

RESEARCH PAPER

Ultrasonic Accelerated Efficient Synthesis of (1*H*-tetrazole-5-yl) Pyrazines Catalyzed by MgFe₂O₄ Nanoparticles as a Reusable Heterogeneous Catalyst

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ABSTRACT

An efficient synthesis of (1*H*-tetrazole-5-yl) pyrazines is achieved by one pot multi- component coupling reaction of α -dicarbonyl compounds, 2,3-diaminomaleonitrile and sodium azide using MgFe₂O₄ nanoparticles as a robust catalyst under ultrasonic irradiation. This novel synthetic method using MgFe₂O₄ nanoparticles is easier, faster than that of using other nanoparticles. MgFe₂O₄ nanoparticles have been characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM). The attractive advantages of the present process are atom economy, wide range of products, high catalytic activity, excellent yields, short reaction times and simple operational procedures. Use of simple and readily available starting materials, experimental simplicity, applying the sono-chemical methodology as an efficient method and innocuous means of activation in synthetic chemistry are some properties of this protocol. The reusability of the MgFe₂O₄ nanoparticles catalyst was examined and it was found that product yields decreased to a small extent on each reuse. The possibility of performing multicomponent reactions with a green catalyst under ultrasonic irradiation plays a prominent role in green chemistry.

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INTRODUCTION

The pyrazine ring system is a structural sector of a great number of biologically active compounds.

The pyrazine derivatives exhibit various pharmacological activities such as antibacterial [1,2], analgesic and anti-inflammatory [3], anti-cancer [4], and anti-bronchospastic [5]. The (1*H*-tetrazole-5-yl) pyrazine derivatives display variable biological properties including antiallergic [6,7], and anti-microbial [8]. Compounds containing the tetrazole moiety are utilized as TNF- α inhibitors [9], antiproliferative, antitumor [10], and antifungal activities [11]. Therefore, the development of easy procedures for the synthesis of (1*H*-tetrazole-5-yl) pyrazines is an

attractive challenge. Recently, the performing multicomponent reactions with a heterogeneous catalyst under ultrasonic irradiation have attracted much attention. The ultrasound approach decreases reaction times, increases yields and minimizes side reactions by providing the activation energy in micro environment [12-14]. The ultrasonic irradiations accelerate an organic transformation at ambient conditions which otherwise require harsh conditions of temperature and pressure [15-17]. The synthesis of tetrazoles has been described in the presence of different catalysts such as CuFe₂O₄ nanoparticles [18], γ -Fe₂O₃ [19], Fe₃O₄@SiO₂ nanoparticles [20], silver nanoparticles [21], NiO nanoparticles [22], and

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nano-ZnS [23]. Many methods for the synthesis of tetrazoles and pyrazines are known, but due to their importance, the improvement of new synthetic approaches by mild reaction conditions remains enough scope for an efficient and reusable catalyst with high catalytic activity for the preparation of (1*H*-tetrazole-5-yl) pyrazines. Herein, we report the use of MgFe₂O₄ nanoparticles as catalyst for the preparation of (1*H*-tetrazole-5-yl) pyrazines by one pot multi-component coupling reaction of α -dicarbonyl compounds, 2,3-diaminomaleonitrile and sodium azide under ultrasonic irradiation (Fig. 1).

MATERIALS AND METHODS

Materials and Apparatus

All organic materials were prepared commercially from Sigma-Aldrich and Merck and were used without further purification. A multiwave ultrasonic generator (Sonicator 3200; Bandelin, MS 73, Germany), equipped with a converter/transducer and titanium oscillator (horn), 12.5 mm in diameter, operating at 20 kHz with a maximum power output of 200 W, was used for the ultrasonic irradiation. The ultrasonic generator automatically adjusted the power level. All melting points are uncorrected and were determined in capillary tube on Boetius melting point microscope. FT-IR 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-*d*₆ as solvent and TMS as internal standard. CHN compositions were measured by Carlo ERBA

Model EA 1108 analyzer. Powder X-ray diffraction (XRD) was carried out on a Philips diffractometer of X'pert Company with monochromatized Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). In order to investigate the particle size and morphology of the synthesis structures nano-MgFe₂O₄, SEM images of the products visualized by a SEM LEO 1455VP.

Preparation of MgFe₂O₄ nanoparticles

In a typical preparation, MgSO₄, Fe(NO₃)₃·9H₂O, NaCl and NaOH were mixed (molar ratio 1:2:10:8) and ground together in an agate mortar for 30 min. The reaction started easily during the mixing procedure, accompanied by release of heat. As the reaction continued, the mixture became mushy and underwent gradual changes in color from colorless to light red (~1 min) and finally brown (~10 min). The mixture was then placed in a quartz crucible, inserted into a quartz tube, annealed at 700 °C for 1 h, and subsequently cooled to room temperature. Samples were collected, washed several times with distilled water, and dried at 120 °C overnight in a drying oven [24].

General procedure for the synthesis of (1*H*-tetrazole-5-yl) pyrazines

A mixture of α -dicarbonyl (1 mmol), 2,3-diaminomaleonitrile (1 mmol), and sodium azide (1.5 mmol or 3 mmol) and nano-MgFe₂O₄ (0.4 mo%) in DMSO (3 mL) was sonicated at 50 W power. After completion of the reaction confirmed by TLC (eluent: EtOAc/*n*-hexane, 1:1),

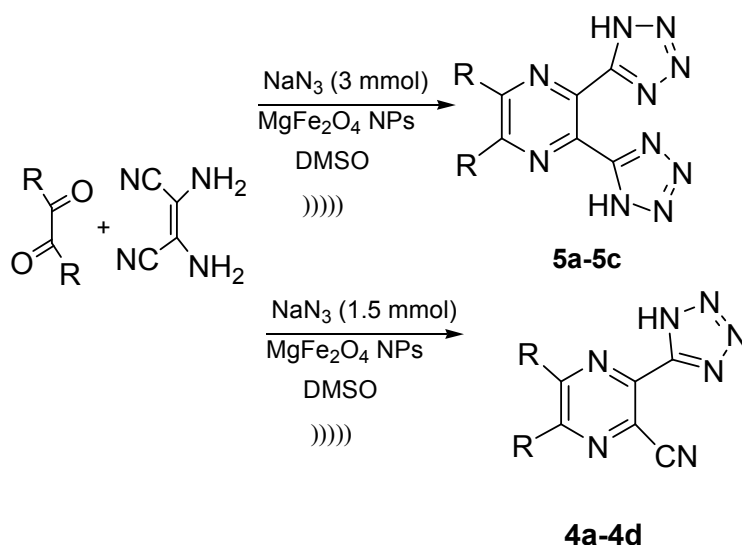


Fig. 1. Synthesis of (1*H*-tetrazole-5-yl) pyrazines using nano- MgFe₂O₄

the catalyst was separated magnetically and the heterogeneous catalyst was recovered. Then the solvent was removed. To the residue was added 10 mL of 2 N HCl with vigorous stirring causing the 3-(1H-tetrazol-5-yl) pyrazines.

Representative spectral data

3-(1H-tetrazol-5-yl)pyrazine-2-carbonitrile (4a):

Cream powder, m.p. 174-176 °C, IR (KBr) cm⁻¹: 3401, 2125, 1670, 1544; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 8.73 (1H, CH), 8.94 (1H, s, CH); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm) 117.2, 126.4, 143.9, 147.9, 148.1, 158.0; Anal. calcd for C₆H₃N₇: C, 41.62; H, 1.75; N, 56.63; Found: C, 41.51; H, 1.64; N, 56.54.

5,6-diphenyl-3-(1H-tetrazol-5-yl)pyrazine-2-carbonitrile (4b):

Yellow powder, m.p. 160-161 °C, IR (KBr) cm⁻¹: 3435, 2230, 1690, 1545, 1448, 708; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 7.36-7.51(10H, m, H-Ar); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm) 115.8, 124.4, 128.7, 128.9, 130.1, 130.4, 130.6, 136.5, 136.6, 139.7, 153.2, 153.5, 154.3; Anal. calcd for C₁₈H₁₁N₇: C, 66.45; H, 3.41; N, 30.14; Found: C, 66.37; H, 3.35; N, 30.09.

5,6-bis(4-methoxyphenyl)-3-(1H-tetrazol-5-yl)pyrazine-2-carbonitrile (4c):

Yellow powder; m.p. 122-124 °C, IR (KBr) cm⁻¹: 3430, 3030, 2236, 1637, 1549, 1282; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 3.85 (3H, s, OCH₃), 3.90 (3H, s, OCH₃), 6.92-7.93 (8H, m, H-Ar); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm) 55.7, 55.8, 114.4, 114.8, 116.2, 123.3, 128.7, 129, 131.5, 131.7, 132.1, 138.7, 152.7, 153.4, 161.2, 161.4; Anal. calcd for C₂₀H₁₅N₇O₂: C, 62.33; H, 3.92; N, 25.44; Found: C, 62.25; H, 3.96; N, 25.54.

9-(1H-tetrazol-5-yl)acenaphtho[1,2-b]pyrazine-8-carbonitrile (4d):

Brown powder; m.p. 238-240 °C; IR (KBr) cm⁻¹: 3434, 3100, 2236, 1613, 1450; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 7.99-8.03 (2H, m, H-Ar), 8.42-8.54 (4H, m, H-Ar); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm) 116.5, 124.3, 127.4, 127.6, 127.9, 128.1, 128.4, 134.5, 143.4, 148.1, 148.8, 158.4; Anal. calcd for C₁₆H₇N₇: C, 64.64; H, 2.37; N, 32.98; Found: C, 64.53; H, 2.32; N, 32.82.

2,3-di(1H-tetrazol-5-yl)pyrazine (5a):

White powder; m.p. 255-256 °C; IR (KBr) cm⁻¹:

¹: 3428, 3123, 1683, 1549; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 9.15 (2H, s, H-Ar); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm) 140.2, 146.5, 153.7; Anal. calcd for C₆H₄N₁₀: C, 33.34; H, 1.87; N, 64.80; Found: C, 33.26; H, 1.81; N, 64.89.

2,3-diphenyl-5,6-di(1H-tetrazol-5-yl)pyrazine (5b):

Cream powder; m.p. 250-252 °C; IR (KBr) cm⁻¹: 3428, 2923, 1683, 699, 761; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 7.41-7.51 (6H, m, H-Ar), 7.60-7.85 (4H, m, H-Ar); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm) 128.8, 130.2, 130.3, 136.7, 137.0, 152.9, 153.6; Anal. calcd for C₁₈H₁₂N₁₀: C, 58.69; H, 3.28; N, 38.03; Found: C, 58.53; H, 3.38; N, 38.09.

2,3-bis(4-methoxyphenyl)-5,6-di(1H-tetrazol-5-yl)pyrazine (5c)

Cream powder; m.p. 207-209 °C; IR (KBr) cm⁻¹: 3538, 1543, 1454; ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 3.79 (6H, s, OMe), 6.973-6.994 (4H, d, *J* = 8.4 Hz, H-Ar), 7.853-7.874 (4H, d, *J* = 8.4 Hz, H-Ar); ¹³C NMR (100 MHz, DMSO-*d*₆): δ (ppm) 55.7, 114.4, 129.4, 131.5, 131.7, 132.0, 151.8, 160.9; Anal. calcd for C₂₀H₁₆N₁₀O₂: C, 56.07; H, 3.76; N, 32.69; Found: C, 56.14; H, 3.85; N, 32.57.

RESULTS AND DISCUSSION

The XRD patterns for MgFe₂O₄ nanoparticle is shown in Fig. 2. The pattern agrees well with the reported pattern for MgFe₂O₄ nanoparticles (JCPDS No. 71-1232). The crystalline size was calculated from FWHM using Scherrer's formula and was observed to be 25-30 nm. The morphology and particle size of MgFe₂O₄ NPs was investigated by scanning electron microscopy (SEM) as shown in Fig. 3. The SEM images prove particles with diameters in the range of nanometers. Initially, we focused on systematic evaluation of diverse catalysts for the model reaction of oxalaldehyde (1 mmol), 2,3-diaminomaleonitrile (1 mmol), and sodium azide (1.5 mmol) in different solvents. We employed various conditions and found that the reaction gave satisfying result in the presence of nano-MgFe₂O₄ under ultrasonic irradiation in DMSO (Table 1). The reaction was carried out with different amounts of nano-MgFe₂O₄ as catalyst. As show in Table 1, 0.40 mol % of nano-MgFe₂O₄ as catalyst was suitable and when the amount of catalyst was increased to 0.60 mol %, but the yield was not improved. The model reaction was carried out in the presence of various catalysts such as nano-MgO, nano-NiO, nano-Fe₃O₄, nano-NiFe₂O₄,

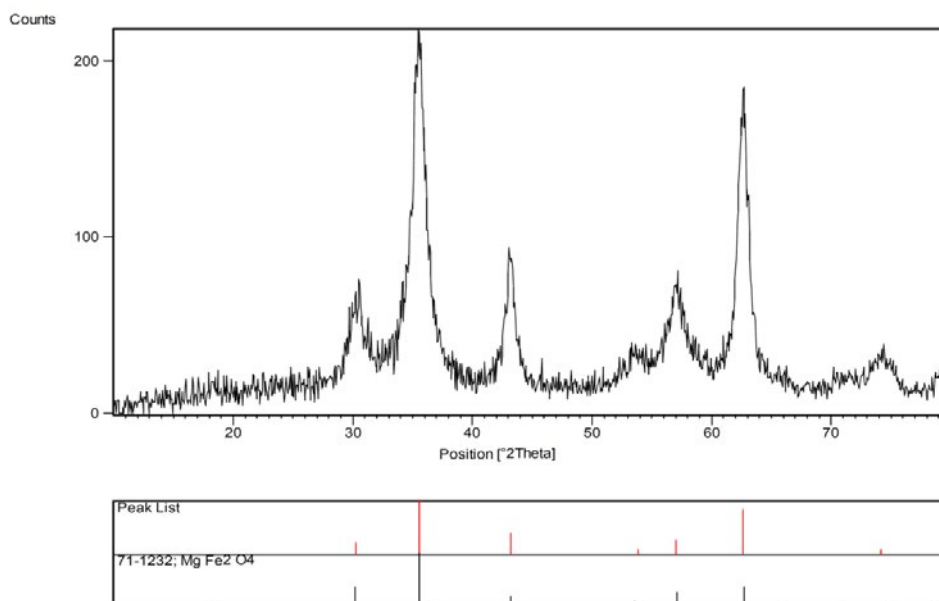


Fig. 2. The XRD pattern of $MgFe_2O_4$ nanoparticles

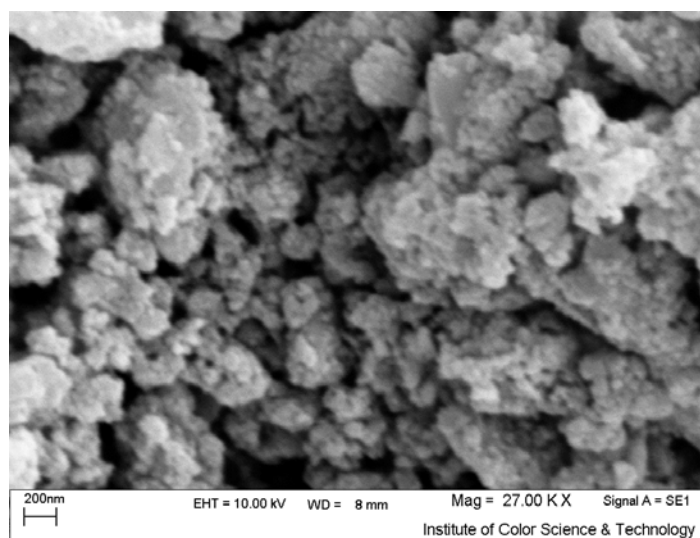


Fig. 3. SEM image of $MgFe_2O_4$ nanoparticles

and nano- $MgFe_2O_4$. When the reaction was carried out using $MgFe_2O_4$ nanoparticles as the catalyst under ultrasonic irradiation, the product could be obtained in good yield.

In continuation of this method, the model reaction was performed with 0.40 mol % of nano- $MgFe_2O_4$ in DMSO in various powers of ultrasonic irradiation to explore the appropriate power of ultrasonic irradiation. It is clear from Table 2 that, reactions under the effect of ultrasound give excellent yields of products in short reaction times

due to inrush of liquid from one side of the surface of the catalyst because of the collapse of the cavitation bubbles. This high pressure jet of the liquid is supposed to activate the surface of the solid catalyst and consequently increase the rate of the reaction [25-27]. Therefore, it was observed that the reaction in the presence of 0.40 mol % of nano- $MgFe_2O_4$ and under ultrasonic irradiation with the power of 50W gave the best result as the obtained product with 95% isolated yield during 10 minutes. With these promising results

Table 1. Optimization of reaction conditions using different catalysts

Entry	Solvent (condition)	Catalyst (mol%)	Time (min)	Yield ^b (%)
1	toluene (reflux)	—	600	trace
2	EtOH (reflux)	—	400	12
3	MeOH (reflux)	—	400	18
4	DMSO (90 °C)	—	140	52
5	DMSO (90 °C)	nano-MgO (0.9 mol%)	100	68
6	DMSO (90 °C)	nano-Fe ₃ O ₄ (0.8 mol%)	100	74
7	DMSO (90 °C)	nano-MgFe ₂ O ₄ (0.6 mol%)	80	85
8	DMSO (90 °C)	nano-NiO (0.9 mol%)	100	62
9	DMSO (90 °C)	nano-NiFe ₂ O ₄ (0.6 mol%)	90	79
10	DMSO (US) ^c	nano-MgFe ₂ O ₄ (0.2 mol%)	10	93
11	DMSO (US)	nano-MgFe ₂ O ₄ (0.4 mol%)	10	95
12	DMSO (US)	nano-MgFe ₂ O ₄ (0.6 mol%)	10	95
13	DMSO (US)	—	10	64
14	DMSO (US)	nano-Fe ₃ O ₄ (0.8 mol%)	10	78
15	DMSO (US)	nano-NiFe ₂ O ₄ (0.6 mol%)	10	82

^a oxalaldehyde (1 mmol), 2,3-diaminomaleonitrile (1 mmol), and sodium azide (1.5 mmol)

^b Isolated yields

^c Ultrasonic irradiation (50 W)

Table 2. Study of the effect of ultrasonic irradiation on the formation of (1H-tetrazole-5-yl) pyrazines

Entry	Power (W)	Time (min)	Yield ^b (%)
1	20	20	76
2	30	20	85
3	40	15	91
4	50	10	95
5	60	10	95

^a oxalaldehyde (1 mmol), 2,3-diaminomaleonitrile (1 mmol), and sodium azide (1.5 mmol), nano-MgFe₂O₄ (0.4 mol%)

^b Isolated yields

in hand, we turned to explore the possibility of the reaction using diverse α -dicarbonyl compounds as substrates under the optimized reaction conditions (Table 3).

After completion of the reaction, the catalyst was separated magnetically from the reaction mixture and washed with Et₂O, air-dried and then reused directly in the model reaction of oxalaldehyde (1 mmol), 2,3-diaminomaleonitrile (1 mmol), and sodium azide (1.5 mmol) by 0.40 mol% of nano-MgFe₂O₄ under ultrasonic irradiation in DMSO. The results demonstrated that the catalyst exhibited high but slowly decreasing activity in six consecutive cycles, which might be attributed to the slight loss of catalyst during the reaction and recovery processes (Fig. 4).

A plausible mechanism for the preparation of (1H-tetrazole-5-yl) pyrazines using nano-

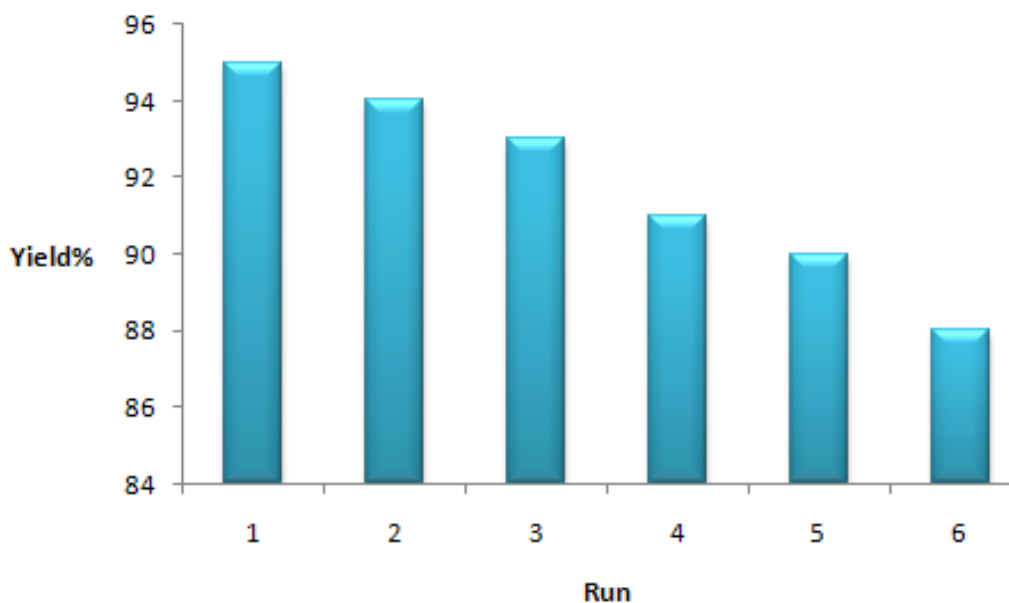


Fig. 4. Reusability of MgFe₂O₄ nanoparticles catalyst for the preparation of 4a

Table 3. Synthesis of (1H-tetrazole-5-yl) pyrazines using nano-MgFe₂O₄ (0.4 mol%) under ultrasonic irradiation (50 W)

Entry	α -dicarbonyl compounds	Product	4a-5c	Time (min)	Yield ^a %
1			4a	10	95
2			4b	10	97
3			4c	15	92
4			4d	10	95
5			5a	10	94
6			5b	10	95
7			5c	15	91

^a Isolated yields

MgFe₂O₄ is shown in Fig. 5. The formation of products can be rationalized by initial formation of pyrazine-2,3-dicarbonitriles by a condensation reaction of α -dicarbonyl compounds and 2,3-diaminomaleonitrile. Subsequent [2+3] cycloaddition reaction of pyrazine-2,3-dicarbonitriles with the sodium azide to afford (1H-tetrazole-5-yl) pyrazines. In this mechanism the nano-MgFe₂O₄ as a highly efficient and green catalyst activates the C=O and C≡N groups for better reaction with nucleophiles.

CONCLUSION

In conclusion, we have developed an atom-efficient, high-yielding protocol for the synthesis of (1H-tetrazole-5-yl) pyrazines by one pot multi-component coupling reaction of α -dicarbonyl compounds, 2,3-diaminomaleonitrile and sodium azide using MgFe₂O₄ nanoparticles as a robust catalyst under ultrasonic irradiation. The attractive advantages of the present process are atom economy, wide range of products, high catalytic activity, excellent yields, short reaction times and simple operational procedures.

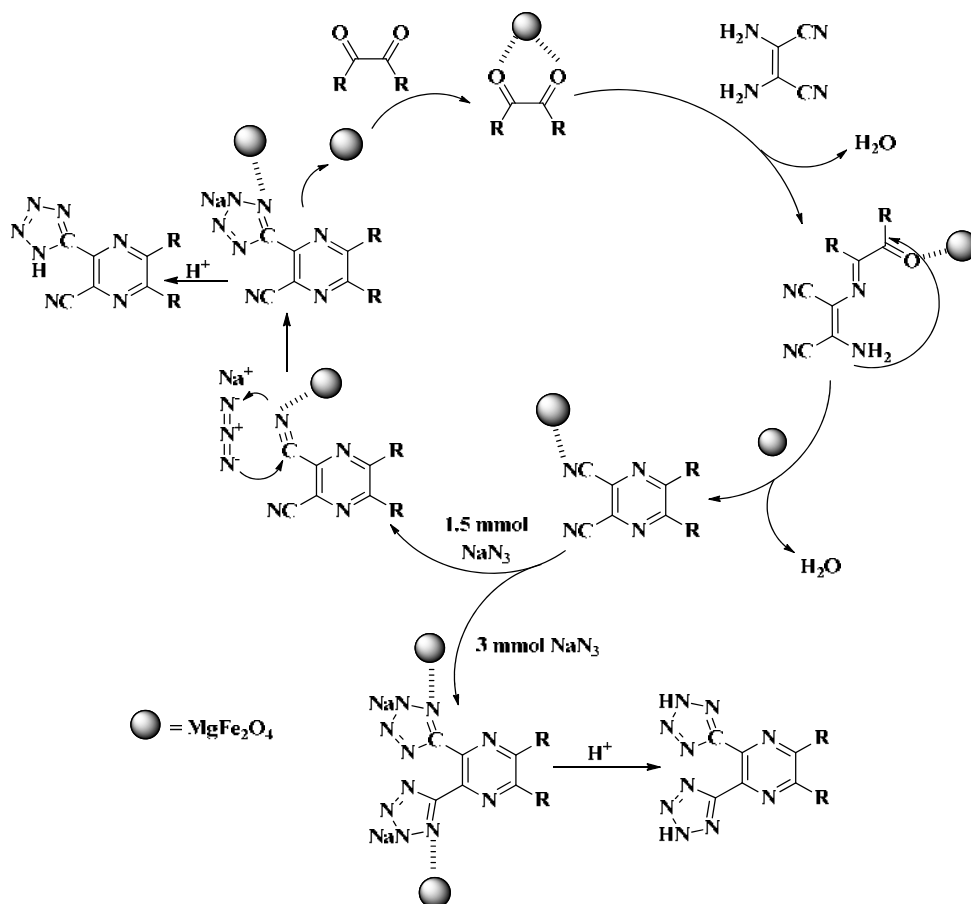


Fig. 5. A plausible mechanism for the preparation of (1H-tetrazole-5-yl) pyrazines using nano- MgFe_2O_4 .

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CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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