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Palladium-catalyzed regioselective direct C–H arylation of pyrazolo[3,4-*d*]pyrimidines

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ABSTRACT

Nitrogenous bicycles are an apparently endless field of organic and biological research. In this study, we disclose an efficient pathway to the synthesis of the pyrazolo[3,4-*d*]pyrimidine scaffold in three steps from allopurinol. This key intermediate was engaged in the first example of regioselective C–H arylation catalyzed by palladium to access a library of 3-substituted-1-methyl-4-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidines.

1. Introduction

Pyrazolo[3,4-*d*]pyrimidines are of pharmaceutical interest in a variety of therapeutic areas [1]. They have attracted much attention in drug discovery programs because of their structural resemblance to purine nucleobases. In recent years, researchers have reported the use of purine derivatives of allopurinol (Scheme 1, compound **1**) as kinase inhibitors [2], antiviral agents [3], adenosine antagonists [3c,4], glutamate modulators [5], and antitubercular agents [6].

Recently, the direct C–H arylation of aromatic and heteroaromatic compounds has proved to be an attractive alternative method to traditional cross-coupling reactions [7]. Contrary to palladium-catalyzed couplings, including Suzuki et al. [8], Stille [9], or Negishi et al. [10] couplings, which require the preparation of organometallic reagents (boronic acids, tin, or zinc derivatives), preliminary functionalization is not carried out in direct C–H arylation. In our research program on the stimulation of C–H activation [11], we suggest a new approach for the direct and selective C-3 arylation of substituted 1*H*-pyrazolo[3,4-*d*]pyrimidines using $Pd(OAc)_2$ as a catalyst, with various ligands and bases. The results obtained with this approach in the present investigation are reported and discussed herein.

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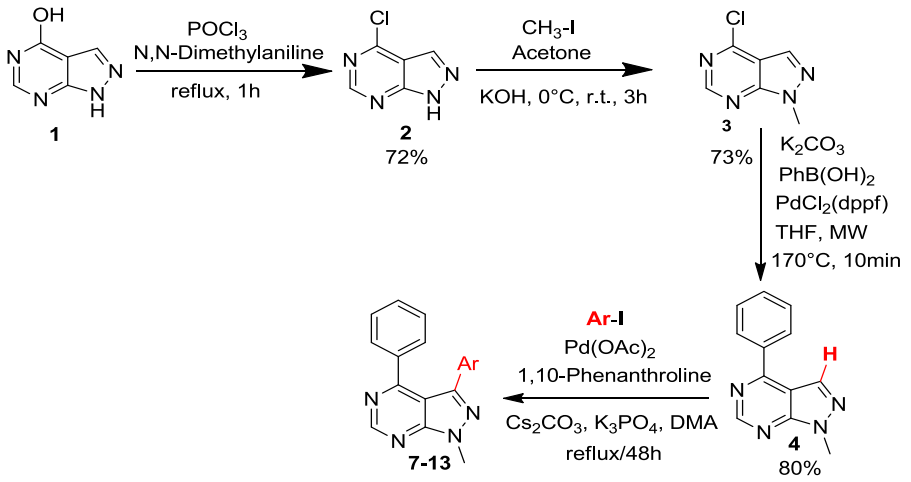


Scheme 1. Strategy for the synthesis of C-3 arylated pyrazolo[3,4-*d*]pyrimidines.

2. Results and discussion

The general synthetic pathway for the preparation of a series of 1*H*-pyrazolo[3,4-*d*]pyrimidines is shown in Scheme 1.

In the pyrazolopyrimidine ring system, the chloro substituent (as a leaving group) was introduced at C-4, which was the most reactive site for nucleophilic attack. The high-temperature reaction of the commercially available allopurinol **1** with excess phosphorus oxychloride in the presence of N—N-dimethylaniline gave an intermediate product **2** [12], which was then allowed to react with methyl iodide so as to afford the corresponding 4-chloro-1-methyl-1*H*-pyrazolo[3,4-*d*]pyrimidine [13] **3**. The use of 1 equiv of methyl iodide allowed us to selectively obtain *N1* methylated isomer. Compound **3** also demonstrated the Suzuki cross-coupling reaction [14]. The 4-chloro substituent of **3** was directly displaced with phenyl to generate compound **4**, affording an additional opportunity for the selective functionalization of this molecule.

We started our optimization by investigating the direct arylation of 4-phenyl-1-methyl pyrazolopyrimidine **4** with 4-bromotoluene **5a**. The reaction conditions were the same as those already used in our previous study [11f]. Compound **4** (1 equiv) was allowed to react with **5a** (2 equiv) in the presence of palladium(II) acetate (10 mol %), triphenylphosphine (20 mol %), and K_2CO_3 (2 equiv) in toluene at 110 °C for 48 h, but no C-3 arylation was observed (Table 1, entry 1). On replacing the ligand by tricyclohexylphosphine (20%), direct C—H arylation then took place at the 3-position of **4** to give product **7** in 28% yield (Table 1, entry 3). Using another base such as cesium carbonate and increasing the amount of $Pd(OAc)_2$ and tricyclohexylphosphine to 20% and 40% mol, respectively, no noticeable improvement in the reaction was detected (Table 1, entries 4 and 5). Using 1,10-phenanthroline instead of tricyclohexylphosphine in the presence of $Pd(OAc)_2$, 4-iodotoluene (2 equiv), and Cs_2CO_3 (3 equiv) in DMA at 165 °C produced **7** in 65% yield (Table 1, entry 7). With K_3PO_4 as additive conversion improved to 80% (Table 1, entry 8), whereas using 4-bromotoluene instead of 4-iodotoluene led to a decrease in conversion to 72% (Table 1, entry 9). When decreasing the amount of $Pd(OAc)_2$ and 1,10-phenanthroline to 10 and 20 mol %, respectively, the yield of the desired product **7** diminished (60%, Table 1, entry 10).

We tested the latter conditions without using 1,10-phenanthroline, but no C3 arylation was detected (Table 1, entry 11). This result displays the crucial role of the ligand in C—H activation.

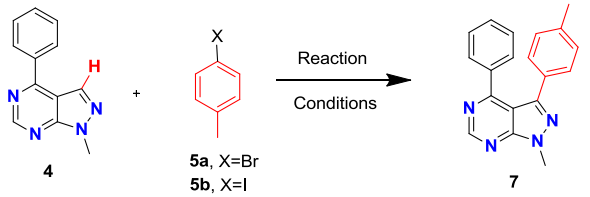
Finally the best results were obtained in the presence of palladium (II) acetate (20 mol %), 1,10-phenanthroline (40 mol %), 4-iodotoluene (2 equiv), Cs_2CO_3 (3 equiv), and K_3PO_4 (2.5 equiv) in DMA at 165 °C for 48 h (entry 8).

Comparable yields were attained using diverse aryl iodides bearing *ortho*, *meta*, and *para* substituents (Table 2, entries 1—7). This result highlights the great adaptability of our strategy. Indeed, we were pleased to observe that aryl-bearing electron-rich (MeO, Me) or electron-poor (CF3, CN, and so forth) substituents in the C-3 position were well tolerated. The molecular structure of compound **7** was confirmed by X-ray crystallographic analysis and proved that C—H arylation occurred regioselectively at position 3 (Fig. 1).

Afterward, we prepared the compound 4-methoxy-1-methyl-1*H*-pyrazolo[3,4-*d*]pyrimidine [15] **14** by reacting 4-chloro-1-methyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **3** with sodium methoxide and methyl alcohol in THF, then we tested the optimal conditions found (Table 1, entry 8) on these compounds functionalized at position 4 (**3** and **14**). No C-3 arylation was detected, and only degradation was observed (Scheme 2).

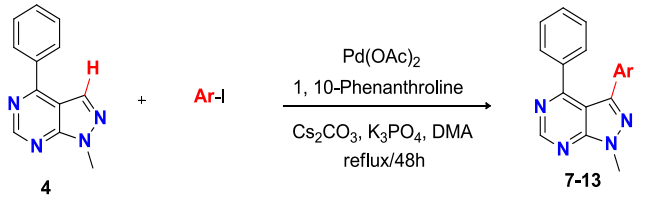
On the basis of the newly reported studies on the C-3 arylation reaction [11b,11c], a probable mechanism for the C-3 arylation on pyrazolopyrimidine is put forward (Scheme 3). $Pd(OAc)_2$ and ligand form a Pd(II) intermediate (complex **1**, Scheme 3) followed by a Pd(II)—pyrazolopyrimidine complex (complex **2**, Scheme 3). Then an oxidative addition of aryl halide (X = I or Br) results in a Pd(IV) complex (complex **3**, Scheme 3). After reductive elimination, the required compound **7** is achieved, and complex **4** is formed. In the end, complex **4** is transformed into complex **1**.

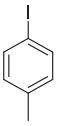
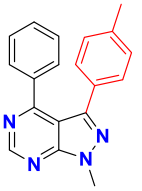
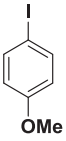
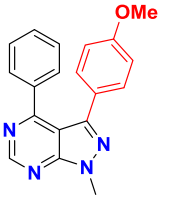
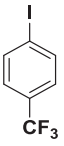
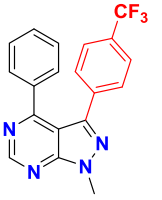
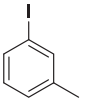
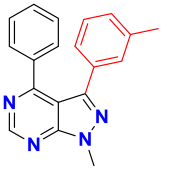
Table 1
Optimization of the C-3 arylation reaction of **4**.



Entry	Catalyst (mol %)	Ligand (mol %)	Base (equiv)	*X* (equiv)	Solvent	*T* (°C)	% Yield
1	$Pd(OAc)_2$ (10%)	PPh_3 (20%)	K_2CO_3 (2.0)	Br (2.0)	Toluene	110	0
2	$Pd(OAc)_2$ (10%)	PPh_3 (20%)	K_2CO_3 (2.0)	I (2.0)	Toluene	110	0
3	$Pd(OAc)_2$ (10%)	PCy_3 (20%)	K_2CO_3 (2.0)	I (2.0)	Dioxane	120	28
4	$Pd(OAc)_2$ (10%)	PCy_3 (20%)	Cs_2CO_3 (2.0)	I (2.0)	Dioxane	120	35
5	$Pd(OAc)_2$ (20%)	PCy_3 (40%)	Cs_2CO_3 (3.0)	I (2.0)	Dioxane	120	40
6	$Pd(OAc)_2$ (20%)	Phen (40%)	K_2CO_3 (3.0)	I (2.0)	DMA	165	48
7	$Pd(OAc)_2$ (20%)	Phen (40%)	Cs_2CO_3 (3.0)	I (2.0)	DMA	165	65
8	$Pd(OAc)_2$ (20%)	Phen (40%)	Cs_2CO_3 (3.0)/K_3PO_4 (2.5)	I (2.0)	DMA	165	80
9	$Pd(OAc)_2$ (20%)	Phen (40%)	Cs_2CO_3 (3.0)/K_3PO_4 (2.5)	Br (2.0)	DMA	165	72
10	$Pd(OAc)_2$ (10%)	Phen (20%)	Cs_2CO_3 (3.0)/K_3PO_4 (2.5)	I (2.0)	DMA	165	60
11	$Pd(OAc)_2$ (20%)	–	Cs_2CO_3 (3.0)/K_3PO_4 (2.5)	I (2.0)	DMA	165	0

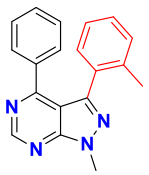
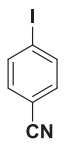
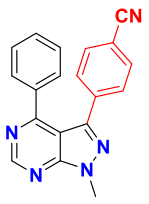
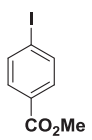
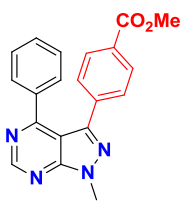
Table 2
Scope and limitation study of C-3 arylation.



Entry	Ar-I	Product	Yield (%)[a]
1			80[b]
2			71[b]
3			65[b]
4			69[b]

(*continued on next page*)

Table 2 (*continued*)

Entry	Ar-I	Product	Yield (%)[a]
5			63[b]
6			49/61[c]
7			43/59[c]

[a] Isolated yield after column chromatography.
[b] Conditions: $Pd(OAc)_2$ (20 mol %), 1,10-phenanthroline (40 mol %), aryl iodide (2.0 equiv), Cs_2CO_3 (3.0 equiv), and K_3PO_4 (2.5 equiv) in DMA.
[c] Using 4 equiv of ArI.

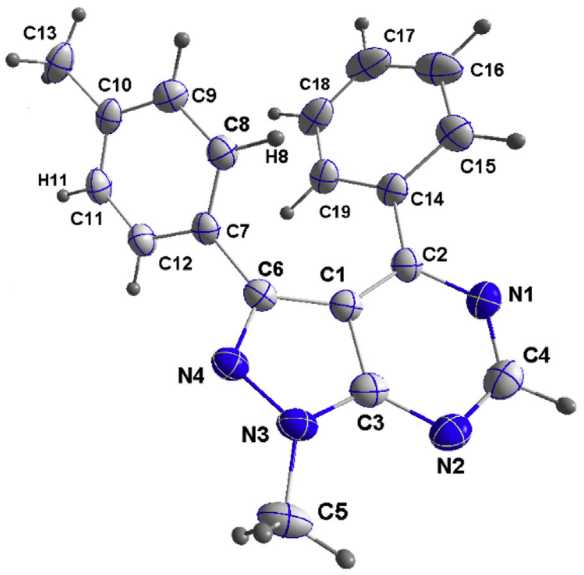


Fig. 1. X-ray structure of compound **7**.

3. Conclusion

It has been shown for the first time that Pd-catalyzed regioselective direct C-3 arylation of substituted 1*H*-pyrazolo[3,4-*d*]pyrimidine is possible by using 1,10-phenanthroline as a ligand. Satisfactory to excellent yields were achieved for a wide range of aryl coupling partners with both electron-rich and electron-poor substituents, providing access to a library of various 1*H*-pyrazolo[3,4-*d*]pyrimidine compounds.

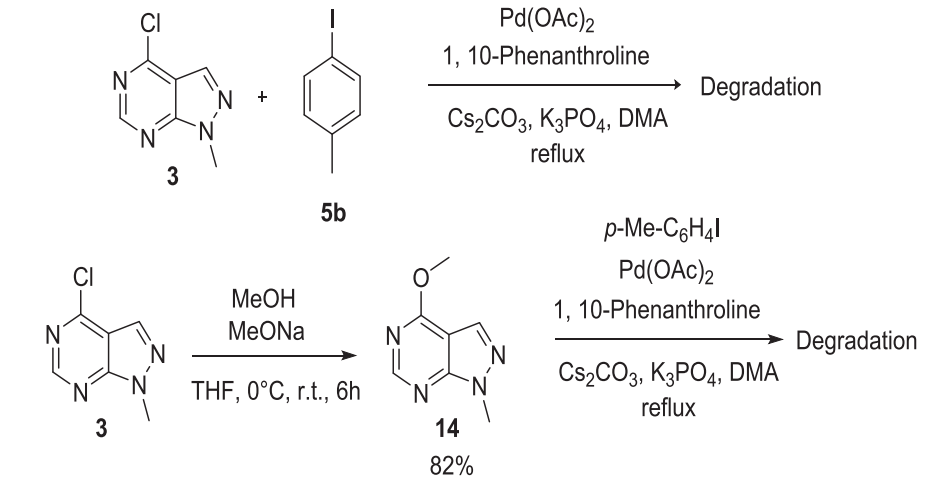
4. Experimental section

4.1. Materials and instrumentation

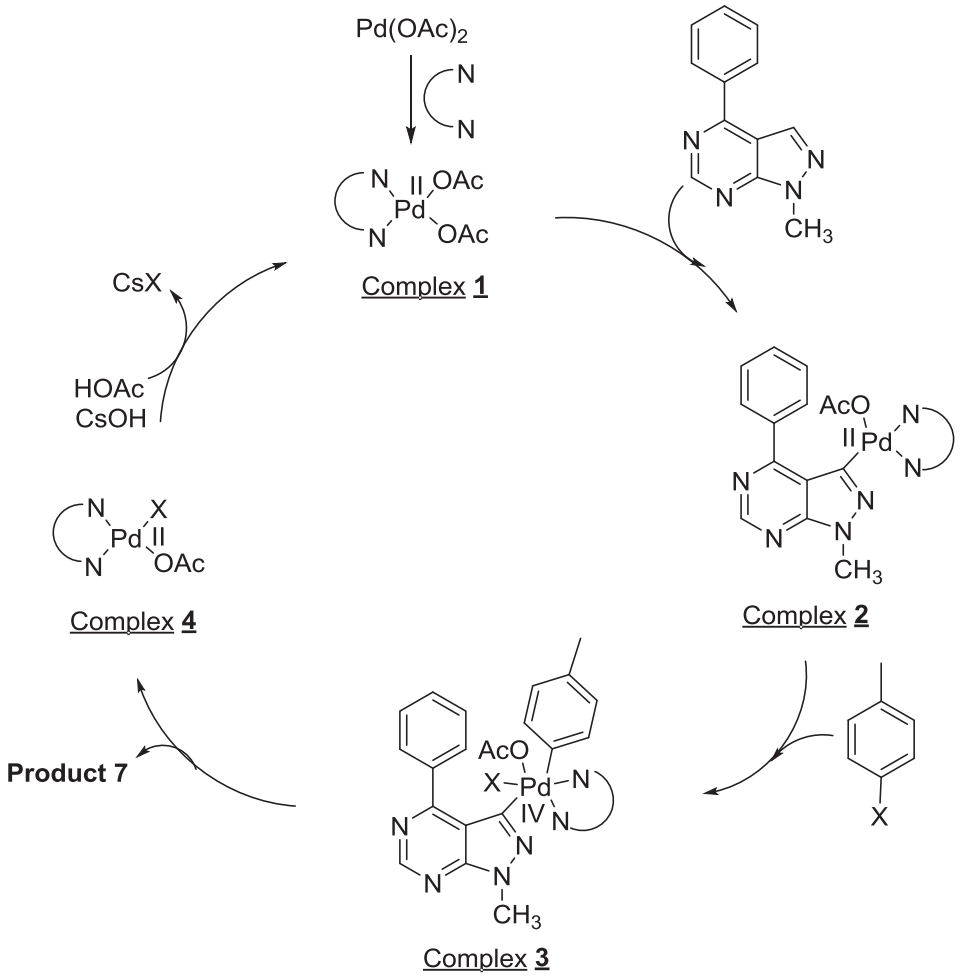
All reagents were purchased from commercial suppliers and were used without further purification. Microwave-assisted reactions were carried out in a Biotage Initiator microwave synthesis instrument and temperatures were measured by an IR sensor. The reactions were monitored by thin-layer chromatography analysis using silica gel (60 F254) plates. Compounds were visualized by UV irradiation. Flash column chromatography was performed on silica gel 60 (230–400 mesh, 0.040–0.063 mm). Melting points (mp [°C]) were taken on samples in open capillary tubes and are uncorrected. ^1H and ^{13}C NMR spectra were recorded on a spectrometer at 250 MHz (13C, 62.9 MHz) or 400 MHz (13C, 100 MHz). Chemical shifts are given in parts per million from tetramethylsilane as an internal standard. The following abbreviations are used for the proton spectra multiplicities: s, singlet; d, doublet; t, triplet; q, quartet; qt, quintuplet; and m, multiplet. Coupling constants (*J*) are reported in Hertz (Hz). High-resolution mass spectra (HRMS) were performed on a Maxis Bruker 4G by the "Federation de Recherche" ICOA/CBM (FR2708) platform.

4.2. General procedure for direct arylation

A mixture 1-methyl-4-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine **4** (0.1 g, 0.47 mmol), aryl iodide **5b** (2 equiv),



Scheme 2. C-3 arylation of **3** and **14**.



Scheme 3. Possible mechanism for C-3 arylation.

Cs_2CO_3 (1.42 mmol, 3 equiv), K_3PO_4 (1.18 mmol, 2.5 equiv), 1,10-phenanthroline (0.19 mmol, 0.4 equiv), and $Pd(OAc)_2$ (0.094 mmol, 0.2 equiv) in DMA (3 mL) was prepared under an atmosphere of argon. The resulting mixture was flushed with argon and heated to 165 °C for 48 h. After completion of the reaction, the mixture was then allowed to cool to room temperature, and the solvent was removed under reduced pressure, water (15 mL) was added, and the resulting aqueous phase was extracted with CH_2Cl_2 (3 × 15 mL). The combined organic layers were dried over $MgSO_4$ and concentrated under vacuum. The residue was purified by column chromatography on silica gel (EtOAc/petroleum ether).

1-Methyl-4-phenyl-3-(*p*-tolyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine (**7**): Yellow solid; Yield (0.114 g, 0.38 mmol), 80%; mp 128–130 °C. ^{1}H NMR (400 MHz, methanol-d_4) δ: 8.91 (s, 1H), 7.41–7.31 (m, 3H), 7.19 (dd, J = 6.8, 1.4 Hz, 2H), 6.95 (dd, J = 10.3, 3.0 Hz, 4H), 4.10 (s, 3H), 2.27 (s, 3H). ^{13}C NMR (101 MHz, methanol-d_4) δ: 163.25, 154.1, 153.9, 145.5, 138.3, 136.2, 130.0, 129.7 (2C), 129.1, 129.0 (2C), 128.1 (2C), 127.7 (2C), 109.1, 32.8, 19.9. HRMS (ESI): calcd for $C_{19}H_{17}N_4$ $[M+H]^+$ 301.1448; found 301.1452.

3-(4-Methoxyphenyl)-1-methyl-4-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidine (**8**): Yellow solid; Yield (0.107 g, 0.34 mmol), 71%; mp 139–141 °C. ^{1}H NMR (400 MHz, $CDCl_3$-*d*) δ: 9.09 (s, 1H), 7.49 (d, J = 7.4 Hz, 2H), 7.39 (t, J = 7.4 Hz, 1H), 7.31–7.19 (m, 2H), 7.15 (d, J = 8.5 Hz, 2H), 6.71 (d, J = 8.5 Hz, 2H), 4.23 (s, 3H), 3.79 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$-*d*) δ: 163.3, 160.0, 154.9, 154.6, 145.3, 136.8, 130.8 (2C), 130.3, 130.2 (2C), 128.2 (2C), 125.0, 113,6 (2C), 109.6, 55.5, 34.1. HRMS (ESI): calcd for $C_{19}H_{17}N_4O$ $[M+H]^+$ 317.1397; found 317.1396.

1-Methyl-4-phenyl-3-[4-trifluoromethyl)phenyl]-1*H*-pyrazolo[3,4-*d*]pyrimidine (**9**): White solid; Yield (0.109 g, 0.31 mmol), 65%; mp 145–147. ^{1}H NMR (400 MHz, methanol-d_4) δ: 9.04 (s, 1H), 7.43 (m, 5H), 7.36 (d, J = 8.0 Hz, 2H), 7.25 (dd, J = 7.5, 3.0 Hz, 2H), 4.22 (s, 3H). ^{13}C NMR (101 MHz, methanol-d_4) δ: 164.6, 155.8, 155.5, 145.2, 137.6, 137.4, 131.65 (2C), 131.3 (q, $^2J_{Cq,F}$ = 32.3 Hz), 131.1 (2C), 131.0 (2C), 129.2 (2C), 125.8 (q, $^3J_{CH,F}$ = 4 Hz), 125.5 (q,$^1J_{Cq,F}$ = 273 Hz), 110.8, 34.4. HRMS (ESI): calcd for $C_{19}H_{14}F_3N_4$ $[M+H]^+$ 355.1165; found 355.1167.

1-Methyl-4-phenyl-3-(*m*-tolyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine (**10**): Yellow oil; Yield (0.099 g, 0.33 mmol), 69%. ^{1}H NMR (400 MHz, methanol-d_4) δ: 8.95 (s, 1H), 7.43–7.32 (m, 3H), 7.21 (dd, J = 11.1, 4.7 Hz, 2H), 7.08–7.04 (m, 3H), 6.76 (s, 1H), 4.14 (s, 3H), 2.04 (s, 3H). ^{13}C NMR (101 MHz, methanol-d_4) δ: 163.2, 154.1, 153.9, 145.5, 137.3, 136.2, 131.6, 130.3, 130.0, 129.7 (2C), 128.8, 127.6 (2C), 127.5, 125.9, 109.2, 32.8, 19.85. HRMS (ESI): calcd for $C_{19}H_{17}N_4$ $[M+H]^+$ 301.1447; found 301.1450.

1-Methyl-4-phenyl-3-(*o*-tolyl)-1*H*-pyrazolo[3,4-*d*]pyrimidine (**11**): Yellow solid; Yield (0.090 g, 0.30 mmol), 63%; mp 132–134 °C. ^{1}H NMR (400 MHz, $CDCl_3$-*d*) δ: 9.11 (s, 1H), 7.42 (t, J = 4.9 Hz, 2H), 7.24 (dd, J = 8.6, 4.0 Hz, 2H), 7.17–7.07 (m, 4H), 7.03 (d, J = 9.3 Hz, 1H), 4.24 (s, 3H), 1.84 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$-*d*) δ: 163.5, 155.45, 154.5, 145.3, 137.8, 136.5, 133.1, 130.8, 130.65, 130.5, 123.0 (2C), 129.3128.2 (2C), 126.1, 111.3, 34.5, 20.1. HRMS (ESI): calcd for $C_{19}H_{17}N_4$ $[M+H]^+$ 301.1448; found 301.1447.

4-(1-Methyl-4-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-3-yl)benzonitrile (**12**): White solid; Yield (0.090 g, 0.29 mmol), 61%; mp 135–137 °C. ^{1}H NMR (400 MHz, methanol-d_4) δ: 9.08 (s, 1H), 7.54 (d, J = 8.0 Hz, 2H), 7.49–7.44 (m, 3H), 7.39 (d, J = 8.0 Hz, 2H), 7.31 (t, J = 7.5 Hz, 2H), 4.25 (s, 3H). ^{13}C NMR (101 MHz, methanol-d_4) δ: 162.5, 156.3, 154.9, 149.1, 140.2, 138.0, 134.7 (2C), 134.5 (2C), 133.1, 130.8 (2C), 130.5 (2C), 119.5, 113.1, 112.9, 34.5. HRMS (ESI): calcd for $C_{19}H_{14}N_5$ $[M+H]^+$ 312.1244; found 312.1243.

Methyl 4-(1-methyl-4-phenyl-1*H*-pyrazolo[3,4-*d*]pyrimidin-3-yl)benzoate (**13**): White solid; Yield (0.097 g, 0.28 mmol), 59%; mp 158–160 °C. ^{1}H NMR (400 MHz, methanol-d_4) δ: 9.07 (s, 1H), 7.83–7.80 (m, 2H), 7.48–7.43 (m, 4H), 7.35–7.30 (m, 3H), 4.24 (s, 3H), 3.91 (s, 3H). ^{13}C NMR (101 MHz, methanol-d_4) δ: 167.5, 164.9, 155.75, 155.5, 147.1, 139.9, 137.8, 131.6, 131.3 (2C), 130.7, 130.6 (2C), 129.75 (2C), 129.3 (2C), 110.7, 52.7, 34.4. HRMS (ESI): calcd for $C_{20}H_{17}N_4O_2$ $[M+H]^+$ 345.1345; found 345.1346.

Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.crci.2017.06.004.

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