EFFICIENT SYNTHESIS OF 2-AMINO-6-ARYL-5,6-DIHYDRO -3*H*-PYRIMIDIN-4-ONE BUILDING BLOCKS VIA DOMINO REACTION

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Abstract

One-pot three component condensation of Meldrum's acid, benzaldehyde derivatives and guanidinium carbonate leads to 2-amino-6-aryl-5,6-dihydro-3*H*-pyrimidin-4-one derivatives in good yields. The products can be utilized in the synthesis of diverse heterocyclic compounds libraries.

Introduction

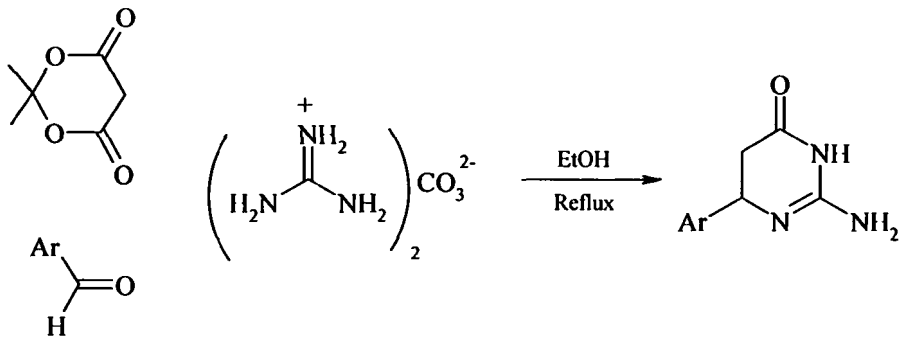
Known heterocycles dihydropyrimidines (DHPMs) represent a heterocyclic system of remarkable pharmacological efficiency, and may exhibit antiviral, antitumor, antimicrobial and anti-inflammatory properties.[1] Several marine natural products containing the DHPM core were found to be potent HIVgp-120-CD4 inhibitors.[2] Some 2-amino-DHPMs were found as novel 5-HT_{5A}-receptor in the limbic brain areas suggests a potential role in the modulation of psychiatric diseases.[3]

There are some reports for the synthesis of 2-amino DHPMs. The reported methods are as follows:

a) Reaction of active methylene compounds, aldehyde and guanidine.[4]
b) Using of resin-bound isothiourea building blocks and multidirectional resin cleavage.[5]
c) Cyclization of 3-guanidinocarboxylic acids by refluxing in hydrochloric acid media.[6]

However, the most general known route to 2-amino-DHPMs is condensation of β-keto esters with guanidine. All the approaches are quite laborious and prolonged; they require initial synthesis of starting materials and give relatively low overall yields.[7] Recently, Gorobets *et al.* reported three component condensation of Meldrum's acid, benzaldehydes and guanidine under conventional and microwave conditions.[8]

Following of our research work about one-pot multicomponent reactions in recent two years,[9] we concentrated on the new Domino Knoevenagel-Michael condensation. In the present work, we report a new three-component reaction of Meldrum's acid, aromatic aldehyde and guanidinum carbonate that provides an easy access to 2-amino-6-aryl-5,6-dihydro-3H-pyrimidin-4-one(2-NH_2-DHPMs) building blocks under reflux condition.



Scheme 1

Results and discussion:

In recent years, domino reactions have attracted considerable attentions. It is a general way to improve synthetic efficiency and also to give access to a multitude of diversified molecules which allow the formation of complex materials from simple starting materials.[10] The quality and importance of a domino reaction can be correlated to the number of bonds generated in such process and increase of complexity. In this work, Meldrum's acid was used as the starting material. It shows several unique features, such as: a) unusual high acidity pK_a= 7.25 b) high activity in electrophilic and nucleophilic reactions c) unique ring –opening reaction d) more synthetic applications as malonic esters.

It is known that Meldrum's acid undergoes standard Knoevenagel condensation with aromatic aldehydes to yield the corresponding arylidene derivatives, which are versatile substrates for different kinds of reactions. Arylidene Meldrum's acid is useful intermediate for different reactions such as: dienophile in hetero-Diels-Alder reaction and also as a Michael acceptor. [11]

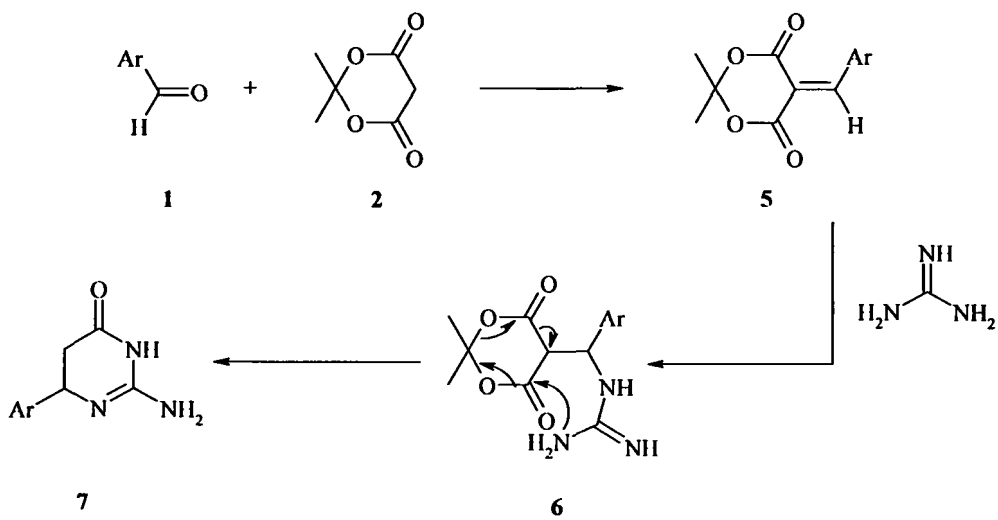
When guanidinium chloride was used for the formation of free guanidine, strong bases such as natrium methoxide must be utilized. [12] In this reaction, guanidinium carbonate which was used had two roles, as a base as well as a source of free guanidine. Free guanidine could be formed only with heating of guanidinium carbonate for 15 min in absolute ethanol or methanol in reflux conditions. In this condition, basic media could accelerate formation of arylidene Meldrum's acid (Knoevenagel condensation) from reaction of Meldrum's acid and aldehydes. The initial reflux of guanidinium carbonate in ethanol is very essential for the progress of reaction. As a model, reaction of Meldrum acid, 4-bromobenzaldehyde and guanidinium carbonate was investigated in two different conditions, a) refluxing of guanidinium carbonate for 15 min and then addition of two other materials reflux for 2.15 h. b) Putting all of three starting materials and heating of the mixture for 2.5 h. In both cases, the product was **4b** with 83 and 30% yields, respectively. (Table 1)

Table1. One-pot Synthesis of 2-amino-6-aryl-5,6-dihydro-3*H*-Pyrimidin-4-ones (2-NH_2-DHPM) **4a-l.**

No.	Ar	Yield* %
4a	C_6H_5	51
4b	*p*-Br-C_6H_4	83
4c	*m*-Cl-C_6H_4	55
4d	*p*-Cl-C_6H_4	77
4e	*2,4*-Cl_2-C_6H_4	72
4f	*p*-CN-C_6H_4	75
4g	*p*-OMe-C_6H_4	57
4h	*p*-Me-C_6H_4	64
4k	*m*-NO_2-C_6H_4	53
4l	*p*-CF_3-C_6H_4	84

*Yields refer to those of pure isolated products characterized by IR, ^1H and ^{13}C NMR spectroscopic data. In all cases, reaction time was 2.5h.

Although we have not established the mechanism for the above reaction in an experimental manner yet, a possible explanation is proposed in scheme 2. Based on this proposed mechanism, reaction could proceed via formation of arylidene Meldrum's acid. When guanidinium carbonate was heated in ethanol, released free guanidine and also the carbonate ion could act as a base for the formation of arylidene Meldrum's acid. Guanidinium carbonate has different roles in this reaction, it is as a source of free guanidine, and also it can provide a mild basic media. Guanidinium carbonate can add the entropy of the reaction which acts as a driving force for this reaction. This mild basic condition accelerates deprotonation of active methylene compound (alkylcyano acetate) and addition of this compound to carbonyl group leads to the desired alkene. The produced alkene acts as a Michael acceptor and reaction proceeds via a tandem Knoevenagel-Michael reaction. In the second step, a Michael addition of free guanidine to arylidene Meldrum's acid afforded intermediate **6** followed by ring closure leading to intermediate **7**. The elimination of acetone, carbon dioxide and water in the next step lead to the formation of desired products **(4a-l)**.



Scheme 2. The proposed mechanism for the synthesis of 2-amino-6-aryl-5,6-dihydro-3H-pyrimidin-4-ones(2-NH_2-DHPM) in reflux condition.

The structure of the products was confirmed by ^1H, ^{13}C-NMR, Mass spectra and CHN analysis data. Formation of arylidene Meldrum's acid was confirmed comparing to the synthetic material. The ^1H-NMR spectra of compound **4a** exhibited a broad singlet peak at δ 7.72 ppm that confirms amide proton and it was also seen as a peak at 3125 cm^{-1} in IR spectra. Meanwhile, there were 3 different protons in the pyrimidinoe structure, -CH_2 protons are diasterotopic protons and they have two different peaks at δ 2.30 ppm and δ 2.52 ppm with $J_{geminal}$= 15.3 Hz, these two doublet peaks could be splitted by C-H proton with J= 8.3 Hz and two protons was shown as doublet of doublet. The second coupling constant was different which was related to different torsion angles. The C-H proton was shown as triplet with J=6.9 Hz. Two (SP^3) carbons showed two peaks in ^{13}C-NMR at δ 38.5 and 52.0ppm and carbonyl group showed a peak at δ=175.8 ppm. This data could confirm the structure of 2-NH_2-DHPM.

In conclusion, we proposed an efficient three-component reaction leading to 2-amino pyrimidinone building blocks. The yield of reactions were 51-84% and with simple work-up and also excellent purity. Thus, they can be applied in generation of diverse 2-NH_2-DHPM libraries.

EXPERIMENTAL

Melting points were determined with Electrothermal 9100 apparatus and were uncorrected. IR spectra were obtained on an ABB FT-IR (FTLA 2000) spectrometer. ^1H and ^{13}C NMR spectra were run on a Bruker DRX-300 instrument at 300 MHz and 75 MHz using TMS as internal standard and DMSO-d_6 as solvent. Mass spectra were recorded on JEOL JMS-700 (HR-EI) spectrometer. Elemental analysis was carried out with CHN-Analyzer Heraeus.

General procedure for the synthesis of compounds 4a-l:

A mixture of Meldrum's acid (283 mg, 2 mmol), corresponding aromatic aldehyde (2 mmol) and guanidinium carbonate (450 mg, 2.5 mmol) in EtOH (40 ml) was heated under reflux condition for 2.5 h. The progress of reaction was monitored by TLC (Eluent, EtOAc: MeOH 10:1). The mixture was cooled and the produced solid was filtered off, washed with water.

Selected data for Compounds 4a-l:

2-Amino-6-phenyl-5,6-dihydropyrimidin-4(3H)-one (**4a**, $C_{10}H_{11}N_3O$):
m.p. 267-268 °C (Lit.[6] m.p. 251-252 °C, Lit.[14] m.p. 266-267 °C, Lit.[8] m.p. 262-263 °C); IR (KBr): ν_{max} = 3330, 3125, 3050, 1636, 1589, 1554, 1498 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.3 (dd, 1H, J = 15.42, 8.3 Hz, H_b), 2.52 (dd, 1H, J = 15.42, 6 Hz, H_c), 4.65 (t, 1H, J = 6.9 Hz, H_a), 6.69 (br s, 2H, NH_2), 7.29-7.39 (m, 5H, H_{Ar}), 7.72 (br s, 1H, NH) ppm;^{13}C-NMR (δ, DMSO-d_6): 40.1, 53.5, 127.7, 129.18, 130.2, 143.4, 163.6, 177.9 ppm; HR-MS (70 eV, EI): $C_{10}H_{11}N_3O$ $[M]^{+\cdot}$ found: 189.0896, calc. 189.0903.

2-Amino-6-(4-bromophenyl)-5,6-dihydropyrimidin-4(3H)-one (**4b**, $C_{10}H_{10}N_3OBr$):
m.p. 289-290 °C (Lit.[8] m.p. 275-276 °C); IR (KBr): ν_{max} = 3355, 3200, 3025, 1628, 1579, 1500, 1399 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.26 (dd, 1H, J = 15.26, 7.7 Hz, H_b), 2.51 (dd, 1H, J = 15.26, 6 Hz, H_c), 4.65 (t, 1H, J = 6.5 Hz, H_a), 6.65 (br s, 2H, NH_2), 7.25 (d, 2H, J = 8 Hz, H_{Ar}), 7.55 (d, 2H, J = 8 Hz, H_{Ar}), 7.7 (br s, 1H, NH) ppm; ^{13}C-NMR (δ, DMSO-d_6): 39.9, 52.9, 122.2, 130.0, 133.0, 143.0, 163.5, 177.4 ppm; Anal. Calc for $C_{10}H_{10}N_3OBr$ (M.W=268.113): C: 44.8, H: 3.8, N: 15.7; Found: C: 44.3, H: 3.7, N: 15.70; HR-MS (70 eV, EI): $C_{10}H_{10}N_3O^{79}Br$ $[M]^{+\cdot}$ found: 266.9998, Calc. 267.1008; $C_{10}H_{10}N_3O^{81}Br$ $[M+2]^{+\cdot}$ found: 268.9963, calc. 268.9987.

2-Amino-6-(3-chlorophenyl)-5,6-dihydropyrimidin-4(3H)-one (**4c**, $C_{10}H_{10}N_3OCl$):
m.p. 260-261 °C; IR (KBr): ν_{max} = 3335, 3115, 3050, 1635, 1572, 1547, 1494 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.32 (dd, 1H, J = 15.34, 7.5 Hz, H_b), 2.5 (dd, 1H, J = 15.34, 6.2 Hz, H_c), 4.69 (t, 1H, J = 6.5 Hz, H_a), 6.69 (br s, 2H, NH_2), 7.26-7.43 (m, 4H, H_{Ar}), 7.76 (br s, 1H, NH) ppm; ^{13}C-NMR (δ, DMSO-d_6): 38.0, 51.2, 124.8, 126.0, 127.5, 130.5, 133.2, 144.4, 161.8, 176.0 ppm; HR-MS (70 eV, EI): $C_{10}H_{10}N_3O^{35}Cl$ $[M]^{+\cdot}$ found: 223.0503, calc. 223.0512; $C_{10}H_{10}N_3O^{37}Cl$ $[M+2]^{+\cdot}$ found: 225.0440, calc. 225.0483.

2-Amino-6-(4-chlorophenyl)-5,6-dihydropyrimidin-4(3H)-one (**4d**, $C_{10}H_{10}N_3OCl$):
m.p. 291-292 °C; IR (KBr): ν_{max} = 3375, 3215, 3095, 1660, 1643, 1556, 1488, 1396 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.26 (dd, 1H, J = 15.02, 7.6 Hz, H_b), 2.5 (dd, 1H, J = 15.02, 6.2 Hz, H_c), 4.67 (br s, 1H, H_a), 6.68 (br s, 2H, NH_2), 7.32 (d, 2H, J = 7.7 Hz, H_{Ar}), 7.42 (d, 2H, J = 7.6 Hz, H_{Ar}), 7.73 (br s, 1H, NH) ppm; ^{13}C-NMR (δ, DMSO-d_6): 38.8, 51.6, 129.0, 132.5, 132.53, 141.4, 162.4, 176.3 ppm; $C_{10}H_{10}N_3O^{35}Cl$ $[M]^{+\cdot}$ found: 223.0497, calc. 223.0512; $C_{10}H_{10}N_3O^{37}Cl$ $[M+2]^{+\cdot}$ found: 225.0472, calc. 225.0483; Anal.Calcd for $C_{10}H_{10}N_3OCl$ (M.W=223.662): C: 53.7, H: 4.5, N: 18.78; Found: C: 52.5, H: 4.4, N: 18.30.

2-Amino-6-(2,4-dichlorophenyl)-5,6-dihydropyrimidin-4(3H)-one (**4e**, $C_{10}H_9N_3OCl_2$):
m.p. 294 °C; IR (KBr): ν_{max} = 3355, 3120, 3085, 1633, 1579, 1545, 1497 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.21 (dd, 1H, J = 15.44, 6.2 Hz, H_b), 2.64 (dd, 1H, J = 15.44, 6.7 Hz, H_c), 4.94 (br s, 1H, H_a), 6.73 (br s, 2H, NH_2), 7.31 (br s, 1H, NH), 7.51 (m, 3H, H_{Ar}) ppm. ^{13}C-NMR (δ, DMSO-d_6): 36.5, 49.1, 128.3, 128.9, 129.6, 132.6, 133.3, 138.6, 162.5, 175.4 ppm; $C_{10}H_9N_3O^{35}Cl_2$ $[M]^{+\cdot}$ found: 257.0097, calc. 257.0123; $C_{10}H_9N_3O^{37}Cl_2$ $[M+2]^{+\cdot}$ found: 259.0060, calc. 259.0093, $C_{10}H_9N_3O^{37}Cl_2$ $[M+4]^{+\cdot}$ found: 261.0056, calc. 261.0064.

2-Amino-6-(4-cyanophenyl)-5,6-dihydropyrimidin-4(3H)-one (**4f**, $C_{11}H_{10}N_4O$):
m.p. 297 °C (Decomposed); IR (KBr): ν_{max} = 3350, 3050, 2195, 1644, 1570, 1540, 1498 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.27 (dd, 1H, J = 15.18, 7.4 Hz, H_b), 2.55 (dd, 1H, J = 15.18, 6.3 Hz, H_c), 4.77 (br s, 1H, H_a), 6.62 (br s, 2H, NH_2), 7.49 (d, 2H, J =7.7 Hz, H_{Ar}), 7.84 (d, 2H, J = 7.9 Hz, H_{Ar}), 7.72 (br s, 1H, NH) ppm; ^{13}C-NMR (δ, DMSO-d_6): 38.4, 51.9, 110.7, 119.2, 127.6, 133.0, 148.2, 162.4, 175.7 ppm. HR-MS (70 eV, EI): $C_{11}H_{10}N_4O$ $[M]^{+\cdot}$ found: 214.0862, calc. 214.0855; Anal.Calcd for $C_{11}H_{10}N_4O$ (M.W=214.227): C: 61.1, H: 4.7, N: 26.15; Found: C: 60.4, H: 4.7, N: 26.20.

2-Amino-6-(4-methoxyphenyl)-5,6-dihydropyrimidin-4(3H)-one (**4g**, $C_{11}H_{13}N_3O_2$):
m.p.278-279 °C (Lit.[8] m.p. 278-279 °C); IR (KBr): ν_{max} = 3360, 3350, 3030, 2825, 1629, 1552, 1501, 1374 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.27 (dd, 1H, J = 15.26, 8.8 Hz, H_b), 2.55 (dd, 1H, J = 15.26, 6 Hz, H_c), 3.73 (s, 3H, CH_3), 4.56 (t, 1H, J = 6 Hz, H_a), 6.47 (br s, 2H, NH_2), 6.91 (d, 2H, J= 8.6 Hz, H_{Ar}), 7.24 (d, 2H, J = 8.6 Hz, H_{Ar}), 7.54 (br s, 1H, NH) ppm; ^{13}C-NMR (δ, DMSO-d_6): 49.5, 53.2, 54.1, 112.0, 125.5, 131.7, 156.8, 160.0, 174.3 ppm; HR-MS (70 eV, EI): $C_{11}H_{13}N_3O_2$ $[M]^{+\cdot}$ found: 219.1019, calc. 219.1008.

2-Amino-6-(4-methylphenyl)-5,6-dihydropyrimidin-4(3H)-one (**4h**, $C_{11}H_{13}N_3O$):
m.p. 274-275 °C; IR (KBr): ν_{max} = 3355, 3150, 3100, 1630, 1577, 1486 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.26 (m, 1H ,H_b), 2.28 (s, 3H, CH_3), 2.46 (m, 1H, H_c), 4.58 (t, 1H, J = 7.1 Hz, H_a), 6.41 (br s, 2H, NH_2), 7.2 (br s, 4H, H_{Ar}), 7.49 (br s, 1H, NH) ppm; ^{13}C-NMR (δ, DMSO-d_6): 21.2, 39.1, 52.1, 126.5, 129.6, 137.3, 139.3, 162.4, 176.6 ppm; HR-MS (70 eV, EI): $C_{11}H_{13}N_3O$ $[M]^{+\cdot}$ found: 203.1046, Calc. 203.1058.; Anal. Calc for $C_{11}H_{13}N_3O$ (M.W=203.2437): C: 65, H: 6.45, N: 20.67; Found: C: 64.4, H: 6.5, N: 20.60.

2-Amino-6-(3-nitrophenyl)-5,6-dihydropyrimidin-4(3H)-one (**4k**, $C_{10}H_{10}N_4O_3$):
m.p.258 °C (Decomposed); IR (KBr): ν_{max} = 3395, 3230, 3120, 1634, 1577, 1515, 1437 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.33 (dd, 1H, J = 15.45, 7.6 Hz, H_b), 2.62 (dd, 1H, J = 15.45, 6.1 Hz, H_c), 4.85 (br s, 1H, H_a), 6.7 (br s, 2H, NH_2), 7.67 (t, 1H, J = 7.9 Hz, H_{Ar}), 7.79 (d, 1H, J =7.5 Hz, H_{Ar}), 7.77 (s, 1H, H_{Ar}), 8.15 (d, 1H, J = 8.0 Hz, H_{Ar}), 8.17 (br s, 1H, NH) ppm; ^{13}C-NMR (δ, DMSO-d_6): 38.4, 51.6, 121.1, 122.9, 130.6, 133.5, 144.8, 148.3, 162.3, 176.0 ppm; HR-MS (70 eV, EI): $C_{10}H_{10}N_4O_3$ $[M]^{+\cdot}$ found: 234.0759, calc. 234.0753.

2-Amino-6-(4-triflouromethylphenyl)-5,6-dihydropyrimidin-4(3H)-one (**4l**, $C_{11}H_{10}N_3OF_3$):
m.p. 301 °C (Decomposed); IR (KBr, cm^{-1}): 3355, 3250, 3035, 1631, 1580, 1540, 1498 cm^{-1}; ^{1}H-NMR (δ, DMSO-d_6): 2.29 (dd, 1H, J = 15.2, 7.2 Hz, H_b), 2.56 (dd, 1H, J = 15.2, 6.1 Hz, H_c), 4.79 (br s, 1H, H_a), 6.65 (br s, 2H, NH_2), 7.51 (d, 2H, J = 7.6 Hz, H_{Ar}), 7.73 (d, 2H, J =7.7 Hz, H_{Ar}), 7.75 (br s, 1H, NH) ppm; HR-MS (70 eV, EI): $C_{11}H_{10}N_3OF_3$ $[M]^{+\cdot}$ found: 257.0757, calc. 257.0776; Anal.Calc for $C_{11}H_{10}N_3OF_3$ (M.W=257.214): C: 51.39, H: 3.92, N: 16.34; Found: C: 51.1, H: 4.0, N: 16.0.

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