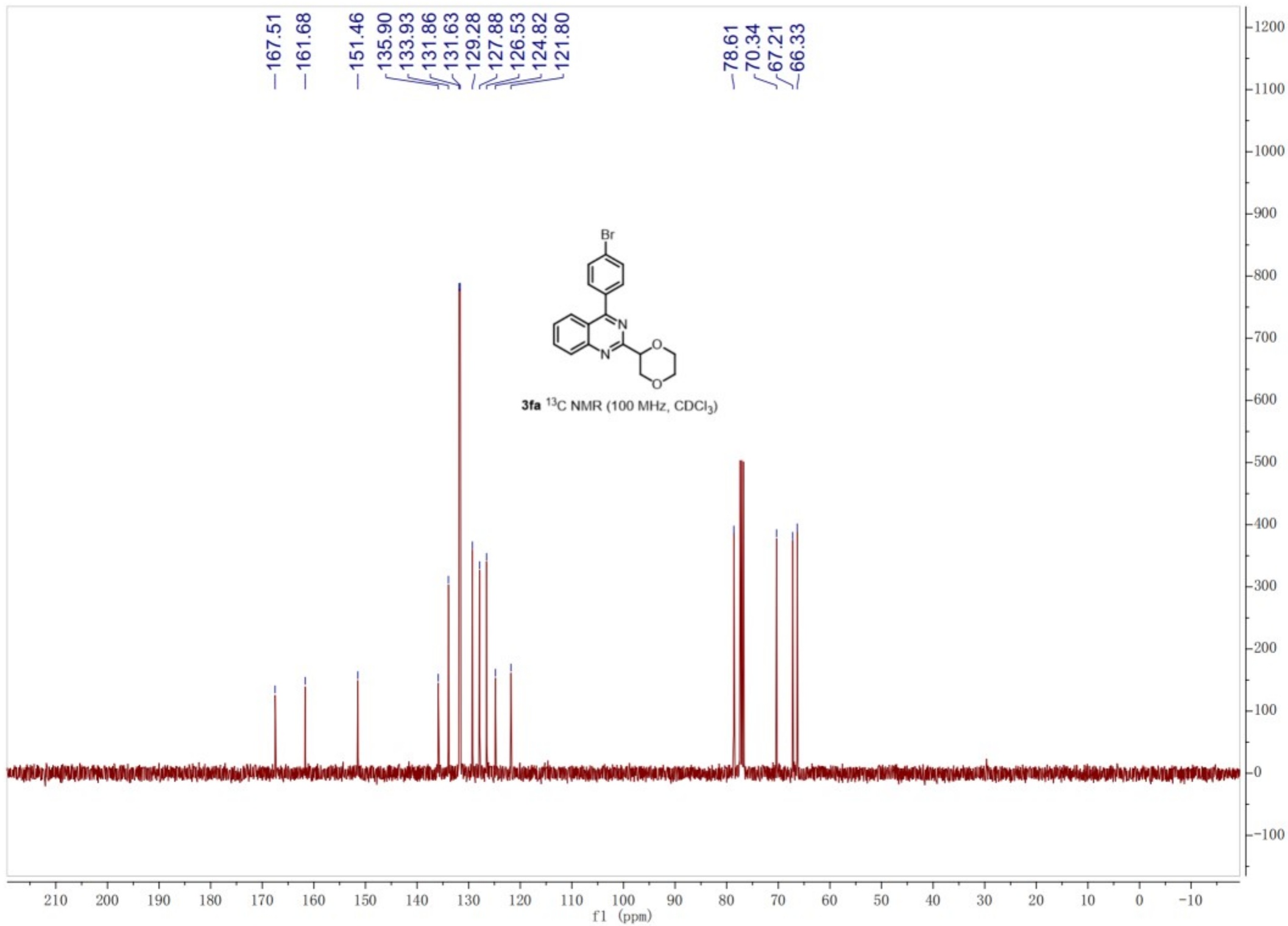
**3ea**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

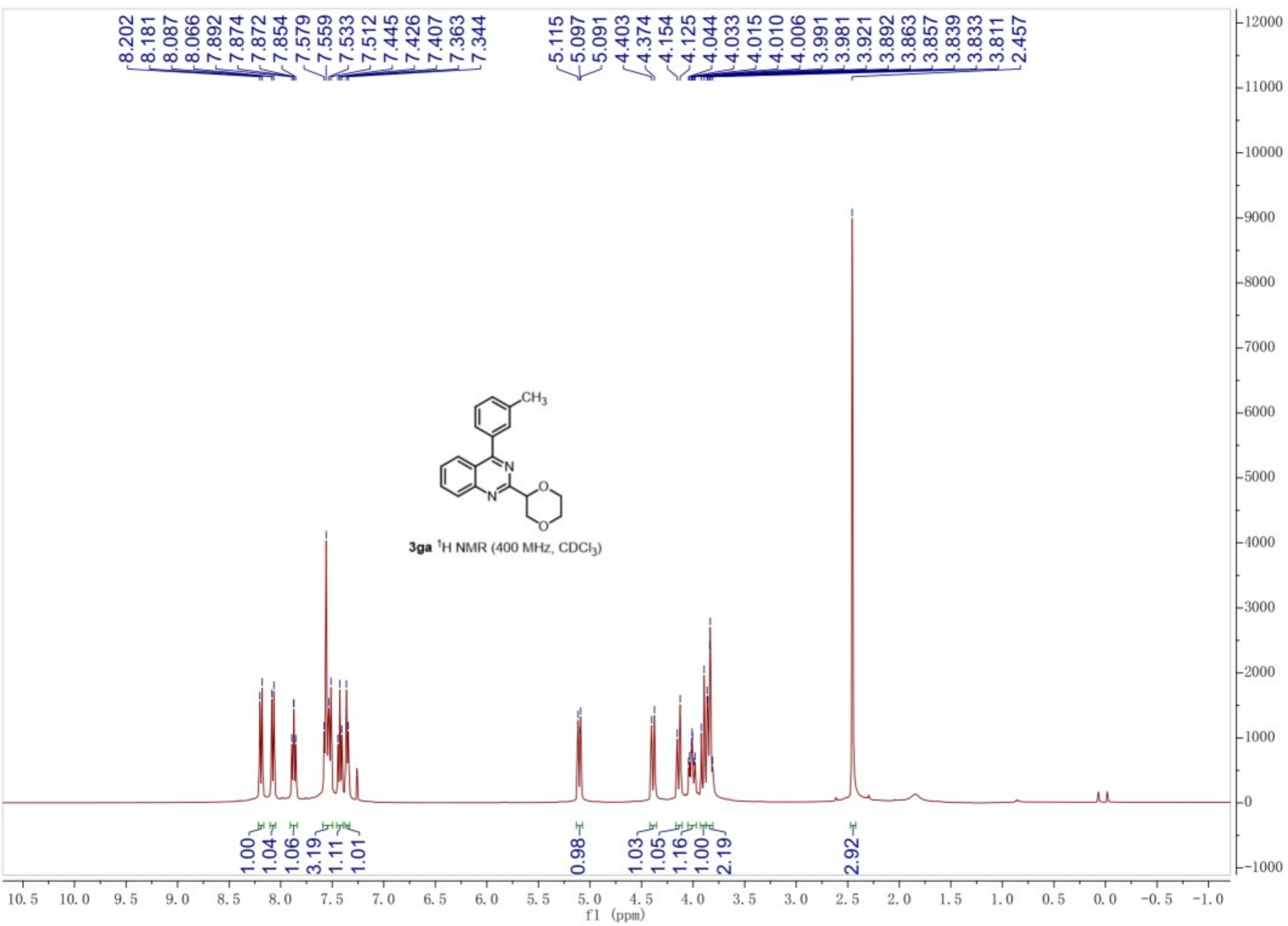
167.49  
161.64  
151.44  
136.52  
135.41  
133.99  
131.44  
129.27  
128.93  
127.92  
126.59  
121.85

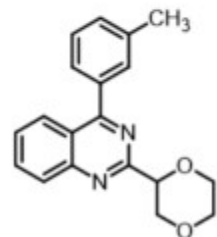
78.62  
70.38  
67.25  
66.34











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

— 169.00  
— 161.64  
— 151.35  
— 138.46  
— 136.99  
— 133.70  
— 130.80  
— 130.54  
— 129.09  
— 128.39  
— 127.56  
— 127.21  
— 127.13  
— 122.14

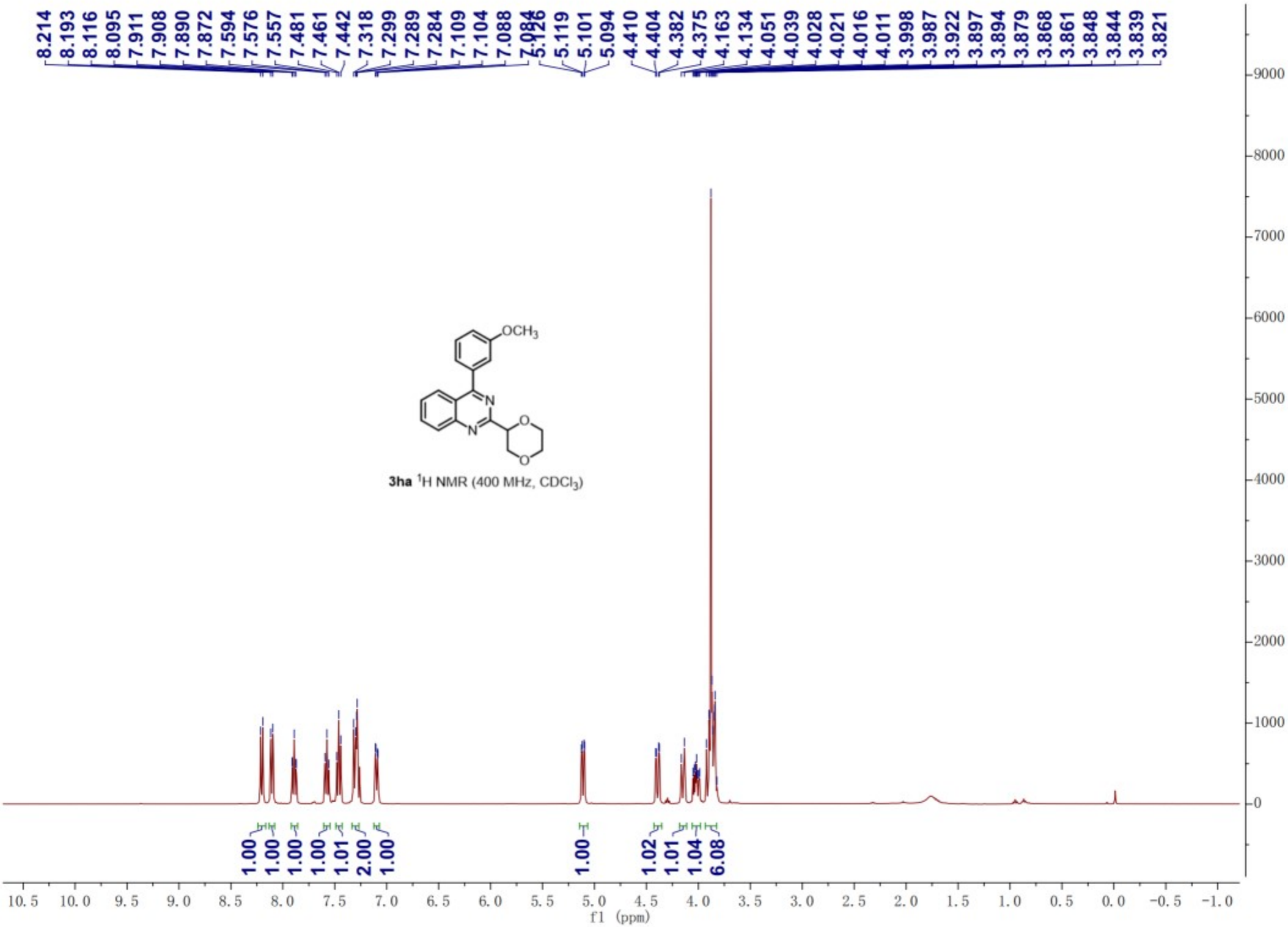
— 78.73  
— 70.43  
— 67.24  
— 66.34

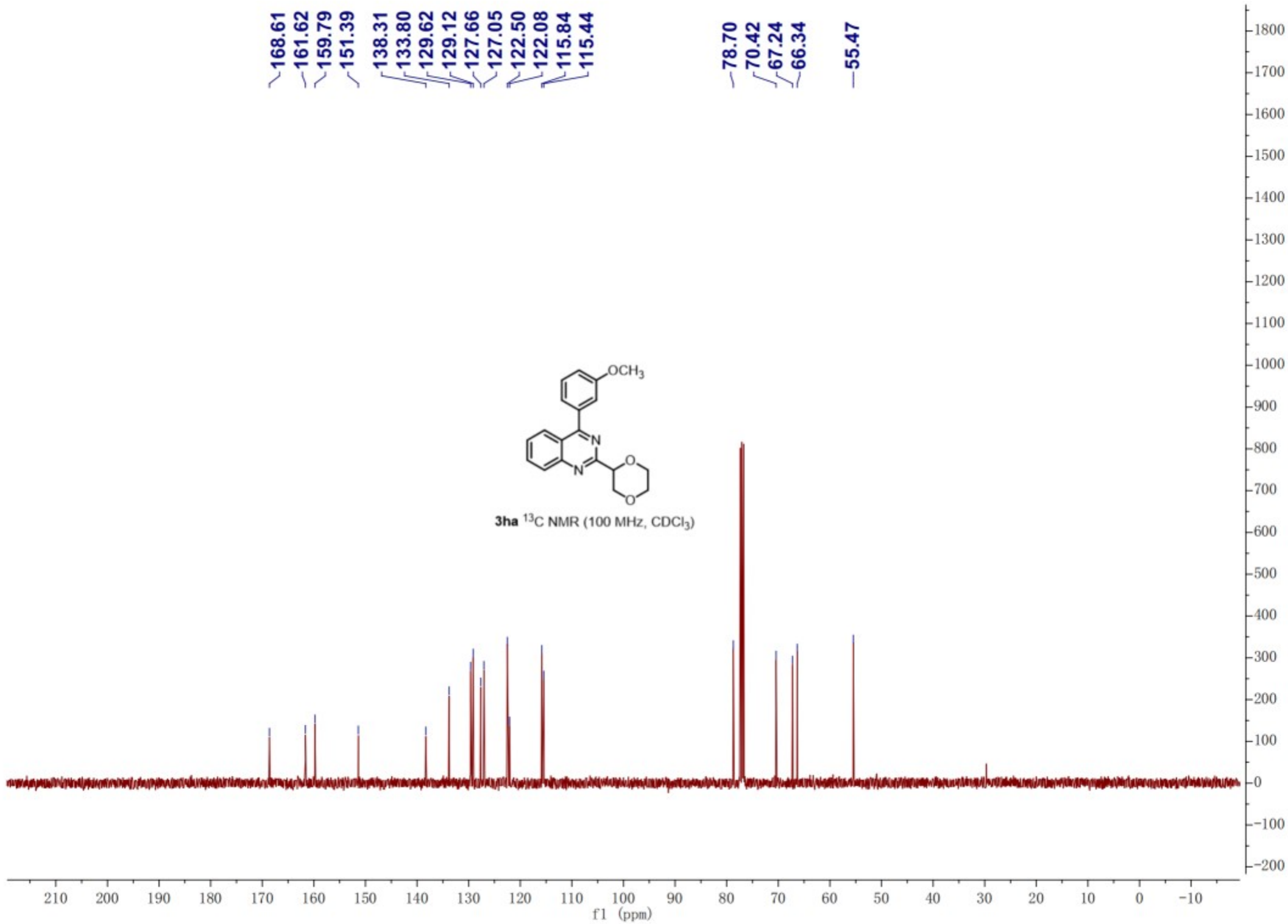
— 21.47

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10  
f1 (ppm)

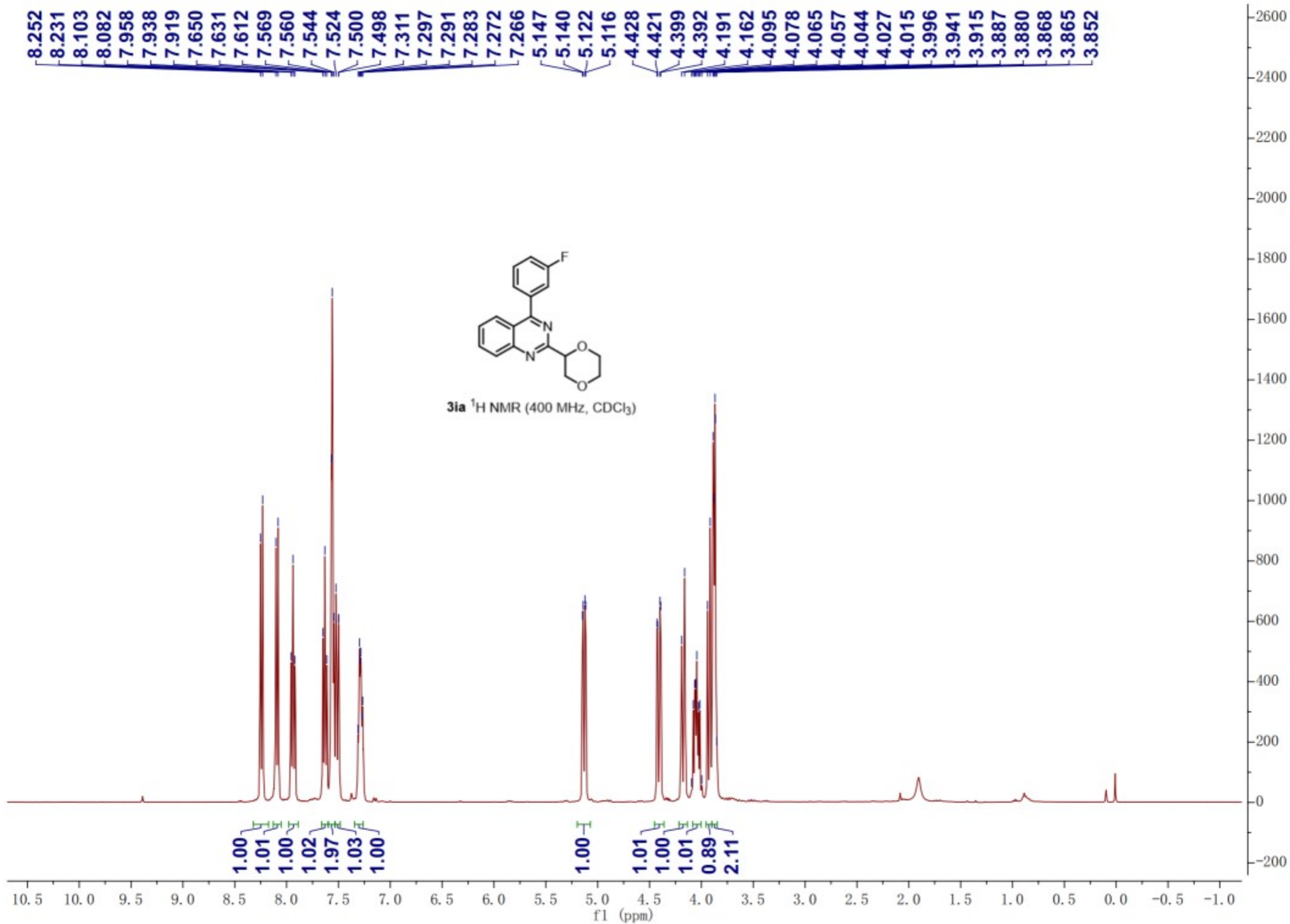
1100  
1000  
900  
800  
700  
600  
500  
400  
300  
200  
100  
0  
-100



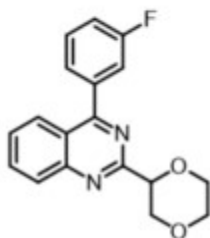




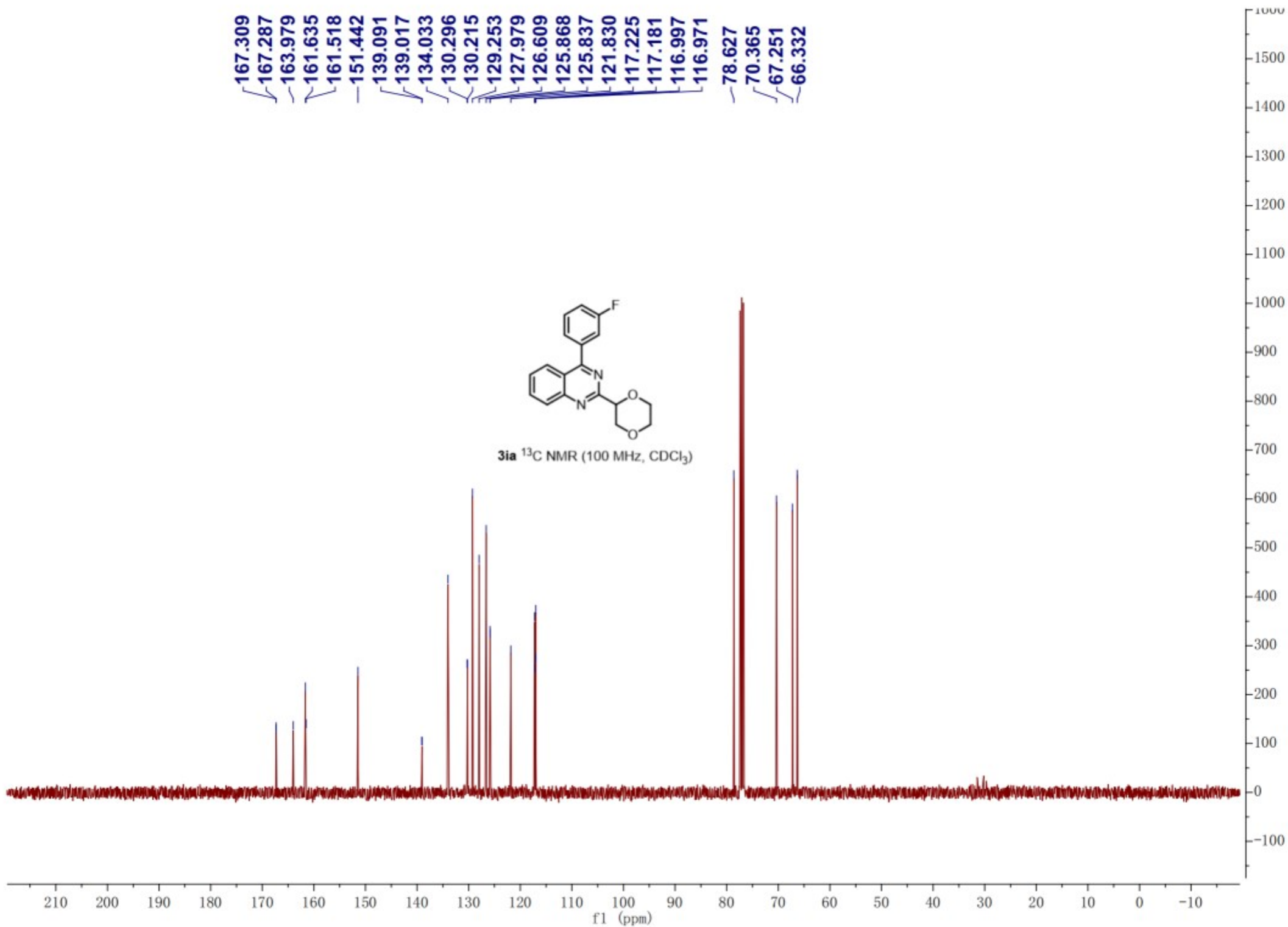


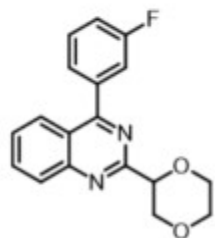


167.309  
167.287  
163.979  
161.635  
161.518  
151.442  
139.091  
139.017  
134.033  
130.296  
130.215  
129.253  
127.979  
126.609  
125.868  
125.837  
121.830  
117.225  
117.181  
116.997  
116.971  
78.627  
70.365  
67.251  
66.332

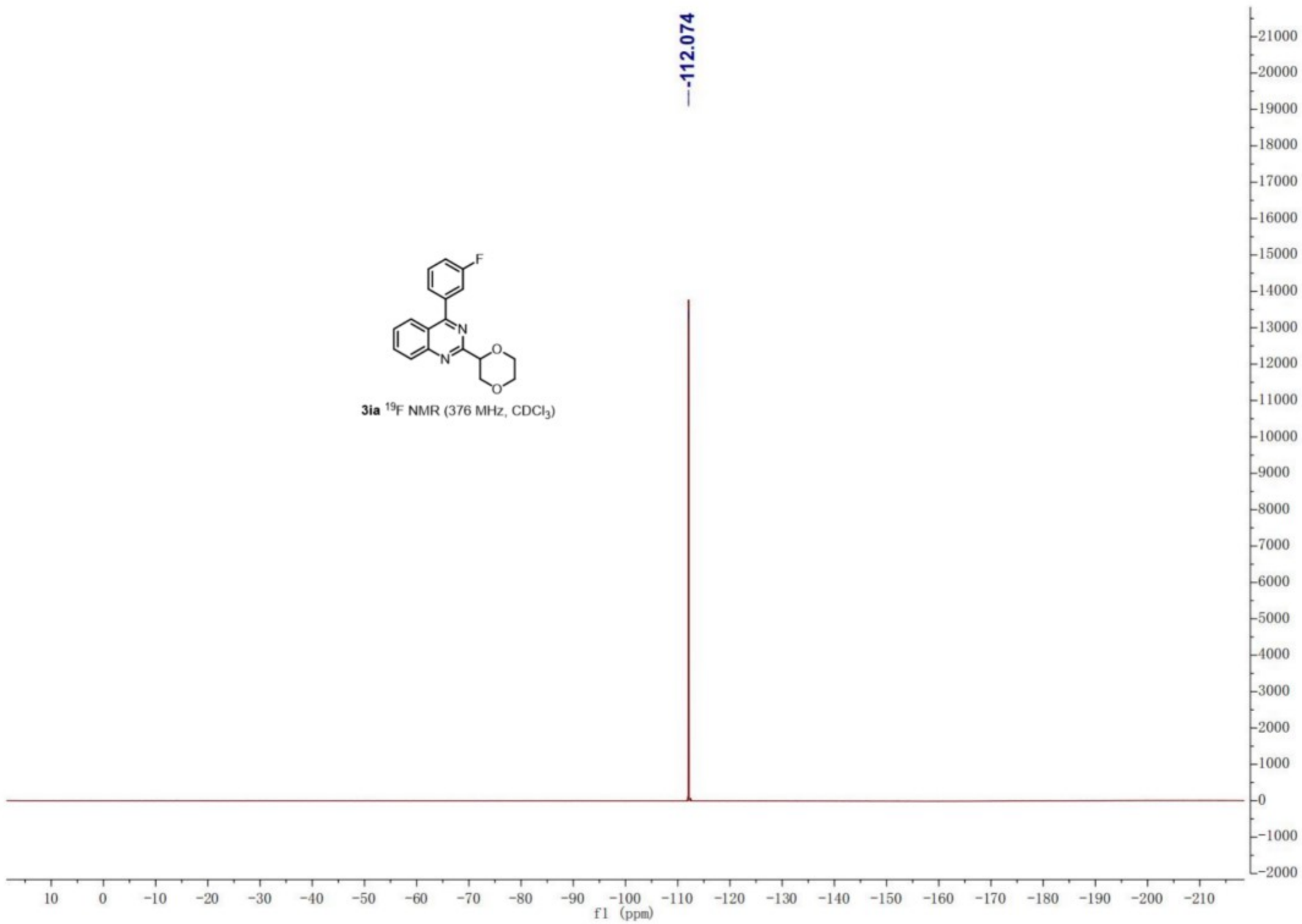


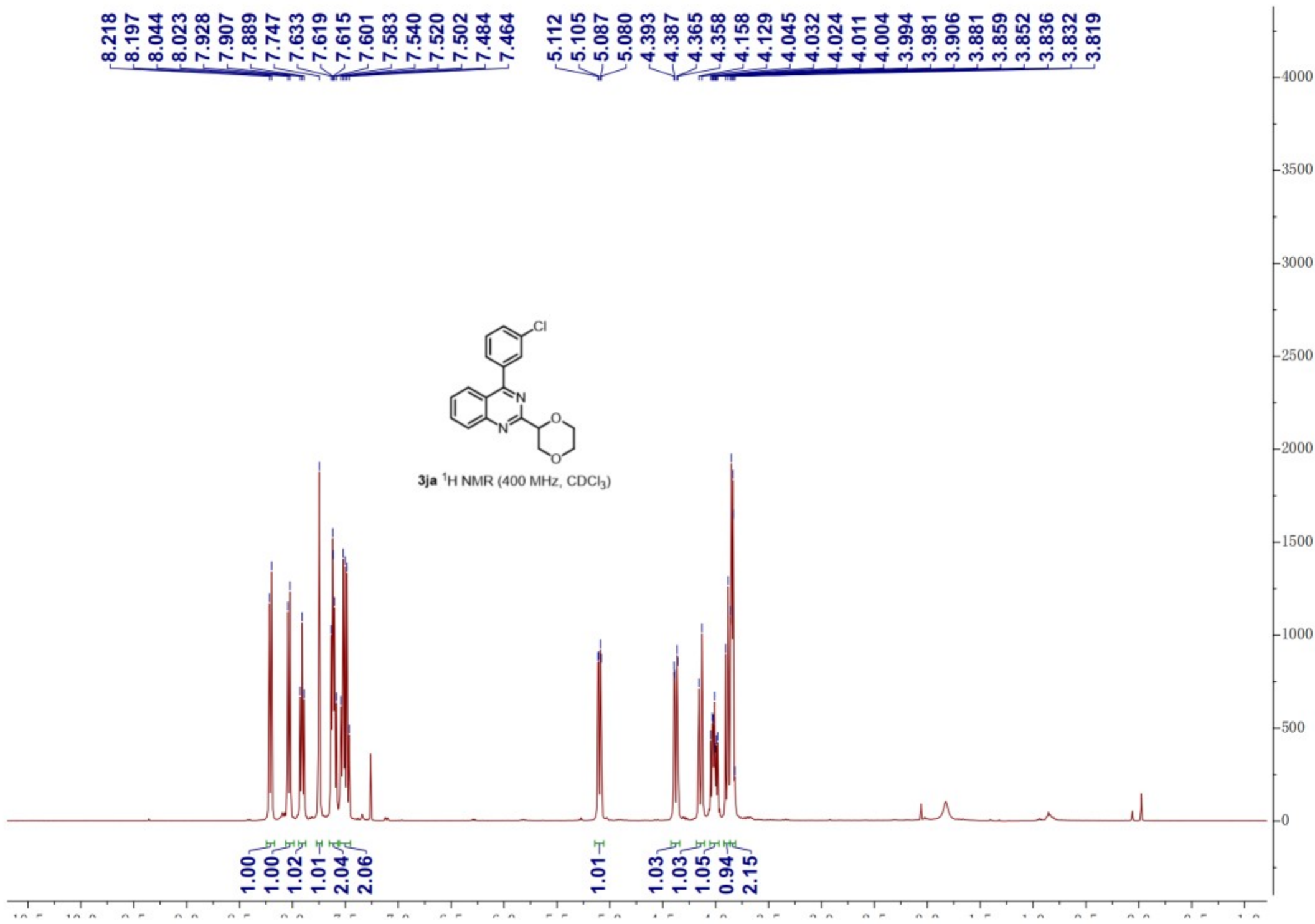
3ia <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

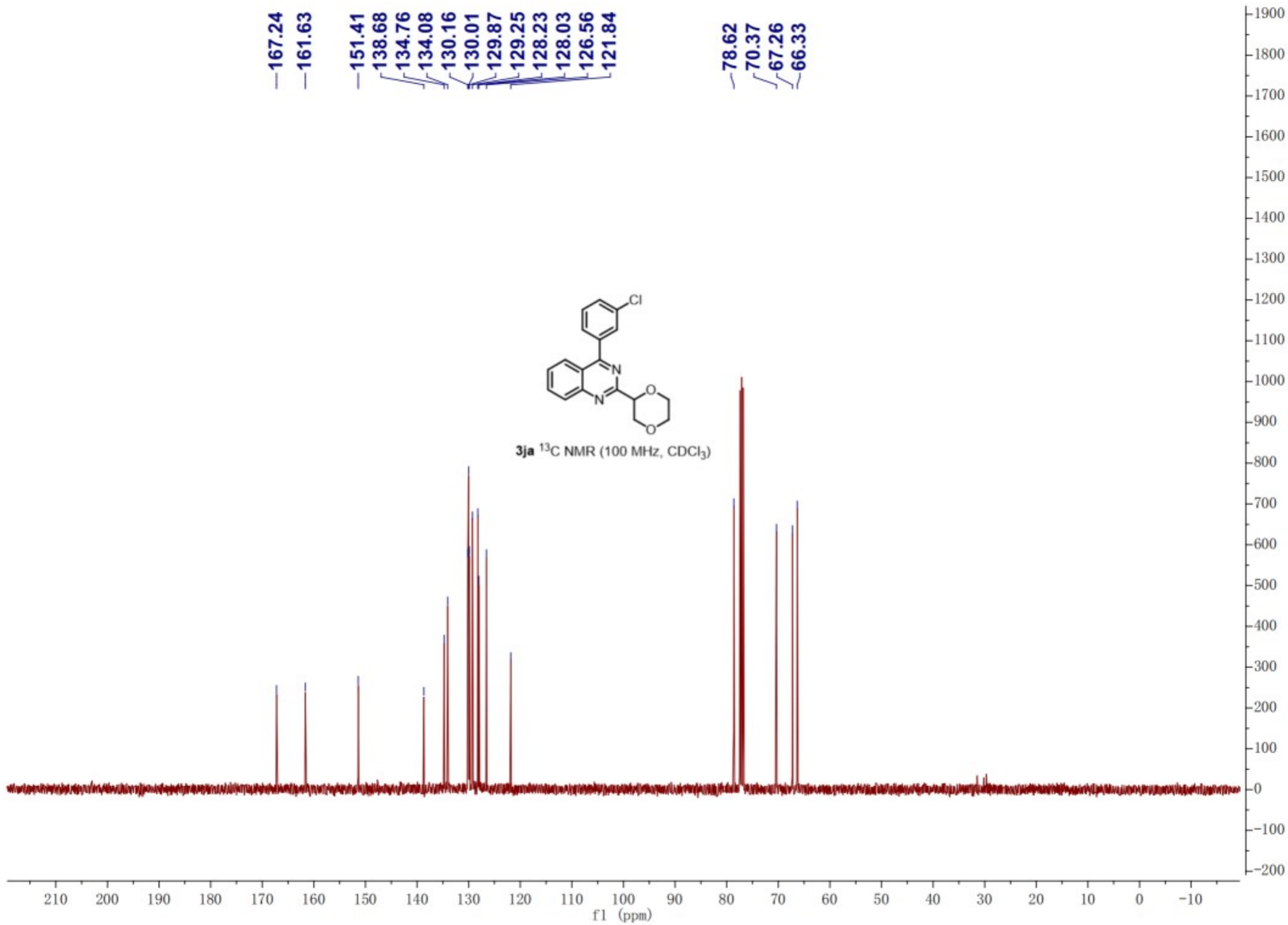


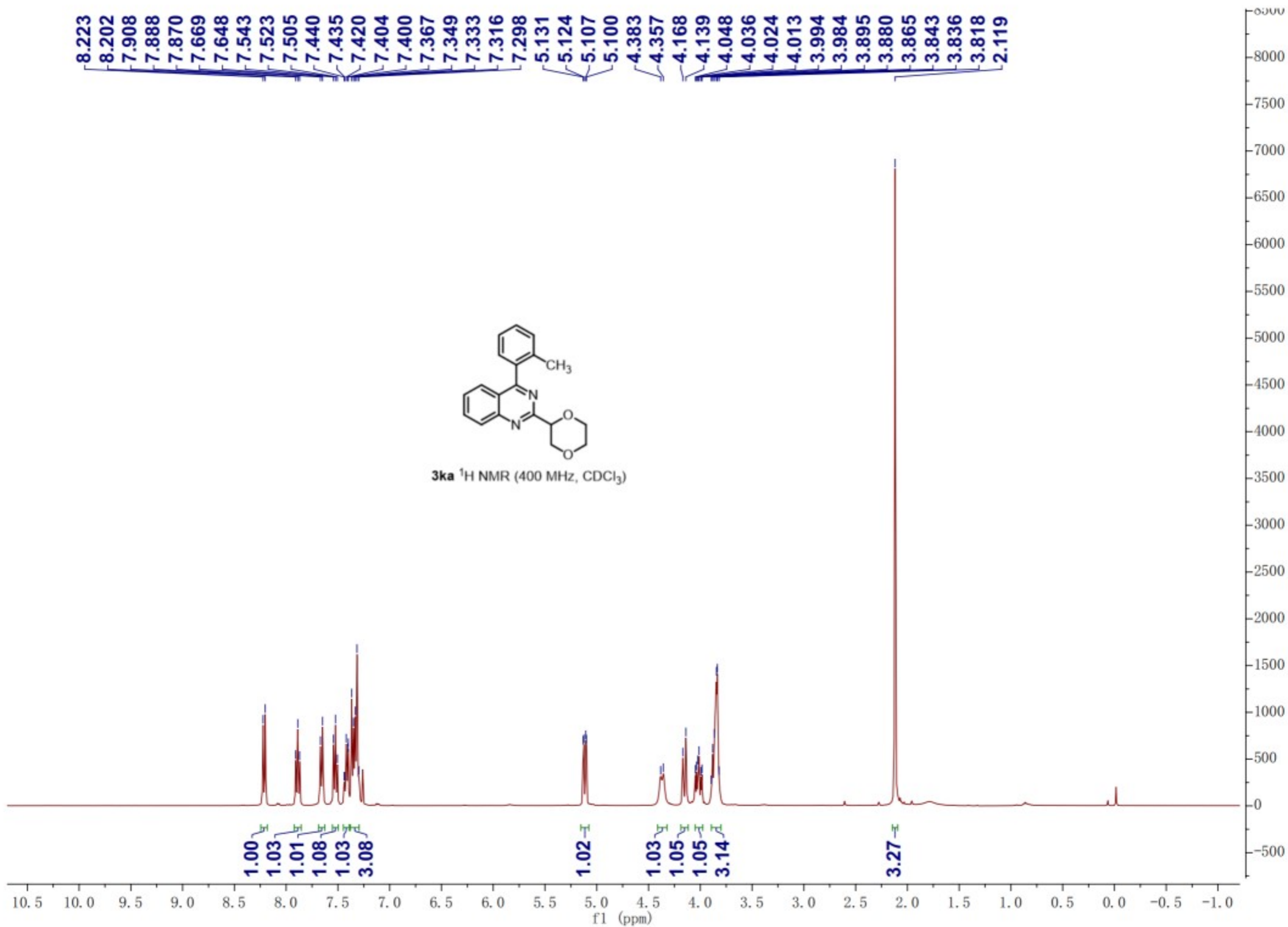


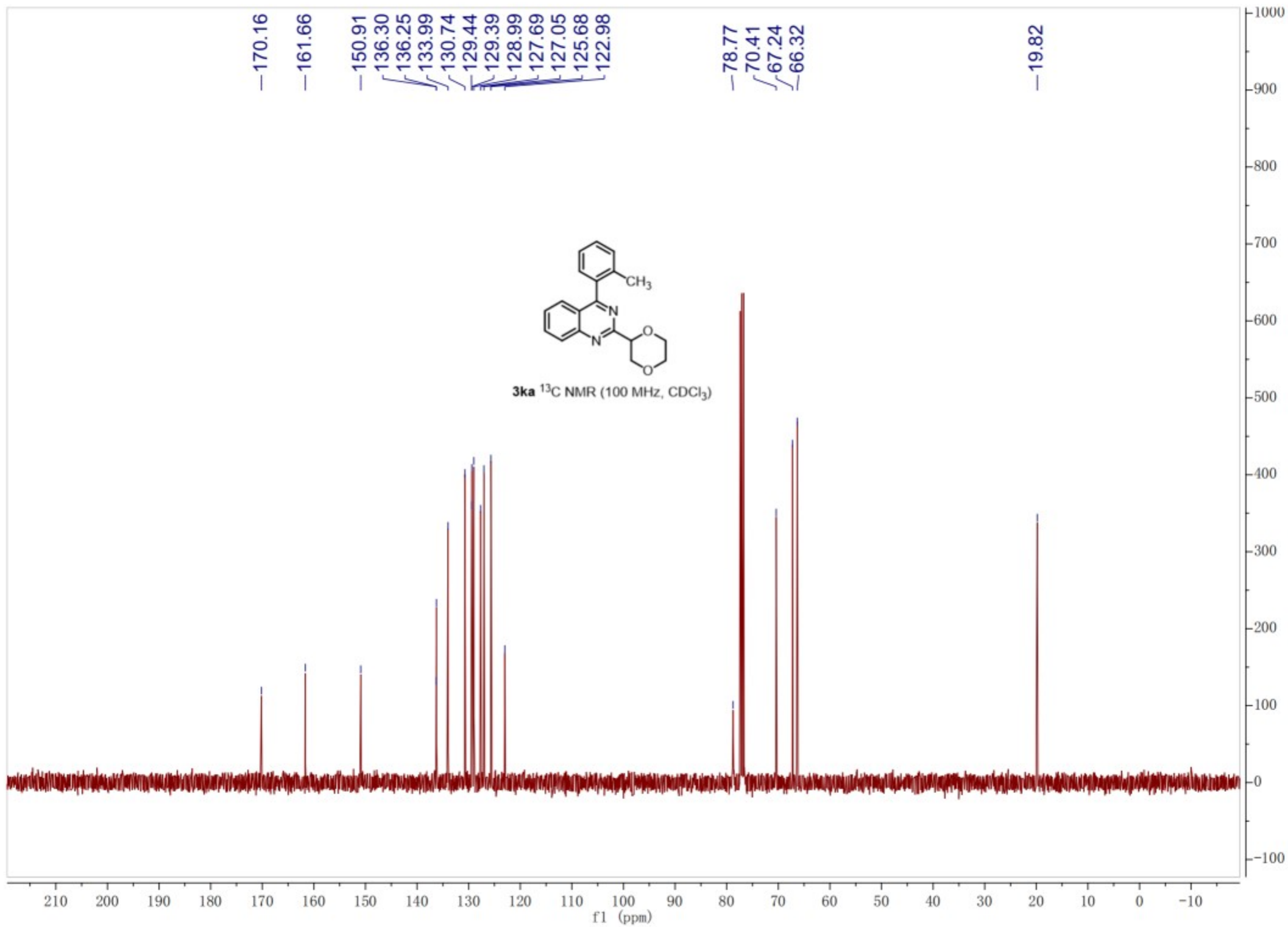
**3ia**  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )



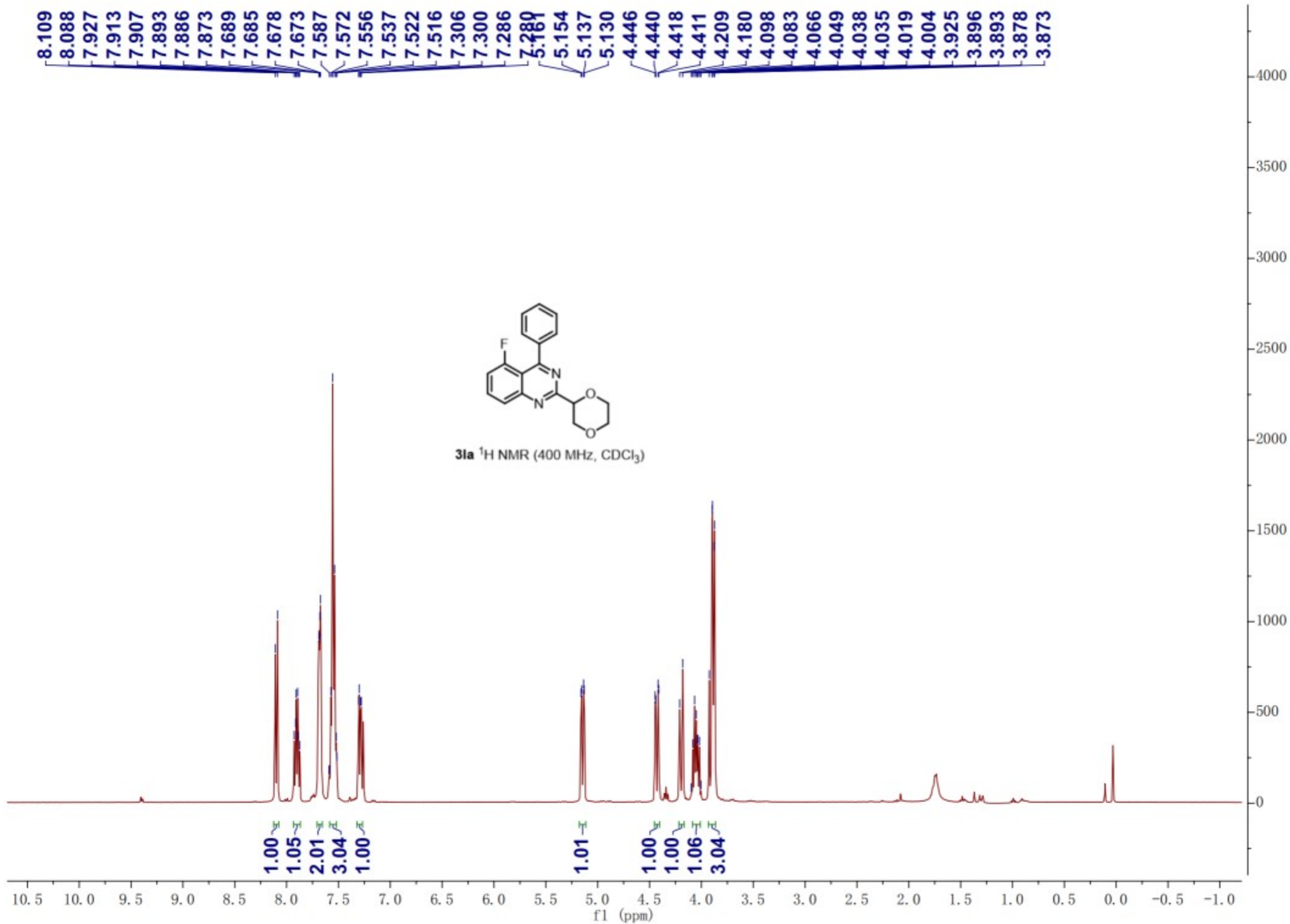








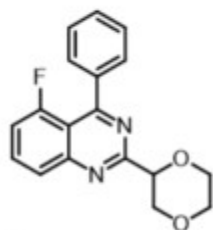




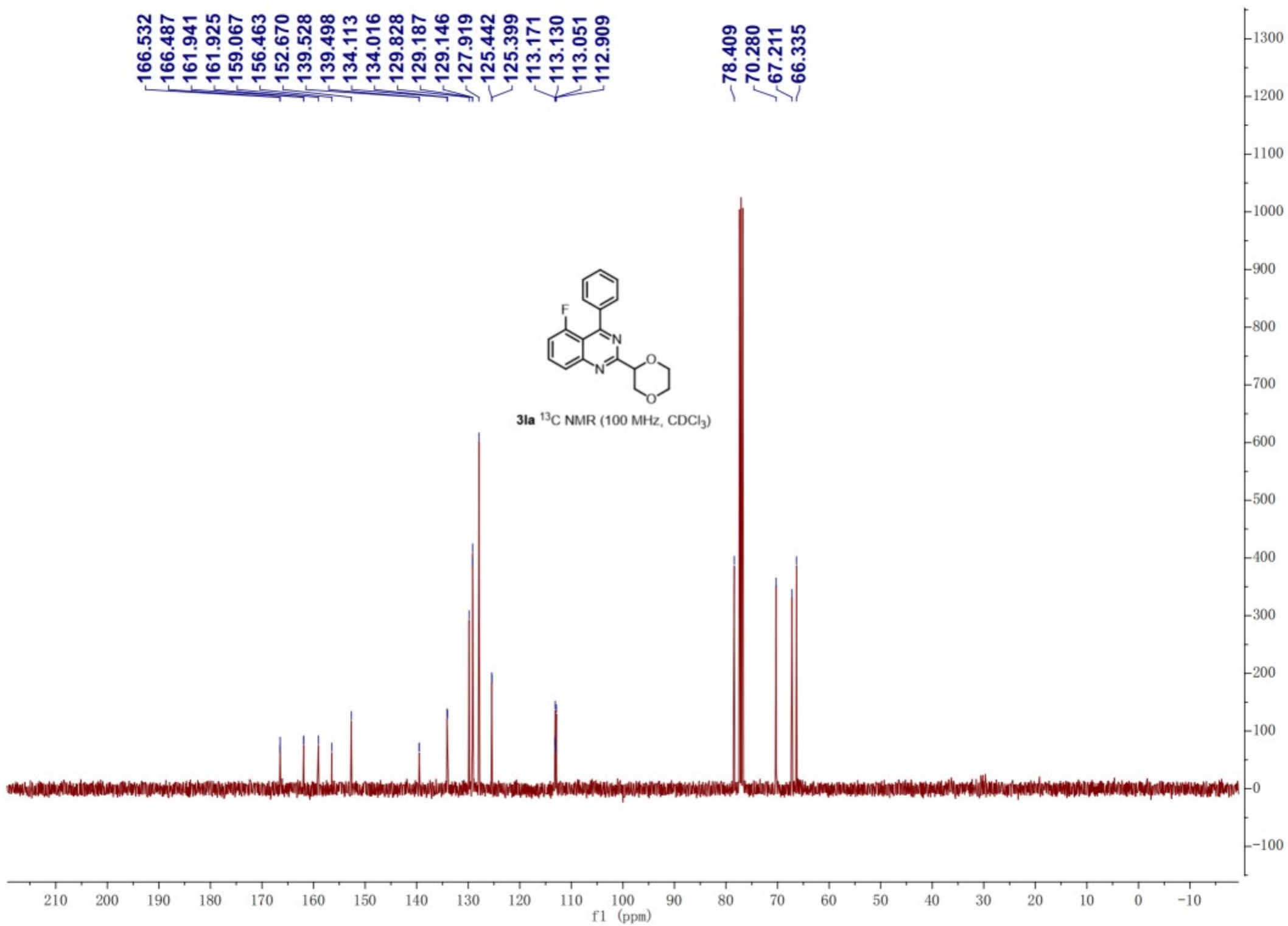


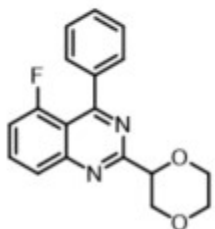
166.532  
166.487  
161.941  
161.925  
159.067  
156.463  
152.670  
139.528  
139.498  
134.113  
134.016  
129.828  
129.187  
129.146  
127.919  
125.442  
125.399  
113.171  
113.130  
113.051  
112.909

78.409  
70.280  
67.211  
66.335



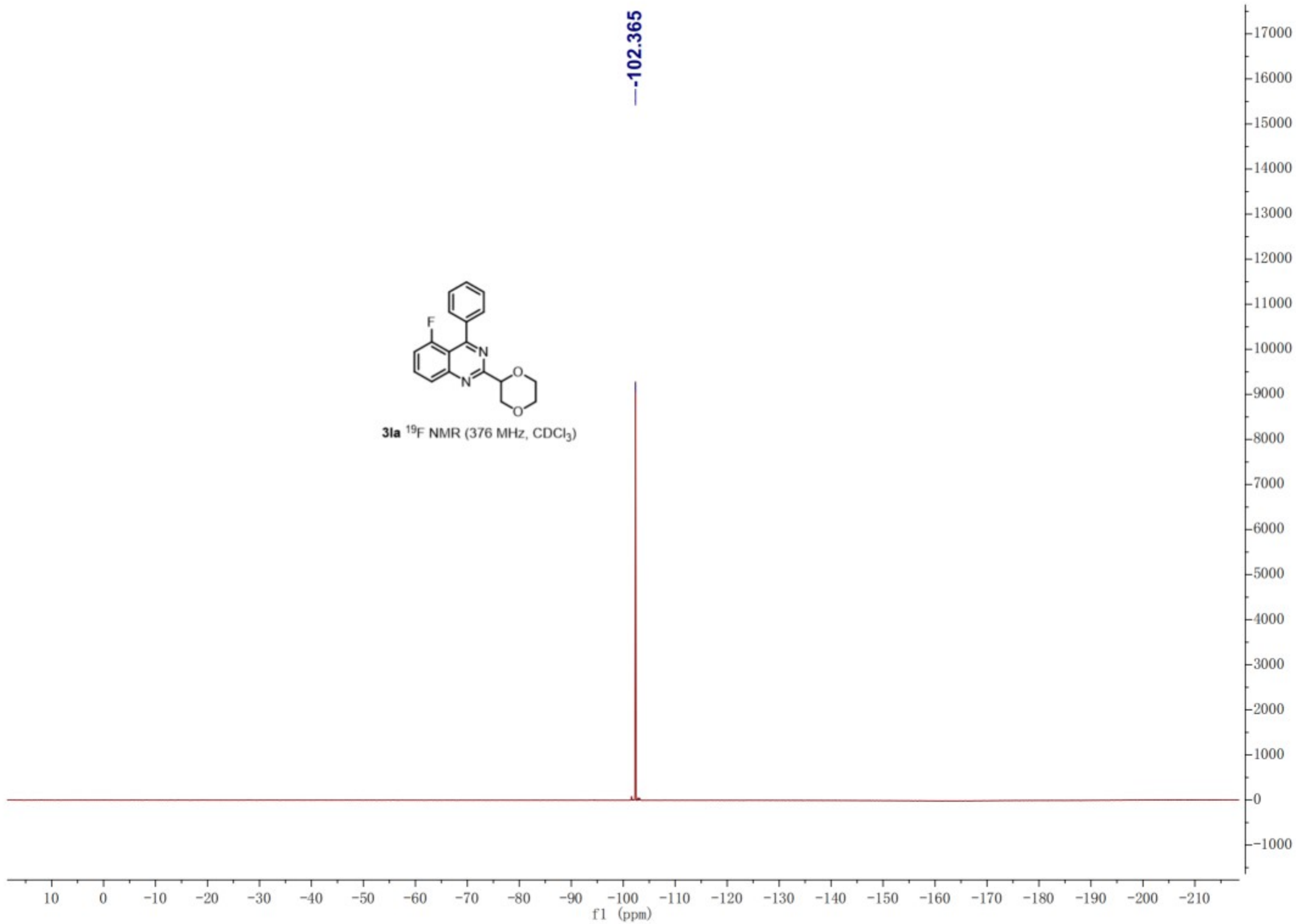
31a <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



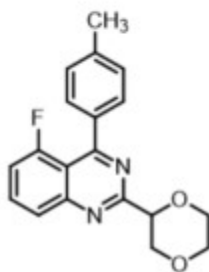


**31a**  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )

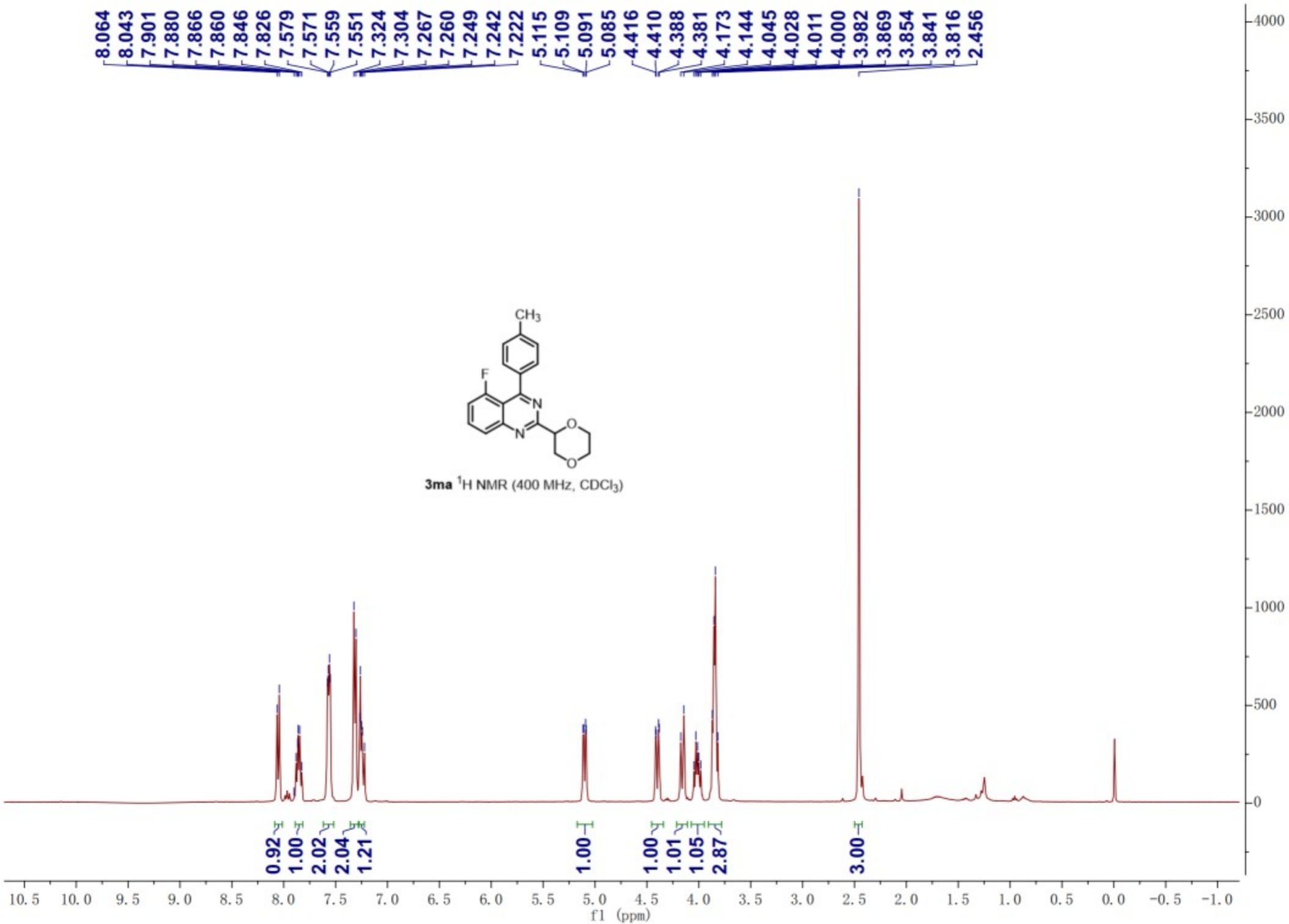
---102.365



8.064  
8.043  
7.901  
7.880  
7.866  
7.860  
7.846  
7.826  
7.579  
7.571  
7.559  
7.551  
7.324  
7.304  
7.267  
7.260  
7.249  
7.242  
7.222  
5.115  
5.109  
5.091  
5.085  
4.416  
4.410  
4.388  
4.381  
4.173  
4.144  
4.045  
4.028  
4.011  
4.000  
3.982  
3.869  
3.854  
3.841  
3.816  
2.456



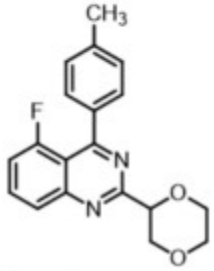
**3ma**  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )



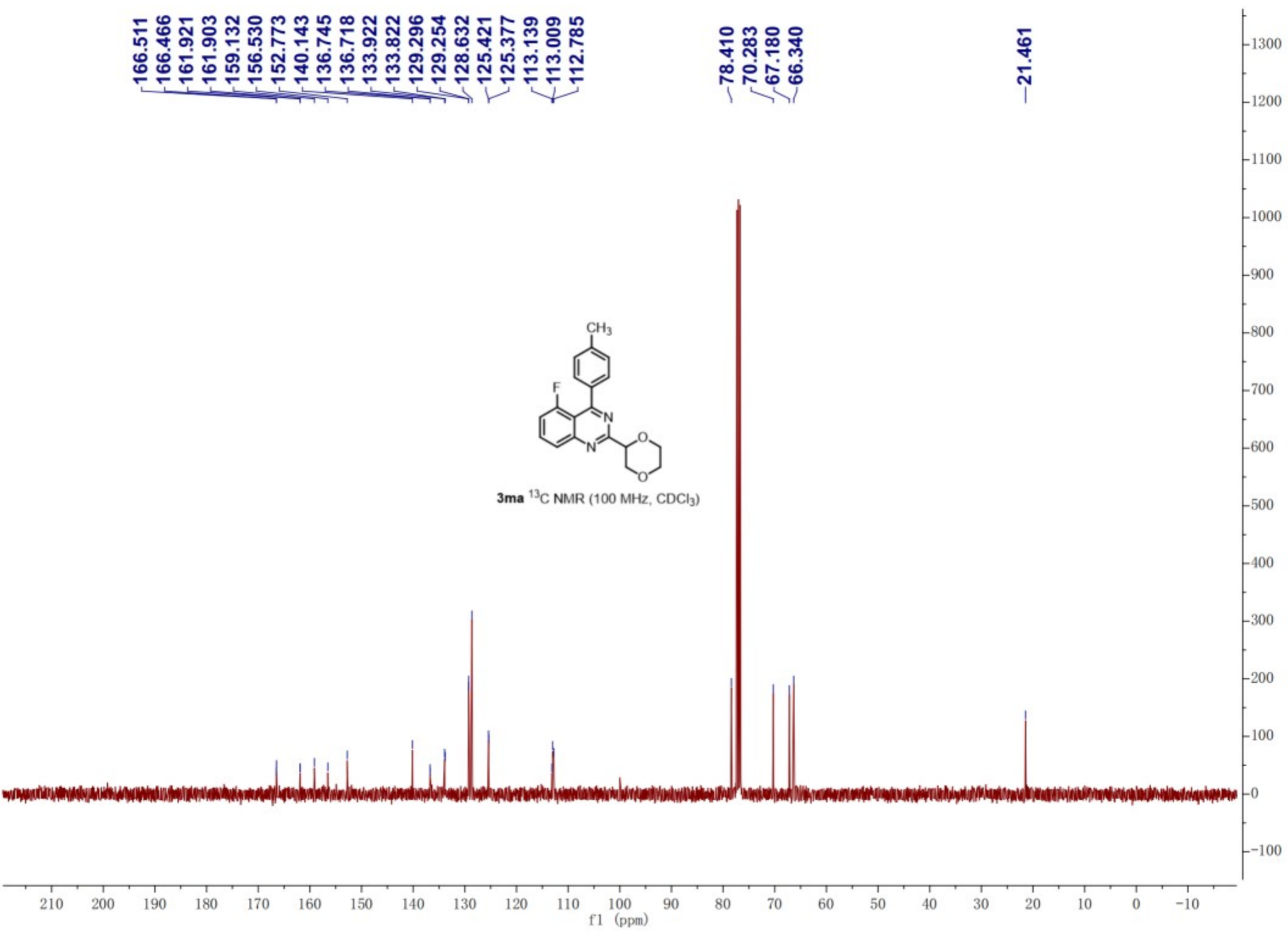
166.511  
166.466  
161.921  
161.903  
159.132  
156.530  
152.773  
140.143  
136.745  
136.718  
133.922  
133.822  
129.296  
129.254  
128.632  
125.421  
125.377  
113.139  
113.009  
112.785

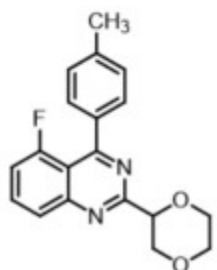
78.410  
70.283  
67.180  
66.340

21.461



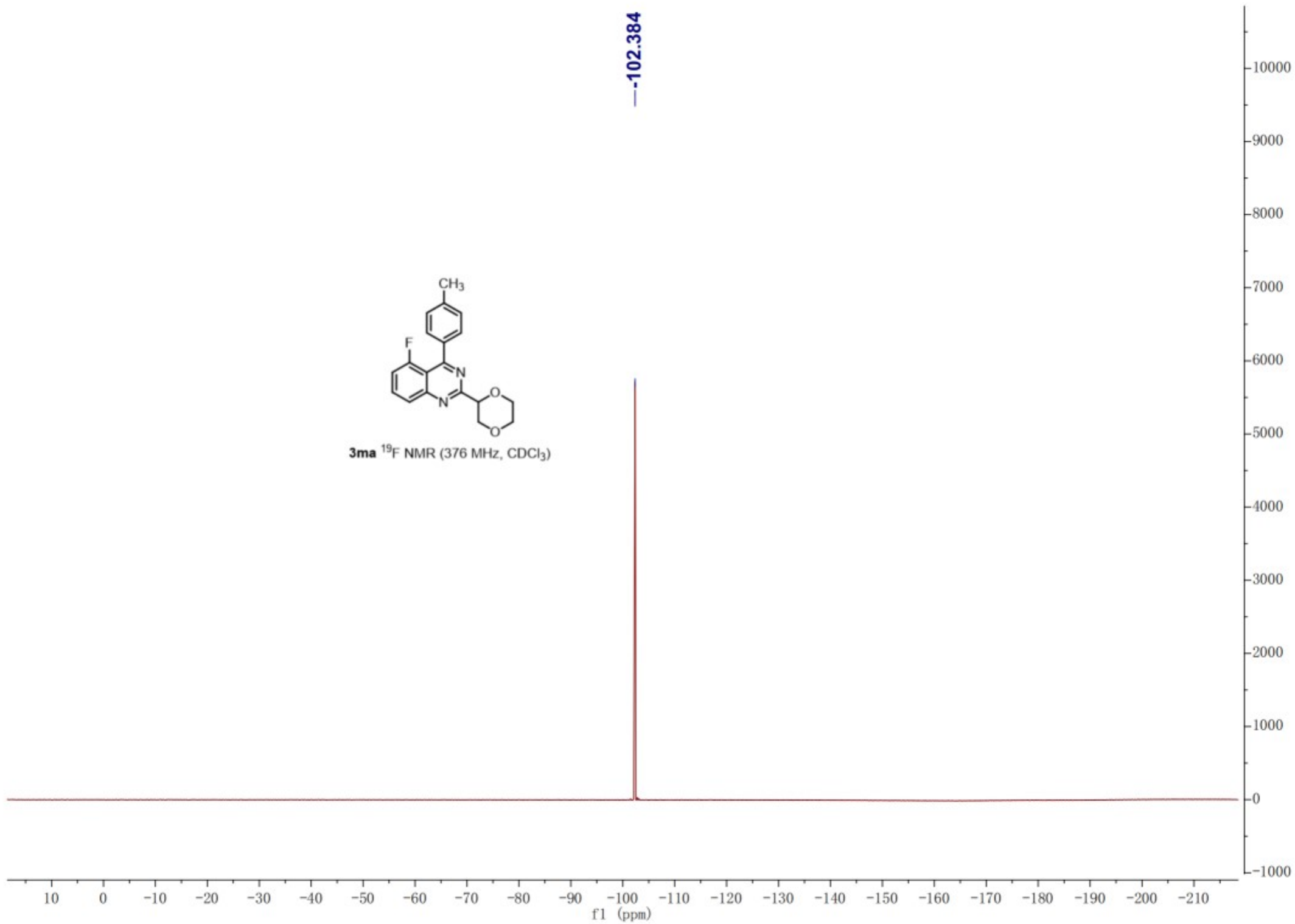
3ma <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)





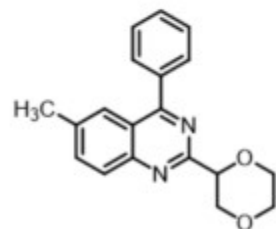
3ma  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )

---102.384



8.109  
8.087  
7.820  
7.748  
7.741  
7.727  
7.706  
7.559  
7.551  
7.545

5.105  
5.098  
5.080  
5.074  
4.399  
4.392  
4.370  
4.363  
4.149  
4.120  
4.041  
4.030  
4.017  
4.006  
3.987  
3.977  
3.905  
3.880  
3.858  
3.852  
3.835  
3.829  
3.809  
2.484



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

1.00  
1.03  
3.07  
3.04

1.00

1.03  
1.01  
1.06  
0.97  
2.16

3.02

10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0

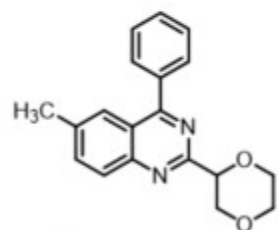
f1 (ppm)

6500  
6000  
5500  
5000  
4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0  
-500

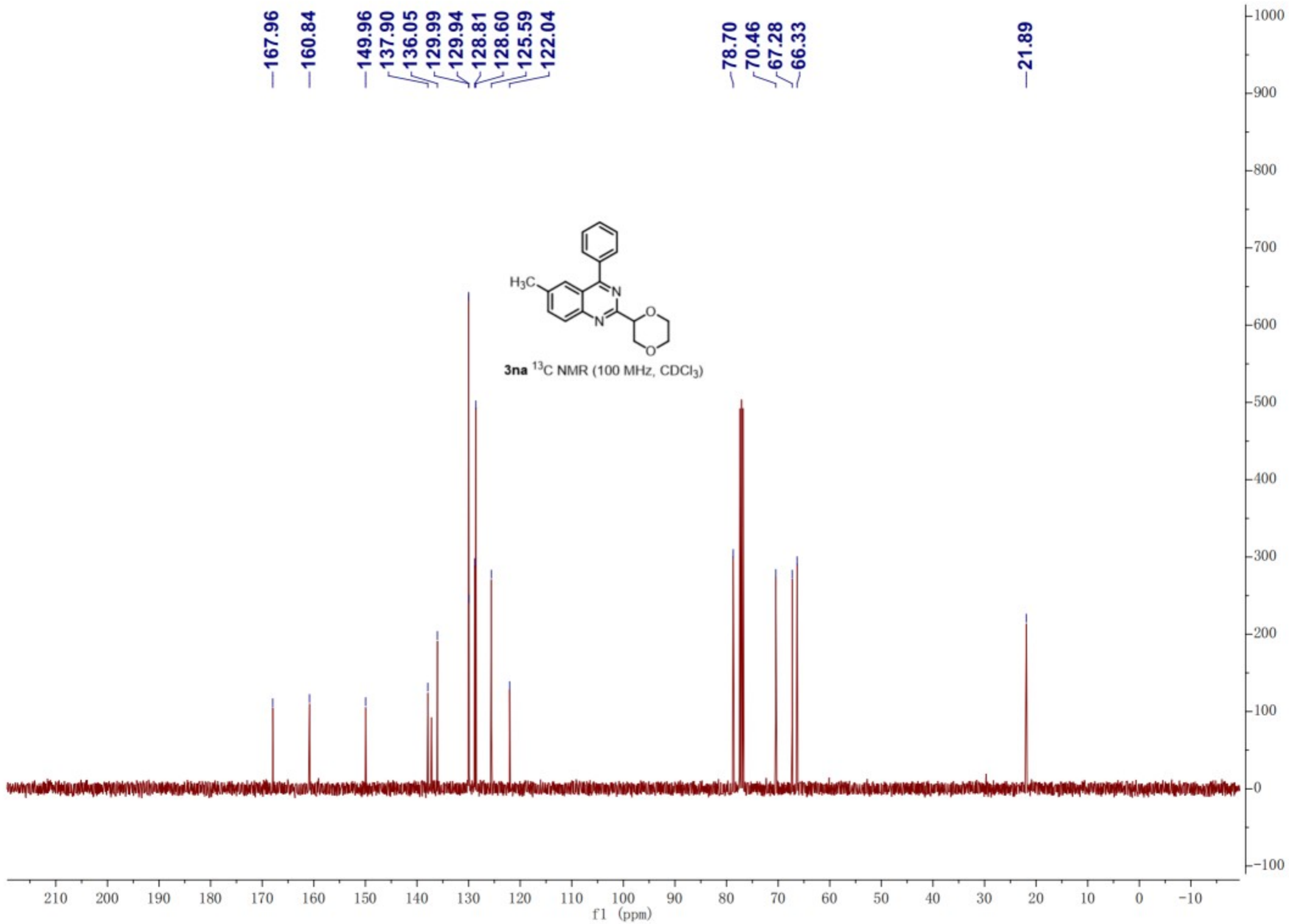
—167.96  
—160.84  
—149.96  
—137.90  
—136.05  
—129.99  
—129.94  
—128.81  
—128.60  
—125.59  
—122.04

—78.70  
—70.46  
—67.28  
—66.33

—21.89



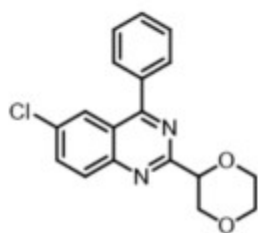
**3na**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )





8.172  
8.149  
8.067  
8.062  
7.846  
7.840  
7.823  
7.818  
7.751  
7.742  
7.733  
7.595  
7.589  
7.580

5.116  
5.110  
5.092  
5.085  
4.409  
4.402  
4.380  
4.374  
4.167  
4.138  
4.064  
4.051  
4.034  
4.016  
4.002  
3.987  
3.971  
3.895  
3.866  
3.862  
3.856  
3.841



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

1.00  
1.00  
1.00  
2.00  
3.05

1.00

1.00  
1.03  
1.11  
3.06

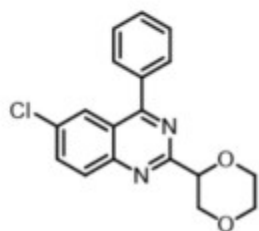
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0

f1 (ppm)

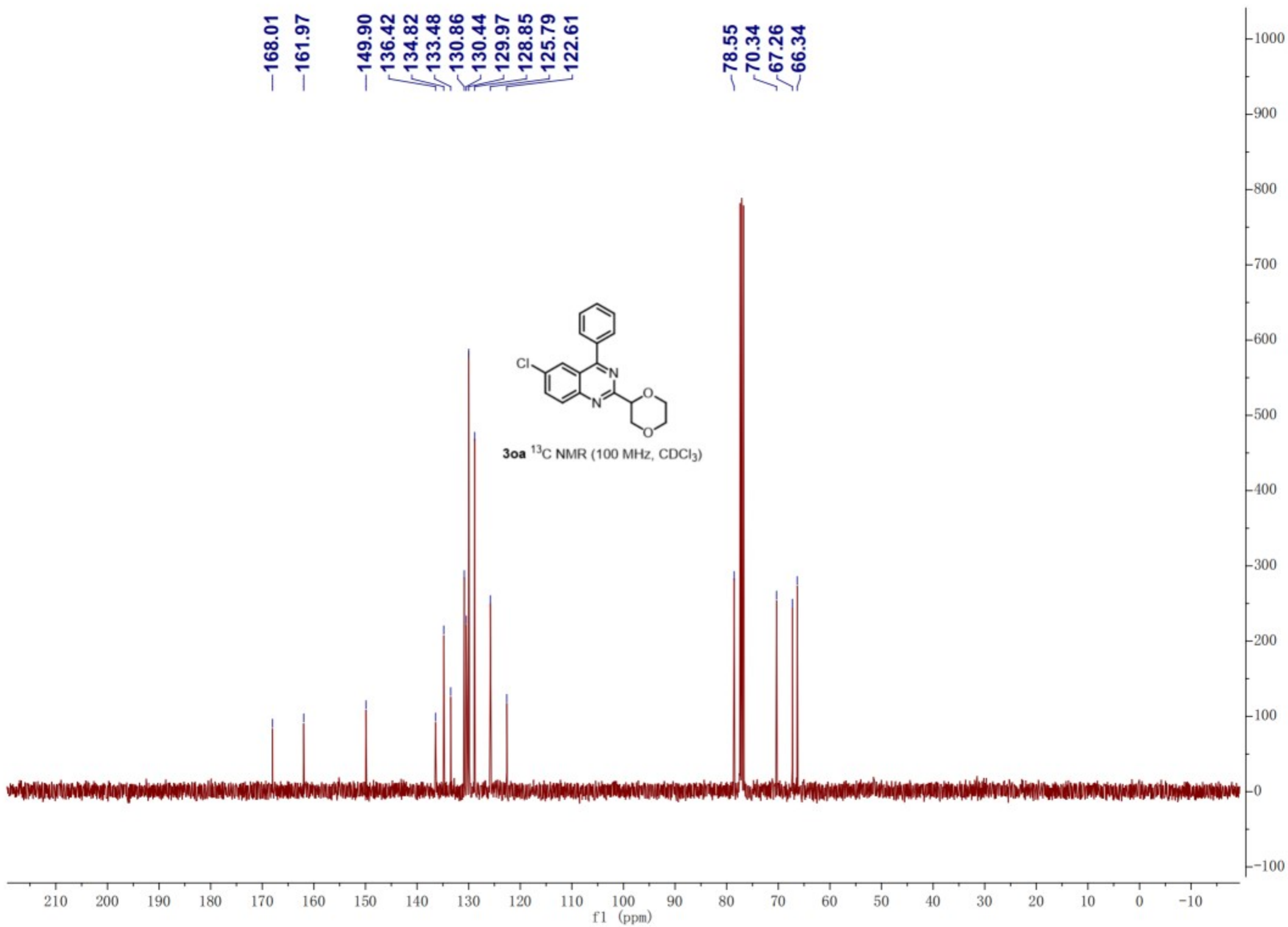
5000  
4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0



—168.01  
—161.97  
—149.90  
—136.42  
—134.82  
—133.48  
—130.86  
—130.44  
—129.97  
—128.85  
—125.79  
—122.61  
—78.55  
—70.34  
—67.26  
—66.34

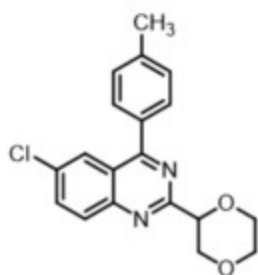


**3oa**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



8.154  
8.132  
8.088  
8.083  
7.824  
7.819  
7.802  
7.796  
7.662  
7.642  
7.394  
7.374

5.102  
5.095  
5.077  
5.071  
4.407  
4.401  
4.379  
4.372  
4.160  
4.131  
4.061  
4.057  
4.042  
4.025  
4.008  
3.998  
3.994  
3.978  
3.963  
3.874  
3.848  
3.833  
3.829  
3.821  
2.469



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

1.00  
0.98  
1.00  
2.00  
2.05

1.00

1.03

1.02

1.10

3.08

3.05

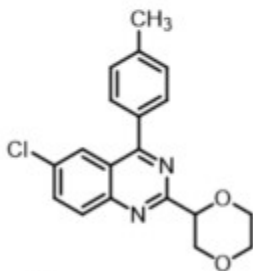
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0  
f1 (ppm)

4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0

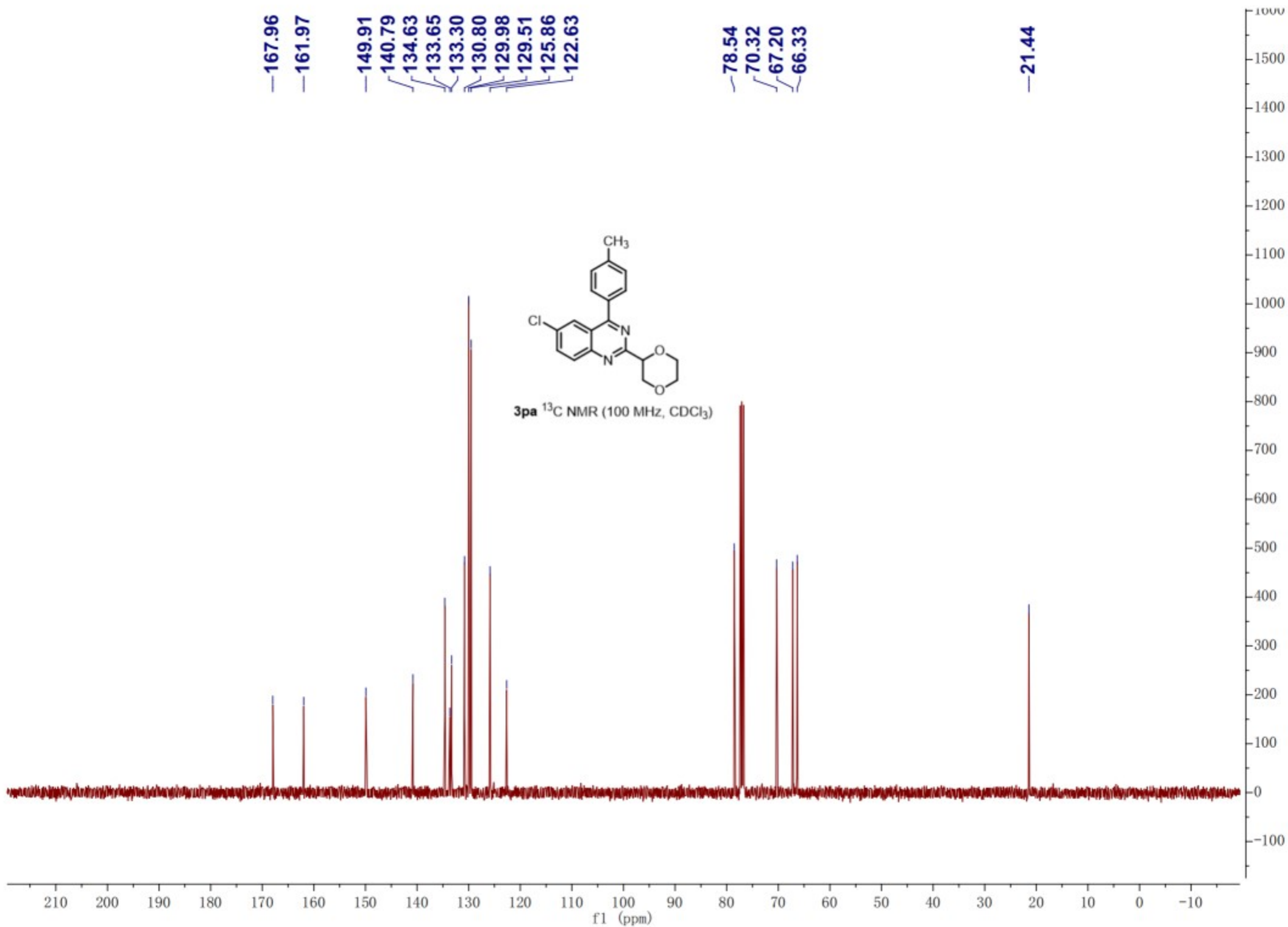
—167.96  
—161.97  
—149.91  
—140.79  
—134.63  
—133.65  
—133.30  
—130.80  
—129.98  
—129.51  
—125.86  
—122.63

—78.54  
—70.32  
—67.20  
—66.33

—21.44

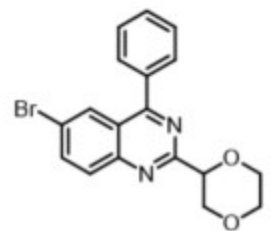


**3pa**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



8.234  
8.230  
8.098  
8.075  
7.972  
7.967  
7.949  
7.944  
7.753  
7.747  
7.738  
7.730  
7.594  
7.588  
7.579

5.106  
5.099  
5.081  
5.075  
4.406  
4.400  
4.378  
4.371  
4.164  
4.135  
4.059  
4.047  
4.030  
4.012  
3.999  
3.983  
3.968  
3.889  
3.859  
3.852  
3.837



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

1.00  
1.00  
1.02  
2.14  
3.19

1.04  
1.04  
1.02  
1.09  
3.10

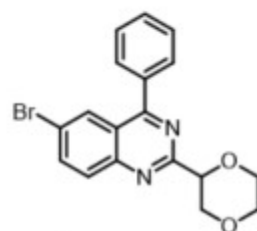
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0  
f1 (ppm)

6000  
5500  
5000  
4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0  
-500

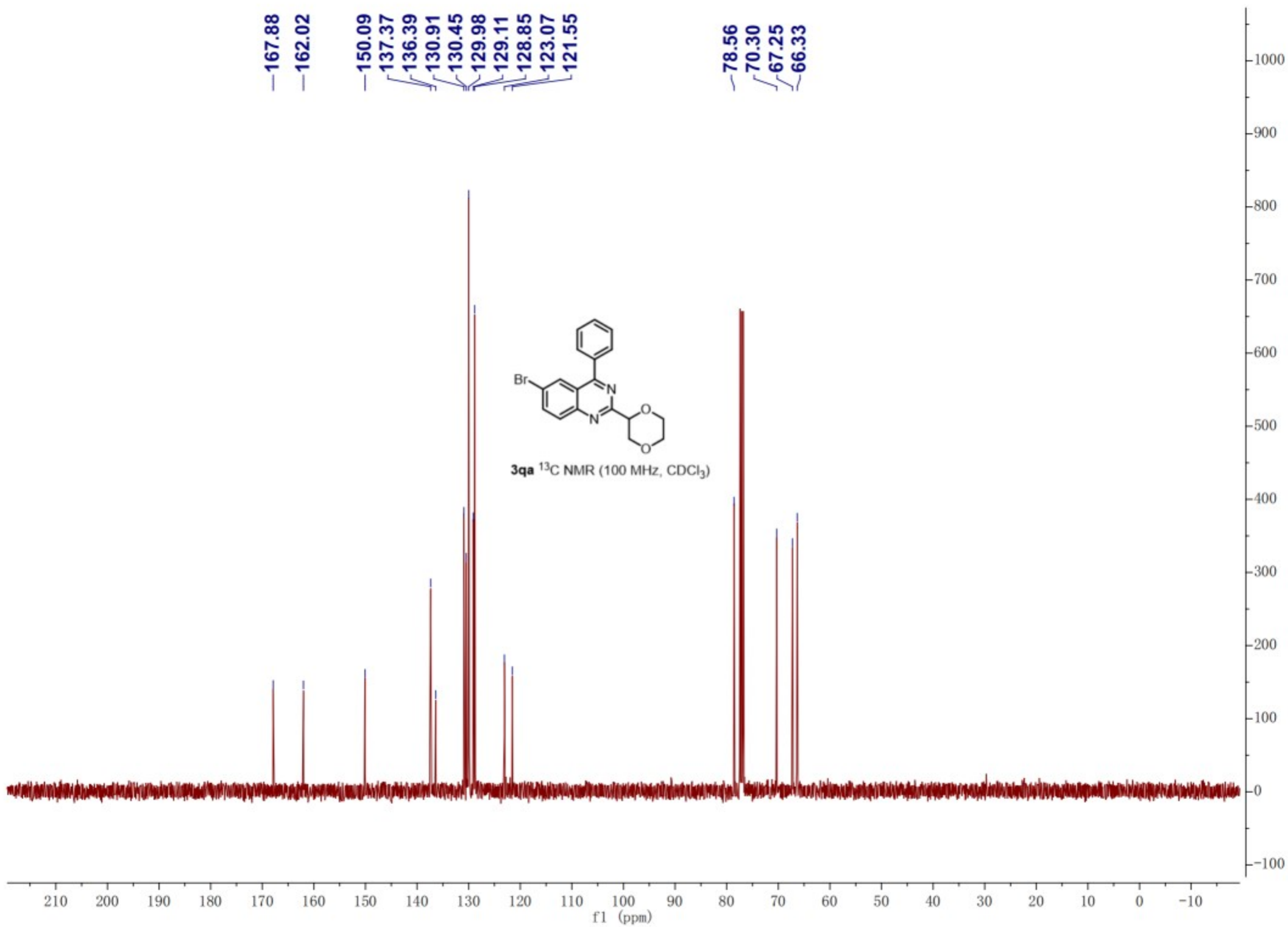
—167.88  
—162.02

—150.09  
—137.37  
—136.39  
—130.91  
—130.45  
—129.98  
—129.11  
—128.85  
—123.07  
—121.55

—78.56  
—70.30  
—67.25  
—66.33

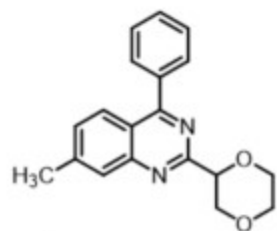


**3qa**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

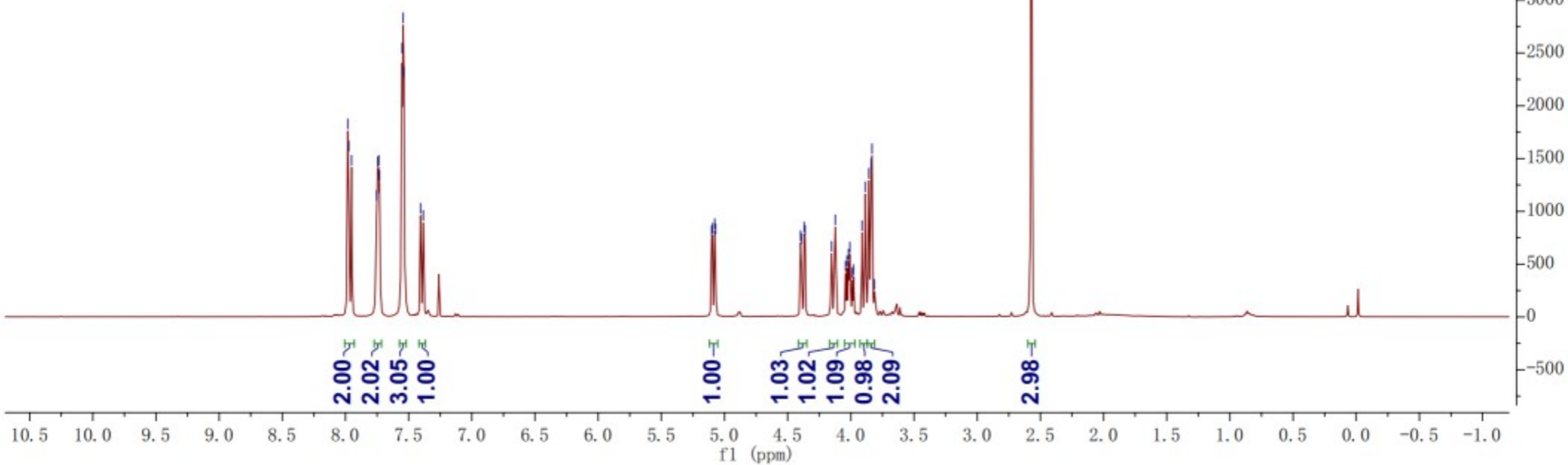


7.982  
7.974  
7.952  
7.754  
7.745  
7.736  
7.730  
7.552  
7.544  
7.536  
7.405  
7.384

5.102  
5.095  
5.078  
5.071  
4.398  
4.391  
4.369  
4.362  
4.152  
4.123  
4.042  
4.031  
4.019  
4.008  
3.989  
3.978  
3.911  
3.886  
3.857  
3.839  
3.832  
3.814  
2.571



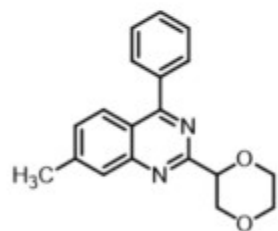
**3ra** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



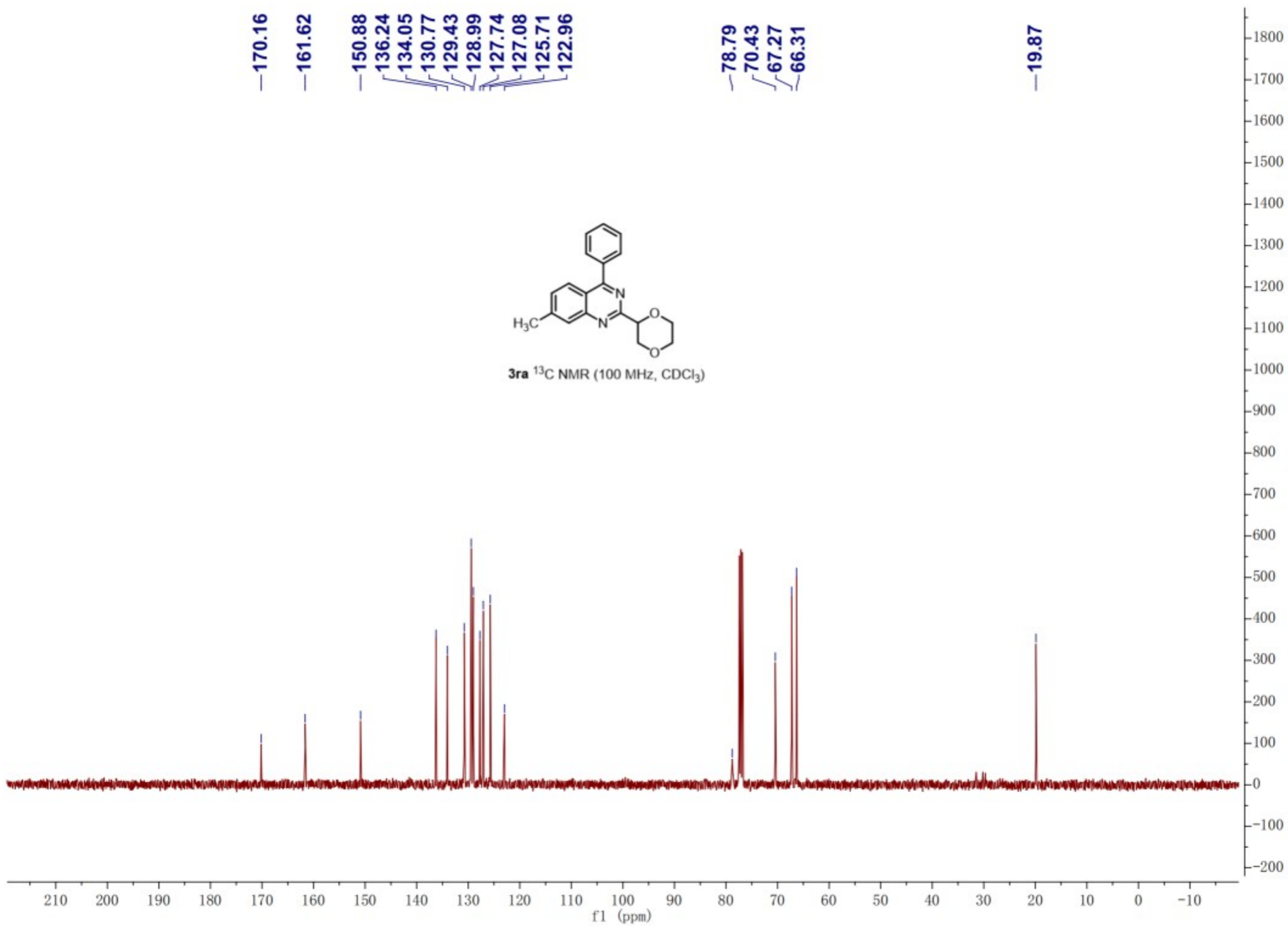
—170.16  
—161.62  
—150.88  
136.24  
134.05  
130.77  
129.43  
128.99  
127.74  
127.08  
125.71  
122.96

—78.79  
70.43  
67.27  
66.31

—19.87



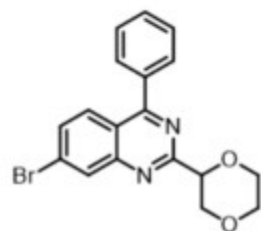
**3ra**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



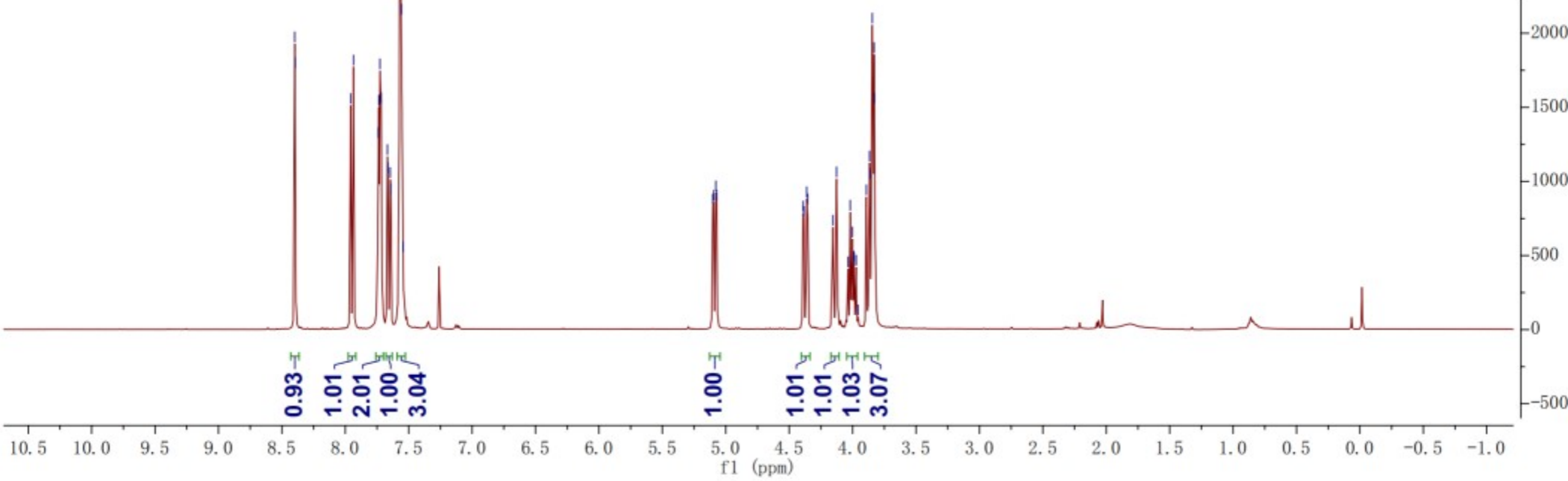


8.399  
8.395  
7.958  
7.935  
7.741  
7.736  
7.727  
7.717  
7.668  
7.664  
7.646  
7.641  
7.570  
7.565  
7.557  
7.545

5.103  
5.096  
5.078  
5.072  
4.390  
4.384  
4.362  
4.355  
4.156  
4.126  
4.036  
4.019  
4.002  
3.991  
3.988  
3.972  
3.958  
3.891  
3.867  
3.863  
3.845  
3.831  
3.827

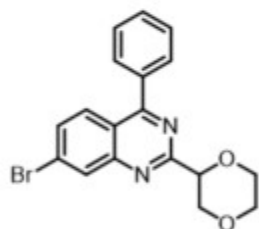


**3sa** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

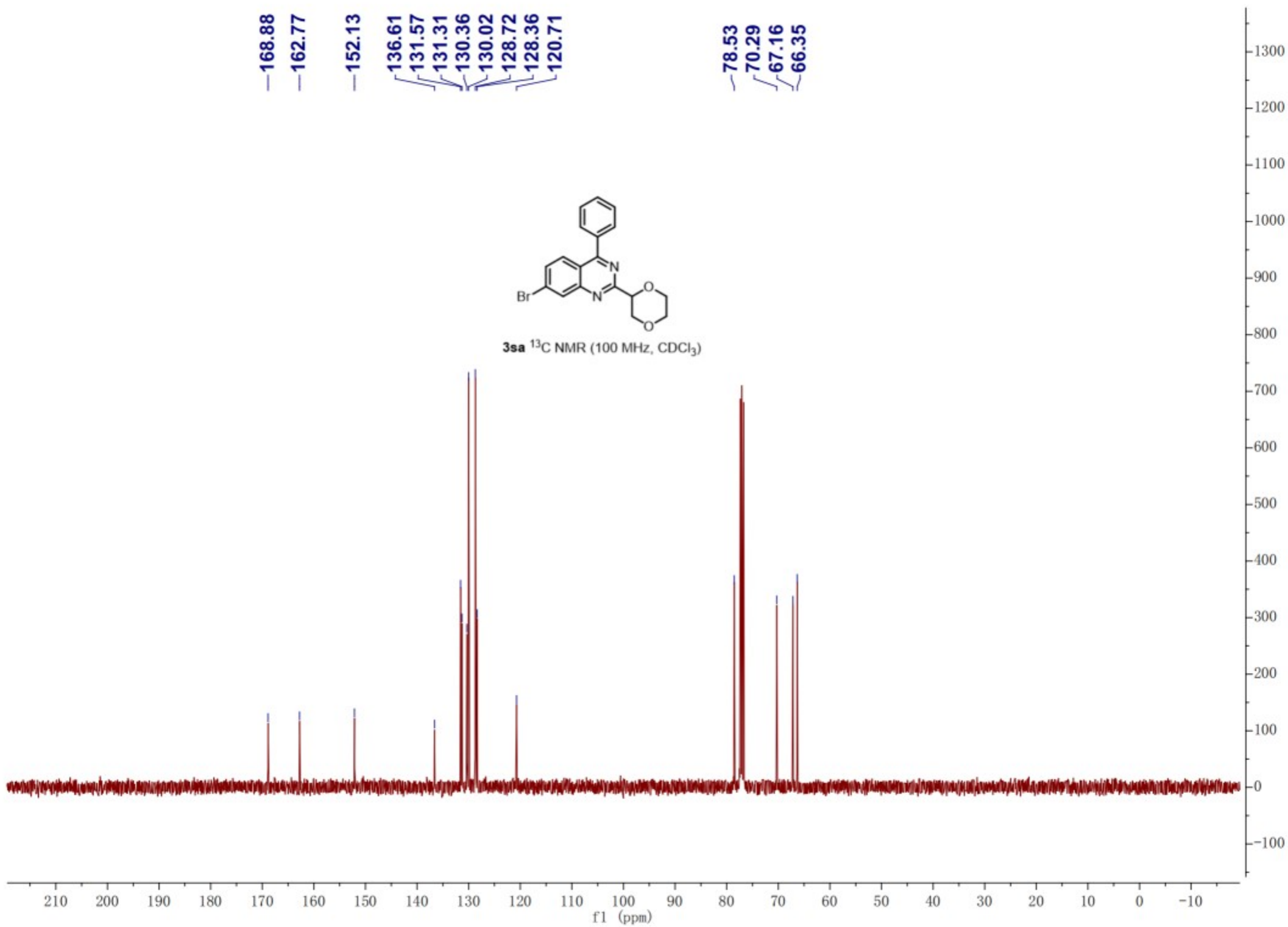




—168.88  
—162.77  
—152.13  
136.61  
131.57  
131.31  
130.36  
130.02  
128.72  
128.36  
120.71  
78.53  
70.29  
67.16  
66.35

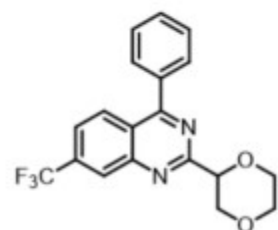


**3sa**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )



8.543  
8.255  
8.233  
7.780  
7.767  
7.744  
7.600  
7.589

5.146  
5.140  
5.122  
5.116  
4.415  
4.410  
4.387  
4.382  
4.179  
4.150  
4.057  
4.040  
4.024  
4.011  
3.994  
3.980  
3.926  
3.899  
3.861  
3.846



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

0.92

1.00

3.01

3.03

0.98

0.99

1.01

1.09

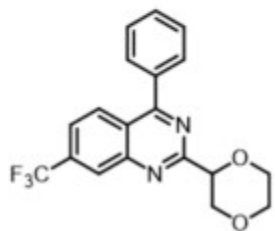
0.87

2.21

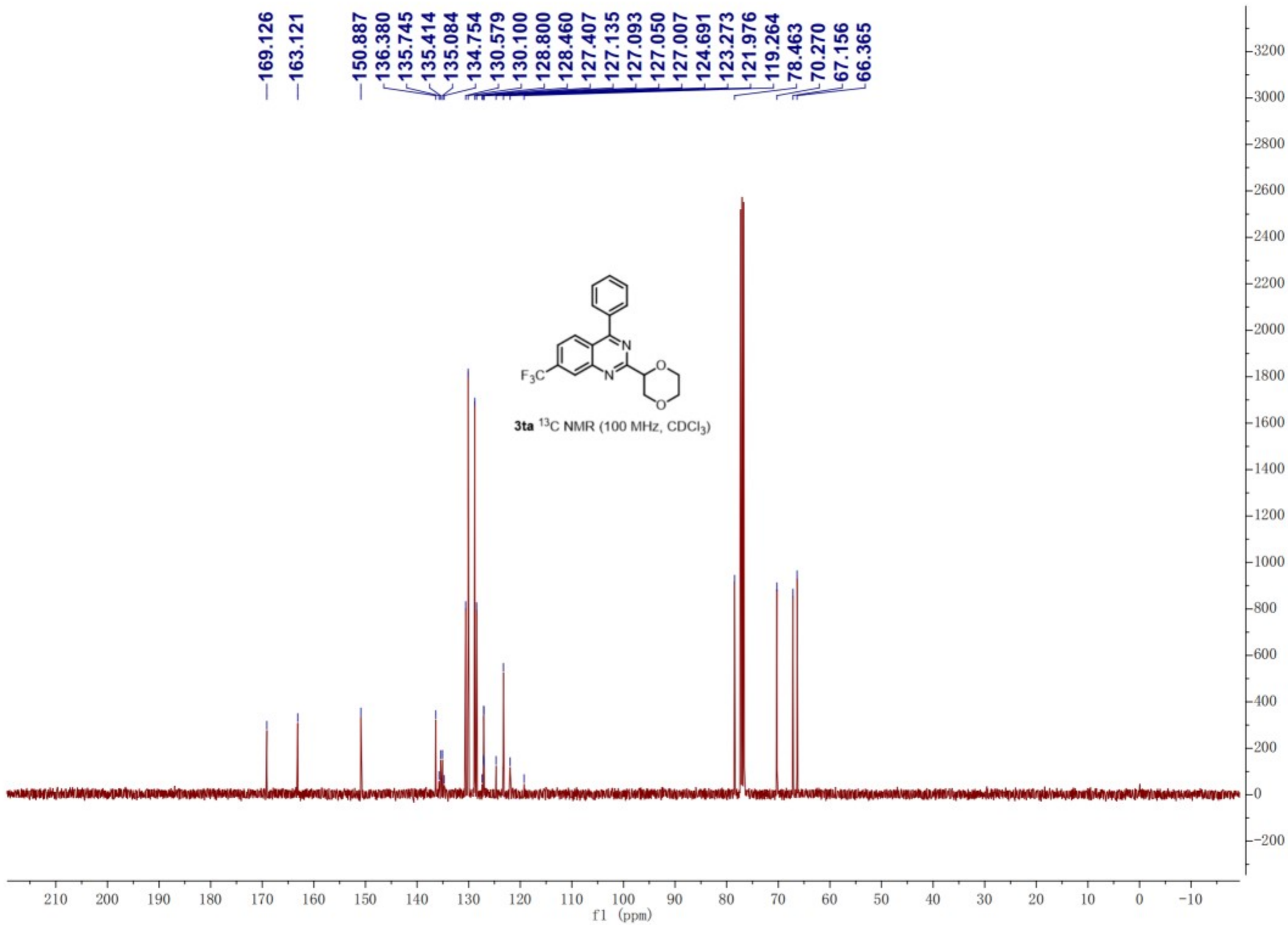
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0  
f1 (ppm)

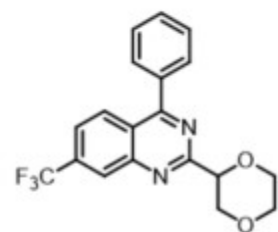
4500  
4000  
3500  
3000  
2500  
2000  
1500  
1000  
500  
0

—169.126  
—163.121  
—150.887  
—136.380  
—135.745  
—135.414  
—135.084  
—134.754  
—130.579  
—130.100  
—128.800  
—128.460  
—127.407  
—127.135  
—127.093  
—127.050  
—127.007  
—124.691  
—123.273  
—121.976  
—119.264  
—78.463  
—70.270  
—67.156  
—66.365



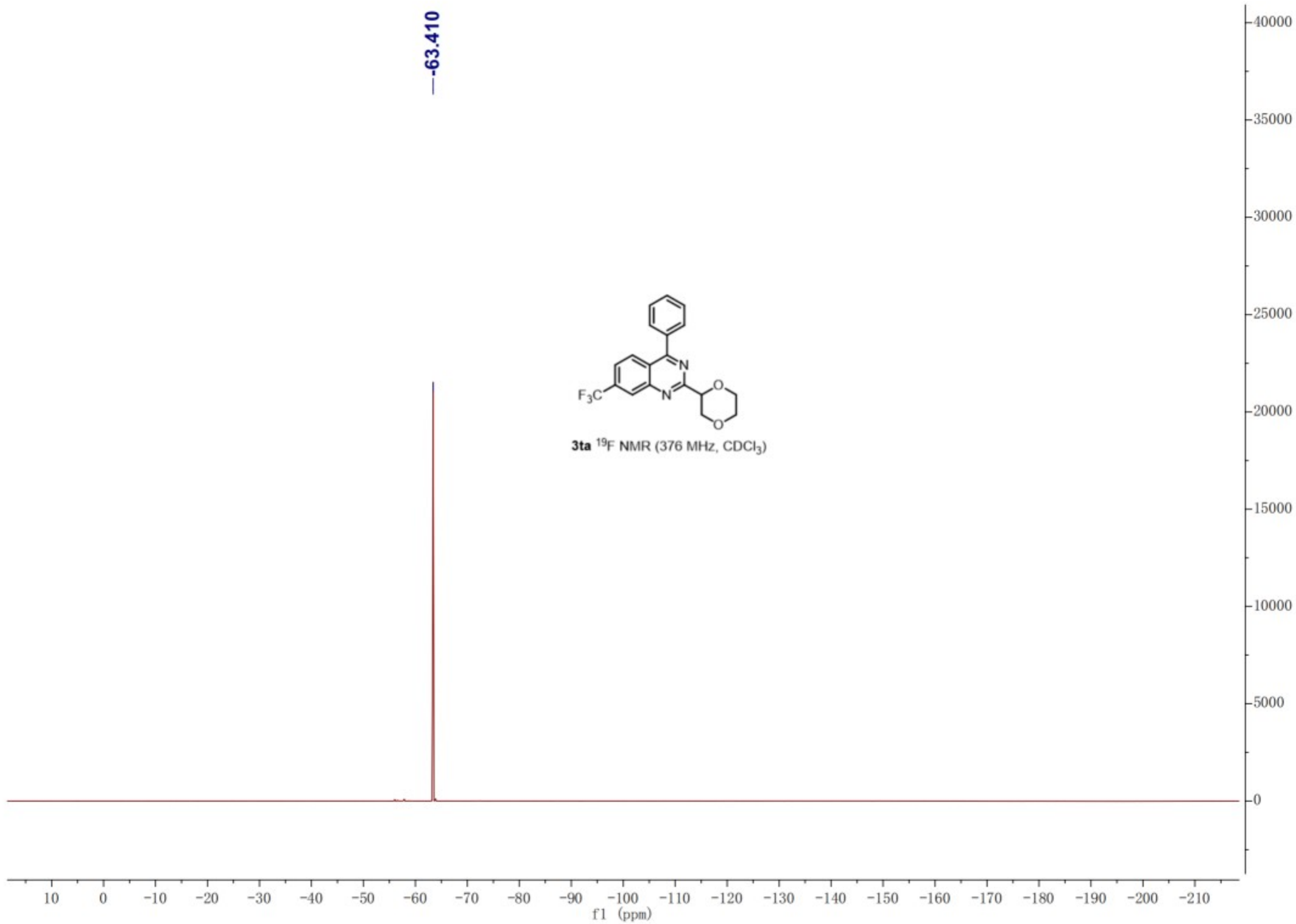
**3ta** <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

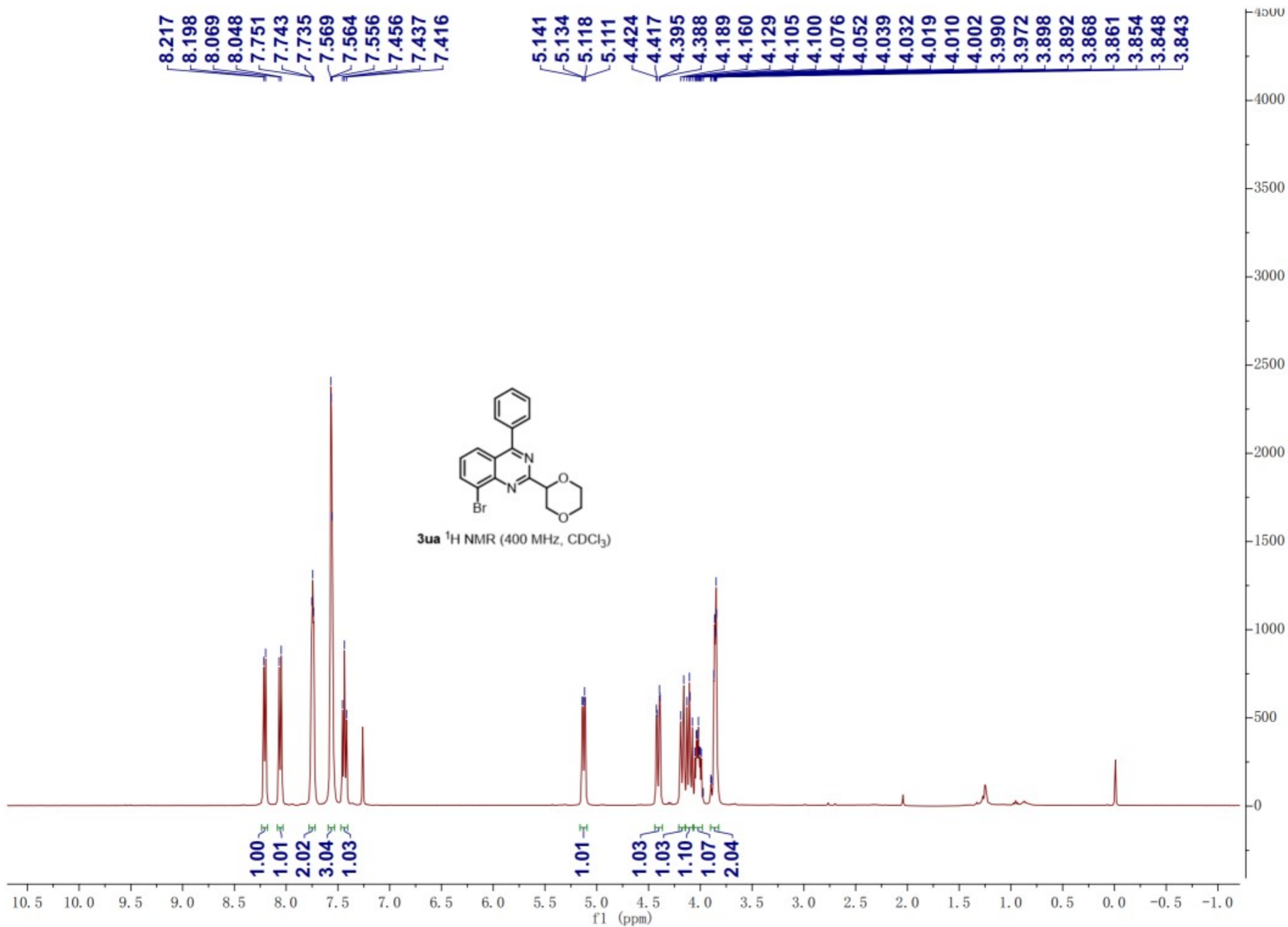




**3ta**  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )

—63.410





—169.76

—162.52

—148.75

137.24

136.77

130.23

130.18

128.63

127.89

126.82

124.63

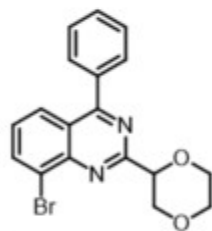
123.46

78.17

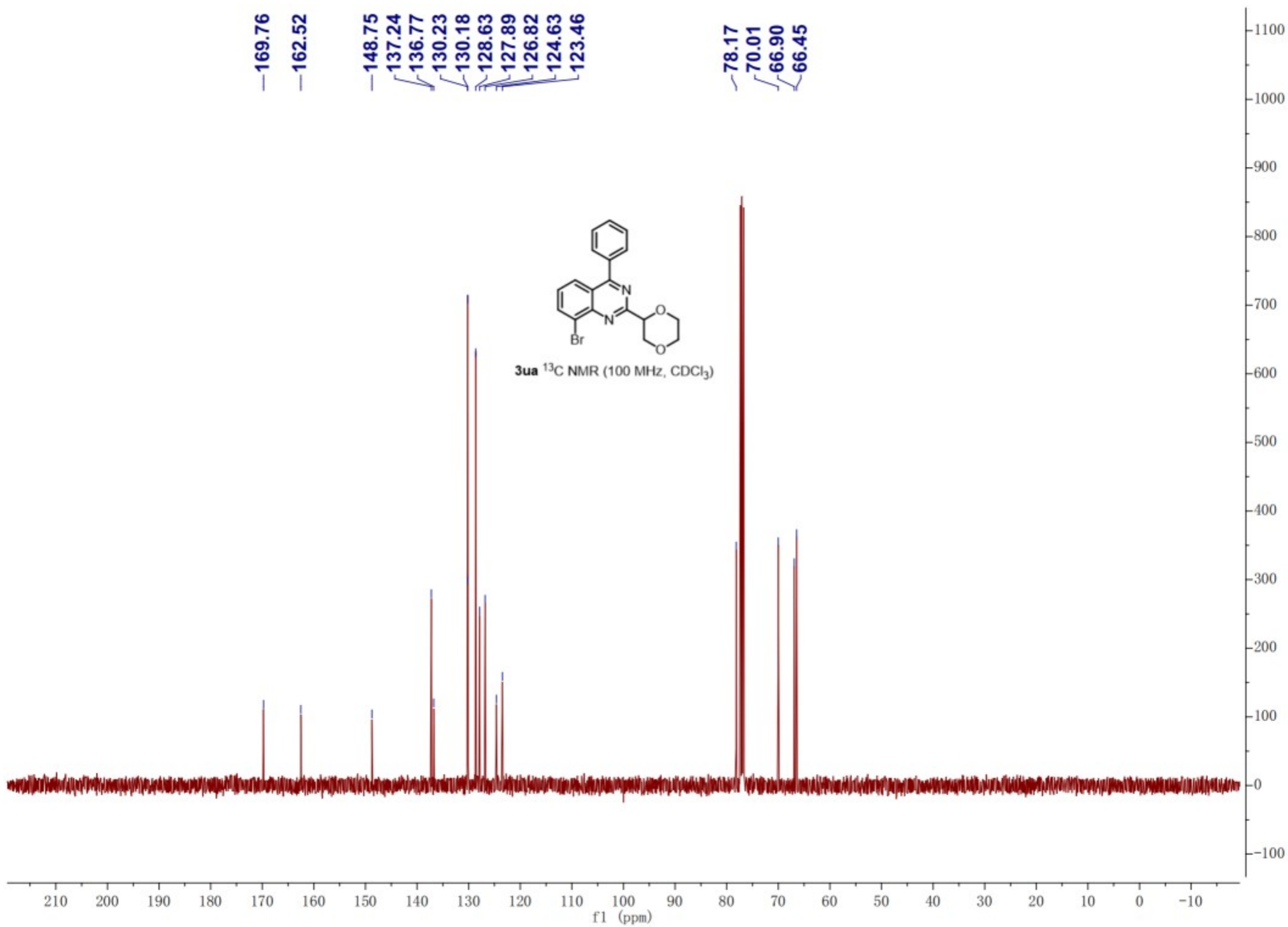
70.01

66.90

66.45

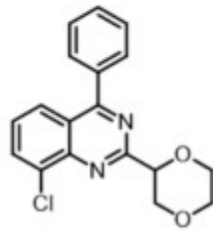


**3ua**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

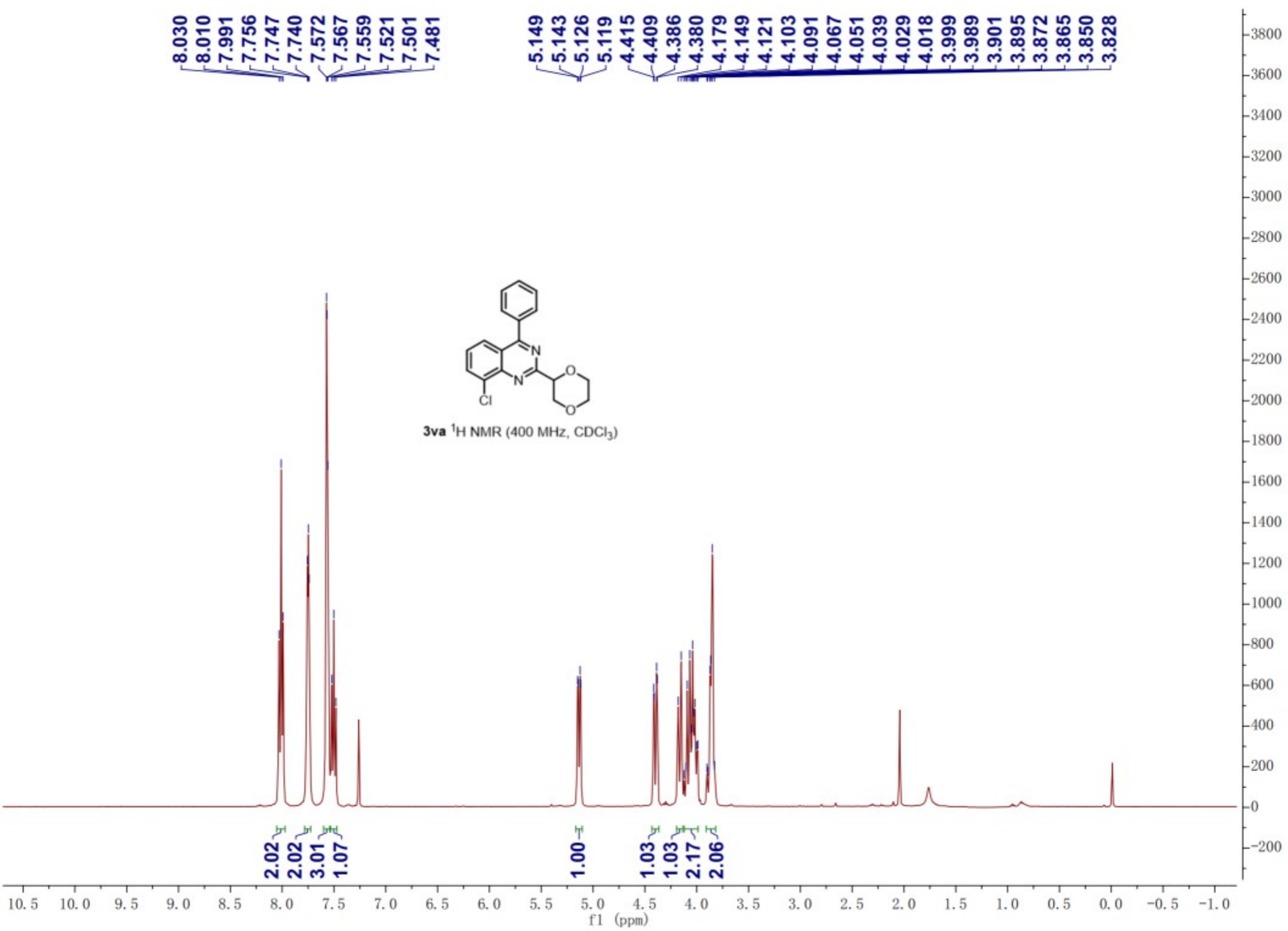


8.030  
8.010  
7.991  
7.756  
7.747  
7.740  
7.572  
7.567  
7.559  
7.521  
7.501  
7.481

5.149  
5.143  
5.126  
5.119  
4.415  
4.409  
4.386  
4.380  
4.179  
4.149  
4.121  
4.103  
4.091  
4.067  
4.051  
4.039  
4.029  
4.018  
3.999  
3.989  
3.901  
3.895  
3.872  
3.865  
3.850  
3.828



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





169.58

162.33

147.98

136.83

133.63

130.26

130.16

128.63

127.32

126.06

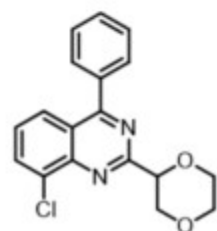
123.40

78.34

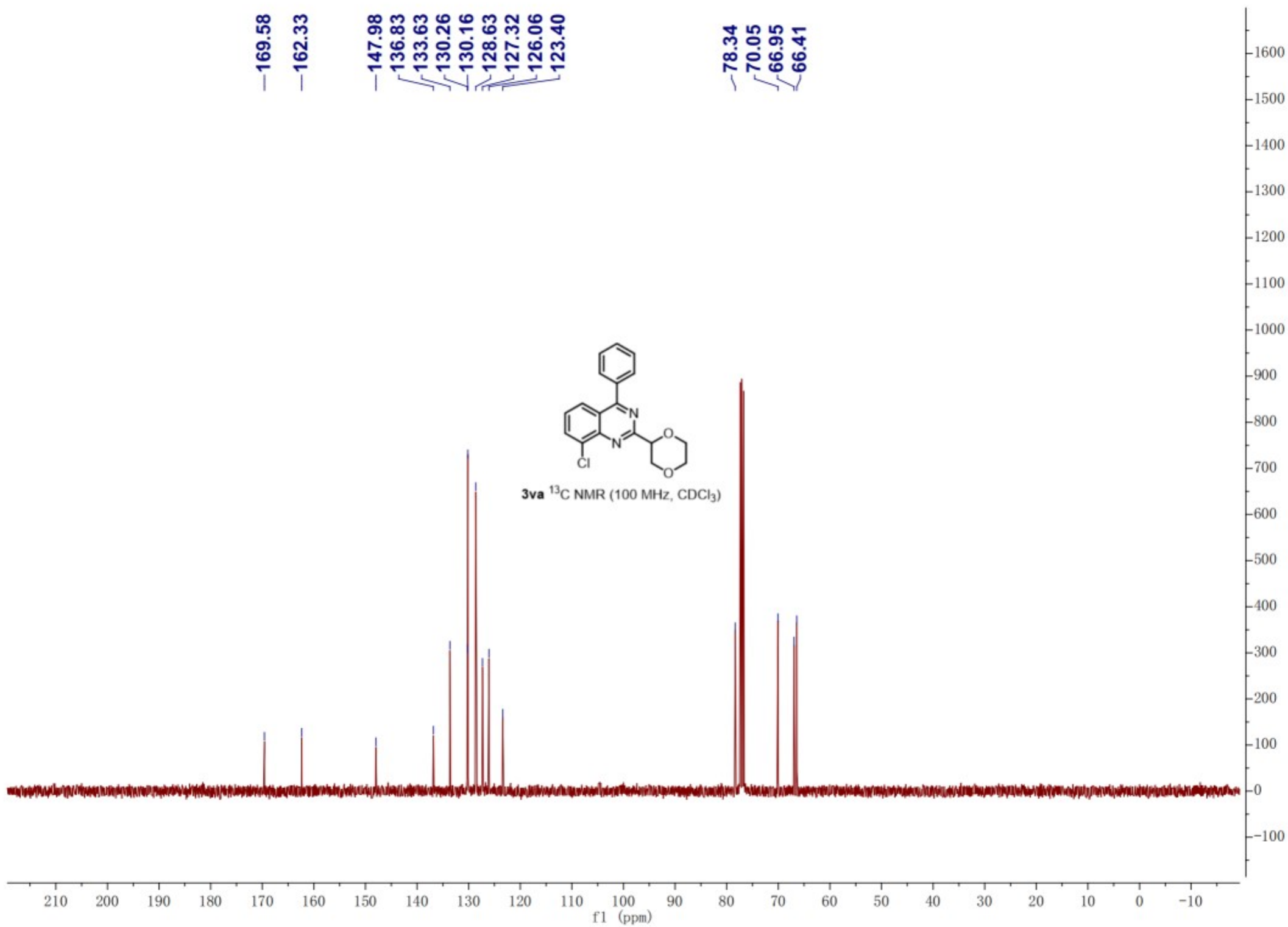
70.05

66.95

66.41



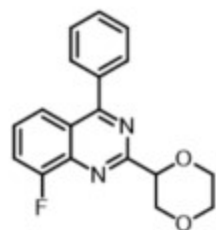
3va <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



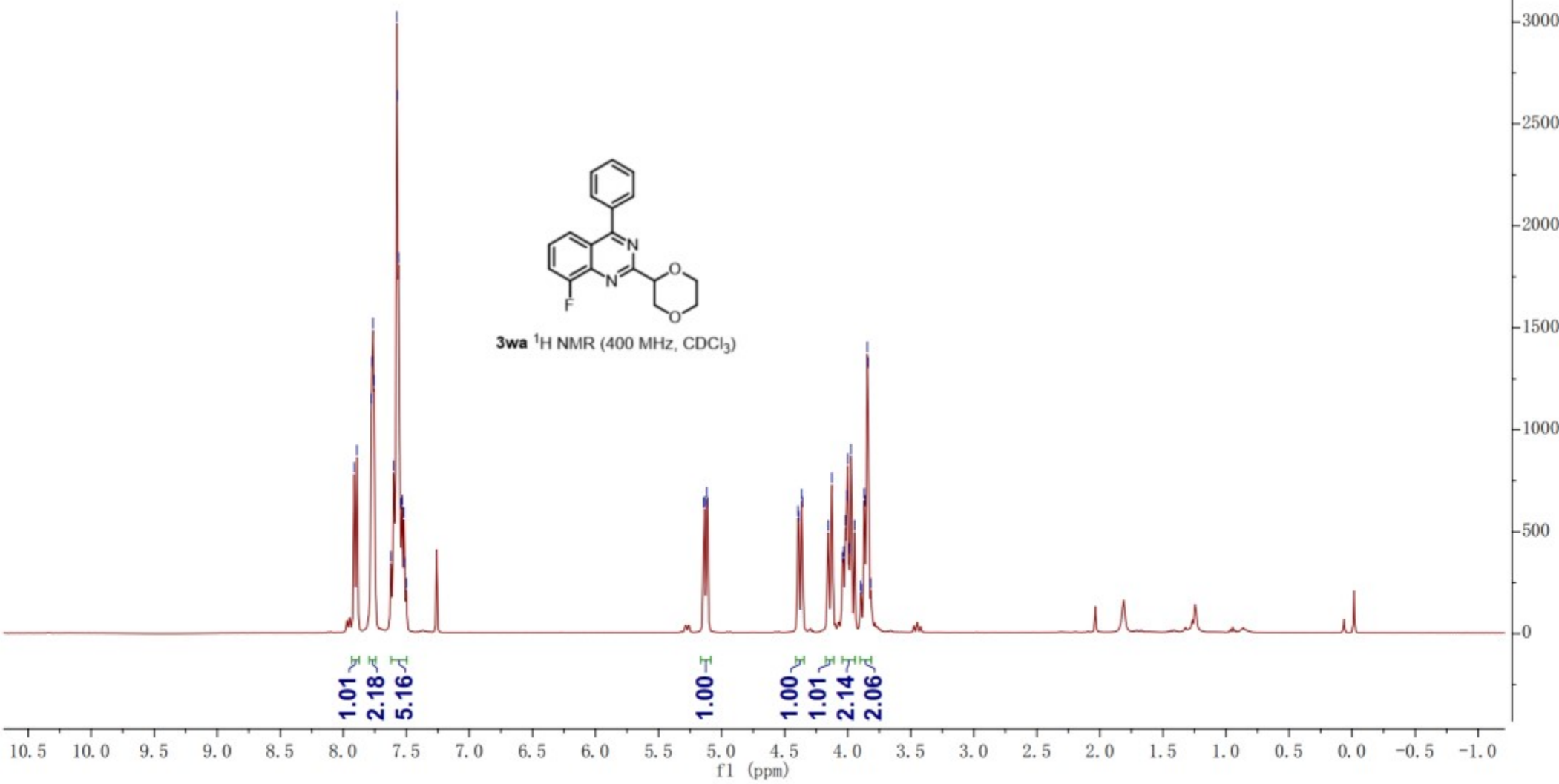


7.913  
7.892  
7.778  
7.772  
7.764  
7.756  
7.623  
7.601  
7.576  
7.571  
7.562  
7.540  
7.532  
7.520  
7.512  
7.500

5.142  
5.135  
5.118  
5.111  
4.394  
4.388  
4.366  
4.359  
4.155  
4.126  
4.042  
4.031  
4.018  
4.007  
3.999  
3.988  
3.974  
3.946  
3.897  
3.891  
3.868  
3.862  
3.845  
3.838  
3.818

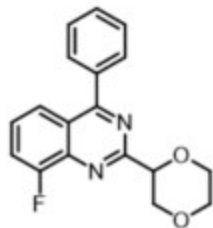


**3wa** <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

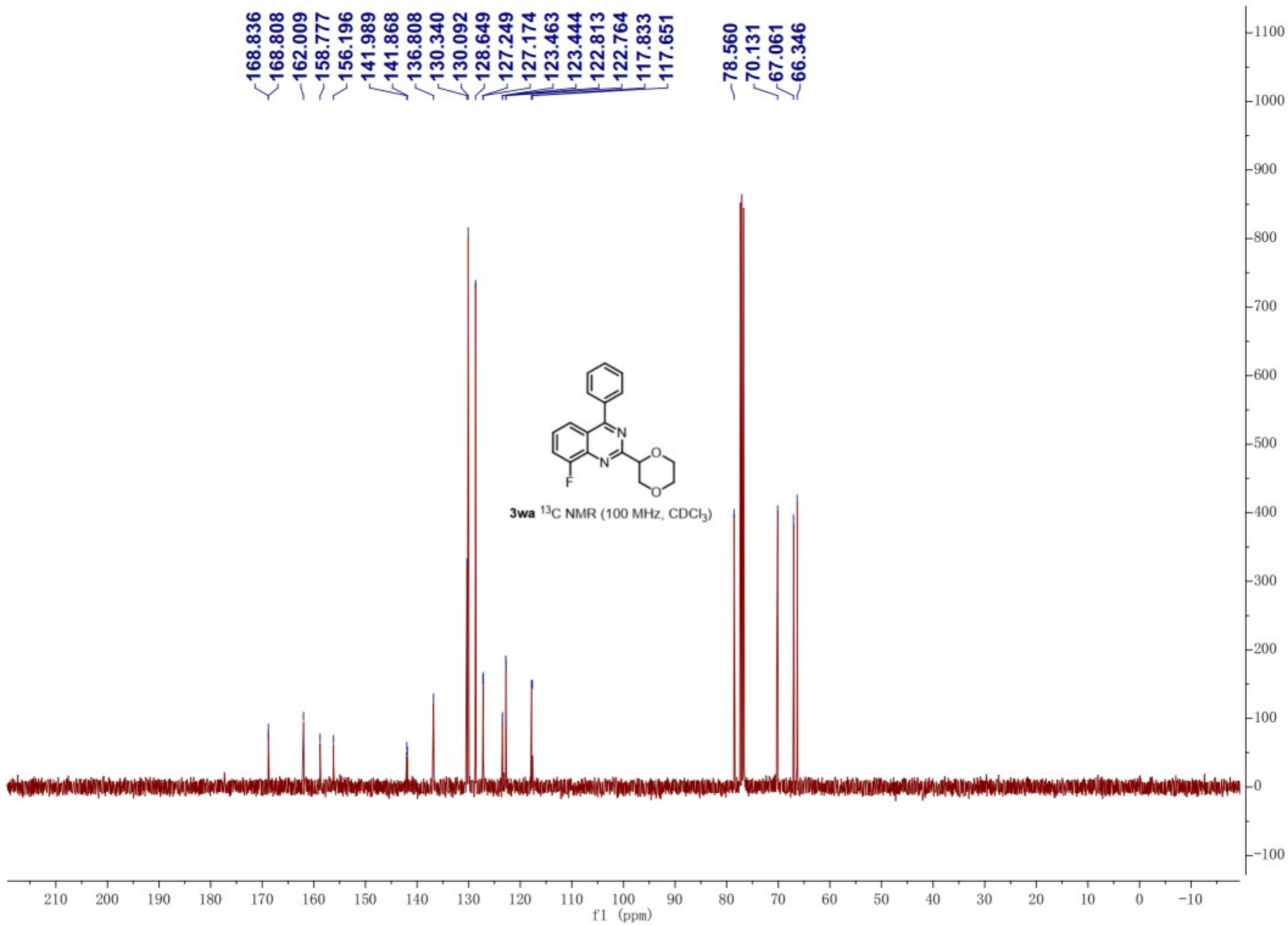


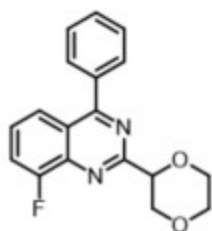
168.836  
168.808  
162.009  
158.777  
156.196  
141.989  
141.868  
136.808  
130.340  
130.092  
128.649  
127.249  
127.174  
123.463  
123.444  
122.813  
122.764  
117.833  
117.651

78.560  
70.131  
67.061  
66.346

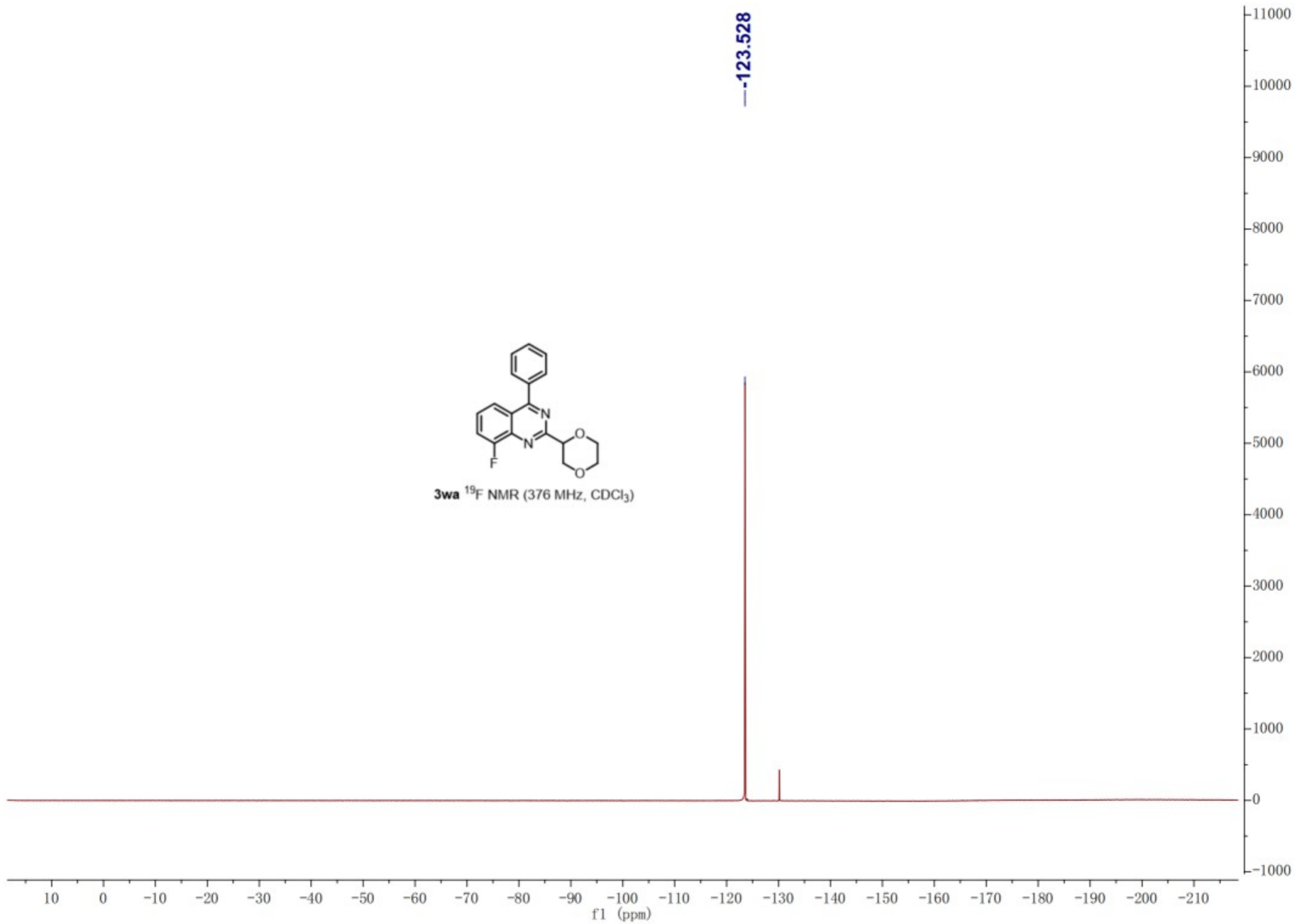


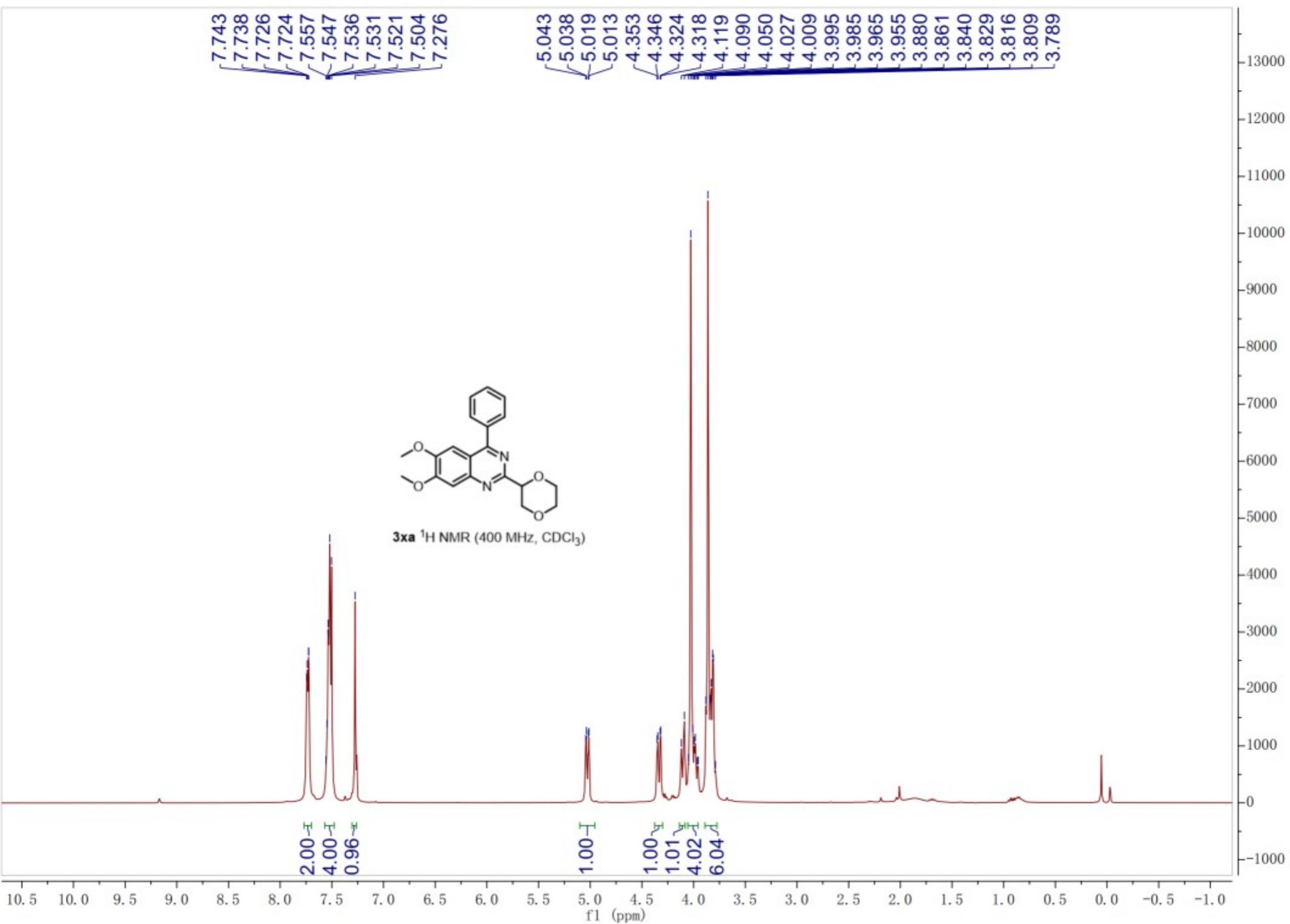
**3wa**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

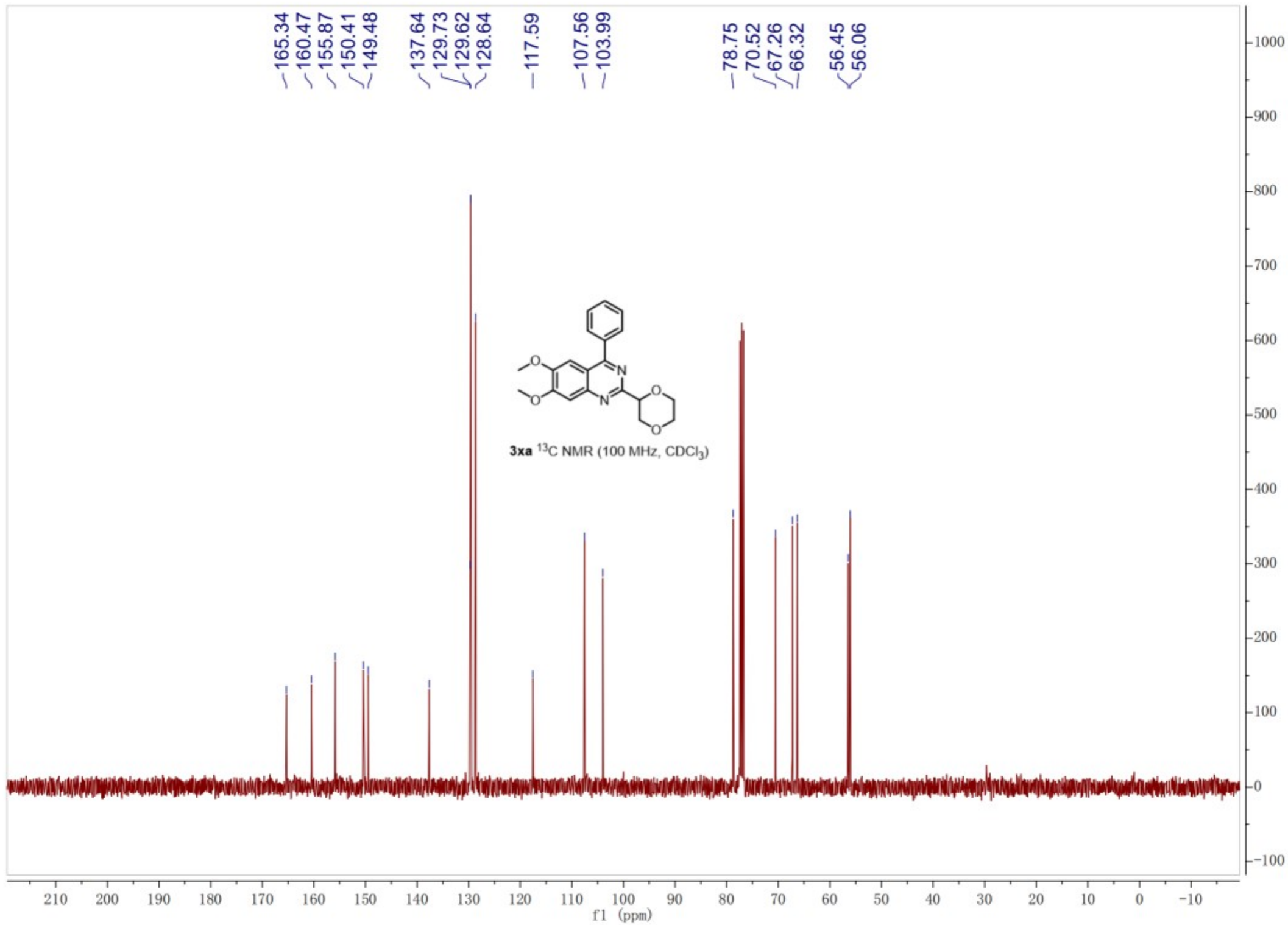


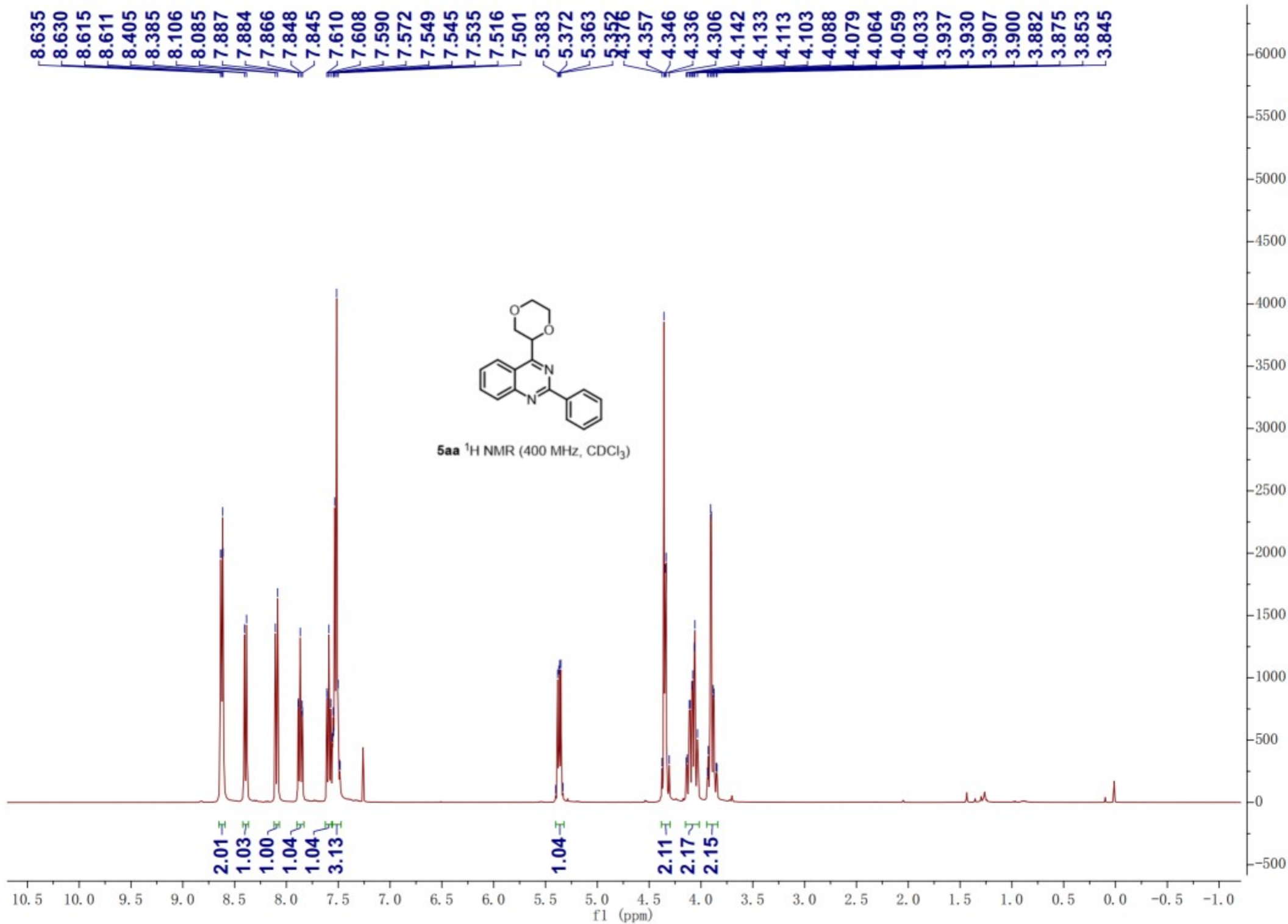


**3wa**  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )



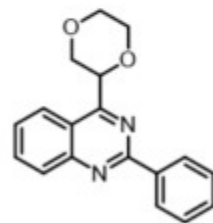




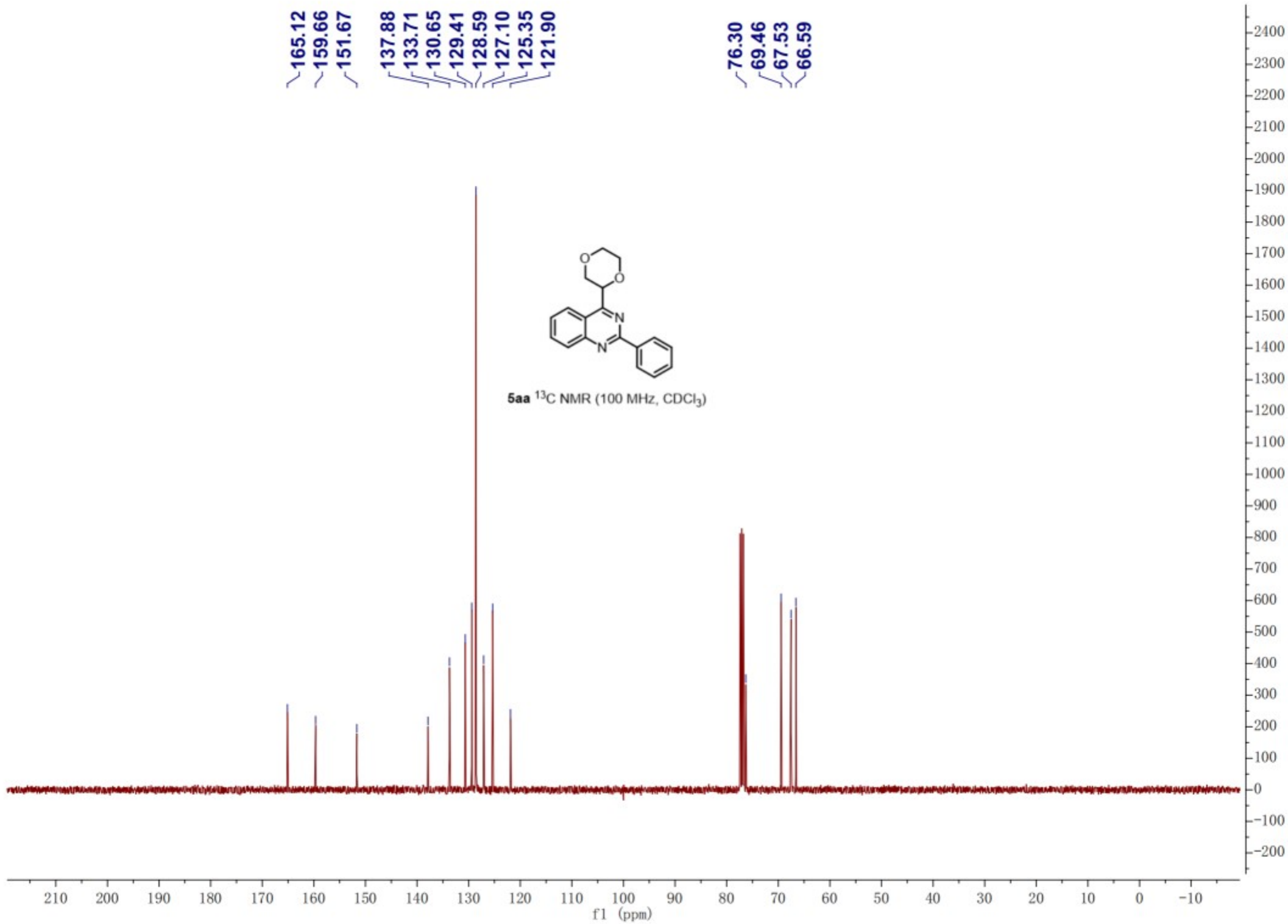


165.12  
159.66  
151.67  
137.88  
133.71  
130.65  
129.41  
128.59  
127.10  
125.35  
121.90

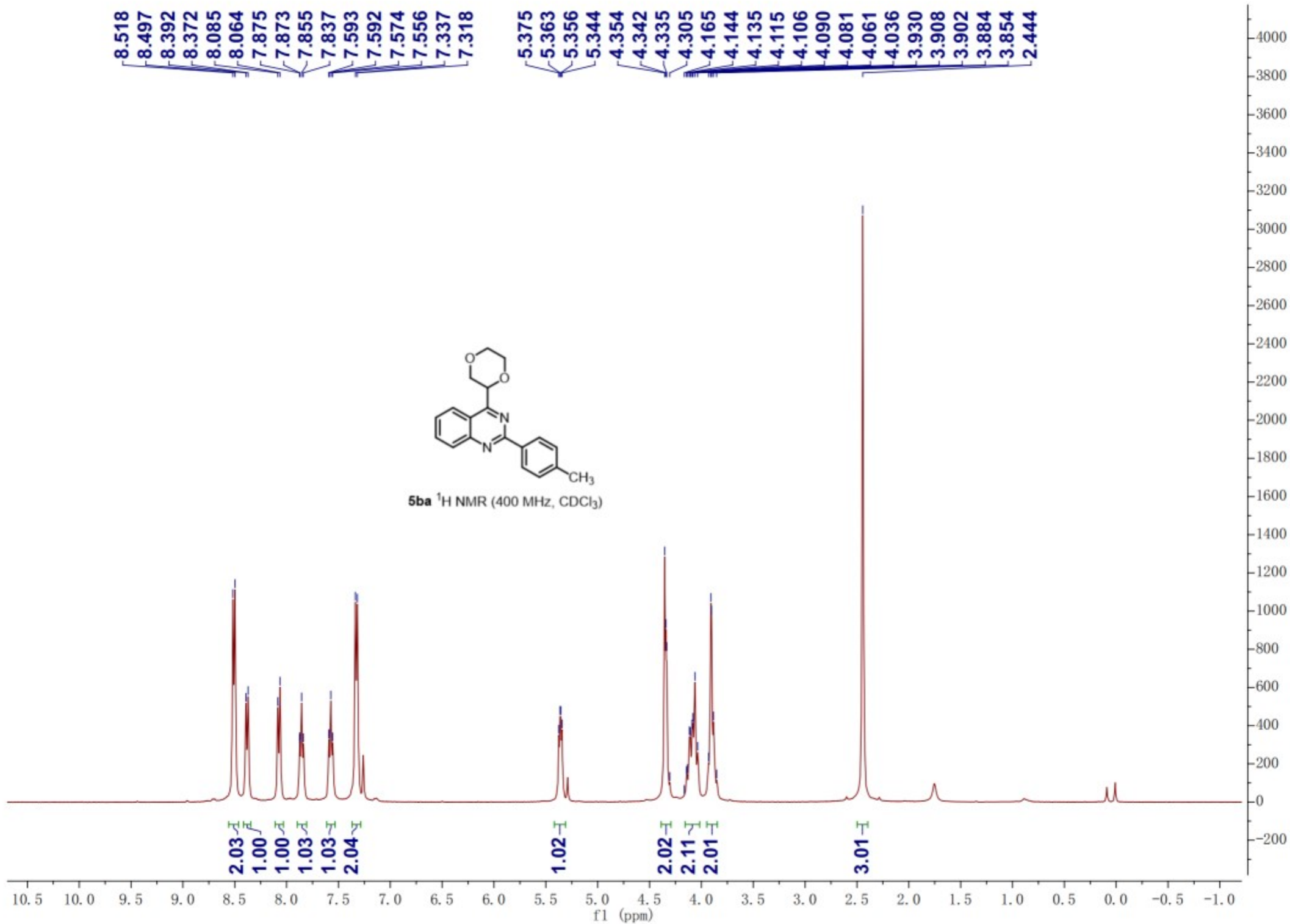
76.30  
69.46  
67.53  
66.59



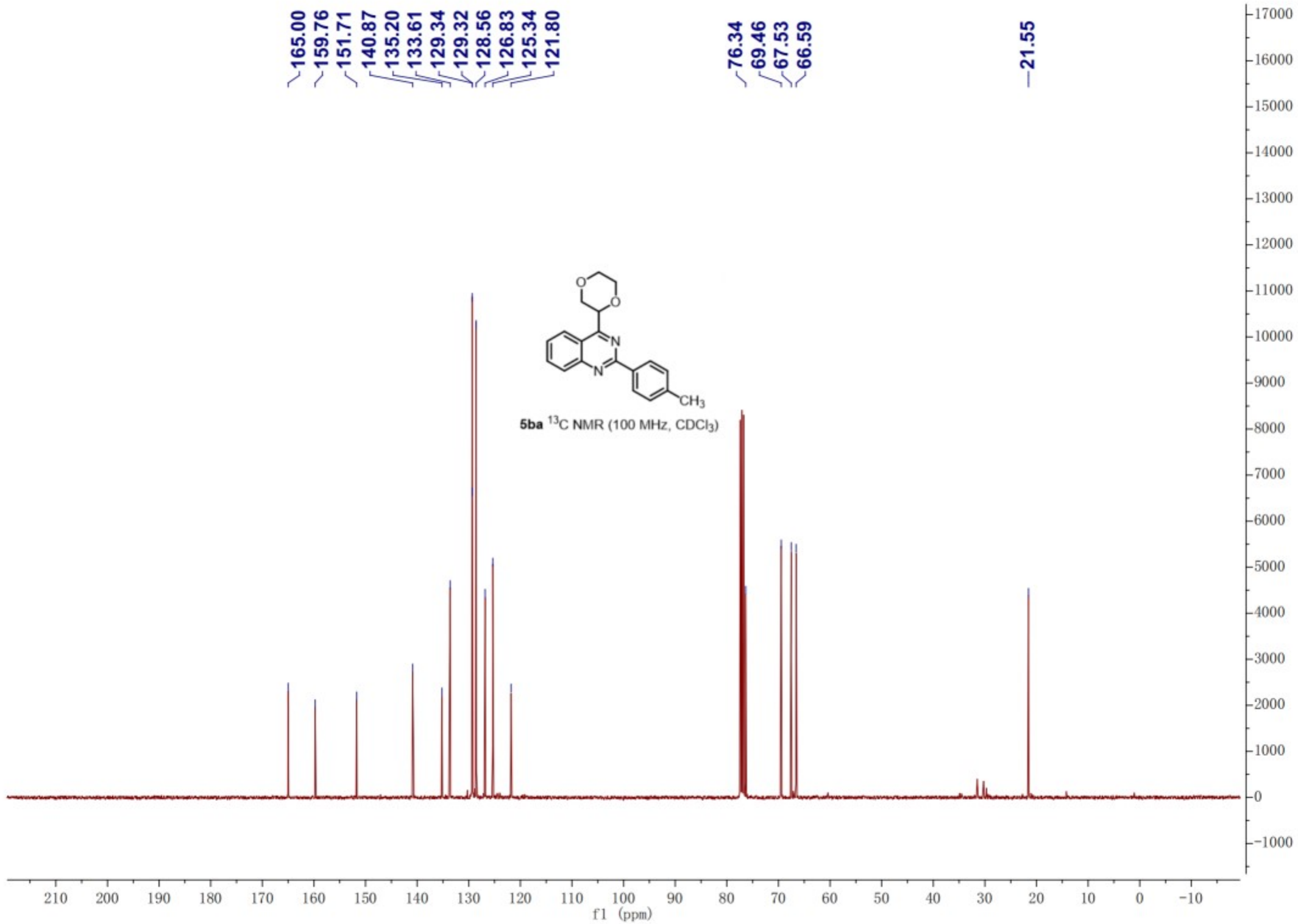
5aa <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

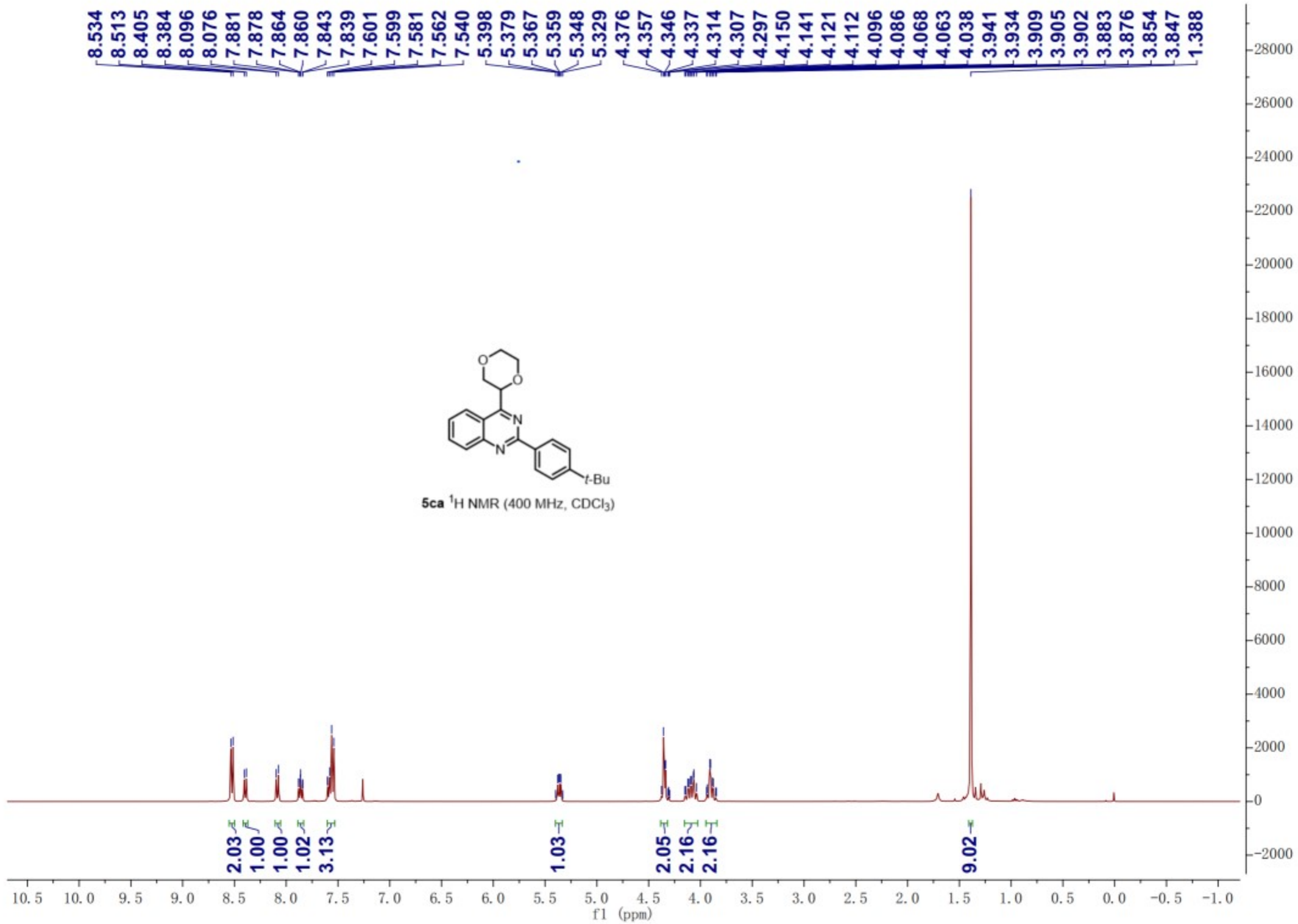








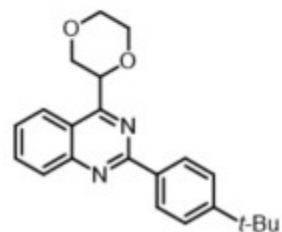




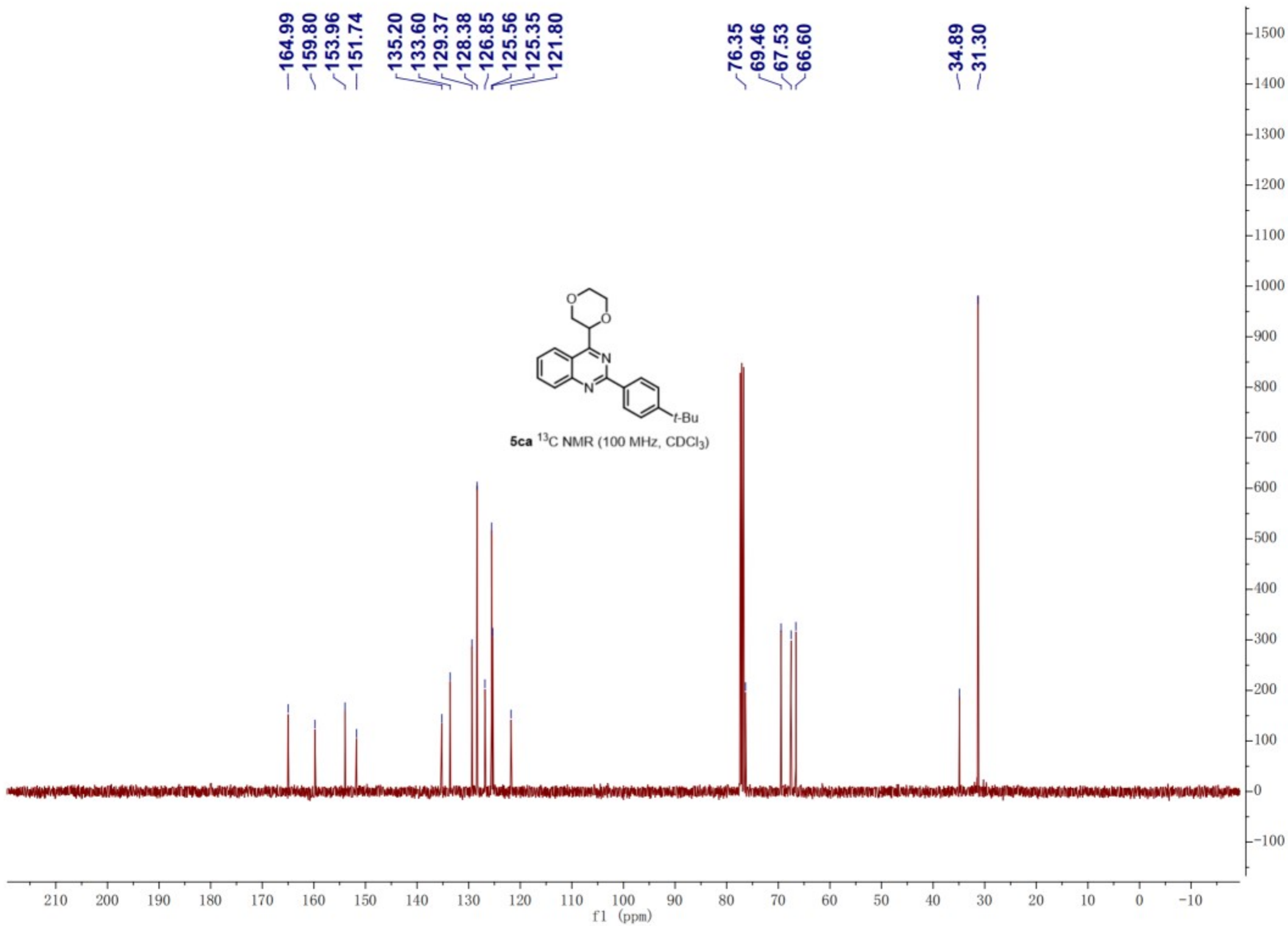
164.99  
159.80  
153.96  
151.74  
135.20  
133.60  
129.37  
128.38  
126.85  
125.56  
125.35  
121.80

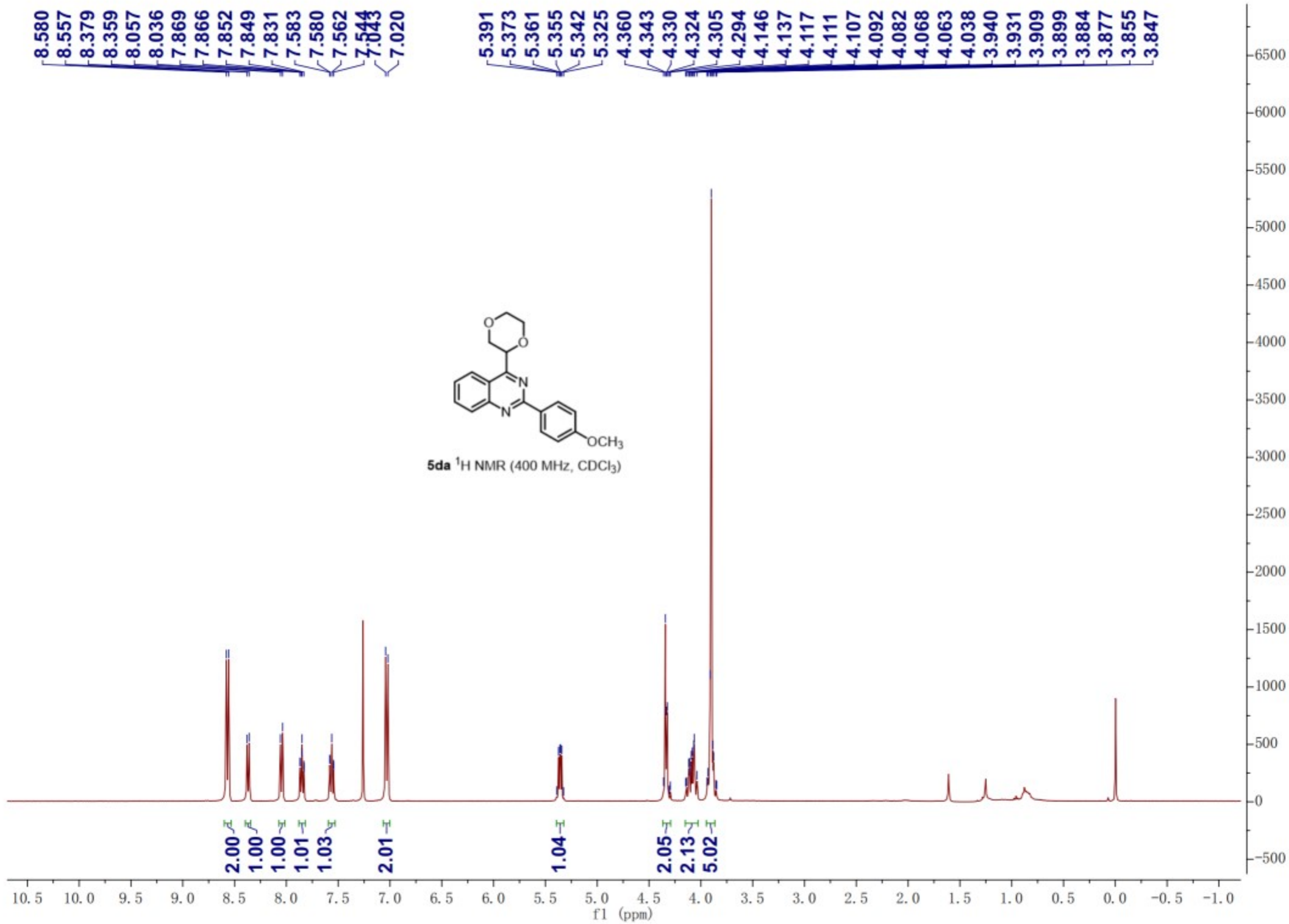
76.35  
69.46  
67.53  
66.60

34.89  
31.30



**5ca**  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

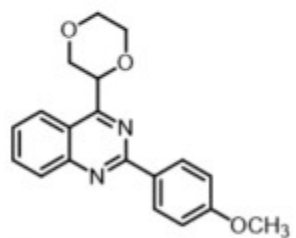




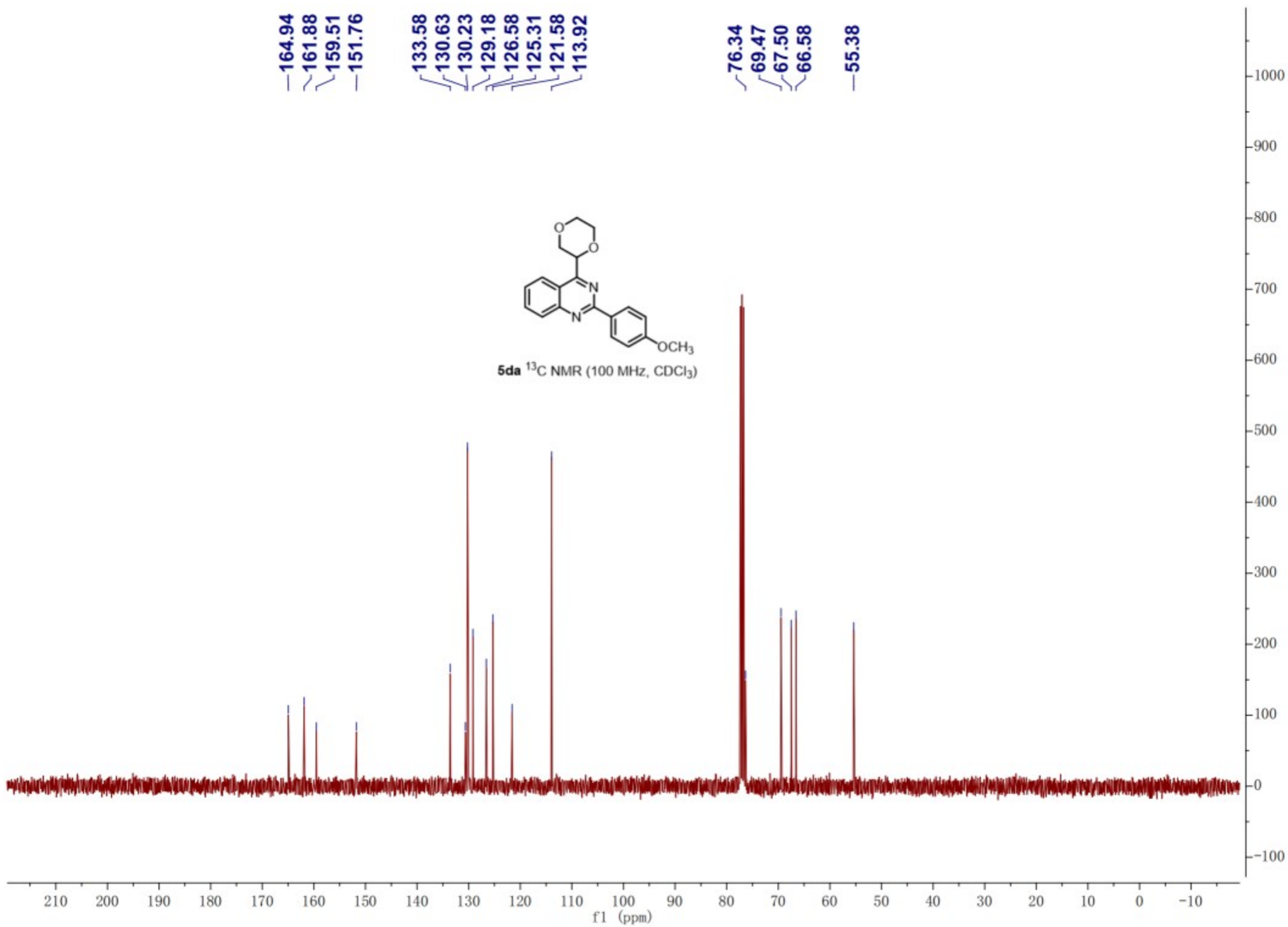
164.94  
161.88  
159.51  
151.76

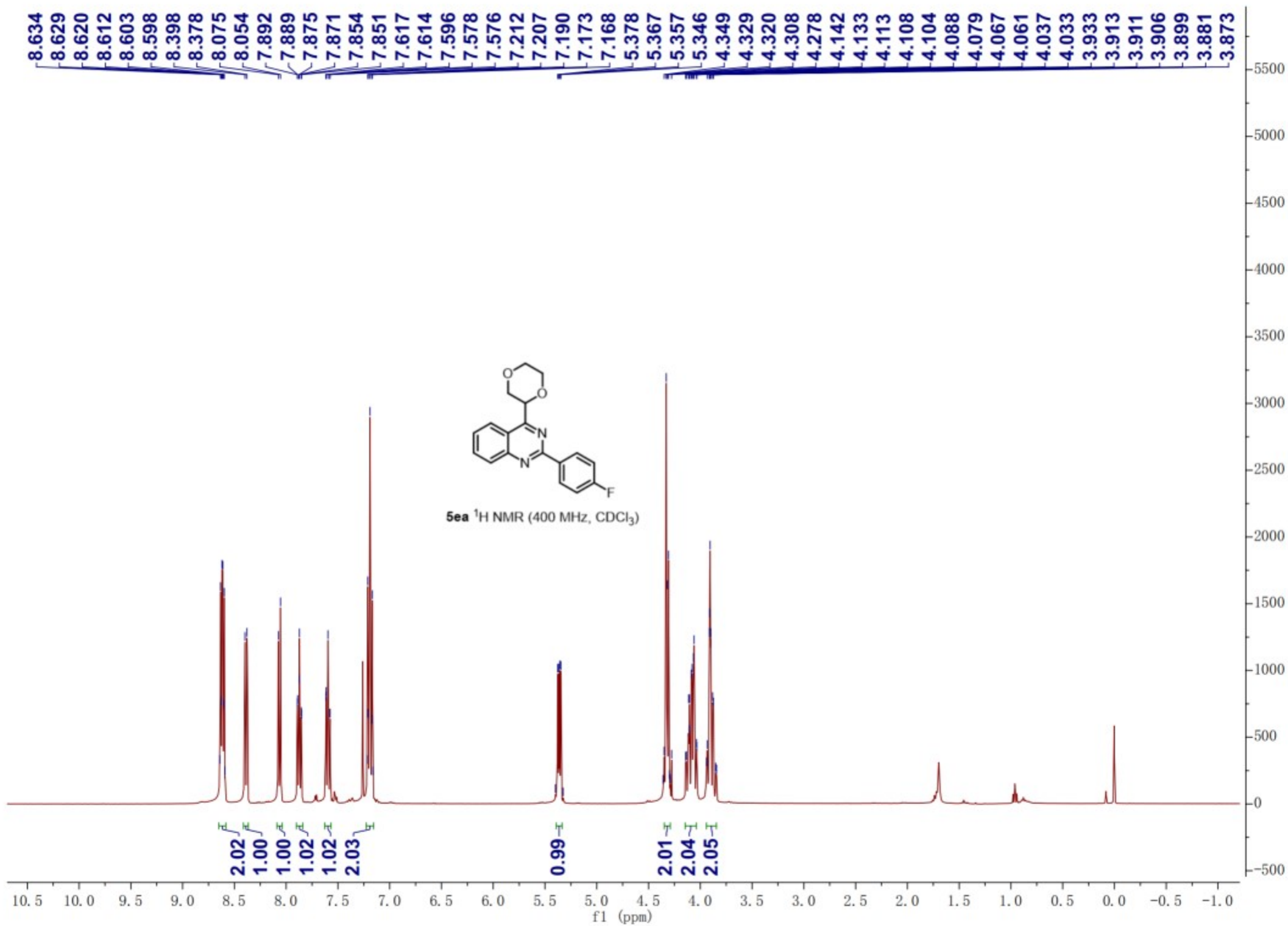
133.58  
130.63  
130.23  
129.18  
126.58  
125.31  
121.58  
113.92

76.34  
69.47  
67.50  
66.58  
55.38



5da  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

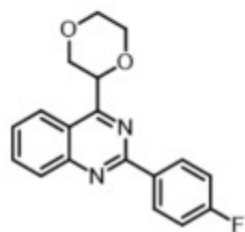




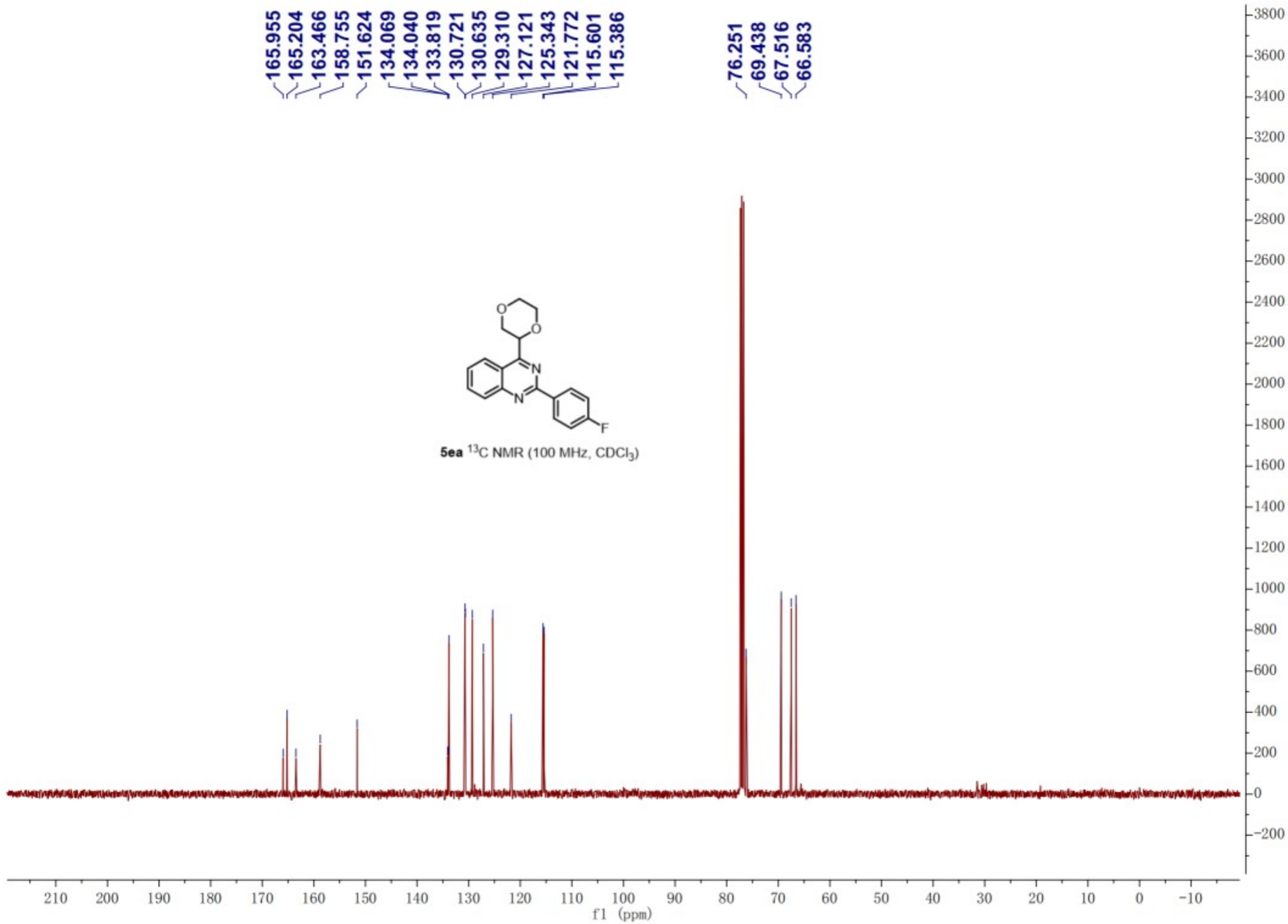


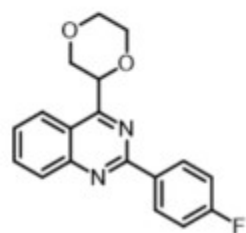
165.955  
165.204  
163.466  
158.755  
151.624  
134.069  
134.040  
133.819  
130.721  
130.635  
129.310  
127.121  
125.343  
121.772  
115.601  
115.386

76.251  
69.438  
67.516  
66.583



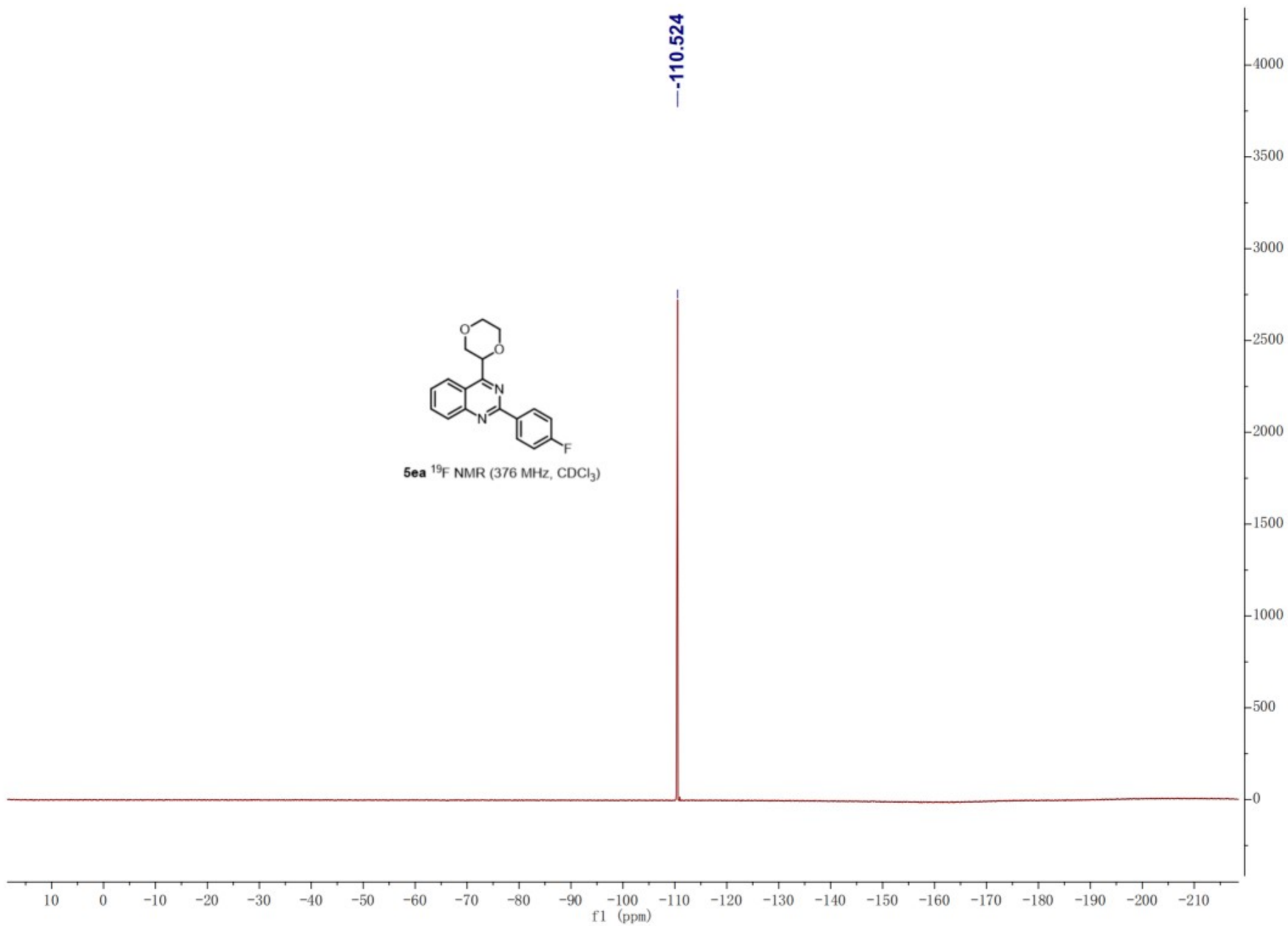
5ea <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)





**5ea**  $^{19}\text{F}$  NMR (376 MHz,  $\text{CDCl}_3$ )

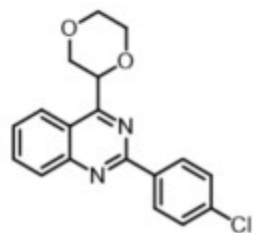
---110.524





8.559  
8.538  
8.397  
8.376  
8.074  
8.053  
7.891  
7.873  
7.853  
7.620  
7.602  
7.582  
7.484  
7.463

5.369  
5.359  
5.348  
5.338  
4.344  
4.322  
4.300  
4.270  
4.137  
4.130  
4.109  
4.101  
4.083  
4.074  
4.062  
4.057  
4.033  
3.931  
3.902  
3.877  
3.870  
3.848  
3.840



5fa <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

2.02  
1.00  
1.00  
1.04  
1.03  
2.01

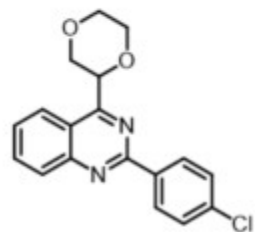
1.00

2.00  
2.06  
2.12

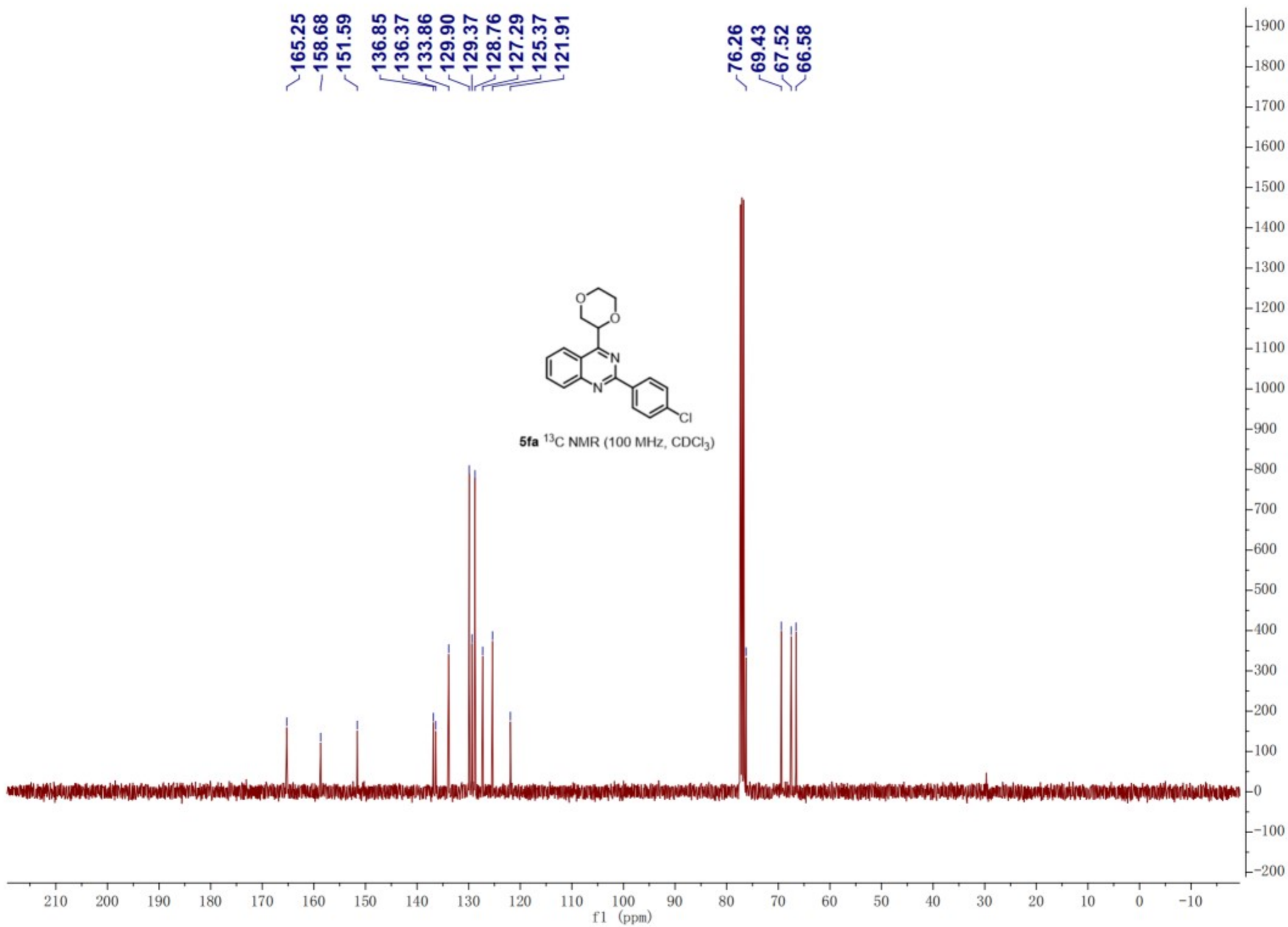
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0  
f1 (ppm)

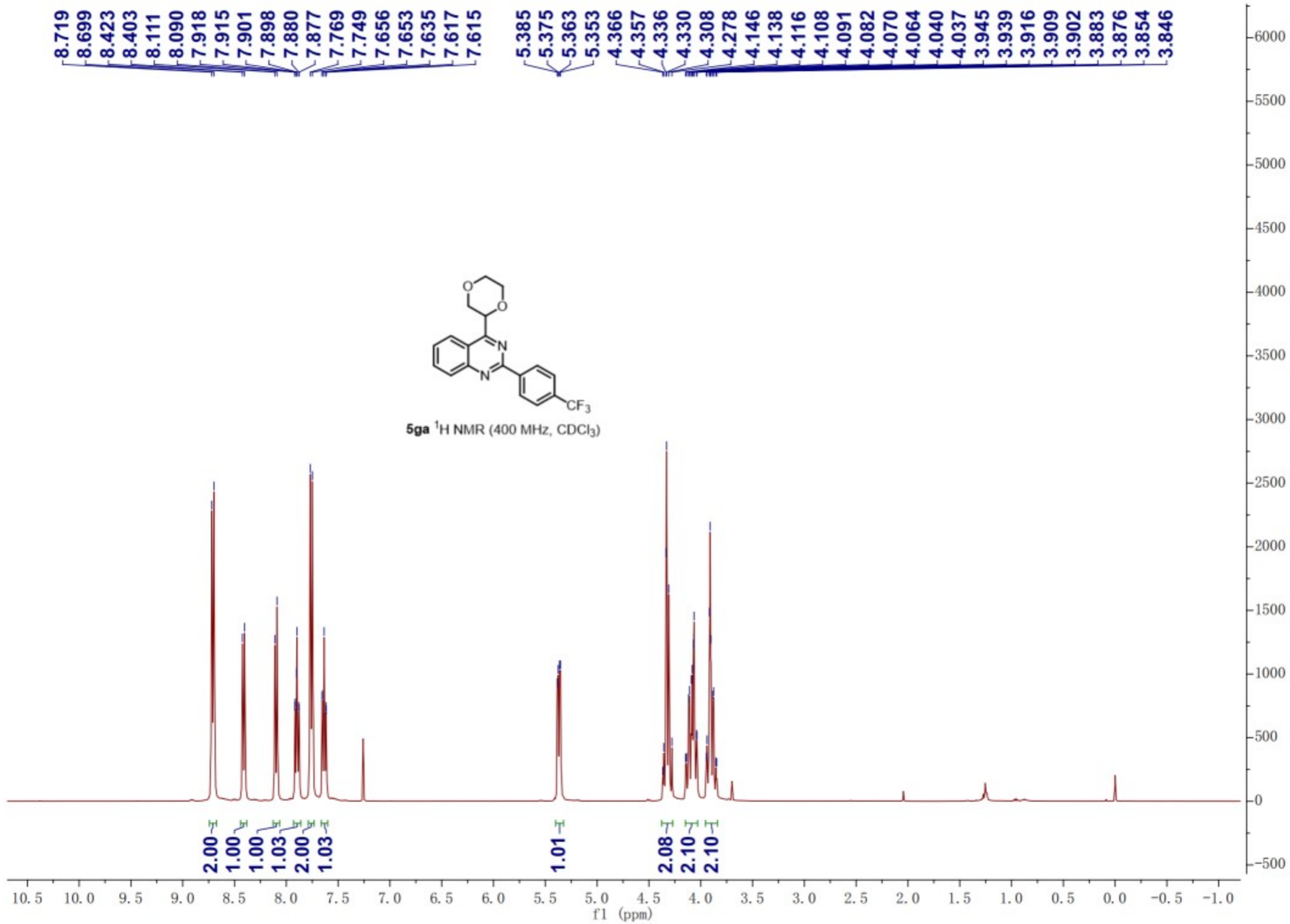
165.25  
158.68  
151.59  
136.85  
136.37  
133.86  
129.90  
129.37  
128.76  
127.29  
125.37  
121.91

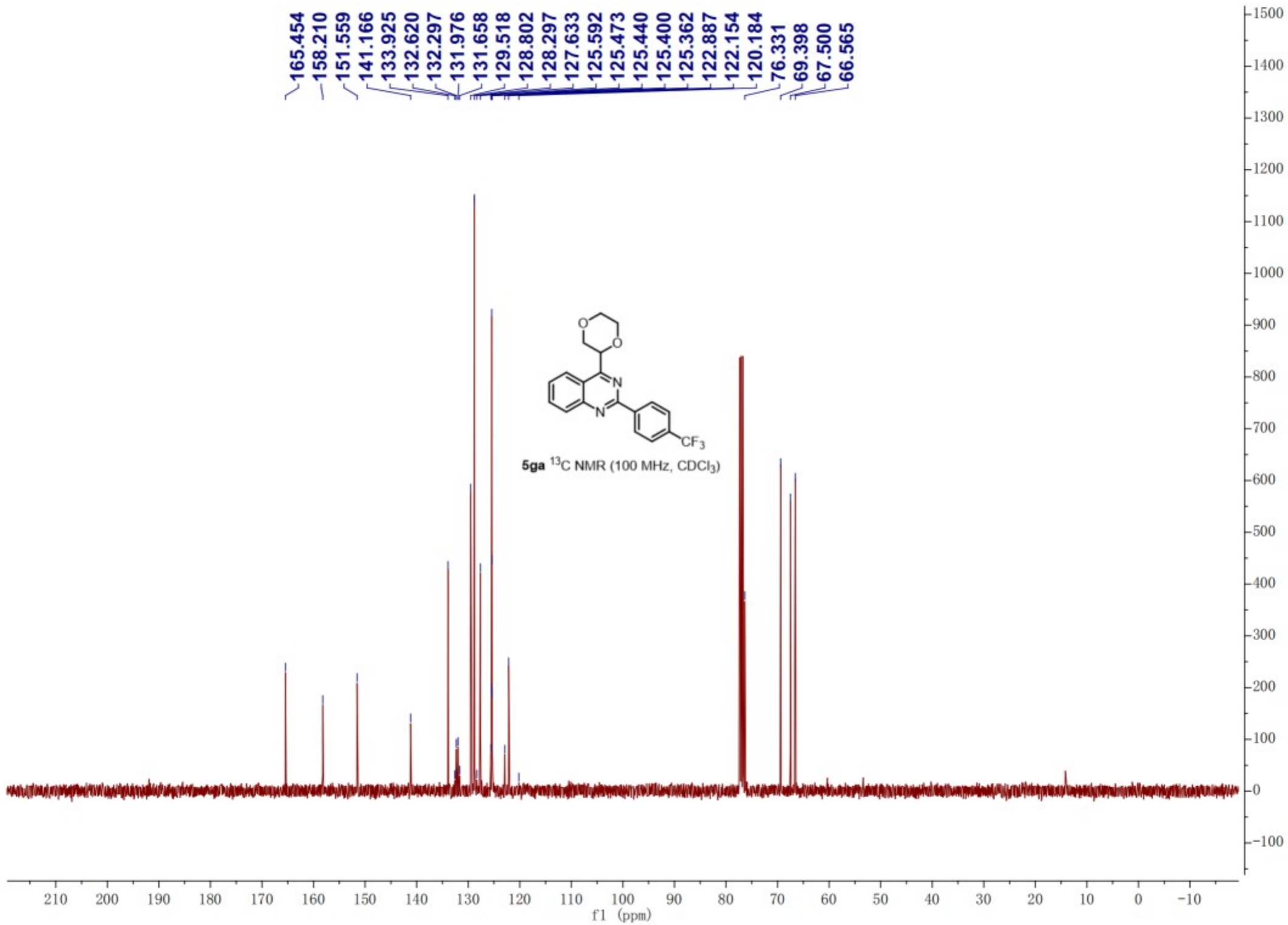
76.26  
69.43  
67.52  
66.58

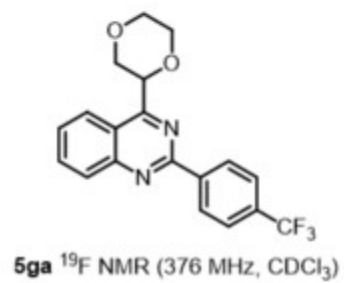


5fa <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

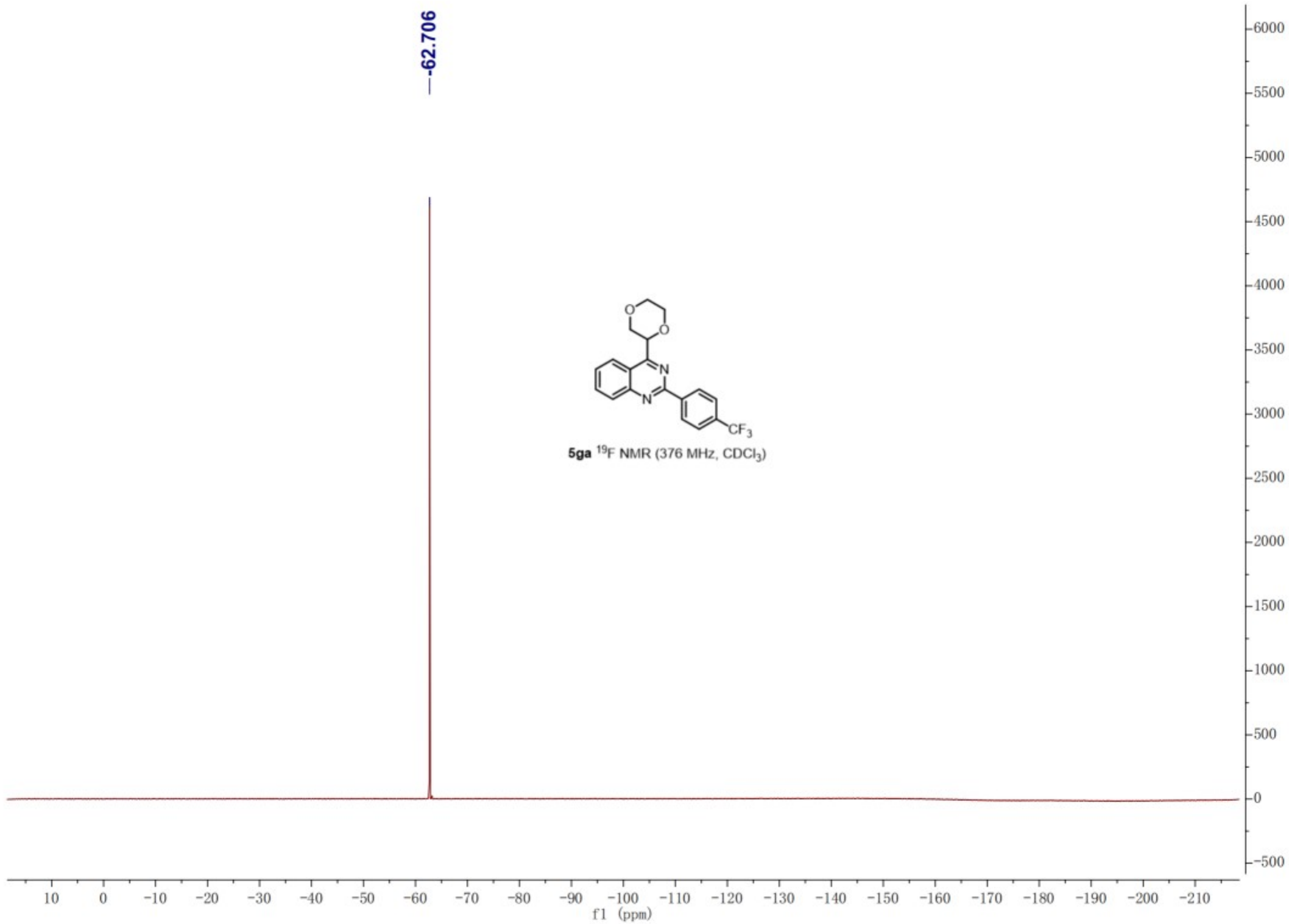


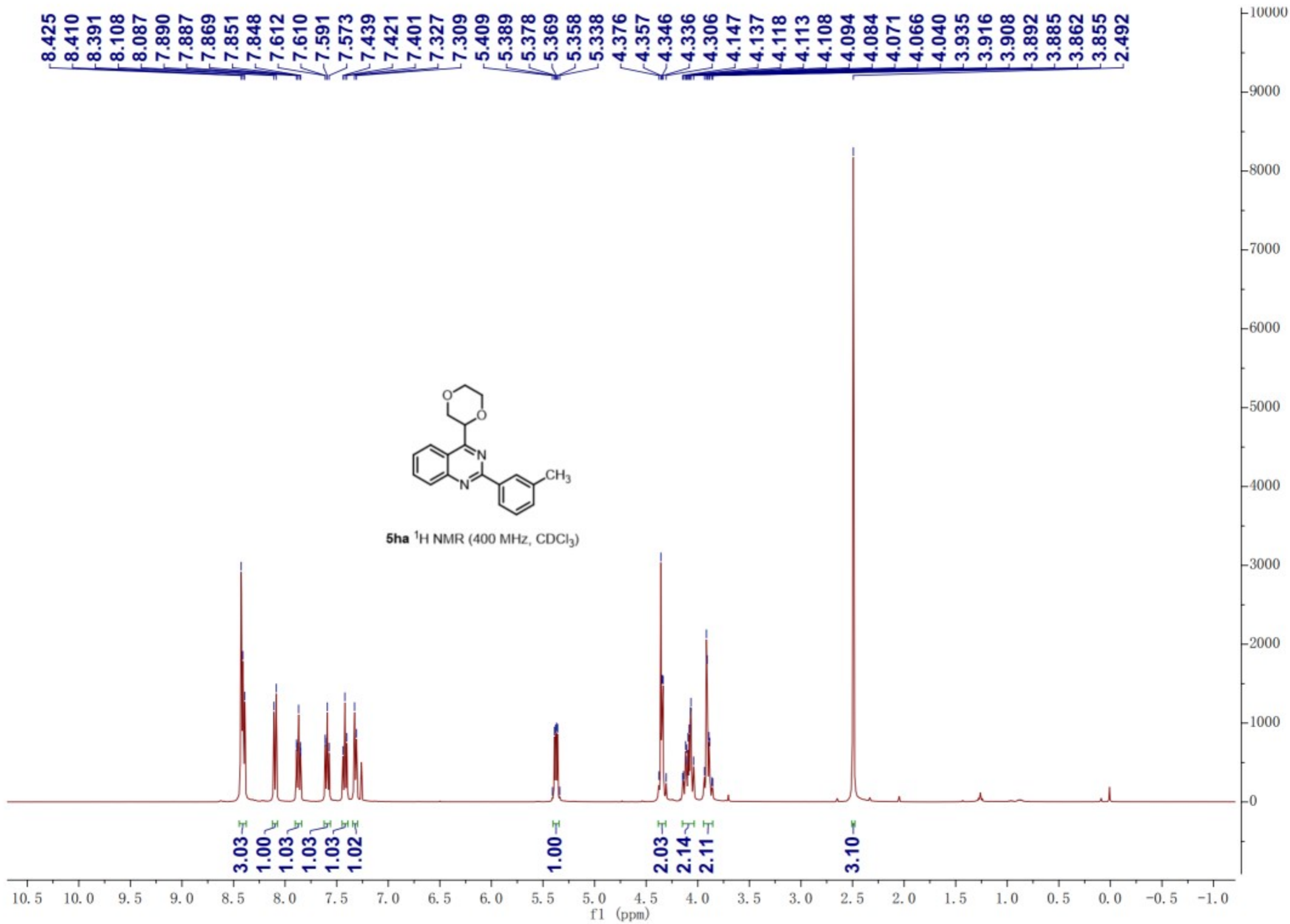


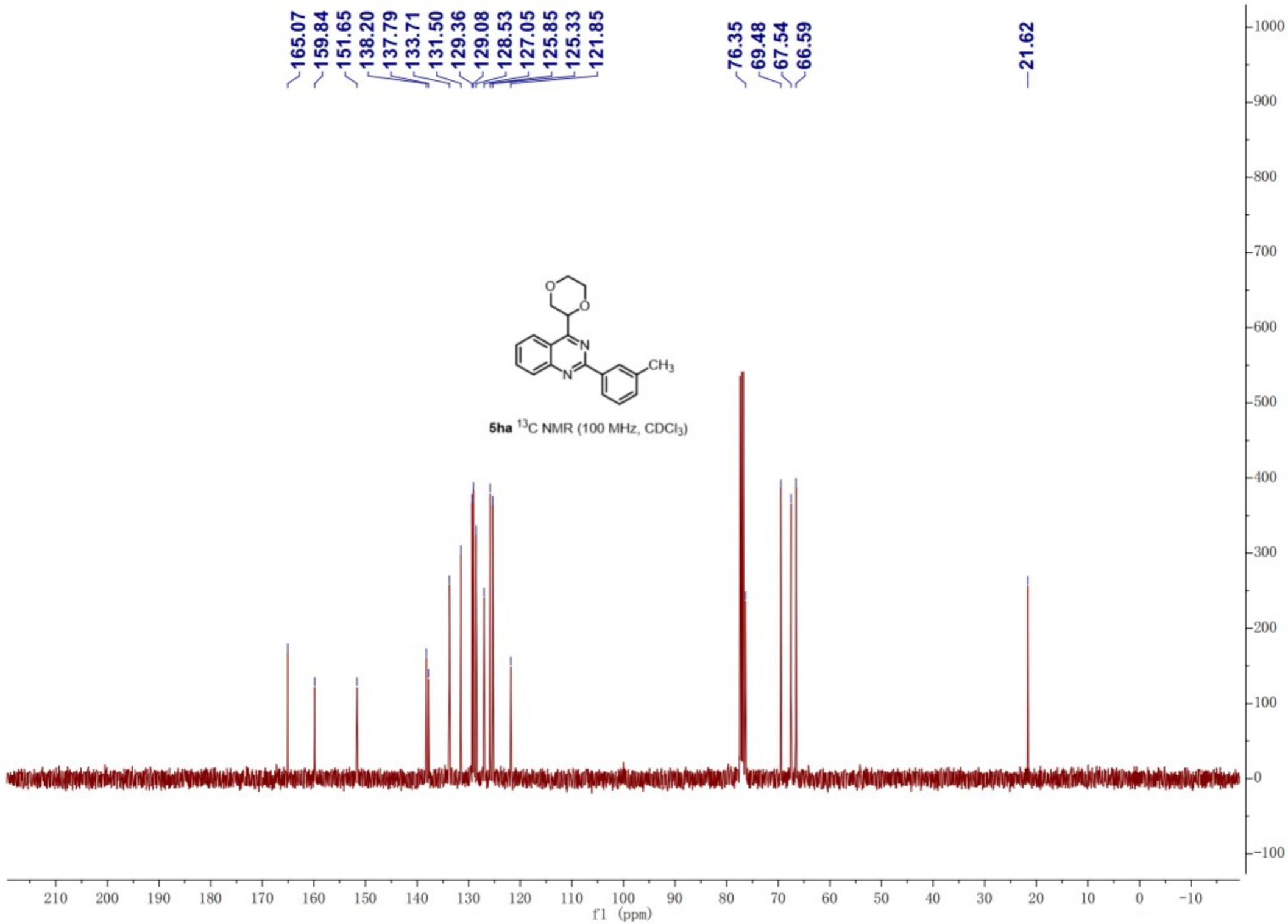




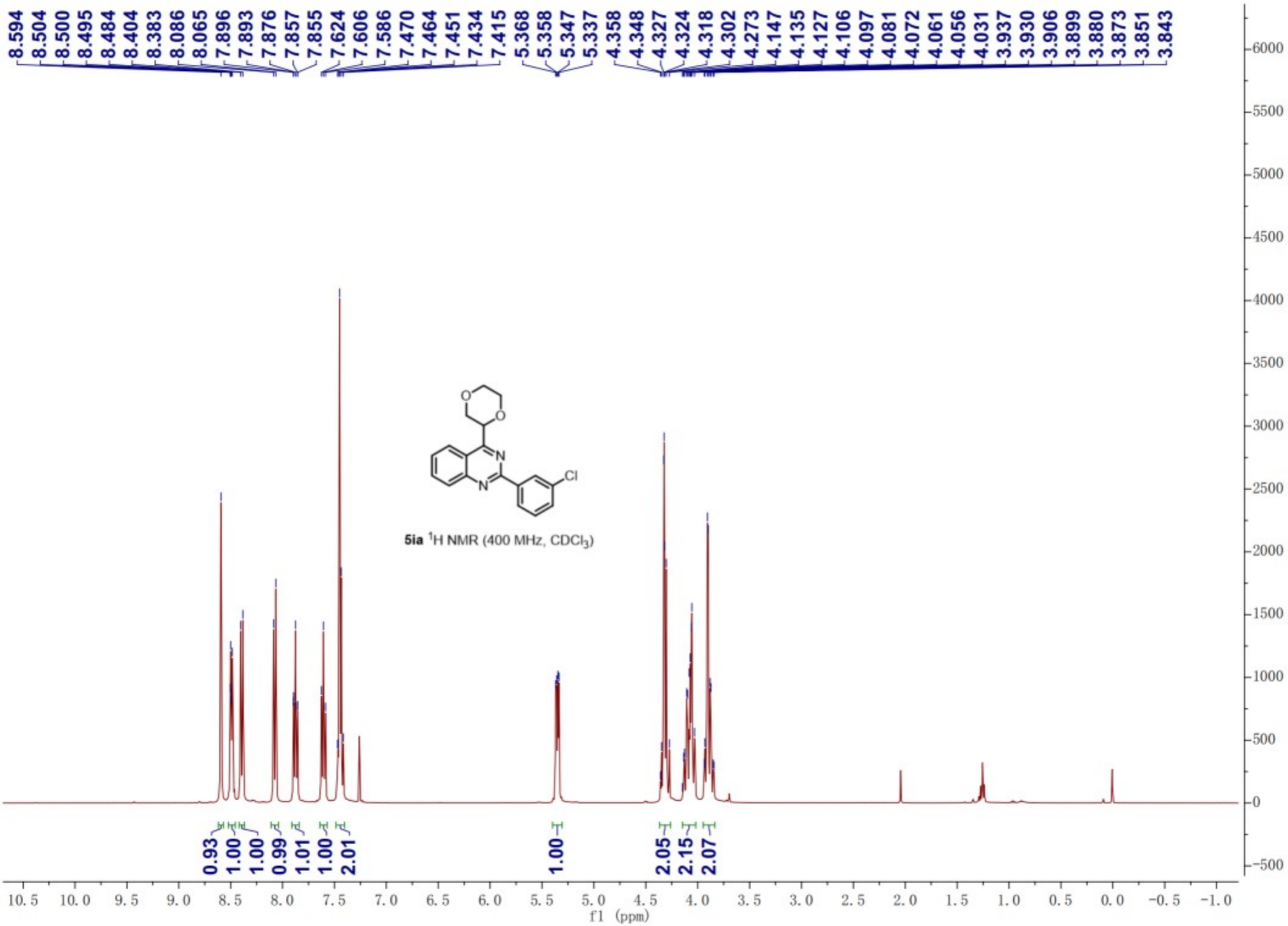
-62.706



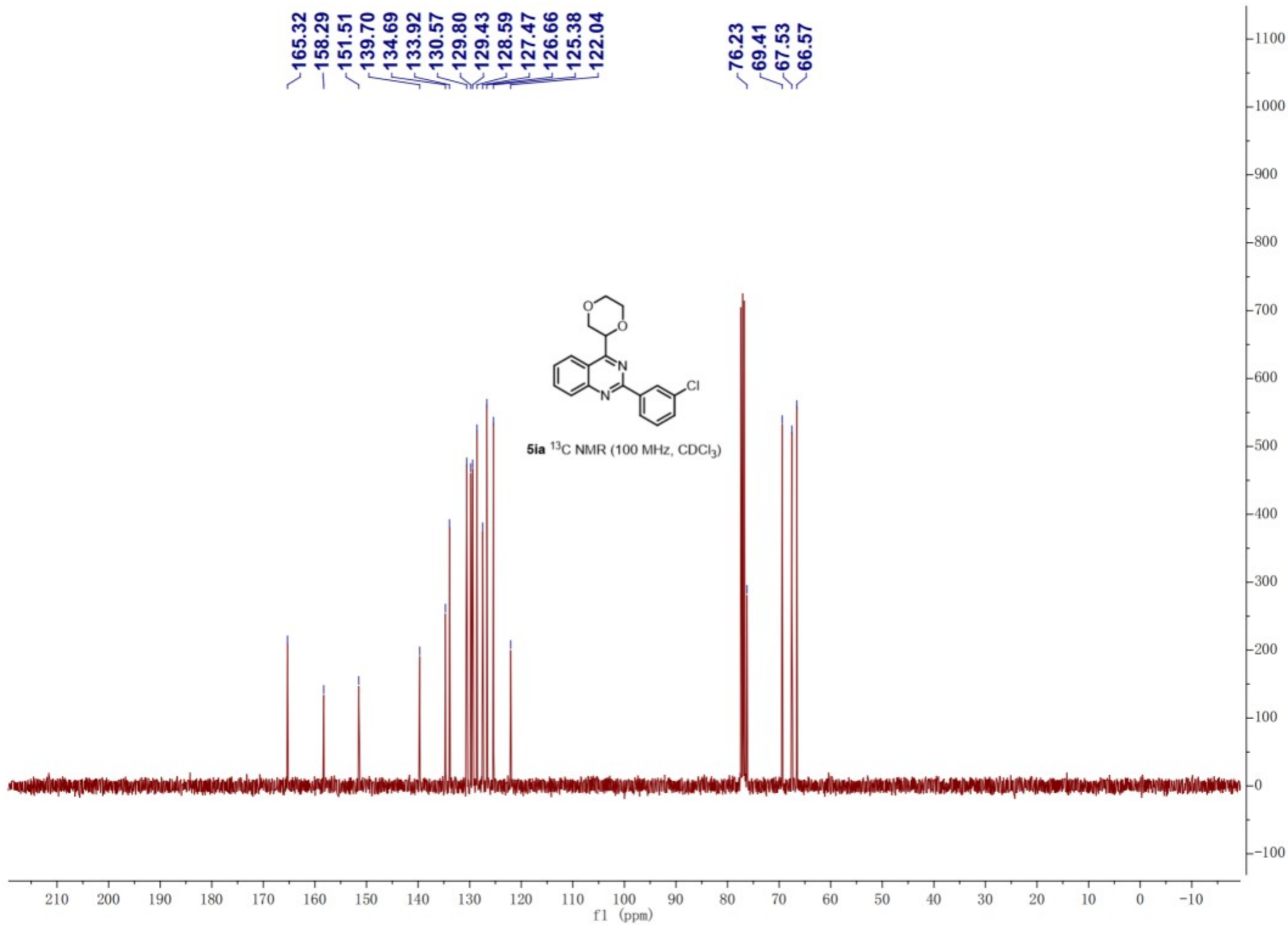


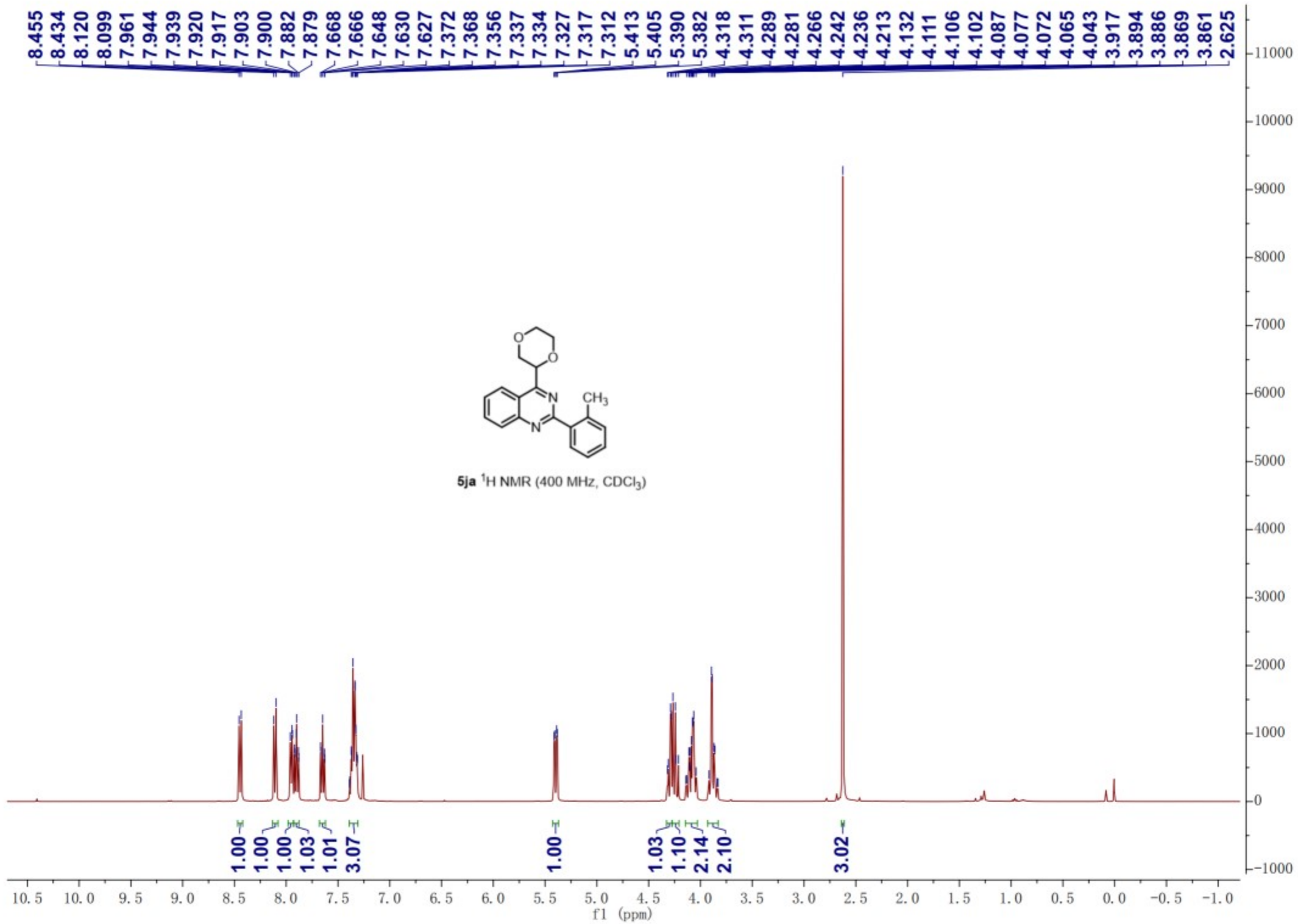


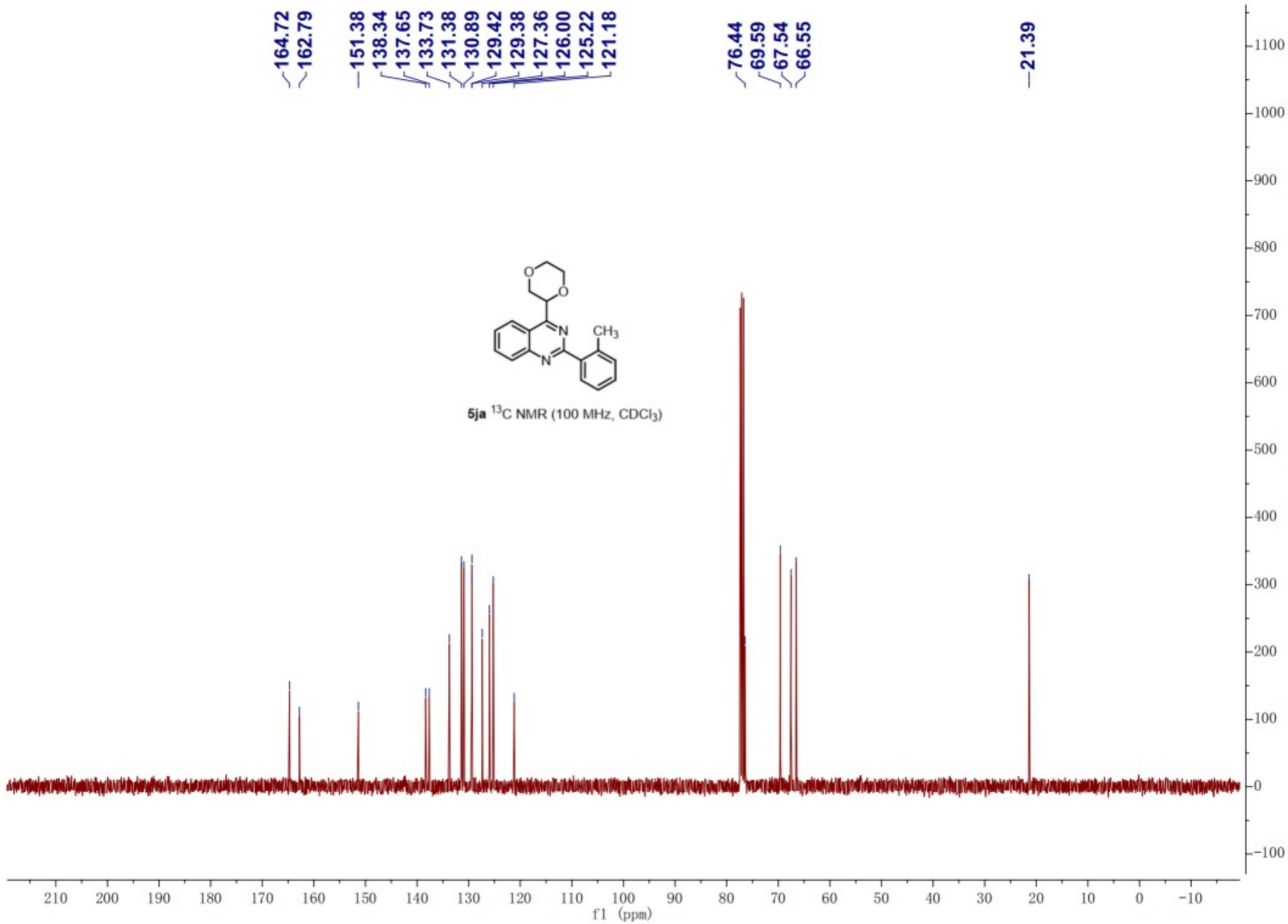


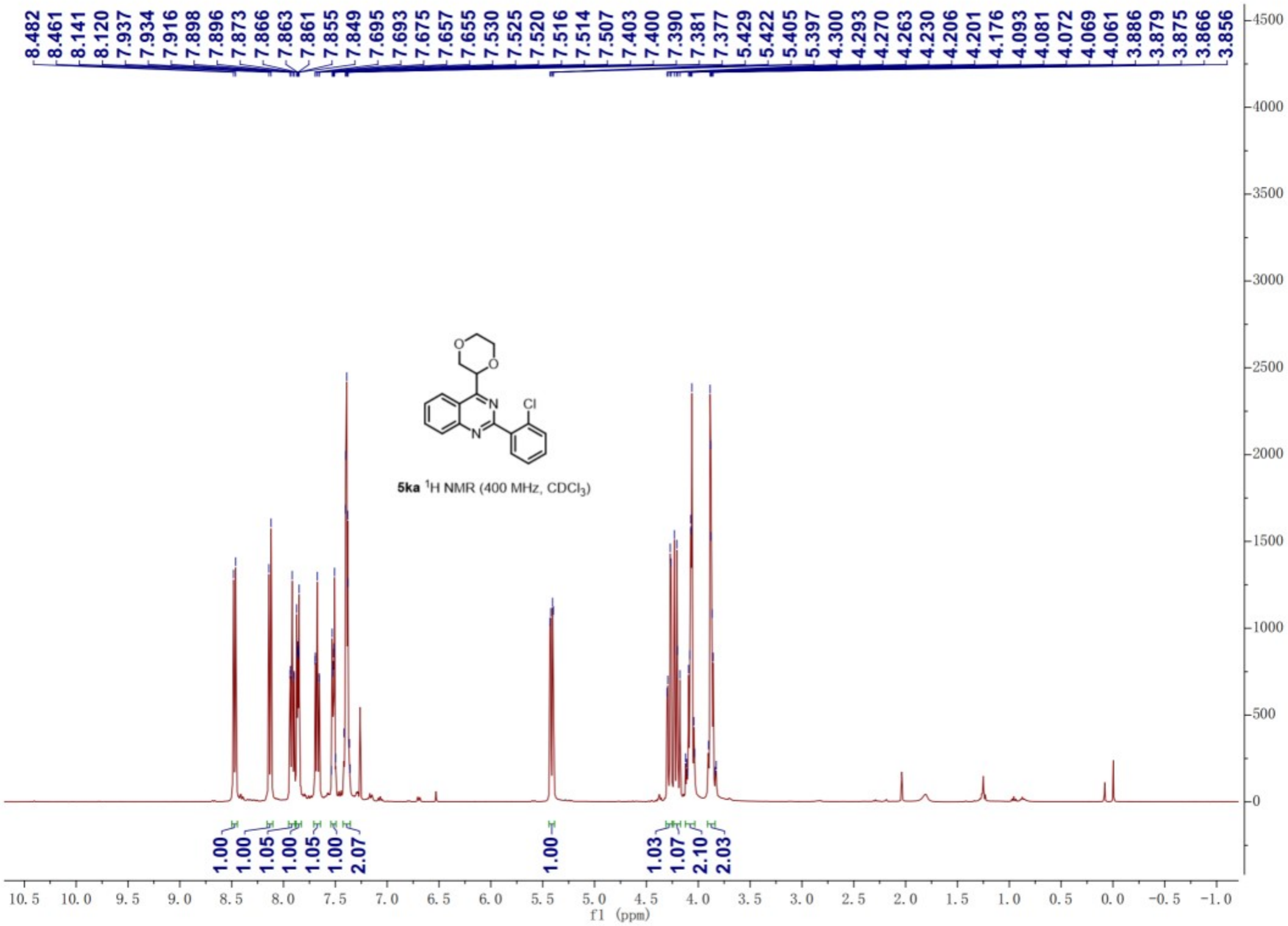


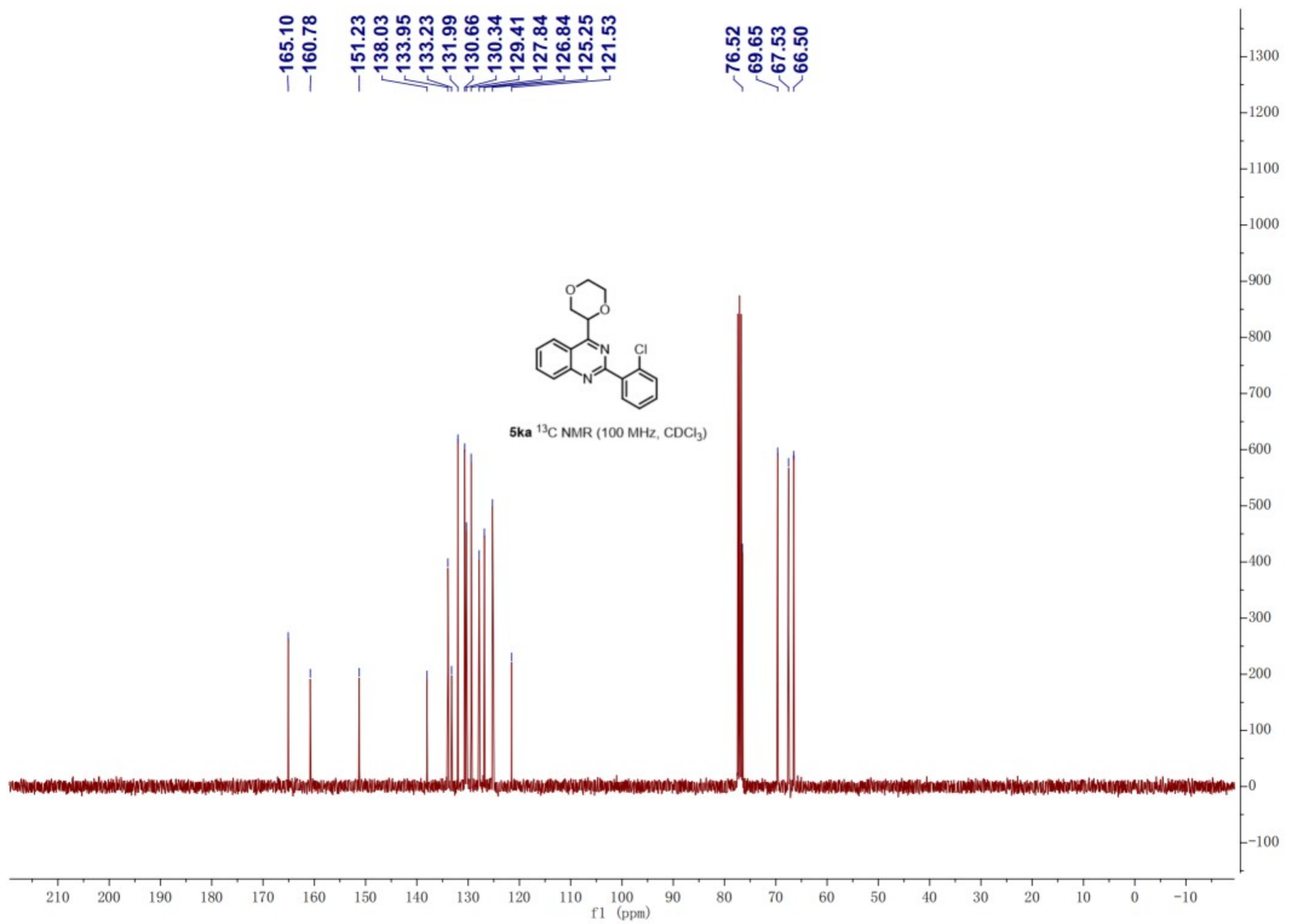


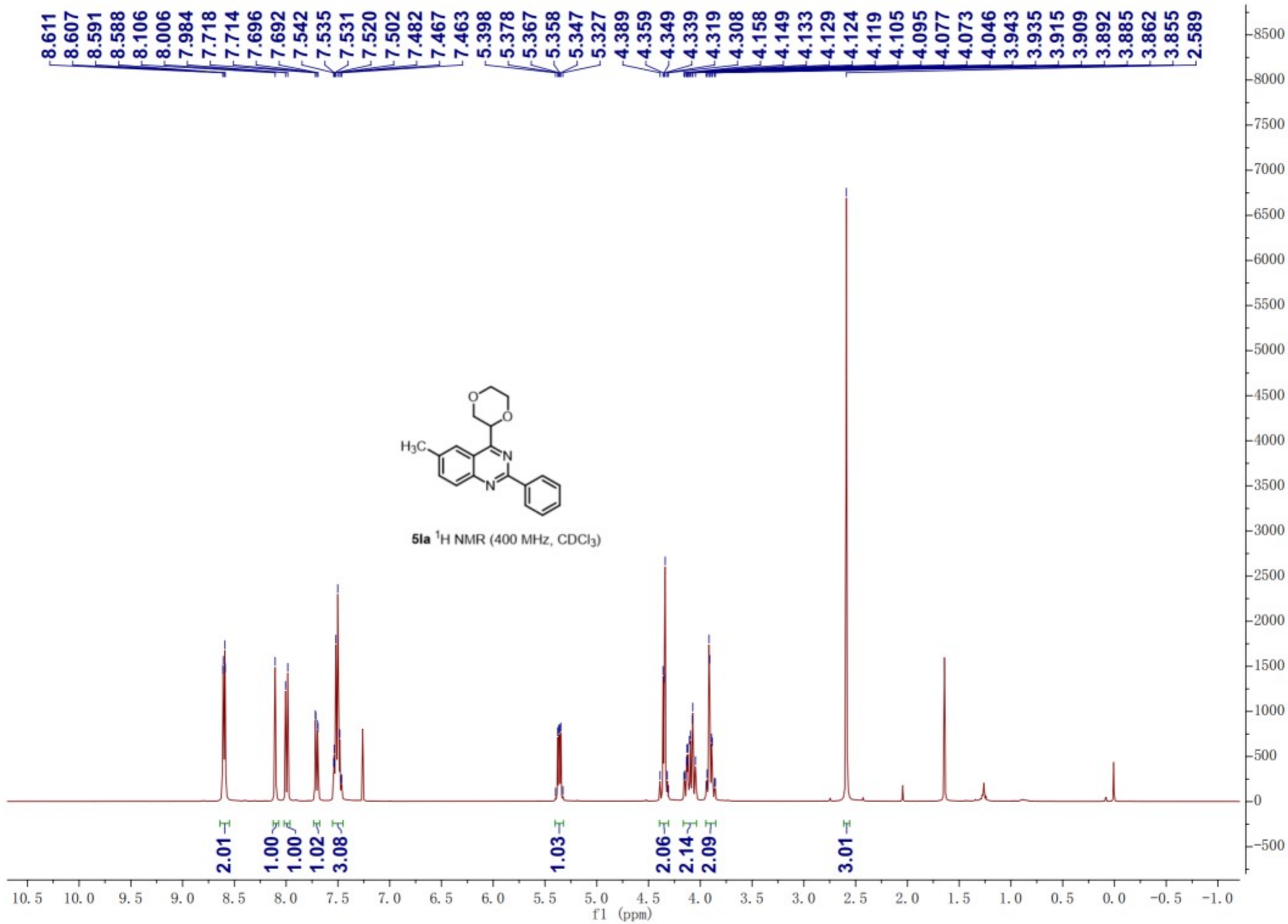




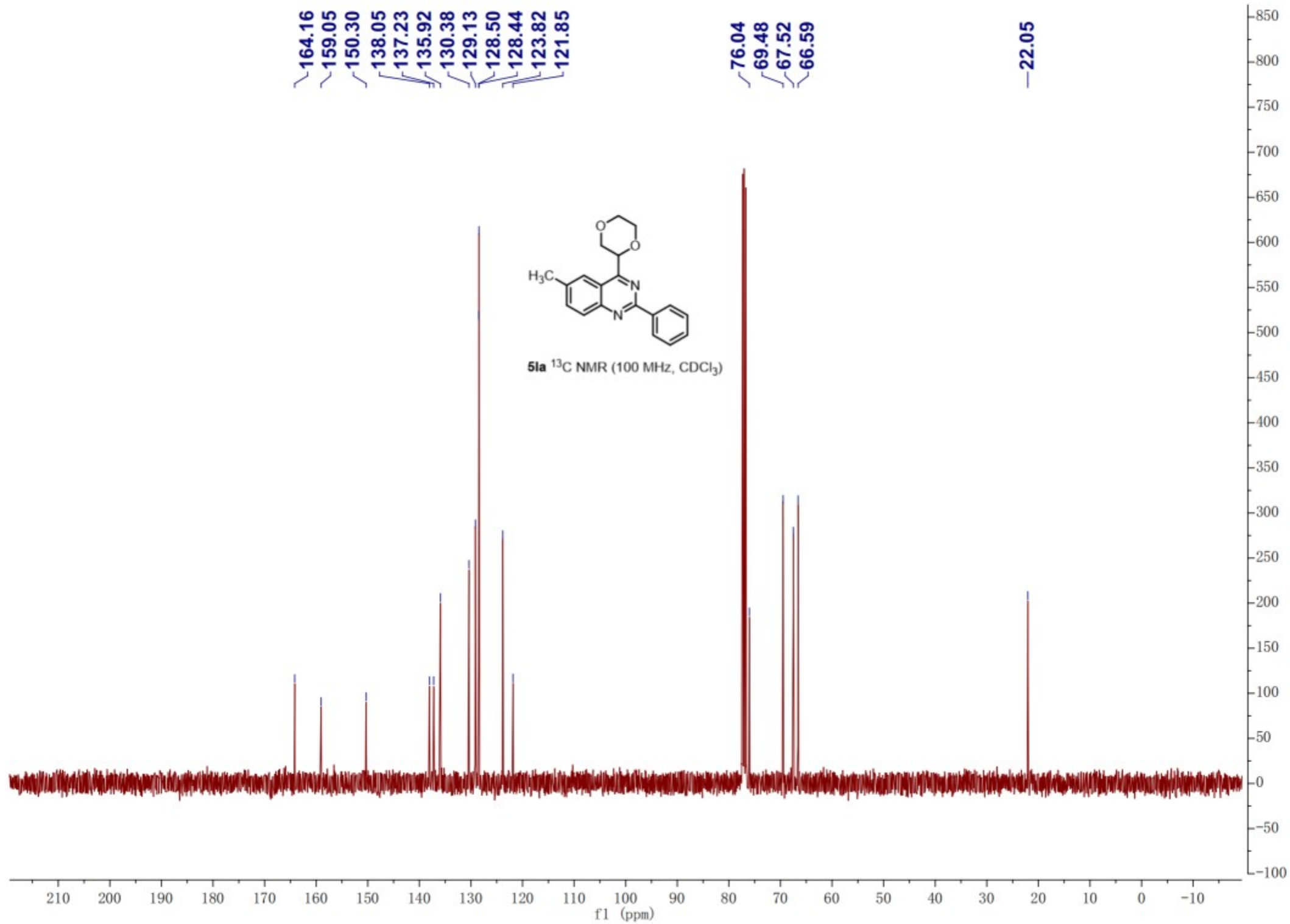




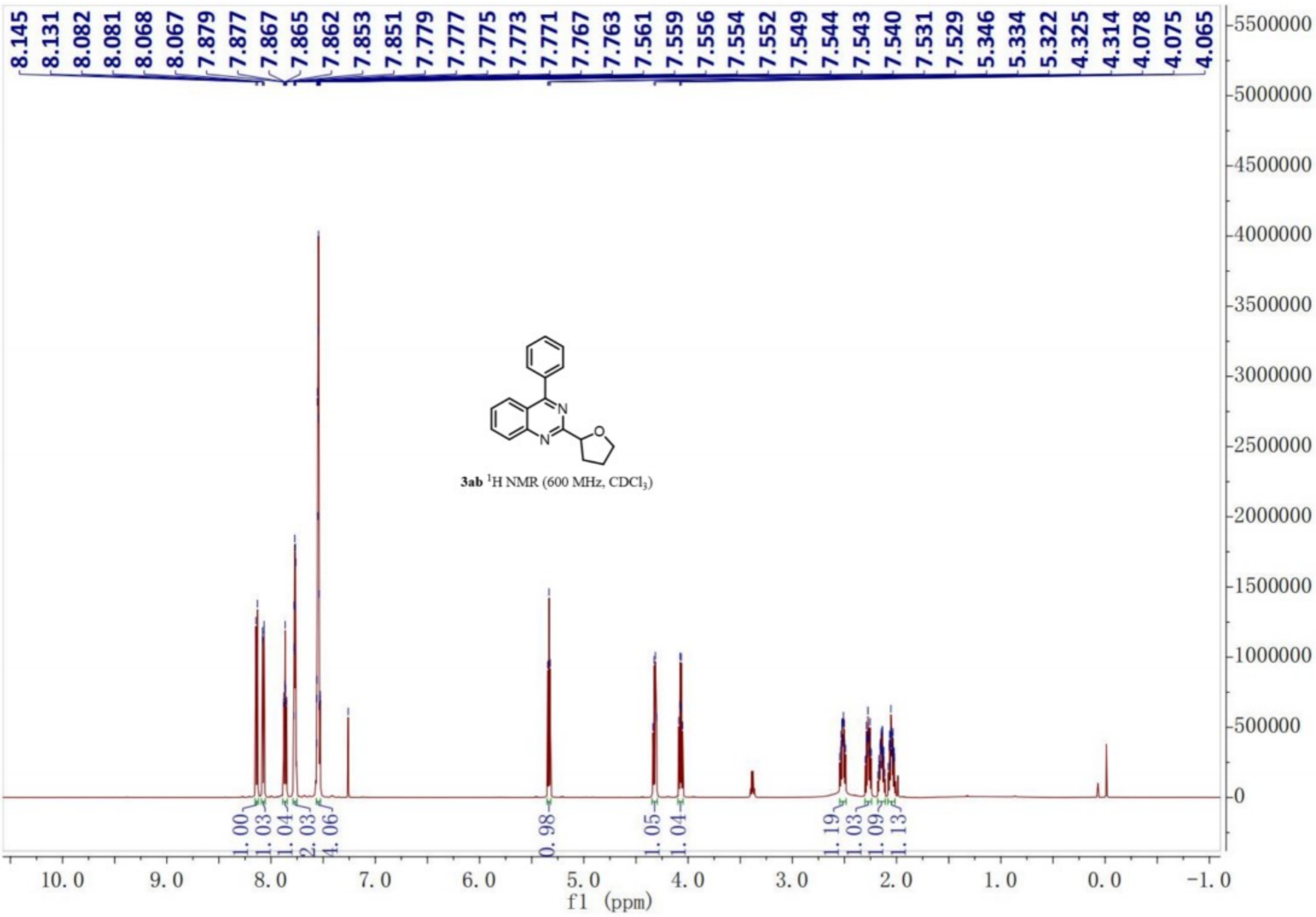












168.77  
166.37

151.33

137.36

133.56

130.09

129.92

129.01

128.55

127.22

126.98

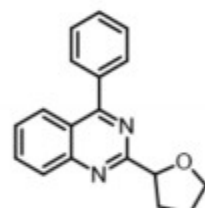
121.90

81.92

69.51

32.38

25.87

**3ab**  $^{13}\text{C}$  NMR (150 MHz,  $\text{CDCl}_3$ )210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10  
f1 (ppm)1000000  
900000  
800000  
700000  
600000  
500000  
400000  
300000  
200000  
100000  
0  
-100000