© 2017 IJSRST | Volume 3 | Issue 9 | Print ISSN: 2395-6011 | Online ISSN: 2395-602X
National Conference on Green Approach towards Environment and Chemical Science

One Pot Synthesis of 6,7-Diimino imidazolo[2,3-b]pyrimido[5,6-e] pyrimido[2,3-b][1,3]benzothiazole Derivatives

Sambhaji P. Vartale*[1], Sandeep G. Sontakke[2] , Prashant N. Ubale[1]
[1]P.G. Research Centre, Department of Chemistry, Yeshwant Mahavidyalaya, Nanded, Maharashtra, India
[2]Department of Chemistry, Dr. B. N. Purandare Arts, Smt.S.G.Gupta Commerce and Science College, Lonavala , Maharashtra, India

ABSTRACT

We have reported the synthesis of 6,7-Diimino imidazolo[2,3-b]pyrimido[5,6-e]pyrimido[2,3-b][1,3]benzothiazole derivatives by condensing 3-Cyano-4-imino-2-methylthio-4H-pyrimido[1,2-a]benzimidazole with 2-Amino benzothiazole derivatives in presence of N,N'-dimethyl formamide and anhydrous potassium carbonate as a catalyst under reflux condition. The structures of all newly synthesized cyclic compounds were confirmed on the basis of spectral analysis, IR, 1H NMR and Mass spectral properties.

Keywords : Benzothiazole, Pyrimido, DMF, Potassium Carbonate, Spectral Properties.

I. INTRODUCTION

Benzothiazole derivatives are significant class of fused heterocycles due to their extensive range of pharmacological potential, in addition to this its value in the synthesis of drug molecules and natural products. It shows anti-inflammatory[1], analgesic[2], antioxidant[3], antidiabetics[4], antimicrobial[5], antituberculosis[6], vasodilator[7], antitumor[8], antiproliferative[9], anticancer activity[10]. These things inspire us to synthesize some pyrimido-benzothiazole derivatives.

II. Material and Methods

All the chemicals used in present works are from analytical grade and used without further purification. Melting points of the products were determined in open capillary tubes on an electrothermal melting point apparatus and were uncorrected. All these reactions were monitored by TLC. IR spectra were recorded on Shimadzu FT-IR spectrophotometer, ^{1}HNMR spectra were obtained on Bruker avance spectrophotometer 500 MHz in DMSO-d6 using TMS as an internal standard. Mass spectrums were analyzed on GC-MS spectrometer using the ESI 70 eVtechnique.

III. General Procedure

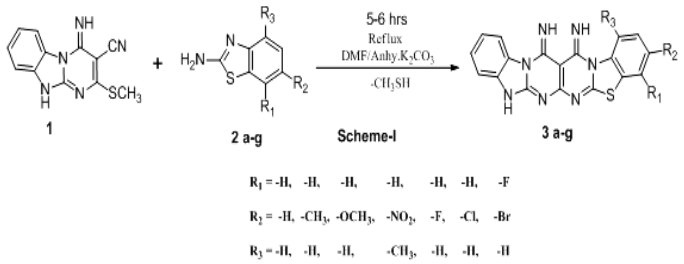
Synthesis of 6,7-Diiminoimidazolo[2,3-*b*]pyrimido[5,6-*e*]pyrimido[2,3-*b*][1,3] benzothiazole and their substituted derivatives (3B.40a-g).

The parent compound (**1**) (0.001 mol) and 2-Amino benzothiazole derivatives such as 2-amino benzothiazole **(2a)**, 2-amino-6-methylbenzothiazole **(2b)**, 2-amino-6-methoxy benzothiazole **(2c)**, 2-amino-4-methyl-6-nitrobenzothiazole **(2d)**, 2-amino-6-fluoro benzothiazole **(2e)**, 2-amino-6-chlorobenzothiazole **(2f)** and 2-amino-6-bromo-4-fluoro benzothiazole **(2g)** (0.001 mol) in 15 ml of DMF and anhydrous K_2CO_3 (10 mg) were refluxed for 5-6 hours. Reaction content cooled at room temperature and mixed with ice cold water then separated solid was filtered, rinse with water and recrystallized using DMF-ethanol mixture to give pure **(3a-g)** respectively.

IV. Result and Discussion

In the present research, we reported one pot synthesis of 6,7-diimino imidazolo[2,3-*b*]pyrimido[5,6-*e*]pyrimido[2,3-*b*][1,3]benzothiazole and their

substituted derivatives **(3a-g).** The reaction started with 3-cyano-4-imino-2-methylthio-4*H*-pyrimido[1,2-*a*]benzimidazole **(2)** were condensed with dissimilar substituted benzothiazoles in presence of refluxing N,N'-dimethyl formamide and anhydrous potassium carbonate to accomplish corresponding target compounds **(3a-g)** shown in scheme-I.



The reaction started with an initial attack of amino group of benzothiazoles on carbon attached to -SCH_3 group resulting in loss of the thiomethyl group in the form of methyl mercaptan. Resulting secondary amine polarizes on to nitrile carbon to give cyclized compounds

The structures of all newly synthesized cyclic compounds **(3Ba-g)** were confirmed on the basis of spectral analysis, IR, ^{1}H NMR and Mass spectral data**.** IR spectra of compounds showed the presence of absorption bands between 3400-3436 cm^{-1} which can be assigned to (=NH stretch). The ^{1}H NMR showed a singlet at δ 3.81 and δ 8.6 which can be assigned to (-NH & =NH) proton. Mass spectra of compounds exhibited the molecular ion peaks which correspond to their molecular weights.

6,7-Diiminoimidazolo[2,3-*b*]pyrimido[5,6 *e*]pyrimido[2,3-*b*][1,3]benzothiazole (3B.40a)
IR:

1577-1662 cm^{-1} (Ar C=C stretch), 3436 cm^{-1} (=NH stretch), **^{1}H NMR** : (500 MHz, DMSO) δ : 3.81 (s, 1H, Ar C-NH), 7.3-8.4 (m, 8H, Ar-H), 8.6 - 9.3 (s, 1H, two =NH) ppm, **Mass:*m/z***=358 (M+1).

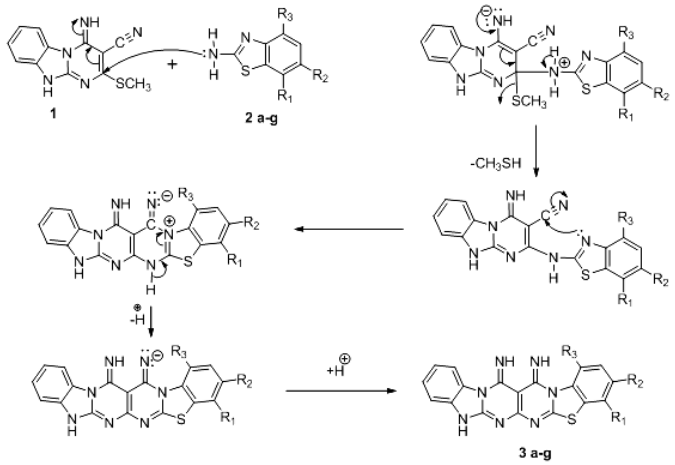
6,7-Diimino–2-methoxy-imidazolo[2,3-*b*] pyrimido [5,6-*e*] pyrimido [2,3-*b*][1,3] benzothiazole (3B.40c)
IR:

1454-1650 cm^{-1} (Ar, stretch), 3460 cm^{-1} (=NH stretch) **^{1}H NMR**: (500 MHz, DMSO) δ: 3.7 (s, 3H, -OCH_3), 3.81(s,1H, NH),7.0 - 8.5 (m, 7H, Ar-H), 8.6, 9.3 (s,1H, two =NH)ppm, **Mass: *m/z***=388 (M+1).

Table 1. Physico-chemical properties of synthesized compounds (3a-g).

Sr. No	Comp.code	Color	M.F.	M. W.	M.P.(^{0} C)	Yield (%)
1.	3a	Yellow	$C_{18}H_{11}N_7S$	357	316-318	80
2.	3b	Brown	$C_{19}H_{13}N_7S$	371	325-327	72
3.	3c	Yellow	$C_{19}H_{13}N_7OS$	387	314-317	70
4.	3d	Yellow	$C_{19}H_{12}N_8O_2S$	416	322-324	65
5.	3e	Yellow	$C_{18}H_{10}FN_7S$	375	312-315	68
6.	3f	Brown	$C_{18}H_{10}ClN_7S$	391	321-323	73
7.	3g	Brown	$C_{18}H_9BrFN_7S$	454	318-320	78

Plausible mechanism of compound (3a-g) (Scheme-I).



V. Conclusion

In the present work we have synthesized 6,7-diimino imidazolo[2,3-*b*]pyrimido[5,6-*e*]pyrimido[2,3-*b*][1,3]benzothiazole and their substituted derivatives **(3a-g)** in good yields. We have used inorganic base potassium carbonate as a catalyst for transformation instead of many other organic bases due to its distinctive features and DMF as refluxing solvent. We have achieved this synthesis in single step rather than multiple steps with minimum reaction time.

VI. Acknowledgement

We thankful to Director CIF, Pune and Vishnu Chemicals Ltd, for providing spectral data.The authors are grateful to principal, Yeshwant Mahavidyalaya, Nanded for providing laboratory facilities.

VII. REFERENCES

[1]. P.Venkatesh and S.N.Pandeya, Int.J. ChemTech Res., 2009, 1(4), 1354-1358.

[2]. F Russo, G Romeo, N Santagati, A Caruso, V Cutuli, D Amore, European Journal of Medicinal Chemistry, 1994, 29, 7-8; 569-578.

[3]. Shivani Choudhary, Suvarna G. Kini and Muhammad Mubeen, Der Pharma Chemica, 2013, 5(4), 213-222.

[4]. Mariappan G, Prabhat P, Sutharson L, Banerjee J, Patangia U, Nath S, Journal of the Korean Chemical Society, 2012, 56(2), 251-256.

[5]. Mariappan G, Prabhat P, Sutharson L, Patangia U, Nath S, Banerjee J, European Journal of Medicinal Chemistry, 2010, 45(7), 2938-2942.

[6]. Priya P. Netalkar, Sandeep P. Netalkar, Srinivasa Budagumpi, Vidyanand K. Revankar, European Journal of Medicinal Chemistry, 2014, 79, 47-56.

[7]. Kochichiro Y, Katsumi G, Kazuya Y, Tominori M, and Goro T, Journal of Medicinal Chemistry, 1990, 33(8), 2192-2196.

[8]. Tashfeen Akhtar, Shahid Hameed, Najim Al-Masoudi, Roberta Loddo, Paolo Colla, Acta Pharmaceutica, 2008, 58(2), 135-149.

[9]. Yaseen A. Al-Soud, Haitham H. Al-Sa'doni, Bahjat Saeed, Ihsan H. Jaber, Mohammad O. Beni-Khalid, Najim A. Al-Masoudi, Tahsin Abdul-Kadir, Paolo La Colla, Bernardetta Busonera, Tiziana Sanna and Roberta Loddo, ARKIVOC, 2008, (xv), 225-238.

[10]. Suvarna Kini, S.P. Swain and A.M. Gandhi, Indian Journal of Pharmaceutical Sciences, 2007, 69(1), 46-50.