

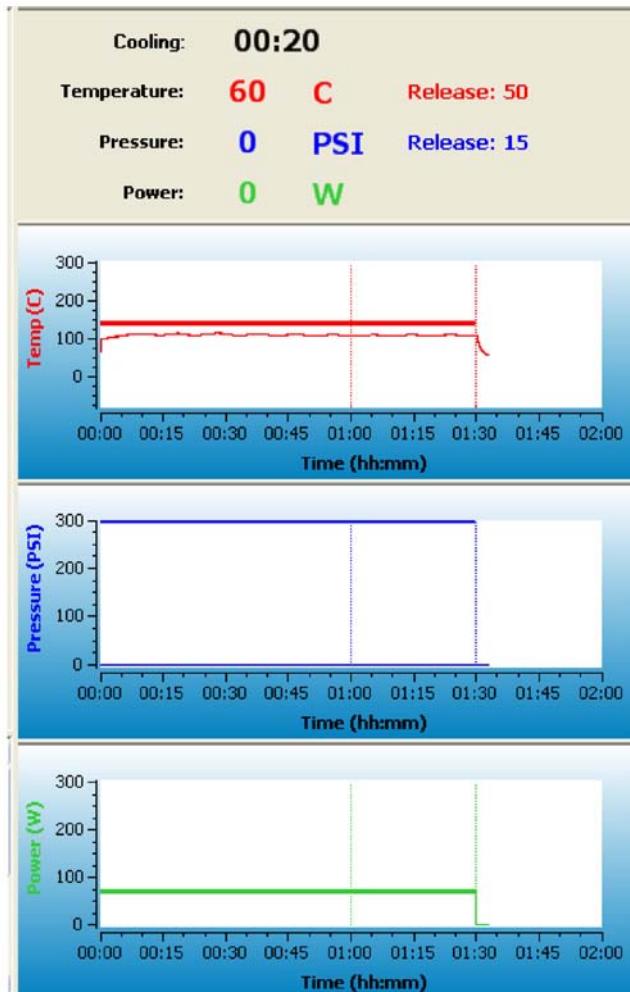
## Microwave-Assisted Synthesis of Nitroketene N,S-Arylaminoacetals

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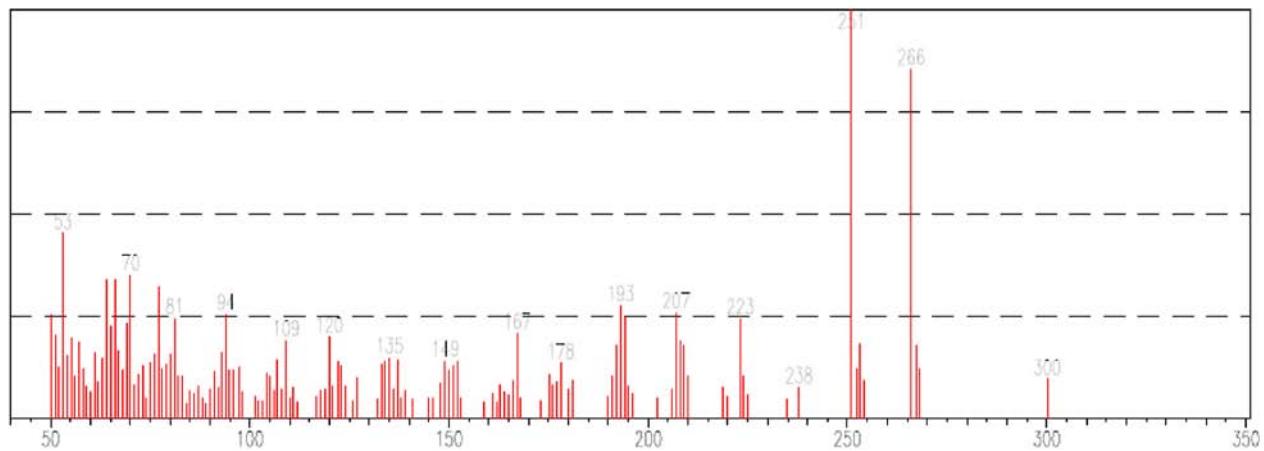
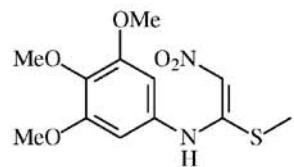
### Experimental

<sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a Bruker ARX-400 (400 and 100 MHz respectively). The IR spectra refer to tablets in KBr and were measured on a Bomem M102 spectrometer. Mass Spectra were recorded on a Shimadzu GCMS-QP5000.

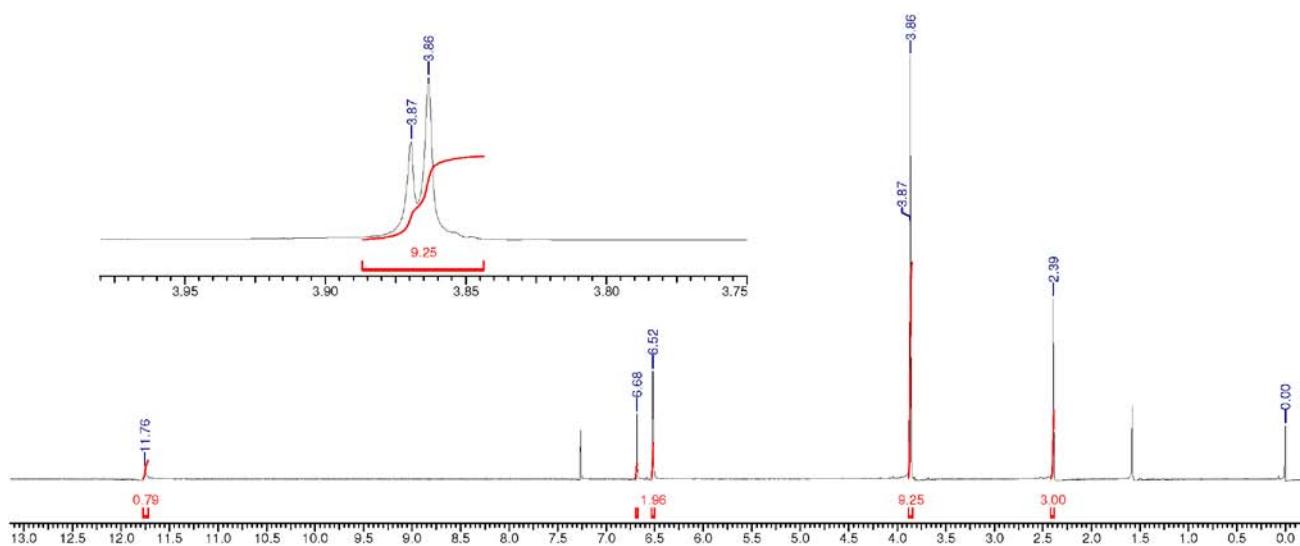


**Figure S1.** Representative graphic of the reaction of aniline with nitroethene **1** irradiated during 90 min in a CEM Discovery® focused microwave oven at 110 °C and 70 W.

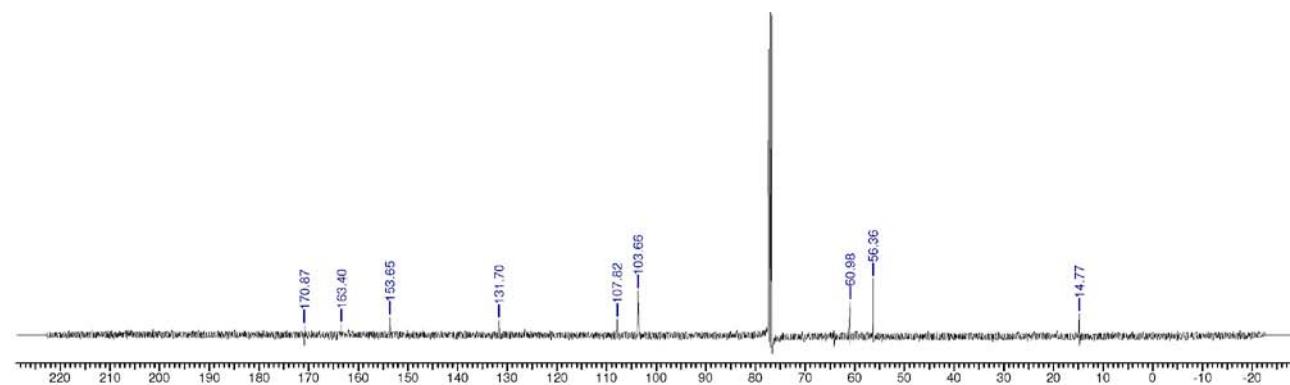
\*e-mail: agcorreia@ufscar.br

*3,4,5-Trimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3a)*

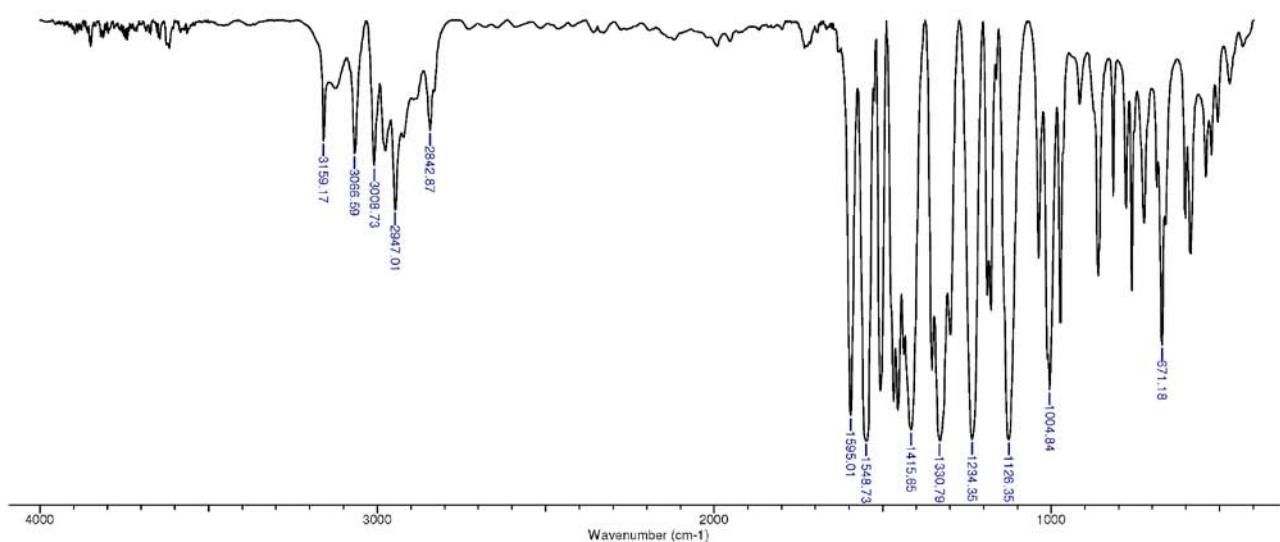
**Figure S2.** GC-MS of *3,4,5-Trimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3a)*.



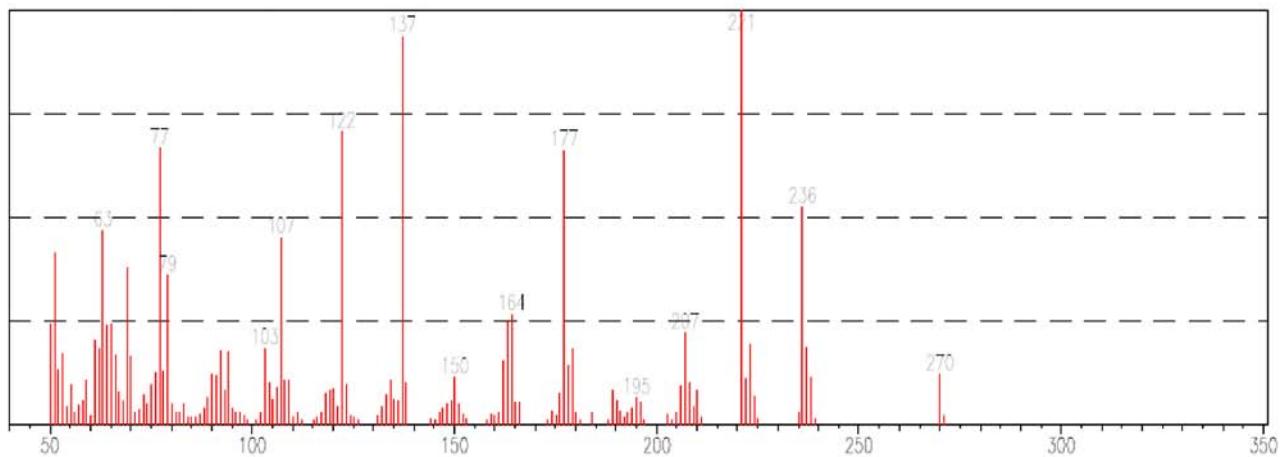
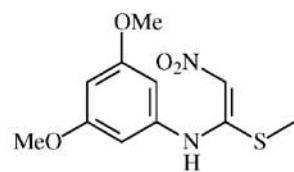
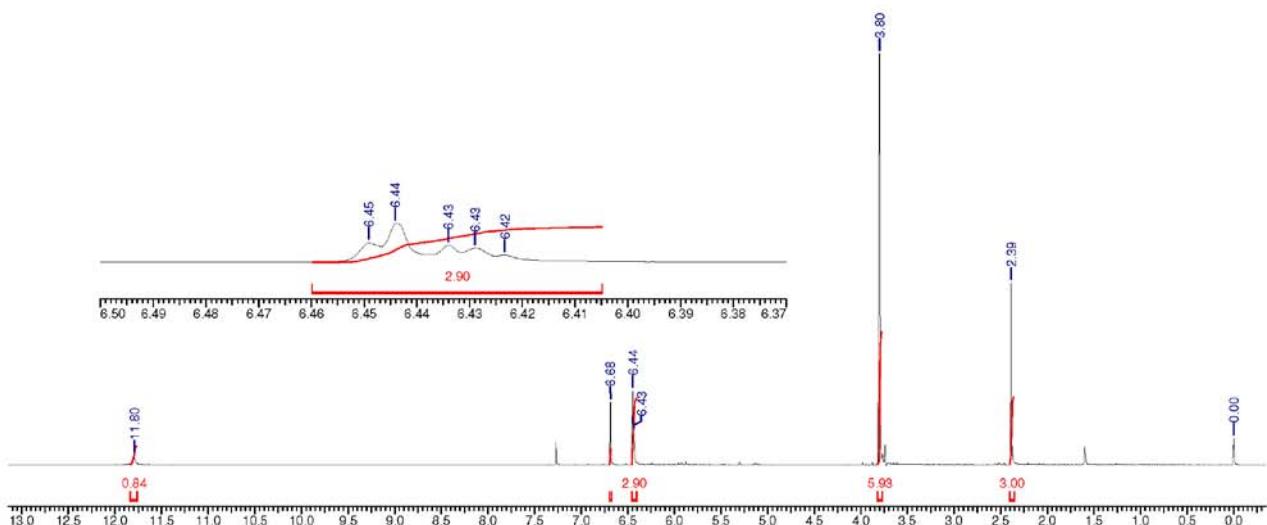
**Figure S3.** <sup>1</sup>H NMR of *3,4,5-Trimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3a)*.

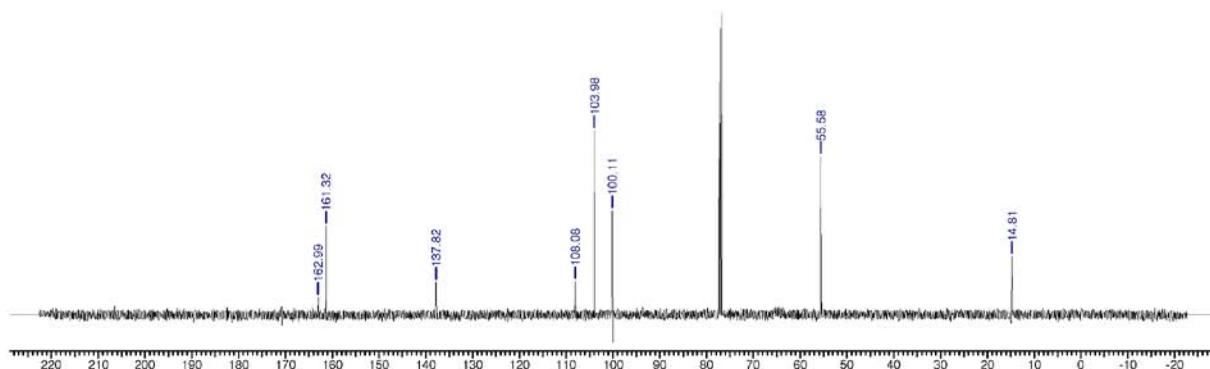


**Figure S4.** <sup>13</sup>C NMR of 3,4,5-Trimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3a**).

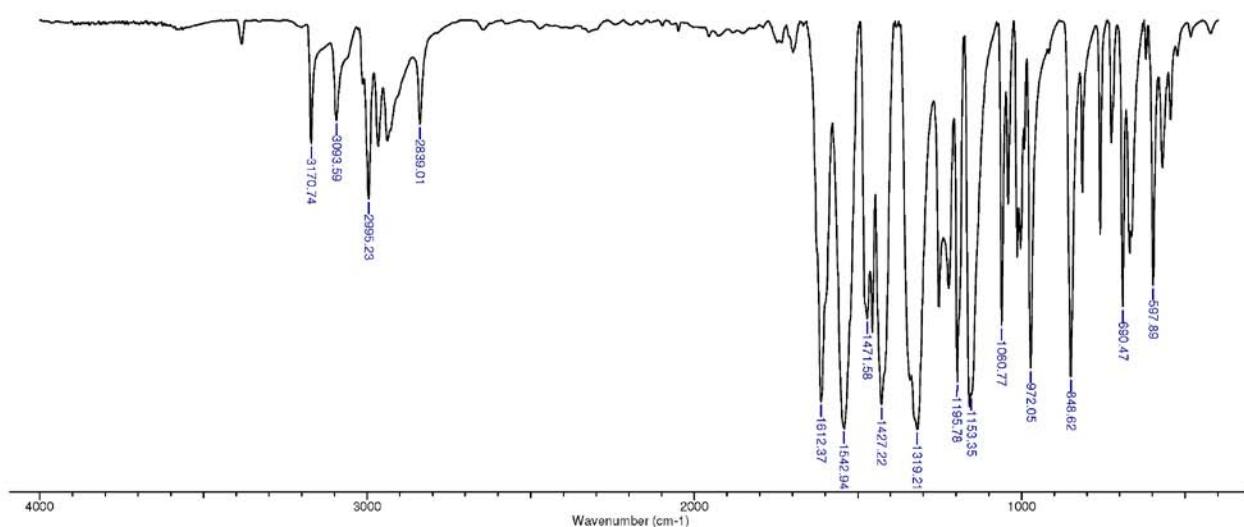


**Figure S5.** IR of 3,4,5-Trimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3a**)

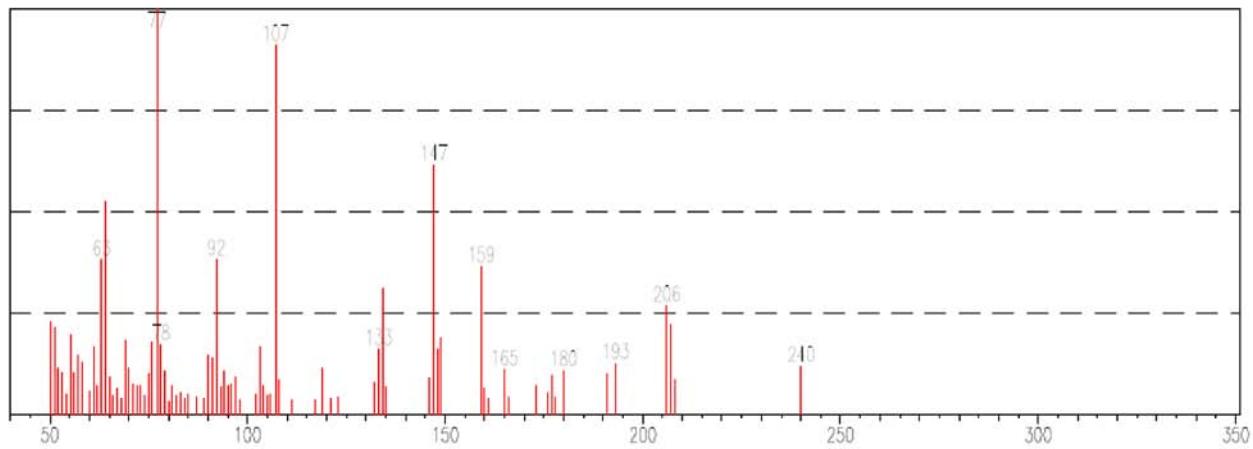
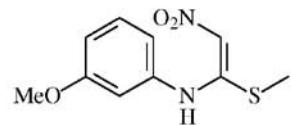
**3,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3b)****Figure S6.** GC-MS of 3,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3b**).**Figure S7.** <sup>1</sup>H NMR of 3,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3b**)



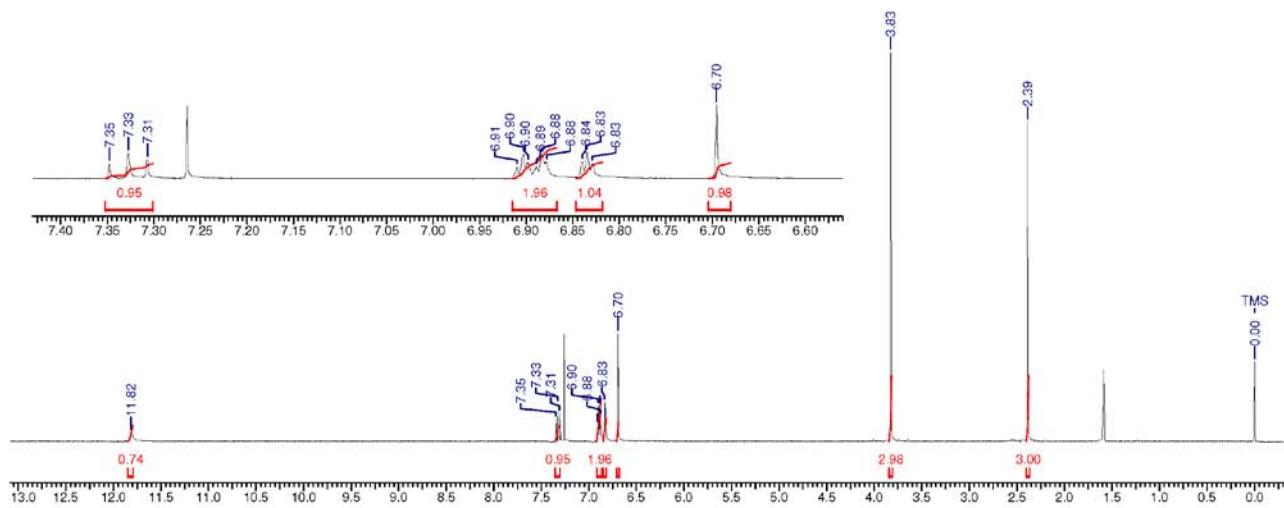
**Figure S8.** <sup>13</sup>C NMR of 3,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3b**).



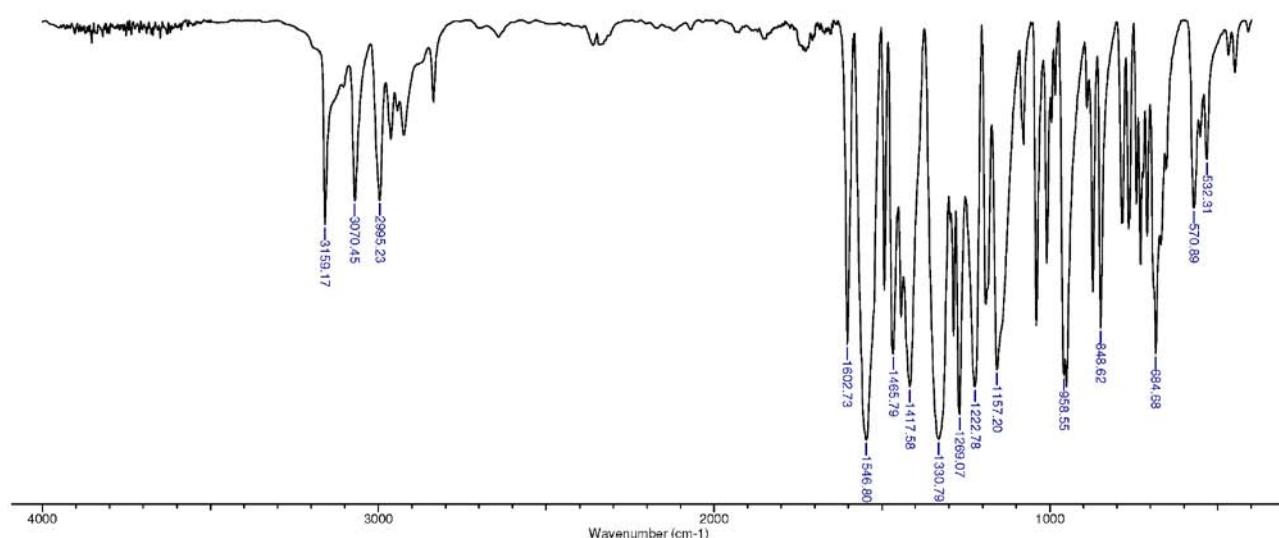
**Figure S9.** IR of 3,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3b**)

*3-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3c)*

**Figure S10.** GC-MS of *3-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3c)*.

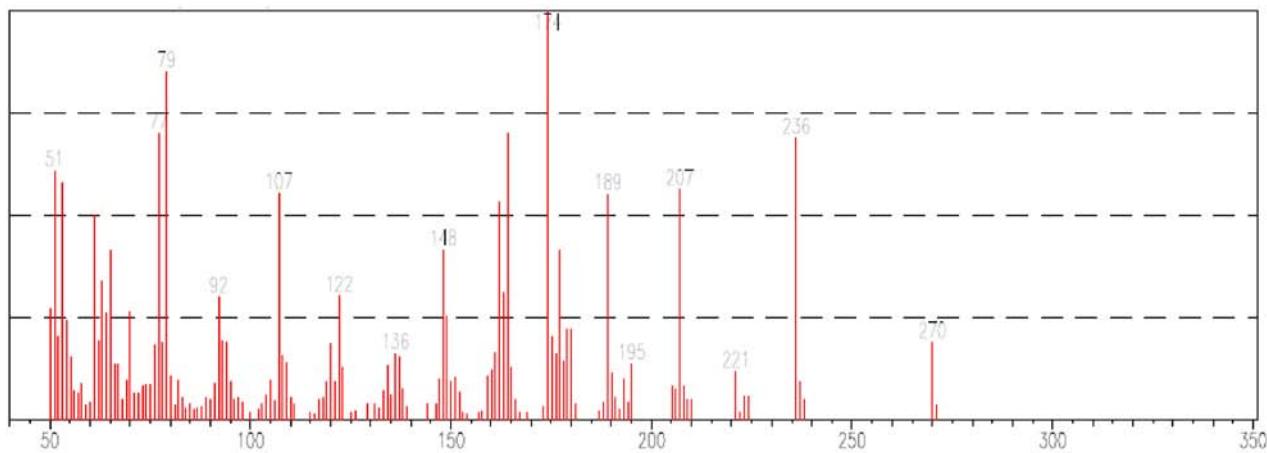
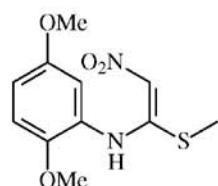


**Figure S11.** <sup>1</sup>H NMR of *3-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3c)*.



**Figure S12.** IR of 3-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3c**).

**2,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3d**)**



**Figure S13.** GC-MS of 2,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3d**).

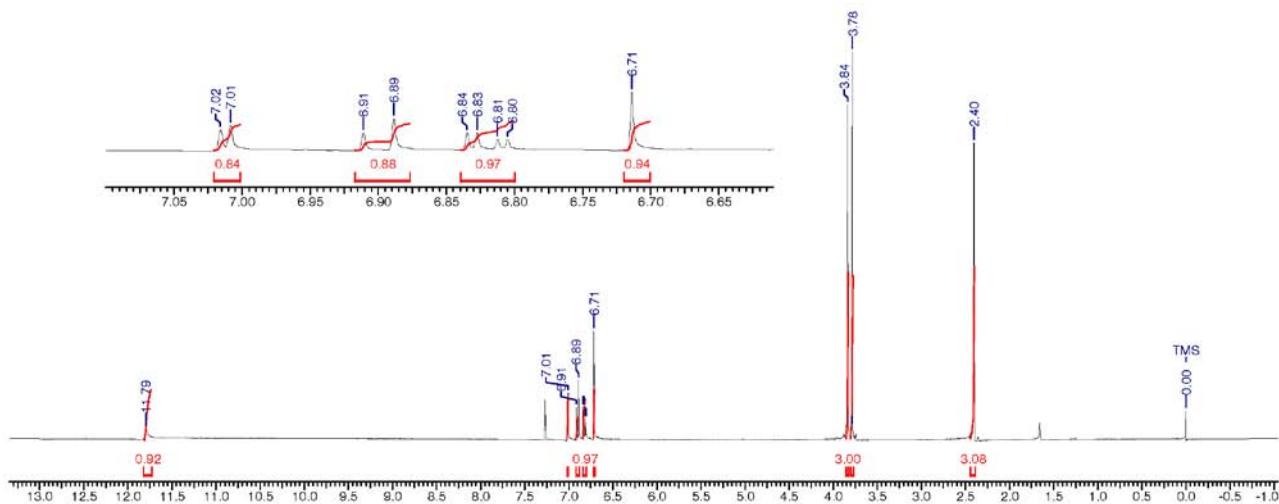


Figure S14. <sup>1</sup>H NMR of 2,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3d).

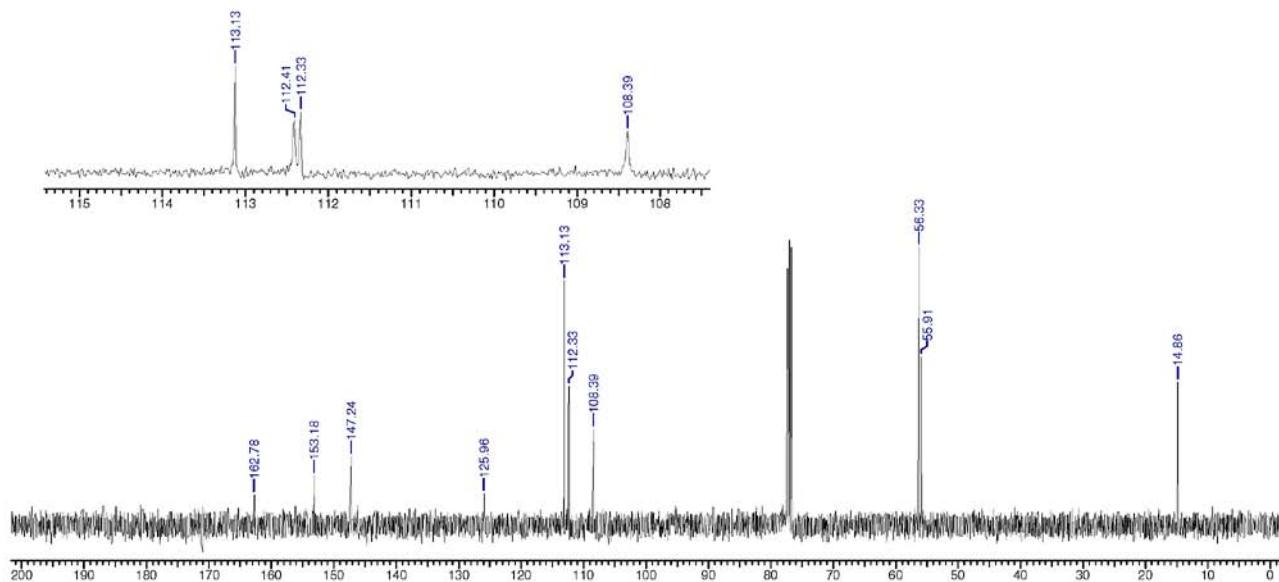
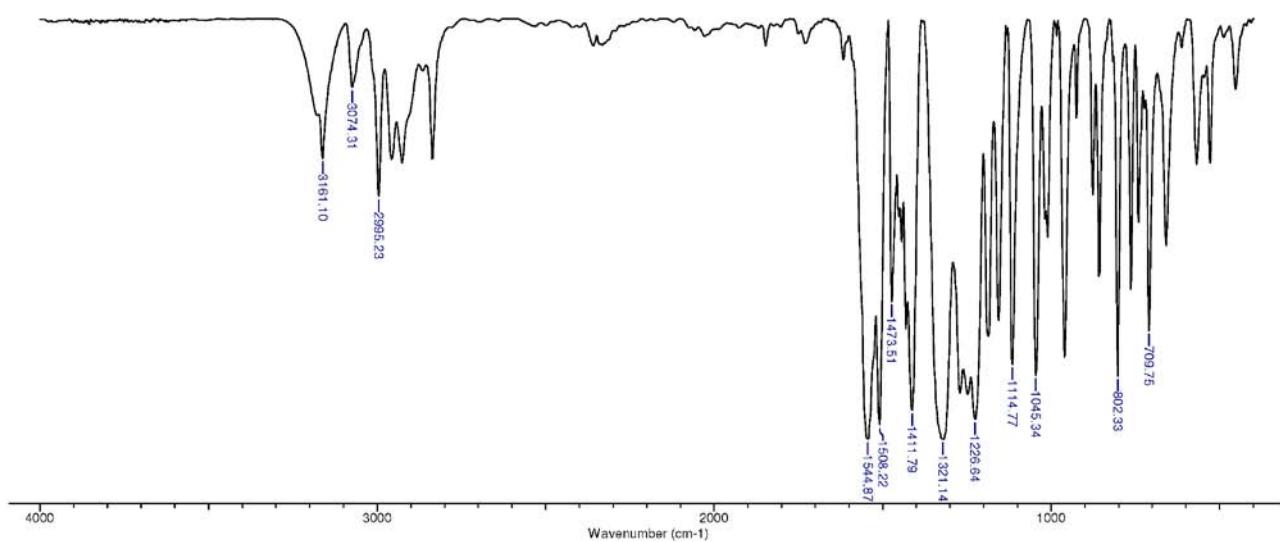
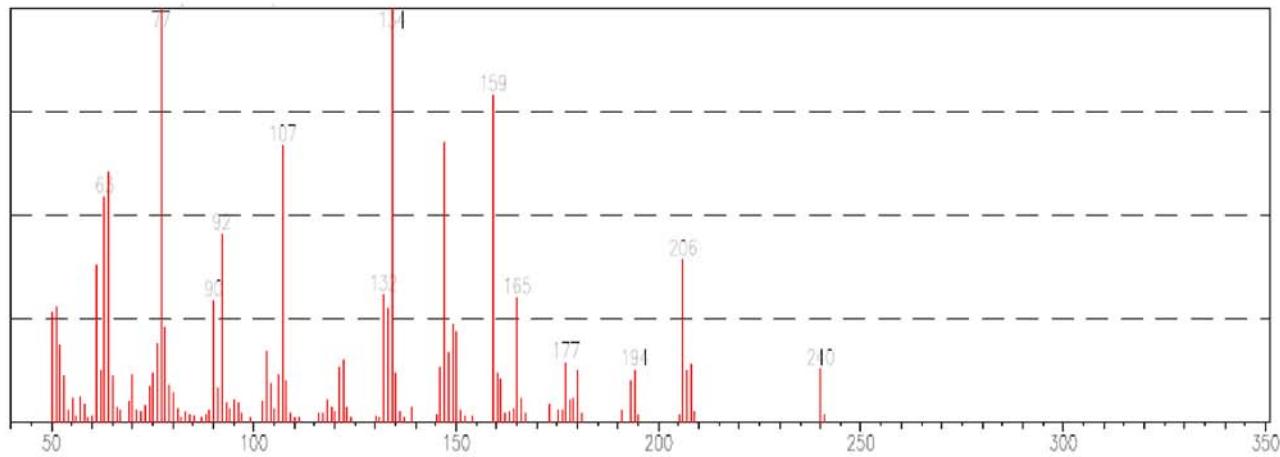
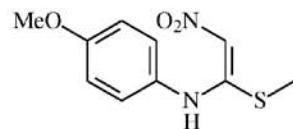


Figure S15. <sup>13</sup>C NMR of 2,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3d).



**Figure S16.** IR of 2,5-Dimethoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3d**).

*4-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3e**)*



**Figure S17.** GC-MS of 4-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3e**).

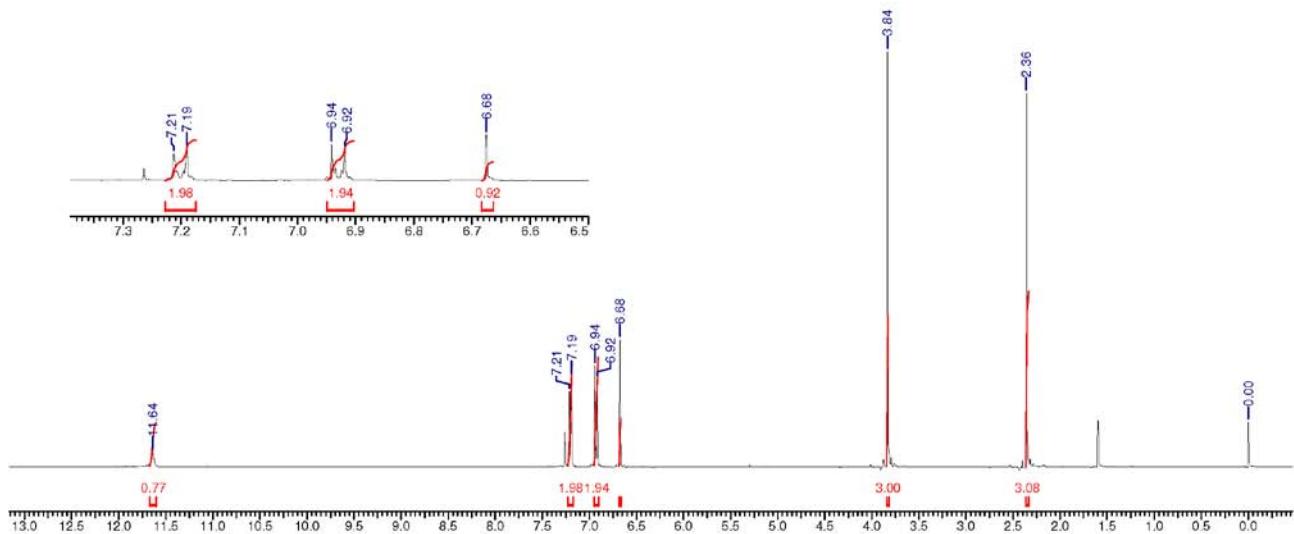


Figure S18.  $^1\text{H}$  NMR of 4-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3e**).

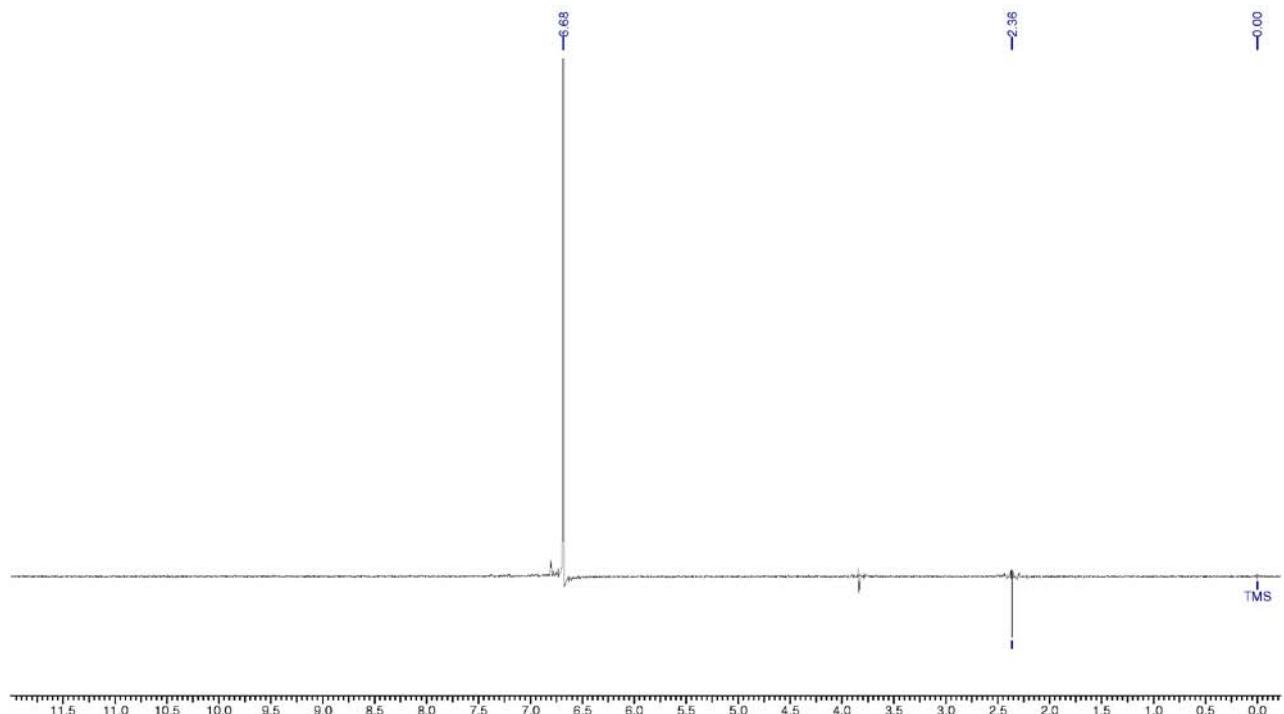
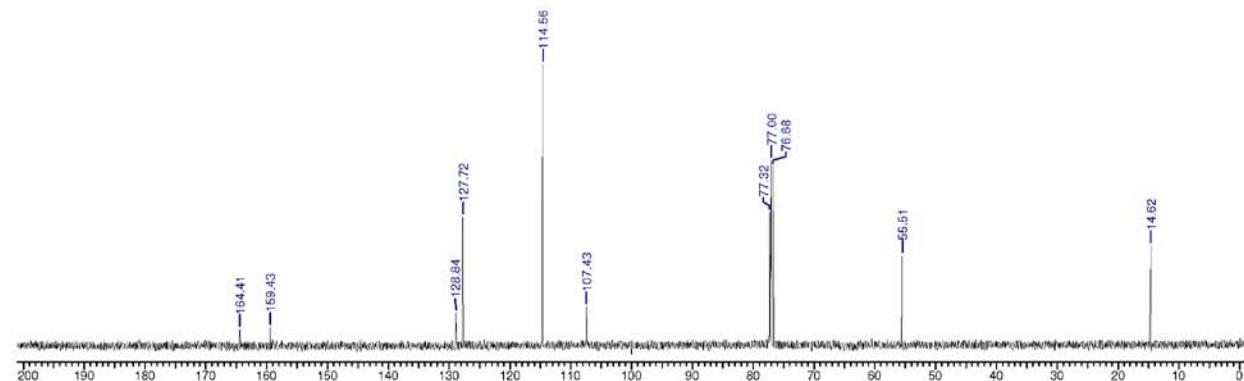
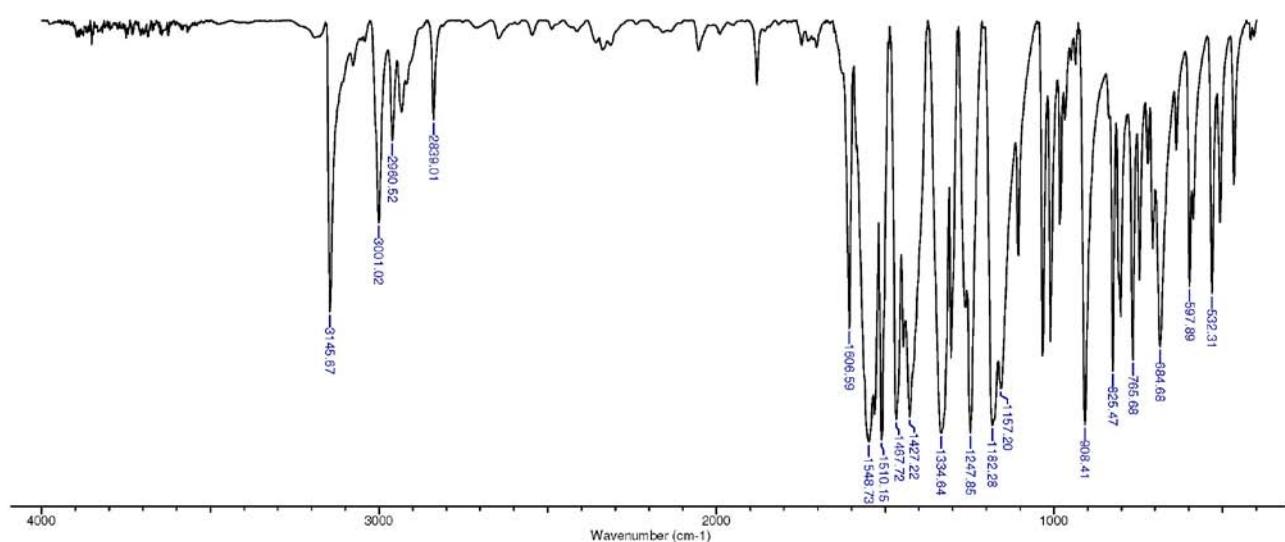


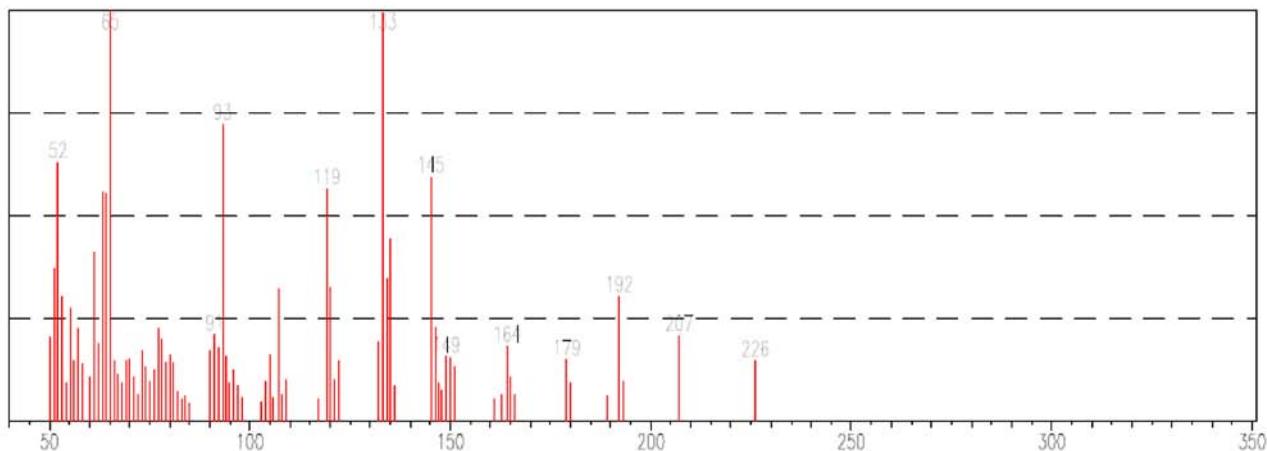
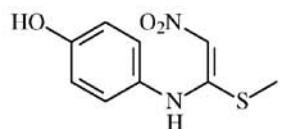
Figure S19.  $^1\text{H}$  NMR- NOE of 4-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3e**).



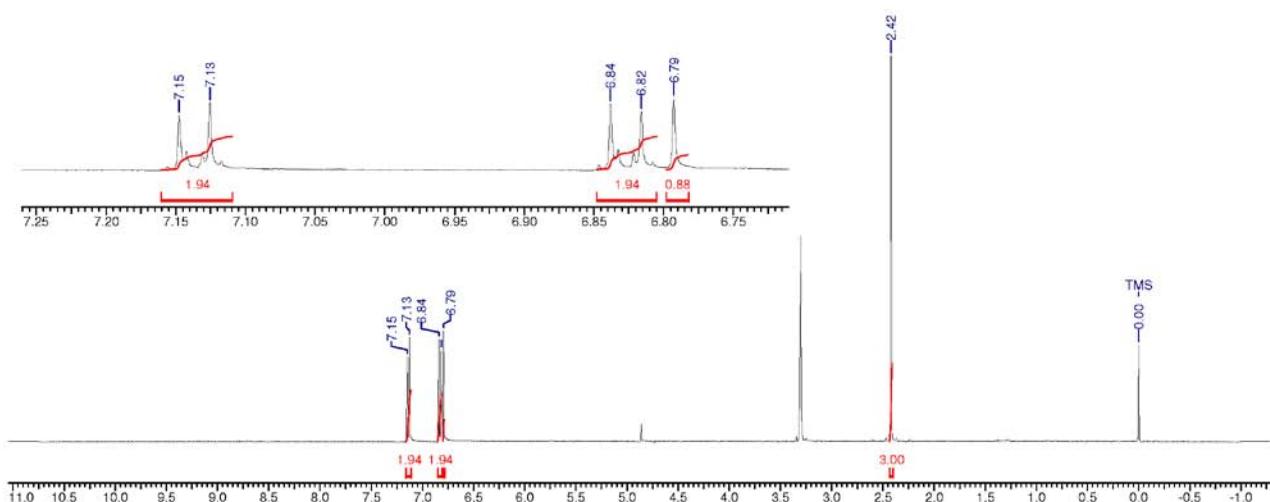
**Figure S20.** <sup>13</sup>C NMR of 4-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3e).



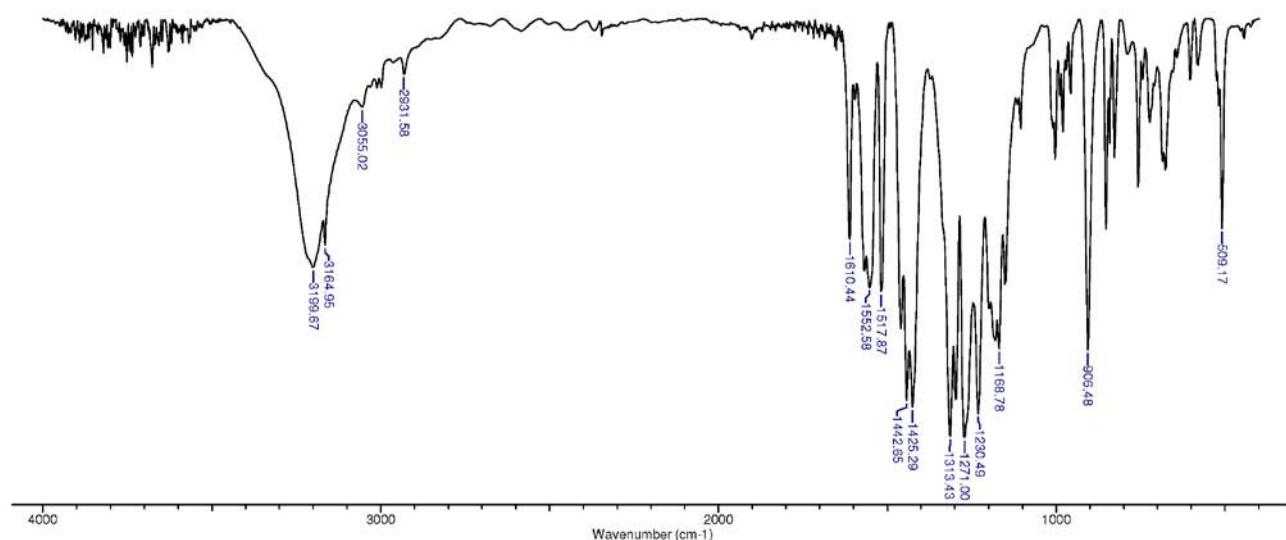
**Figure S21.** IR of 4-Methoxy-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3e).

**4-[[1-(methylthio)-2-nitroethenyl]amino]-phenol (**3f**)**

**Figure S22.** GC-MS of 4-[[1-(methylthio)-2-nitroethenyl]amino]-phenol (**3f**).

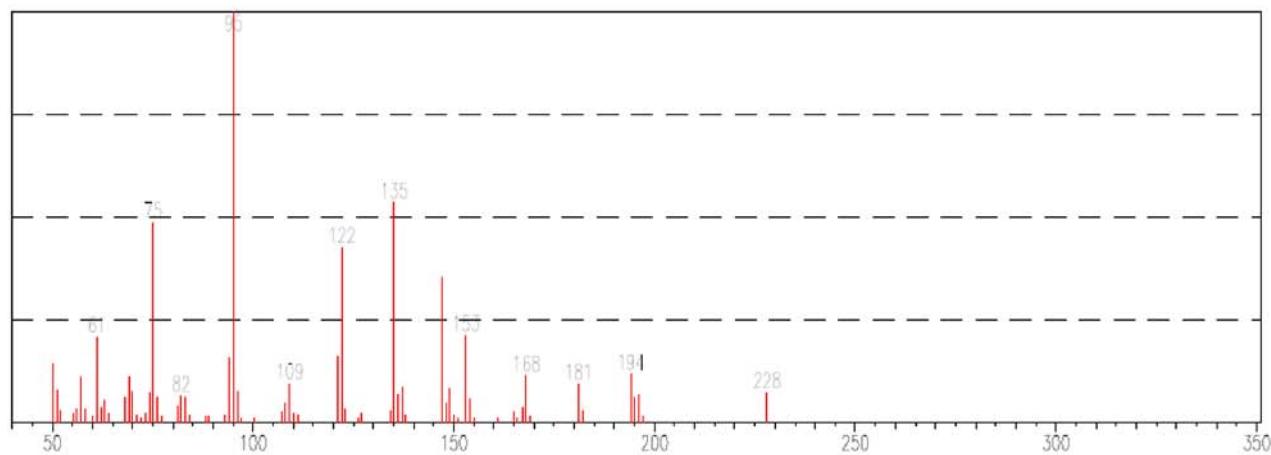
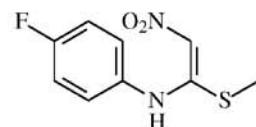


**Figure S23.** <sup>1</sup>H NMR of 4-[[1-(methylthio)-2-nitroethenyl]amino]-phenol (**3f**).

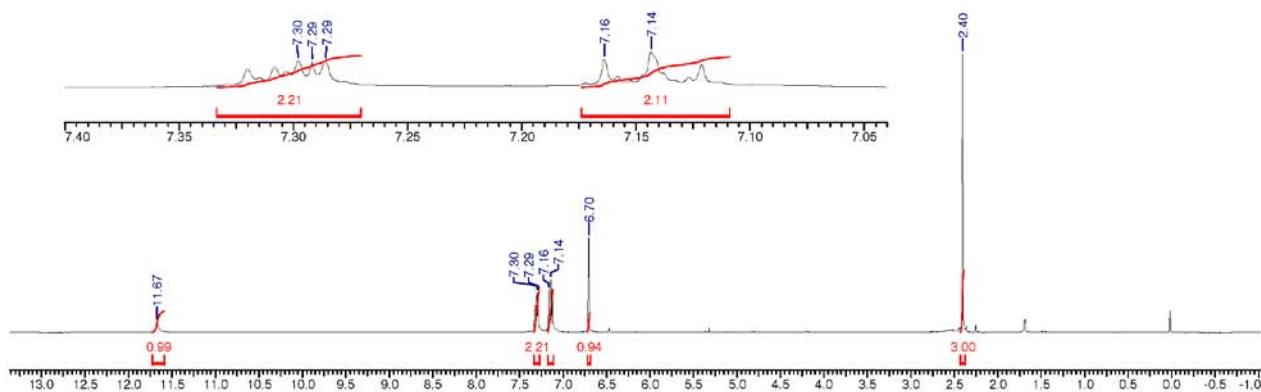


**Figure S24.** IR of 4-[{1-(methylthio)-2-nitroethenyl}amino]-phenol (**3f**).

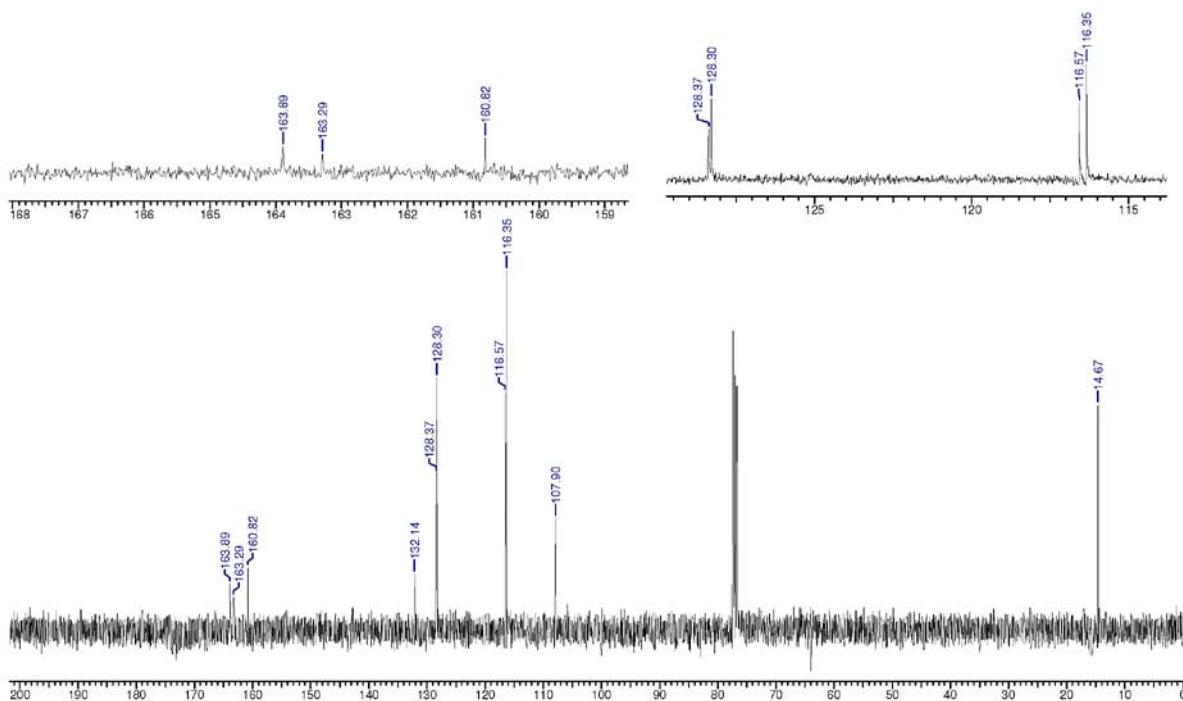
**4-Fluoro-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3h**)**



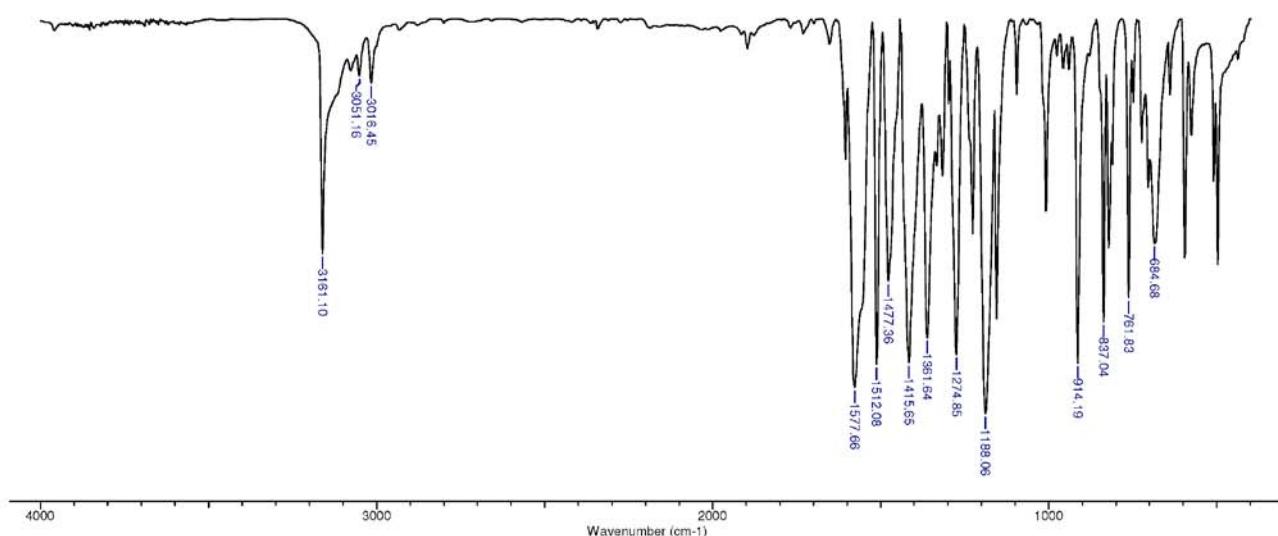
**Figure S25.** GC-MS of 4-Fluoro-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3h**).



**Figure S26.** <sup>1</sup>H NMR of 4-Fluoro-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3h**).

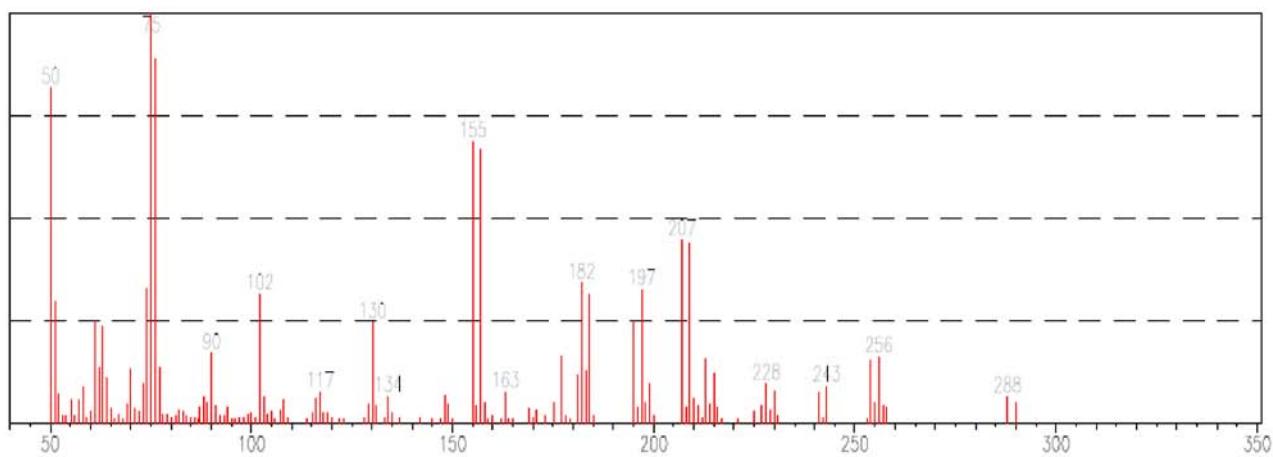
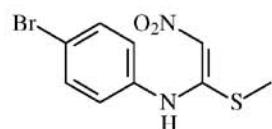


**Figure S27.** <sup>13</sup>C NMR of 4-Fluoro-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3h**).

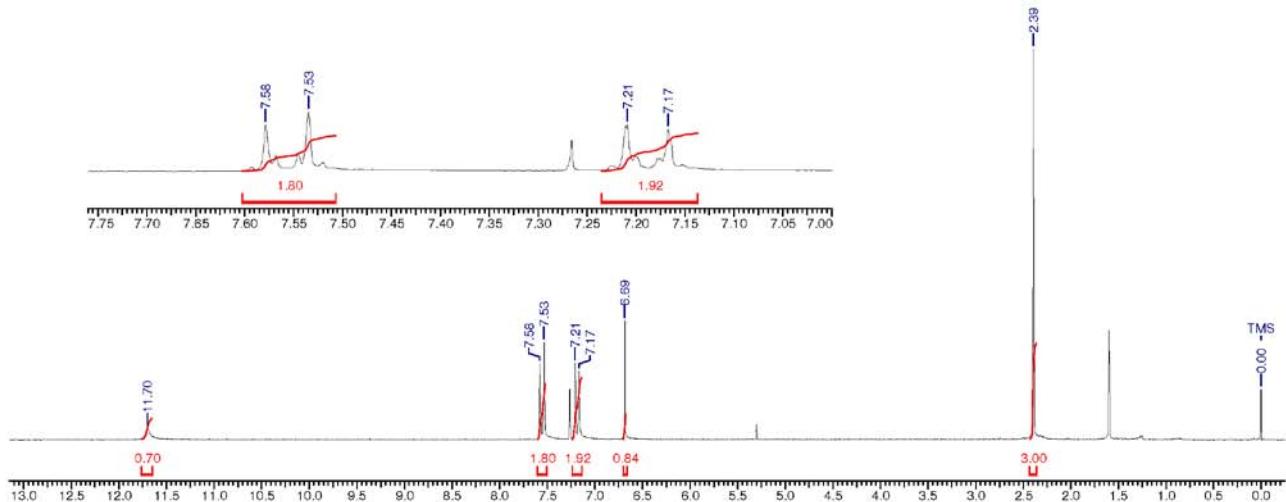


**Figure S28.** IR of 4-Fluoro-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3h**).

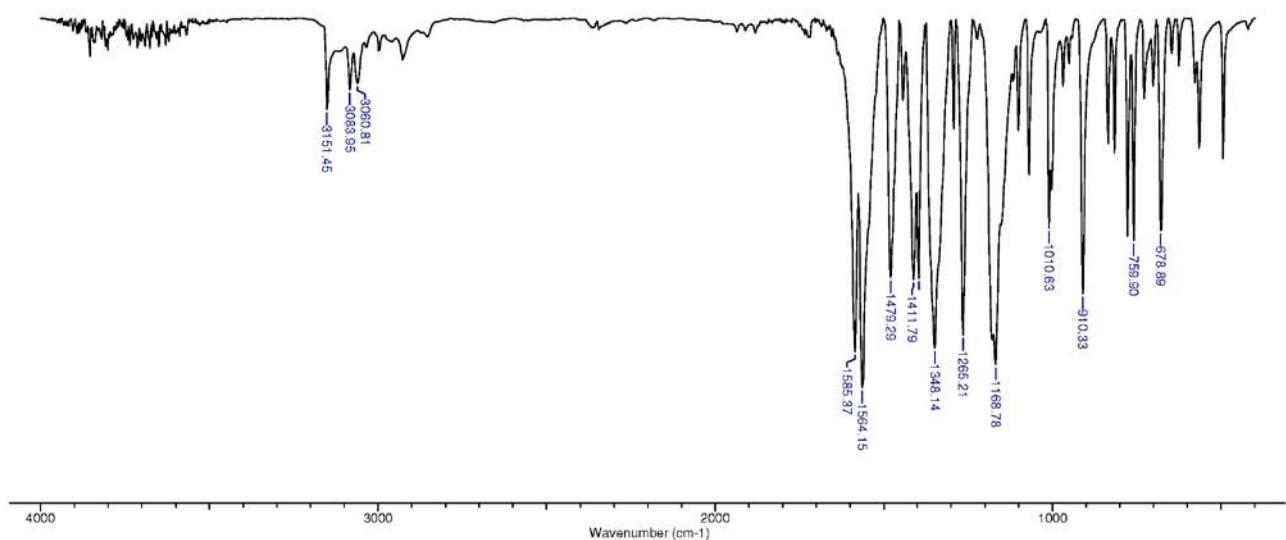
*4-Bromo-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3j**)*



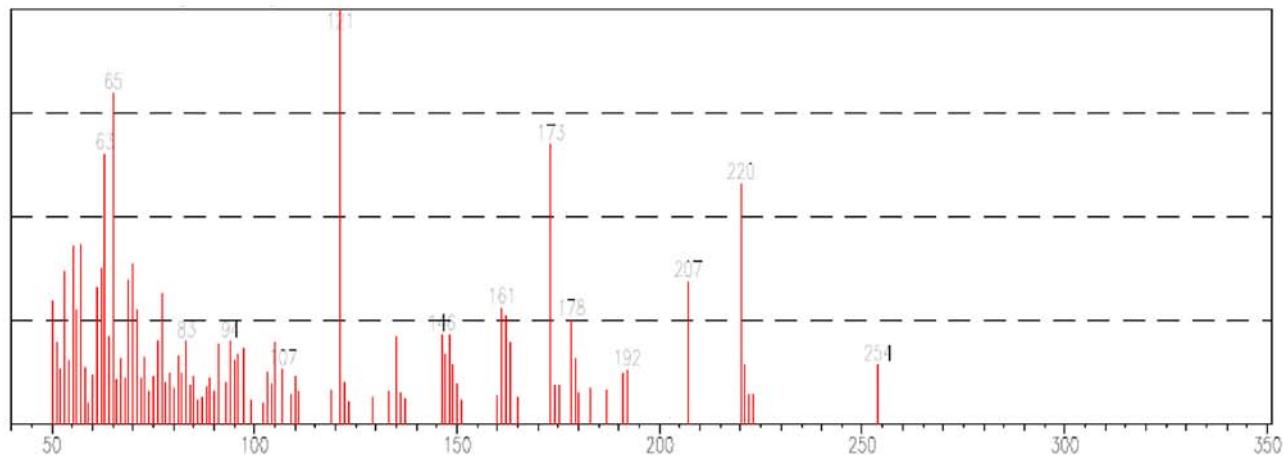
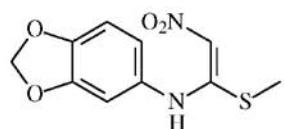
**Figure S29.** GC-MS of 4-Bromo-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3j**).



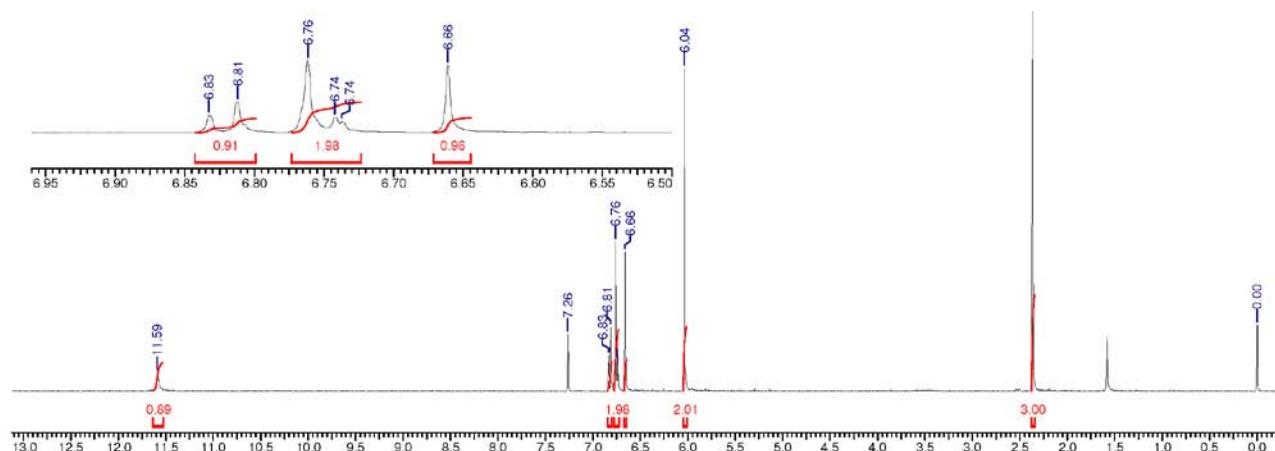
**Figure S30.** <sup>1</sup>H NMR of 4-Bromo-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3j**).



**Figure S31.** IR of 4-Bromo-N-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3j**).

*N-[1-(methylthio)-2-nitroethenyl]-1,3-benzodioxol-5-amine (3I)*

**Figure S32.** GC-MS of *N*-[1-(methylthio)-2-nitroethenyl]-1,3-benzodioxol-5-amine (**3I**).



**Figure S33.** <sup>1</sup>H NMR of *N*-[1-(methylthio)-2-nitroethenyl]-1,3-benzodioxol-5-amine (**3I**).

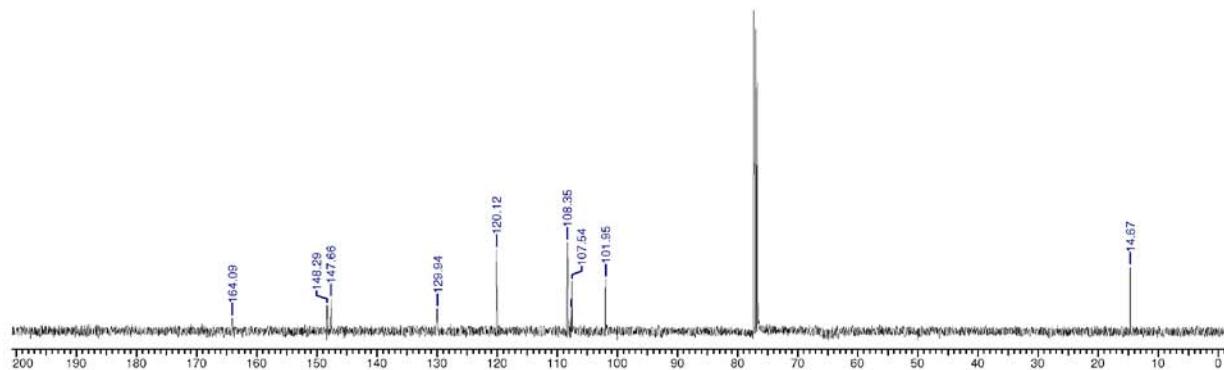


Figure S34.  $^{13}\text{C}$  NMR of *N*-[1-(methylthio)-2-nitroethenyl]-1,3-benzodioxol-5-amine (**3l**).

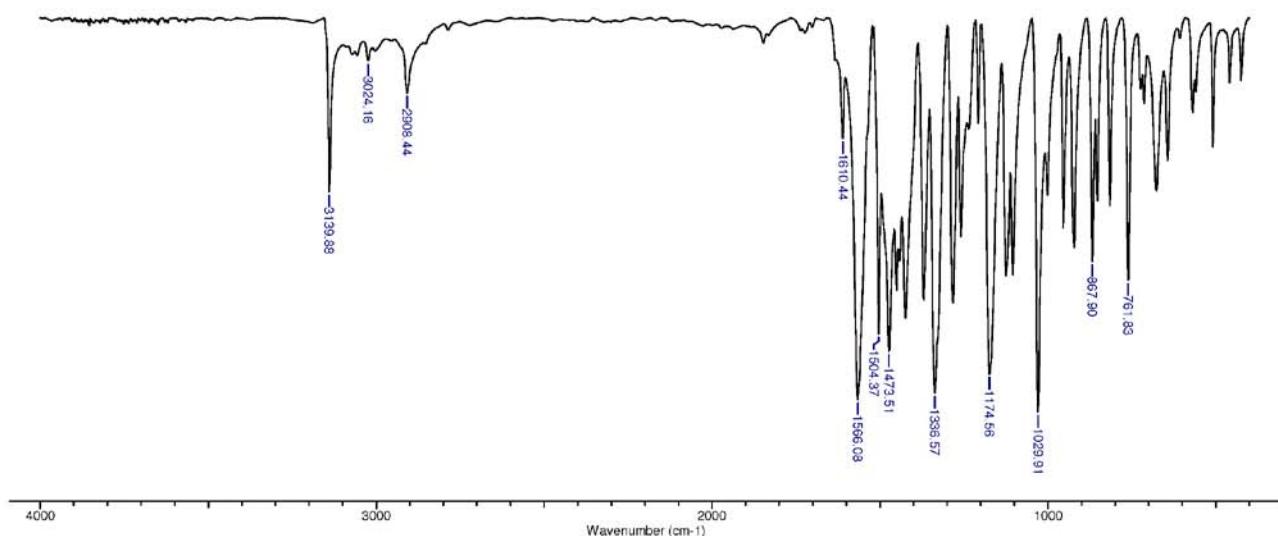
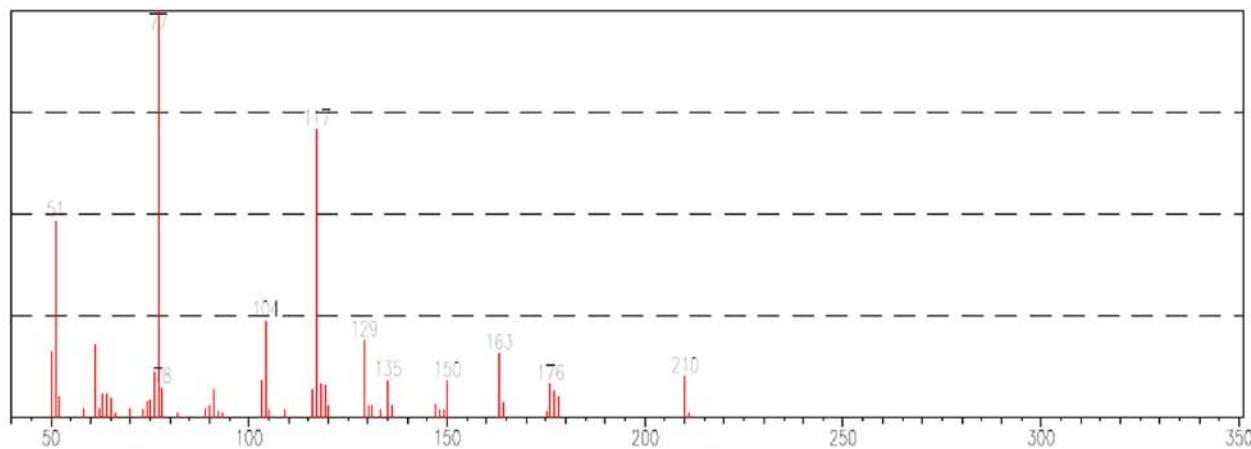
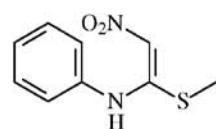
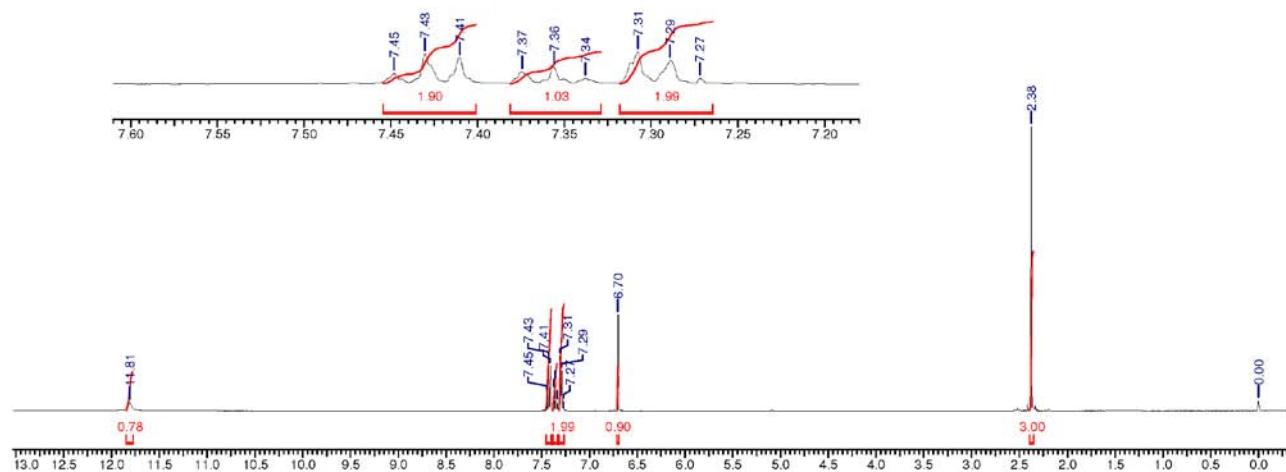


Figure S35. IR of *N*-[1-(methylthio)-2-nitroethenyl]-1,3-benzodioxol-5-amine (**3l**).

*N-[1-(methylthio)-2-nitroethenyl]-benzenamine (3m)*

**Figure S36.** GC-MS of *N*-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3m**).



**Figure S37.**  $^1\text{H}$  NMR of *N*-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3m**).

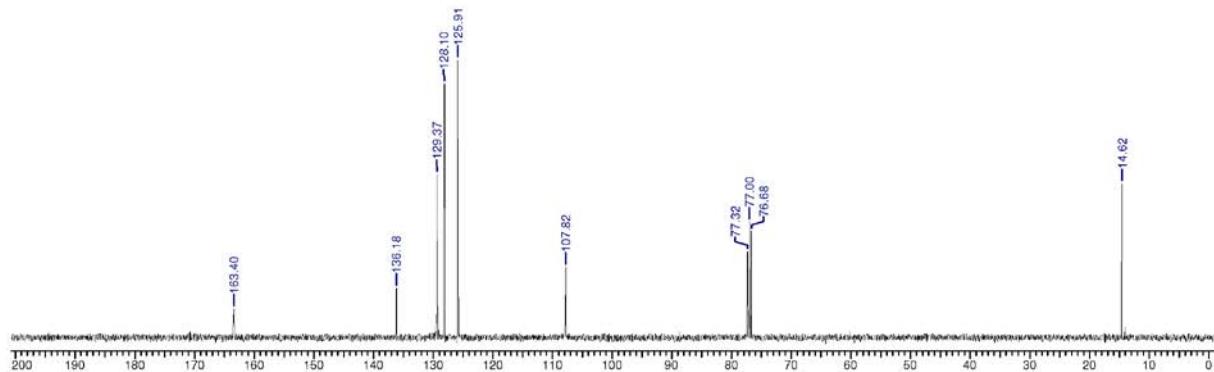


Figure S38. <sup>13</sup>C NMR of *N*-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3m**).

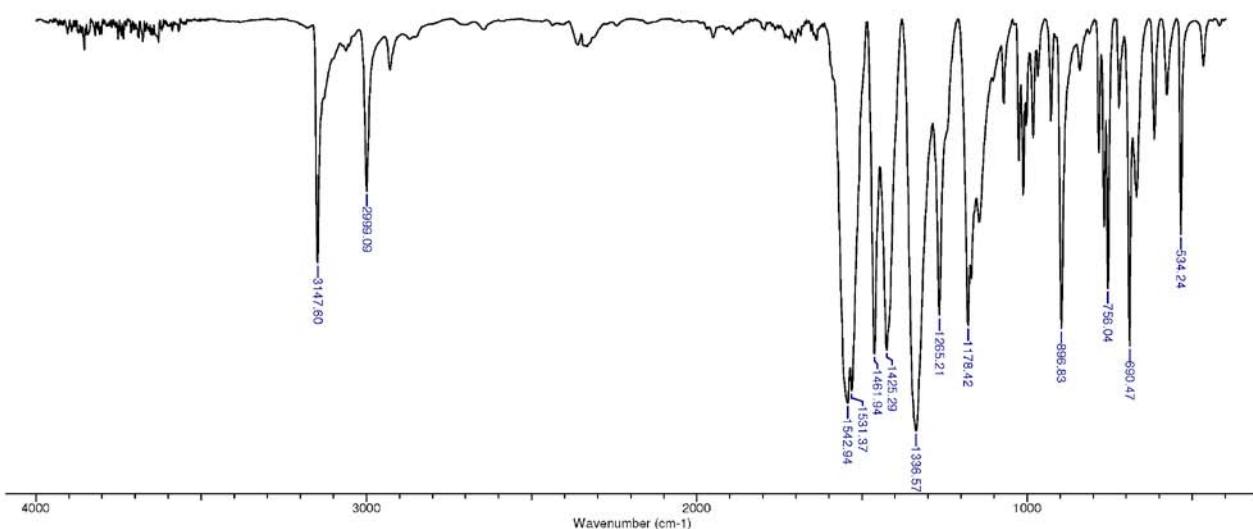


Figure S39. IR of *N*-[1-(methylthio)-2-nitroethenyl]-benzenamine (**3m**).