$CoFe_2O_4$ nanoparticles as a magnetically recoverable and reusable catalyst for the synthesis of arylaminotetrazoles and 5-aryloxytetrazoles

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An efficient and direct method is described for the synthesis of 5-arylamino-1*H*-tetrazoles (Isomer A) or 1-aryl-5-amino-1*H*-tetrazoles (Isomer **B**) and 5-aryloxytetrazoles from arylcyanamides or cynates with nano $CoFe_2O_4$ as a reusable and efficient heterogeneous catalyst. Isomer **A** was obtained from arylcyanamides carrying electron-withdrawing substituents on the aryl ring, while isomer **B** was produced with electron-releasing groups. The significant advantages of this methodology are high yields, elimination of dangerous and harmful hydrazoic acid, simple work-up procedure and the recovery and reusability of the catalyst.

Keywords: arylaminotetrazole, aryloxytetrazole, cyanamides, cyanates, $CoFe_2O_4$ nanoparticles, heterogeneous catalyst

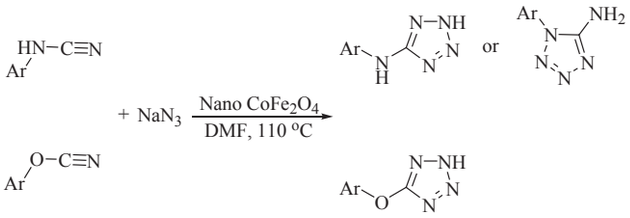
Tetrazoles have been employed as an isostere for the carboxylic acid moiety,[1] as ligands in coordination chemistry and as explosives and rocket propellants.[2,3] Tetrazole is a well-known intermediate in the synthesis of imidoylazides.[4]

Classical methods for the synthesis of tetrazoles have involved the reaction of azide ions with nitriles, cyanates and cyanamides in the presence of acids or other catalysts in organic solvents at high temperature and pressure.[5–16] Among the tetrazoles, arylaminotetrazoles and aryloxytetrazoles have received attention because of their wide utility.[4–16] However, the lack of convenient methods for the preparation of arylaminotetrazoles restricts their potential application in medicine. Generally, arylaminotetrazoles are synthesised by the condensation reaction of cyanamides with hydrazoic acid,[17] HOAc[7] and $FeCl_3$–SiO_2[13] which lead to a mixture of products. Earlier methods for the synthesis of arylaminotetrazoles or 5-aryloxytetrazoles suffer from drawbacks such as the use of strong Lewis acids or expensive and toxic metals, drastic reaction conditions, low yield, long reaction times, low temperatures (–70 °C), tedious work-up procedures due to the application of homogeneous catalyst and hydrazoic acid formation. The latter is explosive and very toxic.[18,19]

Several regiospecific syntheses of arylaminotetrazoles have been reported through the [2+3] cycloaddition of cyanamides using NaN_3 in the presence of catalysts such as $ZnCl_2$,[15] $AlCl_3$[9] and Iranian Natrolite zeolite.[14] Thus, the development of a catalytic synthetic method for tetrazoles still remains an active research area.

There has been an interest in various chemical transformations performed with heterogeneous catalysis[20–24] and, in particular, magnetic nanoparticles have emerged as a useful group.[25–30] The separation of magnetic nanoparticles is simple, and an attractive alternative to filtration or centrifugation as it prevents the loss of catalyst and enhances reusability, making the catalyst more cost-effective and promising for industrial applications. Recently, magnetically separable $CoFe_2O_4$ nanoparticles, have been prepared by a simple procedure and their catalytic activity investigated.[30]

In continuation of our researches on the application of heterogeneous catalyst,[31] we report a new protocol for preparation of arylaminotetrazoles and 5-aryloxytetrazoles from cyanamides and cyanates, respectively using magnetically separable $CoFe_2O_4$ nanoparticles as an efficient catalyst (Scheme 1). This catalyst, can be prepared from inexpensive and readily available materials and is environmentally acceptable and recyclable.



Scheme 1 Synthesis of arylaminotetrazoles and 5-aryloxytetrazoles using magnetically separable $CoFe_2O_4$ nanoparticles.

Results and discussion

No reaction was observed when a mixture of 2,5-dichlorophenylcyanamide and sodium azide was stirred for 5 h at 110 °C. However, addition of the nano $CoFe_2O_4$ to the mixture has rapidly increased the yield of the arylaminotetrazoles.

At the start, the reactivity of various catalysts were investigated in the reaction of 2,5-dichlorophenylcyanamide with sodium azide in different solvents (Table 1). $FeCl_3$–SiO_2[13] and glacial HOAc[7] catalysts gave a mixture of isomers (**A**+**B**), while nano $CoFe_2O_4$ gave just the isomer (**A**). Another method needed 0.4 g of zinc (II) chloride as reagent under reflux in water and involved long reaction times (15 h). $ZnCl_2$[15] is a well-known reagent, but it is sensitive to moisture and is difficult to recycle. Natrolite zeolite[14] is a local catalyst, but there are difficulties in the preparation and availability of the catalyst. Zeolites also have definite advantages and some disadvantages over conventional solid catalysts in a certain number of applications. The main limitations are (i) the great sensitivity of zeolites to deactivation by irreversible adsorption or steric blockage by high molecular weight by-products and (ii) the impossibility of using their microporosity for the synthesis of bulky molecules. Then, we optimised the amount of nano $CoFe_2O_4$ catalyst required for the model reaction (Table 1). Water was not a suitable solvent for this reaction. Sodium azide is insoluble in most organic solvents, which has limited its application in organic synthesis. However, cycloaddition reactions have been carried out at high temperatures. DMF is a suitable solvent for these cycloaddition reactions.[13,14] The optimum amount of nano $CoFe_2O_4$ was found to be 0.1 g in the presence of cyanamides (2 mmol) and sodium azide (3 mmol) in DMF (6 mL). We next examined a variety of structurally divergent phenylcyanamide possessing a wide range of functional groups to understand

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Table 1 Comparison of catalytic activities in the reaction of 2,5-dichlorophenylcyanamide with sodium azide in different solvents

Entry	Catalyst	Solvent	Time/min	Yield/%[a]	Product (**A** or **B**)
1	$FeCl_3$-SiO_2 (0.1 g)	DMF[b]	120	76	**A**+**B**
2	Glacial HOAc (3 mL)	Glacial HOAc[c]	30 h	72	**A**+**B**
3	$ZnCl_2$ (0.4 g)	H_2O[d]	15 h	84	**A**
4	Natrolite zeolite (0.1 g)	H_2O[d]	9 h	49	**A**
5	Natrolite zeolite (0.1 g)	DMF[e]	95	81	**A**
6	Nano $CoFe_2O_4$ (0.05 g)	DMF[b]	105	71	**A**
7	Nano $CoFe_2O_4$ (0.07 g)	DMF[b]	105	78	**A**
8	Nano $CoFe_2O_4$ (0.10 g)	DMF[b]	105	87	**A**
9	Nano $CoFe_2O_4$ (0.10 g)	DMSO	105	87	**A**
10	Nano $CoFe_2O_4$ (0.15 g)	DMF[b]	105	87	**A**

[a]Pure isolated products.
[b]Reaction was carried out at 110 °C.
[c]Room temperature.
[d]Under reflux conditions.
[e]Reaction was carried out at 110–115 °C.

Table 2 Preparation of 5-arylamino-1*H*-tetrazoles (**A**) and 1-aryl-5-amino-1*H*-tetrazoles (**B**) using nano $CoFe_2O_4$ from cyanamides at 110 °C

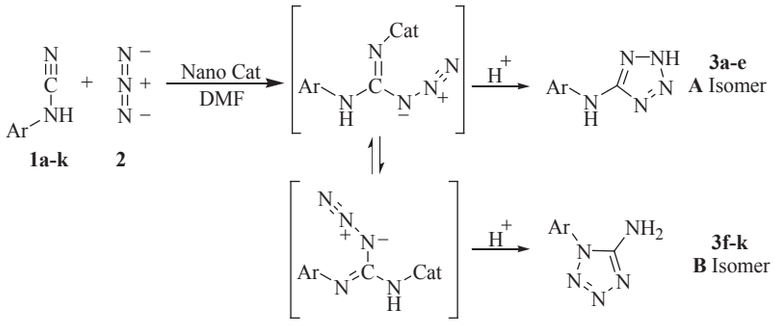
Entry	Substrate	Product (Isomer **A** or **B**)	Time/min	Yield/%[a]	M.p. (lit.)[Ref.]
1	4-$NO_2C_6H_4NHCN$ (**1a**)	4-$NO_2C_6H_4NHTet$ (**3b**) (**A**)	105	84	218–220 (218–220)[11]
2	2-ClC_6H_4NHCN (**1b**)	2-ClC_6H_4NHTet (**3b**) (**A**)	105	86	228–230 (228–230)[11]
3	2,5-$(Cl)_2C_6H_3NHCN$ (**1c**)	2,5-$(Cl)_2C_6H_3NHTet$ (**3c**) (**A**)	100	87	272–274 (272–274)[11]
4	4-BrC_6H_4NHCN (**1d**)	4-BrC_6H_4NHTet (**3d**) (**A**)	100	86	249–250 (249–250)[11]
5	C_6H_5NHCN (**1e**)	C_6H_5NHTet (**3e**) (**A**)	90	85	215–217 (215–217)[11]
6	4-MeC_6H_4NHCN (**1f**)	4-MeC_6H_4NHTet (**3f**) (**B**)	55	86	178–179 (178–179)[11]
7	2,4-$(Me)_2C_6H_3NHCN$ (**1g**)	2,4-$(Me)_2C_6H_3NHTet$ (**3g**) (**B**)	55	85	199–201 (199–201)[11]
8	2-MeC_6H_4NHCN (**1h**)	2-MeC_6H_4NHTet (**3h**) (**B**)	55	85	191–192 (191–192)[11]
9	4-$NHCNC_6H_5NHCN$ (**1i**)	4-$NHTetC_6H_5NHTet$ (**1i**) (**B**)	50	88	264–266 (264–266)[11]
10	4-$OMeC_6H_4NHCN$ (**1j**)	4-$OMeC_6H_4NHTet$ (**1j**) (**B**)	55	86	211–213 (211–213)[11]
11	2,6-$(Me)_2C_6H_3NHCN$ (**1k**)	2,6-$(Me)_2C_6H_3NHTet$ (**1k**) (**B**)	55	87	211–213 (–)

[a]Pure isolated product.

the scope and generality of the nano $CoFe_2O_4$-promoted [2+3] cycloaddition reaction to form arylaminotetrazoles. These results are summarised in Table 2.

As shown in Table 2, cyanamides having electron releasing groups on the rings (entries 6–8, 10, 11) were completed at 110 °C after 55 min, while the species bearing the electron withdrawing species (entries 1–4) required longer reaction times. The influences of various substituents in different *ortho*, *meta* or *para* positions on the type of the products were examined. Substituents on the aromatic ring of cyanamide had an important effect on the type of product (isomers **A** or **B**) and these results were incorporated in Table 2. In the case of cyanamides having the electron releasing groups, 1-aryl-5-amino-1*H*-tetrazoles (**B**) was obtained (entries 6–8, 10 and 11, Table 2). When the substituent was replaced by an electron-withdrawing group, the 5-arylamino-1*H*-tetrazoles (**A**) were obtained (entries 1–4, Table 2). It was reported that the 1-(4-nitrophenyl)-5-amino-1*H*-tetrazole (isomer **B**) was obtained using hydrazoic acid and a mixture of isomers (**A**+**B**) were obtained by the use of $FeCl_3$–SiO_2 or glacial HOAc, while with nano $CoFe_2O_4$ only a single 5-(4-nitrophenyl)amino-1*H*-tetrazole (isomer **A**) was produced. In the presence of two CN groups, **1i** (Table 2, entry 9) afforded the double-addition product. 2,6-Dimethylphenylcyanamide and 2-methylphenylcyanamide (Table 2, entry 11 and 8) interestingly gave **B** isomer, while with HOAc and $FeCl_3$–SiO_2, the **A** isomer and the mixture of isomers (**A**+**B**) were obtained.

The nano $CoFe_2O_4$ probably has an important role in the promotion of the synthesis of arylaminotetrazoles as a Lewis acid. A plausible mechanism is shown in Scheme 2. Nano $CoFe_2O_4$ may show complexation towards nitrile group of cyanamides and thus may enhance its electrophic character. It involves activation of the nitrile group over surface of the



Scheme 2 Mechanism of nano $CoFe_2O_4$ catalysed synthesis of arylaminotetrazoles.

Table 3 Preparation of 5-aryloxytetrazoles using nano $CoFe_2O_4$ from cyanates at 110 °C

Entry	Substrate	Product	Time/min	Yield/%[a]	M.p. (lit.)[Ref.]
1	C_6H_5OCN (**1l**)	C_6H_5OTet (**3l**)	200	81	136–138 (137–138)[33]
2	4-$OMeC_6H_4OCN$ (**1m**)	4-$OMeC_6H_4OTet$ (**3m**)	180	80	150–151 (149–150)[33]
3	2,6-$(Me)_2C_6H_3OCN$ (**1n**)	2,6-$(Me)_2C_6H_3OTet$ (**3n**)	180	78	171–173 (173–174)[33]
4	4-ClC_6H_4OCN (**1o**)	4-ClC_6H_4OTet (**3o**)	220	79	166–168 (166–167)[33]
5	4-MeC_6H_4OCN (**1p**)	4-MeC_6H_4OTet (**3p**)	180	82	139–140 (140–142)[33]
6	4-$NO_2C_6H_4OCN$ (**1q**)	4-$NO_2C_6H_4OTet$ (**3q**)	220	81	161–163 (162–163)[33]

[a]Pure isolated product.

nano $CoFe_2O_4$ and subsequent nucleophilic attack of the sodium azide. A similar mechanism has been reported for the synthesis of arylaminotetrazoles using catalyst ZAS.[13]

The reactivity of several cyanates was also tested in the reaction with sodium azide in the presence of nano $CoFe_2O_4$ under thermal conditions. The results are given in Table 3. The electronic nature or position of the substituents on the aromatic ring of cyanates affected the reaction time. As shown in Table 3, cyanates having the methyl as electron-releasing groups (entries 2,3 and 5) were completed at 110 °C after 180 min, while the species bearing the $–NO_2$ or –Cl electron-withdrawing groups (entries 4 and 6) required longer reaction times.

The structure of the products was confirmed by IR, ^{1}H NMR and ^{13}C NMR spectral data and melting points. 5-Arylamino-1*H*-tetrazoles isomers (**A**) contain two NH bonds [NH of the amine attached to the aryl group (NH^A) and NH of the tetrazole ring (NH^T)] and 1-aryl-5-amino-1*H*-tetrazoles isomers (**B**) contain a NH_2 bond. In the IR spectra of the tetrazoles, the CN peak had disappeared and one absorptions band (NH stretching band) was detected. The appearance of a signal about $\delta = 154–157$ ppm for the tetrazole ring carbon in the ^{13}C NMR spectra was evidence for the formation of tetrazoles.[9,14,15]

The reusability of the catalyst (Table 3) was examined with 4-nitrophenylcyanamide and NaN_3 under the optimised reaction conditions. $CoFe_2O_4$ nanoparticles are recyclable without loss of significant catalytic activity. The catalyst was separated by an external magnet and reused for subsequent experiments under similar reaction conditions. Yields of the product decreased only slightly after four cycles (Table 4). This reusability demonstrated the high stability and turnover of $CoFe_2O_4$ magnetic nano catalyst under operating conditions. Work is in progress to develop novel magnetically recoverable and reusable catalysts and study their application in the other type of reactions.

Table 4 Reusability of the $CoFe_2O_4$ catalyst

Entry	Yield/%	Recovery of $CoFe_2O_4$/%
Refresh	84	99
1	83	99
2	83	98
3	81	98
4	80	97

Conclusion

In conclusion, we have developed a novel and highly efficient method for the synthesis of various arylaminotetrazoles and 5-aryloxytetrazoles by treatment of cyanamides or cyanates with sodium azide in the presence of $CoFe_2O_4$ as a magnetically recoverable and reusable catalyst. The significant advantages of this methodology are the high yields, elimination of dangerous and harmful hydrazoic acid and simple work-up procedure. The catalyst is easily prepared, stable to air and magnetically recoverable from the reaction mixture by applying external magnetic field and reused four times without significant loss of catalytic activity. This methodology may find widespread use in organic synthesis for the preparation of tetrazoles.

Experimental

All the solvents and reagents were purchased at the highest commercial quality and used without further purification. All reaction mixtures were stirred magnetically and were monitored by TLC using 0.25 mm E-Merck silica gel 60 F254 pre-coated glass plates, which were visualised with UV light and then developed by using iodine mixed with silica gel 60–120 mesh. Melting points were recorded on a Buchi R-535 apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer FT-IR 240-C spectrophotometer using KBr optics. ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker Avance 400 MHz spectrometers in $CDCl_3$ and DMSO using TMS as internal standard, with chemical shifts being given in ppm with respect to internal TMS and *J* values quoted in Hz.

Preparation of the catalyst

Two aqueous solutions of $FeCl_3$ (1.5 g, 9.3 mmol, 50 mL) and $CoCl_2{\cdot}6H_2O$ (1 g, 4.2 mmol, 50 mL) were mixed in a 200 mL flat bottom flask and placed in an ultrasonic bath. An aqueous KOH solution (3 M, 25 mL) was added dropwise under argon atmosphere with continuous ultrasonic irradiation (frequency 40 KHz and power of 40 KW). Prior to mixing, all three solutions were sonicated for 30 min to remove dissolved oxygen. The temperature of the sonicator bath was raised to 60 °C and the mixture was further sonicated for 30 min in air. A black precipitate was formed during this time. The reaction mixture was centrifuged (14,000 rpm) at ambient temperature for 15 min. The mixture was subjected to successive sonication (30 min) and centrifugation (15 min) for five times. The black precipitate was then separated, washed with copious amounts of distilled water, ethanol and kept overnight in an incubator at 60 °C for ageing. The precipitate was then dried in an oven at 100 °C for 1 h and subsequently kept under high vacuum (10^{-2} bar) for 1 h. Finally, the black particles were suspended in dry ethanol (50 mL) and subjected to successive sonication (30 min) and centrifugation (15 min) repeatedly until a brown coloured solution appeared. The precipitate was separated, dried and stored for further applications.[30]

Preparation of the arylcyanamides

The arylcyanamides were prepared according to the literature.[13,32]

Preparation of the arylcyanates

The arylcyanates were prepared according to the literature.[33]

Synthesis of arylaminotetrazoles and 5-aryloxytetrazoles; general procedure

A mixture of arylcyanamides (**1a–k**) or arylcyanates (**1l–q**) (2 mmol), sodium azide (3 mmol), nano $CoFe_2O_4$ (0.1 g) and DMF (6 mL) was placed in a round-bottomed flask and stirred for the appropriate time (Table 2) at 110 °C. The progress of the reaction was monitored by TLC. After completion of the reaction, the catalyst was separated by an external magnet, washed with ethyl acetate and activated at 100 °C for 2 h. Then, the resultant mixture was treated with ethyl acetate (35 mL) and 5 N HCl (20 mL). The organic layer was separated and the aqueous

layer was extracted with ethyl acetate (20 mL) again. The combined organic layers were concentrated under reduced pressure. The crude solid was recrystallised with aqueous ethanol to afford the pure product in high yields. All products were characterised by spectral analysis. All compounds were compared with the corresponding compounds prepared by the reported procedure.[4,9,14,15]

*1-(2,6-Dimethylphenyl)-5-amino-1*H*-tetrazole* (**3k**, *Table 2, entry 11*): M.p. 147–149 °C; IR (KBr, cm^{-1}): 3430, 3295, 3070, 2955, 2915, 2850, 1650, 1601, 1586, 1538, 1474, 1440, 1376, 1349, 1260, 1217, 1160, 1114, 917, 869, 760, 700, 662, 636, 546, 516, 496; ^{1}H NMR (500 MHz, DMSO-d_6): δ_H 2.25 (s, 6H), 5.24 (s, 2H), 7.03 (s, 3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ_C 157.5, 137.1, 136.6, 128.3, 126.3, 18.6; Anal. calcd for $C_9H_{11}N_5$: C, 57.12; H, 5.86; N, 37.02; found: C, 57.18; H, 5.90; N, 37.09%.

5-(Phenoxy)tetrazole (**3l**, *Table 3, entry 1*): ^{1}H NMR (500 MHz, DMSO-d_6), δ_H 7.25 (s, 5H), 12.10 (s, br, 1H).

5-(4-Methoxyphenoxy)tetrazole (**3m**, *Table 3, entry 2*): ^{1}H NMR (500 MHz, DMSO-d_6), δ_H 3.91 (s, 3H), 7.11 (d, J = 10.2 Hz, 2H), 7.51 (d, J = 10.2 Hz, 2H), 8.49 (s, 1H).

5-(2,6-Dimethylphenoxy)tetrazole (**3n**, *Table 3, entry 3*): ^{1}H NMR (500 MHz, DMSO-d_6), δ_H 2.42 (s, 3H), 7.34 (s, 4H), 9.90 (s, 1H).

5-(4-Chlorophenoxy)tetrazole (**3o**, *Table 3, entry 4*): ^{1}H NMR (500 MHz, DMSO-d_6), δ_H 7.40 (s, 4H), 8.71 (s, br, 1H).

5-(4-Methylphenoxy)tetrazole (**3p**, *Table 3, entry 5*): ^{1}H NMR (500 MHz, DMSO-d_6), δ_H 2.30 (s, 6H), 7.18 (s, 3H), 10.40 (s, 1H).

5-(4-Nitrophenoxy)tetrazole (**3q**, *Table 3, entry 6*): ^{1}H NMR (500 MHz, DMSO-d_6), δ_H 7.61 (d, J = 8.7 Hz, 2H), 8.39 (d, J = 8.7 Hz, 2H), 13.01 (s, br, 1H).

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