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Microwave assisted one pot conversion of aromatic aldehydes to nitriles

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RESEARCH ARTICLE

ABSTRACT



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Nitriles are versatile organic precursors in organic synthesis and have numerous applications. An efficient microwave assisted method for conversion of aromatic aldehydes to the corresponding nitriles is reported. Aldehydes are readily converted to oxime followed by acetylation and acetic acid elimination to provide nitriles in good yields within minutes. The method proved to be efficient for the synthesis of aromatic and heterocyclic nitriles. The reaction proceeds smoothly by microwave at 150 °C for 5 minutes. The obtained products are isolated simply by filtration or extraction.

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1. Introduction

The importance of nitriles arises from its use for the synthesis of a variety of important functionalities such as amines, amides, aldehydes, carboxylic acids, esters and ketones. Those are considered the scaffolds for agro-chemicals, dyes, pharmaceuticals, and functional materials. Nitriles are abundant in nature, for example in phytochemicals, bitter almond and cassava. Nitrile-containing pharmaceuticals have diverse medicinal indications. Furthermore, several substituted benzonitriles have been developed as selective inhibitors for various enzymes related to chronic diseases. Few demonstrative examples are shown in Figure 1. Finrozole (1) is an aromatase and aldosterone inhibitor. Letrozole (2) is a Food and Drug Administration (FDA) approved drug used as aldosterone inhibitor for the treatment of breast cancer. Milrinone (3) is a *meta*-substituted benzonitrile and a phosphodiesterase inhibitor used for heart failure. Vildagliptin (4) is an aminonitrile used as antidiabetic drug. Entacapone (5) is a vinylic nitrile prescribed for Parkinson's disease. Bosutinib (6), having the core of 3-cyanoquinoline, was recently FDA

approved as an anti-cancer drug. Febuxostat (7) is a xanthine oxidase inhibitor; used as uric acid reducer [1]. 2-Amino-oxazole-5-phenyl-3-cyanoindole (8) is an inosine monophosphate dehydrogenase inhibitor [2]. These factors have prompted to us the importance of developing a fast, low cost, facile and feasible method for the synthesis of aromatic nitriles.

Sandmeyer and Rosenmund-Von Braun developed the early methods to convert aromatic amines and halides to nitrile using CuCN as cyanating agent. Later, several other methods and reagents have been employed for conversion of aldehydes to nitrile *via* their aldoximes [3-17]. Recently, oxo-ammonium salts [18], hydroxylamine-*o*-sulphonic acid [19], and *o*-(diphenyl phosphinyl)hydroxylamine / toluene [20], were reported. In addition, Fe₃O₄ nanoparticles [21] and active silver nanoparticles [22] were used as catalyst. More recently, Cu catalyzed cyanation, [23] and Fe catalyzed dehydration of aldoximes [24] were used for nitrile formation. However, the drawbacks of these methods include harsh reaction conditions, use of toxic and corrosive reagents such as metal cyanides, expensive, exotic or commercially unavailable reagents.

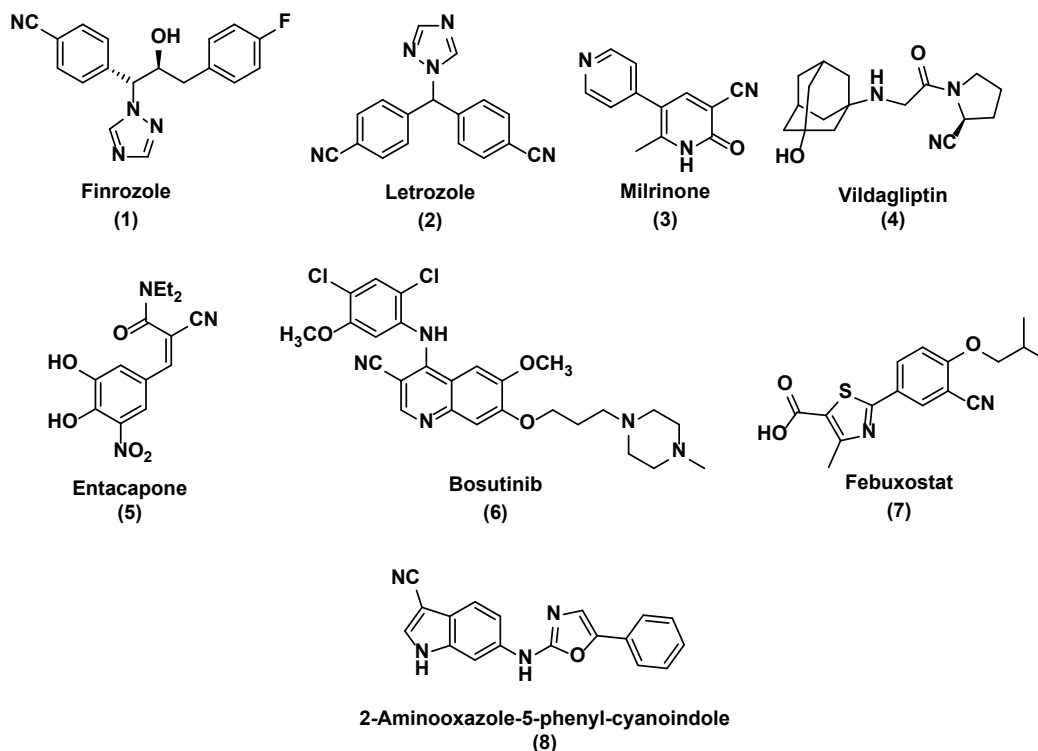


Figure 1. Structures of selected aryl nitriles containing drugs used for various ailments.

In these reactions organic solvents are used, and long reaction times that have shown to generate multiple products in low yields. They involve tedious work-up procedures to isolate and purify the nitrile. Thus, considerable efforts are directed towards developing efficient methods for the conversion of aldehydes to nitriles, or dehydration of amides and aldoximes to nitriles.

Recently, the microwave (MW) irradiation technique has been utilized as a powerful tool for the various organic transformations [25]. The main attained benefits are the significant enhancements of the reaction rates, yields, selectivity, and that the reaction can be done under heterogeneous conditions. These reactions resulted in almost complete conversion. In particular, microwave irradiation proved to be highly effective in promoting the condensation reactions [26-31]. Microwave was applied in nitrile formation with dehydrating agents such as peroxymonosulfate / alumina, sodium hydrogen sulphate / SiO₂, or HY-Zeolite, anhydrous Na₂SO₄ and anhydrous NaHCO₃ [32]. Most of these procedures employed a conventional kitchen microwave oven, in which temperature and pressure cannot be controlled, unlike the case of using standardized microwave reactor. In practice, the MW induced methods utilizing solid supports resulted in low reproducibility due to the heterogeneous nature of the reaction conditions. Furthermore, among the conventional or MW irradiation, only few methods are available to produce nitriles from aldehydes without the use of inorganic salts or solid supports. Acetic anhydride, which is a common and cheap dehydrating agent reported in the conversion of aldoximes to nitrile. However, the reaction time was more than 10 hours and used excessive amount of reagents [33]. The acid-sensitive functional groups such as ester were partly cleaved. In some instances, the hydration of nitriles to the corresponding primary amides was also observed [34]. Herein, we are reporting an improved microwave assisted and environmentally friendly way for the synthesis of substituted aryl nitriles, which are still in high demand.

2. Experimental

2.1. Instrumentation

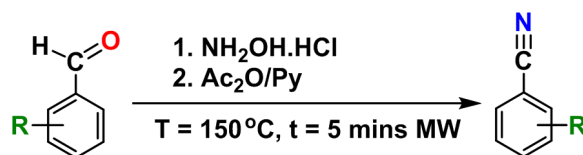
The reactions were carried out in a Biotage Initiator system (Biotage Sweden). The identity of the products is determined by FT-IR Perkin Elmer Spectrum BX Spectrometer and Bruker FTIR Spectrometer ALPHA (ATR for liquid samples). The ¹H and ¹³C NMR spectra were recorded on a Bruker AVANCE-400 spectrometer (Bruker BioSpin, Billerica, MA) operating at 400 and 100 MHz, respectively, using DMSO-*d*₆ as a solvent.

2.2. General procedure for the conversion of aromatic aldehydes to nitriles

The process is optimized using *m*-nitro benzaldehyde (*m*-NBA) as the model substrate. In this procedure, *m*-NBA (1 mmol) and hydroxylamine hydrochloride (1.5 mmol) were placed in a 10 mL microwave vial. To this mixture was added, pyridine (1 mmol), followed by Ac₂O (1.5 mmol). This mixture was stirred and well capped. The vial was placed in the microwave chamber. The reaction was kept under constant stirring using a magnetic stirrer at T = 150 °C for 5 minutes. The reaction progress and completion were monitored by TLC (eluent EtOAc:hexane, 1:4, v:v) and FT-IR Spectrometer. After completion of the reaction, cold water was added to the reaction mixture. The solid products were precipitated. The solids were dissolved in a minimal quantity of ether and dried over anhydrous Na₂SO₄ and kept for crystallization to give a yellow or off white crystals (~80% yield). In the case of liquid products, upon addition of cold water, liquids separated out as a layer. This was extracted with ether, and the ether extract is then washed with brine solution 2-3 times, and then dried over anhydrous Na₂SO₄.

Table 1. The temperature and time optimization for conversion of *m*-NBA to *m*-nitrobenzoxime.

Aldehyde	Time (mins)	Temperature (°C)	Reaction completion
<i>m</i> -NBA	5.0	50	Incomplete
	5.0	100	Incomplete
	5.0	120	Incomplete
	0.5	150	Incomplete
	3.0	180	Completed
	5.0	150	Completed

**Scheme 1.** Conversion of aldehydes to nitriles in a single-pot (R = -H, -NO₂, -OCH₃, -CH₃, -OH, -Cl, -Br).

Finally, the solvent was removed under vacuum using Heidolph Rotary Evaporator (Laborota 4000). All products were characterized by melting points, FT-IR, ¹H NMR, and ¹³C NMR.

3. Results and discussions

3.1. Optimisation of reaction condition in microwave reactor

The reaction is studied initially from room temperature (RT) to 180 °C as shown in Table 1. At 180 °C the reaction reaches complete conversion as monitored by FT-IR analysis. It was observed that the reaction is exothermic as indicated by the sudden rise in temperature from the set temperature when the samples were microwaved from 50 °C onwards. *m*-NBA is reported to take long reaction time [19] while here it took 10 minutes for completion. Other aldehydes took only 5 minutes. For generalization, in our method, aromatic aldehydes (Entries 1-16) were successfully converted to the corresponding nitriles along with formamides in cases of (Entries 1, 2, 3, 7, 8, and 12) under the optimized conditions as mentioned as general procedure under Scheme 1.

3.1.1. The products properties melting point, IR stretch and the ratio of nitrile to formamide

The reaction conditions and yields of the products are presented in Table 2. The NMR data clearly demonstrate the formation of the corresponding formamides obtained *via* Beckmann rearrangement to the extent of 25% conversion along with the nitrile as shown in Scheme 1. The nitrile is formed by acetic acid elimination reaction. The melting point, IR vibrational data and percent yields, as well as the ratio of nitrile to formamides as calculated from the ¹H NMR data are also given in Table 2. This method is applied to a range of aldehydes, *o*-, *m*-, and *p*-substituted aromatic monoaldehydes, dialdehydes, and heterocyclic aldehydes towards the corresponding nitriles in good yields. One advantage of this method over the conventional reaction is that in the case of *o*-, *m*-, *p*-substituted hydroxyl group the esterification of the hydroxyl group under acidic and basic conditions are preserved to give the *o*-, *m*-, and *p*-hydroxyl benzonitrile (Entries 9 and 10). The present method probably proceeds *via* initial formation of *o*-acetylaldoxime intermediates, formed *in situ* followed by acetic acid elimination under the influence of MW irradiation [35]. The detailed mechanism is depicted in Scheme 2.

The conventional method was tried for conversion of *m*-NBA to the nitrile in a two-step process. Firstly, the oxime of *m*-NBA is synthesized, followed by adding pyridine (1 mmol) and stirring at room temperature for 30 minutes. The reaction

progress was monitored by TLC, eluted with ethyl acetate:hexane (1:4, *v:v*) and FT-IR spectrometer. A white solid formed in the reaction mixture. The precipitate was filtered off, and washed with diethyl ether to remove excess starting materials. Secondly, the solid was converted to nitrile by adding acetic anhydride (1.5 mmol) and toluene (2 mL) as solvent in which the mixture is refluxed for 24 hours. The nitrile was not formed under these conditions as evidenced by FT-IR spectrometer. This shows the advantage of using MW over the conventional procedure.

3.2. Synthesis of heterocyclic aromatic nitriles

In the case of furfuraldehyde, an aromatic heterocyclic aldehyde, the reaction was performed under similar conditions, but a new product was obtained instead of the expected nitrile. This challenge was taken and done according to the following two steps:

3.2.1. Synthesis of furan aldoxime

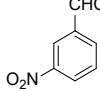

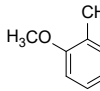

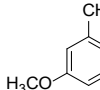

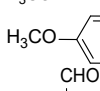

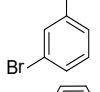

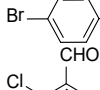

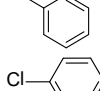

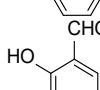

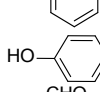

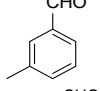

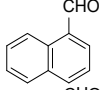

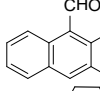

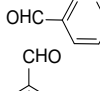

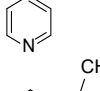

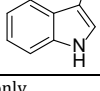
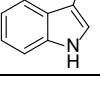
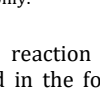
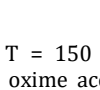
To furfuraldehyde (1 mmol) without further purification, NH₂OH.HCl (1.5 mmol) were added followed by pyridine (1 mmol) and stirred for 3 hours at room temperature until the NH₂OH.HCl is completely solubilized. The reaction mixture was then quenched in water where the furfuraldoxime precipitated. The solid was filtered off, and recrystallized from diethyl ether to give white needle like crystals (Yield = 74%). The product was characterized and confirmed by FT-IR spectrometer (which showed two peaks at 3166 and 1634 cm⁻¹), ¹H NMR and ¹³C NMR, as well as, melting point. The product was divided into three portions and used for synthesising furanonitrile under different thermal conditions.

3.2.2. Synthesis of furonitrile

To furanaldoxime (2.07 mmol), was added acetic anhydride (3.10 mmol) and the mixture was stirred for 3 hours at room temperature until the reaction mixture turned brown. The reaction mixture was then quenched in water where the separated out liquid was extracted with diethyl ether and washed with water to remove excess acetic acid. The ethereal solution was dried by flushing through anhydrous Na₂SO₄ and finally evaporated under vacuum resulting in a brown liquid (Yield: ~90%). The procedure was repeated under MW conditions as shown in Table 3.

The presence of the nitrile was confirmed by FT-IR spectrometer at 2231 cm⁻¹, along with oxime acetate as an intermediate which was confirmed by FT-IR peak at 1769 cm⁻¹ for oxime ester. The structure was further confirmed by ¹H- and ¹³C-NMR (given as supplementary material Figure 19, 20a, 20b and 20c).

Table 2. Aldehydes converted to nitriles under solvent free MW irradiation at T = 150 °C for 5 minutes.

Entry	Aldehyde	Nitrile	Melting point (°C)	CN stretch (v, cm ⁻¹)	Yield (%) and Nitrile: Amide ratio
1			114-116 [22]	2237 (2235) [13]	80 (95:5)
2			-	2229	82 (90:10)
3			20-25	2231	89 (78:22)
4			53-55 [22]	2219 (2227) [13]	86 *
5			36-38	2232	83 *
6			111-113	2225	72 *
7			Mixture	2231 (2214) [36]	94 (75:25)
8			Mixture	2225 (2226) [37]	58 (90:10)
9			76-83	2231 (2226) [22]	61 *
10			109-112	2234	55 *
11			-	2230 (2225) [13]	62 (Impure)
12			Mixture	2222 (2213) [38]	85 (75:25)
13			165-170	2212	31 *
14			216-221 [22]	2232 (2232) [22]	74 *
15			Mixture	2242	40 (Impure)
16			173-176	2224	76 *

* Nitrile only.

The reaction under MW at T = 150 °C for 5 minutes, resulted in the formation of the oxime acetate (FT-IR: 1760 and 1721 cm⁻¹) rather than the nitrile, showing that the elimination did not take place. This means the thermal stability of oxime ester renders it from elimination to give nitrile. The formation of o-acetyl ester is also confirmed *via* benzoyl chloride reaction with furfuraldoxime, to give pale white needle like crystals of o-benzoyl ester. The structure was determined by ¹H NMR, single crystal X-ray diffraction, showing 50% probability displacement ellipsoids (Figure 2). In addition, FT-IR spectrum shows the presence of a strong peak at 1736 cm⁻¹ along with sharp peaks in the range 3139 to 2863 cm⁻¹ corresponding to aromatic and aliphatic C-H stretching vibration, respectively. Similar reaction conversion

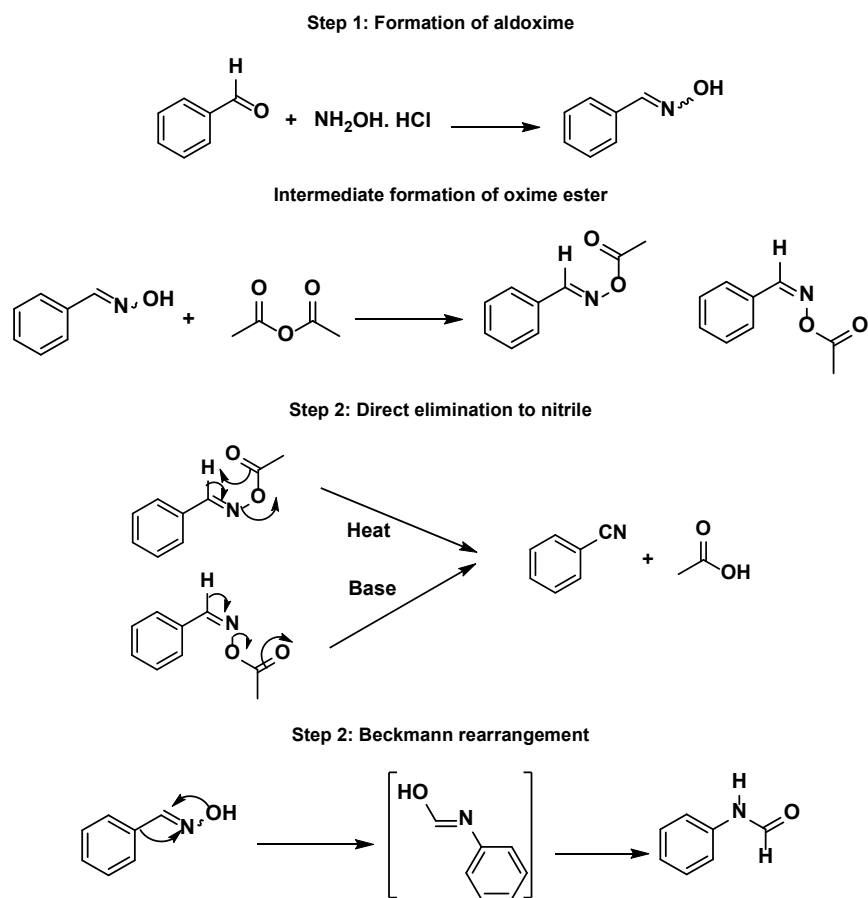
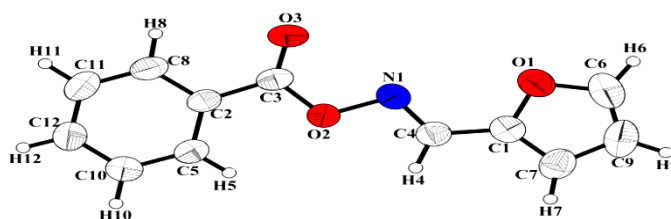
of aldehyde to nitrile through the oxime ester formation using o-benzoyl hydroxylamine has been reported [39].

3.2.3. Synthesis of o-benzoyl ester of furfuraldoxime

Benzoyl chloride (1 mmol) was added slowly (drop-wise) to furfuraldoxime (1 mmol). Since the reaction was vigorous and exothermic, the temperature was maintained at 0 °C in ice bath and stirred for 30 minutes. The reaction mixture was quenched using ice-water, then extracted with ethyl acetate. The organic layer was separated and then washed with NaOH (1 M) solution to remove any remaining benzoic acid and HCl by-products.

Table 3. Reaction conditions for MW-initiated synthesis of furanaldoxime acetate.

Time (Minutes)	Temperature (°C)	Yield (%)
10	100	60
5	150	55

**Scheme 2.** Schematic representation of the reactions mechanisms.**Figure 2.** View of the molecular structure of 2-furaldehyde oxime benzoate [40].

Ethyl acetate solution was dried over anhydrous Na_2SO_4 and evaporated under vacuum to get light brown solid recrystallized from ethanol:ethyl acetate binary solvents to give overall product; Yield: 50%, m.p.: 137-139 °C.

4. Conclusions

We have developed an environmentally benign MW-initiated method for direct synthesis of aryl nitriles from aromatic aldehydes using hydroxylamine hydrochloride and acetic anhydride. The method is simple and efficient which makes it an attractive alternative synthetic methodology towards different classes of aromatic, heterocyclic nitriles.

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Supporting information

Electronic supplementary information (ESI) available: Full experimental and characterization data.

Disclosure statement

Conflict of interests: The authors declare that they have no conflict of interest.

Author contributions: All authors contributed equally to this work.

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