

**PEPPSI SONO SP<sup>2</sup>**: A new highly efficient ligand-free catalyst system for the synthesis of tri-substituted triazine derivatives *via* Suzuki-Miyaura and Sonogashira coupling reactions under green approach

Motakatla Venkata Krishna Reddy,<sup>a</sup> Peddiahgari Vasu Govardhana Reddy,<sup>\*a</sup> Cirandur Suresh Reddy<sup>b</sup>

<sup>a</sup>*Department of Chemistry, Yogi Vemana University, Kadapa-516003, Andhra Pradesh, India.*

<sup>b</sup>*Department of Chemistry, Sri Venkateswara University, Tirupati-517502, Andhra Pradesh, India.*

\*Corresponding Author Phone: +91-9985200965; fax: +91-8562225419

E-mail: [pvgr@yogivemanauniversity.ac.in](mailto:pvgr@yogivemanauniversity.ac.in)

### Supporting Information

<b>I. General Experimental Section</b>	<b>S2</b>
<b>II. Characteristic Data</b>	<b>S2-S11</b>
<b>III. Copies of (<sup>1</sup>H &amp; <sup>13</sup>C) NMR and HRMS Spectra</b>	<b>S12-S28</b>

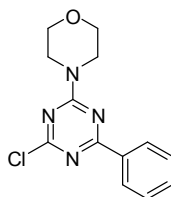
## I. General Experimental Section

All reactions were performed under non-inert atmosphere in solvents such as acetone, acetonitrile, dimethyl sulfoxide, 1, 4-dioxane, ethanol and water. Every reaction was monitored by TLC analysis using Merck silica gel 60 F<sub>254</sub> plates with fluorescent indicator (254 nm) and visualized with UV lamp. All commercially available reagents such as cyanuric chloride, morpholine, boronic acids, acetylenes, bases, palladium reagents and PEPSI-SONO-SP<sup>2</sup> were procured from Aldrich, Acros organics and Merck Scientific companies and used as received without further purification. Analytical grade hexane and ethyl acetate were used as such for purification through column chromatography.

Melting points of the synthesized compounds are uncorrected. Infrared spectra of the title compounds were recorded on Bruker Alpha-Eco ATR-FTIR (Attenuated total reflection-Fourier transform infrared) interferometer with single reflection sampling module equipped with KBr crystal. <sup>1</sup>H and <sup>13</sup>C-NMR spectra were recorded on Bruker top spin 400 and 100 MHz spectrometers in CDCl<sub>3</sub> solvent using TMS as an internal standard. Chemical shifts (δ) are reported in ppm. Multiplicities in the <sup>1</sup>H-NMR spectra are described as: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet; Coupling constants are reported in Hz. High Resolution Mass Spectra were recorded on micrOTOF-Q II Bruker compass mass spectrometer operating 70 eV.

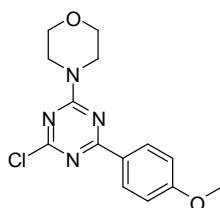
## II. Characteristic Data

### 4-(4-Chloro-6-phenyl-1,3,5-triazin-2-yl)morpholine (3a):



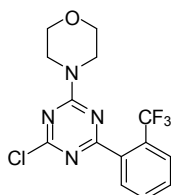
Colorless solid, m. p. 87-89 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ (ppm) 8.41-8.39 (m, 2H, Ar-H), 7.57-7.54 (m, 1H, Ar-H), 7.48-7.45 (m, 2H, Ar-H), 4.06 (t, *J* = 3.6 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.93 (t, *J* = 3.6 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.81-3.77 (m, 4H, -CH<sub>2</sub>-O-CH<sub>2</sub>-).

**4-(4-Chloro-6-(4-methoxyphenyl)-1,3,5-triazin-2-yl)morpholine (3b):**



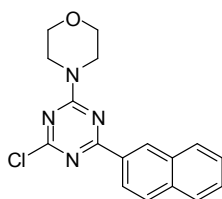
Colorless solid, m. p. 92 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ (ppm) 8.37 (d, *J* = 6.8 Hz, 2H, Ar-H), 6.96 (d, *J* = 11.6 Hz, 2H, Ar-H), 4.03 (t, *J* = 4.0 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.90 (t, *J* = 3.6 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.88 (s, 3H, O-CH<sub>3</sub>), 3.81-3.77 (m, 4H, -CH<sub>2</sub>-O-CH<sub>2</sub>-).

**4-(4-Chloro-6-(2-(trifluoromethyl)phenyl)-1,3,5-triazin-2-yl)morpholine (3c):**



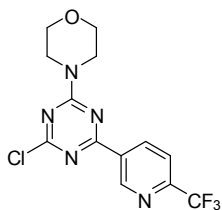
Colorless solid, m. p. 95-97 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ (ppm) 7.85 (d, *J* = 7.6 Hz, 1H, Ar-H), 7.65 (t, *J* = 8.0 Hz, 2H, Ar-H), 7.57 (d, *J* = 7.6 Hz, 1H, Ar-H), 3.97 (t, *J* = 4.0 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.88 (t, *J* = 4.0 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.78-3.75 (m, 4H, -CH<sub>2</sub>-O-CH<sub>2</sub>-).

**4-(4-Chloro-6-naphthalen-2-yl)-1,3,5-triazin-2-yl)morpholine (3d):**



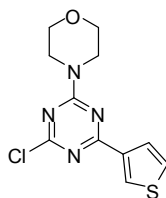
Colorless solid, m. p. 97-99 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>): δ (ppm) 9.01 (s, 1H, Ar-H), 8.49 (d, *J* = 8.4 Hz, 1H, Ar-H), 8.02-7.91 (m, 3H, Ar-H), 7.57 (t, *J* = 7.2 Hz, 2H, Ar-H), 4.06 (t, *J* = 4.0 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.96 (t, *J* = 3.6 Hz, 2H, -CH<sub>2</sub>-N-CH<sub>2</sub>-), 3.85-3.79 (m, 4H, -CH<sub>2</sub>-O-CH<sub>2</sub>-).

**4-(4-Chloro-6-(6-trifluoromethyl)pyridin-3-yl)-1,3,5-triazin-2-yl)morpholine (3e):**



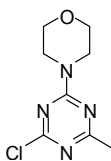
Colorless solid, m. p. 98 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 9.63 (s, 1H, Ar-H), 8.82 (d,  $J = 6.4$ , 1H, Ar-H), 7.80 (d,  $J = 6.4$ , 1H, Ar-H), 4.04 (t,  $J = 4.0$  Hz, 2H,  $-\text{CH}_2-\text{N}-\text{CH}_2-$ ), 3.96 (t,  $J = 4.0$  Hz, 2H,  $-\text{CH}_2-\text{N}-\text{CH}_2-$ ), 3.83-3.79 (m, 4H,  $-\text{CH}_2-\text{O}-\text{CH}_2-$ ).

**4-(4-Chloro-6-(thiophen-3-yl)-1,3,5-triazin-2-yl)morpholine (3f):**



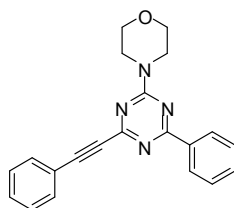
Colorless solid, m. p. 91 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.4 (s, 1H, Ar-H), 7.81 (d,  $J = 4$ , 1H, Ar-H), 7.35 (d,  $J = 2.4$ , 1H, Ar-H), 4.00 (t,  $J = 3.6$  Hz, 2H,  $-\text{CH}_2-\text{N}-\text{CH}_2-$ ), 3.91 (t,  $J = 3.6$  Hz, 2H,  $-\text{CH}_2-\text{N}-\text{CH}_2-$ ), 3.80-3.76 (m, 4H,  $-\text{CH}_2-\text{O}-\text{CH}_2-$ ).

**4-(4-Chloro-6-methyl-1,3,5-triazin-2-yl) morpholine (3g):**



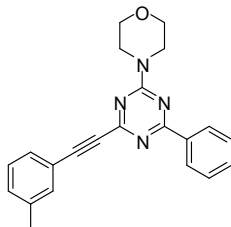
Colorless solid, m. p. 85 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 3.72 (t,  $J = 3.6$  Hz, 2H,  $-\text{CH}_2-\text{N}-\text{CH}_2-$ ), 3.63 (t,  $J = 3.6$  Hz, 2H,  $-\text{CH}_2-\text{N}-\text{CH}_2-$ ), 3.68-3.73 (m, 4H,  $-\text{CH}_2-\text{O}-\text{CH}_2-$ ), 1.3 (s, 3H, Ali- $\text{CH}_3$ ).

**4-(4-Phenyl-6-(phenyl ethynyl)-1,3,5-triazin-2-yl)morpholine (4a):**



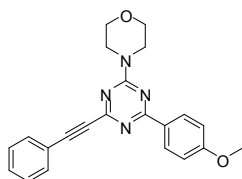
Pale brown solid, m. p. 125 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3059 (Ar-C-H), 2963 (Alk-C-H), 2221 ( $\text{—C}\equiv\text{C—}$ ), 1596 (Ar-C=C), 1272 (C-N), 1114 (C-O);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.47 (d,  $J = 7.6$  Hz, 2H, Ar-H), 7.70 (d,  $J = 7.2$  Hz, 2H, Ar-H), 7.56-7.52 (m, 1H, Ar-H), 7.50 (t,  $J = 7.2$  Hz, 2H, Ar-H), 7.43 (q,  $J = 7.6$  Hz, 3H, Ar-H), 4.03 (s, 4H,  $\text{—CH}_2\text{—N—CH}_2\text{—}$ ), 3.80 (s, 4H,  $\text{—CH}_2\text{—O—CH}_2\text{—}$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 171.16 (Ar-C), 164.28 (Ar-C), 160.43 (Ar-C), 135.92 (Ar-C), 132.81 (Ar-C), 132.22 (Ar-C), 129.88 (Ar-C), 128.74 (Ar-C), 128.42 (Ar-C), 128.41 (Ar-C), 121.23 (Ar-C), 88.56 ( $\text{—C}\equiv\text{C—}$ ), 87.36 ( $\text{—C}\equiv\text{C—}$ ), 66.75 ( $\text{—CH}_2\text{—O—CH}_2\text{—}$ ), 43.78 ( $\text{—CH}_2\text{—N—CH}_2\text{—}$ ); HRMS: Anal. calculated for  $\text{C}_{21}\text{H}_{19}\text{N}_4\text{O}$  (M+H: 343.1559); found: 343.1548.

**4-(4-Phenyl-6-(m-tolylethynyl)-1,3,5-triazin-2-yl)morpholine (4b):**



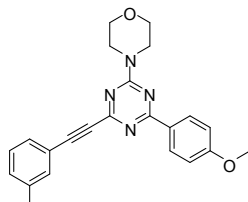
Colorless solid, m. p. 120 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3058 (Ar-C-H), 2957 (Alk-C-H), 2221 ( $\text{—C}\equiv\text{C—}$ ), 1586 (Ar-C=C), 1268 (C-N), 1113 (C-O);  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.46 (s, 2H, Ar-H), 7.51 (d,  $J = 12$  Hz, 5H, Ar-H), 7.23 (s, 2H, Ar-H), 4.01 (s, 4H,  $\text{—CH}_2\text{—N—CH}_2\text{—}$ ), 3.79 (s, 4H,  $\text{—CH}_2\text{—O—CH}_2\text{—}$ ), 2.36 (s, 3H, Ar- $\text{CH}_3$ );  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 171.12 (Ar-C), 164.28 (Ar-C), 160.47 (Ar-C), 138.16 (Ar-C), 135.94 (Ar-C), 133.39 (Ar-C), 132.20 (Ar-C), 130.83 (Ar-C), 129.93 (Ar-C), 128.74 (Ar-C), 128.40 (Ar-C), 128.33 (Ar-C), 121.00 (Ar-C), 88.90 ( $\text{—C}\equiv\text{C—}$ ), 87.62 ( $\text{—C}\equiv\text{C—}$ ), 66.76 ( $\text{—CH}_2\text{—O—CH}_2\text{—}$ ), 43.77 ( $\text{—CH}_2\text{—N—CH}_2\text{—}$ ), 21.21 (Ar- $\text{CH}_3$ ); HRMS: Anal. calculated for  $\text{C}_{22}\text{H}_{21}\text{N}_4\text{O}$  (M+H: 357.1715); found: 357.1708.

#### 4-(4-(4-Methoxyphenyl)-6-(phenylethynyl)-1,3,5-triazin-2-yl)morpholine (4c):



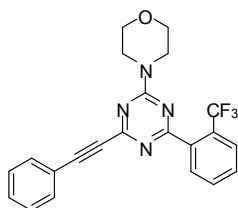
Colorless solid, m. p. 108-110 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3057 (Ar-C-H), 2960 (Alk-C-H), 2219 ( $-\text{C}\equiv\text{C}-$ ), 1574 (Ar-C=C), 1255 (Ar-O-CH<sub>3</sub>), 1269 (C-N), 1111 (C-O); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm) 8.44-8.40 (m, 2H, Ar-H), 7.69-7.67 (m, 2H, Ar-H), 7.42-7.35 (m, 3H, Ar-H), 6.97 (dd,  $J = 2.0$  Hz and 2.0 Hz, 2H, Ar-H), 4.01 (s, 4H, CH<sub>2</sub>-N-CH<sub>2</sub>), 3.87 (s, 3H, Ar-OCH<sub>3</sub>), 3.79 (s, 4H, -CH<sub>2</sub>-O-CH<sub>2</sub>-); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 170.64 (Ar-C), 164.17 (Ar-C), 163.14 (Ar-C), 160.22 (Ar-C), 132.80 (Ar-C), 130.65 (Ar-C), 129.82 (Ar-C), 128.41 (Ar-C), 128.34 (Ar-C), 121.27 (Ar-C), 113.73 (Ar-C), 88.25 ( $-\text{C}\equiv\text{C}-$ ), 87.92 ( $-\text{C}\equiv\text{C}-$ ), 66.77 (-CH<sub>2</sub>-O-CH<sub>2</sub>-), 55.43 (-CH<sub>2</sub>-N-CH<sub>2</sub>-), 43.70 (Ar-OCH<sub>3</sub>); HRMS: Anal. calculated for C<sub>22</sub>H<sub>21</sub>N<sub>4</sub>O<sub>2</sub> (M+H: 373.1665); found: 373.1653.

#### 4-(4-(4-Methoxyphenyl)-6-(*m*-tolylethynyl)-1,3,5-triazin-2-yl)morpholine (4d):



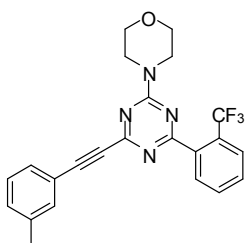
Pale yellow solid, m. p. 121-123 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3056 (Ar-C-H), 2959 (Alk-C-H), 2218 ( $-\text{C}\equiv\text{C}-$ ), 1572 (Ar-C=C), 1253 (Ar-O-CH<sub>3</sub>), 1268 (C-N), 1110 (C-O); <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm) 8.43 (d,  $J = 9.2$  Hz, 2H, Ar-H), 7.51-7.47 (m, 2H, Ar-H), 7.28-7.21 (m, 2H, Ar-H), 6.97-6.95 (dd,  $J = 1.6$  Hz and 2.0 Hz, 2H, Ar-H), 4.01 (s, 4H, CH<sub>2</sub>-N-CH<sub>2</sub>), 3.87 (s, 3H, Ar-OCH<sub>3</sub>), 3.79 (s, 4H, -CH<sub>2</sub>-O-CH<sub>2</sub>-), 2.36 (s, 3H, Ar-CH<sub>3</sub>); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>):  $\delta$  (ppm): 170.60 (Ar-C), 164.17 (Ar-C), 163.10 (Ar-C), 160.25 (Ar-C), 138.12 (Ar-C), 133.36 (Ar-C), 130.75 (Ar-C), 130.63 (Ar-C), 129.90 (Ar-C), 128.37 (Ar-C), 128.29 (Ar-C), 121.05 (Ar-C), 113.70 (Ar-C), 88.56 ( $-\text{C}\equiv\text{C}-$ ), 87.67 ( $-\text{C}\equiv\text{C}-$ ), 66.77 (-CH<sub>2</sub>-O-CH<sub>2</sub>-), 55.41 (-CH<sub>2</sub>-N-CH<sub>2</sub>-), 43.72 (Ar-OCH<sub>3</sub>), 21.21 (Ar-CH<sub>3</sub>); HRMS: Anal. calculated for C<sub>22</sub>H<sub>23</sub>N<sub>4</sub>O<sub>2</sub> (M+H: 387.1821); found: 387.1815.

**4-(4-(Phenylethynyl)-6-(2-(trifluoromethyl)phenyl)-1,3,5-triazin-2-yl)morpholine (4e):**



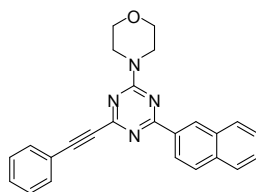
Pale yellow solid, m. p. 102 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3055 (Ar-C-H), 2920 (Alk-C-H), 2219 ( $-\text{C}\equiv\text{C}-$ ), 1572 (Ar-C=C), 1310 (C-N), 1119 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 7.87 (d,  $J = 7.6$  Hz, 1H, Ar-H), 7.80 (d,  $J = 7.6$  Hz, 1H, Ar-H), 7.67- 7.62 (m, 3H, Ar-H), 7.60-7.56 (m, 1H, Ar-H), 7.42-7.35 (m, 3H, Ar-H), 3.98 (d,  $J = 19.6$  Hz, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.79-3.75 (m, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 173.01 (Ar-C), 163.54 (Ar-C), 160.24 (Ar-C), 136.80 (Ar-C), 132.86 (Ar-C), 131.70 (Ar-C), 130.03 (Ar-C), 128.90 (Ar-C), 128.59 (Ar-C), 127.00 (Ar-C), 125.26 (Ar-C), 122.54 (Ar-C), 120.99 (Ar-C), 89.31 ( $-\text{C}\equiv\text{C}-$ ), 87.46 ( $-\text{C}\equiv\text{C}-$ ), 66.70 (Ar- $\text{CF}_3$ ), 43.93 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.79 ( $-\text{CH}_2\text{-N-CH}_2-$ ); HRMS: Anal. calculated for  $\text{C}_{22}\text{H}_{18}\text{F}_3\text{N}_4\text{O}$  (M+H: 411.1433); found: 411.1423.

**4-(4-(m-Tolyethynyl)-6-(2-(trifluoromethyl)phenyl)-1,3,5-triazin-2-yl)morpholine (4f):**



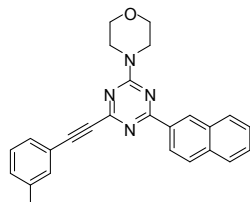
Colorless Solid, m. p. 95 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3054 (Ar-C-H), 2925 (Alk-C-H), 2224 ( $-\text{C}\equiv\text{C}-$ ), 1560 (Ar-C=C), 1308 (C-N), 1113 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 7.87 (d,  $J = 7.6$  Hz, 1H, Ar-H), 7.79 (d,  $J = 7.6$  Hz, 1H, Ar-H), 7.66-7.56 (m, 2H, Ar-H), 7.49-7.45 (m, 2H, Ar-H), 7.28-7.22 (m, 2H, Ar-H), 3.98 (d, 4H,  $J = 20$  Hz,  $\text{CH}_2\text{-N-CH}_2$ ), 3.79- 3.75 (m, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ ), 2.35 (Ar- $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 172.99 (Ar-C), 163.55 (Ar-C), 160.28 (Ar-C), 138.20 (Ar-C), 133.43 (Ar-C), 131.69 (Ar-C), 131.32 (Ar-C), 130.97 (Ar-C), 129.99 (Ar-C), 128.90 (Ar-C), 128.59 (Ar-C), 128.34 (Ar-C), 126.93 (Ar-C), 126.88 (Ar-C), 120.78 (Ar-C), 89.66 ( $-\text{C}\equiv\text{C}-$ ), 87.21 ( $-\text{C}\equiv\text{C}-$ ), 66.71 ( $-\text{CF}_3-$ ), 43.92 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.78 ( $-\text{CH}_2\text{-N-CH}_2-$ ), 21.20 (Ar- $\text{CH}_3$ ); HRMS: Anal. calculated for  $\text{C}_{23}\text{H}_{20}\text{F}_3\text{N}_4\text{O}$  (M+H: 425.1589); found: 425.1594.

#### 4-(4-(Naphthalen-2-yl)-6-(phenylethynyl)-1,3,5-triazin-2-yl)morpholine (4g):



Pale brown solid, m. p. 135 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3027 (Ar-C-H), 2980 (Alk-C-H), 2219 ( $-\text{C}\equiv\text{C}-$ ), 1595 (Ar-C=C), 1213 (C-N), 1108 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 9.04 (s, 1H, Ar-H), 8.50 (d,  $J = 8.4$  Hz, 1H, Ar-H), 8.01-7.87 (m, 3H, Ar-H), 7.72 (d,  $J = 6.8$  Hz, 2H, Ar-H), 7.57-7.50 (m, 2H, Ar-H), 7.41 (t,  $J = 15.2, 7.6$  Hz, 3H, Ar-H), 4.09 (s, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.91 (s, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 171.13 (Ar-C), 164.29 (Ar-C), 160.41 (Ar-C), 135.54 (Ar-C), 133.23 (Ar-C), 133.00 (Ar-C), 129.92 (Ar-C), 129.85 (Ar-C), 129.48 (Ar-C), 128.45 (Ar-C), 128.08 (Ar-C), 127.76 (Ar-C), 126.38 (Ar-C), 124.94 (Ar-C), 121.23 (Ar-C), 88.66 ( $-\text{C}\equiv\text{C}-$ ), 87.91 ( $-\text{C}\equiv\text{C}-$ ), 66.79 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.83 ( $-\text{CH}_2\text{-N-CH}_2-$ ); HRMS: Anal. calculated for  $\text{C}_{25}\text{H}_{21}\text{N}_4\text{O}$  (M+H: 393.1715); found: 393.1692.

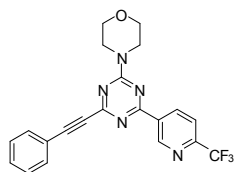
#### 4-(4-(Naphthalen-2-yl)-6-(m-tolylethynyl)-1,3,5-triazin-2-yl)morpholine (4h):



Pale brown solid, m. p. 132 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3028 (Ar-C-H), 2984 (Alk-C-H), 2218 ( $-\text{C}\equiv\text{C}-$ ), 1591 (Ar-C=C), 1216 (C-N), 1105 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 9.04 (s, 1H, Ar-H), 8.51 (d,  $J = 8.4$  Hz, 1H, Ar-H), 8.02-7.87 (m, 3H, Ar-H), 7.54-7.51 (m, 4H, Ar-H), 7.30-7.23 (m, 2H, Ar-H), 4.09 (s, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.82 (s, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ ), 2.37 (s, 3H, Ar- $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 171.10 (Ar-C), 164.31 (Ar-C), 160.45 (Ar-C), 138.18 (Ar-C), 135.53 (Ar-C), 133.43 (Ar-C), 133.25 (Ar-C), 132.99 (Ar-C), 130.85 (Ar-C), 129.97 (Ar-C), 129.83 (Ar-C), 129.48 (Ar-C), 128.33 (Ar-C), 128.06 (Ar-C), 127.75 (Ar-C), 127.72 (Ar-C), 126.36 (Ar-C), 124.94 (Ar-C), 121.01 (Ar-C), 88.99 ( $-\text{C}\equiv\text{C}-$ ), 87.63 ( $-\text{C}\equiv\text{C}-$ ), 66.81 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.83 ( $-\text{CH}_2\text{-N-CH}_2-$ ), 21.23 (Ar- $\text{CH}_3$ ); HRMS: Anal. calculated for  $\text{C}_{26}\text{H}_{23}\text{N}_4\text{O}$  (M+H: 407.1872); found: 407.1861.

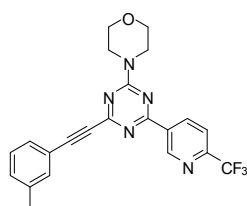


**4-(4-Phenylethynyl)-6-(6-trifluoromethyl)pyridin-3-yl)-1,3,5-triazin-2-yl)morpholine (4i) :**



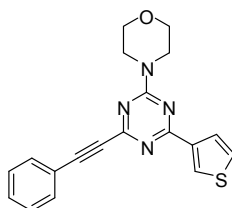
Pale yellow solid, m.p.120 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3057 (Ar-C-H), 2923 (Al-i-C-H), 2221 ( $-\text{C}\equiv\text{C}-$ ), 1587 (Ar-C=C), 1254 (C-N), 1120 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 9.68 (s, 1H, Ar-H), 8.88 (d,  $J = 8$  Hz, 1H, Ar-H), 7.79-7.68 (m, 3H, Ar-H), 7.44-7.39 (m, 3H, Ar-H), 4.01 (s, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.81 (s, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 168.50 (Ar-C), 164.01 (Ar-C), 160.62 (Ar-C), 150.69 (Ar-C), 137.53 (Ar-C), 134.25 (Ar-C), 132.88 (Ar-C), 130.21 (Ar-C), 128.52 (Ar-C), 122.80 (Ar-C), 120.83 (Ar-C), 120.05 (Ar-C), 89.73 ( $-\text{C}\equiv\text{C}-$ ), 87.41 ( $-\text{C}\equiv\text{C}-$ ), 66.63 ( $-\text{CF}_3$ ), 43.98 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.84 ( $-\text{CH}_2\text{-N-CH}_2-$ ); Anal. calculated for  $\text{C}_{21}\text{H}_{17}\text{F}_3\text{N}_5\text{O}$  (M+H: 412.1385); found: 412.1425.

**4-(4-(m-Toly)ethynyl)-6-(6-trifluoromethyl)pyridin-3-yl)-1,3,5-triazin-2-yl)morpholine (4j):**



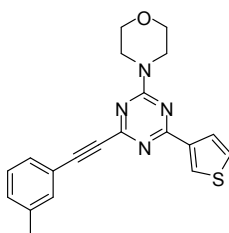
Colorless solid, m.p.110 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3058 (Ar-C-H), 2927 (Al-i-C-H), 2221 ( $-\text{C}\equiv\text{C}-$ ), 1585 (Ar-C=C), 1250 (C-N), 1115 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 9.69 (s, 1H, Ar-H), 8.88 (q,  $J = 8, 1.2$  Hz, 1H, Ar-H), 7.80 (d,  $J = 8$  Hz, 1H, Ar-H), 7.53-7.49 (m, 2H, Ar-H), 7.31-7.25 (m, 2H, Ar-H), 4.01 (s, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.82 (s, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ ), 2.38 (s, 3H, Ar- $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 168.48 (Ar-C), 164.03 (Ar-C), 160.67 (Ar-C), 150.68 (Ar-C), 150.38 (Ar-C), 138.32 (Ar-C), 137.54 (Ar-C), 134.29 (Ar-C), 133.44 (Ar-C), 131.17 (Ar-C), 130.01 (Ar-C), 128.43 (Ar-C), 120.63 (Ar-C), 120.05 (Ar-C), 90.10 ( $-\text{C}\equiv\text{C}-$ ), 87.18 ( $-\text{C}\equiv\text{C}-$ ), 66.64 ( $-\text{CF}_3$ ), 43.93 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.82 ( $-\text{CH}_2\text{-N-CH}_2-$ ), 21.21 (Ar- $\text{CH}_3$ ); Anal. calculated for  $\text{C}_{22}\text{H}_{19}\text{F}_3\text{N}_5\text{O}$  (M+H: 426.1542); found: 426.1571.

**4-(4-Phenylethynyl)-6-(thiophen-3-yl)-1,3,5-triazin-2-yl)morpholine (4k):**



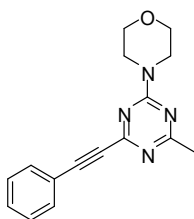
Pale yellow solid, m. p. 105 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3058 (Ar-C-H), 2927 (Alk-C-H), 2221 ( $-\text{C}\equiv\text{C}-$ ), 1585 (Ar-C=C), 1265 (C-N), 1115 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.42 (s, 1H, Ar-H), 7.87 (d,  $J = 4.8$  Hz, 1H, Ar-H), 7.68 (d,  $J = 6.8$  Hz, 2H, Ar-H), 7.42-7.34 (m, 4H, Ar-H), 3.97 (s, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.78 (s, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 167.65 (Ar-C), 164.19 (Ar-C), 160.39 (Ar-C), 140.10 (Ar-C), 132.81 (Ar-C), 130.78 (Ar-C), 129.89 (Ar-C), 128.42 (Ar-C), 127.50 (Ar-C), 125.97 (Ar-C), 121.17 (Ar-C), 88.59 ( $-\text{C}\equiv\text{C}-$ ), 87.75 ( $-\text{C}\equiv\text{C}-$ ), 66.74 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.75 ( $-\text{CH}_2\text{-N-CH}_2-$ ); Anal. calculated for  $\text{C}_{19}\text{H}_{17}\text{N}_4\text{OS}$  (M+H: 349.1123); found: 349.1121.

**4-(4-(Thiophen-3-yl)-6-(m-tolylolethynyl)-1,3,5-triazin-2-yl)morpholine (4l):**



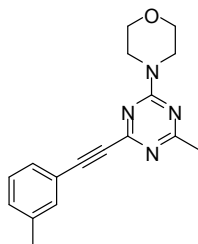
Off white solid, m. p. 95-98 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3059 (Ar-C-H), 2923 (Alk-C-H), 2220 ( $-\text{C}\equiv\text{C}-$ ), 1580 (Ar-C=C), 1274 (C-N), 1113 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 8.42 (s, 1H, Ar-H), 7.87 (d,  $J = 5.2$  Hz, 1H, Ar-H), 7.51-7.47 (m, 2H, Ar-H), 7.36-7.34 (m, 1H, Ar-H), 7.29-7.22 (m, 2H, Ar-H), 3.98 (s, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.79 (s, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ ), 2.36 (s, 3H, Ar- $\text{CH}_3$ );  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 167.64 (Ar-C), 164.21 (Ar-C), 160.44 (Ar-C), 140.14 (Ar-C), 138.16 (Ar-C), 133.38 (Ar-C), 130.83 (Ar-C), 130.74 (Ar-C), 129.92 (Ar-C), 128.31 (Ar-C), 127.51 (Ar-C), 125.93 (Ar-C), 120.97 (Ar-C), 88.93 ( $-\text{C}\equiv\text{C}-$ ), 87.49 ( $-\text{C}\equiv\text{C}-$ ), 66.75 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 43.73 ( $-\text{CH}_2\text{-N-CH}_2-$ ), 21.21 (Ar- $\text{CH}_3$ ); Anal. calculated for  $\text{C}_{20}\text{H}_{19}\text{N}_4\text{OS}$  (M+H: 363.1280); found: 363.1307.

#### 4-(4-Methyl-6-(phenylethynyl)-1,3,5-triazin-2-yl)morpholine (4m):



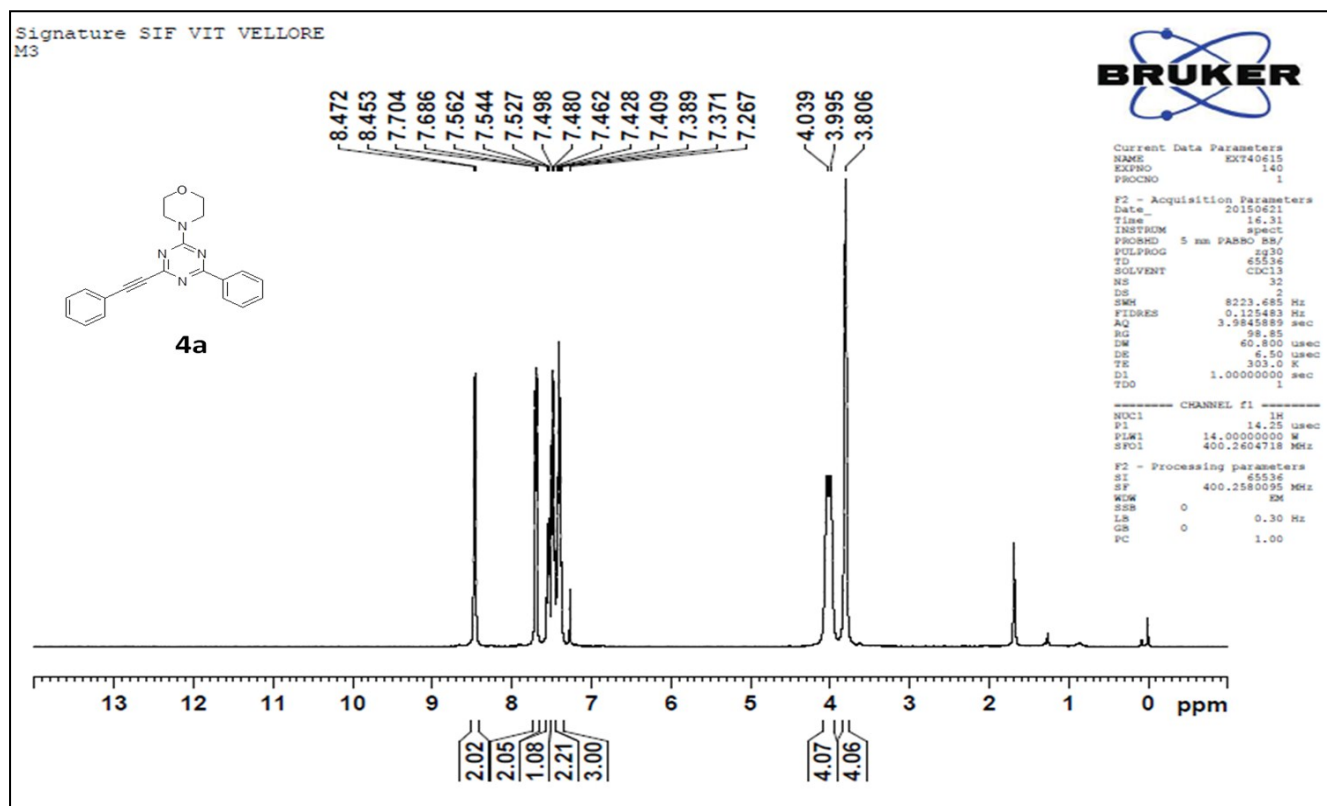
Colorless solid, m. p. 85 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3058 (Ar-C-H), 2960 (Al-i-C-H), 2221 ( $-\text{C}\equiv\text{C}-$ ), 1553 (Ar-C=C), 1273 (C-N), 1109 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 7.64 (d,  $J = 8$  Hz, 2H, Ar-H), 7.41-7.34 (m, 3H, Ar-H), 3.92 (s, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.75 (t,  $J = 9.6$  Hz, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ ), 1.42 (t,  $J = 14.4$  Hz, 3H, ((N-C=N)- $\text{CH}_3$ ));  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 170.37 (Ar-C), 165.58 (Ar-C), 161.17 (Ar-C), 132.82 (Ar-C), 129.93 (Ar-C), 128.43 (Ar-C), 121.09 (Ar-C), 88.47 ( $-\text{C}\equiv\text{C}-$ ), 87.35 ( $-\text{C}\equiv\text{C}-$ ), 66.72 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 44.17 ( $-\text{CH}_2\text{-N-CH}_2-$ ), 14.36 ( $-\text{CH}_3$ ); HRMS: Anal. calculated for  $\text{C}_{16}\text{H}_{17}\text{N}_4\text{O}$  (M+H: 281.1402); found: 281.1389.

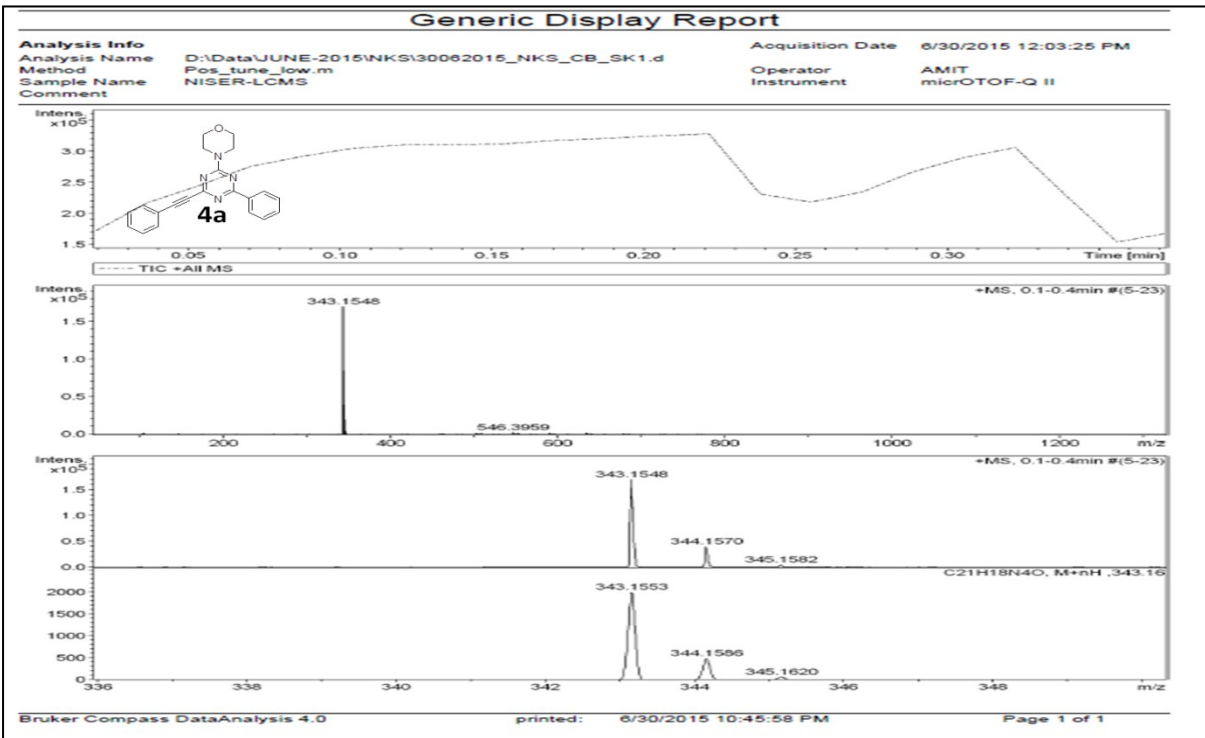
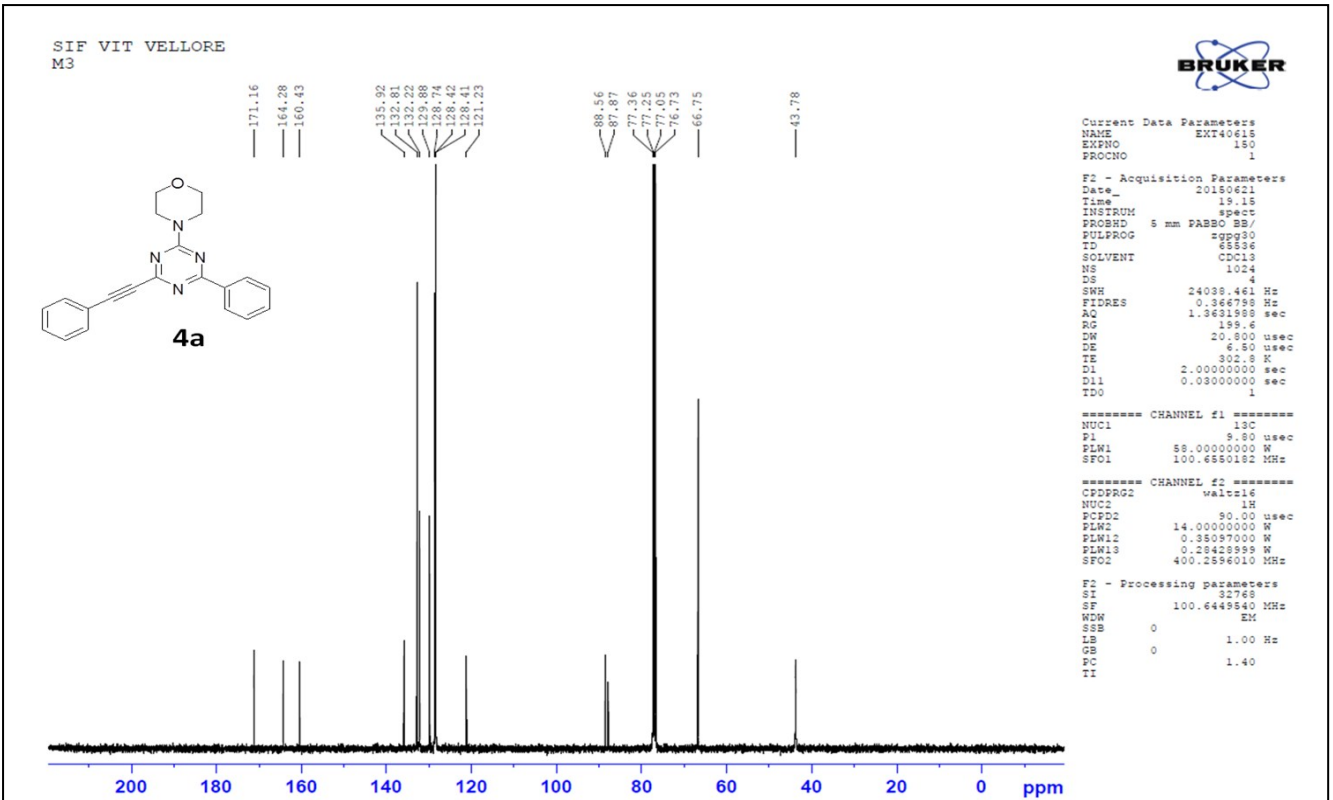
#### 4-(4-Methyl-6-(m-tolyethynyl)-1,3,5-triazin-2-yl)morpholine (4n):

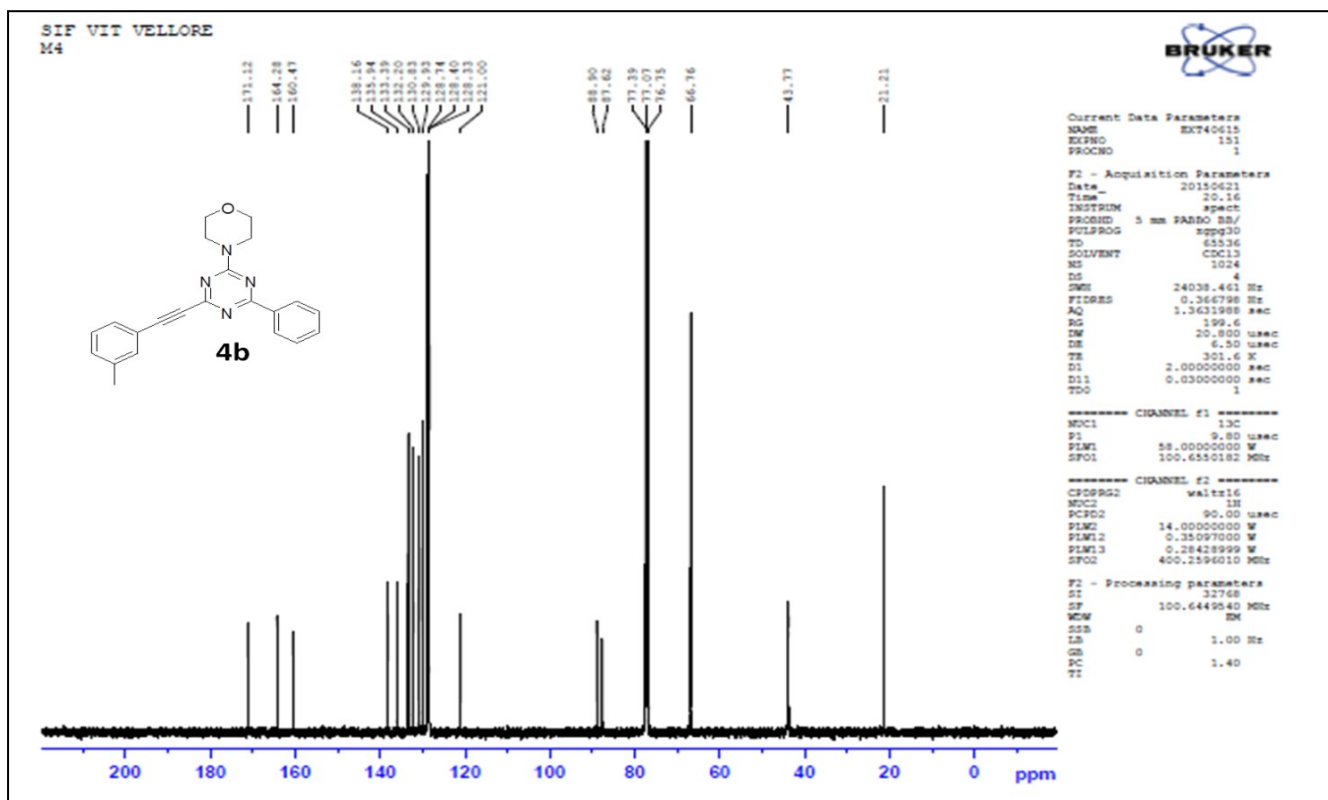
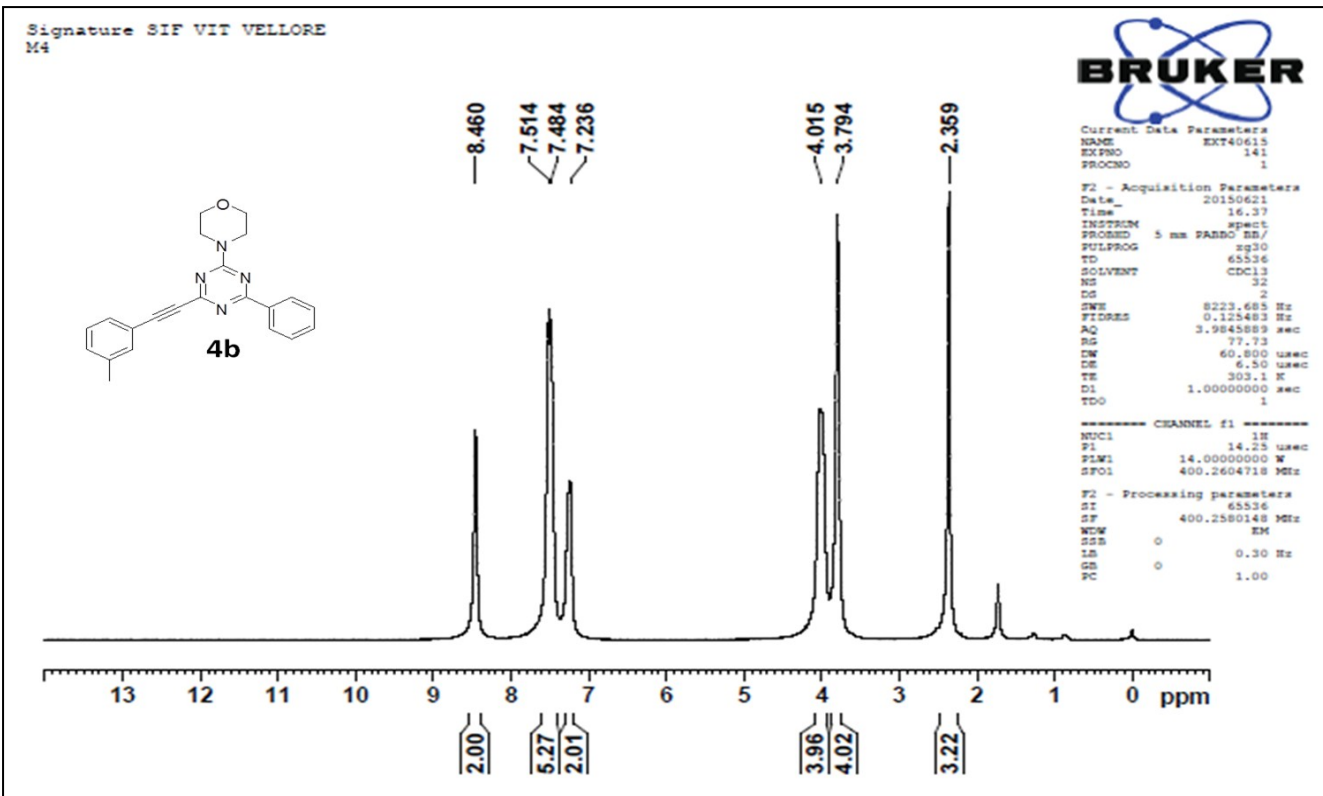


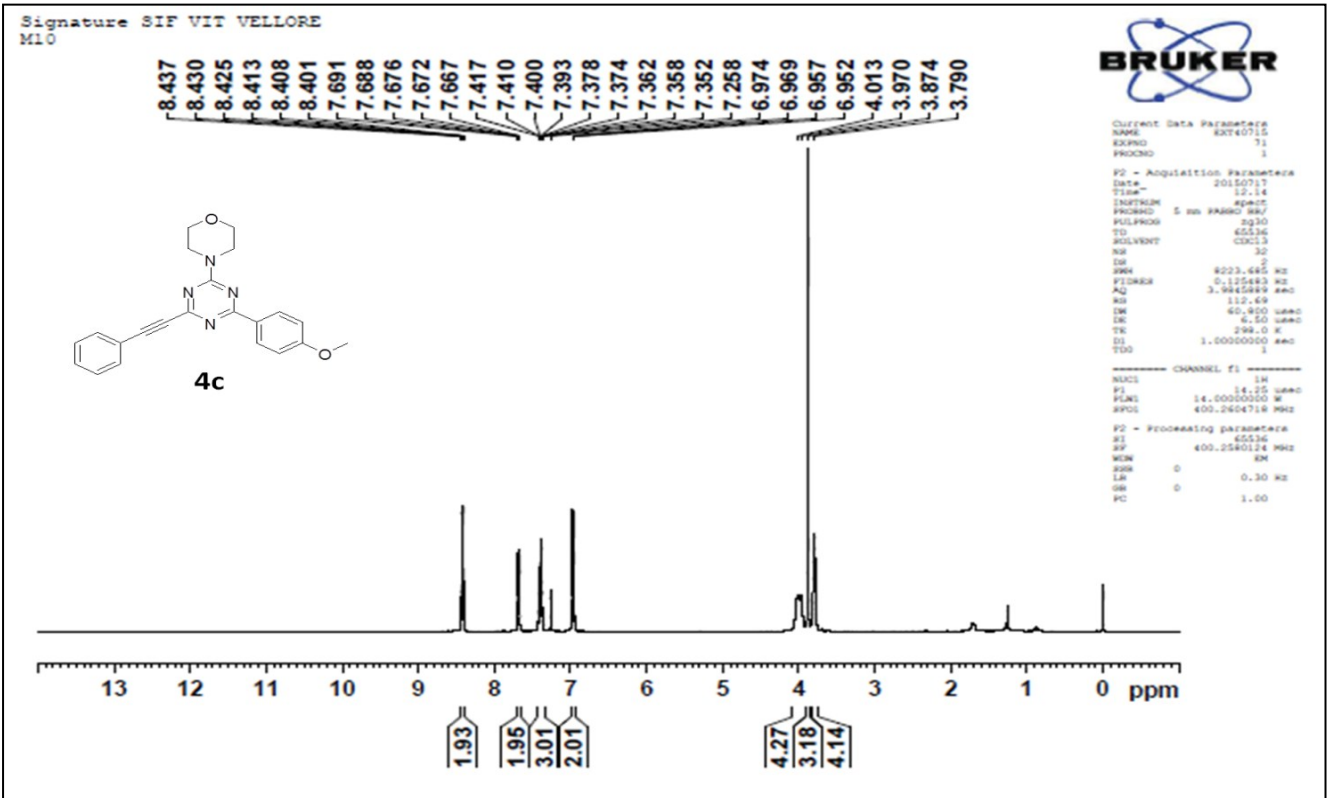
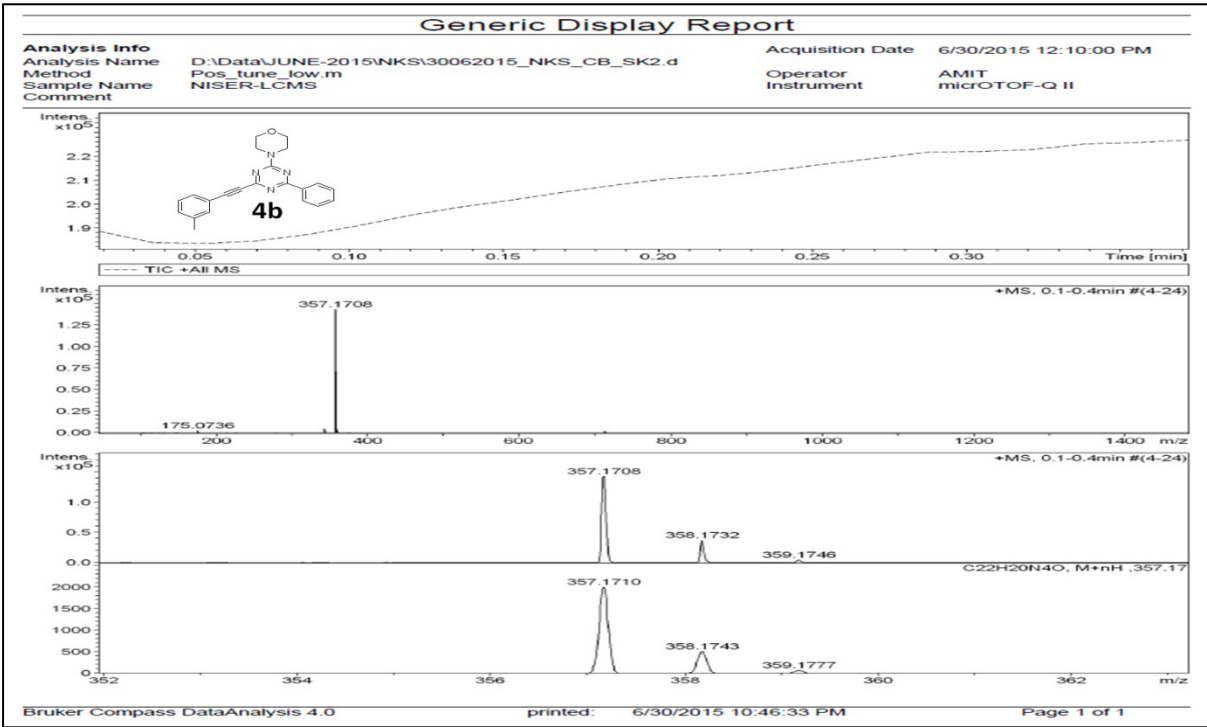
Pale brown solid, m. p. 95 °C;  $\nu_{\text{max}}$  (KBr)/ $\text{cm}^{-1}$ : 3058 (Ar-C-H), 2966 (Al-i-C-H), 2220 ( $-\text{C}\equiv\text{C}-$ ), 1562 (Ar-C=C), 1273 (C-N), 1117 (C-O);  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm) 7.46-7.43 (m, 2H, Ar-H), 7.27-7.20 (m, 2H, Ar-H), 3.92-3.86 (m, 4H,  $\text{CH}_2\text{-N-CH}_2$ ), 3.75 (t,  $J = 9.2$  Hz, 4H,  $-\text{CH}_2\text{-O-CH}_2-$ ), 2.34 (s, 3H, Ar- $\text{CH}_3$ ), 1.42 (t,  $J = 14.4$  Hz, 3H, ((N-C=N)- $\text{CH}_3$ ));  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  (ppm): 170.35 (Ar-C), 165.56 (Ar-C), 161.20 (Ar-C), 138.15 (Ar-C), 133.35 (Ar-C), 130.85 (Ar-C), 129.91 (Ar-C), 128.30 (Ar-C), 120.85 (Ar-C), 88.78 ( $-\text{C}\equiv\text{C}-$ ), 87.10 ( $-\text{C}\equiv\text{C}-$ ), 66.70 ( $-\text{CH}_2\text{-O-CH}_2-$ ), 44.15 ( $-\text{CH}_2\text{-N-CH}_2-$ ), 21.19 (Ar- $\text{CH}_3$ ), 14.35 ((N-C=N)- $\text{CH}_3$ ); HRMS: Anal. calculated for  $\text{C}_{17}\text{H}_{19}\text{N}_4\text{O}$  (M+H: 295.1559); found: 295.1567.

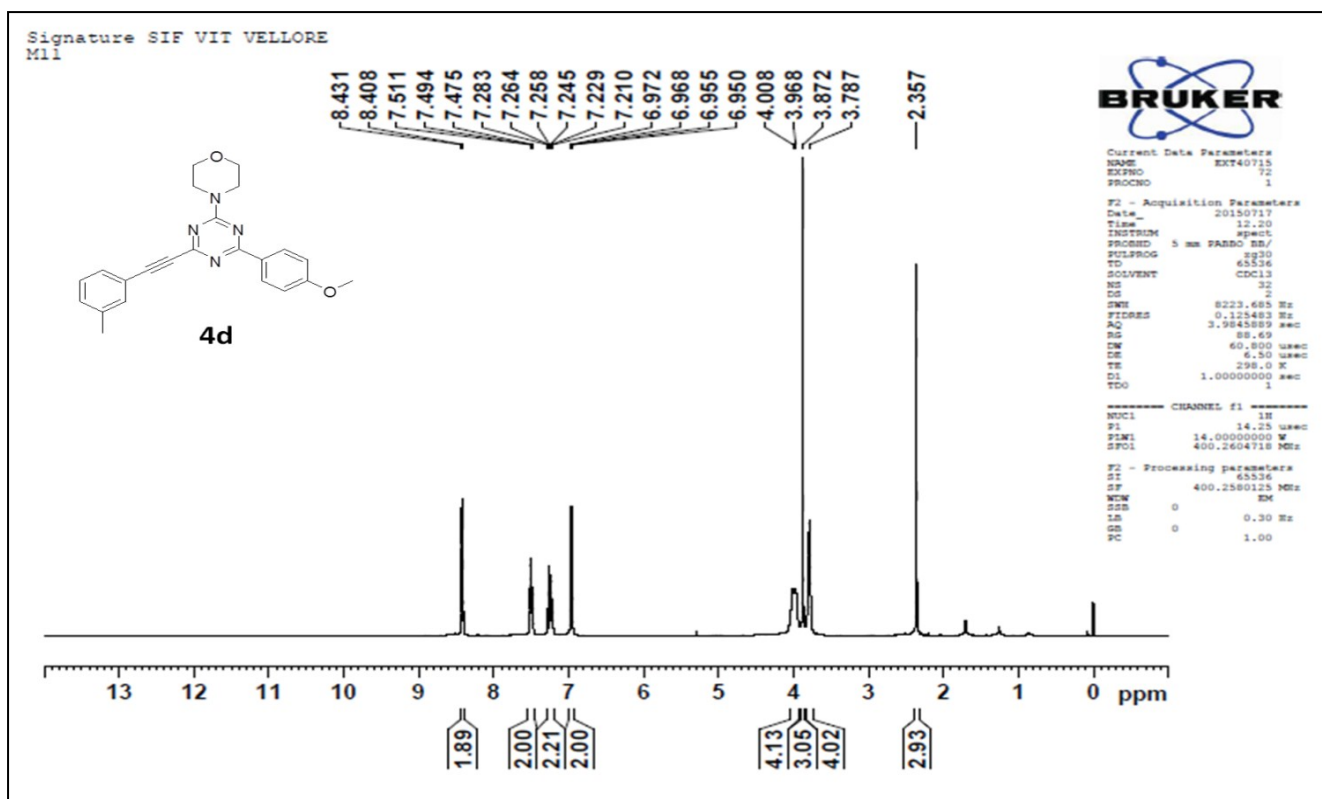
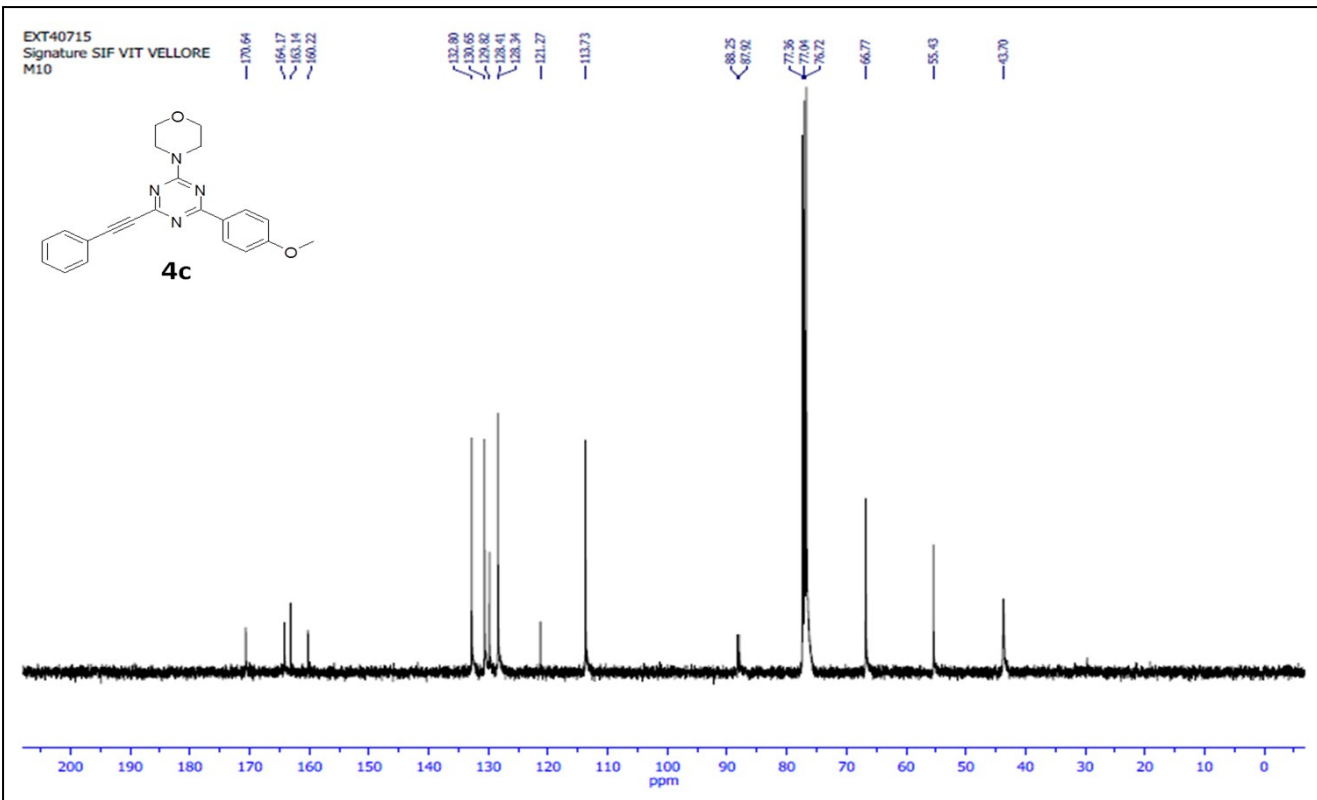
### III. Copies of (<sup>1</sup>H & <sup>13</sup>C) NMR and HRMS Spectra



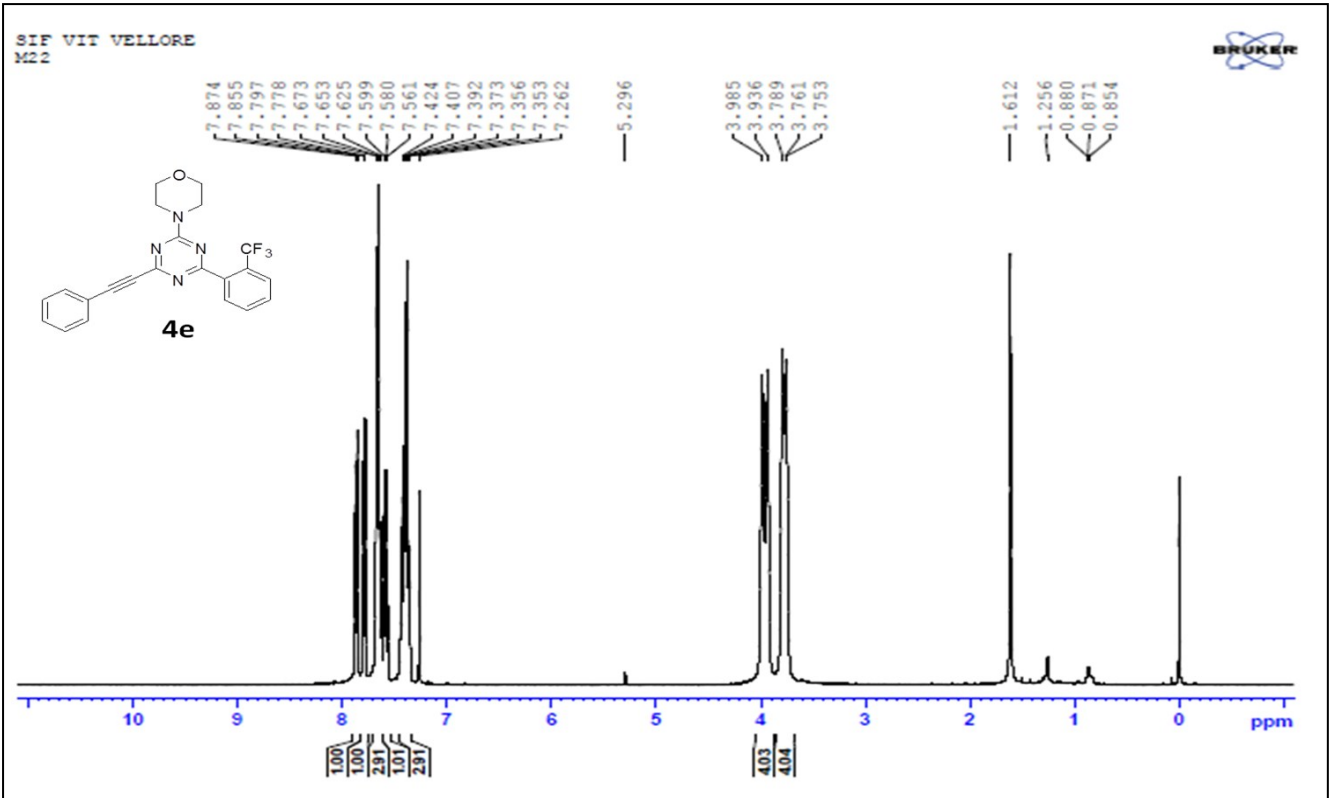
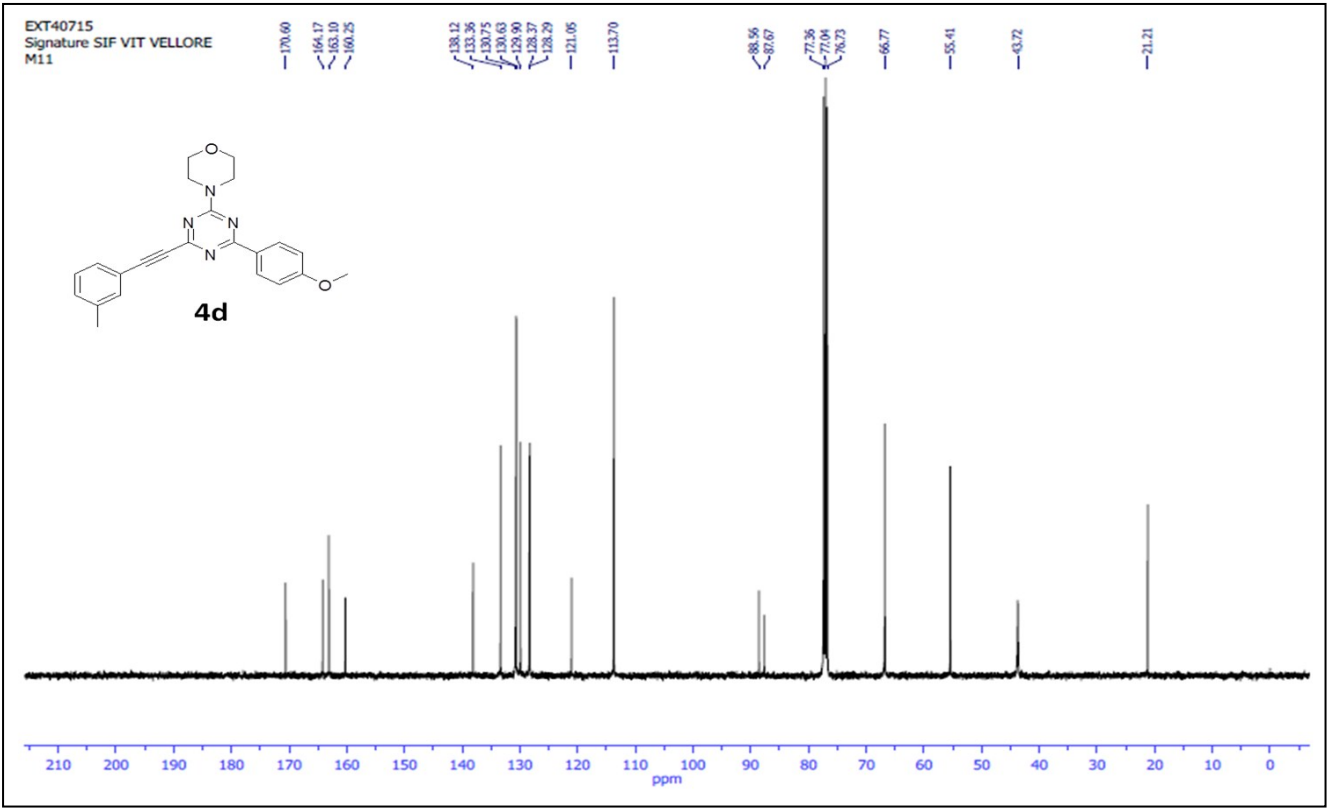


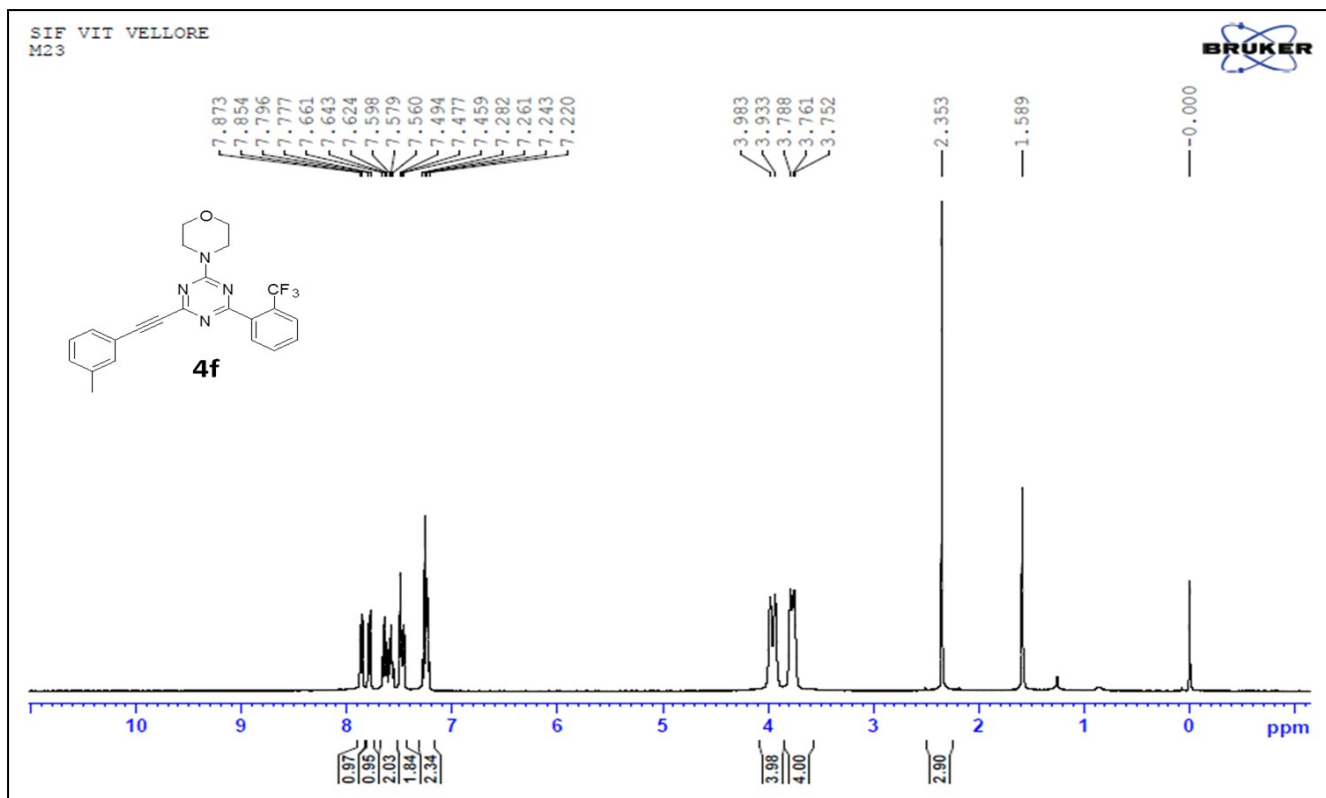
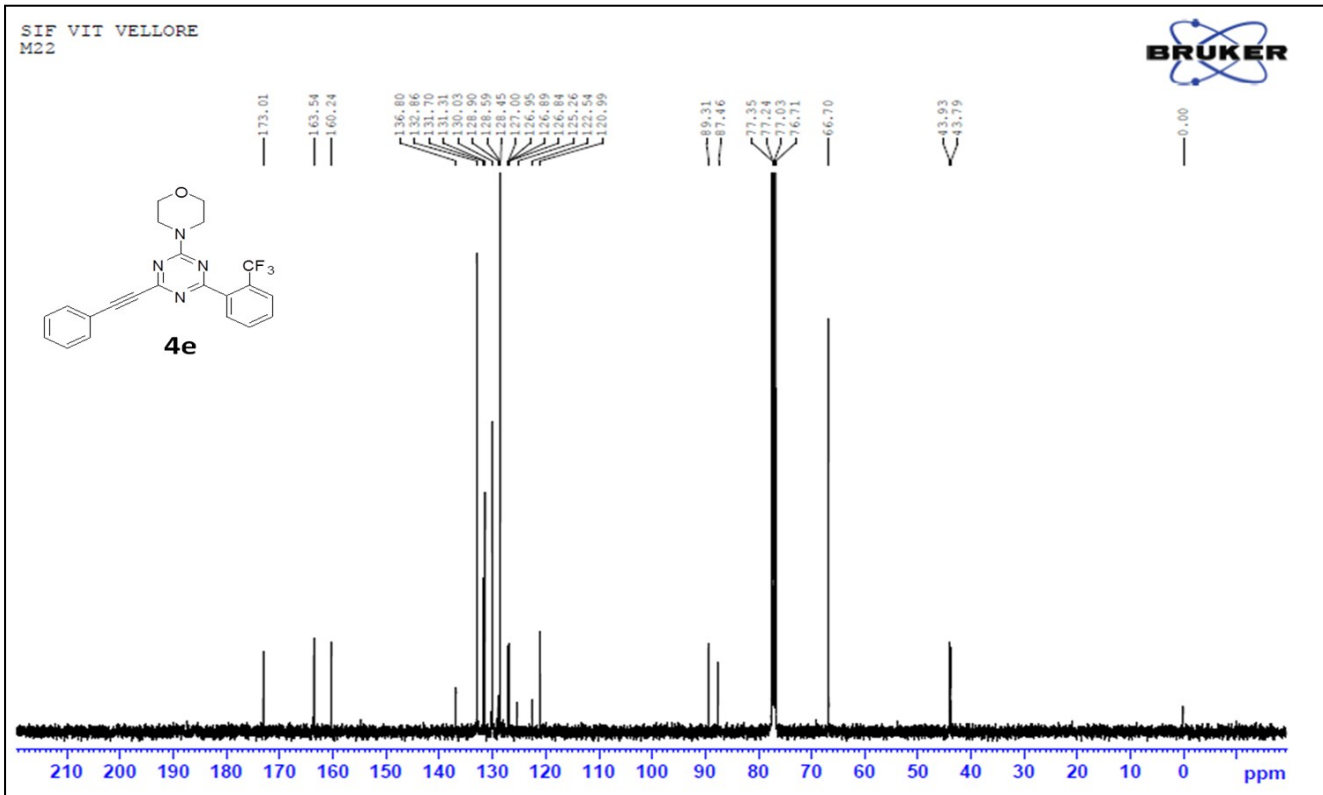


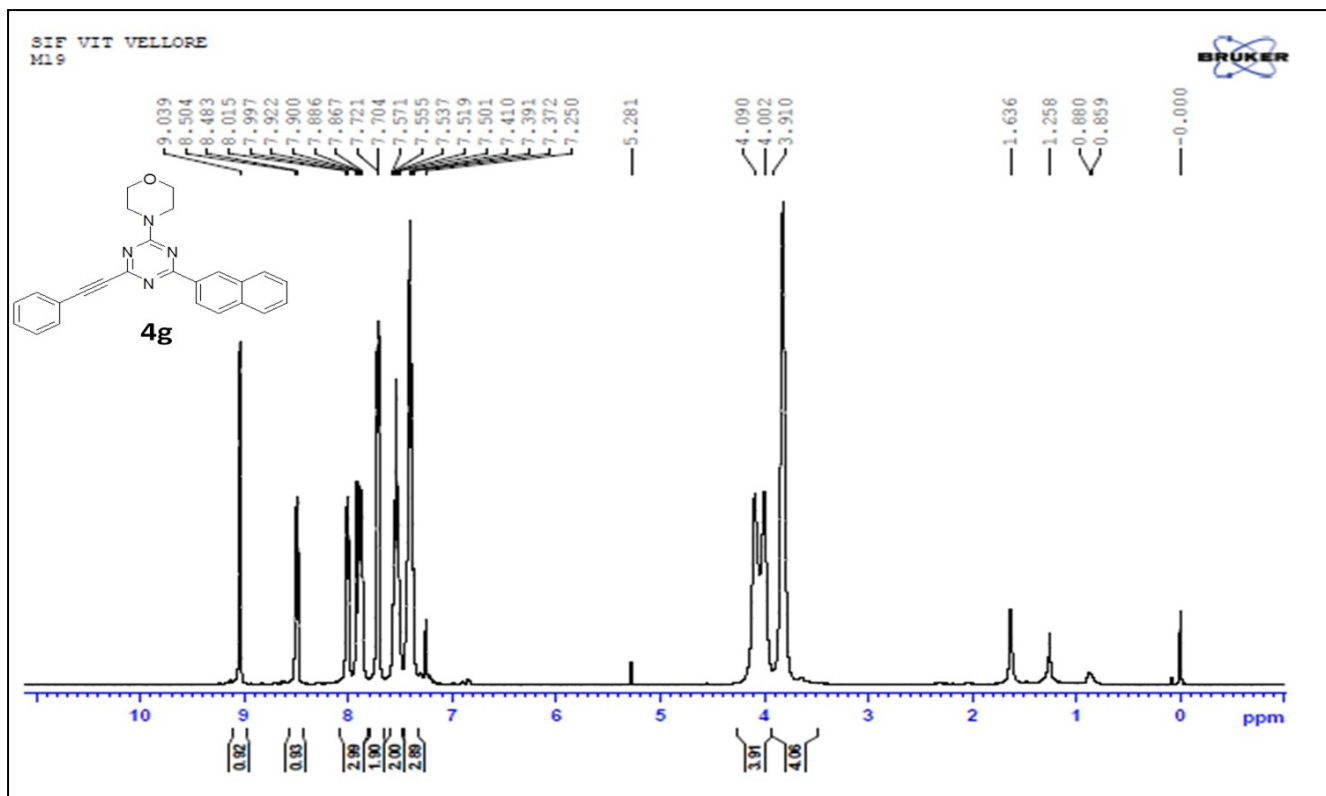
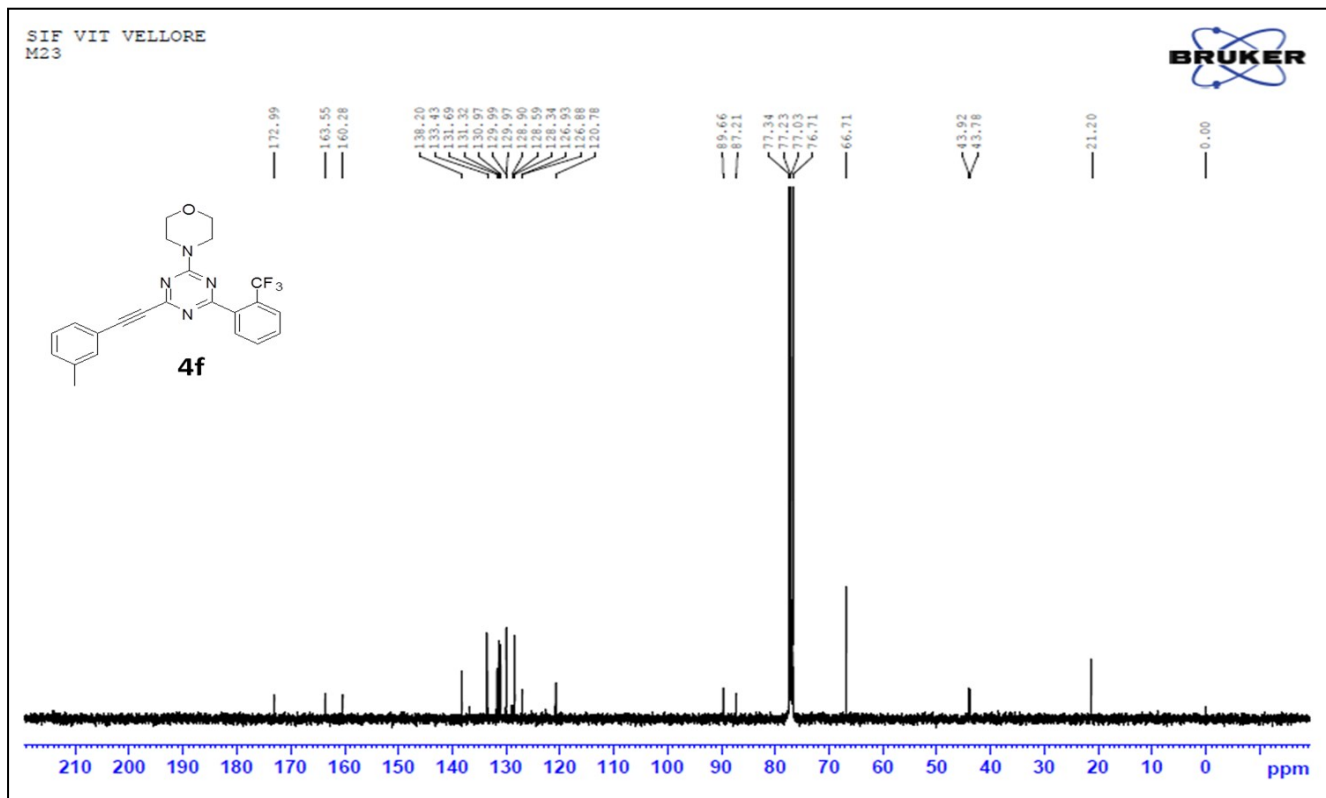




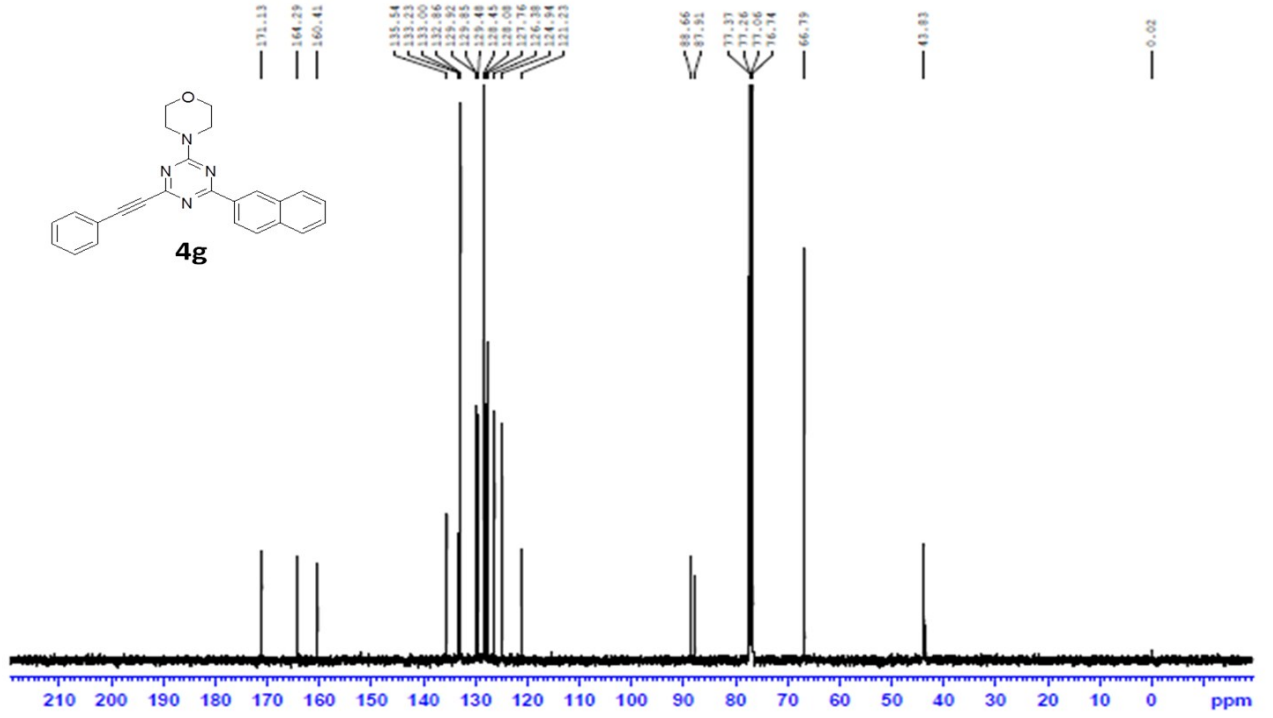
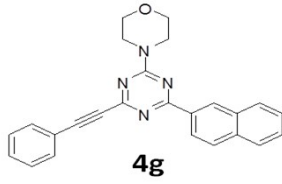




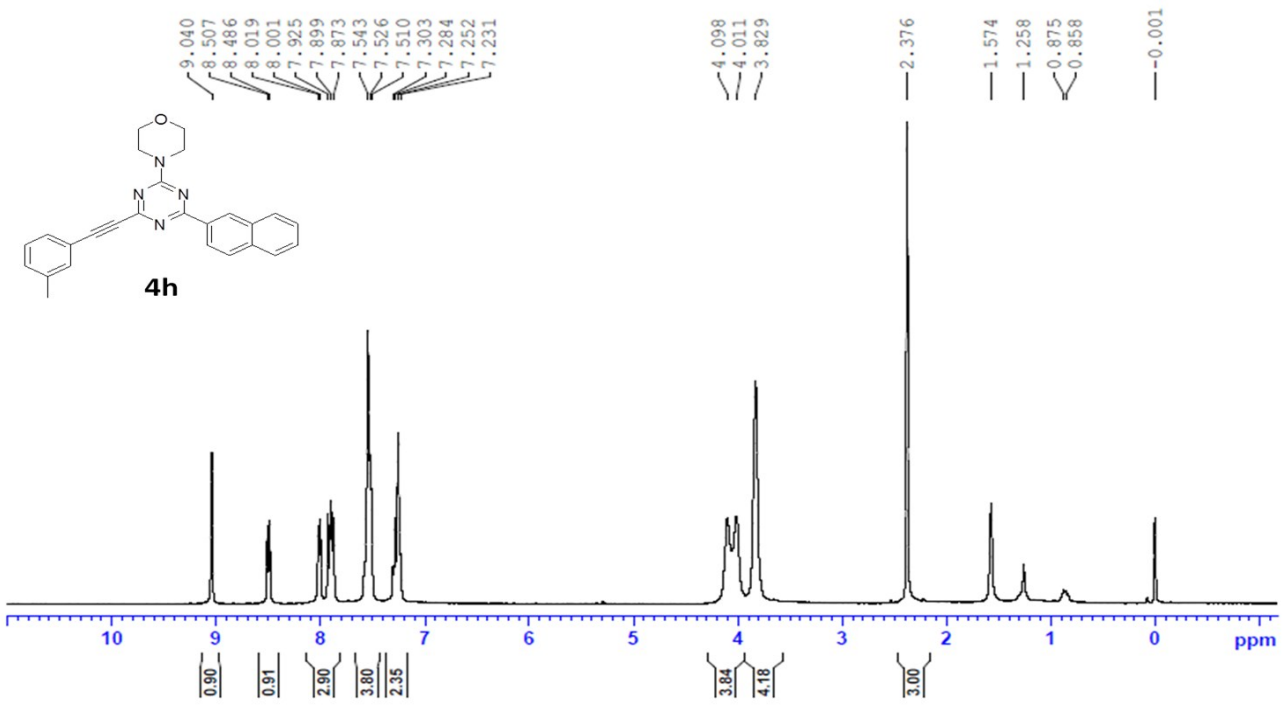
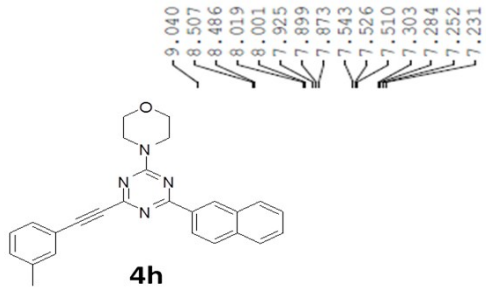




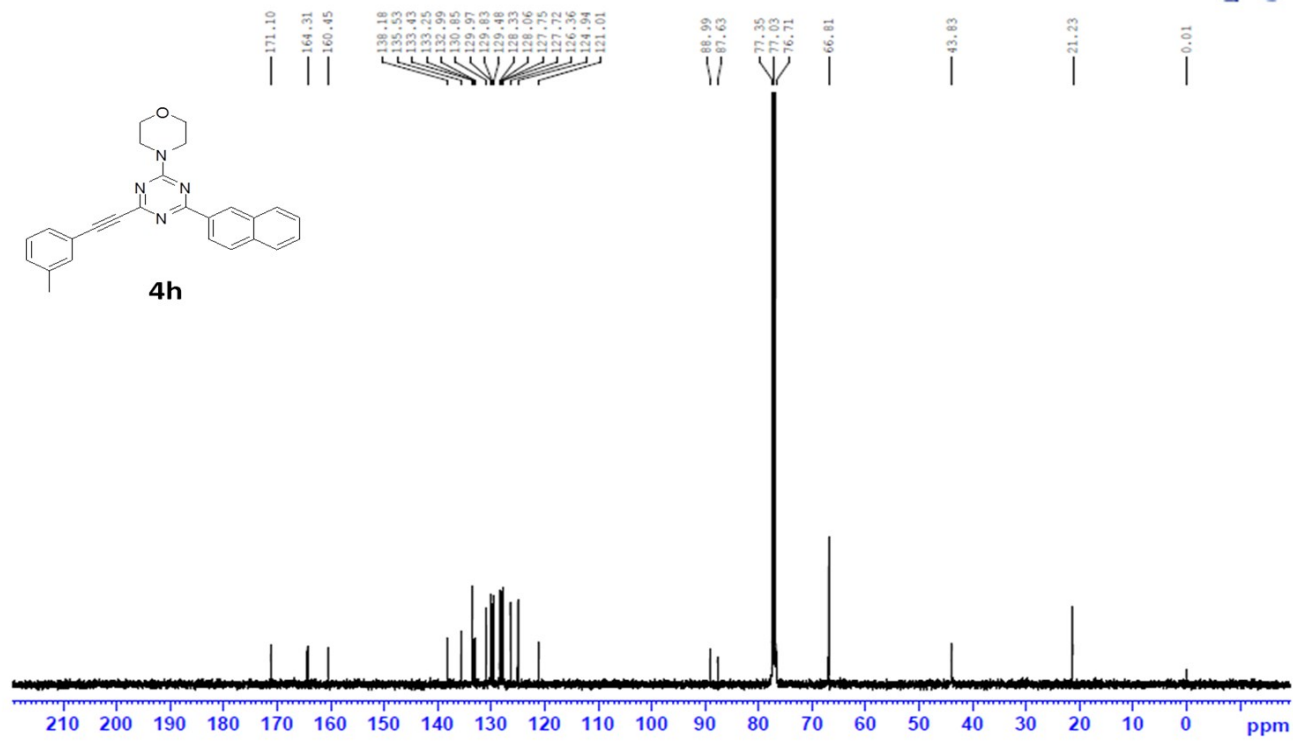
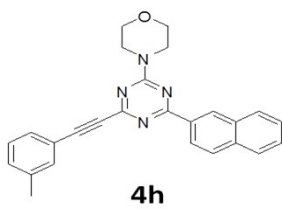
SIF VIT VELLORE  
M19

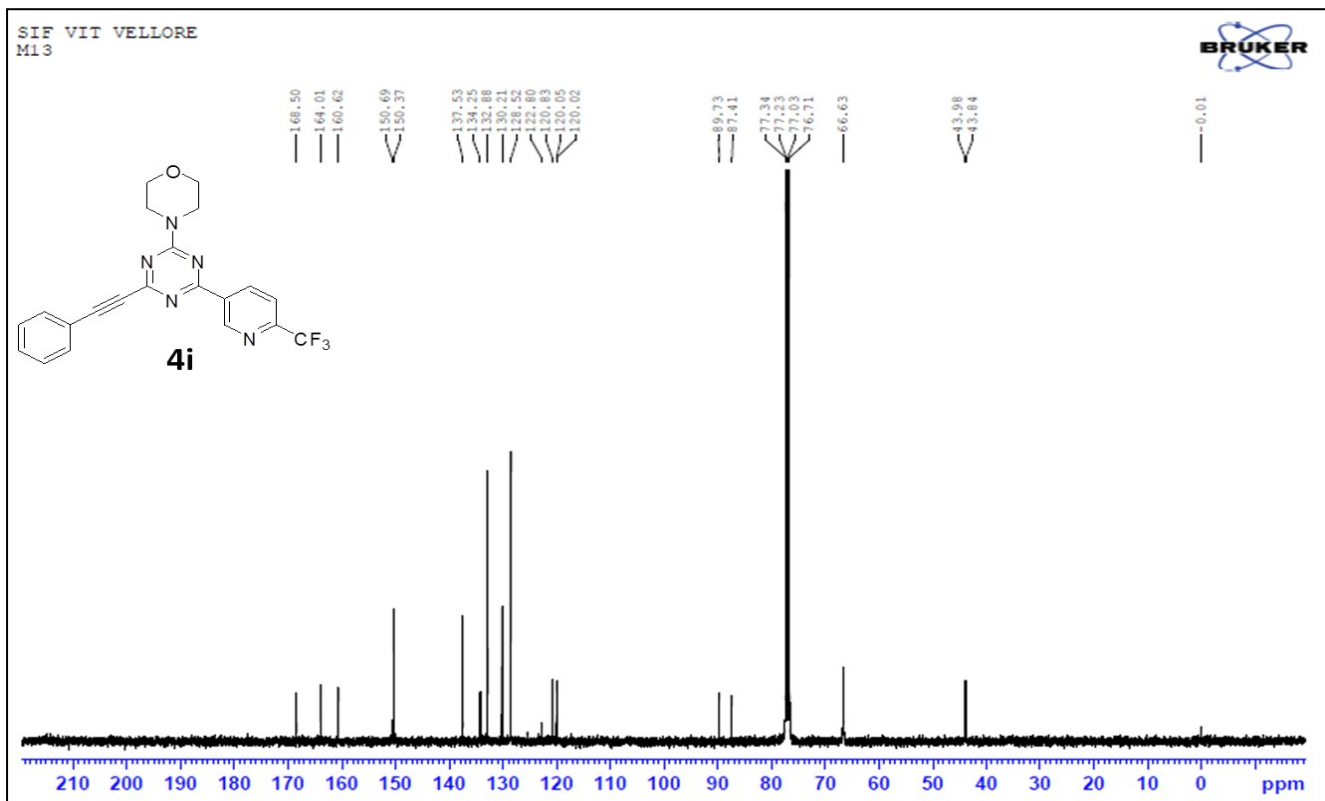
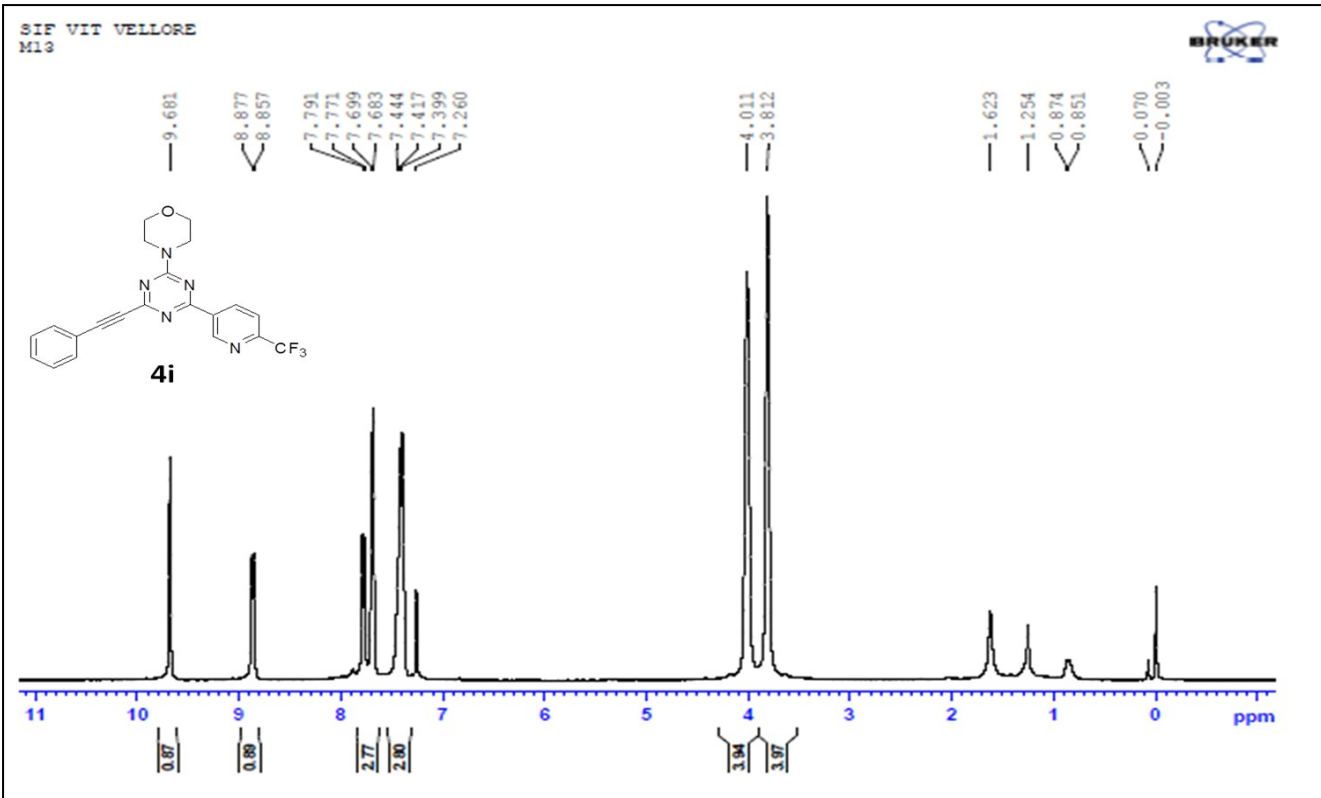


SIF VIT VELLORE  
M20



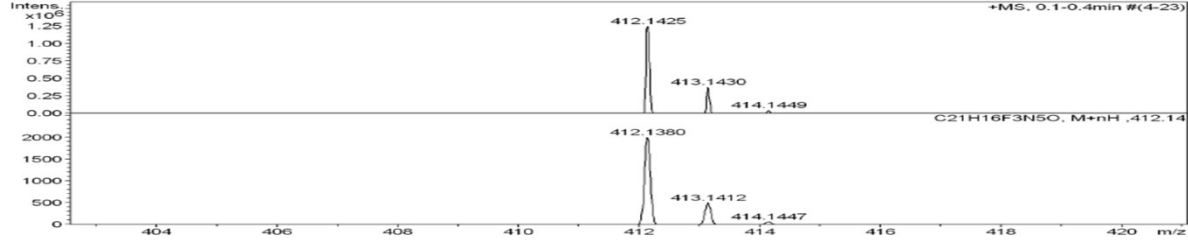
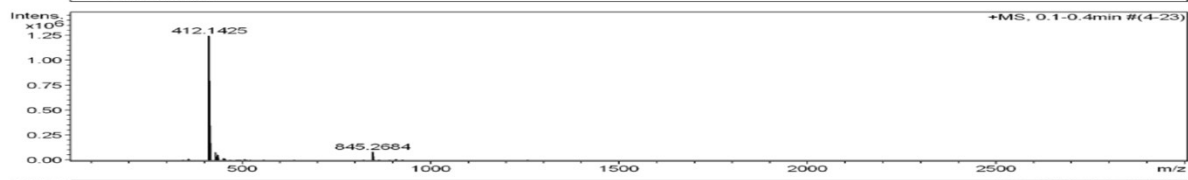
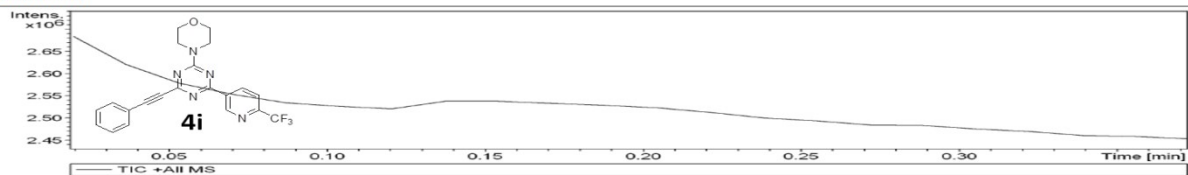
SIF VIT VELLORE  
M20



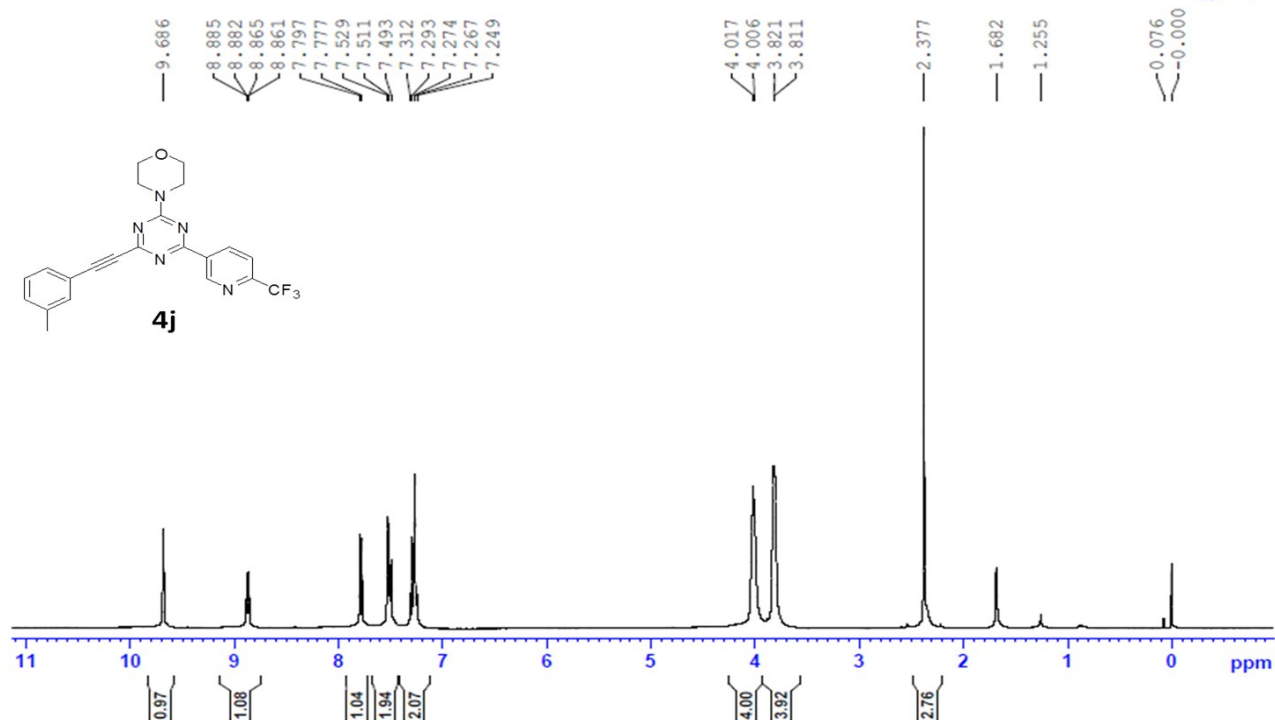


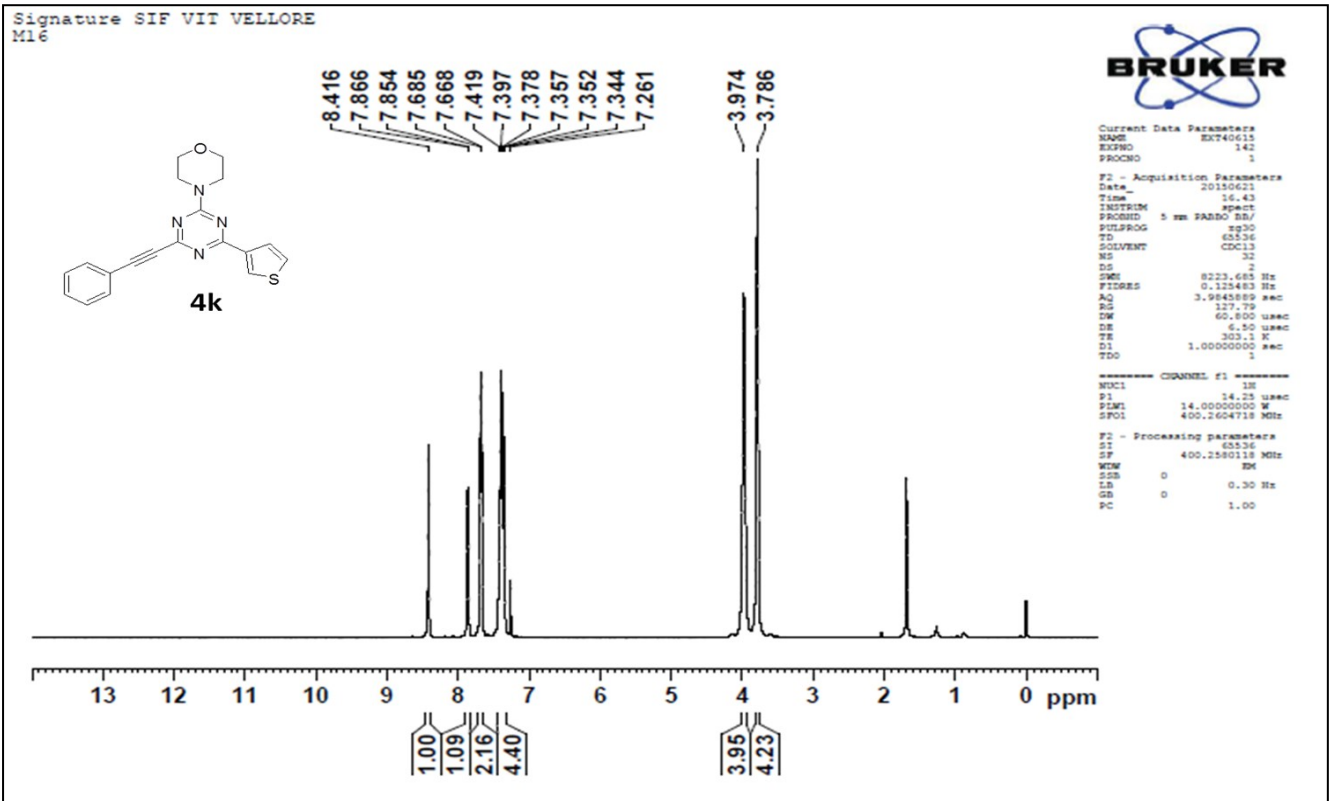
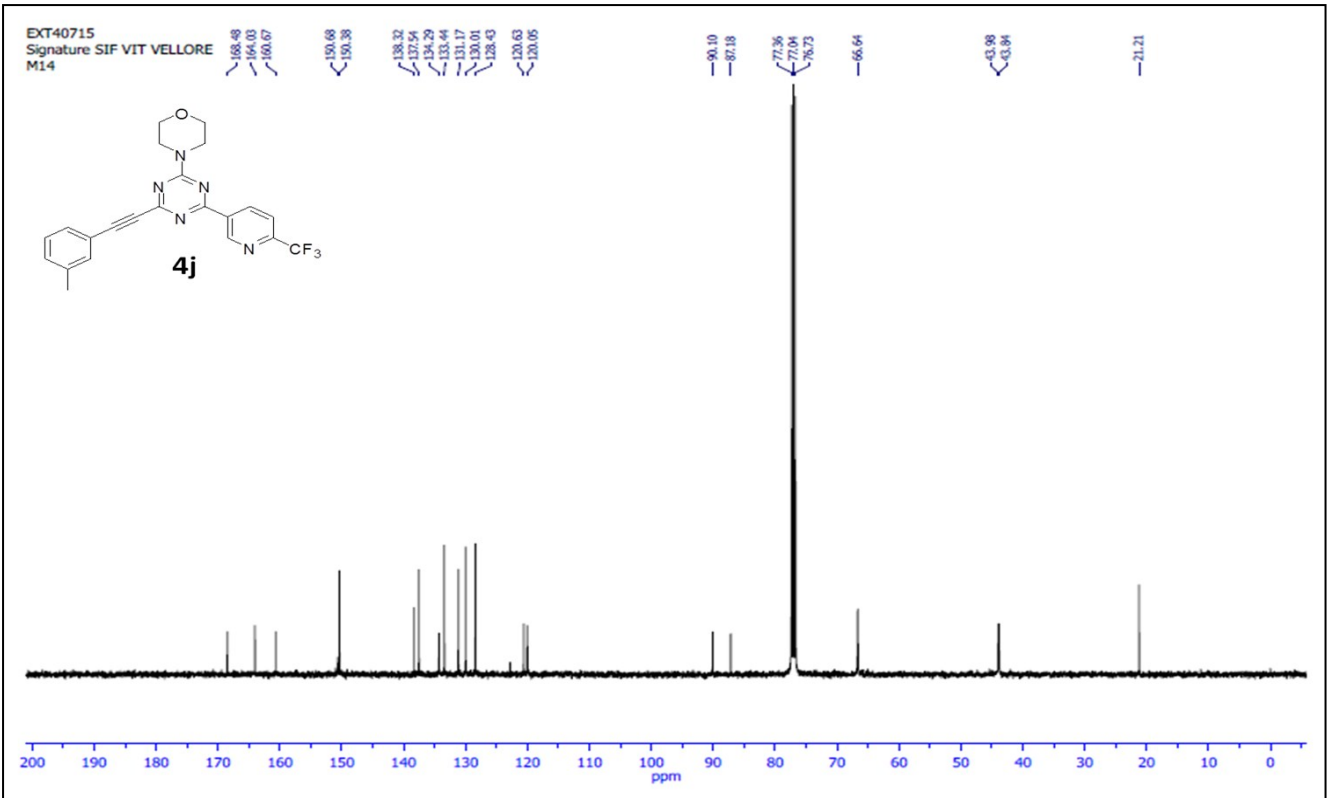
### Generic Display Report

<b>Analysis Info</b>	D:\Data\JUNE-2015\NKS\30062015_NKS_CB_SK3.d	Acquisition Date	6/30/2015 2:33:47 PM
Method	lms_wide.m	Operator	AMIT
Sample Name	NISER-LCMS	Instrument	micrOTOF-Q II
Comment			

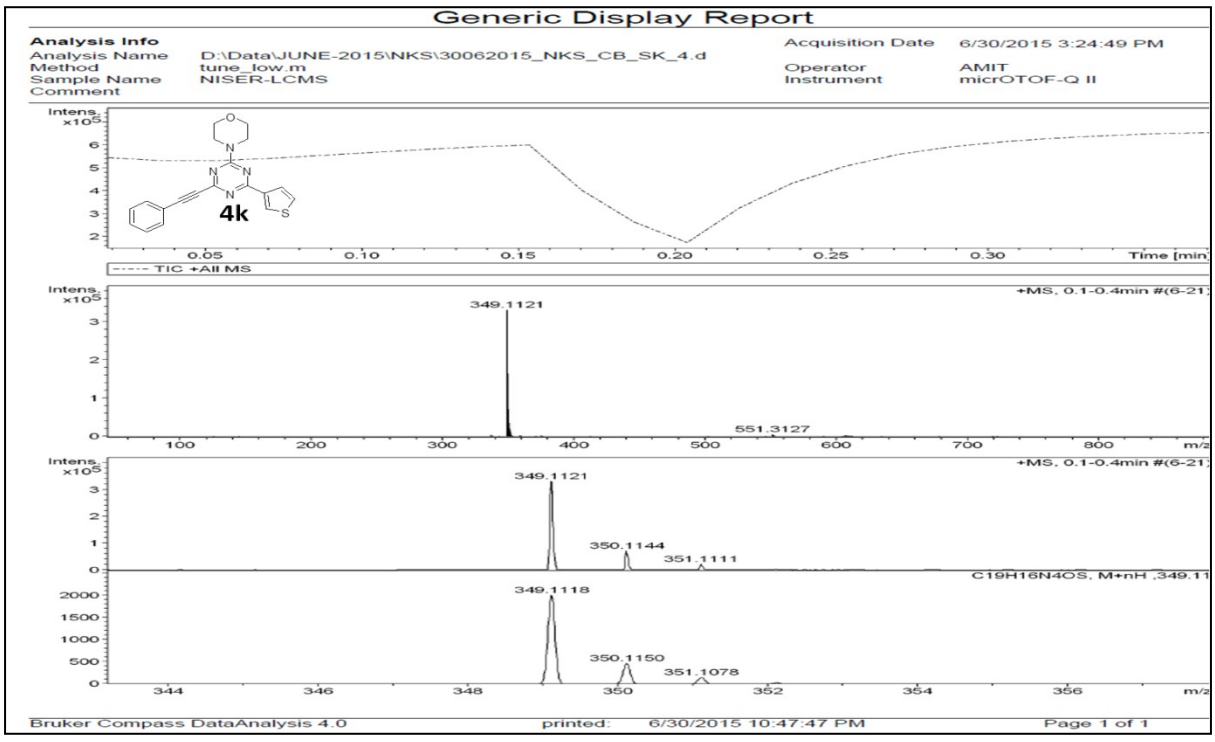
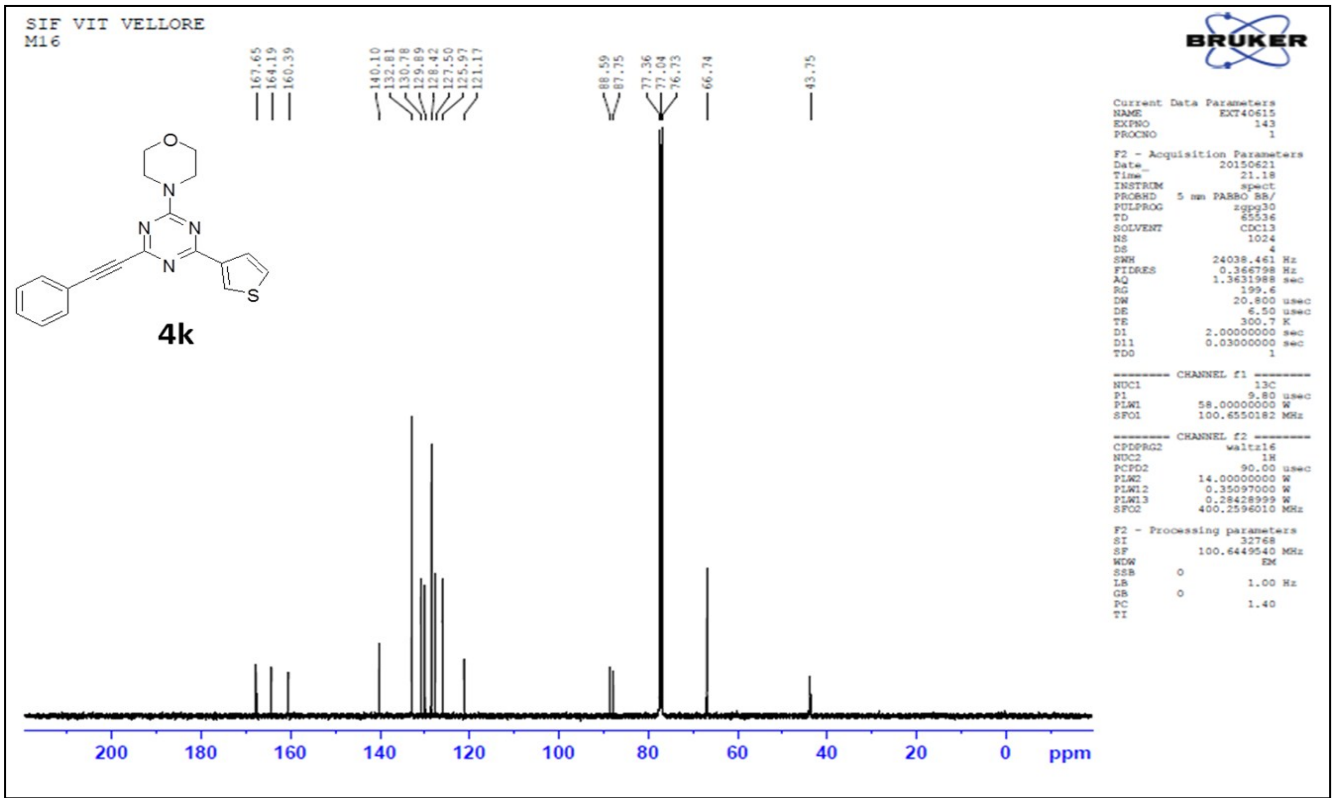


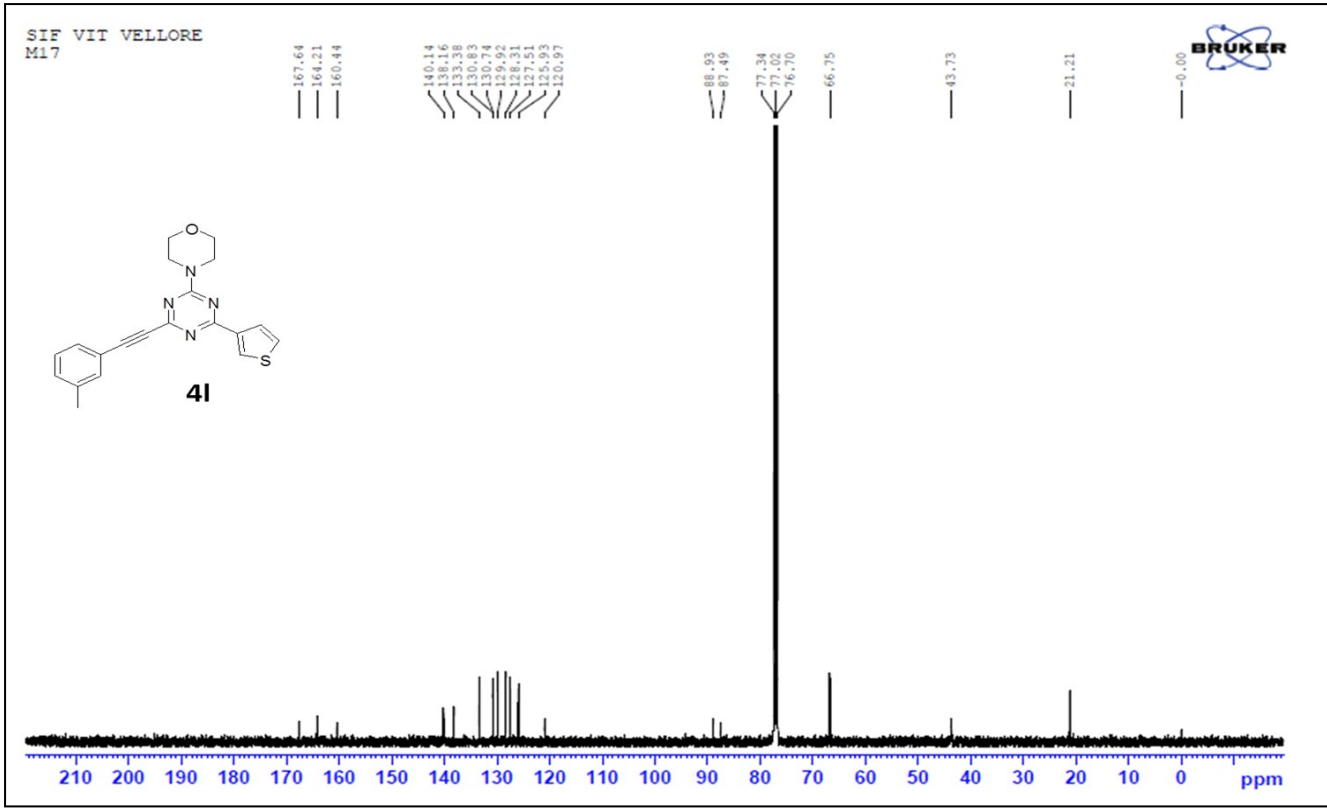
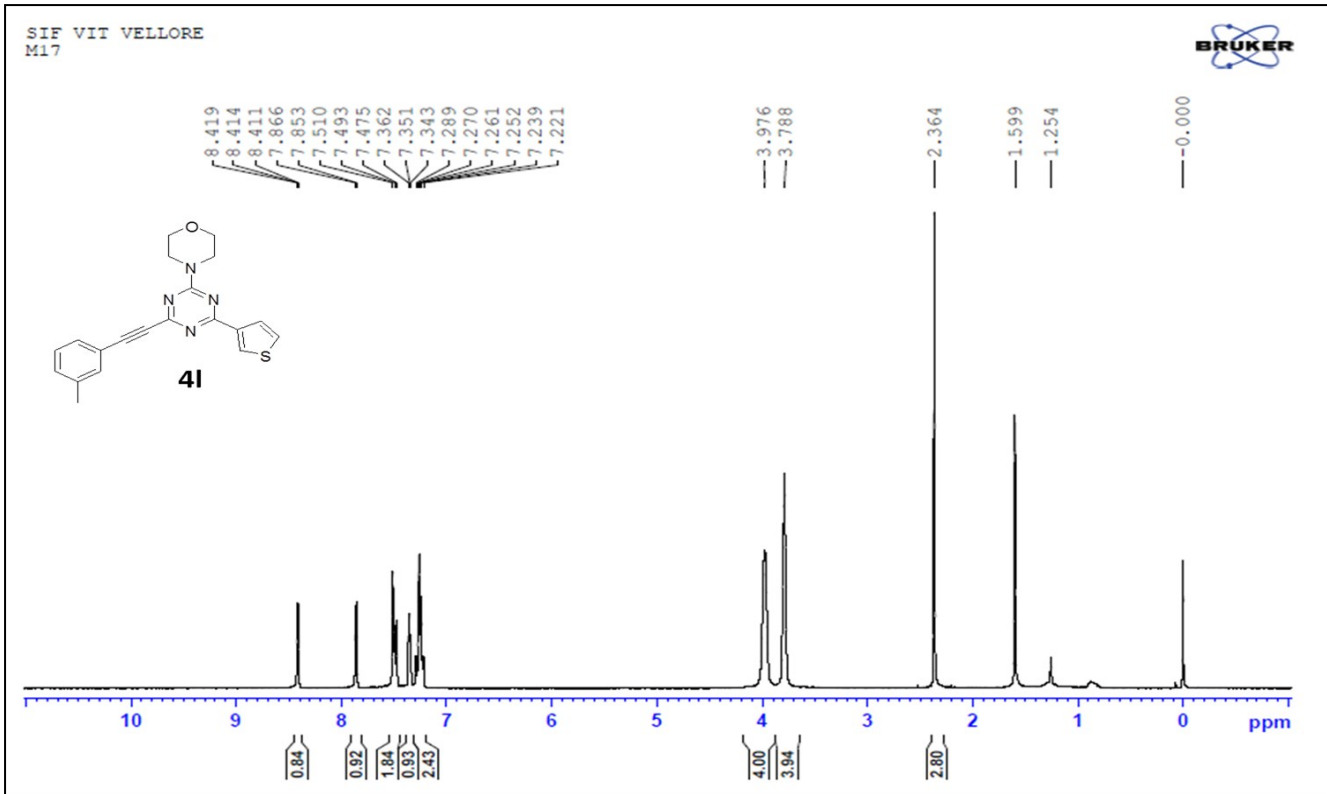
Signature SIF VIJ VELLORE  
M14

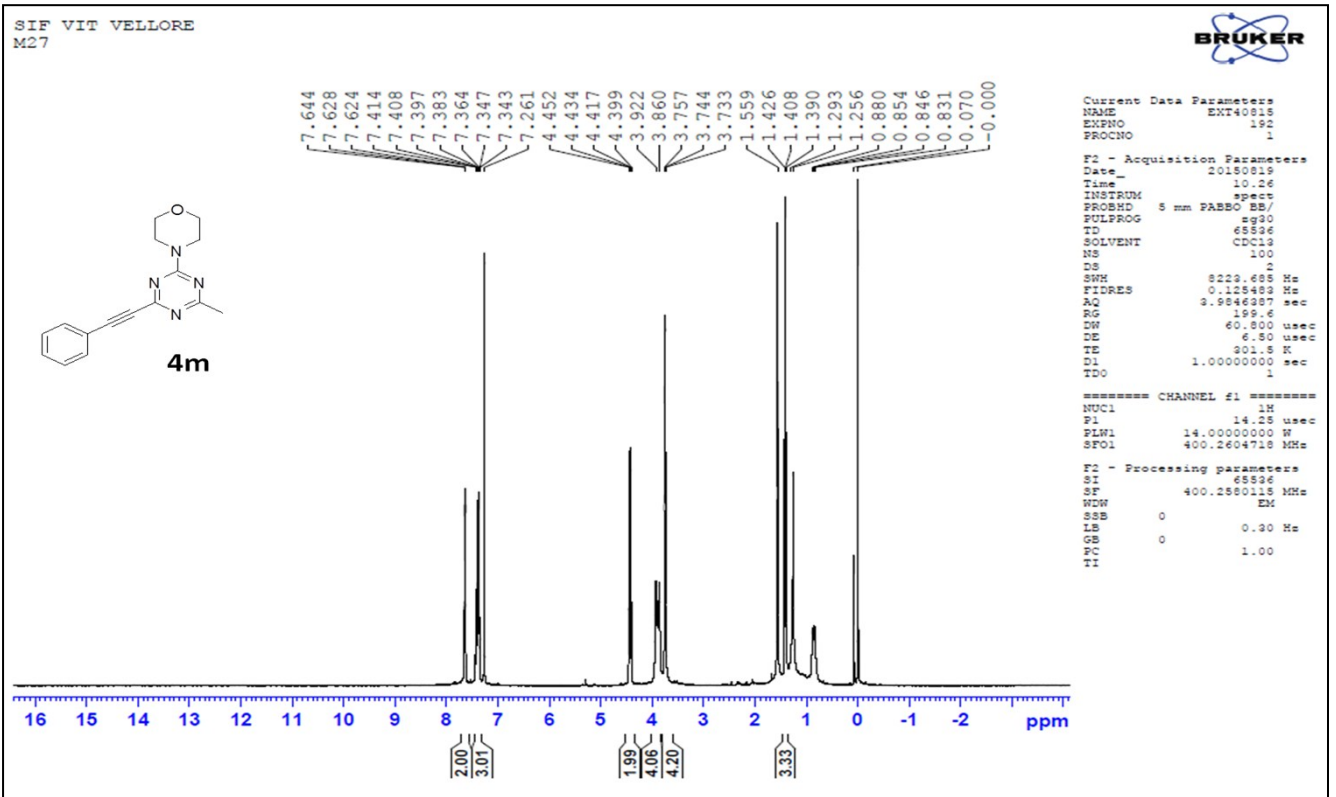
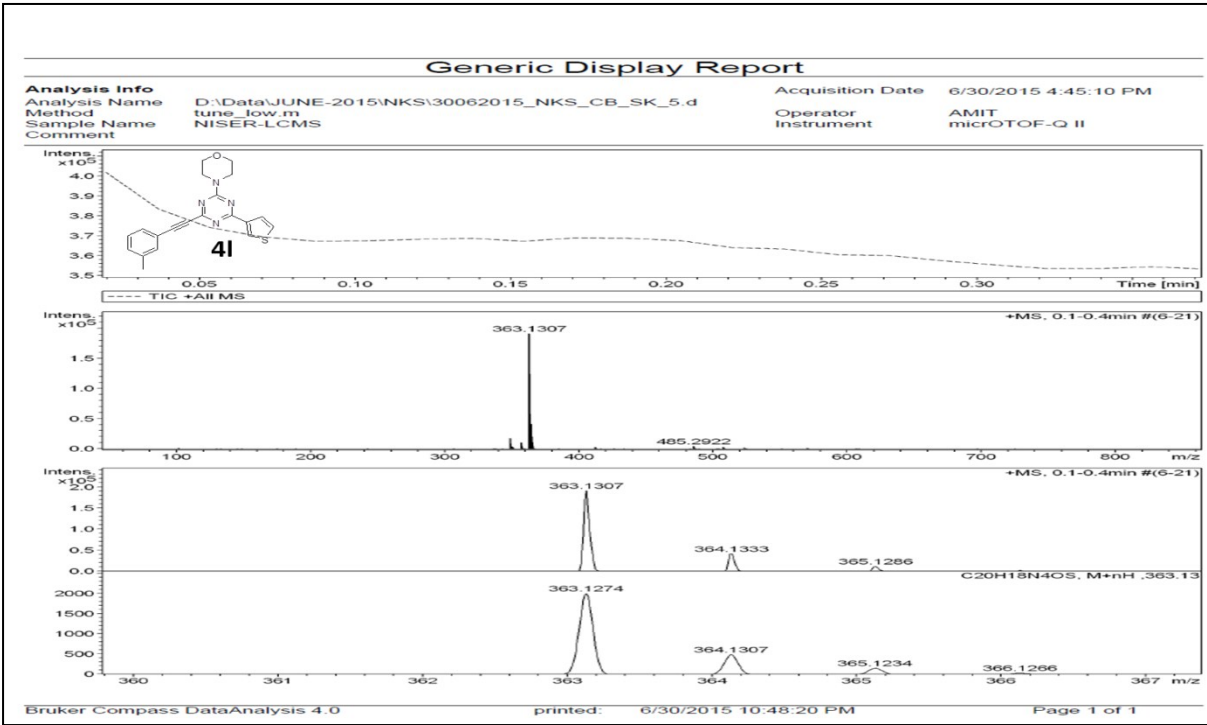


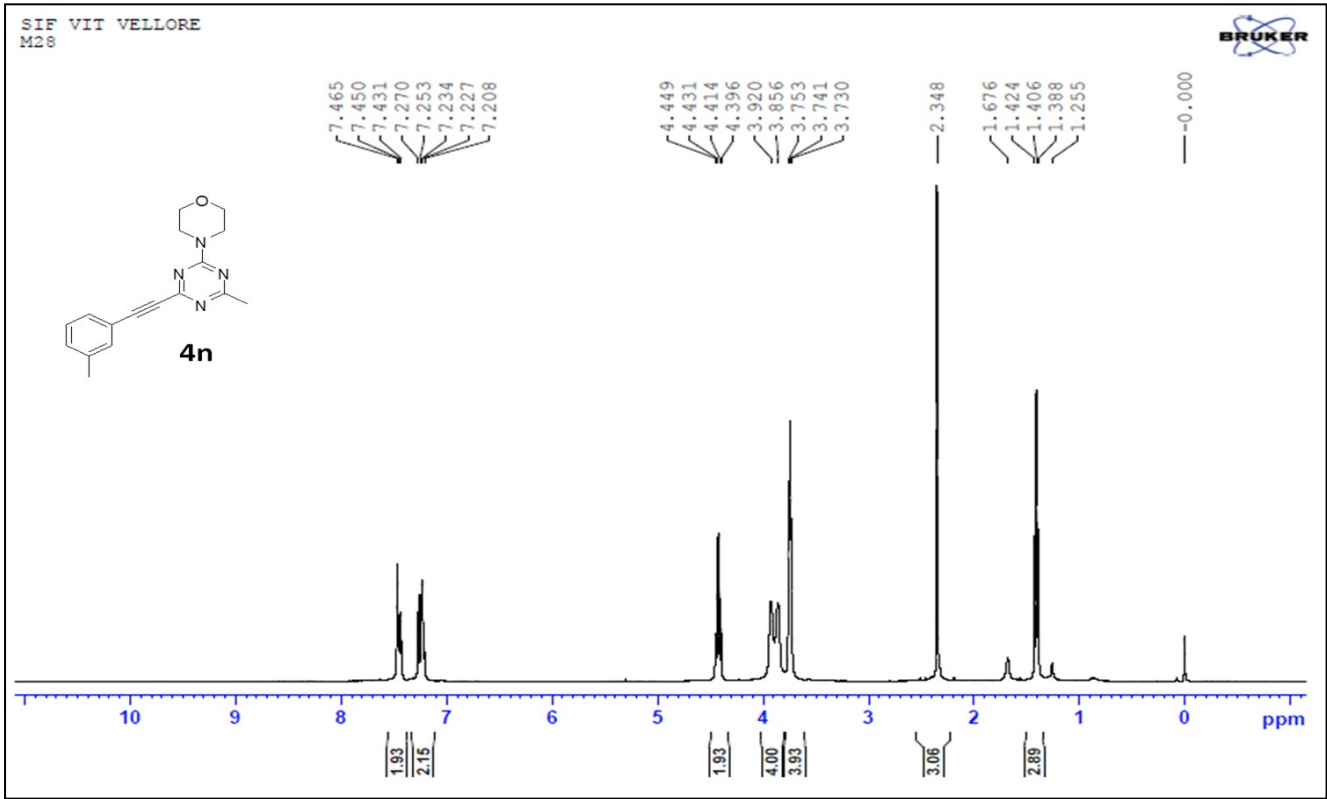
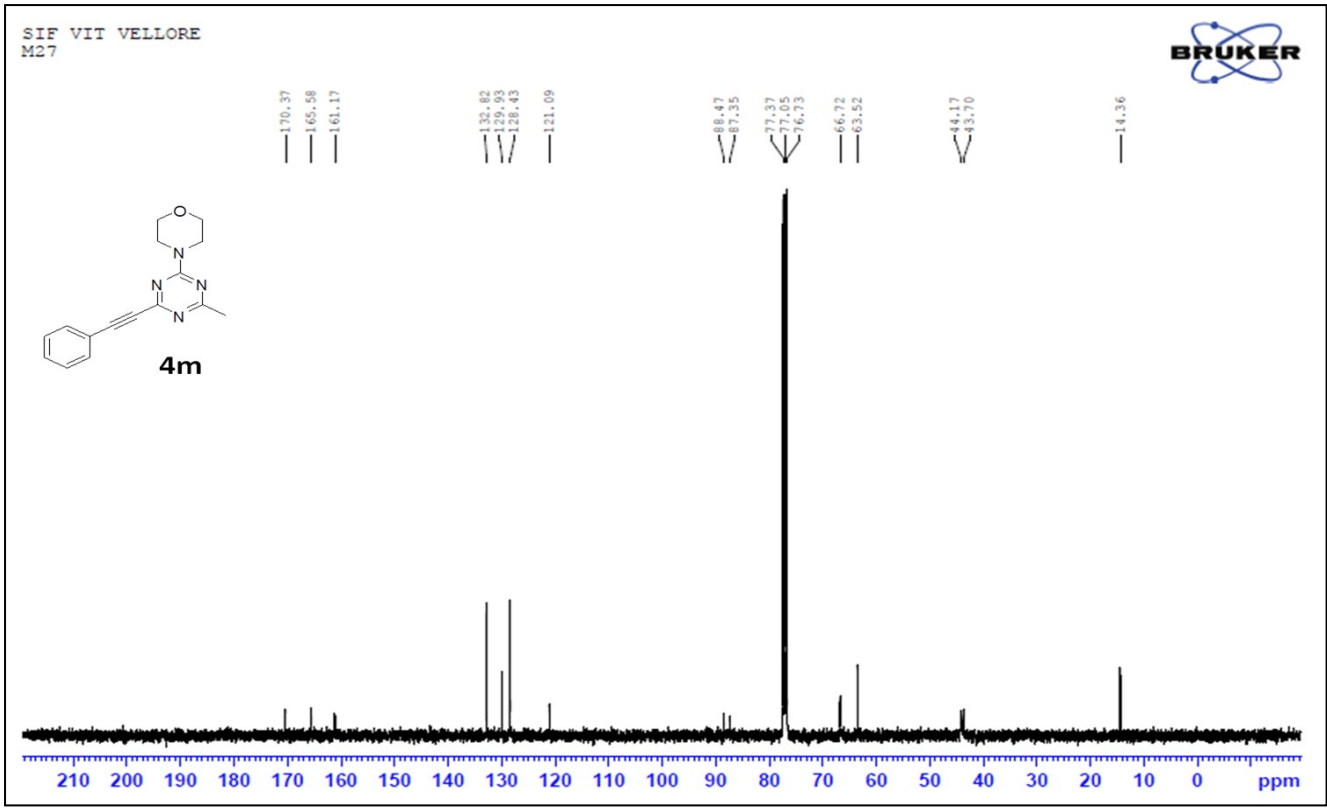












SIF VIT VELLORE  
M28

