

## Supporting Information

### Electrochemical Direct C-H Mono and Bis-Chalcogenation of Indolizine Frameworks Under Oxidant-Free Conditions

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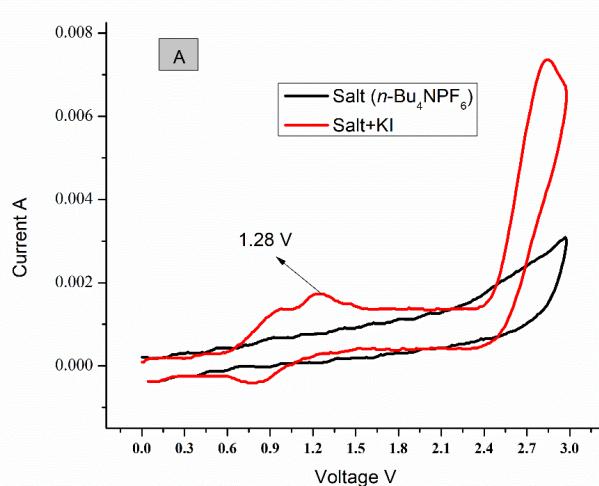
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### General Procedure for Cyclic Voltammetry (CV):

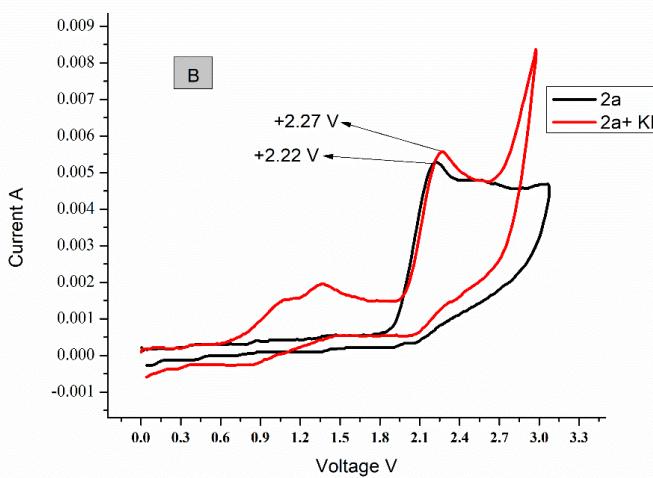
Cyclic voltammetry was performed in a three electrode cell at room temperature. The working electrode was a glassy electrode and the counter electrode was a platinum electrode. The reference was an Ag/AgCl electrode submerged in 3M KCl solution, and separated from the reaction by a salt bridge.

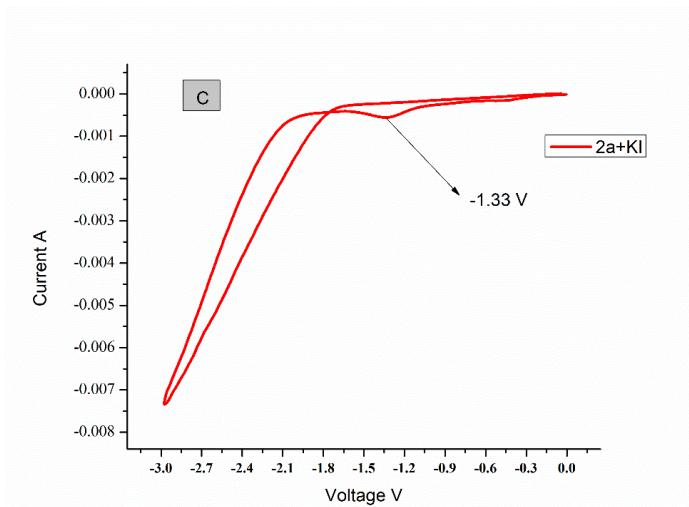
### Cyclic Voltammograms graph for KI



**Figure S1.** Cyclic voltammograms of reactants and mixture in 0.1 M  $n\text{-Bu}_4\text{NPF}_6$  in  $\text{CH}_3\text{CN}$  using a glassy carbon disk electrode, Pt electrode as counter electrode and Ag/AgCl as reference electrode, at a  $200 \text{ mV}\text{s}^{-1}$ . Cyclic voltammograms of salt and salt with KI at a  $200 \text{ mV}\text{s}^{-1}$  (graph A):  $n\text{-Bu}_4\text{NPF}_6$  (0.1 M); (curve salt+KI):  $n\text{-Bu}_4\text{NPF}_6$  (0.1 M), KI (10 mM).

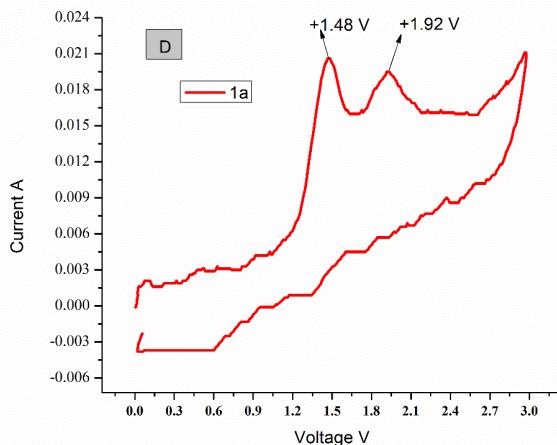
### Cyclic Voltammograms graph of 2a





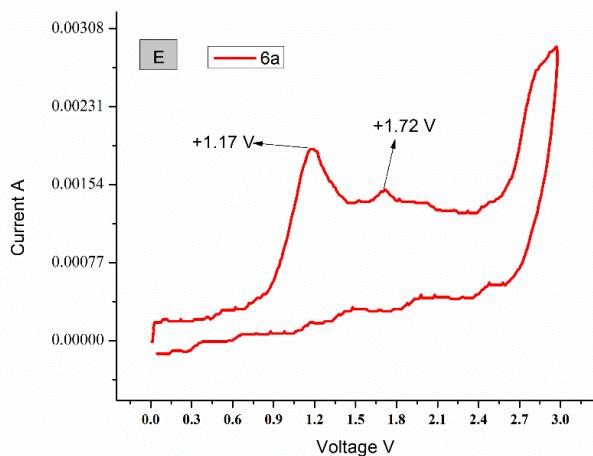
**Figure S2.** Cyclic voltammograms of **2a** in 0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub> in CH<sub>3</sub>CN using a glassy carbon disk electrode, Pt electrode as counter electrode and Ag/AgCl as reference electrode, at a 200 mVS<sup>-1</sup>; (graph B and graph C): **2a** (5 mM), *n*-Bu<sub>4</sub>NPF<sub>6</sub> (0.1 M) and KI (10 mM) in acetonitrile solvent.

#### Cyclic Voltammograms graph for **1a**



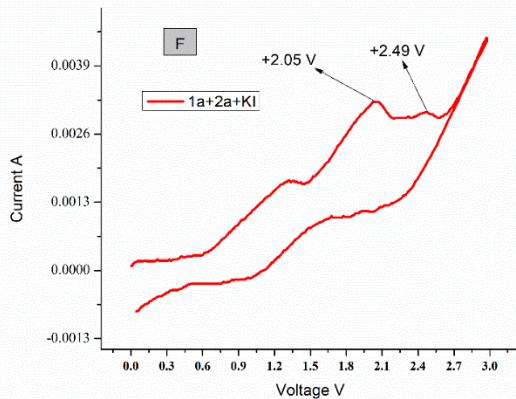
**Figure S3.** Cyclic voltammograms of **1a** in 0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub> in CH<sub>3</sub>CN using a glassy carbon disk electrode, Pt electrode as counter electrode and Ag/AgCl as reference electrode, at a 200 mVS<sup>-1</sup>; (graph D): **1a** (5 mM), *n*-Bu<sub>4</sub>NPF<sub>6</sub> (0.1 M) in acetonitrile solvent.

### Cyclic Voltammograms graph for 6a



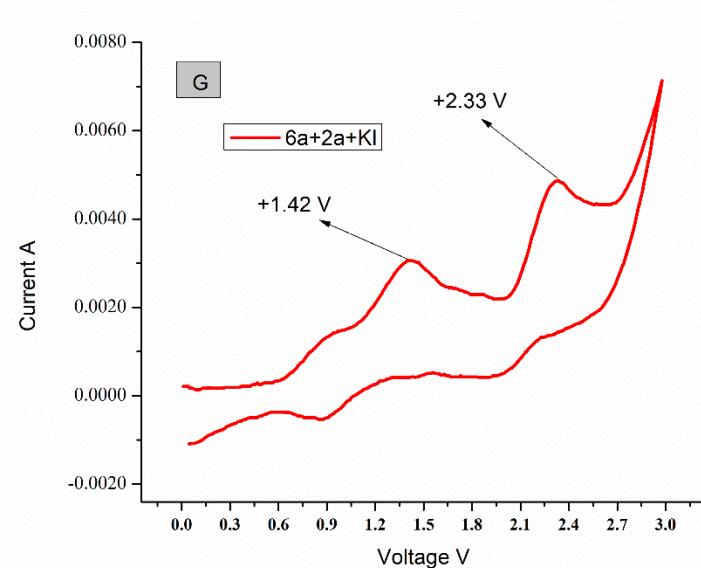
**Figure S4.** Cyclic voltammograms of **6a** in 0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub> in CH<sub>3</sub>CN using a glassy carbon disk electrode, Pt electrode as counter electrode and Ag/AgCl as reference electrode, at a 200 mVS<sup>-1</sup>; (graph E): **6a** (5 mM), *n*-Bu<sub>4</sub>NPF<sub>6</sub> (0.1 M) in acetonitrile solvent.

### Cyclic Voltammograms graph for 1a+2a



**Figure S5.** Cyclic voltammograms of reactants and mixture in 0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub> in CH<sub>3</sub>CN using a glassy carbon disk electrode, Pt electrode as counter electrode and Ag/AgCl as reference electrode, at a 200 mVS<sup>-1</sup>; (graph F): **1a** (5 mM) + **2a** (5 mM)+ *n*-Bu<sub>4</sub>NPF<sub>6</sub> (0.1 M), KI (10 mM) in acetonitrile solvent.

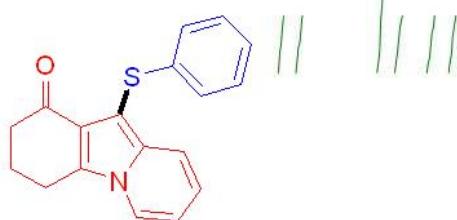
### Cyclic Voltammograms graph for **6a+2a**



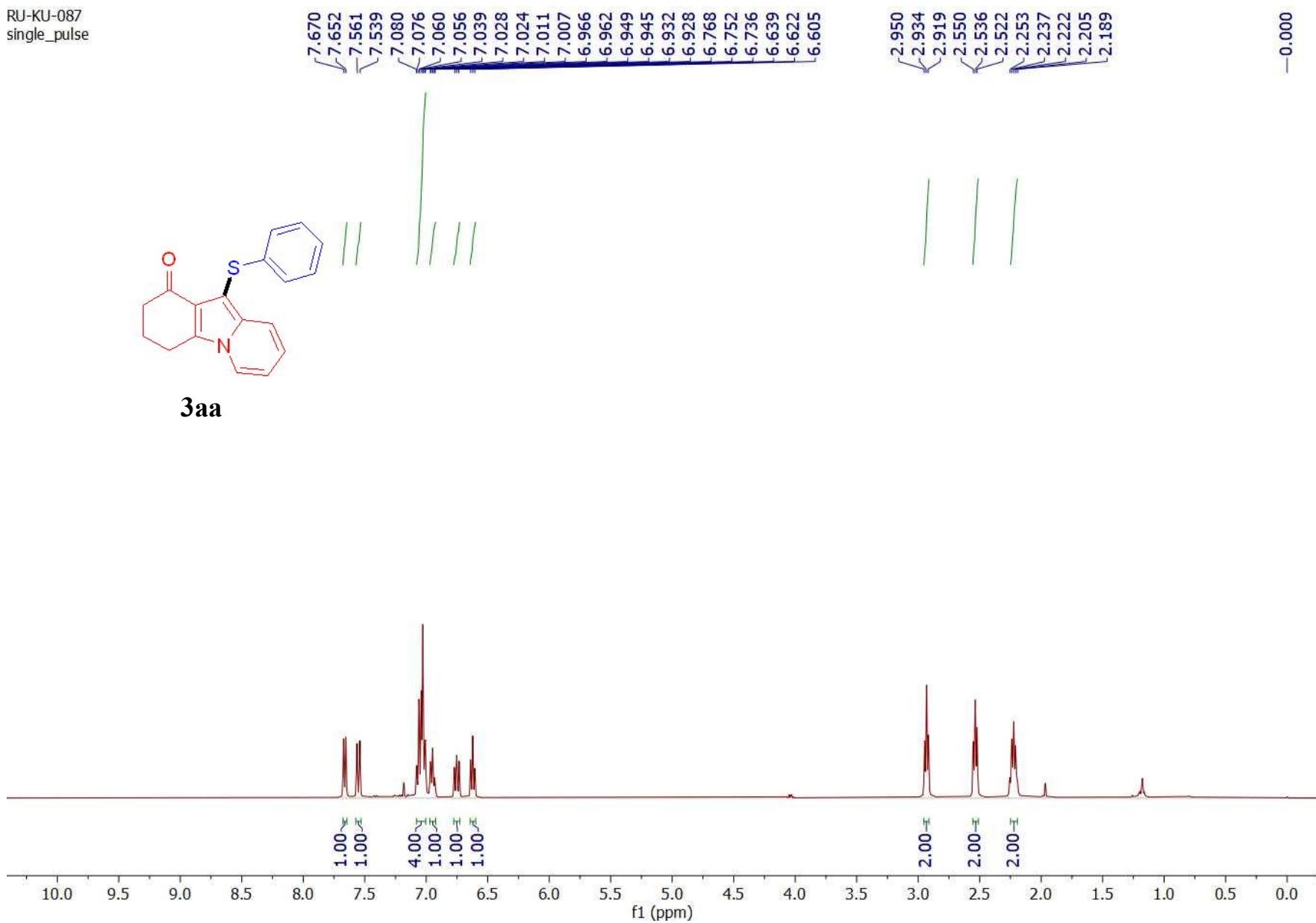
**Figure S6.** Cyclic voltammograms of reactants and mixture in 0.1 M *n*-Bu<sub>4</sub>NPF<sub>6</sub> in CH<sub>3</sub>CN using a glassy carbon disk electrode, Pt electrode as counter electrode and Ag/AgCl as reference electrode, at a 200 mVS<sup>-1</sup>; (graph F): **6a** (5 mM) + **2a** (5 mM)+ *n*-Bu<sub>4</sub>NPF<sub>6</sub> (0.1 M), KI (10 mM) in acetonitrile solvent.

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

RU-KU-087  
single\_pulse



**3aa**



<sup>13</sup>C{H} NMR spectrum of 3aa (100 MHz, CDCl<sub>3</sub>)

RU-KU-087  
single pulse decoupled gated NOE  
— 194.88

139.49  
136.58  
133.50  
128.59  
126.57  
124.91  
123.15  
122.74  
120.37  
119.38  
113.34

— 95.92

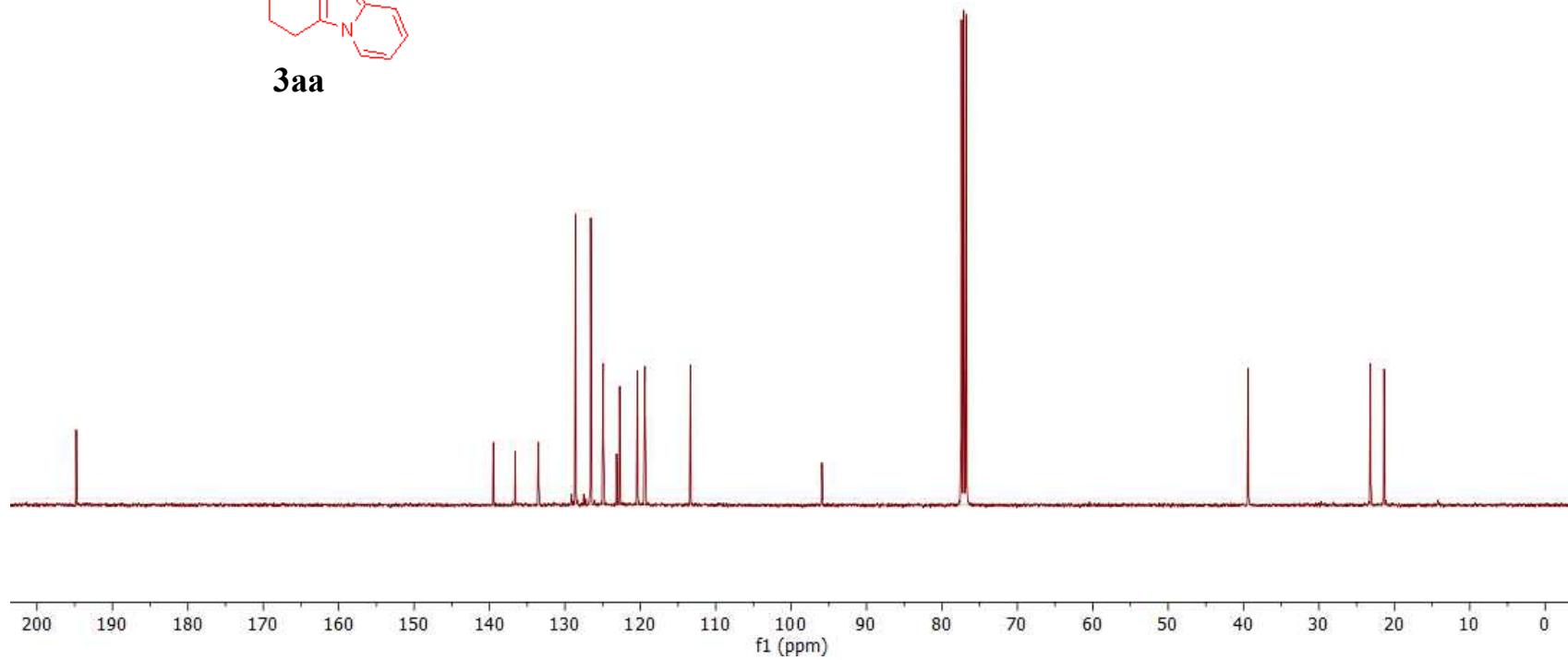
77.42  
77.10  
76.78

— 39.39

— 23.17  
— 21.37

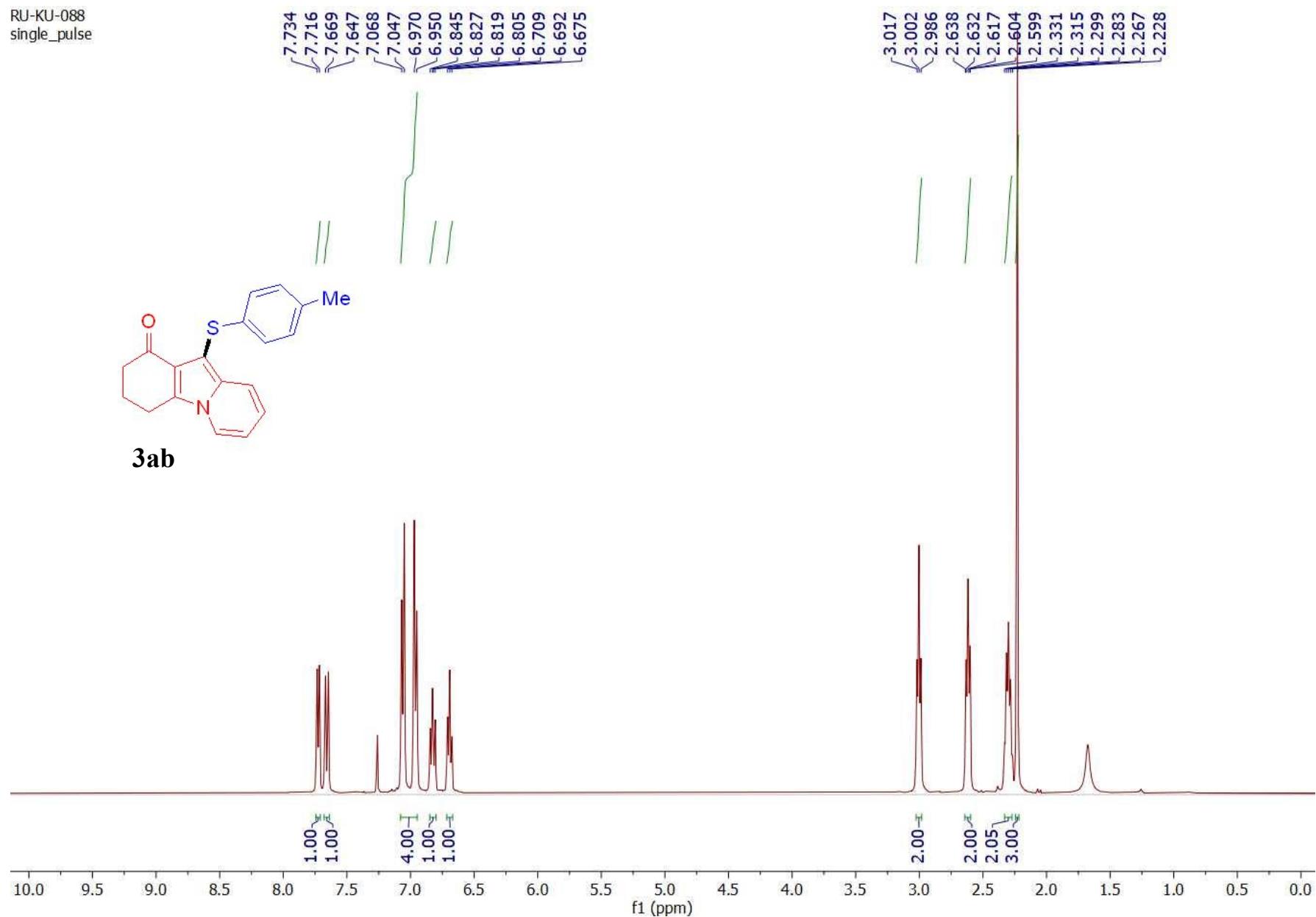


**3aa**



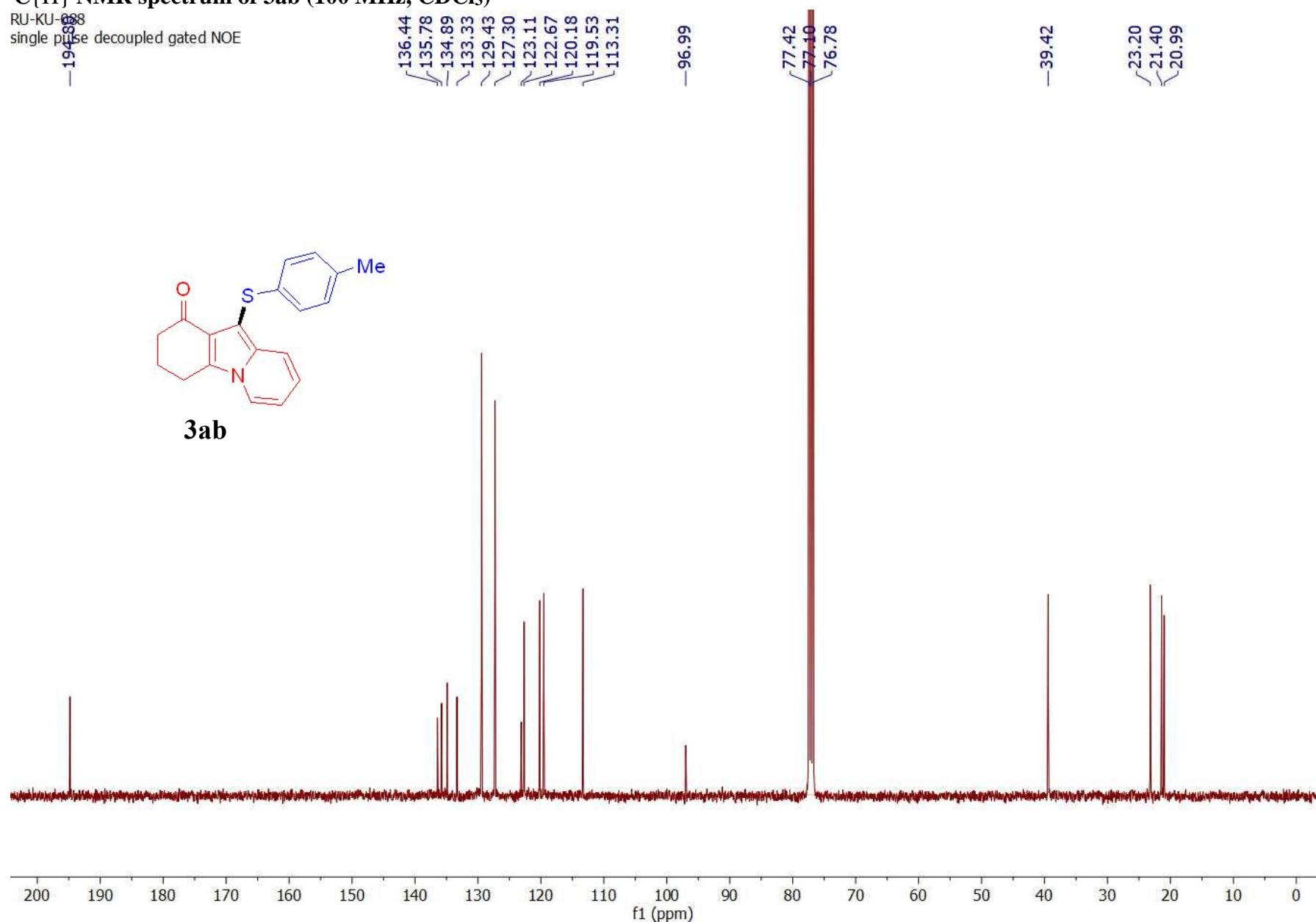
<sup>1</sup>H NMR spectrum of 3ab (400 MHz, CDCl<sub>3</sub>)

RU-KU-088  
single\_pulse



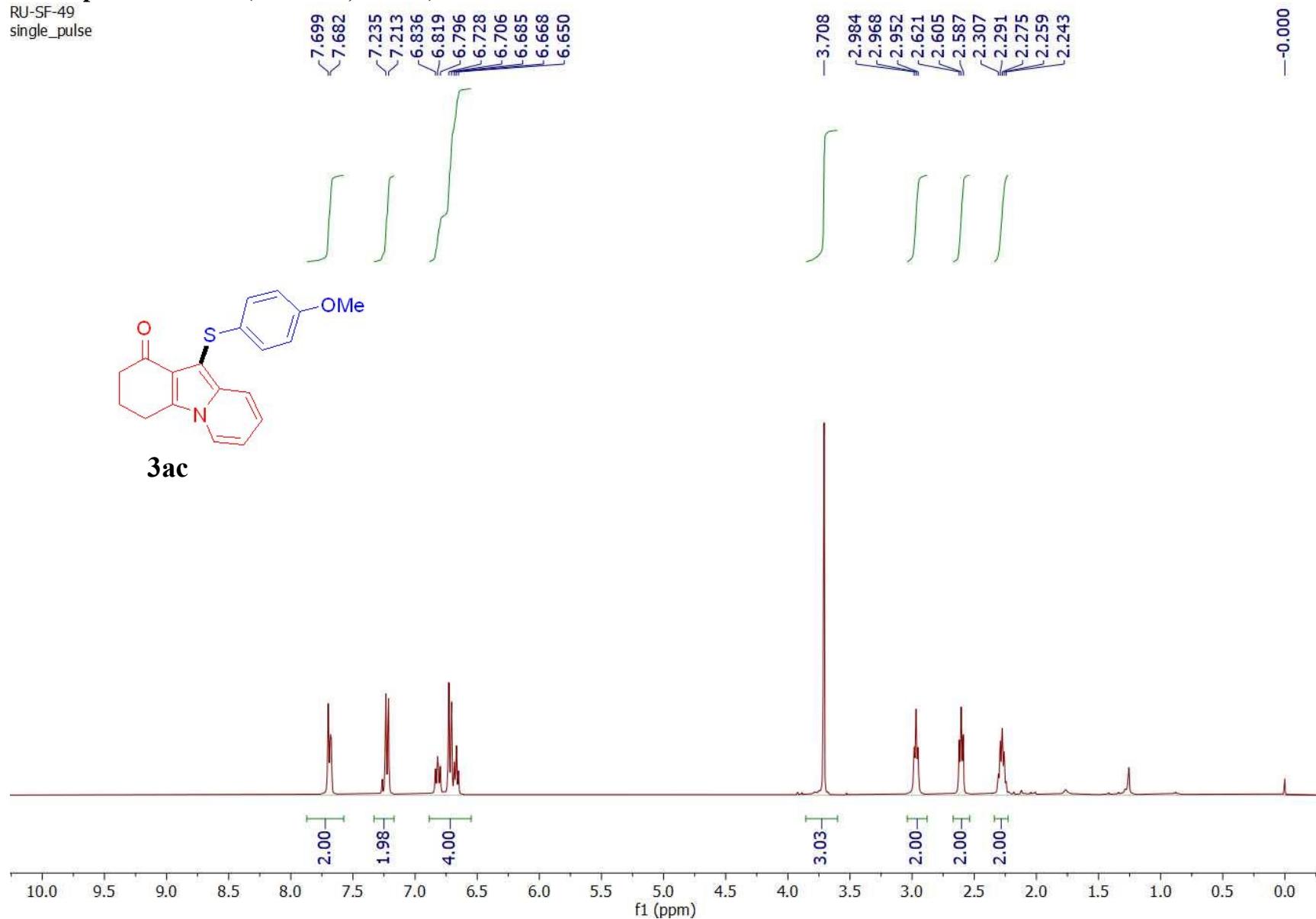
<sup>13</sup>C{H} NMR spectrum of 3ab (100 MHz, CDCl<sub>3</sub>)

RU-KU-088  
single pulse decoupled gated NOE



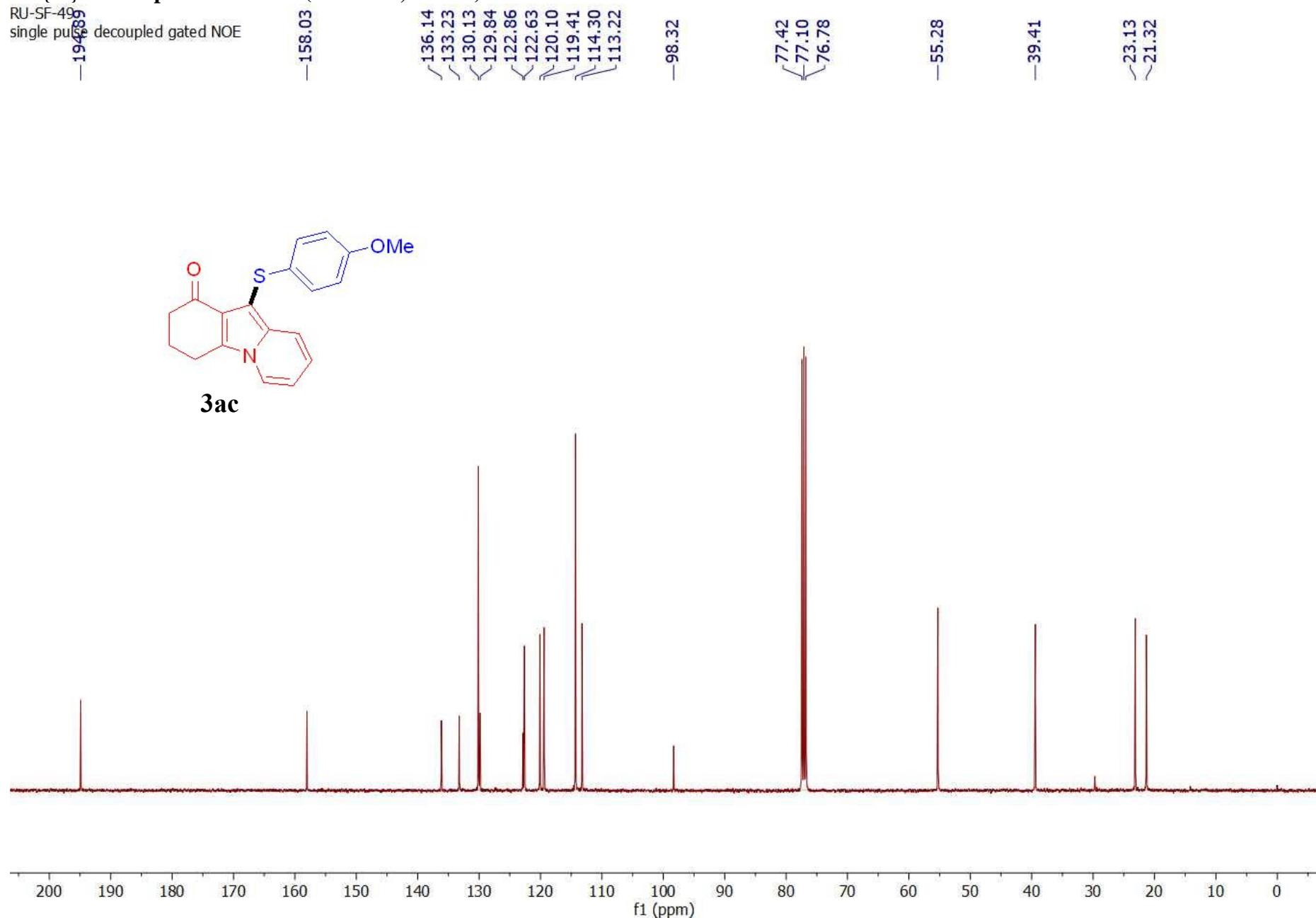
**<sup>1</sup>H NMR spectrum of 3ac (400 MHz, CDCl<sub>3</sub>)**

RU-SF-49  
single\_pulse



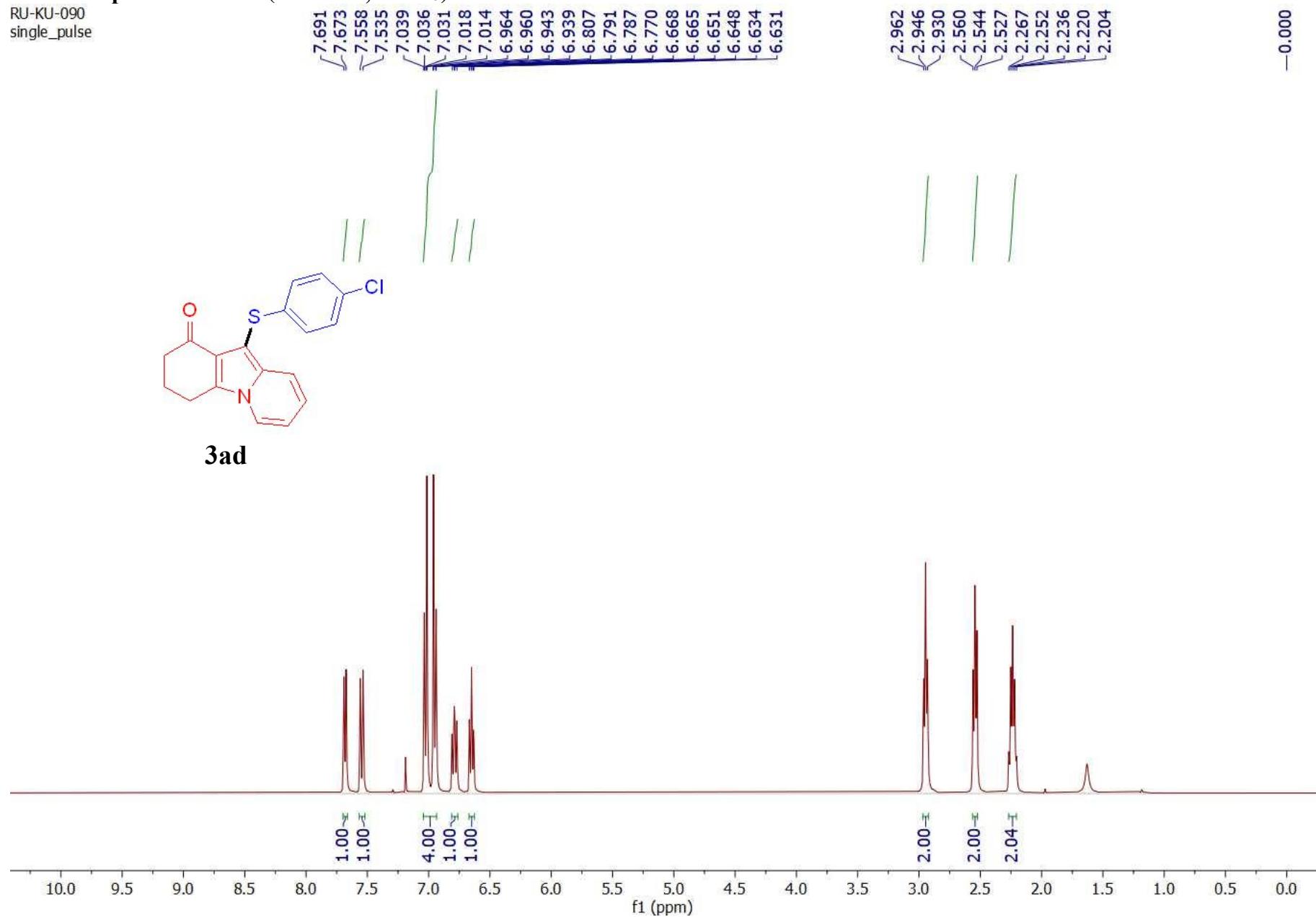
$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 3ac (100 MHz,  $\text{CDCl}_3$ )

RU-SF-499  
single pulse decoupled gated NOE



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

RU-KU-090  
single\_pulse



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 3ad (100 MHz,  $\text{CDCl}_3$ )

RU-KU-000  
single pulse decoupled gated NOE

-194.99

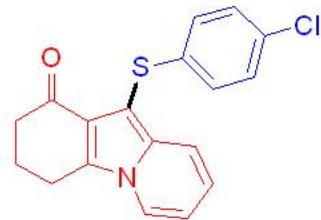
138.18  
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128.68  
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122.83  
120.69  
119.21  
113.46

-95.43

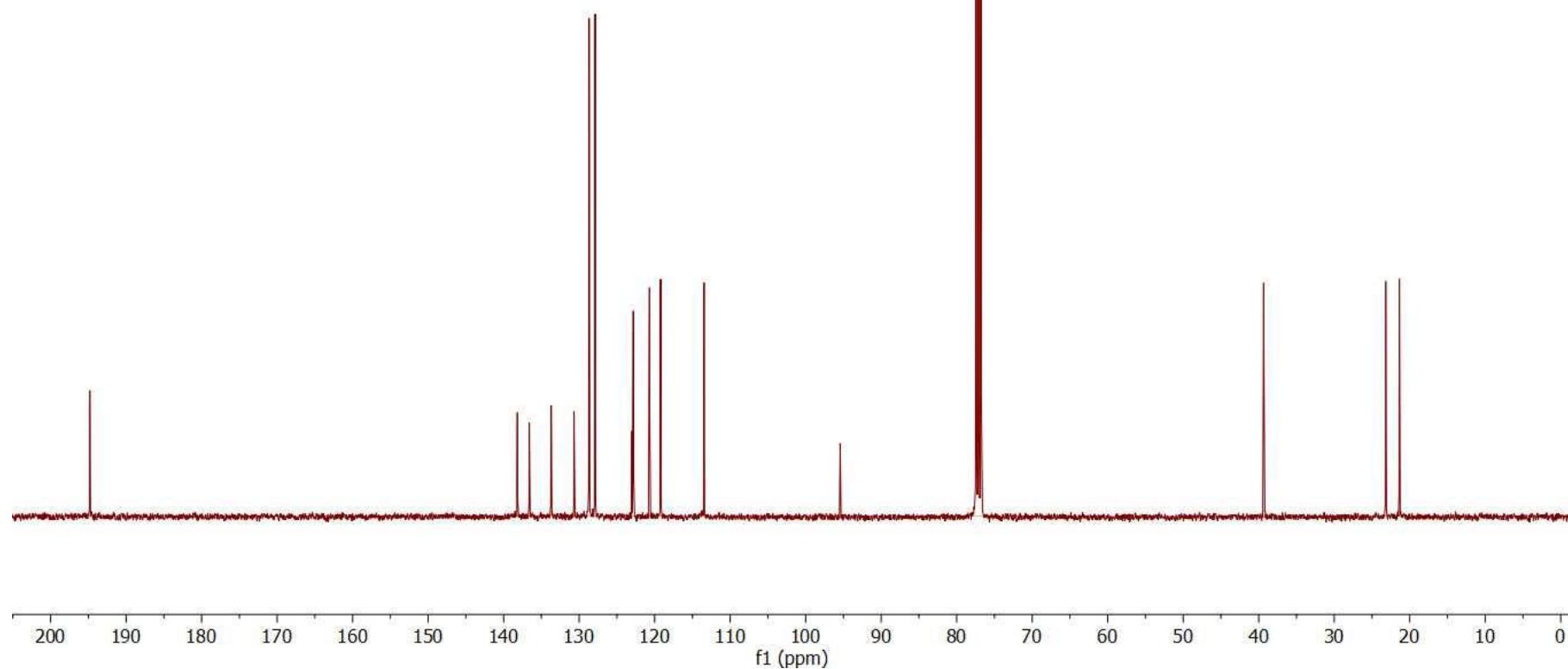
77.42  
77.10  
76.78

-39.35

23.18  
21.37

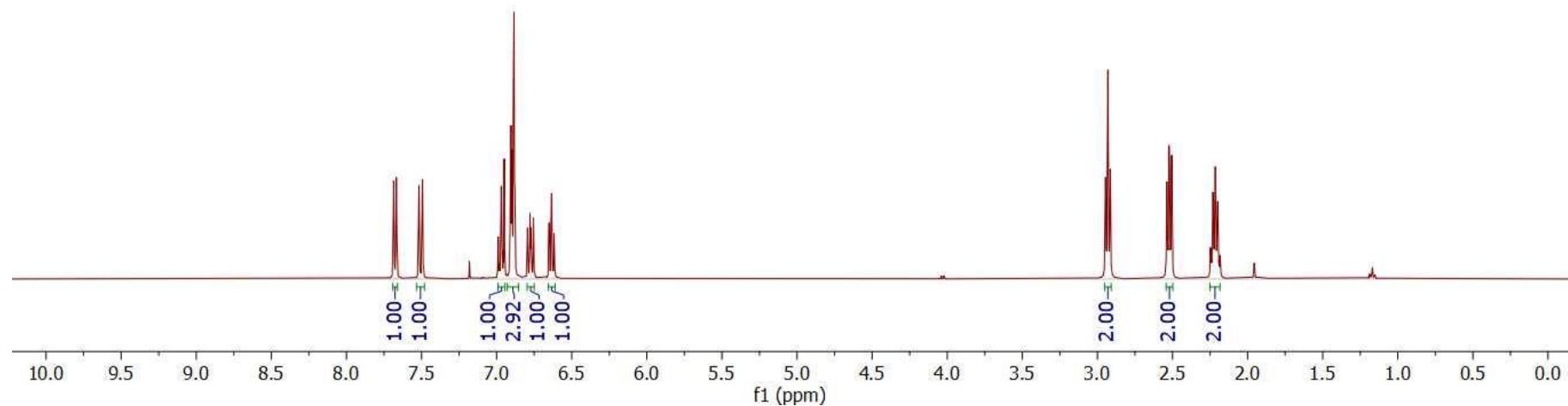
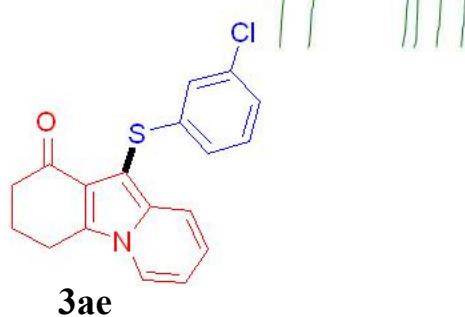
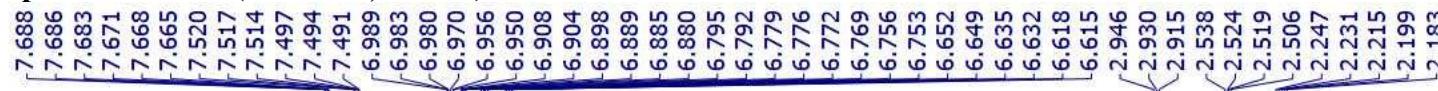


3ad



<sup>1</sup>H NMR spectrum of 3ae (400 MHz, CDCl<sub>3</sub>)

RU-KU-095  
single\_pulse



<sup>13</sup>C{H} NMR spectrum of 3ae (100 MHz, CDCl<sub>3</sub>)

RU-KU-095  
single pulse decoupled gated NOE

-194.75

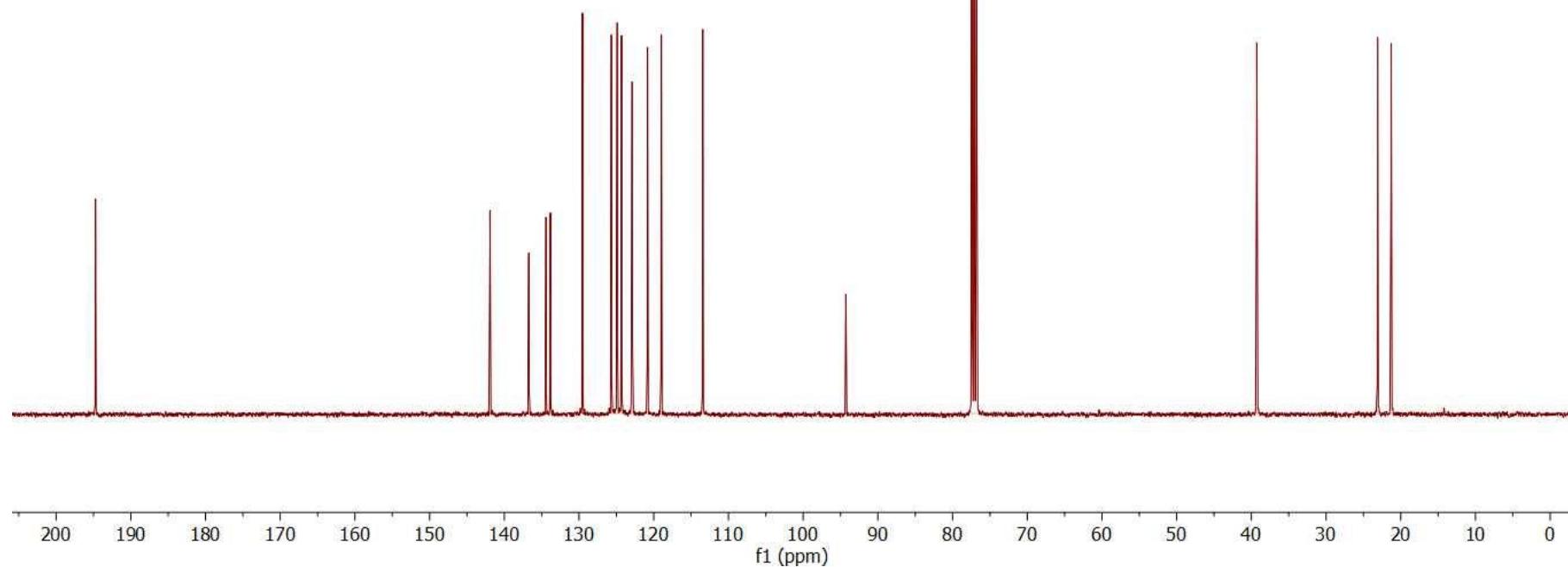
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118.99  
113.44

—94.29

77.42  
77.10  
76.78

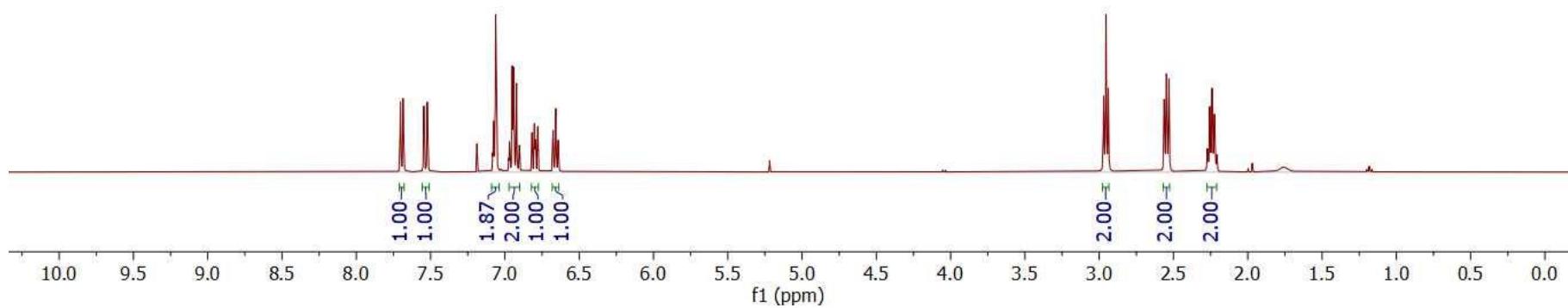
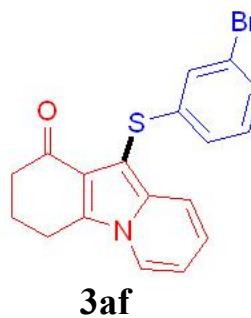
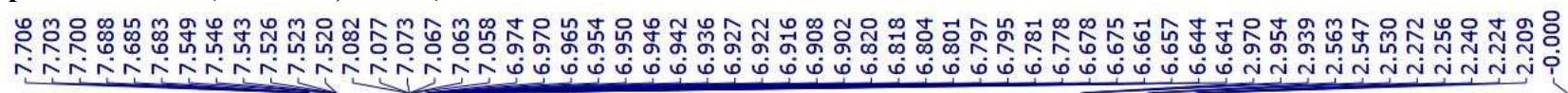
—39.27

~23.10  
~21.29



<sup>1</sup>H NMR spectrum of 3af (400 MHz, CDCl<sub>3</sub>)

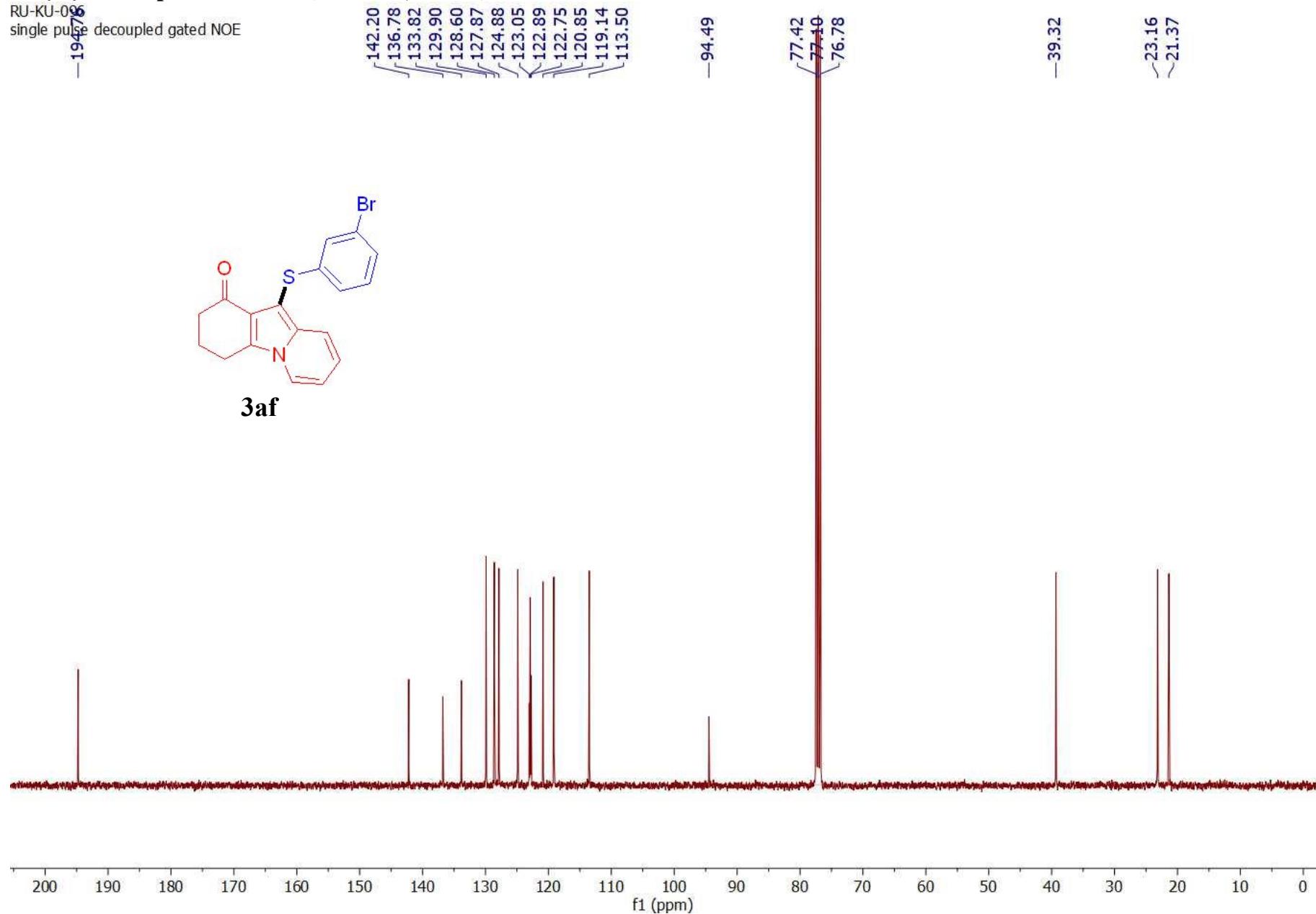
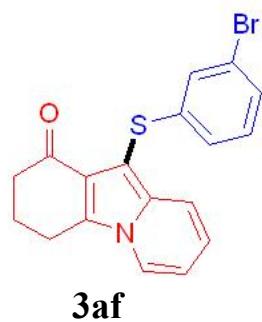
RU-KU-096  
single\_pulse



<sup>13</sup>C{H} NMR spectrum of 3af (100 MHz, CDCl<sub>3</sub>)

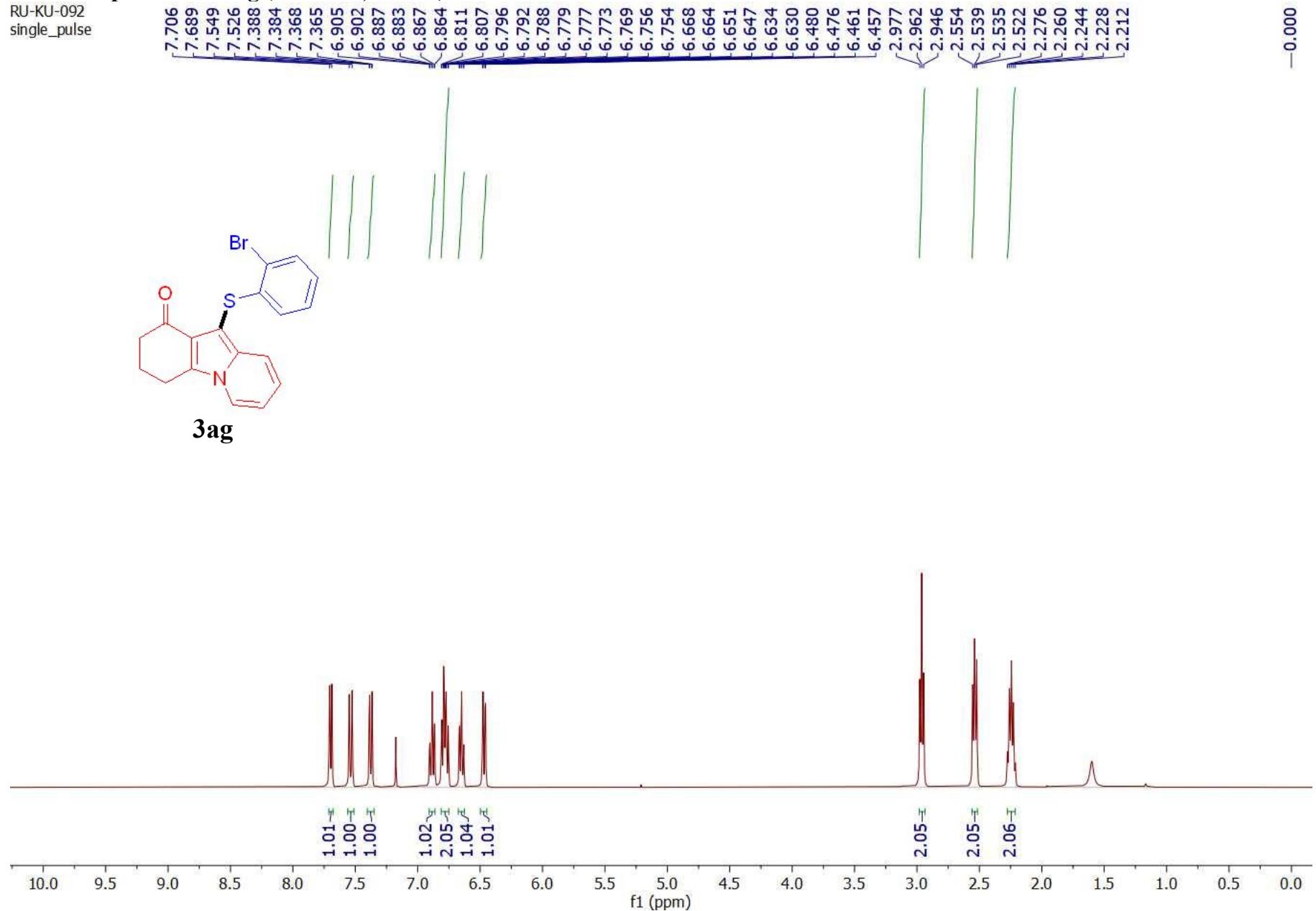
RU-KU-096  
single pulse decoupled gated NOE

-194.76



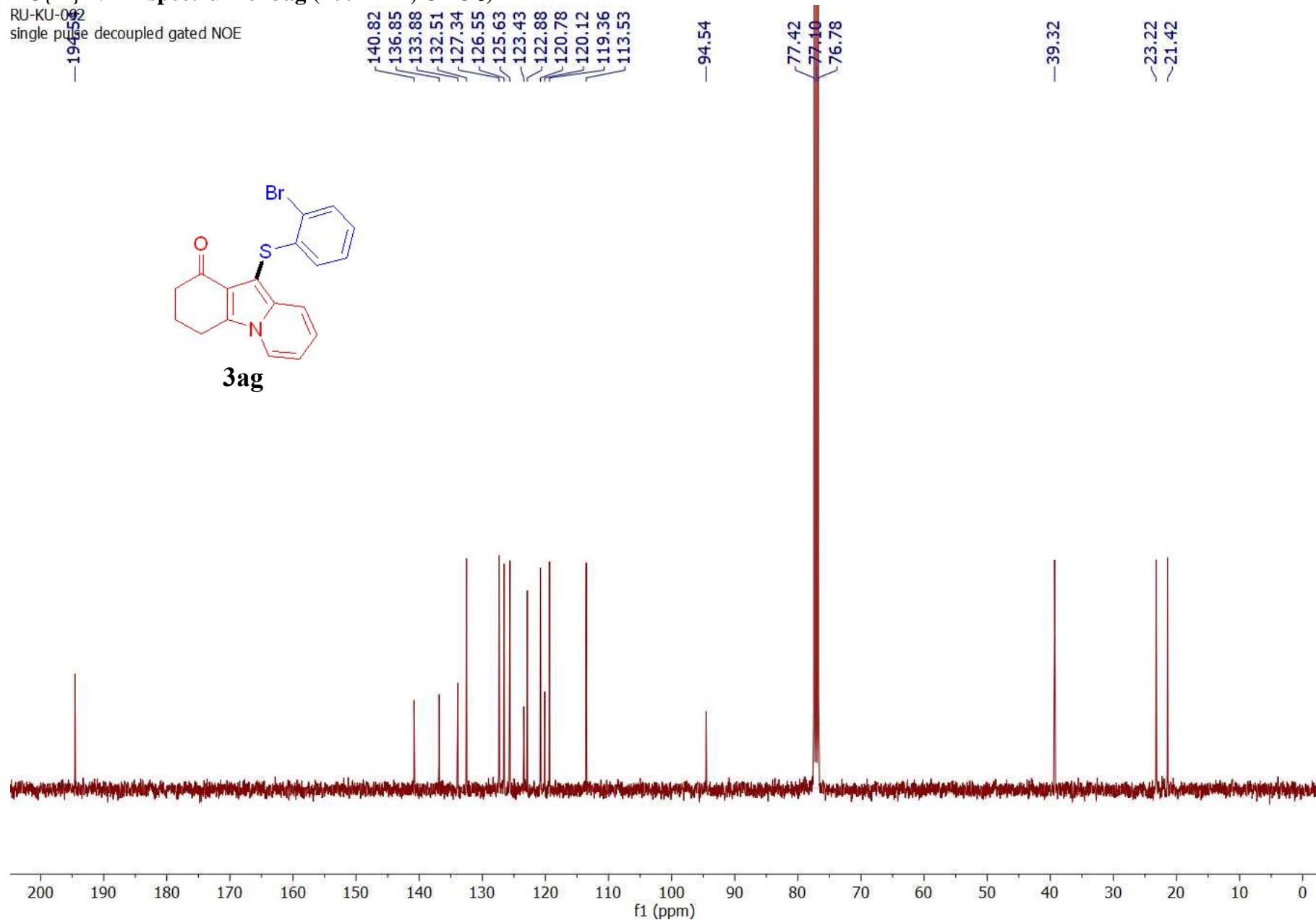
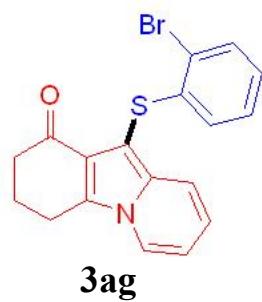
<sup>1</sup>H NMR spectrum of 3ag (400 MHz, CDCl<sub>3</sub>)

RU-KU-092  
single\_pulse



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 3ag (100 MHz,  $\text{CDCl}_3$ )

RU-KU-092  
single pulse decoupled gated NOE  
~~-194.52~~

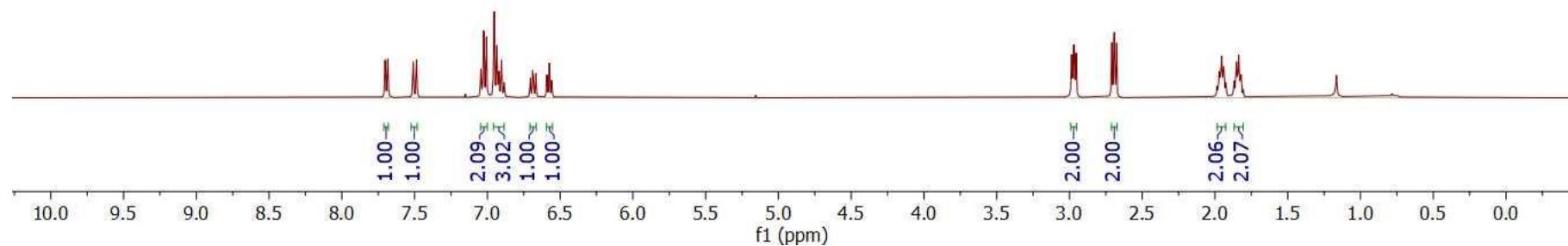
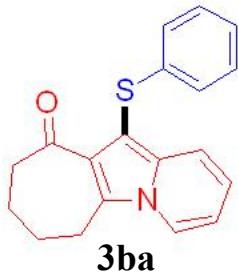


<sup>1</sup>H NMR spectrum of 3ba (400 MHz, CDCl<sub>3</sub>)

RU-KU-101  
single\_pulse



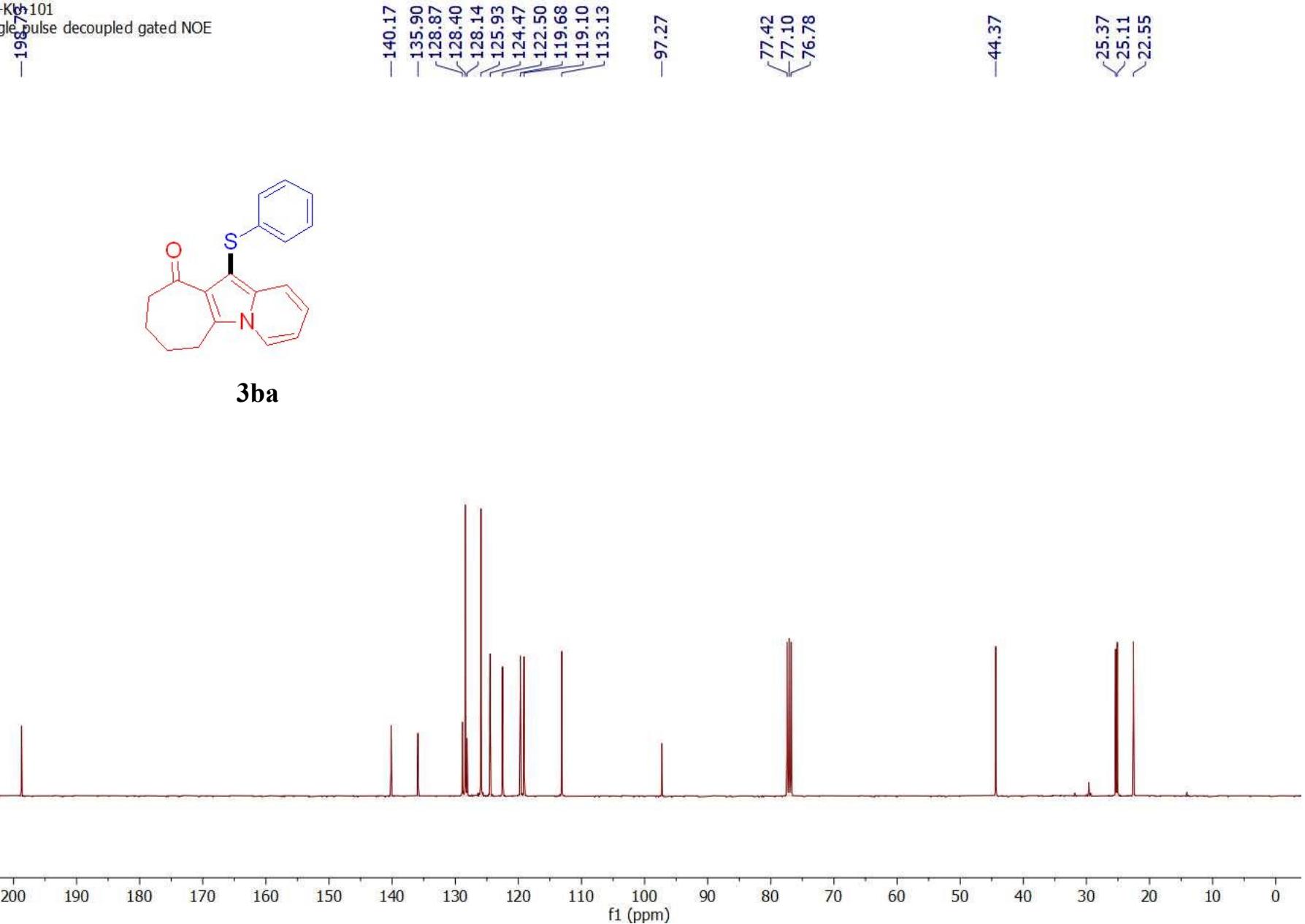
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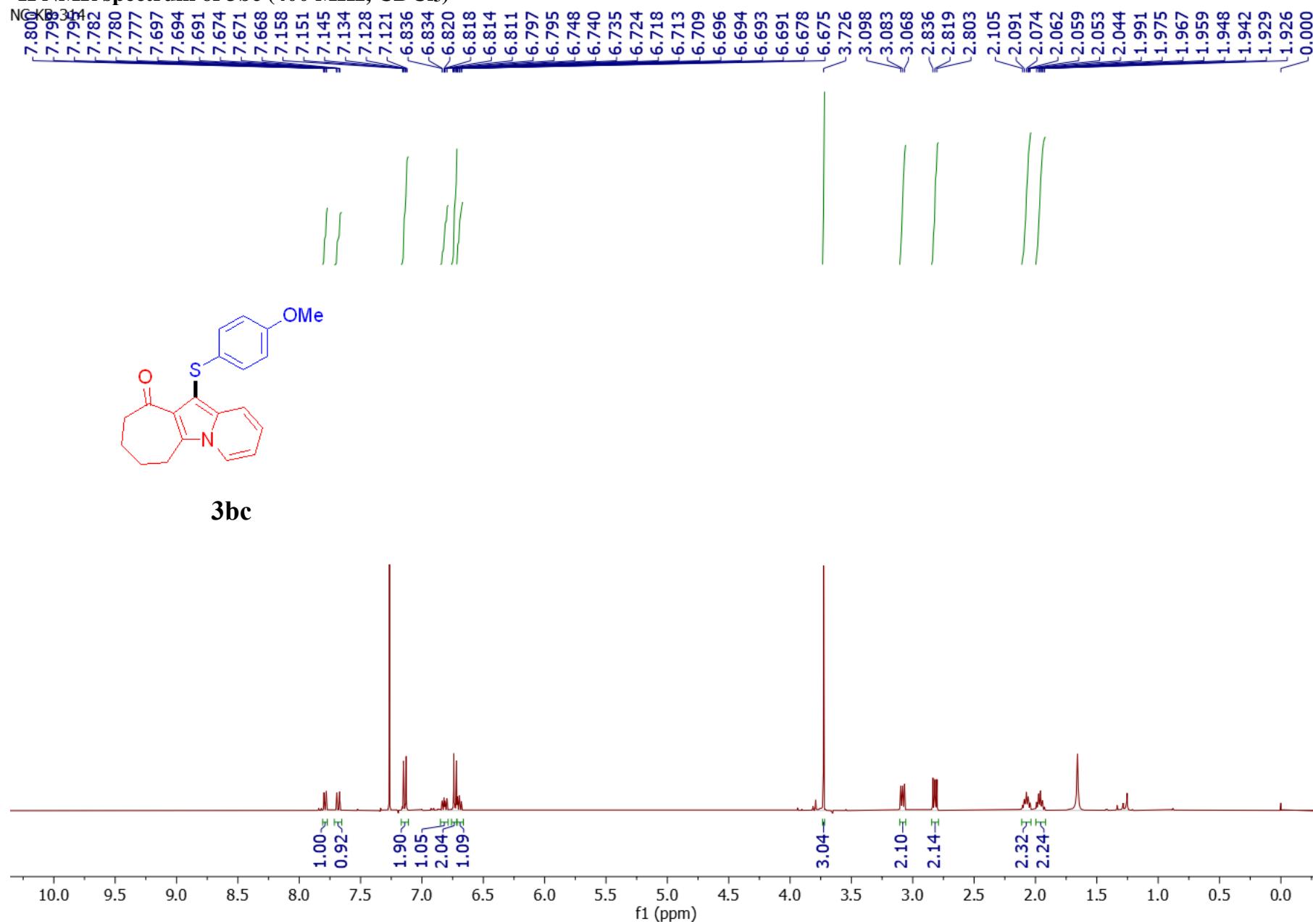
<sup>13</sup>C{H} NMR spectrum of 3ba (100 MHz, CDCl<sub>3</sub>)

RU-KN101

single pulse decoupled gated NOE

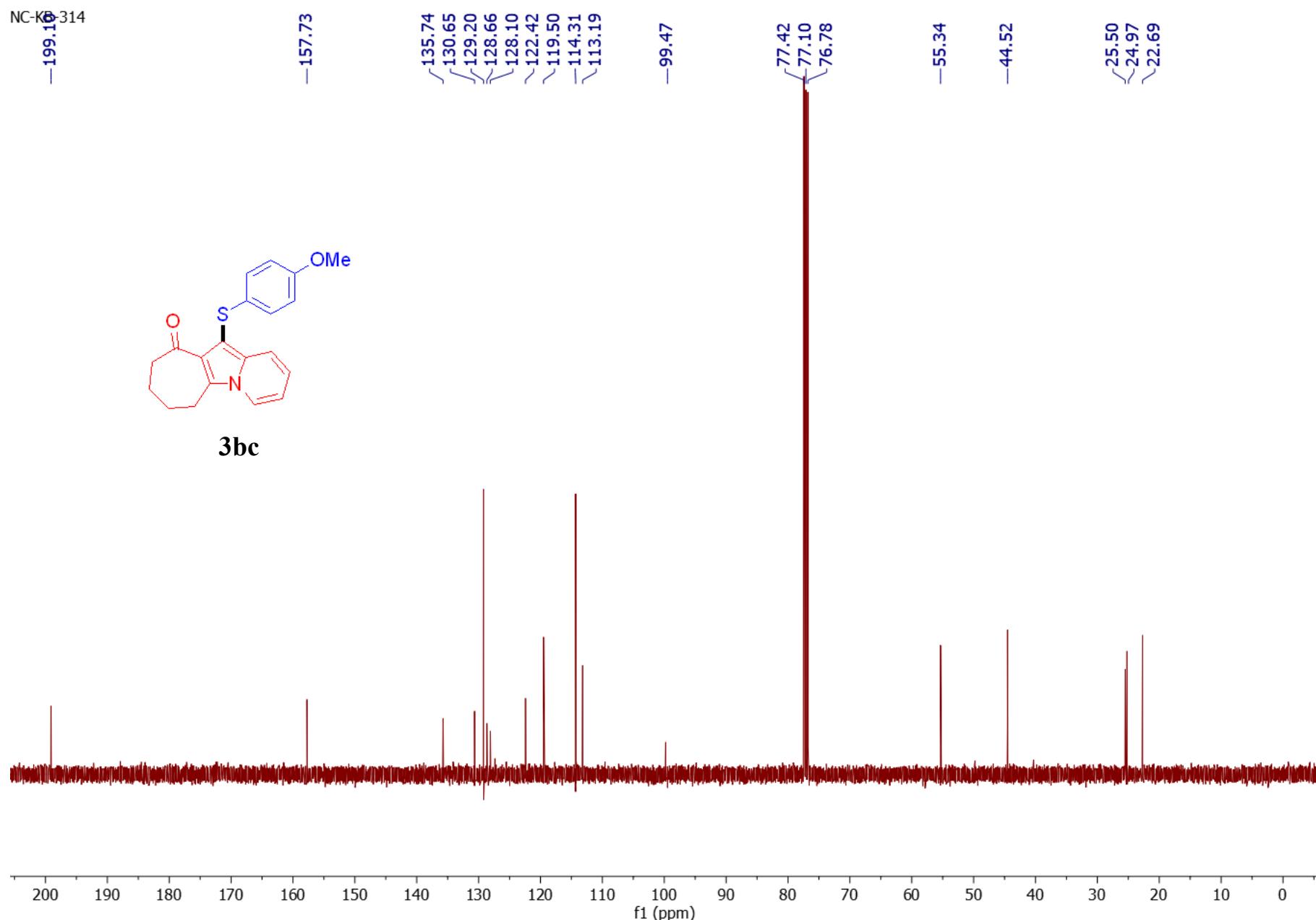


<sup>1</sup>H NMR spectrum of 3bc (400 MHz, CDCl<sub>3</sub>)



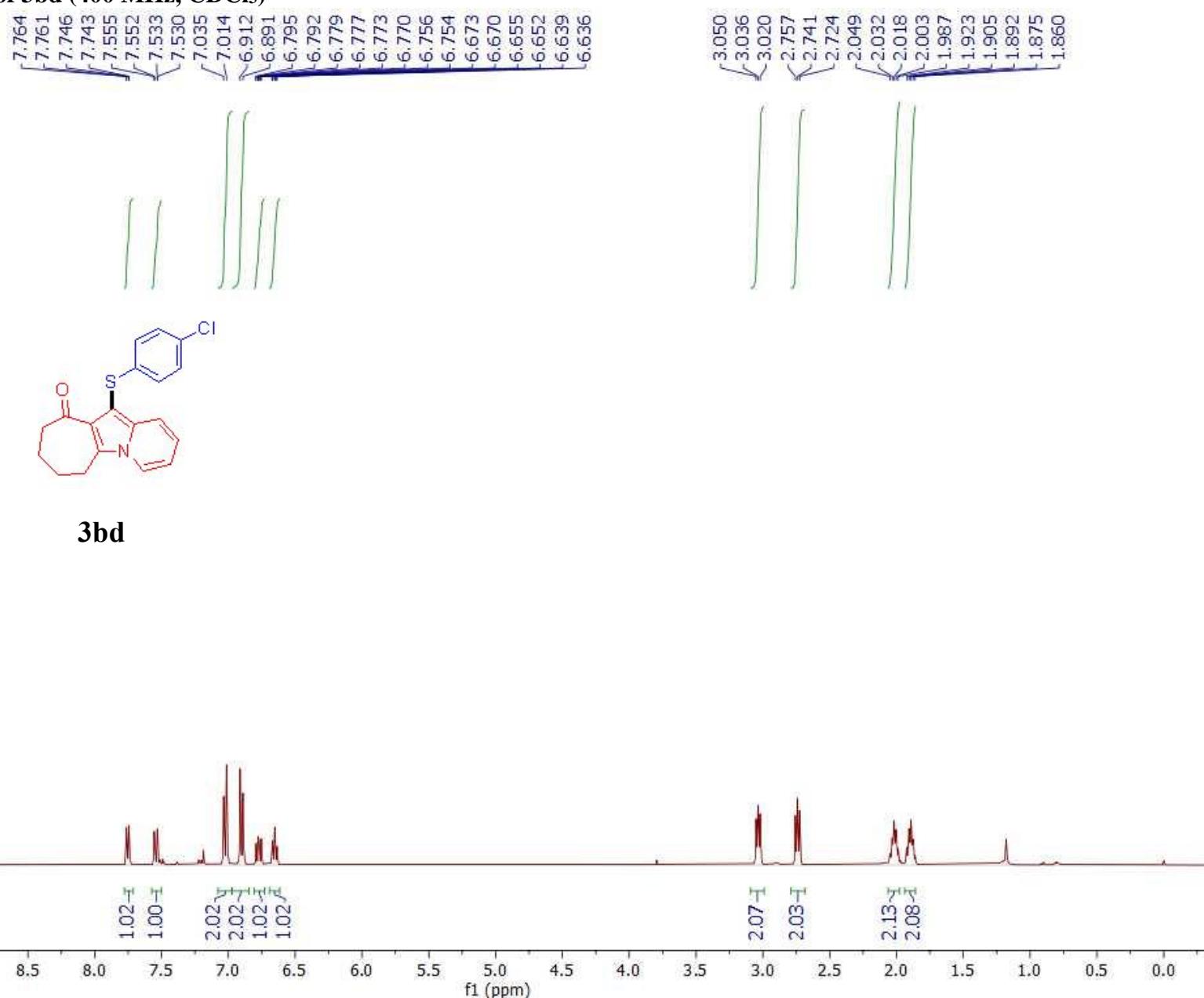
$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 3bc (100 MHz,  $\text{CDCl}_3$ )

NC-KB-314



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

RU-KU-122  
single\_pulse

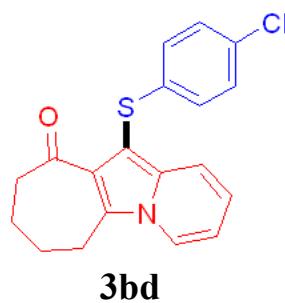


**3bd**

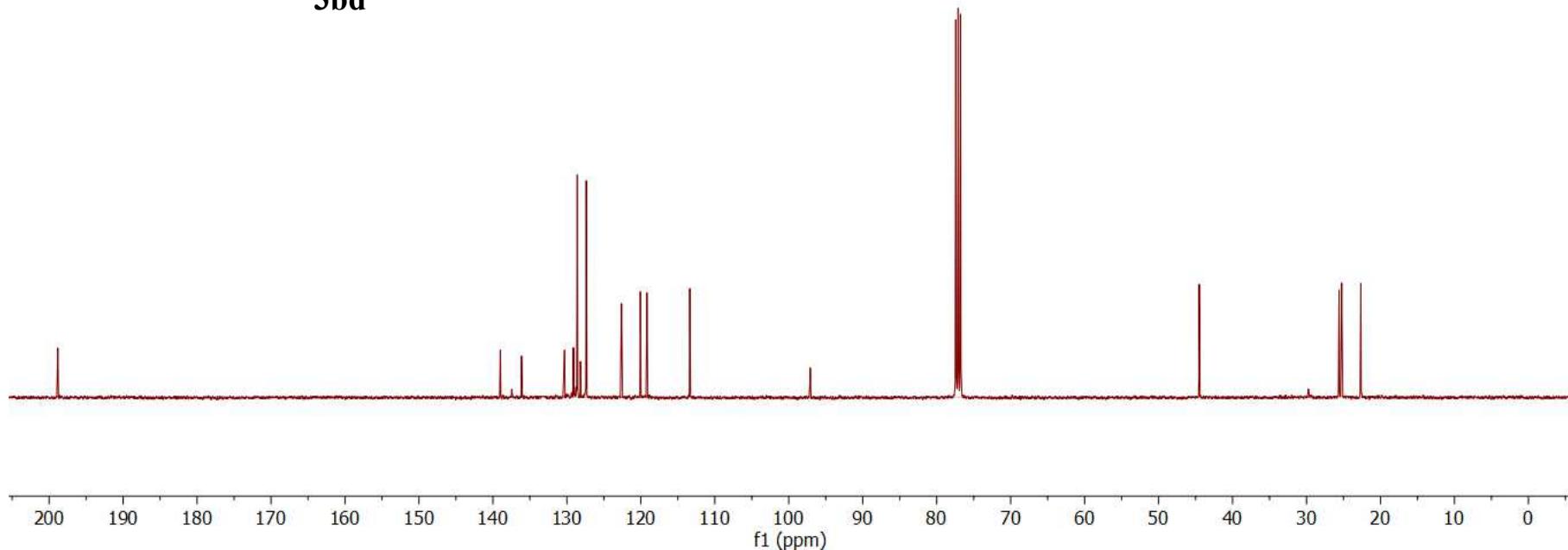
<sup>13</sup>C{H} NMR spectrum of 3bd (100 MHz, CDCl<sub>3</sub>)

RU-Kbr 122  
single pulse decoupled gated NOE  
—198.83

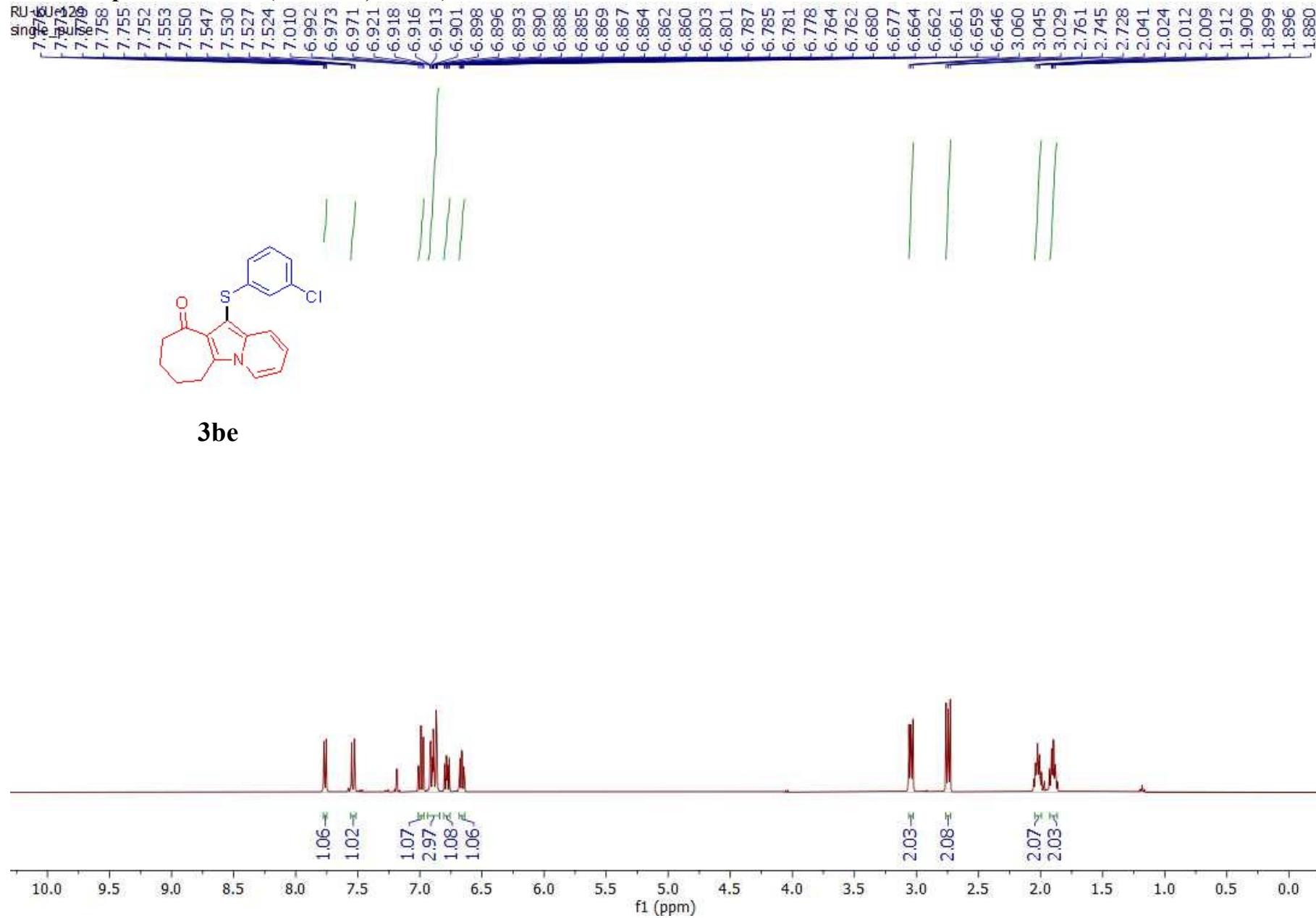
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128.59  
128.16  
127.38  
122.61  
120.06  
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113.38  
—97.10  
77.42  
77.10  
76.78  
—44.51  
25.60  
25.25  
22.65



**3bd**



<sup>1</sup>H NMR spectrum of 3be (400 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C{H} NMR spectrum of 3be (100 MHz, CDCl<sub>3</sub>)

RU-KP<sub>129</sub>  
single pulse decoupled gated NOE  
—198.7

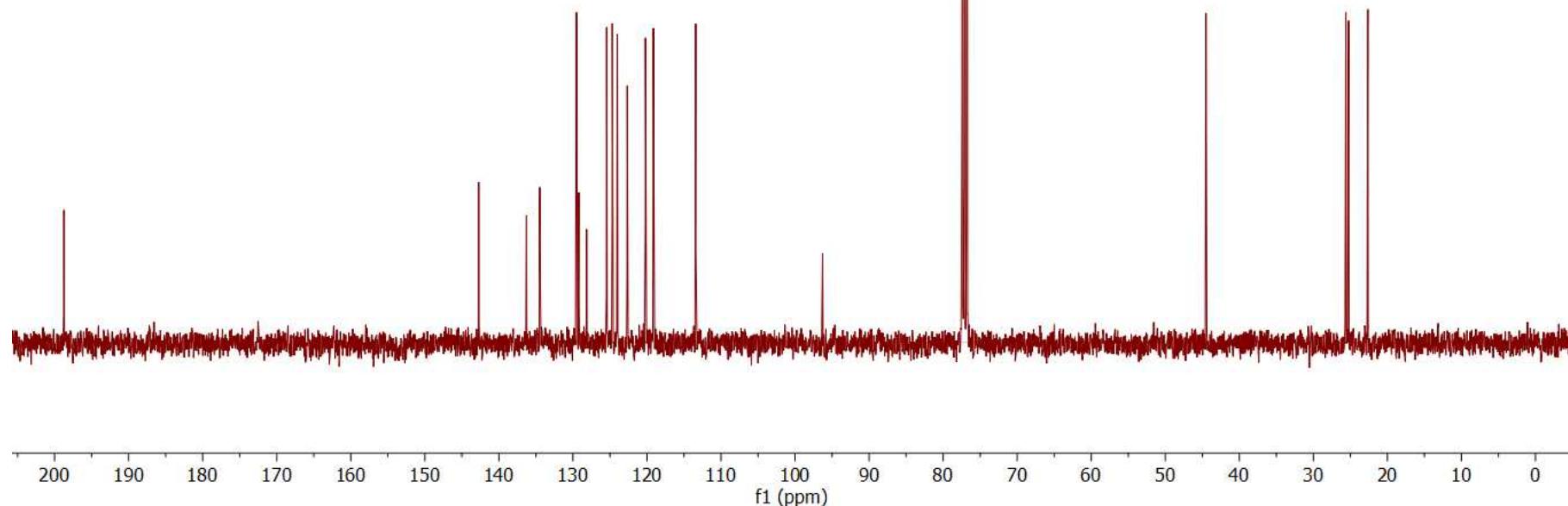
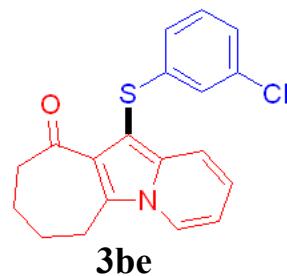
142.72  
136.27  
134.47  
129.51  
129.21  
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122.66  
120.20  
119.13  
113.41

—96.29

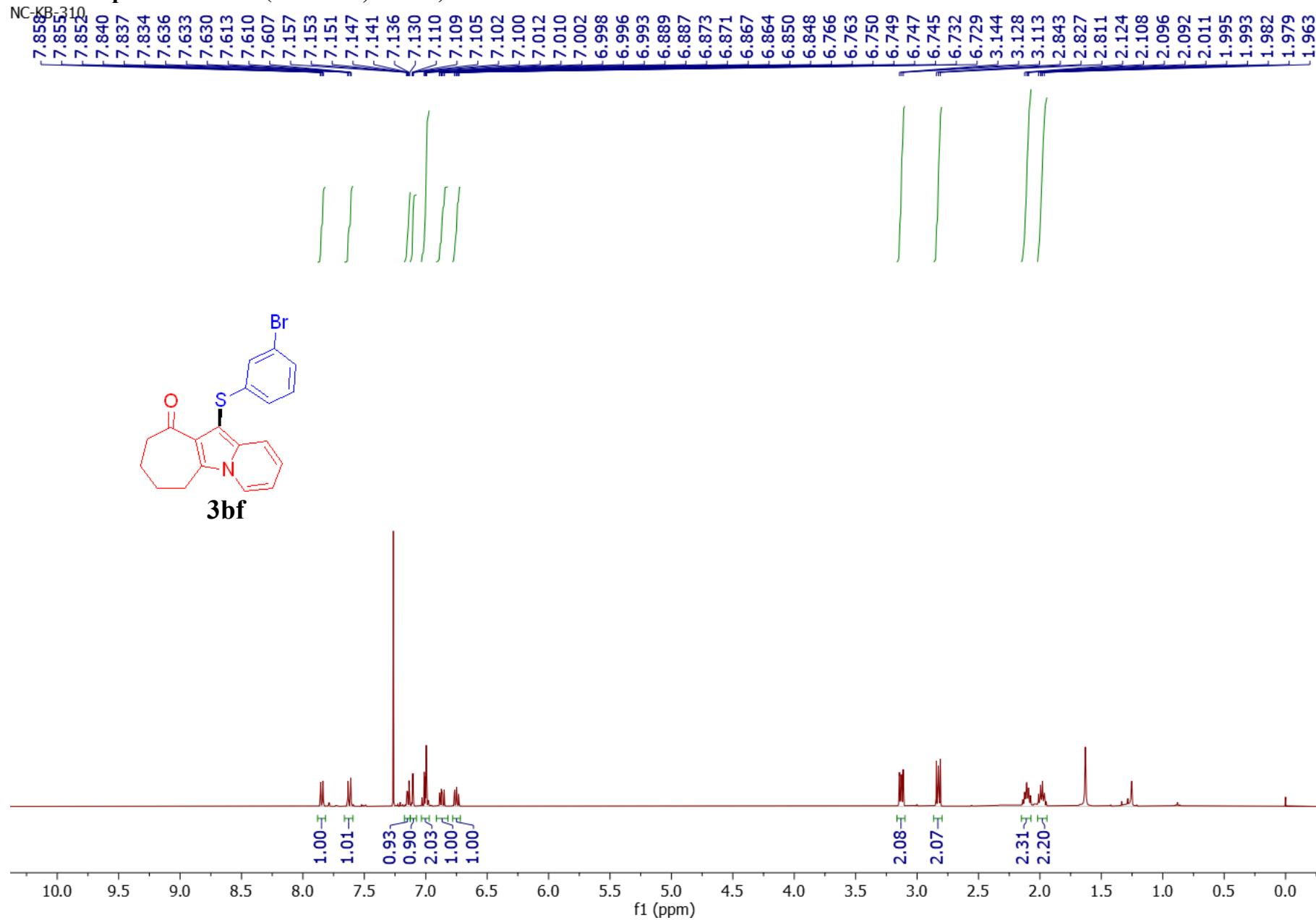
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77.10  
76.78

—44.50

25.62  
25.24  
22.64



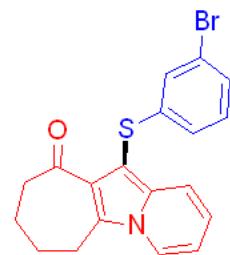
<sup>1</sup>H NMR spectrum of 3bf (400 MHz, CDCl<sub>3</sub>)



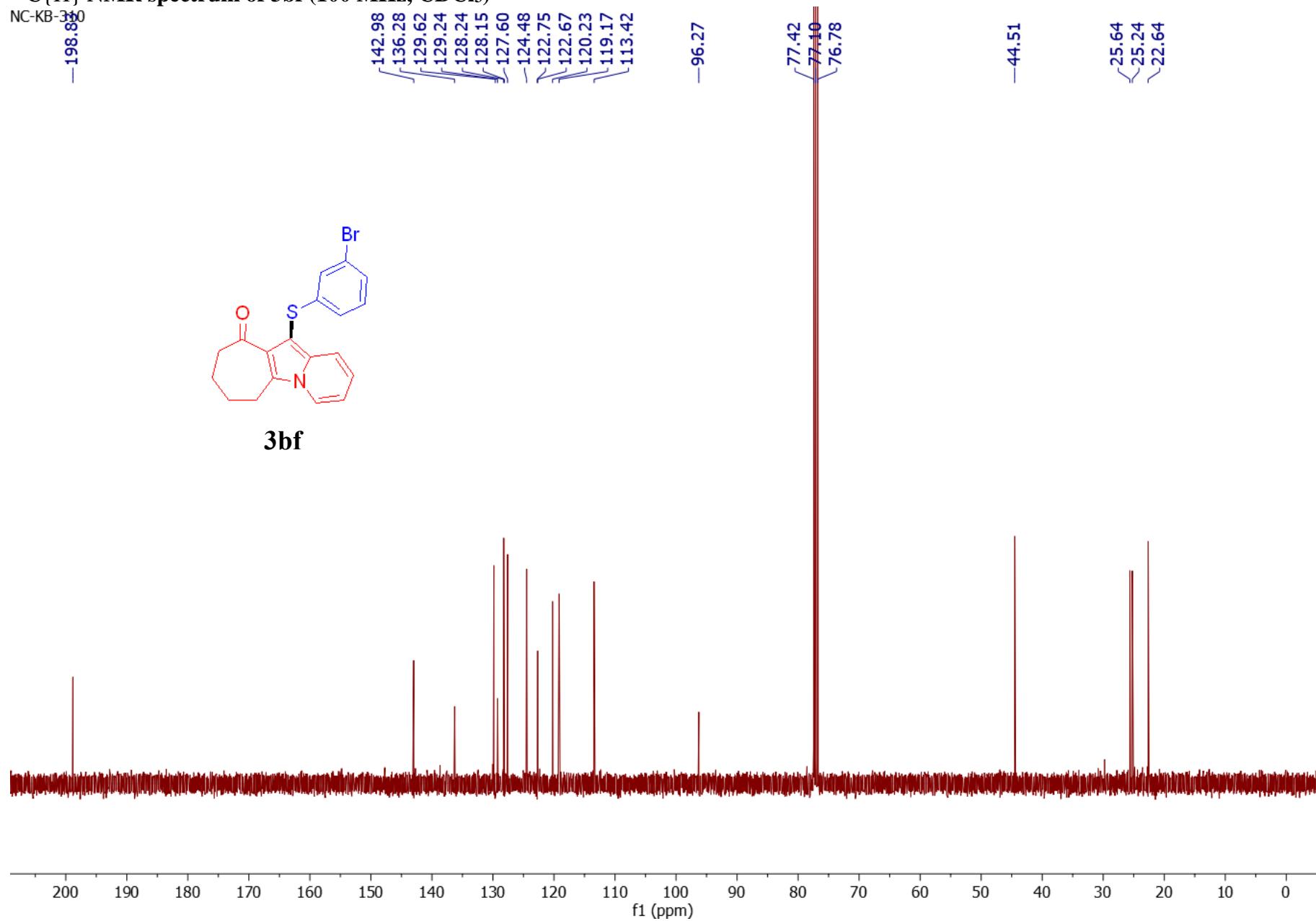
$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 3bf (100 MHz,  $\text{CDCl}_3$ )

NC-KB-340

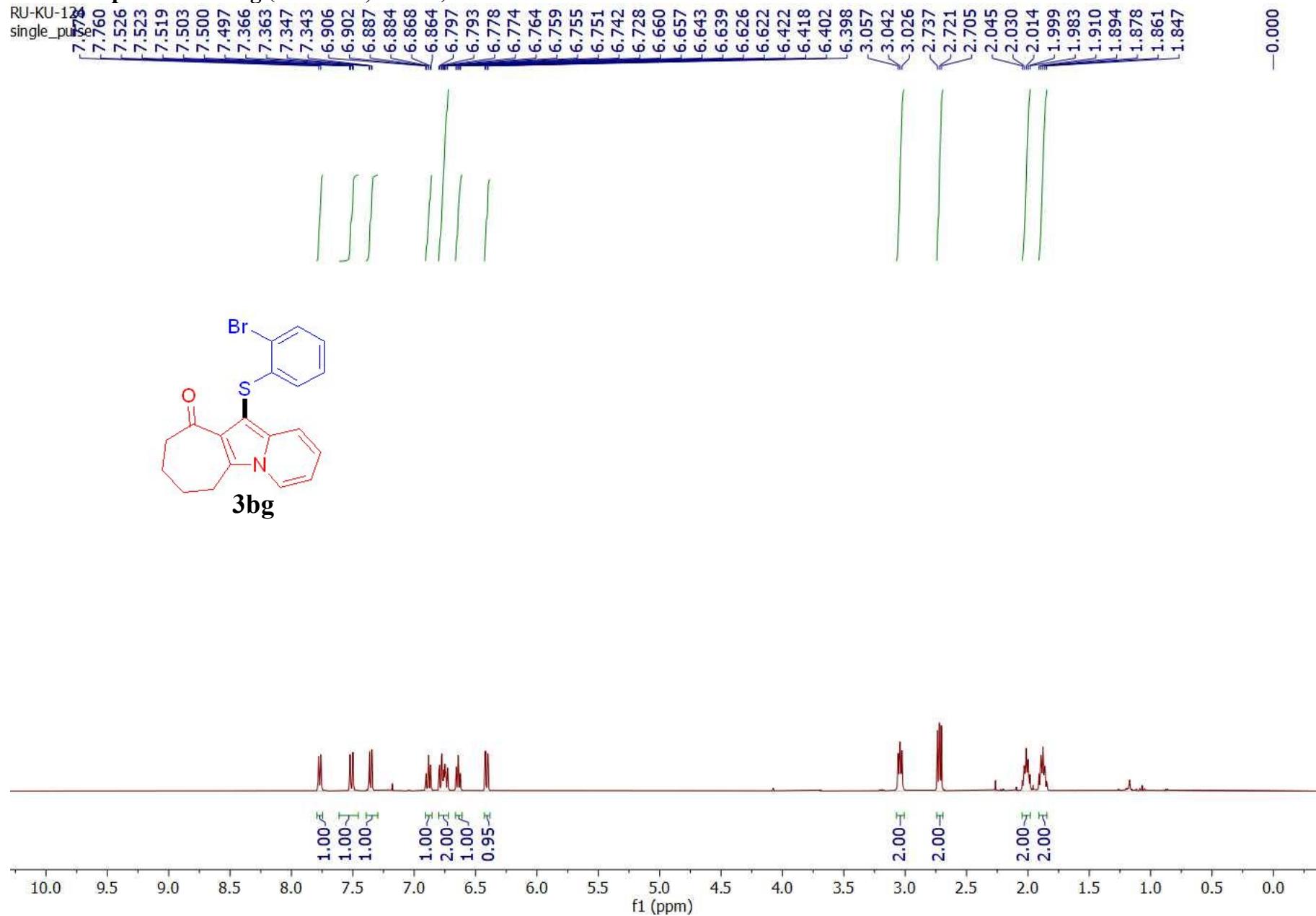
-198.87



3bf



<sup>1</sup>H NMR spectrum of 3bg (400 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 3bg (100 MHz,  $\text{CDCl}_3$ )

RU-101-124

single pulse decoupled gated NOE

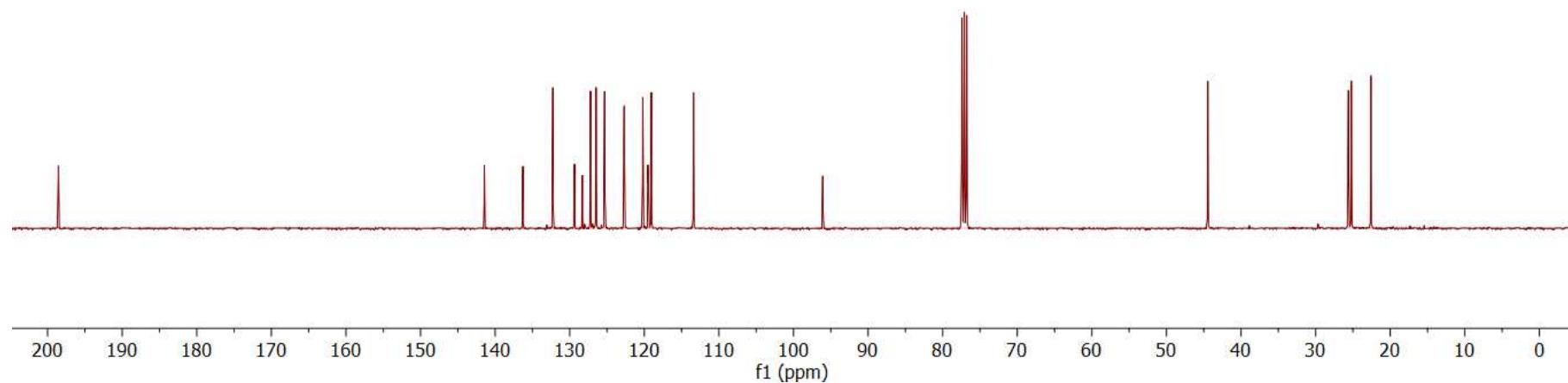
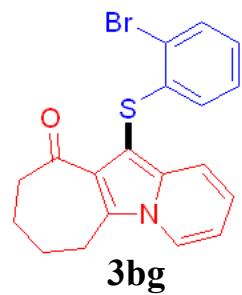
141.45  
136.27  
132.26  
129.36  
128.31  
127.21  
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119.52  
119.06

-96.08

77.42  
77.10  
76.78

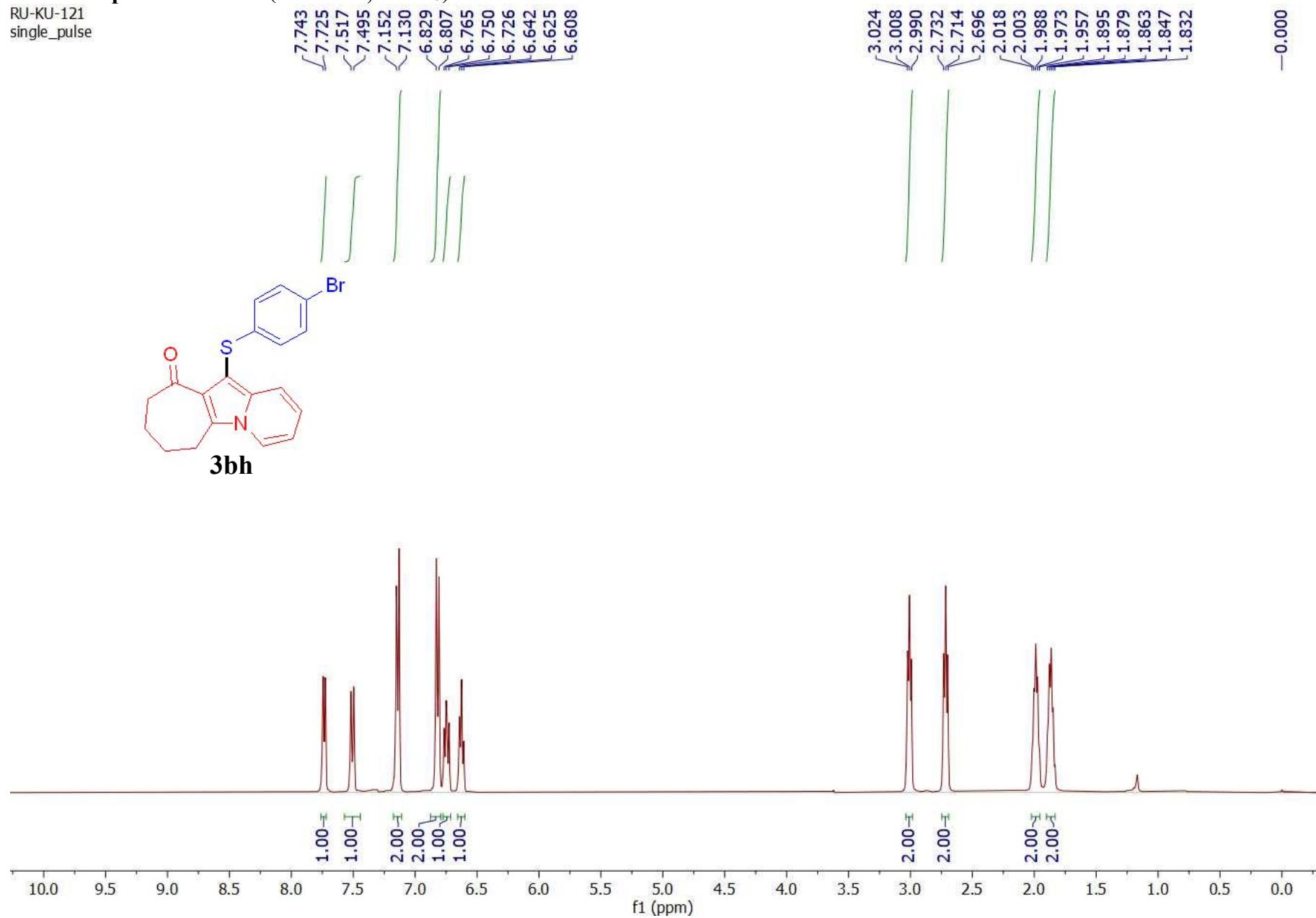
-44.46

25.60  
25.21  
22.59



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

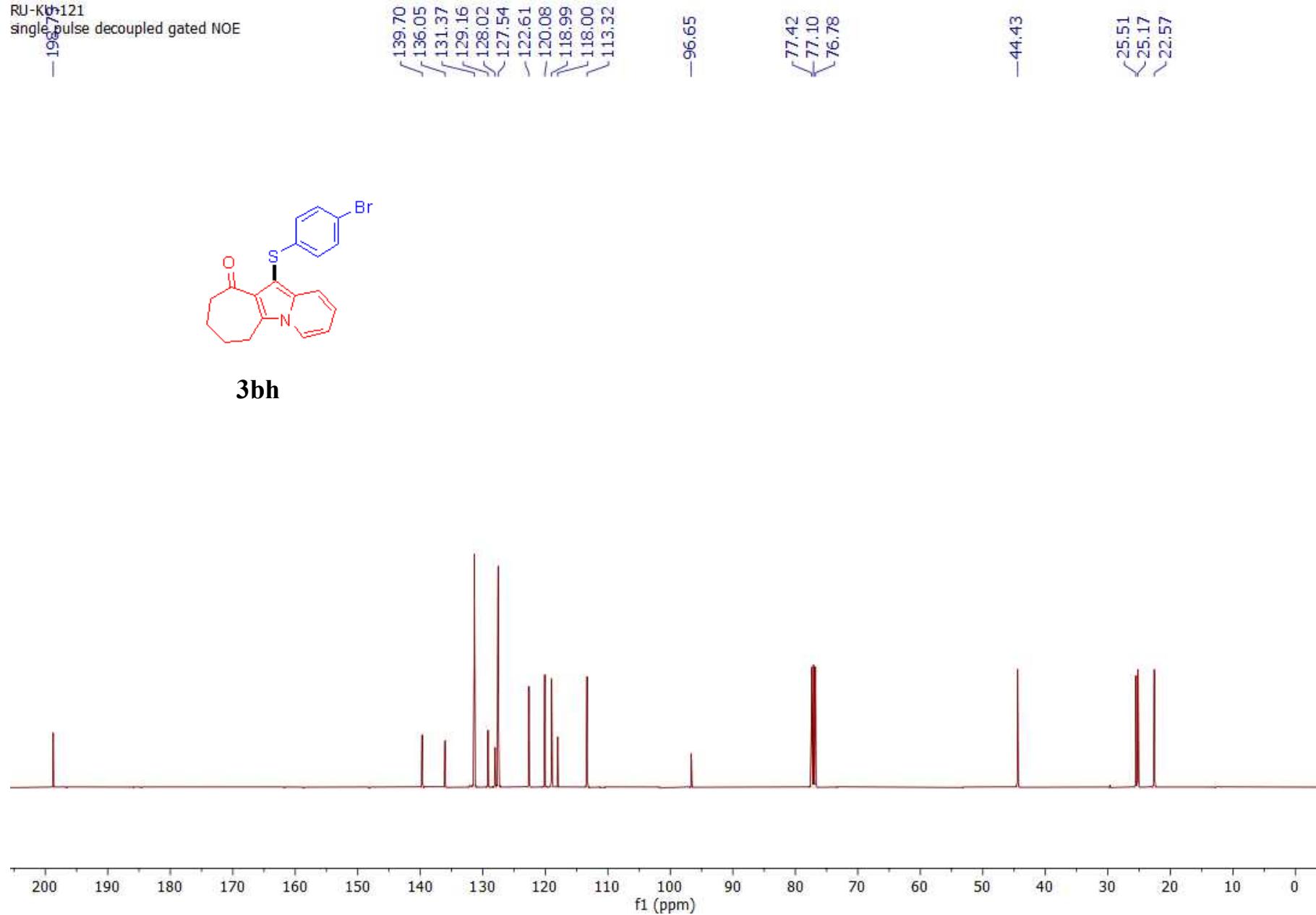
RU-KU-121  
single\_pulse



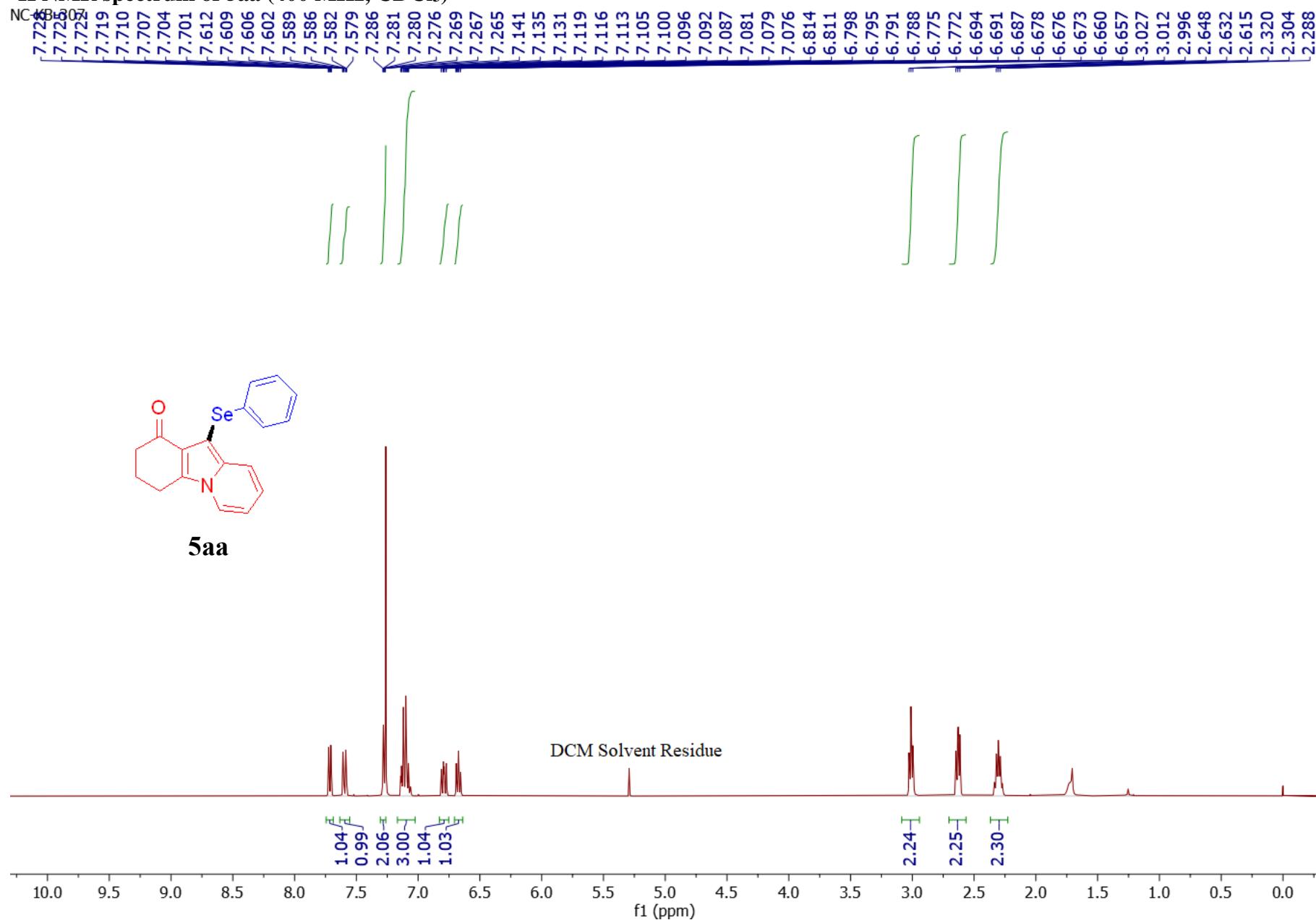
$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 3bh (100 MHz,  $\text{CDCl}_3$ )

RU-Kh121

single pulse decoupled gated NOE



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



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 5aa (100 MHz,  $\text{CDCl}_3$ )

NC-KB-308

-195.16

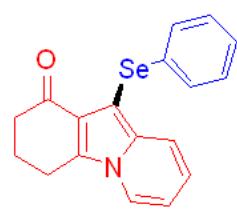
136.48  
134.10  
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120.11  
113.21

-91.21

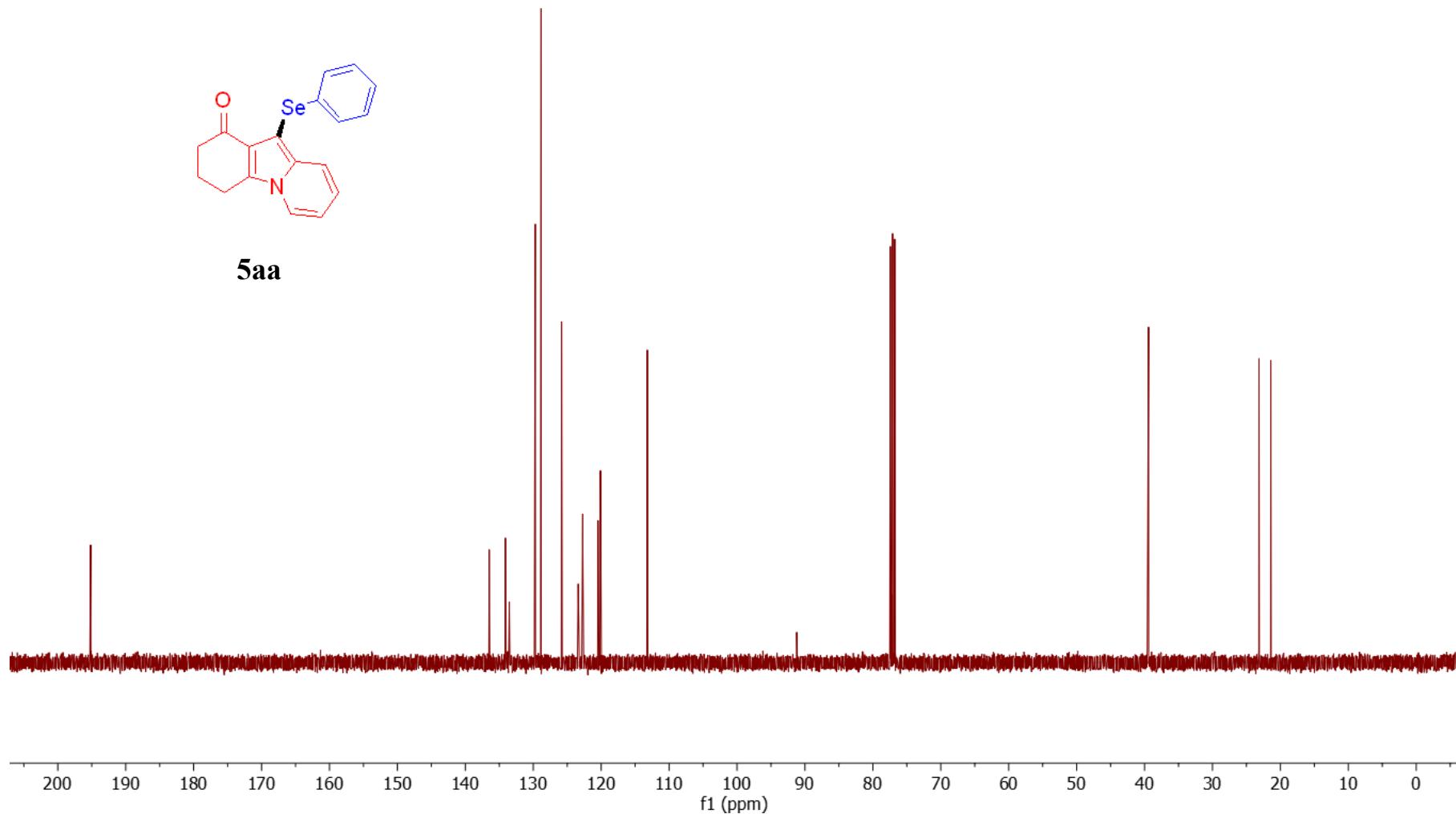
77.42  
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76.78

-39.41

~23.15  
~21.41

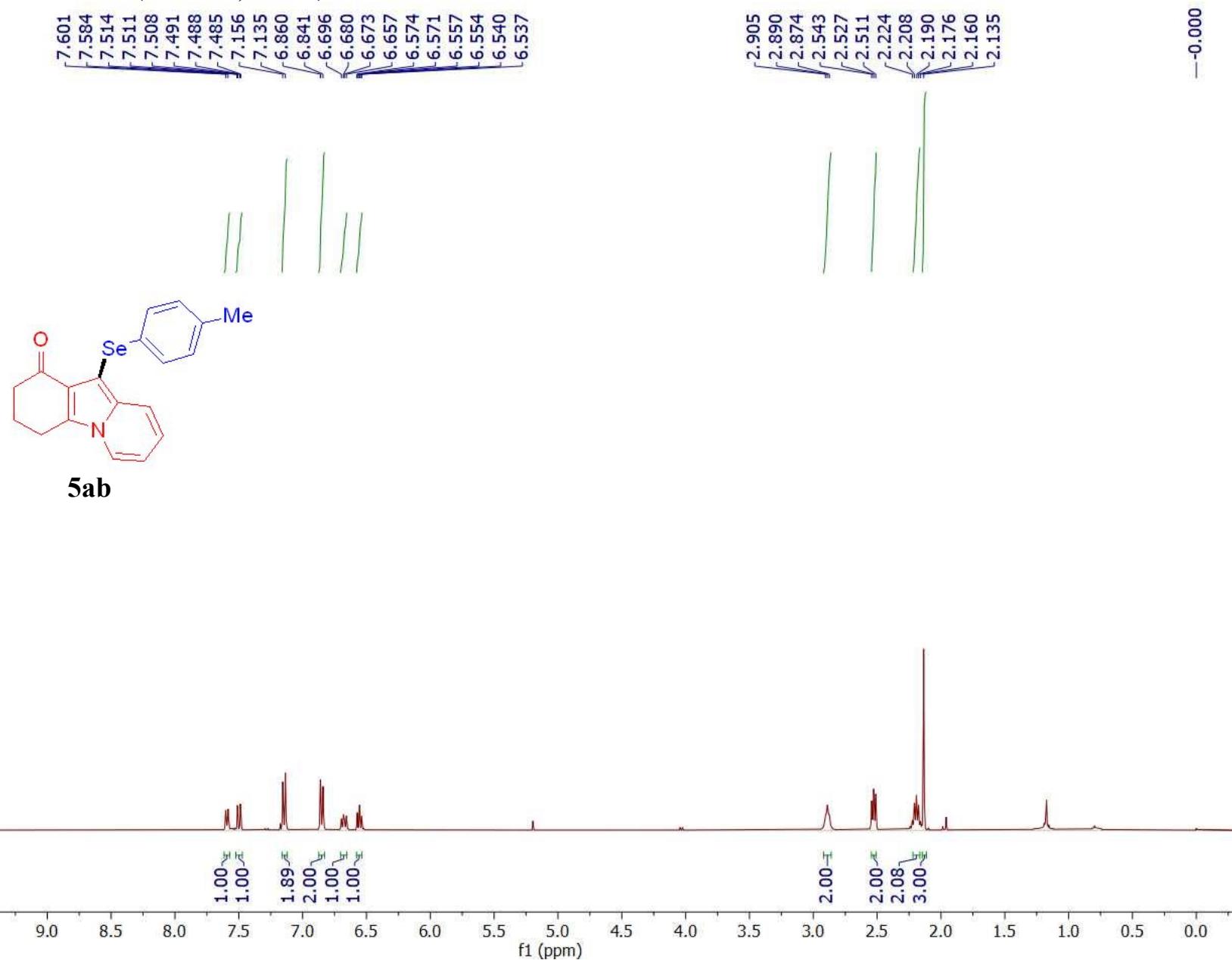


5aa



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

RU-KU-099  
single\_pulse



<sup>13</sup>C{H} NMR spectrum of 5ab (100 MHz, CDCl<sub>3</sub>)

RU-KU 699  
single pulse decoupled gated NOE  
—195.00

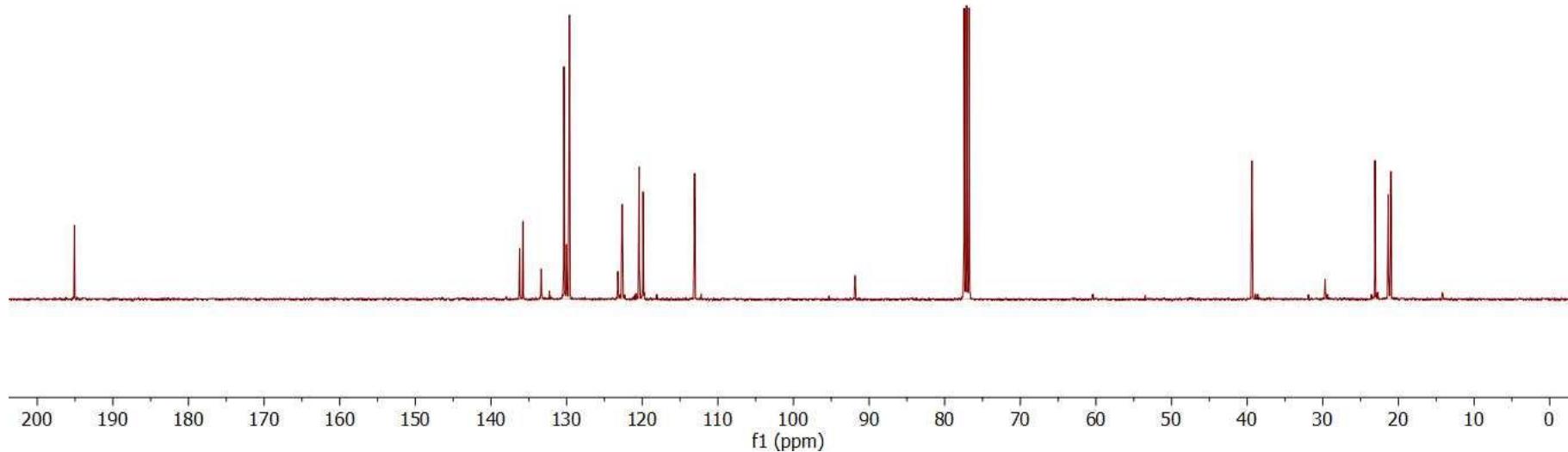
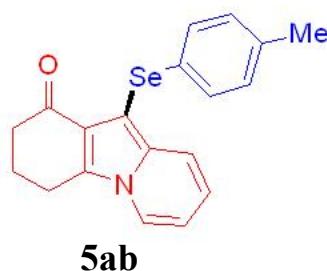
136.20  
135.77  
133.36  
130.36  
130.02  
129.63  
123.23  
122.66  
120.40  
119.88  
113.08

—90.88

77.42  
77.10  
76.78

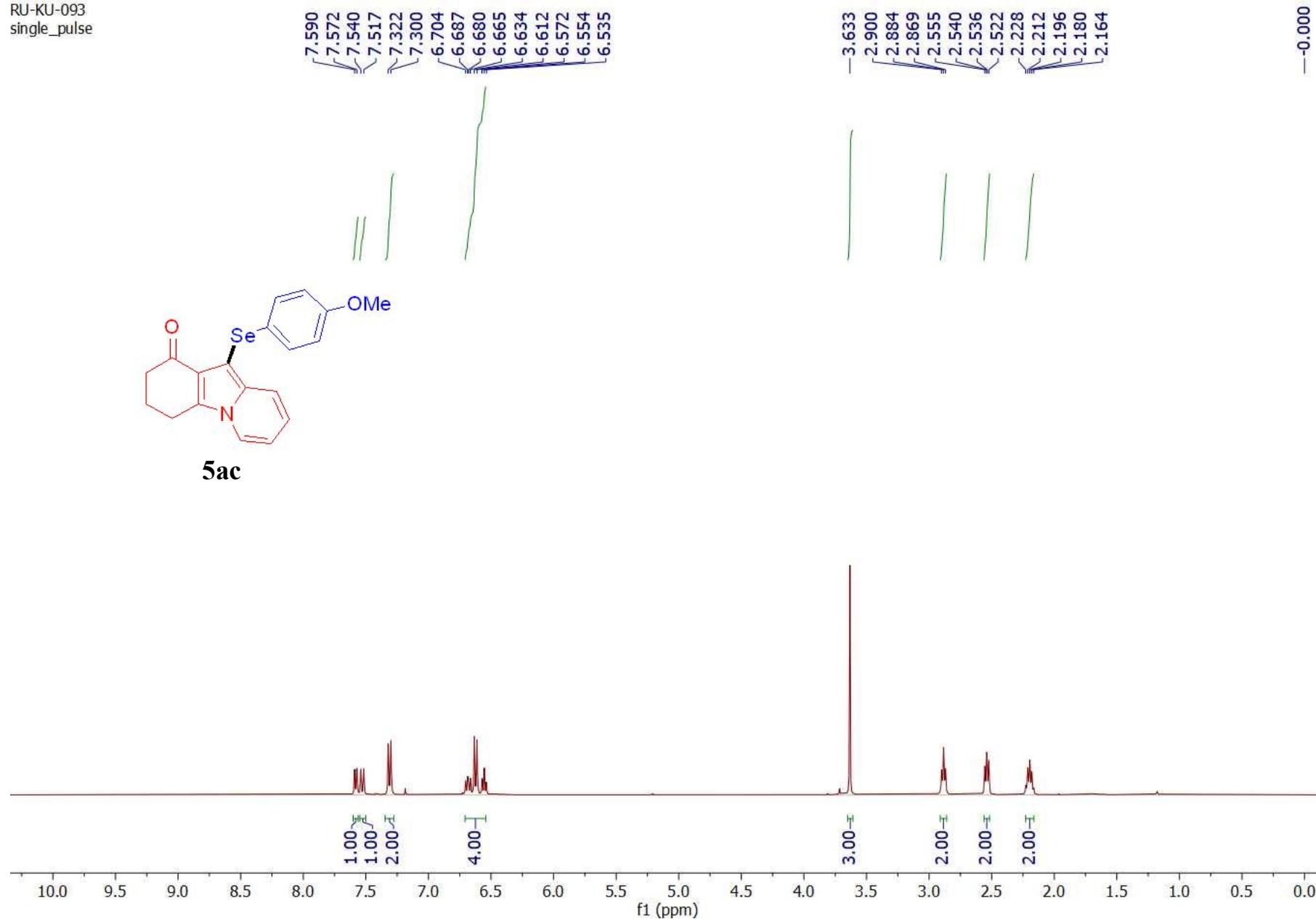
—39.37

23.11  
21.34  
21.01



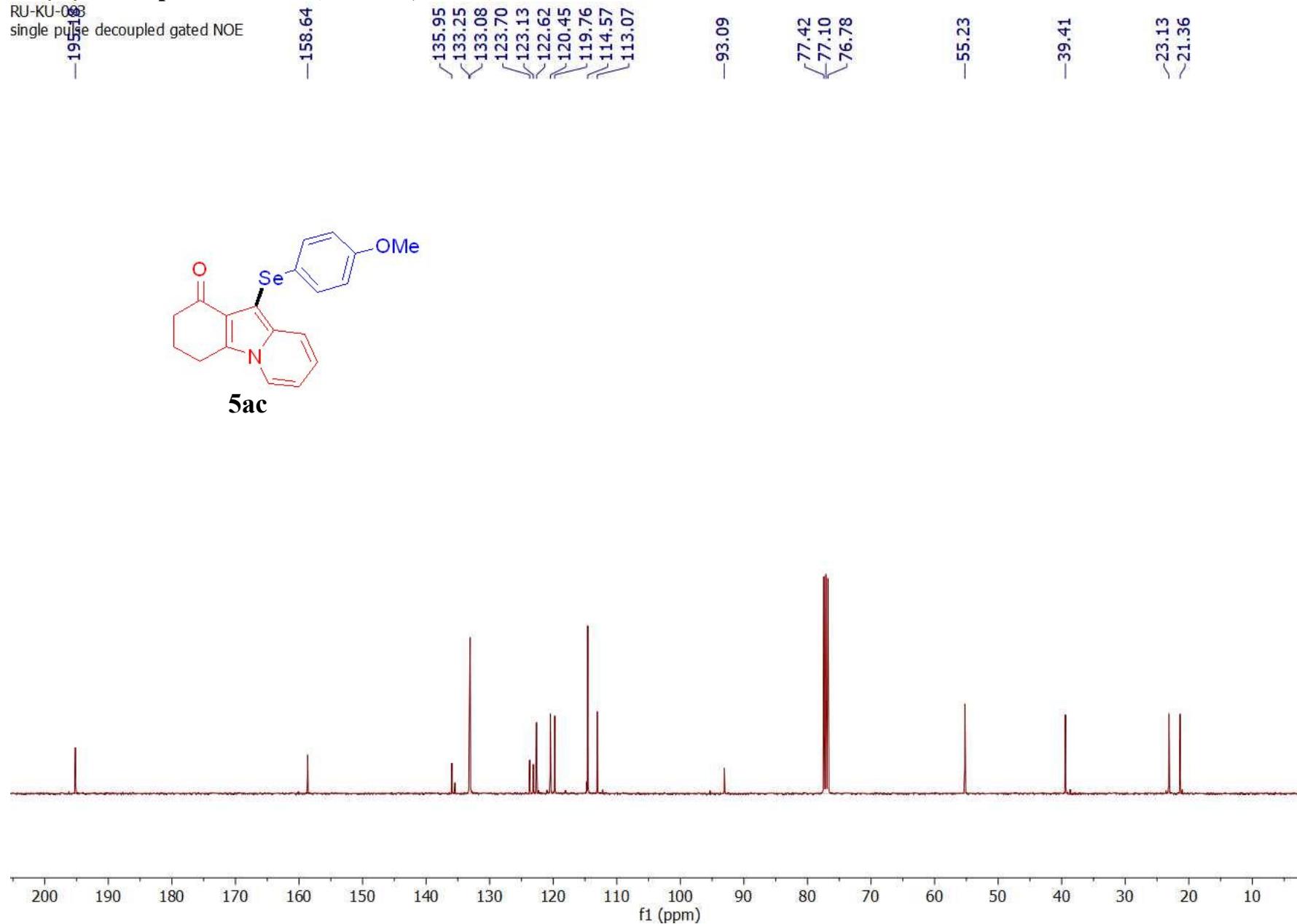
**<sup>1</sup>H NMR spectrum of 5ac (400 MHz, CDCl<sub>3</sub>)**

RU-KU-093  
single\_pulse



<sup>13</sup>C{H} NMR spectrum of 5ac (100 MHz, CDCl<sub>3</sub>)

RU-KU-008  
single pulse decoupled gated NOE



**<sup>1</sup>H NMR spectrum of 5ad (400 MHz, CDCl<sub>3</sub>)**

RU-AC-080  
single\_pulse

7.744

7.738

7.726

7.723

7.720

7.604

7.601

7.598

7.581

7.578

7.575

7.230

7.209

7.135

7.114

6.849

6.847

6.833

6.830

6.827

6.824

6.810

6.807

6.717

6.714

6.700

6.697

6.683

6.680

3.036

3.020

3.005

2.646

2.630

2.614

2.337

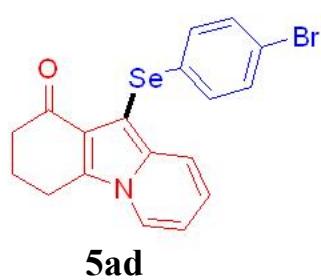
2.322

2.307

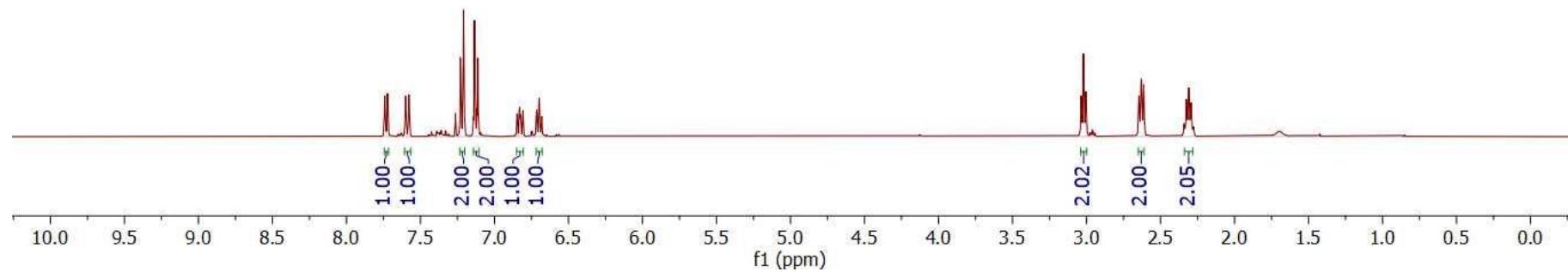
2.293

2.278

-0.000

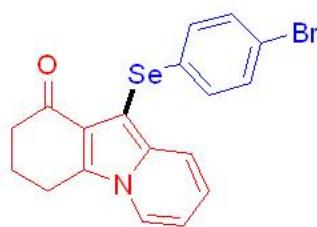
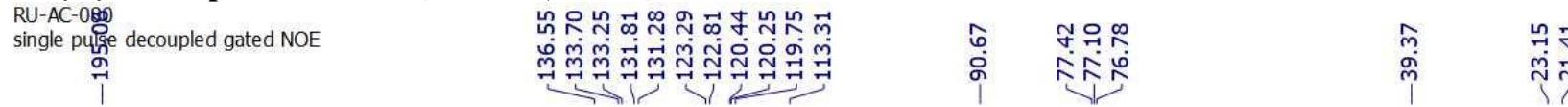


**5ad**

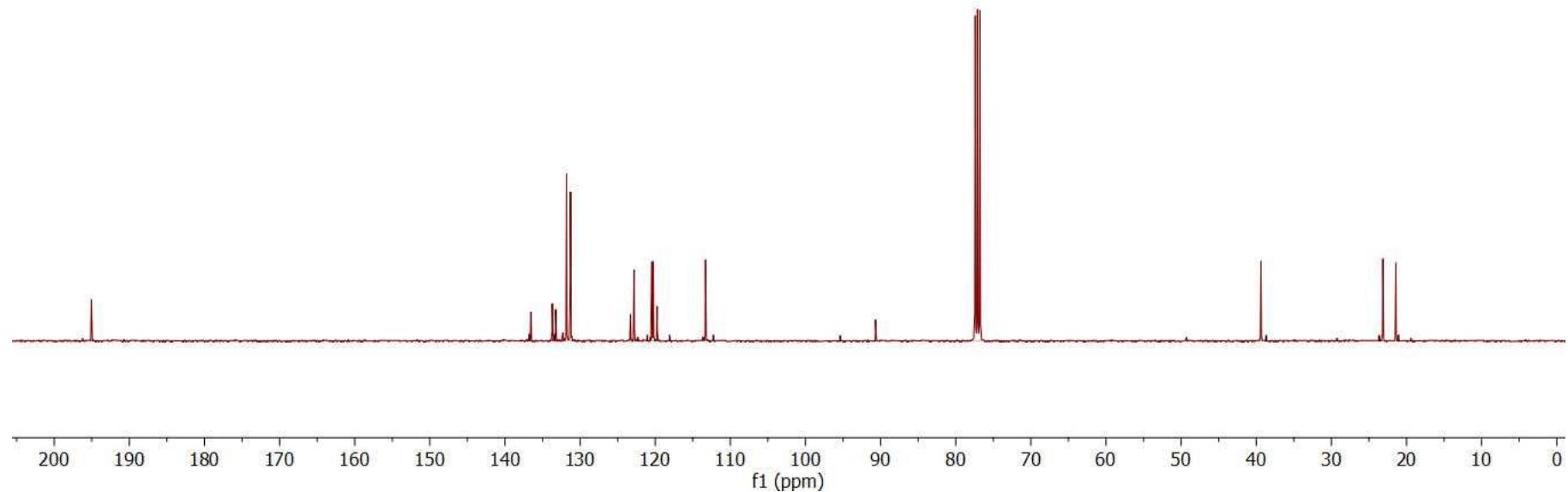


$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 5ad (100 MHz,  $\text{CDCl}_3$ )

RU-AC-080  
single pulse decoupled gated NOE

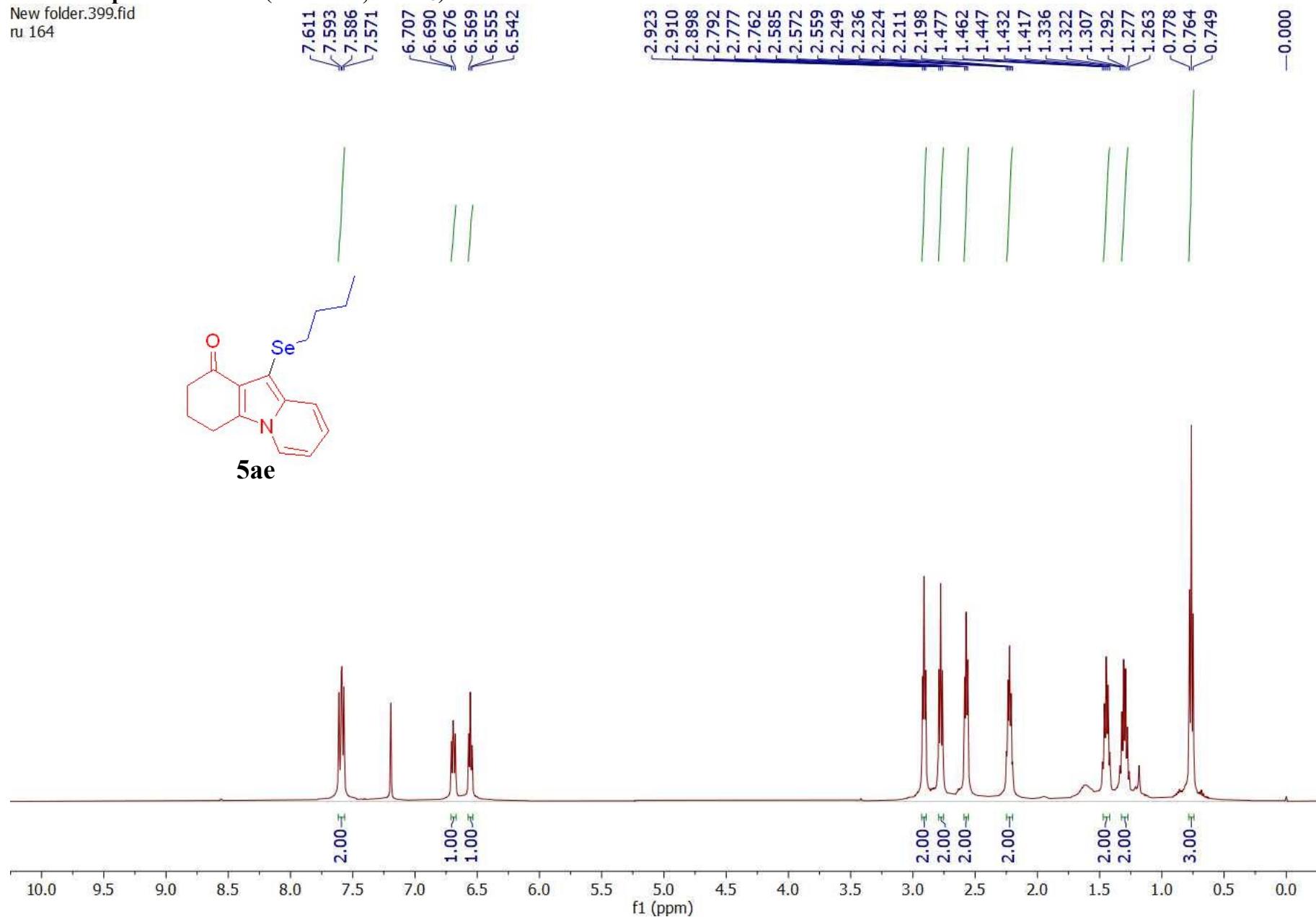


5ad



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

New folder.399.fid  
ru 164



<sup>13</sup>C{H} NMR spectrum of 5ae (100 MHz, CDCl<sub>3</sub>)

New folder 400.fid

ru 164

-195.3

140.05

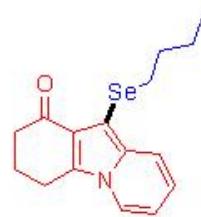
-133.28

-136.05  
-133.28  
123.70  
122.42  
120.89  
119.02  
112.94

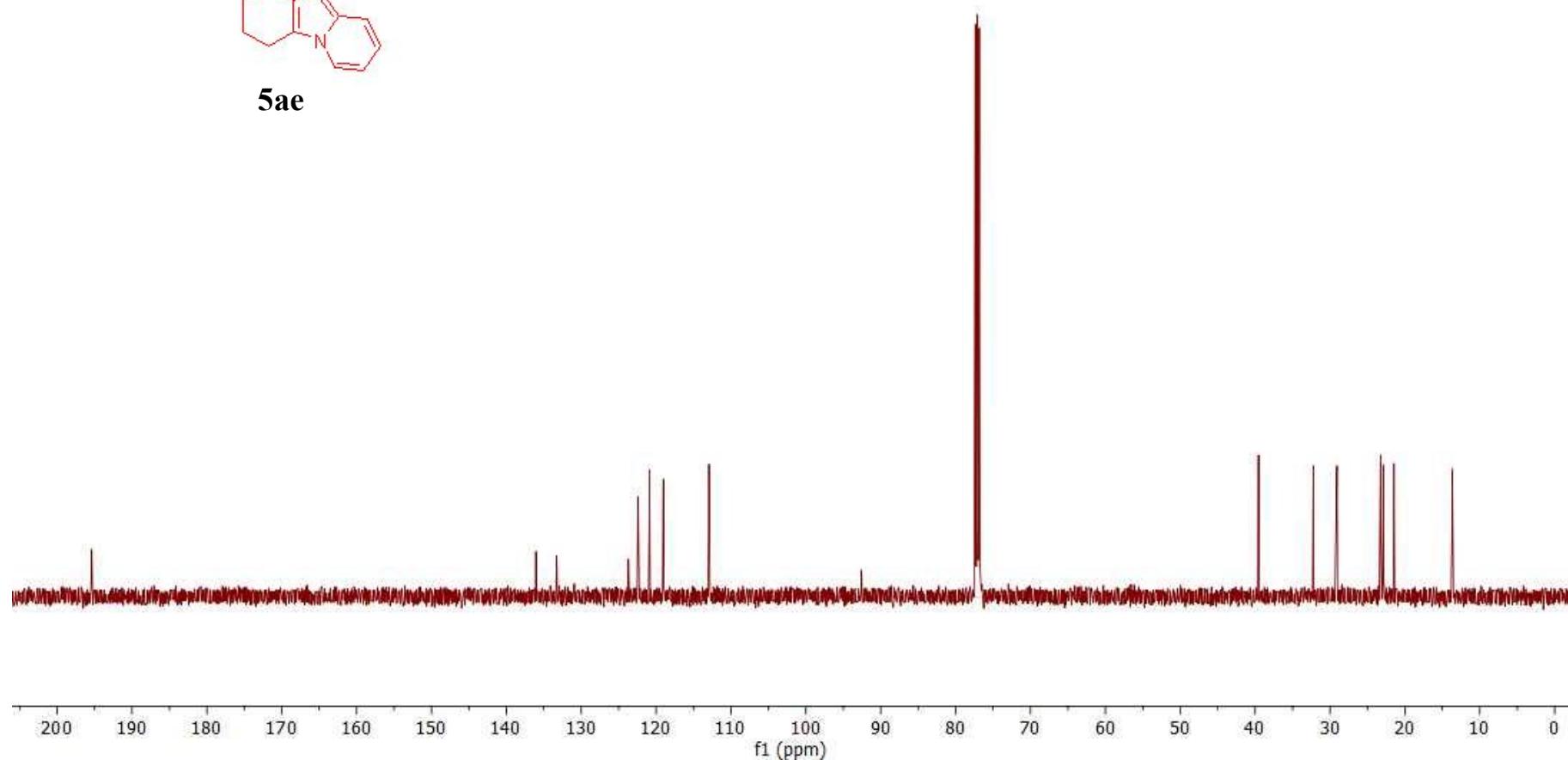
-92.59

77.35  
77.10  
76.85

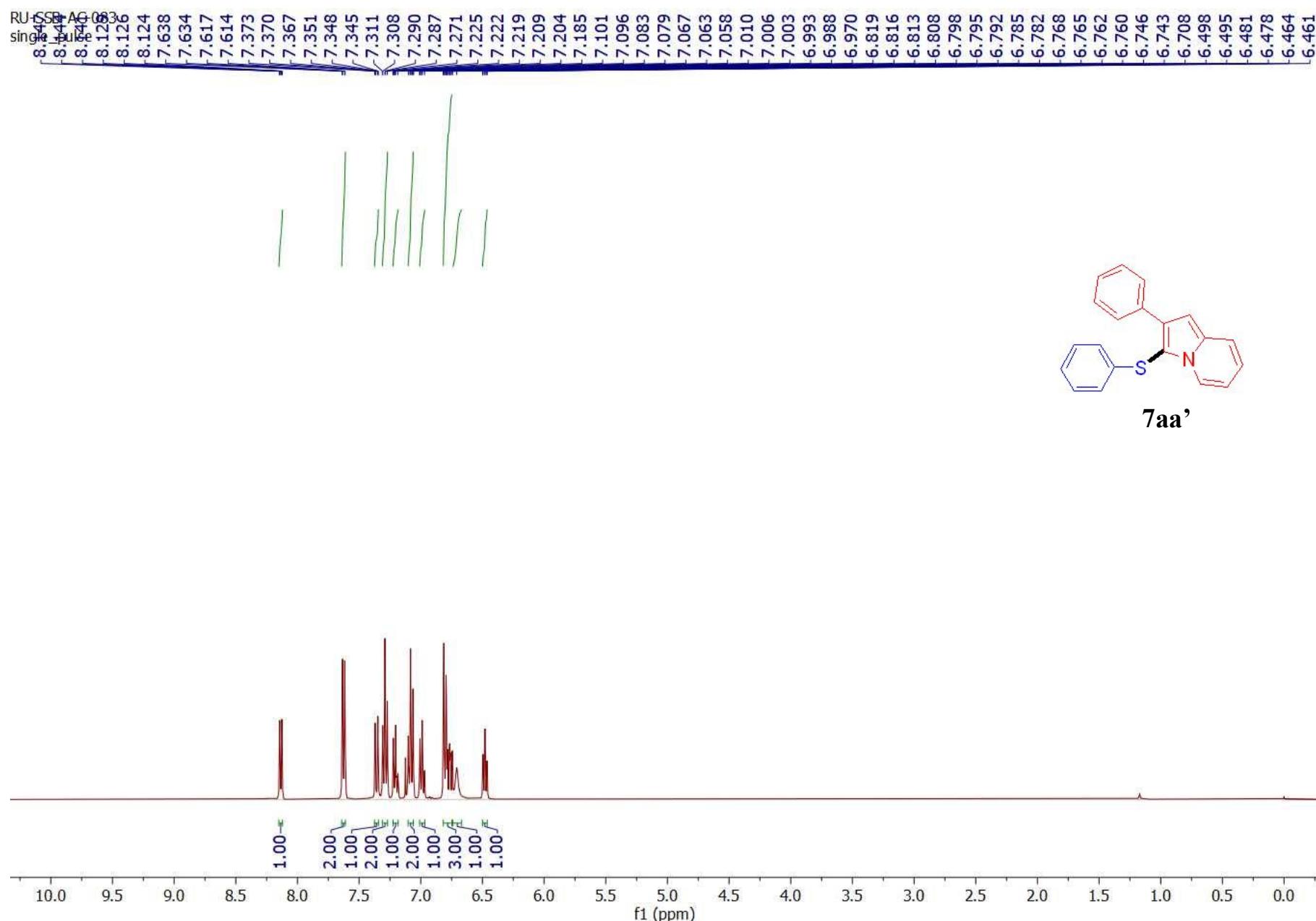
-39.55  
32.23  
29.07  
23.25  
22.89  
21.47  
-13.69



5ae



<sup>1</sup>H NMR spectrum of 7aa' (400 MHz, CDCl<sub>3</sub>)



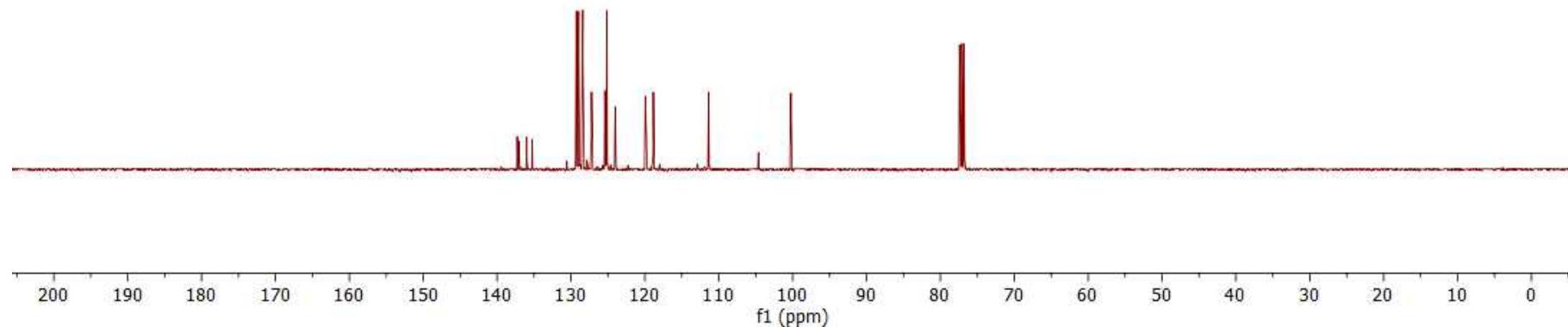
<sup>13</sup>C{H} NMR spectrum of 7aa' (100 MHz, CDCl<sub>3</sub>)

RU-SSB-AC-083

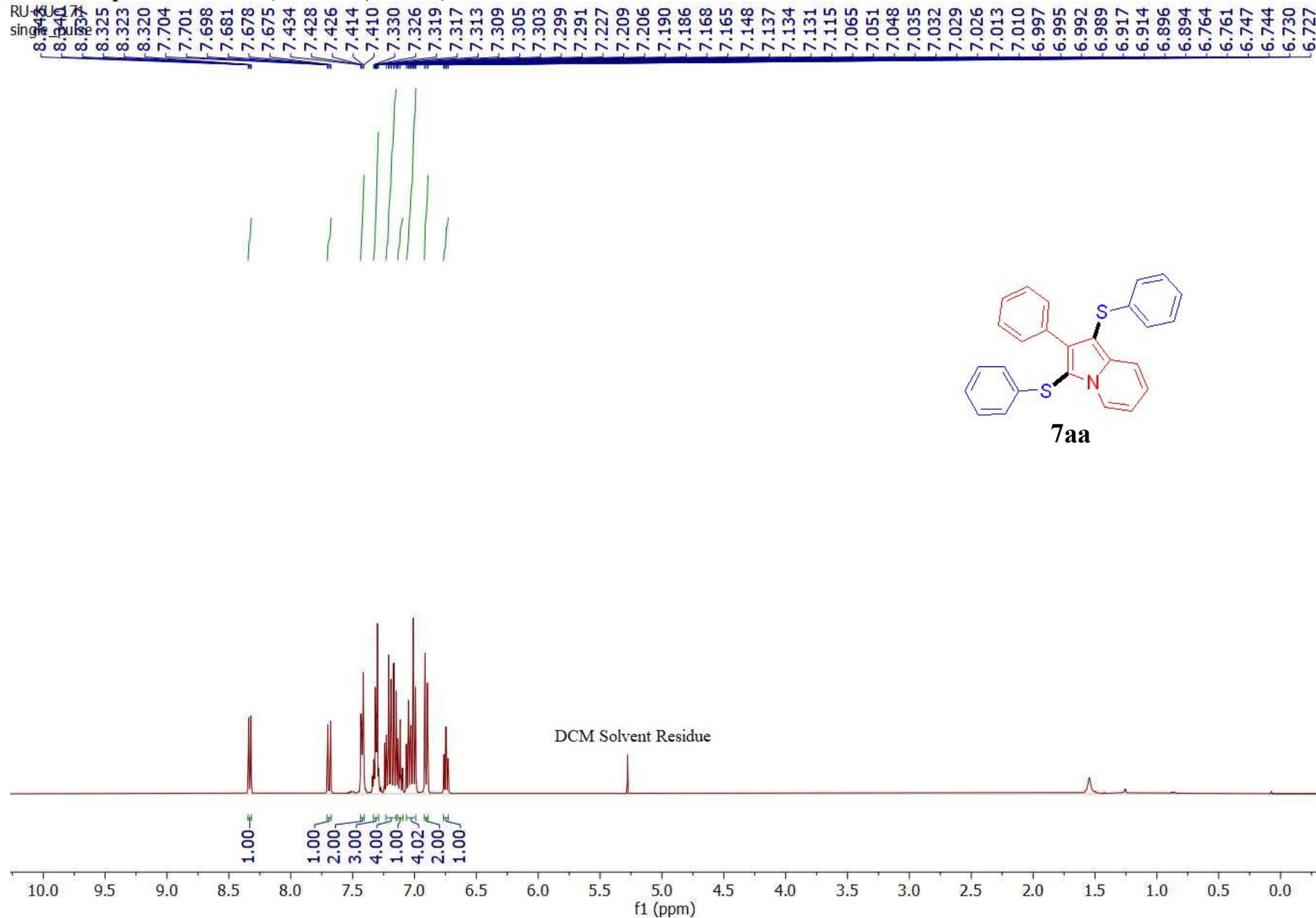
single pulse decoupled gated NOE



7aa'

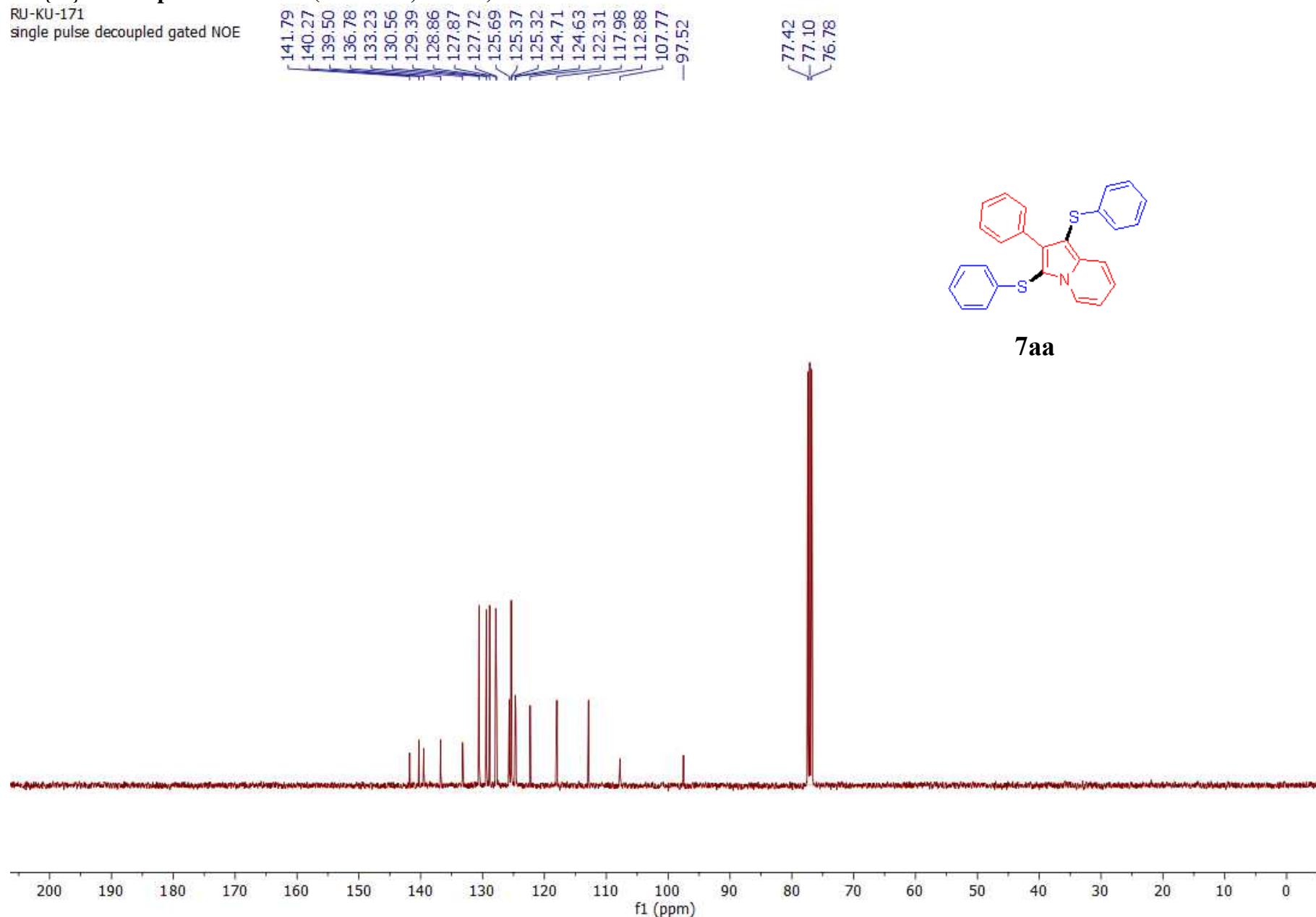


<sup>1</sup>H NMR spectrum of 7aa (400 MHz, CDCl<sub>3</sub>)



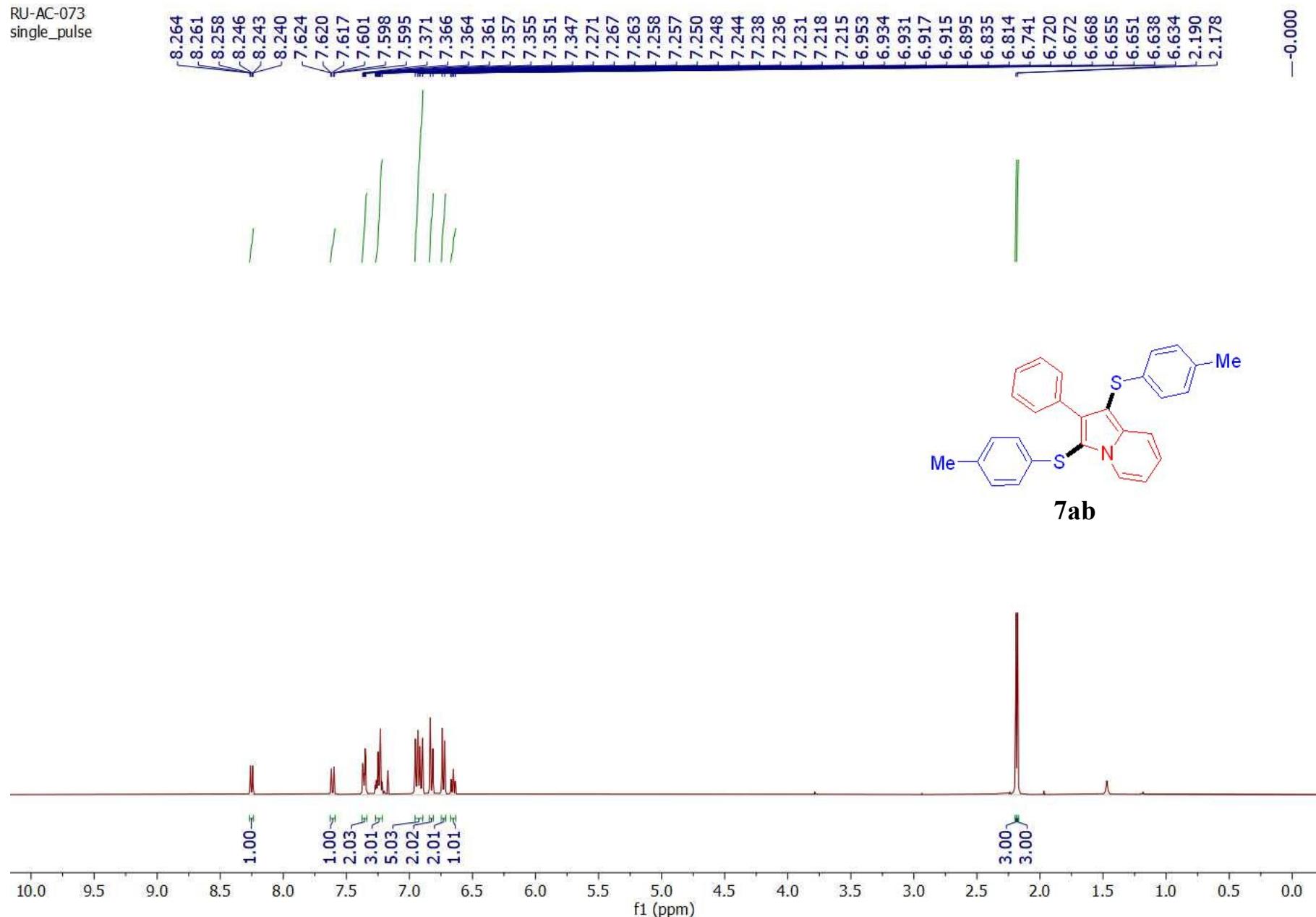
<sup>13</sup>C{H} NMR spectrum of 7aa (100 MHz, CDCl<sub>3</sub>)

RU-KU-171  
single pulse decoupled gated NOE



<sup>1</sup>H NMR spectrum of 7ab (400 MHz, CDCl<sub>3</sub>)

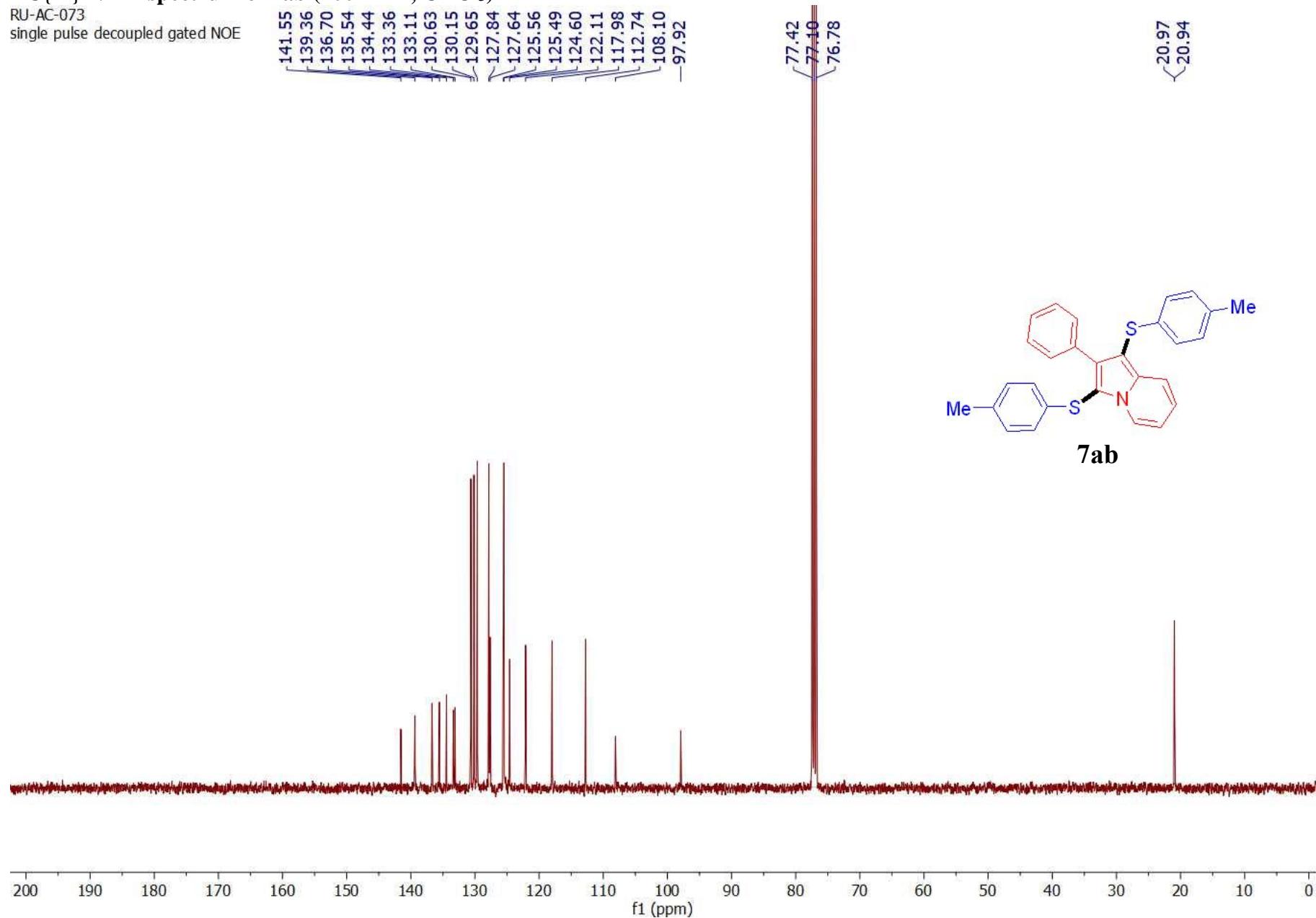
RU-AC-073  
single\_pulse



<sup>13</sup>C{H} NMR spectrum of 7ab (100 MHz, CDCl<sub>3</sub>)

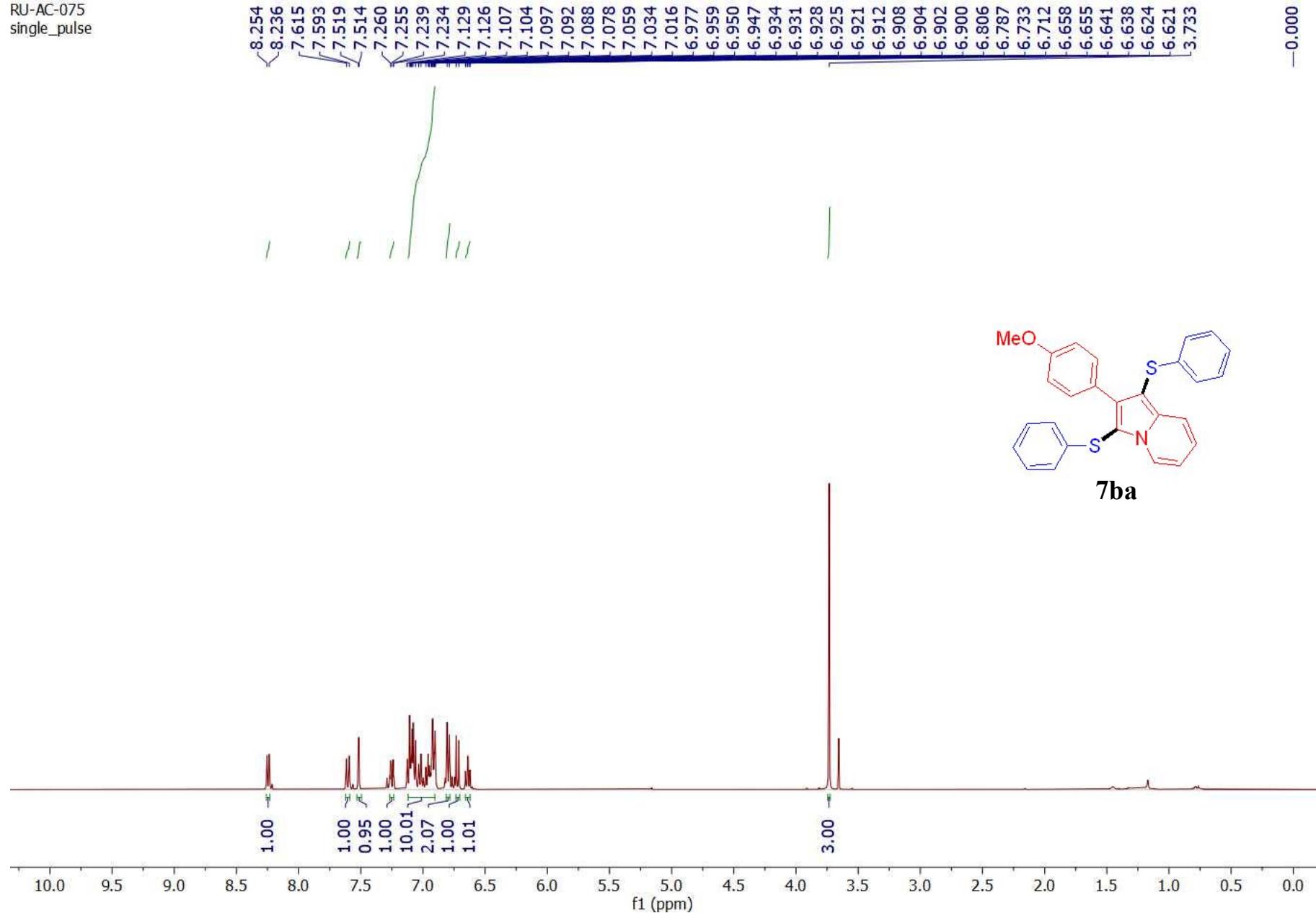
RU-AC-073

single pulse decoupled gated NOE



<sup>1</sup>H NMR spectrum of 7ba (400 MHz, CDCl<sub>3</sub>)

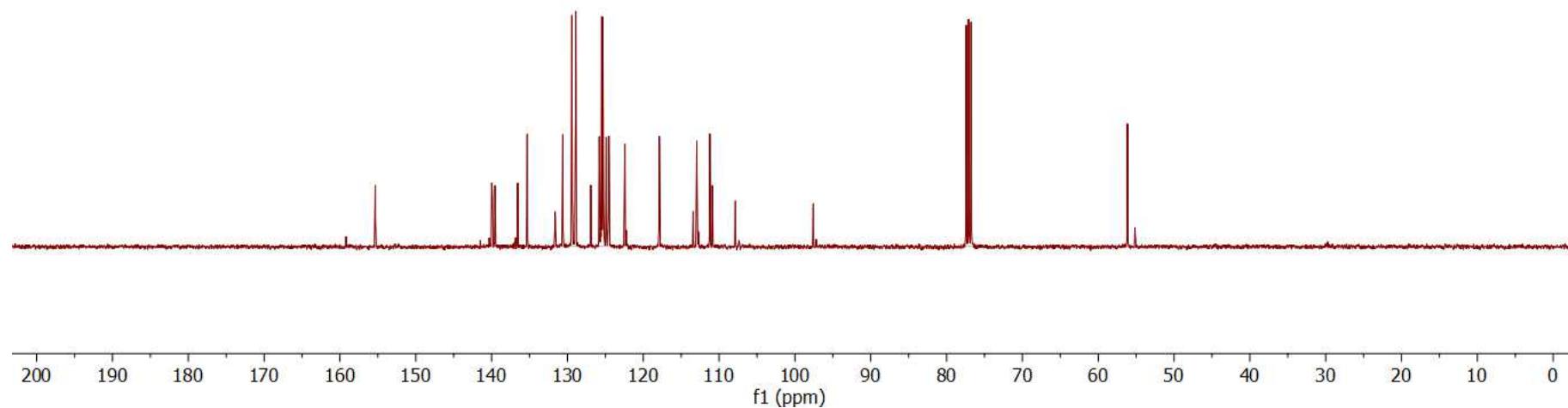
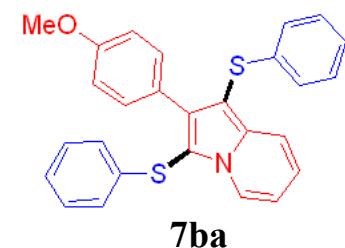
RU-AC-075  
single\_pulse



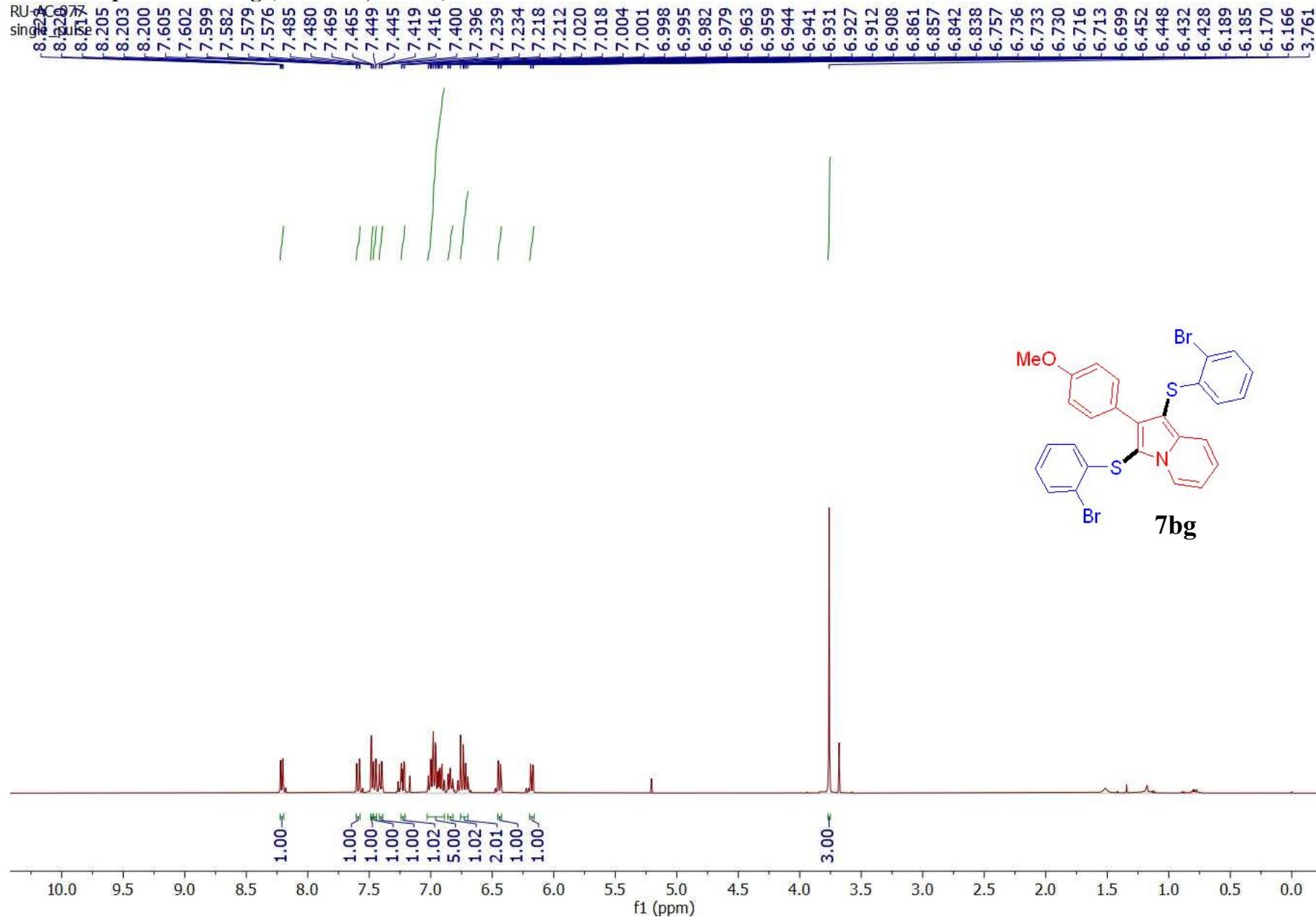
$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 7ba (100 MHz,  $\text{CDCl}_3$ )

RU-AC-075

single pulse decoupled gated NCE



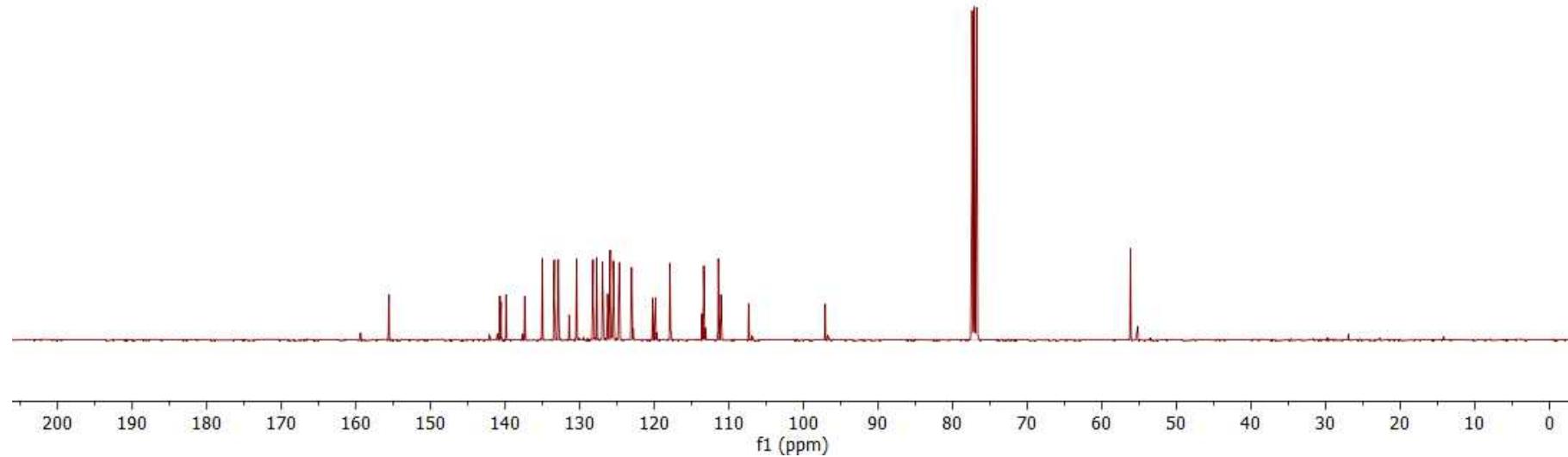
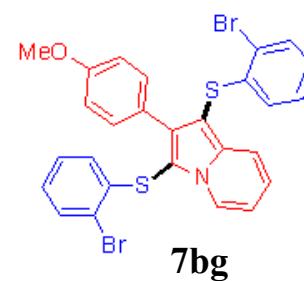
<sup>1</sup>H NMR spectrum of 7bg (400 MHz, CDCl<sub>3</sub>)



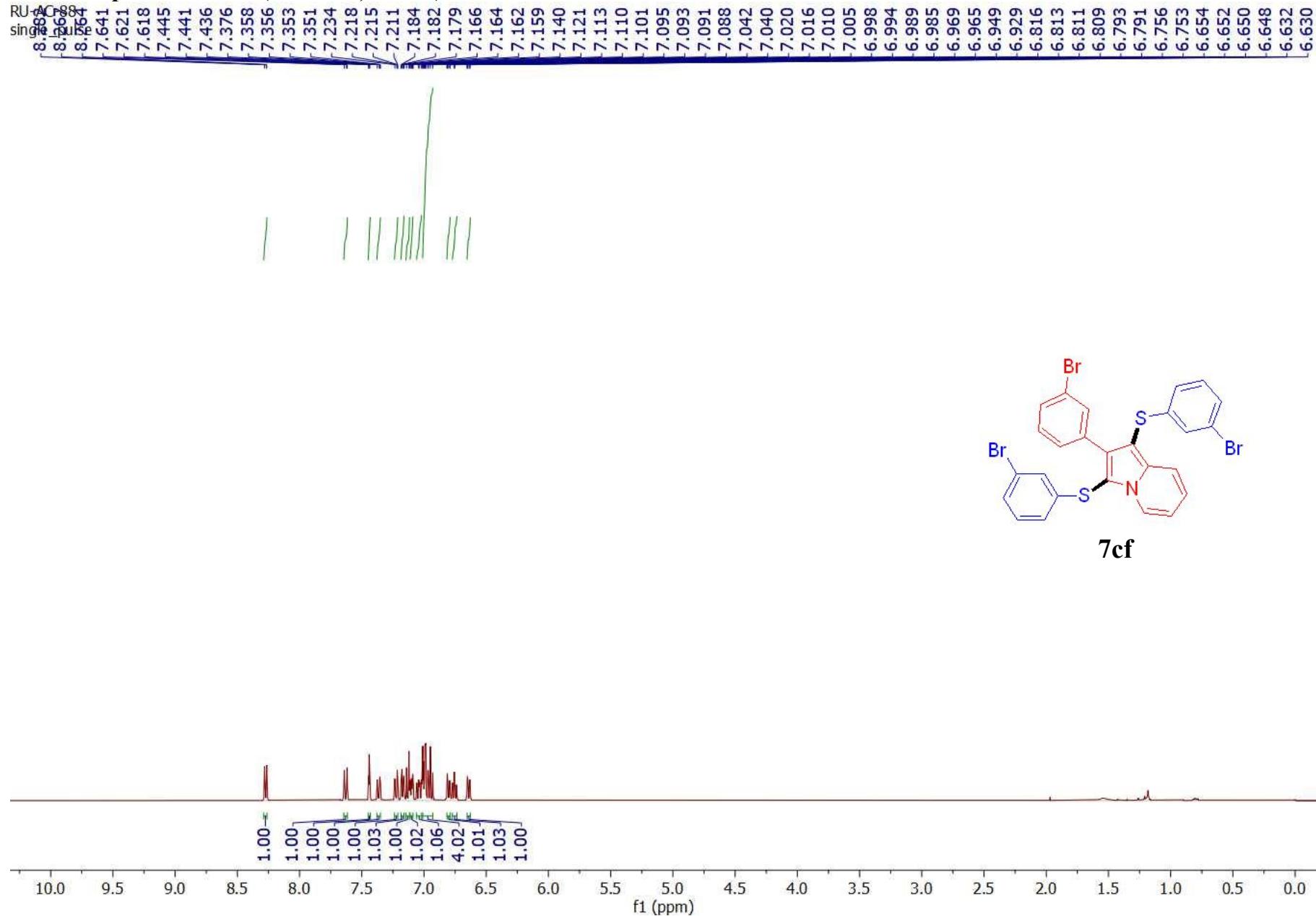
$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 7bg (100 MHz,  $\text{CDCl}_3$ )

RU-AC-077

single pulse decoupled gated NOE



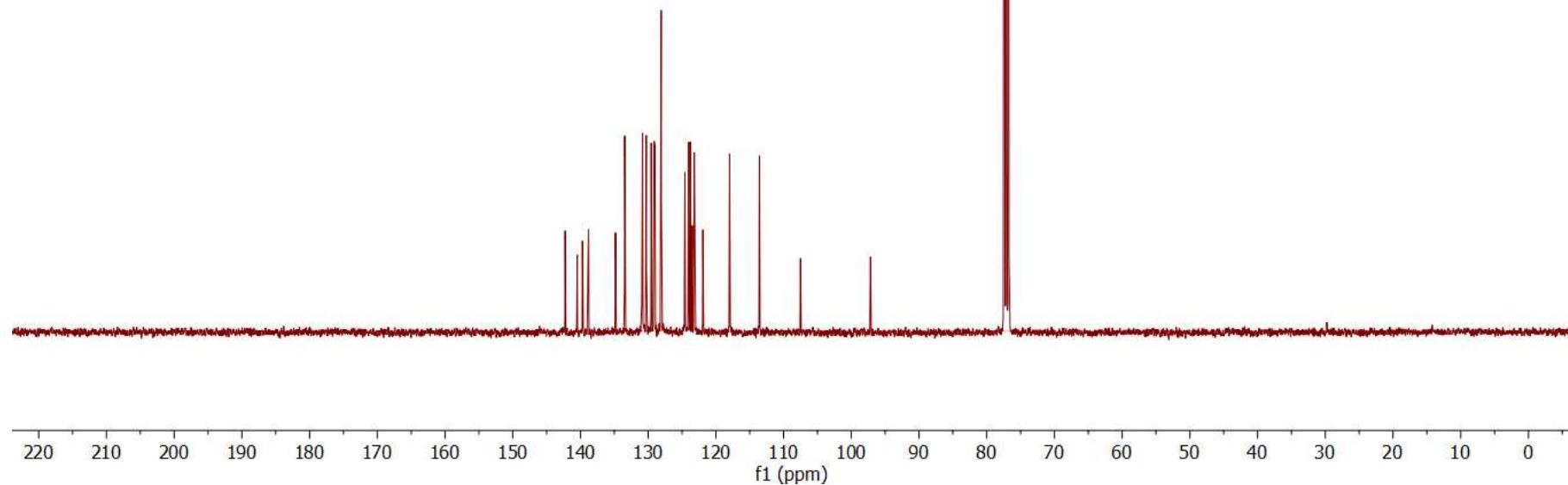
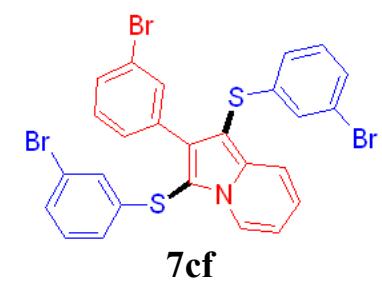
<sup>1</sup>H NMR spectrum of 7cf (400 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C{H} NMR spectrum of 7cf (100 MHz, CDCl<sub>3</sub>)

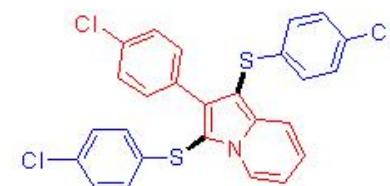
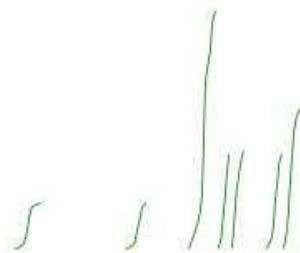
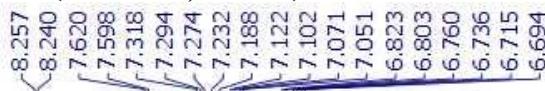
RU-AC-88

single pulse decoupled gated NOE

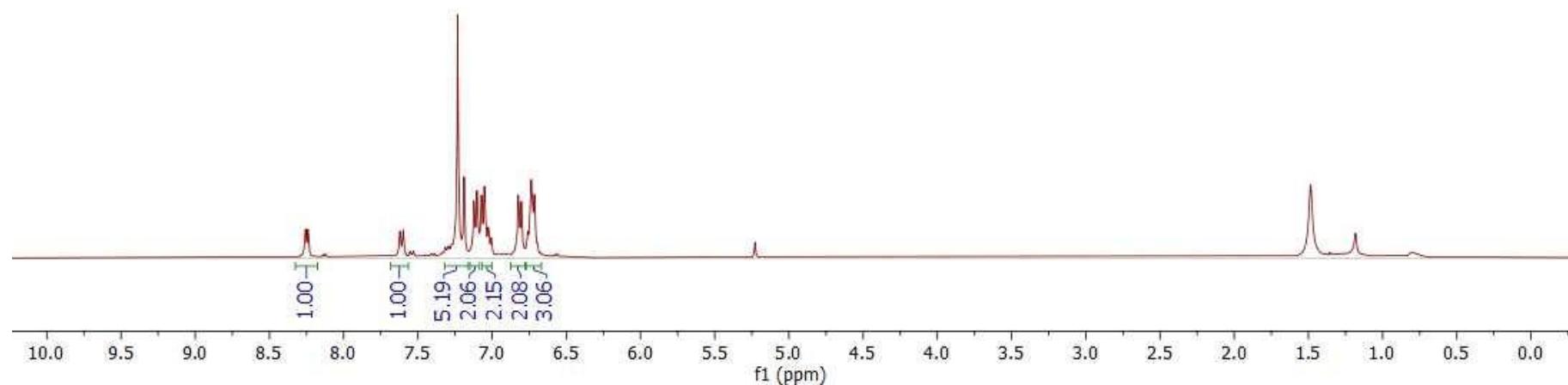


<sup>1</sup>H NMR spectrum of 7dd (400 MHz, CDCl<sub>3</sub>)

RU-AC-87  
single\_pulse



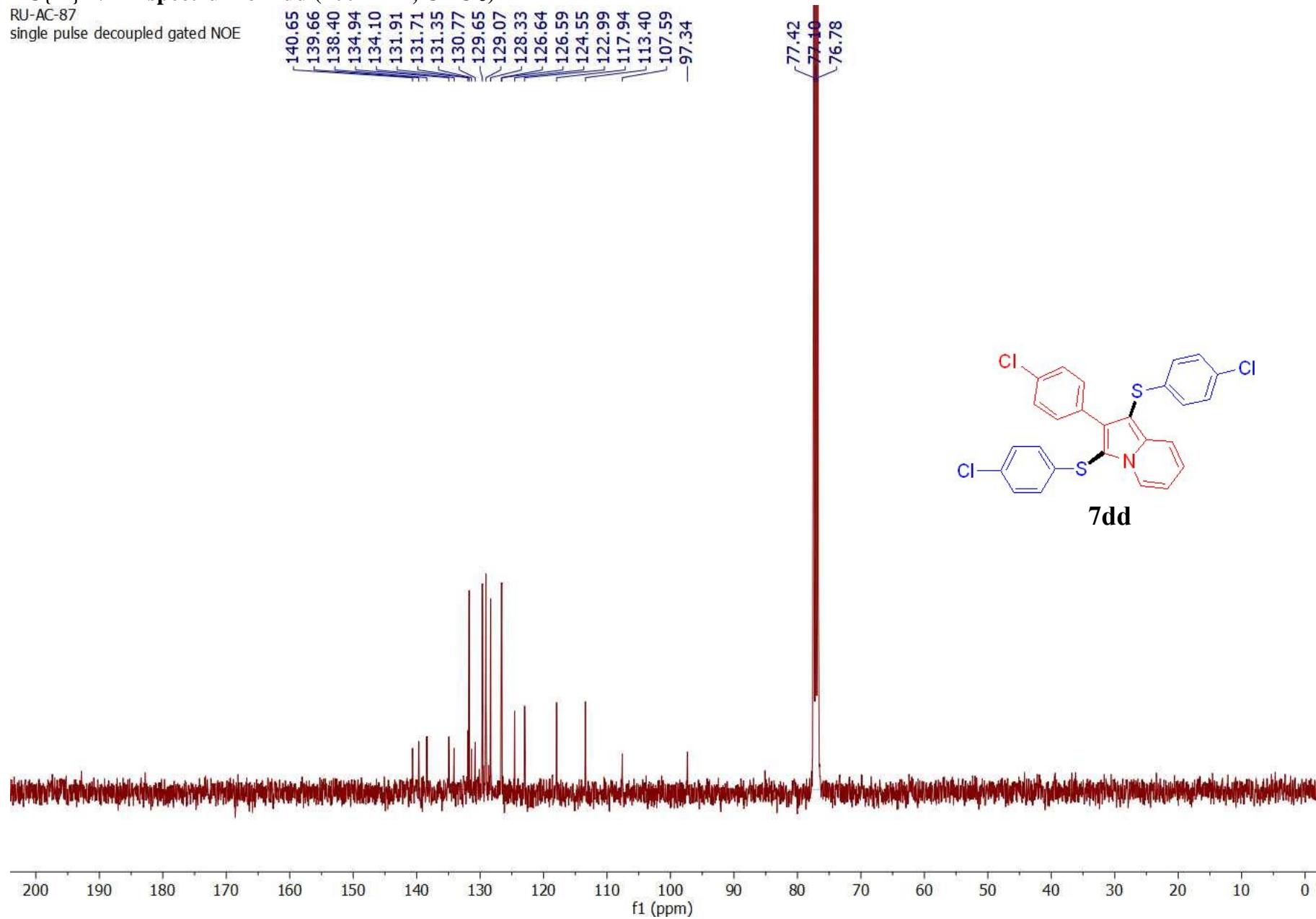
7dd



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 7dd (100 MHz,  $\text{CDCl}_3$ )

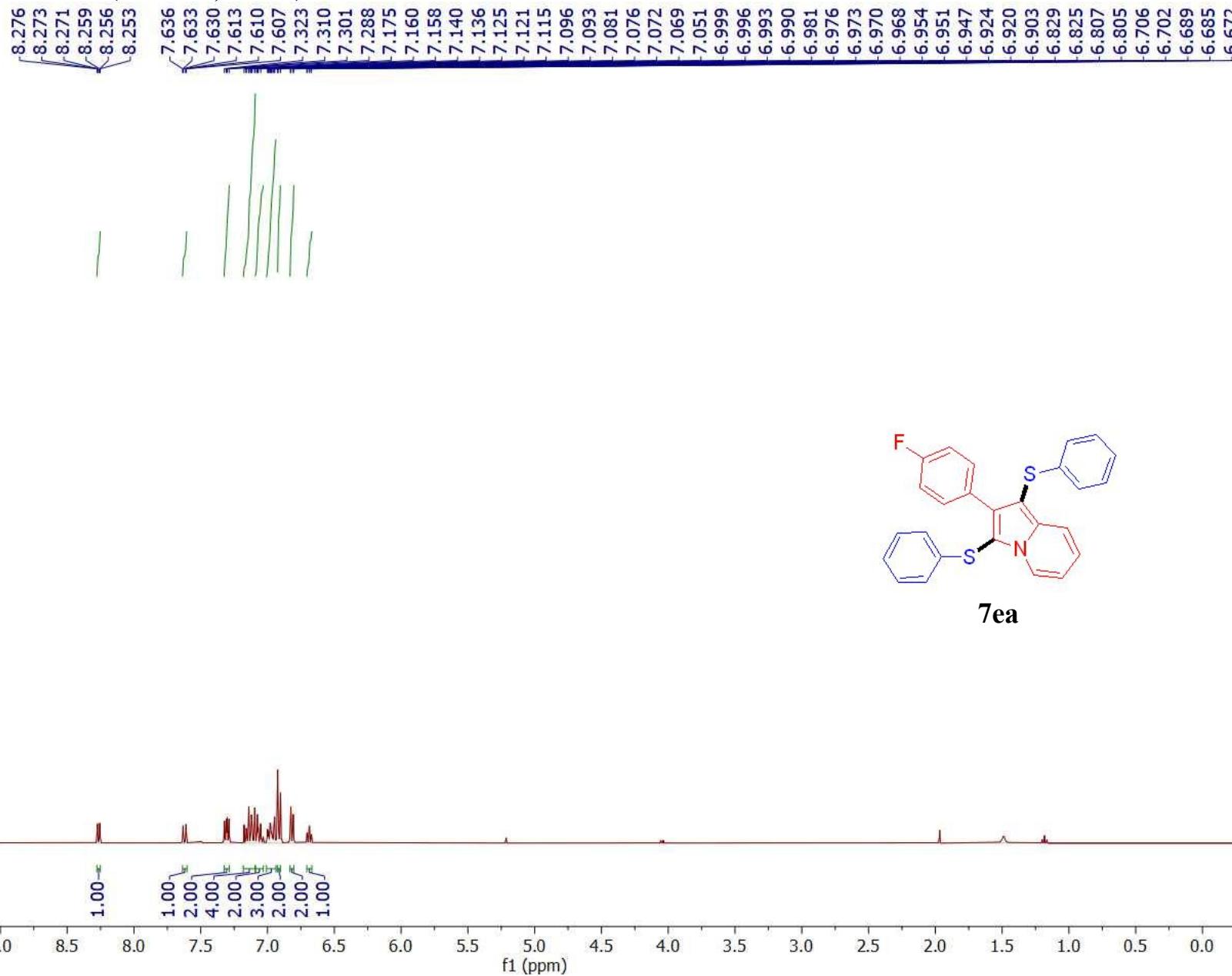
RU-AC-87

single pulse decoupled gated NOE



<sup>1</sup>H NMR spectrum of 7ea (400 MHz, CDCl<sub>3</sub>)

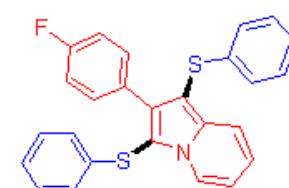
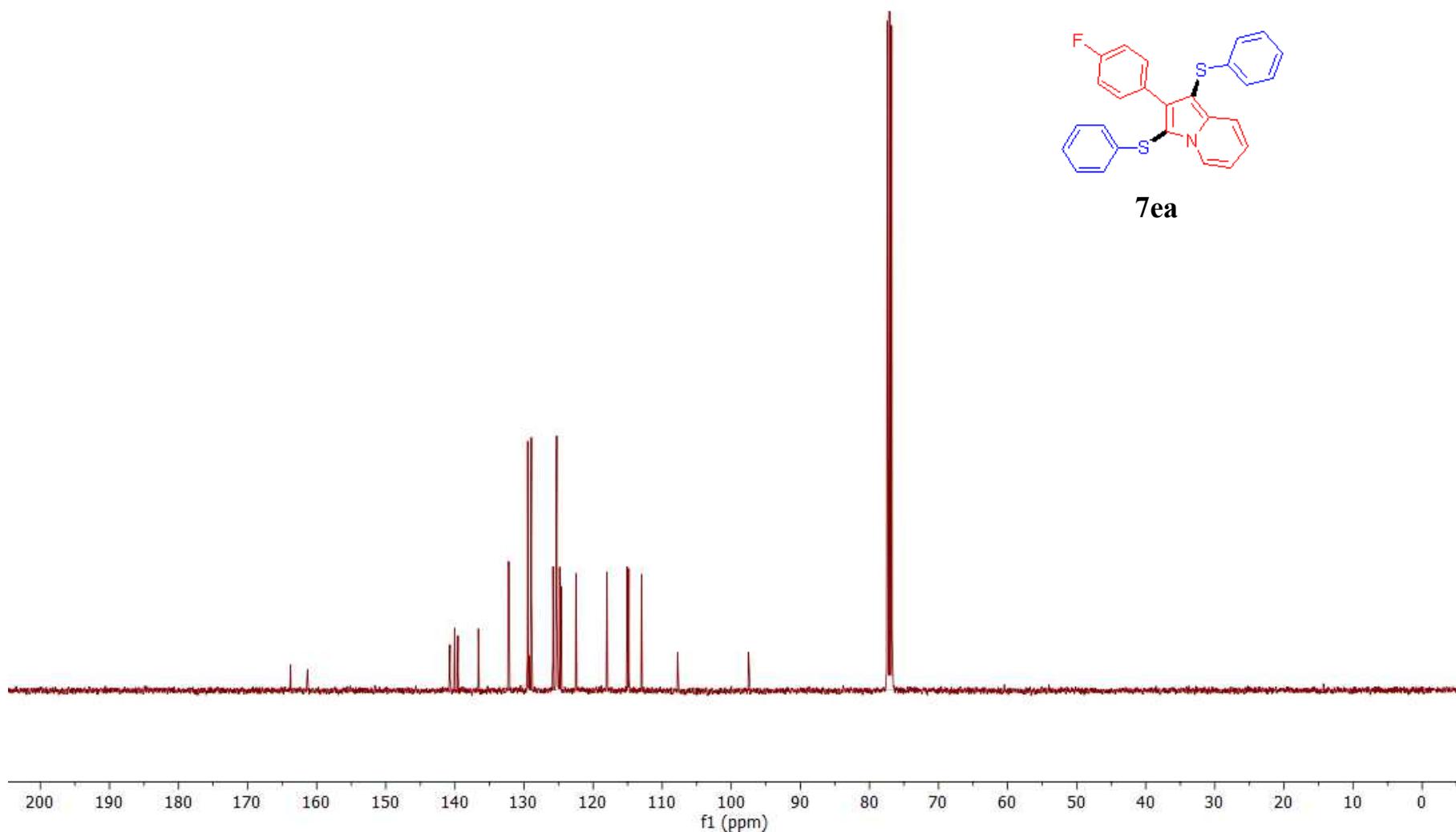
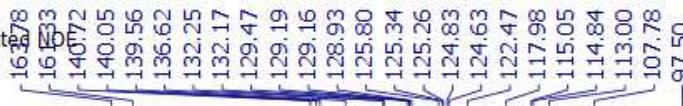
RU-AC-097  
single\_pulse



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 7ea (100 MHz,  $\text{CDCl}_3$ )

RU-AC-097

single pulse decoupled gated DE

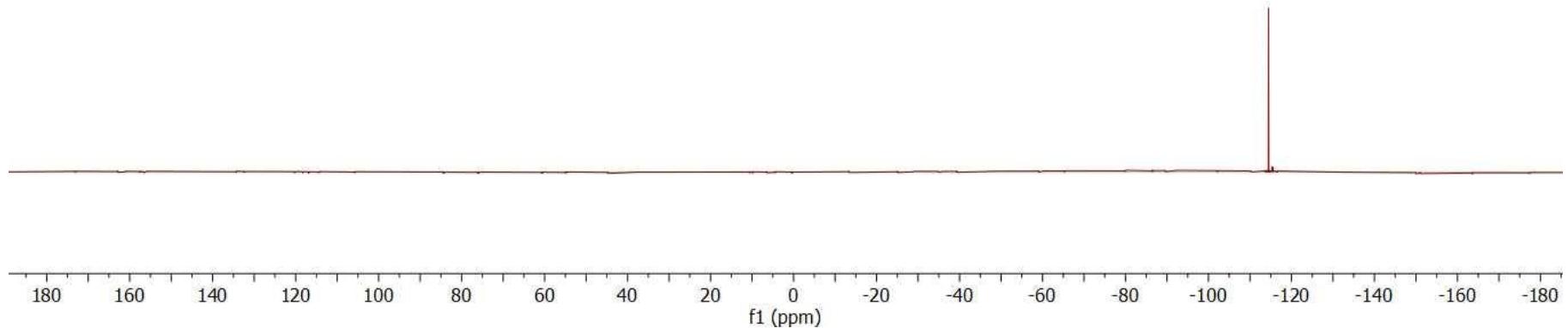
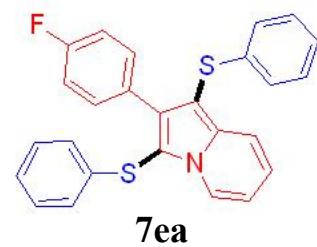


7ea

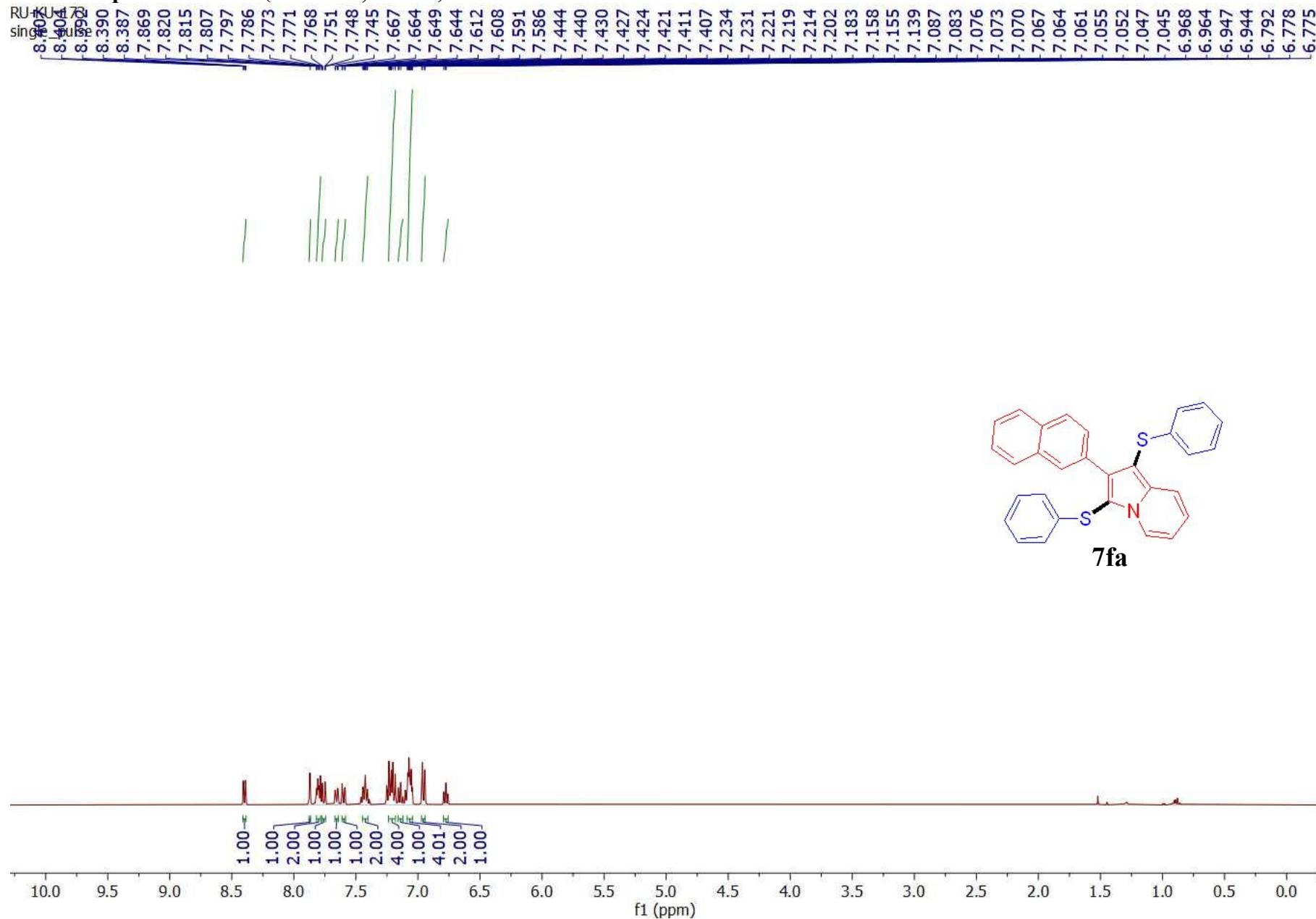
**<sup>19</sup>F NMR spectrum of 7ea (376 MHz, CDCl<sub>3</sub>)**

RU-AC-097  
single\_pulse

-114.47



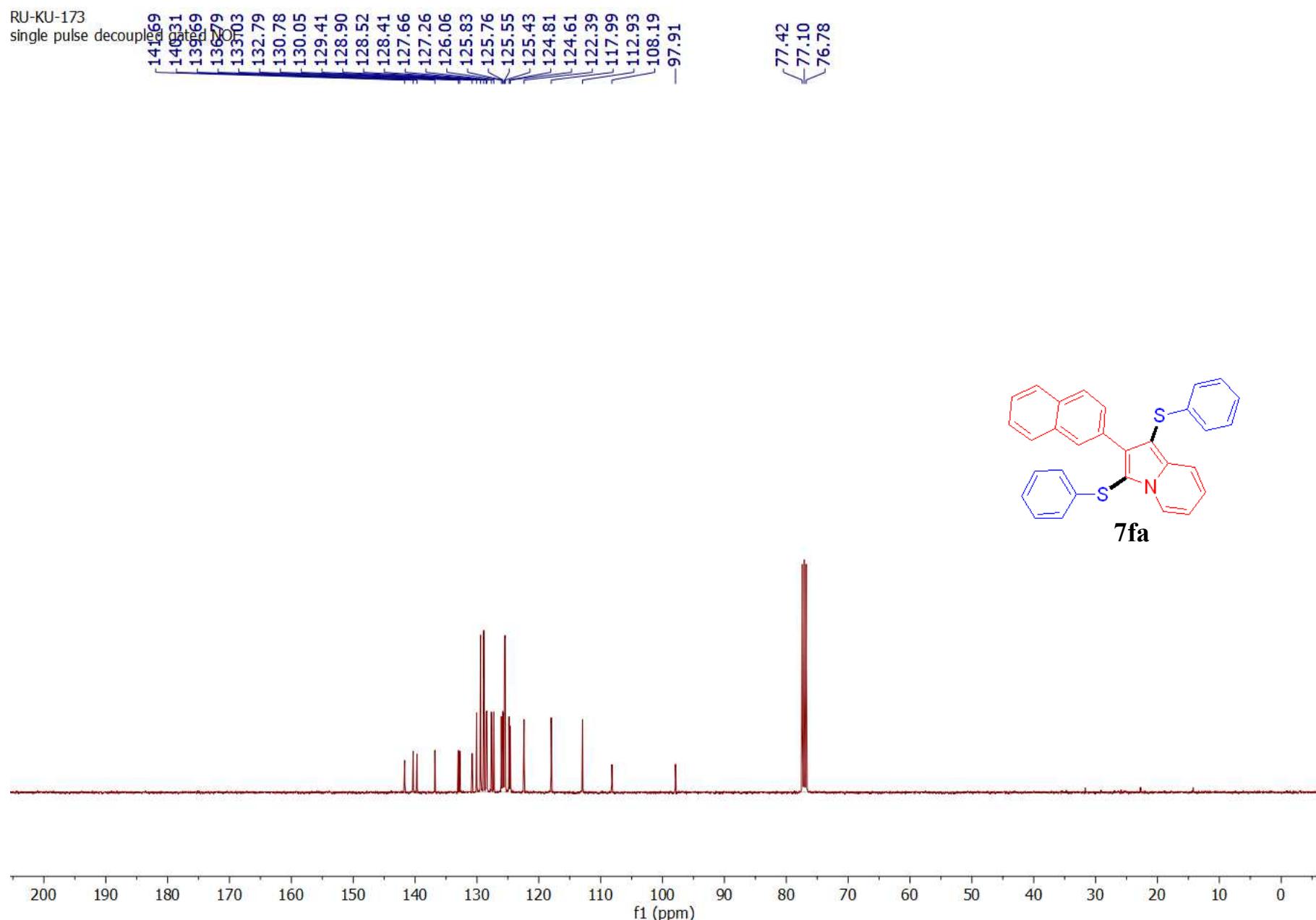
<sup>1</sup>H NMR spectrum of 7fa (400 MHz, CDCl<sub>3</sub>)



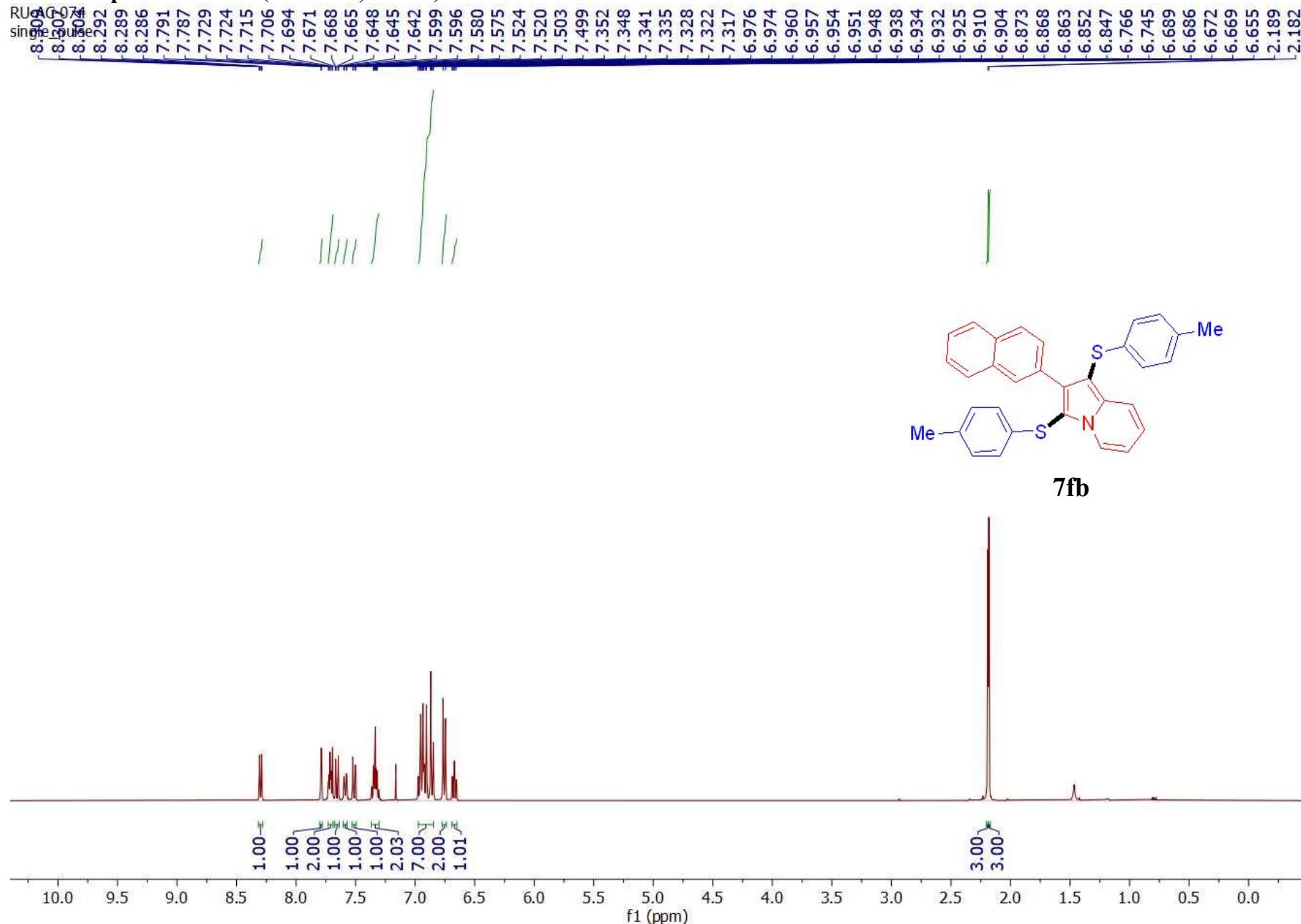
<sup>13</sup>C{H} NMR spectrum of 7fa (100 MHz, CDCl<sub>3</sub>)

RU-KU-173

single pulse decoupled



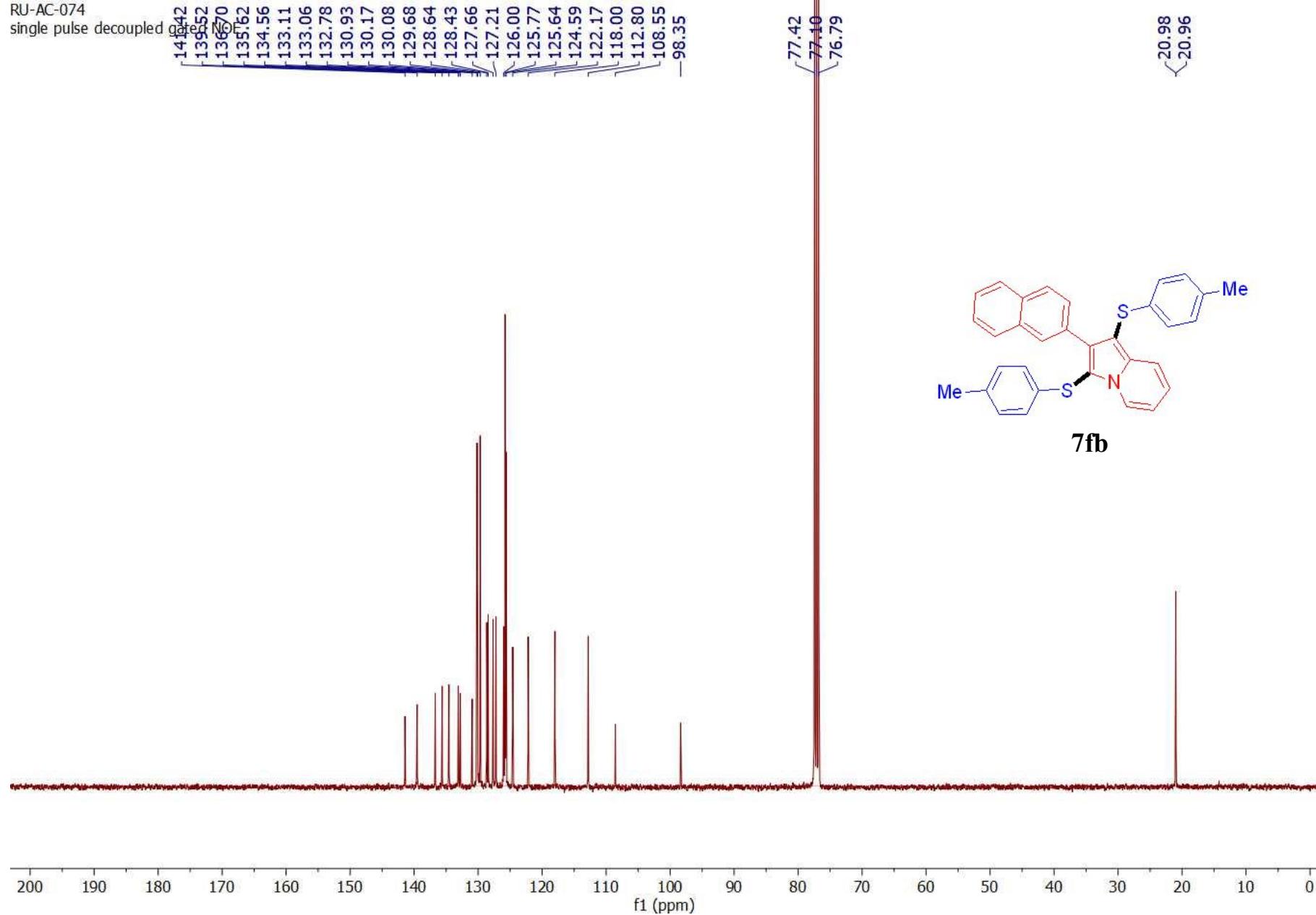
<sup>1</sup>H NMR spectrum of 7fb (400 MHz, CDCl<sub>3</sub>)



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 7fb (100 MHz,  $\text{CDCl}_3$ )

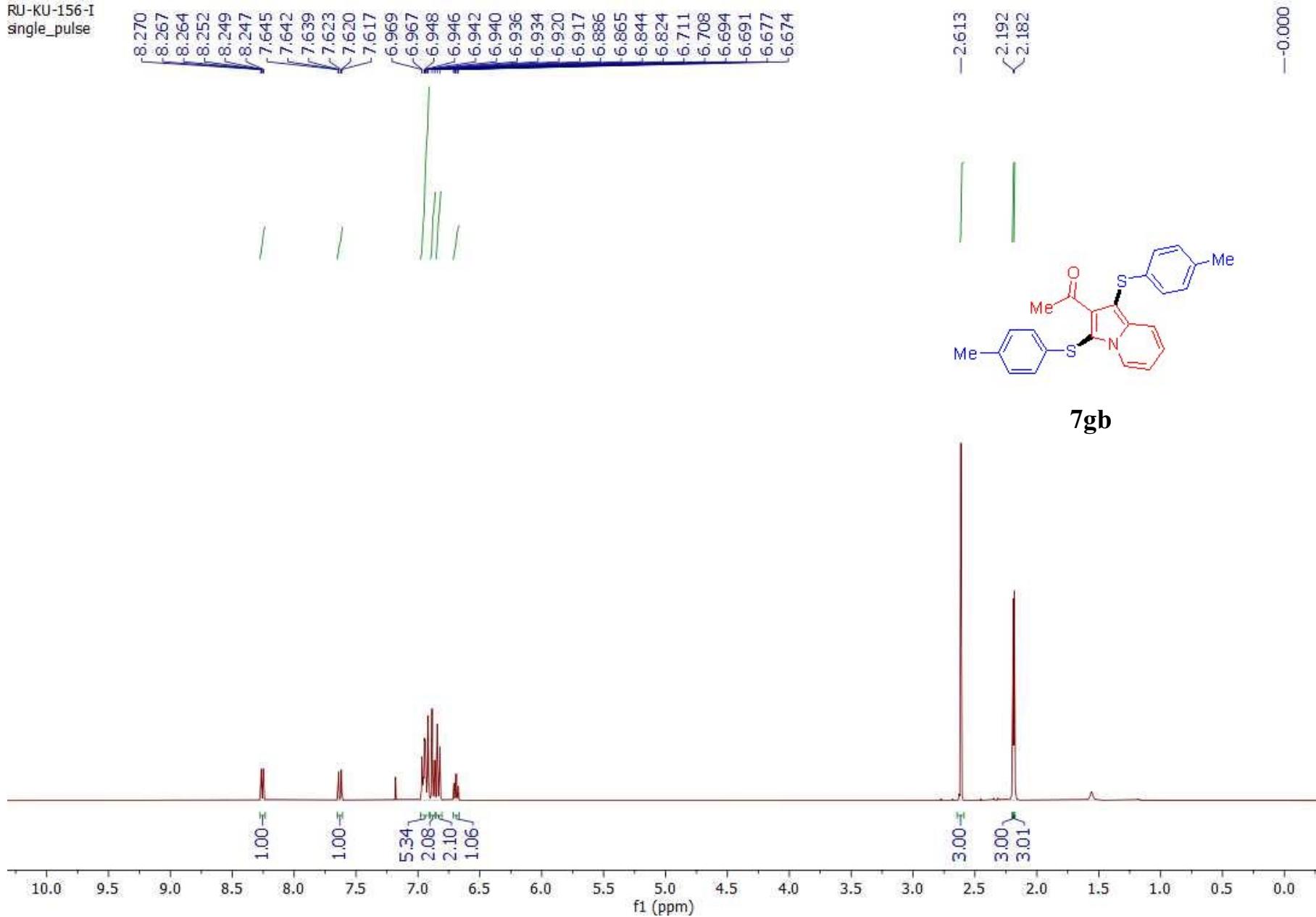
RU-AC-074

single pulse decoupled gated NOE



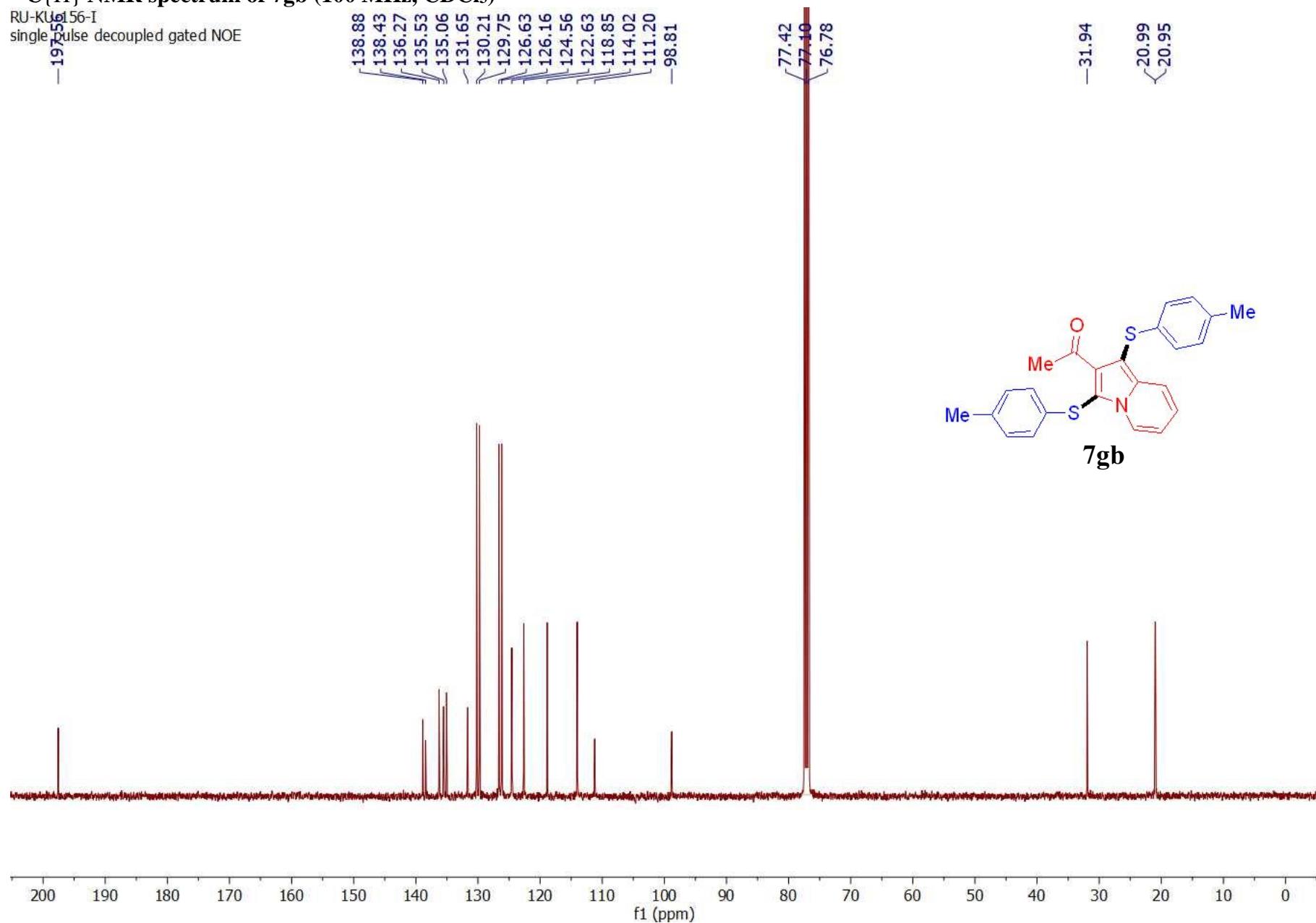
<sup>1</sup>H NMR spectrum of 7gb (400 MHz, CDCl<sub>3</sub>)

RU-KU-156-I  
single\_pulse



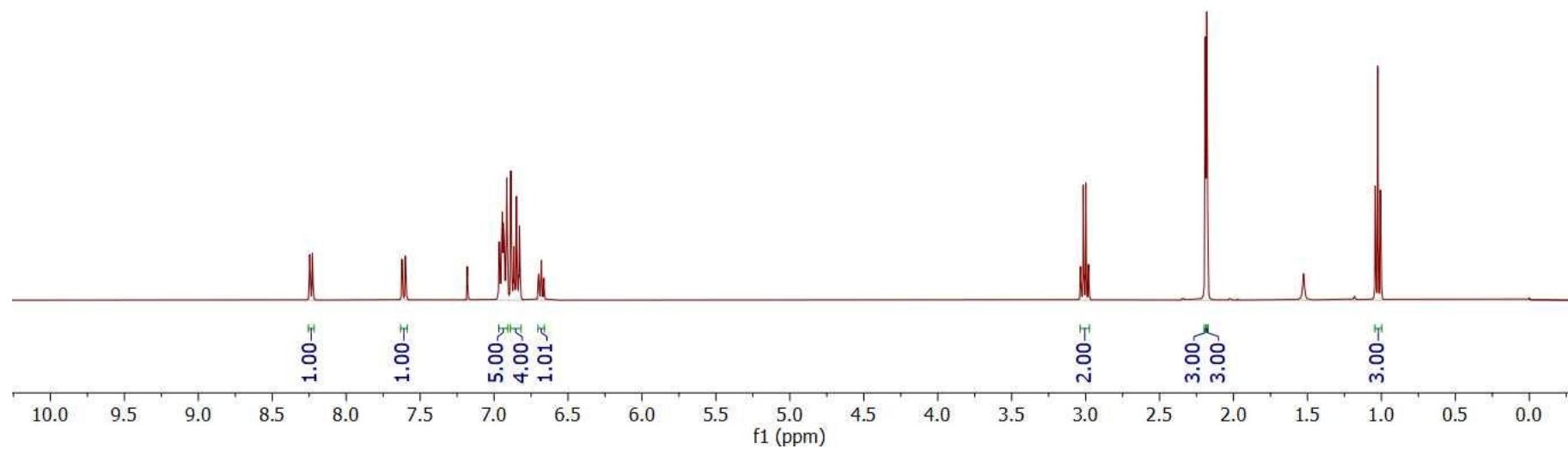
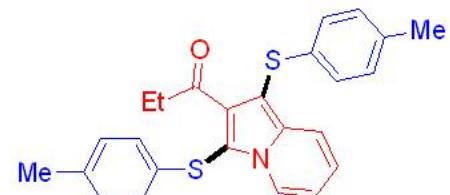
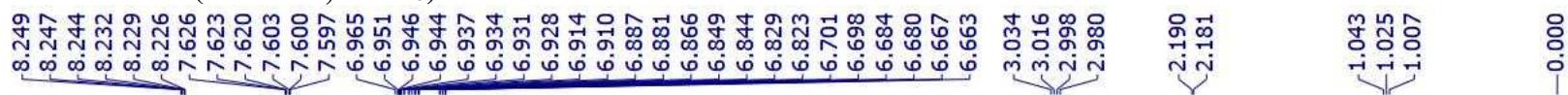
$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 7gb (100 MHz,  $\text{CDCl}_3$ )

RU-KU<sub>ol</sub> 56-I  
single pulse decoupled gated NOE



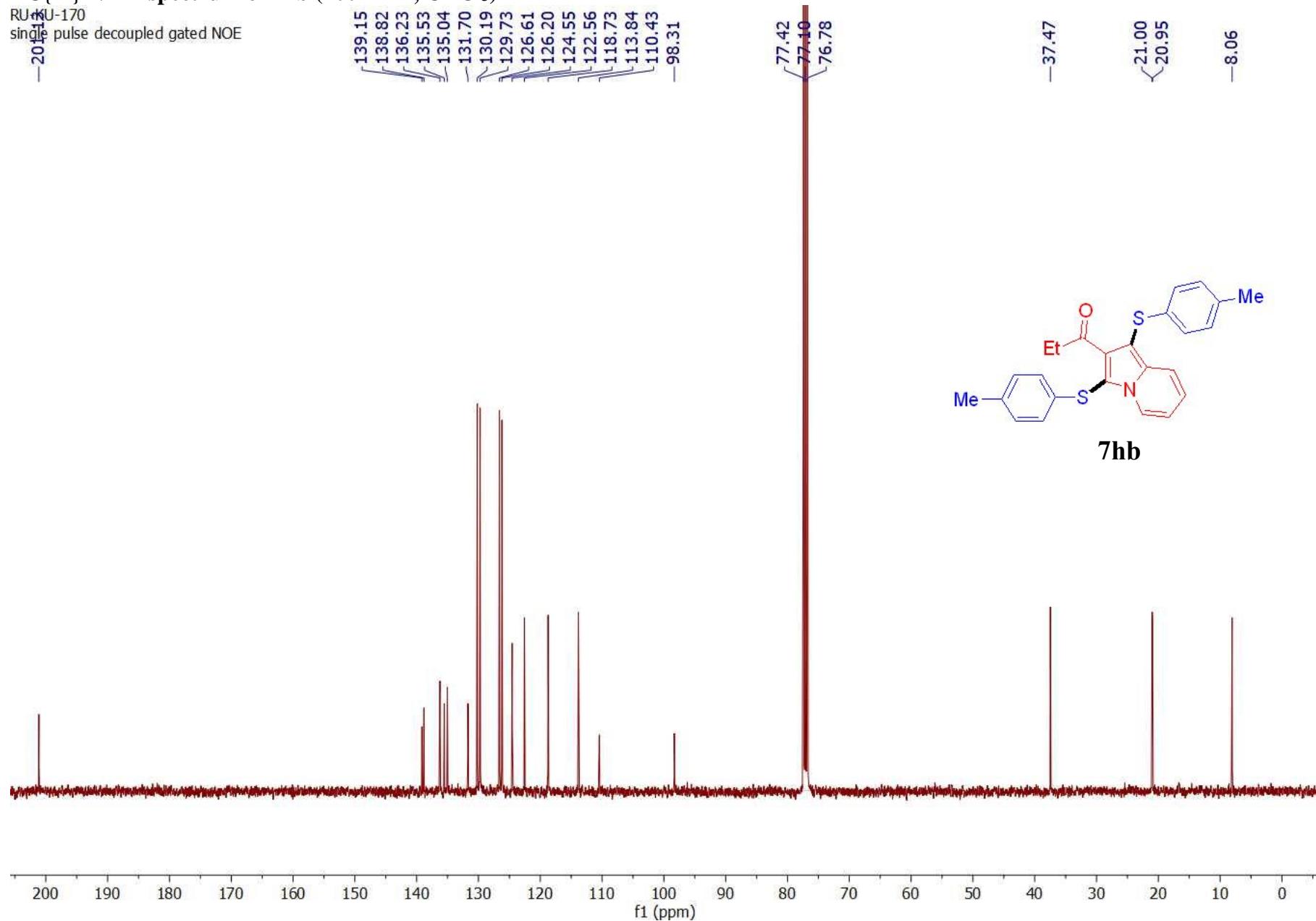
<sup>1</sup>H NMR spectrum of 7hb (400 MHz, CDCl<sub>3</sub>)

RU-KU-170  
single\_pulse

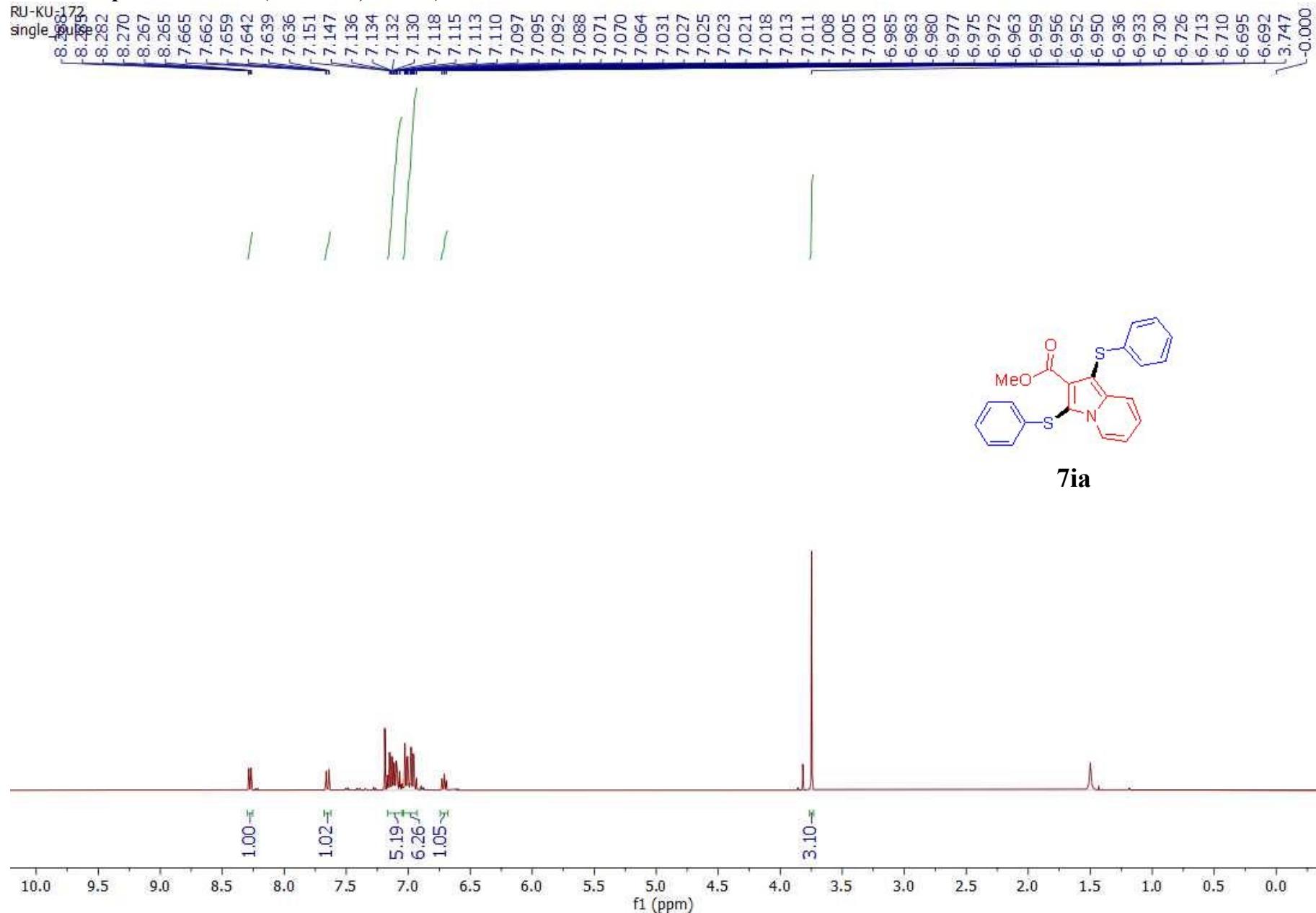


<sup>13</sup>C{H} NMR spectrum of 7hb (100 MHz, CDCl<sub>3</sub>)

RU KU-170  
single pulse decoupled gated NOE  
-201

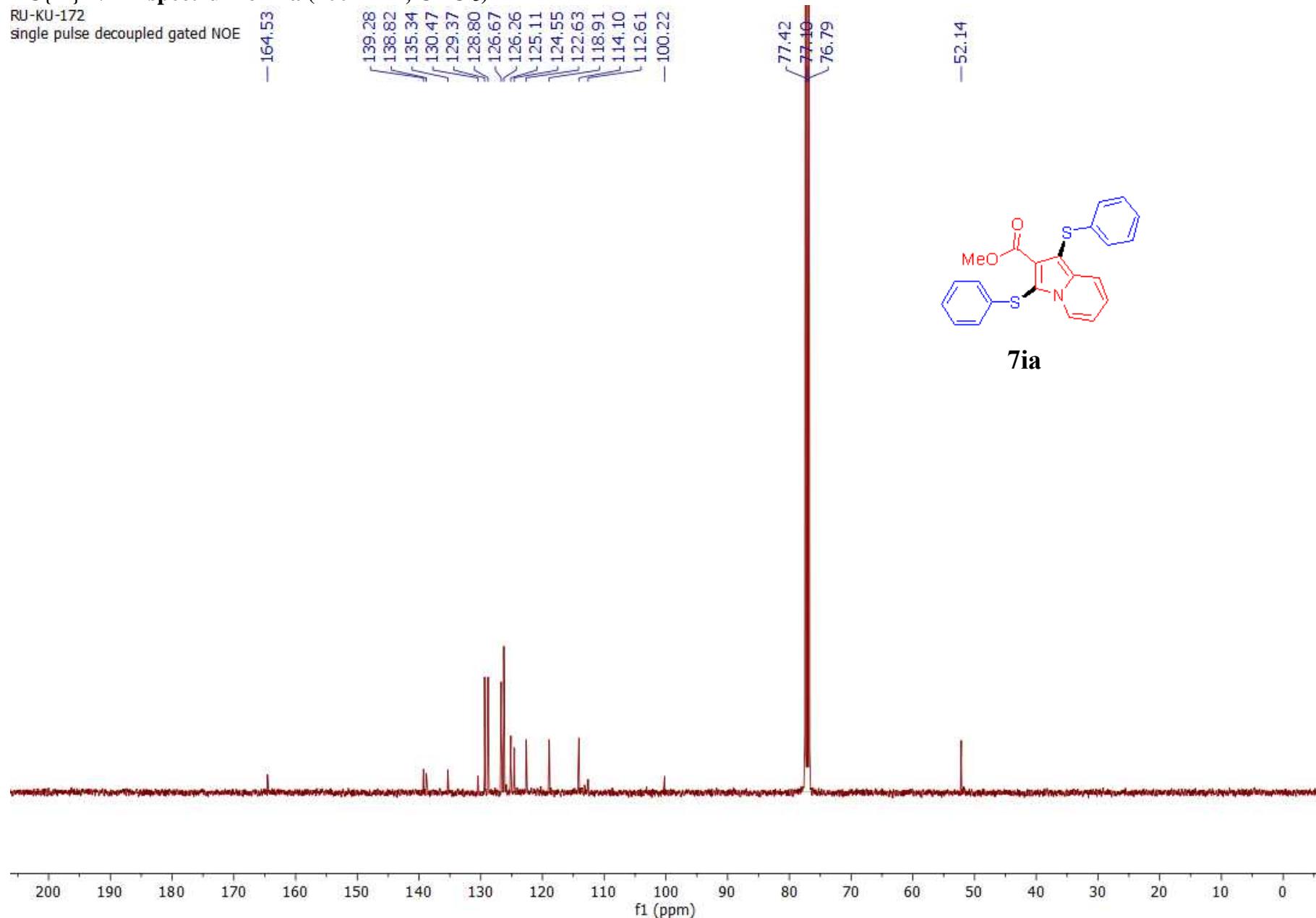


<sup>1</sup>H NMR spectrum of 7ia (400 MHz, CDCl<sub>3</sub>)

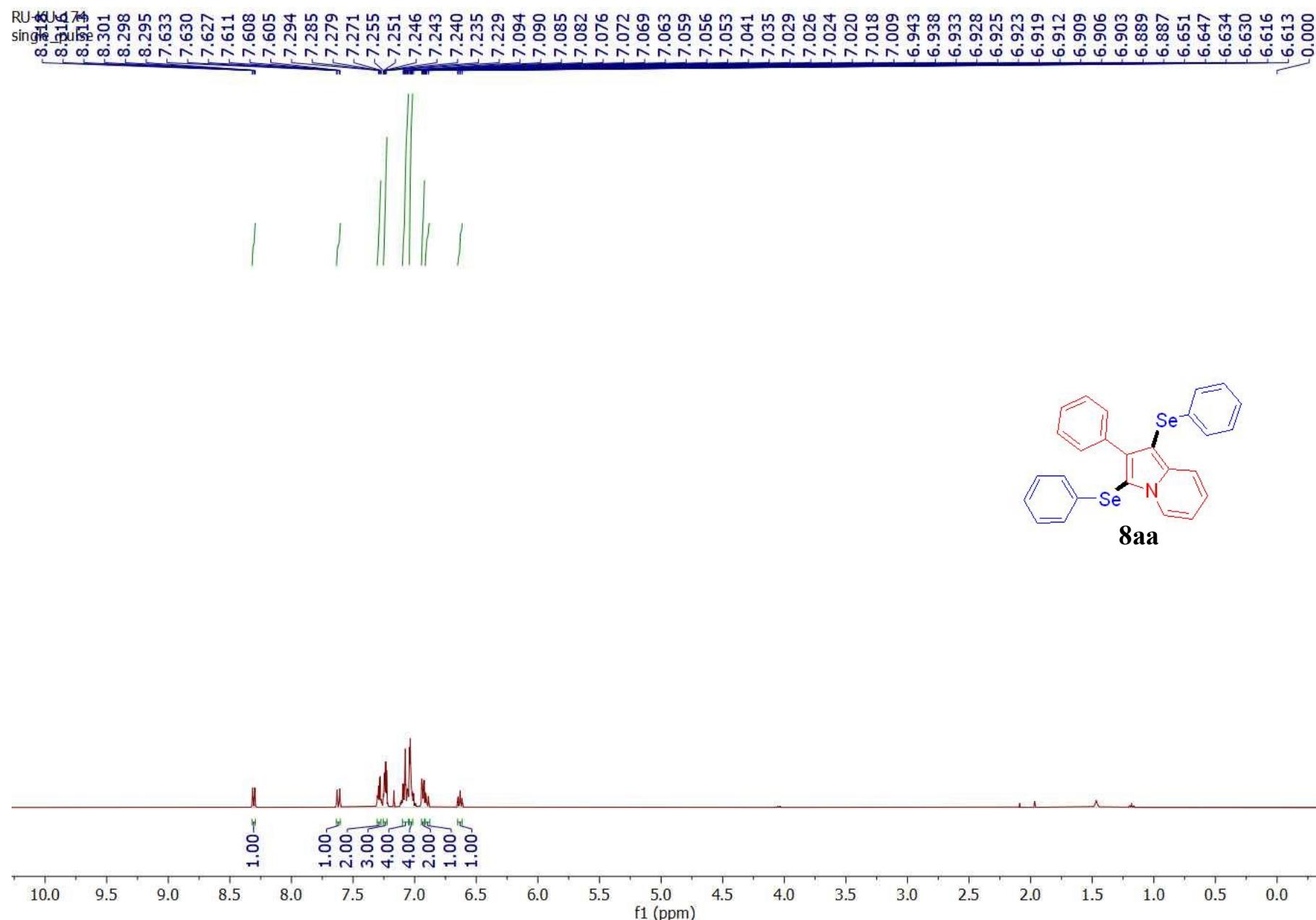


$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 7ia (100 MHz,  $\text{CDCl}_3$ )

RU-KU-172  
single pulse decoupled gated NOE



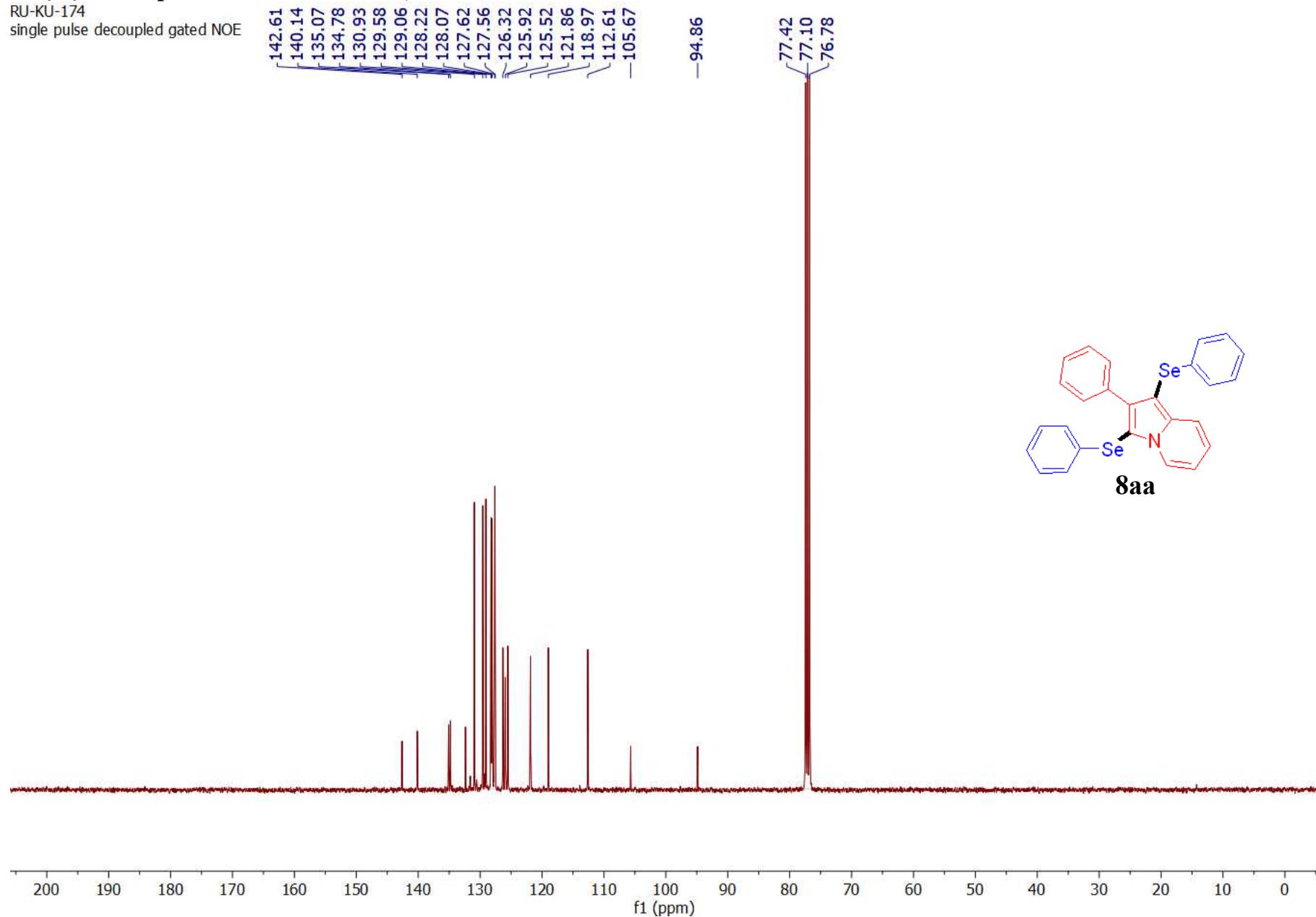
<sup>1</sup>H NMR spectrum of 8aa (400 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C{H} NMR spectrum of 7ia (100 MHz, CDCl<sub>3</sub>)

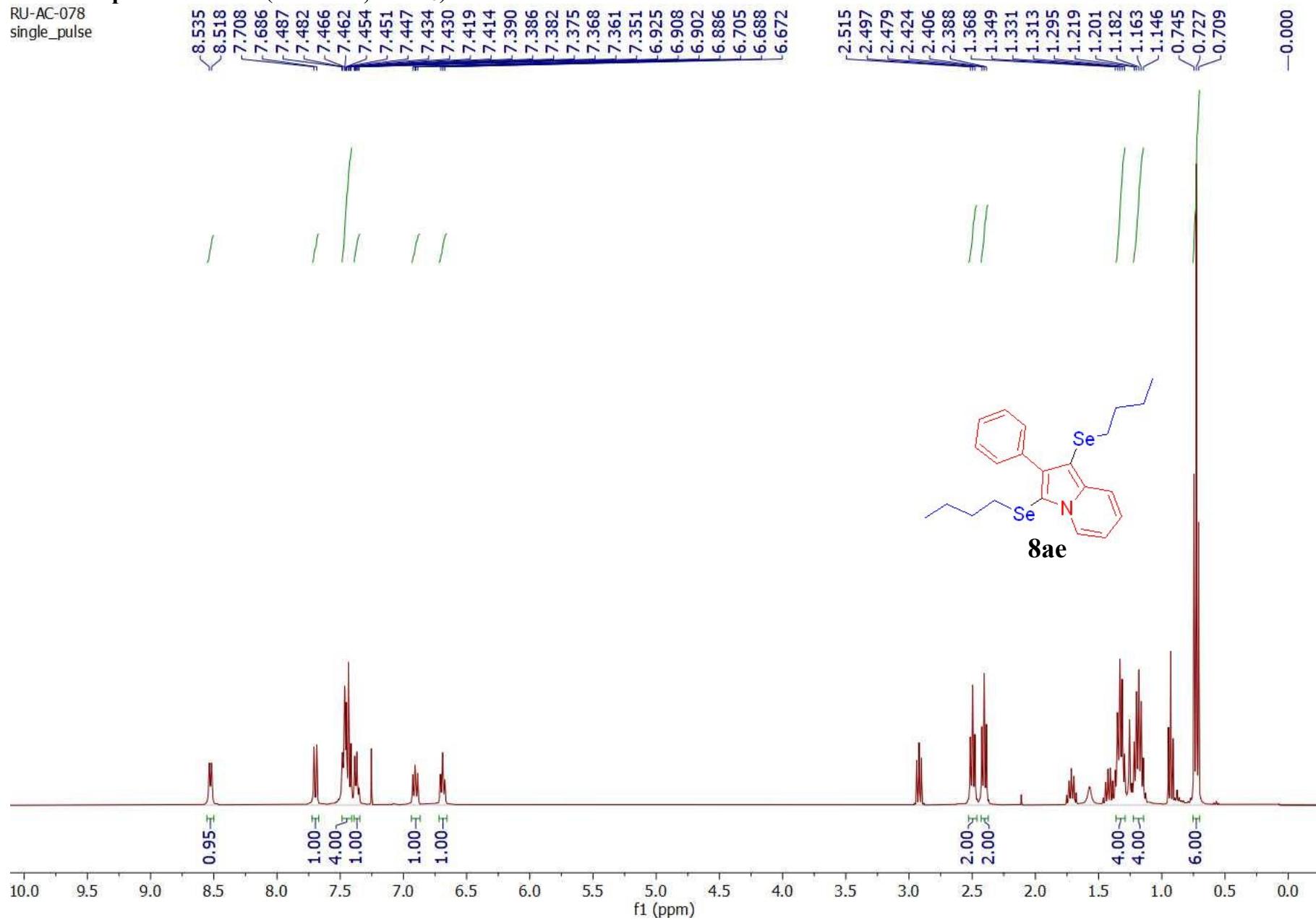
RU-KU-174

single pulse decoupled gated NOE



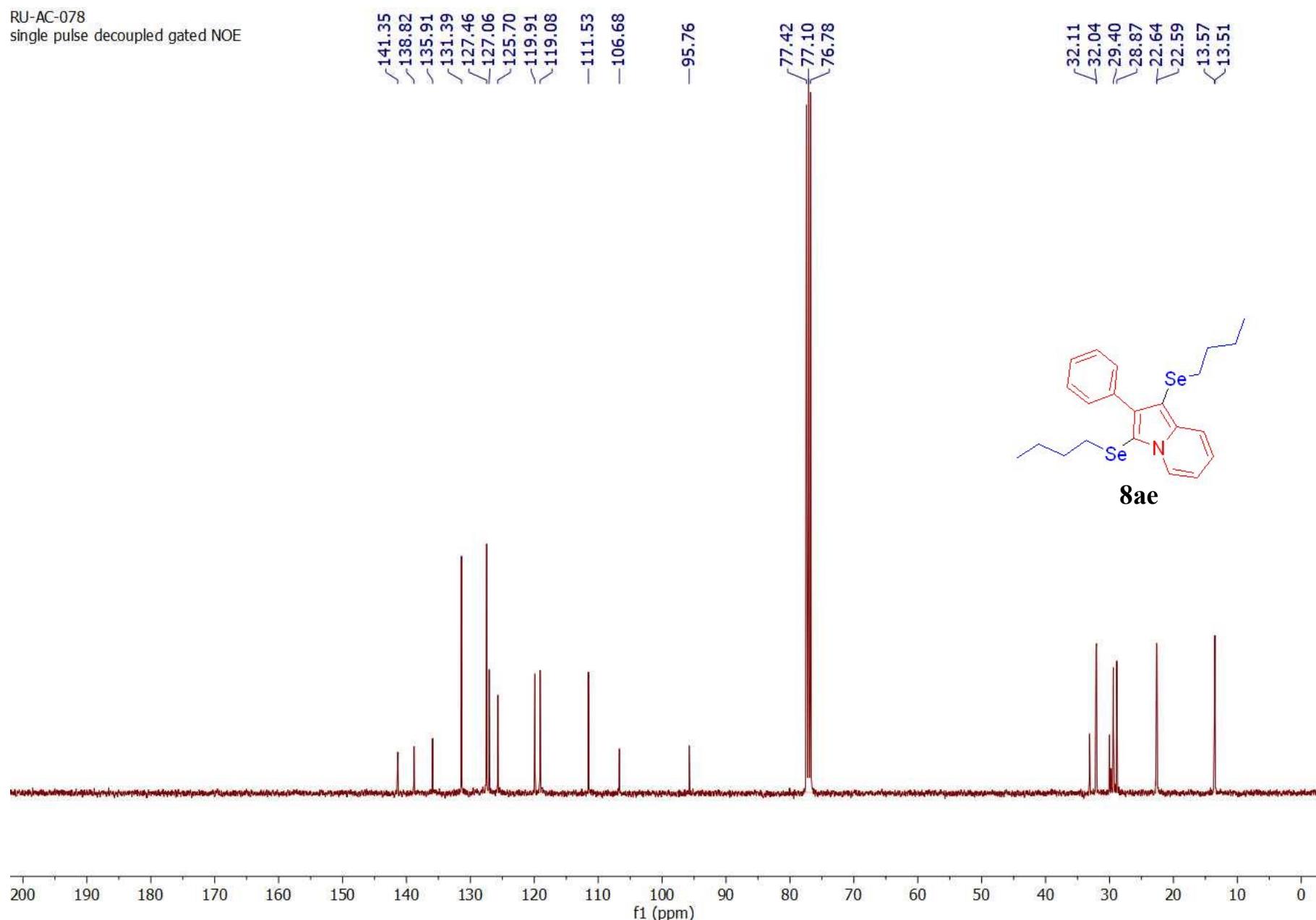
<sup>1</sup>H NMR spectrum of 8ae (400 MHz, CDCl<sub>3</sub>)

RU-AC-078  
single\_pulse



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 8ae (100 MHz,  $\text{CDCl}_3$ )

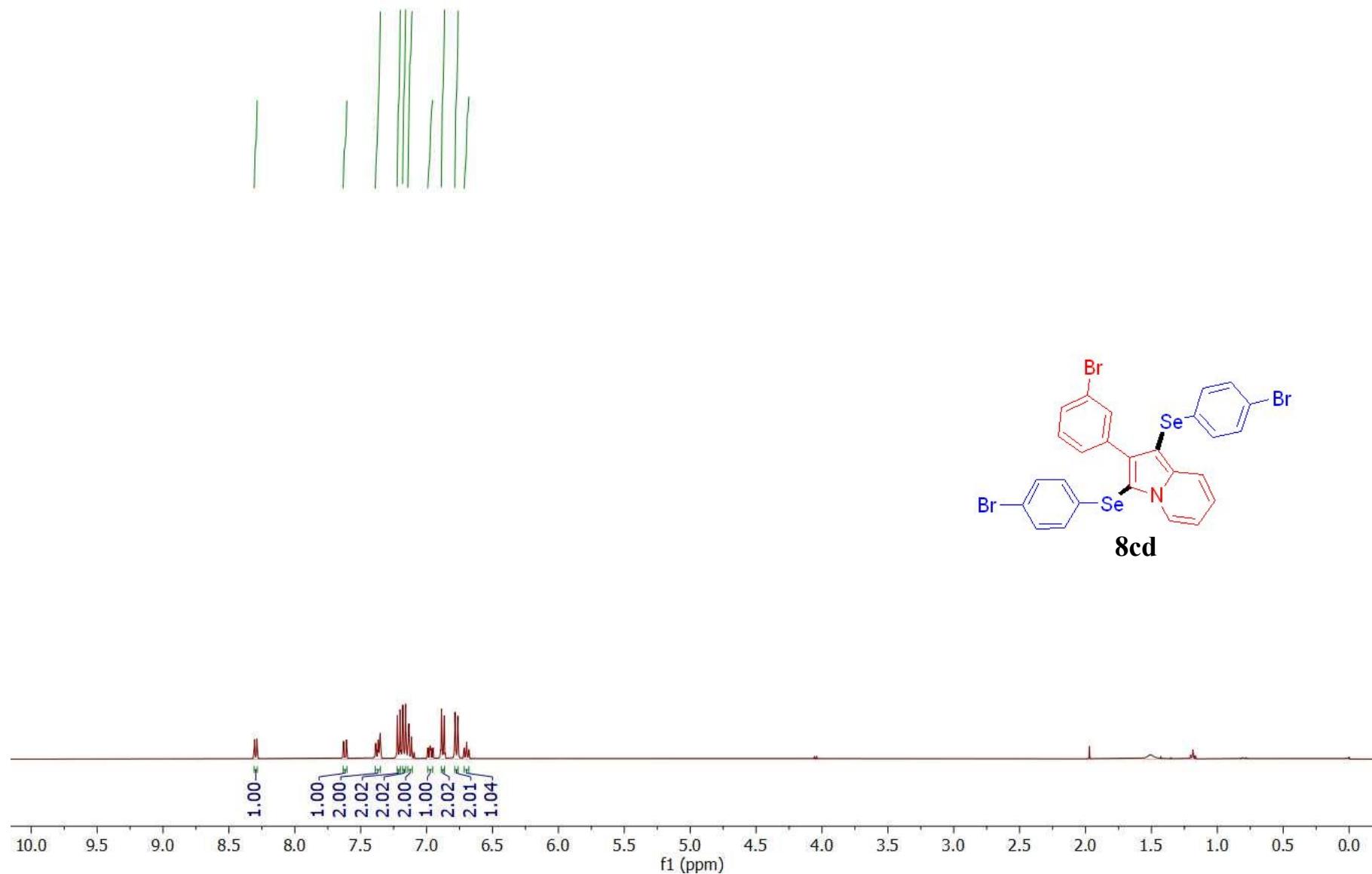
RU-AC-078  
single pulse decoupled gated NOE



<sup>1</sup>H NMR spectrum of 8cd (400 MHz, CDCl<sub>3</sub>)

RU-AC-089  
single\_pulse

-0.000



$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 8cd (100 MHz,  $\text{CDCl}_3$ )

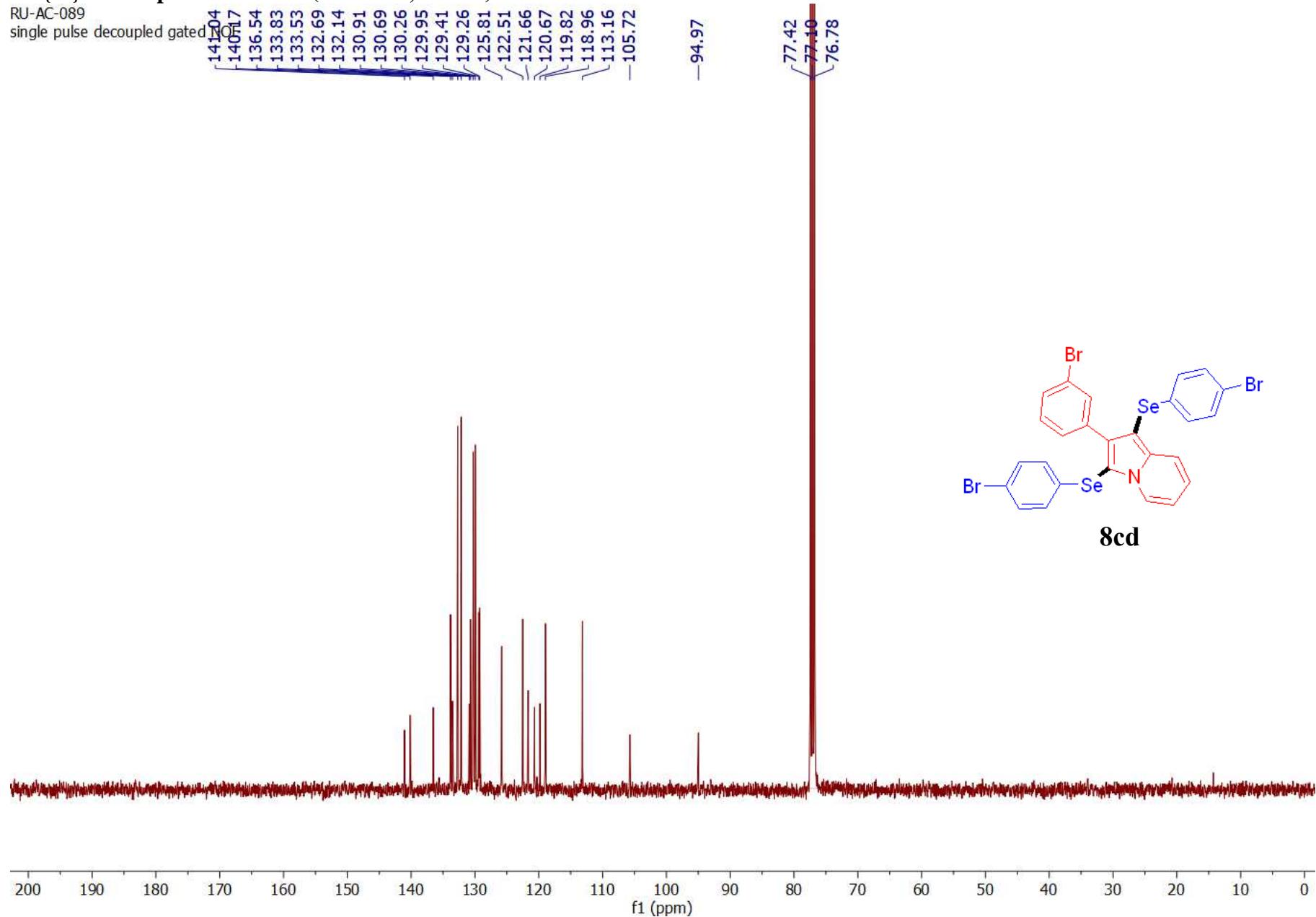
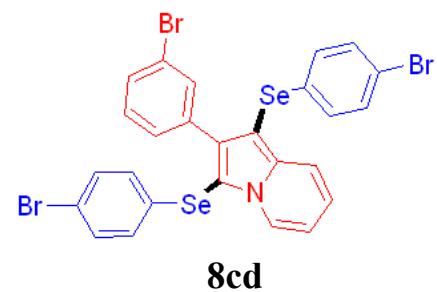
RU-AC-089

single pulse decoupled gated NOE

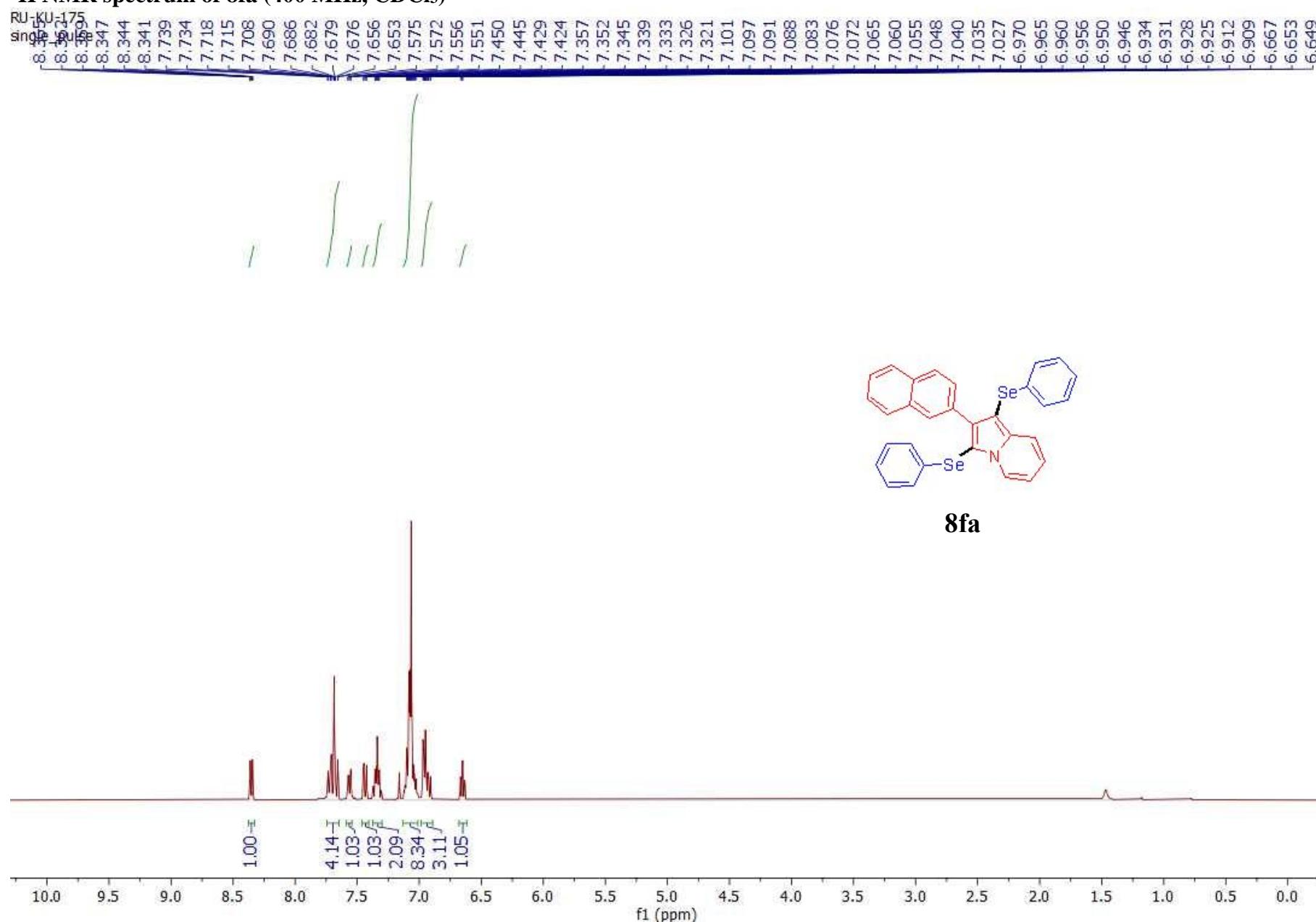
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130.91  
130.69  
130.26  
129.95  
129.41  
129.26  
125.81  
125.51  
121.66  
120.67  
119.82  
118.96  
113.16  
105.72

-94.97

77.42  
77.10  
76.78



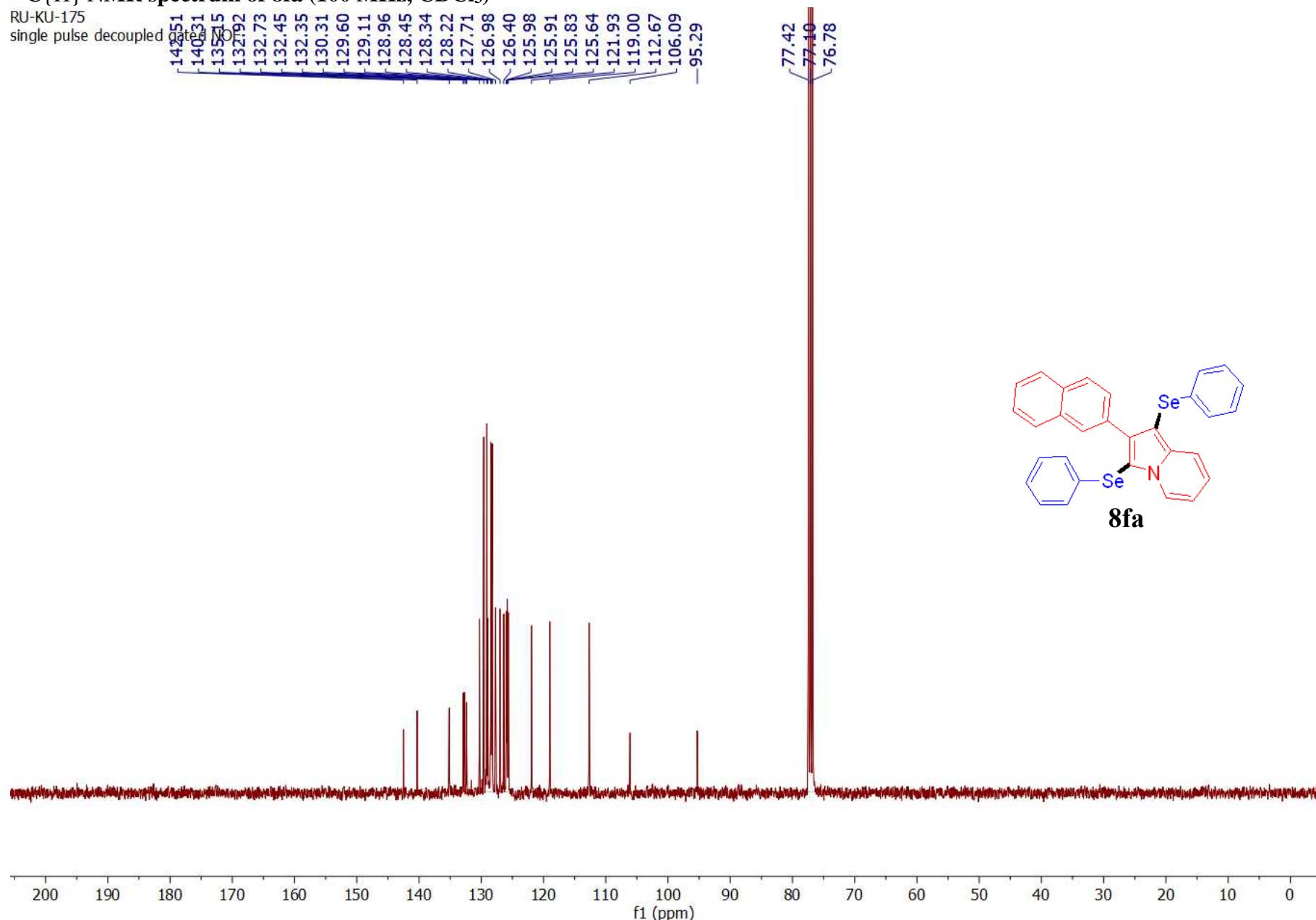
**<sup>1</sup>H NMR spectrum of 8fa (400 MHz, CDCl<sub>3</sub>)**



<sup>13</sup>C{H} NMR spectrum of 8fa (100 MHz, CDCl<sub>3</sub>)

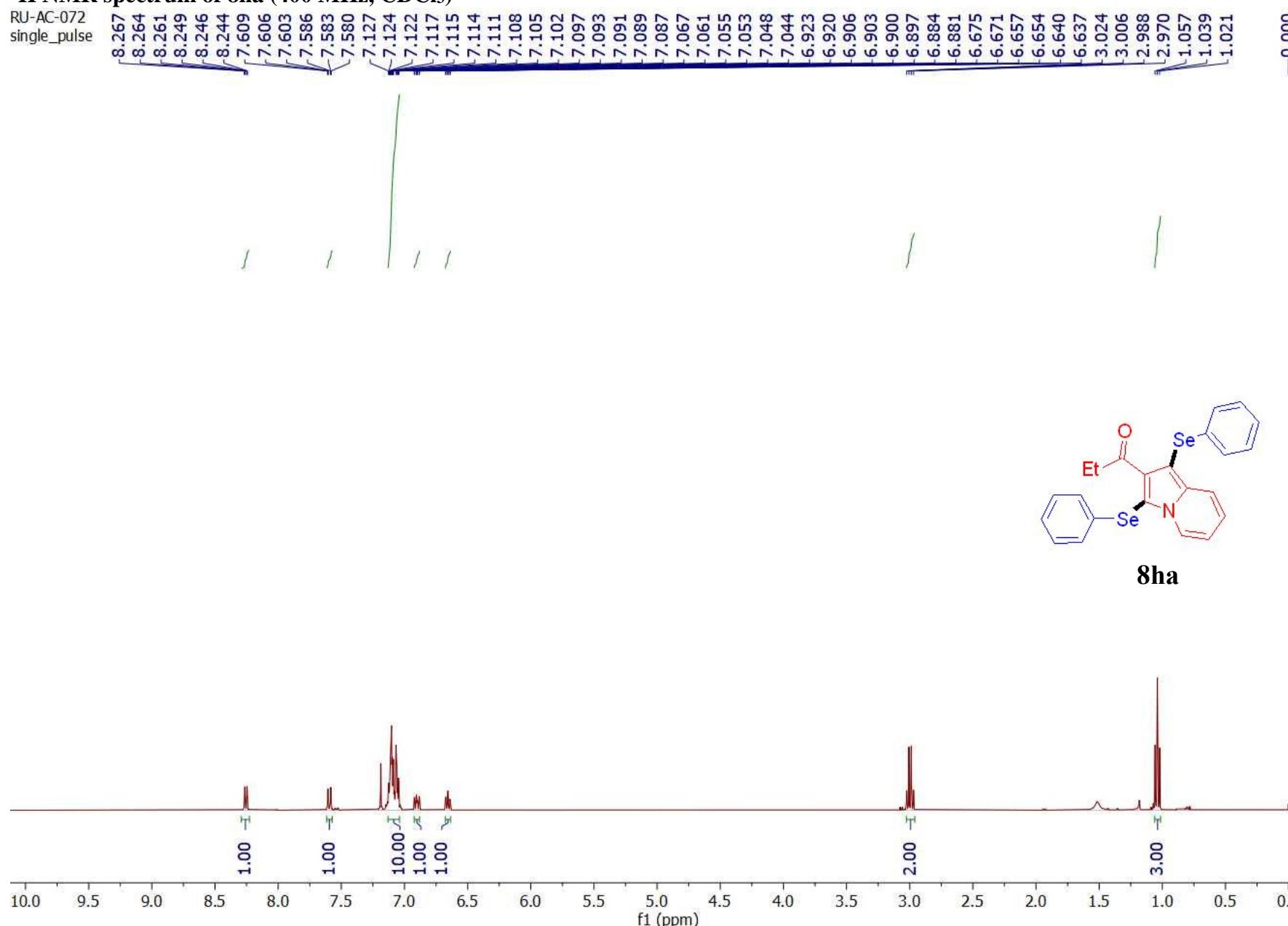
RU-KU-175

single pulse decoupled gated



**<sup>1</sup>H NMR spectrum of 8ha (400 MHz, CDCl<sub>3</sub>)**

RU-AC-072  
single\_pulse



**$^{13}\text{C}\{\text{H}\}$  NMR spectrum of 8ha (100 MHz,  $\text{CDCl}_3$ )**

RU-AC-078  
single pulse SE decoupled gated NOE

