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Regiospecific one-pot synthesis of pyrimido[4,5-*d*]pyrimidine derivatives in the solid state under microwave irradiations

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Abstract—Electron rich 6-[(dimethylamino)methylene]amino uracil **1**, undergoes [4+2] cycloaddition reactions with various in situ generated glyoxylate imine and imine oxides **6** to provide novel pyrimido[4,5-*d*]pyrimidine derivatives of biological significance, after elimination of dimethylamine from the (1:1) cycloadducts and oxidative aromatisation. This procedure provides a convenient method for the direct synthesis of pyrimido[4,5-*d*]pyrimidines in excellent yields when carried out in the solid state and under microwave irradiations.

The versatility of uracil derivatives for the synthesis of nitrogen-containing heteroaromatic species of biological significance has been well documented.[1] Pyrimidopyrimidines, pyrazolopyrimidines, pyridopyrimidines and xanthine derivatives have all been prepared by functionalization of these important heterocyclic building blocks.[2] Among them, pyrimido[4,5-*d*]pyrimidines and pyrido[2,3-*d*]pyrimidines are an important class of annulated uracils of biological importance[3] because of their connection with purine pteridine systems.[4] Several patents have been reported for the preparation of these fused heterocycles, derivatives of which are useful as bronchodilators,[5] vasodilators,[6] antiallergic,[5,7] antihypertensive[8] and anticancer[5] agents. Most of these preparations rely on cyclocondensation reactions from pyrimidine or pyridine intermediates. However, this type of stepwise synthetic strategy limits the synthetic flexibility. Recently pyrimido[4,5-*d*]pyrimidine analogues of folic acid have been screened for anti-tumour activity.[9] Therefore, with the aim of the preparation of these complex molecules, there has been remarkable interest in the synthetic manipulations of uracils[10], although the synthetic exploitation of the nucleophilic double bond of uracil is an undeveloped field in view of a great variety of potential products.[11] 4-Deazatoxaflavin, a member of the pyrimido[4,5-*c*]pyridazines, inhibits the growth of *Pseudomonus 568* and also binds to herring sperm DNA.[12] Another approach to the synthesis of pyrimido[4,5-*d*]pyrimidines reported by Wamhoff and Muhr[13] is the aza-Wittig type reaction of iminophosphoranes of 5-amino uracils leading to functionalized pyrimido[4,5-*d*]pyrimidines. Our synthetic strategy utilizing three- and two-component reaction of various glyoxylate imines (generated in situ) and diaryl imine oxides with 6-[(dimethylamino)-methylene]aminouracil affords regiospecific one-pot synthesis of pyrimido[4,5-*d*]pyrimidines in excellent yields when carried out in the solid state under microwave irradiations based on [4+2] cycloaddition strategy. In the past a cycloaddition approach has had little appeal since the dienophilic nature of the pyrimidine ring is rather limited, and the diene properties of vinylpyrimidines had not yet been established.[14] It was postulated that if a vinylpyrimidine system were appropriately substituted with strong electron-donating groups, cycloaddition might occur with electron-deficient dienophiles. In a report, the diene character of furan was enhanced by incorporation of a dimethylhydrazino group[15] and 1-(dimethylamino)-3-methyl-2-azabutadiene[16] to function as azadienes suggests that the dienic character of vinylpyrimidines would be increased by similar substituents and which is also supported by HOMO calculations.[17] In continuation to our studies[18] on uracil analogues, we report herein the combination of solid state and microwave technique to synthesize various novel

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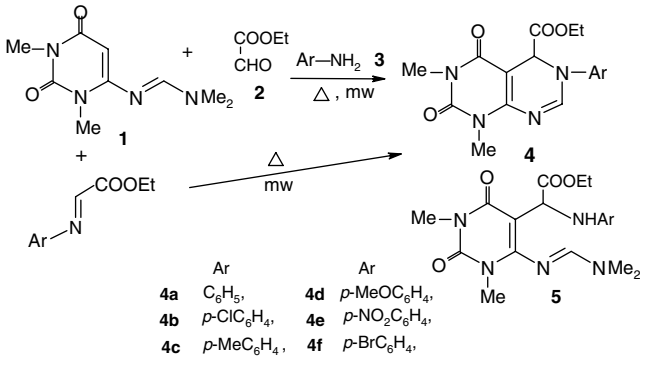
pyrimido[4,5-*d*]pyrimidine derivatives in excellent yields within minutes time. In recent years, the multicomponent one-pot condensation constitutes an attractive synthetic strategy for rapid and efficient library generation due to the fact that products are formed in a single step and the diversity can be achieved simply by varying the reacting components.[19] The reaction can be performed within minutes time and in excellent yields when carried out in a Prolabo Synthwave Microwave Reactor under solvent-free conditions in a three-component one-pot system (Scheme 1).

A mixture of 6-[(dimethylamino)methylene]amino-1,3-dimethyl uracil **1** with an equimolar amount of ethyl glyoxylate **2** and aniline **3** (Ar = C_6H_5) in a reaction vessel is placed in a Prolabo Synthwave Microwave Reactor and irradiated at 110 °C for 3.5 min, which gave, after elimination of dimethylamine from the 1:1 cycloadduct and tautomerism, the pyrimido[4,5-*d*]pyrimidine **4a** as the only product in 95% yield. But the reaction is less effective and takes 5 h to complete when carried out in DMF under reflux and the corresponding pyrimido[4,5-*d*]pyrimidine derivative **4a** was obtained in 63% yield.[20] The structure of product **4a** as a pyrimido[4,5-*d*]pyrimidine derivative was assigned on the basis of its elemental and spectral analysis. The diagnostic signal for the azomethine (formed in situ) proton at δ 8.25 was absent in the cycloadduct, whilst upfield shift of this proton from δ 8.25 to δ 5.45 showed that the cycloaddition had occurred at the C=N bond of the glyoxylate imine. Also, the ^{1}H NMR spectrum showed the absence of the H-5 proton of the uracil **1** and the presence of two methyl groups from the cycloadduct **4a** at δ 3.20 (s, 3H, CH_3) and at 3.62 (s, 3H, NCH_3), and other peaks at 1.20 (t, 3H, OCH_2OCH_3), 4.14 (q, 2H, OCH_2CH_3), 6.85–7.42 (m, 5H, ArH) and 7.75 (s, 1H, CH=N–). The mass spectrum of **4a** revealed a strong molecular ion peak at *m*/*z* 342. Similarly, other pyrimido[4,5-*d*]pyrimidines **4b–f** were prepared in 83–95% yields and their characteristics are recorded in Table 1. Notably, the reaction is also less effective and takes 10–15 min in 60% conversion when glyoxylate imines were reacted directly with **1** in a two-component system in lieu of a three-component system. To enhance the yield, further increase of reaction time did not yield any fruitful results rather decomposition of starting material occurred. This three-component one-pot reaction yields only the pyrimido[4,5-*d*]pyrimidine derivatives **4** and we did not observe the formation of any Michael type of products **5**. Our this finding is in contrast to an earlier report[21] where Sandhu et al. have obtained simple Michael adducts and failed to prepare fused pyrimidines from the reactions of α,β-unsaturated nitro compounds with 6-amino, 6-hydroxylamino and 6-hydrazino-1,3-dimethyluracils. The ^{1}H NMR spectra of **4a** show the absence of the –CH proton in the α-carbon atoms (characteristic peak for Michael adduct) and the presence of a –CH=N– proton at δ 7.75 which rules out the formation of any Michael adduct. The high regiospecificity observed in these reactions is consistent with the electron-donating effect of the dimethylamino substituent increasing the nucleophilicity of the C-5 position. Although, we could not isolate any intermediates from the reaction mixture, a reasonable mechanism for the formation of the product would involve initial electrophilic attack of the in situ generated glyoxylate imine at the C-5 position of the amidine **1** to give the Michael adduct which suffers a subsequent nucleophilic attack on the imino carbon atom eliminating dimethylamine to give product **4**. However, further work is in progress to understand the mechanism in detail.



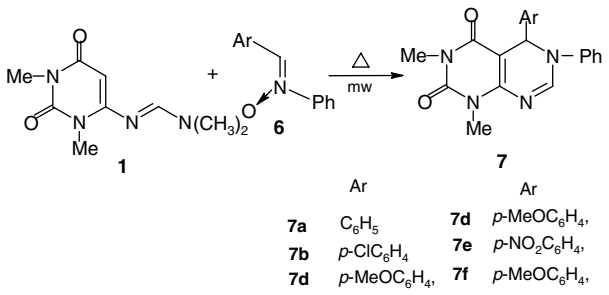
Scheme 1.

Table 1. Physical characteristics of pyrimido[4,5-*d*]pyrimidines **4a–f** and **7a–d**

Product	Ar	Mp (°C)	Reaction time (min)	Yield[a] (%)
4a	C_6H_5	104–06	3.5	95
4b	*p*-ClC_6H_4	130–32	4.0	85
4c	*p*-MeC_6H_4	121–23	3.5	86
4d	*p*-$MeOC_6H_4$	129–31	3.0	95
4e	*p*-$NO_2C_6H_4$	145–46	4.5	84
4f	*p*-BrC_6H_4	140–42	4.0	83
7a	C_6H_5	156–58	3.5	90
7b	*p*-ClC_6H_4	212–14	3.0	88
7c	*p*-MeC_6H_4	240–41	3.5	82
7d	*p*-$MeOC_6H_4$	223–25	3.0	87
7e	*p*-$NO_2C_6H_4$	203–05	3.5	85
7f	*p*-BrC_6H_4	230–32	3.5	80

[a] Yields refer to the isolated pure compounds.

To further investigate the synthetic scope of this cycloaddition reaction, we reacted various diaryl nitrones **6** with amidine **1** under microwave irradiation in the solid state and isolated the corresponding pyrimido[4,5-*d*]pyrimidines **7**, after elimination of 1,3-dimethylamine from the 1:1 cycloadduct and aromatisation, in 80–90% yields, and there was no evidence for the formation of any Michael adducts (Scheme 2). The structure of product **7** as pyrimido[4,5-*d*]pyrimidine was assigned on the basis of its elemental and spectral analysis.[22] The ^{1}H NMR spectrum showed the absence of the H-5 proton of the uracil **1** and two methyl groups from the cycloadduct **7a** at δ 3.32 (s, 3H, NCH_3), 3.55 (s, 3H, NCH_3). The reaction is found to be less effective when carried out in refluxing DMF for 6 h under thermal condition and the corresponding pyrimido[4,5-*d*]pyrimidines were obtained in only 50–60% yields. Further increase of refluxing time did not yield any encouraging results. To generalise this reaction, we reacted various substituted diaryl nitrones **6b–f** with amidine **1** and isolated the corresponding pyrimido[4,5-*d*]pyrimidine derivatives **7b–f** in



Scheme 2.

a one-pot synthesis (Table 1). The reaction is effective with non-conjugated nitrones and when we employed a conjugated nitrone like cinnamaldehyde nitrone or furfural nitrone in lieu of diaryl nitrones and irradiated under MW for 10 min, the reaction did not proceed. Also in an attempt to perform this reaction in a three-component one-pot system, that is, by irradiating a mixture of equimolar quantities of amidine **1**, β-phenylhydroxyl-amine and aromatic aldehyde in the solid state under microwave energy for 10 min it did not proceed effectively and the corresponding pyrimido[4,5-*d*]pyrimidine derivatives **7** were obtained in poor yields. The high regiospecificity observed in these reactions is consistent with the electron-donating effect of the dimethylamine substituent increasing the nucleophilicity of the C-5 position and the established reactivity of the olefins.

In conclusion, our results demonstrate a new, simple and efficient synthesis of novel complex pyrimido[4,5-*d*]pyrimidine derivatives of biological significance in almost excellent yields. These results also illustrate that the title compound **1** is a useful substrate for the generation of an array of fused nitrogen heterocycles. When conventional thermal methods require a considerable reaction time, microwave irradiation can substitute classical methods allowing easy and rapid access to heterocycles of biological significance, reducing the reaction times from hours to minutes with improved yields.

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Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.bmcl.2006.03.088.

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20. Reaction of uracil amidine **1** with ethyl glyoxylate and a primary amine under microwave irradiations and synthesis of novel pyrimido[4,5-*d*]pyrimidine derivatives **4**: equimolar quantities of 6[(dimethylamino)methylene] amino-1,3-dimethyl uracil **1** (0.210 g, 1 mmol), ethyl glyoxylate **2** (0.102 g, 1 mmol) and aniline (0.100 g, 1 mmol) were mixed together in the reaction vassel of the microwave reactor (Synthwave 402 Monomod Reactor from Prolabo) and allowed to react under microwave irradiation at 60% power for 3.5 min. The temperature

was not allowed to increase above 110 °C. The automatic mode stirrer helps in mixing and uniform heating of the reactants. After completion (monitored by TLC), the reaction vessel was cooled to room temperature and the crude product was extracted with ethyl acetate (3× 20 ml) and washed with water. The combined organic phases were dried over anhydrous Na_2SO_4 and subjected to column chromatography to afford the corresponding pyrimido[4,5-*d*]pyrimidine, mp 104–106 °C, in 95% yield. Similarly pyrimido[4,5-*d*]pyrimidines **4b–f** were prepared and characterized as below. Compound **4a**: (whitish solid), IR ν_{max}/KBr/cm^{-1}: 1725, 1690, 1650 (C=O), 1610 (C=N); ^{1}H NMR ($CDCl_3$); δ 7.75 (s, 1H, CH=N–), 6.85–7.42 (m, 5H, ArH), 5.45 (s, 1H, CH-5), 4.15 (q, 2H, OCH_2), 3.62 (s, 3H, NCH_3), 3.20 (s, 3H, NCH_3), 1.20 (t, 3H, OCH_2CH_3); δc 13.72, 27.02, 29.01, 58.55, 66.3, 90.86, 121.62, 124.22, 126.32, 128.51, 128.68, 140.96, 147.88, 150.98, 151.96, 161,21 and 163.4. MS *m*/*z* 342 (M^+). Anal. Calcd for $C_{17}H_{18}N_4O_4$: C, 59.65; H, 5.26; N, 16.37. Found: C, 59.72; H, 5.17; N, 16.45. Compound **4b**: IR ν_{max}/KBr/cm^{-1}: 1720, 1695, 1650 (C=O), 1605 (C=N); ^{1}H NMR ($CDCl_3$) δ 7.62 (s, 1H, CH=N–), 6.98–7.52 (m, 4H, ArH), 5.42 (s, 1H, CH-5), 4.10 (q, 2H, OCH_2), 3.58 (s, 3H, NCH_3), 3.15 (s, 3H, NCH_3), 1.24 (t, 3H, OCH_2CH_3); δc 14.1, 27.1, 28.5, 57.9, 66.4, 90.1, 121.7, 125.4, 126.6, 127.6, 128.0, 140.7, 147.8, 150.9, 151.4, 160.2 and 163.1. MS *m*/*z* 376 (M^+). Anal. Calcd for $C_{17}H_{17}N_4O_4Cl$: C, 54.25; H, 4.52; N, 14.89. Found: C, 54.32; H, 4.44; N, 14.97. Compound **4c**: IR ν_{max}/KBr/cm^{-1}: 1720, 1690, 1660 (C=O), 1610 (C=N); ^{1}H NMR ($CDCl_3$) δ 7.68 (s, 1H, CH=N–), 6.80–7.32 (m, 4H, ArH), 5.36 (s, 1H, CH-5), 4.08 (q, 2H, OCH_2), 3.42 (s, 3H, NCH_3), 3.15 (s, 3H, NCH_3), 2.22 (s, 3H, CH_3), 1.15 (t, 3H, OCH_2CH_3), MS *m*/*z* 356 (M^+). Anal. Calcd for $C_{18}H_{20}N_4O_4$: C, 60.67; H, 5.61; N, 15.73. Found: C, 60.77; H, 5.53; N, 15.80. Compound **4d**: IR ν_{max}/KBr/cm^{-1}: 1720, 1695, 1655 (C=O), 1615 (C=N); ^{1}H NMR ($CDCl_3$) δ 7.72 (s, 1H, CH=N–), 6.90–7.42 (m, 4H, ArH), 5.42 (s, 1H, CH-5), 4.15 (q, 2H, OCH_2), 3.88 (s, 3H, OCH_3), 3.55 (s, 3H, NCH_3), 3.20 (s, 3H, NCH_3), 1.22 (t, 3H, OCH_2CH_3), MS *m*/*z* 372 (M^+). Anal. Calcd for $C_{18}H_{20}N_4O_5$: C, 58.06; H, 5.37; N, 15.05. Found: C, 58.12; H, 5.28; N, 15.11.

Compound **4e**: IR ν_{max}/KBr/cm^{-1}: 1725, 1690, 1655 (C=O), 1605(C=N); ^{1}H NMR ($CDCl_3$) δ 7.72 (s, 1H, CH=N–), 6.88–7.45 (m, 4H, ArH), 5.44 (s, 1H, CH-5), 4.20 (q, 2H, OCH_2), 3.66 (s, 3H, NCH_3), 3.25 (s, 3H, NCH_3), 1.15 (t, 3H, OCH_2CH_3). MS *m*/*z* 387 (M^+). Anal. Calcd for $C_{17}H_{17}N_5O_6$: C, 52.71; H, 4.39; N, 18.07. Found: C, 52.65; H, 4.33; N, 18.15.

Compound **4f**: IR ν_{max}/KBr/cm^{-1}: 1720, 1690, 1655 (C=O), 1610 (C=N); ^{1}H NMR ($CDCl_3$) δ 7.70 (s, 1H, CH=N–), 6.88–7.50 (m, 4H, ArH), 5.35 (s, 1H, CH-5), 4.15 (q, 2H, OCH_2), 3.60 (s, 3H, NCH_3), 3.25 (s, 3H, NCH_3), 1.18 (t, 3H, OCH_2CH_3). MS *m*/*z* 421 (M^+). Anal. Calcd for $C_{17}H_{17}N_4O_4Br$: C, 48.45; H, 4.04; N, 13.30. Found: C, 48.38; H, 4.10; N, 13.25.

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22. Reaction of uracil amidine **1** with aldonitrones **6** and synthesis of pyrimido[4,5-*d*]pyrimidine derivatives 7: equimolar quantities of 6[(dimethylamino)methylene]amino-1,3-dimethyl uracil (0.210 g, 1 mmol) and diphenyl nitrone **6a** (0.198 g, 1 mmol) were added in the reaction vassel of the microwave reactor (Synthwave 402 Monomode Reactor from Prolabo) and allowed to react under microwave irradiation at 80% power for 3 min. The automatic mode stirrer helps in mixing and uniform heating of the reactants. The reaction vessel was cooled to room temperature and the solid compound obtained was recrystallized from ethyl acetate:petroleum ether (40–60) (1:4) to give pyrimido[4,5-*d*]pyrimidine **7a** in 90% yield, Mp 156–158 °C, IR ν_{max}/KBr/cm^{-1}: 1690, 1650 (C=O); ^{1}H NMR ($CDCl_3$): δ_H 8.10 (s, 1H, CH=N–), 7.05–7.88 (m, 4H, ArH), 6.05 (s, 1H, H-5), 3.55 (s, 3H, NCH_3), 3.32 (s, 3H, NCH_3). δc 160.1 (C-2), 151.4 (C-4), 151.1 (C-8a), 121.7, 125.4, 126.6, 127.6, 128.0, 140.7, 140.8, 148.1, (C-7 and C-Ph), 90.1 (C-4a), 57.9 (C-5), 28.5 (C-9), 27.2 (C-10). MS *m*/*z* 346 (M^+). Anal. Calcd for $C_{16}H_{11}N_3O_4$: C, 62.13; H, 3.55; N, 13.59. Found: C, 62.01; H, 3.62; N, 13.44. Similarly pyrimido[4,5-*d*]pyrimidines **7b–f** were prepared and characterized.