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Sulfamic Acid: An Efficient and Green Catalyst for Synthesis of 1,5-Benzodiazepines Under Solvent-Free Conditions

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ABSTRACT: *It is described that sulfamic acid can be utilized as an effective and green catalyst for the synthesis of 1,5-benzodiazepines in high yields from the solvent-free condensation of o-phenylenediamine (**1**) and ketones (**2**).* © 2007 Wiley Periodicals, Inc. Heteroatom Chem 18:354–358, 2007; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.20305

INTRODUCTION

Benzodiazepines and their polycyclic derivatives are a very important family of bioactive compounds. They are widely used as anticonvulsant, antiinflammatory, analgesic, hypnotic, sedative, and antidepressive reagents [1,2] and their new applications have been increasingly found. Besides, benzodiazepines are also significant intermediates in the constructions of fused-ring compounds such as triazolo-, oxadiazolo-, oxazino-, and furanobenzodiazepines [3–6]. Owing to their broad spectrum of biological activity, these compounds have received a great deal of attention in connection with their synthesis. Generally, the routes to them include the condensation of *o*-phenylenediamines with α,β-unsaturated carbonyl compounds [7], β-haloketones, or with ketones [8]. To drive the condensations, the presence of an acidic catalyst is indispensable, like BF_3·OEt [9], polyphosphoric acid [10], $MgO/POCl_3$ [11], $Yb(OTf)_3$ [12], $Me_2S^+BrBr^-$ [13], $Sc(OTf)_3$ [14], PVP-supported $FeCl_3$ [15], I_2 [16], $Ag_3PW_{12}O_{40}$ [17], $InBr_3$ [18], SO_4^{2-}/ZrO_2 [19], [(L)proline]$_2$Zn [20], Al_2O_3/P_2O_5 [21], etc are reported to catalyzed the formation of 1,5-benzodiazepines. Moreover, the approach to these significant species can also be achieved by microwave irradiation in the aid of CH_3COOH [22] or executed in ionic liquid medium [23]. Besides, Zhang et al. described an alternative strategy [24] for the transformation of *o*-nitrophenylazide and ketones to 1,5-benzodiazepines through the reductive cyclization in the $TiCl_4$/Sm system. However, most of these methodologies are associated with several drawbacks, such as prolonged reaction time, harsh reaction conditions, difficulty recycling catalysts, low product yields, occurrence of several side reactions, much expensive catalysts, heavy environment pollution, and so on.

In the past 3 years, owing to its prominent physical and chemical property like outstanding stability, nontoxicity, nonvolatility, odorlessness, ready availability, and extremely cheap price,* sulfamic acid (SA) has increasingly attracted much attention as an alternative acidic catalyst in some heterogeneously catalytic reactions like acetalization [25], esterification [26], acetylation of alcohols and phenols [27], nitrile formation [28], tetrahydropyranylation of alcohols [29], transesterification

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*In China, the price of sulfamic acid is $1 per 100 g.

of β-ketoesters [30], acetolysis of cyclic ethers [31], protection of carbonyls [32], Biginelli [33] and Pechmann condensation [34], Bechmann rearrangement [35], imino-Diels-Alder reaction [36], synthesis of quinolines [37], benzoxanthenes [38], bis(indolyl)methanes [39], and so on. However, there is no report in the literature on the utilization of SA in the synthesis of benzodiazepines via the cyclization of *o*-phenylenediamine and ketones. The distinctive catalytic activity and intrinsic zwitterionic feature of SA makes it much different from conventional acidic catalysts and encourages us to investigate its application in the formation of benzodiazepines. The use of recyclable SA makes the process convenient, economic, and environmentally benign with rapid access to products in good to excellent yields under mild conditions.

EXPERIMENTAL

Apparatus and Analysis

All the compounds used were analytical reagents and some chemicals were further purified by recrystallization or distillation. ^{1}H NMR spectra were obtained on a Bruker Avance DMX 400 MHz instrument using TMS as internal standard in $CDCl_3$. FT-IR spectra were recorded on a Nicolet Avatar spectrophotometer. EI-MS were carried out on a Varian GC-MS spectrometer.

General Procedure

A mixture of *o*-phenylenediamine **1** (1 mmol, 0.103 g), ketone **2** (2.2 mmol), and SA (0.2 mmol, 0.02 g) was stirred under solvent-free conditions at appropriate temperature for appropriate time (see Table 1). After completion of the reaction (determined by TLC), the mixture was extracted with acetone (2 × 10 mL) and the solid was filtrated. The combined organic layers were dried over anhydrous Na_2SO_4, concentrated in vacuo and purified by silica gel column chromatography using petroleum ether/ethyl acetate (4:1) as eluent. The filtrated solid, which was the recovered SA, was washed with ether and activated in vacuo at 70°C for 2 h prior to reuse.

3a: mp 134–136°C (Lit. [12] 137–139°C); FT-IR (KBr) ν: 3294, 2964, 1633, 1594, 1475, 770 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.34 (s, 6H, 2×CH_3), 2.23 (s, 2H, CH_2), 2.37 (s, 3H, N=C—CH_3), 2.97 (br, s, 1H, NH), 6.71–6.74 (m, 1H, Ar—H), 6.97–7.00 (m, 2H, Ar—Hs), 7.13–7.15 (m, 1H, Ar—H); EI-MS (70 eV) *m/z* (%): 188 (M^+, 100), 173 (44), 132 (21), 104 (64), 77 (32), 65 (27).

3b: mp 141–143°C) Lit. [17] 137–139°C); FT-IR (KBr) ν: 3332, 2967, 2932, 1639, 1596, 1471, 750 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.94 (t, $J = 7.6$ Hz, 3H, CH_2 *CH_3*), 1.24 (t, $J = 7.6$ Hz, 3H, N=C—CH_2 *CH_3*), 1.26 (s, 3H, CH_3), 1.56–1.70 (m, 2H, *CH_2*CH_3), 2.14 (d, $J = 13.2$ Hz, 1H, CH_2^a), 2.23 (d, $J = 13.2$ Hz, 1H, CH_2^b), 2.59 (q, $J = 7.6$ Hz, 2H, N=C—CH_2), 3.01 (br, s, 1H, NH), 6.71–6.74 (m, 1H, Ar—H), 6.96–6.98 (m, 2H, Ar—Hs), 7.11–7.14 (m, 1H, Ar—H); EI-MS (70 eV) *m/z* (%): 217 ($M^+ + 1$, 100), 187 (95), 145 (22), 133 (20), 119 (7), 92 (6), 77 (10), 65 (11).

3b′: mp 135–136°C (Lit. [40] 139–141°C); FT-IR (KBr) ν: 3381, 2982, 1635, 1601, 1477, 765 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.98 (t, $J =$ 7.0 Hz, 3H, *CH_3*CH_2—), 1.25 (s, 3H, CH_3—), 1.51–1.64 (m, 2H, *CH_2*CH_3), 1.75 (m, 3H, —CH *CH_3*), 2.51 (s, 3H, N=C—CH_3), 2.85 (m, 1H, N=C—CH), 3.55 (s, br, NH), 6.67–6.70 (m, 1H, Ar—H), 6.94 (m, 2H, Ar—Hs), 7.06–7.11 (m, 1H, Ar—H); EI-MS (70 eV) *m/z* (%): 216 (M^+, 100), 193 (11), 187 (75), 155 (42), 141 (20), 131 (6), 118 (17), 77 (30).

3c: mp 142–144°C (Lit. [12] 144–145°C); FT-IR (KBr) ν: 3323, 2982, 2876, 1654, 1605, 1475, 778 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 0.77–1.10 (m, 9H, *CH_3*CH_2—), 1.23–1.41 (m, 4H, CH_3 *CH_2*—), 1.53–1.67 (m, 3H, —CH*CH_3*), 2.41–2.63 (m, 2H, N=C—*CH_2*CH_3), 2.89 (q, $J = 7.2$ Hz, 1H, N=C—*CH*—CH_3), 3.91 (br, s, 1H, NH), 6.57 (d, $J = 8.0$ Hz, 1H, Ar—H), 6.65 (t, $J = 8.0$ Hz, 1H, Ar—H), 6.93 (t, $J = 8.2$ Hz, 1H, Ar—H), 7.33 (d, $J = 8.2$ Hz, 1H, Ar—H); EI-MS (70 eV) *m/z* (%): 244 (M^+, 30), 229 (25), 215 (100), 114 (15), 77 (25), 65 (13).

3d: mp 134–135°C (Lit. [12] 138–139°C); FT-IR (KBr) ν: 3340, 2966, 2872, 1642, 1605, 1503, 1264, 739 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.51–1.87 (m, 10H, CH_2—), 2.16–2.22 (m, 2H, CH_2—), 2.31–2.52 (m, 2H, N=C— *CH_2*—), 2.80–2.90 (m, 1H, N=C—*CH*), 6.65 (dd, $J = 1.2$, 8.0 Hz, 1H, Ar—H), 6.82 (dt, $J = 1.4$, 7.2 Hz, 1H, Ar—H), 6.95 (dt, $J = 1.4$, 7.2 Hz, 1H, Ar—H), 7.95 (dd, $J = 1.2$, 8.0 Hz, 1H, Ar—H); EI-MS (70 eV) *m/z* (%): 241 ($M^+ + 1$, 100), 225 (60), 210 (10), 182 (11), 133 (15), 92 (6), 77 (5), 65 (13).

3e: mp 136–137°C (Lit. [12] 137–139°C); FT-IR (KBr) ν: 3394, 3047, 2929, 2854, 1640, 1594, 1476, 754 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 1.25–1.84 (m, 16H, —CH_2—), 2.31–2.35 (m, 1H, N=C—*CH*), 2.57 (t, $J = 6.8$ Hz, 2H, N=C—*CH_2*), 3.75 (br, s, 1H, NH), 6.68 (dd, $J = 1.4$ Hz, $J = 7.6$ Hz, 1H, Ar—H), 6.90–6.98 (m, 2H, Ar—Hs), 7.26 (dd, $J = 1.4$ Hz, $J = 7.6$ Hz, 1H, Ar—H); EI-MS (70 eV) *m/z* (%): 267 ($M^+ - 1$, 85), 237 (35), 223 (100), 210 (16), 195 (11), 91 (7), 77 (7), 65 (11).

3f: mp 146–148°C (Lit. [12] 151–152°C); FT-IR (KBr) ν: 3280, 3057, 1633, 1600, 1467, 1329, 762, 749, 700, 688 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ:

1.75 (s, 3H, CH_3), 2.97 (d, $J = 13.2$ Hz, 1H, CH_2^a), 3.13 (d, $J = 13.2$ Hz, 1H, CH_2^b); 3.51 (s, 1H, NH), 6.85 (dd, $J = 1.6$ Hz, $J = 8.0$ Hz, 1H, Ar—H), 7.05 (dt, $J = 1.6$ Hz, $J = 8.0$ Hz, 2H, Ar—Hs), 7.17–7.31 (m, 7H, Ar—Hs), 7.57 (dt, $J = 1.6$ Hz, $J = 8.0$ Hz, 4H, Ar—Hs); EI-MS (70 eV) *m/z* (%): 312 (M^+, 10), 295 (100), 235 (25), 194 (30), 103 (20), 77 (60), 40 (80).

3g: mp 121–123°C (Lit. [41] 117°C); FT-IR (KBr) ν: 3336, 3056, 2960, 1603, 1510, 1467, 1249, 1176, 1032, 832, 756 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ: 1.73 (s, 3H, CH_3), 2.93 (d, $J = 13.2$ Hz, 1H, CH_2^a), 3.06 (d, $J = 13.2$ Hz, 1H, CH_2^b), 3.45 (br, s, 1H, NH), 3.76 (s, 3H, OCH_3), 3.80 (s, 3H, OCH_3), 6.76–6.83 (m, 5H, Ar—Hs), 7.05 (t, $J = 8.8$ Hz, 2H, Ar—Hs), 7.33 (m, 1H, Ar—H), 7.52 (d, $J = 8.8$ Hz, 2H, Ar—Hs), 7.61 (d, $J = 8.8$ Hz, 2H, Ar—Hs); EI-MS (70 eV) *m/z* (%): 373 ($M^+ + 1$, 6), 279 (9), 236 (100), 221 (35), 209 (11), 193 (13), 166 (17), 133 (16), 103 (15), 93 (7), 76 (14).

3h: mp 151–153°C (Lit. [41] 154°C); FT-IR (KBr) ν: 3312, 3074, 1597, 1525, 1468, 1348, 855, 750, 694 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ: 1.84 (s, 3H, CH_3), 3.01 (d, $J = 13.6$ Hz, 1H, CH_2^a), 3.31 (d, $J = 13.6$ Hz, 1H, CH_2^b), 3.64 (br, s, 1H, NH), 6.90 (d, $J = 7.6$ Hz, 1H, Ar—H), 7.11 (t, $J = 7.6$ Hz, 1H, Ar—H), 7.16 (t, $J = 7.6$ Hz, 1H, Ar—H), 7.34 (d, $J = 7.6$ Hz, 1H, Ar—H), 7.68 (d, $J = 8.8$ Hz, 2H, Ar—Hs), 7.76 (d, $J = 8.8$ Hz, 2H, Ar—Hs), 8.06 (d, $J = 8.8$ Hz, 4H, Ar—Hs); EI-MS (70 eV) *m/z* (%): 403 ($M^+ + 1$, 5), 251 (100), 221 (40), 205 (24), 188 (15), 178 (21), 151 (11), 102 (13), 76 (23), 50 (27).

RESULTS AND DISCUSSION

To survey the reaction conditions in the formation of benzodiazepines assisted by SA, the condensation of *o*-phenylenediamine (**1**) with acetone **2a** was selected as a model (Scheme 1) and the results were indicated in Table 1.

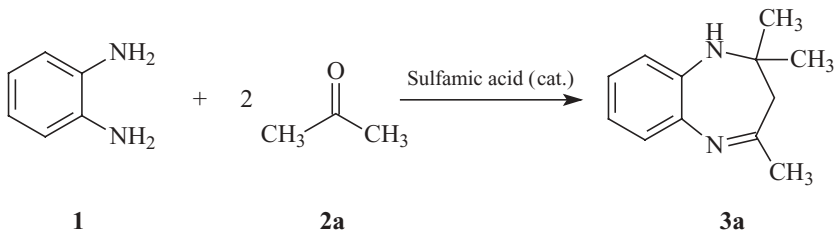
Apparently, the efficiency and the yields of the reaction in organic solution were much less than those obtained under solvent-free conditions. In organic solutions, the conversion of **1** could not be accomplished completely, a substantial amount of **1** was recovered through column chromatography when the condensation was carried out for 10 h. The yield of the reaction increased remarkably, from 69% to 96%, as the amount of SA was raised from 5 to 20 mol%, respectively. It seemed that 20 mol% SA was sufficient to promote the reaction; higher amounts of it could not obviously improve the yield. However, in the absence of SA, the reaction could hardly proceed even after long time (overnight). Moreover, the recovery of the catalyst was also tested. It is well known that SA is insoluble in many organic solvents, especially the non-protonic and low-polar ones. Therefore, in our case, it was ready to separate SA from the acetone solution of products by simple filtration. After activation through washing then heating at 70°C for 2 h, the refreshed SA could be reused in reaction with only a gradual decrease in its activity, the yields were 96%, 91%, 84%, and 72%, respectively, over four runs.

TABLE 1 Reaction Conditions in the Formation of Benzodiazepine **3a**

Entry	*Solvent*	*Catalyst Amount (mol%)*	*Temp (°C)*	*Time (h)*	*Yield (%)*
1	CH_3CN	20	Refluxing	10	65
2	EtOH	20	Refluxing	10	57
3	$CHCl_3$	20	Refluxing	10	44
4	No	5	r.t.	0.5	69
5	No	10	r.t.	0.5	86
6	No	20	r.t.	0.5	96
7	No	No	r.t.	Overnight	Trace

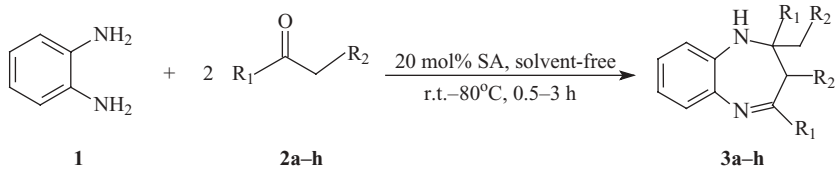
Based on the above results, we extended our investigation to some other ketones (Scheme 2).

Aliphatic and aromatic ketones as well as cyclic ones were screened as substrates to carry out the SA-promoted synthesis of 1,5-benzodiazepine derivatives. It was found that all ketones involved in reactions could successfully undergo the condensations assisted by 20 mol% SA to offer the expected products **3a–h** in 33%–96% yields. Aliphatic and cyclic ketones displayed their high reactivity, condensations



1 **2a** **3a**

SCHEME 1

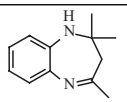
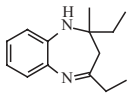
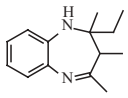
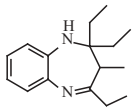
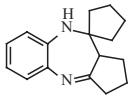
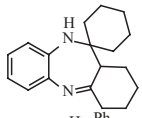
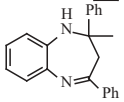
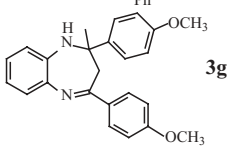
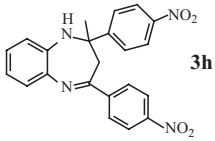


1 **2a–h** **3a–h**

SCHEME 2

involved with them could smoothly be accomplished within 1 h in high yields under mild conditions (Table 2). However, owing to the low activity of carbonyl groups, reactions participated with aromatic ketones should be executed at 50–80°C and the reaction time was prolonged to 3 h (Table 2, entry f, g, and h). The remarkable electron effect existed in the reactions involving aromatic ketones, since the one with an electron-withdrawing group like 4-nitroacetophenone was able to fulfill the condensation in 81% yield, but the other one with an electron-donating group like 4-methoxyacetophenone was sluggish to carry out the condensation, affording the product **3g** only in 33% yield, and the

TABLE 2 SA-promoted Synthesis of 1,5-Benzodiazepines **3a–h**

Entry	*Ketone*	*Product*	*Temp (°C)*	*Time (h)*	*Yield (%)**
1	Acetone	 **3a**	r.t.	0.5	96
2	2-Butanone	 **3b**	45	0.5	87
		 **3b′**			8
3	3-Pentanone	 **3c**	45	0.5	90
4	Cyclopentanone	 **3d**	r.t.	1	86
5	Cyclohexanone	 **3e**	r.t.	1	95
6	Acetophenone	 **3f**	50	2	86
7	4-Methoxyacetophenone	 **3g**	80	3	33
8	4-Nitroacetophenone	 **3h**	80	1.5	81

*Yield refers to the isolated pure products after column chromatography.

unconverted substrates of *o*-phenylenediamine and 4-methoxyacetophenone could be recovered by column chromatography.

CONCLUSION

Herein we described a mild and effective protocol for the solvent-free synthesis of 1,5-benzodiazepine derivatives through the condensation of *o*-phenylenediamine and ketones using sulfamic acid as a green and recyclable catalyst. The simple procedure combined with the ease of recovery and reuse of this novel catalyst made our method very simple, convenient, and environmentally benign for the construction of various 1,5-benzodiazepine compounds.

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