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Microwave-assisted multicomponent reaction of aryl amidines: regiospecific synthesis of new polysubstituted thiopyrano-, and pyrano[4,3-*d*]pyrimidines

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ABSTRACT

A series of new functionalized thiopyrano-, pyrano[4,3-*d*]pyrimidine derivatives with benzyl group residing in 8-position of fused pyrimidine nucleus were synthesized through multicomponent reactions of aromatic aldehydes, tetrahydrothiopyran-4-one(tetrahydropyran-4-one), and aryl amidines using *t*-BuOK as a base under microwave heating. The procedure is facile, avoiding time-consuming and costly syntheses, tedious work-up, and purifications of precursors as well as protection/deprotection of functional groups. This method is very efficient due to short reaction times and easy work up and provides a four-component strategy for the construction of the thiopyrano-, and pyrano[4,3-*d*] pyrimidine skeletons.

The search for the efficient formations of multifunctionalized complex products by using simple reactants has been an active objective in organic synthesis.[1] In this regard, multi-component domino reactions that are very useful for the total syntheses of natural products and versatile building blocks have become increasingly attractive because of their green characteristics of atom-economy, bond-forming economy, and structural economy.[2] These reactions can avoid time-consuming and costly processes for the purification of various precursors and tedious steps of protection and deprotection of functional groups. In addition, these reactions are environmentally friendly, and often proceed with excellent chemoselectivities.[3] Therefore; the design of new selective multi-component domino reactions is a continuing challenge at the forefront of organic chemistry.

Pyrimidines compose an important class of heterocycles[4] and their structural framework is a key constituent of numerous natural biologically active compounds.[5] The pyrano-fused pyrimidines as a key pyrimidine family showed a broad range of biological activities, such as antitubercular and antimicrobial agents,[6] antimicrobial agent,[7] and antiplatelet,[8] antifungal agents[9] as well as antiviral activities.[10] In the other hand, the thiopyrano-fused pyrimidines also exhibited antidiabetic[11] and hypoglycemic[12] activities. Because of these biological activities they exhibit, these compounds have distinguished themselves as heterocycles of profound chemical and biological significance. Thus the synthesis of these molecules has attracted considerable attention.[13] In the other hand, amidines and related compounds possessing 1,3-binucleophilic centers arAe versatile synthetic intermediates in organic chemistry.[14] They are frequently applied in the preparation of pyrimidine-containing heterocycles.[14,15] Many pyrimidines have been synthesized by the reactions of amidines, aldehydes, and appropriate ketones via various methods.[15] However, most of these compounds belong to pyrimidines **I**[15a,b] or fused pyrimidines **II**[15c–e] (Fig. 1). To the best of our knowledge, the construction of pyrano- or thiopyrano[4,3-*d*]pyrimidine skeleton (type **III**) with benzyl group regiospecifically residing in 8-position of fused pyran (thiopyran) nucleus has not been reported so far.

Over the past several years, our group has developed various domino reactions that can offer easy access to useful functionalized multiple ring structures of chemical and pharmaceutical interest.[16] For example, a new four-component domino reaction was established as to provide an easy access to the synthesis of multifunctionalized quinazoline[16a] and tricyclo[6.2.2.0^{1,6}]dodecanes derivatives.[16b] Recently, we have also found that the domino reaction of Meldrum's acid, aromatic aldehydes, and electron-rich

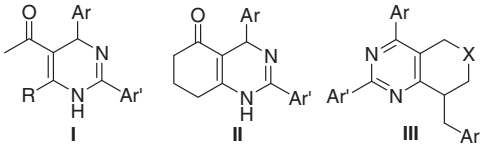

Figure 1. The synthesis of structurally diverse pyrimidines via the reaction of arylamidines, aldehydes, and different ketones.

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heteroaryl-amines in aqueous phase under microwave irradiation (MW) led to the multifunctionalized spiro{[1,3]dioxanes-pyridine}-4,6-dione with high chemo- regio-, and stereoselectivities and good yields.[16c]

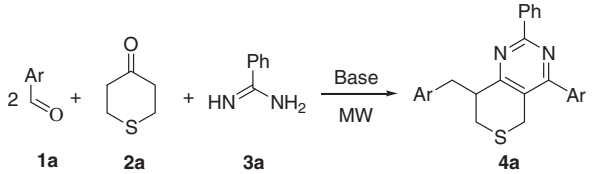
As part of our continuing interest in the development of new domino reaction in heterocyclic compounds,[17,18] in this paper we would like to report a new route to a set of fused pyrimidine derivatives with benzyl group regiospecifically residing in 8-position of fused pyran (thiopyran) nucleus. This reaction was achieved by using readily available aromatic aldehydes **1**, tetrahydrothiopyran-4-one(tetrahydropyran-4-one) **2,** and aryl amidines **3** under microwave heating through one-pot four-component strategy shown in Scheme 1.

To optimize the reaction conditions for the formation of the target compounds, we started this study by treating 4-chlorobenzaldehyde (**1a**) and tetrahydrothiopyran-4-one (**2a**) with benzamidine (**3a**) in a ratio of 2:1:1.1 under microwave irradiation. Various reaction conditions were investigated, including bases, solvents, and temperatures. Initially, we employed various bases to determine one which can give the best results. The results of theses comparative experiments are summarized in Table 1. It was found that with alkaline enhancement of the reaction system, the yield of product **4a** was improved (Table 1, entries 1–5). In this reaction *t*-BuOK demonstrated superior activity and gave the 70% yield of **4a** at 100 °C for 15 min. Subsequently, the reaction was performed in *t*-BuOH and repeated many times at different temperatures in a sealed vessel under microwave heating for 15 min. As shown in Table 1, the yield of product **4a** increased from 70% to 82% as the temperature was raised from 100 to 140 °C (Table 1, entries 5–7). However, the yields leveled off when the temperature was further raised to 150 °C (Table 1, entry 8).

With this result in hand, we went on to study the scope of the methodology. Under the optimized conditions, a variety of structurally diverse aromatic aldehydes were investigated and a series of new thiopyrano[4,3-*d*]pyrimidines with benzyl group regiospecifically residing in 8-position of fused thiopyran nucleus were afforded in good yields. As shown in Table 2, at the beginning, we explored the aldehyde substrate scope, using tetrahydrothiopyran-4-one (**2a**) and benzamidine (**3a**) as model substrates (Table 2, entries 1–5). The results indicated that aromatic aldehydes bearing either electron-donating or -withdrawing functional groups, such as fluoro, chloro, or methoxy, were suitable for the synthesis of compound **4**. Moreover, the heterocyclic aldehyde thiophene-2-carbaldehyde (Table 2, entry 6) still displayed high reactivity under the standard conditions. It is noted that this result is significant because there is no literature precedent for the synthesis of highly functionalized thiopyrano[4,3-*d*]pyrimidines with high regiosele ctivity.

To further expand the scope of the reaction, different aldehydes and tetrahydrothiopyran-4-one (**2a**) were used as substrates with 4-chlorobenzimidamide (**3b**) and 4-bromobenzimidamide (**3c**). In all these cases, the reactions proceeded smoothly to give the corresponding thiopyrano[4,3-*d*]pyrimidines in good yields of 79–87% (Table 2, entries 7–16). Indeed, the protocol provides a straightforward pathway for the construction of highly functionalized thiopyrano[4,3-*d*]pyrimidines.

Table 1
Optimization of bases for the synthesis of **4a** under MW



1a **2a** **3a** **4a**

Ar = 4-Chlorophenyl

Entry	Base	Equiv of base	Solvent	*T* (°C)	Yield [a] (%)
1	NaOH	2.0	Glycol	100	30
2	KOH	2.0	Glycol	100	40
3	MeONa	2.0	MeOH	100	52
4	EtONa	2.0	EtOH	100	58
5	*t*-BuOK	2.0	*t*-BuOH	100	70
6	*t*-BuOK	2.0	*t*-BuOH	120	74
7	*t*-BuOK	2.0	*t*-BuOH	140	82
8	*t*-BuOK	2.0	*t*-BuOH	150	80

[a] Isolated yield.

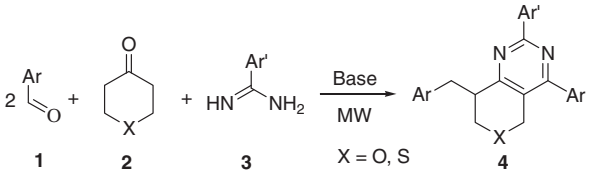
Table 2
Multicomponent synthesis of fused pyrimidines **4**[19]

Entry	4	Ar [a]	3	X	Time [b]	Yield [c] (%)
1	**4a**	4-Chlorophenyl (**1a**)	**3a**	S	15	82
2	**4b**	4-Fluorophenyl (**1b**)	**3a**	S	16	83
3	**4c**	4-Methoxyphenyl (**1c**)	**3a**	S	16	90
4	**4d**	3,4-Dimethoxyphenyl (**1d**)	**3a**	S	14	83
5	**4e**	3,4,5-Trimethoxyphenyl (**1e**)	**3a**	S	13	81
6	**4f**	2-Thienyl (**1f**)	**3a**	S	17	88
7	**4g**	4-Chlorophenyl (**1a**)	**3b**	S	14	83
8	**4h**	4-Fluorophenyl (**1b**)	**3b**	S	15	85
9	**4i**	4-Bromophenyl (**1g**)	**3b**	S	15	82
10	**4j**	4-Chlorophenyl (**1a**)	**3c**	S	14	81
11	**4k**	4-Fluorophenyl (**1b**)	**3c**	S	17	79
12	**4l**	4-Methoxyphenyl (**1c**)	**3c**	S	15	84
13	**4m**	3,4-Dimethoxyphenyl (**1d**)	**3c**	S	16	87
14	**4n**	3,4,5-Trimethoxyphenyl (**1e**)	**3c**	S	14	80
15	**4o**	2-Thienyl (**1f**)	**3c**	S	13	86
16	**4p**	4-Toyl (**1h**)	**3c**	S	16	79
17	**4q**	4-Chlorophenyl (**1a**)	**3a**	O	12	86
18	**4r**	4-Fluorophenyl (**1b**)	**3a**	O	17	89
19	**4s**	Phenyl (**1i**)	**3a**	O	16	86
20	**4t**	4-Chlorophenyl (**1a**)	**3c**	O	17	84
21	**4u**	Phenyl (**1i**)	**3c**	O	15	83

[a] Reagents and conditions: *t*-BuOK (2.0 equiv), 140 °C, *t*-BuOH (2.0 mL), MW.
[b] Min.
[c] Isolated yield.

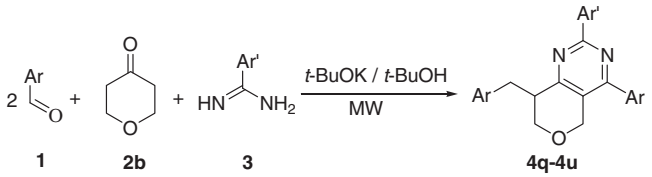
To examine further scope of the methodology, tetrahydropyran-4-one (**2b**) was employed to subject with aldehydes **1** and arylamidine **3** under the optimized conditions described above, the reactions smoothly proceeded to generate the corresponding pyrano[4,3-*d*]pyrimidine derivatives **4q**–**4u** in good to excellent yields (Scheme 2; Table 2, entries 17–21).

The structures of all the products **4** were unambiguously characterized by IR, ^{1}H NMR, and HRMS (ESI). Furthermore, the structure of **4s** was further established by an X-ray crystallographic analysis (Fig. 2).[18]



1 **2** **3** **4**

Scheme 1. The multicomponent synthesis of fused pyrimidines.



1 **2b** **3** **4q-4u**

Scheme 2. The synthesis of pyrano[4,3-*d*]pyrimidines.

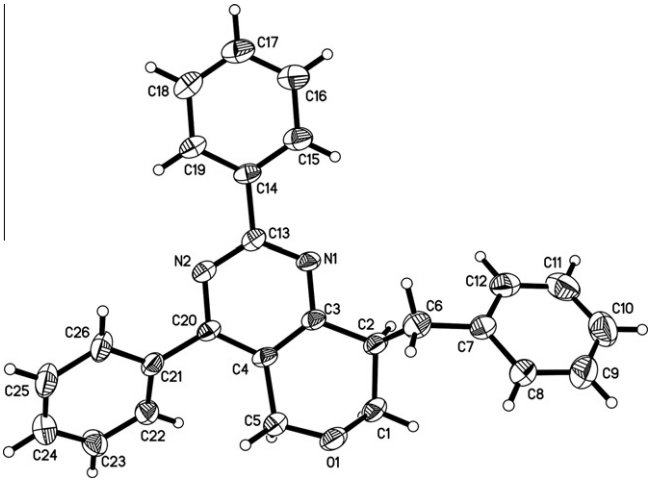
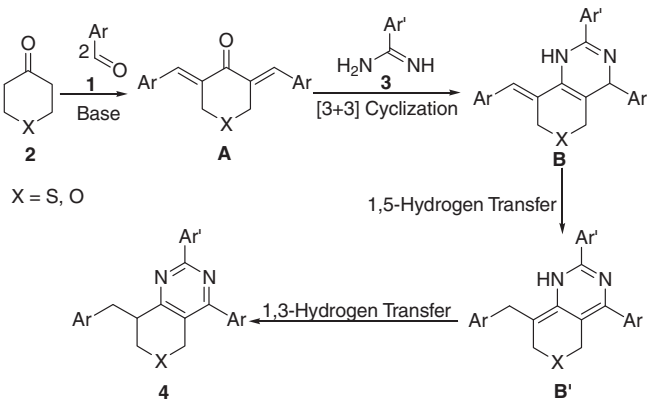


Figure 2. X-ray crystallography structure of compound **4s**.[20]



Scheme 3. Proposed mechanism for the formations of fused pyrimidines **4**.

A reasonable mechanism for the formation of **4** was proposed in Scheme 3. The formation of **4** is expected to proceed via initial condensation of aromatic aldehydes and heterocyclic ketones **2** to afford 2,6-dibenzylidene heterocyclic ketones **A**, and then the [3+3] cycloaddition between **A** and aryl amidine **3** would provide intermediate **B,** which undergoes sequential 1,5-, and 1,3-hydrogen transfer to afford the final products **4**.

Besides a high efficiency in the formation of multiple bonds as a domino process, this reaction has the following advantages: (1) the starting materials are readily available and the reagents are not expensive; (2) the convenient work-up which only needs simple filtration since the products directly precipitate out after the reaction system is neutralized with acid and when its mixtures are diluted with cold water; (3) short reaction times; (4) the regiospecific construction of fused pyrimidine derivatives with benzyl group residing in 8-position of thiopyranopyrimidine nucleus. The novelty of the present multicomponent domino reaction is shown by the fact that multiple chemical bonds breaking and forming were simultaneously achieved in an intermolecular manner and in a one-pot operation.

In summary, new multi-component domino reactions for regiospecific construction of polysubstituted thiopyrano- or pyranopyrimidine skeletons have been established. The reactions showed high regioselectivity and broad substrate scope which further can employ in a wide range of common commercial starting materials.

Acknowledgments

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Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.tetlet.2011.12.128.

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19. General procedure for the synthesis of polysubstituted fused pyrimidines **4** under microwave irradiation: In a 10-mL reaction vial, aromatic aldehyde **1** (2 mmol), tetrahydrothiopyran-4-one or trahydropyran-4-one **2** (1 mmol), *t*-BuOK (2.0 mmol), *t*-BuOH (2.0 mL) were mixed and stirred at room temperature for 10 min. Subsequently aryl amidine **3** (1.1 mmol) was added into the mixture and then capped. The mixture was irradiated for a given time at 140 °C. Upon completion as shown by TLC monitoring, the reaction mixture was cooled to room temperature and neutralized by protonic acids and then diluted with cold water. The solid product was filtered, washed with water and acetone, and subsequently dried and then recrystallized from acetone to give the pure product. **8-Benzyl-2,4-diphenyl-7,8-dihydro-5*H*-pyrano[4,3-*d*]pyrimidine** (**4s**) White solid, mp: 134–136 °C; IR (KBr, ν, cm^{-1}): 3058, 3024, 2841,1541, 1494, 1466, 1452, 1397, 1381, 1365, 1115, 1062, 954, 920, 756, 696; ^{1}H NMR (400 MHz, DMSO-d_6) (δ ppm): 8.46–8.44 (m, 2H, ArH), 7.73–7.71 (m, 2H, ArH), 7.58–7.55 (m, 3H, ArH), 7.54–7.53 (m, 3H, ArH), 7.37–7.32 (m, 4H, ArH), 7.28–7.23 (m, 1H, ArH), 4.96–4.76 (m, 2H, CH_2), 3.92–3.88 (m, 1H, CH_2), 3.82–3.78 (m, 1H, CH_2), 3.47–3.43 (m, 1H, CH_2), 3.31–3.26 (m, 1H, CH_2), 2.92–2.86 (m, 1H, CH); ^{13}C NMR (100 MHz, DMSO-d_6) (δ ppm): 165.9, 162.0, 161.3, 139.5, 137.2, 137.0, 130.6, 129.8, 129.1, 128.7, 128.5, 128.5, 128.4, 127.7, 126.2, 123.8, 67.2, 65.5, 41.4, 37.9, 30.6; HRMS (ESI): *m*/*z* calcd for: $C_{26}H_{23}N_2O$, 379.1805 $[M+H]^+$, found: 379.1822.
20. The single-crystal growth was carried out in DMF at room temperature. X-ray crystallographic analysis was performed with a Siemens SMART CCD and a Siemens P4 diffractometer (graphite monochromator, Mo Kα radiation λ = 0.71073). Crystal data for **4s**: $C_{26}H_{22}N_2O$, crystal dimension 0.43 × 0.21 × 0.13 mm, Triclinic, space group $P\bar{1}$, a = 10.7052(14) Å, b = 13.4963(15) Å, c = 15.4034(16) Å, α = 71.4730(10)°, β = 80.451(2)°, γ = 75.7860(10)°, V = 2036.2(4) Å^3, Mr = 378.46, Z = 4, Dc = 1.235 g/cm^3, λ = 0.71073 Å, μ (Mo Kα) = 0.076 mm^{-1}, $F(000)$ = 800, R_1 = 0.0898, wR_2 = 0.1977.