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A NEW REAGENT FOR SYNTHESIS OF NITRILES FROM ALDOXIMES USING ALUMINIUM (III) TRIFLATE

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ABSTRACT. We report a new, highly efficient, and straightforward method for the dehydration of aldoximes to nitriles using aluminium (III) triflate [$Al(OTf)_3$] as a catalyst in acetonitrile. This one-step transformation proceeds with moderate to excellent yields (44–94%) and tolerates a wide range of substrates, including those with aromatic, functionalized aryl, heteroaryl, alkyl, and cycloalkyl moieties. This approach serves as a practical method for the synthesis of nitriles from aldoximes, yielding valuable synthetic intermediates for the pharmaceutical and agrochemical industries. In addition, the method's operational simplicity and the convenient isolation of the final product are its key advantages.

Keywords: *Aluminum triflate; Nitriles; Reaction mechanism; Catalysis; Dehydration*

INTRODUCTION

Compounds containing the nitrile derivatives are widely used industrially important in the fields of organic chemistry for production of agrochemicals, pharmaceuticals as well as material sciences [1-6]. Deoximation for the regeneration of nitriles from the corresponding oximes is still a worthwhile topic of study in organic chemistry [7-16]. Therefore, the versatily of the recent literature revealed that numerous reagents have been used for this purpose with different degrees of success. A number of efficient examples from literature include methyl N-(triethylammonium-sulfonyl)carbamate (Burges reagent) [17], $Cu(OAc)_2$/ultrasound [18], Pd/Mn [19], diethyl phosphorocyanidate

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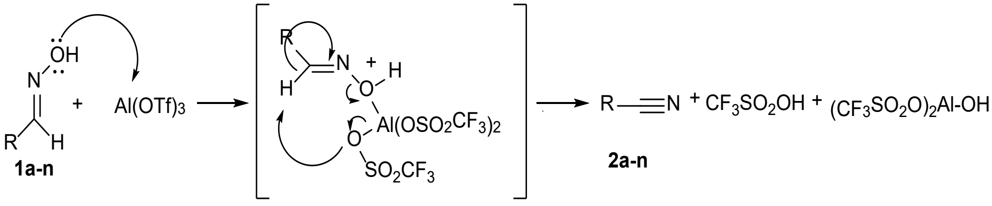
[20], Rosenmund-von Braun reaction [21], [$RuCl_2$(*p*-cymene)]$_2$/molecular sieves [22], $Ga(OTf)_3$ [23], Ceric ammononium nitrate (CAN) [24], Ph_3PO/ /$(COCl)_2$ [25], NCS/PPh_3 [26], Ac_2O/K_2CO_3/DMSO [27], and PEG-SO_3H [28] has also been used to carry out deoximation. Additionally, Recognizing the limitations of current aldoxime-to-nitrile conversion methods, including harsh conditions, low yields, lack of generality, and the use of costly or less accessible reagents, as well as high microwave power. To advance synthetic transformations and introduce a new perspective in organic chemistry, and the key compound, aldoximes, was prepared by following the literature for the deoximation reaction [29-32]. Firstly, according to our research, different aldehydes were used to make aldoximes with different structures and electron-withdrawing or electron-donating properties. For this purpose, aluminium (III) triflate was selected for preliminary studies, and the optimal conditions were determined to afford the corresponding nitrile.

Herein, Our aim was to develop a new method for the synthesis of nitriles from aldoximes using an aluminium (III) triflate-catalyzed dehydration reaction. and also work on understanding the mechanism of aluminium (III) triflate-mediated deoximation (Scheme 1).

Considering several advantages from this reaction, the development of a new method, good to excellent yields, and a wide scope of applicability, we report an investigation into the effect of substituents on the rate of deoximation using various derivatives bearing electron-withdrawing and electron-donating groups. In contrast to previous methods, which require in situ preparation or involve commercially less accessible reagents, the use of aluminum triflate afforded the desired products in moderate to good isolated yields for the first time in our study. Another advantage is that aluminum (III) triflate has been found to be a highly effective Lewis acid catalyst for the deoximation reaction.

RESULTS AND DISCUSSION

We discovered from our literature study that aluminum triflate has not been used in very much work for deoximation reaction. Previously, we reported that trifluoromethanesulfonic anhydride [15] and methoxymethyl bromide [16] are efficient and mild reagents for dehydration of aldoximes to nitriles under mild conditions. In continuation of our interests in developing a new method for conversion of aldoximes to nitriles, we herein wish to report the first application of aluminum triflate to the efficient synthesis of nitriles from aldoximes under efficient method as such a reagent. This study, therefore, examined the effectiveness of aluminium (III) triflate as a Lewis acid for the dehydration of aldoximes. At this point, our initial work commenced with screening of solvent so as to obtain optimal reaction condition. In order to obtain the scope and efficiency of the optimum reaction conditions, benzaldehyde oxime **1** was reacted with various solvents. The results are shown in Table 1. From the results in Table 1 it is evident that use of acetonitrile with aluminium (III) triflate led to the efficient conversion of aldoxime an in good yield 79% upon refluxing for 3 hours as model substrates for this reaction (Entry 9). Several other solvents were evaluated, including DMSO, CH_2Cl_2, THF, EtOH, DMF, MeOH, dioxane, and toluene, but were all found to be much less efficient than MeCN, and the results are summarized in Table 1. Thus, we observed that in the presence of aluminium (III) triflate, dehydration of aldoximes gave nitriles upon refluxing after 2-4 h. A wide range of organic aldoxime compounds, mainly aromatic, functionalized aryl, heteroaryl, alkyl, and cycloalkyl moieties, was used as starting materials and the starting oximes were synthesised according to the literature [30]. All reactions were checked by TLC, FT-IR, and MS analysis, and final products were determined by ^1H NMR and ^{13}C NMR. The spectral analyses of nitriles verified the cyano group with the appearance of specific peaks at around 2200 cm-1 (-C≡N). The procedure for obtaining benzonitrile **2a** was then successfully extended to aldoximes **1b-n** (Scheme 1). To further expand the scope of the process, we examined the reaction mechanism between benzaldoxime **1a** and aluminium (III) triflate. As expected, in the first stage, the OH group attacks aluminium, which is a Lewis acid, due to its high Lewis acidity. This efficiency arises from the triflate anion (-OTf), which is a very weak base and does not strongly interact with the Lewis acid Al^{+3} ion. The triflate anion (-OTf) formed as an anion removes the hydrogen bonded to the C=N bond, resulting in the formation of -CN. We also investigated how the temperature affected this reaction's result. The results revealed that increasing the temperature to refluxing led to an increase in the yield **2a** to 79% **(**Table 1, entry 9**).** Acetonitrile was identified as the optimal solvent for the conversion of aldoximes to the corresponding nitriles (Table 1). We conducted a series of test experiments in which **1a** was reacted with aluminium (III) triflate under different reaction conditions. On the other hand, the desired benzonitrile product **2a** was obtained in a 61% yield when the reaction was carried out with 1 equiv of **1a** and 1 equiv of aluminium (III) triflate at reflux in acetonitrile. In another experiment, using 1 equivalent of 1a and 2 equivalents of aluminum (III) triflate resulted in the formation of compound 2a with a yield of 79%. When using 1 equiv of **1a** and 3 equiv of aluminium (III) triflate under the same conditions, the reaction yield remained unchanged. Thus, the optimization studies showed that the best reaction conditions for the formation of **2a** were reflux in acetonitrile with 2 equiv of aluminium (III) triflate. All other aldoxime substrates were efficiently converted into the corresponding substituted nitriles in high yields using aluminium (III) triflate (Table 2). This transformation is tolerated for various substituents on the phenyl ring of the aldoxime substrates, including both electron-donating and electron-withdrawing groups (**1b-1h**). However, substitution with a 4-trifluoromethyl group resulted in a slightly lower yield because of its strong electron-withdrawing effect (Table 2, entry 5). In addition, aldoximes containing electron-donating groups such as 4-Me (**2f**) and 4-MeO (**2g**) provided their corresponding nitrile derivatives (**2f**, 92% and **2g,** 91%) in excellent yields. Furthermore, heteroatom-containing substrates (S, N, and O) afforded yields in the range of 53–64%. 1-Naphthonitrile was also synthesized in high yield (90%). Subsequently, alkyl and cycloalkyl aldoximes were smoothly converted into nitriles in good yields (Table 2, entries 9, 10).



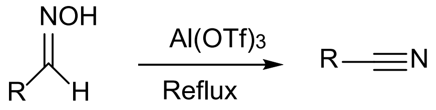
Scheme 1. Synthesis and mechanistic proposal for the conversion of aldoximes to nitriles

Table 1 Optimization of reaction conditions

Entry	Oxime	Solvent	T (^{0} C)	Yield **2a**
1	**1a**	DMSO	Reflux	63
2	**1a**	CH_2Cl_2	Reflux	41
3	**1a**	THF	Reflux	30
4	**1a**	EtOH	Reflux	65
5	**1a**	DMF	Reflux	61
6	**1a**	MeOH	Reflux	67
7	**1a**	MeCN	rt	10
8	**1a**	MeCN	50	55
9	**1a**	MeCN	Reflux	**79**
10	**1a**	Dioxane	Reflux	66
11	**1a**	Toluene	Reflux	54

Aluminum (III) triflate (2 mmol) and benzaldehyde oxime (**1a**, 1 mmol) were used in the reaction, which was conducted in a variety of solvents.

Table 2. Efficient conversion of aldoximes to nitriles using $Al(OTf)_3$



1a-n **2a-n**

R = aryl, heteroaryl, alkyl, cycloalkyl

Entry	R	Time(h)	Isolated yield (%)	m.p. (^{0}C)
1	C_6H_5-	3	79	Oil
2	4-FC_6H_4-	3	66	Oil
3	4-ClC_6H_4-	4	72	92-94 [19]
4	4-BrC_6H_4-	5	84	112-114 [19]
5	4-$CF_3C_6H_4$-	4	41	Oil
6	4-$CH_3C_6H_4$-	2	92	Oil
7	4-$CH_3OC_6H_4$	2	91	56-57 [19]
8	4-$NO_2C_6H_4$-	3	76	147-149 [38]
9	Cyclohexyl-	2	72	Oil
10	*n*-C_6H_{13}-	4	79	Oil
11	2-Pyridyl-	3	74	Oil
12	2-Furan-	2	52	Oil
13	2-Thiophene-	2	63	Oil
14	1-Naphtyl-	2	90	Oil

CONCLUSIONS

In conclusion, we have developed a highly efficient and straightforward method for the preparation of nitriles from various aldoximes. This aluminium (III) triflate-assisted dehydration protocol, performed in acetonitrile, effectively converts a broad range of substrates, including aromatic, functionalized aryl, heteroaryl, alkyl, and cycloalkyl aldoximes. The method's key advantages its operational simplicity, mild conditions, high yields, and short reaction time—make it a practical alternative for deoximation reactions, which are an important transformation in organic synthesis and synthetic industry. Thus, our new dehydration protocol could afford a practical method for synthesizing nitriles from various aldoximes.

EXPERIMENTAL SECTION

^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-400 MHz High-Performance Digital FT-NMR spectrometer, with operating frequencies of 400 MHz for ^{1}H and 101 MHz for ^{13}C. The compounds were dissolved in $CDCl_3$ using tetramethylsilane (TMS) as the internal standard. Infrared (IR) spectra were obtained on a 1000 FT-IR spectrometer using the KBr pellet technique. Mass spectra were recorded on a GEC-21 100B Finnigan Mat 1210 mass spectrometer. Thin-layer chromatography (TLC) was performed on silica gel 60 F254 plates. For purification, silica gel 60 (230-400 mesh, Merck) was used. All solvents were purified according to international standards.

Typical general procedure for the synthesis of 2a-n. To a solution of benzaldoxime **1a** (121.1 mg, 1.0 mmol) was dissolved in 30 mL of MeCN under an atmosphere of nitrogen, and then $Al(OTf)_3$ (948.4 mg, 2.0 mmol) was added in one portion. The resulting mixture was heated reflux for 2-4 hour. Reaction progress was monitored by thin-layer chromatography (TLC, ethyl acetate) to monitor the progress of the reaction. Then it was cooled, the solvent was removed and treated with a 10% aqueous solution of NaHSO4 (20 mL) and extracted with methylene chloride (2 × 30 mL). The combined organic extracts were washed with a saturated aqueous solution of NaCl, dried over anhydrous Na_2SO_4 and filtered. After the solvent was removed under reduced pressure, the crude oil product was purified by column chromatography on silica gel using ethyl acetate. The solvent was then evaporated to yield the corresponding nitrile **2a-n**.

Benzonitrile (2a) [19]. Oil, 81.3 mg, 79%. IR spectrum ν, cm^{-1}: 3071, 2265, 1488, 1451, 751; ^{1}H NMR ($CDCl_3$), δ: 7.42-7.48 (m, 2H, ArH), 7.56-7.61 (m,1H, ArH), 7.66-7.68 (m,2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 111.5, 117.9, 128.1, 131.2, 131.8; MS (EI, 70 eV): m/z (%) 103 [M$^+$] (100), 76 (30).

4-Fluorobenzonitrile (2b) [19]. Oil, 79.7 mg, 66%; IR spectrum ν, cm^{-1}: 3071, 2988, 2234, 1610, 1503, 1241, 848, 681, 542. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.09 (t, *J* = 8.3 Hz, 2H, ArH), 7.57-7.62 (m, 2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 106.5 (d, $J_{C\text{-}F}$ = 3.4 Hz), 114.8 (d, $J_{C\text{-}F}$ = 21.5 Hz), 116.0, 134.7 (d, $J_{C\text{-}F}$ = 9.3 Hz), 165.0 (d, $J_{C\text{-}F}$ = 251.9 Hz). MS (EI, 70 eV): m/z (%) 122 (11), 121 [M$^+$] (70), 105 (14).

4-Chlorobenzonitrile (2c) [33]. Crystallized from diethyl ether, mp 92-94°C (lit. 93-94 °C), 99.7 mg, 72%. IR spectrum ν, cm^{-1}: 3448, 3081, 2221, 1587, 1476, 1079, 831,540. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.43 (d, *J* = 8.1

Hz, 2H, ArH), 7.63(d, *J* = 8.4 Hz, 2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 111.8, 117.0, 128.7, 134.4, 138.6; MS (EI, 70 eV): m/z (%) 139 (28), 137 [M$^+$] (100), 101 (44).

4-Bromobenzonitrile (2d) [34]. Crystallized from diethyl ether, mp112-114 °C (lit. 113-114 °C), 153.0 mg, 84%. IR spectrum ν, cm^{-1}: 3272, 2222, 1474, 1065, 1010, 826, 542. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.51 (d, *J* = 8.5 Hz, 2H, ArH), 7.66 (d, *J* = 8.4 Hz, 2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 112.3, 117.1, 127.0, 131.7, 134.4; MS (EI, 70 eV): m/z (%) 183 (61), 181 [M$^+$] (55), 102 (100).

4-(Trifluoromethyl)benzonitrile (2e) [19]. Oil, 69.3 mg, 41%; %. IR spectrum ν, cm^{-1}: 3062, 2234, 1731, 1687, 1264, 1091, 1022, 814, 678. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.58 (d, *J* = 8.3 Hz, 2H, ArH), 7.71 (d, *J* = 8.3 Hz, 2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 114.0, 118.5, 122.1 (d, $J_{C\text{-}F}$ = 274.4 Hz), 127.2 (q, $J_{C\text{-}F}$ = 3.5 Hz), 133.7, 135.5 (d, $J_{C\text{-}F}$ = 32.3Hz); MS (EI, 70 eV): m/z (%) 172 [M$^+$+1] (11), 147 (18).

4-Methylbenzonitrile (2f) [34]. Oil, 108.3 mg, 92%. IR spectrum ν, cm^{-1}: 3052, 2981, 2221, 1611, 1173, 814, 535. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.68 (s, 3H, CH_3-), 7.29 (d, J = 8.3 Hz, 2H, ArH), 7.55 (d, J = 8.3 Hz, 2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 22.8, 108.3, 118.2, 127.9, 131.0, 144.7; MS (EI, 70 eV): m/z (%) 118 (11), 117 [M$^+$] (100), 116 (54).

4-Methoxybenzonitrile (2g) [33]. Crystallized from petroleum ether, mp 56-57 °C (lit. 55-57 °C), 121.2 mg, 91%. IR spectrum ν, cm^{-1}: 3014, 2988, 2211, 1611, 1505, 1261, 1168, 1017, 841, 678, 543. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.63 (d, *J* = 8.1 Hz, 2H, ArH), 6.93 (d, *J* = 9.1 Hz, 2H, ArH), 3.81 (s, 3H, CH_3O-). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 54.6, 103.0, 113.8, 118.3, 133.0, 163.9; MS (EI, 70 eV): m/z (%) 134 (23), 133 [M$^+$] (100), 103 (36).

4-Nitrobenzonitrile (2h) [35]. Crystallized from ether, mp 147–149 °C (Lit.149 °C), 112.2 mg, 76%; IR spectrum ν, cm^{-1}: 3052, 2967, 2922, 2851, 2227, 1731, 1681,1578, 1508, 1377, 1348, 1238, 1151, 1066, 1011, 963, 804, 771, 694, 583, 451. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.96 (d, *J* = 8.7 Hz, 2H, ArH), 8.32 (d, *J* = 8.6 Hz, 2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 115.8, 117.3, 123.2, 134.4, 151.0; MS (EI) m/z (%): 148 [M$^+$].

Cyclohexanecarbonitrile (2i) [36]. As a clear oil, 78.8 mg, 72%. IR spectrum ν, cm^{-1}: 2938, 2221, 1645, 1283, 1043. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.31-1.43 (m, 4H), 1.60-1.71 (m, 4H), 1.74-1.83 (m, 2H), 2.54-2.65 (m, 1H). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 23.0, 24.3, 27.9, 28.5, 123.6; MS (EI, 70 eV): m/z (%) 110 (32), 109 [M^{+}] (100), 68 (36).

Heptanenitrile (2j) [19]. Oil, 88.2 mg, 79%. IR spectrum ν, cm^{-1}: 2952, 2931, 2858, 2243, 1640, 1461, 1422, 1378, 1271. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.32 (t, *J* = 7.1 Hz, 2H), 1.62 (t, *J* = 7.4 Hz, 2H), 1.47 (t, *J* = 7.3 Hz, 2H), 1.31-1.36 (m, 4H), 0.93 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 14.9, 18.1, 23.3, 26.3, 29.3, 31.9, 118.8.

2-Pyridinecarbonitrile (2k) [37]. Oil, 77.2 mg, 74%. IR spectrum ν, cm^{-1}: 3051, 2923, 2234, 1681, 1644, 1579, 1461, 1431, 1358, 1288, 991, 780, 733, 554; ^{1}H NMR ($CDCl_3$), δ: 7.63 (s, 1H, ArH), 7.78 (s, 1H, ArH), 7.94 (s, 1H, ArH), 8.73(s, 1H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 116.2, 126.1, 127.6, 134.9, 136.2, 152.1; MS (EI, 70 eV): m/z (%) 104 (100) [M^{+}], 78 (5).

Furan-2-carbonitrile (2l) [19]. Oil, 48.5 mg, 52%. IR spectrum ν, cm^{-1}: 3191, 2244, 1258, 1093, 1021, 807, 753, 691. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 6.43-7.46 (m, 1H, ArH), 7.02 (d, *J* = 3.5 Hz, 1H, ArH), 7.54 (s, 1H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 111.4, 122.9, 124.4, 145.3. MS (EI, 70 eV): m/z (%), 93 [M^{+}] (5), 76 (7).

Thiophene-2-carbonitrile (2m) [19]. Oil, 68.6 mg, 63%. IR spectrum ν, cm^{-1}: 3028, 2221, 1441, 1380, 1265, 1070, 807, 778, 450. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.10-7.16 (m, 1H, ArH), 7.63-7.67 (m, 2H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 107.6, 113.2, 128.7, 134.7, 136.4. MS (EI, 70 eV): m/z (%) 109 [M^{+}] (100).

1-Naphthonitrile (2n) [24]. Oil,138.1 mg, 90%. IR spectrum ν, cm^{-1}: 3066, 2229, 1511, 1374, 1217, 810, 770. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 7.22-7.61 (m, 3H, ArH), 7.86-8.09 (m, 3H, ArH), 8.18 (t, *J* = 8.3 Hz, 1H, ArH). ^{13}C NMR spectrum, δ, ppm (*J*, Hz): 111.1, 116.8, 123.9, 126.0, 128.0, 129.6, 129.7, 133.3, 133.6, 133.9, 134.3. MS (EI, 70 eV): m/z (%), 154 (15), 153 [M^{+}] (100).

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