An Efficient One-Pot Synthesis of Pyridazinones and Phthalazinones Using HY-Zeolite

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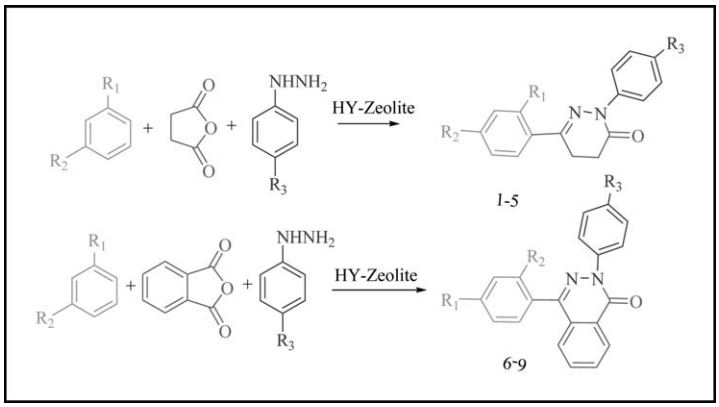

The first one-pot synthesis of pyridazinones and phthalazinones from arenes, cyclic anhydrides, and $ArNHNH_2$ in the presence of efficient recyclable heterogeneous catalyst, HY-zeolite, in high yield and short reaction time is reported.

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INTRODUCTION

Pyridazinones are useful compounds with a broad array of biologically activities. They possessed notable hypertensive [1], platelet aggregation inhibiter [2], phosphodiesterase [3], antiasthmatic [4], antisecretory and antiulcer [5], antidepressant [6], antibacterial [7], antifungal [8], α-adrenoceptor antagonists [9], analgesic [10], anti-inflammatory [11], antianemic [12], nephrotropic [13], cardiotonic [14], anticancer [15], and pesticidal and herbicidal [16] properties.

Previously reported synthetic routes to synthesis of pyridazinones includes reaction of γ-ketoacids and their derivatives with alkylhydrazines or phenylhydrazines [17], condensation of Wittig reagents with arylhydrazones or condensation of α-ketoesters with hydrazinocarbonyl-acetic acid esters [18], catalytic reactions of alkynes with arylhydrazines (hydrohydrazination) [19], condensation of hydrazine with appropriate substituted lactones [20] is reported.

Zeolites are used as catalysts for wide range of processes, from simple drying to complicated catalytic reactions. Acidic zeolite such as HY-zeolite is unique acid catalyst that has become popular over the last two decades. HY-Zeolite contains a framework system of supercages, which are connected by a three-dimensional array of large diameter channels and this array enables a much easier diffusion of reactants and products [21]. This heterogeneous catalyst has been used in various chemical transformations [22].

RESULTS AND DISCUSSION

The remarkable catalytic activity together with easy availability, operational simplicity of HY-zeolite and our continued interests for the development of efficient and environmentally friendly procedures for the synthesis of heterocyclic compounds [23] triggered us to synthesis of pyridazinones and phthalazinones in a ''one-pot'' reaction in the presence of HY-zeolite. This class of reactions is of particular interest in combinatorial chemistry because it allows the production of vast arrays of molecules in an efficient mode.

To optimize the catalyst loading, 0.01, 0.02, 0.03, and 0.05 g of HY-Zeolite per 1 mmol of Arenes each was tested, respectively. The results are summarized in Table 1. A 0.02 g/mmol loading of HY-Zeolite was sufficient to push the reaction forward and 0.01 g/mmol of HY-Zeolite was not enough. Higher amounts of HY-Zeolite did not lead to significant change in the reaction yields (Scheme 1).

To find the optimal solvent for this reaction, the synthesis of 4a was carried out at refluxing temperature

Table 1

The effect about amounts of HY-zeolite when synthesizing 1 in dicholoroethane.

Entry	HY-Zeolite (g/mmol of substrate)	Reaction time (h)	Isolated yield (%)
1	0.01	10	72
2	0.02	8	80
3	0.03	8	76
4	0.05	8	75

Table 2

Solvent optimization for 0.02 g of HY-zeolite per 1 mmol of benzene synthesis of 1 at refluxing temperature.

Entry	Solvent	Reaction time (h)	Isolated yield (%)
1	THF	9	70
2	$CHCl_3$	8.5	73
3	$ClCH_2CH_2Cl$	8	80
4	Nitrobenzene	8	71
5	Solvent free	10	75

using THF, $CHCl_3$, $ClCH_2CH_2Cl$, nitrobenzene, and solvent-free condition, as solvents, respectively. It is shown in Table 2 that the reactions with dicholoroethane as solvent resulted in shorter reaction time and higher yield than other solvents. So $ClCH_2CH_2Cl$ was chosen as the solvent of this reaction.

Under these optimized reaction conditions (10 mL $ClCH_2CH_2Cl$, at refluxing temperature, 0.02 g of HY-Zeolite per 1mmol of substrate), a series of pyridazinones and phthalazinones derivatives were synthesized. The results are summarized in Table 3.

Apart from the mild conditions of the process and its excellent results, the simplicity of product isolation and the possibility to recycle the catalyst HY-Zeolite offer a significant advantage. Because products are soluble with $ClCH_2CH_2Cl$ and HY-Zeolite can be directly separated by filtration. The HY-Zeolite can be recycled. Studies using **1** as model substrates showed that the recovered catalyst could be successively recycled in subsequent reactions without any decrease of yields (Table 4). It is shown that HY-Zeolite has been recycled sixth rounds, catalytic efficiency of HY-Zeolite not be decreased even in the seventh round.

The mechanism of reaction goes through Friedel-Crafts acylation between arenes and anhydride to prepare keto-carboxylic acids. Arenes bearing electron-releasing substituent such as toluene, anisole and *m*-xylene decrease the rate of this reaction and improve the reaction yield. Intermolecular hydrazone formation followed by intramolecular cyclization over an efficient acidic catalyst led to the formation of pyridazinones **1-5** and phthalazinones **6-9**. For these reasons, we used HY-zeolite because of its environmental compatibility, reusability, operational simplicity, no toxicity, no corrosiveness, low cost, and ease of isolation (Scheme 2).

This one-pot reaction is applicable to some of arenes corresponding pyridazinones and phthalazinones in varying yields (Table 3). The structures of the synthesized compounds were elucidated by IR, NMR, and elemental analysis results.

In conclusion, a convenient one-pot zeolite-catalyzed synthesis of pyridazinones and phthalazinones from arenes, anhydrides, and $ArNHNH_2$ in high yield and short reaction time was developed. To the best of our knowledge, this is the first report on the one-pot synthesis of pyridazinones and phthalazinones. This one-pot reaction has several advantages such as virtue of their convergence, productivity, ease of execution, and work-up, the small amount of waste, short reaction time, reusability of catalyst coupled with replacement of carcinogenic solvents, such as benzene and nitrobenzene with dichloroethane.

Scheme 1

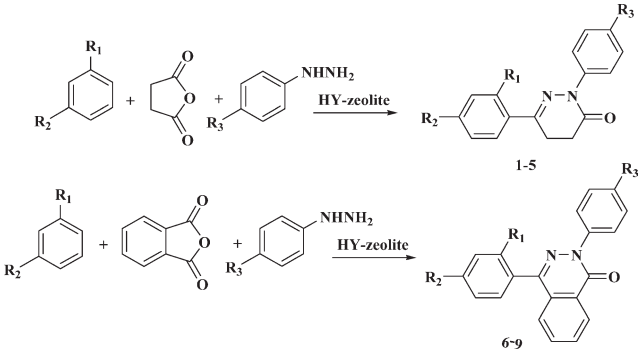


Table 3

Synthesis of pyridazinones and phthalazinones by the one-pot reaction

Entry	R_1	R_2	R_3	Yield[a] (%)	Total time (h)	m.p. (°C)[b]
1	H	H	H	80	8	92–94
2	H	CH_3	H	82	7	oil
3	CH_3	CH_3	H	75	7.5	oil
4	H	Cl	H	70	8	oil
5	H	OCH_3	OCH_3	80	7	oil
6	H	H	H	75	8	161–163
7	H	CH_3	H	77	7	124–126
8	CH_3	CH_3	H	75	7	156–158
9	H	Cl	H	70	8	164–165

[a] Isolated yield.
[b] Identified by spectroscopic analysis (IR, ^{1}H NMR, ^{13}C NMR, and elemental analysis).

Table 4

Studies on the reuse of HY-zeolite in the preparation of 1.

Round	1	2	3	4	5	6	7
Yield	80	78	76	77	75	73	75

EXPERIMENTAL

Chemicals were purchased from Merck and Fluka in Kimiaexir. Co., a branch of Merck, Fluka and Aldrich in Iran, Tehran. HY-zeolite was purchased from Aldrich. Melting points were measured on an Electro-thermal 9100 apparatus and are uncorrected. ^{1}H NMR spectra were obtained on a Bruker DRX 500, 250, and those of ^{13}C NMR spectra on a Bruker DRX 125 Avance spectrometer in $CDCl_3$ as solvent and with TMS as internal standard. FT-IR spectra were recorded on a Shimadzu FT-IR-8400S spectrometer. Elemental analyses were recorded on a Carlo-Erba EA1110CNNO-S analyzer.

General procedure for the synthesis of pyridazinones and phthalazinones. The general procedure for the synthesis of pyridazinones **1-5** and phthalazinones **6-9** is follows: A mixture of anhydride and arenes (10 mmol) each and 0.2 g HY-zeolite in dichloroethane (20 mL) was refluxed for the required reaction time. After completion of the reaction, $ArNHNH_2$ (10 mmol) was added to the mixture and the mixture was refluxed for 7–8 h. The progress of the reaction was monitored by TLC (EtOAc: petroleum ether 1:4). Subsequently, the catalyst was removed by filtration and the product **1-9** was purified by column chromatography (EtOAc: petroleum ether 1:4) to furnish the desired pyridazinones and phthalazinones. The pure products were collected in 70–82% yields.

2, 6-Diphenyl-4, 5-dihydropyridazin-3(2H)-one, (1). M.p. 92–94°C; as an off white solid; IR (KBr): 1680 (C=O), 1600, 1541 (C=C), 1330, 1490 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ_H 2.84 (t, 2H, J = 8.4 Hz), 3.15 (t, 2H, J = 8.5 Hz), 7.30–7.33 (m, 1H), 7.45–7.48 (m, 5H), 7.64–7.66 (d, 2H, J = 7.5 Hz), 7.85–7.86 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 23.33, 28.47, 125.31, 126.51, 127.00, 129.07, 130.42, 135.91, 141.68, 151.93, 165.69; Anal. Calcd for $C_{16}H_{14}N_2O$: C, 76.78; H, 5.64; N, 11.19. Found: C, 76.62; H, 5.77; N, 11.10.

6-(4-Methylphenyl)-4,5-dihydropyridazin-3(2H)-one, (2). As a Red oil; IR (KBr): 1680 (C=O),1595, 1492 (C=C), 1326 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ_H 2.4 (s, 3H), 2.78 (t, 2H, J = 8.5 Hz), 3.06 (t, 2H, J = 8.5 Hz), 7.27–7.30 (d, 2H, J = 8.1 Hz), 7.32–7.35 (t, 1H, J = 7.4 Hz), 7.47–7.50 (t, 2H, J = 7.6 Hz), 7.67–7.69 (d, 2H, J = 8.3 Hz), 7.75–7.77 (d, 2H, J = 8.3 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 21.86, 23.21, 125.33, 126.53, 126.96, 128.48, 128.97, 129.01, 129.82, 133.12, 140.71, 152.16, 165.87; Anal. Calcd for $C_{17}H_{16}N_2O$: C, 77.25; H, 6.10; N, 10.60. Found: C, 77.26; H, 6.33; N, 10.58.

6-(2, 4-Dimethylphenyl)-2-phenyl-4, 5- dihydropyridazin-3(2H)-one, (3). As a Red oil; IR (KBr): 1677 (C=O), 1600, 1494 (C=C), 1326 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ_H 2.4 (s, 3H), 2.5 (s, 3H), 2.8 (t, 2H, J = 8.4 Hz), 3.0 (t, 2H J = 8.5 Hz), 7.10 (s, 1H), 7.12 (s, 1H), 7.28–7.33 (m, 2H), 7.43–7.46 (t, 2H, J = 8.2 Hz), 7.61–7.62 (d, 2H, J = 7.6 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 21.60, 21.61, 26.95, 28.74, 125.30, 126.93, 127.12, 128.93, 132.51, 134.02, 136.22, 139.66, 141.49, 155.60, 165.75; Anal. Calcd for $C_{18}H_{18}N_2O$: C, 77.67; H, 6.52. N, 10.06; Found: C, 77.56; H, 6.23; N, 10.18.

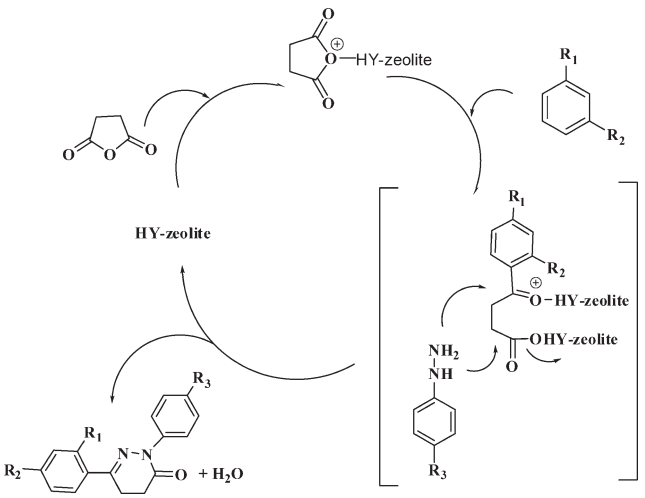
6-(4-Chlorophenyl)-2-phenyl-4, 5-dihydropyridazin-3(2H)-one, (4). As Red oil; IR (KBr): 1683 (C=O), 1595, 1490 (C=C), 1326, 1012 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ_H 2.8 (t, 2H, J = 8.5 Hz), 3.06–3.10 (t, 2H, J = 8.5 Hz), 7.32–7.34 (t, 1H, J = 7.3 Hz), 7.41–7.42 (d, 2H, J = 8.6 Hz), 7.46–7.49 (t, 2H, J = 8.2 Hz), 7.62–7.63 (d, 2H, J = 7.4 Hz), 7.76–7.79 (d, 2H, J = 8.6 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 23.17, 28.31, 125.26, 125.39, 127.14, 127.91, 129.00, 129.29, 136.44, 141.59, 150.68, 165.53; Anal. Calcd for $C_{16}H_{13}ClN_2O$: C, 67.49; H, 4.60; N, 9.84. Found: C, 67.21; H, 4.58; N, 9.81.

2, 6-Bis (4-methoxyphenyl)-4, 5-dihydropyridazin-3(2H)-one, (5). As Red oil; IR (KBr): 1683 (C=O), 1595, 1490 (C=C), 1326 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ_H 2.72–2.75 (t, 2H, J = 8.5 Hz), 3.01–3.04 (t, 2H, J = 7.6 Hz), 3.81 (s, 3H), 3.83 (s, 3H), 6.90–6.94 (m, 4H), 7.46–7.48 (d, 2H, J = 8.9 Hz), 7.72–7.74 (d, 2H, J = 8.9 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 23.23, 28.37, 55.81, 55.92, 114.24, 126.83, 128.07, 128.42, 134.95, 151.61, 158.50, 161.53, 165.86; Anal. Calcd for $C_{18}H_{18}N_2O_3$: C, 69.66; H, 5.85; N, 9.03. Found: C, 69.51; H, 5.69; N, 9.45.

2, 4-Diphenylphthalazin-1(2H)-one, (6). M.p. 161–163°C; as a Light brown solid; IR (KBr): 1656 (C=O), 1580, 1487 (C=C), 1325 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ_H 7.40–7.43 (t, 1H, J = 7.3 Hz),7.51–7.57 (m, 5H), 7.69–7.70 (d, 2H, J = 5.5 Hz), 7.78–7.79 (d, 2H, J = 7.6 Hz), 7.84–7.87 (m, 3H), 8.66–8.67 (d, 1H, J = 7.4 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 126.18, 127.18, 128.08, 128.20, 129.11, 129.40, 129.87, 130.06, 131.98, 132.18, 133.59, 135.47, 142.43, 148.05, 159.37; Anal. Calcd for $C_{20}H_{14}N_2O$: C, 80.52; H, 4.73; N, 9.39. Found: C, 80.25; H, 4.51; N, 9.27.

2-Phenyl-4-o-tolyl-2H phthalazin-1-one, (7). M.p. 124–126°C; as a Brown solid; IR (KBr): 1662 (C=O), 1595, 1490 (C=C), 1330 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz): δ_H 2.5 (s, 3H), 7.37–7.42 (m, 3H), 7.51–7.54 (t, 2H, J = 8.0 Hz), 7.58–7.60 (d, 2H, J = 7.9 Hz), 7.79–7.80 (d, 2H, J = 7.7 Hz), 7.82–7.86 (m, 3H), 8.65–8.67 (d, 1H, J = 7.6 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 21.80, 126.26, 127.35, 128.00, 128.15, 129.11, 129.41, 129.65, 129.74, 129.88, 131.99, 132.62, 133.51, 139.67, 142.50, 148.07, 159.37; Anal. Calcd for

Scheme 2



$C_{21}H_{16}N_2O$: C, 80.75; H, 5.16; N, 8.97. Found: C, 80.35; H, 5.87; N, 8.43.

4-(2, 4-Dimethylphenyl)-2-phenyl-2H phthalazin-1-one, (8). M.p.156–158°C, as a Brown solid; IR (KBr): 1662, 1583, 1492, 1330 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz): δ_H 2.25 (s, 3H), 2.45 (s, 3H), 7.17–7.19 (d, 1H, $J = 7.6$ Hz), 7.21(s, 1H), 7.30–7.31 (d, 1H, $J = 7.5$ Hz), 7.38–7.43 (m, 2H), 7.50–7.53 (t, 2H, $J = 7.7$ Hz), 7.76–7.79 (d, 3H, $J = 7.8$ Hz), 7.83–7.86 (t, 1H, $J = 7.3$ Hz), 8.63–8.65 (d, 1H, $J = 7.8$ Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 20.28, 21.70, 126.17, 126.35, 127.07, 127.28, 127.98, 129.14, 130.30, 131.78, 131.98, 133.54, 133.74, 137.30, 139.54, 142.41, 148.42, 159.48; Anal. Calcd for $C_{22}H_{18}N_2O$: C, 80.96; H, 5.56; N, 8.58. Found: C, 80.80; H, 5.57; N, 8.39.

4-(2-Chlorophenyl)-2-phenyl-2H phthalazin-1-one, (9). M.p. 164–165°C; as a Brown solid; IR (KBr): 1662, 1593, 1569, 1490, 1323, 1139; ^1H NMR ($CDCl_3$, 500 MHz): δ_H 7.40–7.43 (t, 1H, $J = 7.4$ Hz), 7.51–7.59 (m, 5H), 7.68–7.73 (d, 2H, $J = 7.2$ Hz), 7.78–7.80 (d, 2H, $J = 7.6$ Hz), 7.83–7.88 (m, 2H), 8.66–8.67 (d, 1H, $J = 7.5$ Hz); ^{13}C NMR ($CDCl_3$, 125 MHz): δ_C 126.27, 127.30, 128.08, 128.18, 129.07, 129.15, 129.40, 129.55, 129.66, 129.98, 132.08, 133.59, 135.48, 142.44, 148.04, 159.37; Anal. Calcd for $C_{20}H_{13}ClN_2O$: C, 72.18; H, 3.94; N, 8.42. Found: C, 72.52; H, 3.57; N, 8.27.

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