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OPPI BRIEF

One-Pot Synthesis of Bicyclic *ortho*-Aminocarbonitrile and Multisubstituted Cyclohexa-1,3-dienamine Derivatives

Behrooz Maleki, Roghaie Rooky, Esmail Rezaei-Seresht, and Reza Tayebee

Department of Chemistry, Hakim Sabzevari University, Sabzevar, 96179-76487, Iran

Creation of molecular functionality and diversity[1] from common starting materials while mindful of economic and environmental considerations constitutes a great challenge in modern organic chemistry.[2–4] Multi-component reactions (MCRs) are the best agents for reaching this ideal goal. MCRs are convergent reactions, in which three or more starting materials react to form a product, where all or most of the atoms contribute to the newly formed product. The major advantages of MCRs include lower cost, shorter reaction time, high atom-economy, energy saving, and the avoidance of time consuming and expensive purification processes.[5–8]

Derivatives of bicyclic *ortho*-aminocarbonitrile and multisubstituted cyclohexa-1, 3-dienamine are considered important organic intermediates[9-10] due to their applications in the synthesis of heterocyclic compounds.[11] Moreover, these compounds are useful precursors for the preparation of their respective dicyanoanilines[12] which are significant for their optical properties.[13]

These features make the preparation of bicyclic ortho-aminocarbonitriles and multisubstituted cyclohexa-1,3-dienamines very attractive to synthetic chemists, and consequently several methods have been introduced in recent years for their synthesis.[14-20] One of the simplest and most general methods involves the condensation of aldehydes (1 equiv.) and malononitrile (2 equiv.) with ketones (1 equiv.) under a variety of conditions. This reaction has been carried out using catalysts or reagents such as 1-methylpiperidin-4-one,[21] *N*-butylpyridinium tetrafluoroborate [BPy]BF_4,[22] ammonium acetate,[23] imidazole,[24] Borax,[25] ethanediamine,[26] and β-1-imidazol-2,3,4,6-tetraacetyl-D-glucopyranosyl bromide ($[Bmim\text{-}G]^+[Br]^-$).[27] However, many of these catalysts have disadvantages, such as low yields, the need to activate the catalyst prior to use, the use of commercially unavailable reactants or catalysts, harsh conditions, tedious work-up procedures, the need to use high catalyst loading, and multistep procedures.

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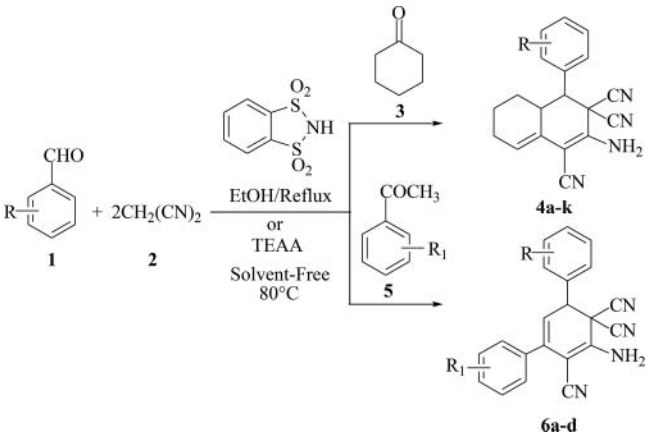
Address correspondence to Behrooz Maleki, Department of Chemistry, Hakim Sabzevari University, Sabzevar, 96179-76487, Iran. E-mail: malekibehrooz@gmail.com

Considering the importance of these compounds, and as a continuation of our development of efficient environmentally benign catalysts,[28-36] we now report an efficient, eco-



Scheme 1

friendly, safe, green, and practical method for the synthesis of bicyclic *ortho*-aminocarbonitrile and multisubstituted cyclohexa-1,3-dienamine derivatives using *ortho*-benzenedisulfonimide (OBS) and triethylammonium acetate ($Et_3NH^+OAc^-$) as catalysts (*Scheme 1*).

We have recently reported the use of catalytic amounts of OBS as a safe, nonvolatile, and noncorrosive Brønsted acid in several acid-catalyzed organic reactions that gave excellent results.[37–38]

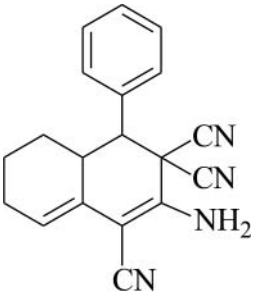
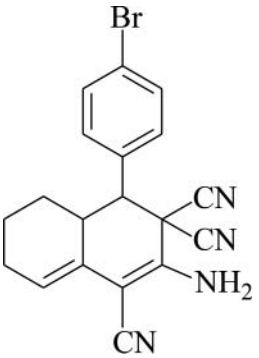
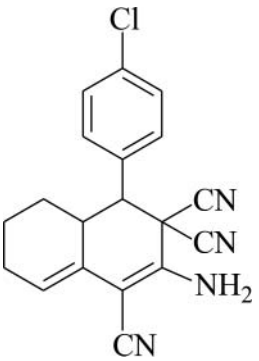
In general, all synthetic methods should have mild reaction conditions, short reaction times, good selectivity and the absence of by-products. It is worthwhile to highlight the further valuable aspect of the above reactions, namely the fact that OBS can easily be recovered from the reaction mixtures, in good to high yield, due to its complete solubility in water. This permits its reuse in catalytic amounts in other reactions, either directly or after a fast purification run on a cation-exchange resin, without the loss of catalytic activity. This obviously has economic and ecological advantages.[39–41]

In order to optimize the reaction conditions and obtain the best catalytic activity, the reaction of benzaldehyde (1 mmol), malononitrile (2 mmol), and cyclohexanone (1 mmol) was used as a model and conducted with varying reaction parameters such as solvent, temperature and amount of catalyst. The best results were obtained using 25 mol% of OBS as catalyst under reflux conditions in ethanol.

A series of bicyclic *ortho*-aminocarbonitrile derivatives was prepared successfully from different aromatic aldehydes bearing electron-withdrawing and electron-donating groups, cyclohexanone or aromatic ketones and malononitrile at 80°C. In all cases, the corresponding products **4a-k, 6a-d** were obtained in good to excellent yields (*Table 1*).

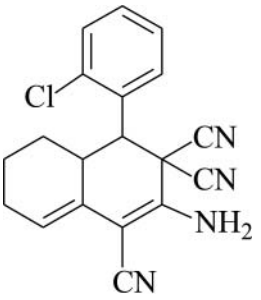
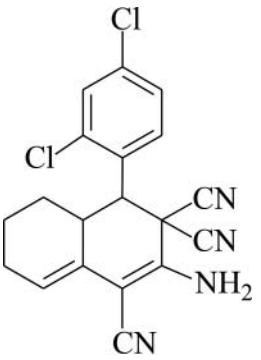
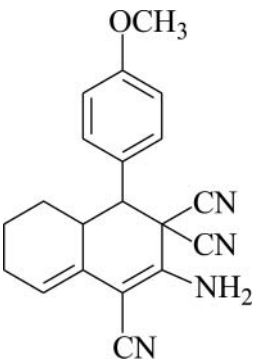
Due to the increase in environmental consciousness in chemical research and industry, the challenge for sustainable chemistry calls for clean procedures that avoid the use of harmful organic solvents. In recent years, ionic liquids (ILs) have attracted increasing interest and been successfully used in a variety of reactions as environmentally benign solvents and catalysts due to their relatively low viscosity and vapor pressure, high thermal and chemical stability, high conductivity, wide electrochemical window and the high solubility of inorganic, organic, polymeric materials and gases. Much attention is currently being focused on organic reactions with ILs as catalysts or solvents, and many chemical reactions have been performed in ILs

Table 1
Synthesis of Bicyclic *ortho*-Aminocarbonitrile and Multisubstituted Cyclohexa-1,3-dienamine Derivatives in the Presence of OBS and TEAA

Entry	Products **(4 a-k, 6 a-d)**	Yield[a] (%) **I-II**	Time (h) **I-II**	mp (°C) Found	mp (°C) Lit.
4 a		93–93	6-3	255–257	255–257[22]
4b		90–91	5-4	265–267	265–268[22]
4 c		90–88	5–4	244–246	248–250[22]

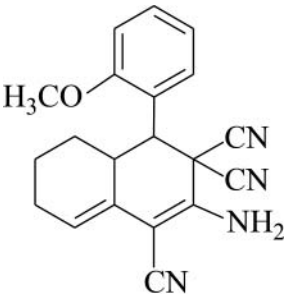
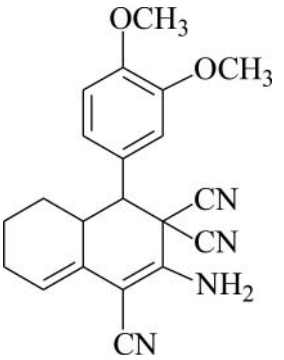
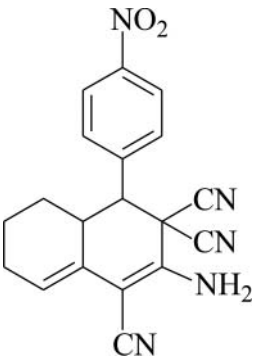
(*Continued on next page*)

Table 1
Synthesis of Bicyclic *ortho*-Aminocarbonitrile and Multisubstituted Cyclohexa-1,3-dienamine Derivatives in the Presence of OBS and TEAA *(Continued)*

Entry	Products **(4 a-k, 6 a-d)**	Yield[a] (%) **I-II**	Time (h) **I-II**	mp (°C) Found	mp (°C) Lit.
4 d		82–84	7–6	266–268	271–272[22]
4e		92–94	5-4	262-264	257-260[22]
4f		92–90	5-4	255–257	261–262[22]

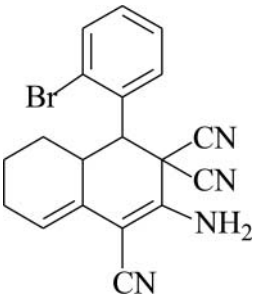
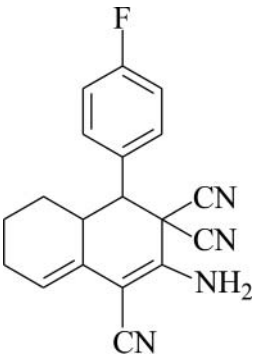
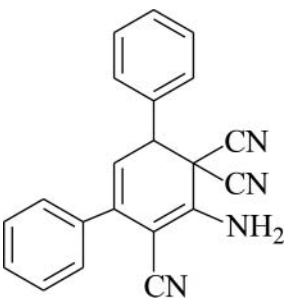
(*Continued on next page*)

Table 1
Synthesis of Bicyclic *ortho*-Aminocarbonitrile and Multisubstituted Cyclohexa-1,3-dienamine Derivatives in the Presence of OBS and TEAA *(Continued)*

Entry	Products **(4 a-k, 6 a-d)**	Yield[a] (%) **I-II**	Time (h) **I-II**	mp (°C) Found	mp (°C) Lit.
4 g		86–88	6-3	265–266	264–265[22]
4 h		82–90	7-5	250–252	254–255[22]
4i		90–90	6-3	264-266	266-268[18]

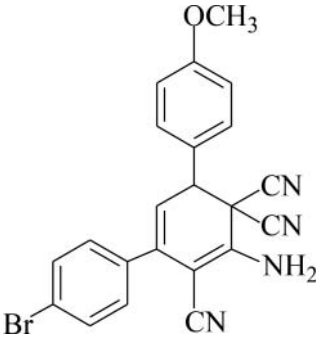
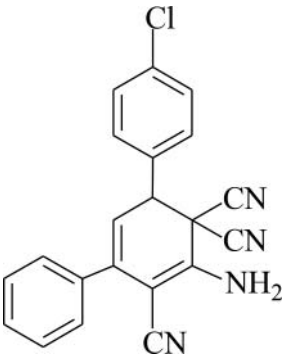
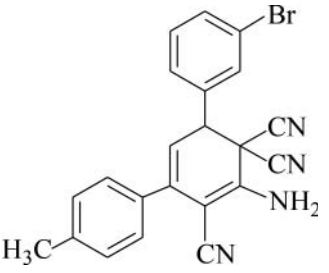
(*Continued on next page*)

Table 1
Synthesis of Bicyclic *ortho*-Aminocarbonitrile and Multisubstituted Cyclohexa-1,3-dienamine Derivatives in the Presence of OBS and TEAA *(Continued)*

Entry	Products **(4 a-k, 6 a-d)**	Yield[a] (%) **I-II**	Time (h) **I-II**	mp (°C) Found	mp (°C) Lit.
4j		80–83	7-6	239–241	245–248[22]
4 k		85–89	5–4	262–264	265–267[22]
6 a		86–90	6–5	156–158	158–160[26]

(*Continued on next page*)

Table 1
Synthesis of Bicyclic *ortho*-Aminocarbonitrile and Multisubstituted Cyclohexa-1,3-dienamine Derivatives in the Presence of OBS and TEAA *(Continued)*

Entry	Products **(4 a-k, 6 a-d)**	Yield[a] (%) **I-II**	Time (h) **I-II**	mp (°C) Found	mp (°C) Lit.
6b		90–92	7–5	252–254	253–255[24]
6 c		88–90	7–6	245–247	246–248[24]
6 d		89–91	6–5	202–204	205–209[26]

[a]The yields and times by OBS (**I**), and TEAA (**II**).

with excellent outcomes.[42–49] In continuation of our recent investigations on the synthesis and applications of the ionic liquids,[50–51] we now report that triethylammonium acetate (TEAA) is a green catalyst for the synthesis of bicyclic *ortho*-aminocarbonitriles (*Scheme 1*).

To check the suitability of TEAA for this MCR, initially the reaction of benzaldehyde (1 mmol), malononitrile (2 mmol), and cyclohexanone (1 mmol) was tested in the presence of TEAA (0.1 ml) under solvent–free conditions at 80°C. Interestingly, in this case we also

achieved a 93% yield of **4a** within 3 h. Then, different reaction parameters such as solvent, temperature and amount of catalyst were investigated. Also, in comparison with TEAA, the use of Et_3NH^+ $HCOO^-$ (TEAF), Et_3NH^+ $HSO4^-$ (TEAS), Et_3NH^+ HNO_3^- (TEAN), Et_3NH^+ $H_2PO_4^-$ (TEAP), and Et_3NH^+ $ClSO_3^-$ (TEAClS) as catalysts afforded lower yields in the model reaction under solvent-free conditions at 80°C. This investigation showed that TEAA was the most efficient catalyst in terms of the yield of **4a**.

Further, to show the generality and versatility of TEAA as a catalyst, this protocol was extended to the synthesis of bicyclic *ortho*-aminocarbonitrile derivatives (**4a-k**) and multisubstituted cyclohexa-1,3-dienamines (**6a-d**) by a one-pot reaction of benzaldehyde (1 mmol), malononitrile (2 mmol), and cyclohexanone or aromatic ketones (1 mmol) in the presence of TEAA (0.1 ml) under solvent–free conditions at 80°C (*Table 1*).

The recycling of OBS was investigated using the synthesis of **4a**. The catalyst was recovered by evaporation of the combined aqueous layer and aqueous washings under reduced pressure. The residue was passed through a column of acidic Dowex AG 50 W-X8 ion-exchange resin (100–200 mesh, 160 mg resin/500 mg product) with water as eluent. After removal of the water under reduced pressure, the pure OBS was recovered as a white solid; mp 192–193°C (lit.[38] 192–193°C). It was re-used in the model reactions to give **4a** in yields of 93%, 89%, and 88%, for three consecutive runs.

The re-usability of the TEAA was also evaluated by performing the model reaction under the optimized conditions. After the completion of the reaction, the catalyst was recovered and could be re-used three times without any substantial decrease in the yield of product (93, 90, and 89%) for three consecutive runs.

In conclusion, efficient, simple, and safe procedures for the preparation of the *ortho*-aminocarbonitriles and multisubstituted cyclohexa-1,3-dienamines were reported in this work. This protocol has the advantages of including high yields, ready availability, lower cost of the catalyst, operational simplicity, and minimization of cost and waste generation owing to recycling of the catalyst. The reagents are non-volatile and non-corrosive. Column chromatographic separation is not required.

Experimental Section

All reagents were obtained from commercial sources and were used without purification. The ionic liquids TEAA, TEAF, TEAS, TEAN, TEAP, and TEAClS were prepared according to our previous procedure.[51] IR spectra were recorded as KBr pellets on a Shimadzu 435-U-04 spectrophotometer. ^1H and ^{13}C NMR spectra were determined on a Bruker DRX-300 Avance spectrometer in DMSO-d_6, and shifts are given in δ downfield from tetramethylsilane (TMS) as an internal standard. Melting points were determined using an Electrothermal 9200 apparatus and are uncorrected.

General Procedure for the Synthesis of Compounds 4a-k, 6a-d using OBS

o-Benzenedisulfonimide (0.055 g, 0.25 mmol), was added to a mixture of the aldehydes (1 mmol), malononitrile (0.14 g, 2 mmol), cyclohexanone or aromatic ketones (1 mmol) and 2 mL of ethanol in a 10 mL flask fitted with a reflux condenser. The resulting mixture was heated to reflux (oil bath) for the appropriate time (*see Table 1*) with stirring. After completion of the reaction as determined by TLC (silica gel; hexane-ethyl acetate, 4:1),

the mixture was poured onto crushed ice and the solid product that separated out was collected, washed with water, dried and recrystallized from ethanol (96%, 10 ml) to afford the pure compound **4a-k, 6a-d**.

General Procedure for the Synthesis of Compounds 4a-k, 6a-d using TEAA

TEAA (0.1 ml), was added to a mixture of the aldehydes (1 mmol), malononitrile (0.14 g, 2 mmol) and cyclohexanone or aromatic ketones (1 mmol) in a 10 mL flask at room temperature. The temperature was then raised to 80°C and maintained for the appropriate time (*see Table 1*) with stirring. After completion of the reaction as determined by TLC (silica gel; hexane-ethyl acetate, 4:1), hot 96% EtOH (2 ml) was added and the mixture was stirred for 2 min. It was then poured onto crushed ice and the solid product that separated out was collected, washed with water, dried and recrystallized from ethanol (96%, 6 ml) to afford the pure compounds **4a-k, 6a-d**.

To recover the catalyst, the filtrate from above [(containing the catalyst (TEAA)] was extracted with CH_2Cl_2 (5 ml) to remove non-ionic organic impurities. Then the water was evaporated from the aqueous phase and the residue was dried at 60°C under reduced pressure for 2 h and re-used it for subsequent reactions.

Analytical Data for Selected Compounds

*2-Amino-4-(phenyl)-4 a,5,6,7-tetrahydronaphthalene-1,3,3-(4 H)-tricarbonitrile (**4a**).* IR (KBr): 3420, 3340, 3250, 3007, 2950, 2870, 2830, 2215, 1640, 1590, 1480, 1440, 1430, 1410, 1390, 1340, 1310, 1270, 1210, 1160, 950; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.64-0.82 (m, 1 H, CH), 1.34-1.42 (m, 2 H, CH_2), 1.60-1.82 (m, 1 H, CH), 1.92-2.25 (m, 2 H, CH_2), 2.89 (m, 1 H, CH), 3.54 (d, 1 H, CH), 5.76 (s, 1 H, CH), 7.28-7.89 (m, 7 H, NH_2 and ArH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 21.36, 24.67, 27.56, 34.28, 43.08, 51.34, 81.96, 112.97, 117.89, 121.84, 126.78, 129.07, 133.56, 136.01, 143.92.

*2-Amino-4-(4-nitrophenyl)-4 a,5,6,7-tetrahydronaphthalene-1,3,3-(4 H)-tricarbonitrile (**4i**).* IR (KBr): 3480, 3350, 3240, 3002, 2900, 2890, 2240, 1670, 1600, 1530, 1450, 1400, 1390, 1260, 1220, 1160, 880; ^{1}H NMR (300 MHz, DMSO-d_6): δ 0.81–0.92 (m, 1 H, CH), 1.42–1.51 (m, 2 H, CH_2), 1.68–1.74 (m, 1 H, CH), 2.12–2.27 (m, 2 H, CH_2), 2.79–2.88 (m, 1 H, CH), 3.68 (d, 1 H, CH), 5.89 (s, 1 H, CH), 7.23 (d, 1 H, ArH), 7.39–7.41 (m, 3 H, NH_2 and ArH), 7.82–7.88 (m, 2 H, ArH); ^{13}C NMR (75 MHz, DMSO-d_6): δ 22.67, 25.09, 27.98, 34.69, 45.87, 51.66, 82.81, 113.07, 117.88, 122.09, 128.08, 129.86, 133.86, 136.75, 145.02.

*2-Amino-6-(4-chlorophenyl)-4-phenylcyclohexa-2,4-diene-1,1,3-tricarbonitrile (**6c**).* IR (KBr): 3360, 3340, 3210, 3070, 3050, 2220, 2215, 1660, 1610, 1550, 1530, 1490, 1440, 1400, 1310, 1215, 1090, 1010, 970; ^{1}H NMR (300 MHz, DMSO-d_6): δ 6.49 (d, 1 H), 7.25 (d, 2 H), 7.42–7.56 (m, 1 H), 7.65 (d, 2 H), 8.45 (s, 1 H), 9.13 (s, 1 H); ^{13}C NMR (75 MHz, DMSO-d_6): δ 44.12, 48.09, 81.94, 110.45, 112.87, 114.76, 115.90, 116.07, 116.82, 129.32, 129.41, 130.04, 132.56, 133.18, 135.09, 145.12, 163.34.

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