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RESEARCH ARTICLE

Facile Synthesis of *(E)*-5-Styrylpyrimidines *via* Wittig Reaction Using Sodium Tripolyphosphate in Water

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Abstract: An inexpensive and eco-friendly Wittig olefination protocol has been developed to prepare novel *(E)*-5-styrylpyrimidines. The reaction of pyrimidine phosphonium ylide with different aryl/heteroaryl aldehydes underwent smoothly in the presence of sodium tripolyphosphate (STPP) in aqueous condition giving *(E)*-5-styrylpyrimidines (**10a-t**) in very high yields (77-96 %).

Keywords: Wittig reaction, (*E*)-5-styrylpyrimidines, green chemistry, water-mediated, sodium tripolyphosphate.

1. INTRODUCTION

Heterocyclic compounds, especially pyrimidine scaffolds, either naturally occurring or synthetic, are well known for their critical roles in medicinal chemistry [1]. For instance, pyrimidine nucleus constitutes many bioactive natural products, such as vitamins (vitamin B1) [2], antibiotics (bacimethrin, sparsomycin, bleomycin) [3], alkaloids (heteromines, crambescins, manzacidins, variolins, meridianins, psammopemmins, *etc*.) [4], and toxins [5]. Owing to their medicinal properties, over the years, diversely substituted pyrimidine derivatives have been synthesized [6] and reported to have a plethora of biological activities, including antiviral [7], anti-HIV [8], anti-microbial [9], anti-tubercular [10], anticancer [11], anti-malarial [12], diuretic [13], cardiovascular [14], analgesic and anti-inflammatory [15]. Further, pyrimidine nucleus bearing styryl motif (compound **A** and **B,** Figure **1**) showed potent *in vitro* as well as *in vivo* antileishmanial activity against L. donovani/hamster model [16]. On the other hand, the drug Rosuvastatin (**C**, Figure **1**) is a member of statin family used to treat high cholesterol and related conditions to prevent cardiovascular diseases [17]. These reports underline the biological significance of styrylpyrimidines and the need of developing novel methodology to access them.

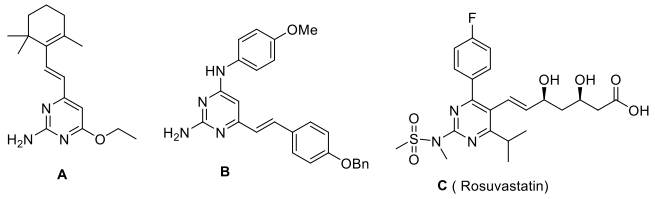

Fig. (1). Bioactive Styrylpyrimidines.

The typical synthetic strategy towards styrylpyrimidines has involved base mediated Wittig olefination between phosphonium salt of an appropriately functionalized pyrimidine and terminal aldehyde [18]. Generally, in the Wittig reaction, stabilized ylides furnish products with high *E*-selectivity of alkenes in solvents ranging from hexane to DMSO [19]. Further, several ecofriendly reaction conditions for Wittig reaction have been reported using microwave irradiation [20], light [21], ionic solvents [22], water [23], and gas hydrate water molecules [24]. Although water has been reported as an effective medium, the application of these strategies is restricted due to poor performance of bases under aqueous conditions and non-compatibility of water insoluble ylides. Hence, the development of a facile protocol for Wittig olefination using heteroaryl phosphonium ylides with a compatible base in water is highly required.

In our continuous efforts towards developing a facile synthetic protocol for bioactive heterocycles [25], herein, we

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describe an efficient method for novel *(E)*-5-styryl-pyrimidines (Figure **2**) *via* Wittig olefination of pyrimidine phosphonium ylide using sodium tripolyphosphate (STPP) in aqueous condition.

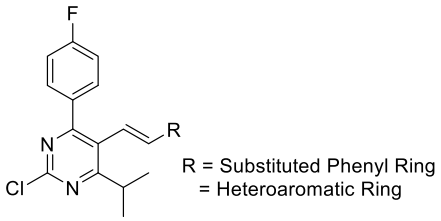


Fig. (2). Targeted novel 5-styrylpyrimidines.

2. RESULT AND DISCUSSION

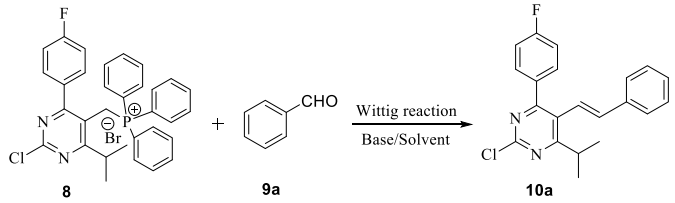
Scheme **1** disclosed the five-step approach for the synthesis of requisite key intermediate pyrimidine phosphonium ylide. The synthesis was initiated with multi-component Biginelli reaction of 4-flurobenzaldehyde (**1**), methyl isobutyrate acetate (**2**) and urea (**3**) in the presence of etidronic acid to obtain methyl 4-(4-fluorophenyl)-6-isopropyl-2-oxo-1,2,3,4-tetrahydropyrimidine-5-carboxylate (**4**, DHPM), as previously described [26]. Further, oxidative dehydrogenation of **4** using 60% Con. HNO_3 yielded 2-hydroxypyrimidine derivative (**5**) [27]. The chlorination of 2-hydroxypyrimidine (**5**) using $POCl_3$ afforded 2-chloropyrimidine derivative (**6**) with a 92% yield [27]. The ester functional group of compound (**6**) was reduced to alcohol by the treatment of diisobutylaluminium hydride (DIBAL) in toluene at 0 °C to give (2-chloro-4-(4-fluorophenyl)-6-isopropyl pyrimidin-5-yl)methanol (**7**) in good yield.

The bromination of compound (**7**) by phosphorous tribromide in DCM at 0 °C followed by treatment with triphenyl phosphine in toluene at reflux temperature afforded ylide (**8**) viz. triphenyl[2-chloro-{4-(4-flourophenyl)-6-isopropyl-pyrimidin-5-lmethyl}phosphonium]bromide in excellent yield (90%).

After establishing facile access of pyrimidine phosphonium ylide (**8**), we set to prepare a library of novel *(E)*-5-styrylpyrimidines by employing Wittig olefination reaction. Initially, we treated compound **8** with benzaldehyde (**9)** using K_2CO_3 in DMSO, as reported earlier [18]. However, even after several replicates, the yield of the desired 5-styrylpyrimidine (**10a**) was unexpectedly low (Table **1**, Entry-1).

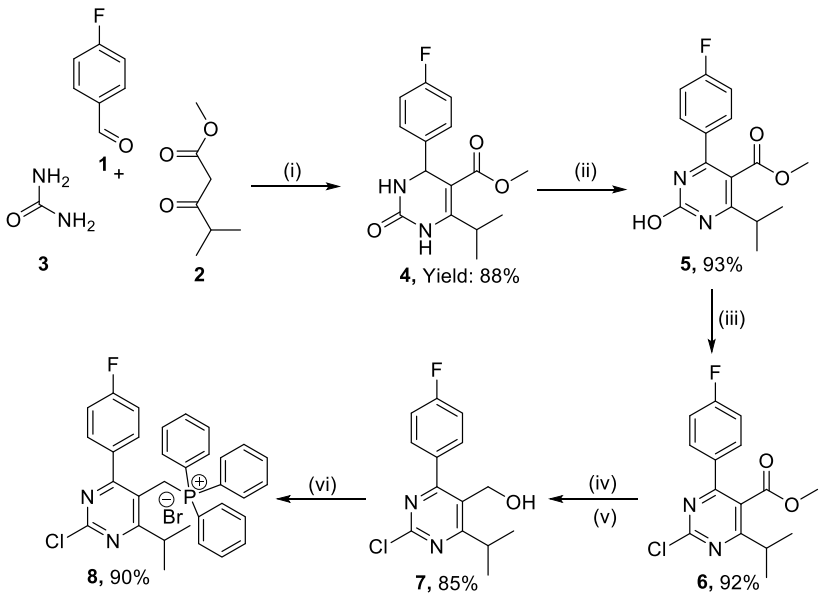
In an effort to improve yield, other bases like Na_2CO_3, $NaHCO_3$, TEA, and DABCO have been tried for the olefination reaction of pyrimidine phosphonium ylide (**8**) with benzaldehyde (**9a**), but results were unsatisfactory, giving low to a moderate yield of **10a** (Table **1**, Entries 2-5).

Table 1. Optimization of reaction condition for the synthesis of 10a using variety of base and solvent.



Entry	Base[a]	Solvent	Time(h)	Yield[b]
1	K_2CO_3	DMSO	24	32
2	Na_2CO_3	DMSO	24	28
3	$NaHCO_3$	DMSO	24	14
4	TEA	DMSO	12	18
5	DABCO	DMSO	12	26
6	NaH_2PO_4	DMSO	24	35
7	Na_2HPO_4	DMSO	12	40
8	$Na_5P_3O_{10}$	DMSO	7	81
9	$Na_5P_3O_{10}$	THF	7	73
10	$Na_5P_3O_{10}$	Dioxane	7	65
11	$Na_5P_3O_{10}$	Water	3	95

[a]***Reaction Conditions****:* Pyrimidine phosphorous ylide (**8**, 1.0 mmol), benzaldehyde (**9**, 1.0 mmol) and base (1.2 mmol) at 70 °C; [b]Isolated Yield.



Scheme 1. Synthesis of key intermediate pyrimidine triphenylphosphonium ylide (**8**).
Reagent and condition: (i) Etidronic acid, EtOH, reflux, 8 h; (ii) Con. HNO_3, 0.5 h; (iii) $POCl_3$, 100 °C, 2 h; (iv) di-isobutylaluminium hydride (DIBAL-H), toluene, 0 °C, 3 h; (v) PBr_3, DCM, 0 °C, 3 h; (vi) PPh_3, toluene, reflux, 4 h.

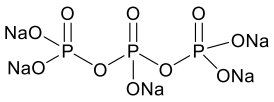


Fig. (3). Sodium Tripolyphosphate (STPP).

In the literature, sodium tripolyphosphate (STPP) (Figure **3**) has been described as a compatible base to prepare α-Bromoisobutyric acid *tert*-butyl ester from *tert*-butyl alcohol and *α*-bromoisobutyryl bromide [28]. Further, sodium tripolyphosphate has several advantages of low cost, stability, high aqueous solubility, and low toxicity. These attributes encouraged us to try out various sodium polyphosphates in our olefination reaction.

When different combinations of NaH_2PO_4, Na_2HPO_4 and $Na_5P_3O_{10}$ with DMSO, THF, and water as solvents were tested for reaction optimization, significant improvement in the yields of **10a** (Table **1**, Entries 6-10) were found. Further, sodium tripolyphosphate ($Na_5P_3O_{10}$, 1.2 eq.) in water turned out to be the best combination for the olefination, providing compound **10a** in excellent yield (95%) within 3 h (Table **1**, entry 11). To investigate the generality of this reaction, we treated a variety of substituted aryl and heteroaryl aldehydes (**10b-t)** with pyrimidine ylide **(8**) using sodium tripolyphosphate in aqueous media. As disclosed in Table **2**, the olefination reaction underwent smoothly furnishing *(E)*-5-styrylpyrimidines (**10b-t**) in very high yields (77 to 96 %), which underline the compatibility of STPP as a base for Wittig reaction.

The protocol was equally effective for aromatic aldehydes bearing electron-withdrawing and donating functionalities (Table **2**, Entry **10b** *vs*. **10n**, **10d** *vs*. **10o**). Moreover, the Wittig olefination worked very well with valuable heteroaromatic aldehydes to furnish (E)-5-styrylpyrimidines **10k-m** in 82-90% isolated yields. It is important to note that the product isolation and purification procedure were extremely simple, wherein the residue obtained after work-up were crystallized from isopropyl alcohol to give analytically pure products (**10a-t**).

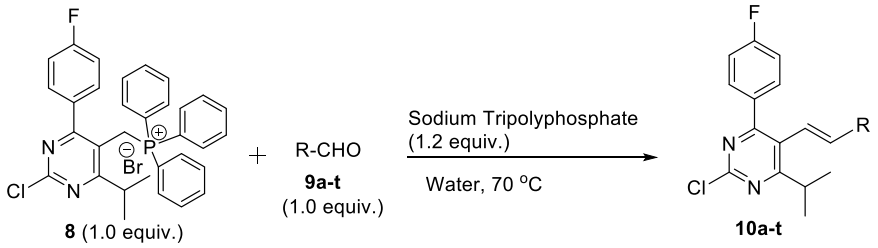
The structures of **10a-t** were established on the basis of their spectroscopic data (IR, MS, ^{1}H NMR, ^{13}C NMR) and elemental analysis. It is noteworthy that all 5-styrylpyrimidines were obtained with *E*-configured vinylic system (J_{trans} ~ 16.0 Hz), as evident by the ^{1}H NMR spectrum. The observed *E* stereochemistry in 5-styrylpyrimidines might be the result of the formation of stabilized ylides, as often observed in the Wittig reactions [19]. Additionally, the exact molecular structure of compound **10d** was unambiguously assured by X-ray analysis, which featured *E*-configuration of 5-styrylpyrimidines (Fig. **4**). Crystallographic data of **10d** have been deposited with the Cambridge Crystallographic Data Center (CCDC 1003862).

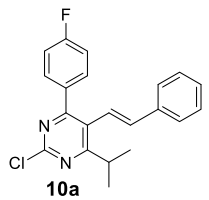
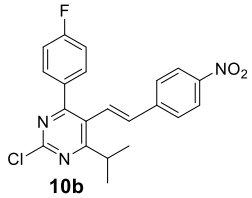
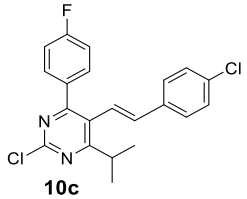
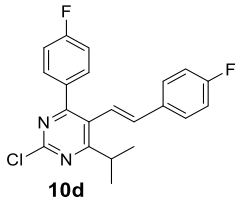
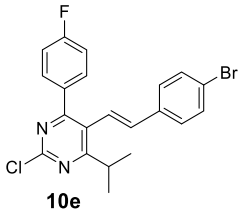
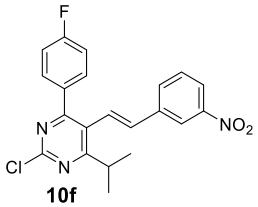
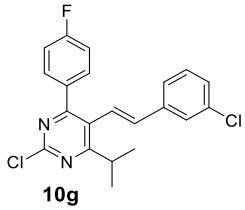
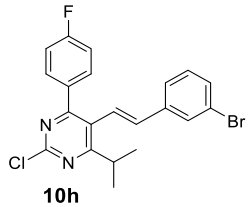
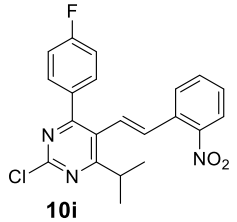
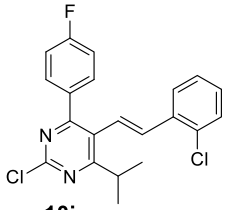
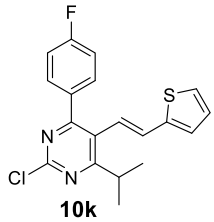
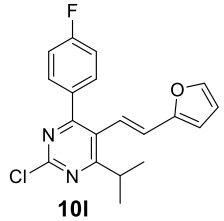
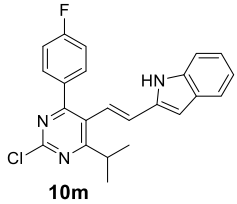
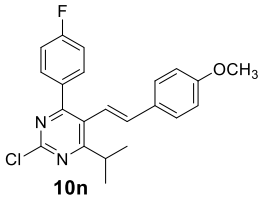
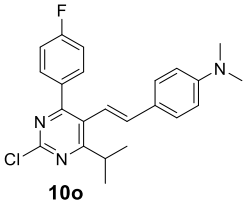
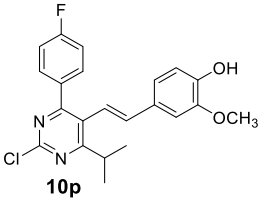
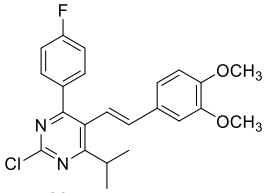
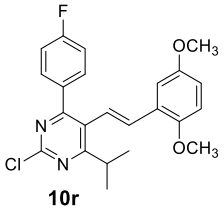
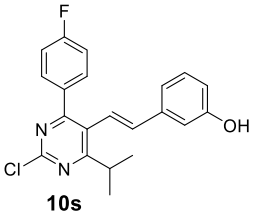
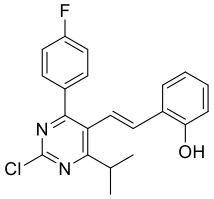
3. EXPERIMENTAL SECTION

3.1. General Method

Chemical reagents and organic solvents were purchased from Sigma-Aldrich & Loba Chemie, and used without further purification. The purity of compounds was checked by Thin-layer chromatography on 0.2-mm precoated plates of silica gel G60 F254 (Merck). Visualization was made with UV light (254 and 365 nm). Infrared spectra were recorded on an FTIR-8400 spectrophotometer using DRS prob. Mass spectra were determined using a direct inlet probe on a GCMS-QP 2010 mass spectrometer (Shimadzu). Nuclear magnetic resonance spectra (^{1}H NMR and ^{13}C NMR) were measured on a Bruker DPX 300 & 400 MHz spectrometers. Chemical shifts (δ) were reported in parts per million (ppm) relative to the TMS peak. Solvents were evaporated with a BUCHI rotary evaporator. Melting points were measured in open capillaries and uncorrected.

Table-2. Synthesis of (*E*)-5-Styrylpyrimidines (10a-t).



 **10a** Reaction time: 3 h, Yield: 95%	 **10b** 2.5 h, 92%	 **10c** 3 h, 84%	 **10d** 3.5 h, 93%
 **10e** 4.5 h, 90%	 **10f** 4 h, 94%	 **10g** 3.5 h, 85%	 **10h** 4 h, 83%
 **10i** 4 h, 79%	 **10j** 3 h, 81%	 **10k** 3.5 h, 89%	 **10l** 4 h, 90%
 **10m** 3.5 h, 82%	 **10n** 3.5 h, 96%	 **10o** 3 h, 82%	 **10p** 3.5 h, 83%
 **10q** 3 h, 95%	 **10r** 3 h, 89%	 **10s** 4 h, 77%	 **10t** 3 h, 88%

3.2. General Synthetic Procedure for 5-(E)-styrylpyrimidine (10a-t)

To a stirred solution of pyrimidine phosphonium bromide ylide (**8**, 1.0 mmol) in water (20 mL) was added aldehyde (**9a-t**, 1.0 mmol) and sodium tripolyphosphate (1.2 mmol). The resulting mixture was heated at 60-70 °C for the time indicated in Table **2**. The progress of the reaction was monitored by thin-layer chromatography. After completion, the

reaction mixture was cooled to room temperature, diluted with water (50 mL), and extracted with DCM (2x50 mL). The combined organic layer was washed with water (2x50 mL), dried over anhydrous sodium sulfate, and evaporated to dryness under *vacuo*. The residue was crystallized from isopropyl alcohol to afford analytically pure products **10a-t.**

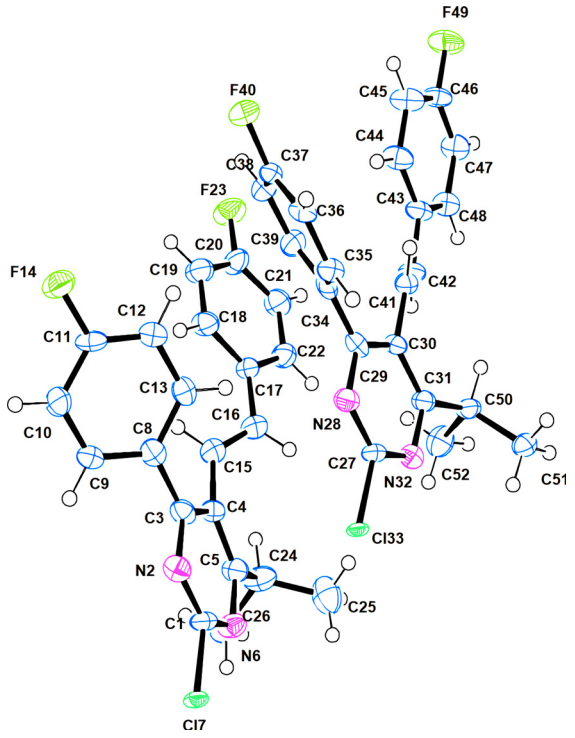


Fig. (4). ORTEP diagram of compound **10d** (showing 50 % probability displacement ellipsoids). (*A higher resolution / colour version of this figure is available in the electronic copy of the article*).

3.2.1. 2-Chloro-4-(4-flurophenyl)-6-isopropyl-(E)-5-styrylpyrimidine (10a)

White solid; R_f 0.75 (8:2 hexane-EtOAc); mp 80-82°C; IR (KBr): 3087, 3070, 3040, 2930, 2856, 1658, 1450, 1430, 1368, 830, 753, 700 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 1.30-1.33 (d, 6H, J = 6.4 Hz, 2 x $^iprCH_3$), 3.03-3.08 (m, 1H, ^iprCH), 6.41-6.47(d, 1H, J =16.4 Hz, ethylene-H), 6.75-6.81 (d, 1H, J =16.4 Hz, ethylene-H), 7.04-7.23 (m, 3H, Ar-H), 7.53-7.56 (d, 2H, J = 8.6 Hz, Ar-H), 7.72-7.74 (t, 2H, J = 8.6 Hz, Ar-H), 8.23-8.26 (d, 2H, J = 8.6 Hz, Ar-H); ^{13}C NMR (DEPT) (75 MHz, $CDCl_3$): 21.6, 32.2, 115.3 (d, J = 21 Hz, CH), 124.3, 126.4, 127.5, 128.5, 132.3 (d, J = 8.2 Hz, CH), 133.0, 135.2; MS (*m/z*): 353 (M^+); Anal. Calcd for $C_{21}H_{18}ClFN_2$: C, 71.49; H, 5.14; N, 7.94; Found: C, 71.45; H, 5.17; N, 7.97.

CONCLUSION

We have developed a greener and expeditious methodology for the synthesis of novel *(E)*-5-styrylpyrimidines *via* Wittig reaction, using sodium tripolyphosphate in aqueous media. The protocol is endowed with several advantages like hefty substrate scope, inexpensive and eco-friendly base, shorter reaction time, easy isolation, and purification of products, would imply that this method may have industrial applications pertaining to Wittig reaction.

CONSENT FOR PUBLICATION

Not applicable.

CONFLICT OF INTEREST

Authors declare that they do not have any conflict of interest.

AVAILABILITY OF DATA AND MATERIALS

Not applicable.

FUNDING

None.

ACKNOWLEDGEMENTS

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SUPPLEMENTARY DATA

Supplementary information (Experimental detail for compound **4** to **8**, Characterization data for **10b-t**, ^1H and ^{13}C spectra, X-ray crystallography data of compounds **10d)** for this article is available in the online version.

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