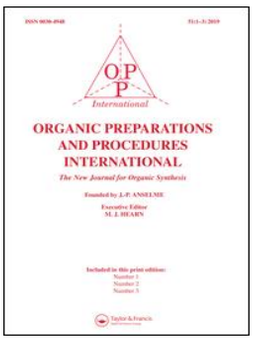

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Nanostructural Cu-Doped ZnO Hollow Spheres as an Economical and Recyclable Catalyst in the Synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones and Pyrazolo[1,2-*a*][1,2,4]triazole-1,3-diones

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EXPERIMENTAL PAPER

Nanostructural Cu-Doped ZnO Hollow Spheres as an Economical and Recyclable Catalyst in the Synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones and Pyrazolo[1,2-*a*][1,2,4]triazole-1,3-diones

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In the synthesis of organic compounds, heterogeneous catalysts have gained significance versus their homogeneous counterparts, due to both environmental and economic concerns.[1–4] Homogeneous catalytic systems cannot as readily be commercialized owing to difficulties encountered with equipment corrosion, solvent recycling, isolation, and reusability of the catalyst. In recent years, metallic nanoparticles have found use as heterogeneous catalysts.[5–9] Nanometal oxides possess a surface Lewis acid (metal cation) and Lewis base (oxygen anion),[10] and they enhance organic reactions because they act as excellent adsorbents for a wide variety of organic molecules. The high surface area to volume ratio of the metal oxide nanoparticles is in large part responsible for their catalytic activities.[11] In connection with this, zinc oxide is of low cost and has been widely used in industry.[12–15] In recent years, ZnO nanostructures, including nanotowers, nanovolcanoes, nanorods, nanotubes, nanowires, nanoflowers, and nanospheres have been reported. Among the ZnO nanostructures, zinc oxide hollow structures have attracted considerable attention.[16–18]

The use of multi-component reactions (MCRs)[19–26] is becoming more prominent in organic synthesis and represents an attractive possibility for the preparation of biologically active pyrazoles.[27–32] For example, extensive studies have been accomplished on the synthesis of pyrazolo[1,2-*b*]phthalazine-diones and pyrazolo[1,2-*a*][1,2,4]triazole-1,3-diones. These compounds are known as antimicrobial, antitumor, anti-inflammatory, analgesic, antihypotoxic, anticonvulsant, antipyretic, and antifungal agents.[30–32]

We now report the use of Cu-ZnO hollow sphere nanostructures as a catalyst for the MCR preparation of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones and pyrazolo[1,2-*a*][1,2,4]triazole-1,3-diones, formed from phthalhydrazide or phenylurazole, malononitrile and aldehydes (*Scheme 1*). The catalyst was rigorously characterized using the techniques of x-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FT-IR),

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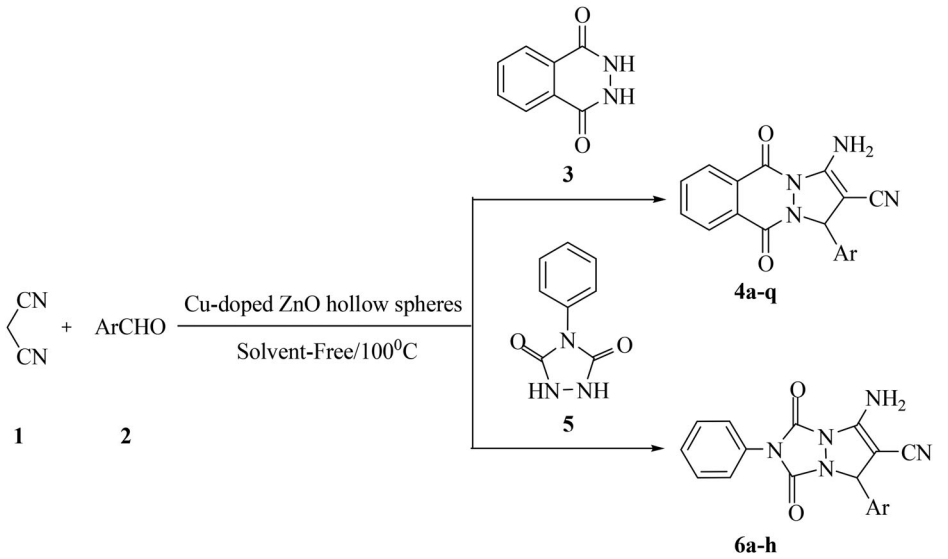


Scheme 1. Synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones and pyrazolo[1,2-*a*][1,2,4]triazole-1,3-diones.

field emission scanning electron microscopy (FE-SEM), and energy dispersive x-ray (EDX) analysis.

In order to establish the conditions for the title reactions, we first examined the model condensation reaction among malononitrile **1** (1 mmol), benzaldehyde **2a** (1 mmol) and phthalhydrazide **3** (1 mmol). The effects of solvents, and conditions on the reaction were studied (Table 1). The reaction worked best at 100 °C under solvent-free conditions using 0.020 g Cu-ZnO hollow spheres as catalyst.

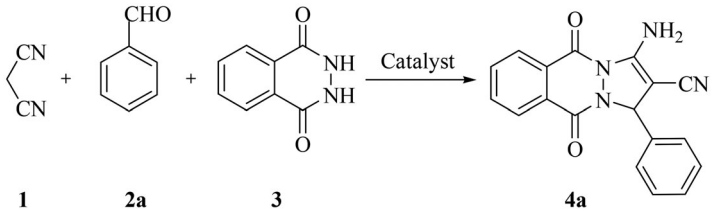
To develop the scope of these reactions, several other aromatic aldehydes **2** were subjected to condensation with malononitrile **1** under the optimized conditions. In general, the reactions proceeded smoothly in relatively short reaction times (8-35 min) to afford the respective 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones **4a-q** in good yields (81-93%). The experimental results are summarized in *Table 2*.

In a similar manner, we optimized the preparation of pyrazolo[1,2-*a*][1,2,4]triazole-1,3-diones and found that the best conditions were 80 °C, solvent-free, 0.020 g Cu-ZnO hollow sphere nanostructures. These results are summarized in *Table 3*.

The reusability of the Cu-ZnO hollow spheres nanostructures was examined in the model reaction among malononitrile **1** (1 mmol), benzaldehyde (1 mmol) and phthalhydrazide **3** (1 mmol). The catalyst was readily separated from the reaction mixture after completion of the reaction, washed with hot ethyl acetate, and used directly for four successive runs in yields of 91%, 88%, 86% and 83% respectively, demonstrating that the catalyst activity declined only slightly. The ease of separation and reusability of the catalyst are two of the highly desirable features of this preparation.

In conclusion, we have developed a convenient one-pot three component cyclocondensation reaction for the preparation of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones and pyrazolo[1,2-*a*][1,2,4]triazole-1,3-dione derivatives, using nano hollow sphere Cu-ZnO

Table 1. Optimization of reaction conditions.



1 **2a** **3** **4a**

Entry	Conditions	Solvent	Catalyst (g)	Time (min)	Yield (%)[b]
1	rt	EtOH	Cu-ZnO hollow spheres (0.030)	100	Trace
2	100 °C	Solvent-free	None	100	–
3	100 °C	Solvent-free	Cu-ZnO hollow spheres (0.005)	20	58
4	100 °C	Solvent-free	Cu-ZnO hollow spheres (0.010)	20	80
5	100 °C	Solvent-free	Cu-ZnO hollow spheres (0.020)	20	91
6	100 °C	Solvent-free	Cu-ZnO hollow spheres (0.030)	20	91
7	100 °C	H_2O/Reflux	Cu-ZnO hollow spheres (0.020)	60	51
8	80 °C	EtOH/Reflux	Cu-ZnO hollow spheres (0.020)	60	68
9	80 °C	H_2O: EtOH (1:1)/Reflux	Cu-ZnO hollow spheres (0.020)	60	62

[a]Conditions: malononitrile (1 mmol), phthalhydrazide (1 mmol), benzaldehyde (1 mmol).
[b]Isolated yield.

as an economical catalyst under solvent-free conditions. This method has such advantages as high yields, short reaction times, easy work-up, ease of separation and reusability of the catalyst. We hope that our work will stimulate further research on this useful catalyst for the preparation of heterocyclic systems.

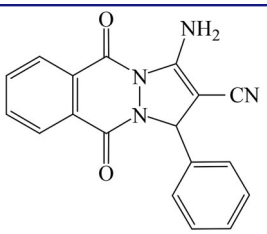
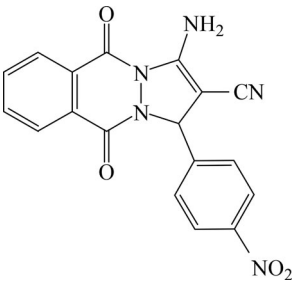
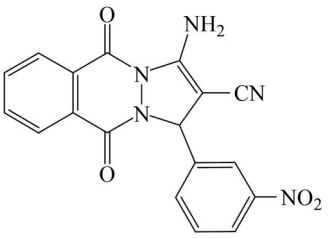
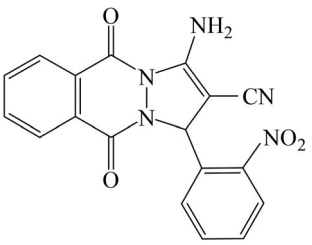
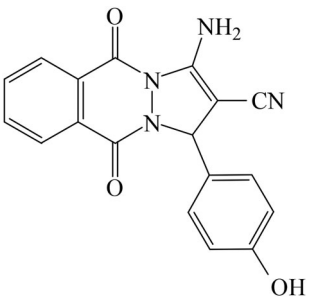
Experimental section

Melting points were measured on an SMPI apparatus. All reagents for the synthesis and analysis were commercially available from Merck Company and used as received. The infrared spectra were recorded on a Nicolet Fourier Transform IR spectrometer in the range 400-4000 cm^{-1} using the KBr disk technique. ^1H NMR spectra were obtained using a Bruker 300 MHz spectrometer in DMSO-d_6 or $CDCl_3$ and using TMS as the internal reference. Powder X-ray diffraction (PXRD) measurements were performed using a Philips X'pert diffractometer with monochromated Cu-Kα radiation (λ= 1.54056 Å). Also, the morphology of samples was characterized by a field emission scanning electron microscope (FE-SEM) (Mira TESCAN) with gold coating. Powder XRD patterns were recorded at room temperature using a Philips X'pert 1710 diffractometer with Co Kα radiation (λ = 1.78897 Å). The corresponding EDX spectrum was obtained with a Holland Philips XL30 microscope.

Carbon spheres

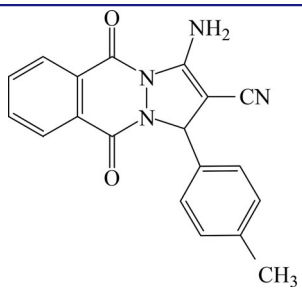
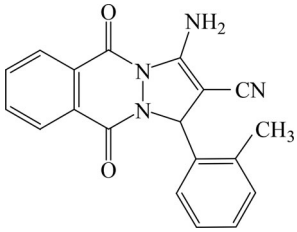
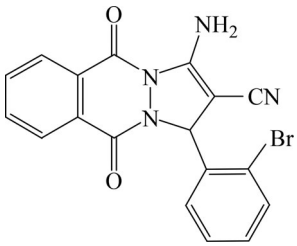
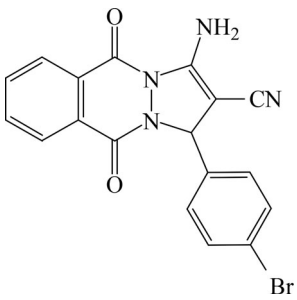
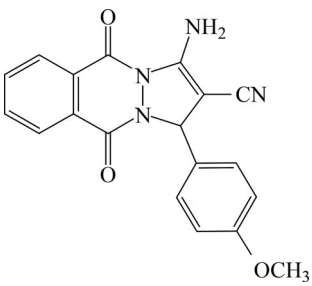
The preparation route used for carbon spheres is as follows:[29] Initially, fructose (10 g) was dissolved in 100 ml of deionized water to obtain a transparent solution. Then, the formed homogeneous solution was sealed in a Teflon-lined stainless steel autoclave. The

Table 2. One-pot synthesis of 1*H*-pyrazolo[1,2-*b*]phthalazine-5,10-diones (**4a-q**) catalyzed by Cu-ZnO hollow spherical nanostructures.

				Mp (°C)	
Entry	Products	Time (min)	Yield (%)	Found	Reported
4a		20	91	275-276	276-278[34]
4b		8	93	227-229	228-229[35]
4c		10	93	268-270	269-271[36]
4d		17	91	265-266	262-264[37]
4e		20	89	232-233	270-272[38]

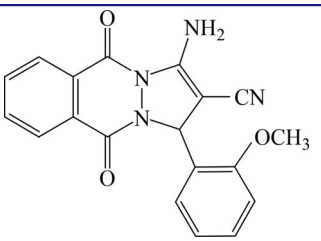
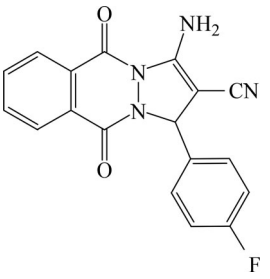
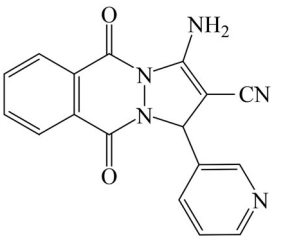
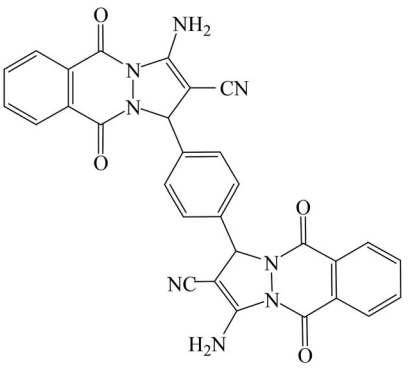
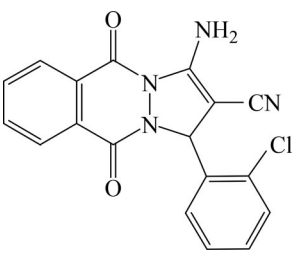
(*continued*)

Table 2. Continued.

Entry	Products	Time (min)	Yield (%)	Mp (°C) Found	Mp (°C) Reported
4f		20	85	250-252	253-255[34]
4g		25	81	247-251	248-250[36]
4h		10	87	248-250	248-250[18]
4i		10	90	263-264	267-268[38]
4j		25	85	190-193	192-194[34]

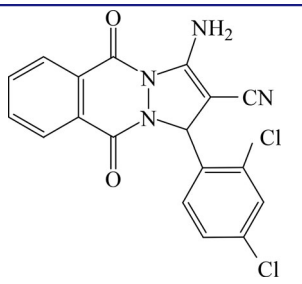
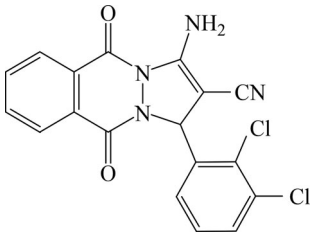
(*continued*)

Table 2. Continued.

Entry	Products	Time (min)	Yield (%)	Mp (°C) Found	Mp (°C) Reported
4k		30	81	154-156	155-157[38]
4l		10	87	263-264	265-267[18]
4m		12	90	268-271	267-270[18]
4n		35	87	269-270	271-273[18]
4o		15	88	269-272	268-270[34]

(*continued*)

Table 2. Continued.

Entry	Products	Time (min)	Yield (%)	Mp (°C) Found	Mp (°C) Reported
4p		12	92	240-243	242-244[39]
4q		14	91	281-283	282-284[39]

autoclave was heated to 160 °C for 4 h. The hot solution was then gradually cooled to room temperature, and the resultant carbon spheres were separated from the suspension and subsequently washed three times with deionized water. Finally, the spheres were dried at 80 °C for 2 h in a way that excessive water was evaporated.

Cu-doped ZnO hollow spheres

In order to prepare x-Cu-ZnO (x = 10 wt % Cu) hollow spheres, the stoichiometric quantities of zinc(II) acetate dihydrate and cupper(II) acetate dihydrate of analytical reagent grade were dissolved in 40 mL deionized double distilled water (solution A). Separately, a solution was prepared by dispersing carbon microspheres (1 g) in 20 ml of deionized double distilled water (solution B). Solution B was dispersed in the ultrasonic bath at room temperature for 1 h and solution A was mixed dropwise into this solution with constant stirring. The mixed solution was then aged for one day under ambient conditions. Subsequently, the product was concentrated by centrifugation and washed three times with 20 mL deionized water in order to remove impurities. Finally, the product was annealed from room temperature to 500 °C at 1 °C min^{-1} rate and placed at 500 °C for 2 hours in air to obtain the Cu-ZnO hollow spherical nanostructures with a dark gray color. The catalyst was rigorously characterized using the techniques of x-ray diffraction, infrared spectroscopy, field emission scanning electron microscopy, and energy dispersive x-ray analysis. The data were submitted for review and are available from the corresponding author upon request.

Synthesis of 1H-pyrazolo[1,2-b]phthalazine-5,10-diones and pyrazolo[1,2-a][1,2,4]triazole-1,3-diones

A mixture of aldehyde **2** (1.0 mmol), malononitrile **1** (1.0 mmol), phthalhydrazide **3** (1.0 mmol) or phenylurazole **5** (1.0 mmol) and Cu-ZnO hollow spheres (0.02 g) were placed in a test tube. The reaction mixture was then heated at 100 °C for an appropriate time (Tables 2 and 3) until the completion of the reaction was achieved as monitored by TLC. Then, the crude product was dissolved in hot ethyl acetate and then filtered to remove the solid catalyst. The filtrate was cooled to give the pure organic product, identified by comparison of the observed melting point with the literature value. Representative spectroscopic data for selected examples are shown below, for the sake of completeness.

3-Amino-1-(2,4-dichlorophenyl)-5,10-dihydro-5,10-dioxo1H-pyrazolo[1,2-b]phthalazine-2-carbonitrile (4q)

^{1}H-NMR (300 MHz, DMSO-d_6): δ 6.43 (s, 1H, CH), 7.39–8.25 (m, 9H, Ar–H and NH_2) ppm; ^{13}C-NMR (75 MHz, DMSO-d_6): δ 61.2, 62.5, 116.5, 127.1, 127.9, 128.6, 128.9, 129.2, 129.8, 130.3, 130.5, 131.5, 134.4, 138.2, 138.9, 151.6, 154.1, 157.0; IR (KBr, cm^{-1}): 3364, 3262, 2197, 1681, 1660.

7-Amino-1,3-dioxo-2,5-phenyl-1,2,3-trihydropyrazolo[1,2-a][1,2,4]triazole-6-carbonitrile (6a)

^{1}H NMR (500 MHz, DMSO-d_6): δ 5.95 (1H, s, CH), 7.08–8.12 (12H, m, H–Ar and NH_2). ^{13}C NMR (125 MHz, DMSO-d_6): δ 61.2, 63.3, 116.2, 126.1, 126.9, 127.7, 128.3, 128.7, 129.8, 133.4, 145.1, 150.1, 150.5, 153.3; IR (KBr, cm^{-1}): 3365, 3301, 2191, 1651, 1578.

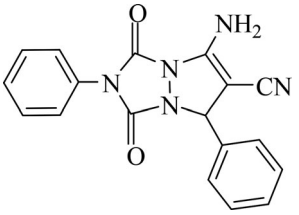
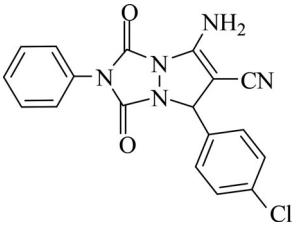
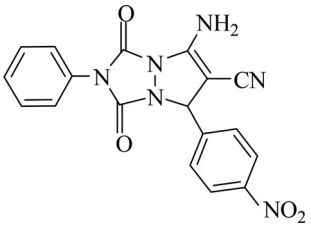
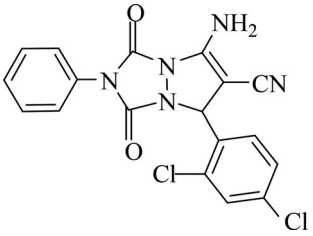
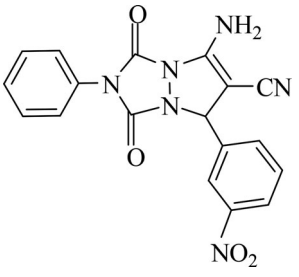
7-Amino-5-(4-chlorophenyl)-1,3-dioxo-2-phenyl-1,2,3-trihydropyrazolo[1,2-a][1,2,4]triazole-6- carbonitrile (6b)

^{1}H NMR (500 MHz, DMSO-d_6): δ 5.89 (1H, s, CH), 7.42–7.67 (11H, m, H–Ar and NH_2). ^{13}C NMR (125 MHz, DMSO-d_6): δ 61.9, 64.5, 116.8, 127.5, 128.9, 129.2, 129.5, 130.1, 134.6, 137.9, 145.7, 150.4, 150.6, 153.8; IR (KBr, cm^{-1}): 3364, 3260, 2187, 1654, 1567.

7-Amino-1,3-dioxo-5-(4-nitrophenyl)-2-phenyl-1,2,3-trihydropyrazolo[1,2-a][1,2,4]triazole-6-carbonitrile (6c)

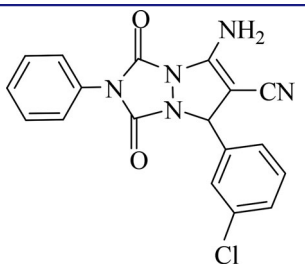
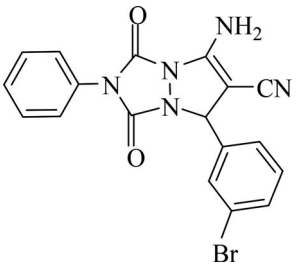
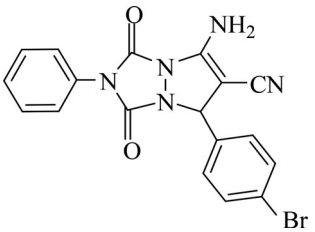
^{1}H NMR (500 MHz, DMSO-d_6): δ 5.97 (1H, s, CH), 7.44–8.32 (11H, m, H–Ar and NH2). ^{13}C NMR (125 MHz, DMSO-d_6): δ 61.0, 63.2, 116.37, 124.2, 126.9, 128.4, 128.9, 129.1, 129.6, 131.0, 146.2, 150.37, 150.6, 153.7; IR (KBr, cm^{-1}): 3363, 3255, 2289, 1648, 1565.

Table 3. One-pot synthesis of pyrazolo[1,2-*a*][1,2,4]triazole-1,3-diones (**6a-h**) catalyzed by Cu-ZnO hollow spheres nanostructures.

Entry	Products	Time (min)	Yield (%)	Mp (°C) Found	Mp (°C) Reported
6a		35	89	210	210[40]
6b		15	90	221	221[40]
6c		20	92	217	218[40]
6d		20	91	200	200[40]
6e		18	90	214	214[40]
6f		25	88	217	216[40]

(*continued*)

Table 3. Continued.

Entry	Products	Time (min)	Yield (%)	Mp (°C) Found	Mp (°C) Reported
					
6g		30	89	220	221[40]
6h		20	91	224	224[40]

7-Amino-5-(2,4-dichlorophenyl)-1,3-dioxo-2-phenyl-1,2,3-dihydropyrazolo[1,2-a][1,2,4]triazole-6-carbonitrile (6d)

^{1}H NMR (500 MHz, DMSO-d_6): δ 6.22 (1H, s, CH), 7.43–7.70 (10H, m, H–Ar and NH_2). ^{13}C NMR (125 MHz, DMSO-d_6): δ 60.5, 61.1, 116.0, 126.7, 128.7, 129.0, 129.2, 129.9, 130.9, 131.0, 132.7, 134.0, 135.1, 150.2, 150.5, 153.5; IR (KBr, cm^{-1}): 3363, 3262, 2191, 1647, 1565.

7-Amino-1,3-dioxo-5-(3-nitrophenyl)-2-phenyl-1,2,3-trihydropyrazolo[1,2-a][1,2,4]triazole-6-carbonitrile (6e)

^{1}H NMR (300 MHz, DMSO- d_6): δ 6.08 (1H, s, CH), 7.43–8.35 (11H, m, H–Ar and NH_2); ^{13}C NMR (75 MHz, DMSO-d_6): δ 60.8, 63.0, 116.3, 121.5, 123.6, 126.8, 128.7, 129.0, 129.5, 130.9, 133.6, 141.5, 148.1, 150.3, 150.5, 153.7; IR (KBr, cm^{-1}): 3369, 3252, 2103, 1629, 1556.

7-Amino-5-(3-chlorophenyl)-1,3-dioxo-2-phenyl-1,2,3-trihydropyrazolo[1,2-a][1,2,4]triazole-6- carbonitrile (6f)

^{1}H NMR (500 MHz, DMSO-d_6): δ 6.17 (1H, s, CH), 7.23–7.80 (11H, m, H–Ar and NH_2). ^{13}C NMR (125 MHz, DMSO-d_6): δ 61.2, 63.2, 116.3, 121.1, 126.8, 127.2, 128.1,

128.8, 129.8, 130.7, 135.2, 135.9, 145.6, 150.3, 150.6, 153.6; IR (KBr, cm^{-1}): 3370, 3233, 2209, 1655, 1563.

7-Amino-5-(3-bromophenyl)-1,3-dioxo-2-phenyl-1,2,3-trihydropyrazolo[1,2-a][1,2,4]triazole-6-carbonitrile (6g)

^{1}H NMR (500 MHz, DMSO-d_6): δ 5.88 (1H, s, CH), 7.40–7.68 (11H, m, H–Ar and NH_2). ^{13}C NMR (125 MHz, DMSO-d_6): δ 61.1, 63.1, 116.4, 121.9, 126.0, 128.6, 128.9, 129.6, 130.9, 131.0, 131.4, 141.8, 145.4, 150.1, 150.4, 153.5; IR (KBr, cm^{-1}): 3450, 3273, 2203, 1661, 1526.

7-Amino-5-(4-bromophenyl)-1,3-dioxo-2-phenyl-1,2,3-trihydropyrazolo[1,2-a][1,2,4]triazole-6-carbonitrile (6h)

^{1}H NMR (500 MHz, DMSO-d_6): δ 5.77 (1H, s, CH), 7.00–7.60 (11H, m, H–Ar and NH_2). ^{13}C NMR (125 MHz, DMSO-d_6): δ 60.8, 61.3, 116.4, 126.5, 127.8, 128.7, 129.0, 129.9, 130.2, 131.1, 133.3, 137.7, 144.5, 150.1, 153.3; IR (KBr, cm^{-1}): 3372, 3276, 2212, 1673, 1603.

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