$ZnFe_2O_4$ nanoparticles as a robust and reusable magnetically catalyst in the four component synthesis of [(5-hydroxy-3-methyl-1*H*-pyrazol-4yl) (phenyl) methyl]propanedinitriles and substituted 6-amino-pyrano[2,3-*c*]pyrazoles

Javad Safaei-Ghomi*, Hossein Shahbazi-Alavi and Elham Heidari-Baghbahadorani

Department of Organic Chemistry, Faculty of Chemistry, University of Kashan, Kashan, PO Box 87317-51167, Iran

A simple method has been developed for the synthesis of [(5-hydroxy-3-methyl-1H-pyrazol-4-yl)(phenyl)methyl]propanedinitriles and 6-amino-4-aryl-3-methyl-2,4-dihydro pyrano[2,3-*c*]pyrazole-5-carbonitrile derivatives through a one-pot four-component condensation reaction of aromatic aldehydes, malononitrile, ethyl acetoacetate and hydrazine hydrate using $ZnFe_2O_4$ nanoparticles under solvent-free conditions. This has the advantages of excellent yields, short reaction times, simple workup and environmentally benign.

Keywords: pyranopyrazoles, pyrazolpropanedinitriles, $ZnFe_2O_4$ nanoparticles, one-pot condensation reaction, solvent-free conditions, robust catalyst

The pyranopyrazole ring is one of the important heterocyclic systems which show some pharmacological and biological properties such as Molluscicidal,[1] analgesic and anti-inflammatory,[2] Chk1 inhibitors[3] activities. The development of new, rapid and clean synthetic routes towards libraries of such compounds is of value to both medicinal and synthetic chemistsThe synthesis of pyranopyrazoles through multicomponent reactions (MCRs) received attention owing to their excellent synthetic efficiency, inherent atom economy, procedural simplicity, and environmental friendliness.[4-6] Substituted 6-aminopyrano[2,3-*c*]pyrazoles were first synthesised by a reaction between 3-methyl-5-pyrazolone with tetracyanoethylene.[7] Various 2,4-dihydropyrano [2,3-*c*] pyrazole-5-carbonitriles were synthesised using γ-alumina,[8] piperidine,[9] imidazole,[10] glycine,[11] $[(CH_2)_4SO_3HMIM][HSO_4]$,[12] as a catalyst. Although many methods for the synthesis of pyrano[2,3-*c*]pyrazoles are known, some have drawbacks, including long reaction times, difficult purification, high catalyst loading and non-reusable catalyst, and may require special conditions. Our method provides several advantages including mild reaction condition, applicability to wide range of substrates, easy purification, reusability of the catalyst and low catalyst loading.

Organic reactions under solvent free conditions have attracted interest from chemists particularly from the viewpoints of green chemistry. Green chemistry emphasises the development of environmentally benign chemical processes. From this point of view, solvent-free MCRs are appealing procedures.[13,14]

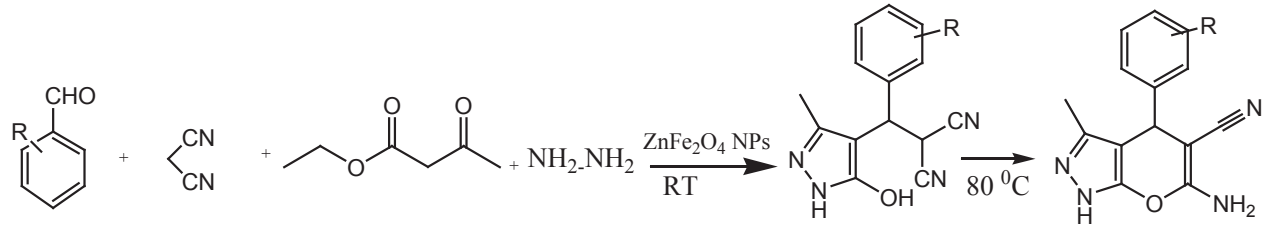
Expansion of new catalytic transformations with simple separation and recyclability of the catalyst is an essential task in chemical synthesis.[15] To overcome the separation problems of the nano catalysts, magnetic materials have emerged as recoverable catalysts. Separation of magnetic nanoparticles is simple, convenient, economical and environmentally benign.[16] We now report the synthesis of [(5-hydroxy-3-methyl-1*H*-pyrazol-4-yl) (phenyl)methyl]propanedinitriles and 6-amino-4-aryl-3-methyl-2,4-dihydropyrano[2,3-*c*] pyrazole-5-carbonitrile derivatives by a one-pot four-component condensation reaction of aromatic aldehydes, malononitrile, ethyl acetoacetate and hydrazine hydrate catalysed by $ZnFe_2O_4$ NPs (Scheme 1).

Results and discussion

In order to optimise the reaction conditions, the condensation reaction of 4-nitrobenzaldehyde, malononitrile, hydrazine hydrate and ethyl acetoacetate was selected as a model. We studied the effects of the catalyst and solvent on the synthesis of 6-amino-4-(4-nitrophenyl)-3-methyl-2,4-dihydropyrano [2,3-*c*]pyrazole-5-carbonitrile (Scheme 2). A wide variety of catalysts including P_2O_5, $ZnCl_2$, CaO, CuO, ZrO_2, $ZrOCl_2$ and $ZnFe_2O_4$ NPs were employed to test their efficacy for the synthesis of pyranopyrazoles. Next, we optimised the amount of $ZnFe_2O_4$ NPs required; the optimum amount was found to be 8 mol %. We examined the reaction in different solvents including acetonitrile, ethanol, water and solvent-free. The best results were obtained under solvent-free conditions. The results are presented in Table 1. The generality of this four-component reaction was studied under optimal conditions by varying the structure of the aldehydes (Table 2).

An important feature of this method is that both electron-releasing and withdrawing groups give excellent yields. A decrease in the temperature led to a decrease of cyclisation rate, so under these conditions only compound **5** was produced.

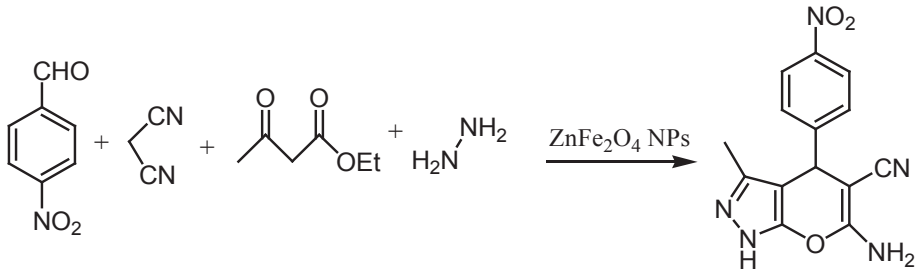
The recoverability of the nano–$ZnFe_2O_4$ catalyst was examined for the synthesis of product **6f** and it was found that



1 **2** **3** **4** **5** **6 a-j**

Scheme 1 Synthesis of pyranopyrazoles from aldehydes, malononitrile, ethyl acetoacetate, and hydrazine hydrate using $ZnFe_2O_4$ nanoparticles.

* Correspondent. E-mail: safaei@kashanu.ac.ir



Scheme 2 The model reaction for the preparation of 6-amino-4-(4-nitrophenyl)-3-methyl-2,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitrile **6f**.

Table1 Optimisation of reaction condition using different catalysts [a]

Entry	Solvent	Conditions	Catalyst/mol	Time/min	Yield/%[b]
1	EtOH	Reflux	P_2O_5 (3)	50	31
2	CH_3CN	40° C	$ZnCl_2$ (3)	60	29
3	H_2O	Reflux	CaO (5)	55	38
4	CH_3CN	40 °C	CuO (5)	45	43
5	H_2O	80 ° C	ZrO_2 (4)	50	37
6	EtOH	50 °C	H_3PO_4 (2)	60	49
7	CH_3CN	40° C	$ZrOCl_2$ (5)	40	62
8	EtOH	Reflux	ZrO_2 (4)	40	58
9	EtOH	Reflux	$ZnFe_2O_4$NPs (8)	30	73
10	Solvent-free	80 °C	$ZnFe_2O_4$NPs (5)	15	82
11	**Solvent-free**	**80 °C**	$ZnFe_2O_4$NPs (**8**)	**15**	**92**
12	Solvent-free	80 °C	$ZnFe_2O_4$NPs (12)	15	91

[a] 4-Nitrobenzaldehyde (1mmol), malononitrile (1mmol), ethyl acetoacetate (1mmol) hydrazine hydrate (1mmol) for synthesis 6-amino-4-(4-nitrophenyl)-3-methyl-2,4-dihydropyrano[2,3-c] pyrazole-5-carbonitrile .
[b]Isolated yield.

Table 2 Synthesis of [(5-hydroxy-3-methyl-1*H*-pyrazol-4-yl)(phenyl)methyl]propanedinitriles (**5a**,**b**) and 6-amino-4-aryl-3-methyl-2,4-dihydropyrano[2,3-*c*]pyrazole-5-carbonitriles (**6a–j**)

Entry	Aldehyde (R)	Products	Time/min	Yield/·[b]	M.p./º C [ref]
1	H	**5a**	9	91	256–258[18]
2	4-OMe	**5b**	12	88	207–208[18]
3	H	**6a**	15	88	244–246[8]
4	4-Me	**6b**	14	90	206–208[8]
5	2-Me	**6c**	15	89	209–211
6	3-Me	**6d**	17	89	212–214
7	2-OMe	**6e**	19	85	215–217
8	4-NO_2	**6f**	15	92	251–253[8]
9	4-Cl	**6g**	14	92	234–236[8]
10	2-F	**6h**	15	90	163–165
11	4-Br	**6i**	14	91	178–180[8]
12	4-OMe	**6j**	16	87	210–212[8]

[a]Hyrazine hydrate (1 mmol), ethyl acetooacetate (1 mmol), malononitrile (1mmol), aldehydes (1 mmol)with $ZnFe_2O_4$ nanoparticles.
[b]Isolated yield.

product yields decreased to a small extent on each reuse (run 1, 92%; run 2, 91%; run 3, 91%; run 4, 90%; run 5, 90%). In the recycling procedure of $ZnFe_2O_4$ NPs, after completion of the reaction, 5 mL ethanol was added and magnet was introduced into the mixture in the form of a magnetic stirrer bar and catalyst was separated magnetically. It is important to note that the catalyst could be recovered magnetically and washed with acetone to remove the residual product.

In conclusion, it was found that under the optimal conditions the reaction between malononitrile, hydrazine hydrate, ethyl acetoacetate and aromatic aldehydes in the presence of 8 mol % of $ZnFe_2O_4$ at 80 °C leads to pyrano[2,3-*c*]pyrazoles selectively in excellent yields. A decrease in the temperature led to decrease of cyclisation rate, so that only [(5-hydroxy-3-methyl-1*H*-pyrazol-4-yl) (phenyl) methyl] propanedinitrile derivatives were formed. Mild reaction conditions, operational simplicity, the use of green catalyst, no organic solvent and high isolated yields of pure products are significant advantages of the method described here.

Experimental

All organic materials were purchased commercially from Sigma-Aldrich and Merck and were used without further purification. All melting points were uncorrected and were determined in a capillary tube on a Boetius melting point microscope or on Electro thermal 9200. The elemental analyses (C, H, N) were obtained from a Carlo ERBA Model EA 1108 analyser. FTIR spectra were recorded with KBr pellets using a Magna-IR, spectrometer 550 Nicolet. NMR spectra

were recorded on a Bruker 400 MHz spectrometer with DMSO as solvent and TMS as an internal standard. Powder XRD was carried out on a Philips diffractometer of X'pert Company. Microscopic morphology of the products was visualised by SEM (MIRA 3 TESCAN).

Synthesis of $ZnFe_2O_4$ nanoparticles

$ZnFe_2O_4$ nanoparticle was prepared according to the procedure reported in the literature.[17] Iron (III) chloride hexahydrate ($FeCl_3.6H_2O$), zinc (II) chloride ($ZnCl_2$), sodium hydroxide (NaOH) and acetone were analytical grade. A mixed aqueous solution was prepared by dissolving the required weights of iron and zinc chloride with the molar ratio of Fe to Zn as 2:1, in distilled water (100 mL). An aqueous solution of 1.5 M NaOH (50 mL) was used as the precipitating agent. Metal chloride and NaOH solutions were added dropwise from two separate burettes into a reaction vessel containing 100 mL of distilled water for obtaining uniform particle size distribution. The reaction vessel was heated up to the desired temperature under magnetic stirring. The resultant precipitates were collected and centrifuged at 6000 rpm and then washed with distilled water and acetone for several times and finally dried in air.

*Synthesis of [(5-hydroxy-3-methyl-1*H*-pyrazol-4-yl)(phenyl)methyl] propanedinitrile derivatives (***5a**,**b***); general procedure*

Hydrazine monohydrate (1 mmol) and ethyl acetoacetate (1 mmol) were mixed, then, aromatic aldehydes (1 mmol), malononitrile (1 mmol) and $ZnFe_2O_4$ NPs (8 mol%) as catalyst were added and stirred at room temperature under solvent-free conditions for the specific time. After completion of the reaction, ethanol (5 mL) was added and magnet was introduced into the mixture in the form of a magnetic stirrer bar and catalyst was separated magnetically. The precipitate was filtered off and washed with a mixture of ethyl acetate/hexane (20:80).

*Synthesis of 6-amino-4-aryl-3-methyl-2,4-dihydro pyrano[2,3-*c*] pyrazole-5-carbonitrile derivatives (***6a–j***); general procedure*

Hydrazine monohydrate (1 mmol) and ethyl acetoacetate(1 mmol) were mixed and then the aromatic aldehyde (1 mmol), malononitrile (1 mmol) and $ZnFe_2O_4$ NPs (8 mol%) as the catalyst were added. The mixture was stirred at 80° C under solvent-free conditions for the specific time (Table 1). After completion of the reaction, ethanol (5 mL) was added. A magnet in the form of a magnetic stirrer bar was added and the catalyst was separated magnetically. The precipitated solid was filtered and washed with a mixture of ethyl acetate/hexane (20:80). The purity of obtained products was assessed by ^{1}H NMR spectroscopy.

*[(5-Hydroxy-3-methyl-1*H*-pyrazol-4-yl) (phenyl)methyl] propane-dinitrile* (**5a**): M.p. 254–256 °C, (lit.[18] 256-258); IR (KBr): ν_{max} 3383, 3300, 2207 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.09 (3H, s, CH_3), 4.65 (1H, d, *J*=11.2 Hz, CH), 5.52 (1H, d, *J*= 11.2 Hz, CH),7.20-7.55 (m, 5H, Ar), 10.5-11.5 (br s, 2H, NH, OH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 9.6, 18.4, 27.6, 99.8, 113.9 (2 C), 114.1, 128.8 (2 C), 131.6, 137.7, 158.5, 159.0. Anal. calcd for $C_{14}H_{12}N_4O$: C, 66.65; H, 4.79; N, 22.21; found: C 66.60; H, 4.70; N, 22.19%.

*[(5-Hydroxy-3-methyl-1*H*-pyrazol-4-yl) (4-methoxyphenyl)methyl] malononitrile* (**5b**): M.p. 207–209 °C, (lit.[18] 207–208); IR (KBr): ν_{max} 3481, 3354, 3255, 2950, 2192, 1641 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): δ 2.08 (s, 3H, CH_3), 3.74 (s, 3H, OCH_3), 4.59 (d, *J* = 11.4 Hz, 1H, CH), 5.45 (d, *J* = 11.4 Hz, 1H, CH), 6.93 (d, *J* = 8.5 Hz, 2H, Ar), 7.42 (d, *J* = 8.5 Hz, 2H, Ar), 10.86 (br s, 2H, NH and OH); ^{13}C NMR (100 MHz, DMSO-d_6): δ 9.7, 18.5, 27.7, 55.1, 98.9, 113.9 (2 C), 114.1, 128.9 (2 C), 131.8, 137.8, 158.6, 159.0; Anal calcd for $C_{15}H_{14}N_4O_2$: C, 63.82; H, 5.00; N, 19.85; found: C, 63.71; H, 5.09; N, 19.78%.

*6-Amino-3-methyl-4-phenyl-2,4-dihydro pyrano[2,3-*c*]-pyrazole-5-carbonitrile* (**6a**): M.p. 244–246°C, (lit.[8] 244–246); IR (KBr): ν_{max} 3371, 3248, 2192 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): 1.76 (s, CH_3, 3H), 4.57 (s, 1H), 6.89–7.30 (m, 7H), 12.09 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.17, 36.67, 57.64, 99.14, 121.24, 127.19, 127.91, 128.89, 136.03, 144.89, 155.21, 161.31 ppm. Anal. calcd for $C_{14}H_{12}N_4O$: C, 66.65; H, 4.79; N, 22.21; found: C, 66.60, H, 4.83, N, 22.26%.

*6-Amino-2,4-dihydro-3-methyl-4-*p*-tolylpyrano[2,3-*c*]pyrazole-5-carbonitrile* (**6b**): M.p. 208–210 °C, (lit.[8] 206–208); IR (KBr): ν_{max} 3405, 3315, 3190, 2191, 1644, 1601 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): 1.76 (s, CH_3, 3H), 2.25 (s, CH_3, 3H), 4.52 (s, 1H), 6.85-7.11 (m, 6H), 12.08(1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.25, 21.23, 36.69, 57.65, 99.14, 121.24, 127.19, 127.91, 128.89, 136.03, 144.89, 155.21, 161.31 ppm. Anal. calcd for $C_{15}H_{14}N_4O$: C, 67.65; H, 5.30; N, 21.04; found: C, 67.71; H, 5.21; N, 20.98%.

*6-Amino-2,4-dihydro-3-methyl-4-*o*-tolylpyrano[2,3-*c*]pyrazole-5-carbonitrile* (**6c**): M.p. 209-211 °C, IR (KBr): ν_{max} 3399, 3311, 3168, 2925, 2190, 1649, 1468 cm^{-1}; ^{1}H NMR (400 MHz, DMSO-d_6): 1.65 (s, CH_3, 3H), 2.24 (s, CH_3, 3H), 4.81 (s, 1H), 6.81–7.09 (m, 6H), 12.05 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.21, 21.22, 36.52, 57.65, 99.14, 121.24, 127.19, 127.31,127.95, 128.83, 129.22, 136.02, 144.85, 155.20, 161.33 ppm. Anal. calcd for $C_{15}H_{14}N_4O$: C, 67.65; H, 5.30; N, 21.04; found: C, 67.73; H, 5.23; N, 20.96%.

*6-Amino-4-(3-methylphenyl)-3-methyl-2,4-dihydropyrano[2,3-*c*] pyrazole-5-carbonitrile* (**6d**): M.p. 212–214 °C, IR (KBr): ν_{max} 3470, 3307, 3181, 2923, 2189, 1639 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.79 (s, CH_3, 3H), 2.25 (s, CH_3, 3H), 4.53 (s, 1H), 6.87-7.25 (m, 6H), 12.09 (1H, NH) ppm ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.25, 21.54, 36.61, 57.67, 98.15, 121.35, 125.16, 127.93, 128.36, 129.1, 136.06, 137.98, 144.99, 155.27, 161.33. Anal. calcd for $C_{15}H_{14}N_4O$: C, 67.65; H, 5.30; N, 21.04; found: C 67.72; H, 5.25; N, 20.98%.

*6-Amino-2,4-dihydro-4-(2-methoxyphenyl)-3-methylpyrano [2,3-*c*]pyrazole-5-carbonitrile* (**6e**): M.p. 215–217 °C, IR (KBr): ν_{max} 3376, 3163, 2928, 2193, 1654, 1604, 1488 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.77 (s, CH_3, 3H), 3.77 (s, OCH_3, 3H), 4.95 (s, 1H), 6.79–7.18 (m, 6H), 12.00 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.23, 36.55, 55.95, 57.65, 99.18, 121.28, 127.19, 127.36,127.97, 128.83, 129.25, 136.01, 144.82, 155.22, 161.34 ppm. Anal. calcd for $C_{15}H_{14}N_4O_2$: C, 63.82; H, 5.00; N, 19.85; found: C, 63.78; H, 5.09; N, 19.72%.

*6-Amino-2,4-dihydro-3-methyl-4-(4-nitrophenyl)pyrano[2,3-*c*] pyrazole-5-carbonitrile* (**6f**): M.p. 250–252 °C, (lit.[8] 251–253) IR (KBr): ν_{max} 3425, 3231, 2925, 2192, 1644, 1403 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.78 (s, CH_3, 3H), 4.96 (s, 1H), 7.07 (NH_2, 2H), 7.44–7.46 (d, *J*= 8 Hz, 2H), 8.25–8.27 (d, *J*= 8 Hz, 2H), 12.14 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.20, 36.36, 56.39, 97.01, 120.96, 124.35, 129.30, 136.37, 146.84, 152.56, 155.13, 161.61 ppm. Anal. calcd for $C_{14}H_{11}N_5O_3$: C, 56.56; H, 3.73; N, 23.56; found: C, 56.48; H, 3.65; N, 23.61%.

*6-Amino-4-(4-chlorophenyl)-3-methyl-2,4-dihydropyrano[2,3-*c*] pyrazole-5-carbonitrile* (**6g**): M.p. 228–230 °C, (lit.[8] 234–236); IR (KBr): ν_{max} 3408, 3369, 3307, 2188, 1643 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.77 (3H, s, CH_3), 4.62 (s, 1H), 6.94 (2H, s, NH_2), 7.17–7.19 (2H, d, *J*= 8Hz), 7.35–7.37 (2H, d, *J*= 8Hz) 12.09 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.2, 36.3, 56.7, 99.0, 120.6, 128.4, 129.3, 131.2, 135.6, 143.4, 154.7,160.9 ppm. Anal. calcd for $C_{14}H_{11}ClN_4O$: C, 58.65; H, 3.87; N, 19.54; found: C 58.60; H, 3.80; N, 19.48%.

*6-Amino-4-(2-fluorophenyl)-2,4-dihydro-3-methylpyrano[2,3-*c*]pyrazole-5-carbonitrile* (**6h**): M.p. 163–165°C, IR (KBr): ν_{max} 3366, 3312, 2186, 1641 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.78 (3H, s, CH_3), 4.84 (s, 1H), 6.96 (2H, s, NH_2), 7.14–7.16 (m, 3H), 7.26–7.28 (m, 1H), 12.13 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 9.6, 36.0, 56.5, 98.5, 120.6, 121.3, 121.4, 128.4, 129.3, 129.5, 131.3,135.6, 143.3, 154.8,161.1 ppm. Anal. calcd for $C_{14}H_{11}FN_4O$: C, 62.22; H, 4.10; N, 20.73; found: C, 62.15; H, 4.16; N, 20.67%.

*6-Amino-4-(4-bromophenyl)-2,4-dihydro-3-methylpyrano[2,3-*c*] pyrazole-5-carbonitrile* (**6i**): M.p. 179–181 °C, (lit.[8] 178–180); IR (KBr): ν_{max} 3470, 3234, 3117, 2192, 1645 cm^{-1}, ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.77 (3H, s, CH_3), 4.61 (s, 1H), 6.92 (2H, s, NH_2), 7.16–7.18 (2H, d, *J*= 8Hz), 7.34-7.36 (2H, d, *J*= 8Hz), 12.12 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.3, 36.6, 56.8, 99.1, 120.7, 128.5, 129.4, 131.3, 135.6, 143.6, 154.7,161.1 ppm. Anal. calcd for

$C_{14}H_{11}BrN_4O$: C, 50.77; H, 3.35; N, 16.92; found: C, 50.65; H, 3.26; N, 16.88%.

6-Amino-2,4-dihydro-4-(4-methoxyphenyl)-3-methylpyrano[2,3-c] pyrazole-5-carbonitrile (**6j**): M.p. 208–210 °C, (lit.[8] 210–212); IR (KBr): ν_{max} 3420, 3249, 2899, 2201, 1644 cm^{-1}, ^1H NMR (400 MHz, DMSO-d_6): δ 1.75 (3H, s, CH_3), 3.70 (3H, s, OCH_3), 4.51 (s, 1H), 6.83–7.06 (m, 6H), 12.07 (1H, NH) ppm; ^{13}C NMR (100 MHz, DMSO-d_6): δ 10.21, 36.52, 55.90, 57.62, 99.09, 121.28, 127.19, 127.97, 128.82, 136.01, 144.72, 155.16, 161.24 ppm. Anal. calcd for $C_{15}H_{14}N_4O_2$: C, 63.82; H, 5.00; N, 19.85; found: C, 63.91; H, 5.10; N, 19.74%.

Electronic Supplementary Information

The spectral data of products are described in the ESI available through: stl.publisher.ingentaconnect.com/content/stl/jcr/supp−data.

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