Chinese Chemical Letters xxx (2013) xxx–xxx

Contents lists available at ScienceDirect

Chinese Chemical Letters

journal homepage: www.elsevier.com/locate/cclet

Original article

An efficient, rapid and facile procedure for conversion of aldoximes to nitriles using triphenylphosphine and *N*-halo sulfonamides

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ARTICLE INFO

Article history:
Received 28 March 2013
Received in revised form 20 June 2013
Accepted 25 June 2013
Available online xxx

Keywords:
Aldoxime
Nitrile
N,N,N′,N′-Tetrabromobenzene-1,3-disulfonamide
N,N,N′,N′-Tetrachlorobenzene-1,3-disulfonamide
Triphenylphosphine (PPh_3)

ABSTRACT

N,N,N′,N′-Tetrabromobenzene-1,3-disulfonamide (TBBDA)/triphenylphosphine and *N,N,N′,N′*-tetrachlorobenzene-1,3-disulfonamide (TCBDA)/triphenylphosphine have been introduced as highly efficient systems for the versatile conversion of aldoxime derivatives into nitriles. The process reported here is operationally simple and reactions have been mildly performed in dichloromethane at room temperature.

1. Introduction

Nitriles are important precursors in organic synthesis, used as intermediates for the synthesis of esters, amides, carboxylic acids, amines, and nitrogen-containing heterocycles [1]. There are many reported procedures for the synthesis of nitriles, such as nucleophilic substitution of alkyl halides with metal cyanide for alkyl nitriles [2], and the Sandmeyer and ammoxidation reaction for aromatic nitriles (benzonitriles) [3]. Dehydration of aldoximes to nitriles is one of the cleanest routes, avoiding inorganic cyanides. Many methods have been used for the dehydration of aldoximes into nitriles; such as $Pd(OAc)_2/PPh_3$ in CH_3CN [4a], benzotriazole phosphonium hexafluorophosphate derivative/DBU in CH_2Cl_2 [4b], *N*-chlorosuccinimide/pyridine in CH_3CN [4c], DMF at 135 °C [4d], tungsten–tin mixed hydroxide in *o*-xylene at 149 °C [4e], diethylchlorophosphate in toluene [4f], molecular sieves under flash vacuum pyrolysis [4g], ZnO/CH_3COCl [4h], chlorosulfonic acid in toluene [4i], dimethylthiocarbonate/Et_3N in dioxane [4j], diethylchlorophosphite in $CHCl_3$ [4k], zeolite under microwave irradiation [4l], $AlCl_3{\cdot}6H_2O/KI/H_2O/CH_3CN$ [4m], Preyssler's anion, $[NaP_5W_{30}O_{110}]$ [4n], Burgess reagent [4o], thionyl chloride [4p], and PPh_3/NCS [4q], *N*-chlorosuccinimide/pyridine [4c], $[pmim]BF_4$ [4r], trichloroisocyanuric acid [4s], *N*-triflylimidazole [4t], triphenylphosphine oxide/oxalyl chloride [4u], MFR-H_2SO_4 [4v], as dehydrating agents. However, these procedures have some drawbacks, such as low yields, long reaction times, expensive reagents, heavy metal contaminations and harsh reaction conditions. Therefore, to improve the mentioned limitations, we decided to apply a new reaction media for the conversion of aldoximes to nitriles.

N,N,N′,N′-Tetrabromobenzene-1,3-disulfonamide (TBBDA) and *N,N,N′,N′*-tetrachlorobenzene-1,3-disulfonamide (TCBDA) are halogenating agents, and are effective catalysts and reagents for various organic transformations [5]. Since TCBDA and TBBDA contain bromine and chlorine atoms which are attached to the nitrogen atoms, it is very probable that they release *in situ* Br^+ and Cl^+, which can act as an electrophilic species, and interaction of Ph_3P with TBBDA or TCBDA would generate phosphonium halides as the reactive phosphonium species.

2. Experimental

2.1. Typical procedure for the conversion of 4-nitrobenzaldehyde oxime to 4-nitrobenzonitrile with TCBDA and PPh_3

To a mixture of PPh_3 (0.315 g, 1.2 mmol) and TCBDA (0.12 g, 0.32 mmol) in dichloromethane (5 mL), 4-nitrobenzaldehyde oxime (0.166 g, 1 mmol) was added. The mixture was stirred at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction (Table 1), the solvent was evaporated. The crude products were purified by short-column

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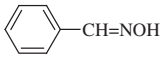
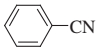
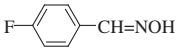
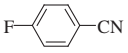
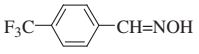
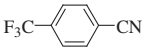
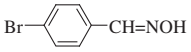
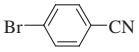
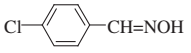
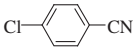
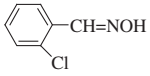
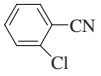
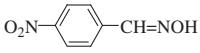
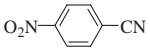
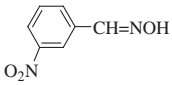
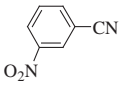
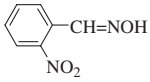
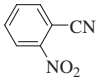
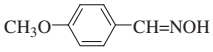
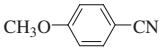
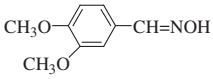
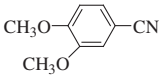
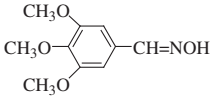
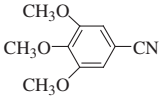
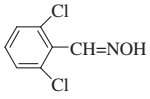
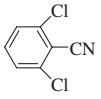
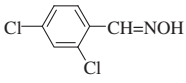
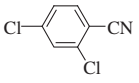
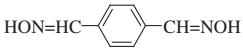
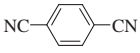
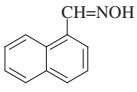
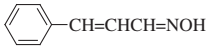
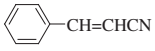
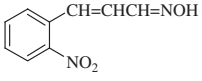
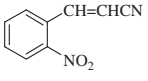
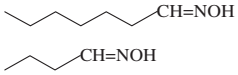
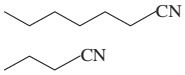
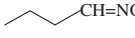
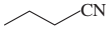
http://dx.doi.org/10.1016/j.cclet.2013.07.020

Table 1
Dehydration of the aldoximes to the corresponding nitriles with TCBDA and TBBDA.[a]

Entry	Substrate	Product[b]	Yield (%)[d]		Reference
			TBBDA[c]	TCBDA[c]	
1			95	90	[7a]
2			89	92	[7b]
3			90	93	[7b]
4			92	91	[7c]
5			96	94	[7d]
6			90	91	[7d]
7			92	95	[7d]
8			95	92	[7d]
9			94	90	[7e]
10			91	93	[7a]
11			93	95	[7f]
12			94	90	[7g]
13			96	90	[7h]
14			90	90	–
15[e]			97	94	[7i]
16			94	91	[7f]
17			90	85	[7j]
18			94	90	–
19			88	83	[7k]
20			80	82	[7k]

[a] The reaction time with TCBDA/PPh_3 is immediately and with TBBDA/PPh_3 is 10 min.
[b] Product were characterized from their physical properties, by comparison with authentic samples, and by spectroscopic methods.
[c] The molar ratio for TCBDA/PPh_3 is (0.32/1.2) and for TBBDA/PPh_3 is (0.53/2).
[d] Isolated yield.
[e] Double amount of molar ratio TCBDA/PPh_3 and TBBDA/PPh_3 were used.

Please cite this article in press as: R. Ghorbani-Vaghei, et al., An efficient, rapid and facile procedure for conversion of aldoximes to nitriles using triphenylphosphine and *N*-halo sulfonamides, Chin. Chem. Lett. (2013), http://dx.doi.org/10.1016/j.cclet.2013.07.020

chromatography (packed with silica gel, using *n*-hexane/ethyl acetate (8:2) as eluent) to achieve the desired 4-nitrobenzonitrile with 0.14 g, 95% yield.

2.2. Typical procedure for the conversion of 4-nitrobenzaldehyde oxime to 4-nitrobenzonitrile with TBBDA and PPh_3

To the mixture of PPh_3 (0.525 g, 2 mmol) and TBBDA (0.287 g, 0.53 mmol) in dry acetonitrile (5 mL), 4-nitrobenzaldehyde oxime (0.166 g, 1 mmol) was added. The mixture was stirred at room temperature. The progress of the reaction was monitored by TLC. After completion of the reaction (Table 1), the solvent was evaporated. The crude products were purified by short-column chromatography (packed with silica gel, using *n*-hexane/ethyl acetate (8:2) as eluent) to achieve the desired 4-nitrobenzonitrile with 0.13 g, 92% yield.

4-Nitrobenzonitrile (Table 1, entry 7): mp 143–147 °C: IR (KBr, cm^{-1}): 3106, 2233, 1602, 1525, 1349; ^1H NMR (400 MHz, $CDCl_3$): δ 8.39 (d, 2H, J = 8.8 Hz), 7.92 (d, 2H, J = 8.8 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 150.06, 133.50, 124.32, 118.36, 116.82.

3-Nitrobenzonitrile (Table 1, entry 8): mp 114–117 °C: IR (KBr, cm^{-1}): 3110, 2232, 1609, 1525, 1344; ^1H NMR (400 MHz, $CDCl_3$): δ 8.58 (s, 1H), 8.52 (d, 1H, J = 9.6 Hz), 8.03 (d, 1H, J = 8 Hz), 7.78 (t, 1H, J = 8 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.27, 137.61, 130.68, 127.56, 127.27, 116.55, 114.18.

2-Nitrobenzonitrile (Table 1, entry 9): mp 105–109 °C: IR (KBr, cm^{-1}): 3112, 2232, 1613, 1530, 1344; ^1H NMR (400 MHz, $CDCl_3$): δ 8.39 (m, 1H), 7.96 (m, 1H), 7.88 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.68, 135.63, 134.31, 133.70, 125.60, 114.95, 108.17.

4-Methoxybenzonitrile (Table 1, entry 10): mp 57–59 °C: IR (KBr, cm^{-1}): 3086, 2972, 2224, 1599; ^1H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, 2H, J = 8.8 Hz), 6.98 (d, 2H, J = 8.8 Hz), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.86, 134.02, 119.27, 114.76, 103.98, 55.57.

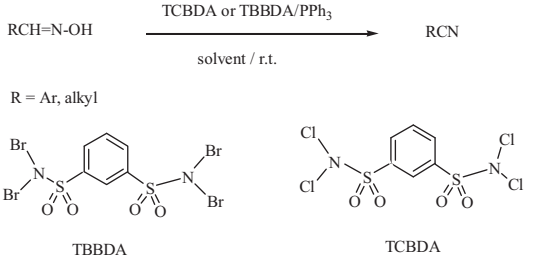
2,6-Dichlorobenzonitrile (Table 1, entry 13): mp 142–144 °C: IR (KBr, cm^{-1}): 3092, 2233, 1572, 802; ^1H NMR (400 MHz, $CDCl_3$): δ 7.53–7.45 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.55, 133.86, 128.18, 114.47, 113.39.

2,4-Dichlorobenzonitrile (Table 1, entry 14): mp 56–58 °C: IR (KBr, cm^{-1}): 3066, 2233, 1581, 821; ^1H NMR (400 MHz, $CDCl_3$): δ 7.65 (d, 1H, J = 8.4 Hz), 7.58 (d, 1H, J = 2 Hz), 7.41 (d, 1H, J = 8.4 Hz). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.13, 137.86, 134.61, 130.31, 127.88, 115.27, 111.91.

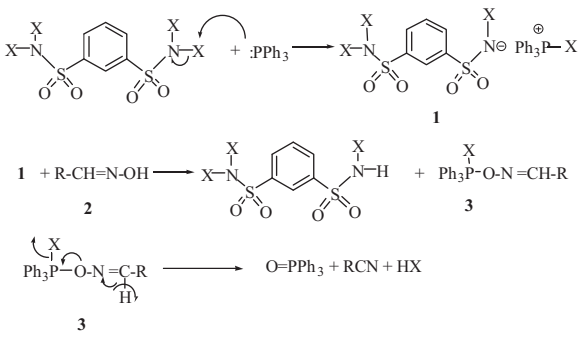
2-Nitrocinnamonitrile (Table 1, entry 18): mp 87–90 °C: IR (KBr, cm^{-1}): 3075, 2226, 1605, 1568, 1523, 1342; ^1H NMR (400 MHz, $CDCl_3$): δ 8.16 (d, 1H, J = 9.2 Hz), 8.0 (d, 1H, J = 17.6 Hz), 7.77–7.60 (m, 3H), 5.89 (d, 1H, J = 16.4 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz): δ 147.48, 146.68, 134.09, 131.36, 129.68, 128.75, 125.36, 116.96, 101.55.

3. Results and discussion

In continuation of our interest in the application of TBBDA and TCBDA, in organic synthesis [5], herein we report a simple and improved protocol for the preparation of nitriles from aldoximes.



Scheme 1. Synthesis of nitriles from aldoximes.



Scheme 2. Proposed mechanism for the preparation of nitriles from aldoximes.

The reactions proceeded *via* combination of TBBDA or TCBDA with Ph_3P in solvent at room temperature under mild conditions (Scheme 1).

In order to optimize the reaction conditions, we first examined the effect of different molar ratios of TBBDA/triphenylphosphine in CH_3CN or TCBDA/triphenylphosphine in CH_2Cl_2 at room temperature for the conversion of 4-chlorobenzaldehye oxime to 4-chlorobenzonitrile as a model reaction. We found that the optimized molar ratio for transformation was 1/0.32/1.2 (4-chlorobenzaldehye oxime/TCBDA/PPh_3) and 1/0.53/2 (4-chlorobenzaldehye oxime/TBBDA/PPh_3). This method is general and can be easily applied for the conversion of aldoximes to their corresponding nitriles using the optimized molar ratios of TCBDA and/or TBBDA and Ph_3P (Table 1).

Dehydration reactions of aldoxime with TBBDA and TCBDA do not proceed in the absence of PPh_3. Triphenylphosphine is a relatively general reducing agent, and its reactions with *N*-halo compounds can lead to the formation of phosphonium intermediates. The phosphorus has positive charge in these intermediates, so its reaction as a strong oxophilic reagent in most cases is driven to the formation of thermodynamically preferred triphenylphosphine oxide. Enthalpies of formation of triphenylphosphine and triphenylphosphine oxide are $\Delta_f H^0$ = +207.02 kJ/mol and $\Delta_f H^0$ = −116.41 kJ/mol, respectively [6]. As such, the formation of $Ph_3P{=}O$ is the major driving force in the proposed mechanism (Scheme 2).

In this potential pathway, triphenylphosphine (PPh_3) attacks *N*-halosufonamide compounds, and leads to intermediate **1** in which the phosphorus atom is more electrophilic. Then, nucleophilic attacks of aldoxime **2** on this intermediate give compound **3**. Finally, intermediate **3** can be converted to the product *via* formation of triphenylphosphine oxide and HX.

4. Conclusion

In conclusion, TBBDA/Ph_3P and TCBDA/Ph_3P are mild and efficient reagent systems for the conversion of aldoximes into nitriles. Simple work-up, high yields, short reaction times and mild reaction temperature could also be considered advantages of this methodology.

Acknowledgment

We are thankful to Bu-Ali Sina University, Center of Excellence and Development of Chemical Methods (CEDCM) for financial support.

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Please cite this article in press as: R. Ghorbani-Vaghei, et al., An efficient, rapid and facile procedure for conversion of aldoximes to nitriles using triphenylphosphine and *N*-halo sulfonamides, Chin. Chem. Lett. (2013), http://dx.doi.org/10.1016/j.cclet.2013.07.020