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RESEARCH ARTICLE

One Pot Multicomponent Synthesis of Novel Thiazolopyrimidines

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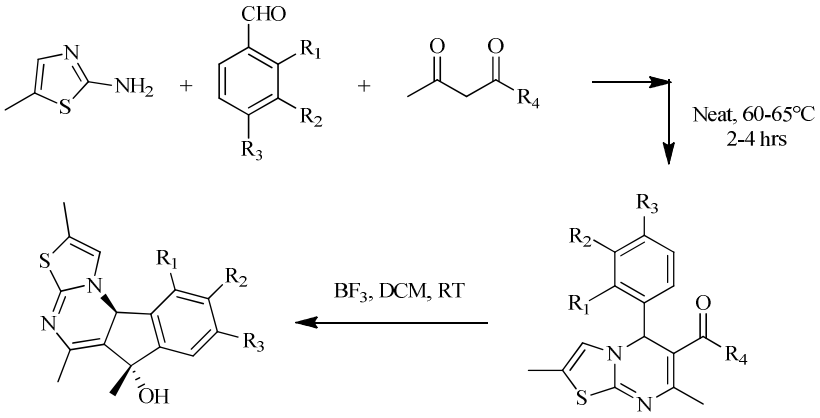
Abstract: A novel, solvent-free, simple, eco-friendly and one-pot multicomponent synthesis of various thiazolopyrimidines is studied. Conventional heating the mixture of amine, aldehyde and *β*-ketoester or 1,3-diketone under solvent free conditions afforded the thiazolopyrimidines compound in good to excellent yields. Further, a boron trifluoride mediated intermolecular cyclization to provide a novel tetra cyclic system is also reported. ^{1}H NMR, ^{13}C NMR, IR and Mass spectroscopy and single crystal analysis were used for identification of these compounds.

Keywords: Thiazolopyrimidines, Multicomponent reaction, Solvent-free method, Tetracyclic system, Eco-friendly reactions

Introduction

Heterocyclic compounds play a significant role in drug discovery as they possess diverse activities such as antifungal, anti-inflammatory, anti-HIV, antimicrobial and anti-psoriatic *etc*[1]. Studies on multicomponent reactions (MCRs) are gaining interest due to the advantages such as mild reaction conditions and shorter reaction steps involved in the synthesis of complex molecules[2-6]. Green chemistry involves atom efficiency, reduced use of solvents, reusable solvents, reagents *etc*.,[7,8]. Jan Svetlik synthesized novel pyrazolopyridine, benzo-pyranopyrazolopyridine involving three-component reaction of 3-hydroxy-benzaldehyde with methyl acetoacetate and 5-amino-3-methyl-1*H*-pyrazole, refluxing the reactants in ethanol for 1.5 hours afforded pyrazolopyridine[9,10]. In this paper we report a novel multicomponent reaction (MCR) strategy to synthesize thiazolopyrimidines of potential medicinal interest in good yields.

In the pursuit to synthesize medicinally active compounds in shorter reaction steps and eco-friendly conditions, we have come across an easy and efficient method for the construction of several substituted/unsubstituted thiazolopyrimidine derivatives. Here we wish to report a simple, one pot multicomponent, efficient and solvent-free synthesis of various thiazolopyrimidines (**1a-1m**) represented in the following scheme.



Compound: 2 **Compound: 1a-1m**

Compound	R_1	R_2	R_3	R_4
1a	H	H	F	OC_2H_5
1b	H	F	H	OC_2H_5
1c	F	H	H	OC_2H_5
1d	H	F	H	OCH_3
1e	H	H	H	OCH_3
1f	H	H	OH	OC_2H_5
1g	H	OCH_3	OH	OCH_3
1h	H	H	H	CH_3
1i	F	H	H	CH_3
1j	H	F	H	CH_3
1k	H	H	F	CH_3
1l	H	H	OCH_3	CH_3
1m	H	OCH_3	OCH_3	CH_3
2	H	H	H	CH_3

Scheme 1. Synthesis of thiazolopyrimidines

The reaction involves the use of 1:1:1 of amine, aldehyde and *β*-ketoester or 1,3-diketone, heating the reaction mixture to about 60-65 °C for 2-4 hours without any solvent followed by a simple work-up procedure. The reaction mixture was cooled to room temperature after confirming the completion of the reaction by TLC, a solvent like ethyl acetate was then added in case of thick reaction mass (if necessary) and the product separated as a solid was collected by filtration, followed by washing with a little quantity of nonpolar solvent like petroleum ether. The solid was dried in vacuum to furnish the product in good yield and found to be pure enough for spectral analysis. In order to reduce the reaction time, we felt that the same reaction carried out under microwave conditions might improve the yield and reduce reaction time. Accordingly, we have carried out the reaction in a microwave oven to obtain compound **1a** but found that the reaction was not clean hence did not proceed further. This observation led us to believe that the method presented in this communication can be considered as a green methodology to obtain novel thiazolopyrimidines.

The products were characterized by the spectral data like ^{1}H NMR, ^{13}C NMR, Mass, IR *etc*. Particularly structure of **1f** is confirmed by single crystal x-ray analysis (Figure 1).

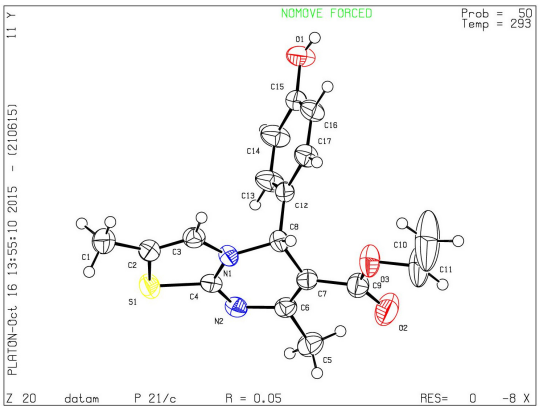


Figure 1. Single crystal x-ray analysis of **1f**

The compound **1h** is converted into a novel tetracyclic compound **2** using boron trifluoride in dichloromethane as solvent at room temperature in a couple of hours. The reaction after a simple work-up and purification on silica gel column chromatography provided the cyclized compound **2** in pure form which was characterized by the spectral data. The above results demonstrate not only a versatile multicomponent reaction strategy to thiazolopyrimidine derivatives but also a chemical transformation of one of the products into a novel tetra cyclic system 2. Further studies are in progress with regard to their biological activity profile and shall be communicated in due course.

Experimental

Reagents purchased from Sigma-Aldrich or Acros and used as such without purification. All solvents were distilled and used. Reactions were carried out in an inert atmosphere. Percentage yields refer to the isolated products after column chromatography if applicable. Silica gel (200 mesh) was used for column chromatography. Precoated silica coated aluminium sheets were used for TLC. Iodine or UV lamp was used to identify the products on TLC plates. NMR was recorded on Bruker 400 MHz, Mass on Apex, IR on Bruker. Either $CDCl_3$ or DMSO-d_6 are used as solvents in NMR. Agilent HPLC instrument was used for HPLC chromatogram involving standard mobile phases like buffer and acetonitrile. Microwave reactions were carried out in a microwave reactor at 150 watt.

General procedure

The amine (10 mmol), aldehyde (10 mmol), *β*-ketoester or 1,3-diketone (10 mmol) were mixed well in a 100 mL round bottom flask with condenser and calcium guard tube and heated to about 60-65 °C. The reaction progress was monitored by TLC with Hexane and ethyl acetate mobile phase (3:7). The reaction mixture was stirred at 60-65 °C until reaction completion. Cool to 25-30 °C, a solvent like ethyl acetate is then added in case of thick reaction mass (if necessary). The product was collected as solid by simple filtration and wash with minimum quantity of petroleum ether. Dried the material at 50 °C and obtained a pale yellow to yellow coloured solid.

*Ethyl 5-(4-fluorophenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (**1a**)*

Yellow coloured solid; Yield 84%; HPLC purity: 95%; mp: 115-116 °C; IR (cm^{-1}): 3376, 3070, 1699, 1616; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33-7.26 (2H, d, J=8 Hz), 7.00-6.96 (2H, d, J=8 Hz), 6.21 (1H, s), 6.06 (1H, s), 4.09-4.00 (2H, m), 2.41 (3H, s), 2.11 (3H, s), 1.17-

1.14 (3H, t, J=8 Hz); ^{13}C NMR (100 MHz, DMSO): δ 165.6, 164.3, 160.6, 155.7, 139.4, 139.3, 128.8, 123.7, 118.5, 115.5, 115.3, 98.4, 59.1, 57.9, 23.2, 14.1, 12.2; MS(EI): *m/z* 333; Elemental analysis: Calculated for $C_{17}H_{17}FN_2O_2S$: C, 61.43; H, 5.16; O, 9.63; N, 8.43; S, 9.65; F, 5.72; found: C, 61.51; H, 5.22; N, 8.51.

*Ethyl 5-(3-fluorophenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (**1b**)*

Pale Yellow coloured solid; Yield 78%; HPLC purity: 97%; mp: 130-134.6 °C; IR (cm^{-1}): 3069, 1696, 1615; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.29-7.24 (1H, t, J=8 Hz), 7.12-7.05 (1H, d, J=8 Hz), 7.05-7.02 (1H, d, J=8 Hz), 6.98-6.93 (1H, m), 6.22 (1H, s), 6.06 (1H, s), 4.10-4.01 (2H, m); 2.41 (3H, s), 2.12 (3H, s), δ 1.18-1.14 (t, 3H, J=8 Hz); ^{13}C NMR (100 MHz, DMSO): δ 165.5, 164.4, 160.9, 156.2, 145.6, 145.6, 130.8, 122.6, 118.3, 115.0, 113.5, 98.0, 58.9, 58.0, 23.3, 14.0, 12.2; MS(EI): *m/z* 333; Elemental analysis: Calculated for $C_{17}H_{17}FN_2O_2S$: C, 61.43; H, 5.16; F, 5.72; N, 8.43; O, 9.63; S, 9.65 found: C, 61.61; H, 5.26; N, 8.30.

*Ethyl 5-(2-fluorophenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (**1c**)*

Yellow coloured solid; Yield 87%; HPLC purity: 98%; mp: 158.7-162.3 °C; IR (cm^{-1}): 3070, 1693, 1614; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44-7.40 (1H, t, J=8 Hz), 7.28-7.22 (1H, d, J=8 Hz), 7.13-7.10 (1H, d, J=8 Hz), 7.05-7.00 (1H, t, J=8 Hz), 6.49 (1H, s), 6.36 (1H, s), 4.04-3.96 (2H, m), 2.48 (3H, s), 2.28-2.11 (3H, s), 1.10-1.07 (t, 3H, J=8 Hz); ^{13}C NMR (100 MHz, DMSO): δ 165.3, 164.4, 159.8, 156.6, 130.4, 129.8, 129.7, 129.5, 124.9, 118.3, 115.5, 96.9, 58.8, 52.8, 23.4, 13.8, 12.2; MS(EI): *m/z*: 333; HRMS: Molecular formula of the compound is $C_{17}H_{17}N_2O_2SFH$ Calculated mass: 333.1073 (M+H); Elemental analysis: calculated for $C_{17}H_{17}FN_2O_2S$: C, 61.43; H, 5.16; N, 8.43; F, 5.72; found: C, 61.62; H, 5.27; N, 8.34.

*Methyl 5-(3-fluorophenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (**1d**)*

Yellow coloured solid; Yield 72.3%; HPLC purity: 96%; mp: 120-123 °C; IR (cm^{-1}): 3079, 1692, 1611; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30-7.24 (1H, t, J=8 Hz), 7.12-7.04 (1H, d, J=8 Hz), 7.02-7.01 (1H, d, J=8 Hz), 6.98-6.93 (1H, m), 6.23 (1H, s), 6.06 (1H, s), 3.61 (3H, s), 2.41 (3H, s), 2.13-2.12 (3H, s); ^{13}C NMR (100 MHz, DMSO): δ 166.0, 164.6, 160.9, 156.6, 145.4, 145.3, 130.9, 122.5, 118.6,115.1, 113.3), 97.6, 57.8, 50.6, 23.3, 12.2; MS(EI): *m/z*: 319; Elemental analysis: calculated for $C_{16}H_{15}FN_2O_2S$: C, 60.36; H, 4.75; N, 8.80; F, 5.97; found: C, 60.52; H, 4.87; N, 8.94.

*Methyl 5-phenyl-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (**1e**)*

Yellow coloured solid; Yield 78.5%; HPLC purity: 99%; mp: 113.2-115.5 °C; IR (cm^{-1}): 3080, 1691, 1611; ^{1}H NMR (400 MHz, $CDCl_3$); δ 7.35-7.26 (5H, m), 6.23 (1H, s), 6.06 (1H, s), 3.59 (3H, s), 2.41 (3H, s), 2.11 (3H, s); ^{13}C NMR (100 MHz, DMSO): δ 166.1, 164.5, 156.3, 142.9, 128.7, 128.1, 126.4, 123.8, 118.0, 98.1, 58.4, 50.4, 23.5, 12.3; MS(EI): *m/z*: 301; Elemental analysis: calculated for $C_{16}H_{16}N_2O_2S$: C, 63.98; H, 5.37; N, 9.33; found: C, 64.12; H, 5.47; N, 9.48.

*Ethyl 5-(4-hydroxyphenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (**1f**)*

Yellow coloured solid; Yield 90.8%; HPLC purity: 98%; mp: 229.0-231.6 °C; IR (cm^{-1}): 3070, 1703, 1607; ^{1}H NMR(400 MHz, DMSO-d_6):δ 9.46 (1H, s), 7.09-7.08 (2H, d, J=8 Hz),

6.84 (1H, s), 6.68-6.66 (2H, d, J=8 Hz), 6.02 (1H, s), 3.95-3.89 (2H, m), 2.26 (3H, s), 2.07 (3H, s), 1.06-1.02 (t, 3H, J=8 Hz); ^{13}C NMR (100 MHz, DMSO): δ 166.7, 164.7, 157.8, 156.4, 134.2, 128.3, 124.3, 118.2, 115.7, 98.8, 58.4, 50.9, 23.9, 14.2, 12.7; MS(EI): *m/z* 331.1; Elemental analysis: calculated for $C_{17}H_{18}N_2O_3S$: C, 60.80; H, 5.49; N, 8.48; found: C, 60.98; H, 5.05; N, 9.04.

*Methyl 5-(4-hydroxy-3-methoxyphenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidine-6-carboxylate (**1g**)*

Yellow coloured solid; Yield 89.5%; HPLC purity: 97%; mp: 225.7-227.6 °C; IR (cm^{-1}): 3060, 1671, 1610; ^{1}H NMR (400 MHz, DMSO-d_6): δ 9.03 (1H, s), 6.92-6.91 (1H, d, J=8 Hz), 6.87 (1H, s), 6.71-6.64 (2H, m), 6.03 (1H, s), 3.71 (3H, s), 3.49-3.32 (3H, s), 2.49-2.48 (3H, s), 2.27-2.08 (3H, s); ^{13}C NMR (400 MHz, DMSO): δ 166.7, 164.7, 156.4, 147.8, 147.0, 134.7,124.3, 119.6, 118.2, 116.0, 111.4, 98.7, 58.7, 56.0, 50.9, 23.9, 12.7; MS(EI): *m/z*: 347; HRMS: The molecular formula of the compound is $C_{17}H_{18}N_2O_4SH$ Calculated mass: 347.1066 (M+H); Elemental analysis: calculated for $C_{17}H_{18}N_2O_4S$: C, 58.94; H, 5.24; N, 8.09; found: C, 59.24; H, 5.19; N, 8.24.

*1-(2,7-Dimethyl-5-phenyl-5H-thiazolo[3,2-a]pyrimidin-6-yl)ethan-1-one (**2a**)*

Yellow coloured solid; Yield 65%; HPLC purity: 97%; mp: 110-112 °C; IR (cm^{-1}): 1700 (C=O); ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.26-7.34 (m, 5H), 6.3 (s, 1H), 6.2 (s, 1H), 2.4 (s, 3H), 2.2 (s, 3H), 2.0 (s, 3H); ^{13}C NMR (DMSO-D6, 100 MHz): 194, 165, 160, 155, 142, 128, 126, 123, 119, 111, 31, 25, 12; MS(EI): *m/z* 284; Elemental analysis: calculated for $C_{16}H_{16}N_2OS$: C, 67.58; H, 5.67; N, 9.85; found: C, 67.78; H, 5.79; N, 9.99.

*1-(5-(2-Fluorophenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidin-6-yl)ethan-1-one (**2b**)*

Yellow coloured solid; Yield: 60%; HPLC purity: 96%; mp: 120-122 °C; IR (cm^{-1}): 1700 (C=O); ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.3 (d, J = 8Hz, 1H), 7.2 (d, J = 8Hz, 1H), 7.0 (d, J = 8Hz, 2H), 6.5 (s, 1H), 6.4 (s, 1H), 2.4 (s, 3H), 2.2 (s, 3H), 2.1 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 194, 165, 160, 157, 130, 129, 125, 122, 119, 115, 108, 77, 30, 25, 12; MS(EI): *m/z* 303.1; Elemental analysis: calculated for $C_{16}H_{15}FN_2OS$: C, 63.56; H, 5.00; F, 6.28; N, 9.26; S, 10.60; found: C, 63.78; H, 5.19; N, 9.39.

*1-(5-(3-Fluorophenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidin-6-yl)ethan-1-one (**2c**)*

Yellow coloured solid; Yield: 67%; HPLC purity: 97%; mp: 120-122 °C. IR (cm^{-1}): 1700 (C=O). ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.3 (d, J = 8 Hz, 1H), 7.2 (t, J = 8 Hz, 1H), 7.0 (d, J = 8 Hz, 2H), 6.5 (s, 1H), 6.4 (s, 1H), 2.4 (s, 3H), 2.2 (s, 3H), 2.1 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): 194, 161, 157, 130, 122, 123, 122, 119, 115, 114, 59, 29, 12; MS(EI): *m/z*: 303.1; Elemental analysis: calculated for $C_{16}H_{15}FN_2OS$: C, 63.56 ; H, 5.00 ; F, 6.28; N, 9.26; found: C, 63.74 ; H, 5.17 ; N, 9.37.

*1-(5-(4-Fluorophenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidin-6-yl)ethan-1-one (**2d**)*

Yellow coloured solid; Yield: 61%; HPLC purity: 95%; mp: 115-117 °C; IR (cm^{-1}): 1700 (C=O); ^{1}HNMR($CDCl_3$, 400 MHz): δ 7.2 (d, J = 8Hz, 2H), 7.0 (d, J = 8Hz, 2H), 6.3 (s, 1H), 6.1 (s, 1H), 2.4 (s, 3H), 2.3 (s, 3H), 2.1 (3H, s); ^{13}C NMR ($CDCl_3$, 100MHz): 194, 165,160, 157, 139,130, 129, 125, 122, 77, 52, 30, 25, 12; MS(EI): *m/z* 303.2; Elemental analysis: calculated for $C_{16}H_{15}FN_2OS$: C, 63.56; H, 5.00; F, 6.28; N, 9.26; found: C, 63.75; H, 5.18; N, 9.34.

*1-(5-(4-Methoxyphenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidin-6-yl)ethan-1-one (**2e**)*

Yellow coloured solid; Yield: 62%; HPLC purity: 96%; mp: 132-134 °C; IR(cm^{-1}): 1700 (C=O); ^{1}HNMR ($CDCl_3$, 400 MHz): δ 7.3 (d, J = 8Hz, 1H), 7.0 (t, J = 8Hz, 1H), 6.5 (s, 1H), 6.4 (s, 1H), 2.4 (s, 3H), 2.2 (s, 3H), 2.1 (s, 3H); ^{13}C NMR ($CDCl_3$, 100MHz): 194, 149, 135, 123, 119, 111, 110, 77, 59, 31, 25, 12, 11; MS(EI): *m/z* 315; Elemental analysis: calculated for $C_{17}H_{18}N_2O_2S$: C, 64.94; H, 5.77; N, 8.91; found: C, 65.18; H, 5.89; N, 9.09.

*1-(5-(3,4-Dimethoxyphenyl)-2,7-dimethyl-5H-thiazolo[3,2-a]pyrimidin-6-yl)ethan-1-one (**2f**)*

Yellow coloured solid; Yield: 60%; HPLC purity: 96%; mp: 144-146 °C; IR (cm^{-1}): 1700 (C=O); ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.2 (s, 1H), 6.8 (d, J = 8Hz, 1H), 6.6 (d, J = 8Hz, 1H), 6.3 (s, 1H), 6.1 (s, 1H), 3.7 (s, 3H), 2.8 (s, 3H), 2.4 (s, 3H), 2.2 (s, 3H), 2.1 (s, 3H); ^{13}C NMR ($CDCl_3$, 100MHz): 194, 165, 155, 149, 135, 123, 119, 111, 110, 77, 59, 31, 25, 12; MS(EI): *m/z*: 344; Elemental analysis: calculated for $C_{18}H_{20}N_2O_3S$: C, 62.77 ; H, 5.85 ; N, 8.13; found: C, 62.98 ; H, 5.79 ; N, 8.29.

*2,5,6-Trimethyl-6,10b-dihydroindeno[2,1-e]thiazolo[3,2-a]pyrimidin-6-ol (**2**)*

To 1 mmole of **1h** in dichloromethane (3 mL), boron trifluoride etherate (3 drops) is added and stirred at room temperature for 2 h. The reaction progress was monitored by TLC, diluted with dichloromethane, washed with water, dried the dichloromethane layer with anhydrous sodium sulfate, removed the solvent, dried to furnish **2** which was used as such for spectral study. Yield: 90%; HPLC: 94%; mp: 102-103 °C; IR (cm^{-1}): OH stretching 3403; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.5 (d, J = 8 Hz, 1H), 7.4 (d, J = 8 Hz, 1H), 7.3 (m, 2H), 6.2 (s, 1H), 5.3 (s, 1H), 2.4 (s, 3H), 2.2 (s, 3H), 1.2 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) : δ 194.7, 141.1, 129.9, 129.6 -126.5, 127.0, 127.0, 123.7, 122.8, 60.4, 31.3, 29.7, 12.7. MS(EI): *m/z*: 284. Elemental analysis: Calculated: C, 67.58; H, 5.67; N, 9.85; obtained: C, 67.56; H, 7.66; N, 9.84.

Conclusion

We have described herein a very simple and easily scalable, multicomponent and eco-friendly method for the synthesis of novel thiazolopyrimidine derivatives of potential medicinal interest. Conventional heating conditions furnished the products in good yields involving simple work-up procedure. Also, an easy synthetic route to a novel tetracyclic system **2** is reported in this work. All the products were characterized by the spectral techniques.

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