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A task specific basic ionic liquid, [bmIm]OH-promoted efficient, green and one-pot synthesis of tetrahydrobenzo[*b*]pyran derivatives

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A basic ionic liquid, 1-butyl-3-methyl imidazolium hydroxide, [bmIm]OH has been used as an efficient catalyst for the synthesis of a variety of 4*H*-benzo[*b*]pyran derivatives by a one-pot three component condensation of aldehydes, cyclohexa-1,3-diones and malononitrile/ethyl cyano acetate at room temperature. The reactions are very fast (5-20 min), high yielding (85-98%) and do not require any organic solvent.

Keywords: Ionic liquid, aldehyde, multi-component condensation, 4*H*- benzo[*b*]pyran, green procedure

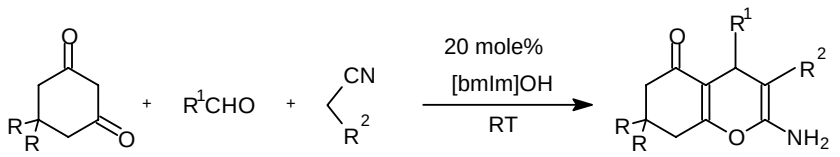
Recently ionic liquids have emerged as very potential green alternatives to the volatile and hazardous organic solvents and have been used as efficient and recyclable reaction media for a variety of important reactions[1]. We have been actively engaged in exploring new facets of ionic liquids as effective catalysts[2] and reagents[3] for last few years and as a part of this program we have recently introduced an apparently safe task specific ionic liquid, 1-butyl-3-methylimidazolium hydroxide, [bmIm]OH as an efficient catalyst for Michael reaction[2h] and Knoevenagel condensation[2l] and we will report here a simple synthesis of benzo[*b*]pyran derivatives by multi-component condensation using this ionic liquid. One pot multi-component condensation represents a possible instrument for ideal synthesis[4a] because by this way complex molecules could be built-up with maximum simplicity and brevity[4b].

4*H*-benzo[*b*]pyran derivatives are of considerable interest because of their useful biological and pharmacological properties, such as spasmolytic, diuretic, anti-coagulant, anti-cancer and anti-anaphylactic activity[5]. They are also used for the treatment of neurodegenerative disease, AIDS associated dementia and Down's syndrome as well as for the treatment of schizophrenia and myoclonus[6]. Besides, polysubstituted 4*H*-benzo[*b*]pyrans constitute structural unit of many natural products[7] and 2-amino-4*H*-benzo[*b*]pyrans are useful as photo active materials[8]. Thus, several methods were reported for the synthesis of these compounds[9-17]. However, many of these methods were associated with use of hazardous organic solvents, long duration of reaction, low yields of products and lack of general applicability; particularly synthesis of substituted 4*H*-benzo[*b*]pyrans was rarely addressed. This prompted us to develop a convenient method for the synthesis of 4-alkyl- and 4-aryl-4*H*-benzo[*b*]pyran derivatives and we report here a one pot synthesis of 4-alkyl- and 4-aryl-4*H*-benzo[*b*]pyran derivatives *via* a tandem Knoevenagal and cyclo-condensation reactions catalyzed by [bmIm]OH (**Scheme I**).

Results and Discussion

The experimental procedure is very simple. A mixture of an aldehyde, a 1,3-cyclohexadione derivative and an active methylene compound was stirred at RT in presence of an ionic liquid, [bmIm]OH for a certain period of time as required for completion (TLC). Recrystallization of the crude product provided pure benzo[*b*]pyran derivatives.

A variety of aliphatic (entries 1-8 in **Table I**), aromatic (entries 9-16 in **Table I**) and heteroaromatic (entries 14 and 15 in **Table I**) aldehydes underwent condensation with active methylene compounds (malononitrile and ethyl cyano acetate) and cyclohexa-1,3-dione (or dimedone) by this procedure to provide the corresponding 4-alkyl- and 4-aryl substituted 4*H*-benzo[*b*]pyran derivatives. The results are summarized in **Table I**.



Scheme I

Table I — Synthesis of tetrahydrobenzo[*b*]pyran derivatives catalyzed by [bmIm]OH

Entry	R	R^1	R^2	Time (min)	Yield (%)[a]	m.p. (^oC)	Ref.
1	Me	C_2H_5	CN	15	85	174-176	-
2	H	C_2H_5	CN	15	86	180-182	-
3	H	^iPr	CN	16	86	178-180	-
4	H	C_3H_7	CN	17	85	192-194	-
5	Me	C_3H_7	CN	16	88	168-170	-
6	H	C_7H_{15}	CN	20	86	161-163	-
7	Me	C_7H_{15}	CN	20	87	156-158	-
8	Me	C_9H_{19}	CN	20	85	134-136	-
9	Me	C_6H_5	CN	5	95	224-226	14
10	Me	4-$NO_2C_6H_4$	CN	5	98	156-158	14
11	Me	4-$MeOC_6H_4$	CN	6	97	202-204	14
12	Me	4-$MeOC_6H_4$	CO_2Et	10	89	182-184	17
13	Me	2-BrC_6H_4	CN	8	95	188-190	-
14	Me	2-Furyl	CN	8	90	196-198	14
15	Me	2-Thionyl	CN	7	92	210-212	14
16	Me	4-$SMeC_6H_4$	CN	5	94	208-209	-

[a]Yields refer to those of the pure isolated products characterized by spectroscopic (IR, ^1H and ^{13}C NMR) data and elemental analysis.

All these reactions are very fast (5-20 min) and high yielding (85-98%) compared to other existing procedures. No side product was observed in any reaction. Only 20 mole % of [bmIm]OH was sufficient to carry out the condensation and without [bmIm]OH reactions did not proceed. The ionic liquid acts here as catalyst as well as reaction medium. The ionic liquid was reused upto 5 runs without loss of catalytic activity. Although the ionic liquids, such as [bmIm]Br, [omIm]PF_6 and [bmIm]BF_4 (Ref.15) were used for the synthesis of tetrahydrobenzo[*b*] pyran derivatives by the same protocol, they were not effective for reactions with aliphatic aldehydes.

The reaction conditions are mild (RT) accepting several functional groups, such as Cl, Br, OMe, SMe, dioxomethylene, NO_2 and CO_2Et present in molecules. The highly sensitive molecules, furyl- and thionyl aldehydes participated in this reaction without any difficulty. The products are obtained by simple filtration followed by crystallization from ethanol, thus avoiding hazardous organic solvents completely. All products are easily identified by their spectroscopic (IR, ^1H and ^{13}C NMR) data.

In conclusion, an efficient catalytic activity of [bmIm]OH is demonsrated for a one-pot synthesis of 4*H*-benzo[*b*]pyran derivatives by a one-pot three

component condensation of aldehydes, cyclohexa-1,3-diones and malononitrile/ethyl cyano acetate under organic solvent free conditions. The significant advantages offered by this methodology are: i) operational simplicity, ii) general applicability to all types of aldehydes, iii) mild reaction conditions, iv) excellent yields of products and v) green procedure avoiding hazardous organic solvents and providing reusability of ionic liquid catalyst. Moreover, this demonstrates the potential of this ionic liquid in organic synthesis and leaves promise for further useful applications.

Experimental Section

The ionic liquid [bmIm]OH was prepared according to the procedure described in our earlier communication[2h]. IR spectra were recorded using samples on KBr pellets for solids on a Shimadzu 8300 FTIR spectrophotometer. ^{1}H and ^{13}C NMR spectra were recorded at 300 and 75 MHz respectively (Bruker 300 DPX instrument).

General procedure for the synthesis of tetrahydro benzo[*b*]pyran derivatives catalyzed by [bmIm]OH. Representative procedure for 2-amino-3-cyano-4-phenyl-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo[*b*]pyran (entry 9, Table I)

The ionic liquid, I-butyl-3-methylimidazolium hydroxide [bmIm]OH (47 mg, 20 mole%) was added to a mixture of benzaldehyde (106 mg, 1 mmole) and malononitrile (79 mg, 1.2 mmole) and the resulting mixture was stirred for 1-2 min. To this was added dimedone (140 mg, 1 mmole) in one portion and during stirring for 3 min the whole mass solidified. The solid was filtered through a small Buckner funnel under vacuum, washed with ethanol-H_2O (1:2) (2 × 3 mL) to leave the crude product which was purified by recrystallization from ethanol to provide pure 2-amino-3-cyano-4-phenyl-7,7-dimethyl-5-oxo-4*H*-5,6,7,8-tetrahydrobenzo[*b*]pyran as a white crystalline solid (288 mg, 98%), m.p. 227-29°C (lit.[14] m.p. 228-30°C); IR (KBr): 3380, 3299, 2964, 2198, 1684, 1654, 1600, 1508, 1369 cm^{-1}; ^{1}H NMR (DMSO-d_6, 300 MHz): δ 0.95 (s, 3H), 1.03 (s, 3H), 2.08 (d, *J* = 16.0 Hz, 1H), 2.24 (d, *J* = 16.0 Hz, 1H), 2.51 (s, 2H), 4.19 (s, 1H), 6.98 (s, 2H), 7.14-7.19 (m, 3H), 7.26-7.31 (m, 2H); ^{13}C NMR (DMSO-d_6, 75 MHz): δ 27.7, 29.3, 32.6, 36.8, 40.9, 50.9, 59.2, 113.6, 120.6, 127.4, 128.0 (2C), 129.2 (2C), 145.6, 159.4, 163.3, 196.5. These values are in good agreement with the literature[14].

This procedure was followed for the preparation of all tetrahydrobenzo[*b*]pyran derivatives listed in **Table I**. Several of these compounds are known and these were identified by comparison of their m.p. and spectroscopic data with those reported (See **Table I**). Those which are new were characterized by their IR, ^{1}H and ^{13}C NMR spectroscopic data and elemental analysis.

Spectroscopic data of these unknown products are given below in order of their entries.

2-Amino-4-ethyl-7,7-dimethyl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile (entry 1, Table I)

IR (KBr): 3423, 3317, 2183, 1674, 1651, 1600, 1379 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 0.76 (t, *J* = 7.5 Hz, 3H), 1.06 (s, 3H), 1.07 (s, 3H), 1.49-1.69 (m, 2H), 2.30 (s, 2H), 2.33 (s, 2H), 3.38 (t, *J* = 4.4 Hz, 1H), 4.68 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 8.8, 26.9, 27.4, 29.0, 30.1, 31.9, 40.6, 50.8, 60.0, 113.2, 119.3, 159.1, 162.9, 196.7. Anal. Calcd. for $C_{14}H_{18}N_2O_2$: C, 68.27; H, 7.37; N, 11.37. Found: C, 68.10, H, 7.21, N, 11.24%.

2-Amino-4-ethyl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-carbonitrile (entry 2, Table I)

IR (KBr): 3400, 3320, 2183, 1675, 1650, 1600, 1380 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 0.68 (t, *J* = 7.2 Hz, 3H), 1.32-1.51 (m, 2H), 1.85-2.00 (m, 2H), 2.21-2.36 (m, 2H), 2.45-2.49 (m, 2H), 3.16 (t, *J* = 4.0 Hz, 1H), 6.67 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 6.8, 18.4, 24.9, 25.3, 28.5, 34.8, 53.5, 111.7, 118.4, 158.2, 163.3, 194.5; Anal. Calcd. for $C_{12}H_{14}N_2O_2$: C, 66.03; H, 6.47; N, 12.84. Found: C, 65.91; H, 6.29; N, 12.69%.

2-Amino-4-isopropyl-5-oxo-3,4,5,6,7,8-hexahydro-2*H*-chromene-3-carbonitrile (entry 3, Table I)

IR (KBr): 3363, 3327, 3192, 2189, 1681, 1658, 1602, 1382 cm^{-1}; ^{1}H NMR (DMSO-d_6): δ 0.87 (d, *J* = 6.7 Hz, 3H), 1.12 (d, *J* = 6.9 Hz, 3H), 1.89-1.95 (m, 1H), 2.10-2.27 (m, 2H), 2.46-2.64 (m, 2H), 2.72 (t, *J* = 6.0 Hz, 2H), 3.34 (d, *J* = 2.5 Hz, 1H), 6.69 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 15.1, 18.4, 18.5, 25.2, 31.8, 33.6, 35.1, 51.6, 113.2, 119.6, 159.5, 163.6, 194.6. Anal. Calcd. for $C_{13}H_{16}N_2O_2$: C, 67.22; H, 6.94; N, 12.06. Found: C, 67.15, H, 6.82; N, 12.01%.

2-Amino-5-oxo-4-propyl-3,4,5,6,7,8-hexahydro-2*H*-chromene-3-carbonitrile (entry 4, Table I)

IR (KBr): 3386, 3328, 2958, 2192, 1678, 1654, 1601, 1380, 1213 cm^{-1}; ^1H NMR (DMSO-d_6): δ 0.82 (t, *J* = 6.9 Hz, 3H), 1.02 (s, 3H), 1.07 (s, 3H), 1.09-1.19 (m, 2H), 1.28-1.42 (m, 2H), 2.21 (s, 3H), 2.37 (s, 3H), 3.14 (t, *J* = 4.0 Hz, 1H), 6.69 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 14.5, 18.5, 27.6, 29.5, 30.0, 32.5, 38.0, 40.9, 51.1, 56.6, 113.8, 121.0, 160.7, 163.7, 196.8. Anal. Calcd. for $C_{15}H_{20}N_2O_2$: C, 69.20; H, 7.74; N, 10.76. Found: C, 69.04; H, 7.62; N, 10.61%.

2-Amino-7,7-dimethyl-5-oxo-4-propyl-3,4,5,6,7,8-hexahydro-2*H*-chromene-3-carbonitrile (entry 5, Table I)

IR (KBr): 3385, 3327, 2959, 2193, 1679, 1654, 1600, 1380, 1214 cm^{-1}; ^1H NMR (DMSO-d_6): δ 0.78 (t, *J* = 6.8 Hz, 3H), 1.08-1.15 (m, 2H), 1.27-1.35 (m, 2H), 1.84-1.89 (m, 2H), 2.27-2.69 (m, 2H), 2.43-2.45 (m, 2H), 3.11 (t, *J* = 4.1 Hz, 1H), 6.73 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 14.7, 18.4, 20.7, 27.2, 29.8, 37.2, 38.1, 56.5, 114.7, 121.2, 160.7, 166.2, 198.0. Anal. Calcd. for $C_{13}H_{16}N_2O_2$: C, 67.22; H, 6.94; N, 12.06. Found: C, 67.10; H, 6.80; N, 12.12%.

2-Amino-4-heptyl-5-oxo-3,4,5,6,7,8-hexahydro-2*H*-chromene-3-carbonitrile (entry 6, Table I)

IR (KBr): 3465, 3394, 3327, 2196, 1682, 1665, 1603, 1367 cm^{-1}; ^1H NMR (DMSO-d_6): δ 0.81 (t, *J* = 5.9 Hz, 3H), 0.98 (s, 3H), 1.00 (s, 3H), 1.18-1.42 (m, 12H), 2.12-2.45 (m, 4H), 3.13 (t, *J* = 3.9 Hz, 1H), 6.82 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 14.3, 22.4, 24.5, 26.9, 29.0, 29.1, 29.3, 29.6, 31.5, 32.0, 34.8, 40.0, 50.5, 55.9, 113.1, 120.5, 160.3, 163.6, 196.6. Anal. Calcd. for $C_{19}H_{28}N_2O_2$: C, 72.12; H, 8.92; N, 8.85. Found: C, 72.01; H, 8.79; N, 8.70%.

2-Amino-4-heptyl-7,7-dimethyl-5-oxo-3,4,5,6,7,8-hexahydro-2*H*-chromene-3 carbonitrile (entry 7, Table I)

IR (KBr): 3397, 3328, 2195, 1680, 1650, 1601, 1368 cm^{-1}; ^1H NMR (DMSO-d_6): δ 0.85 (t, *J* = 6.9 Hz, 3H), 1.17-1.41 (m, 10H), 1.42-1.53 (m, 2H), 1.93-2.05 (m, 2H), 2.27-2.52 (m, 4H), 3.38 (t, *J* = 4.7 Hz, 1H), 4.58 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 14.4, 20.6, 23.0, 25.1, 27.4, 29.4, 29.6, 29.7, 32.0, 32.2, 35.4, 37.3, 59.9, 115.4, 120.1, 159.6, 165.0, 197.5. Anal. Calcd. for $C_{17}H_{26}N_2O_2$: C, 70.31; H, 9.02; N, 9.65. Found: C, 70.20; H, 8.89; N, 9.49%.

2-Amino-7,7-dimethyl-4-nonyl-5-oxo-3,4,5,6,7,8-hexahydro-2*H*-chromene-3-carbonitrile (entry 8, Table I)

IR (KBr): 3460, 3390, 3328, 2190, 1882, 1600, 1365 cm^{-1}; ^1H NMR (DMSO-d_6): δ 0.85 (t, *J* = 6.9 Hz, 3H), 1.02 (s, 3H), 1.04 (s, 3H), 1.09-1.30 (m, 4H), 1.45-1.58 (m, 2H), 2.33 (s, 2H), 2.51 (s, 2H), 3.38 (t, *J* = 3.9 Hz, 1H), 4.54 (s, 2H); ^{13}C NMR (DMSO-d_6): δ 14.1, 22.6, 24.9, 27.4, 28.2, 29.1, 29.3, 29.5, 31.8, 32.0, 34.7, 46.2, 50.8, 54.1, 57.3, 114.0, 119.3, 158.8, 162.6, 197.0. Anal. Calcd. for $C_{21}H_{32}N_2O_2$: C, 73.22; H, 9.36; N, 8.13. Found: C, 73.09; H, 9.29; N, 8.02%.

2-Amino-4-(3-bromo-phenyl)-7,7-dimethyl-5-oxo-3,4,5,6,7,8-hexahydro-2*H*-chromene-3-carbonitrile (entry 13, Table I)

IR (KBr): 3460, 3392, 3327, 2195, 1680, 1650, 1600, 1360 cm^{-1}; ^1H NMR (DMSO-d_6): δ 0.95 (s, 3H), 1.00 (s, 3H), 2.03 (d, *J* = 10.0 Hz, 1H), 2.20 (d, *J* = 10.0 Hz, 1H), 2.49 (s, 2H), 4.70 (s, 1H), 7.00 (s, 2H), 7.05-7.12 (m, 2H), 7.24-7.29 (m, 1H), 7.48-7.51 (m, 1H); ^{13}C NMR (DMSO-d_6): δ 26.5, 27.9, 31.3, 34.5, 40.2, 55.6, 56.6, 111.6, 118.6, 122.2, 127.6, 128.0, 129.4, 132.2, 142.9, 158.1, 162.6, 195.0. Anal. Calcd. for $C_{18}H_{17}BrN_2O_2$: C, 57.92; H, 4.59; N, 7.51. Found: C, 57.84; H, 4.45; N, 7.40%.

2-Amino-7,7-dimethyl-4-(4-methylsulfanyl-phenyl)-5-oxo-3,4,5,6,7,8-hexahydro-2*H*-chromene-3-carbonitrile (entry 16, Table I)

IR (KBr): 3465, 3395, 3327, 2195, 1680, 1650, 1602, 1450, 1436, 1360 cm^{-1}; ^1H NMR (DMSO-d_6): δ 0.96 (s, 3H), 1.04 (s, 3H), 2.07 (d, *J* = 16.2 Hz, 1H), 2.18 (d, *J* = 16.2 Hz, 1H), 2.38 (s, 3H), 2.45 (s, 2H), 4.16 (s, 1H), 6.50 (s, 2H), 7.04-7.09 (m, 4H); ^{13}C NMR (DMSO-d_6): δ 13.8, 25.8, 27.3, 30.5, 33.8, 38.8, 49.0, 57.5, 114.7, 118.4, 124.7 (2C), 126.5 (2C), 134.9, 139.9, 157.2, 161.0, 194.7. Anal. Calcd. for $C_{19}H_{20}N_2O_2S$: C, 67.03; H, 5.92; N, 8.23. Found: C, 66.91; H, 5.80; N, 8.02%.

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