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One-Pot Facile Synthesis of Acridine Derivatives under Solvent-Free Condition

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N,N'-Dibromo-*N,N'*-1,2-ethanediyl*bis*(*p*-toluenesulfonamide) [BNBTS] as a reusable catalyst promoted one-pot synthesis of benzo[c]acridines in good to high yields under three-component reaction from anilines, aldehydes and cyclic 1,3-dicarbonyl compounds under solvent-free conditions.

Keywords: Benzo[c]acridine, Naphthalen-1-amine, Aldehyde, Cyclic 1,3-dicarbonyl compound, Solvent-free, BNBTS

INTRODUCTION

Development of simple synthetic routes for nitrogen-containing heterocyclic systems from ready reagents is an essential task in organic synthesis [1]. Multi-component reactions (MCR_S) are significant tools for the rapid and efficient synthesis of a wide variety of organic compounds. These reactions have been investigated extensively in organic and diversity oriented syntheses, primarily due to their ability to complex molecular functionality from simple starting materials *via* one-pot reactions. In recent years, extensive research on the synthesis of tricycle compounds containing the 1,4-dihydro-pyridines, such as acridine derivatives, has been reported. Acridine derivatives have been found to possess useful biological activities such as antitumor properties [2], carcinogenic activity [3], anti-malaria activity [4] and heart defibrillation [5]. Due to its planar structure, acridine chromophore moiety sometimes has excellent DNA binding properties. Recently, the synthesis of polyacridinic compounds [6], as potential *bis*-intercalating agents, has been extensively studied. Moreover, new acridine (naphthalene-quinolines) skeleton fused with a five or six-membered ring yields polycyclic derivatives, which also play important roles as some DNA-intercalating anticancer drugs [7]. Thus, the synthesis of acridine derivatives is an important and principal task in organic chemistry. In the current context, due to the industrial production of synthetic fuels, that may contain nitrogen analogues of polycyclic aromatic hydrocarbons (PAH), the acridine derivatives are becoming of increasing concern [3].

A straightforward method for the synthesis of these compounds involves a condensation between aldehydes, dimedone and naphthalen-1-amine that is catalyzed by various compounds such as poly-phosphoric acid [8], TEBAC [9], organic solvents [10], ionic liquids [11] and microwave irradiation [12].

In continuation of our interest in the application of *N,N'*-dibromo-*N,N'*-1,2-ethanediyl*bis*(*p*-toluenesulfonamide) [BNBTS] [13], in organic synthesis [14,15], we report here a convenient method for the preparation of acridines from naphthalen-1-amine, dimedone and various aldehydes in the presence of BNBTS under solvent-free conditions (Scheme 1).

EXPERIMENTAL

General

Substrates, solvents and other chemicals were purchased from Fluka, Merck and Aldrich chemical companies. The

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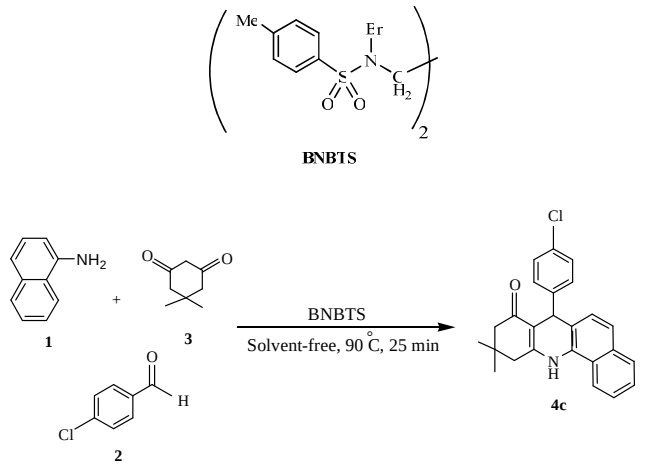


Scheme 1

progress of the reaction was monitored by (TLC SiO_2-*n*-hexane:acetone). IR spectra (KBr) were recorded on Shimadzu infrared Fx-90 spectrophotometer and the nuclear magnetic resonance (NMR) spectra were conducted on a Jeol 90 and 400 MHz spectrometer using TMS as the internal standard. BNBTS was prepared according to our previously reported procedure [13].

Synthesis of Benzo[c]acridine Derivatives Using BNBTS under Solvent-Free Conditions

A mixture of the aldehyde (2 mmol), naphthalene-1-amine or various anilines (2 mmol), 5,5-dimethylcyclohexane-1,3-dione (2 mmol) and BNBTS (0.1 g, 0.2 mmol) was placed in a test-tube at 90 °C and stirred. The progress of the reaction was monitored by TLC (8:2, n-hexane/acetone). After completion of the reaction, ethanol (15 ml) was added and filtered to yield recovered products which were then recrystallized from ethanol to afford the pure products.

Synthesis of 1,8-Dioxo-decahydroacridines Using BNBTS under Solvent-Free Conditions

A mixture of the aldehyde (1 mmol), various anilines or ammonium acetate (1 mmol), 5,5-dimethylcyclohexane-1,3-dione (2 mmol) and BNBTS (0.1 g, 0.2 mmol) was placed in a test-tube at 60 °C (for product **7**, 90 °C) and stirred. The progress of the reaction was monitored by TLC (9:2, n-hexane/acetone). After completion of the reaction, the mixture was cooled, ethanol (15 ml) was added and filtered to yield recovered products which were then recrystallized from ethanol to afford the pure products.

Analytical Data for Compounds which are not Reported Earlier

Compound (2). Yellow solid; m.p.: 250-252 °C; IR (KBr): ν 3330, 2930, 1589, 1530, 1497, 1385, 1346, 1261, 1151 cm^{-1}. ^1H NMR (FT-90 MHz, DMSO-d_6): δ 9.39 (s, 1H), 8.45 (d, *J* = 6.2 Hz, 1H), 8.05 (d, *J* = 6.3 Hz, 1H), 7.76-7.43 (m, 8H), 5.41 (s, 1H), 2.48 (d, *J* = 10.7 Hz, 1H), 2.36 (d, *J* = 10.7 Hz, 1 H), 2.20 (d, *J* = 9.8 Hz, 1 H), 2.09 (d, *J* = 9.8 Hz, 1 H), 1.03 (s, 3H), 0.96 (s, 3H). ^{13}C NMR (FT-90 MHz, DMSO-d_6): δ 25.1, 26.2, 28.9, 31.9, 37.2, 49.9, 105.9, 119.6, 121.2, 124.4, 125.6, 126.5, 126.6, 127.4, 127.7, 130.3, 130.9, 131.5, 132.8, 134.3, 144.4, 151.2, 192.8. MS: (*m/z*) 398 (M^+). Anal. Calcd. for $C_{25}H_{22}N_2O_3$: C, 72.17; H, 5.49; N, 6.75%. Found: C, 72.11; H, 5.28; N, 6.73%.

Compound (9). Wight solid; m.p.: 238-240 °C; IR (KBr): ν 3336, 2926, 1604, 1573, 1493, 1374, 1262, 1141 cm^{-1}. ^1H NMR (FT-90 MHz, DMSO-d_6): δ 9.33 (s, 1H), 8.49 (d, *J* = 10.7 Hz, 1H), 7.92 (d, *J* = 9.8 Hz, 1H) 7.72-7.67 (m, 3H), 7.51-7.72 (m, 3H), 7.44-7.30 (m, 5H), 5.38 (s, 1H), 2.84 (d, *J* = 9.8 Hz, 1H), 2.73 (d, *J* = 9.8 Hz, 1H), 2.19 (d, *J* = 8.0 Hz, 1H), 2.01 (d, *J* = 8.0 Hz, 1H), 1.06 (s, 3H), 0.97 (s, 3H). ^{13}C NMR (FT-90 MHz, DMSO-d_6): δ 25.8, 25.9, 29.2, 31.6, 37.9,38.2, 106.5, 106.7, 120.6, 121.5, 122.2, 122.6, 124.0, 124.7, 125.1, 125.3, 125.9, 126.8, 127.1, 127.5, 130.4, 131.2, 131.6, 132.5, 134.1, 145.1, 145.7, 152.5, 193.1. MS (*m/z*) 403 (M^+). Anal. Calcd. for $C_{29}H_{25}NO$: C, 81.74; H, 6.21; N, 2.99%. Found: C, 81.66; H, 5.93; N, 3.05%.

Compound (5b). Yellow solid; m.p.: 152-154 °C; IR (KBr): ν 3288, 2966, 1705, 1604, 1510, 1386, 1247, 1091 cm^{-1}. ^1H NMR (FT-400 MHz, $CDCl_3$): δ 8.02 (s, 1H), 7.06-7.72 (m, 7H), 5.24 (s, 1H), 3.79 (s, 3H), 2.06-2.23 (m, 4H), 1.23 (s, 3H), 0.98 (s, 3H). ^{13}C NMR (FT-400 MHz, $CDCl_3$): 27.2, 29.2, 29.7, 32.3, 41.8, 42.9, 50.6, 55.5, 114.9, 127.1, 128.4, 128.7, 129.4, 129.9, 130.7, 131.3, 136.6, 142.0, 143.5, 150.5, 195.9. MS (*m/z*) 368 (M^+).

Compound (5c). Yellow solid; m.p.: 295-297 °C; IR (KBr): ν 3273, 2958, 1650, 1521, 1473, 1374, 1278, 1087 cm^{-1}. ^1H NMR (FT-400 MHz, $CDCl_3$): δ 8.46 (s, 1H), 7.18-7.50 (m, 7H), 5.25 (s, 1H), 2.21-2.25 (m, 3H), 1.70-1.81 (d,

J = 8.7 Hz, 1H), 1.21 (s, 3H), 0.98 (s, 3H). ^{13}C NMR (FT-400 MHz, $CDCl_3$): 25.8, 27.2, 29.8, 32.3, 32.6, 42.0, 42.1, 50.0, 50.1, 114.9, 125.5, 128.2, 128.3, 129.5, 131.0, 131.8, 144.1, 144.7, 148.0, 148.4, 195.5. MS (*m/z*) 384 $[M+1]^+$.

Compound (6a). Wight solid; m.p.: 146-148 °C; IR (KBr): ν 3288, 2950, 1597, 1460, 1332, 1270, 1060 cm^{-1}. ^{1}H NMR (FT-400 MHz, $CDCl_3$): δ 7.74 (s, 1H), 3.85 (t, *J* = 12.8 Hz, 1H), 2.24-2.45 (m, 8H), 2.01-2.10 (m, 2H), 0.97-1.29 (m, 12H), 0.85 (t, *J* = 12.8 Hz, 3H). ^{13}C NMR (FT-400 MHz, $CDCl_3$): δ 13.5, 21.8, 22.2, 25.5, 29.7, 31.1, 31.6, 45.9, 115.4, 127.1, 189.5, 190.1. MS (*m/z*) 301 (M^+).

Compound (6b). Grey solid; m.p.: 150-152 °C; IR (KBr): ν 3288, 2960, 1597, 1498, 1332, 1240, 1062 cm^{-1}. ^{1}H NMR (FT-400 MHz, $CDCl_3$): δ 11.94 (s, 1H), 7.11-7.56 (m, 8H), 5.56 (s, 1H), 2.20-2.56 (m, 8H), 0.97-1.25 (m, 12H). ^{13}C NMR (FT-400 MHz, $CDCl_3$): δ 21.5, 27.4, 29.8, 31.4, 32.7, 42.9, 45.4, 125.8, 127.1, 128.2, 136.4, 189.9, 190.0. MS (*m/z*) 350 $[M+1]^+$.

Compound (6c). Yellow solid; m.p.: 194-196 °C; IR (KBr): ν 3250, 3059, 2960, 1602, 1495, 1363, 1261, 1014 cm^{-1}. ^{1}H NMR (FT-400 MHz, $CDCl_3$): δ 11.94 (s, 1H), 7.11-7.31 (m, 5H), 5.57 (s, 1H), 2.01-2.76 (m, 8H), 1.04-1.26 (m, 12H). ^{13}C NMR (FT-400 MHz, $CDCl_3$): δ 27.4, 29.6, 31.4, 32.7, 45.4, 125.8, 126.7, 127.9, 128.2, 138.0, 189.4, 190.4. MS (*m/z*) 350 $[M+1]^+$.

Compound (7). Yellow solid; m.p.: 153-155 °C; IR (KBr): ν 3066, 2933, 2825, 1600, 1598, 1581, 1521, 1473, 1298, 1178, 840 cm^{-1}. ^{1}H NMR (FT-400 MHz, $CDCl_3$): δ 7.04-8.08 (m, 8H), 5.24 (s, 1H), 3.99 (s, 3H), 1.64-2.42 (m, 8H), 0.81-1.25 (m, 12H). ^{13}C NMR (FT-400 MHz, $CDCl_3$): δ 21.5, 26.7, 32.4, 42.1, 50.1, 55.3, 55.6, 115.3, 124.6, 128.1, 129.7, 129.4, 129.2, 130.9, 131.4, 135.6, 144.8, 159.8, 195.9, 197.0. MS (*m/z*) 489 (M^+).

RESULTS AND DISCUSSION

Initially, we decided to explore the role of our catalyst for the synthesis of acridines, from 4-chlorobenzaldehyde, naphthalen-1-amine and dimedone as model compounds (Scheme 1).

In the absence of the catalyst, no acridine synthesis was observed, even after prolonged reaction time. Since the synthesis of acridine from the model compounds failed in the absence of the catalyst, the effect of the catalyst was also investigated in various conditions and the results are presented in Table 1.

In the solvent system, the best results were achieved using H_2O/C_2H_5OH (140 min, 77%, Entry 11). In recent years, there has been an increasing interest in reactions that proceed in the absence of solvents due to the reduced pollution, low cost,

Table 1. Synthesis of 4c under Different Conditions[a]

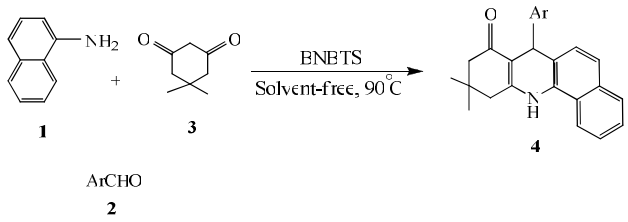
Entry	Conditions	Cata. (g)	Time (min)	Yield (%)[b]
1	Solvent-free, 50 °C	0.10	71	85
2	Solvent-free, 90 °C	0.05	100	58
3	Solvent-free, 90 °C	0.08	43	75
4	Solvent-free, 90 °C	0.10	25	93
5	Solvent-free, 90 °C	0.12	35	87
6	Solvent-free, 110 °C	0.10	15	93
7	Solvent-free, rt[c]	0.10	320	67
8	Solvent-free, rt	0.08	331	62
9	H_2O/CH_3CH_2OH (30/70), rt	0.10	180	69
10	H_2O/CH_3CH_2OH (30/70), 30 °C	0.12	121	70
11	H_2O/CH_3CH_2OH (30/70), 30 °C	0.10	140	77
12	H_2O/CH_3CH_2OH (30/70), 30 °C	0.08	153	74
13	H_2O, 50 °C	0.10	480	trace

[a]Catalyst and conditions: **1** (2 mmol), **2c** (2 mmol), **3** (2.2 mmol). [b]Isolated yield. [c]Room temperature.

simplicity in process and handling. Therefore, we decided to test this reaction under solvent-free condition using various ratios of the catalyst.

We found that the reaction was rapid and gave excellent yields of products when catalyzed by *N,N'*-dibromo-*N,N'*-1,2-ethanediyl*bis*(*p*-toluenesulfonamide) [BNBTS] (0.1 g) (25 min, 93%, Entry 4, Table 1).

To test the generality and versatility of this procedure in the synthesis of acridines, we examined the synthesis of a number of acridines using the optimized conditions (Scheme 2, Table 2).



Scheme 2

It is noteworthy that various acridines with electron-donating or electron-withdrawing groups were synthesized under solvent-free conditions using *N,N'*-dibromo-*N,N'*-1,2-ethanediyl*bis*(*p*-toluenesulfonamide) [BNBTS] in good to high yields (Table 2).

Functional groups such as methoxy (Entry 6), hydroxyl

Table 2. Preparation of Benzo[c]acridines under Solvent-Free Conditions

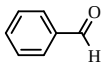
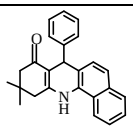
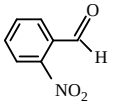
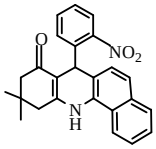
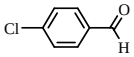
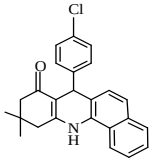
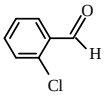
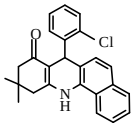
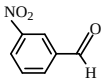
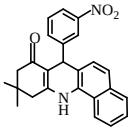
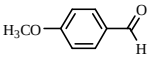
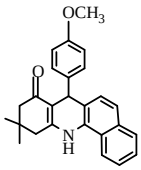
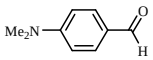
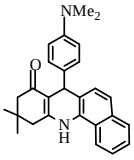
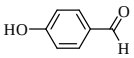
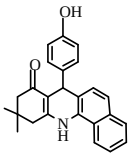
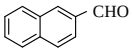
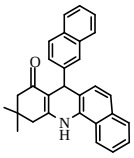
Entry	Ar	Products[a]	Time (min)	Yields (%)	Ref.
1			35	82	[9]
2			60	96	-
3			25	93	[9]
4			25	82	[9]
5			50	86	[9]
6			83	84	[9]

Table 2. Continued

7			20	85	[9]
8			5	90	[9]
9			60	92	-

[a]Products were characterized from their physical properties, by comparison with autheneic samples, and by spectroscopic methods.

(Entry 8), amine (Entry 7), hallogen (Entries 3, 4) and nitro (Entries 2, 5) were unchanged during the reaction. Furthermore, when polycyclic aromatic aldehyde (Entry 9) was treated with naphthalen-1-amine and dimedone in the presence of BNBTS, the corresponding benzoacridine was obtained in high yield under thermal solvent-free condition. However, aliphatic aldehydes were not suitable for this reaction and could not produce benzoacridine derivatives in good yields.

Our experiments also indicated that BNBTS was a reusable catalyst and after four runs, the catalytic activity of the catalyst was almost the same as before (Table 3).

N,*N'*-Dibromo-*N*,*N'*-1,2-ethanediyl*bis*(*p*-toluenesulfonamide) [BNBTS] is inexpensive and non-hazardous catalyst. It can be conveniently handled and can be removed from the reaction mixture by simple filtration.

1,4-Dihydropyridines (DHPs) can be synthesized by different methods. Therefore, we prepared acridine derivatives by using different molar ratios from dimedone, aldehyde, aniline, aniline substituted and naphthalen-1-amine in the presence of BNBTS under solvent-free conditions.

We also examined this reaction with aniline and anilines substituted with electron-donating and electron-withdrawing groups and aldehydes in the presence of BNBTS under solvent-free conditions in which tetrahydroacridinones were obtained in good to high yields (Scheme 3). The results are shown in Table 4.

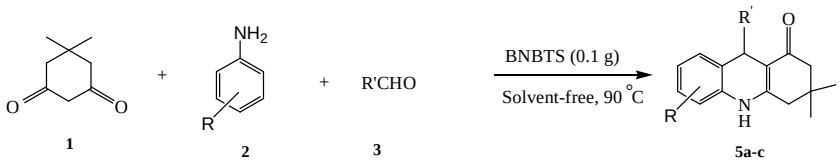
Table 3. The Recycling of BNBTS in the Synthesis of *p*-Chlorobenzo[c]acridine

Entry	Time (min)	Yield (%)
1	25	93
2	25	90
3	25	87
4	25	85

Furthermore, when 1 mmol of dimedone was used, **4** or **5** was obtained, but if 2 mmol of dimedone was added to the mixture of aldehydes and various anilines, 1,8-dioxo-decahydroacridines were obtained (Scheme 4). The results are summarized in Table 5.

It is noteworthy that compound **6c** (60%, 24 min) can also be synthesized by NH_4OAc (Scheme 5).

Moreover, we can synthesize compound **7** by catalytic amount of BNBTS and molar ratio 2:1:1 from dimedone, 4-chlorobenzaldehyde and 4-methoxy aniline with high yield under thermal solvent-free conditions (Scheme 6).

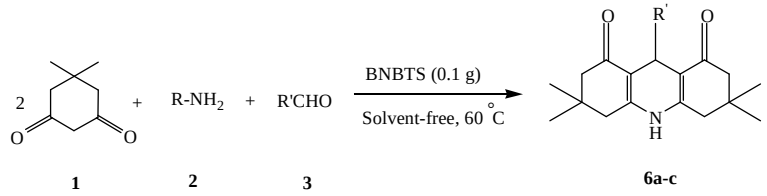


5a R = H, R' = Phenyl
5b R = 4-Methoxy, R' = 4-Chlorophenyl
5c R = 4-Nitro, R' = 4-Chlorophenyl

Scheme 3

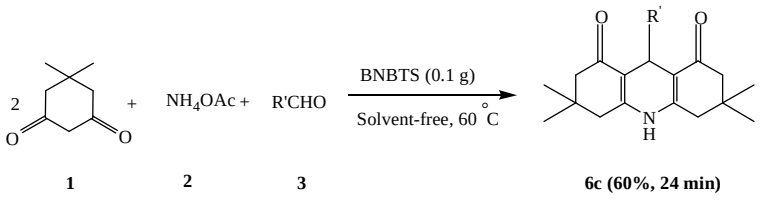
Table 4. Yield and Reaction Time for the Synthesis of Compounds **5a-c**

Compd. No.	R	R'	M.P. (°C)	Time (min)	Yield (%)
5a	Phenyl	Phenyl	193-195	25	90
5b	4-Methoxy	4-Chlorophenyl	152-154	144	80
5c	4-Nitro	4-Chlorophenyl	295-297	150	87



6a R = Phenyl, R' =Ethyl
6b R = 4-Methoxyphenyl, R' = phenyl
6c R = 4-Nitrophenyl, R' = phenyl

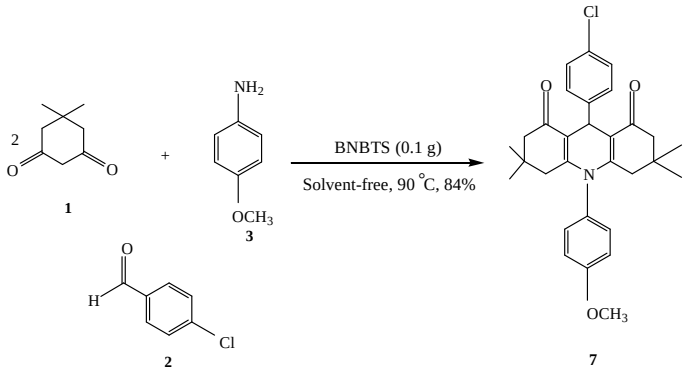
Scheme 4



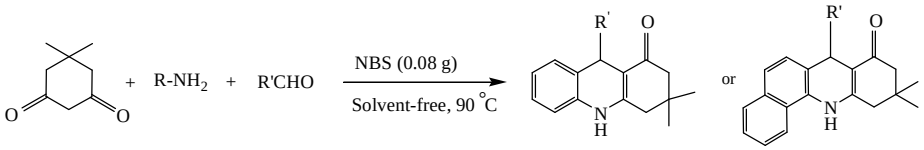
Scheme 5

Table 5. Yield and Reaction Time for the Preparation of Compounds **6a-c**

Compd. No.	R	R'	M.P. (°C)	Time (min)	Yield (%)
6a	Phenyl	Ethyl	146-148	30	70
6b	4-Methoxyphenyl	Phenyl	150-152	28	85
6c	4-Nitrophenyl	Phenyl	194-196	135	88



Scheme 6



R = Phenyl, R' = Phenyl
R = 4-Methoxyphenyl, R' = 4-Chlorophenyl
R = 1-Naphthyl, R' = 4-Chlorophenyl
R = Phenyl, R' =Ethyl

Scheme 7

Table 6. Yield and Reaction Time for Preparation of Acridine Derivatives Catalyzed by NBS

Entry	R	R'	Time (min)	Yield (%)
1	Phenyl	Phenyl	50	84
2	4-Methoxyphenyl	4-Chlorophenyl	93	80
3	1-Naphthyl	4-Chlorophenyl	40	75
4	Phenyl	Ethyl	144	70

Table 7. Reaction Time and Yield for the Proposed Method and Some of the Previously Published Methods

Entry	Aldehyde	Condition	Time	Yield (%)	Ref.
1	4-ClC_6H_4	BNBTS (0.1 g), Solvent-free, 90 °C	25 min	93	This work
2	4-ClC_6H_4	NBS (0.08 g), Solvent-free, 90 °C	40 min	75	This work
3	4-ClC_6H_4	TEBAC (0.1 g), H_2O, 100 °C	12 h	93	[16]
4	4-ClC_6H_4	Ultrasound irradiation, EtOH/25-30 °C	1 h	90	[17]
5	C_6H_5	BNBTS (0.1 g), Solvent-free, 90 °C	35 min	82	This work
6	C_6H_5	NBS (0.08 g), Solvent-free, 90 °C	50 min	84	This work
7	C_6H_5	Reflux, Absolute EtOH	4 h	62	[18]
8	C_6H_5	MW, EtOH, Neutral Alumina	9 min	75	[18]
9	C_6H_5	MW, EtOH, Basic Alumina	8.5 min	80	[18]
10	C_6H_5	MW, Neat	2 min	87	[18]

For the sake of comparison, in a set of experiments, we used NBS as a catalyst for this reaction. *N*-Bromosuccinimide accelerated this reaction at 90 °C under solvent-free conditions to give benzoacridine derivatives (Scheme 7). The best result was obtained when 0.08 g of NBS was used at 90 °C. *N*-Bromosuccinimide promoted the reaction between naphthalen-1-amine, substituted aniline, various aldehydes and dimedone under solvent-free conditions to give tetrahydroacridinones with moderate to good yields (Scheme 7). The results are shown in Table 6.

By comparison, BNBTS was found to be more efficient than NBS. The advantages of our catalyst over the common catalysts in the synthesis of benzoacridine derivatives are demonstrated in Table 7.

CONCLUSIONS

In summary, in this study we have introduced a new and useful solvent-free application of BNBTS as an efficient catalyst for the synthesis of acridines under mild reaction conditions. The method has the advantages of high product yields, selectivity, operational simplicity (easy workup of reaction) and solid state reaction.

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