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A general synthetic approach to pyrazolo[4,3-*d*]pyrimidines is reported. Aldehydes, arylideneanilines, carboxylic acids and orthoesters are used as one-carbon units for bridging the two amino functions of 4-amino-1-alkyl-3-propylpyrazole-5-carboxamides.

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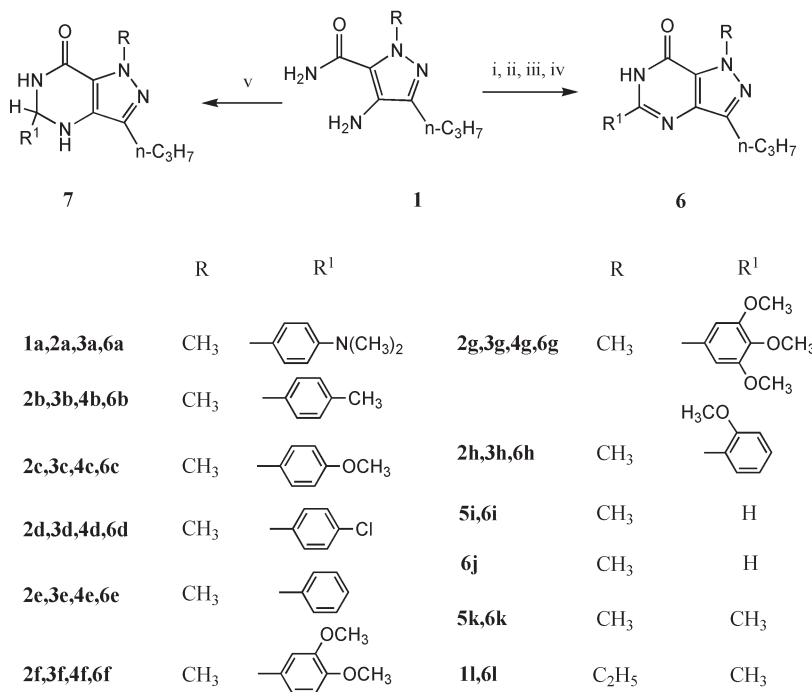
Pyrazolo[4,3-*d*]pyrimidine derivatives [1-10] are the structural analogues of purines. Their excellent adenosine receptor antagonist activity [9] and other useful pharmacological properties such as vasodilator [10,11] and antibacterial [12] activities made them interesting targets for synthesis.

Herein, we report a facile general approach for the synthesis of pyrazolo[4,3-*d*]pyrimidin-7-one derivatives (**6a-l**) from 4-amino-1-methyl (or ethyl)-3-propylpyrazole-5-carboxamide **1a** [13] or **11** using one-carbon electrophilic sources. Pyrazolo[4,3-*d*]pyrimidin-7-ones (**6a-h**) were obtained by refluxing **1a** with appropriate aldehydes **2a-h**

in acetic acid containing a catalytic amount of *p*-toluenesulfonic acid. Tetrahydropyrazolo[4,3-*d*]pyrimidin-7-one derivative **7** was obtained when **1a** was reacted with 4-*N,N*-dimethylamino benzaldehyde in acetic acid at ambient temperature. The intermediacy of **7** in the formation of **6a** was proved by the independent conversion of **7** into **6a** in refluxing acetic acid in the presence of *p*-toluenesulfonic acid.

The versatility of arylideneanilines as aldehyde source-cum-dehydrogenating agents in heterocyclisation reactions was earlier demonstrated in benzimidazole [14,15] and quinazolinone [16,17] chemistry. Therefore, we surmised

Scheme 1



Reaction conditions: i) R<sup>1</sup>-CHO (**2**), AcOH, reflux, *p*-toluenesulfonic acid; ii) 2R<sup>1</sup>-CH=NPh (**3**), AcOH, r.t.; iii) R<sup>1</sup>-CO<sub>2</sub>H (**4**), PPA, 90°C; iv) R<sup>1</sup>-C(OEt)<sub>3</sub> (**5**), xylene, reflux; v) R<sup>1</sup>-CHO(**2**), AcOH, r.t.

Table 1  
Yield and Melting Point of 1-Alkyl-5-aryl-3-propyl-6,7-dihydro-1*H*-pyrazolo[4,3-*d*]pyrimidin-7-ones (**6a-l**)

Entry	Compd. No.	Yield (%)				m.p (°C)
		Method-A	Method-B	Method-C	Method-D	
1	<b>6a</b>	60	55	--	--	245
2	<b>6b</b>	58	60	51	--	240
3	<b>6c</b> [18]	61	52	48	--	242
4	<b>6d</b> [18]	75	59	66	--	239
5	<b>6e</b> [18,19]	57	53	51	--	227
6	<b>6f</b>	68	66	62	--	210
7	<b>6g</b>	70	65	63	--	212
8	<b>6h</b> [20]	55	52	--	--	148
9	<b>6i</b> [21]	--	--	--	83	226
10	<b>6j</b> [22]	--	--	--	83	176
11	<b>6k</b> [23]	--	--	--	78	214
12	<b>6l</b>	--	--	--	77	205

All the compounds were purified by recrystallization from ethyl acetate/*n*-hexane (1:1); Method-A: From the reaction of **1a** with aldehyde in acetic acid under reflux condition; Method-B: From the reaction of **1a** with arylideneaniline (1:2) in acetic acid at room temperature; Method-C: From the reaction of **1a** with aromatic carboxylic acid in PPA; Method-D: From the reaction of **1a** or **1l** with orthoesters.

that, arylideneaniline may as well react with **1a** and provide **6** in one step. Thus 1:2 molar reaction of **1a** and arylideneanilines **3a-h** in acetic acid at ambient temperature directly afforded pyrazolo[4,3-*d*]pyrimidin-7-ones (**6a-h**). On the other hand, an equimolar reaction of **1a** and arylideneaniline under similar experimental conditions provided **7**. Obviously, the second mole of arylideneaniline acted as a dehydrating agent.

Aromatic carboxylic acids **4b-g** also afforded the corresponding pyrazolo[4,3-*d*]pyrimidin-7-one derivatives **6b-g** in one step on heating with **1a** in polyphosphoric acid (PPA), albeit in moderate yields. Triethyl orthoesters **5i** and **5k** directly provided the pyrazolo[4,3-*d*]pyrimidin-7-one derivatives **6i-l** in good yields on reacting with pyrazole carboxamides **1a** / **1l** in refluxing xylene.

IR spectra of compounds **6a-h** showed characteristic amide NH and carbonyl absorptions at ~3200 and ~1680 cm<sup>-1</sup>, respectively. <sup>1</sup>H-NMR spectra of these compounds are characterized by the presence of signals due to *n*-propyl and N-methyl groups of pyrazole moiety in the up field region.

In conclusion, we have developed a facile general method for the synthesis of pyrazolo[4,3-*d*]pyrimidin-7-one derivatives from 4-amino-1-alkyl-3-propyl-1*H*-pyrazole-5-carboxamides by inserting a one-carbon unit derived from suitable aldehydes, arylideneanilines, carboxylic acids and orthoesters. Readily accessible starting materials and simple reaction conditions render this synthesis procedure attractive.

## EXPERIMENTAL

All the melting points were determined in capillaries using

Polman digital melting point apparatus (model no MP 96) and reported in degree centigrade. Ultraviolet spectra were recorded in MeOH on Shimadzu 1601 PC model UV visible spectrophotometer and the absorption maxima are presented in nm. Infrared spectra were recorded in KBr pellets on Shimadzu 435 instrument. The absorptions are quoted in ± 2.5 cm<sup>-1</sup>. Proton magnetic resonance spectra were recorded on Varian Gemini (200 MHz) spectrometer with TMS as internal standard. Chemical shift values are given in δ scale. The solvent in which the NMR spectrum was recorded is indicated at the appropriate places. Mass spectra were recorded on VG-Micro Mass 70-70H instrument with direct inlet probe.

Preparation of 5-Aryl-1-methyl-1,6-dihydro-7*H*-pyrazolo[4,3-*d*]pyrimidin-7-ones (**6a-h**).

General Procedure A.

4-Amino-1-methyl-3-propyl-1*H*-pyrazole-5-carboxamide **1a** (0.01 mol) and the appropriate aldehydes **2a-h** (0.01 mol) were dissolved in acetic acid (20 mL) and catalytic amount of *p*-toluenesulfonic acid (10 mg) was added. The reaction mixture was refluxed for 6 h, cooled to room temperature and diluted with water (60 mL). The crude solid which separated was collected by filtration, dried, and recrystallised from an appropriate solvent to afford 5-aryl-1-methyl-1,6-dihydro-7*H*-pyrazolo[4,3-*d*]pyrimidin-7-ones **6a-h** in good yields.

General Procedure B.

4-Amino-1-methyl-3-propyl-1*H*-pyrazole-5-carboxamide **1a** (0.01 mol) and the appropriate arylideneaniline **3a-h** (0.02 mol) in acetic acid (20 mL) were stirred at room temperature for 4 h. The reaction mixture was diluted with water (100 mL) and the solid that separated was collected by filtration, washed with *n*-hexane (25 mL), dried and recrystallised from suitable solvent to give the 5-aryl-1-methyl-1,6-dihydro-7*H*-pyrazolo[4,3-*d*]pyrimidin-7-ones **6a-h** in good yields.

General Procedure C.

A mixture of 4-amino-1-methyl-3-propyl-1*H*-pyrazole-5-car-

Table 2  
Spectral Data of 1-Alkyl-5-aryl-3-propyl-6,7-dihydro-1*H*-pyrazolo[4,3-*d*]pyrimidin-7-ones (**6a-l**)

Sl. No.	Compd No.	UV $\lambda_{\text{max}}$ nm	IR (KBr, $\text{cm}^{-1}$ ) NH C=O	$^1\text{H}$ NMR ( $\delta$ ppm DMSO- $d_6$ )	$^{13}\text{C}$ NMR ( $\delta$ , ppm)	Mass (m/e)	CHN Analysis
1	<b>6a</b>	295, 250, 220	3176, 1685	1.0 (t, 3H, CH <sub>3</sub> ), 1.85 (m, 2H, CH <sub>2</sub> ), 2.9 (t, 2H, CH <sub>2</sub> ), 3.1 (s, 6H, 2 x CH <sub>3</sub> ), 4.2 (s, 3H, N-CH <sub>3</sub> ), 6.7 (d, 2H, $J=11.5$ Hz, Ar-H), 8.0 (d, 2H, $J=11.5$ Hz, Ar-H), 10.6 (br s, 1H, NH).	---	311 (M <sup>+</sup> ), 296, 283, 282, 155, 147, 145, 140, 137, 134, 119, 91, 77, 67, 53.	Calcd: C, 65.57; H, 6.80; N, 22.49 Found: C, 65.49; H, 6.99; N, 22.55.
2	<b>6b</b>	293, 240, 210	3162, 1681	0.98 (t, 3H, CH <sub>3</sub> ), 1.70 (m, 2H, CH <sub>2</sub> ), 2.40 (s, 3H, Ar-CH <sub>3</sub> ), 2.7 (t, 2H, CH <sub>2</sub> ), 4.1 (s, 3H, N-CH <sub>3</sub> ), 7.1 (d, 2H, $J=11.5$ Hz, Ar-H), 7.3 (d, 2H, $J=11.5$ Hz, Ar-H), 10.3 (br s, 1H, NH).	14.0, 22.3, 27.7, 38.0, 55.4, 114.0, 124.0, 125.3, 128.8, 139.4, 146.5, 149.4, 156.1, 161.8	282 (M <sup>+</sup> ), 267, 254, 253, 239, 118, 116, 91, 77, 65, 57.	Calcd: C, 68.06; H, 6.43; N, 19.84 Found: C, 67.90; H, 6.31; N, 19.79.
3	<b>6c</b>	294, 242, 225	3146, 1675	1.0 (t, 3H, CH <sub>3</sub> ), 1.9 (m, 2H, CH <sub>2</sub> ), 2.9 (t, 2H, CH <sub>2</sub> ), 3.9 (s, 3H, OCH <sub>3</sub> ), 4.2 (s, 3H, N-CH <sub>3</sub> ), 7.0 (d, 2H, $J=11.0$ Hz, Ar-H), 8.2 (d, 2H, $J=11.0$ Hz, Ar-H), 11.7 (br s, 1H, NH).	13.8, 20.8, 21.6, 27.1, 37.8, 127.3, 129.0, 130.1, 137.9, 140.5, 144.8, 150.1, 154.6.	---	---
4	<b>6d</b>	293, 240, 210	3294, 1650	0.8 (t, 3H, CH <sub>3</sub> ), 1.6 (m, 2H, CH <sub>2</sub> ), 2.6 (t, 2H, CH <sub>2</sub> ), 4.0 (s, 3H, N-CH <sub>3</sub> ), 7.2 (d, 2H, $J=12.0$ Hz, Ar-H), 7.9 (d, 2H, $J=12.0$ Hz, Ar-H), 11.8 (br s, 1H, NH).	---	302 (M <sup>+</sup> ), 287, 274, 259, 111, 98, 81, 69, 55, 43.	---
5	<b>6e</b>	288, 238	3184, 1672	1.05 (t, 3H, CH <sub>3</sub> ), 1.8 (m, 2H, CH <sub>2</sub> ), 2.9 (t, 2H, CH <sub>2</sub> ), 4.3 (s, 3H, N-CH <sub>3</sub> ), 7.4 (d, 2H, Ar-H), 7.5-7.6 (m, 3H, Ar-H), 10.8 (br s, 1H, NH).	---	---	---
6	<b>6f</b>	295, 210	3142, 1682	1.0 (t, 3H, CH <sub>3</sub> ), 1.9 (m, 2H, CH <sub>2</sub> ), 2.9 (t, 2H, CH <sub>2</sub> ), 3.9 (s, 3H, OCH <sub>3</sub> ), 4.0 (s, 3H, OCH <sub>3</sub> ), 4.25 (s, 3H, N-CH <sub>3</sub> ), 6.9 (d, 2H, Ar-H), 7.4 (d, 1H, Ar-H), 11.5 (br s, 1H, NH).	13.98, 22.27, 6, 38.0, 55.9, 56.1, 110.5, 110.6, 120.0, 124.0, 125.4, 139.2, 149.1, 149.4, 151.5, 156.0.	328 (M <sup>+</sup> ), 313, 300, 285, 283, 269, 255, 241, 237, 164, 136, 106, 77, 65.	Calcd: C, 62.18; H, 6.14; N, 17.06 Found: C, 62.05; H, 6.26; N, 17.23.
7	<b>6g</b>	292, 225	3280, 1652	1.0 (t, 3H, CH <sub>3</sub> ), 1.8 (m, 2H, CH <sub>2</sub> ), 3.0 (t, 2H, CH <sub>2</sub> ), 3.9 (s, 3H, 4-Ar-OCH <sub>3</sub> ), 4.0 (s, 6H, 3,5-di-Ar-OCH <sub>3</sub> ), 4.3 (s, 3H, N-CH <sub>3</sub> ), 7.3 (s, 2H, Ar-H), 11.1 (br s, 1H, NH).	---	---	Calcd: C, 60.32; H, 6.19; N, 15.63 Found: C, 60.66; H, 6.31; N, 15.82.
8	<b>6h</b>	292	3279, 1652	1.1 (t, 3H, CH <sub>3</sub> ), 1.7 (t, 3H, CH <sub>3</sub> ), 1.9 (m, 2H, CH <sub>2</sub> ), 2.95 (t, 2H, CH <sub>2</sub> ), 4.1 (s, 3H, N-CH <sub>3</sub> ), 4.2 (q, 2H, O-CH <sub>2</sub> ), 7.0 (d, 1H, Ar-H), 7.15 (t, 1H, Ar-H), 7.4 (t, 1H, Ar-H), 8.4 (d, 1H, Ar-H), 11.0 (br s, 1H, NH).	---	312 (M <sup>+</sup> ), 297, 284, 279, 269, 255, 240, 193, 182, 166, 136, 120, 102, 91, 77, 43.	---
9	<b>6i</b>	288, 227	3142, 1681	1.0 (t, 3H, CH <sub>3</sub> ), 1.8 (m, 2H, CH <sub>2</sub> ), 2.8 (t, 2H, CH <sub>2</sub> ), 4.2 (s, 3H, N-CH <sub>3</sub> ), 7.7 (s, 1H, methane proton), 12.0 (br s, 1H, NH).	---	192 (M <sup>+</sup> ), 177, 164, 149, 81, 67, 53.	---
10	<b>6j</b>	287, 228	3160, 1695	1.0 (t, 3H, CH <sub>3</sub> ), 1.5 (t, 3H, N-C-CH <sub>3</sub> ), 1.8 (m, 2H, CH <sub>2</sub> ), 2.9 (t, 2H, CH <sub>2</sub> ), 4.6 (q, 2H, N-CH <sub>2</sub> ), 7.7 (s, 1H, methane proton), 12.0 (br s, 1H, NH).	---	---	---
11	<b>6k</b>	291, 229	3045, 1682	1.0 (t, 3H, CH <sub>3</sub> ), 1.8 (m, 2H, CH <sub>2</sub> ), 2.4 (s, 3H, CH <sub>3</sub> ), 2.9 (t, 2H, CH <sub>2</sub> ), 4.2 (s, 3H, N-CH <sub>3</sub> ), 11.8 (br s, 1H, NH).	---	---	---
12	<b>6l</b>	290, 230	3193, 1687	1.05 (t, 3H, CH <sub>3</sub> ), 1.5 (t, 3H, N-C-CH <sub>3</sub> ), 1.8 (m, 2H, CH <sub>2</sub> ), 2.5 (s, 3H, CH <sub>3</sub> ), 2.85 (t, 2H, CH <sub>2</sub> ), 4.55 (q, 2H, N-CH <sub>2</sub> ), 11.9 (br s, 1H, NH).	---	---	Calcd: C, 59.98; H, 7.32; N, 25.44 Found: C, 60.11; H, 7.19; N, 25.67.

boxamide **1a** (0.01 mol) the appropriate aromatic carboxylic acid **4b-g** (0.01 mol) and polyphosphoric acid (PPA, 10 mL) was heated at 85-90 °C for 2 h. The reaction mixture was poured onto crushed ice (200 g). The separated crude product was collected by filtration, washed with water, dried, and recrystallised from a suitable solvent to afford 5-aryl-1-methyl-1,6-dihydro-7H-pyrazolo[4,3-d]pyrimidin-7-ones **6b-g** in good yields.

#### General Procedure D.

A solution containing 4-amino-1-methyl (or ethyl)-3-propyl-1H-pyrazole-5-carboxamide **1a** or **1l** (1 mmoles) and the appropriate triethyl orthoester **5i** or **5k** (1.5 mmoles) in xylene (20 mL), was heated under reflux for 4 h. The reaction mixture was cooled to room temperature and diluted with *n*-hexane (75 mL). The crude product that separated was collected by filtration, washed with *n*-hexane (10 mL) and recrystallised from suitable solvent to give 5-methyl (or unsubstituted)-1-methyl (or ethyl)-1,6-dihydro-7H-pyrazolo[4,3-d]pyrimidines **6i-l** in good yields.

5-(4-Dimethylaminophenyl)-1-methyl-3-propyl-4,5,6,7-tetrahydro-7H-pyrazolo[4,3-d]pyrimidin-7-one (**7**).

A solution containing 4-amino-3-propyl-1-methyl-1H-pyrazole-5-carboxamide **1l** (0.01 moles) and 4-(dimethylamino) benzaldehyde **2a** (0.01 moles) in glacial acetic acid (10 mL) was stirred at room temperature for 4 h. The reaction mixture was cooled to room temperature and diluted with water (100 mL). The separated crude product was collected by filtration, air-dried and recrystallised from a 1:1 mixture of ethyl acetate and *n*-hexane to give **7** in 71 % yield, m.p. 193 °C; MS: m/z. 313 (M<sup>+</sup>); IR (KBr): 1667 (C=O), 3155 (NH), 3305 (NH); <sup>1</sup>H-NMR (CDCl<sub>3</sub>): δ 1.0 (s, 3H, CH<sub>3</sub>), 1.7 (m, 2H, CH<sub>2</sub>), 2.7 (t, 2H, CH<sub>2</sub>), 3.08 (s, 6H, 2 x N-CH<sub>3</sub>), 4.2 (s, 3H, N-CH<sub>3</sub>), 5.6 (br s, 1H, NH), 6.75 (d, 2H, *J*=11.5 Hz, Ar-H), 7.65 (d, 2H, *J*=11.5 Hz, Ar-H), 8.4 (s, 1H, methane CH), 8.7 (br s, 1H, NH).

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