

**Lewis Acid-Promoted Synthesis of 3-Tosylisoindolinone from 3-Hydroxyisoindolinone:  
Access to Complex Fuopyrroloisoindoleione**

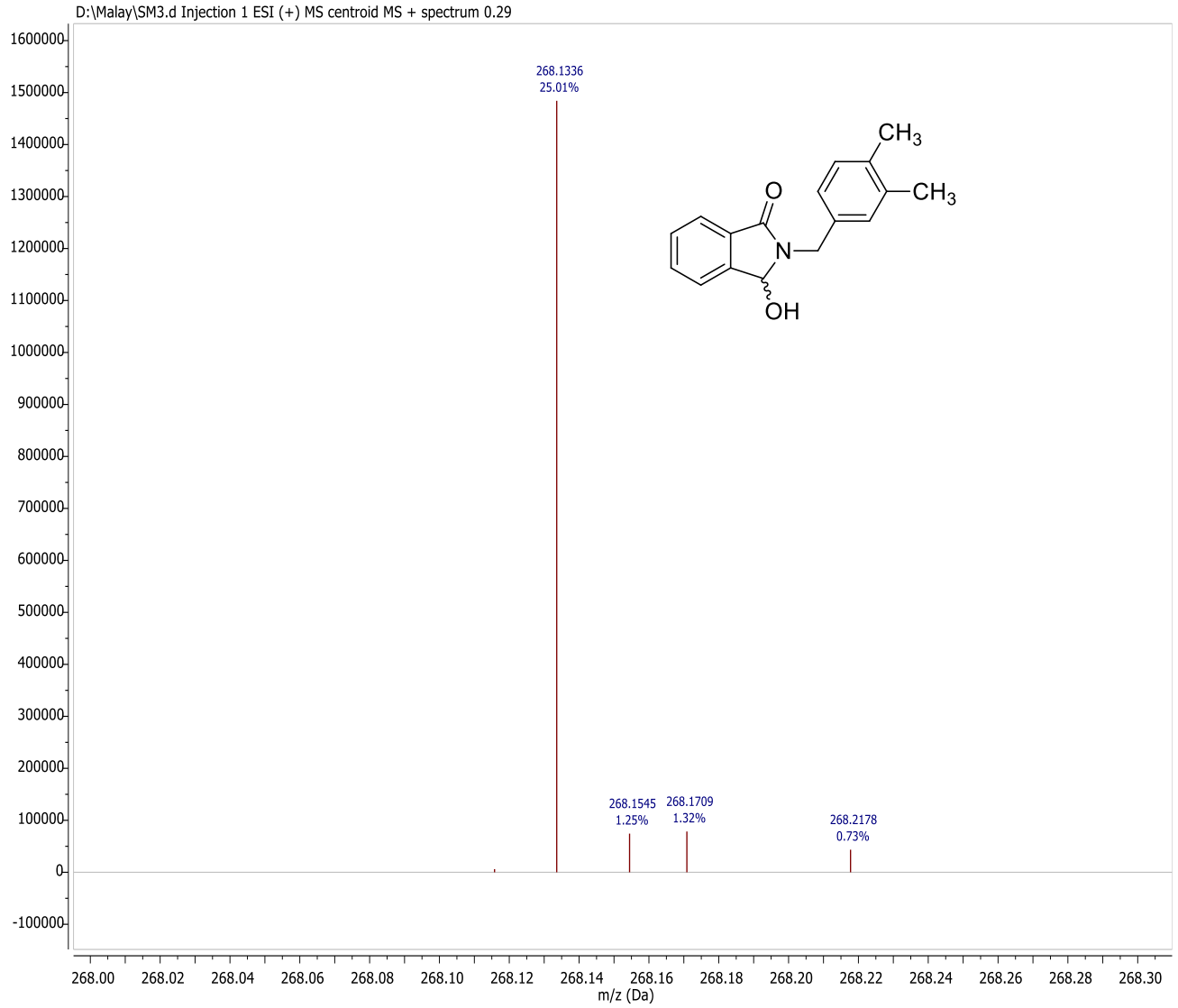
Malay Das, Virendra Kumar, Pallav Jyoti Arandhara, Subhamoy Biswas and Anil K. Saikia\*

Department of Chemistry, Indian Institute of Technology Guwahati, Guwahati-781039, India, e-mail: [asaikia@iitg.ac.in](mailto:asaikia@iitg.ac.in)

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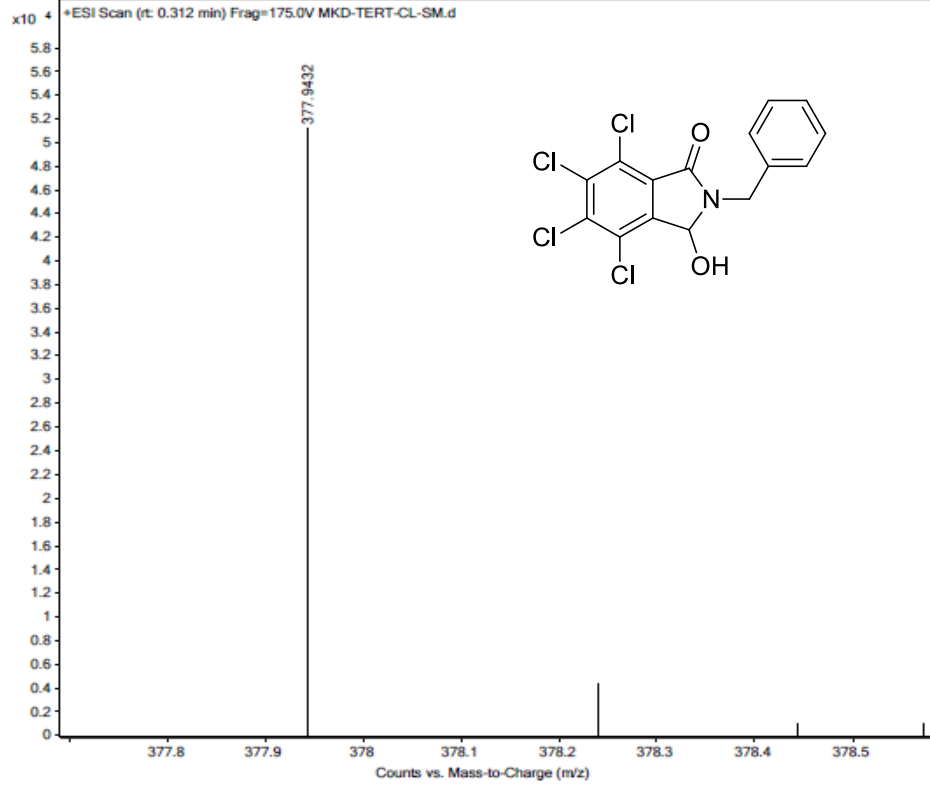
|                                      |               |
|--------------------------------------|---------------|
| 1. HRMS spectra of all new compounds | <b>S2-S28</b> |
|--------------------------------------|---------------|

# HRMS spectrum of compound **1c**

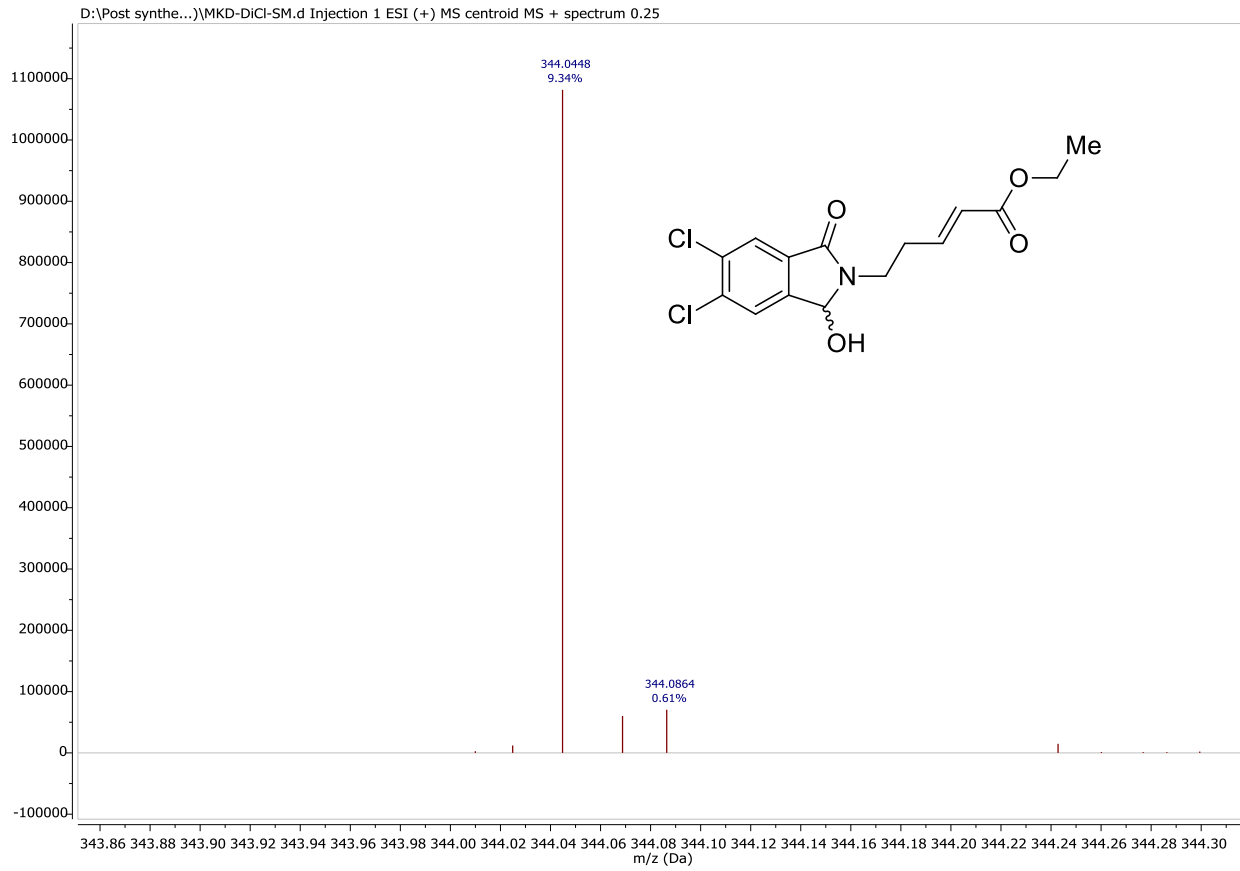


# HRMS spectrum of compound 11

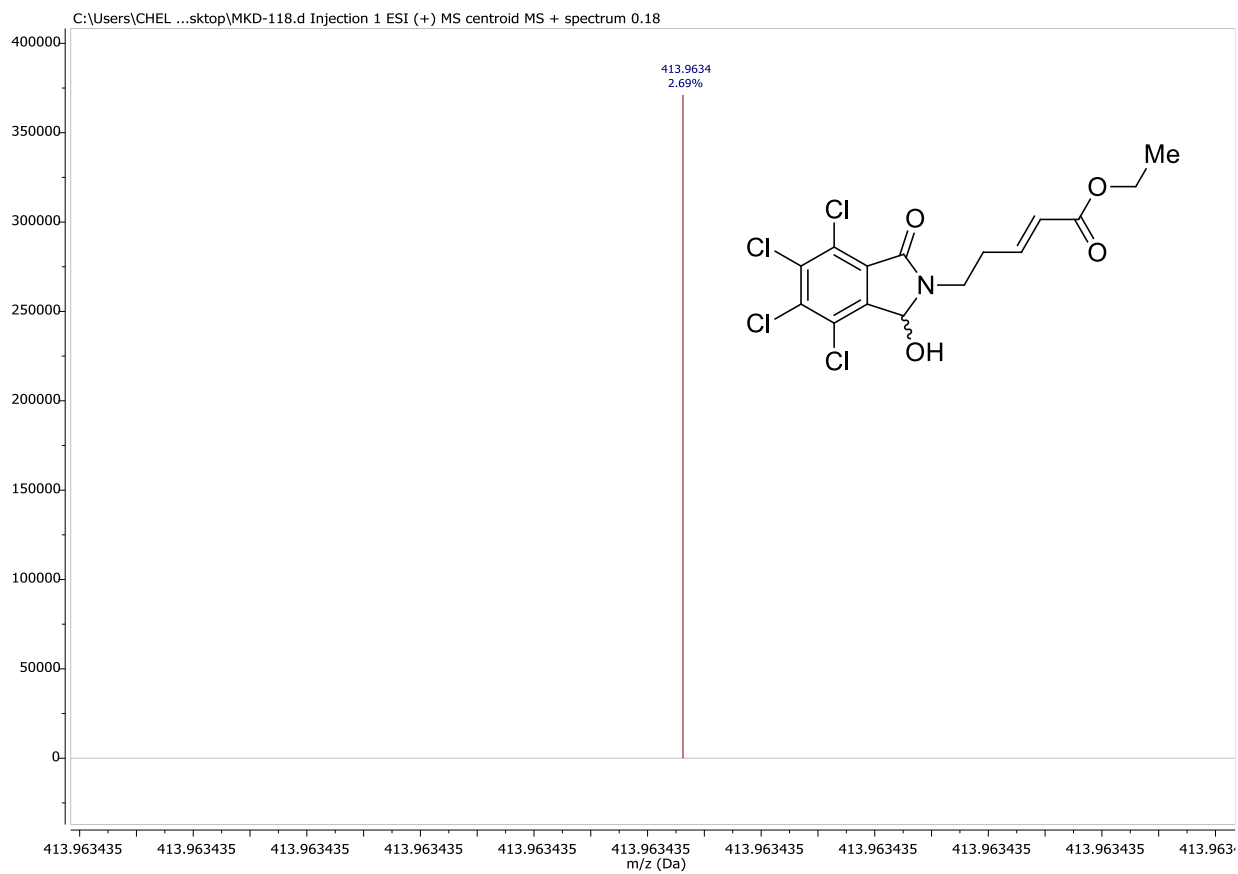
|                    |                   |                               |         |                        |                                    |
|--------------------|-------------------|-------------------------------|---------|------------------------|------------------------------------|
| <b>Sample Name</b> | WASH              | <b>Position</b>               | P1-B1   | <b>Instrument Name</b> | Instrument 1                       |
| <b>User Name</b>   |                   | <b>Inj Vol</b>                | 20      | <b>InjPosition</b>     |                                    |
| <b>Sample Type</b> | Sample            | <b>IRM Calibration Status</b> | Success | <b>Data Filename</b>   | MKD-TERT-CL-SM.d                   |
| <b>ACQ Method</b>  | ESI ALS 100-600.m | <b>Comment</b>                |         | <b>Acquired Time</b>   | 14-12-2020 12:08:46 PM (UTC+05:30) |



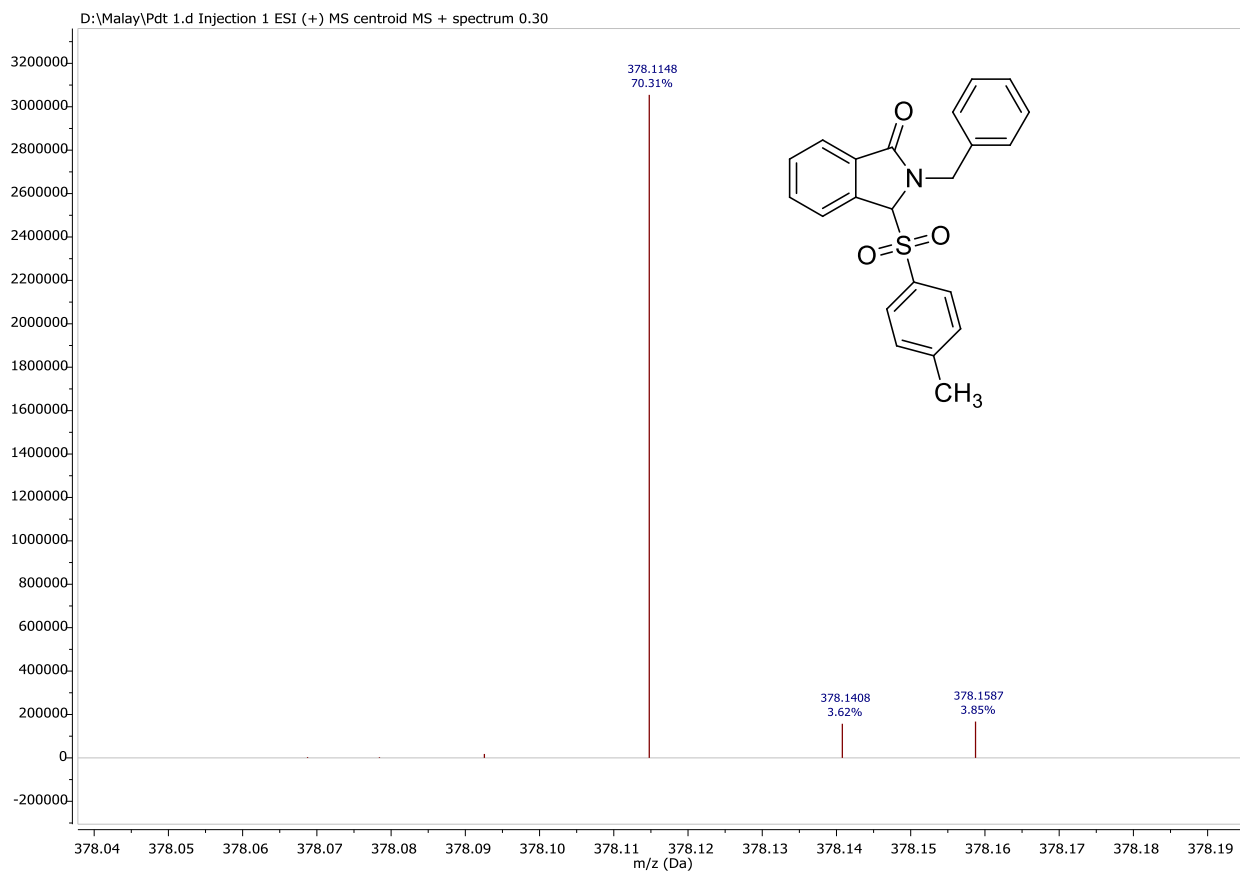
# HRMS spectrum of compound 1s



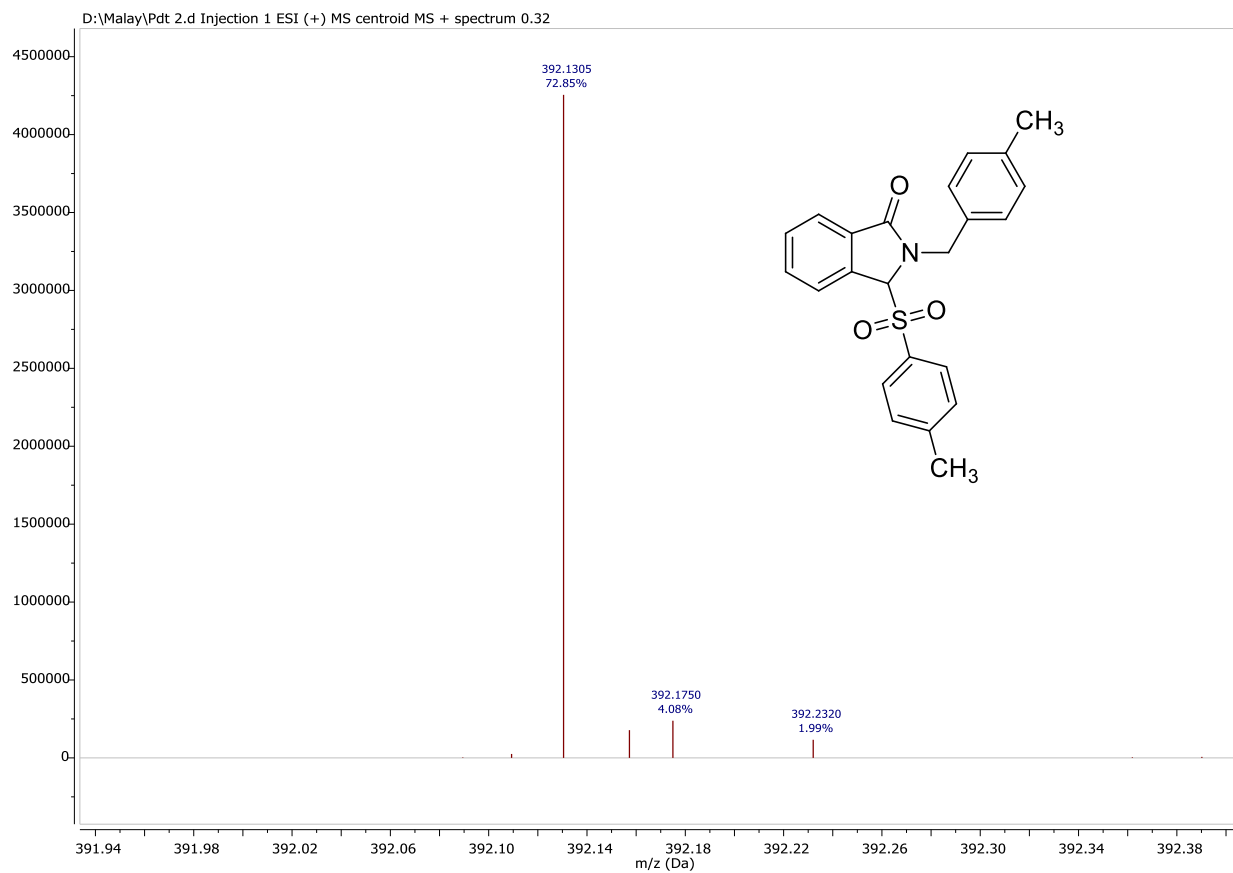
# HRMS spectrum of compound **1t**



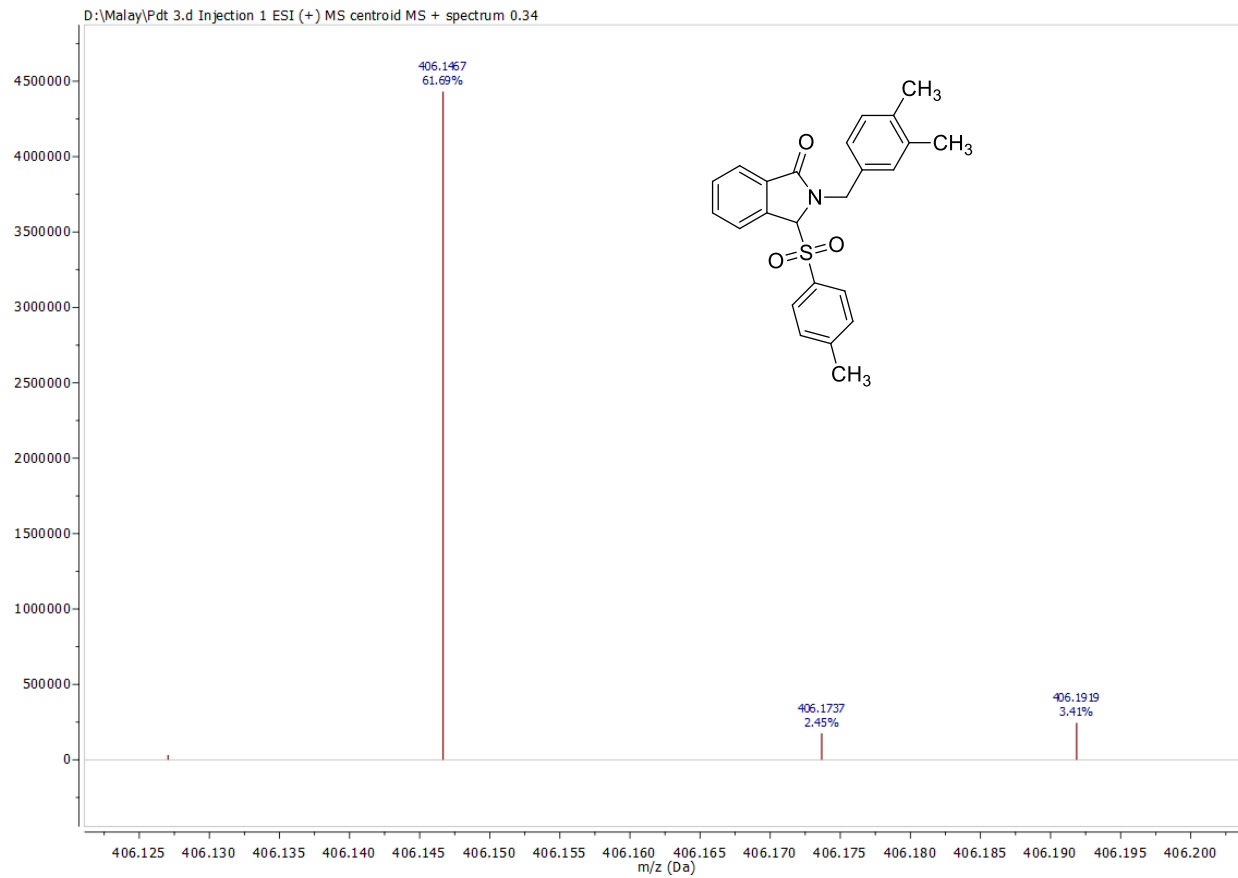
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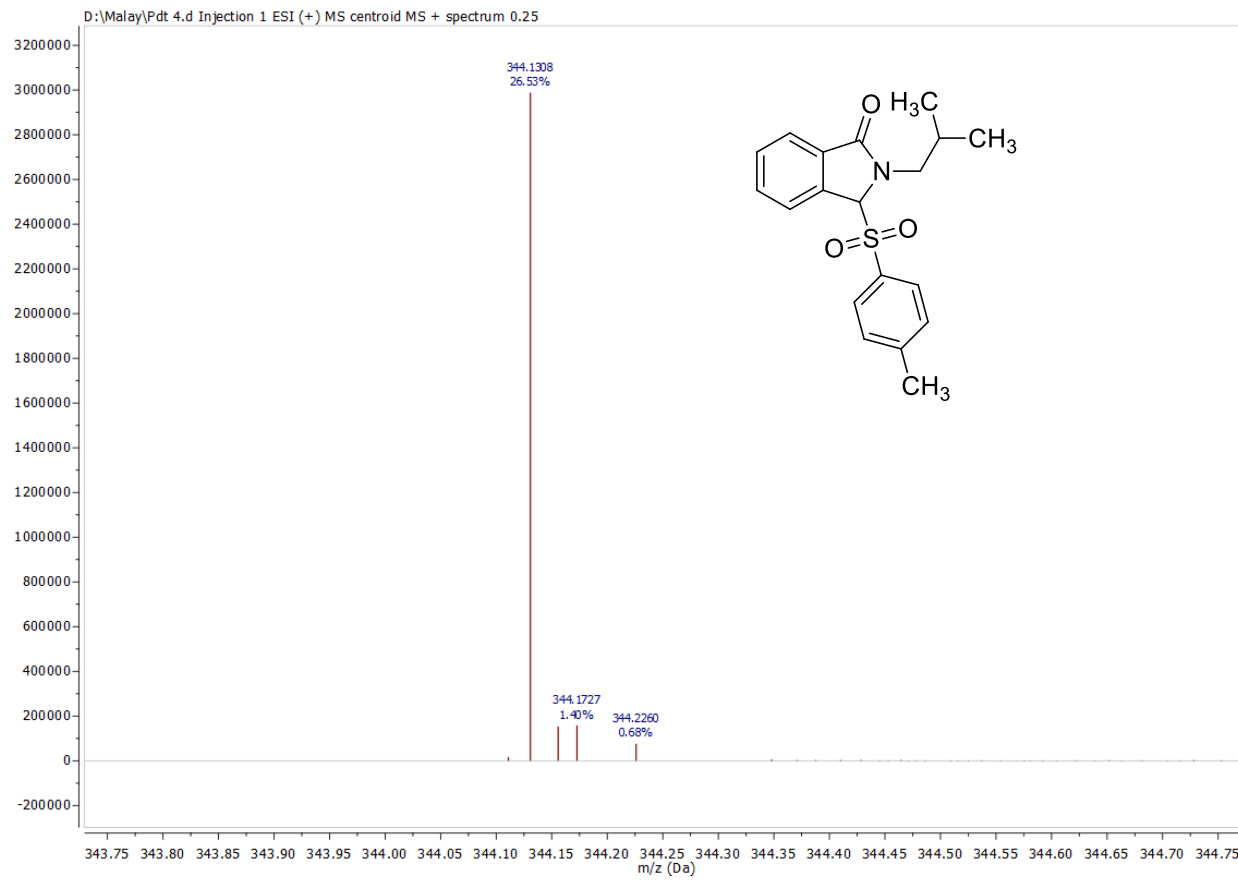
# HRMS spectrum of compound **3b**



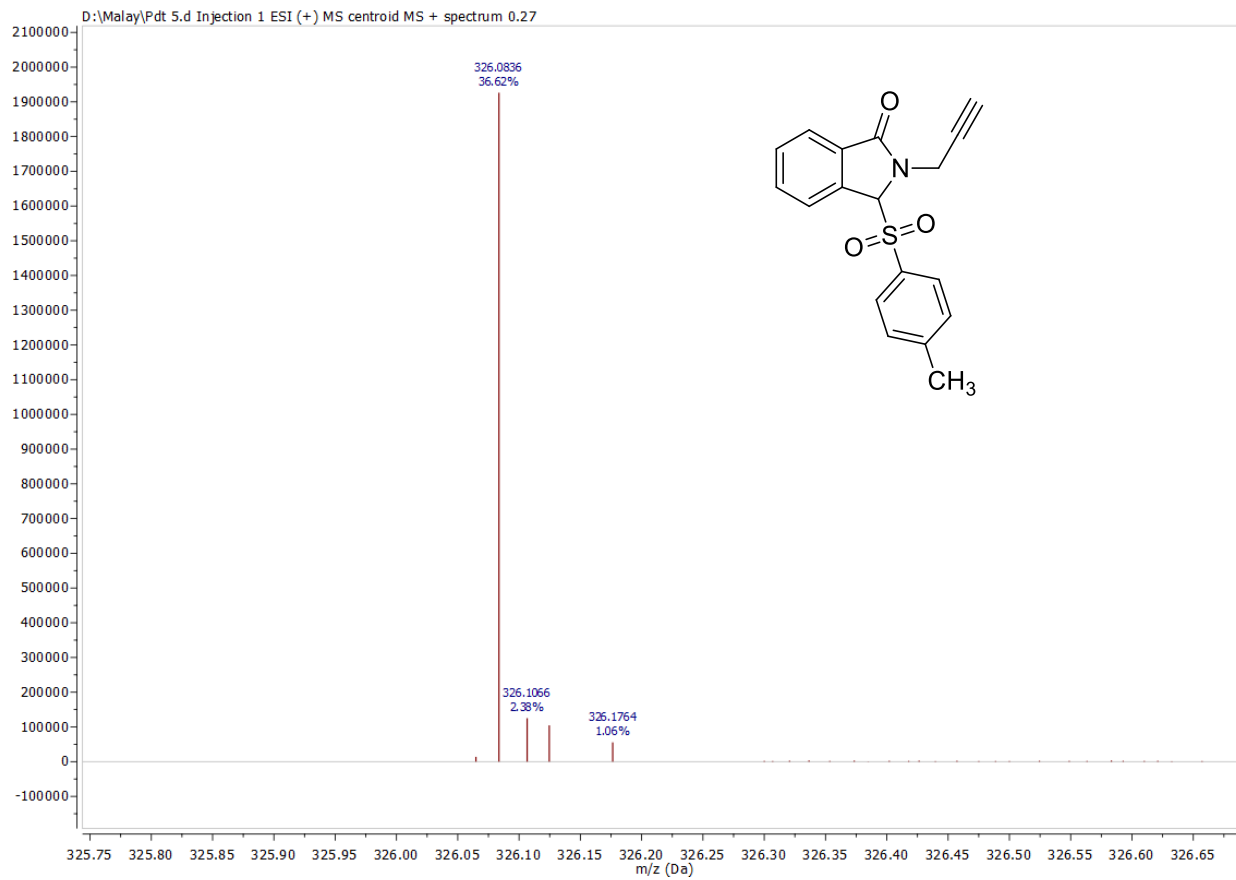
# HRMS spectrum of compound **3c**



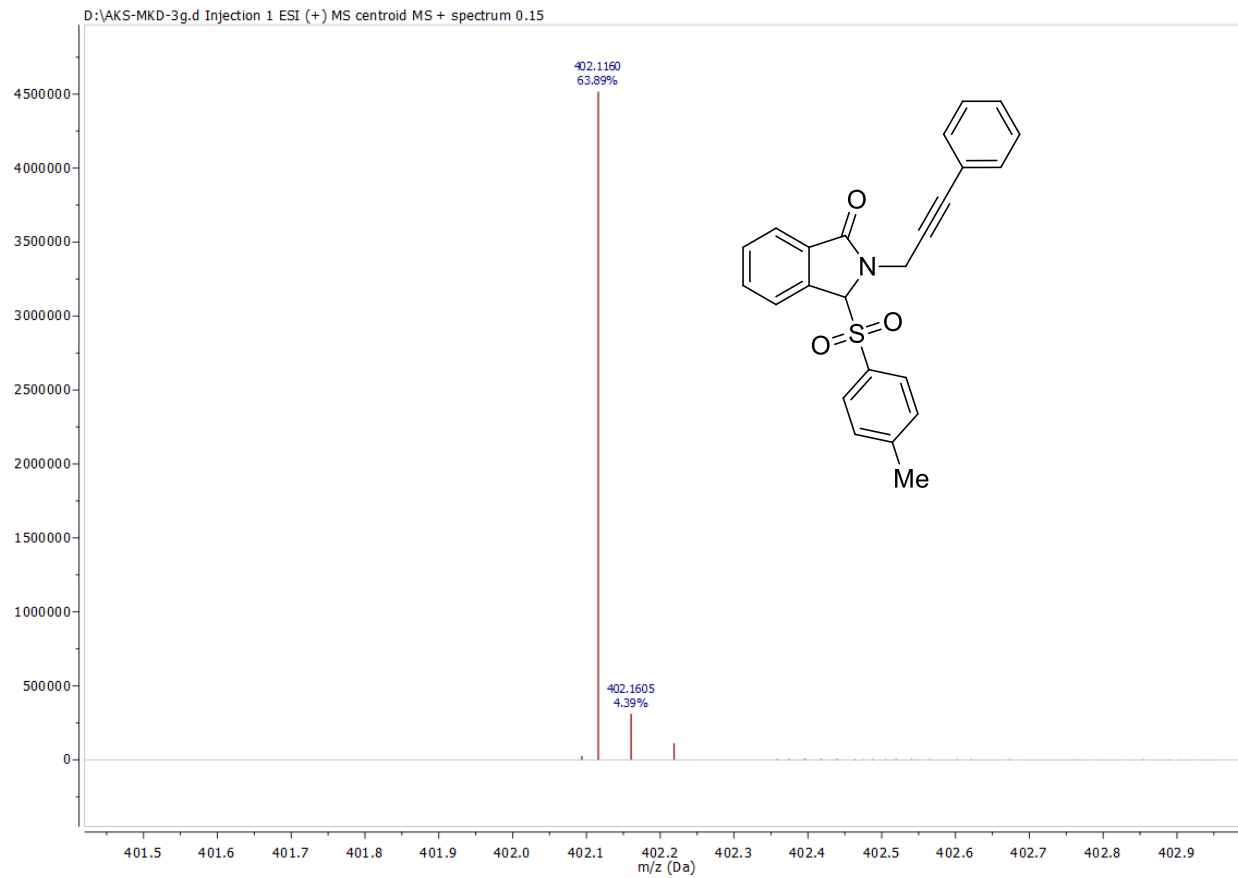
# HRMS spectrum of compound **3d**



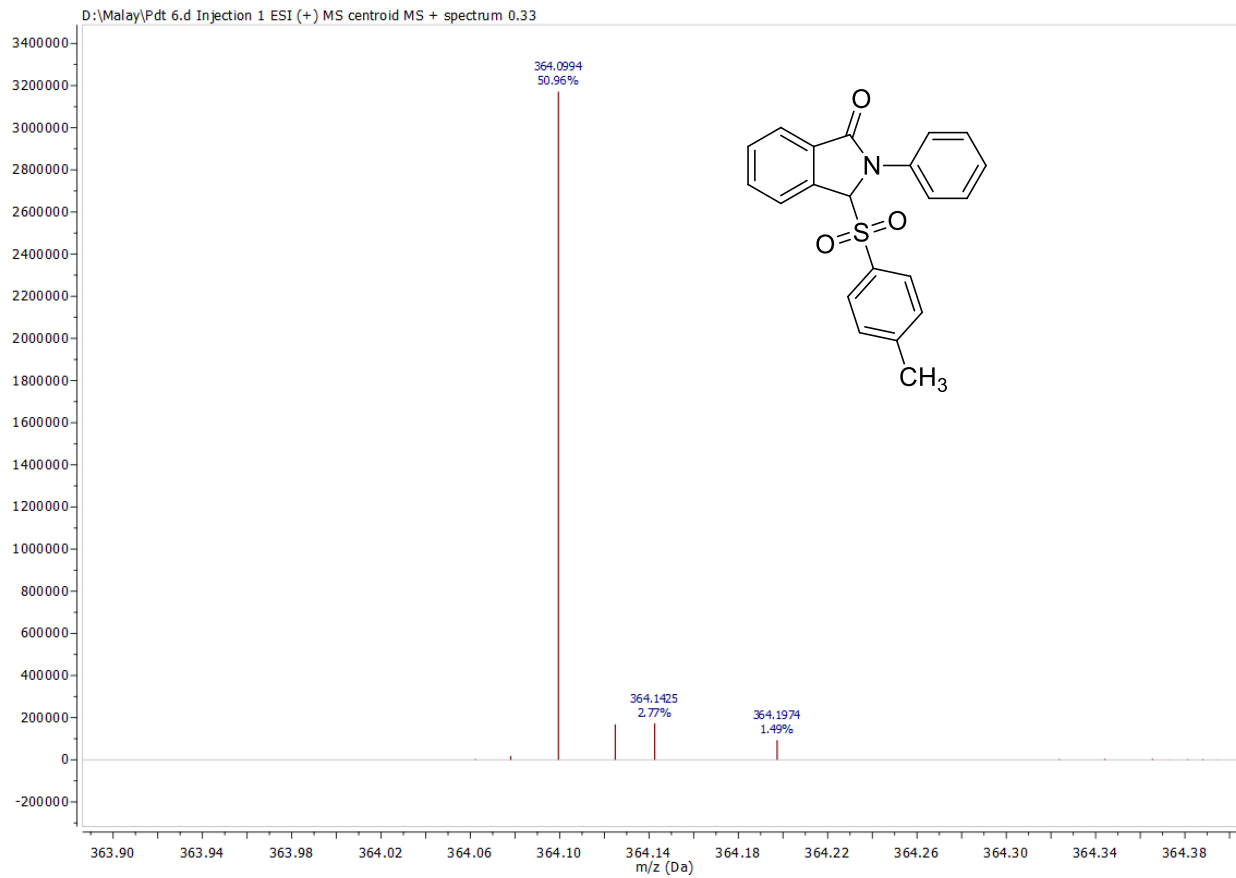
# HRMS spectrum of compound **3e**



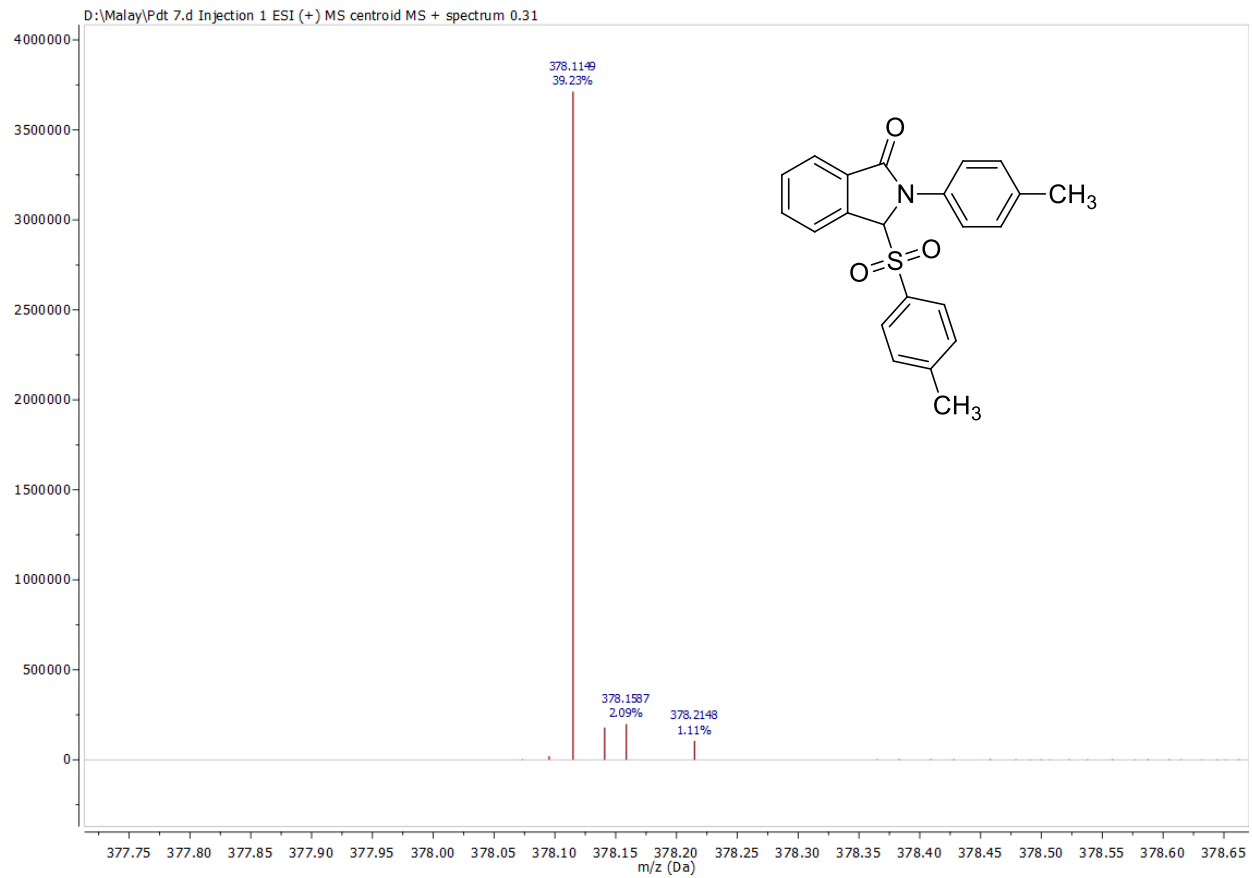
# HRMS spectrum of compound **3f**



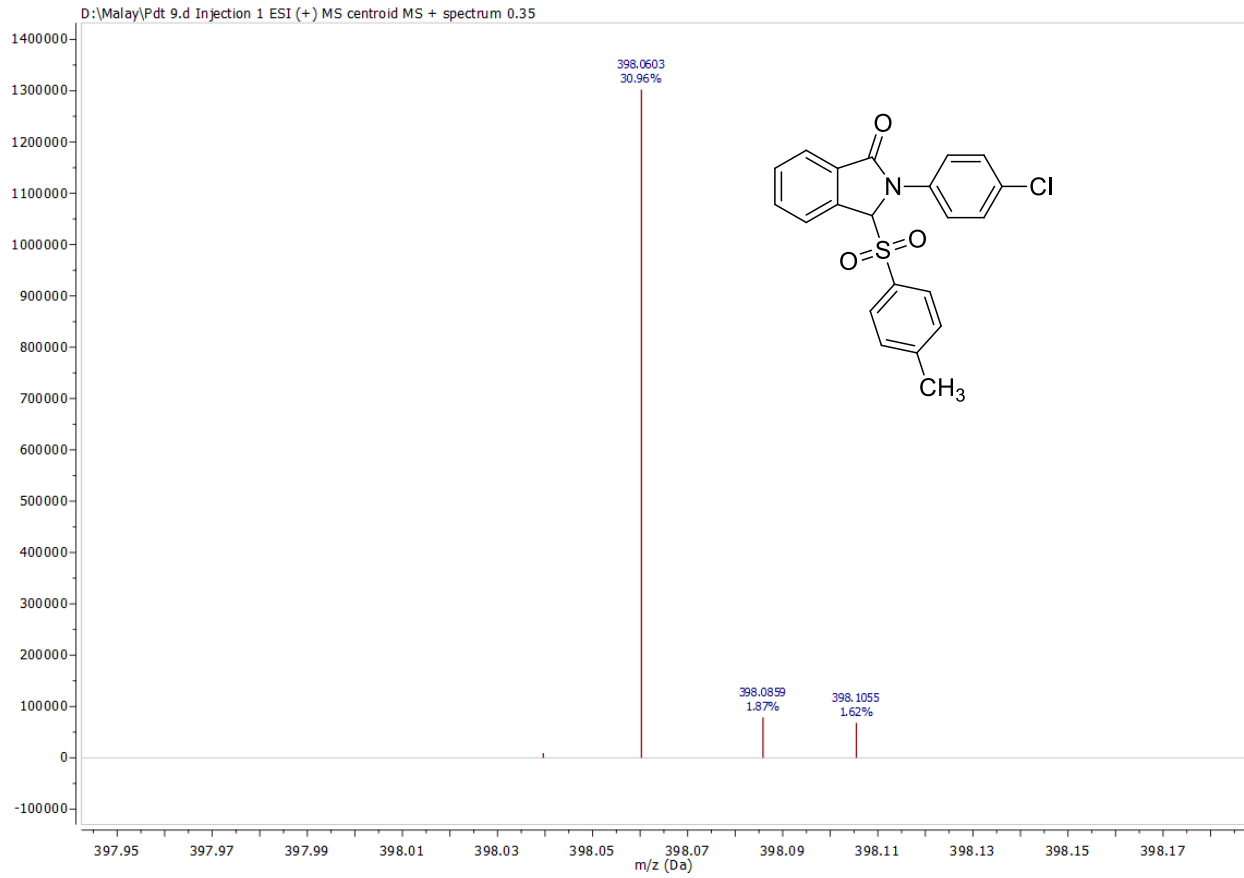
# HRMS spectrum of compound **3g**



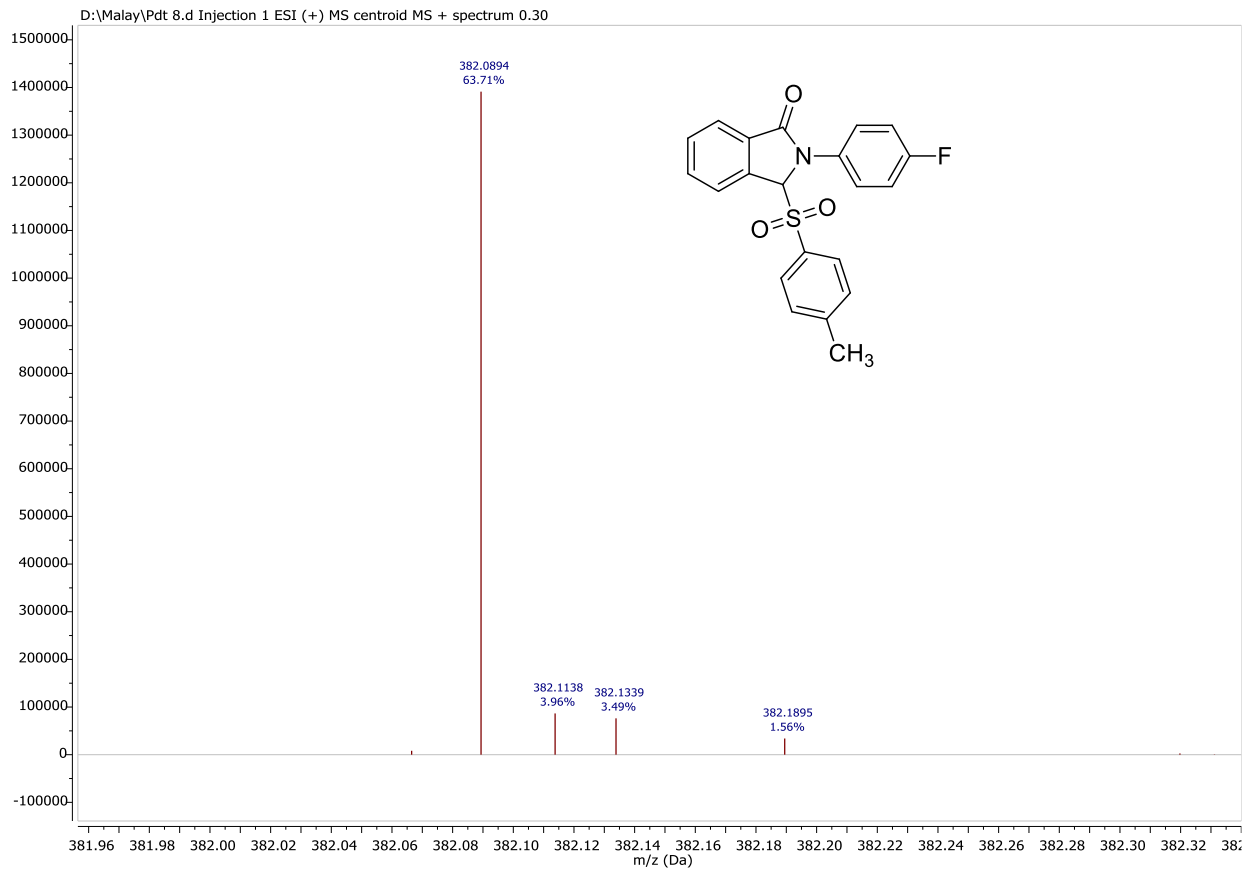
# HRMS spectrum of compound **3h**



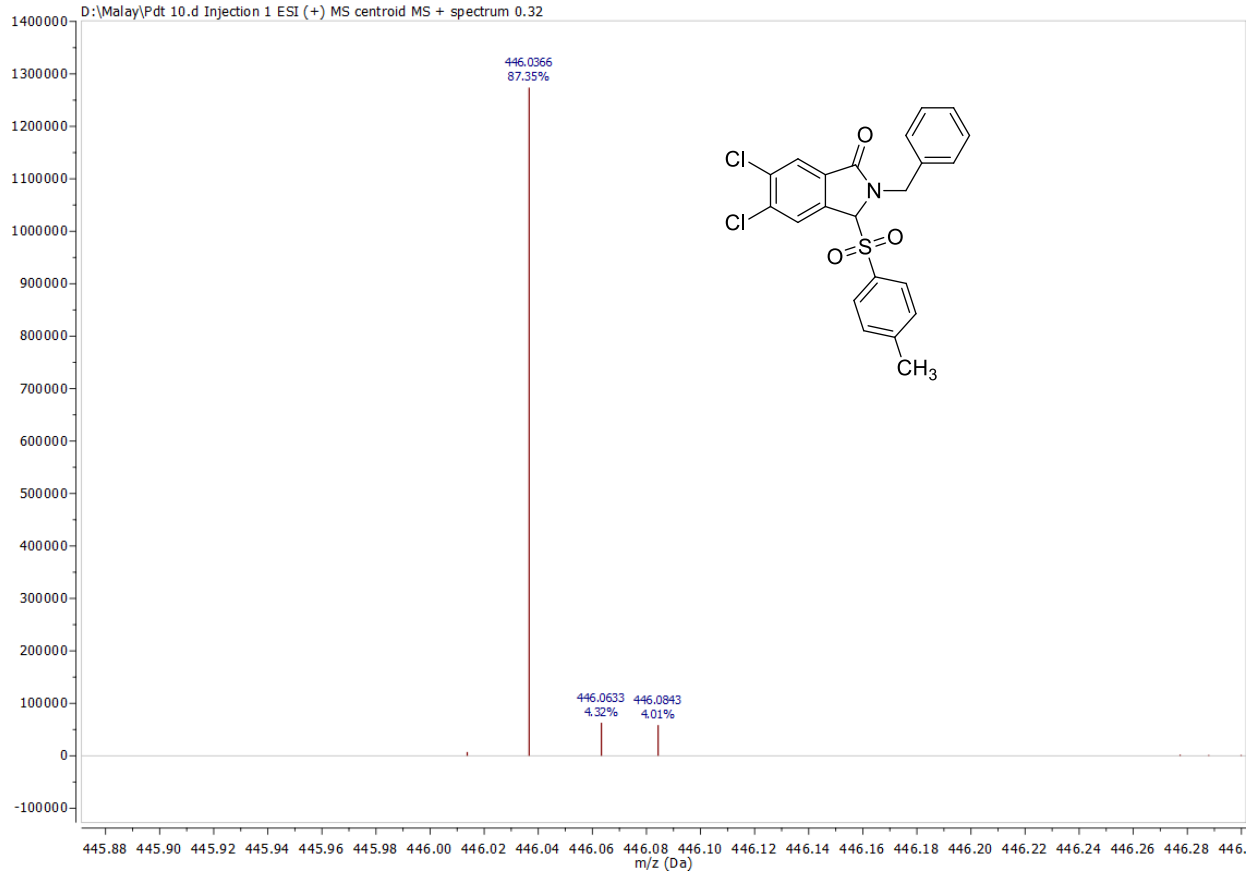
# HRMS spectrum of compound **3i**



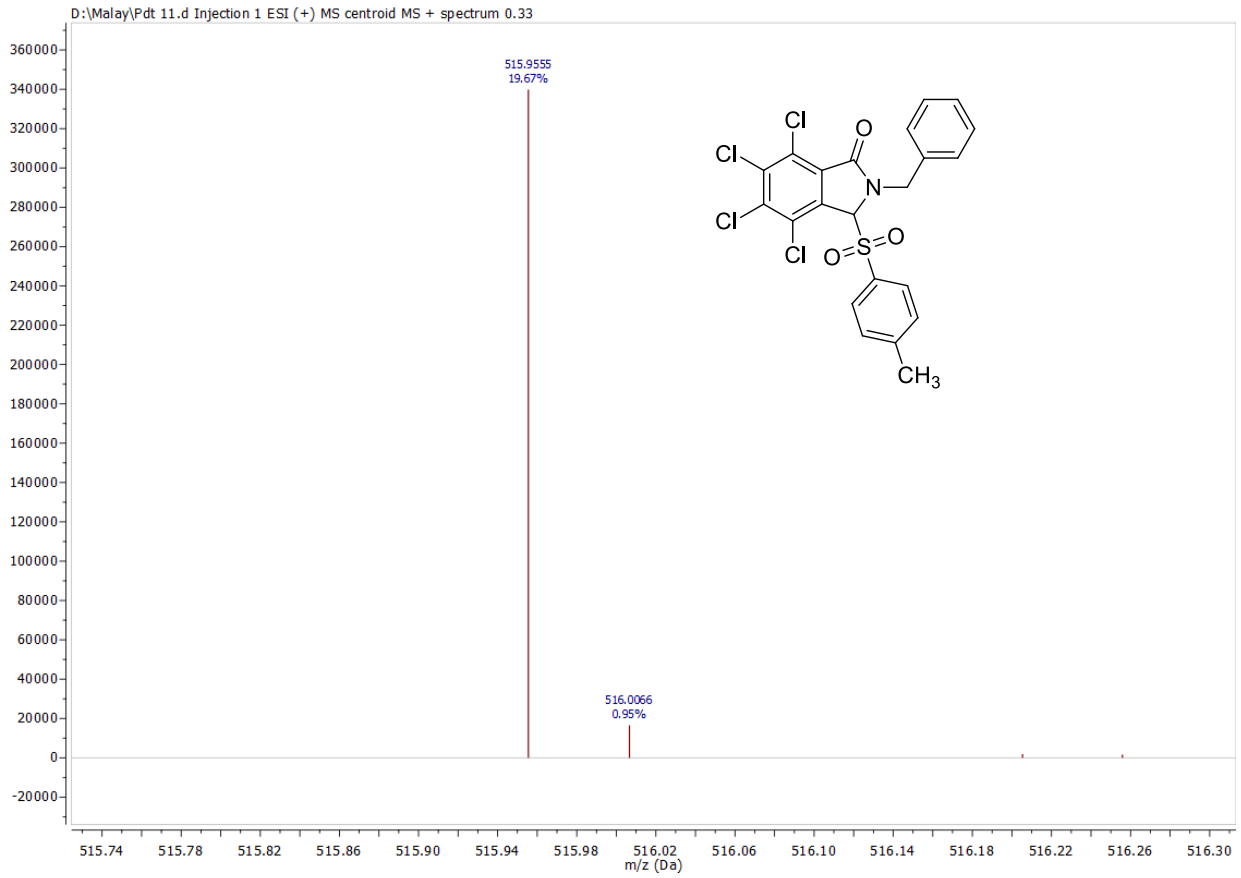
# HRMS spectrum of compound 3j



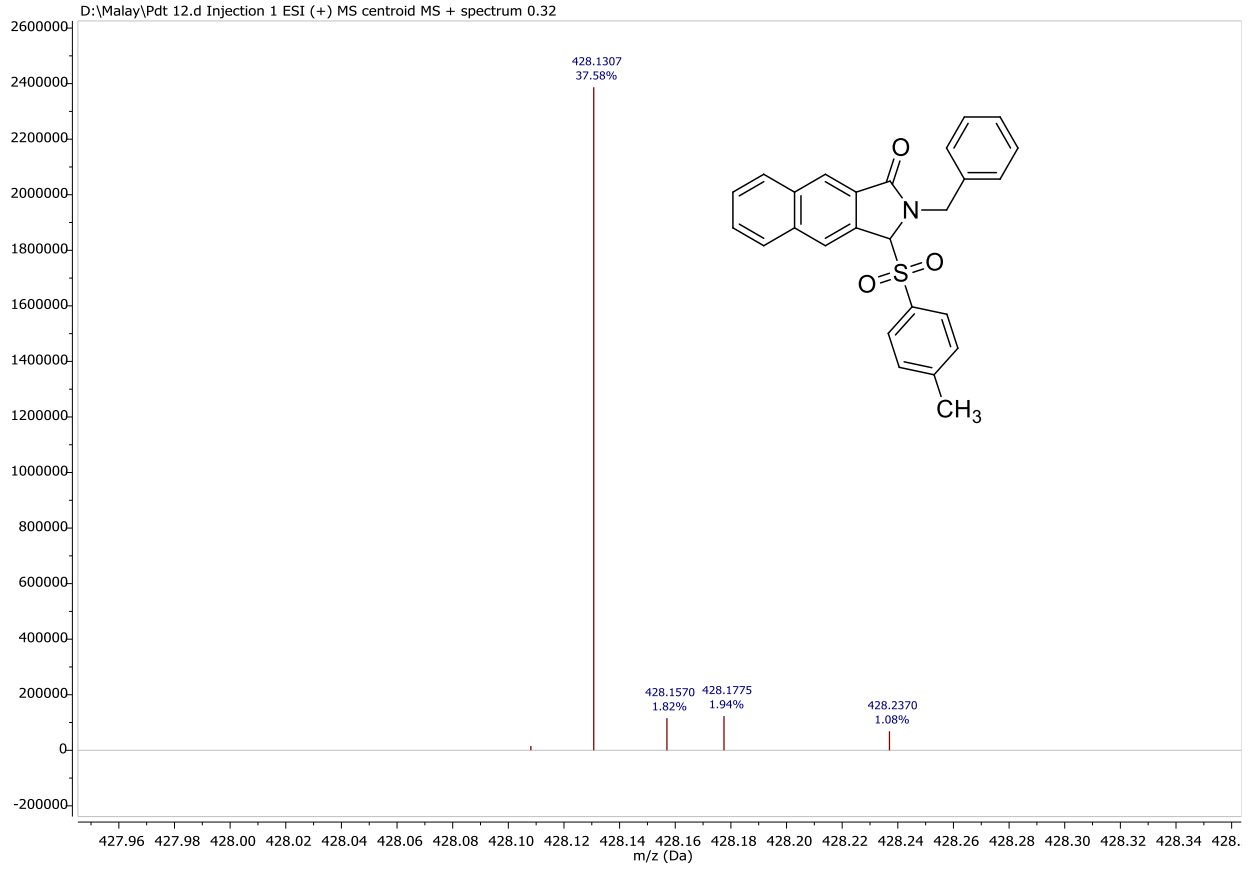
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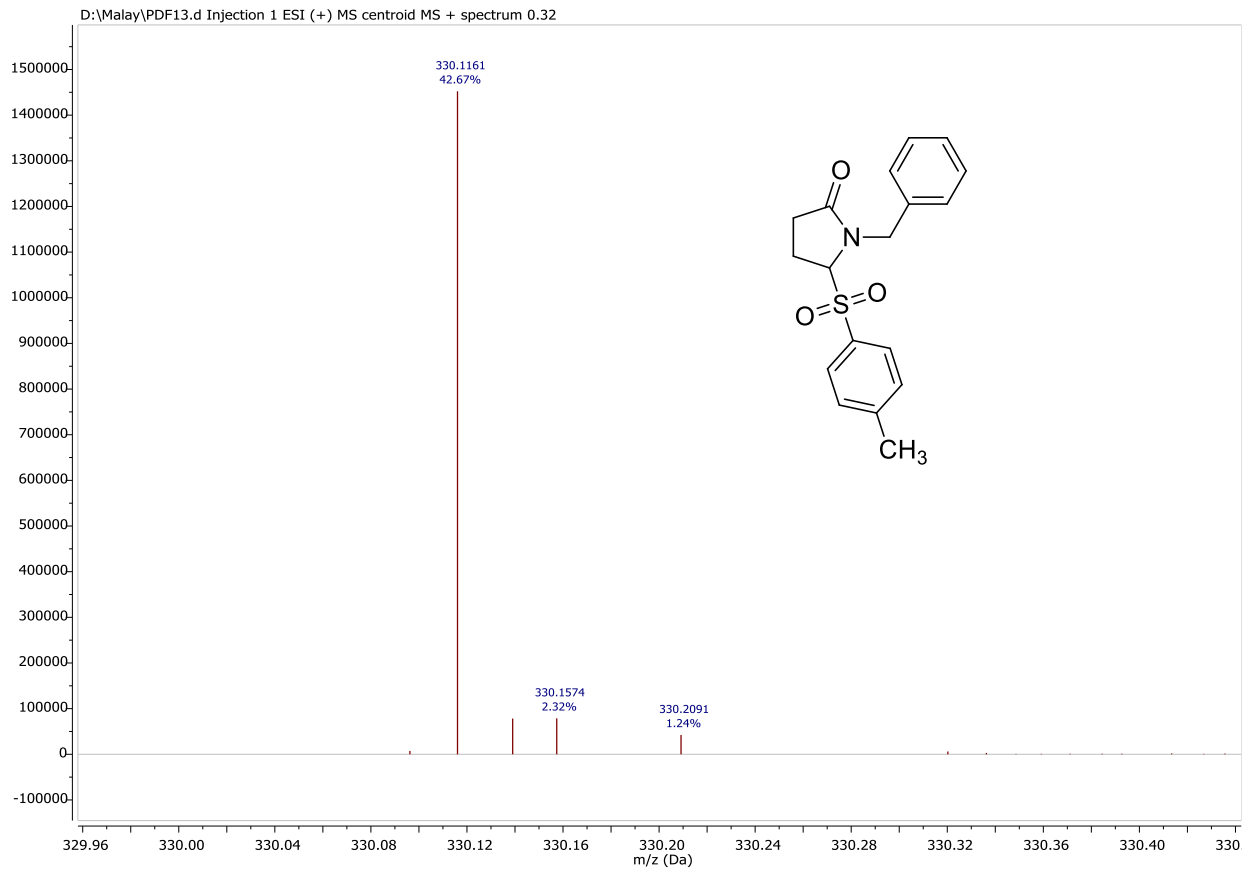
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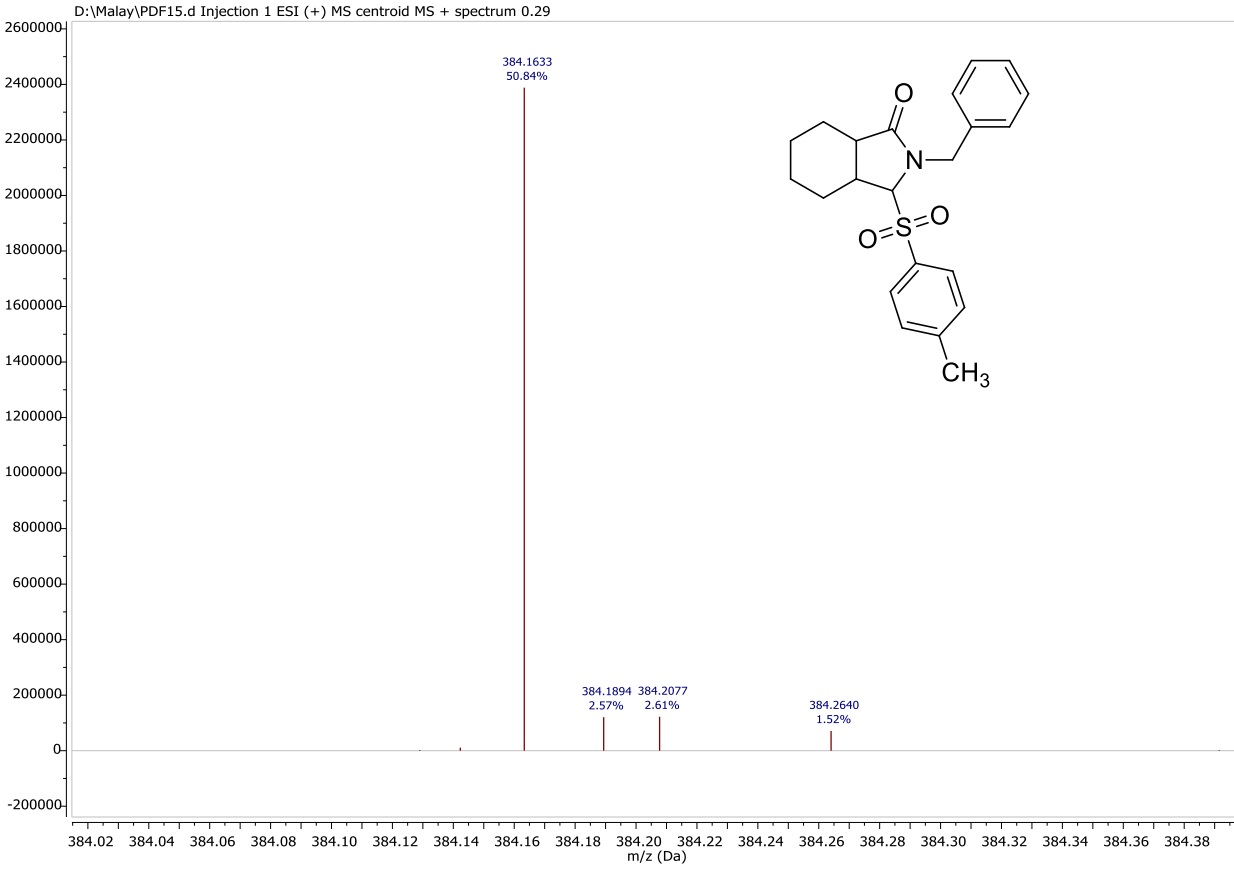
# HRMS spectrum of compound **3m**



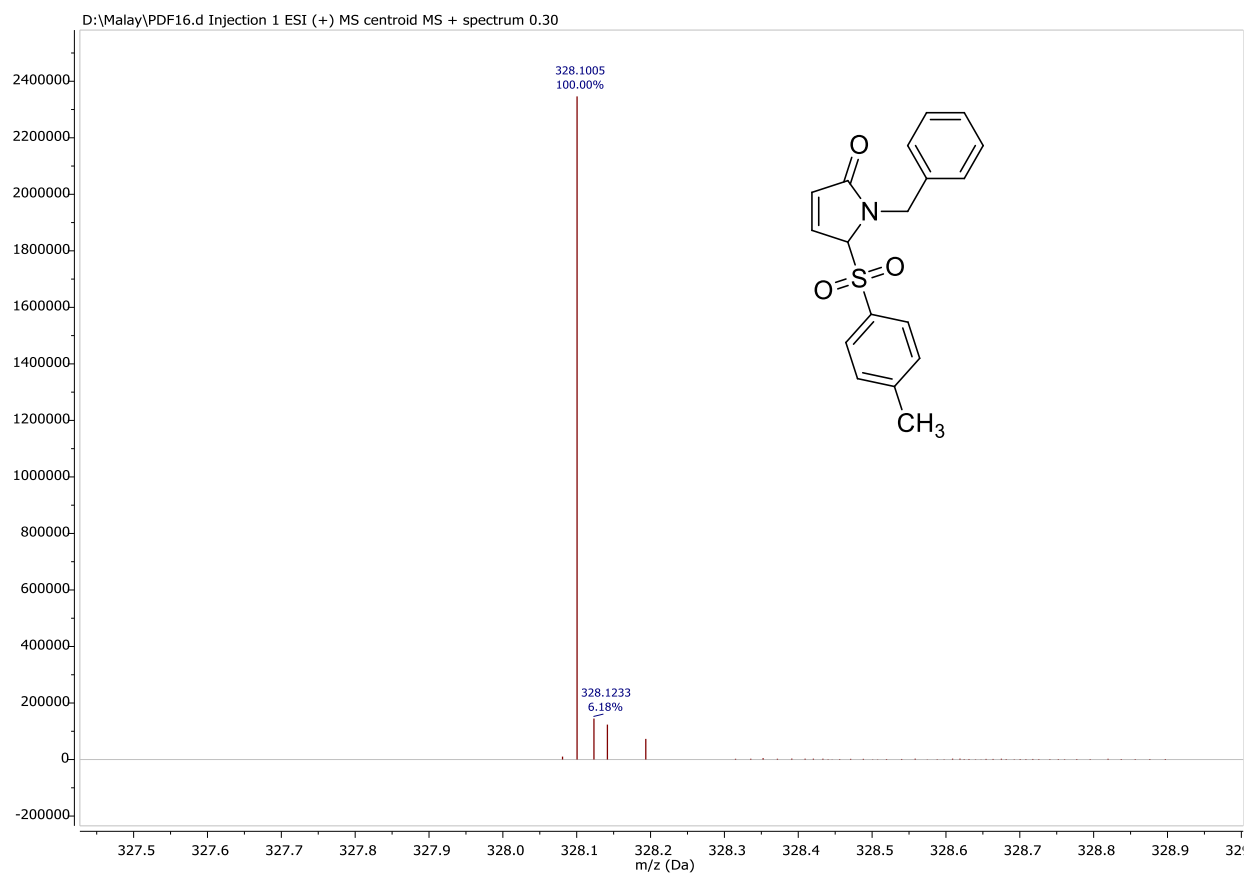
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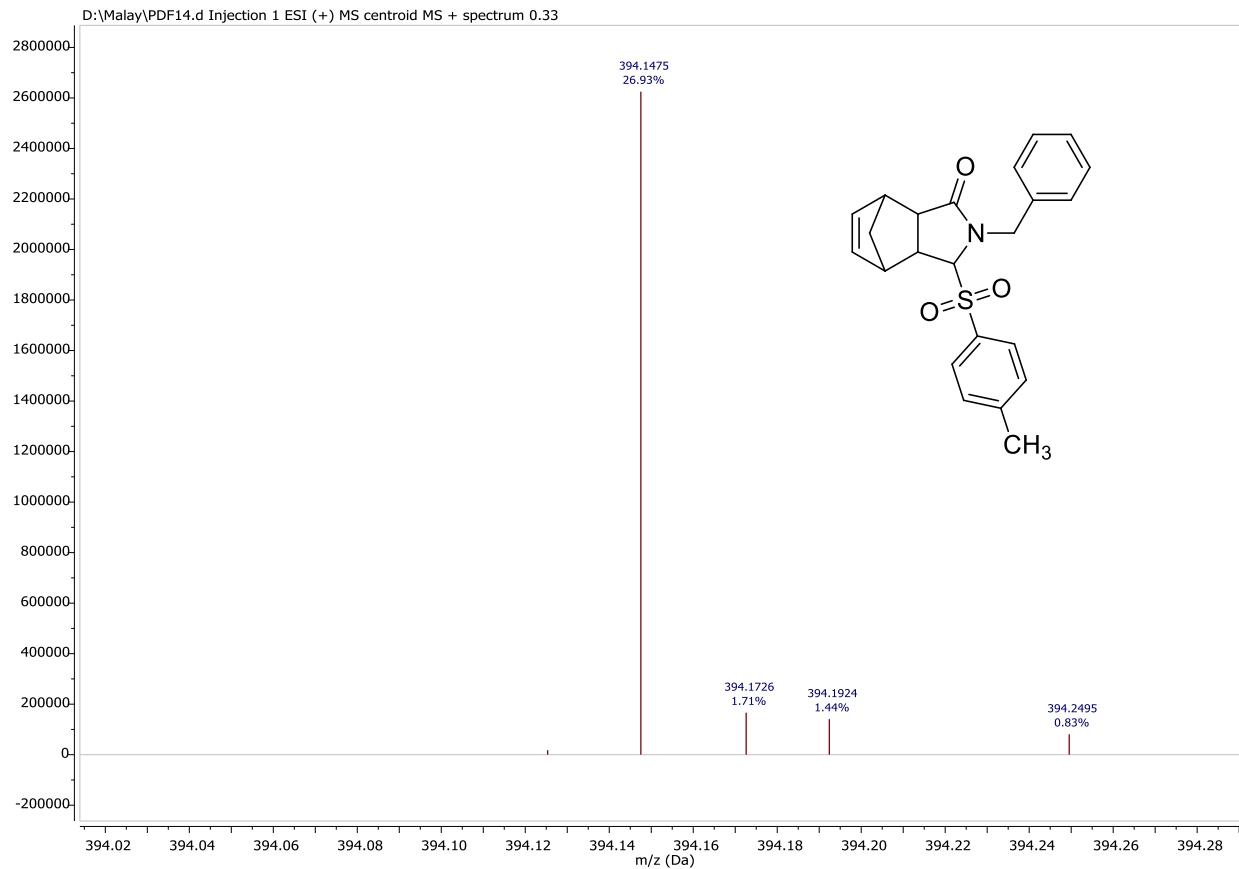
# HRMS spectrum of compound **3o**



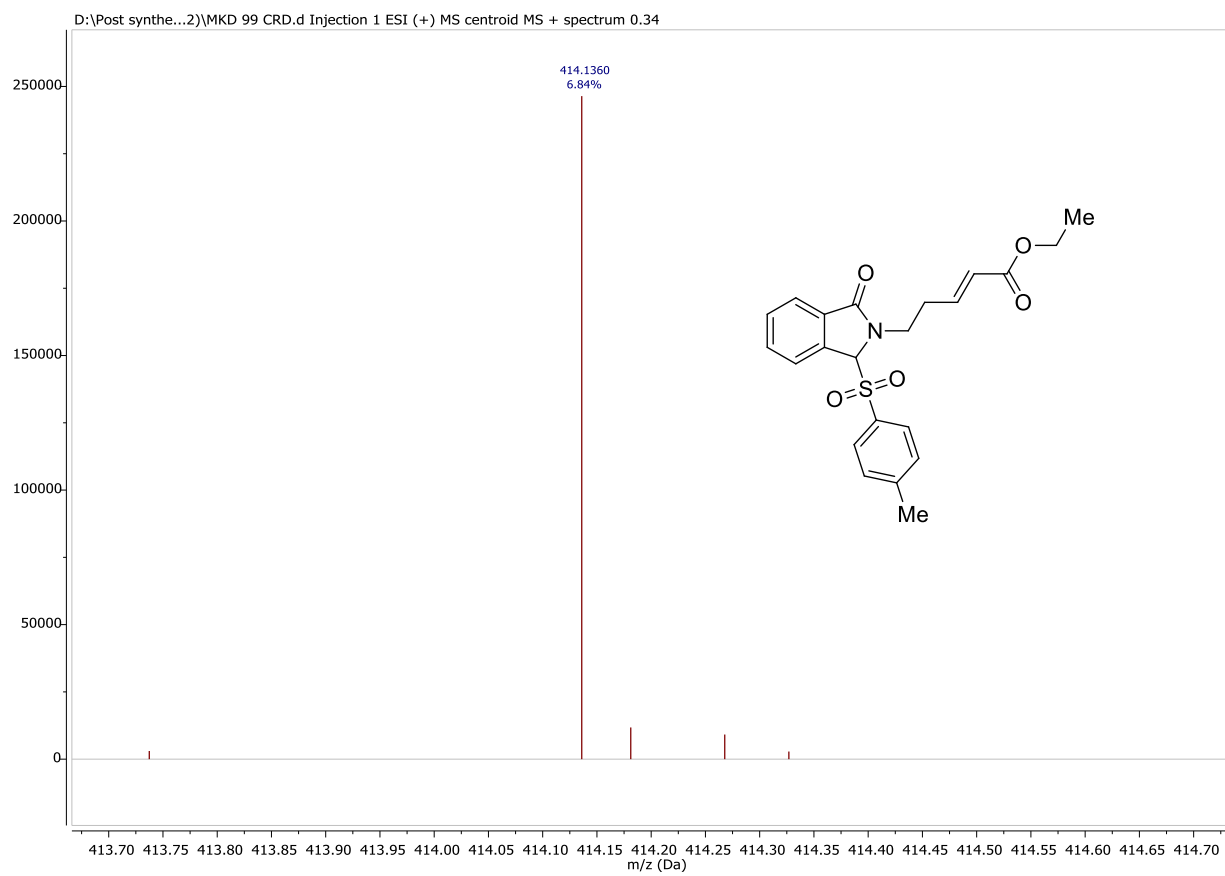
# HRMS spectrum of compound **3p**



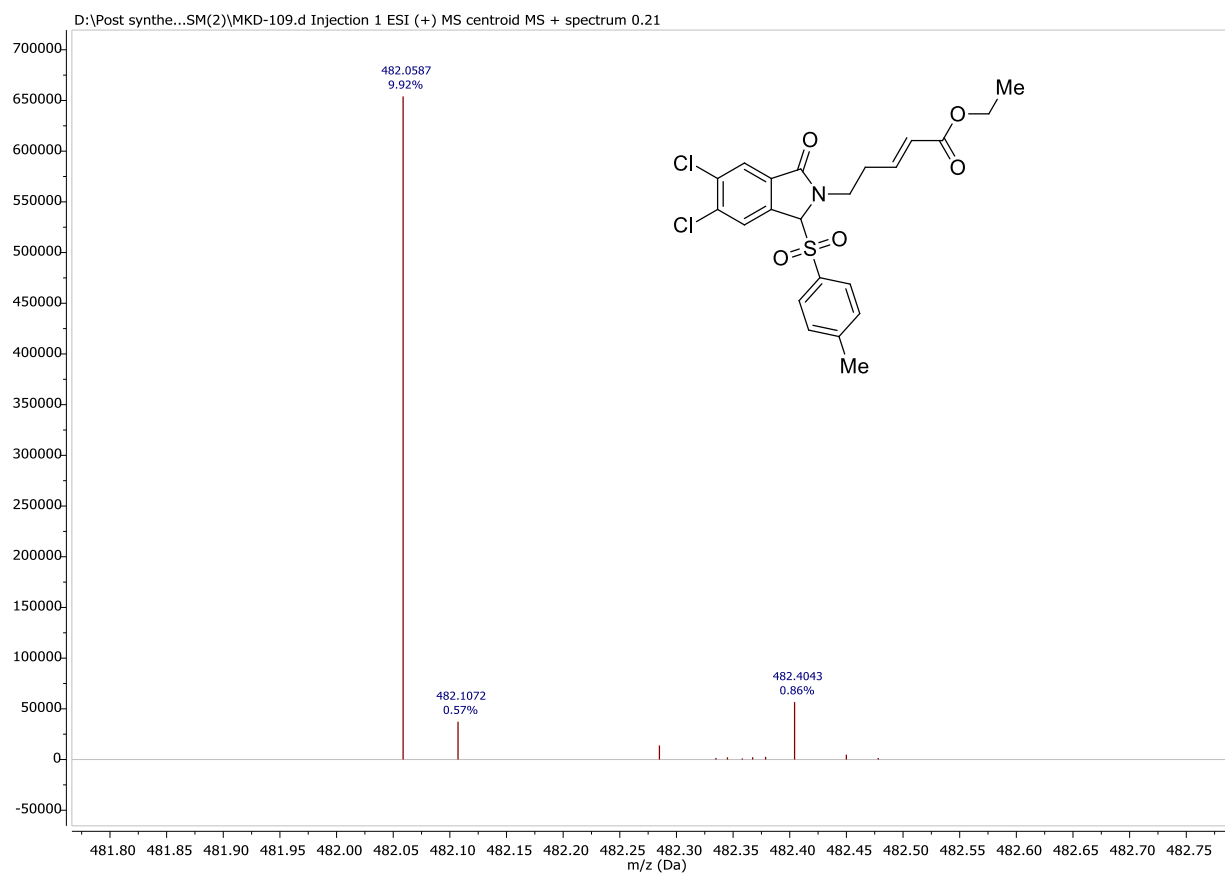
# HRMS spectrum of compound **3q**



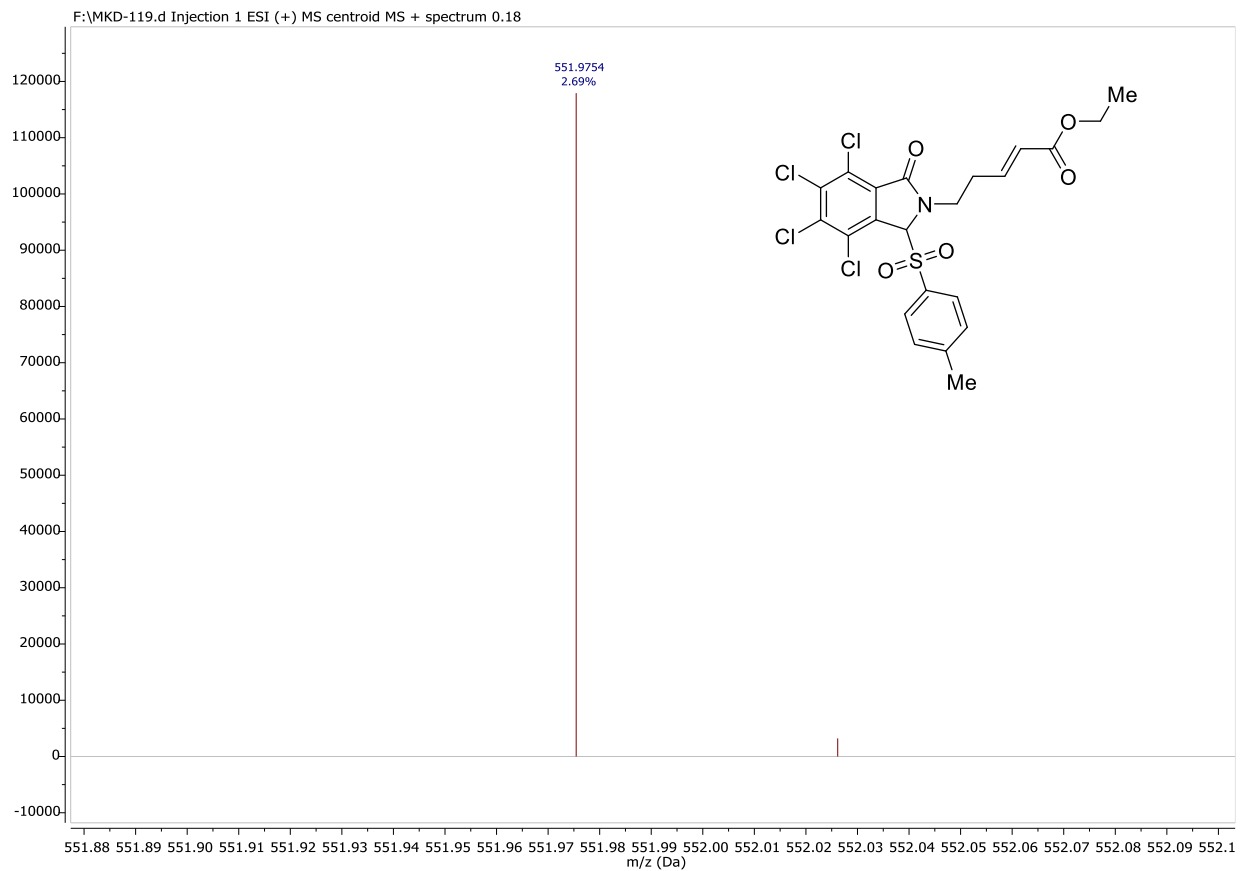
# HRMS spectrum of compound **3r**



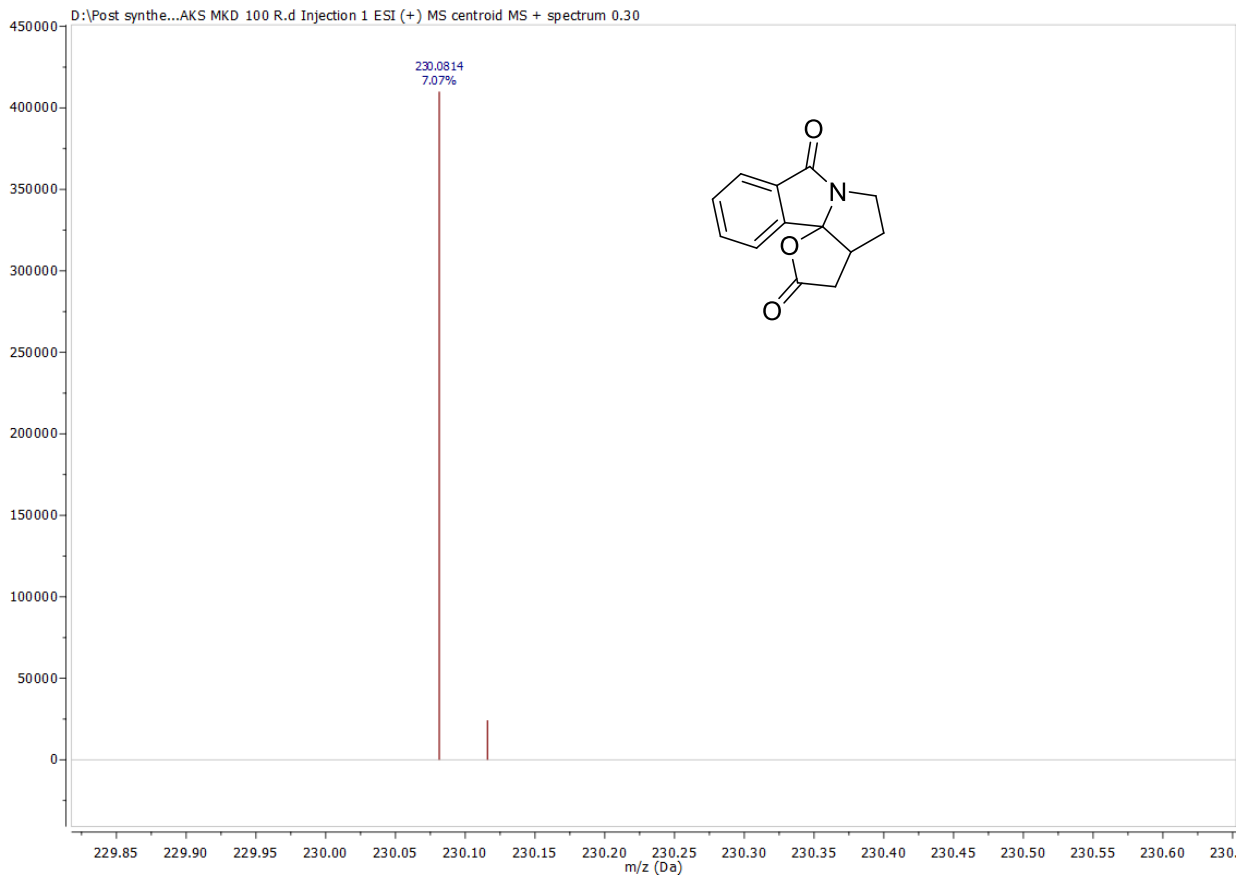
# HRMS spectrum of compound **3s**



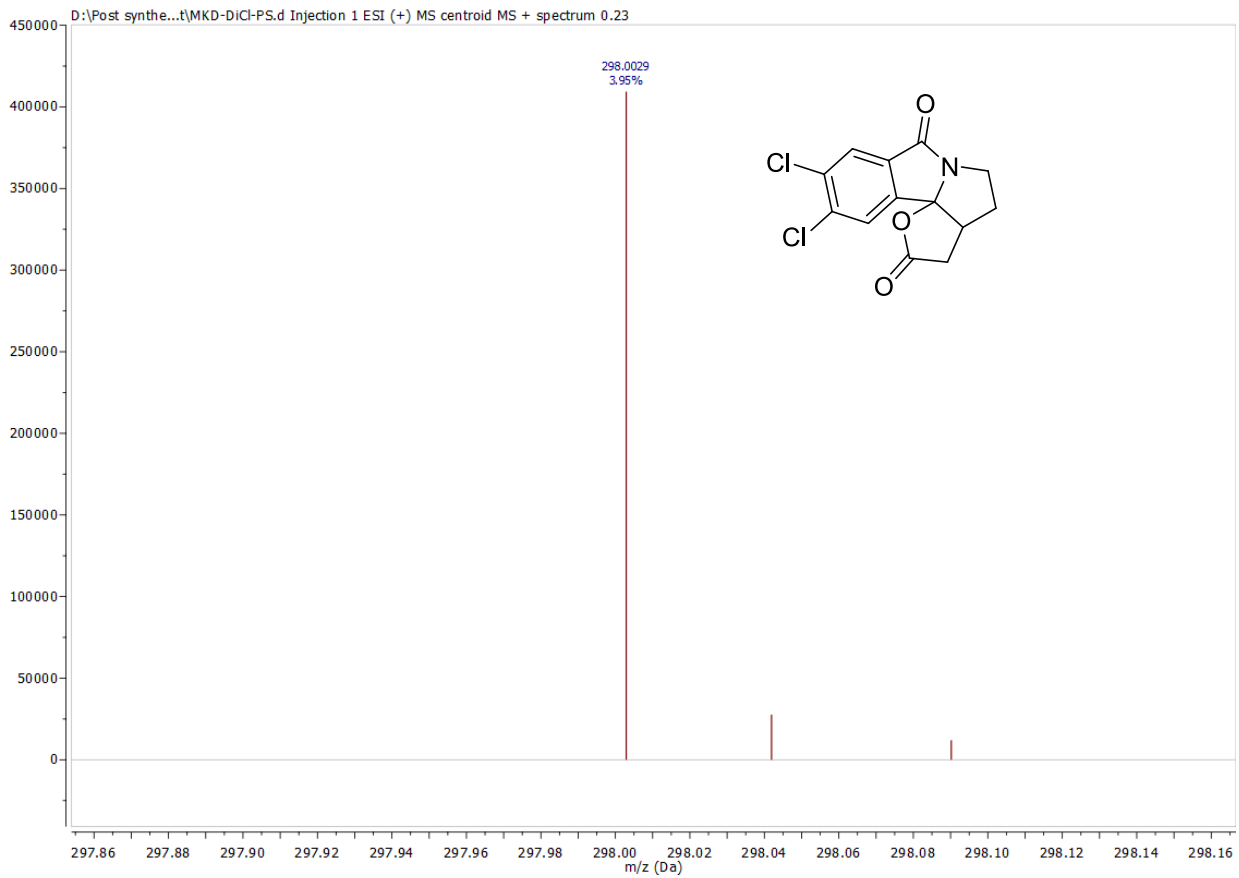
# HRMS spectrum of compound **3t**



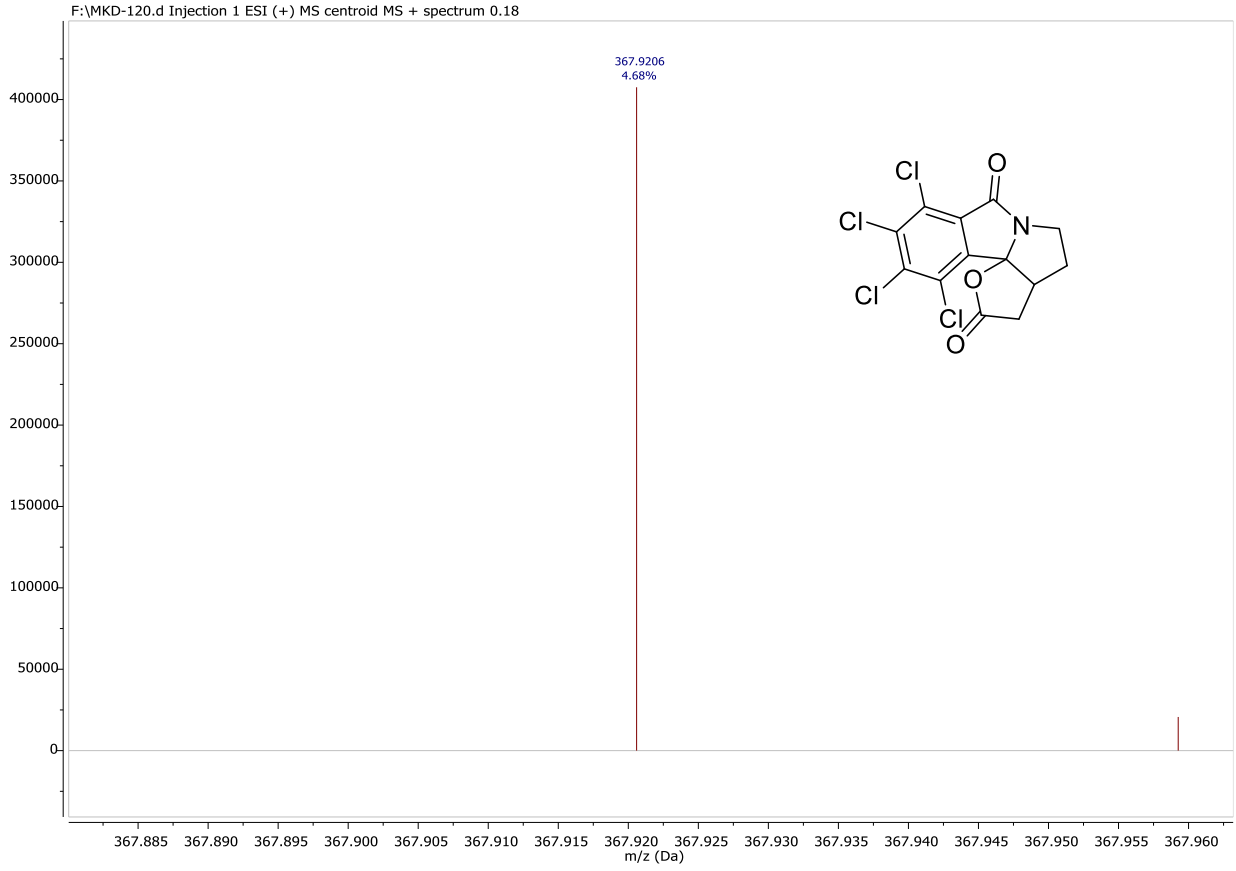
# HRMS spectrum of compound **4a**



# HRMS spectrum of compound **4b**



# HRMS spectrum of compound **4c**



**Supporting Information for Publication**

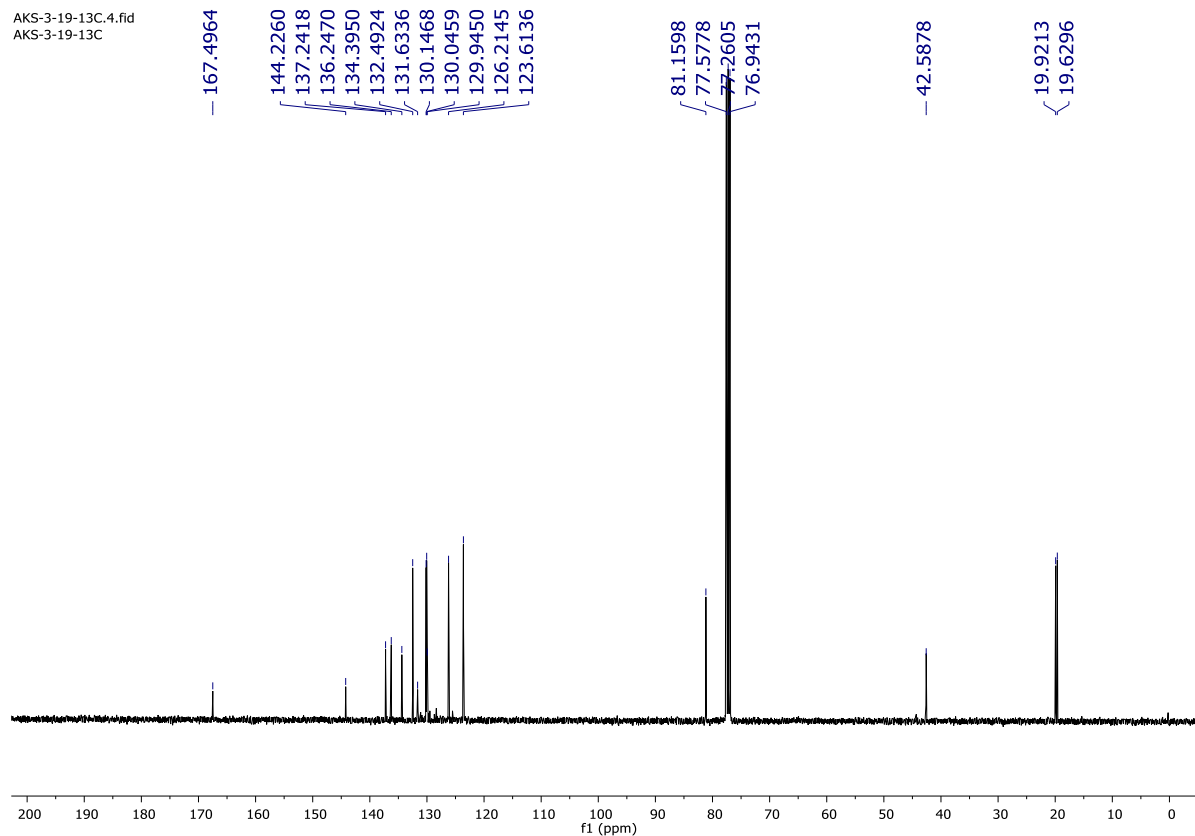
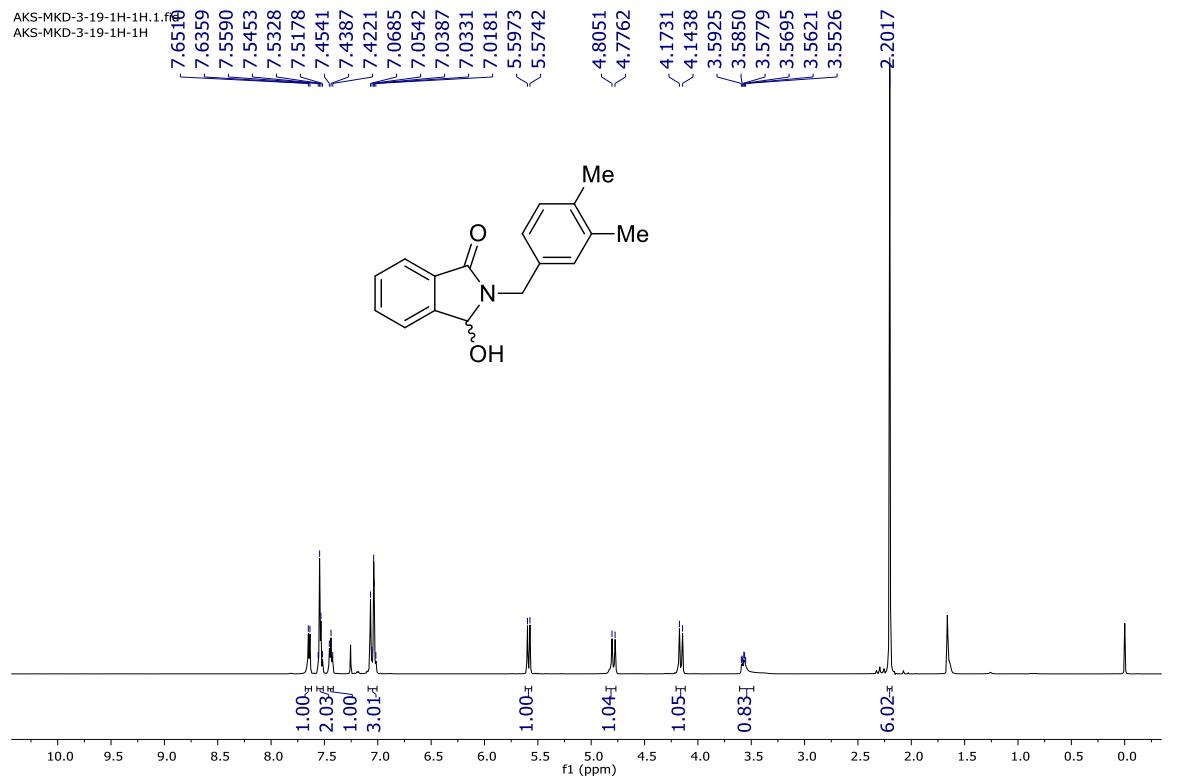
**Lewis Acid-Promoted Synthesis of 3-Tosylisoindolinone from 3-Hydroxyisoindolinone: Access to Complex Fuopyrroloisoindole-dione**

Malay Das, Virendra Kumar, Pallav Jyoti Arandhara, Subhamoy Biswas and Anil K. Saikia\*

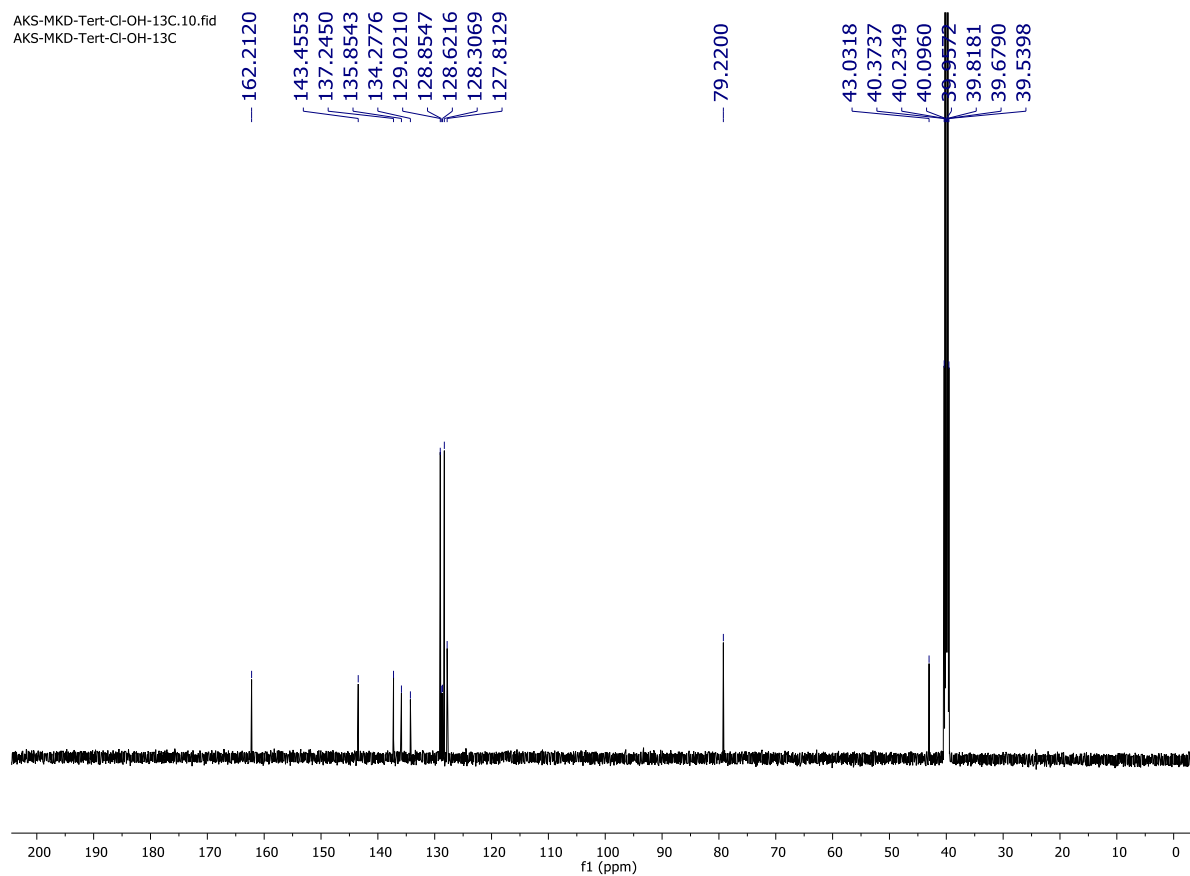
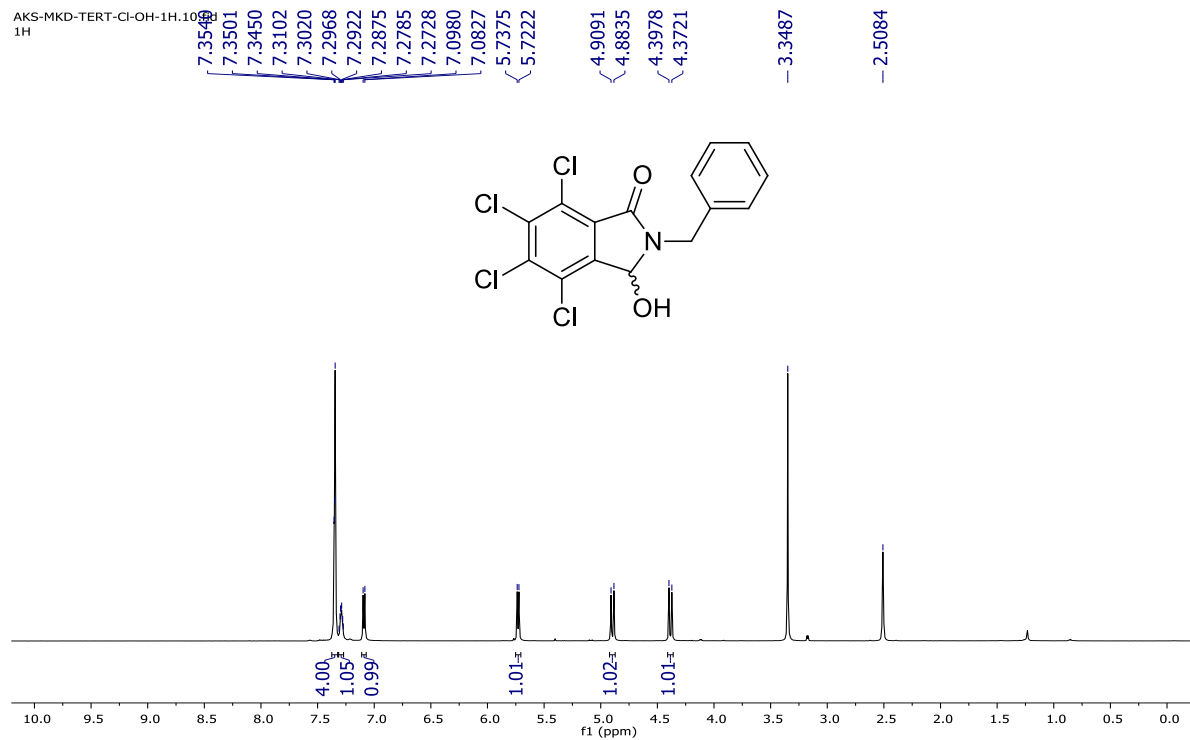
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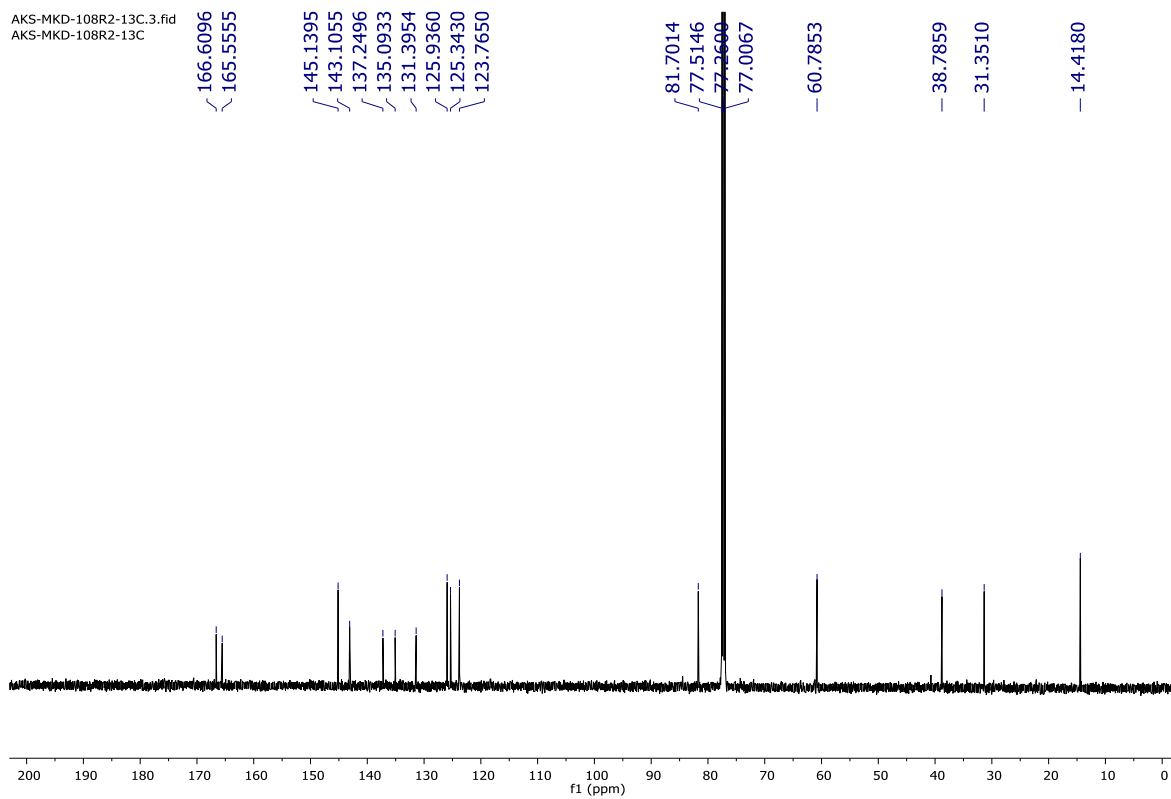
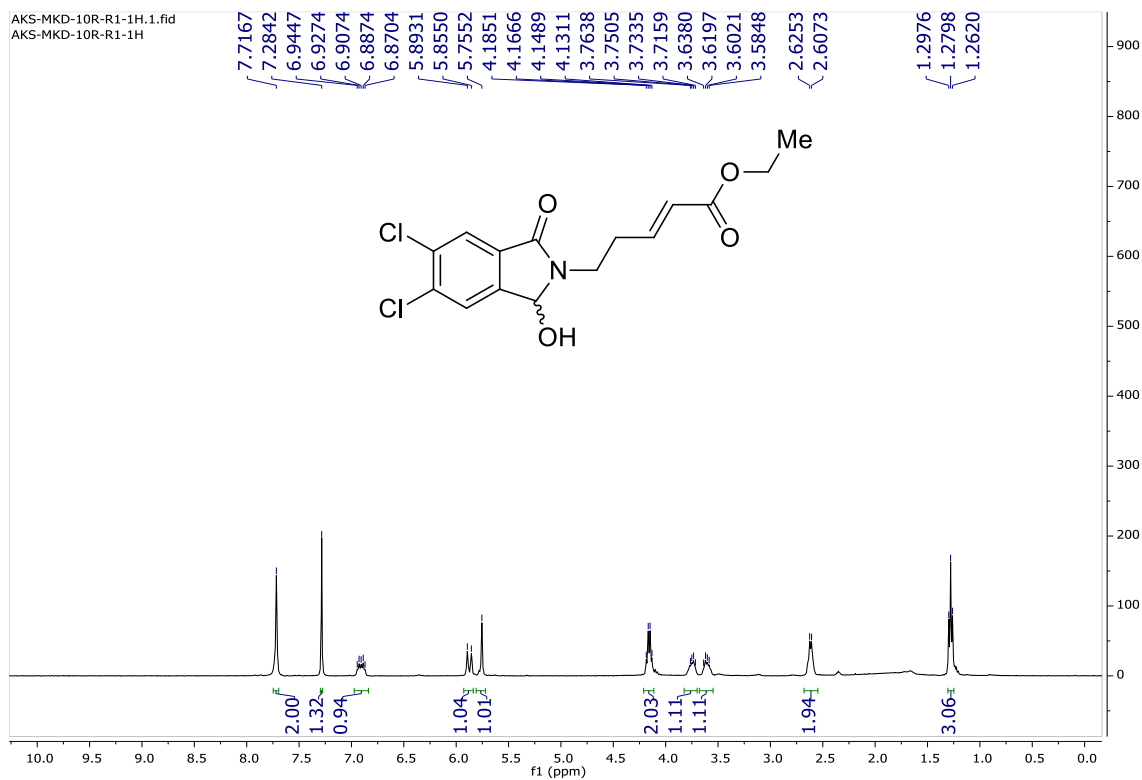
# $^1\text{H}$ and $^{13}\text{C}\{^1\text{H}\}$ (500 and 100 MHz, $\text{CDCl}_3$ ) NMR spectra of compound 1c.



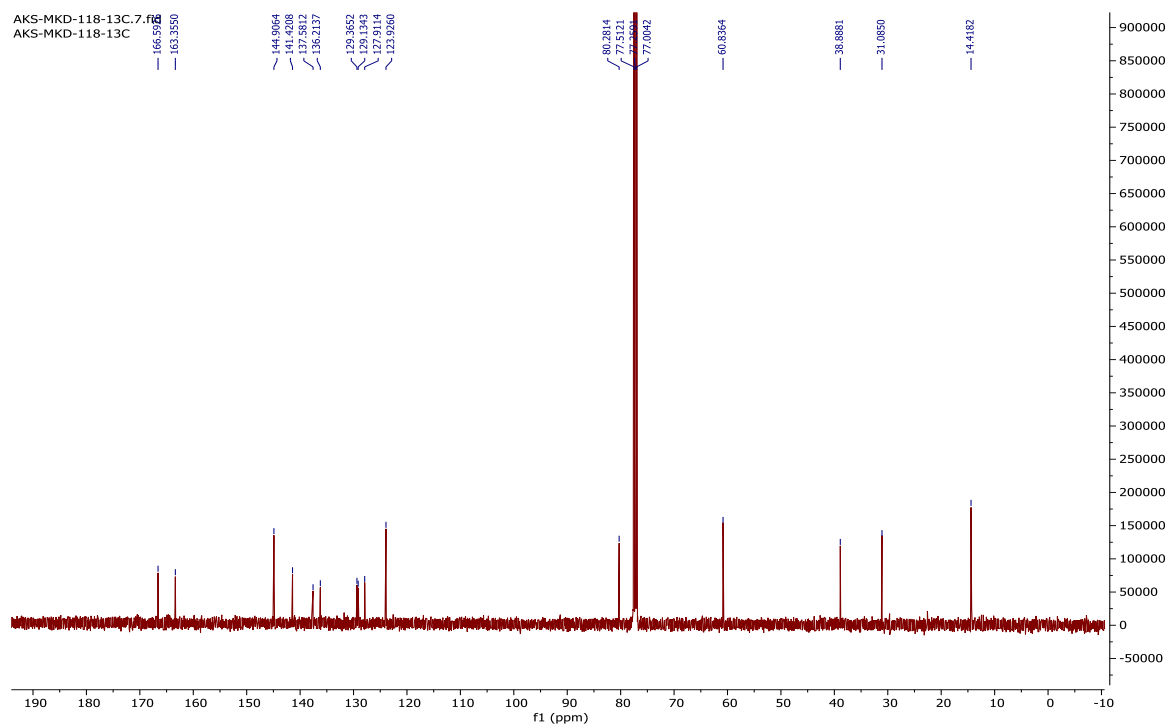
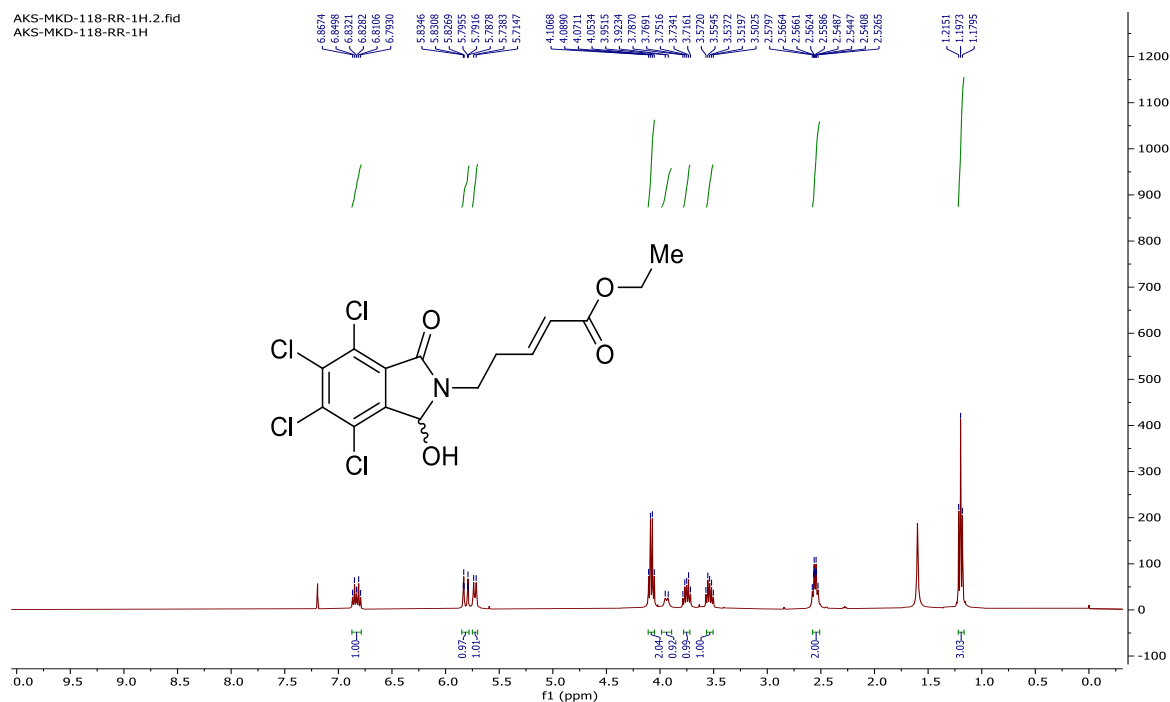
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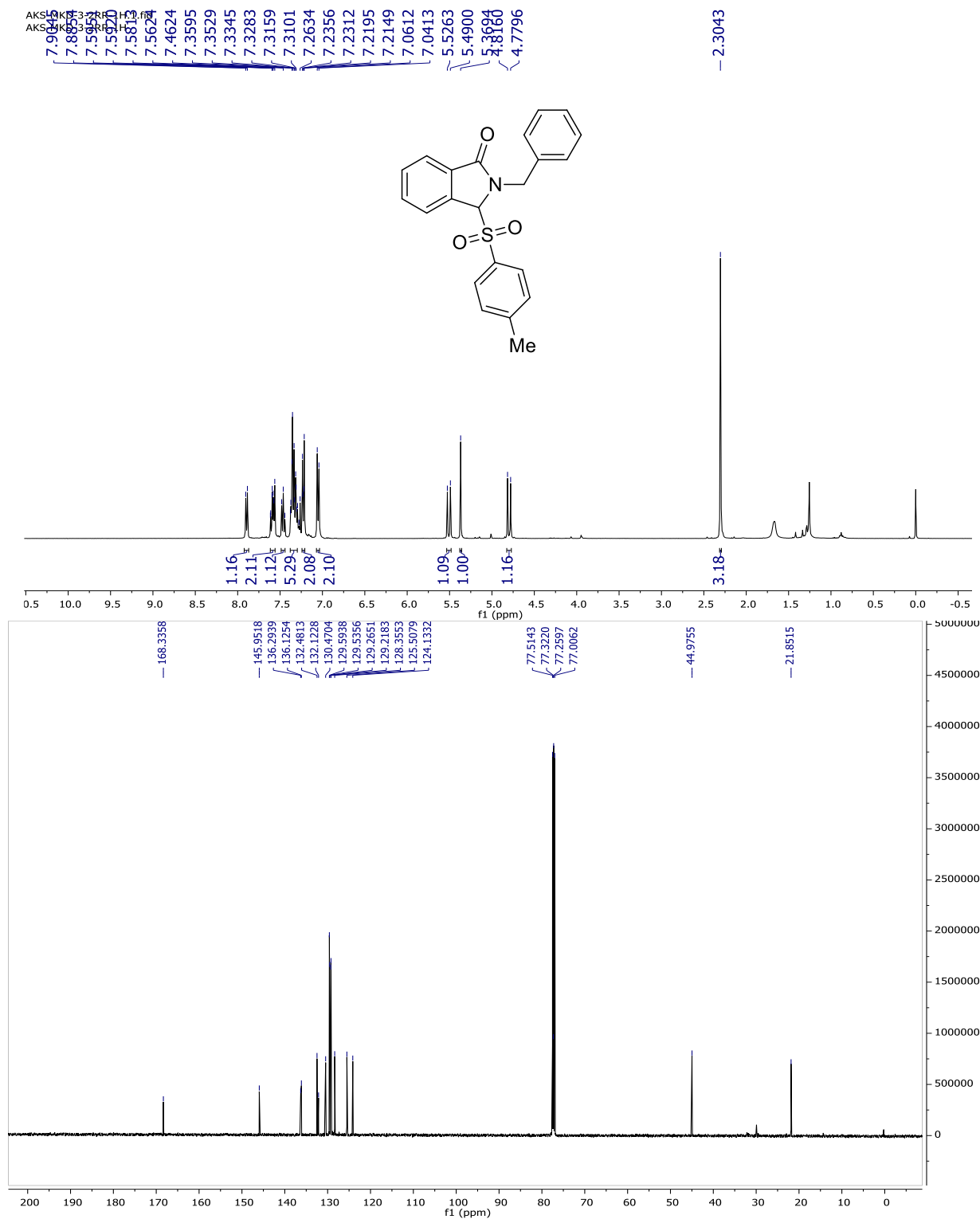
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 125 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 1s.**



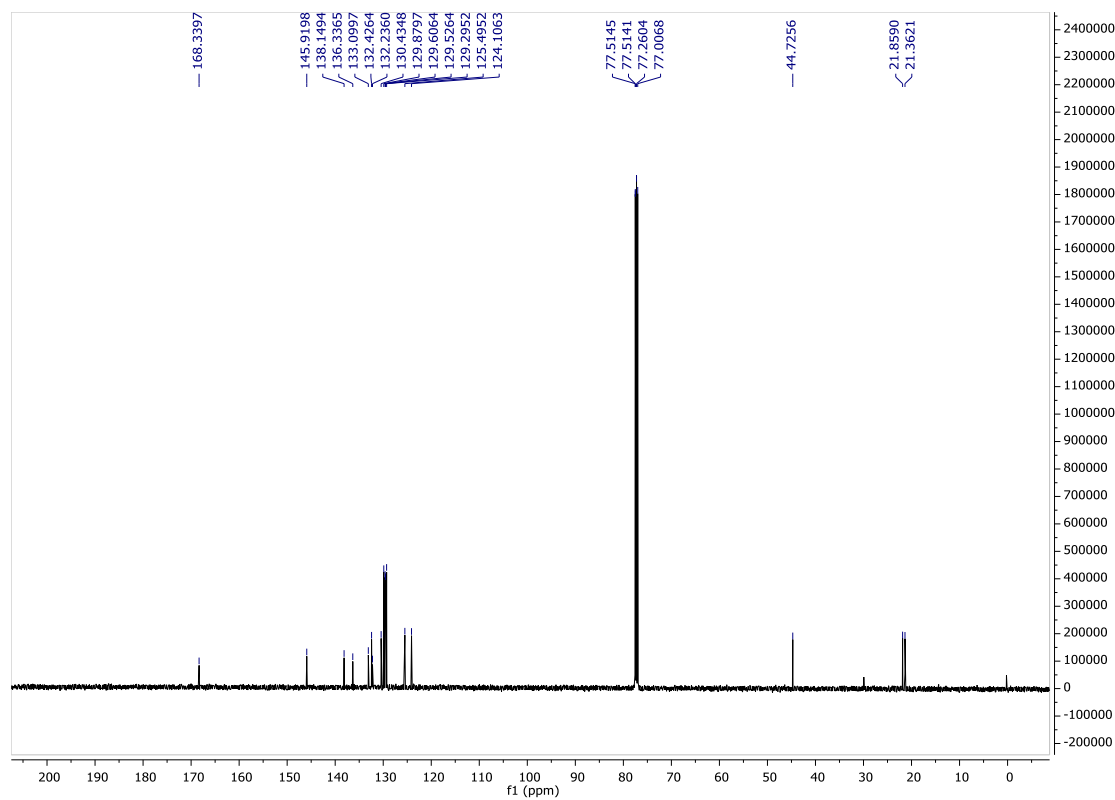
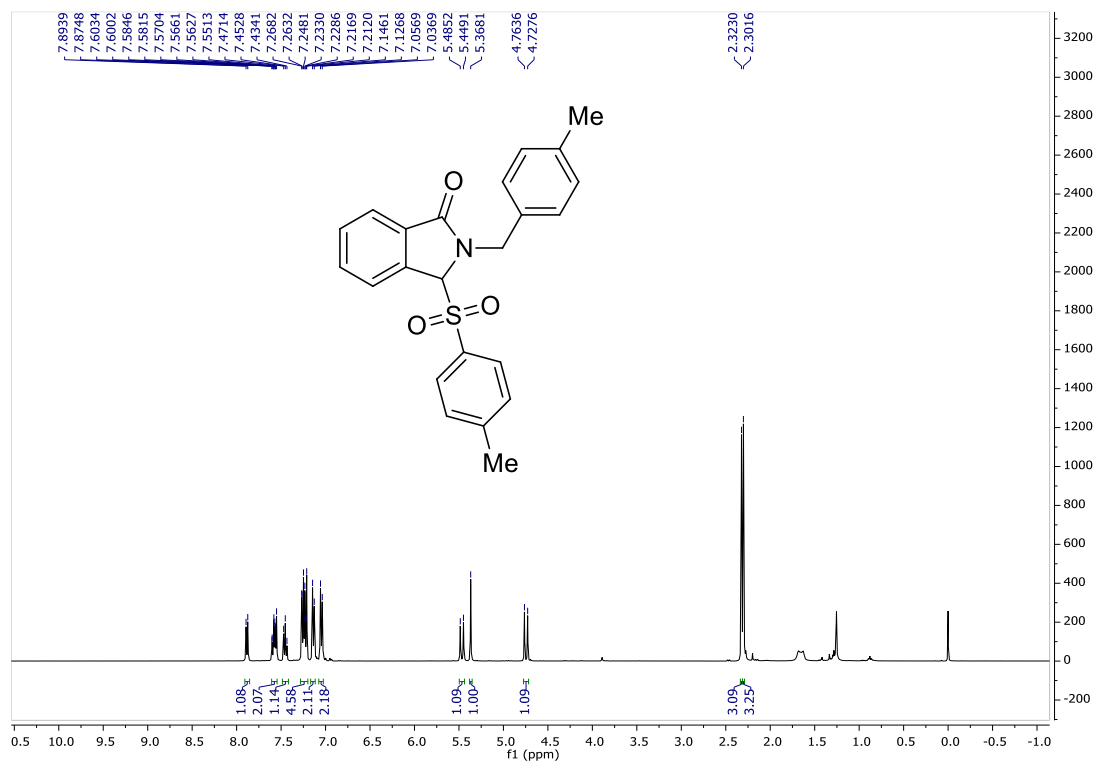
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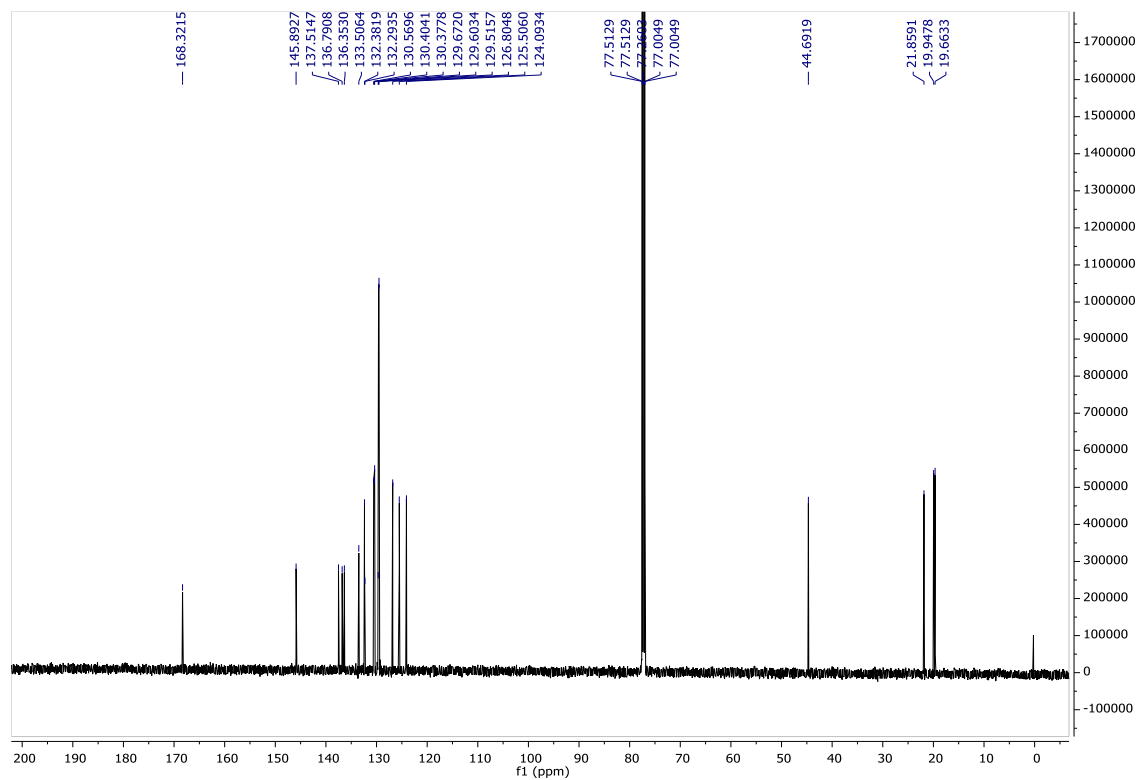
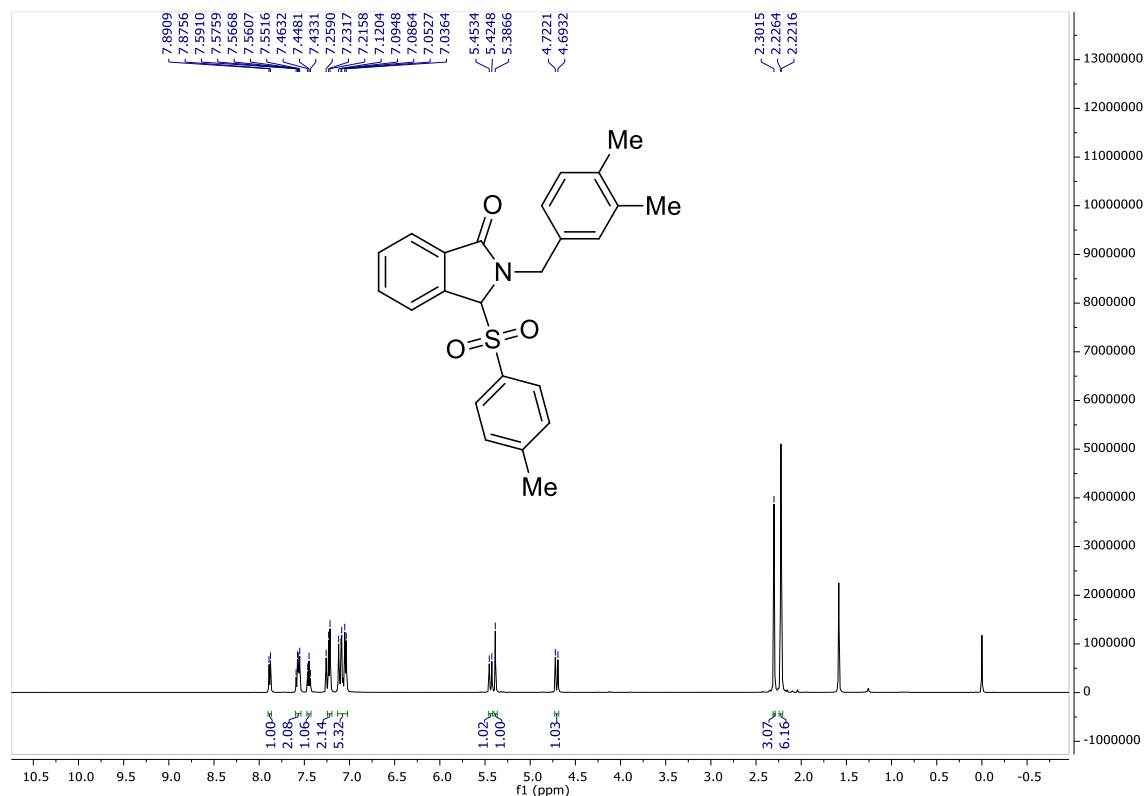
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 100 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3a.**



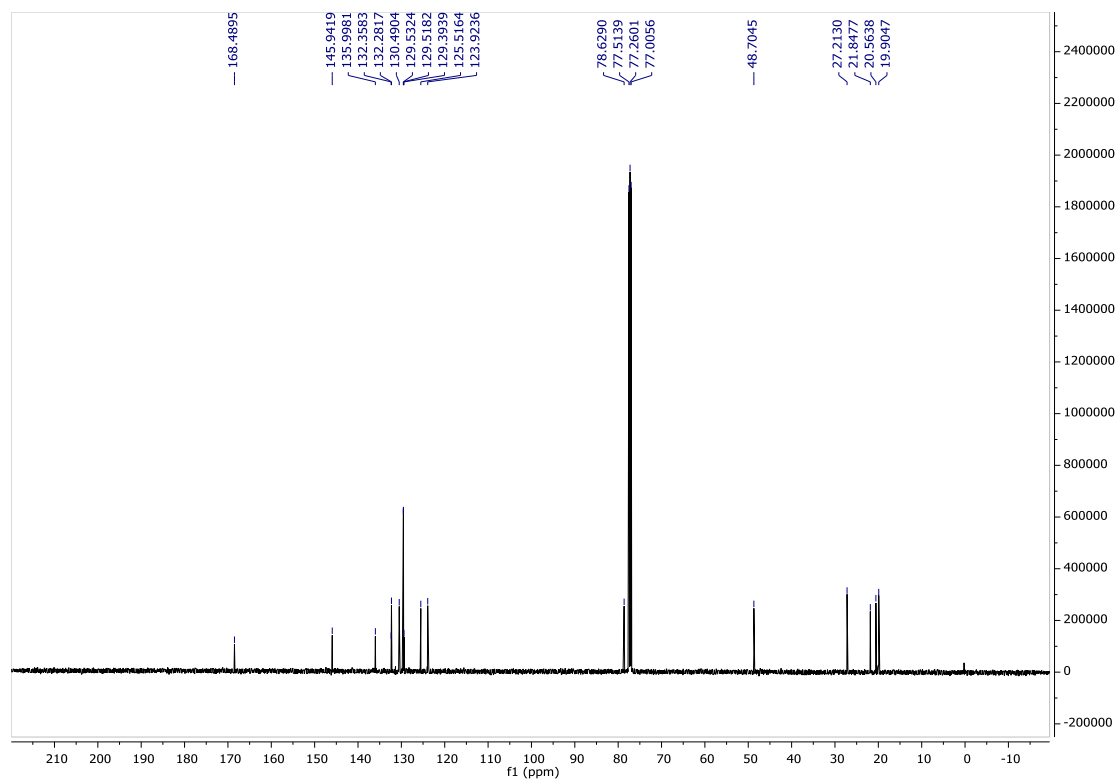
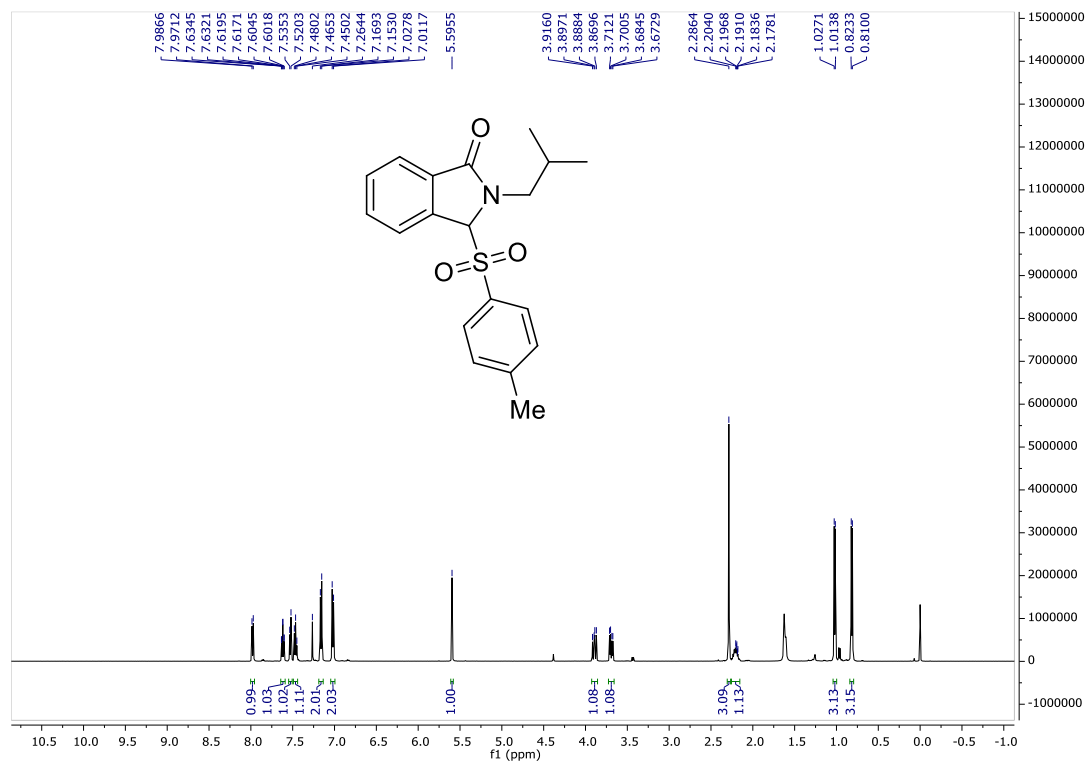
$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 100 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3b.



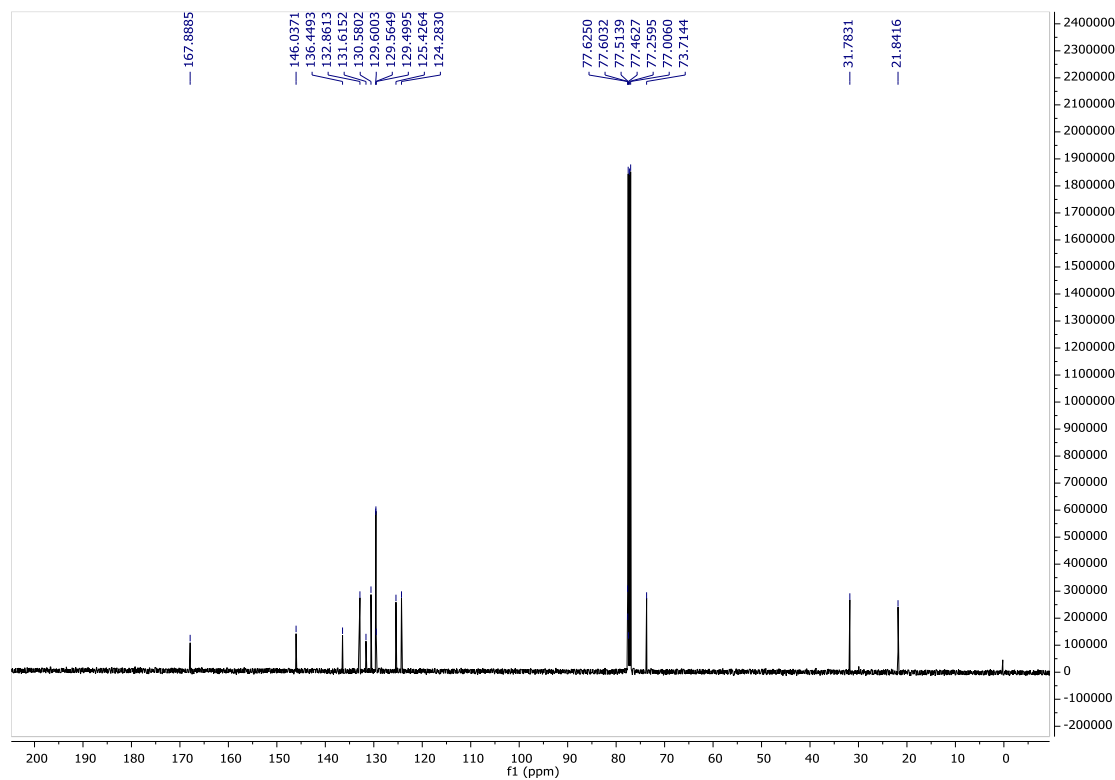
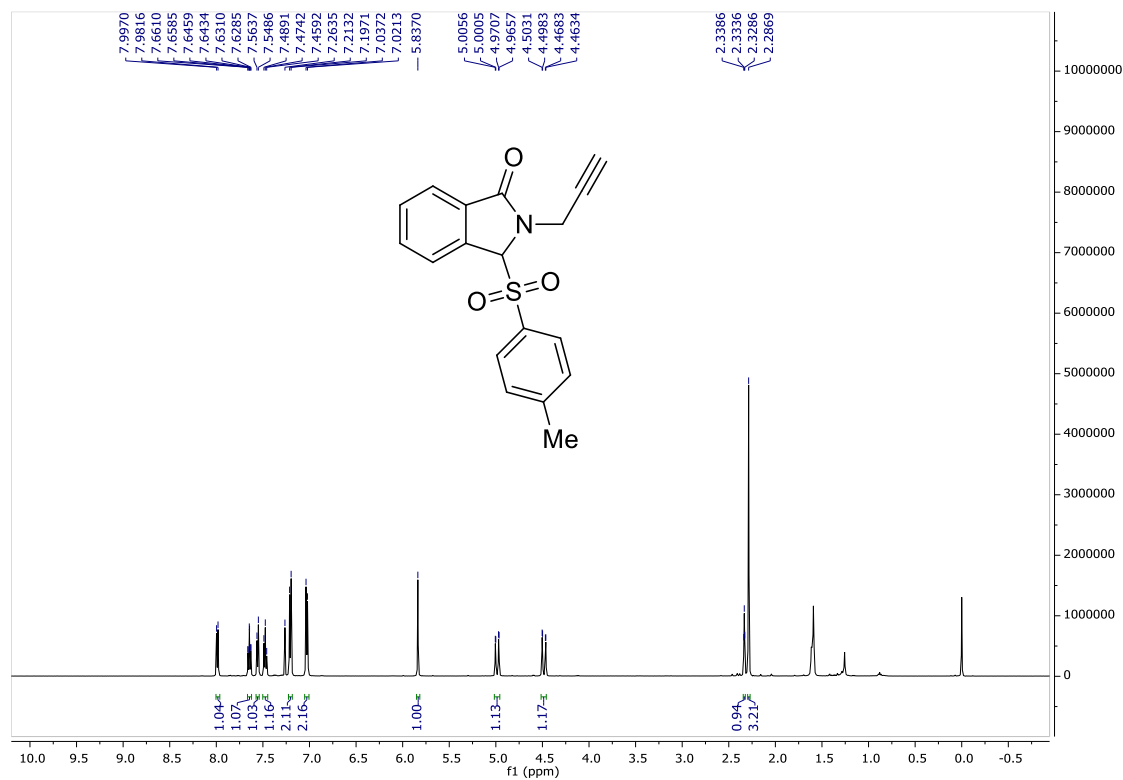
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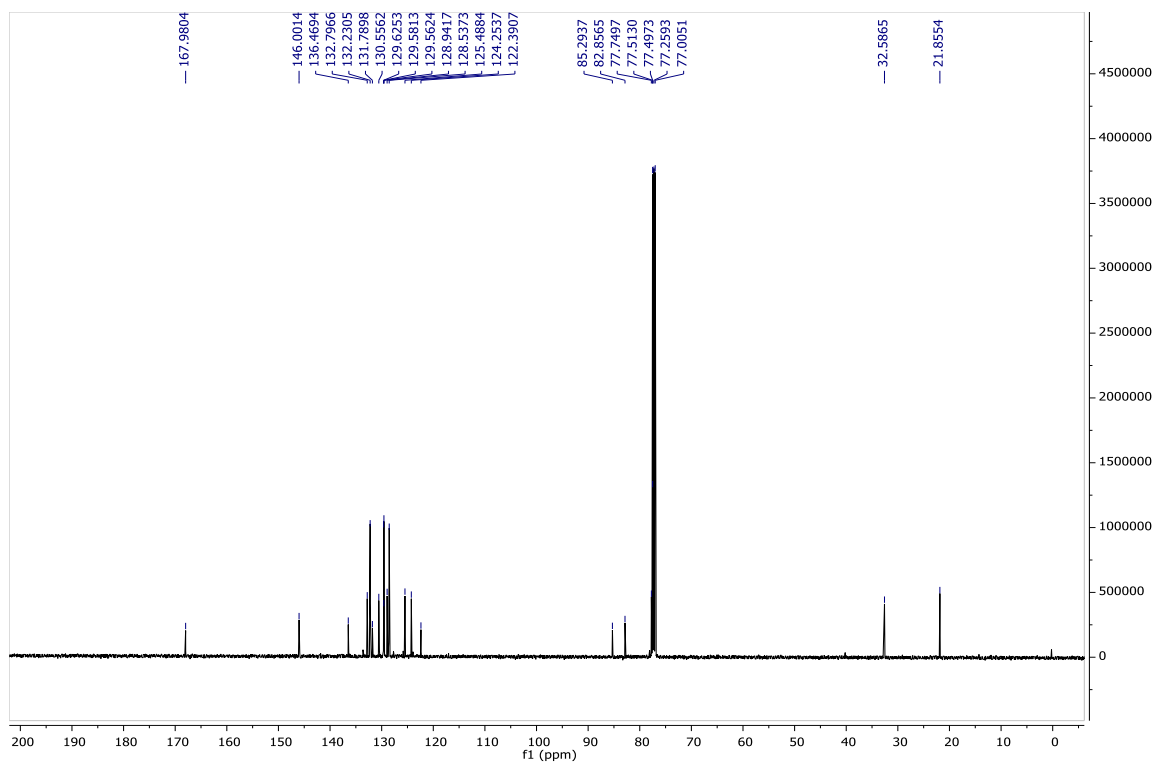
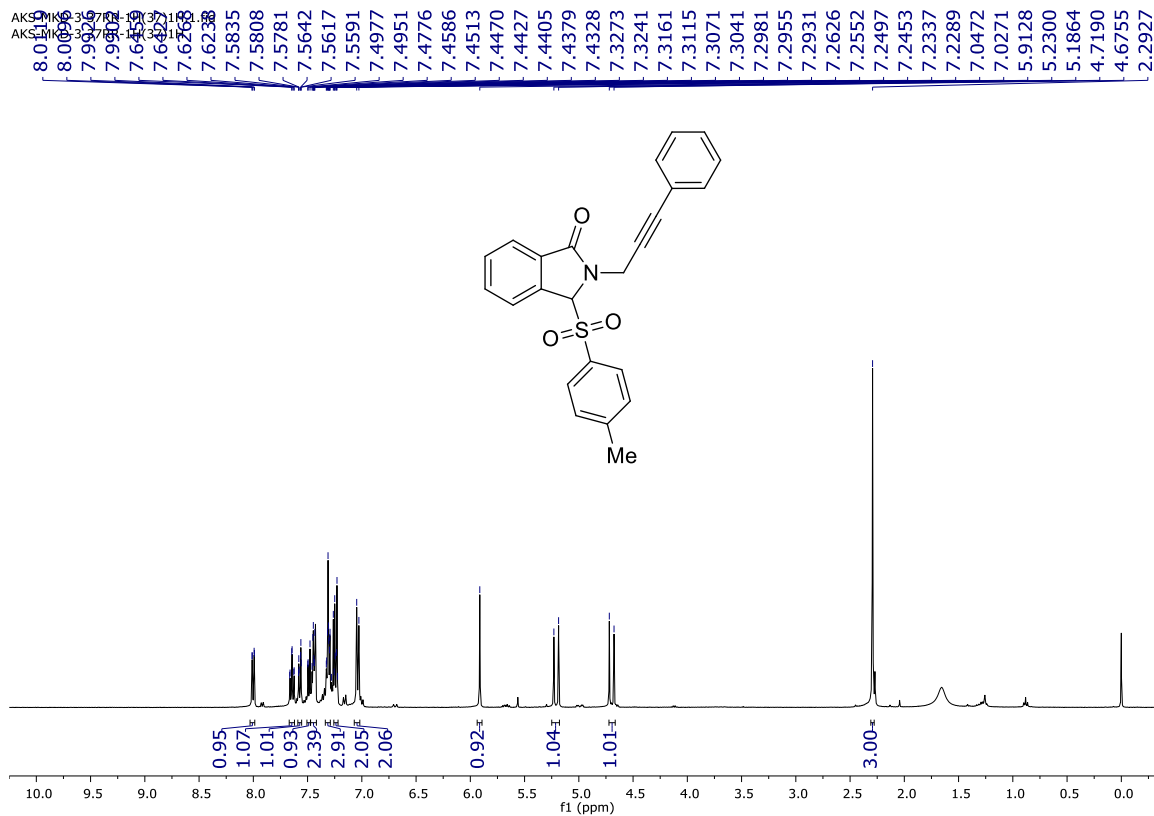
$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 100 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3d.



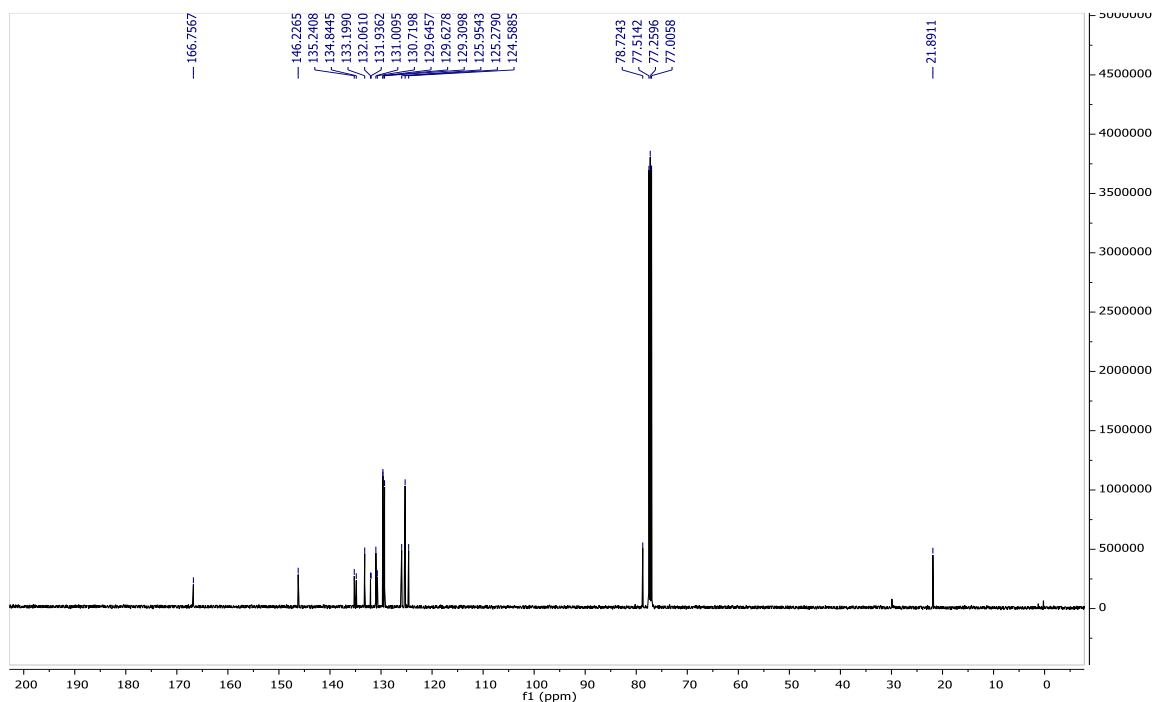
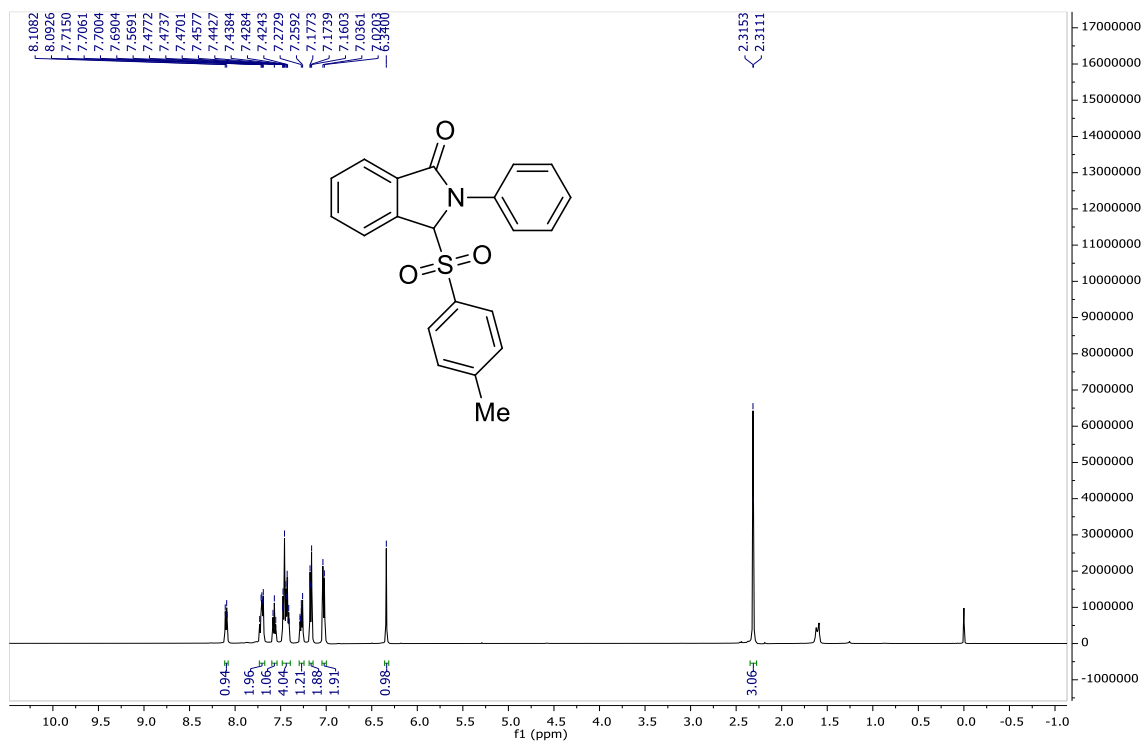
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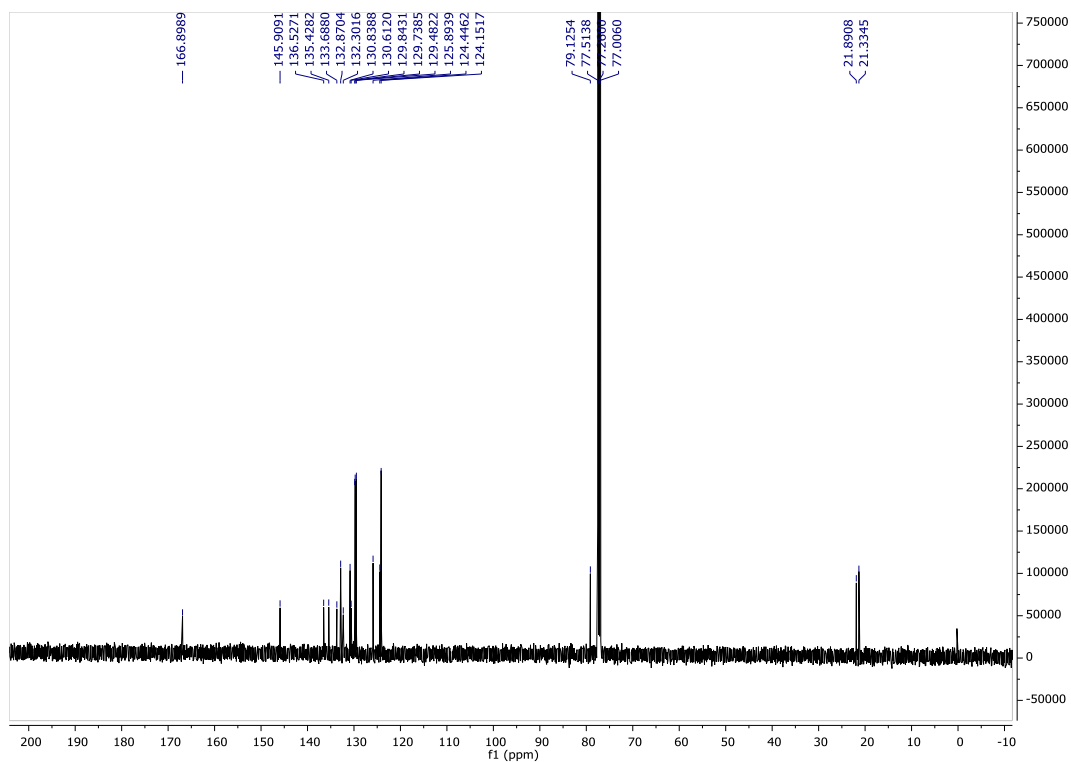
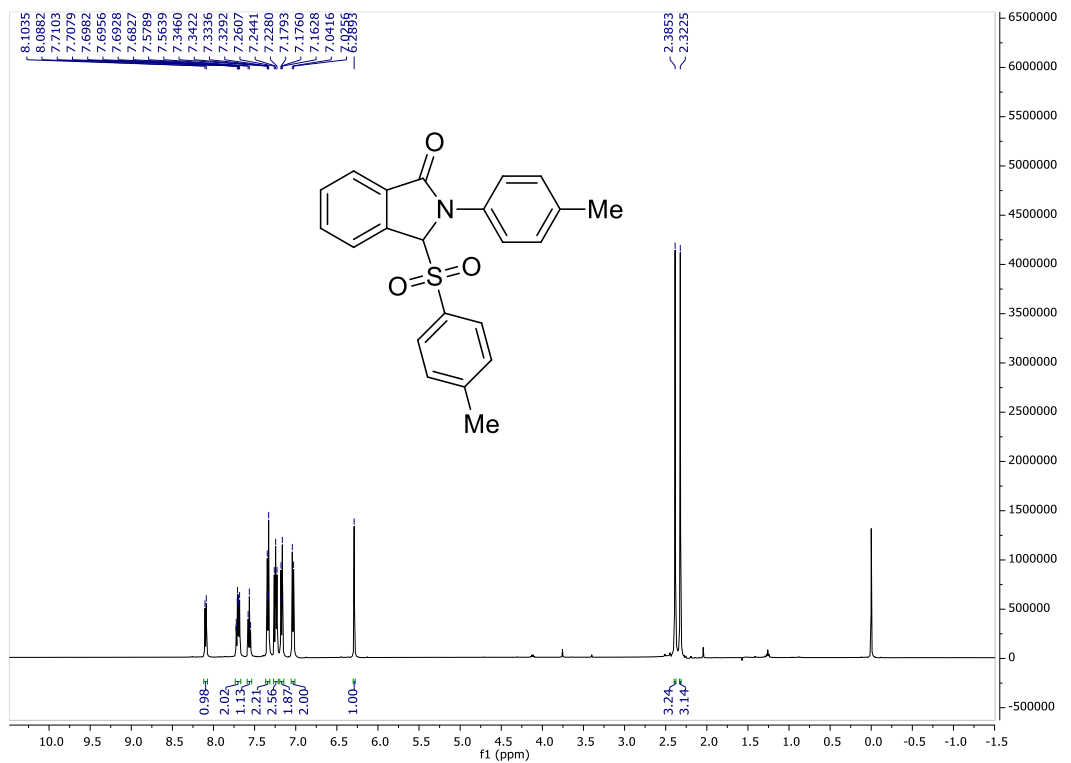
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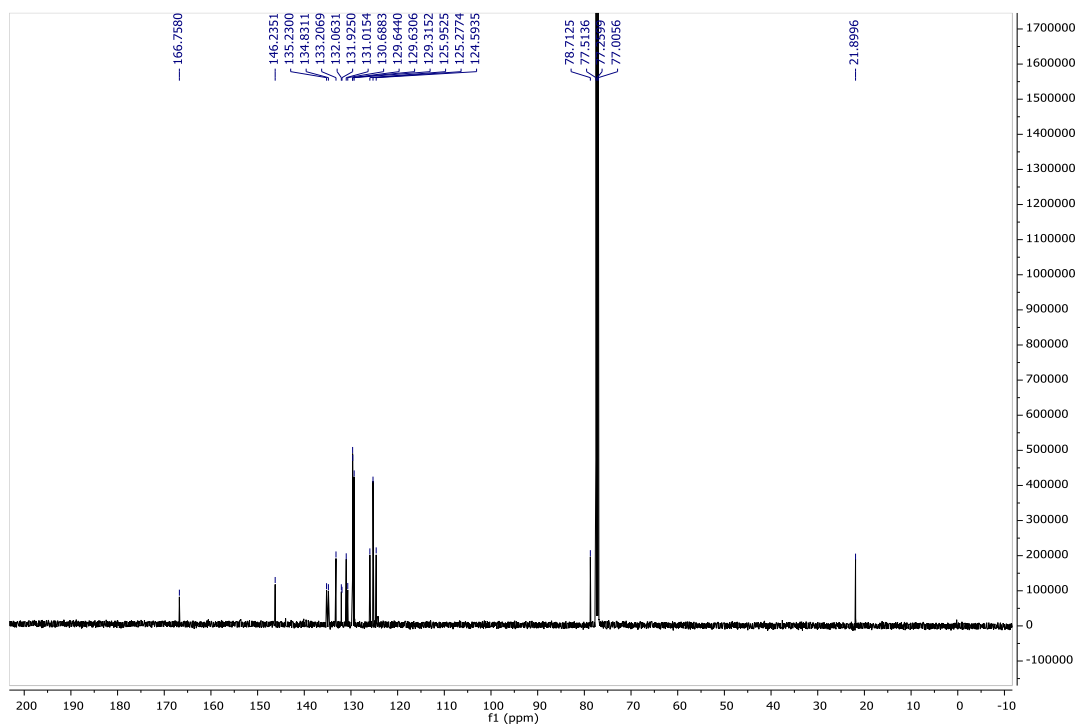
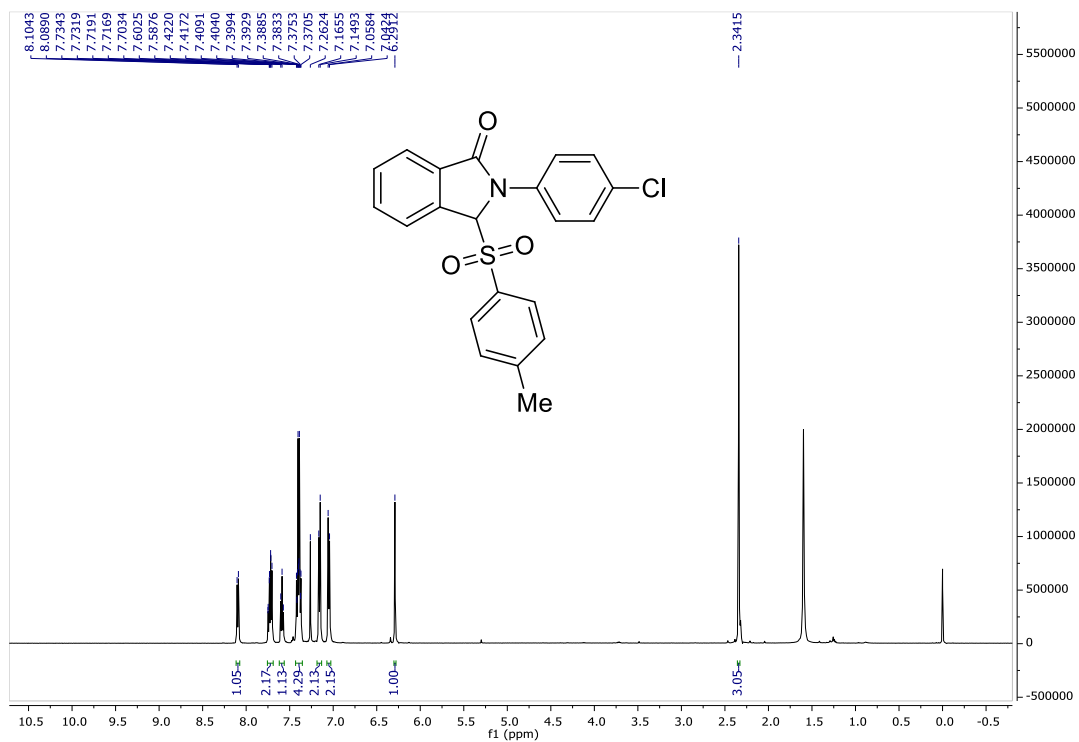
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 100 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3g.**



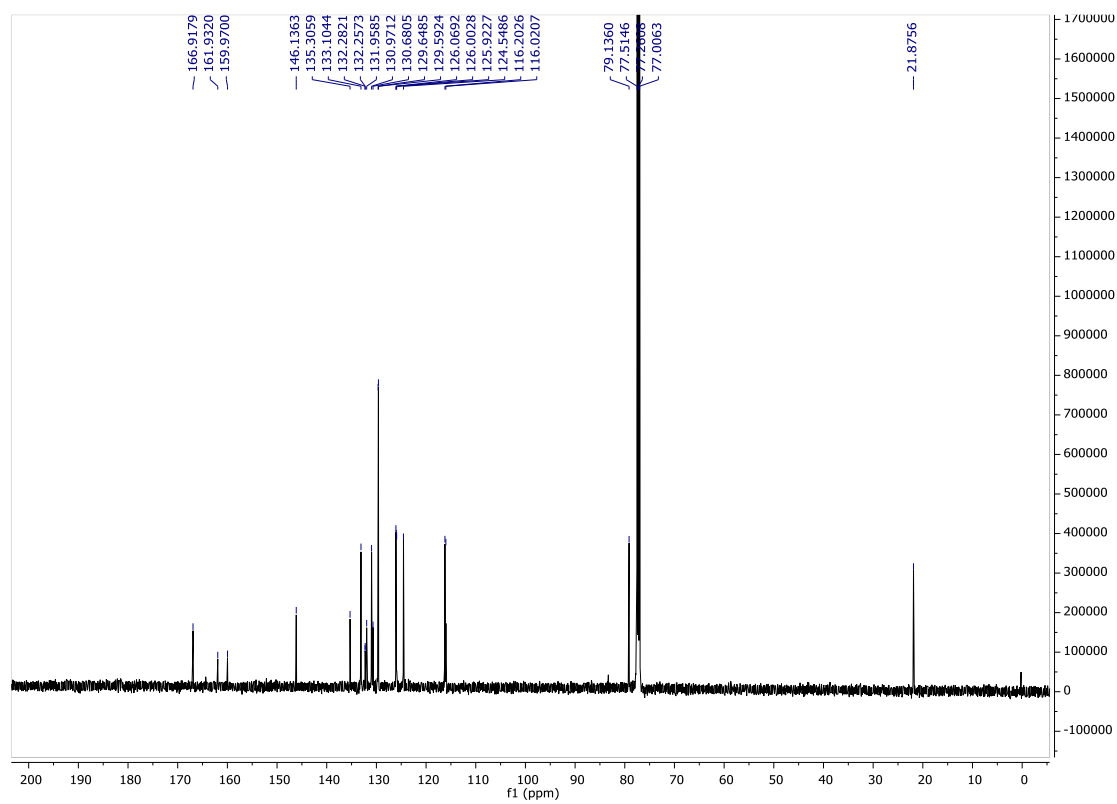
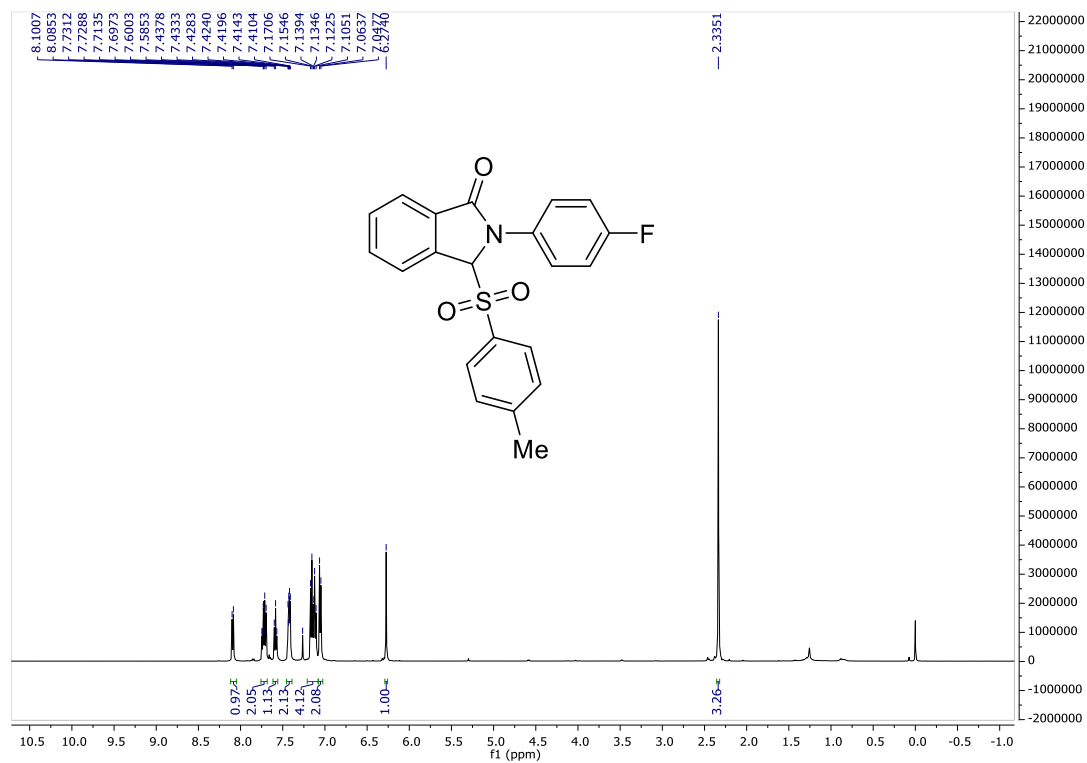
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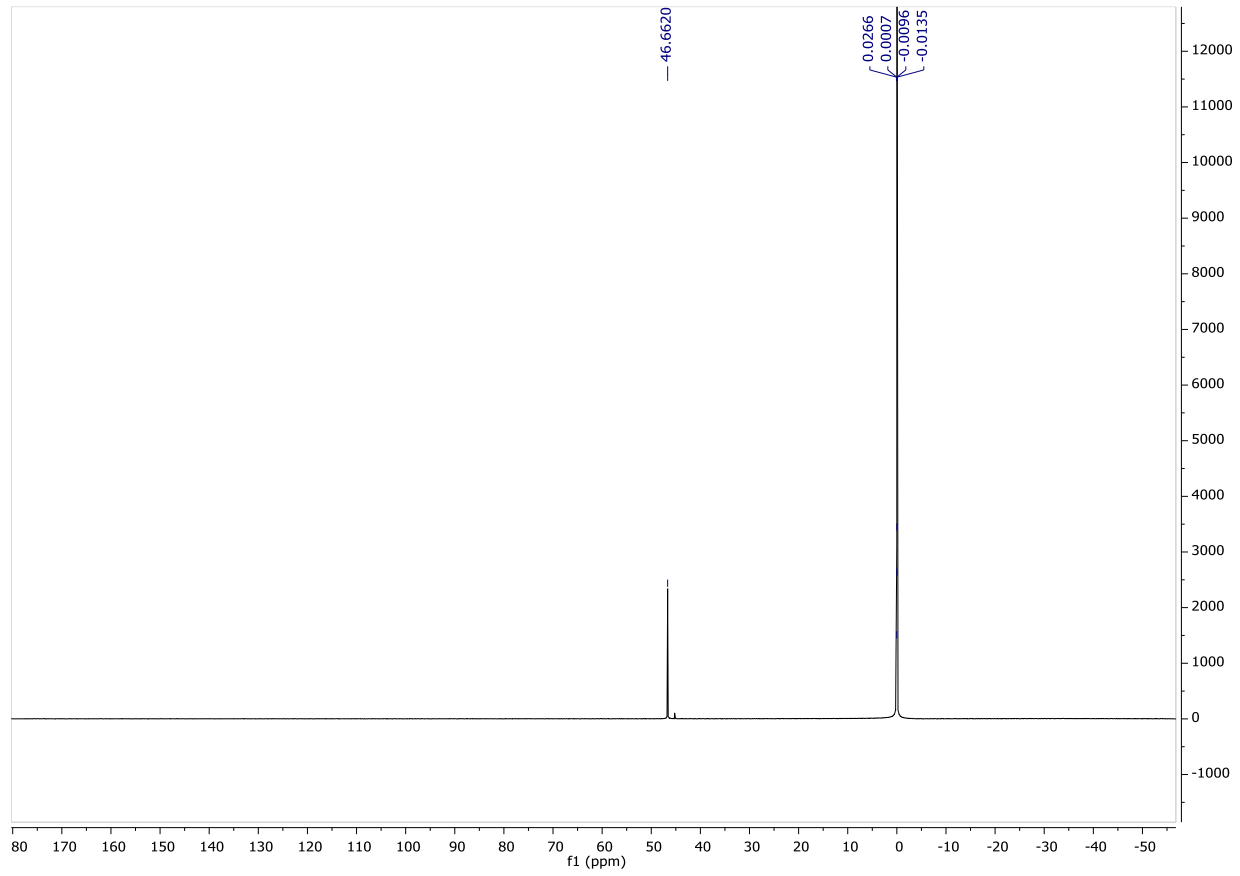
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 100 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3i.**



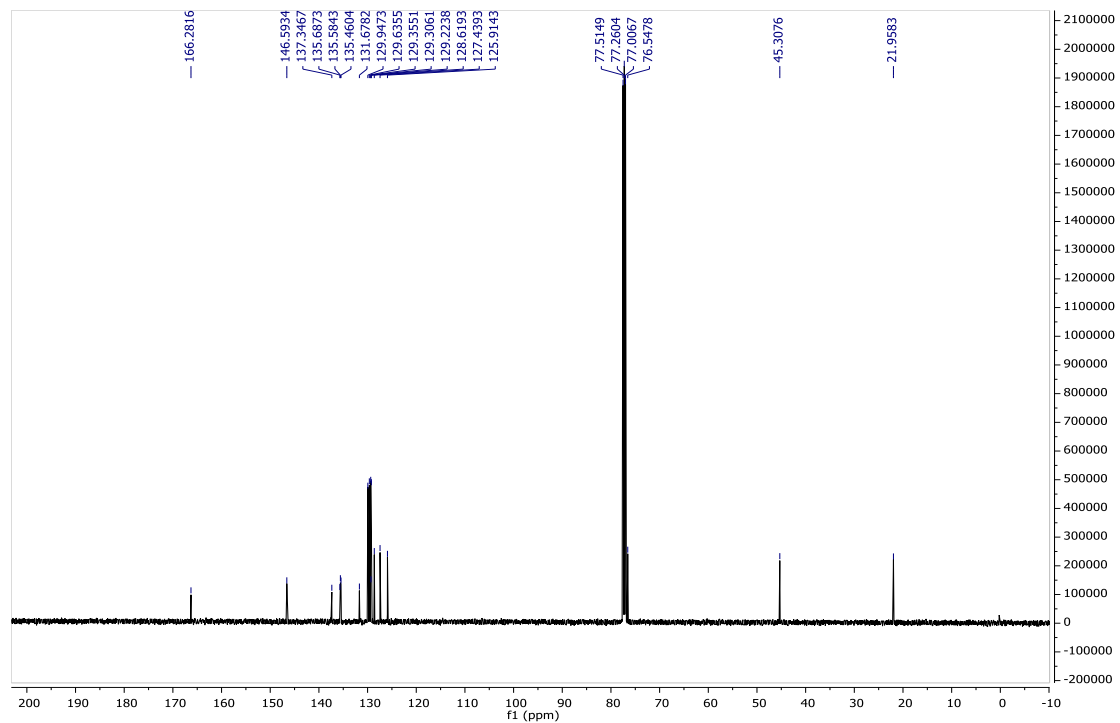
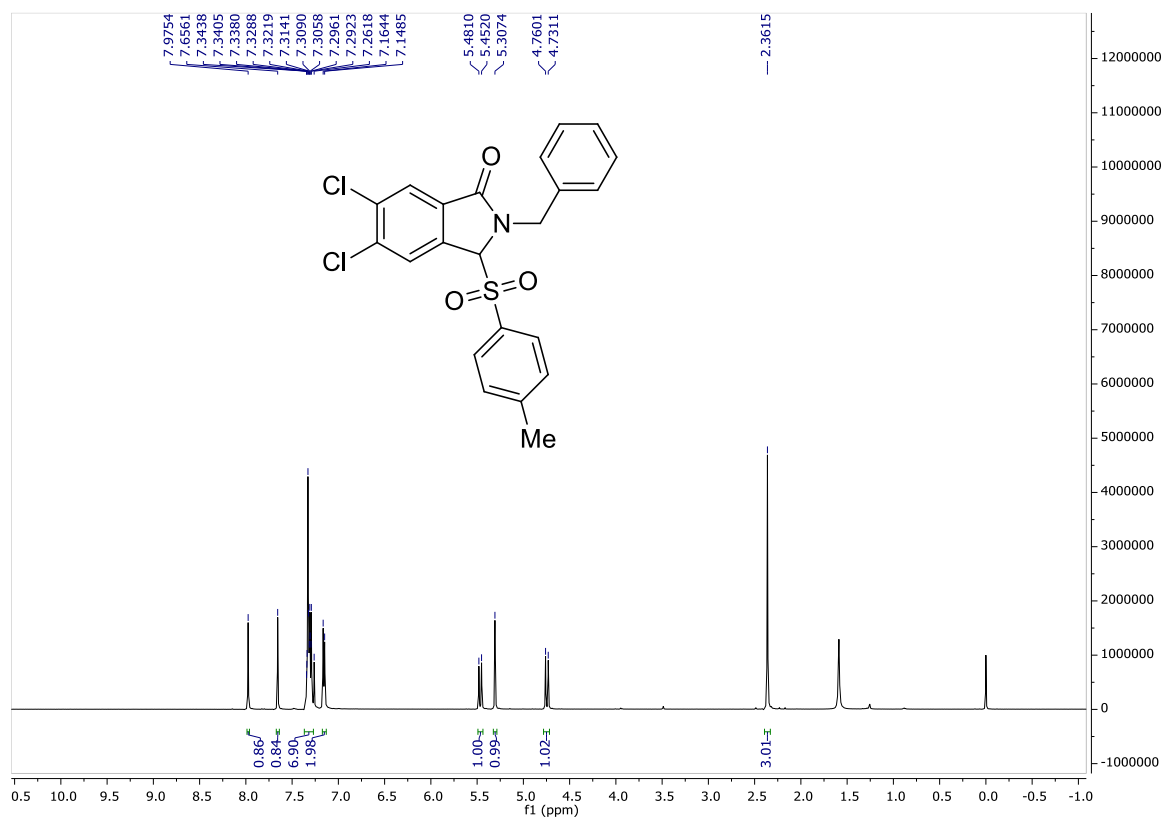
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (500 and 125 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3j**



**$^{19}\text{F}$  (376 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3j**



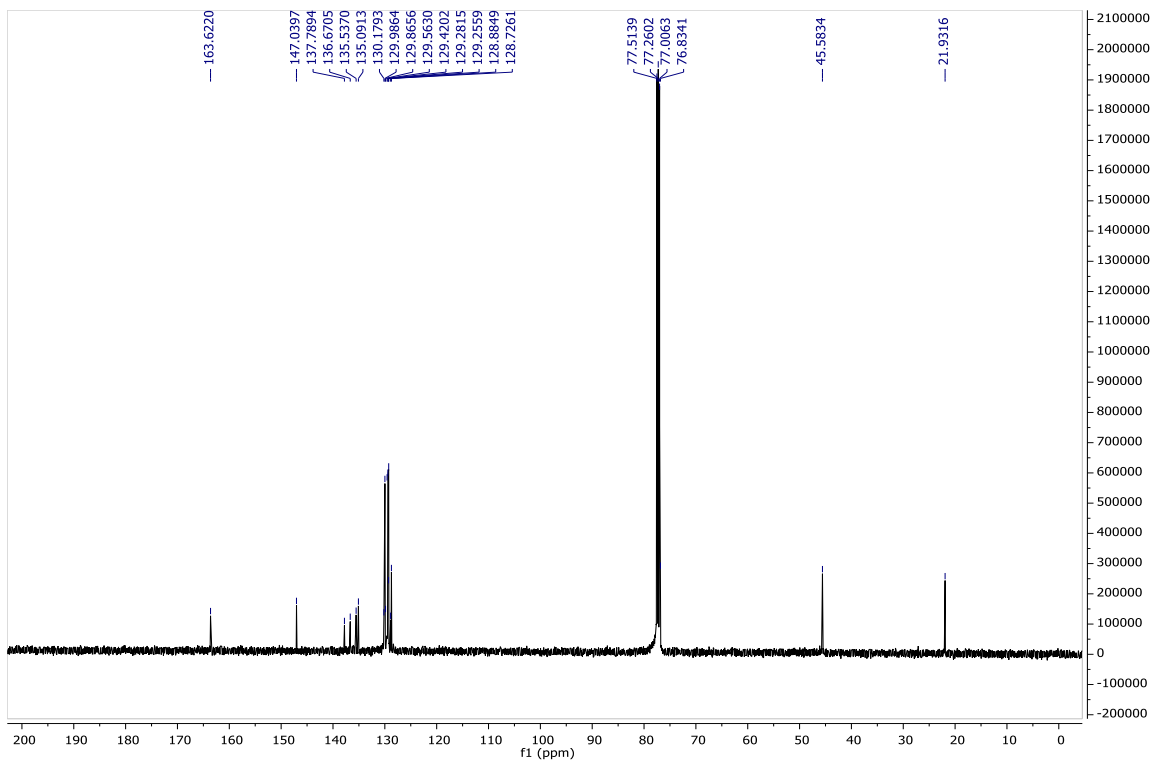
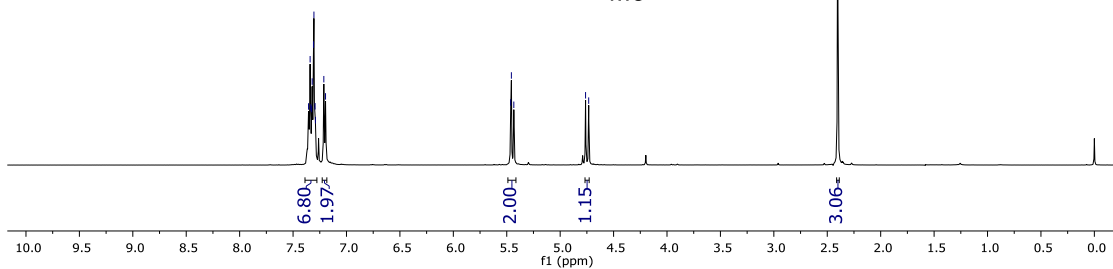
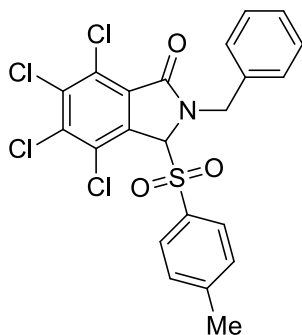
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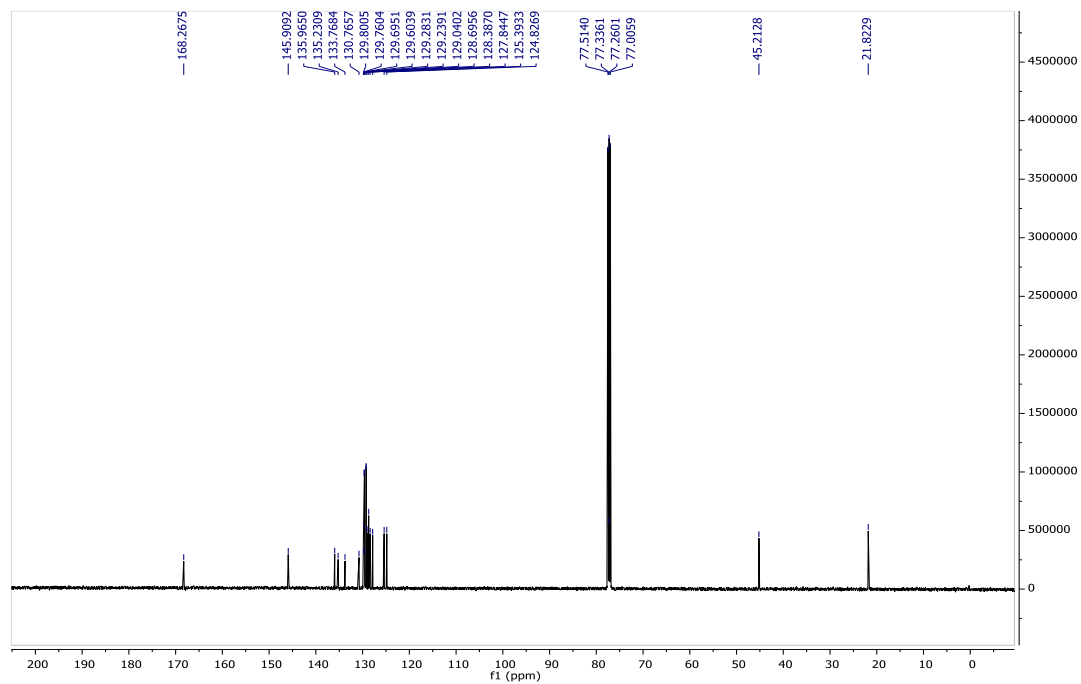
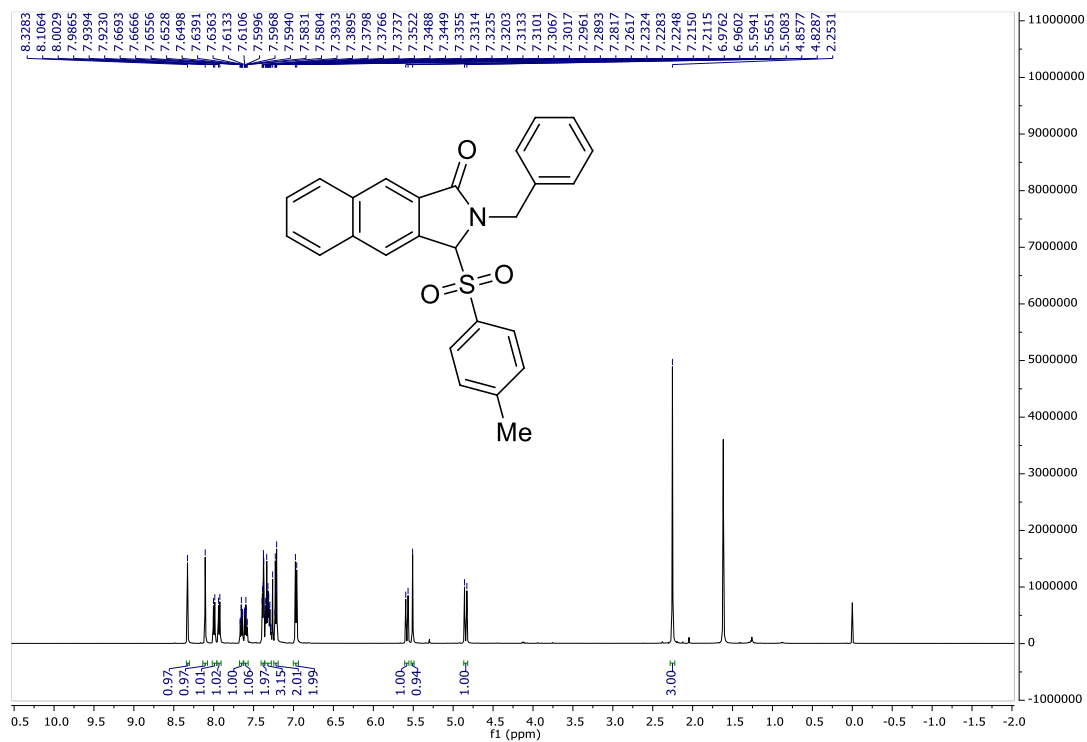
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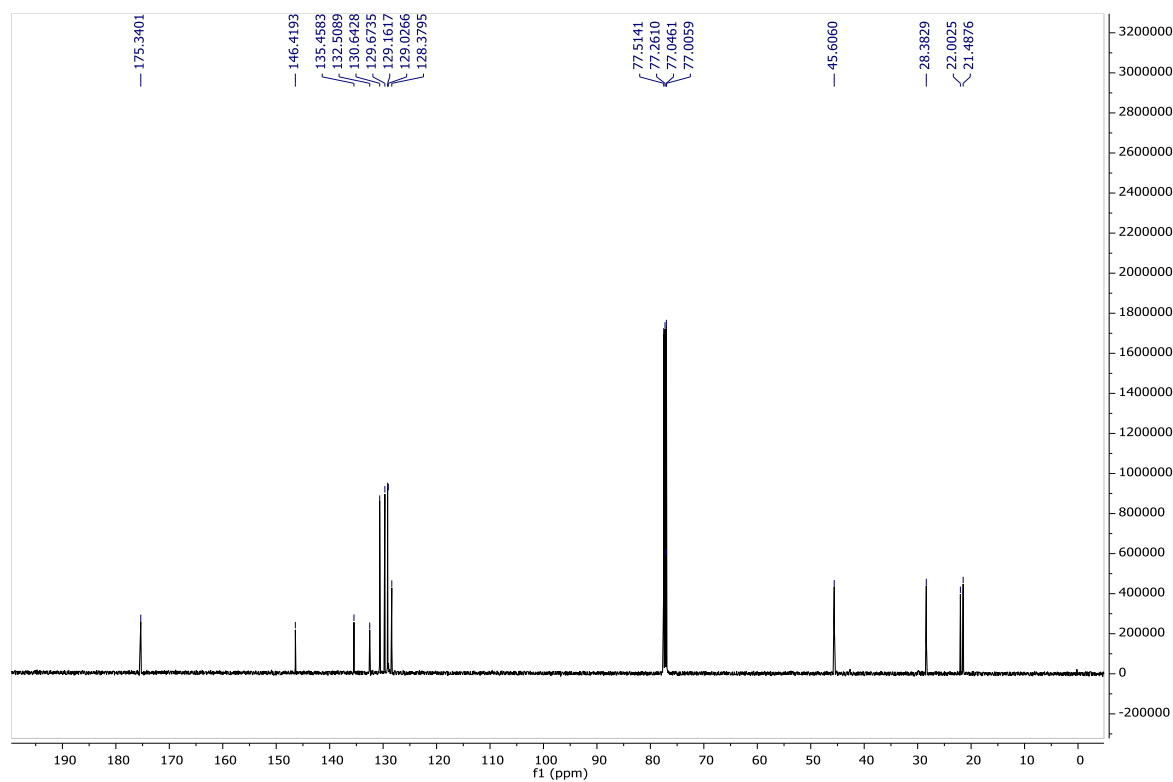
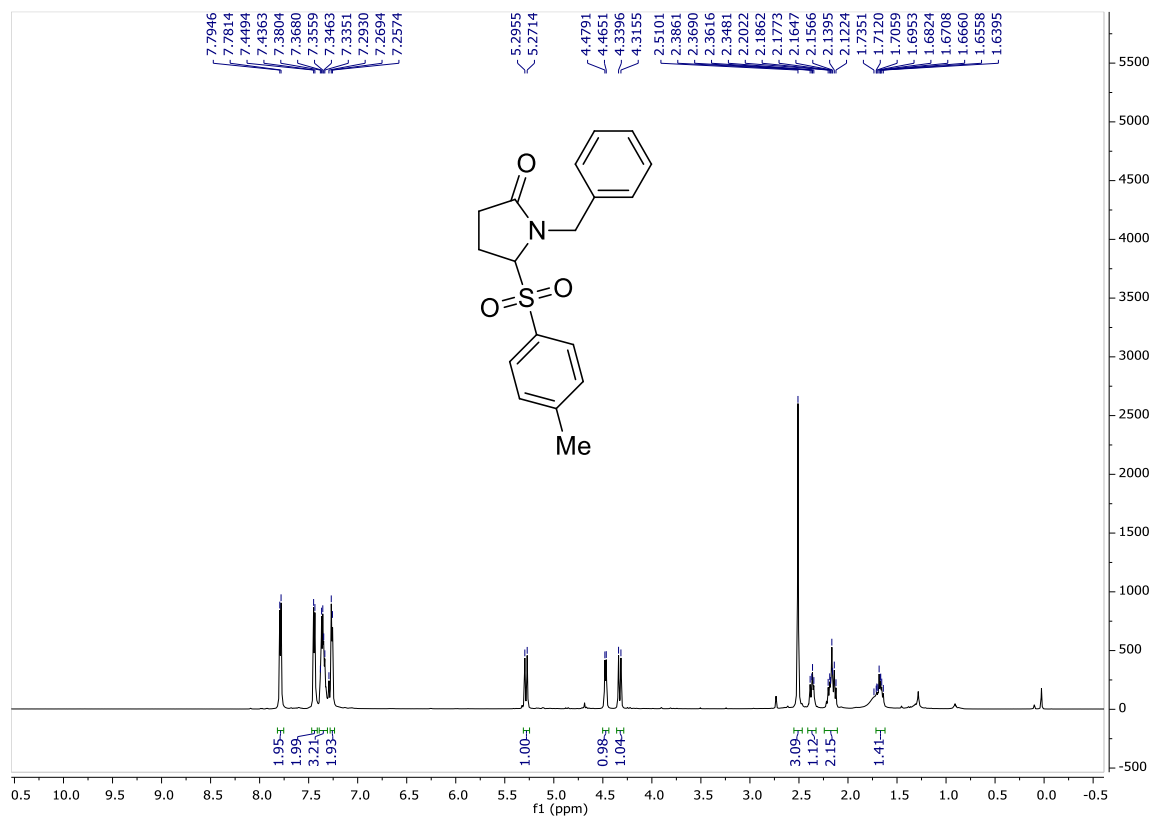
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- 2.4023



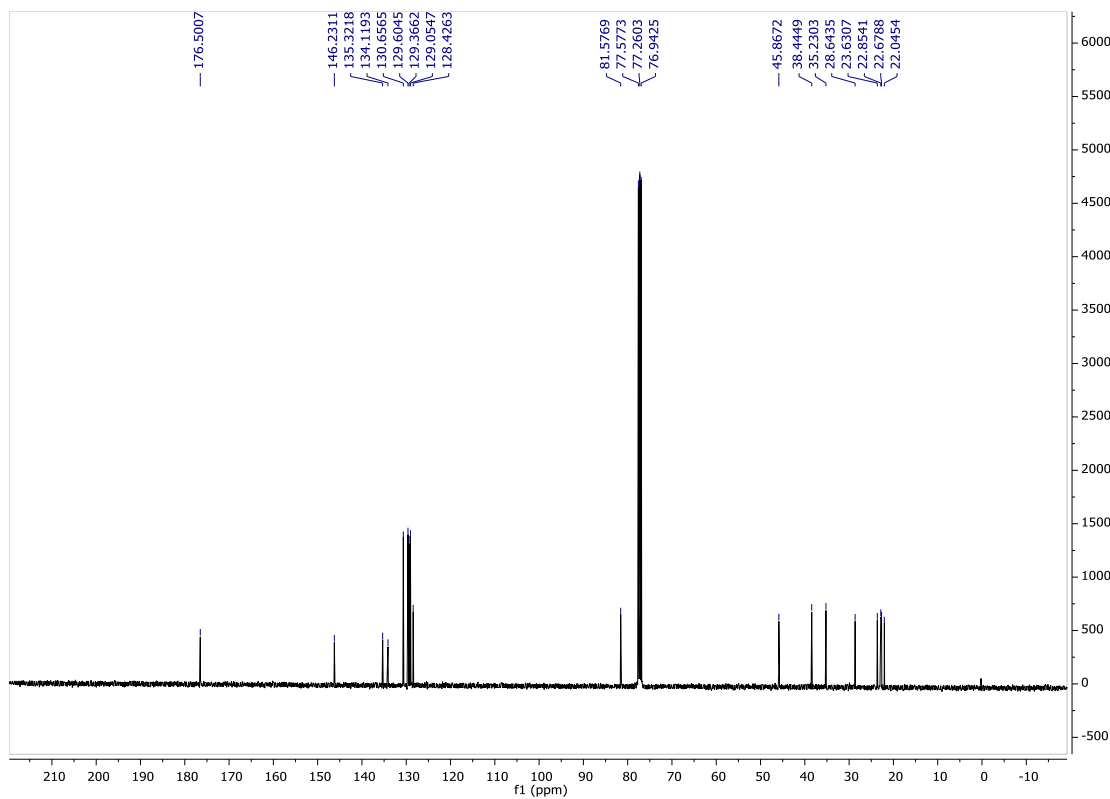
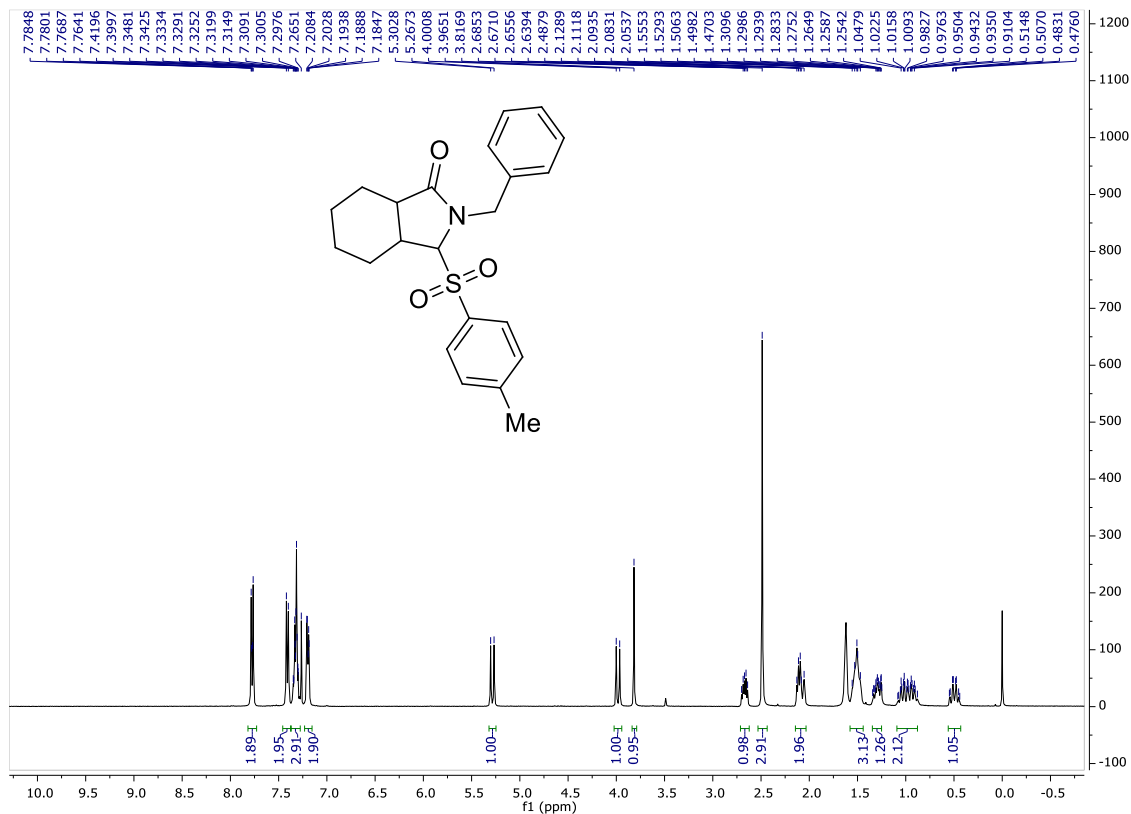
**<sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} (400 and 100 MHz, CDCl<sub>3</sub>) NMR spectra of compound 3m**



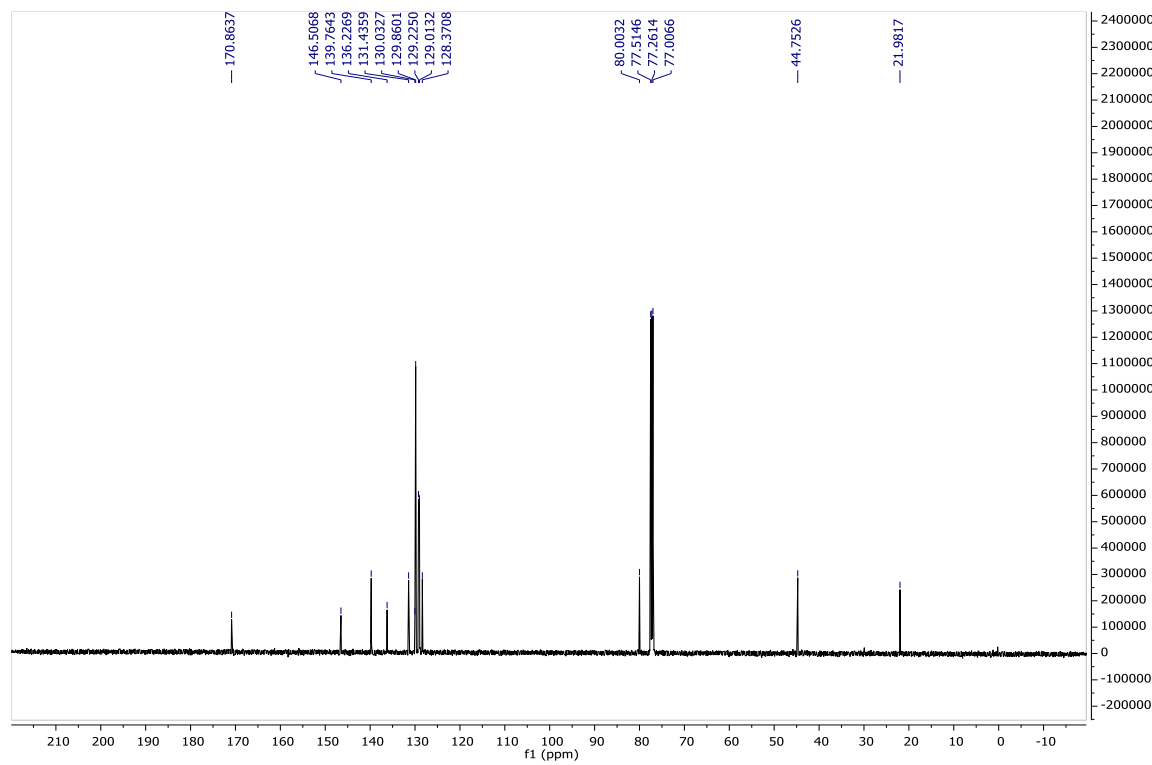
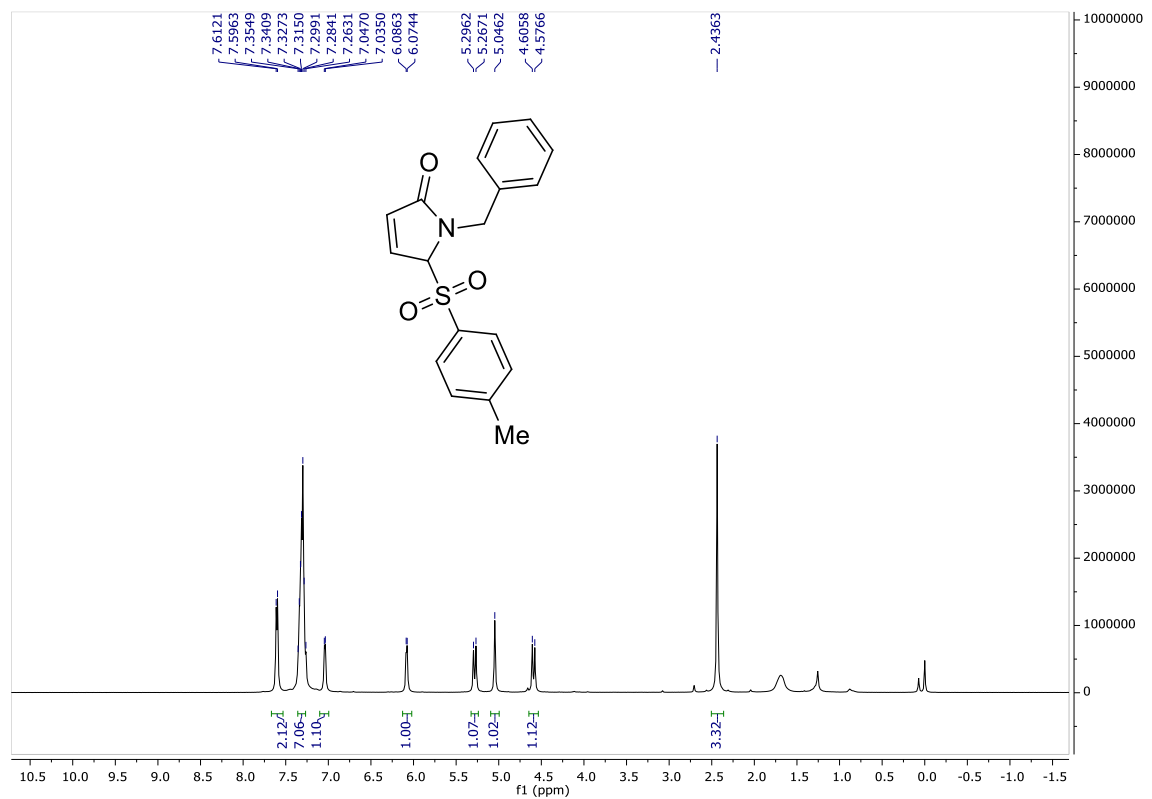
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 100 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3n**



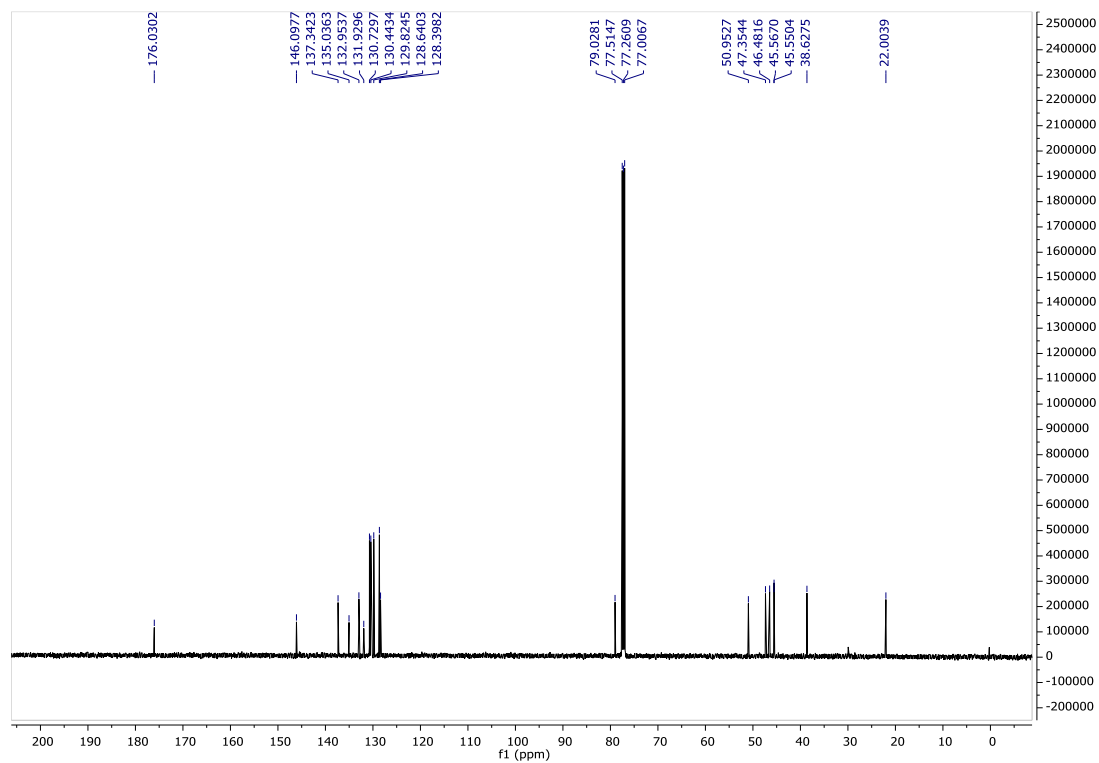
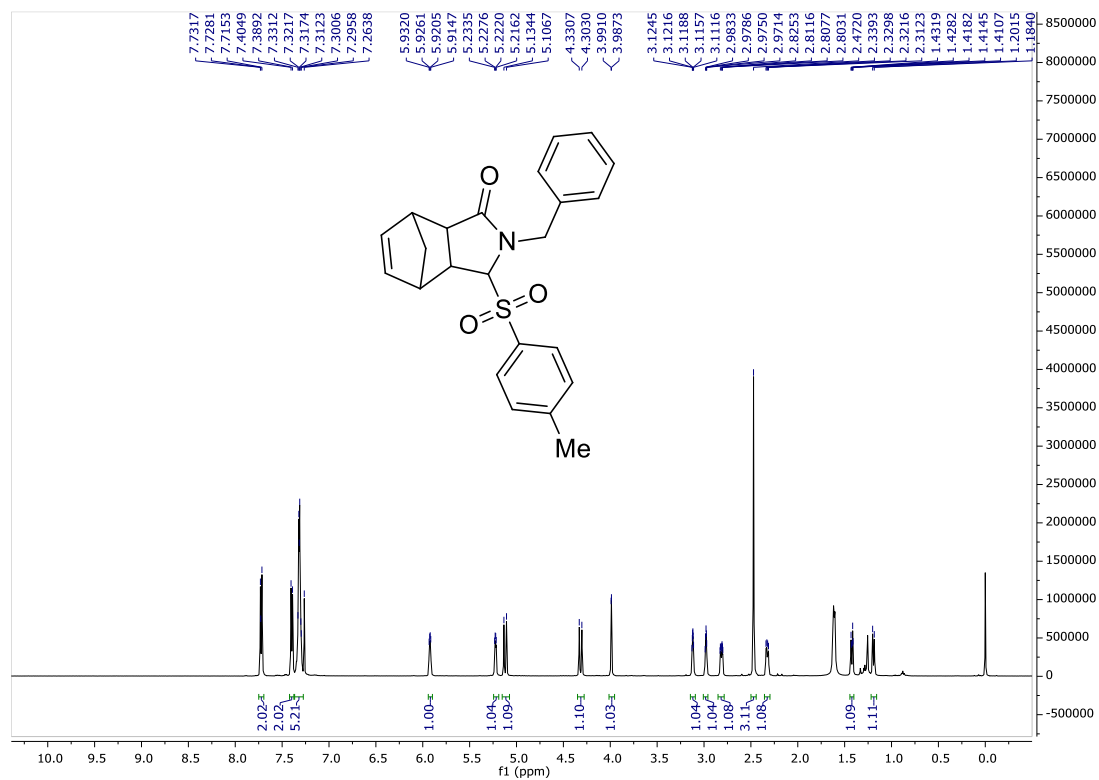
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 100 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3o**



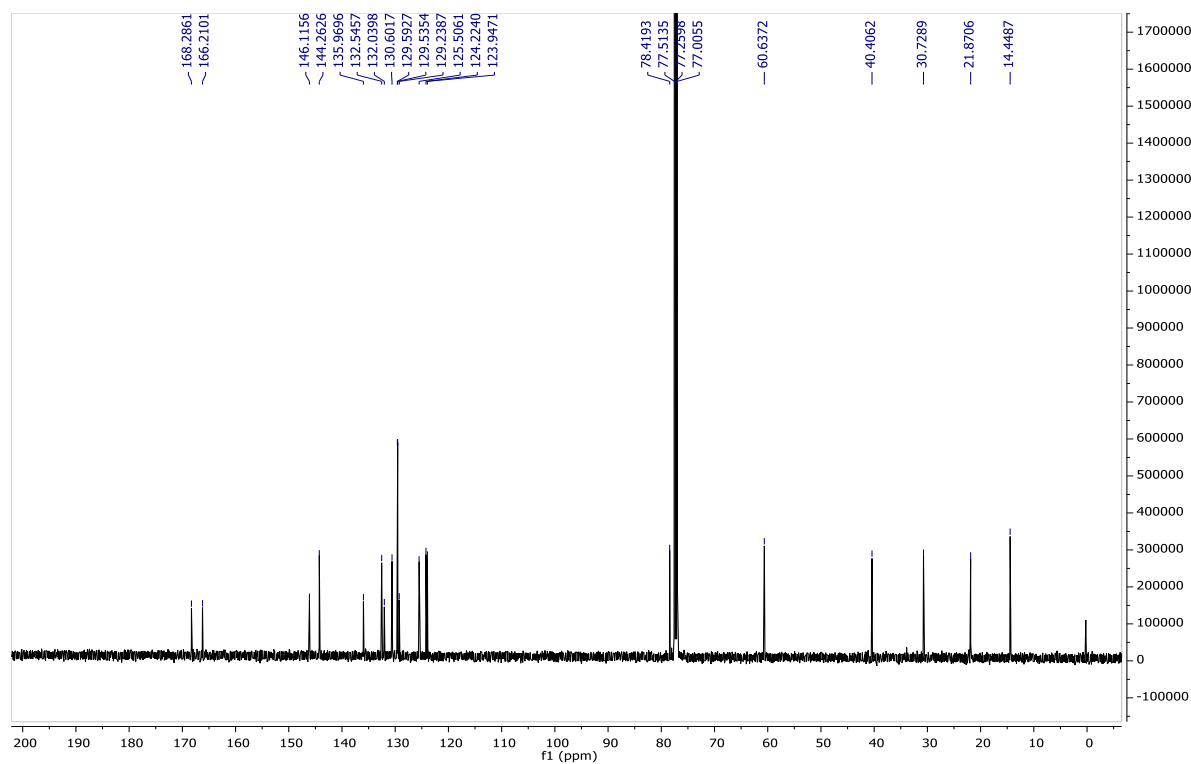
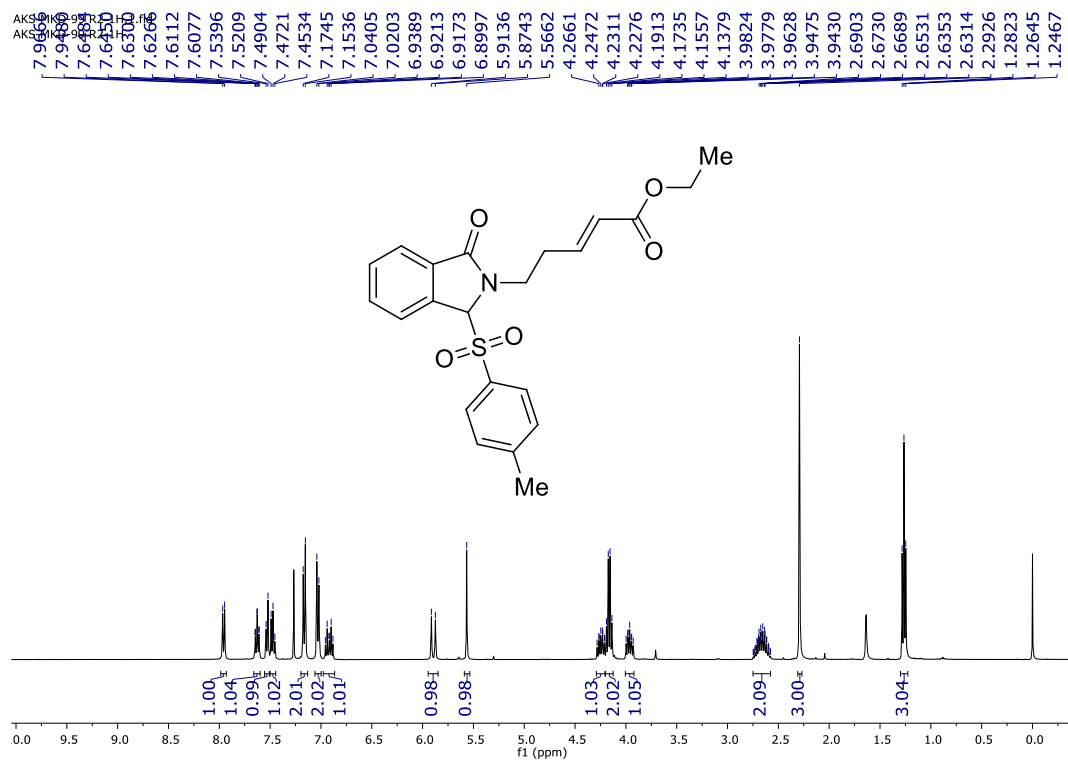
**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (500 and 125 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3p**



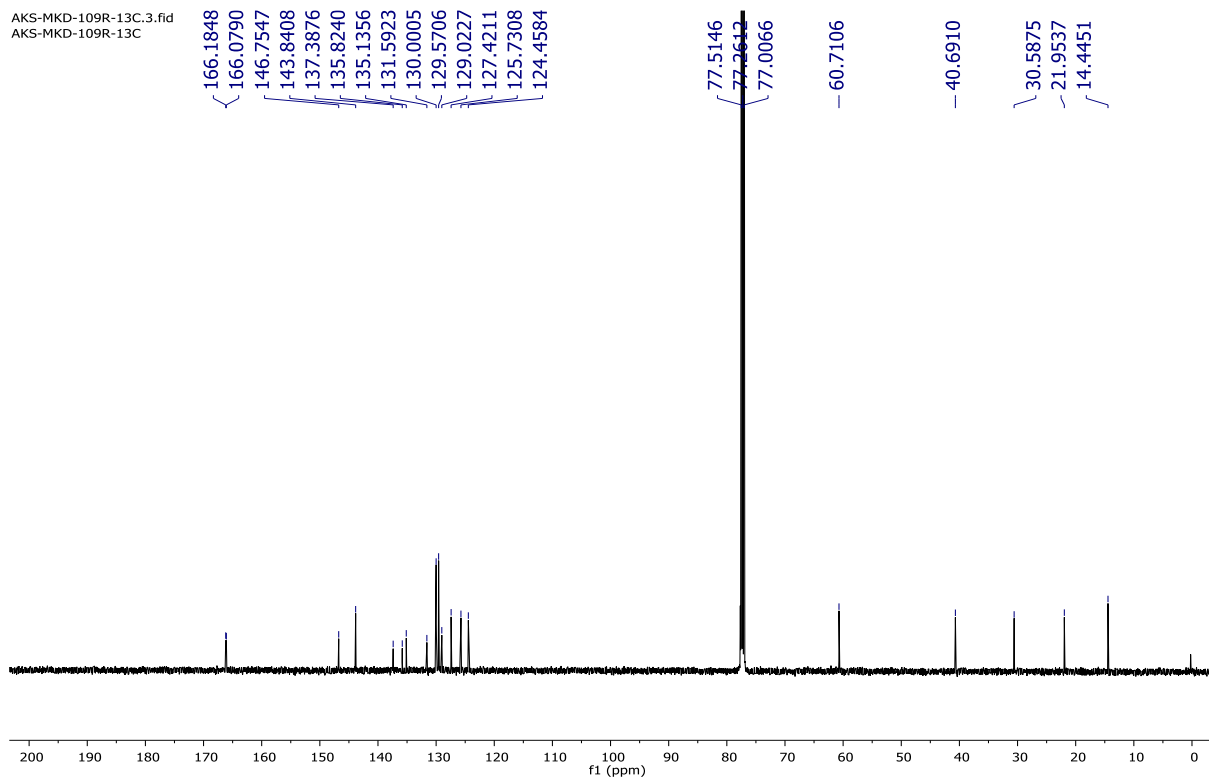
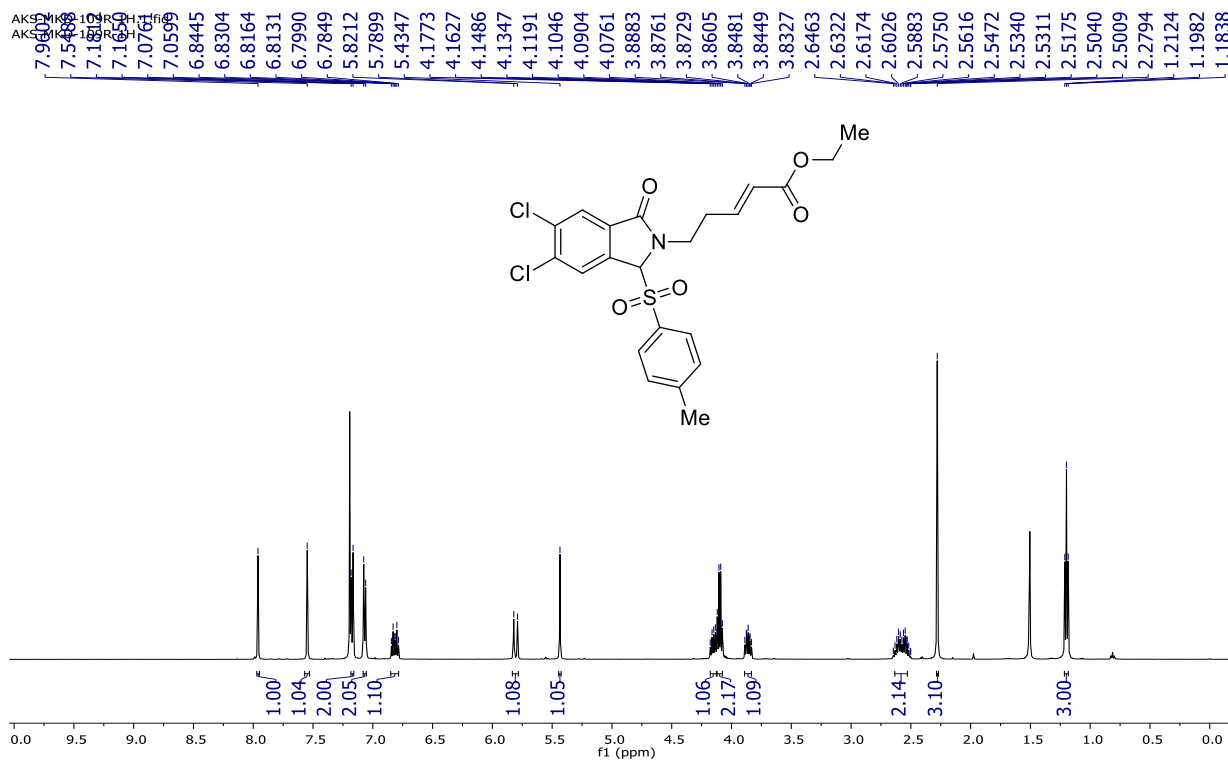
# $^1\text{H}$ and $^{13}\text{C}\{^1\text{H}\}$ (400 and 100 MHz, $\text{CDCl}_3$ ) NMR spectra of compound 3q



**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (400 and 125 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3r**

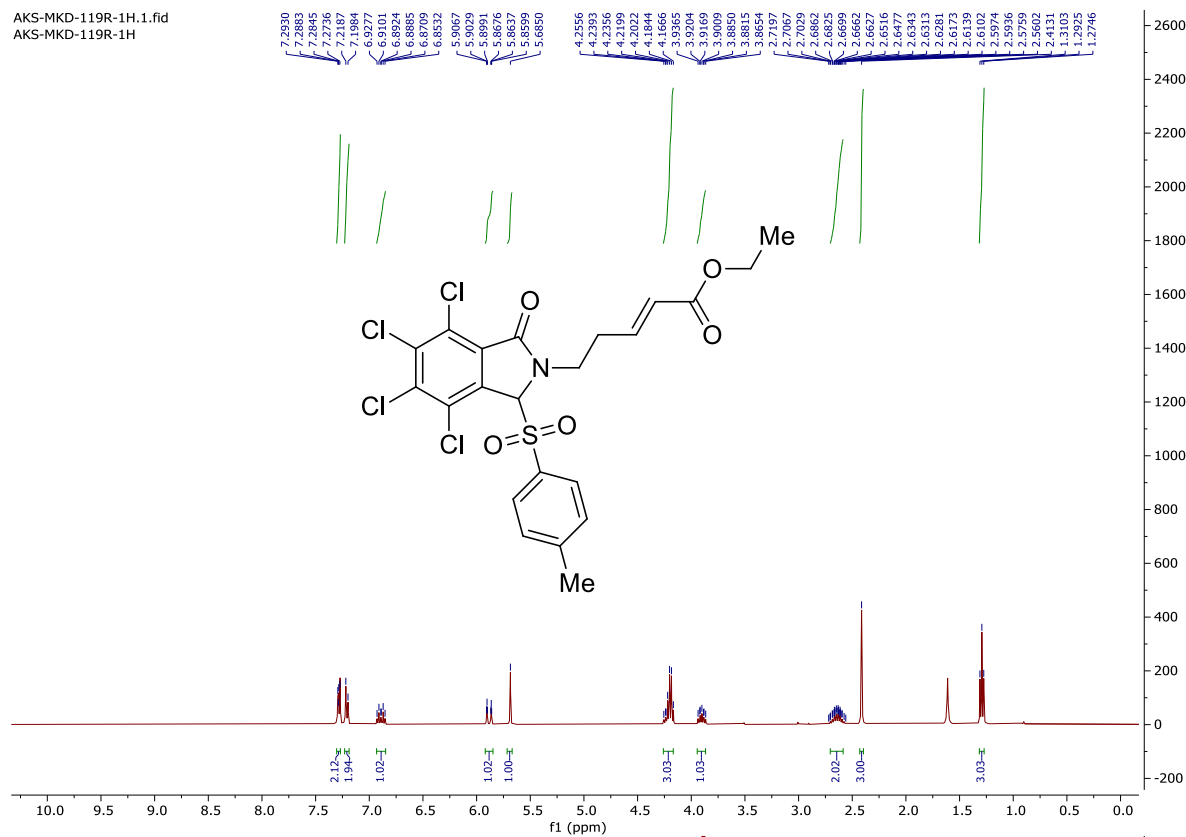


**$^1\text{H}$  and  $^{13}\text{C}\{^1\text{H}\}$  (500 and 125 MHz,  $\text{CDCl}_3$ ) NMR spectra of compound 3s**

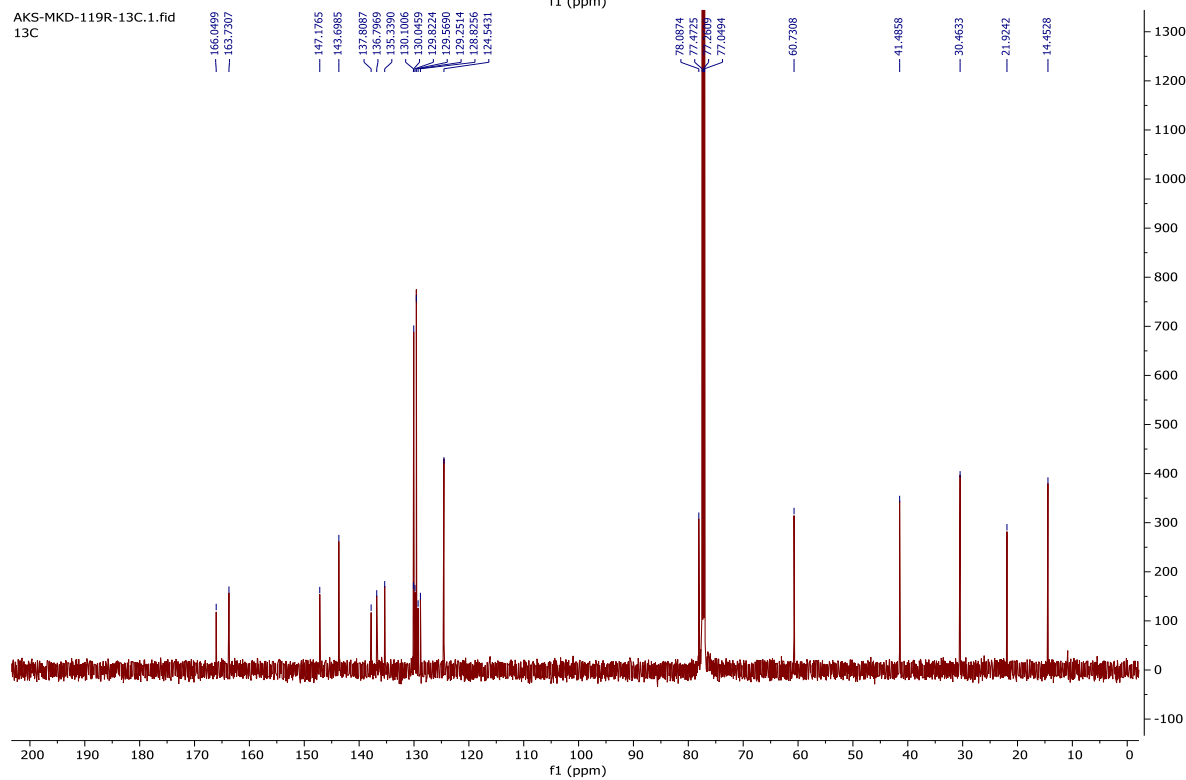


# $^1\text{H}$ and $^{13}\text{C}\{^1\text{H}\}$ (400 and 150 MHz, $\text{CDCl}_3$ ) NMR spectra of compound 3t

AKS-MKD-119R-1H.1.fid  
AKS-MKD-119R-1H

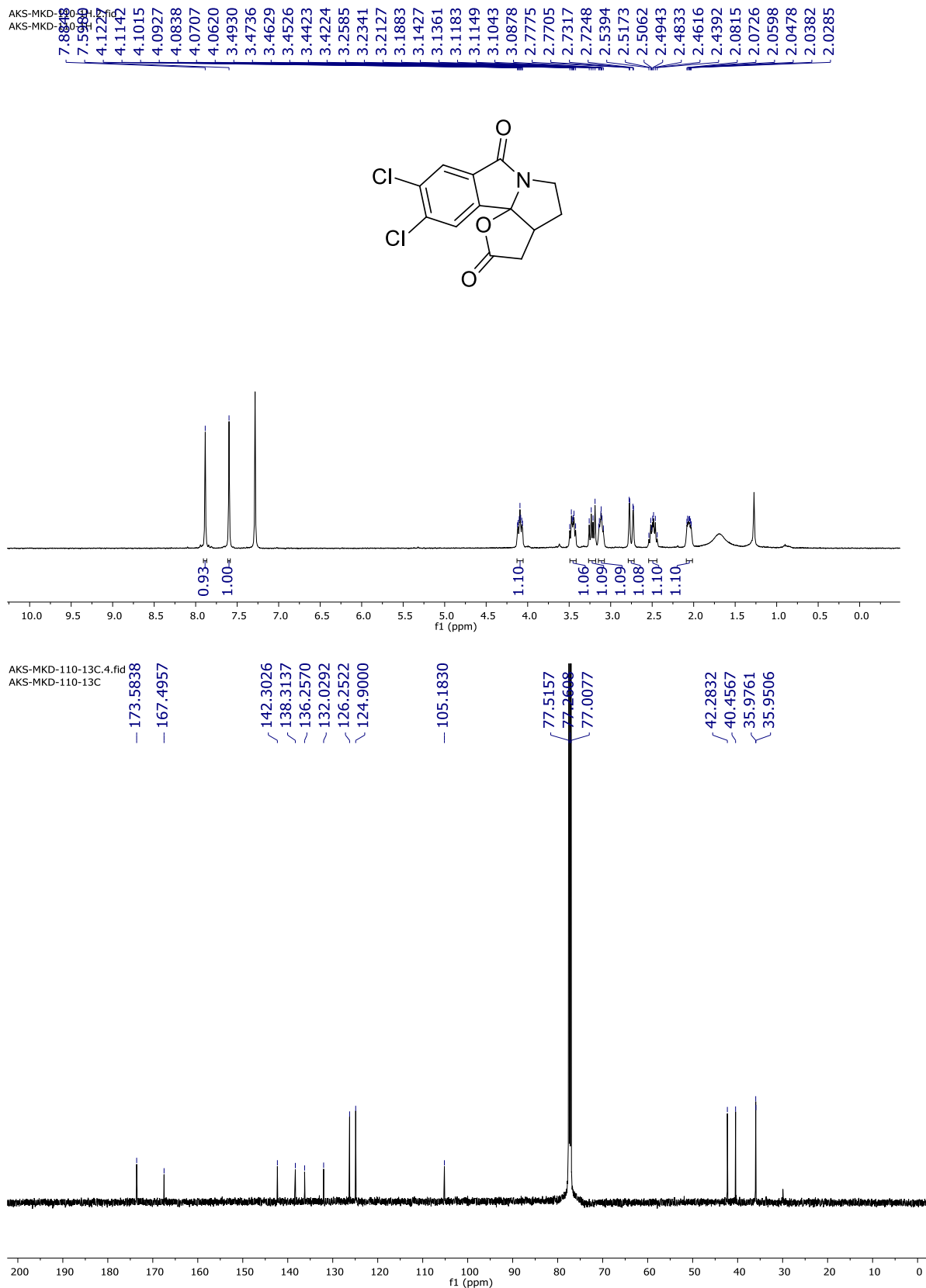


AKS-MKD-119R-13C.1.fid  
13C



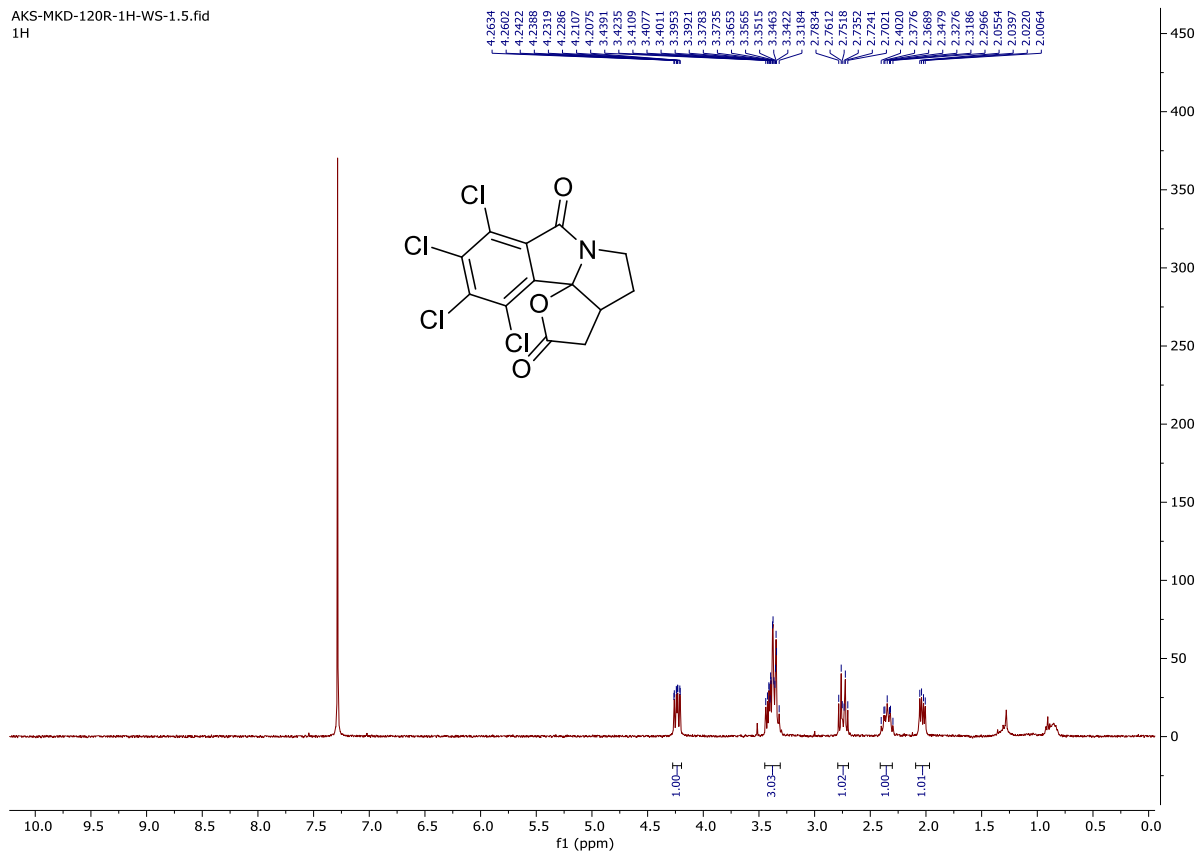


# $^1\text{H}$ and $^{13}\text{C}\{^1\text{H}\}$ (400 and 125 MHz, $\text{CDCl}_3$ ) NMR spectra of compound 4b

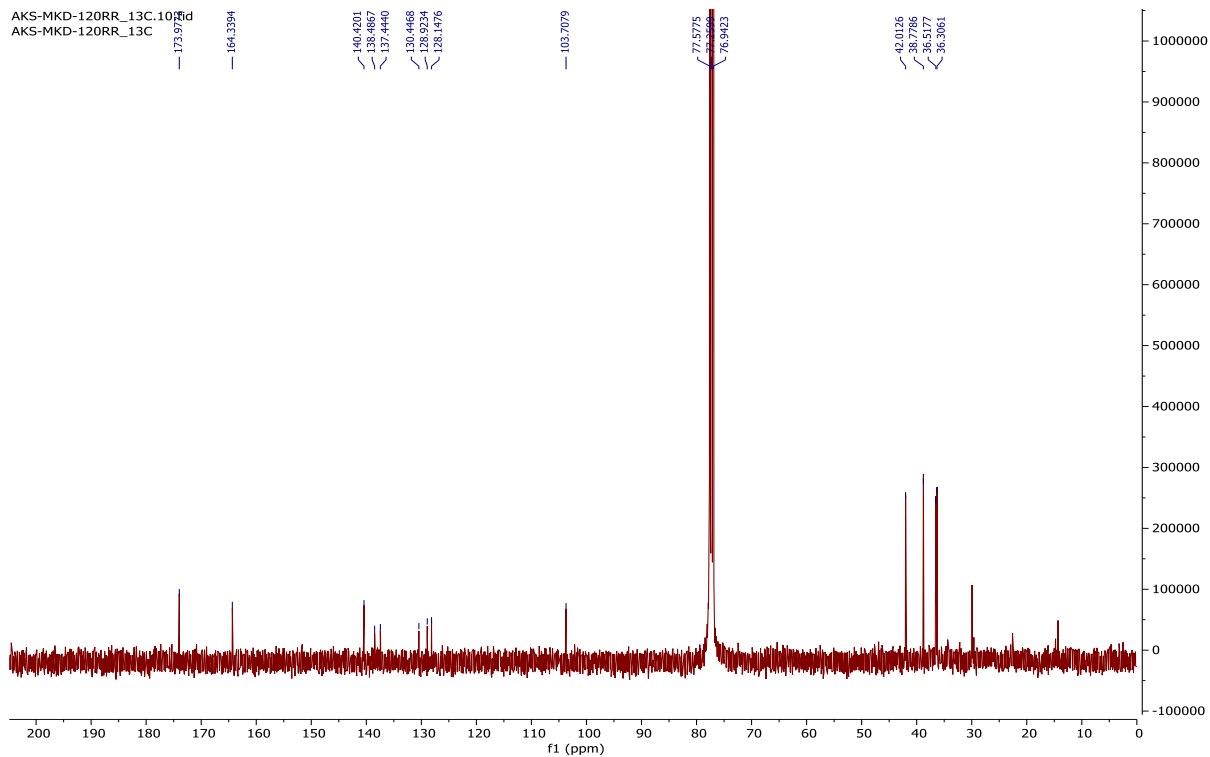


# <sup>1</sup>H and <sup>13</sup>C{<sup>1</sup>H} (400 and 100 MHz, CDCl<sub>3</sub>) NMR spectra of compound 4c

AKS-MKD-120R-1H-WS-1.5.fid  
1H



AKS-MKD-120RR\_13C.102.fid  
AKS-MKD-120RR\_13C



### Single crystal X-ray diffraction

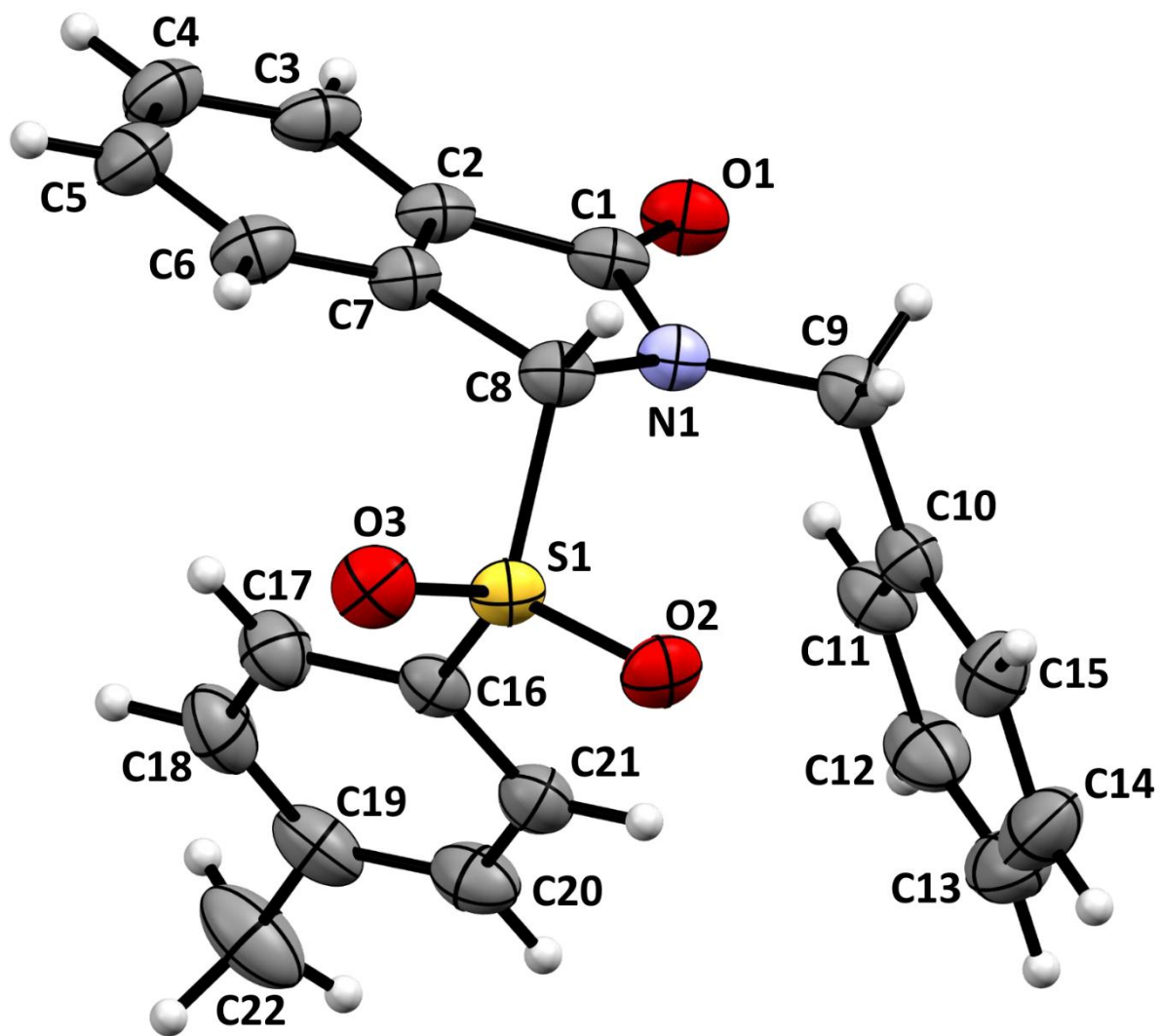
Single crystals of compounds **3a** and **4a** was obtained by slow evaporation of ethyl acetate and hexane solution (1:9) and dichloromethane and hexane(1:9) respectively. Bruker APEX-II CCD diffractometer was used to collect the intensity data. The instrument is equipped with a fine focus 1.75 kW sealed tube Mo K $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) at 297(2) and 294(2) K. The data acquisition was done with the APEX4 software. APEX4 software was implemented for data integration and reduction. Multi-scan empirical absorption corrections were employed to the data using the program APEX4. Structures were solved by direct methods using SHELXL-2019 and refined with full-matrix least-squares on F2 using SHELXL-2019/1.<sup>1</sup> Structural illustrations have been drawn with ORTEP-3 for Windows.<sup>2</sup> The detailed data collection and structure refinement are summarized in Tables S1 and S2. CCDC-2430245 (for **3a**) and CCDC-2430246 (for **4a**) contained supplementary crystallographic data for this paper.

### References:

- 1) Sheldrick G M, SHELXS-2014, Program for the crystal structure solution; University of Göttingen: Göttingen, Germany, 2014.
- 2) Farrugia L J, XRDIF: Simulation of X-ray diffraction patterns, *J Appl Crystallogr*, 30 (1997) 565.

**Table S1.** The crystal parameters of compound **3a**

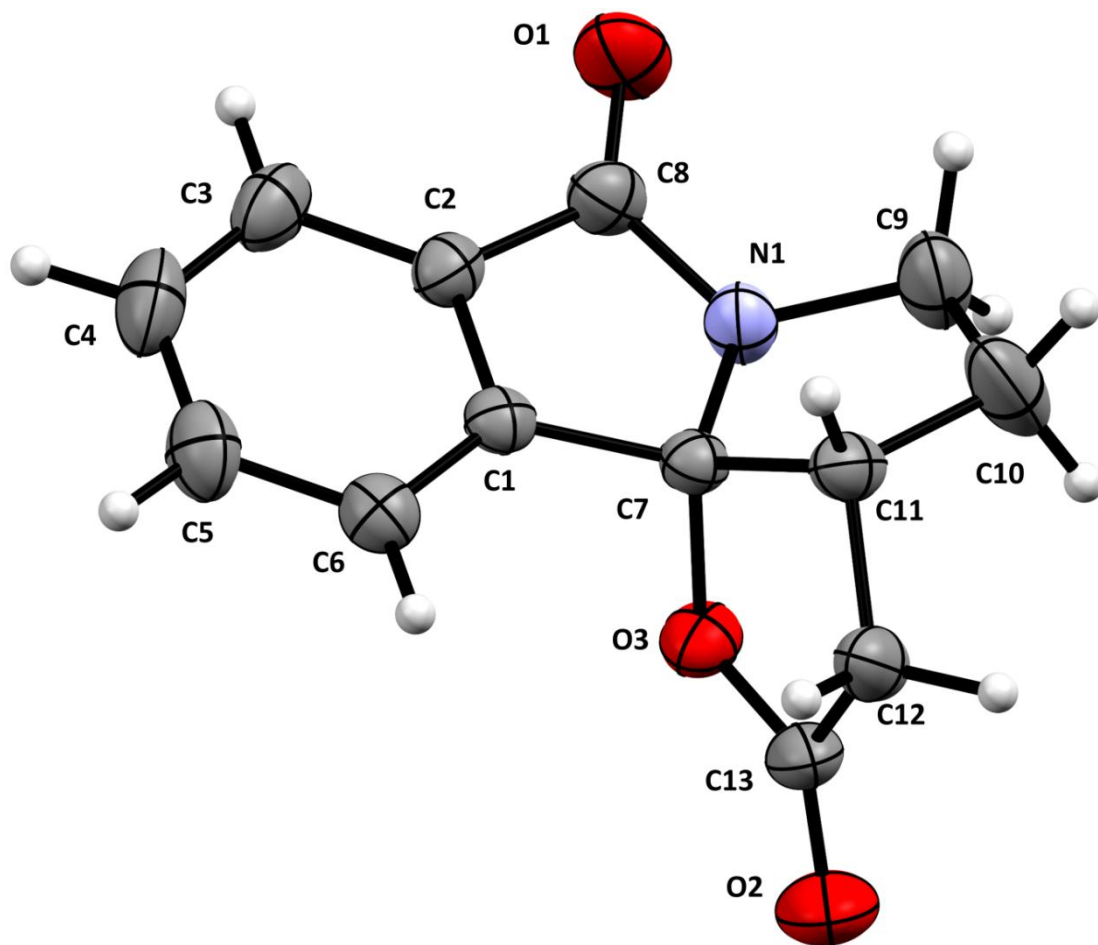
|   | CCDC 2430245                                      |
|---|---|
| Formula   | C <sub>22</sub> H <sub>19</sub> NO <sub>3</sub> S |
| Formula weight                                      | 377.44  |
| <i>T</i> /K   | 297(2)  |
| Crystal system                                      | Triclinic   |
| Space group   | P -1  |
| <i>a</i> /Å   | 9.267(2)  |
| <i>b</i> /Å   | 9.645(2)  |
| <i>c</i> /Å   | 12.292(3)   |
| $\alpha$ /°   | 75.762(5)   |
| $\beta$ /°  | 69.530(5)   |
| $\gamma$ /°   | 65.948(5)   |
| <i>V</i> /Å <sup>3</sup>                            | 933.1(4)  |
| <i>Z</i>  | 2   |
| Abs. Coeff./mm <sup>-1</sup>                        | 0.196   |
| Abs. Correction                                     | none  |
| GOF on <i>F</i> <sup>2</sup>                        | 1.553   |
| Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )] | <i>RI</i> = 0.0601<br><i>wR2</i> = 0.1982         |
| <i>R</i> indices [all data]                         | <i>RI</i> = 0.0736<br><i>wR2</i> = 0.2137         |



**Fig S1.** ORTEP diagram of compound **3a**, thermal ellipsoids are drawn on 30% probability level.

**Table S2.** The crystal parameters of compound **4a**

|   | CCDC 2430246                                    |
|---|---|
| Formula   | C <sub>13</sub> H <sub>11</sub> NO <sub>3</sub> |
| Formula weight                                      | 229.23  |
| <i>T</i> /K   | 294(2)  |
| Crystal system                                      | Monoclinic                                      |
| Space group   | P 1 21/c 1                                      |
| <i>a</i> /Å   | 7.5670(6)                                       |
| <i>b</i> /Å   | 10.9049(9)                                      |
| <i>c</i> /Å   | 13.5634(12)                                     |
| $\alpha$ /°   | 90.00   |
| $\beta$ /°  | 103.996(2)                                      |
| $\gamma$ /°   | 90.00   |
| <i>V</i> /Å <sup>3</sup>                            | 1085.99(16)                                     |
| <i>Z</i>  | 4   |
| Abs. Coeff./mm <sup>-1</sup>                        | 0.101   |
| Abs. Correction                                     | None  |
| GOF on <i>F</i> <sup>2</sup>                        | 1.091   |
| Final <i>R</i> indices [ <i>I</i> > 2σ( <i>I</i> )] | <i>R</i> 1 = 0.0769<br><i>wR</i> 2 = 0.1666     |
| <i>R</i> indices [all data]                         | <i>R</i> 1 = 0.1192<br><i>wR</i> 2 = 0.1935     |



**Fig S2.** ORTEP diagram of compound **4a**, thermal ellipsoids are drawn on 30% probability level.