

5-Aminouracil as a Building Block in Heterocyclic Synthesis: Part III. One-pot Synthesis of Novel Pyrimido[5,4-*b*]quinoline-2,4,9-triones and Pyrimido[5,4-*c*]isoquinolines

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An efficient and direct procedure for the synthesis of pyrimido[5,4-*b*]quinoline-2,4,9-trione and pyrimido[5,4-*c*]isoquinoline derivatives has been described. The products were characterized by elemental analyses, IR, ¹H NMR, ¹³C NMR and MS spectra.

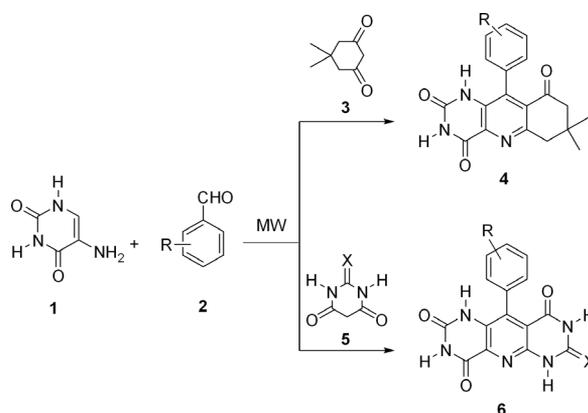
Key words: 5-Aminouracil, Dimedone, One-pot Synthesis, Pyrimido[5,4-*b*]quinoline-2,4,9-trione, Pyrimido[5,4-*c*]isoquinoline

Introduction

Polyfunctionalized heterocyclic compounds play important roles in the drug discovery process, and analysis of drugs in late development or on the market shows that 68 % of them are heterocycles [1]. Therefore, it is not surprising that research on the synthesis of polyfunctionalized heterocyclic compounds has received significant attention. Pyrimidoquinolines have been an object of great interest to organic, medicinal and materials scientists over many years, as they are present in a number of biologically active organic compounds which exhibit antimalarial [2, 3], anticancer [4], antitumor [5] antimicrobial [6], antiviral [7], analgesic [8], anti-oxidant [8], and anti-inflammatory activities [8, 9]. Furthermore, multi-component reactions (MCRs) play an increasingly important role in organic and medicinal chemistry for their convergence, productivity, ease of execution, excellent yield, and broad application in combinational chemistry [10–18].

Recently, we reported a simple and efficient synthesis of pyrimido[5,4-*b*]quinoline-2,4,9-triones (**4**) [17], and pyrido[3,2-*d*:6,5-*d'*]dipyrimidines (**6**) [18] via the reaction of 5-aminouracil (**1**), benzaldehyde derivatives **2** and dimedone (**3**) or barbituric acid derivatives **5** under microwave irradiation without catalyst. These compounds could have interesting effects on biological targets (Scheme 1).

Considering the above reports and in continuation of our work in the development of new and simple

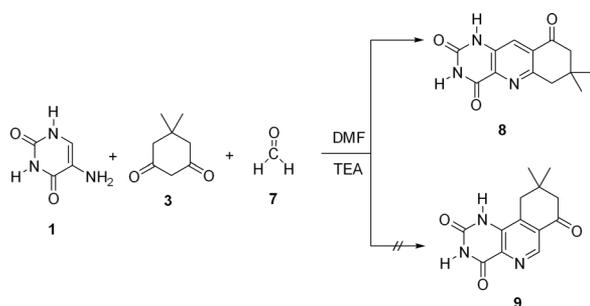


Scheme 1. Synthesis of pyrimido[5,4-*b*]quinolines **4** and pyrido[3,2-*d*:6,5-*d'*]dipyrimidines **6**.

methods for the synthesis of polyfunctionally substituted heterocyclic compounds [17–28], we wish to report a novel and efficient one-pot method for the synthesis of pyrimido[5,4-*b*]quinoline-2,4,9-trione and pyrimido[5,4-*c*]isoquinoline derivatives with the purpose of investigating in the future their possible biological activity. The mechanism of the reaction has been proved *via* the synthesis of a proposed intermediate.

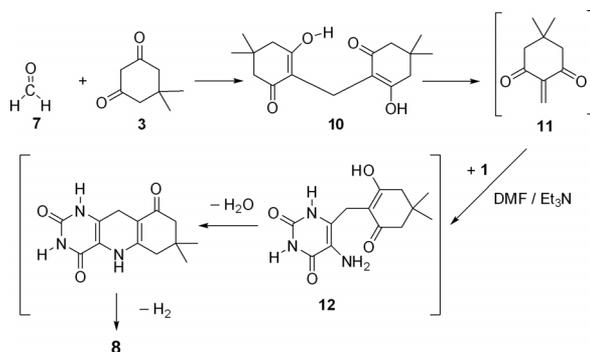
Results and Discussion

The reaction of 5-aminouracil (**1**), dimedone (**3**) and paraformaldehyde (**7**) in DMF and in the pres-

Scheme 2. Synthesis of **8**.

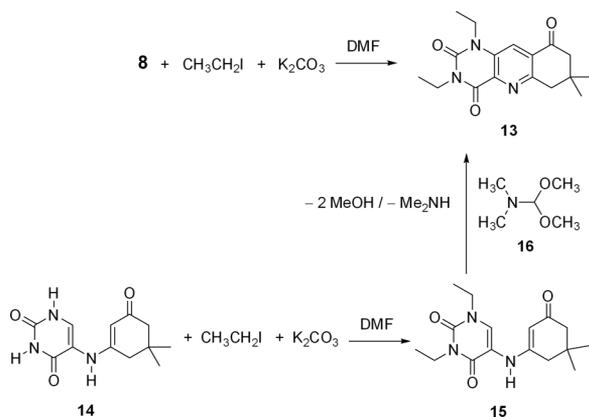
ence of triethylamine as a catalyst gave a solid product of the composition $C_{13}H_{13}N_3O_3$ ($m/z = 259$ (30%), $[M]^+$) which may be formulated as the pyrimido[5,4-*b*]quinoline-2,4,9-trione **8** or its isomer **9** (Scheme 2). The molecular structure of **8** was indicated by its 1H NMR spectrum which revealed three characteristic, relatively sharp singlets at 11.56, 11.25 and 7.93 ppm. The two former ones are assigned to the two NH groups, 3-NH and 1-NH, respectively, and the latter to H-10. Two singlets appear at 3.00 and 2.62 ppm corresponding to the two CH_2 groups at positions 6 and 8, respectively, in addition to the singlet for the two methyl groups. Moreover, the ^{13}C NMR spectrum of **8** showed signals at $\delta_C = 28.41$ (CME_2), 33.02 (C-7), 41.88 (C-6), 50.37 (C-8), 129.01 (C-10), 133.73 (C-9a), 139.98 (C-4a), 144.62 (C-10a), 151.31 (C-6a), 162.08 (C-2), 163.31 (C-4), and 197.09 (C-9). With these spectroscopic data the proposed linear structure of **8** is identified. In the angular structure **9** the pyridine proton signal should have appeared at a higher field [29].

A proposed reaction mechanism that accounts for the multicomponent reaction is shown in Scheme 3. Thus, the reaction may occur *via* a condensation, addition, cyclization, and elimination mechanism. We

Scheme 3. Proposed mechanism to pyrimido[5,4-*b*]quinoline-2,4,9-trione **8**.

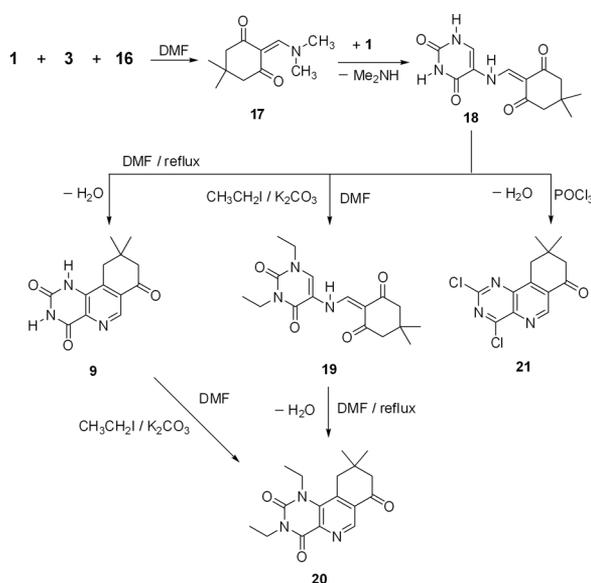
assume the initial formation of the 2:1 dimedone/formaldehyde adduct **10**, which gives the Knoevenagel adduct intermediate **11**. A subsequent Michael-type addition reaction of the nucleophile C-6 in 5-aminouracil (**1**) leads to the formation of intermediate **12**, which undergoes cyclization with loss of a water molecule and partial oxidation to render compound **8**. Neither adduct **10** nor **11** were isolated in the reaction under study. To confirm the assumed three-component condensation route we synthesized 2,2'-methylene-bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**10**) [30] and brought it into reaction with compound **1** under the previous conditions. The target product formed in approximately the same yield and was identical in all aspects to compound **8** (Scheme 3).

We also studied the alkylation of **8** with ethyl iodide. The reaction was carried out at r.t. in DMF and in the presence of anhydrous potassium carbonate to afford the ethylated derivative **13** (Scheme 4). The structure of the product **13** was proved by elemental analysis and spectral data. This compound is not the *O*-ethylation product. Its IR spectrum contains bands around $1662-1708\text{ cm}^{-1}$, characteristic of carbonyl absorptions, and the 1H NMR spectrum revealed that the alkylation occurs at the N-1 and N-3 atoms. Thus, the 1H NMR spectrum of compound **13** contained signals from the N^1CH_2 ($\delta = 3.81$) and N^3CH_2 protons ($\delta = 3.88$ ppm). Furthermore, the structure assigned to **13** was fully supported by its mass spectrum, which showed a molecular formula $C_{17}H_{21}N_3O_3$ ($m/z = 315$ (35%), $[M]^+$). Further confirmation of structure **13** was achieved *via* the synthesis of the ethylated enaminoketone **15**, prepared by ethylation of the enaminoketone **14** with ethyl iodide in DMF and in the

Scheme 4. Ethylation of **8** and **14**.

presence of anhydrous potassium carbonate. The IR, MS, ^1H NMR as well as the ^{13}C NMR spectra agreed with the proposed structure **15**. Gentle heating of a dimethylformamide solution of **15** and dimethylformamide dimethyl acetal (**16**) at 125–135 °C for 18 h yielded a product identical in all aspects to compound **13** (Scheme 4).

In addition, the structure of **8** was confirmed further by an alternative synthesis of its isomer **9** (Scheme 5). Thus, reacting a mixture of 5-aminouracil (**1**), dime-done (**3**) and DMFDMA (**16**) in DMF without catalyst under reflux for 8 h afforded the pyrimidine-2,4(1*H*,3*H*)-dione **18** (Scheme 5). The structure of compound **18** was confirmed by its elemental and spectral analyses, which showed the molecular ion peak at $m/z = 277.14$ (98%). Its ^1H NMR spectrum showed characteristic singlets at $\delta = 11.09$ and 11.57 ppm for two NH groups, a doublet at $\delta = 12.29$ ($J = 15$ Hz) for an exocyclic NH group, a doublet at $\delta = 8.36$ ($J = 15$ Hz) due to N-CH=, a singlet at $\delta = 7.99$ due to CH-uracil, in addition to three singlet signals for the methyl and dime-done protons. Furthermore, the structure of compound **18** was confirmed by an independent synthesis of the same compound from an equimolar amount of **1** and 2-dimethylaminomethylidencyclohexane-1,3-dione (**17**) in DMF under reflux to afford a product identical in all aspects to compound **18** (Scheme 5).



Scheme 5. Synthesis of pyrimido[5,4-*c*]isoquinolines **9**, **20** and **21**.

The formation of **18** can be described in terms of the initial formation of the intermediate **17**. A reaction of the latter with **1**, which is accompanied by elimination of the readily leaving dimethylamino group, gives rise to **18** (Scheme 5). Heating a dimethylformamide solution of compound **18** affords the pyrimido[5,4-*c*]isoquinoline **9**, the ^1H NMR spectrum of which revealed the absence of an exocyclic NH proton and the presence of a singlet at $\delta = 8.45$ ppm due to a pyridine-CH proton. The structure of **9** was confirmed by its elemental and spectral analyses, which showed a molecular formula $\text{C}_{13}\text{H}_{13}\text{N}_3\text{O}_3$ ($m/z = 259$ (83.3%), $[\text{M}]^+$). Comparison of the data of **9** with those of **8** showed differences in melting point, IR, and ^1H NMR data which confirmed the structure of **9** for our product.

Moreover, the ethylated derivative **20** was obtained by reacting **9** with ethyl iodide in DMF and in the presence of anhydrous potassium carbonate (Scheme 5). The structure of **20** was confirmed by its elemental and spectral analyses, which showed the molecular ion peak at $m/z = 315$ (15%). Also, the ^1H NMR spectrum of **20** displayed no NH protons, but the presence of signals from the N^1CH_2 ($\delta = 3.86$) and N^3CH_2 protons ($\delta = 4.10$ ppm). Further confirmation of structure **20** was obtained *via* the synthesis of compound **19**, prepared by ethylation of compound **18**. The structure of **19** was established by its correct elemental analysis and compatible spectroscopic data. Subsequent heating of **19** in refluxing DMF smoothly converted it to the final product **20** (Scheme 5). The identity of the products of the cyclization of **19** with those obtained by the ethylation of **9** was confirmed by comparison of their IR and ^1H NMR spectra.

Finally, cyclization and chlorination of compound **18** using phosphoryl chloride gave the dichloro derivative **21** (Scheme 5). The structure of the latter product was confirmed on the basis of correct elemental analysis and spectral data. Thus, the IR and ^1H NMR spectra of **21** revealed the absence of NH groups and signals attributable to the CH-uracil and NH protons of **18**. Also, its MS gave the characteristic fragmentation pattern due to the presence of two chlorine atoms and showed the molecular ion peak at $m/z = 296$ (19.80%) in agreement with its molecular formula $\text{C}_{13}\text{H}_{11}\text{Cl}_2\text{N}_3\text{O}$.

In conclusion, the reported three-component one-step procedure is a simple, practical and very regioselective method for the synthesis of novel pyrimido[5,4-*b*]quinoline-2,4,9-trione and pyrimido[5,4-*c*]isoquinoline derivatives.

Experimental Section

General procedures

Melting points were measured with a Gallenkamp apparatus and are uncorrected. The reactions and purity were monitored by thin layer chromatography (TLC) on aluminum plates coated with silica gel with fluorescence indicator (Merck, 60 F₂₅₄) using CHCl₃-CH₃OH (10 : 1) as eluent. Infrared spectra were recorded in potassium bromide disks on a Jasco FT/IR-450 Plus infrared spectrophotometer. NMR spectra were obtained on a JHA-LAA 400 WB-FT spectrometer (300 MHz for ¹H NMR, 75 MHz for ¹³C NMR), with deuterated chloroform (CDCl₃) or dimethylsulfoxide ([D₆]DMSO) as solvent. Chemical shifts are quoted in δ and are referenced to TMS or the solvent signal. Mass spectra were recorded on a Trace GC 2000/Finngan Mat SSQ 7000 and a Shimadzu GCMS-QP-1000EX mass spectrometer at 70 eV. Elemental analyses were measured with a Vario EL III CHNOS Elemental Analyzer in the Microanalytical Center of Cairo University. Compounds **10** [30], **14** [17] and **17** [31] were synthesized using the published procedures.

7,7-Dimethyl-7,8-dihydropyrimido[5,4-*b*]quinoline-2,4,9(1*H*,3*H*,6*H*)-trione (**8**)

Method A: A mixture of 5-aminouracil (**1**) (0.13 g, 1 mmol), dimedone (**3**) (0.28 g, 2 mmol) and paraformaldehyde **7** (0.64 g, 2 mmol) along with triethylamine (0.05 g, 0.5 mmol) in DMF (10 mL) was refluxed for 20 h (TLC control using a solvent system of chloroform-methanol (5 : 2)). The solvent was evaporated under vacuum; the resulting solid was then collected and crystallized from dioxane; yield: 81 %.

Method B: A solution of equimolar amounts of 2,2'-methylenebis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**10**) (0.29 g, 1 mmol), **1** (0.13 g, 1 mmol) and triethylamine (0.05 g, 0.5 mmol) in DMF (10 mL) was refluxed for 25 h. Product **8** was isolated as described above; yield: 83 %. Brown powder, m. p. 288–290 °C. – IR (film): $\nu = 3440, 2923, 1674 \text{ cm}^{-1}$. – ¹H NMR (300 MHz, [D₆]DMSO): $\delta = 1.03$ (s, 6H, 2CH₃), 2.62 (s, 2H, CH₂), 3.00 (s, 2H, CH₂), 7.93 (s, 1H, 6-H), 11.25 (s, 1H, NH), 11.56 (s, 1H, NH). – ¹³C NMR (75 MHz, [D₆]DMSO): $\delta = 28.41, 33.02, 41.88, 50.37, 129.01, 133.73, 139.98, 144.62, 151.31, 162.08, 163.31, 197.09$. – MS (EI, 70 eV): m/z (%) = 260 (16.7) [M+1]⁺, 259 (30) [M]⁺. – Anal. for C₁₃H₁₃N₃O₃ (259.26): calcd. C 60.22, H 5.05, N 16.21; found C 60.39, H 5.17, N 16.34.

Ethylation of **8** and **14**

Ethyl iodide (2.81 g, 18 mmol) was added to a mixture of **8** and/or **14** (3 mmol) and anhydrous potassium carbonate (0.83 g, 6 mmol) in DMF (20 mL). The reaction mixture was

stirred for 48–55 h at r. t. and then poured into cold water and extracted with chloroform (3 × 20 mL). The combined organic extracts were washed with water and dried (anhydrous magnesium sulfate). After evaporation of some of the solvent (~ 10 mL), petroleum ether (40 mL) was added, and the resulting precipitate was collected, dried and recrystallized from petroleum ether-chloroform and/or *n*-hexane-benzene to give **13** (yield 40 %), and/or **15** (yield 70 %), respectively.

1,3-Diethyl-7,7-dimethyl-7,8-dihydropyrimido[5,4-*b*]quinoline-2,4,9(1*H*,3*H*,6*H*)-trione (**13**)

Dark-brown powder, m. p. 209–212 °C. – IR (film): $\nu = 3521, 2924, 2877, 1708, 1662.5 \text{ cm}^{-1}$. – ¹H NMR (300 MHz, [D₆]DMSO): $\delta = 0.99$ (s, 6H, 2CH₃), 1.12 (t, 3H, *J* = 6 Hz, CH₃), 1.23 (t, 3H, *J* = 6 Hz, CH₃), 2.32 (s, 2H, CH₂), 2.40 (s, 2H, CH₂), 3.81 (q, 2H, *J* = 6 Hz, CH₂), 3.88 (q, 2H, *J* = 6 Hz, CH₂), 8.41 (s, 1H, 10-H). – MS (EI, 70 eV): m/z (%) = 316 (40) [M+1]⁺, 315 (35) [M]⁺. – Anal. for C₁₇H₂₁N₃O₃ (315.37): calcd. C 64.74, H 6.71, N 13.32; found C 64.89, H 6.59, N 13.39.

5-(5,5-Dimethyl-3-oxocyclohex-1-enylamino)-1,3-diethylpyrimidine-2,4(1*H*,3*H*)-dione (**15**)

Yellow crystals, m. p. 238–240 °C. – IR (film): $\nu = 3205, 3106, 2954, 2877, 1708, 1654 \text{ cm}^{-1}$. – ¹H NMR (300 MHz, CDCl₃): $\delta = 1.05$ (s, 6H, 2 CH₃), 1.19 (t, 3H, *J* = 6 Hz, CH₃), 1.28 (t, 3H, *J* = 6 Hz, CH₃), 2.18 (s, 2H, CH₂), 2.31 (s, 2H, CH₂), 3.80 (q, 2H, *J* = 9 Hz, CH₂), 4.00 (q, 2H, *J* = 6 Hz, CH₂), 5.41 (s, 1H, dimedone), 6.43 (s, 1H, CH uracil), 7.24 (s, 1H, NH). – ¹³C NMR (75 MHz, CDCl₃): $\delta = 12.69, 14.23, 28.14, 32.70, 37.26, 43.77, 45.31, 50.11, 99.99, 114.35, 131.12, 148.86, 158.59, 160.09, 197.95$. – MS (EI, 70 eV): m/z (%) = 305.15 (35.58) [M]⁺, 290 (100) [M-CH₃]⁺. – Anal. for C₁₆H₂₃N₃O₃ (305.37): calcd. C 62.93, H 7.59, N 13.76; found C 62.82, H 7.71, N 13.81.

An alternative synthesis of compound **13**

A solution of **15** (0.61 g, 2 mmol) and **16** (0.36 g, 3 mmol) in DMF (5 mL) was gently heated (125–135 °C) (TLC control using a solvent system of toluene-acetone (5 : 4)). After 18 h, product **13** was isolated as described above; yield 50 %.

5-((4,4-Dimethyl-2,6-dioxocyclohexylidene)methylamino)-pyrimidine-2,4(1*H*,3*H*)-dione (**18**)

Method A: A solution of **1** (0.25 g, 2 mmol), **3** (0.28 g, 2 mmol) and **16** (0.24 g, 2 mmol) in DMF (10 mL) was refluxed for 8 h (TLC control). The solvent was evaporated under vacuum; the resulting solid was then collected and crystallized from DMF; yield: 80 %.

Method B: A solution of equimolar amounts of **17**(0.4 g, 2 mmol) and **1** (0.25 g, 2 mmol) in DMF (10 mL)

was refluxed for 8 h. Product **18** was isolated as described for method A; yield 85 %. Pale-yellow crystals, m. p. 350–352 °C. – IR (film): $\nu = 3127, 3081, 2954, 2929, 1731, 1666, 1583 \text{ cm}^{-1}$. – $^1\text{H NMR}$ (300 MHz, $[\text{D}_6]\text{DMSO}$): $\delta = 0.99$ (s, 6H, 2Me), 2.30 (s, 2H, CH₂), 2.37 (s, 2H, CH₂), 7.99 (s, 1H, CH *uracil*), 8.36 (d, 1H, $J = 15 \text{ Hz}$, C=H), 11.09 (s, 1H, NH *uracil*), 11.57 (s, 1H, NH *uracil*), 12.29 (d, 1H, $J = 15 \text{ Hz}$, NH *exocyclic*). – MS (EI, 70 eV): m/z (%) = 278.28 (14) $[\text{M}+1]^+$, 277.14 (98) $[\text{M}]^+$. – Anal. for C₁₃H₁₅N₃O₄ (277.28): calcd. C 56.31, H 5.45, N 15.15; found C 56.22, H 5.53, N 15.11.

9,9-Dimethyl-9,10-dihydropyrimido[5,4-c]isoquinoline-2,4,7(1H,3H,8H)-trione (9)

A solution of compound **18** (0.28 g, 1 mmol) in DMF (20 mL) was refluxed for 70 h (TLC control). The solvent was evaporated under vacuum; the resulting solid was crystallized from dioxane, yield 45 %. Brown powder, m. p. 288–290 °C. – IR (film): $\nu = 3430, 3340, 3175, 2957, 1687 \text{ cm}^{-1}$. – $^1\text{H NMR}$ (300 MHz, $[\text{D}_6]\text{DMSO}$): $\delta = 1.12$ (s, 6H, 2CH₃), 2.32 (s, 2H, CH₂), 2.40 (s, 2H, CH₂), 8.45 (s, 1H, 6-H), 11.30 (s, 1H, NH), 11.60 (s, 1H, NH). – MS (EI, 70 eV): m/z (%) = 259 (83.3) $[\text{M}]^+$. – Anal. for C₁₃H₁₃N₃O₃ (259.26): calcd. C 60.22, H 5.05, N 16.21; found C 60.28 H 5.21, N 16.29.

5-((4,4-Dimethyl-2,6-dioxocyclohexylidene)methylamino)-1,3-diethylpyrimidine-2,4(1H,3H)-dione (19)

Ethyl iodide (1.87 g, 12 mmol) was added to a mixture of **18** (0.56 g, 2 mmol) and anhydrous potassium carbonate (0.83 g, 6 mmol) in DMF (20 mL). The reaction mixture was stirred for 36 h at r. t., concentrated to 3 mL and then poured into cold water. After stirring for 15 min, the precipitate was collected by filtration, washed with water, dried and crystallized from petroleum ether/chloroform; yield 55 %. Yellow crystals, m. p. 232–234 °C. – IR (film): $\nu = 3440, 2927, 2873, 1651, 1581 \text{ cm}^{-1}$. – $^1\text{H NMR}$ (300 MHz, CDCl₃): $\delta = 1.04$ (s, 6H, 2 CH₃), 1.22 (t, 3H, $J = 6 \text{ Hz}$, CH₃), 1.35 (t, 3H, $J = 6 \text{ Hz}$, CH₃), 2.36 (s, 2H, CH₂), 2.42 (s, 2H, CH₂), 3.88 (q, 2H, $J = 9 \text{ Hz}$, CH₂), 4.03 (q, 2H, $J = 6 \text{ Hz}$, CH₂), 7.43 (s,

1H, *uracil*), 8.27 (d, 1H, $J = 15 \text{ Hz}$), 12.45 (d, 1H, $J = 9 \text{ Hz}$, NH). – $^{13}\text{C NMR}$ (75 MHz, CDCl₃): $\delta = 12.64, 14.30, 28.46, 31.02, 37.28, 45.47, 51.30, 51.51, 115.73, 125.35, 127.97, 149.02, 158.36, 163.31, 197.40, 197.73$. – MS (EI, 70 eV): m/z (%) = 335.15 (8.01) $[\text{M}+2]^+$, 334.15 (42.27) $[\text{M}+1]^+$, 333.15 (100) $[\text{M}]^+$. – Anal. for C₁₇H₂₃N₃O₄ (333.38): calcd. C 61.25, H 6.95, N 12.60; found C 61.36, H 7.04, N 12.69.

1,3-Diethyl-9,9-dimethyl-9,10-dihydropyrimido[5,4-c]isoquinoline-2,4,7(1H,3H,8H)-trione (20)

Method A: This compound was prepared in 42 % isolated yield by ethylation of **9** using the procedure described for the synthesis of **13**. The solid product was isolated and crystallized from *n*-hexane/benzene.

Method B: This compound was prepared in 50 % isolated yield by heating **19** in refluxing DMF for 60 h. The product **20** was isolated as described above. Dark-brown powder, m. p. 182–185 °C. – IR (film): $\nu = 2931, 2877, 1708, 1651, 1577 \text{ cm}^{-1}$. – $^1\text{H NMR}$ (300 MHz, CDCl₃): $\delta = 1.08$ (s, 6H, 2Me), 1.26 (t, 3H, $J = 6 \text{ Hz}$, CHMe), 1.35 (t, 3H, $J = 6 \text{ Hz}$, CHMe) 2.65 (s, 2H, CH₂), 2.85 (s, 2H, CH₂), 3.86 (q, 2H, $J = 9 \text{ Hz}$, CH₂), 4.10 (q, 2H, $J = 6 \text{ Hz}$, CH₂), 8.27 (s, 1H, CH). – MS (EI, 70 eV): m/z (%) = 315 (15) $[\text{M}]^+$, 314 (10) $[\text{M}-1]^+$. – Anal. for C₁₇H₂₁N₃O₃ (315.37): calcd. C 64.74, H 6.71, N 13.32; found C 64.79, H 6.82, N 13.38.

2,4-Dichloro-9,9-dimethyl-9,10-dihydropyrimido[5,4-c]isoquinoline-7(8H)-one (21)

Compound **18** (0.56 g, 2 mmol) was refluxed in phosphorus oxychloride (5 mL) for 12 h. The mixture was cooled and poured onto ice-water to give a precipitate which was filtered off, dried and recrystallized from petroleum ether/methylene chloride to afford **21**. Yield 50 %. Brown powder, m. p. 222–224 °C. – IR (film): $\nu = 2958, 2831, 1697, 1512 \text{ cm}^{-1}$. – $^1\text{H NMR}$ (300 MHz, $[\text{D}_6]\text{DMSO}$): $\delta = 1.07$ (s, 6H, 2Me), 2.72 (s, 2H, CH₂), 2.86 (s, 2H, CH₂), 8.32 (s, 1H, CH). – MS (EI, 70 eV): m/z (%) = 297 (21.05) $[\text{M}+1]^+$, 296 (19.75) $[\text{M}]^+$, 295.00 (13.25) $[\text{M}-1]^+$. – Anal. for C₁₃H₁₁Cl₂N₃O (296.15): calcd. C 52.72, H 3.74, N 14.19; found C 52.81, H 3.81, N 14.25.

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