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## A MILD AND EFFICIENT SYNTHESIS OF NEW 3-PHENYL- THIENOTRIAZOLOPYRIMIDINE DERIVATIVES USING IODOBENZENE DIACETATE

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**Abstract** – New 3-phenylthieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine **9a-i** and 3-phenylthieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine **10a-i** derivatives were easily synthesized at rt in high yield by the oxidative cyclization of thienopyrimidinyl hydrazones using iodobenzene diacetate.

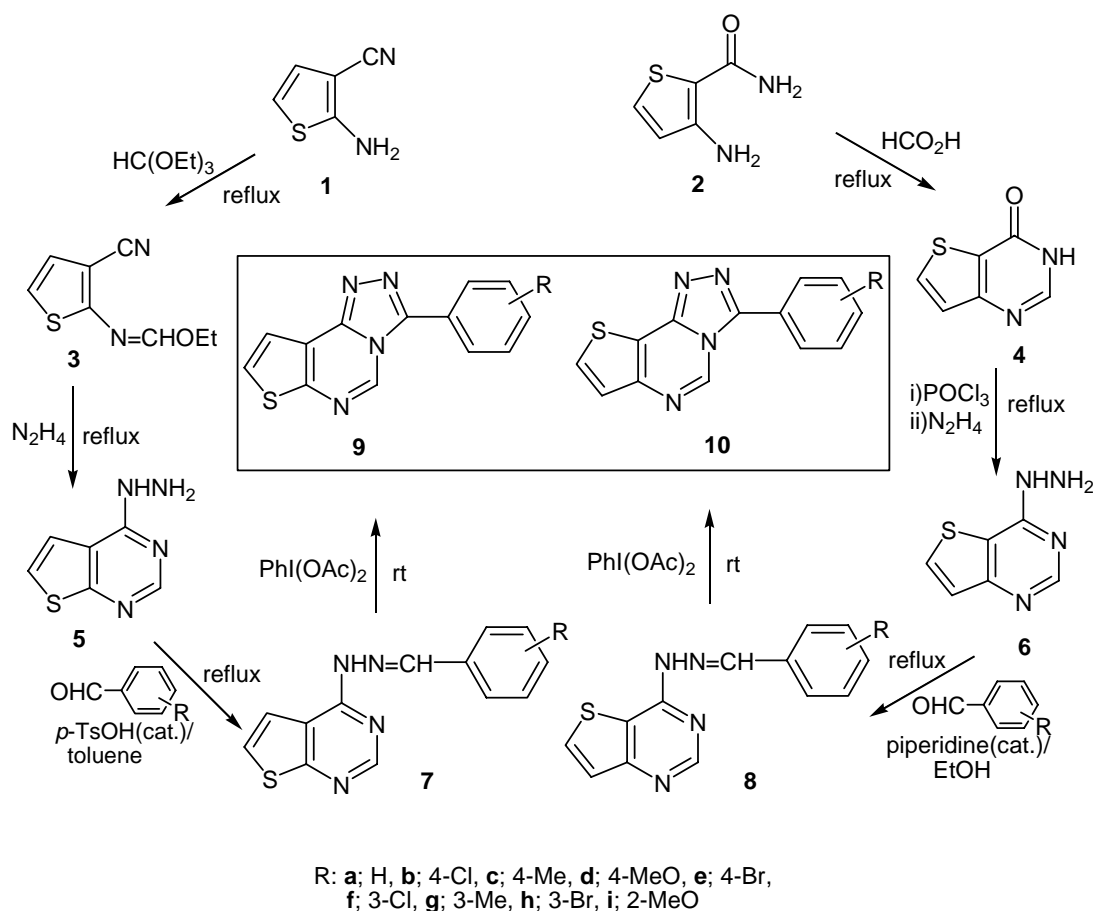
### INTRODUCTION

Various thienopyrimidines have been synthesized and investigated in relation with their biological and pharmacological activities. Recently, a novel class of thieno[3,2-*d*]pyrimidine has been identified as potent inhibitors of VEGF receptor-2 kinase, which is a key component of the signaling pathway responsible for the sprouting and maturation of new blood vessels from tumors.<sup>1</sup> New thieno[2,3-*d*]pyrimidine derivatives were also prepared and studied as selective and potent ligands for the 5-HT<sub>3</sub> receptor.<sup>2</sup> Furthermore, derivatives of 1,2,4-triazole have also received considerable attention because of their therapeutic importance. The 1,2,4-triazole moiety is present, for example, in certain antiviral,<sup>3</sup> antiasthmatic<sup>4</sup> and antibacterial<sup>5</sup> drugs.

Therefore, we have planned to synthesize new 3-phenylthienotriazolopyrimidine derivatives **9** and **10** that are fused with thienopyrimidine and 1,2,4-triazole moiety, which may have a broad range of biological activities.

Many substituted thienotriazolopyrimidine derivatives have been prepared since the first synthesis of thienotriazolopyrimidine by Robba *et al.*<sup>6</sup> But, literature survey showed that only two 3-substituted thienotriazolopyrimidine derivatives have been reported.<sup>6</sup>

As a continuation of our previous work on thienopyrimidines,<sup>7</sup> we wish to report herein the synthesis of new 3-phenylthienotriazolopyrimidine derivatives using iodobenzene diacetate in oxidative cyclization reaction.



Scheme 1

## RESULTS AND DISCUSSION

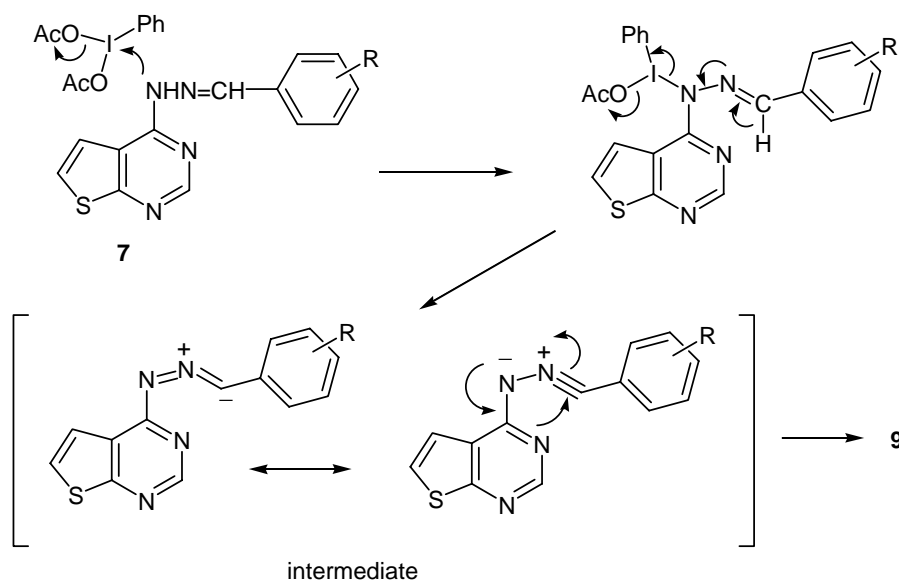
The oxidative cyclization of pyrimidine hydrazone to triazolopyrimidine compound has been usually accomplished using bromine/acetic acid or thionyl chloride. These reagents are often associated with several drawbacks such as toxicity, poor yield, and need of high reaction temperature. Iodobenzene diacetate as one of organohypervalent iodine reagent has been used for many chemical transformations due to its low toxicity, ready availability and ease of handling.<sup>8</sup>

The target compounds **9** and **10** were prepared as shown Scheme 1 through a series of reactions starting with 2-aminothiophene-3-carbonitrile (**1**) or ethyl 3-amino-thiophene-2-carboxamide (**2**)<sup>7</sup> which were obtained by the method of Gewald.<sup>9</sup> Cyclization of **1** and **2** with triethyl orthoformate or formic acid afforded *N*-(3-cyanothiophen-2-yl)formimidic acid ethyl ester (**3**) or 3*H*-thieno[3,2-*d*]pyrimidin-4-one (**4**), respectively. Reaction of **3** with hydrazine hydrate gave 4-hydrazinothieno[2,3-*d*]pyrimidine (**5**), while treatment of **4** with phosphorus oxychloride in the presence of dimethylaniline as catalyst led to 4-chlorothieno[3,2-*d*]pyrimidine which then underwent nucleophilic substitution with hydrazine hydrate resulting in 4-hydrazinothieno[3,2-*d*]pyrimidine (**6**). The hydrazone derivatives, **7a-i** or **8a-i**, were synthesized by condensation of **5** or **6** with the corresponding benzaldehydes in toluene in the presence of

catalytic amount of *p*-TsOH. For **8a-i**, marked improvement in yield could be made when the reaction took place in absolute EtOH in the presence of few drops of piperidine as catalyst.

The oxidative cyclization of **7a-i** and **8a-i** to 3-phenylthieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine **9a-i** and 3-phenylthieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine **10a-i** derivatives, respectively, was achieved easily using iodobenzene diacetate. For instance, when a solution of **7a** or **8a** in dichloromethane was treated with iodobenzene diacetate at rt for 1 h, rapid reaction occurred to give **9a** or **10a** as crystalline solid in 75–85% yield.

The mechanism of cyclization reaction is not certain, but it is assumed that the reaction took place via cyclization of intermediate through the electrophilic attack of iodobenzene diacetate on **7**, followed by elimination of iodobenzene and acetic acid (Scheme 2).



Scheme 2

The structures of all compounds were confirmed by elemental analyses and spectroscopic studies.

In conclusion, the facile and mild synthesis of new 3-phenylthienotriazolopyrimidine derivatives using iodobenzene diacetate in oxidative cyclization reaction was developed.

## EXPERIMENTAL

Melting points were determined in capillary tubes on Büchi apparatus and are uncorrected. Each compound of the reactions was checked on thin-layer chromatography of Merck Kieselgel 60F<sub>254</sub> and purified by column chromatography using Merck silica gel (70-230 mesh). The <sup>1</sup>H NMR spectra were recorded on Bruker DRX-300 FT-NMR spectrometer (300 MHz) with Me<sub>4</sub>Si as internal standard and

chemical shifts are given in ppm ( $\delta$ ). Electron ionization mass spectra were recorded on a HP 59580 B spectrometer. Elemental analyses were performed on a Carlo Erba 1106 elemental analyzer.

**General procedure for the preparation of *N*-substituted benzylidene-*N'*-thieno[2,3-*d*]pyrimidin-4-ylhydrazine 7a-i and *N*-substituted benzylidene-*N'*-thieno[3,2-*d*]pyrimidin-4-ylhydrazine 8a-i derivatives:**

A mixture of 4-hydrazinothieno[2,3-*d*]pyrimidine (**5**) (0.1 mol), each benzaldehyde (0.12 mol) and *p*-TsOH monohydrate (0.3 g) in toluene (200 mL) was stirred and refluxed for 24 h with a Dean-Stark trap. The reaction mixture was cooled in an ice-water bath and the solid which had formed was collected by filtration, washed with Et<sub>2</sub>O and crystallized to give **7a-i**.

A mixture of 4-hydrazinothieno[3,2-*d*]pyrimidine (**6**) (0.1 mol), each benzaldehyde (0.12 mol) and piperidine (0.1 g) in absolute EtOH (100 mL) was stirred and refluxed for 24 h. The solvent was evaporated and the residue was purified by recrystallization in CHCl<sub>3</sub> or column chromatography in EtOAc-hexane to give **8a-i**.

**General procedure for the preparation of 3-phenylthieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine 9a-i and 3-phenylthieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine 10a-i derivatives:**

To a solution of *N*-substituted benzylidene-*N'*-thieno[2,3-*d*]pyrimidin-4-yl hydrazine **7a-i** or *N*-substituted benzylidene-*N'*-thieno[3,2-*d*]pyrimidin-4-yl hydrazine **8a-i** (0.01 mol) in dry CH<sub>2</sub>Cl<sub>2</sub> (20 mL), iodobenzene diacetate (0.01 mol) was slowly added. The reaction mixture was stirred for 1 h at rt. After evaporation the precipitate was filtered and recrystallized from CHCl<sub>3</sub>.

**3-Phenylthieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9a):**

85% Yield, mp 206 - 207 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  (ppm) 9.00 (s, 1H, H-5), 7.97 (d, *J* = 5.9 Hz, 1H, thiophene H-8), 7.87 (d, 2H, H-2' and H-6'), 7.72 (d, *J* = 5.9 Hz, 1H, H-9), 7.63 (m, 3H, H-3', H-4' and H-5'); MS: (m/z) 252 (M<sup>+</sup>), 149, 122. *Anal.* Calcd for C<sub>13</sub>H<sub>8</sub>N<sub>4</sub>S: C, 61.89; H, 3.20, N, 22.21. Found: C, 61.80; H, 3.14; N, 22.30.

**3-(4-Chlorophenyl)thieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9b):**

82% Yield, mp 269 - 270 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  8.97 (s, 1H, H-5), 7.97 (d, *J* = 5.9 Hz, 1H, H-8), 7.86 (d, 2H, H-3' and H-5'), 7.74 (d, *J* = 5.9 Hz, 1H, H-9), 7.65 (d, 2H, H-2' and H-6'); MS: (m/z) 286 (M<sup>+</sup>), 152, 134. *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>ClN<sub>4</sub>S: C, 54.45; H, 2.46, N, 19.54. Found: C, 54.39; H, 2.40; N, 19.62.

**3-*p*-Tolylthieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9c):**

75% Yield, mp 195 - 196 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>):  $\delta$  8.99 (s, 1H, H-5), 7.97 (d, *J* = 6.0 Hz, 1H, H-8), 7.78 (d, 2H, H-2' and H-6'), 7.71 (d, *J* = 5.9 Hz, 1H, H-9), 7.46 (d, 2H, H-3' and H-5'), 2.49 (s, 3H, methyl); MS: (m/z) 266 (M<sup>+</sup>), 149, 132. *Anal.* Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>S: C, 63.14; H, 3.78, N, 21.04. Found: C, 62.99; H, 3.68; N, 21.92.

**3-(4-Methoxyphenyl)thieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9d):**

83% Yield, mp 239 - 240 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.97 (s, 1H, H-5), 7.98 (d, *J* = 6.0 Hz, 1H, t H-8), 7.79 (d, 2H, H-2' and H-6'), 7.71 (d, *J* = 5.9 Hz, 1H, H-9), 7.17 (d, 2H, H-3' and H-5'), 3.93 (s, 3H, methyl); MS: (m/z) 282 (M<sup>+</sup>), 151, 132. *Anal.* Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>OS: C, 59.56; H, 3.57, N, 19.85. Found: C, 60.48; H, 3.62; N, 20.12.

**3-(4-Bromophenyl)thieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9e):**

80% Yield, mp 258 - 259 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.97 (s, 1H, H-5), 7.96 (d, *J* = 6.0 Hz, 1H, H-8), 7.79 - 7.71 (m, 4H, H-2', H-3', H-5' and H-6'), 7.61 (d, *J* = 6.0 Hz, 1H, H-9); MS: (m/z) 331 (M<sup>+</sup>), 251, 149, 135. *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>BrN<sub>4</sub>S: C, 47.15; H, 2.13, N, 16.92. Found: C, 46.98; H, 2.22; N, 17.03.

**3-(3-Chlorophenyl)thieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9f):**

79% Yield, mp 242 - 243 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.00 (s, 1H, H-5), 7.98 (d, *J* = 6.0 Hz, 1H, H-8), 7.91 (s, 1H, H-2'), 7.74 - 7.72 (m, 2H, H-9, H-4'), 7.60 - 7.56 (m, 2H, H-5' and H-6'); MS: (m/z) 286 (M<sup>+</sup>), 149, 135, 122. *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>ClN<sub>4</sub>S: C, 54.45; H, 2.46, N, 19.54. Found: C, 54.88; H, 2.32; N, 19.39.

**3-*m*-Tolylthieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9g):**

70% Yield, mp 198 - 199 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.62 (s, 1H, H-5), 7.99 (d, *J* = 6.0 Hz, 1H, H-8), 7.82 (d, 1H, H-6'), 7.70 (d, *J* = 6.0 Hz, 1H, H-9), 7.68 (s, 1H, H-2'), 7.30 (t, 1H, H-5'), 7.26 (d, 1H, H-4'); MS: (m/z) 265 (M<sup>+</sup>-H), 149, 132. *Anal.* Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>S: C, 63.14; H, 3.78, N, 21.04. Found: C, 62.88; H, 3.64; N, 21.43.

**3-(3-Bromophenyl)thieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9h):**

84% Yield, mp 262 - 263 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.00 (s, 1H, H-5), 8.06 (s, 1H, H-2') 7.98 (d, *J* = 6.0 Hz, 1H, H-8), 7.84 - 7.77 (m, 3H, H-9, H-4' and H-6'), 7.56 (t, 1H, H-5'); MS: (m/z) 331 (M<sup>+</sup>), 196, 148. *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>BrN<sub>4</sub>S: C, 47.15; H, 2.13, N, 16.92. Found: C, 46.88; H, 2.31; N, 17.01.

**3-(2-Methoxy phenyl)thieno[3,2-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (9i):**

80% Yield, mp 162 - 164 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.01 (s, 1H, H-5), 7.96 (d, *J* = 6.0 Hz, 1H, H-8), 7.71 (d, *J* = 5.9 Hz, 1H, H-9), 7.64 - 7.55 (m, 2H, H-4' and H-6'), 7.50 (t, 1H, H-4') 7.41 (d, 1H, H-3'), 2.50 (s, 3H, methyl); MS: (m/z) 282 (M<sup>+</sup>). *Anal.* Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>OS: C, 59.56; H, 3.57, N, 19.85. Found: C, 59.88; H, 3.74; N, 20.26.

**3-Phenylthieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10a):**

75% Yield, mp 156 - 157 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.30 (s, 1H, H-5), 8.34 (m, 2H, H-2' and H-6'), 7.87 (d, *J* = 5.9 Hz, 1H, thiophene H-8), 7.66 (d, *J* = 5.9 Hz, 1H, H-7), 7.54 - 7.45 (m, 3H, H-3', H-4' and H-5'); MS: (m/z) 252 (M<sup>+</sup>), 149, 118. *Anal.* Calcd for C<sub>13</sub>H<sub>8</sub>N<sub>4</sub>S: C, 61.89; H, 3.20, N, 22.21. Found: C, 62.30; H, 3.26; N, 22.50.

**3-(4-Chlorophenyl)thieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10b):**

77% Yield, mp 264 – 265 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.02 (s, 1H, H-5), 7.87 - 7.79 (m, 3H, H-8, H-3' and H-5'), 7.65 - 7.60 (m, 3H, H-7, H-2' and H-6'); MS: (m/z) 286 (M<sup>+</sup>), 134. *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>ClN<sub>4</sub>S: C, 54.45; H, 2.46, N, 19.54. Found: C, 54.29; H, 2.41; N, 19.68.

**3-*p*-Tolylthieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10c):**

70% Yield, mp 206 – 208 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.20 (s, 1H, H-5), 8.23 (m, 2H, H-2' and H-6'), 7.86 (d, *J* = 5.9 Hz, 1H, H-8), 7.63 (d, *J* = 6.0 Hz, 1H, H-7), 7.35 (m, 2H, H-3' and H-5'); MS: (m/z) 266 (M<sup>+</sup>), 131, 91. *Anal.* Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>S: C, 63.14; H, 3.78, N, 21.04. Found: C, 62.85; H, 3.71; N, 21.35.

**3-(4-Methoxyphenyl)thieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10d):**

70% Yield, mp 142 – 143 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.29 (s, 1H, H-5), 8.25 (m, 2H, H-2' and H-6'), 7.87 (d, *J* = 5.9 Hz, 1H, H-8), 7.64 (d, *J* = 6.0 Hz, 1H, H-7), 7.05 (m, 2H, H-3' and H-5'), 3.90 (s, 3H, methyl); MS: (m/z) 282 (M<sup>+</sup>), 132. *Anal.* Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>OS: C, 59.56; H, 3.57, N, 19.85. Found: C, 60.20; H, 3.68; N, 20.22.

**3-(4-Bromophenyl)thieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10e):**

78% Yield, mp 168 – 169 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.01 (s, 1H, H-5), 7.96 (d, *J* = 6.0 Hz, 1H, H-8), 7.75 - 7.66 (m, 4H, H-2', H-3', H-5' and H-6'), 7.65 (d, *J* = 6.0 Hz, 1H, H-7); MS: (m/z) 331 (M<sup>+</sup>). *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>BrN<sub>4</sub>S: C, 47.15; H, 2.13, N, 16.92. Found: C, 46.84; H, 2.29; N, 17.26.

**3-(3-Chlorophenyl)thieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10f):**

81% Yield, mp 158 – 159 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.04 (s, 1H, H-5), 7.91 (d, *J* = 6.0 Hz, 1H, H-8), 7.81 (s, 1H, H-2'), 7.75 - 7.72 (m, 2H, H-7, H-4'), 7.62 - 7.59 (m, 2H, H-5' and H-6'); MS: (m/z) 286 (M<sup>+</sup>). *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>ClN<sub>4</sub>S: C, 54.45; H, 2.46, N, 19.54. Found: C, 54.63; H, 2.30; N, 19.30.

**3-*m*-Tolylthieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10g):**

72% Yield, mp 274 – 276 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.06 (s, 1H, H-5), 7.79 (d, *J* = 6.0 Hz, 1H, H-8), 7.72 (s, 1H, H-2'), 7.67 (d, 1H, H-6'), 7.62 (d, *J* = 6.0 Hz, 1H, H-7), 7.55 (t, 1H, H-5'), 7.44 (d, 1H, H-4'); MS: (m/z) 266 (M<sup>+</sup>), 149, 132. *Anal.* Calcd for C<sub>14</sub>H<sub>10</sub>N<sub>4</sub>S: C, 63.14; H, 3.78, N, 21.04. Found: C, 63.43; H, 3.60; N, 21.33.

**3-(3-Bromophenyl)thieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10h):**

77% Yield, mp 186 – 187 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 8.98 (s, 1H, H-5), 8.05 (s, 1H, H-2') 7.95 (d, *J* = 6.0 Hz, 1H, H-8), 7.86 - 7.79 (m, 3H, H-9, H-4' and H-6'), 7.59 (t, 1H, H-5'); MS: (m/z) 331 (M<sup>+</sup>). *Anal.* Calcd for C<sub>13</sub>H<sub>7</sub>BrN<sub>4</sub>S: C, 47.15; H, 2.13, N, 16.92. Found: C, 46.94; H, 1.91; N, 17.10.

**3-(2-Methoxyphenyl)thieno[2,3-*e*][1,2,4]triazolo[4,3-*c*]pyrimidine (10i):**

70% Yield, mp 191 – 192 °C; <sup>1</sup>H NMR (CDCl<sub>3</sub>): δ 9.00 (s, 1H, H-5), 7.94 (d, *J* = 6.0 Hz, 1H, H-8), 7.75 (d, *J* = 5.9 Hz, 1H, H-9), 7.68 - 7.57 (m, 2H, H-4' and H-6'), 7.52 (t, 1H, H-4') 7.46 (d, 1H, H-3'), 2.52 (s,

3H, methyl); MS: (m/z) 282 ( $M^+$ ). *Anal.* Calcd for  $C_{14}H_{10}N_4OS$ : C, 59.56; H, 3.57, N, 19.85. Found: C, 59.48; H, 3.78; N, 19.66.

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