



## A NOVEL SYNTHETIC PROTOCOL FOR THE BIS-TRIAZOLONES DERIVATIVES THROUGH CORRESPONDING N<sup>1</sup>-ETHOXYCARBONYL-N<sup>1</sup>-TOSYLHYDRAZONATES

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### ABSTRACT

A simple method has been developed for the synthesis of bis-triazolones **2a-n** starting from N<sup>1</sup>-ethoxycarbonyl-N<sup>1</sup>-tosylhydrazonates **1** and aliphatic diamine. It affords a number of bis-triazolones **2a-n** in reasonable yields. The structures of all new compounds were elucidated using infrared, <sup>1</sup>H and <sup>13</sup>C NMR studies as well as elemental analysis. Some of these reactions provide successful means to produce biologically important structures.

### Keywords

N<sup>1</sup>-ethoxycarbonyl-N<sup>1</sup>-tosylhydrazonates; Bis-Triazolones; Infrared spectroscopy; Nuclear magnetic resonance.



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## INTRODUCTION

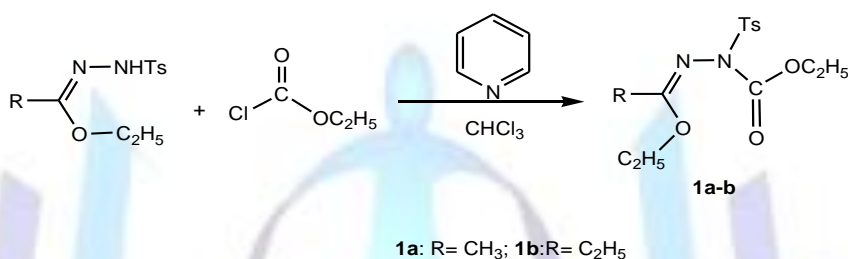
The synthesis of bis-heterocyclic compounds has received considerable attention as it demonstrates a broad spectrum of biological activities such as anticancer, antibacterial, anti-tumor, and anti-mycobacterial activities [1-10]. Among these compounds, we can mention the bis-triazoles derivatives [11-15].

Various methods of the synthesis of 1,2,4- triazolones have been described in the literature [16-18]. Recently, the synthesis of Bis-triazolones, yielded by the reaction of bis-amidrazones with ethyl chloroformate has been illustrated [19].

The present paper proposes a novel method for obtaining Bis-triazolones **2**, as a result of the reaction of N<sup>1</sup>-ethoxycarbonyl-N<sup>1</sup>-tosylhydrazonates **1** with diamine derivatives.

## RESULTS AND DISCUSSION

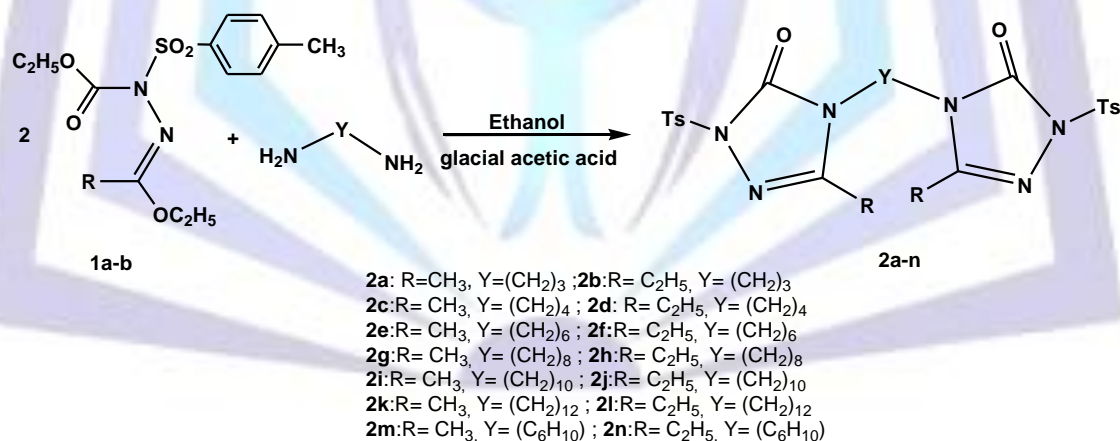
The N<sup>1</sup>-ethoxycarbonyl-N<sup>1</sup>-tosylhydrazonates **1a-b**, used in this study, were prepared by the addition of N<sup>1</sup>-tosylhydrazonates to the ethyl chloroformate using a previously reported method [20].



**Scheme 1:** Synthetic route to N<sup>1</sup>-ethoxycarbonyl N<sup>1</sup>- tosylhydrazonate **1a-b**

These compounds have two electrophilic centers at positions 1, 4 that are very reactive compared with compounds containing a nucleophilic center (e.g., diamines).

In fact, the condensation of two equiv. of N<sup>1</sup>-ethoxycarbonyl-N<sup>1</sup>-tosylhydrazonates **1a-b** in ethanol with one equiv. of diamine derivatives, in the presence of a catalytic amount of glacial acetic acid produced, with a good yield, a cyclized product. The latter is identified as 1,Y-bis-(5-alkyl-2-tosyl-1,2,4-triazol-3-one-4-yl) alkane **2** (Scheme 2).



**Scheme 2:**Synthetic route to bis-triazolones **2 a-n**

The structural assignments of the new compounds were based on their elemental analysis and spectral (IR, <sup>1</sup>H NMR and <sup>13</sup>C NMR) data. The characterisation data of all new compounds is presented in the experimental section. The IR spectra of compounds **2** shows absorption bands in the range of 1590-1610 cm<sup>-1</sup>, which are attributed to the C=N group. However, carbonyl stretching vibration bands are found to appear at around 1735cm<sup>-1</sup>. The NMR spectrum of **2** essentially shows the absence of the signal of ethoxy group of the parent N<sup>1</sup>-ethoxycarbonyl-N<sup>1</sup>-tosylhydrazonates **1**. It also illustrates the presence of the characteristic resonance signals of protons introduced by the diamine derivatives. The <sup>13</sup>C NMR signal for the C=O group is observed at around 151 ppm, whereas the signal of the C=N group is noted at values in the range of 147-150 ppm. Moreover, the elemental analyses are consistent with the proposed formula.



Some of these compounds have already been synthesized from the condensation of bis-amidrazones with ethyl chloroformate as described above[19]. Given the substantially reduced number of bis-amidrazones, the synthesis presented in this paper is found to be advantageous following a more general and lower-cost approach. Furthermore, the proposed method is also proven to have better yields than the others.

## CONCLUSION

The present research study reports the success of a novel strategy in the synthesis and characterization of new 1,Y-bis(5-alkyl-2-tosyl-1,2,4-triazol-3-one-4-yl) alkane derivatives in good yields.

## EXPERIMENTAL

The melting points were determined by Electrothermal 9100 apparatus. The infrared spectra were determined on a Perkin Elmer Spectrum Version 10.00.00 with a precision of  $2\text{ cm}^{-1}$  covering field  $400\text{--}4000\text{ cm}^{-1}$ . NMR spectra were recorded in  $\text{CDCl}_3$  or in  $\text{DMSO-d}_6$  on a Bruker Avance spectrometer (400 MHz for  $^1\text{H}$ , 100 MHz for  $^{13}\text{C}$ ).  $^1\text{H}$  and  $^{13}\text{C}$  chemical shifts are given on the  $\delta$  scale (ppm) and referenced to internal T.M.S. The multiplicities of the signals are indicated by the following abbreviations: s: singlet, d: doublet, t: triplet, q: quadruplet, m: multiplet, br: broad and coupling constants are expressed in Hz. The elemental analyses were performed on a Thermo Finnigan EA 1112 apparatus, both at the Service Commun d'Analyses, University of Lorraine, Nancy.

## GENERAL EXPERIMENTAL PROCEDURE

A mixture of compound 1 (0.01 mol), diamino-alkane (represented in the scheme 1) (0.005 mol) and 4–5 drops of glacial acetic acid in ethanol was heated to reflux for 8 h. The resulting solution was cooled to room temperature and the precipitated solid was filtered and washed with cold ethanol, then dried to get pure product.

### Spectral Data of New Compounds

**1,3-bis(5-methyl-3-tosyl-1,3,4-triazol-2-onyl)propane (2a).** yield (67%), mpq  $130\text{--}132\text{ }^\circ\text{C}$ ; IR spectrum ( $\text{v}/\text{cm}^{-1}$ ): 1711 (C=O), 1602 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.99 (m, 2H), 2.19 (s, 6H), 2.44 (s, 6H), 3.54 (t,  $J=6.9\text{ Hz}$ , 4H), 7.34–7.97 (2AB(Ts),  $J=7.8\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (150.7, 2C=O), (146.1, 2C=N), (21.2, 2CH<sub>3</sub>(Ts)), (11.4, 2CH<sub>3</sub>), (38.7, 2CH<sub>2</sub>-N), (26.9, CH<sub>2</sub>), Carom 127.6–145.5; Anal. Calcd. for  $\text{C}_{23}\text{H}_{26}\text{N}_6\text{O}_6\text{S}_2$ : C, 50.54; H, 4.79; N, 15.37. Found: C, 50.90; H, 5.00; N, 15.60.

**1,3-bis(5-ethyl-3-tosyl-1,3,4-triazol-2-onyl)propane (2b).** yield (57%), mp  $136\text{--}138\text{ }^\circ\text{C}$ ; IR spectrum ( $\text{v}/\text{cm}^{-1}$ ): 1709 (C=O), 1597 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 0.83 (t,  $J=7.5\text{ Hz}$ , 3H), 1.53 (m, 2H), 1.98 (q,  $J=7.5\text{ Hz}$ , 4H), 2.28 (s, 6H), 3.21 (t,  $J=6.9\text{ Hz}$ , 4H), 7.39–7.65 (2AB(Ts),  $J=8.1\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (151.4, 2C=O), (145.4, 2C=N), (21.4, 2CH<sub>3</sub>(Ts)), (9.0, 2CH<sub>3</sub>), (13.8, 2CH<sub>2</sub>), (38.3, 2CH<sub>2</sub>-N), (26.2, CH<sub>2</sub>), Carom 127.6–139.0; Anal. Calcd. for  $\text{C}_{25}\text{H}_{30}\text{N}_6\text{O}_6\text{S}_2$ : C, 52.25; H, 5.26; N, 14.62. Found: C, 52.50; H, 5.50; N, 14.85.

**1,4-bis(5-methyl-3-tosyl-1,3,4-triazol-2-onyl)butane (2c).** yield (55%), mp  $260\text{--}262\text{ }^\circ\text{C}$ ; IR spectrum,  $\text{v}$ ,  $\text{cm}^{-1}$ : 1716 (C=O), 1593 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{DMSO-d}_6$ ):  $\delta$  (ppm): 1.44 (m, 4H), 2.16 (s, 6H), 2.41 (s, 6H), 3.47 (m, 4H), 7.45–7.83 (2AB(Ts),  $J=8.1\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{DMSO-d}_6$ ):  $\delta$  (ppm): (151.1, 2C=O), (148.4, 2C=N), (21.0, 2CH<sub>3</sub>(Ts)), (11.5, 2CH<sub>3</sub>), (41.5, 2CH<sub>2</sub>-N), (24.7, 2CH<sub>2</sub>), Carom 127.3–145.7; Anal. Calcd. for  $\text{C}_{24}\text{H}_{28}\text{N}_6\text{O}_6\text{S}_2$ : C, 51.42; H, 5.03; N, 14.99. Found: C, 51.75; H, 5.35; N, 15.30.

**1,4-bis(5-ethyl-3-tosyl-1,3,4-triazol-2-onyl)butane (2d).** yield (64%), mp  $226\text{--}228\text{ }^\circ\text{C}$ ; IR spectrum ( $\text{v}/\text{cm}^{-1}$ ): 1735 (C=O), 1591 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{DMSO-d}_6$ ):  $\delta$  (ppm): 1.12 (t,  $J=7.5\text{ Hz}$ , 3H), 1.43 (m, 4H), 2.40 (s, 6H), 2.51 (q,  $J=7.5\text{ Hz}$ , 2H), 3.45 (m, 4H), 7.44–7.84 (2AB(Ts),  $J=7.8\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{DMSO-d}_6$ ):  $\delta$  (ppm): (152.0, 2C=O), (151.3, 2C=N), (9.0, 2CH<sub>3</sub>), (18.5, 2CH<sub>2</sub>), (21.0, 2CH<sub>3</sub>(Ts)), (24.8, 2CH<sub>2</sub>), (38.9, 2CH<sub>2</sub>-N), Carom 127.3–145.7; Anal. Calcd. for  $\text{C}_{26}\text{H}_{32}\text{N}_6\text{O}_6\text{S}_2$ : C, 53.05; H, 5.48; N, 14.28. Found: C, 53.05; H, 5.62; N, 13.99.

**1,6-bis(5-methyl-2-tosyl-1,2,4-triazol-3-one-4-yl)hexane (2e).** yield (80%), mp  $190\text{--}192\text{ }^\circ\text{C}$ ; IR spectrum ( $\text{v}/\text{cm}^{-1}$ ): 1722 (C=O), 1601 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.24 (m, 4H), 1.54 (m, 4H), 2.18 (s, 6H), 2.40 (s, 6H), 3.45 (t,  $J=6.9\text{ Hz}$ , 4H), 7.31–7.93 (2AB(Ts),  $J=7.8\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (151.3, 2C=O), (147.1, 2C=N), (21.8, 2CH<sub>3</sub>(Ts)), (12.2, 2CH<sub>3</sub>), (41.6, 2CH<sub>2</sub>-N), (28.4, 2CH<sub>2</sub>), (25.8, 2CH<sub>2</sub>), Carom 128.2–145.9; Anal. Calcd. for  $\text{C}_{26}\text{H}_{32}\text{N}_6\text{O}_6\text{S}_2$ : C, 53.05; H, 5.48; N, 14.28. Found: C, 53.05; H, 5.62; N, 13.99.

**1,6-bis(5-ethyl-2-tosyl-1,2,4-triazol-3-one-4-yl)hexane (2f).** yield (75%), mp  $194\text{--}196\text{ }^\circ\text{C}$ ; IR spectrum ( $\text{v}/\text{cm}^{-1}$ ): 1735 (C=O), 1594 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.20 (m, 10H), 1.49 (m, 4H), 2.35 (s, 6H), 2.42 (q,  $J=7.2\text{ Hz}$ , 4H), 3.39 (t,  $J=7.2\text{ Hz}$ , 4H), 7.27–7.89 (2AB(Ts),  $J=8.1\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (152.0, 2C=O), (151.3, 2C=N), (22.2, 2CH<sub>3</sub>(Ts)), (19.9, 2CH<sub>2</sub>-CH<sub>3</sub>), (10.2, 2CH<sub>3</sub>-CH<sub>2</sub>), (41.8, 2CH<sub>2</sub>-N), (28.8, 2CH<sub>2</sub>), (26.2, 2CH<sub>2</sub>), Carom 128.5–146.2; Anal. Calcd. for  $\text{C}_{28}\text{H}_{36}\text{N}_6\text{O}_6\text{S}_2$ : C, 54.53; H, 5.88; N, 13.63. Found: C, 54.49; H, 5.81; N, 13.42.





**1,8-bis (5-methyl-2-tosyl-1,2,4-triazol-3-one-4-yl)octane (2g).** yield (85%), mp 162-164 °C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1732 (C=O), 1593 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.00 (m, 4H), 1.38 (m, 4H), 2.11 (s, 6H), 2.31 (s, 6H), 2.43 (m, 4H), 3.39 (t,  $J=6.8\text{Hz}$ , 4H), 7.39-7.74 (2AB(Ts),  $J=7.9\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (151.5, 2C=O), (149.1, 2C=N), (21.5, 2CH<sub>3</sub>(Ts)), (12.0, 2CH<sub>3</sub>), (41.5, 2CH<sub>2</sub>-N), (28.6, 2CH<sub>2</sub>), (28.0, 2CH<sub>2</sub>), (26.0, 2CH<sub>2</sub>), Carom 127.8-146.2; Anal. Calcd for C<sub>28</sub>H<sub>36</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 54.53; H, 5.88; N, 13.63. Found: C, 54.50; H, 5.82; N, 13.59.

**1,8-bis (5-ethyl-2-tosyl-1,2,4-triazol-3-one-4-yl)octane (2h).** yield 82%, mp 174-176 °C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1727 (C=O), 1588 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.04-1.15 (m, 14H), 1.49 (m, 4H), 2.38(s, 6H), 2.53(q,  $J=7.5\text{ Hz}$ , 4H), 3.45(t,  $J=7.3\text{ Hz}$ , 4H), 7.46-7.82 (2AB(Ts),  $J=8.2\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (151.6, 2C=O), (151.0, 2C=N), (21.5, CH<sub>3</sub>(Ts)), (19.0, 2CH<sub>2</sub>-CH<sub>3</sub>), (9.3, 2CH<sub>3</sub>-CH<sub>2</sub>), (41.3, 2CH<sub>2</sub>-N), (28.6, 2CH<sub>2</sub>), (28.0, 2CH<sub>2</sub>), (26.0, 2CH<sub>2</sub>), Carom 128.1-146.7; Anal. Calcd for C<sub>30</sub>H<sub>40</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 55.88; H, 6.25; N, 13.03. Found: C, 55.61; H, 6.23; N, 12.92.

**1,10-bis (5-methyl-2-tosyl-1,2,4-triazol-3-one-4-yl)decane (2i).** yield (90%), mp 180-182 °C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1736 (C=O), 1603 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.20-1.28 (m, 12H), 1.54 (m, 4H), 2.18 (s, 6H), 2.40 (s, 6H), 3.45 (t,  $J=7.2\text{ Hz}$ , 4H), 7.31-7.93 (2AB(Ts),  $J=8.4\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (151.3, 2C=O), (147.2, 2C=N), (21.8, 2CH<sub>3</sub>(Ts)), (12.2, 2CH<sub>3</sub>), (41.9, 2CH<sub>2</sub>-N), (29.2, 2CH<sub>2</sub>), (28.9, 2CH<sub>2</sub>), (28.6, 2CH<sub>2</sub>), (26.4, 2CH<sub>2</sub>), Carom 128.2-145.9; Anal. Calcd for C<sub>30</sub>H<sub>40</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 55.88; H, 6.25; N, 13.03. Found: C, 55.57; H, 6.19; N, 12.82.

**1,10-bis (5-ethyl-2-tosyl-1,2,4-triazol-3-one-4-yl)decane (2j).** yield (76%), mp 188-190 °C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1735 (C=O), 1600 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.08-1.23 (m, 18H), 1.51 (m, 4H), 2.31-2.48 (m, 10H), 3.41 (t,  $J=6.5\text{ Hz}$ , 4H), 7.26-7.92 (2AB(Ts),  $J=7.3\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (151.6, 2C=O), (150.9, 2C=N), (21.8, 2CH<sub>3</sub>(Ts)), (18.2, 2CH<sub>2</sub>-CH<sub>3</sub>), (10.9, 2CH<sub>3</sub>-CH<sub>2</sub>), (41.9, 2CH<sub>2</sub>-N), (29.2, 2CH<sub>2</sub>), (28.9, 2CH<sub>2</sub>), (28.6, 2CH<sub>2</sub>), (26.4, 2CH<sub>2</sub>), Carom 128.2-145.6; Anal. Calcd for C<sub>32</sub>H<sub>44</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 57.12; H, 6.59; N, 12.49. Found: C, 56.99; H, 6.41; N 12.32.

**1,12-bis (5-methyl-2-tosyl-1,2,4-triazol-3-one-4-yl)dodecane (2k).** yield (87%), mp 166-168 °C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1732 (C=O), 1605 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.14 (m, 16H), 1.50 (m, 4H), 2.14 (s, 6H), 2.36 (s, 6H), 3.41 (m, 4H), 7.26-7.90 (2AB(Ts),  $J=6.9\text{ Hz}$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (150.8, 2C=O), (146.6, 2C=N), (21.3, 2CH<sub>3</sub>(Ts)), (11.7, 2CH<sub>3</sub>), (41.5, 2CH<sub>2</sub>-N), (28.8, 2CH<sub>2</sub>), (28.5, 2CH<sub>2</sub>), (28.5, 2CH<sub>2</sub>), (28.3, 2CH<sub>2</sub>), (26.0, 2CH<sub>2</sub>), Carom 127.7-145.3; Anal. Calcd for C<sub>32</sub>H<sub>44</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 57.12; H, 6.59; N, 12.49. Found: C, 57.11; H, 6.61; N; 12.58.

**1,12-bis (5-ethyl-2-tosyl-1,2,4-triazol-3-one-4-yl)dodecane (2l).** yield (47%), mp 166-168 °C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1730 (C=O), 1607 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 0.98 (t,  $J=7.5\text{Hz}$ , 3H), 1.59 (m, 16H), 2.10 (q,  $J=7.5\text{Hz}$ , 2H), 2.37 (s, 6H), 2.80 (m, 4H), 4.66 (m, 4H), 7.31-7.79 (2AB(Ts),  $J=8.1\text{ Hz}$ , 8Harom); Anal. Calcd for C<sub>32</sub>H<sub>44</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 57.12; H, 6.59; N, 12.49. Found: C, 57.11; H, 6.61; N; 12.58.

**1,2-bis(5-methyl-3-tosyl-1,3,4-triazol-2-onyl)cyclohexane (2m).** yield (60%), mp 180-182°C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1721 (C=O), 1585 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.36 (m, 4H), 1.76 (m, 4H), 2.00 (s, 6H), 2.45 (s, 6H), 4.63 (t,  $J=6.3\text{ Hz}$ , 2H), 7.33-7.82 (2AB(Ts),  $J=8.1$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (150.4, 2C=O), (146.9, 2C=N), (11.2, 2CH<sub>3</sub>), (21.7, 2CH<sub>3</sub>(Ts)), (24.4, 2CH<sub>2</sub>), (29.2, 2CH<sub>2</sub>), (53.5, 2CH-N), Carom 127.5-145.6; Anal. Calcd. for C<sub>26</sub>H<sub>30</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 53.23; H, 5.25; N, 14.32. Found: C, 53.05; H, 5.62; N, 13.99.

**1,2-bis(5-éthyl-3-tosyl-1,3,4-triazol-2-onyl)cyclohexane (2n).** yield (30%), mp 190-192°C; IR spectrum ( $\nu/\text{cm}^{-1}$ ): 1718 (C=O), 1595 (C=N);  $^1\text{H}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): 1.09 (t,  $J=7.5\text{Hz}$ , 6H), 1.18 (m, 4H), 1.57 (m, 4H), 1.97 (q,  $J=7.5\text{Hz}$ , 2H), 2.23 (s, 6H), 3.97 (t,  $J=6.9\text{ Hz}$ , 2H), 7.42-7.67 (2AB(Ts),  $J=8.1$ , 8Harom);  $^{13}\text{C}$  NMR spectrum ( $\text{CDCl}_3$ ):  $\delta$  (ppm): (151.7, 2C=O), (148.6, 2C=N), (8.9, 2CH<sub>3</sub>), (14.3, 2CH<sub>2</sub>), (21.3, 2CH<sub>3</sub>(Ts)), (29.2, 2CH<sub>2</sub>), (42.4, 2CH<sub>2</sub>), (61.4, 2CH-N), Carom 123.9-143.4; Anal. Calcd. for C<sub>28</sub>H<sub>34</sub>N<sub>6</sub>O<sub>6</sub>S<sub>2</sub>: C, 54.71; H, 5.57; N, 13.67. Found: C, 54.49; H, 5.81, N, 13.42.

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