

## A solvent- and catalyst-free domino reaction for the efficient synthesis of 3-aryltiazolidine-2-thiones under microwave irradiation

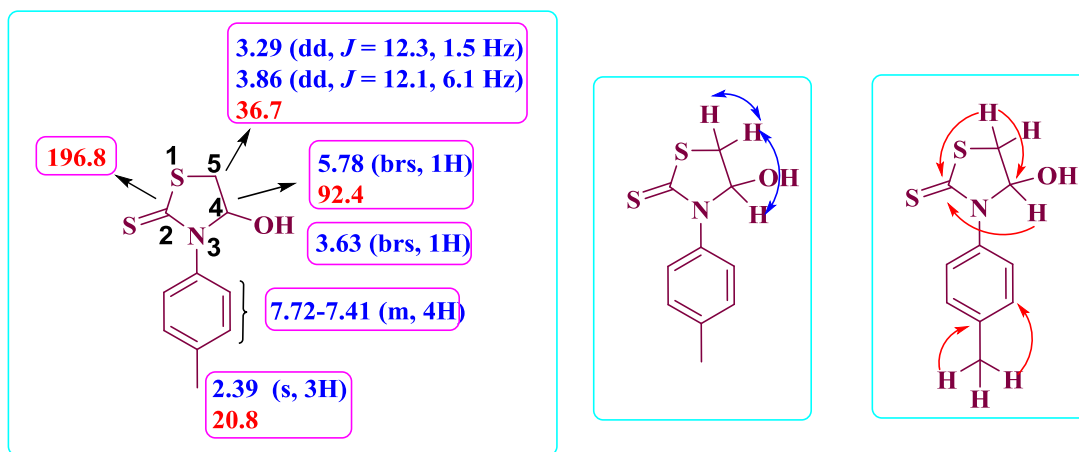
Sundaravel Vivek Kumar, Shanmugam Muthusubramanian and Subbu Perumal\*

Department of Organic Chemistry, School of Chemistry, Madurai Kamaraj University,  
Madurai – 625021, Tamil Nadu, India

### Supporting Information

#### Structure determination using NMR spectroscopic data of **3e**

The structure of 4-hydroxy-3-(*p*-tolyl)thiazolidine-2-thione **3e** was deduced from one- and two-dimensional NMR spectroscopic data. The structural elucidation using NMR spectroscopy is discussed below.



In the  $^1\text{H}$  NMR spectrum of **3e**, the H-5 hydrogens appear as a broad singlet 5.78 ppm and it shows a C-H COSY correlation with the carbon signal at 92.4 ppm. Further, the H-5 have a H-H COCY correlation with a hydrogen at 3.86 ppm ( $J = 12.1, 6.1$  Hz) assignable to H-4a. Moreover, the H-5 protons show a HMB correlation with C-2 at 196.8 ppm. The diastereotopic protons, H-4 appear as doublets of doublets at 3.86 ppm ( $J = 12.1, 6.1$  Hz) and 3.29 ( $J = 12.3, 1.5$  Hz) which show HMBCs with C-2 and C-4 at 196.8 and 92.4 ppm respectively. The H-H COSY correlation reveals that one of the diastereotopic H-4a protons couples with H-5 proton and the protons, H-

4a and H-4b couple with each other. The hydrogens, H-4a and H-4b show a C-H COSY correlation with the carbon signal at 36.7 ppm.

The aromatic protons appear as a multiplet at 7.72-7.41 ppm. The methyl hydrogens appear as a singlet at 2.39 ppm which show HMBs with C-3' and C-4' at 127.8 and 136.7 ppm respectively. The hemiaminalic OH appears as a broad singlet at 3.63 ppm. The >C=S carbon appears at 196.8 ppm, which is a characteristic carbon signal for the 4-hydroxy-3-arylthiazolidine-2-thione system. Finally the structure of the product was also confirmed by ESI mass analysis.

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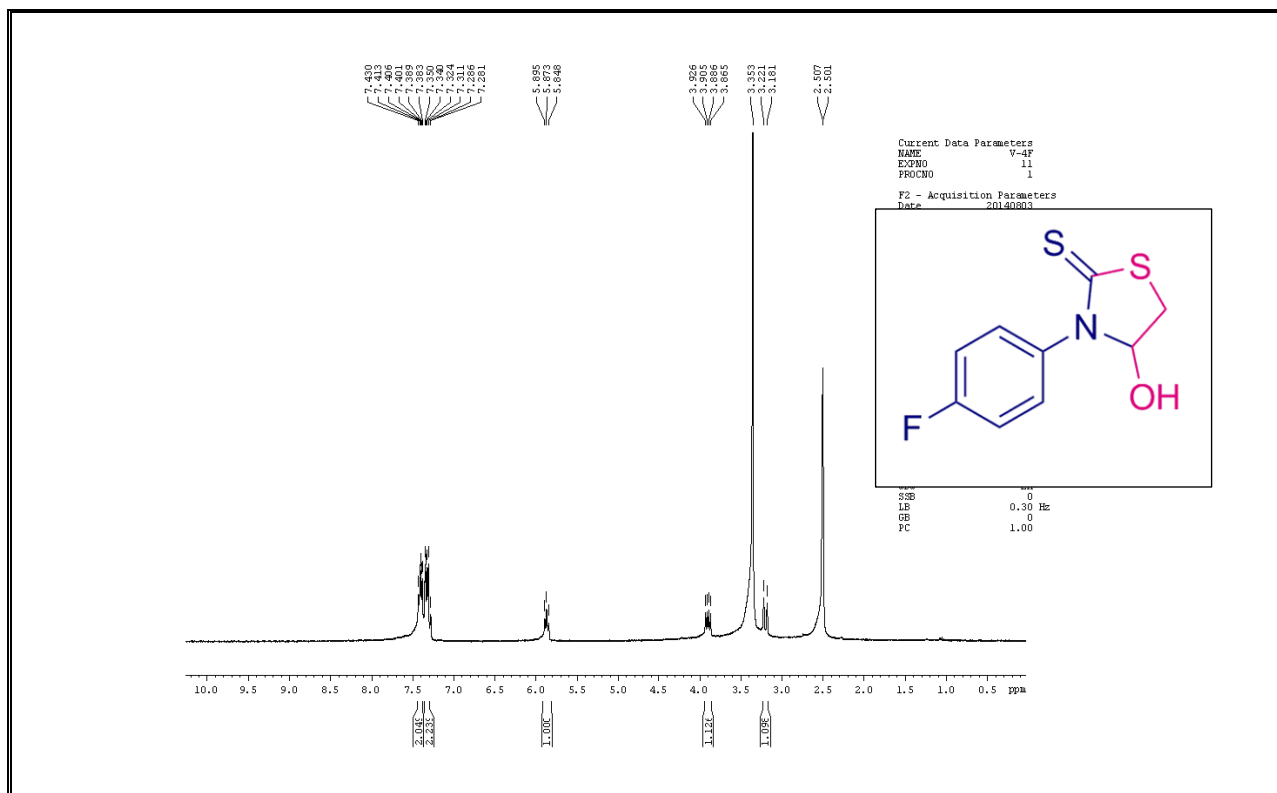


Fig.1 <sup>1</sup>H NMR Spectrum 3a(DMSO-d<sub>6</sub>)

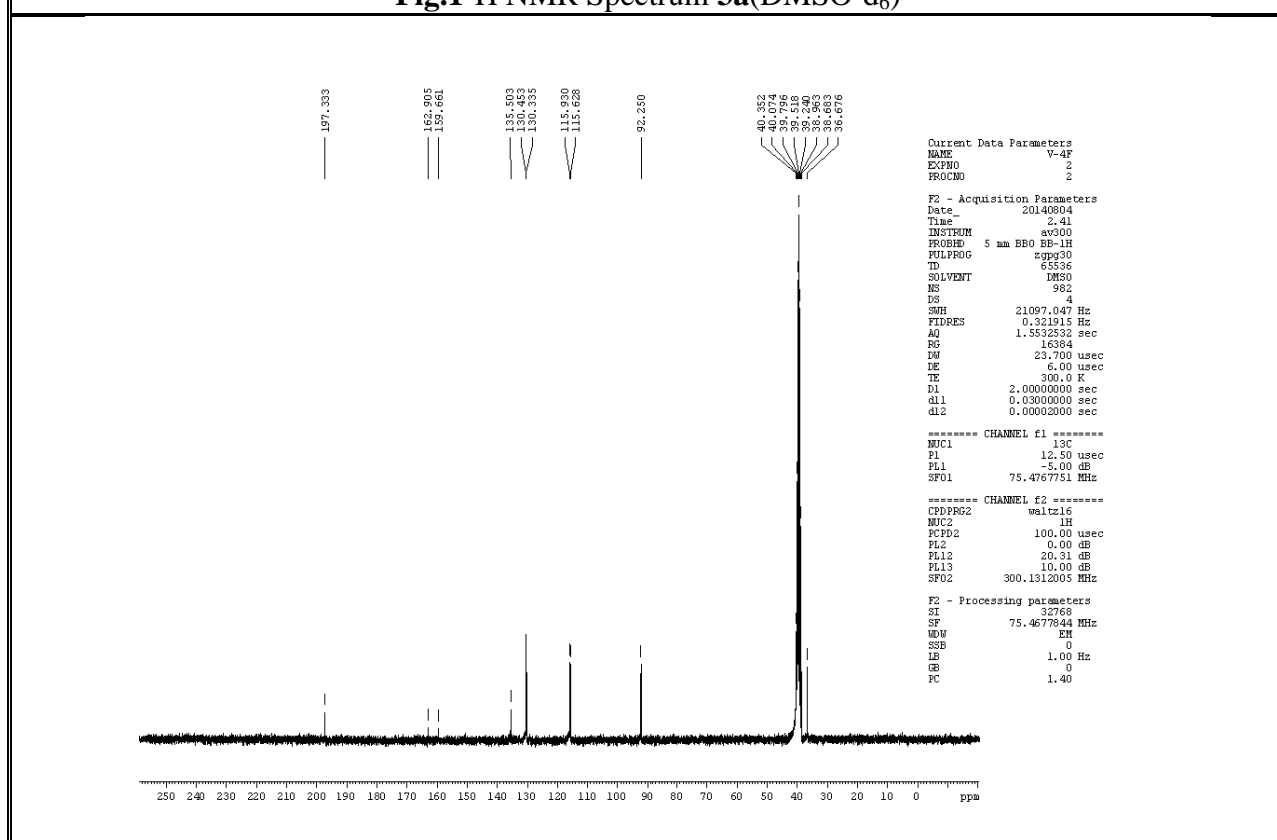


Fig.2 <sup>13</sup>C NMR Spectrum of 3a(DMSO-d<sub>6</sub>)

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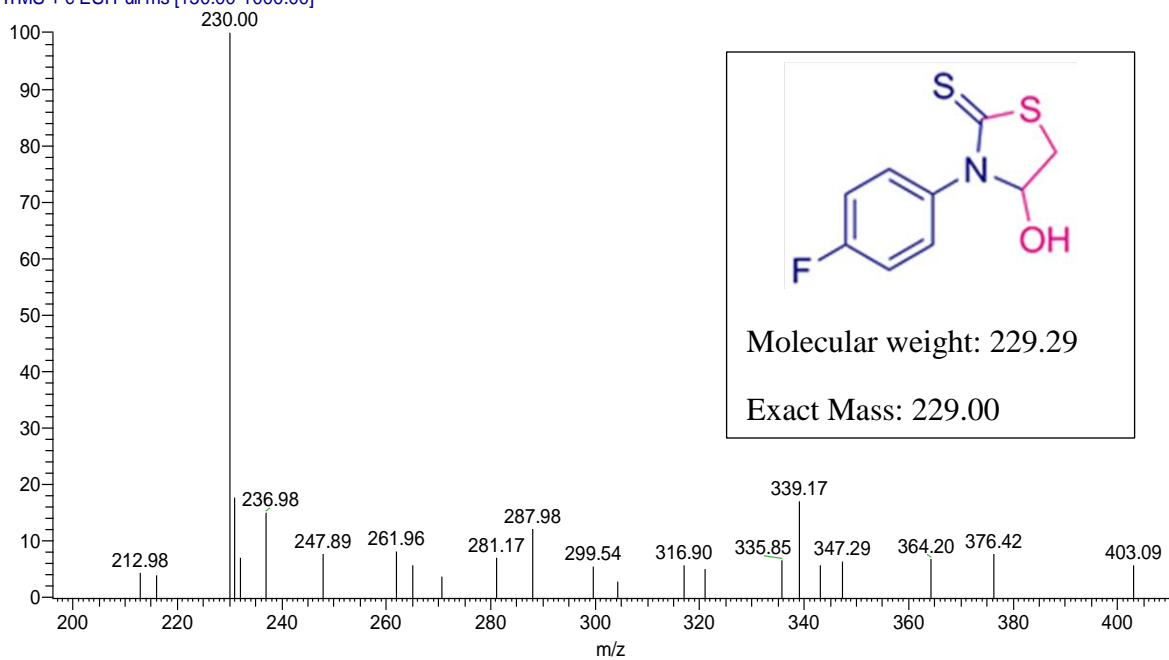


Fig.3 ESI mass spectrum of 3a

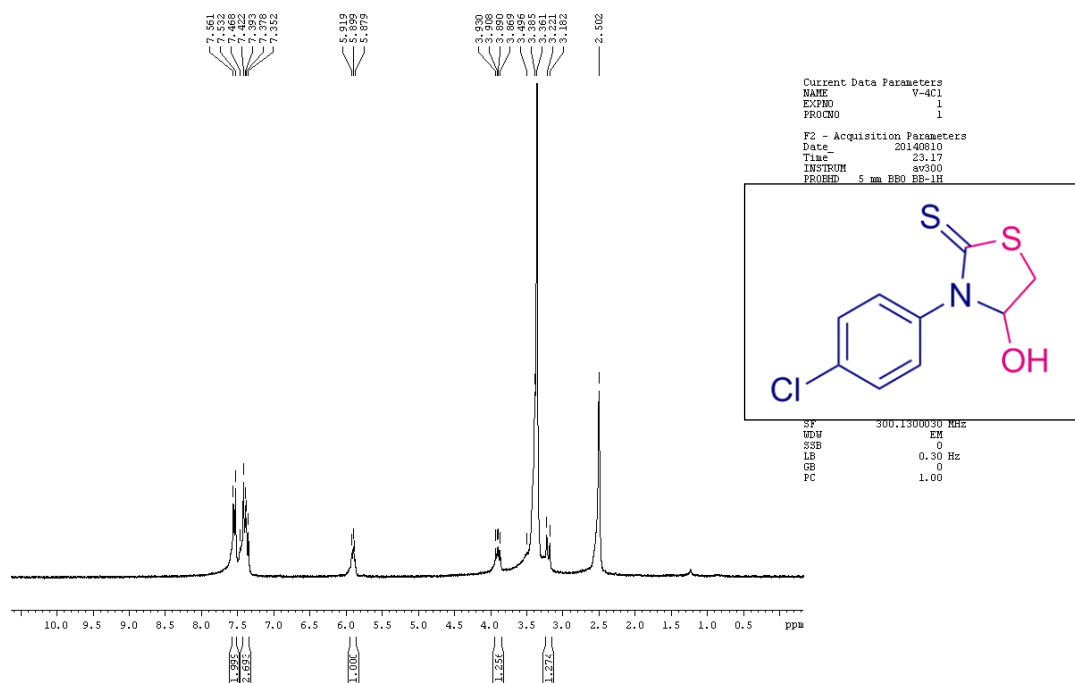
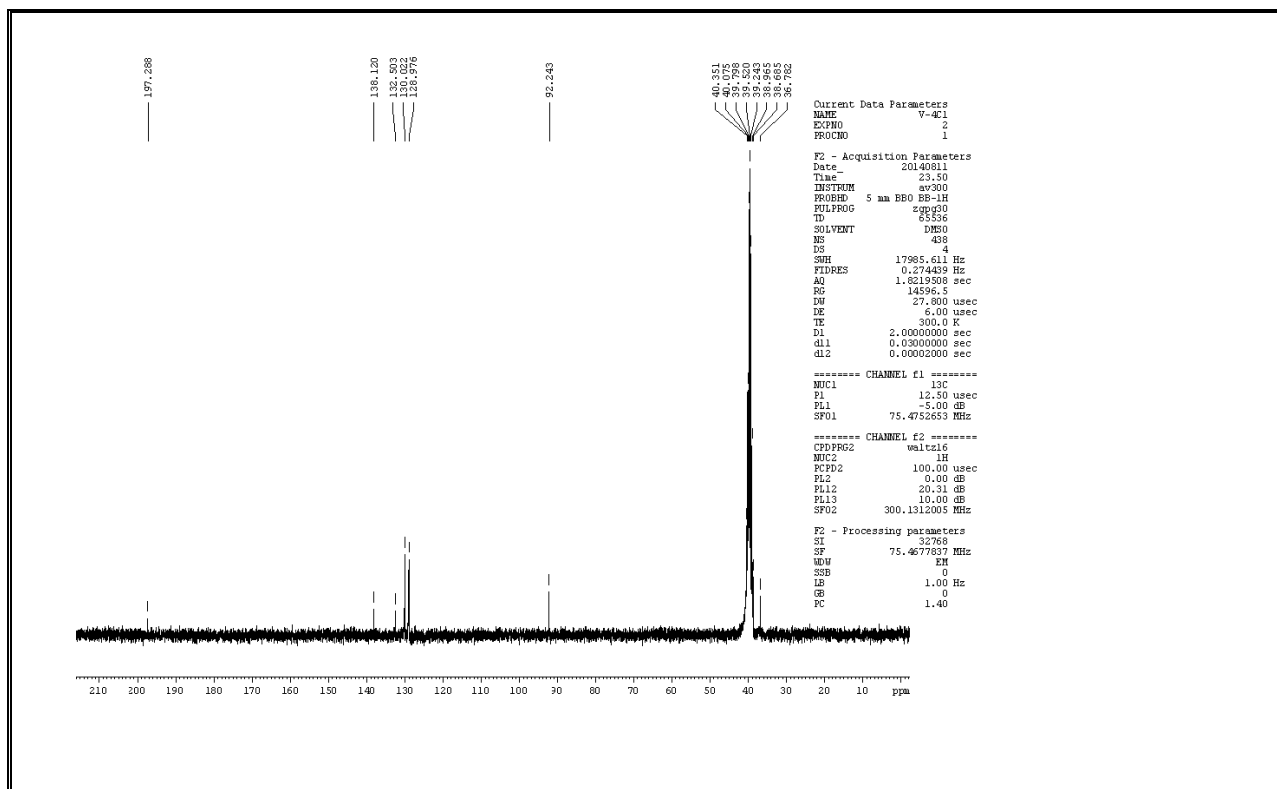
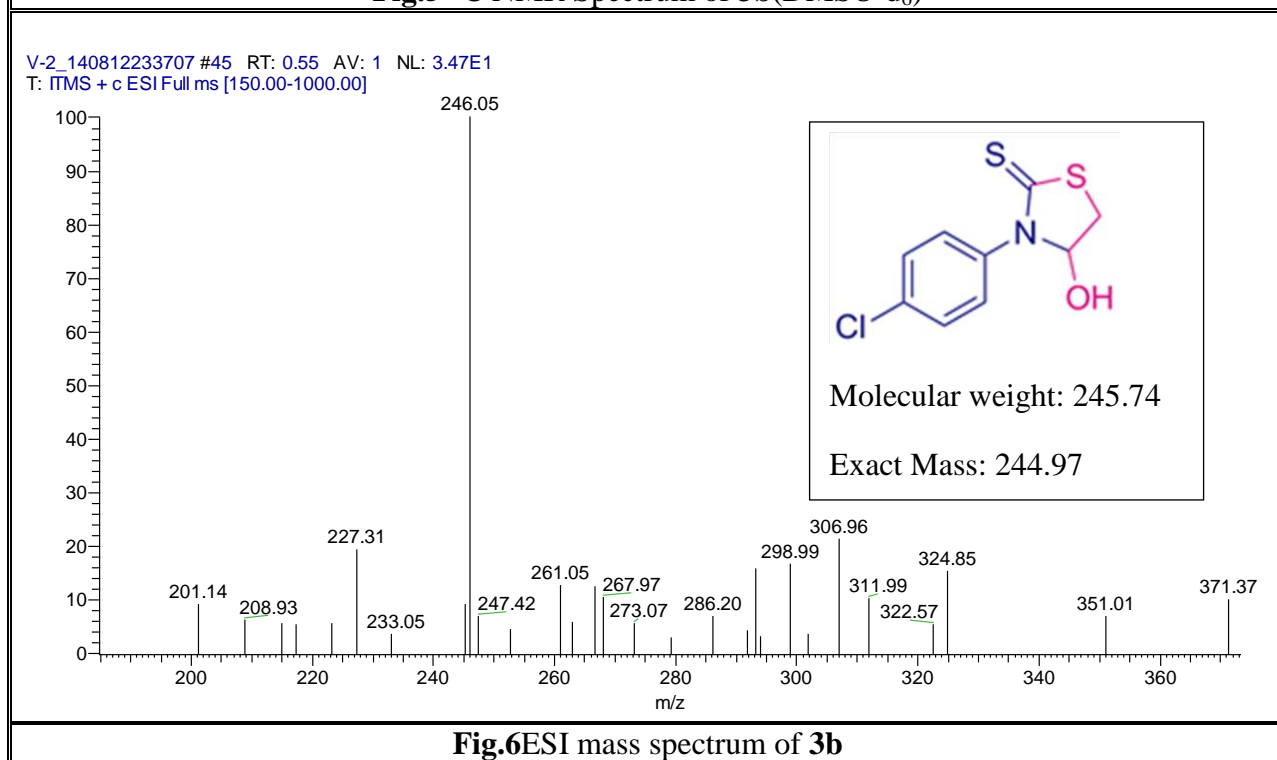


Fig.4 <sup>1</sup>H NMR Spectrum 3b(DMSO-d<sub>6</sub>)



**Fig.5**  $^{13}\text{C}$  NMR Spectrum of **3b**(DMSO- $d_6$ )



**Fig.6** ESI mass spectrum of **3b**



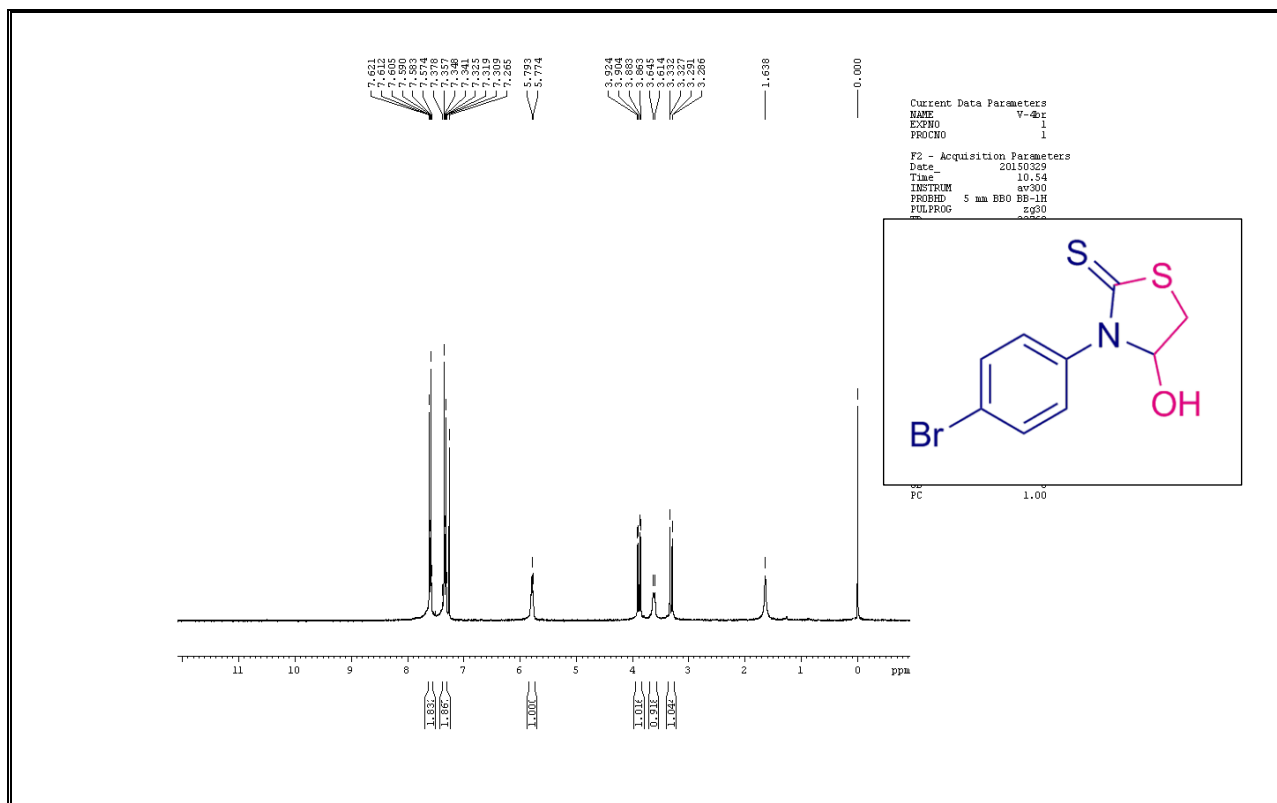


Fig.7  $^1\text{H}$  NMR Spectrum **3c**( $\text{CDCl}_3$ )

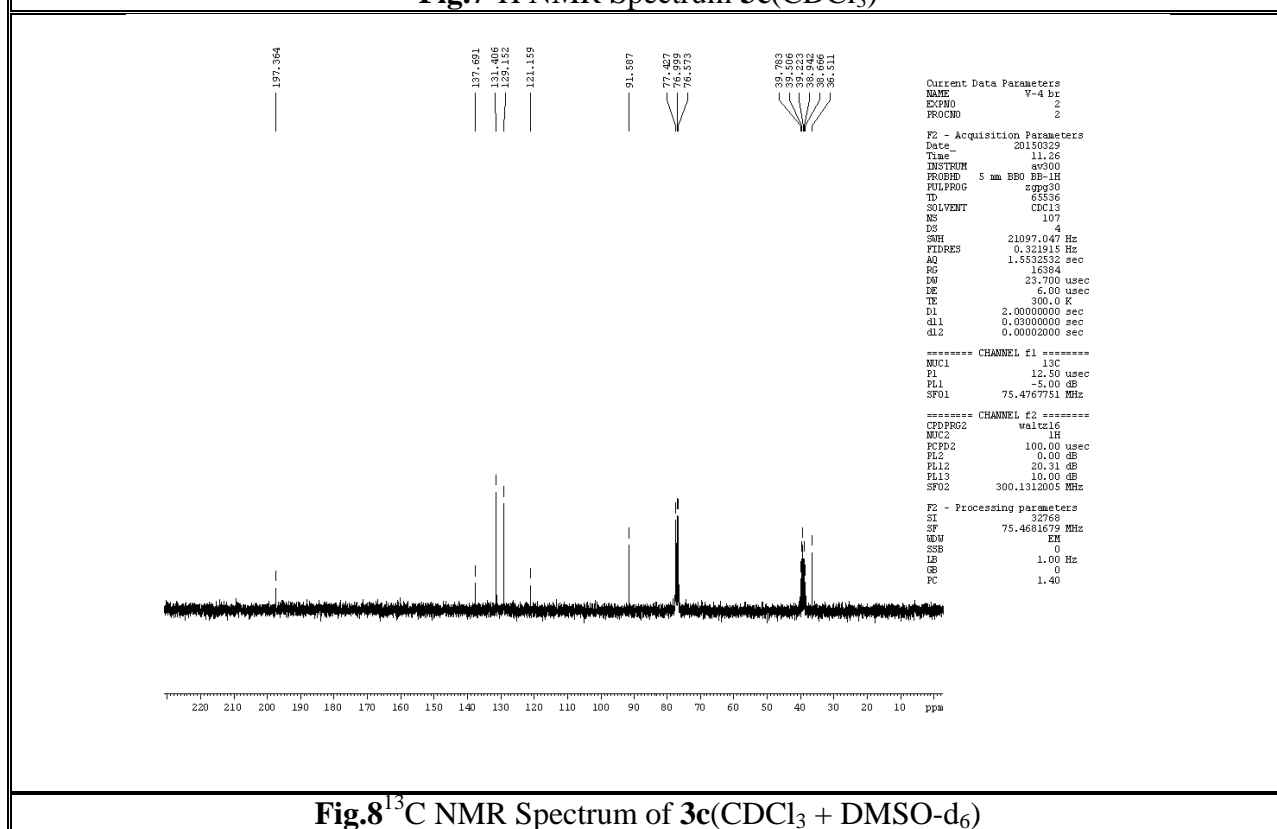


Fig.8  $^{13}\text{C}$  NMR Spectrum of **3c**( $\text{CDCl}_3 + \text{DMSO-d}_6$ )

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T: ITMS - c ESI Full ms [50.00-1000.00]

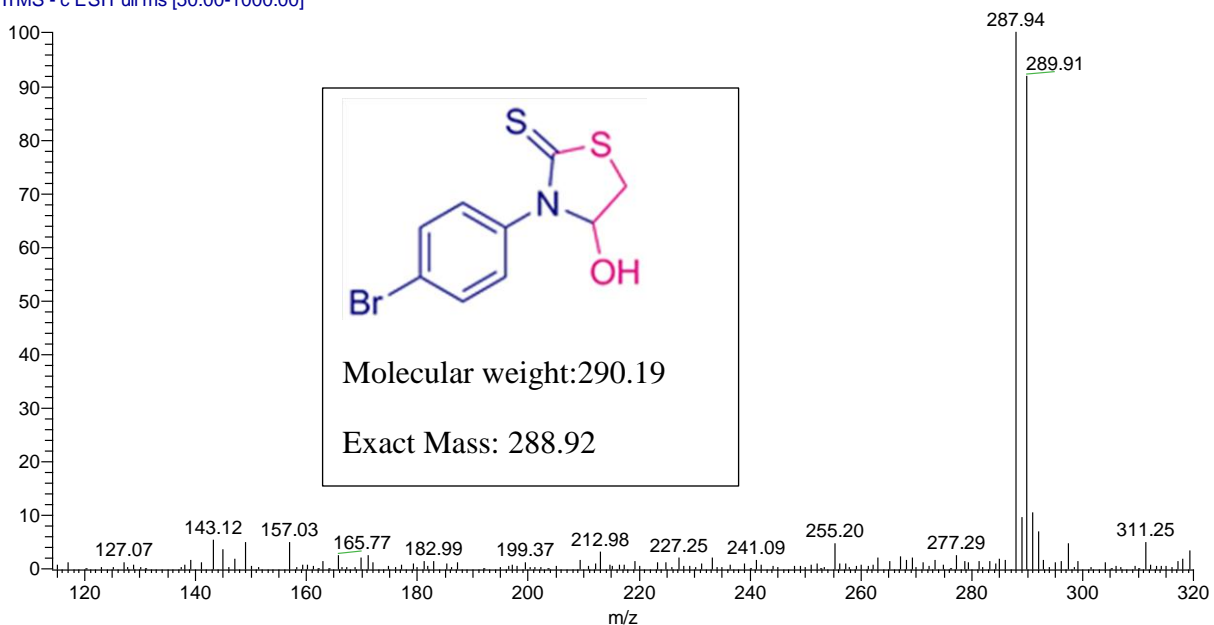


Fig.9 ESI mass spectrum of 3c

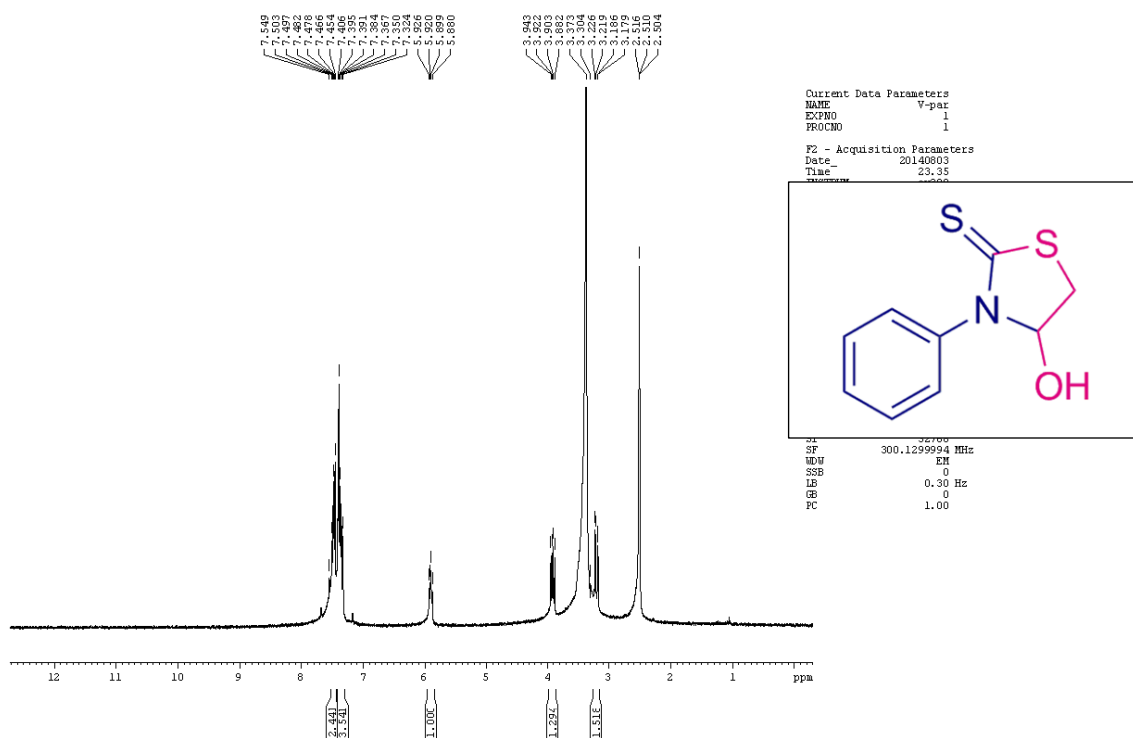
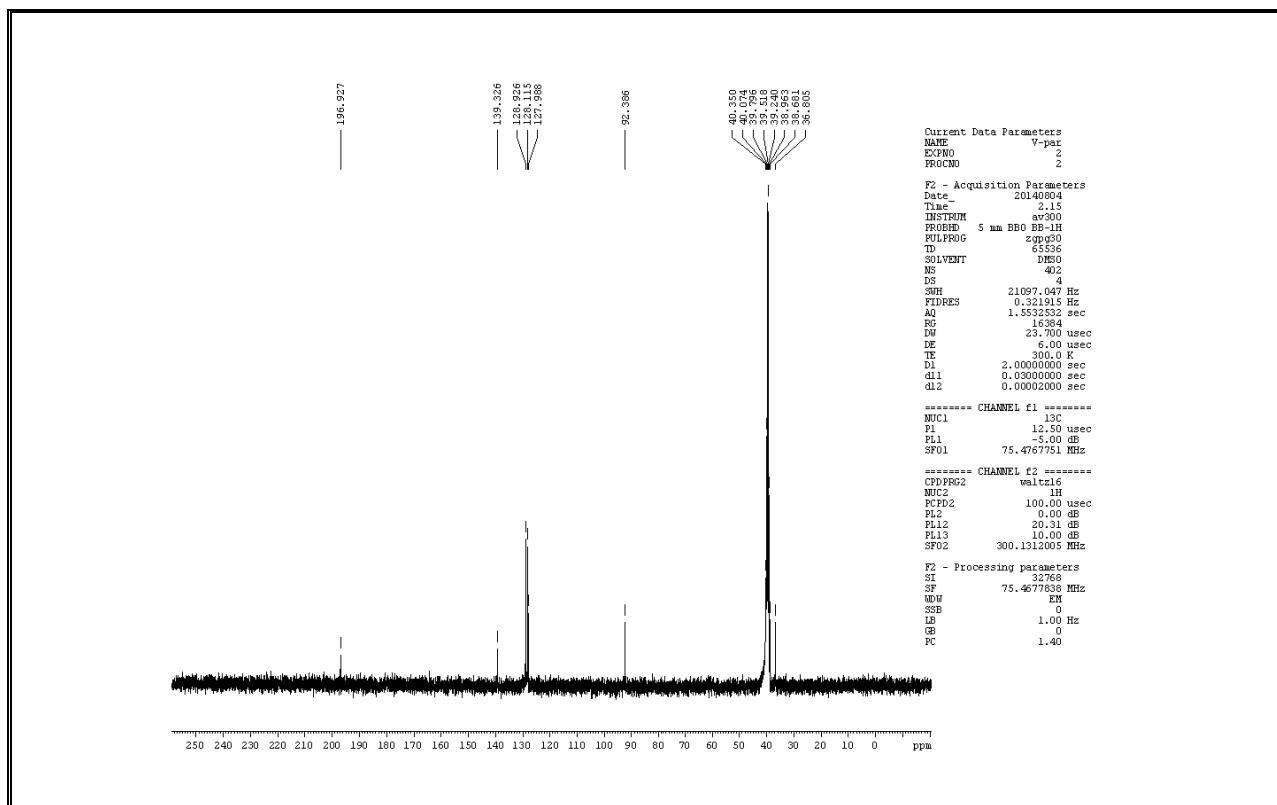
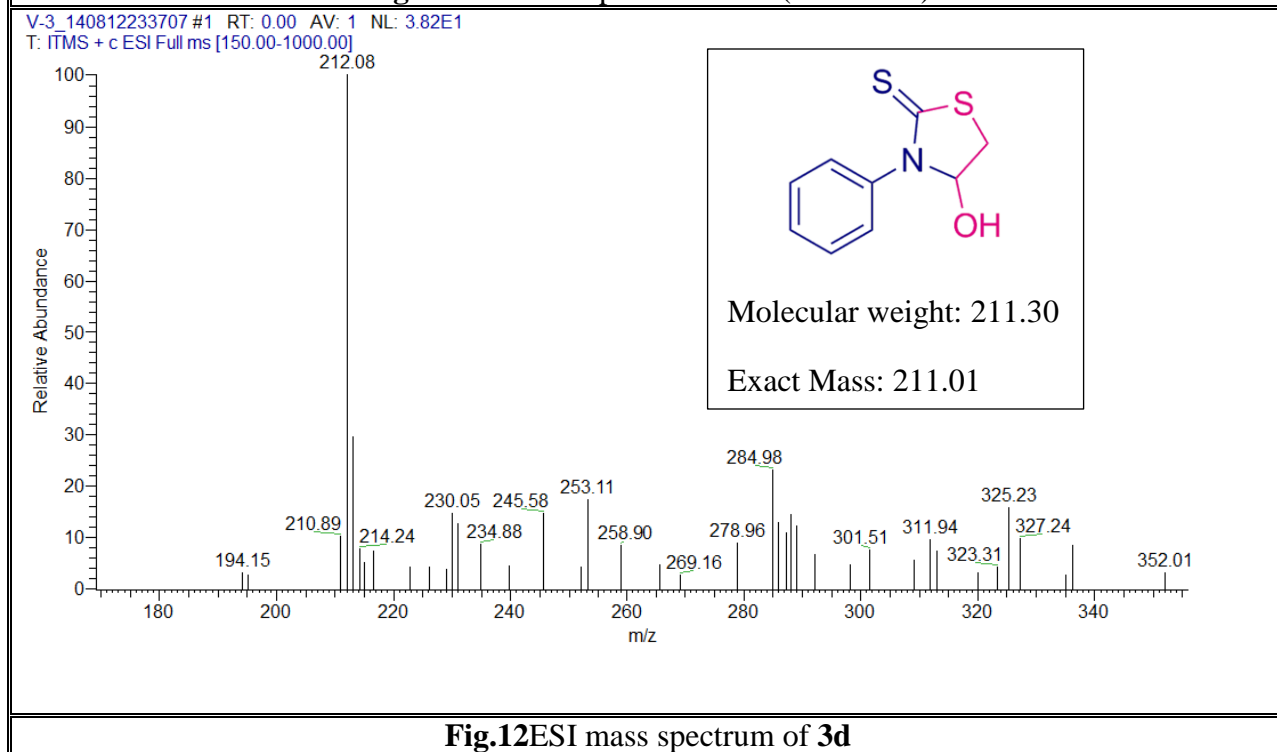


Fig.10 <sup>1</sup>H NMR Spectrum 3d(DMSO-d<sub>6</sub>)



**Fig.11**  $^{13}\text{C}$  NMR Spectrum of **3d**(DMSO- $\text{d}^6$ )



**Fig.12**ESI mass spectrum of **3d**

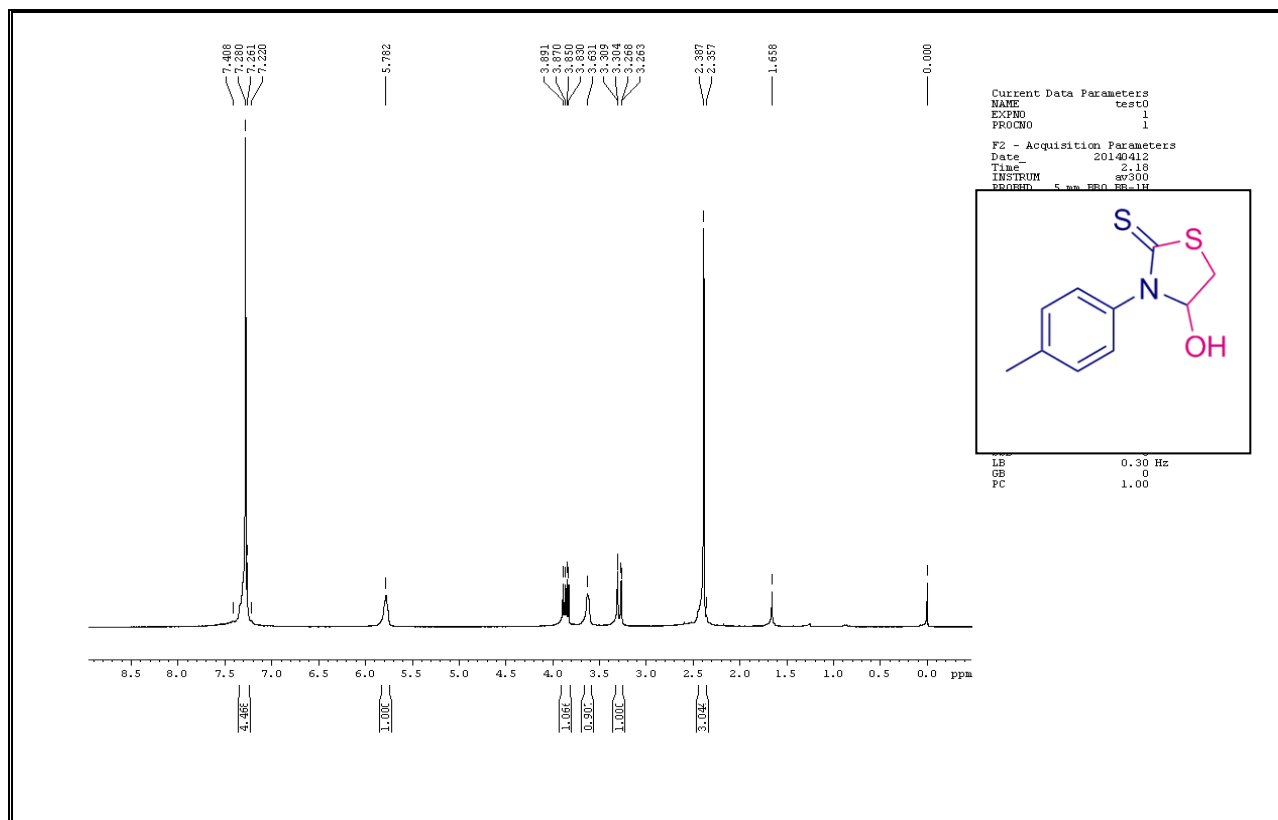


Fig.13  $^1\text{H}$  NMR Spectrum **3e**( $\text{CDCl}_3$ )

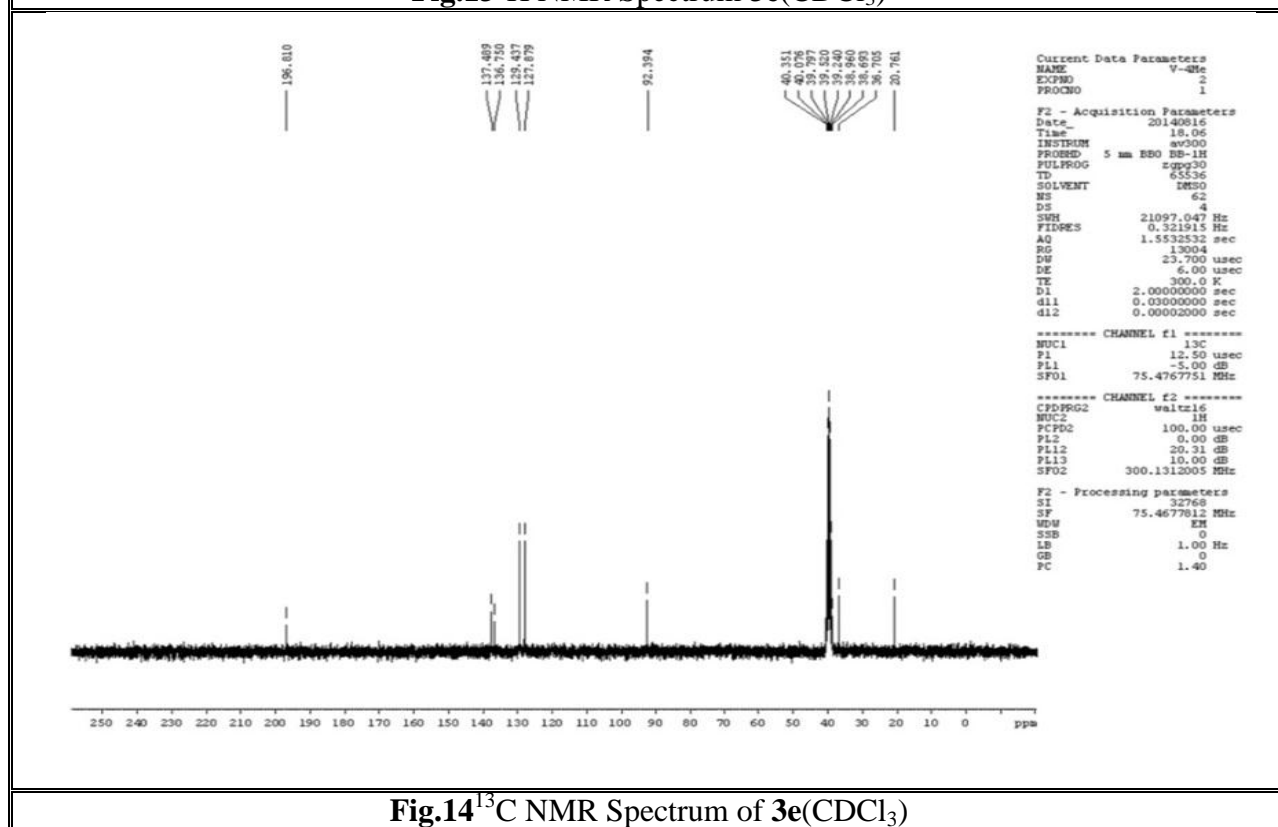


Fig.14  $^{13}\text{C}$  NMR Spectrum of **3e**( $\text{CDCl}_3$ )

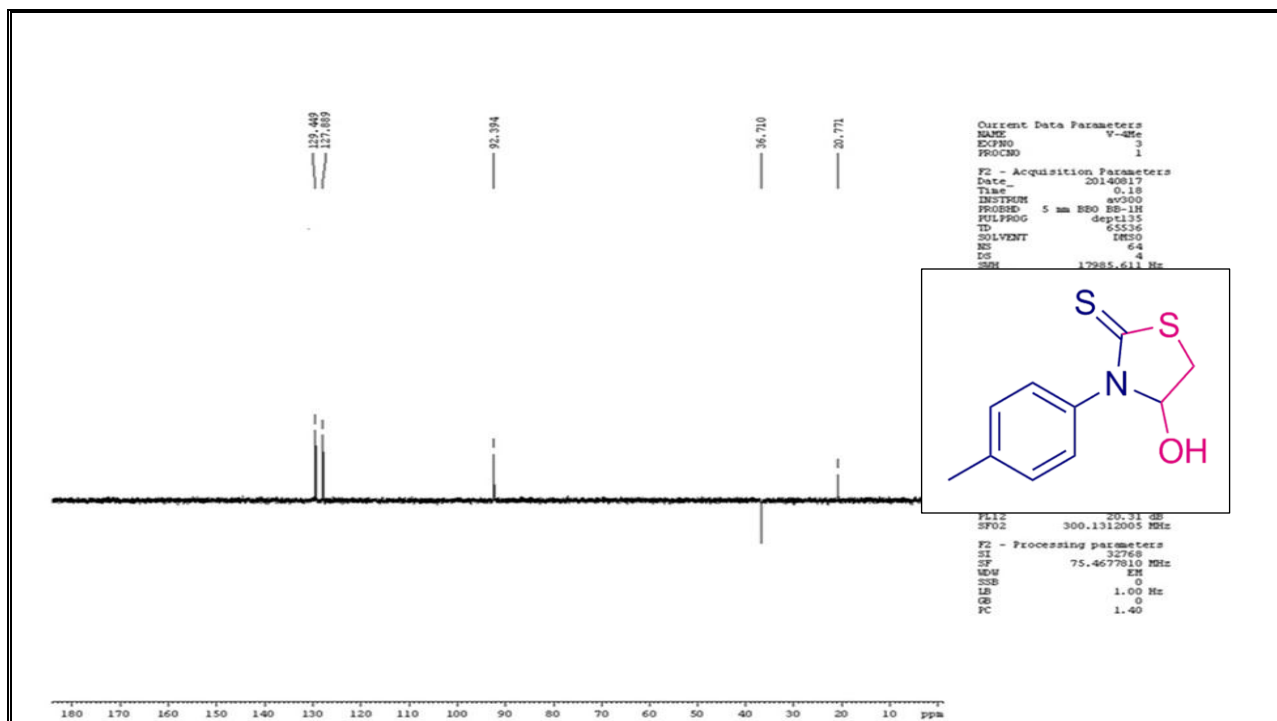


Fig.15 DEPT Spectrum of 3c (CDCl<sub>3</sub>)

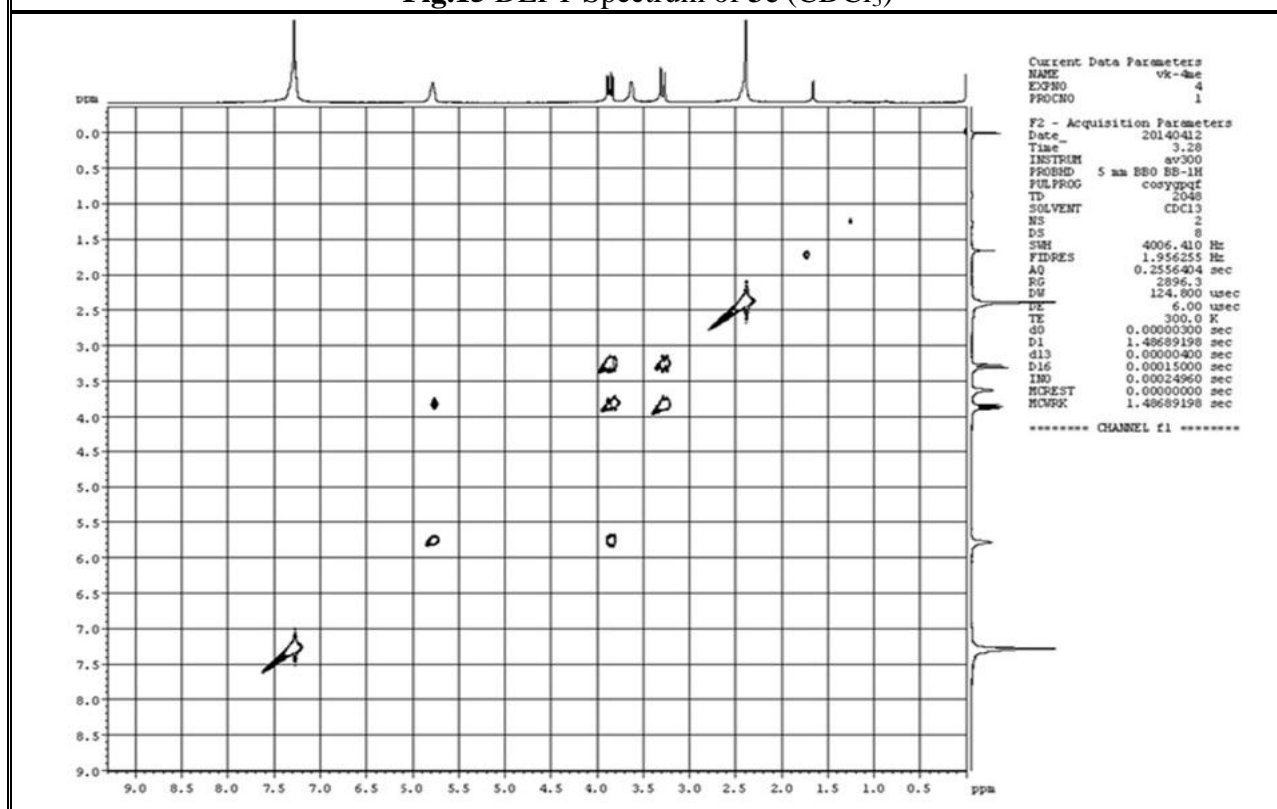


Fig.16 H, H-COSY Spectrum of 3c(CDCl<sub>3</sub>)

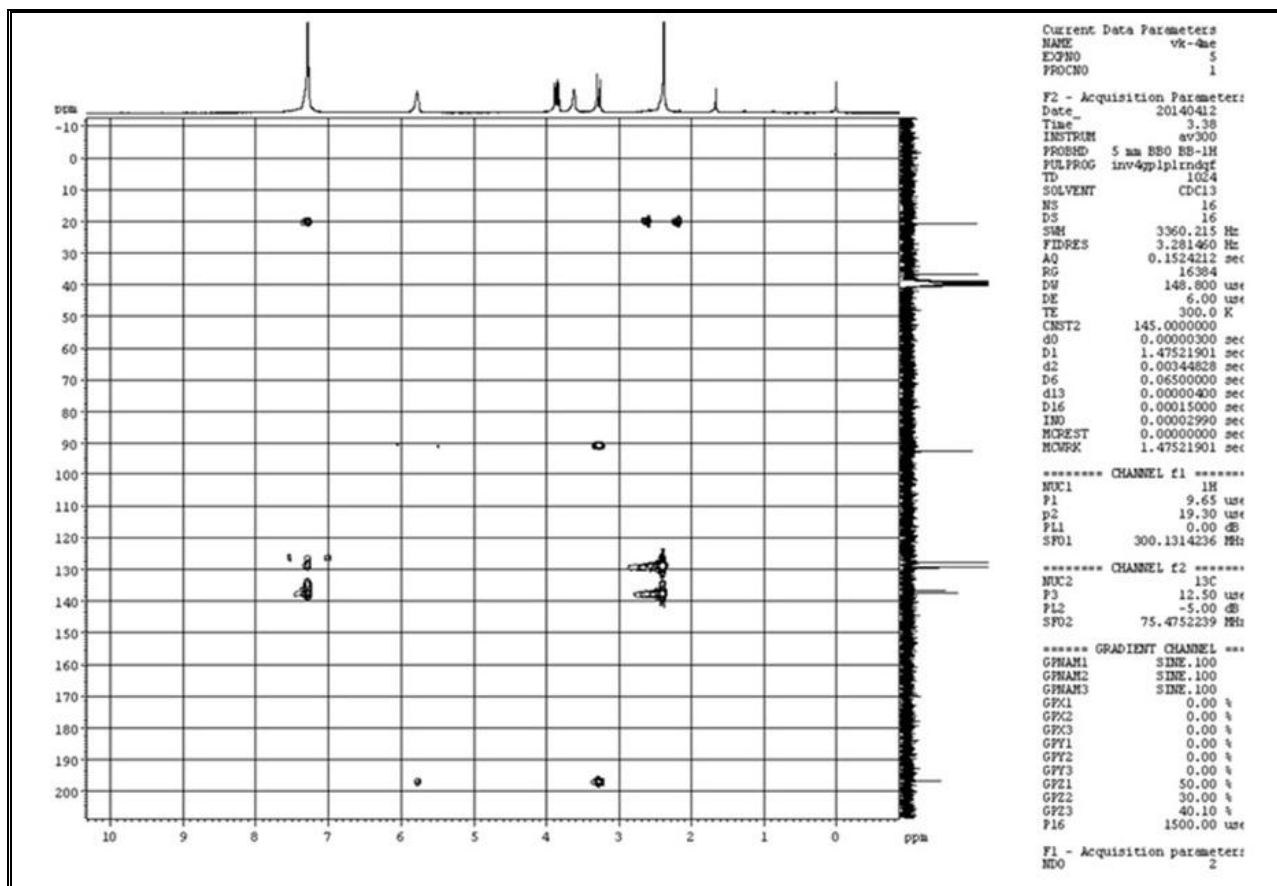


Fig.17 HMBC Spectrum of 3c (CDCl<sub>3</sub>)

V-4\_140812233707 #1 RT: 0.00 AV: 1 NL: 7.16E1  
T: ITMS + c ESI Full ms [150.00-1000.00]

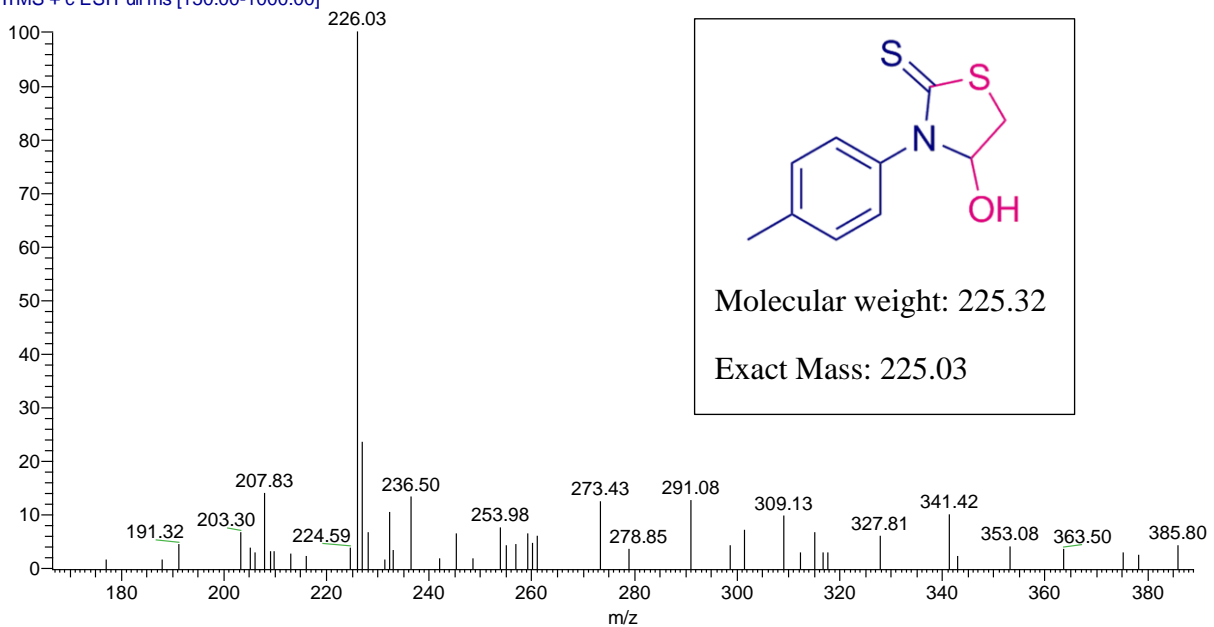


Fig.18ESI mass spectrum of 3e

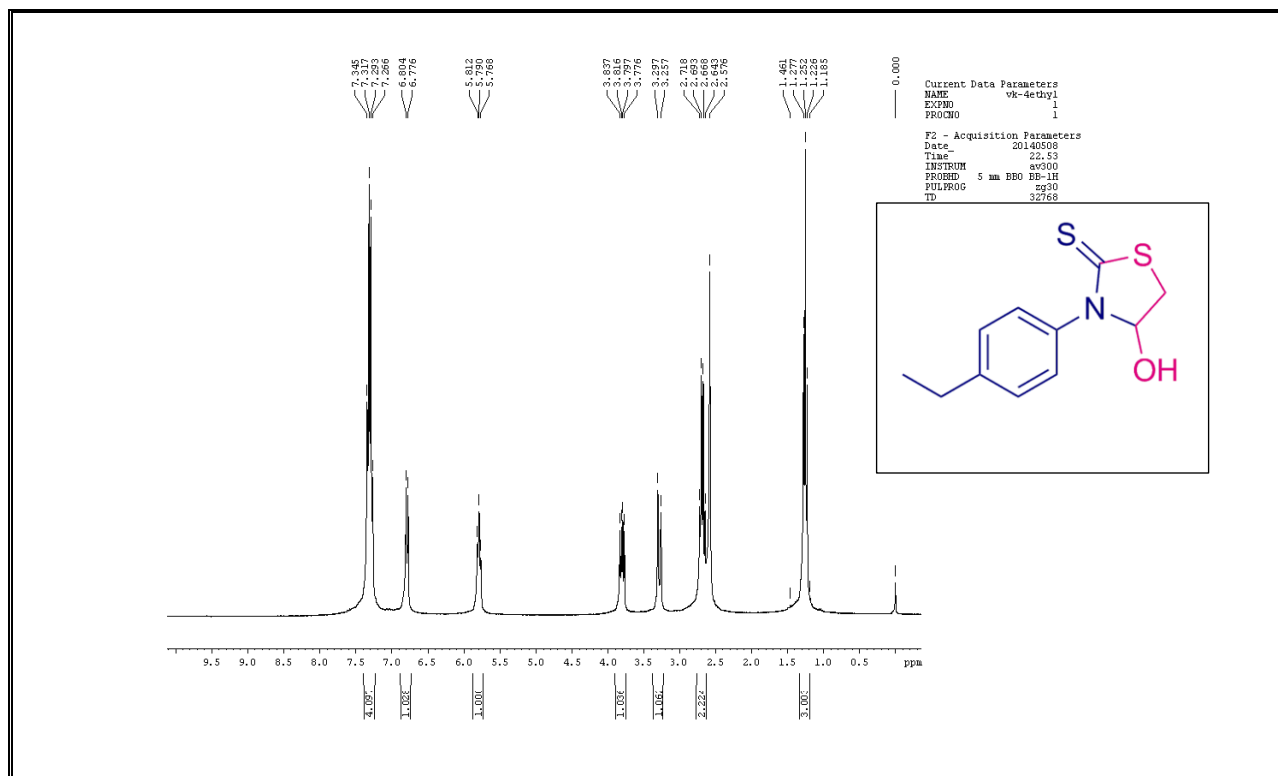


Fig.19  $^1\text{H}$  NMR Spectrum 3f(CDCl<sub>3</sub> + DMSO-d<sub>6</sub>)

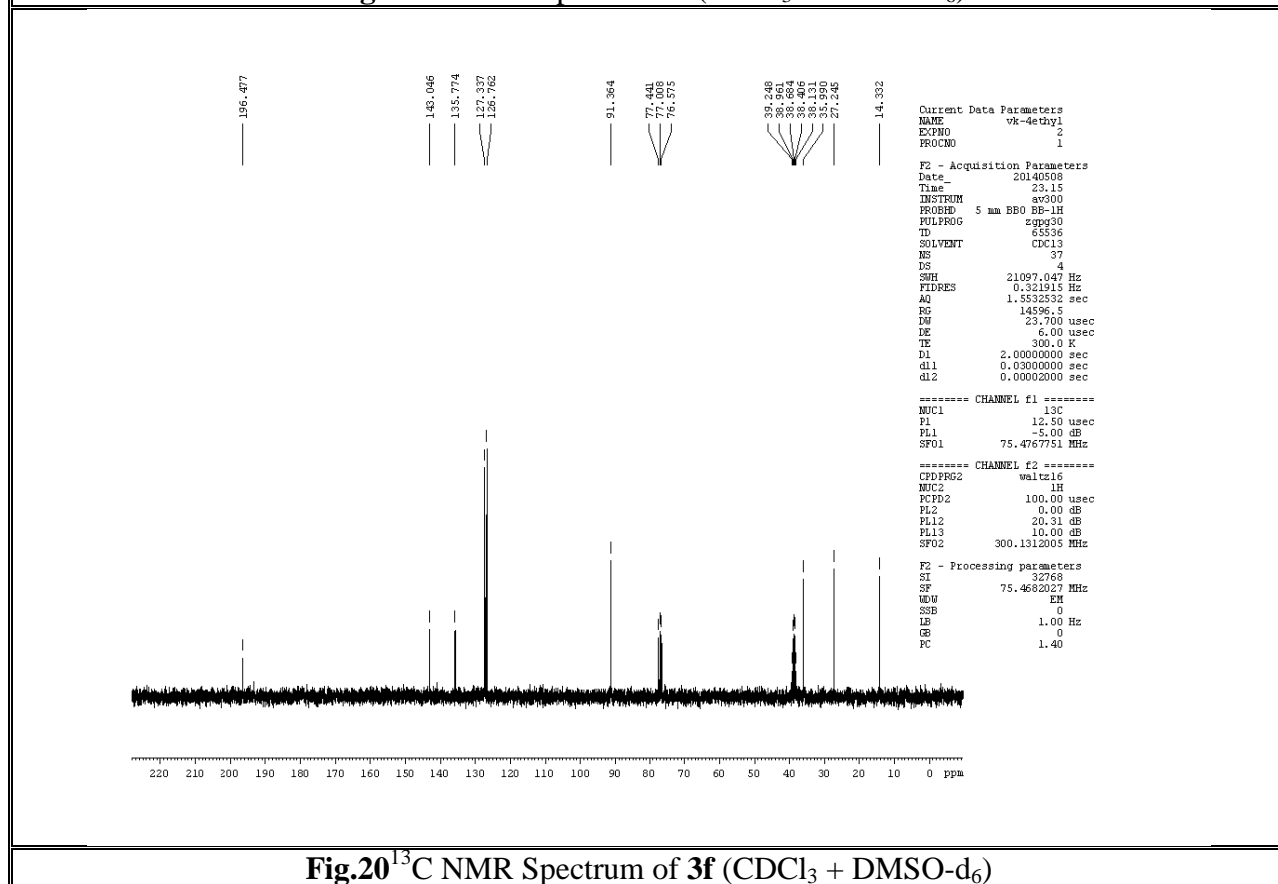


Fig.20  $^{13}\text{C}$  NMR Spectrum of 3f (CDCl<sub>3</sub> + DMSO-d<sub>6</sub>)

V-5\_140812233707 #1 RT: 0.00 AV: 1 NL: 1.59E2  
T: ITMS + c ESI Full ms [150.00-1000.00]

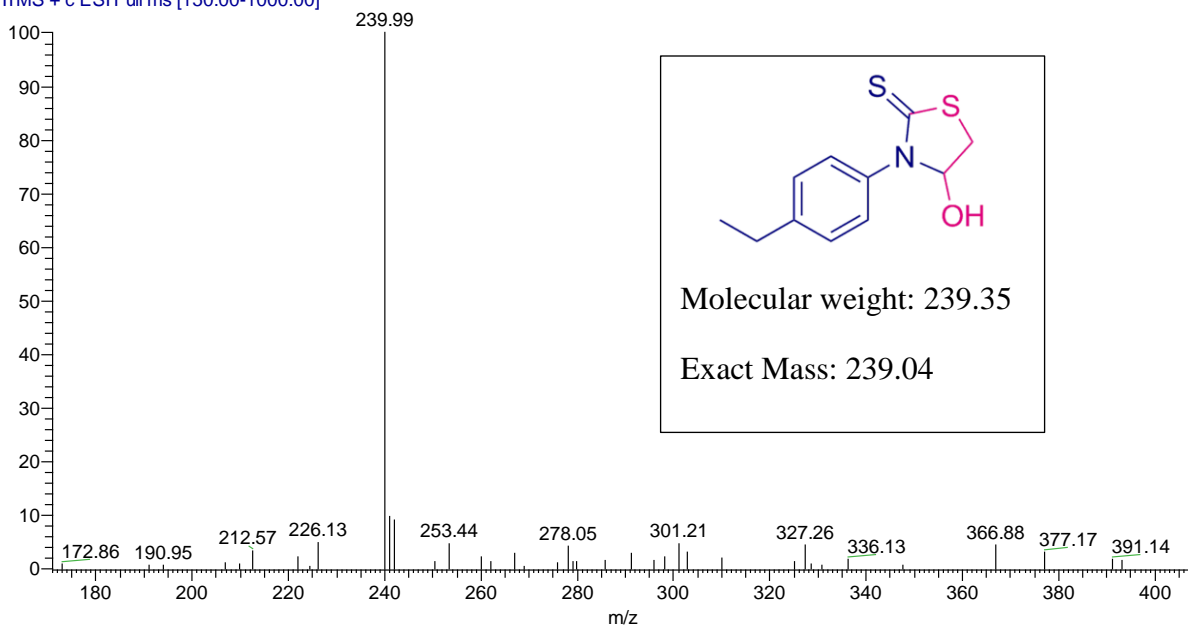


Fig.21 ESI mass spectrum of 3f

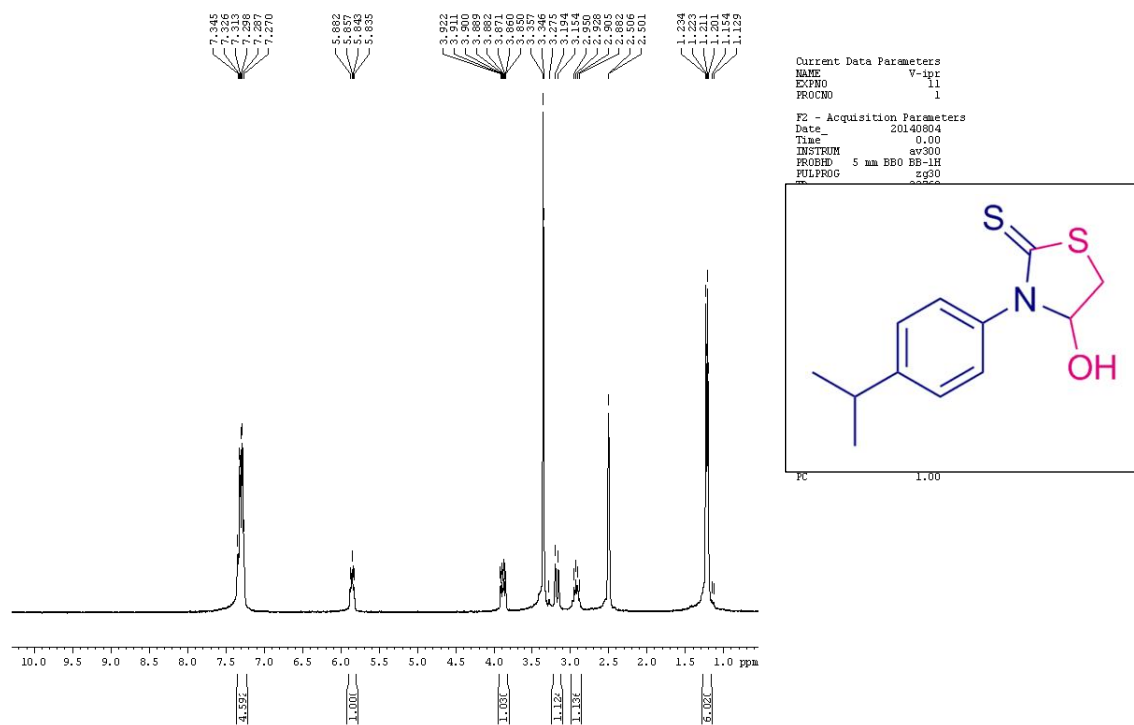
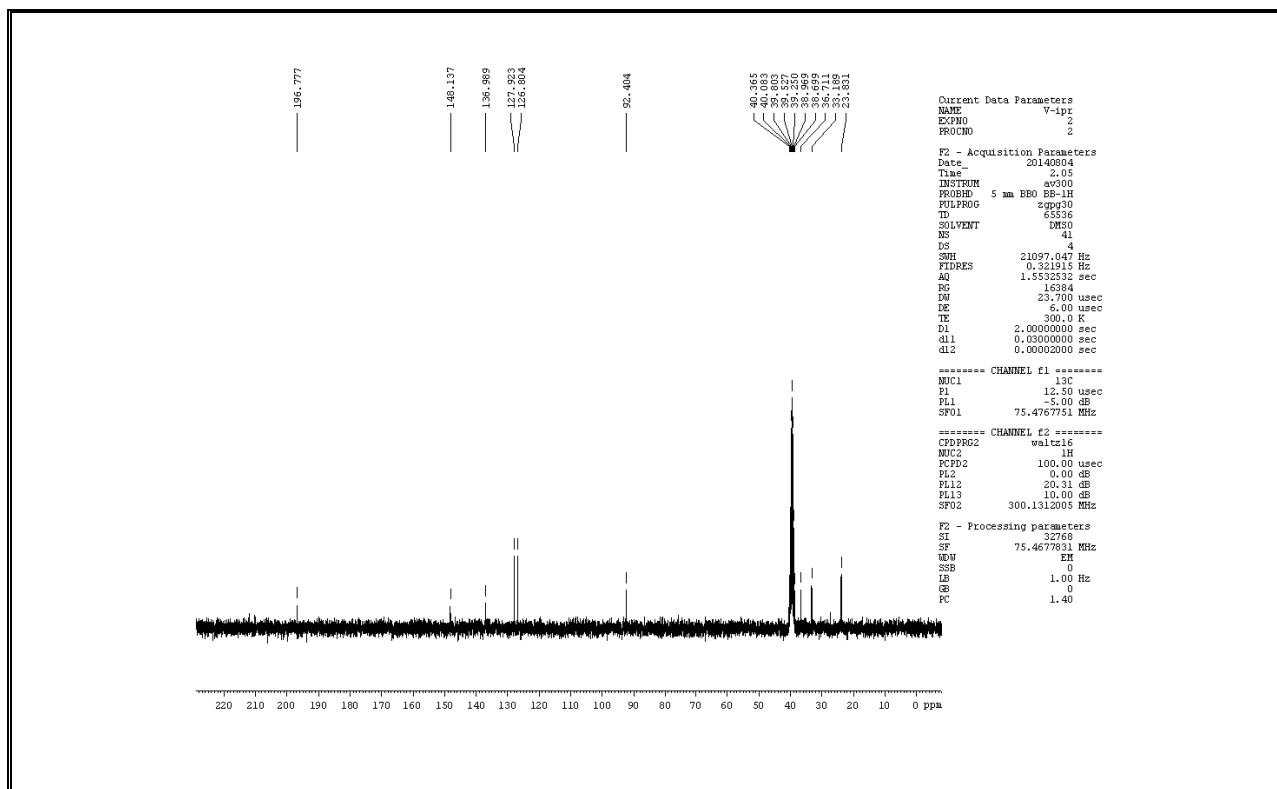


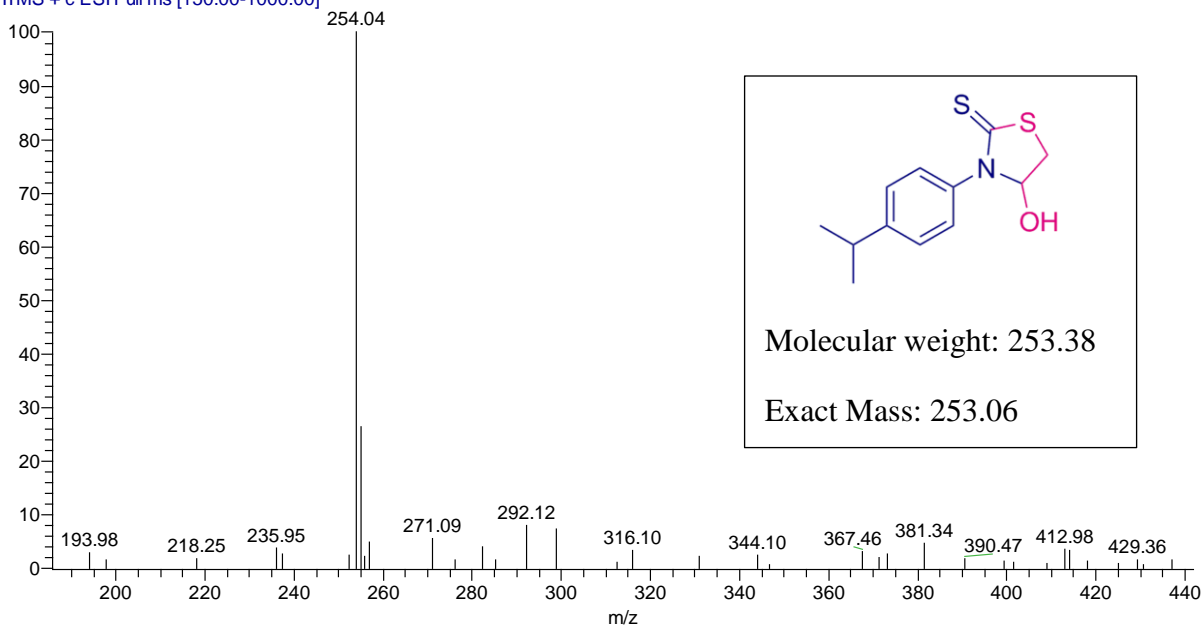
Fig.22 <sup>1</sup>H NMR Spectrum 3g (DMSO-d<sub>6</sub>)





**Fig.23**  $^{13}\text{C}$  NMR Spectrum of **3g** (DMSO- $d_6$ )

V-6\_140812233707 #39 RT: 0.48 AV: 1 NL: 1.39E2  
T: ITMS + c ESI Full ms [150.00-1000.00]



**Fig.24** ESI mass spectrum of **3g**

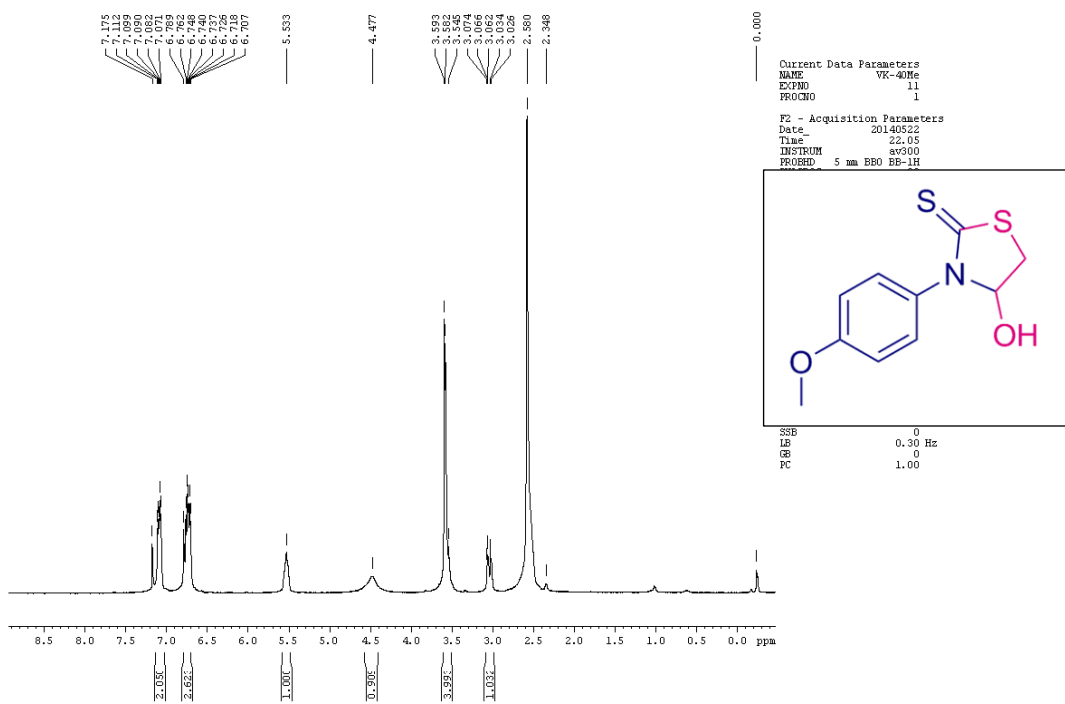


Fig.25  $^1\text{H}$  NMR Spectrum **3h** ( $\text{CDCl}_3 + \text{DMSO-d}_6$ )

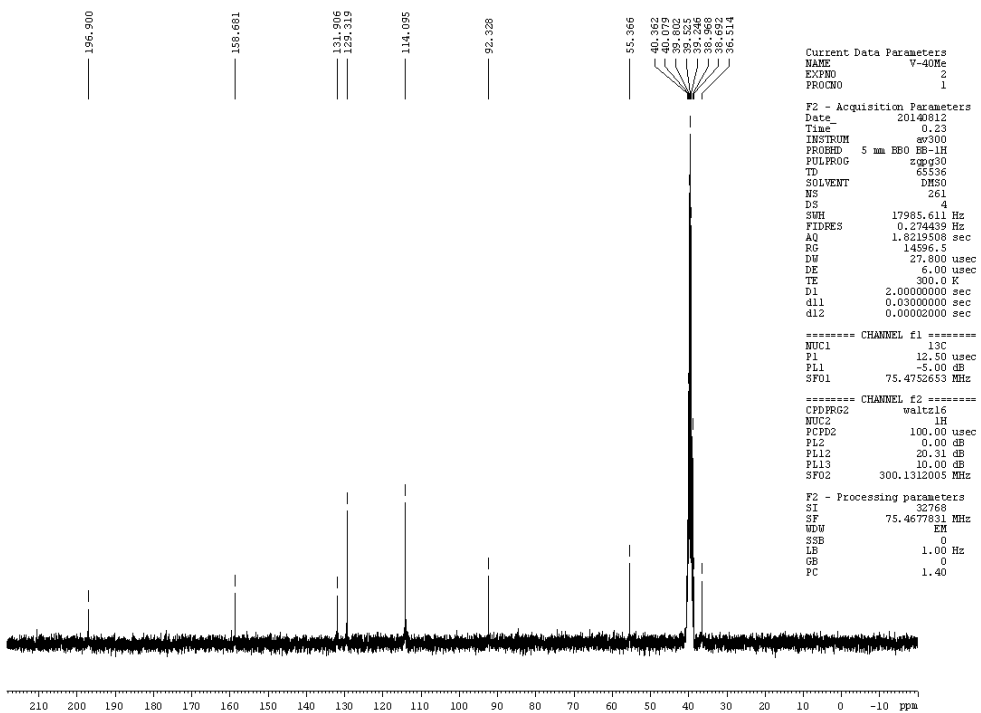


Fig.26  $^{13}\text{C}$  NMR Spectrum of **3h** ( $\text{DMSO-d}_6$ )

V-7\_140812233707 #32 RT: 0.39 AV: 1 NL: 3.87E1  
T: ITMS + c ESI Full ms [150.00-1000.00]

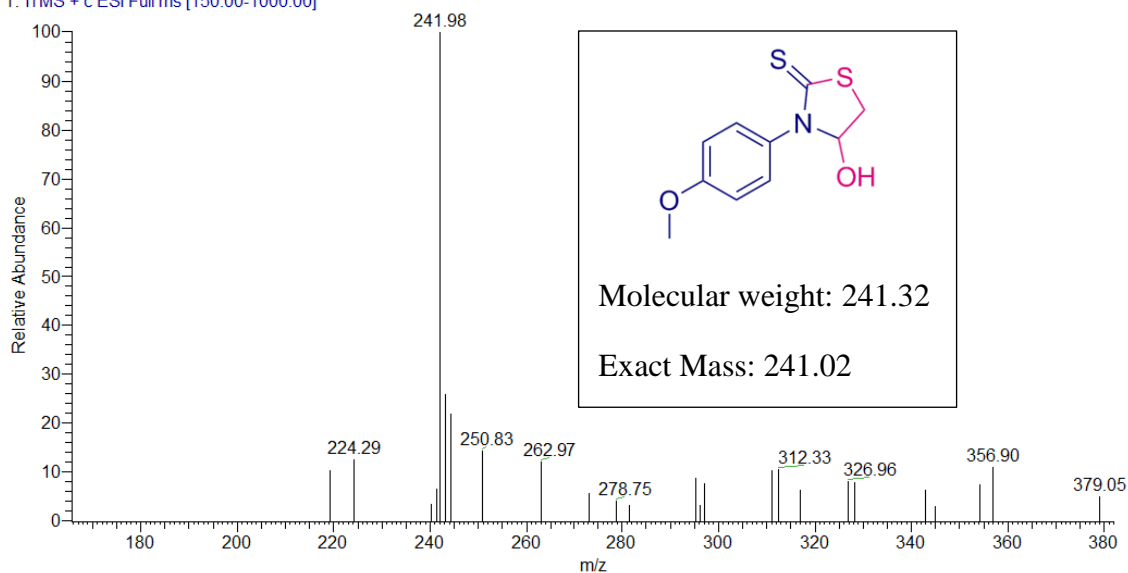


Fig.27 ESI mass spectrum of 3h

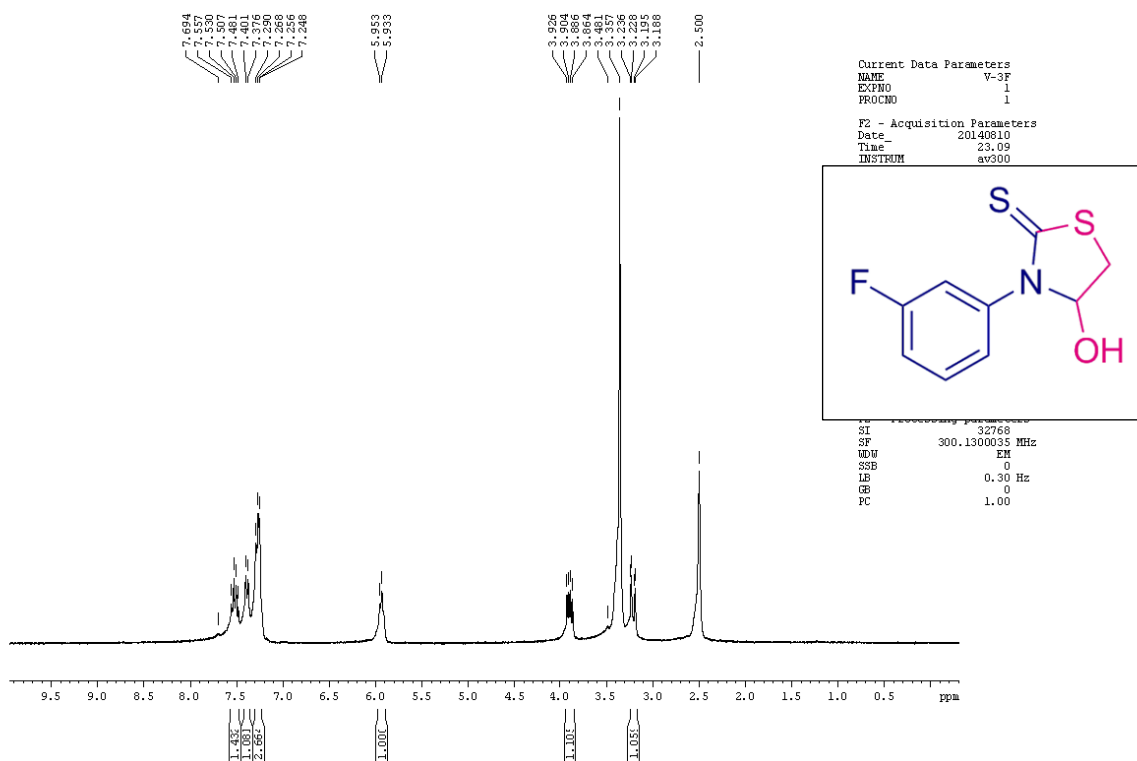
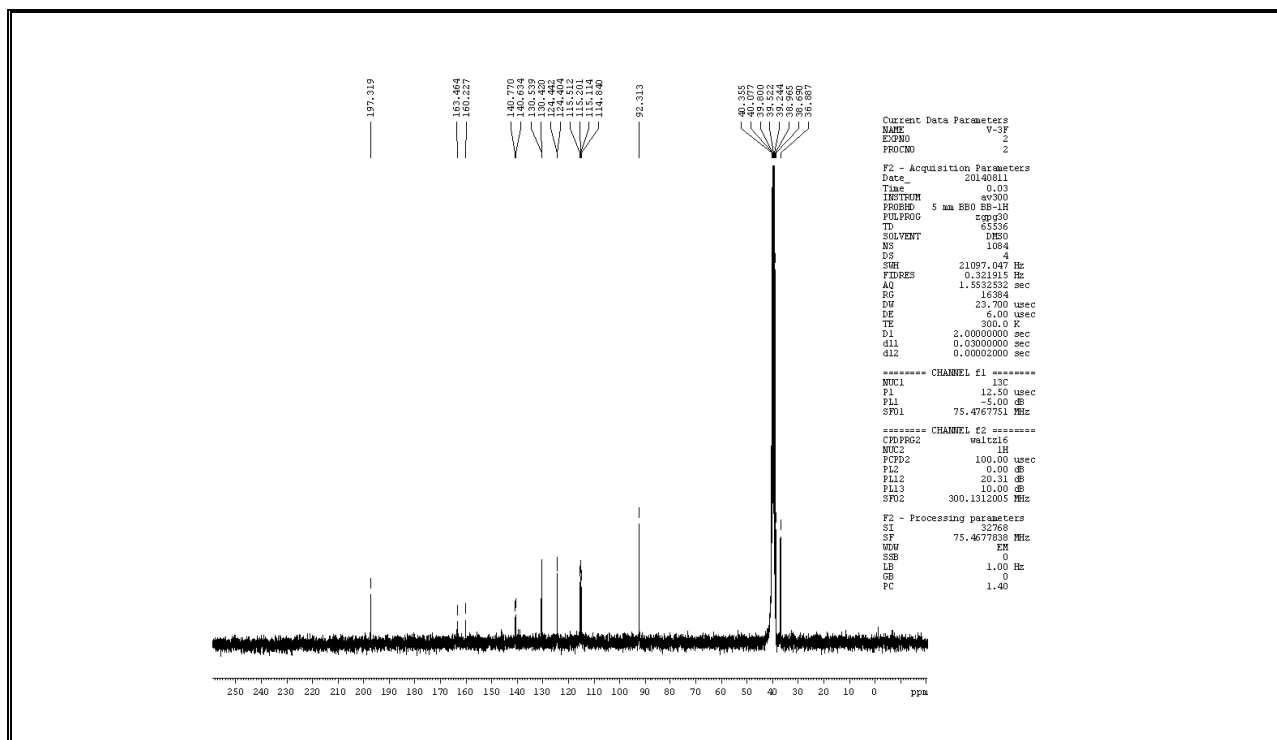
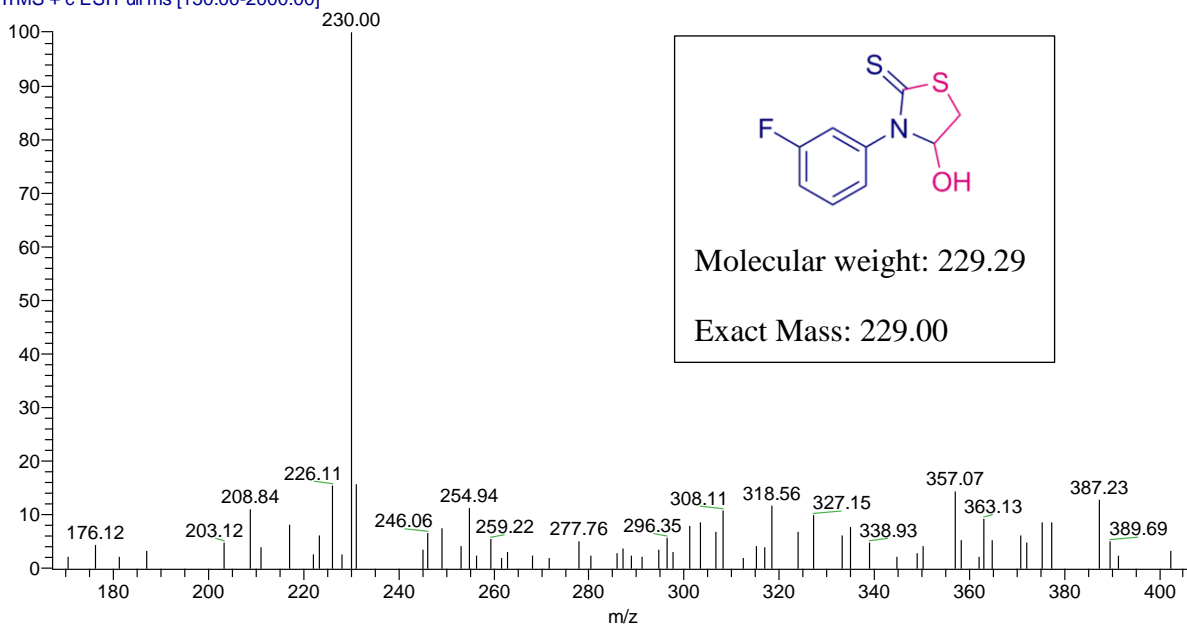


Fig.28 <sup>1</sup>H NMR Spectrum 3i (DMSO-d<sub>6</sub>)



**Fig.29**  $^{13}\text{C}$  NMR Spectrum of **3i** (DMSO- $d_6$ )

V-11\_140812233707 #26 RT: 0.42 AV: 1 NL: 5.62E1  
T: ITMS + c ESI Full ms [150.00-2000.00]



**Fig.30** ESI mass spectrum of **3i**

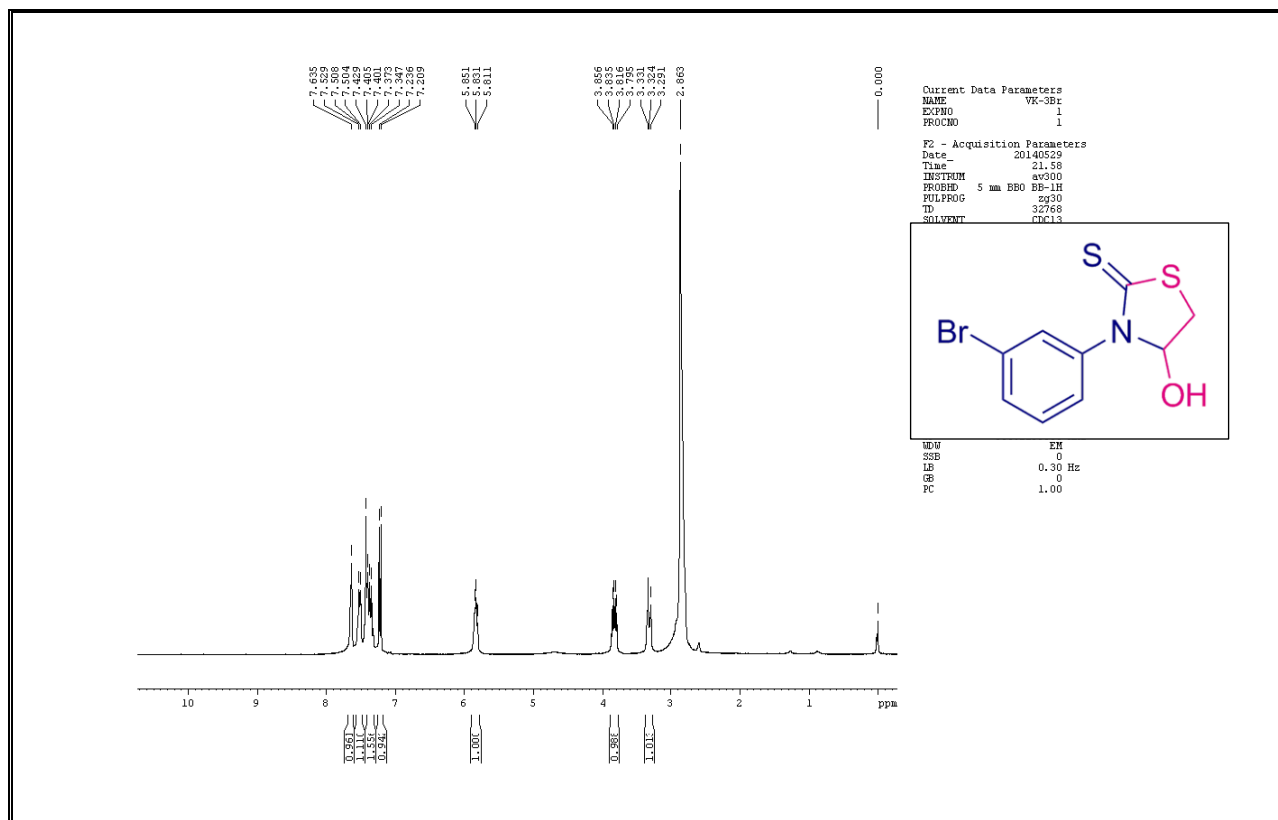


Fig.31 <sup>1</sup>H NMR Spectrum 3j (CDCl<sub>3</sub> + DMSO-d<sub>6</sub>)

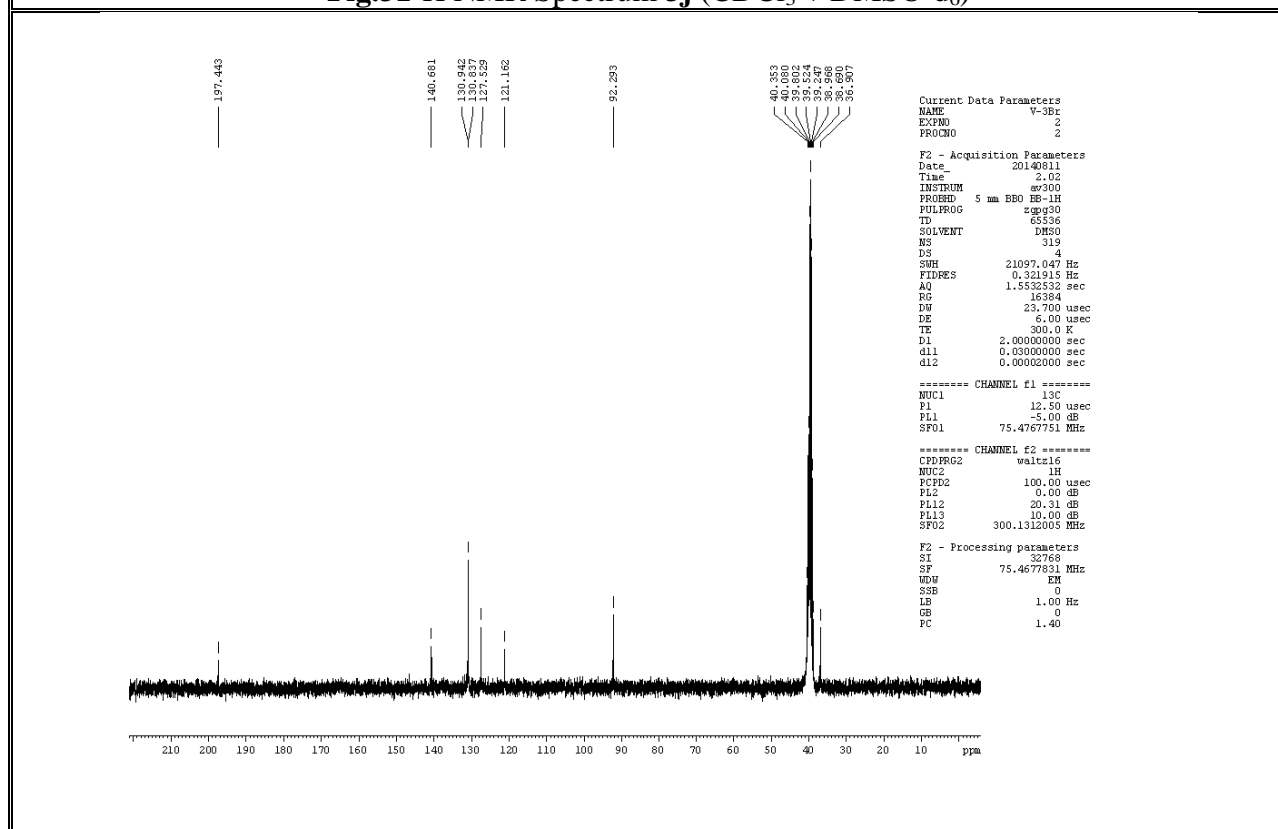


Fig.32 <sup>13</sup>C NMR Spectrum of 3j (DMSO-d<sub>6</sub>)

V-13\_140812233707 #53 RT: 0.65 AV: 1 NL: 1.42E2  
T: ITMS + c ESI Full ms [150.00-1000.00]

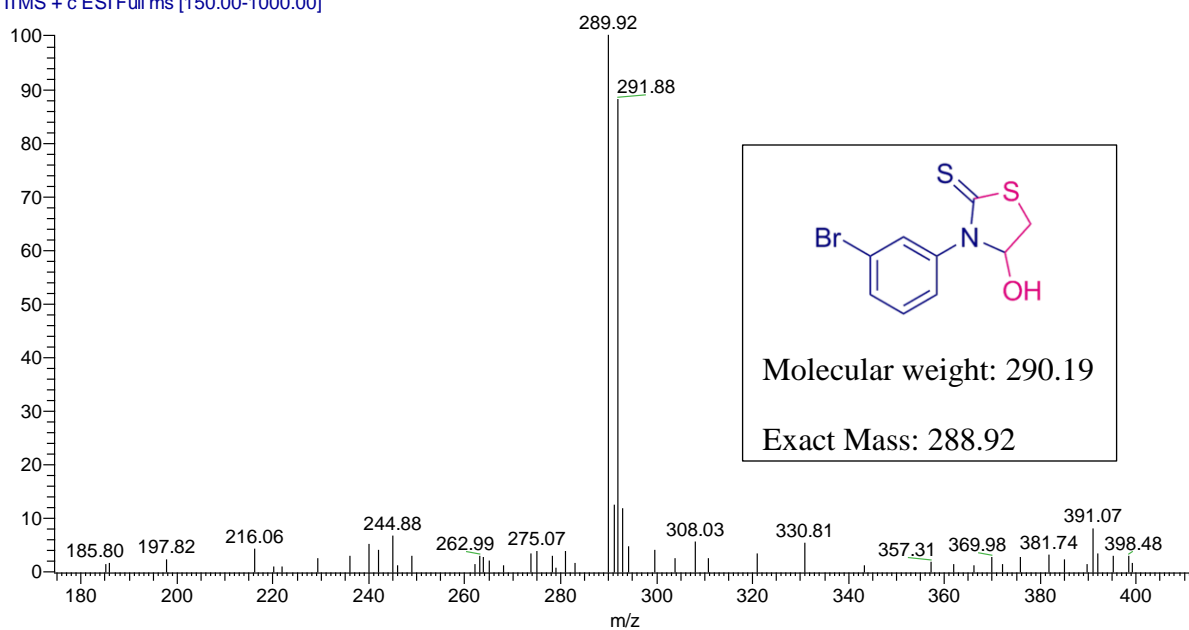


Fig.33 ESI mass spectrum of **3j**

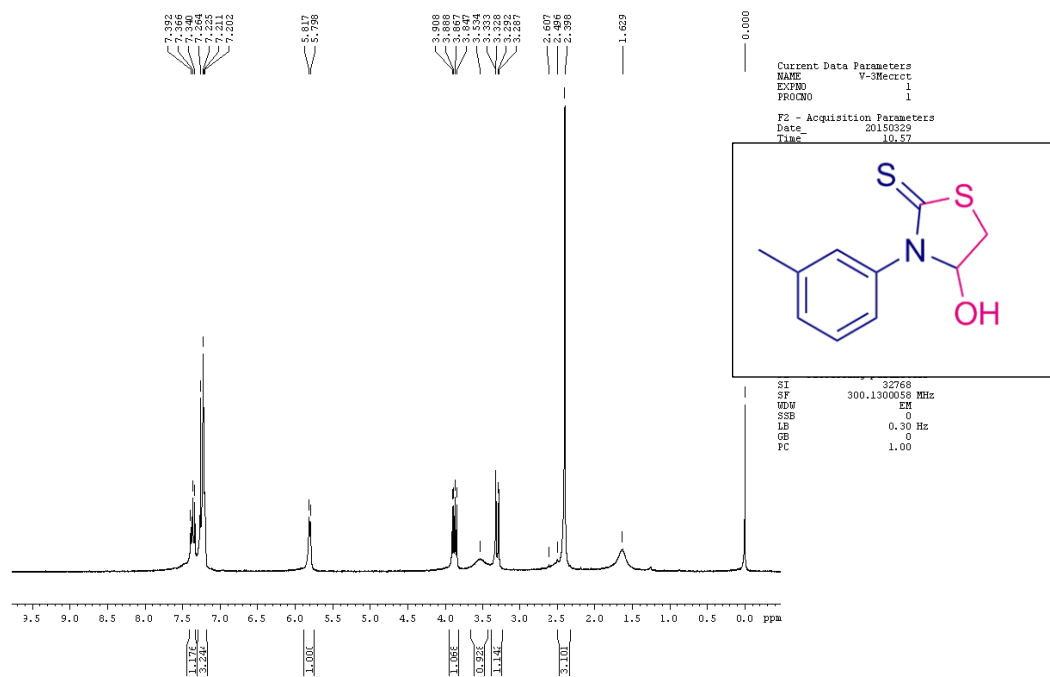
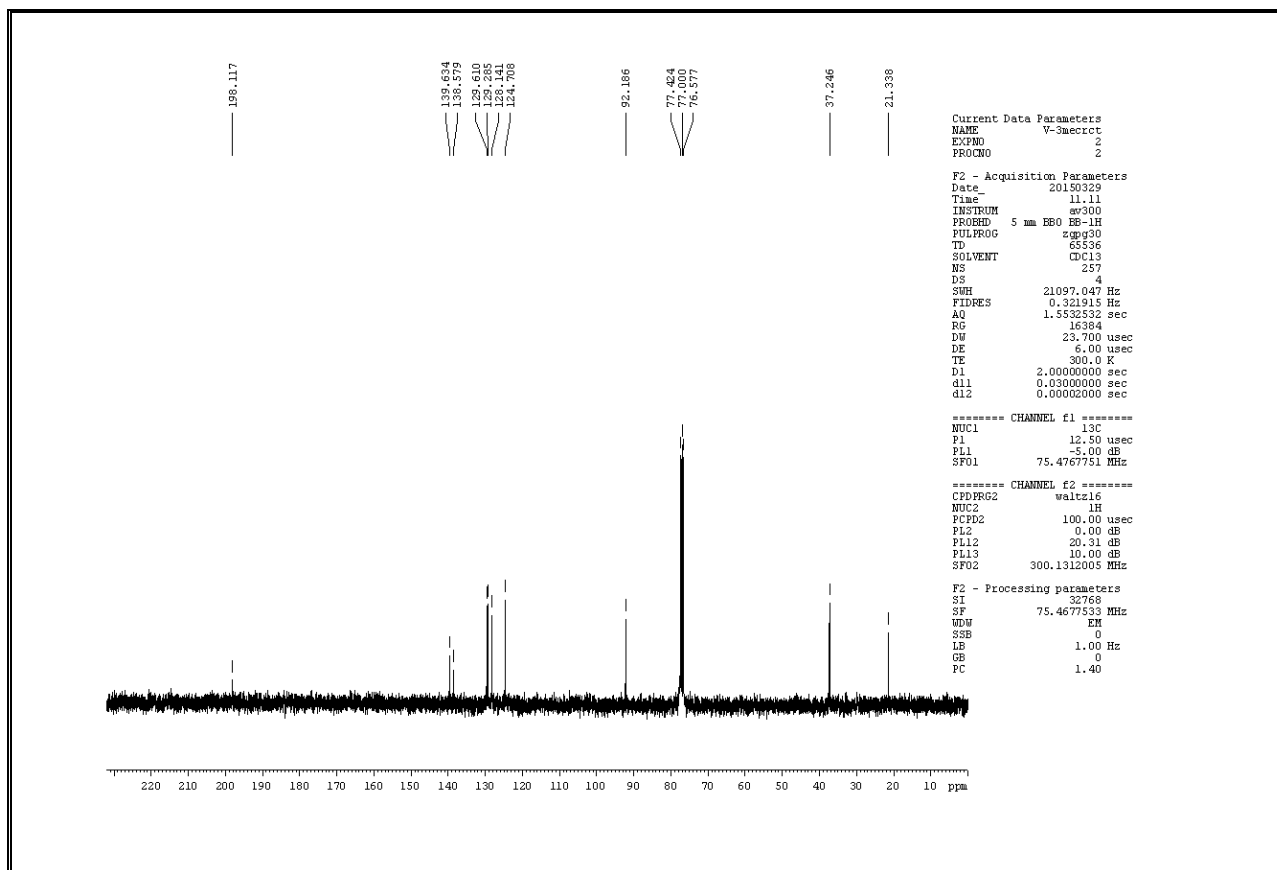
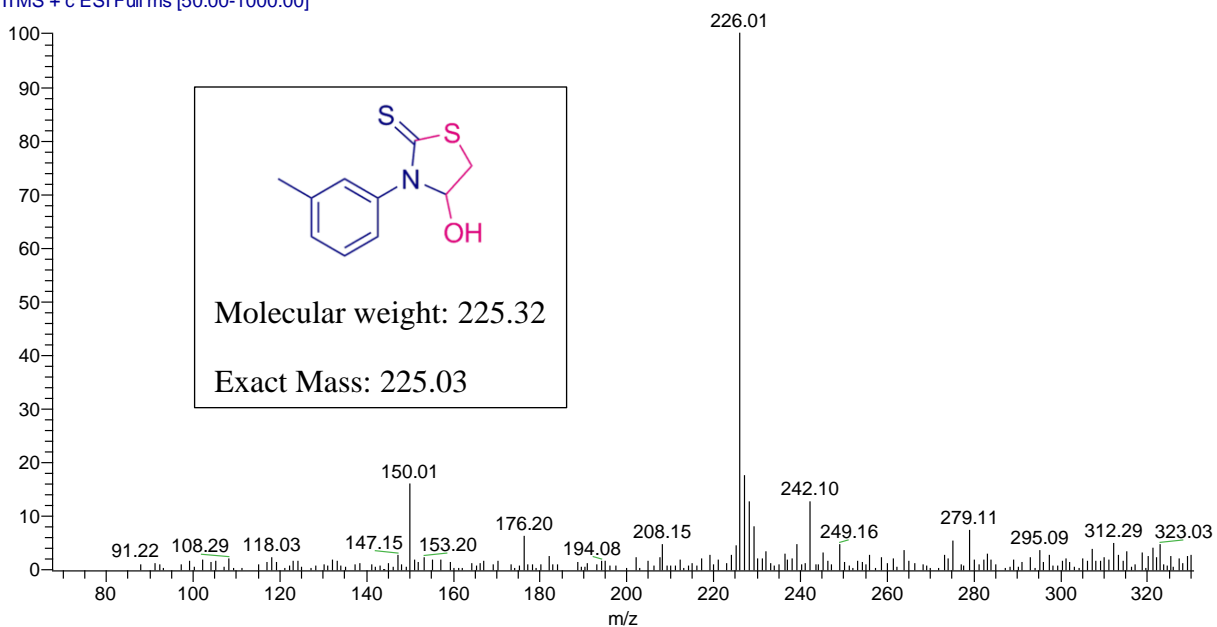


Fig.34  $^1\text{H}$  NMR Spectrum **3k** ( $\text{CDCl}_3$ )



**Fig.35**  $^{13}\text{C}$  NMR Spectrum of **3k** ( $\text{CDCl}_3$ )

3ME #15 RT: 0.19 AV: 1 NL: 3.26E3  
T: ITMS + c ESI Full ms [50.00-1000.00]



**Fig.36** ESI mass spectrum of **3k**

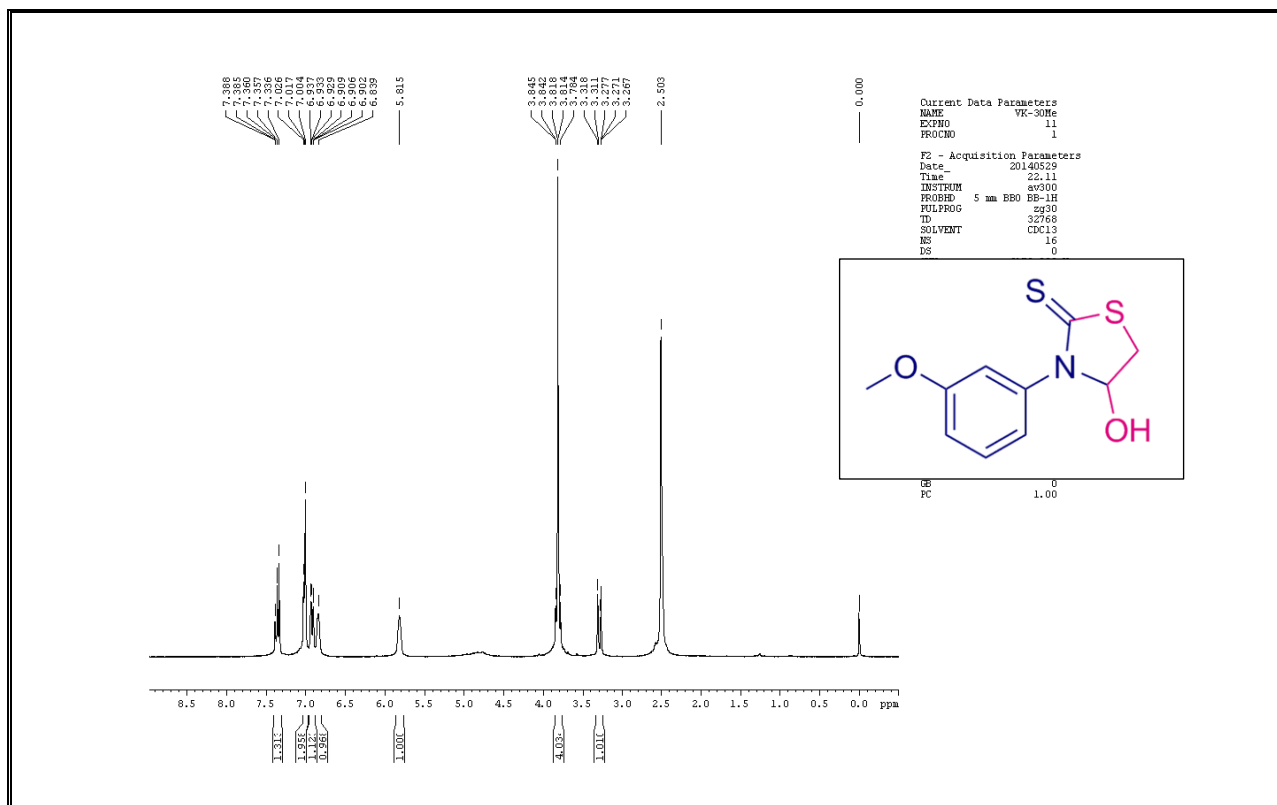


Fig.37 <sup>1</sup>H NMR Spectrum 3l (CDCl<sub>3</sub> + DMSO-d<sub>6</sub>)

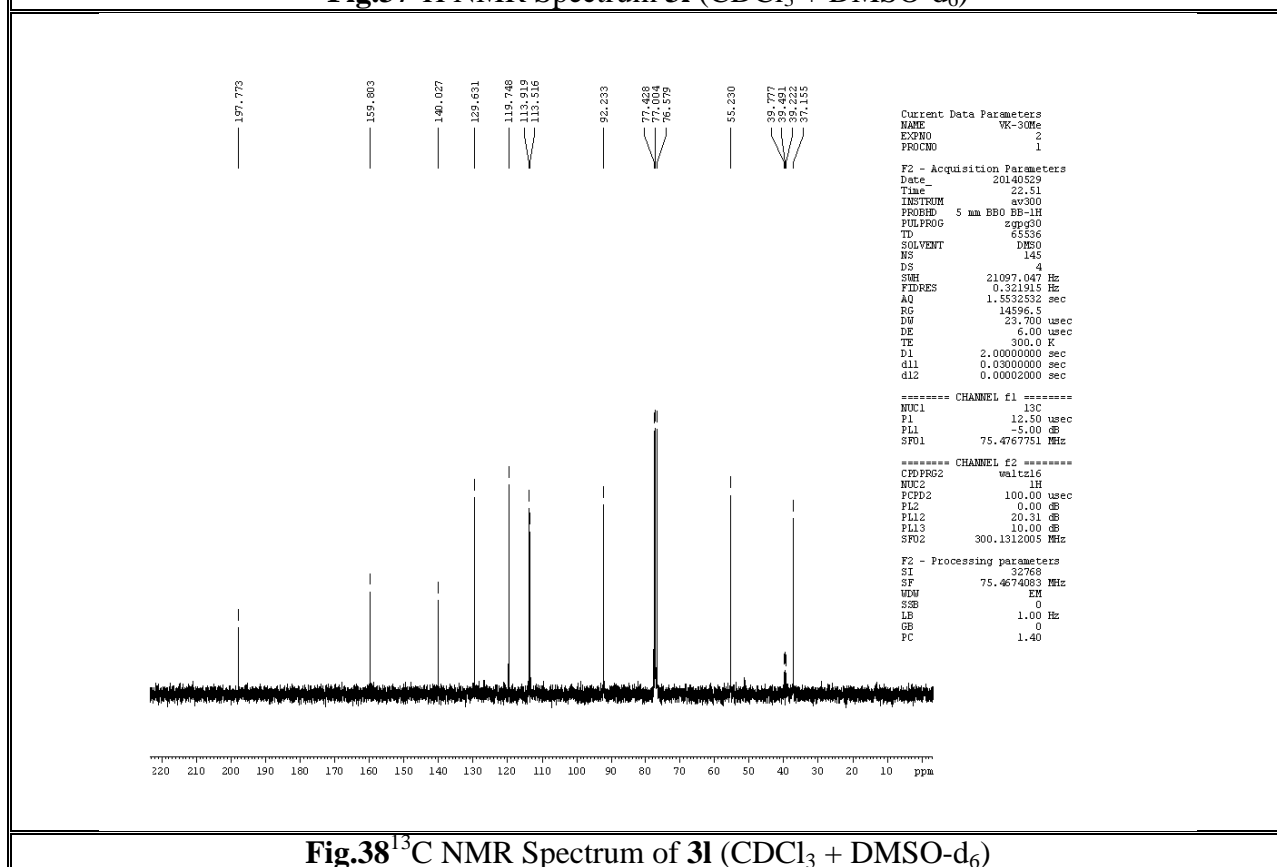


Fig.38 <sup>13</sup>C NMR Spectrum of 3l (CDCl<sub>3</sub> + DMSO-d<sub>6</sub>)



V-14\_140812233707 #29 RT: 0.35 AV: 1 NL: 1.68E2  
T: ITMS + c ESI Full ms [150.00-1000.00]

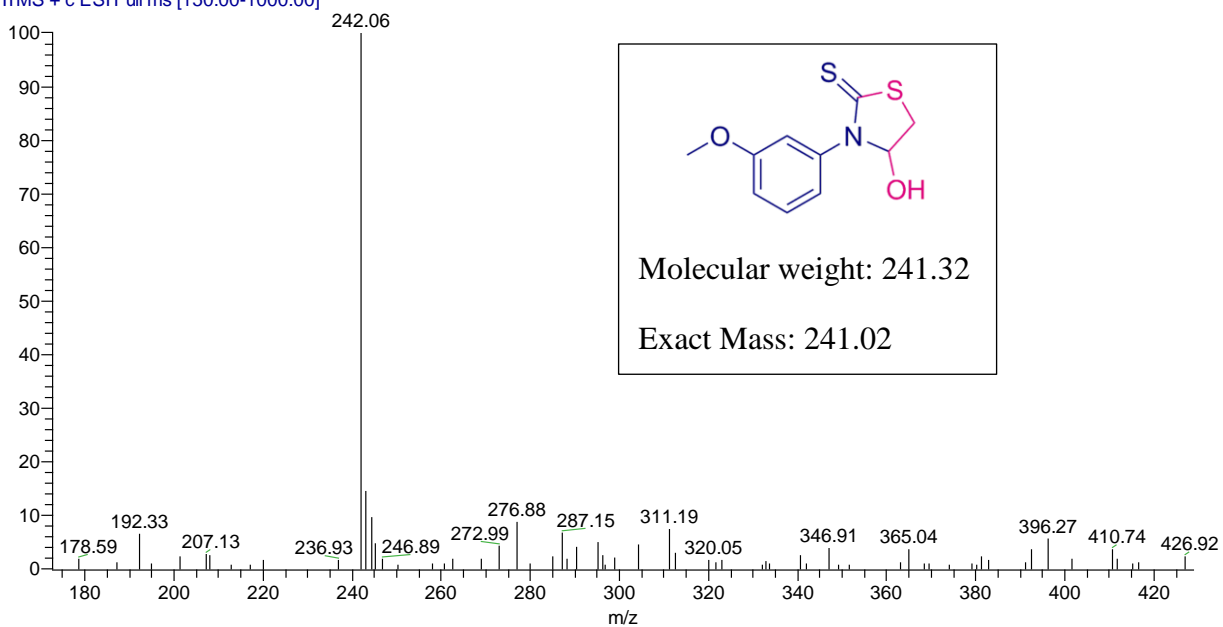


Fig.39 ESI mass spectrum of 31

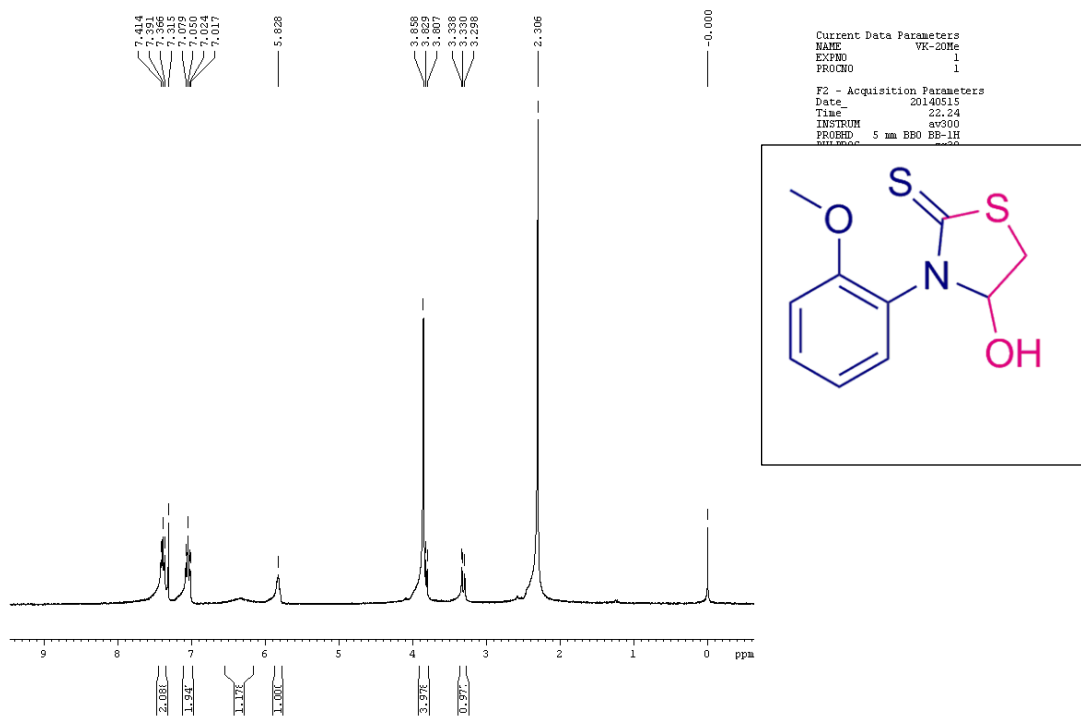


Fig.40  $^1\text{H}$  NMR Spectrum 3m ( $\text{CDCl}_3 + \text{DMSO-d}_6$ )

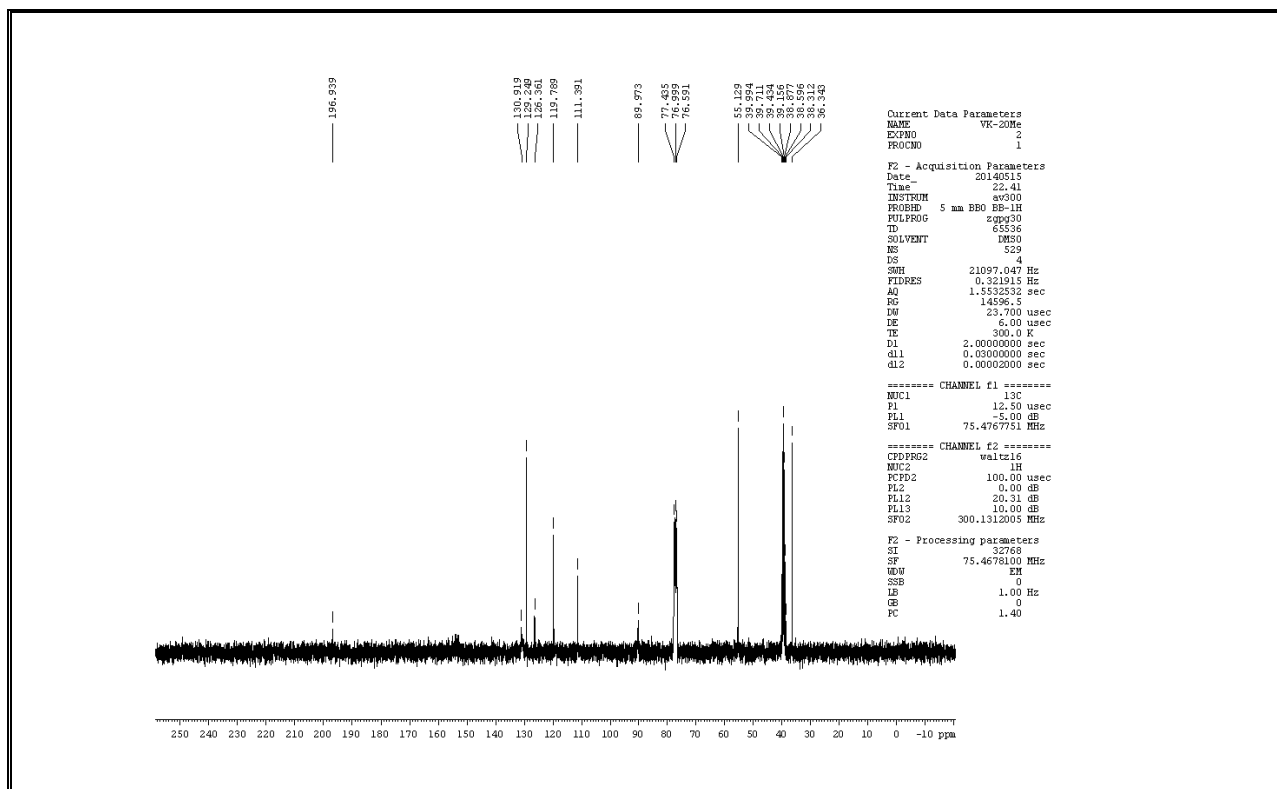


Fig.41 <sup>13</sup>C NMR Spectrum of 3m(CDCl<sub>3</sub> + DMSO-d<sub>6</sub>)

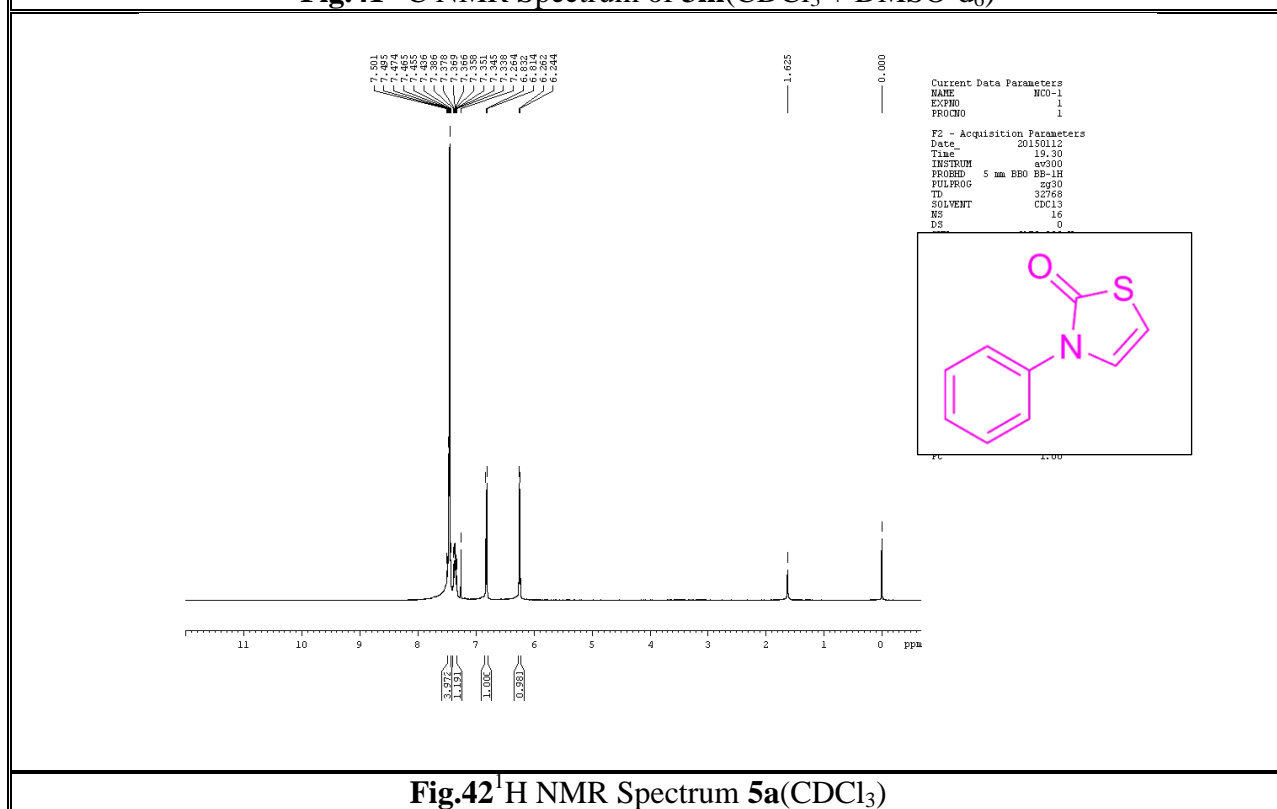


Fig.42 <sup>1</sup>H NMR Spectrum 5a(CDCl<sub>3</sub>)

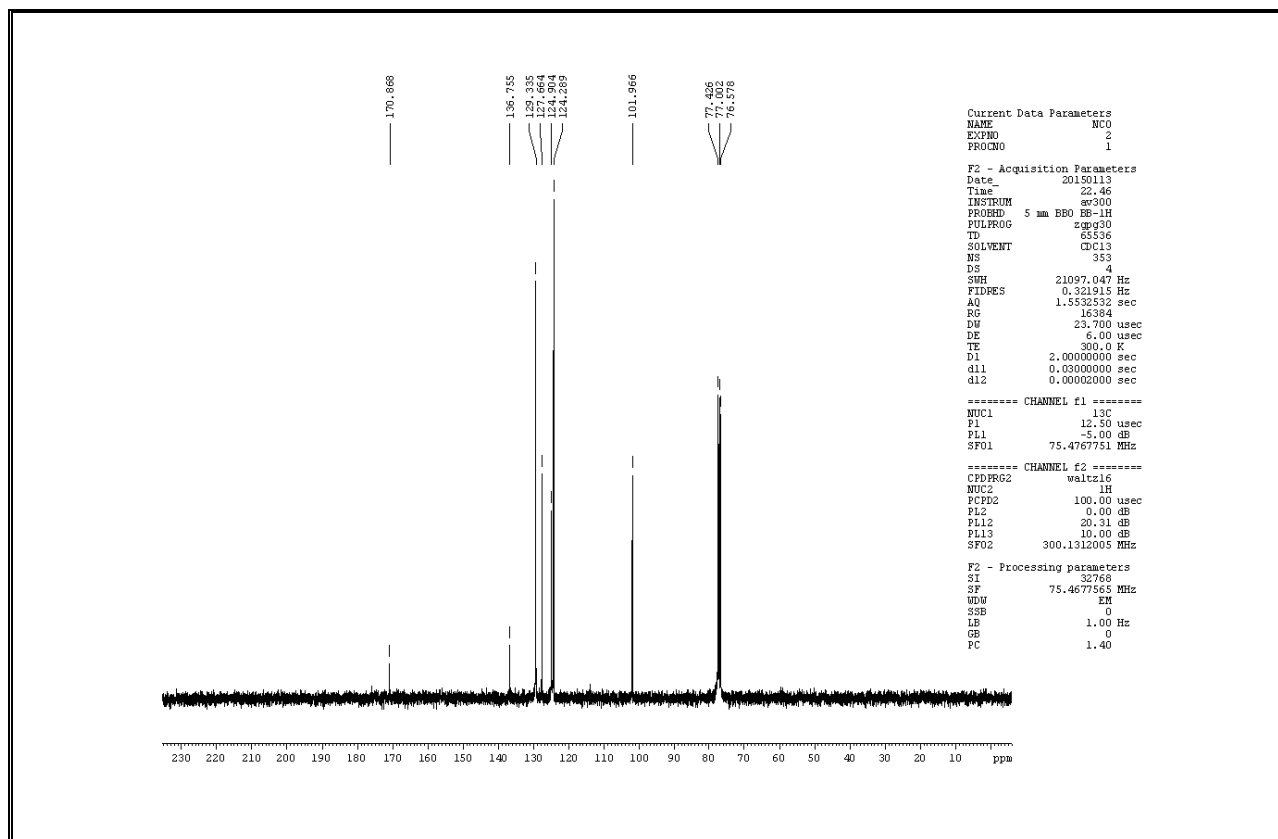


Fig.43  $^{13}\text{C}$  NMR Spectrum of **5a**( $\text{CDCl}_3$ )

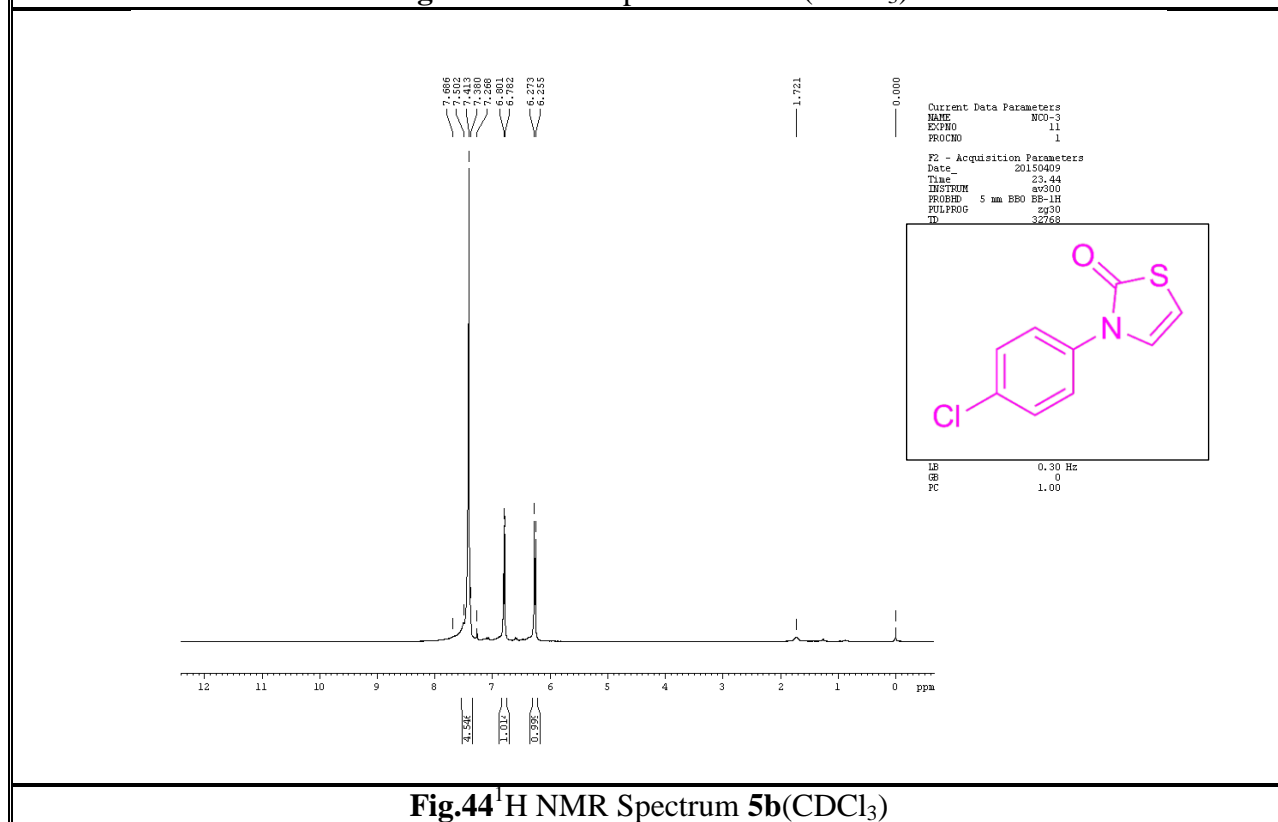


Fig.44  $^1\text{H}$  NMR Spectrum **5b**( $\text{CDCl}_3$ )

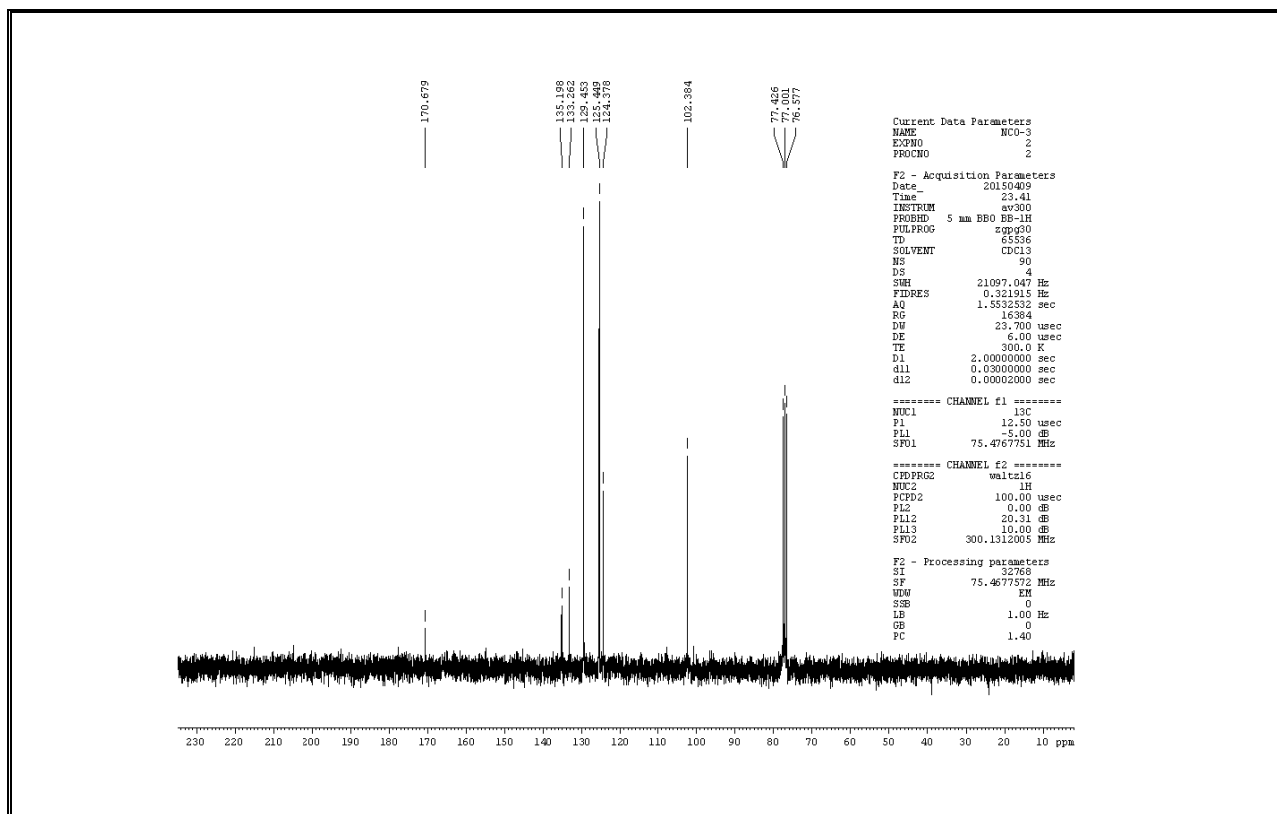


Fig.45  $^{13}\text{C}$  NMR Spectrum of **5b**( $\text{CDCl}_3$ )

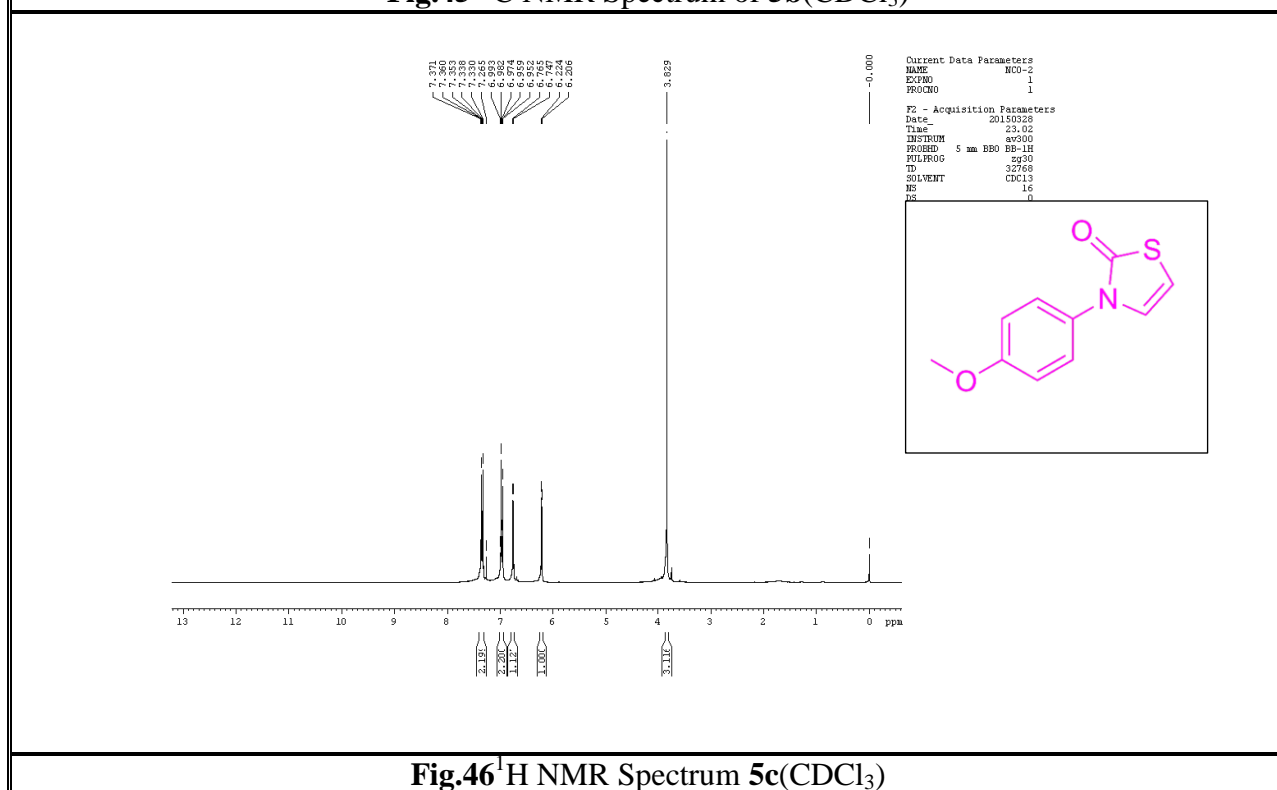


Fig.46  $^1\text{H}$  NMR Spectrum **5c**( $\text{CDCl}_3$ )

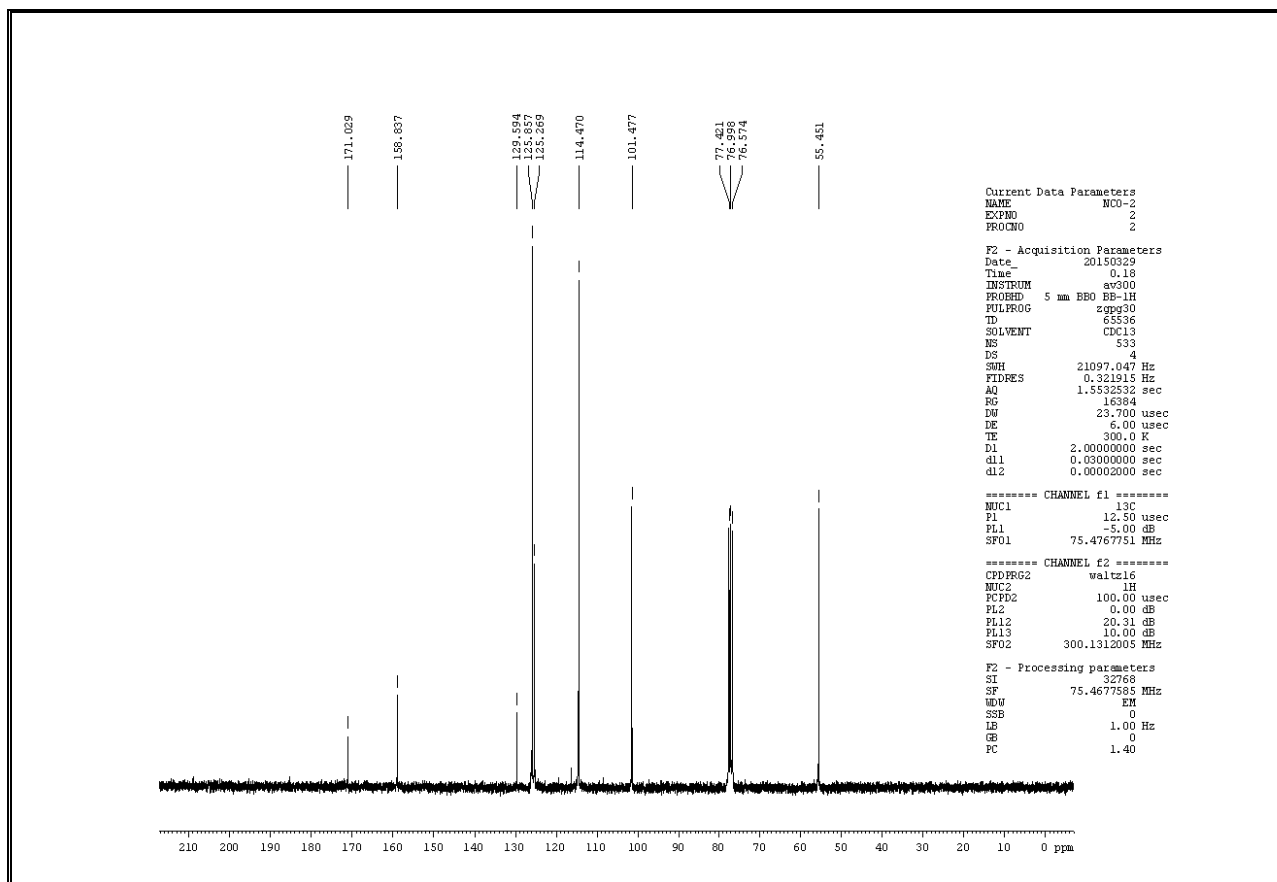


Fig.47 <sup>13</sup>C NMR Spectrum of 5c(CDCl<sub>3</sub>)

NCO-2 #19 RT: 0.24 AV: 1 NL: 5.99E3  
 T: ITMS + c ESI Full ms [50.00-1000.00]

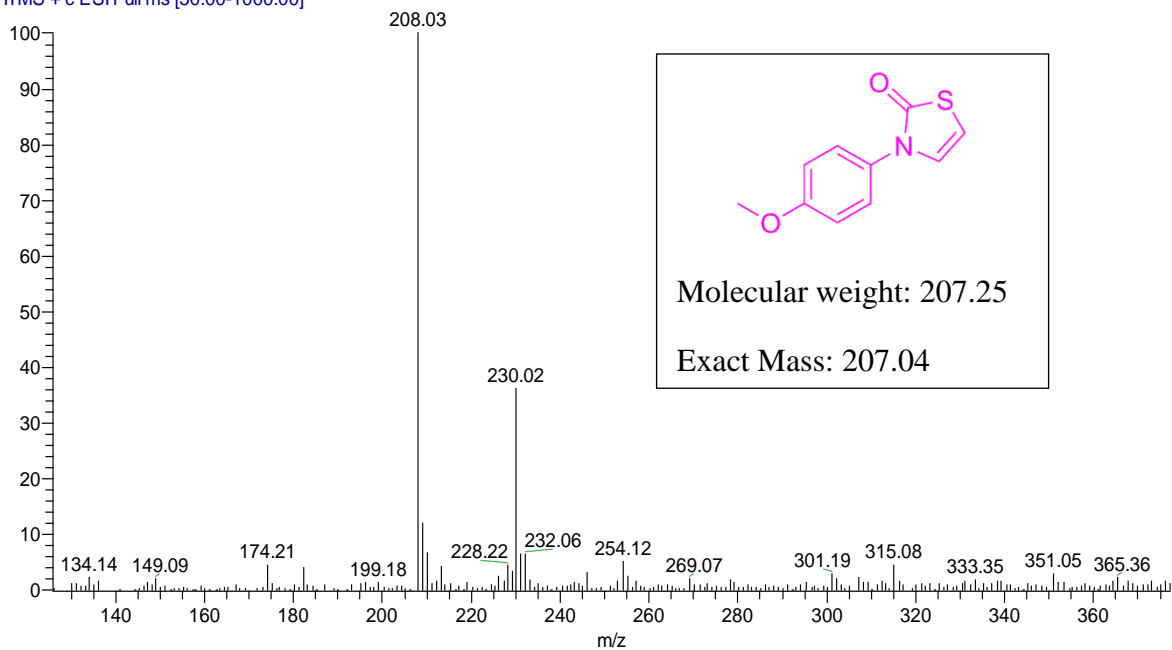


Fig.48 ESI mass spectrum of 5c

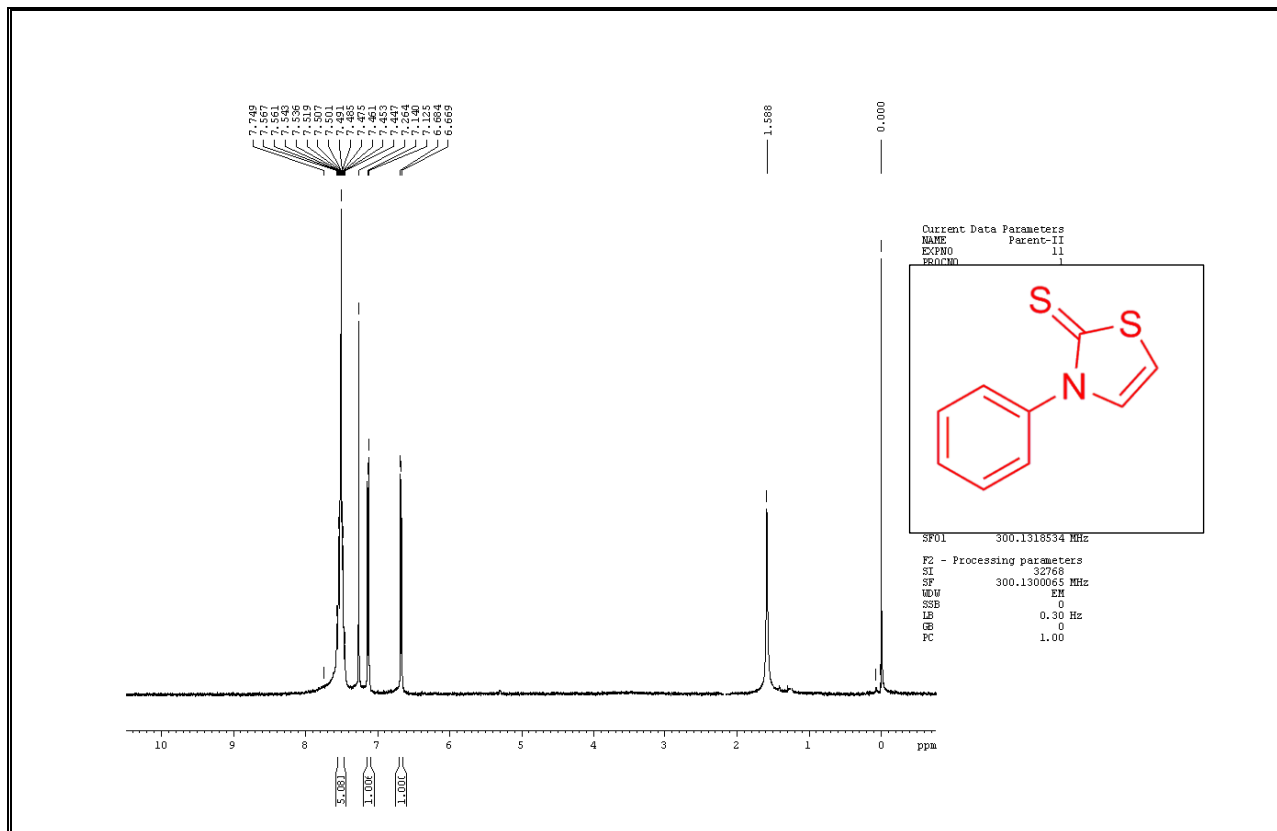


Fig.49 <sup>1</sup>H NMR Spectrum 6a(CDCl<sub>3</sub>)

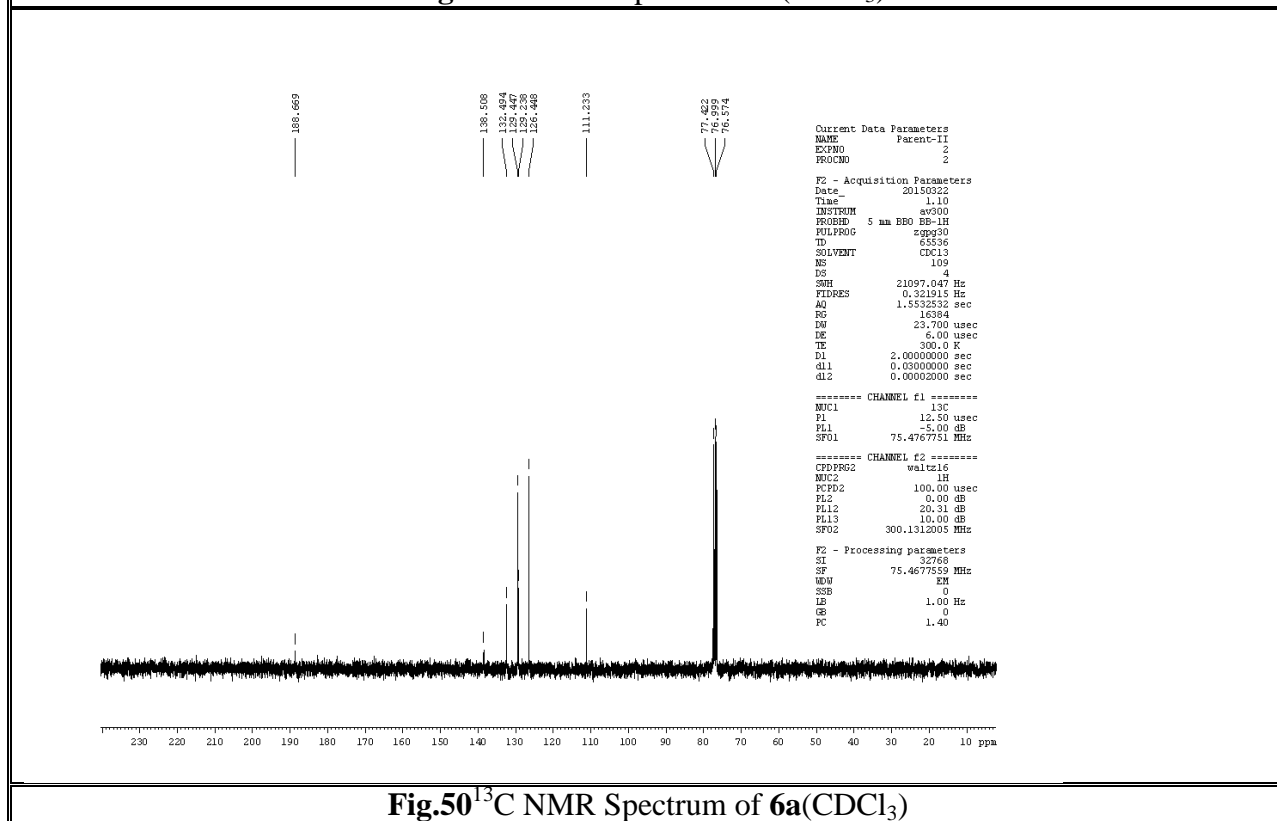


Fig.50 <sup>13</sup>C NMR Spectrum of 6a(CDCl<sub>3</sub>)

H-II #29 RT: 0.36 AV: 1 NL: 3.40E3  
T: ITMS + c ESI Full ms [50.00-1000.00]

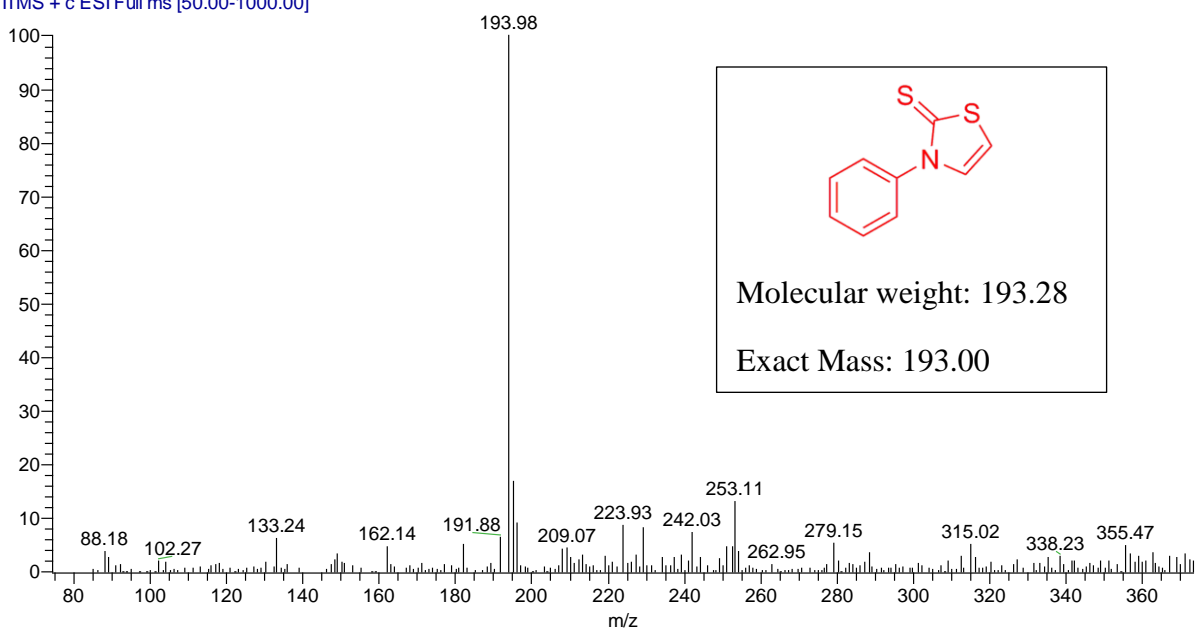


Fig.51 ESI mass spectrum of 6a

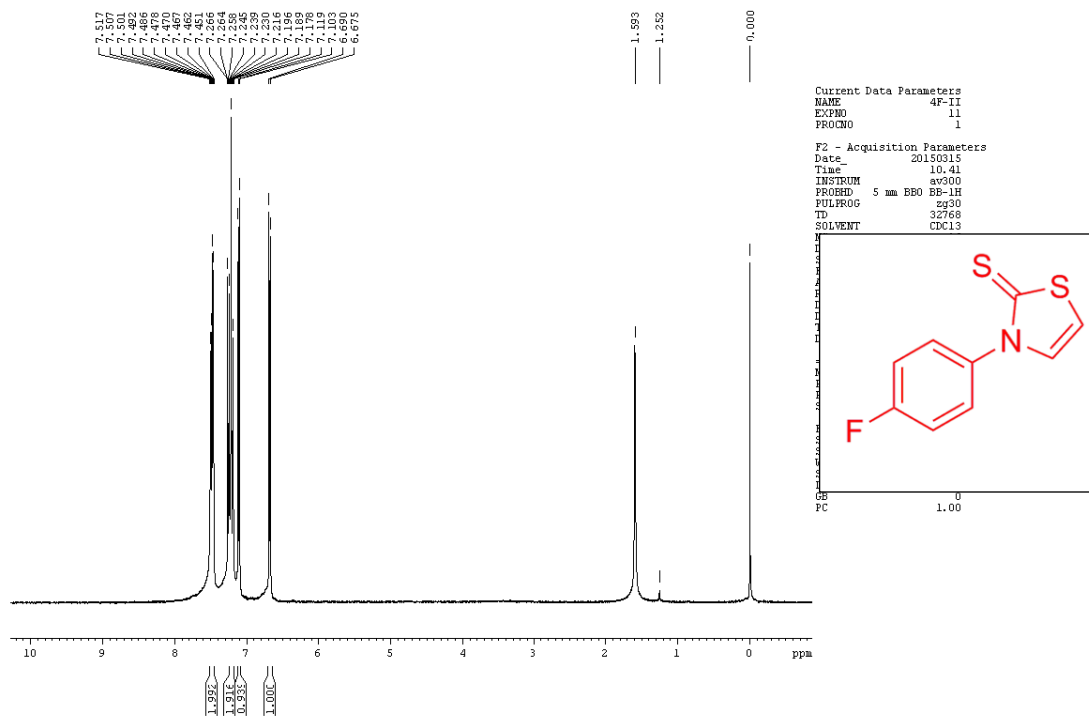


Fig.52 <sup>1</sup>H NMR Spectrum 6b(CDCl<sub>3</sub>)

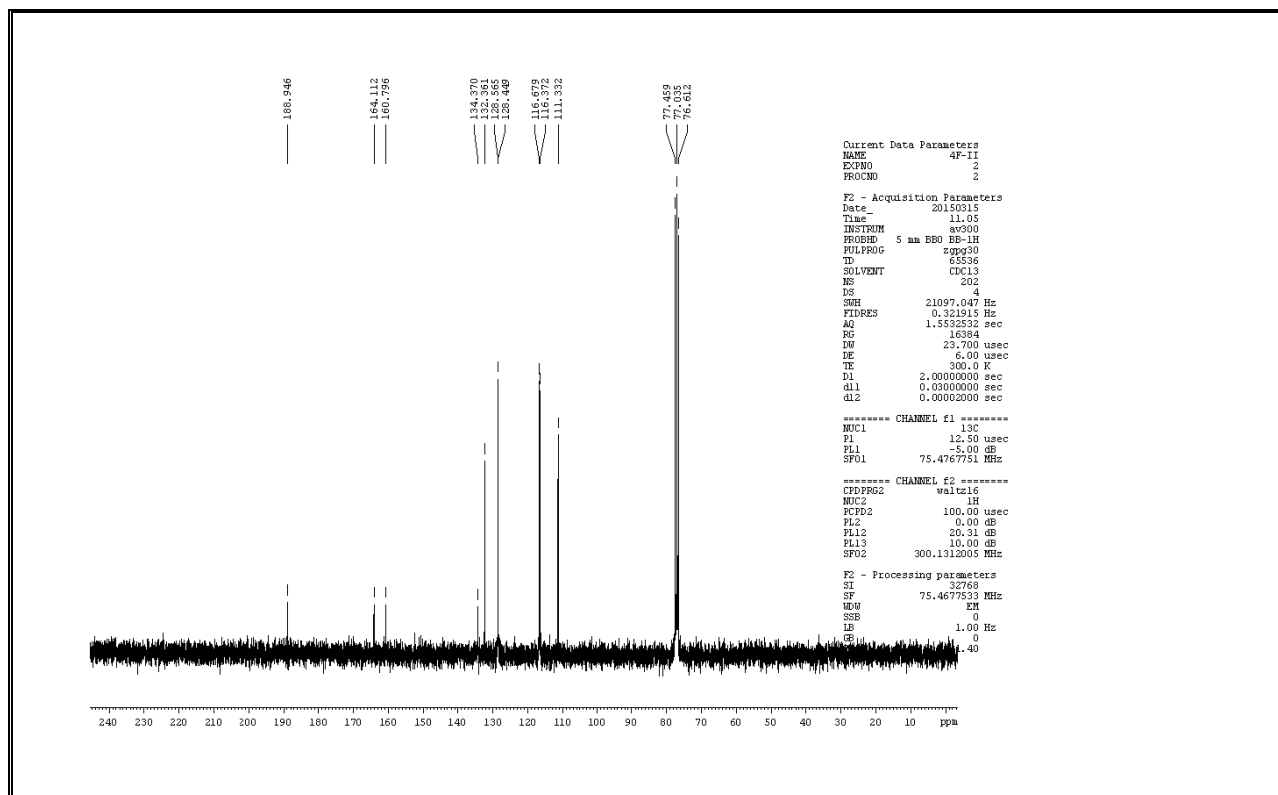


Fig.53 <sup>13</sup>C NMR Spectrum of **6b**(CDCl<sub>3</sub>)

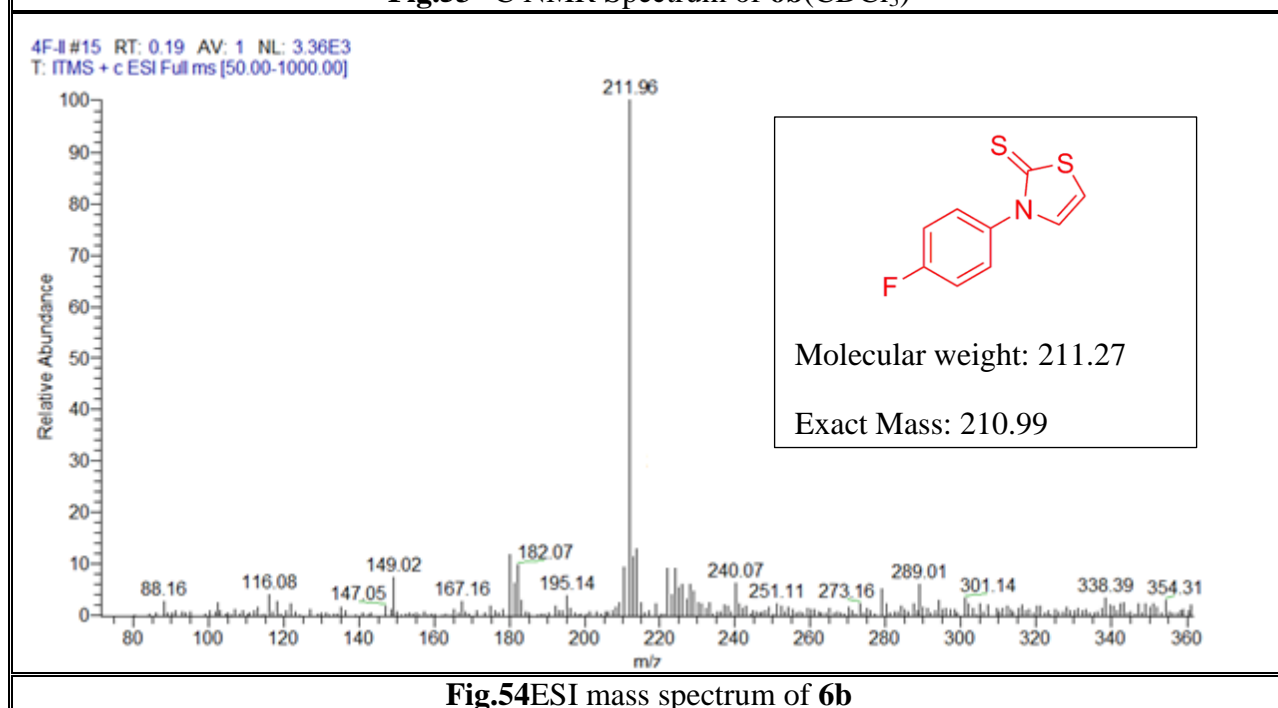


Fig.54 ESI mass spectrum of **6b**



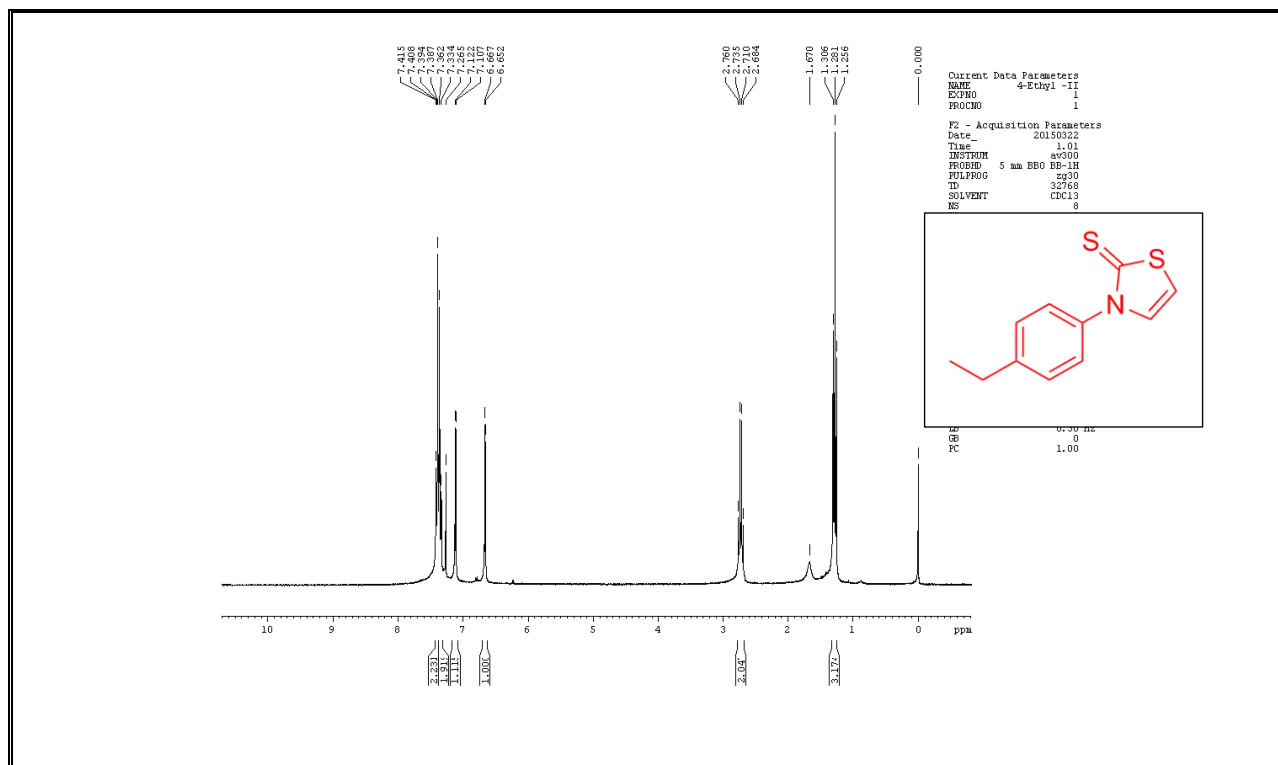


Fig.55  $^1\text{H}$  NMR Spectrum **6c**( $\text{CDCl}_3$ )

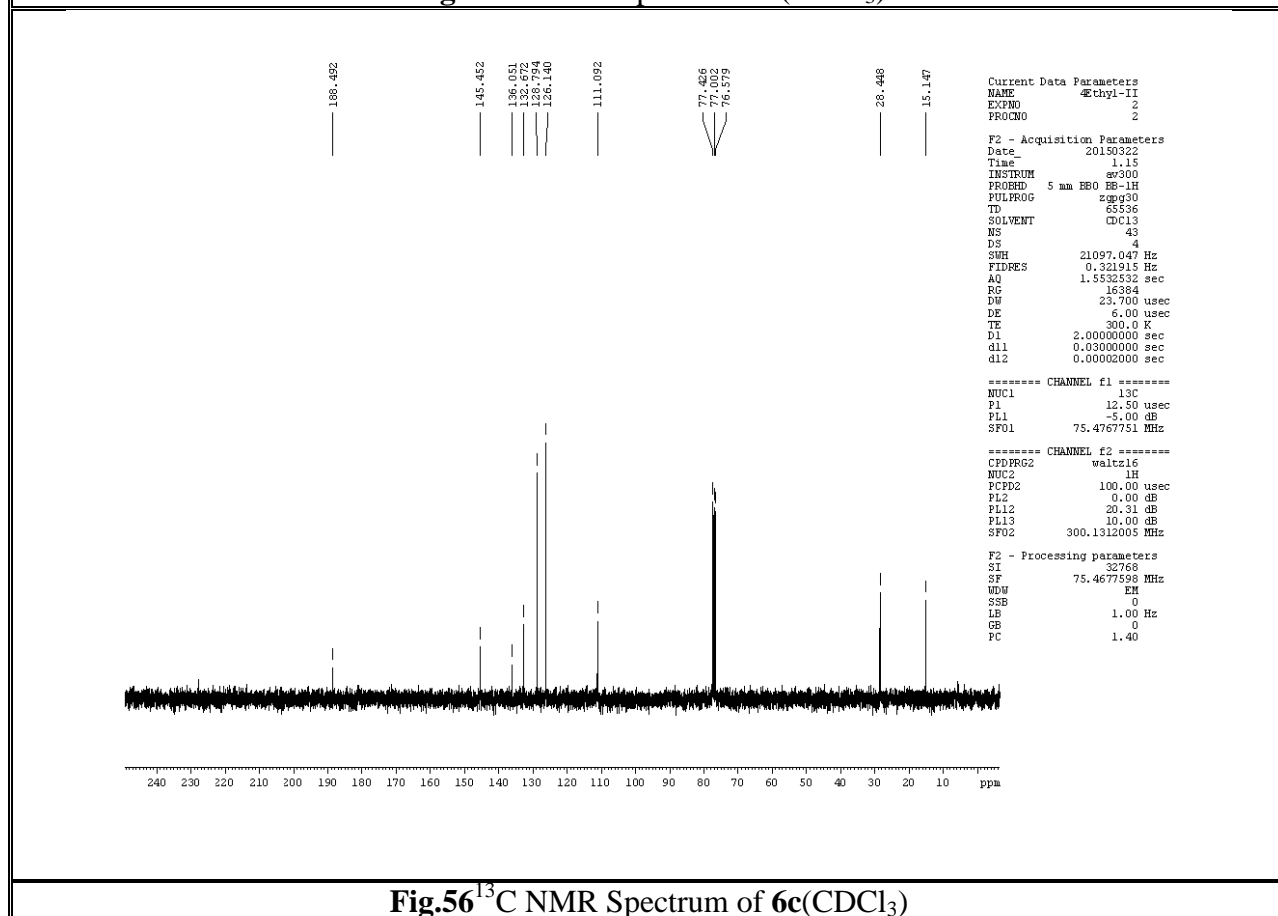


Fig.56  $^{13}\text{C}$  NMR Spectrum of **6c**( $\text{CDCl}_3$ )

4ET-II #33 RT: 0.41 AV: 1 NL: 5.58E3  
T: ITMS + c ESI Full ms [50.00-1000.00]

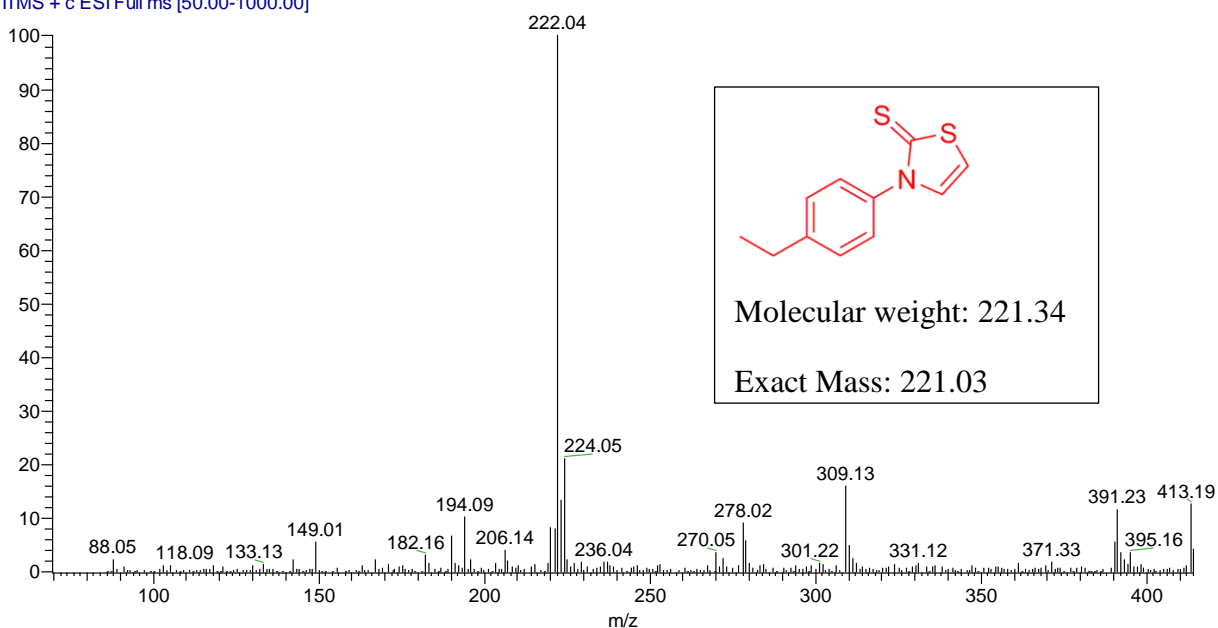


Fig.57 ESI mass spectrum of 6c

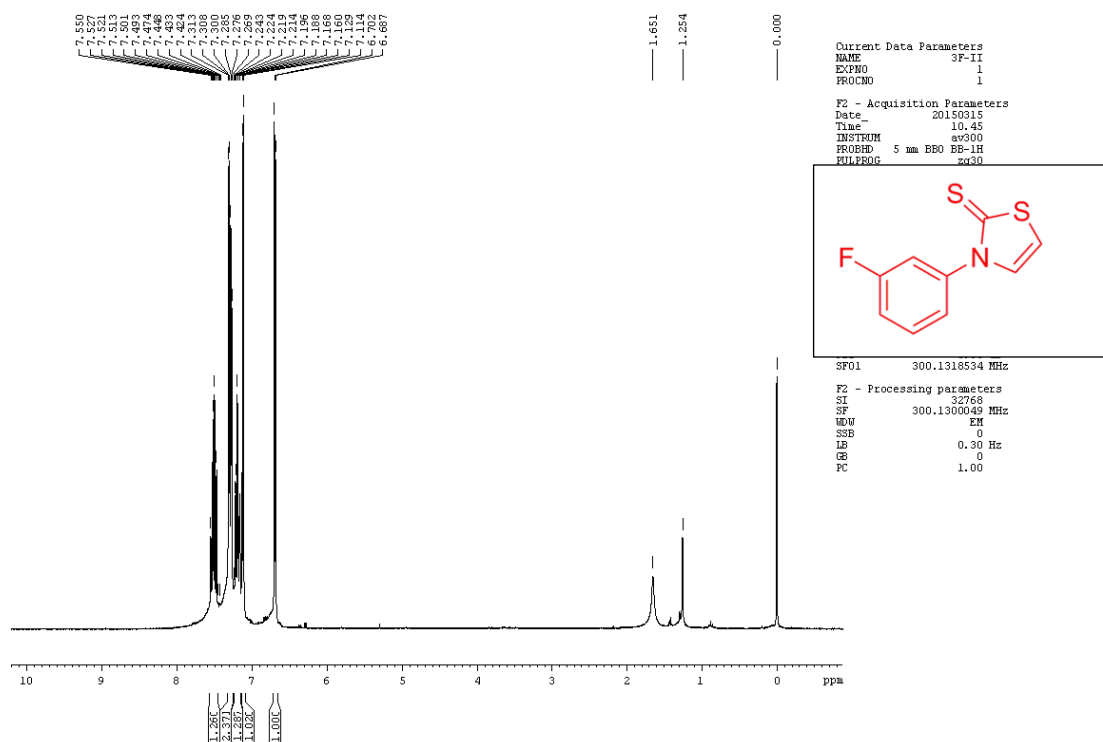


Fig.58 <sup>1</sup>H NMR Spectrum 6d(CDCl<sub>3</sub>)

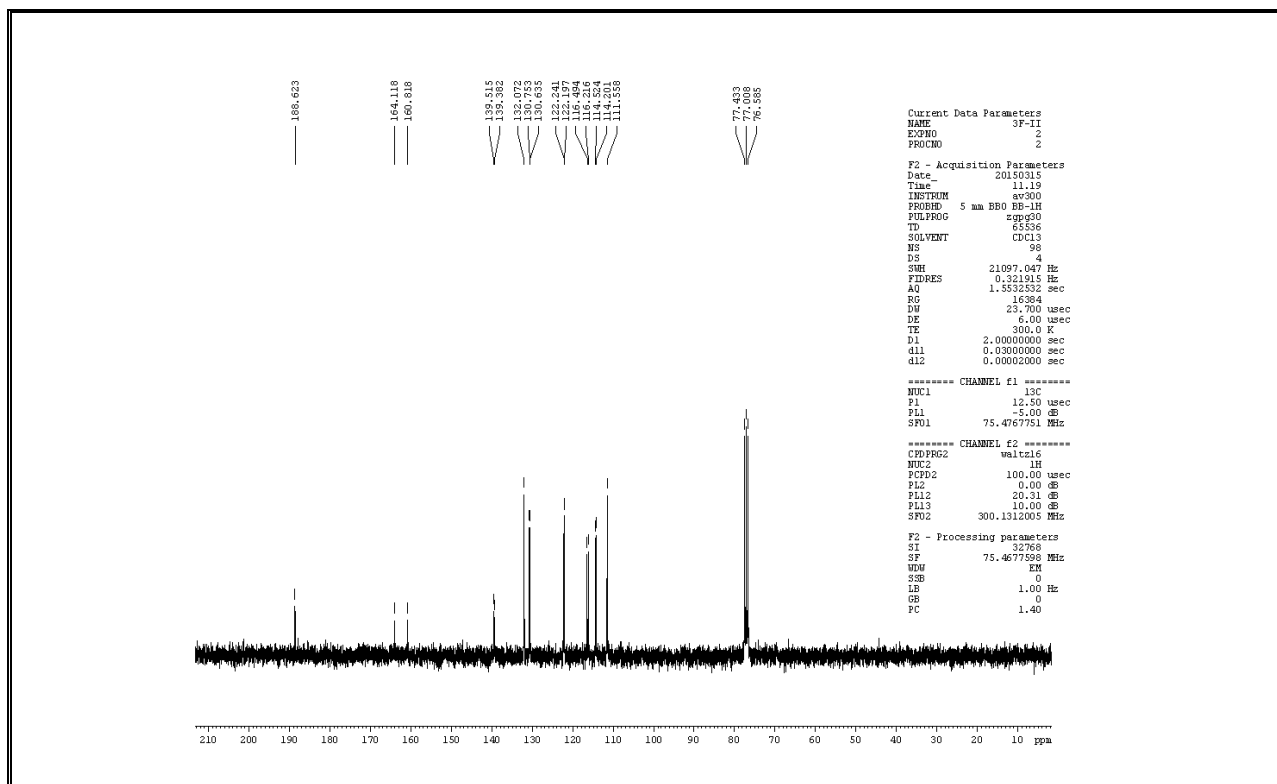


Fig.59  $^{13}\text{C}$  NMR Spectrum of **6d**( $\text{CDCl}_3$ )

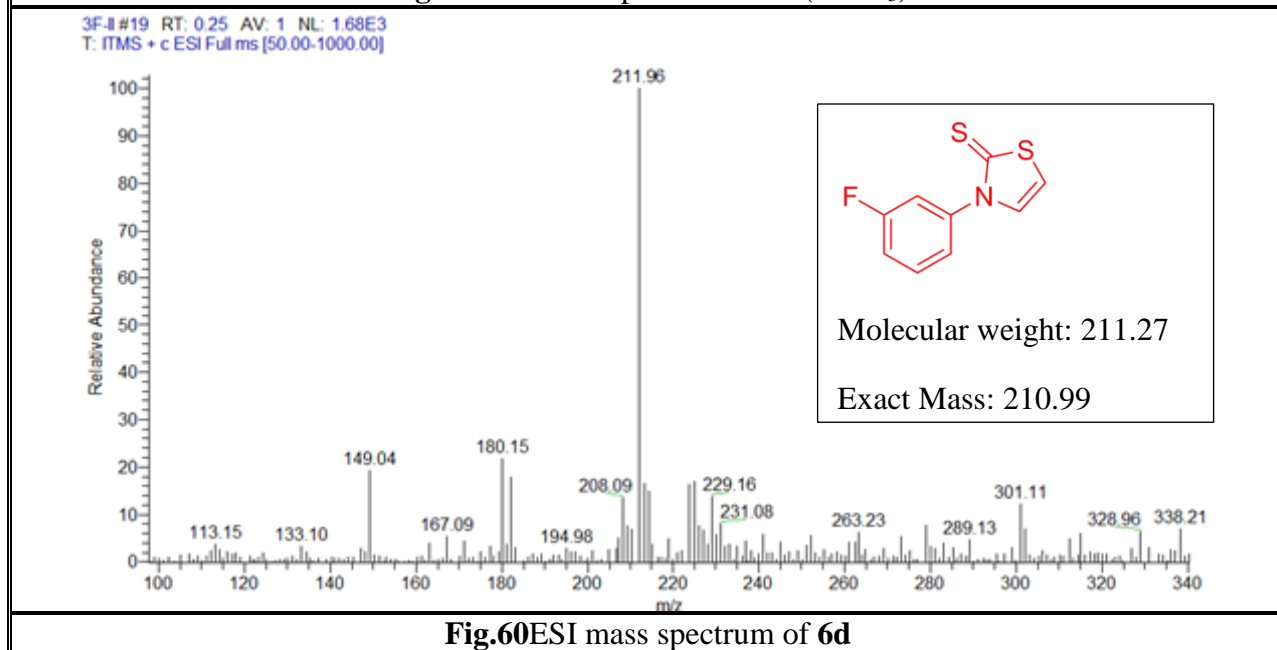


Fig.60 ESI mass spectrum of **6d**

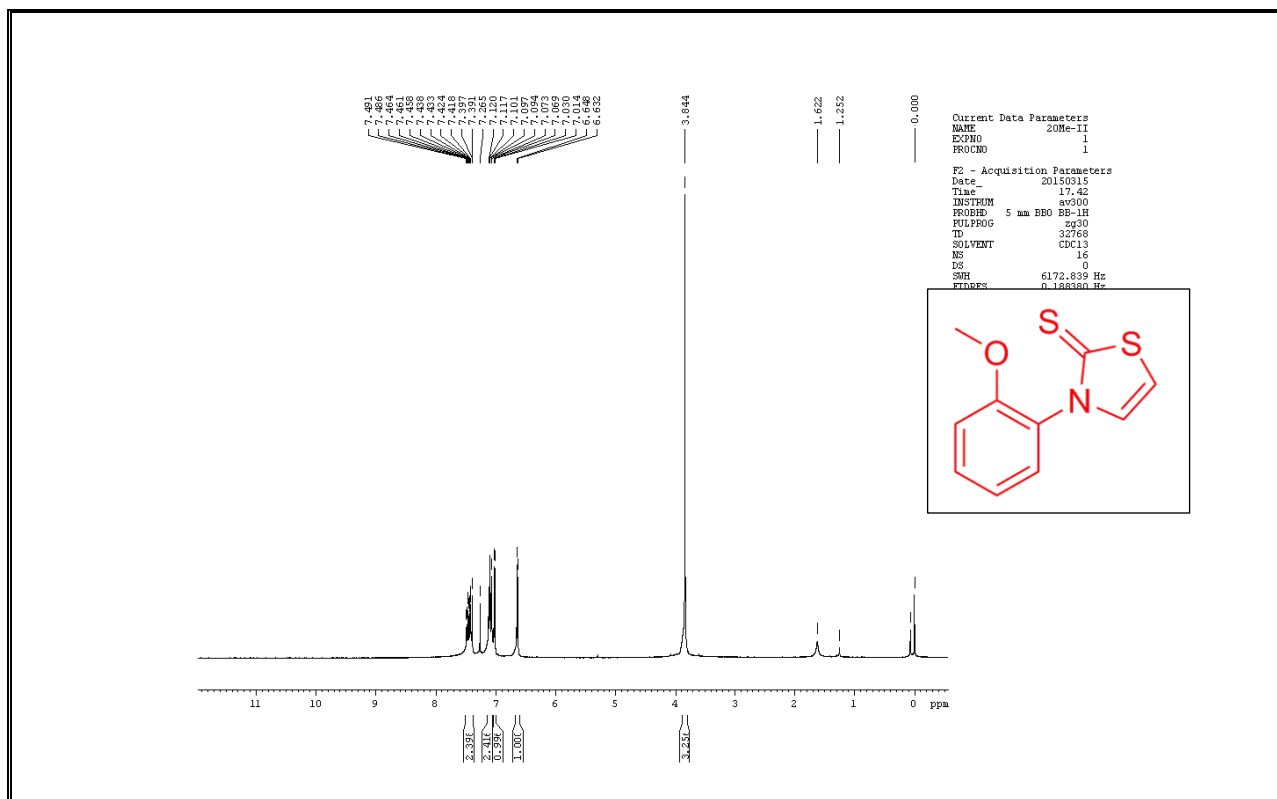


Fig.61 <sup>1</sup>H NMR Spectrum 6e(CDCl<sub>3</sub>)

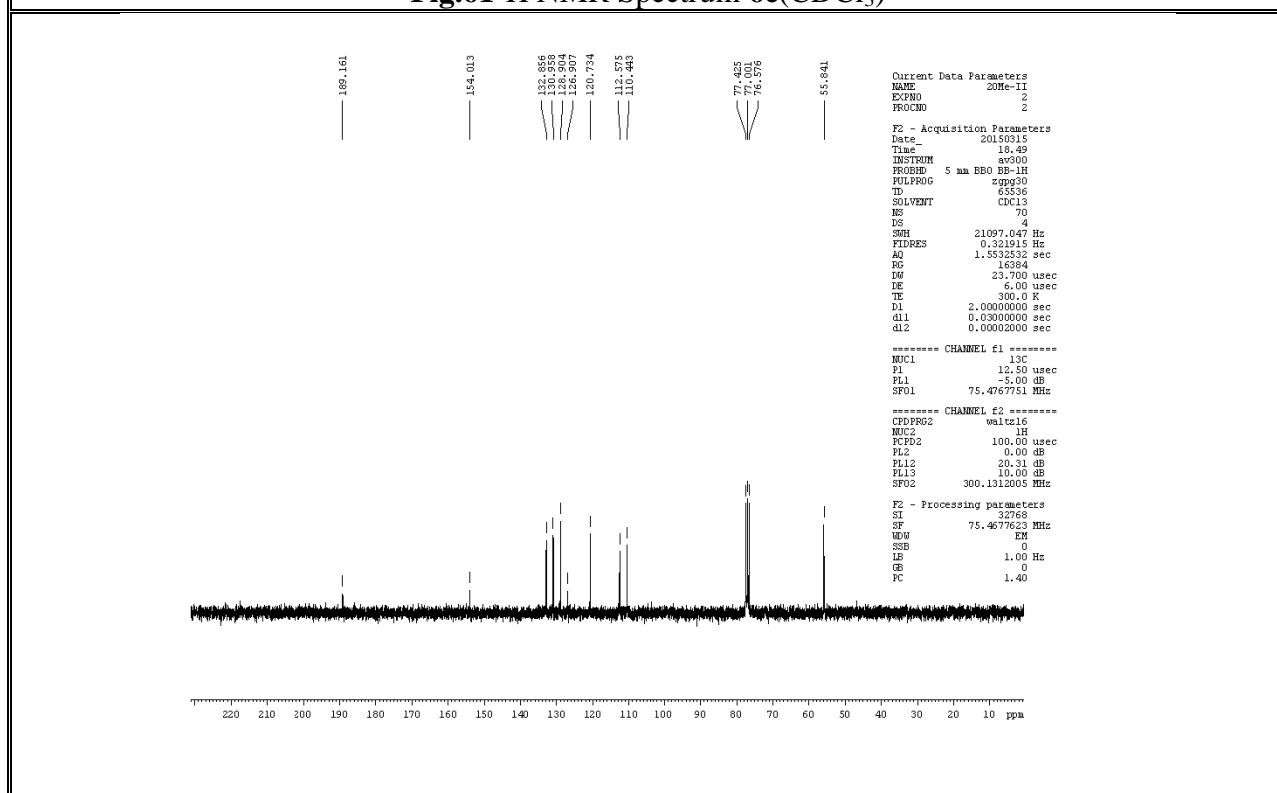


Fig.62 <sup>13</sup>C NMR Spectrum of 6e(CDCl<sub>3</sub>)