One-Pot Synthesis of Polyfunctionalized Pyrans Catalyzed by Basic Ionic Liquid in Aqueous Media

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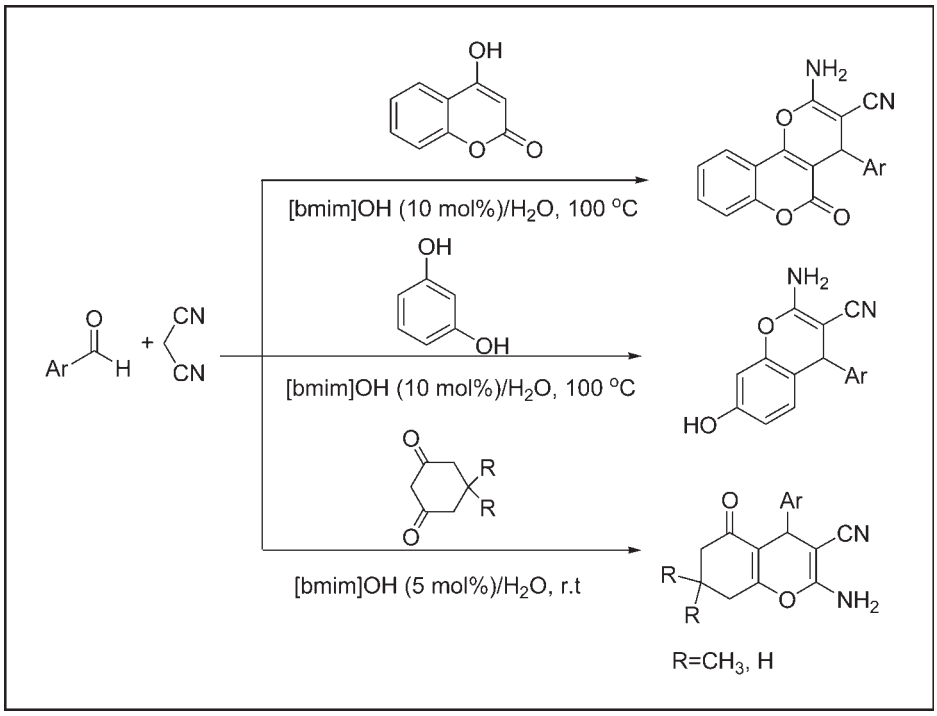
Received February 7, 2009
DOI 10.1002/jhet.193
Published online 2 November 2009 in Wiley InterScience (www.interscience.wiley.com).



An efficient and convenient method for the synthesis of polyfunctionalized 4*H*-pyrans has been achieved through the one-pot condensation of aromatic aldehydes, malononitrile, and 4-hydroxycoumarin, phenols or active methylene carbonyl compounds such as 1, 3-cyclohexanedione and dimedone in the presence of 1-butyl-3-methyl imidazolium hydroxide ([bmim]OH) as catalyst in aqueous media. This method offers several advantages short reaction time, high yields, and simple procedure.

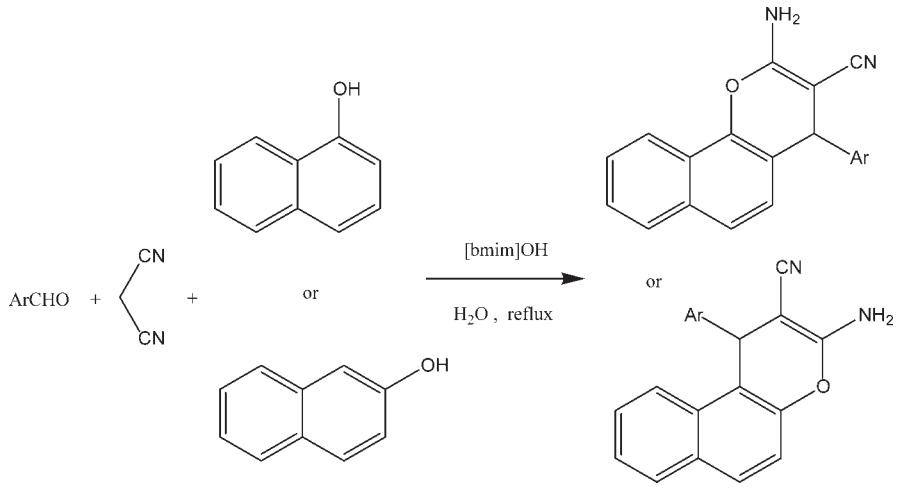
J. Heterocyclic Chem., **46**, 1145 (2009).

INTRODUCTION

The 4*H*-pyrans and their derivatives have attracted considerable attention from organic and medicinal chemists due to their useful biological and pharmacological properties [1], such as spasmolytic, diuretic, anticoagulant, anticancer, and antianaphylactic activity [2]. Furthermore, those compounds can be used as pigments [3], as photoactive materials [4], and as potential biodegradable agrochemicals [5]. Thus, the synthesis of 4*H*-pyran derivatives currently is of much importance. Various methods have been reported for the synthesis of 4*H*-pyrans, including the reaction of aromatic aldehydes, malononitrile with naphthol for the synthesis 2-amino-2-chromenes [6], the condensation of aromatic aldehydes, malononitrile with 4-hydroxycoumarin [7], and the condensation of aromatic aldehydes, malononitrile with active methylene carbonyl compounds [8]. Those reactions have been reported in the presence of a catalyst, such as piperidine [9], KF-Alumina [10], phase-transfer catalyst [11], and $(NH_4)HPO_4$ [12]. Pyrans have also been synthesized under microwave and ultrasound irradiations [13]. These methods also suffer from some disadvantages such as long reaction times, low yields, the use of organic solvent, special apparatus, and tedious workup procedures. Thus, the need for the development of an efficient and facile method for the synthesis of 4*H*-pyran derivatives is in high demand.

Our recent interest has been in the development of new synthetic methods on using ionic liquids as reaction media and catalyst [14]. Herein, we would like to report a highly efficient, convenient, and facile method for the synthesis of 4*H*-pyrans in the presence of basic ionic liquid [bmim]OH as catalyst in aqueous medium.

Scheme 1. Synthesis of 2-amino-2-chromenes.



Scheme 2. Synthesis of 2-amino-4-aryl-3-cyano-5-oxo-4*H*,5*H*-pyrano[3,2-*c*]chromenes (**4a-4l**).

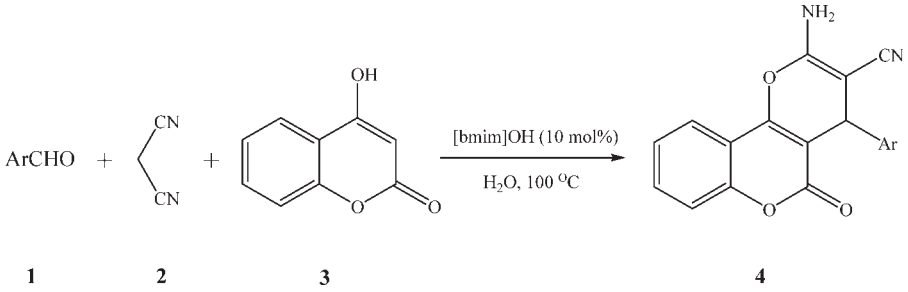


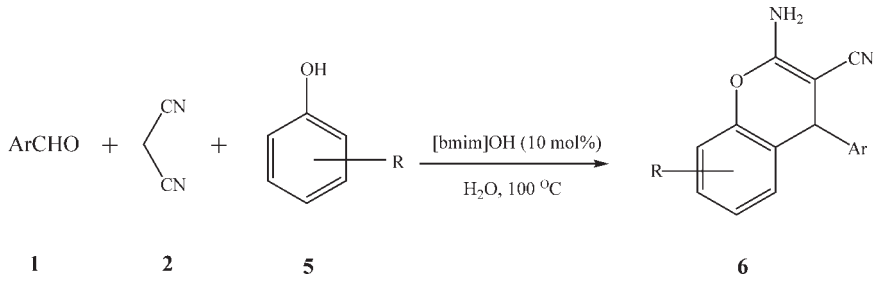
Table 1

Synthesis of **4a–4l** using [bmim]OH as catalyst in aqueous medium.[a]

Entry	Ar	Time (min)	Product	Yield (%)[b]	MP (°C)	
					Found	Reported
1	C_6H_5	8	**4a**	92	257–258	256–258[12a]
2	2-Cl-C_6H_4	15	**4b**	91	265–266	266–268[11b]
3	4-Cl-C_6H_4	5	**4c**	97	264–266	265–265[12a]
4	2,4-Cl_2-C_6H_3	15	**4d**	95	253–255	257–259[12a]
5	4-F-C_6H_4	5	**4e**	96	261–263	260–262[11b]
6	4-Br-C_6H_4	5	**4f**	93	255–257	256–258[10b]
7	2-NO_2-C_6H_4	6	**4g**	94	254–256	255–256[10b]
8	3-NO_2-C_6H_4	5	**4h**	93	258–260	262–264[12a]
9	4-NO_2-C_6H_4	5	**4i**	96	259–260	258–260[12a]
10	4-CH_3-C_6H_4	8	**4j**	90	258–260	259–261[10b]
11	4-CH_3O-C_6H_4	10	**4k**	89	240–242	240–244[12a]
12	4-OH-C_6H_4	30	**4l**	90	260–261	261–262[10b]

[a] Reaction conditions: aldehyde (5 mmol), malononitrile (5 mmol), 4-hydroxycoumarin (5 mmol), [bmim]OH (0.5 mmol), H_2O (2 mL), 100°C.
[b] Isolated yield.

Scheme 3. Synthesis of 2-amino-4-aryl-3-cyano-4*H*-chromenes (**6a-6e**).



1 2 5 6

RESULTS AND DISCUSSION

Very recently, we reported a simple and convenient method for the synthesis of 2-amino-2-chromenes by condensation of aromatic aldehydes, malononitrile with α- or β-naphthol using [bmim]OH as catalyst in aqueous medium (Scheme 1) [15]. This versatility of this ionic liquid encourages us to study its use in other organic reactions. We have now extended the use of this functionalized ionic liquid to catalyze the reaction of aromatic aldehydes, malononitrile with 4-hydroxycoumarin (Scheme 2). The synthesis of 2-amino-4-aryl-5-oxo-4*H*,5*H*-pyrano-[3,2-*c*]chromence-3-carbonitrile was achieved by the three-component condensation of an aromatic aldehyde, malononitrile with 4-hydroxycoumarin in the presence of 10 mol% [bmim]OH at 100°C. The results are summarized in Table 1. In all cases, the corresponding dihydropyrano[*c*]chromenes were obtained in good to excellent yields. However, when aldehydes with electron-withdrawing groups (such as nitro group and halide) are reactants, the reaction time is shorter than that with electron-donating groups (such as methoxy group and hydroxyl group).

Encouraged by these results, we replaced the substituted phenols **5** instead of 4-hydroxycoumarin **3** in the same conditions (Scheme 3). The results are summarized in Table 2. No significant formation of product (**6a**) was observed, when the reaction of benzaldehyde, malononitrile, and phenol was preceded in the same conditions, even prolonged reaction time to 24 hours. However, resorcinol is an activated phenol, when used in the reaction instead of phenol, the yields were promoted greatly with shorter reaction time, which indicated that resorcinol can be successfully used in this reaction.

The versatility of this ionic liquid encourages us to study its utility for one-pot synthesis of other heterocyclic compounds. The reaction of aromatic aldehyde, malononitrile, and active methylene carbonyl compounds for synthesis of 4*H*-benzo[*b*]pyrans has been reported. We then investigated the three-component condesation of aromatic aldehydes, malononitrile, and active methylene carbonyl compounds using [bmim]OH as a catalyst (Scheme 4). When 5 mol% [bmim]OH was used, the corresponding 4*H*-benzo[*b*]pyrans were obtained in good to excellent yields at room temperature, and the results are summarized in Table 3.

As shown in Table 3, the substituents of aromatic aldehydes affect the reaction times, and the yields of 4*H*-benzo[*b*]pyrans to a large extent. When aromatic aldehydes with electron-withdrawing groups (such as nitro group and halide) are reactants, the reaction time is shorter than that with electron-donating groups (such as methoxy group and hydroxyl group).

CONCLUSION

In summary, we have developed an efficient and general method for the synthesis of polyfunctionalized 4*H*-

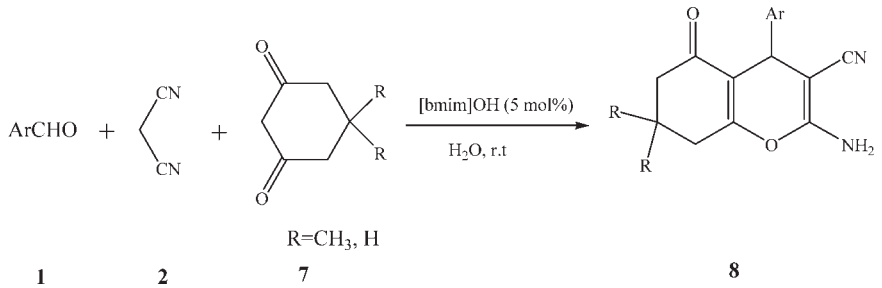
Table 2

Synthesis of **6a–6e** using [bmim]OH as catalyst in aqueous medium.[a]

Entry	Ar	R	Time (hr)	Product	Yield (%)[b]	Mp (°C) Found	Mp (°C) Reported
1	C_6H_5	H	2	**6a**	–		
2	C_6H_5	3-OH	2	**6b**	90	230–232	232–234[13a]
3	2-Cl-C_6H_4	3-OH	2	**6c**	88	95–96	94–95[13c]
4	4-Cl-C_6H_4	3-OH	2	**6d**	94	162–163	161–162[13c]
5	4-CH_3O-C_6H_4	3-OH	2	**6e**	90	112–113	112–114[13c]

[a] Reaction conditions: aldehyde (5 mmol), malononitrile (5 mmol), phenol (5 mmol), [bmim]OH (0.5 mmol), H_2O (2 mL), 100°C.
[b] Isolated yield.

Scheme 4. Synthesis of 4*H*-benzo[*b*]pyrans (**8a-8z**).



pyrans via [bmim]OH-catalyzed the one-pot, three-component reaction of aromatic aldehyde, malononitrile, and 4-hydroxycoumarin or active methylene carbonyl compounds. The attractive features of this protocol are simple procedure, short reaction time, excellent yields, mild conditions, and the easy workup procedure. In addition, water was chosen as a green solvent.

EXPERIMENTAL

General. Melting points were determined on an X6-data microscopic melting points apparatus and were uncorrected. Infrared (IR) spectra were recorded on a BRUKER VECTER 22. ^{1}H NMR spectra were obtained from solution $CDCl_3$ or DMSO-d_6 with TMS as internal standard using a BRUKER DRX-500 (500 MHz) spectrometer. Mass spectra were obtained with an automated Finnigan Trace Ultra-Trace DSQ GC/MS spectrometer.

The synthesis of this task-specific ionic liquid has been carried out from a similar method in the literature [16]. The ionic liquid was formed quantitatively and in high purity as assessed by ^{1}H NMR. All other chemicals (AR grade) were commercially available and used without further purification.

General procedure for the synthesis of 4a–4l and 6a–6e. The mixture of the aromatic aldehyde **1** (5 mmol), malononitrile **2** (5 mmol), phenol **3** or **5** (5 mmol), [bmim]OH (0.5

Table 3

Synthesis of 4*H*-benzo[*b*]pyrans catalyzed by [bmim]OH in aqueous media.[a]

Entry	Ar	R	Time (min)	Product	Yield (%)[b]	Mp (°C) Found	Mp (°C) Reported
1	C_6H_5	CH_3	10	**8a**	92	223–224	224[8b]
2	2-Cl-C_6H_4	CH_3	12	**8b**	90	211–212	214–215[8e]
3	4-Cl-C_6H_4	CH_3	5	**8c**	94	208–209	207–209[8b]
4	2,4-Cl_2-C_6H_3	CH_3	10	**8d**	90	192–193	192–194[8d]
5	4-F-C_6H_4	CH_3	5	**8e**	93	190–191	192–194[8a]
6	2-NO_2-C_6H_4	CH_3	15	**8f**	91	223–224	222–223[8d]
7	3-NO_2-C_6H_4	CH_3	10	**8g**	92	208–209	208–210[8b]
8	4-NO_2-C_6H_4	CH_3	5	**8h**	96	179–180	179[8b]
9	4-CH_3-C_6H_4	CH_3	20	**8i**	93	217–218	218[8c]
10	4-CH_3O-C_6H_4	CH_3	20	**8j**	87	198–200	201[8b]
11	4-OH-C_6H_4	CH_3	60	**4k**	85	204–205	205–206[8e]
12	4-NMe_2-C_6H_4	CH_3	20	**8l**	86	201–202	198–200[8b]
13	2-furyl	CH_3	20	**8m**	91	215–216	218–220[8c]
14	C_6H_5	H	15	**8n**	90	218–220	
15	2-Cl-C_6H_4	H	15	**8o**	91	212–214	213–215[8a]
16	4-Cl-C_6H_4	H	6	**8p**	93	227–228	226–228[8e]
17	2,4-Cl_2-C_6H_4	H	10	**8q**	90	224–225	225–227[8a]
18	4-F-C_6H_4	H	8	**8r**	92	213–215	
19	2-NO_2-C_6H_4	H	10	**8s**	91	196–198	
20	3-NO_2-C_6H_4	H	8	**8t**	92	201–202	198–200[13b]
21	4-NO_2-C_6H_4	H	6	**8u**	95	235–236	235–236[8b]
22	4-CH_3-C_6H_4	H	10	**8v**	90	214–216	
23	4-CH_3O-C_6H_4	H	30	**8w**	90	192–193	193–195[8a]
24	4-OH-C_6H_4	H	60	**8x**	91	234–236	
25	4-NMe_2-C_6H_4	H	40	**8y**	85	168–170	
26	2-furyl	H	20	**8z**	92	222–224	

[a] Reaction conditions: aldehyde (5 mmol), malononitrile (5 mmol), 5, 5-dimethyl-1, 3-cyclohexanedione or 1, 3-cyclohexanedione (5 mmol), [bmim]OH (0.25 mmol), H_2O (2 mL), room temperature.
[b] Isolated yield.

mmol) in H_2O (2 mL) was stirred at 100°C for the appropriate time (monitored by thin-layer chromatography [TLC]). After completion of the reaction, the solid compound obtained was filtered off and washed with H_2O (10 mL). The crude products were purified by recrystallization from EtOH (95%).

Data of selected compounds are given below:

Compound **4a**: IR (KBr): ν_{max} = 3379, 3290, 3179, 2196, 1712, 1671, 1603 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 4.67 (1 H, s, H-4), 4.80 (2 H, s, NH_2), 7.35–7.49 (6 H, m, ArH), 7.61–7.64 (1 H, t, ArH), 7.82–7.84 (1 H, d, ArH) ppm. ESI^+-MS: *m/z* (%) 316.2 (M^+, 23), 239.1 (100).

Compound **4c**: IR (KBr): ν_{max} = 3382, 3315, 3189, 2194, 1715, 1673, 1606 cm^{-1}. ^1H NMR (DMSO-d_6): δ = 4.52 (1 H, s, H-4), 7.32 (2 H, d, *J* = 8.2 Hz, ArH), 7.36 (2 H, s, NH_2), 7.38 (2 H, brs, ArH), 7.42 (1 H, d, *J* = 8.2 Hz, ArH), 7.50 (1 H, t, *J* = 7.6 Hz, ArH), 7.70 (1 H, t, *J* = 7.8 Hz, ArH), 7.93 (1 H, t, *J* = 7.6 Hz, ArH) ppm.

Compound **6b**: IR (KBr): ν_{max} = 3429, 3211, 2191, 1651, 1506, 1458 cm^{-1}. ^1H NMR (DMSO-d_6): δ = 4.62 (1 H, s, H-4), 6.41 (1 H, s, ArH), 6.48 (1 H, d, *J* = 8.3 Hz, ArH), 6.80 (1 H, d, *J* = 9.2 Hz, ArH), 6.85 (2 H, s, NH_2), 7.16–7.22 (3 H, m, ArH), 7.31 (2 H, t, *J* = 7.1 Hz, ArH), 9.68 (1 H, s, ArOH) ppm. ESI^--MS: *m/z* (%) 263.1 (M^-, 20), 218.0 (85), 197.0 (100).

General procedure for the synthesis of 4H-benzo[b] pyrans (8a–8z). The mixture of the aromatic aldehyde **1** (5 mmol), malononitrile **2** (5 mmol), 5, 5-dimethyl-1, 3-cyclohexanedione, or 1, 3-cyclohexanedione **7** (5 mmol), [bmim]OH (0.25 mmol) in H_2O (2 mL) was stirred at room temperature for the appropriate time (monitored by TLC). After completion of the reaction, the solid compound obtained was filtered off and washed with H_2O (10 mL). The crude products were purified by recrystalization from EtOH (95%).

Data of selected compounds are given below:

Compound **8a**: IR (KBr): ν_{max} = 3394, 3251, 2965, 2197, 1670, 1603 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 1.04 (3 H, s, CH_3), 1.11 (3 H, s, CH_3), 2.18–2.26 (2H, q, CH_2), 2.42–2.49 (2 H, m, CH_2), 4.40 (1 H, s, H-4), 4.52 (2 H, s, NH_2), 7.18–7.30 (5 H, m, ArH) ppm. ESI^--MS: *m/z* (%) 293.2 (M^+, 38), 65.1 (100).

Compound **8h**: IR (KBr): ν_{max} = 3451, 3321, 3209, 2193, 1655, 1599 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 1.05 (3 H, s, CH_3), 1.14 (3 H, s, CH_3), 2.12–2.26 (2 H, q, CH_2), 2.50 (2 H, s, CH_2), 4.53 (1 H, s, H-4), 4.68 (2 H, s, NH_2), 7.43 (2 H, d, *J* = 8.5 Hz, ArH), 8.18 (2 H, d, *J* = 8.5 Hz, ArH) ppm.

Compound **8i**: IR (KBr): ν_{max} = 3424, 3324, 2958, 2921, 2191, 1676, 1511 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 1.04 (3 H, s, CH_3), 1.11 (3 H, s, CH_3), 2.18–2.26 (2 H, q, CH_2), 2.29 (3 H, s, CH_3), 2.45 (2 H, s, CH_2), 4.36 (1 H, s, H-4), 4.50 (2 H, s, NH_2), 7.08–7.12 (4 H, m, ArH) ppm.

Compound **8n**: IR (KBr): ν_{max} = 3326, 3172, 2926, 2193, 1685, 1650 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 1.99–2.08 (2 H, m, CH_2), 2.34–2.39 (2 H, m, CH_2), 2.57–2.65 (2H, m, CH_2), 4.43(1 H, s, H-4), 4.52 (2 H, s, NH_2), 7.20–7.31 (5 H, m, ArH) ppm. ESI^--MS: *m/z* (%) 265.1 (M^+, 10), 65.1 (100).

Compound **8r**: IR (KBr): ν_{max} = 3412, 3334, 3257, 2928, 2192, 1651, 1603, 1503 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 2.02–2.08 (2 H, m, CH_2), 2.36–2.41 (2 H, m, CH_2), 2.59–2.64 (2H, m, CH_2), 4.44 (1 H, s, H-4), 4.66 (2 H, s, NH_2), 6.98–7.01 (2 H, m, ArH), 7.22–7.25 (2 H, m, ArH) ppm. ESI^--MS: *m/z* (%) 283.1 (M^+, 10), 65.1 (100).

Compound **8s**: IR (KBr): ν_{max} = 3415, 3337, 3250, 3139, 2954, 2189, 1669, 1597 cm^{-1}. ^1H NMR($CDCl_3$): δ = 1.94–2.06 (2 H, m, CH_2), 2.29–2.32 (2 H, m, CH_2), 2.57–2.61 (2H, m, CH_2), 4.68 (1 H, s, H-4), 5.17 (2 H, s, NH_2), 7.32–7.35 (2 H, t, ArH), 7.51–7.54 (1 H, t, ArH), 7.77 (1 H, d, *J* = 8.0 Hz, ArH) ppm.

Compound **8v**: IR (KBr): ν_{max} = 3406, 3330, 3257, 3210, 2921, 2196, 1655, 1607 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 2.01–2.08 (2 H, m, CH_2), 2.32 (3 H, s, CH_3), 2.36–2.40 (2 H, m, CH_2), 2.58–2.66 (2H, m, CH_2), 4.42 (1 H, s, H-4), 4.52 (2 H, s, NH_2), 7.11 (2 H, d, *J* = 7.7 Hz, ArH), 7.15 (2 H, d, *J* = 7.7 Hz, ArH) ppm. ESI^--MS: *m/z* (%) 279.0 (M^+, 10), 65.1 (100).

Compound **8x**: IR (KBr): ν_{max} = 3740, 3518, 3378, 3317, 3199, 2893, 2817, 2199, 1672, 1643, 1600 cm^{-1}. ^1H NMR (DMSO-d_6): δ = 1.87–1.97 (2 H, m, CH_2), 2.22–2.33 (2 H, m, CH_2), 2.51–2.63 (2H, m, CH_2), 4.09 (1 H, s, H-4), 6.65–6.67 (2 H, d, ArH), 6.89 (2 H, s, NH_2),6.94–6.95 (2 H, d, ArH), 9.22 (1 H, s, OH) ppm.

Compound **8y**: IR (KBr): ν_{max} = 3741, 3440, 3446, 3317, 3178, 2894, 2201, 1683, 1649, 1611, 1560 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 2.01–2.07 (2 H, m, CH_2), 2.35–2.40 (2 H, m, CH_2), 2.57–2.62 (2H, m, CH_2), 3.16 (6 H, s, $N(CH_3)_2$), 4.38 (1 H, s, H-4), 4.51 (2 H, s, NH_2), 6.70–6.72 (2 H, d, ArH), 7.80–7.82 (2 H, d, ArH) ppm.

Compound **8z**: IR (KBr): ν_{max} = 3397, 3322, 3254, 2961, 2188, 1651, 1507 cm^{-1}. ^1H NMR ($CDCl_3$): δ = 2.05–2.10 (2 H, m, CH_2), 2.39–2.48 (2 H, m, CH_2), 2.57–2.66 (2H, m, CH_2), 4.57 (2 H, s, NH_2), 4.62 (1 H, s, H-4), 6.21 (1 H, m, ArH), 6.30 (1 H, m, ArH), 7.23 (1 H, m, ArH) ppm.

Acknowledgments. This work is supported by the Innovation Foundation of NJUST (NanJing University of Science and Technology) for PhD Graduates.

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