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HYPERVALENT IODINE(III) SULFONATE MEDIATED SYNTHESIS OF 5-BENZOYLDIHYDRO-2(3*H*)-FURANONE IN IONIC SOLVENT

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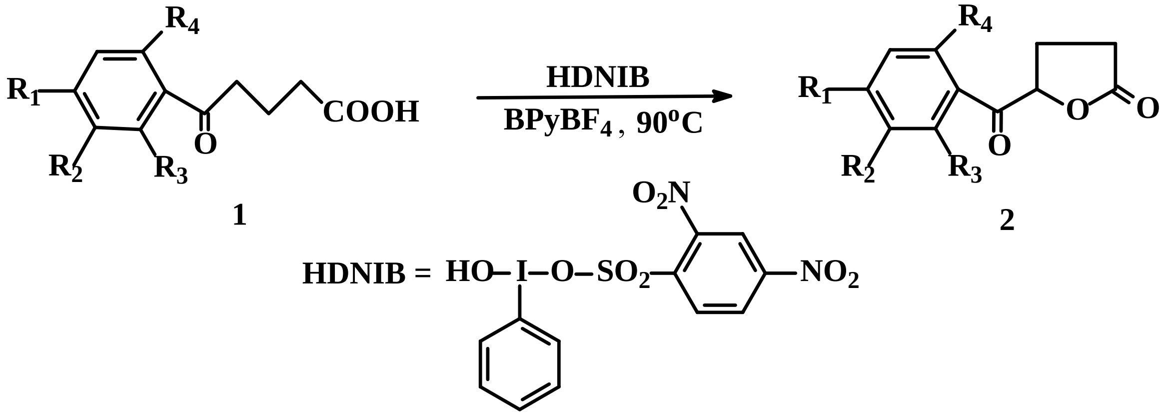
Abstract**—**The room temperature ionic liquid, *n*-butylpyridinium tetrafluoroborate ($BPyBF_4$) is used as a “green” recyclable solvent for the reaction of 4-benzoylbutyric acid with [hydroxy-(2,4-dinitrobenzenesulfonyloxy)iodo]-benzene (HDNIB) to prepare 5-benzoyldihydro-2(3*H*)-furanone.

Room temperature ionic liquids (RTIL) are liquids that are composed entirely of ions. In fact, ionic liquids can now be produced which remain liquid at room temperature and below (even as low as -90℃) and appear to be undemanding and inexpensive to manufacture.[1] Ionic liquids offer an attractive alterative to conventional organic liquids for clean synthesis, as they are easy to recycle, lack flammability, and possess effectively no vapour pressure. Compared with classical molecular solvents, the ionic liquids are environmentally benign reaction media.[2] To date some of the more important reactions have been carried out and investigated in ionic liquids, for example, Friedel–Crafts reaction,[3] alkoxycarbonylation,[4] hydrogenation,[5] Diels–Alder reaction,[6] Wittig reaction,[7] Heck reaction,[8] Trost-Tsuji coupling,[9] ring-closing metathesis (RCM),[10] Suzuki cross-coupling,[11] Fischer indole synthesis,[12] 1,3-dipolar cycloaddition reaction,[13] Beckmann rearrangement,[14] the Knoevenagel, and Robinson annulation reactions,[15] *etc*.

Lactonisation methodology plays an important role in modern organic synthetic chemistry not only because lactones occur in nature in great abundance and variety,[16] but also because they constitute a particularly useful class of synthons.[17] The preparation of lactones was *via* cyclization of acyclic olefinic carboxylic acids.[18] Recently, Moriarty *et al*. have developed a simple and useful route to synthesize 5-benzoyldihydro-2(3*H*)-furanone by using [hydroxy(tosyloxy)iodo]benzene (HTIB). [19] As part of a program to investigate the range of organic reactions possible in ionic liquids, we were interested in the reaction of 4-benzoylbutyric acid with [hydroxy-(2,4-dinitrobenzenesulfonyloxy)iodo]benzene (HDNIB) to prepare 5-benzoyldihydro-2(3*H*)-furanone in ionic liquids. The required HDNIB was prepared in satisfory yields from the reaction of 2,4-dinitrobenzenesulfonic acid with phenyliodine(III) diacetate(PIDA).[20]

For this study, the accessible and cheaper ionic liquid, *n*-butylpyridinium tetrafluoroborate ($BPyBF_4$) was a strong candidate and was synthesized according to the procedures reported in the literature.[1] The scope of the reaction of various 4-benzoylbutyric acids (**1**) with HDNIB in $BPyBF_4$ was investigated. We found that the reaction of 4-benzoylbutyric acids (**1**) with HDNIB occurred easily in $BPyBF_4$ at 90℃ for 2 h to give the desired 5-benzoyldihydro-2(3*H*)-furanones (**2**) in good yields (Scheme I). The results are summarized in Table 1. When the reaction was conducted in the classical solvents, such as dichloromethane, the preparation of 5-benzoyldihydro-2(3*H*)-furanone (**2a**) needs refluxing for 15 h by using HTIB.[19]



Scheme I

Table1. Preparation of 5-benzoyldihydro-2(3*H*)-furanones (**2**)

Entry	Product	R_1	R_2	R_3	R_4	Yield(%)
1	**2a**	H	H	H	H	76
2	**2b**	Me	H	H	H	72
3	**2c**	MeO	H	H	H	70
4	**2d**	Cl	H	H	H	78
5	**2e**	Br	H	H	H	73
6	**2f**	Me	H	H	Me	71
7	**2g**	H	Me	H	Me	75
8	**2h**	i-pr	H	H	H	80
9	**2i**	Me	H	Me	Me	84

Our experiments showed that after the isolation of the product, the ionic liquid $BPyBF_4$ can be resused several times without loss of activity (Table 2).

Table 2. Results obtained using recycled ionic liquid

Entry	Product	Cycle	Yield(%)
1	**2a**	1	75
2	**2a**	2	72
3	**2a**	3	73

In summary, the method described herein provides a good approach for the synthesis of 5-benzoyldihydro-2(3*H*)-furanones by the reaction of 4-benzoylbutyric acids with HDNIB in room temperature ionic liquid $BPyBF_4$ at 90℃ and gives good yields. Separation of products from the ionic liquids is very straight forward, as is recycling of the ionic liquid.

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EXPERIMENTAL

All melting points are uncorrected. The IR spectra were recorded on a Shimadzu IR-27 G spectrophotometer. ^1H NMR spectra were recorded on a Varian Unity Plus 400 MHz. Chemical shifts (δ) were measured in ppm with respect to TMS. MS were obtained on a JEOL JMS D-300 instrument. 5-Oxo-5-phenylpentanoic acid (**1a**) was commercially available from Aldrich.

Typical procedure for the preparation of 4-benzoylbutyric acids (1)[21]

A solution of glutaric andydride (1.14 g, 10 mmol) in dry toluene (10 mL) was added with stirring to an ice cold suspension of powdered andydrous $AlCl_3$ (3.33 g, 25 mmol) in dry toluene (5 mL). The mixture was kept at rt for 12 h and then warm at 60-65 ℃ for 0.5 h. The product was decomposed with ice and 37 % HCl and the excess of toluene removed by distillation. The solid product was extracted with hot $NaHCO_3$, the keto acid was precipitate with 37 % HCl to give **1b**.

5-Oxo-5-*p*-tolylpentanoic acid (1b)

mp 146-148 ℃ (AcOEt/hexane). IR (KBr) ν : 2937, 1701, 1654 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.08 (q, *J* = 7.2 Hz, 2H), 2.41 (s, 3H), 2.49 (t, *J* = 7.2 Hz, 2H), 3.05 (t, *J* = 6.8 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.86 (d, *J* = 8.4 Hz, 2H). EI-MS: 206 (M$^+$), 119, 91. Anal. Calcd for $C_{12}H_{14}O_3$: C, 69.89; H, 6.84. Found : C, 69.63; H, 6.72.

5-(4-Methoxyphenyl)-5-oxopentanoic acid (1c)

mp 136-138 ℃ (AcOEt/hexane). IR (KBr) ν : 2961, 1705, 1667 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.08 (q, *J* = 7.2 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 3.03 (t, *J* = 7.0 Hz, 2H), 3.87 (s, 3H), 6.93 (d, *J* =7.2 Hz, 2H), 7.94 (d, *J* =7.2 Hz, 2H). EI-MS: 222 (M$^+$), 150, 135. Anal. Calcd for $C_{12}H_{14}O_4$: C, 64.85; H, 6.35. Found : C, 64.63; H, 6.57.

5-(4-Chlorophenyl)-5-oxopentanoic acid (1d)

mp 118-120 ℃ (AcOEt/hexane). IR (KBr) ν : 2900, 1727, 1694 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.08 (q, *J* = 7.2 Hz, 2H), 2.50 (t, *J* = 7.0 Hz, 2H), 3.05 (t, *J* = 7.2 Hz, 2H), 7.44 (d, *J* = 8.4 Hz, 2H), 7.90 (d, *J* = 8.8 Hz, 2H). EI-MS: 226 (M$^+$), 141, 139. Anal. Calcd for $C_{11}H_{11}O_3Cl$: C, 58.29; H, 4.89. Found : C, 58.45; H, 5.08.

5-(4-Bromophenyl)-5-oxopentanoic acid (1e)

mp 116-118 ℃ (AcOEt/hexane). IR (KBr) ν : 2964, 1700, 1640 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.09 (q, *J* = 7.2 Hz, 2H), 2.48-2.52 (m, 2H), 3.03-3.10 (m, 2H), 7.55-7.62 (m, 2H), 7.95-7.98 (m, 2H). EI-MS: 273 (M$^+$+2), 271 (M$^+$), 185, 183. Anal. Calcd for $C_{11}H_{11}O_3Br$: C, 48.73; H, 4.09. Found : C, 48.51; H, 4.23.

5-(2,4-Dimethylphenyl)-5-oxopentanoic acid (1f)

mp 117-119 ℃ (AcOEt/hexane). IR (KBr) ν : 2937, 1704, 1679 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.05 (q, *J* = 7.2 Hz, 2H), 2.35 (s, 3H), 2.47 (t, *J* = 7.2 Hz, 2H), 2.49 (s, 3H), 2.99 (t, *J* = 7.2 Hz, 2H), 7.06-7.07 (m, 2H), 7.59 (d, *J* = 8.4 Hz, 1H). EI-MS: 220 (M$^+$), 133, 105. Anal. Calcd for $C_{13}H_{16}O_3$: C, 70.89; H, 7.32. Found : C, 69.54; H, 7.16.

5-(2,5-Dimethylphenyl)-5-oxopentanoic acid (1g)

mp 68-70 ℃ (AcOEt/hexane). IR (KBr) ν : 2937, 1704, 1679 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.05 (q, *J* = 7.2 Hz, 2H), 2.35 (s, 3H), 2.44 (s, 3H), 2.49 (t, *J* = 7.2 Hz, 2H), 2.98 (t, *J* = 7.2 Hz, 2H), 7.12 (d, *J* = 8.0 Hz, 1H), 7.18 (dd, *J* = 1.4, 8.0 Hz, 1H), 7.43 (s, 1H). EI-MS: 220 (M$^+$), 133, 105. Anal. Calcd for $C_{13}H_{16}O_3$: C, 70.89; H, 7.32. Found : C, 71.06; H, 7.54.

5-(4-Isopropylphenyl)-5-oxopentanoic acid (1h)

Oily compound. IR (neat) ν : 2959, 1691, 1604 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 1.25 (s, 6H), 2.08 (q, *J* = 7.2 Hz, 2H), 2.49 (t, *J* = 7.0 Hz, 2H), 2.85-2.99 (m, 1H), 3.06 (t, *J* = 7.2 Hz, 2H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.90 (d, *J* = 8.4 Hz, 2H). EI-MS: 234 (M$^+$), 147. HRMS (EI) Calcd for $C_{14}H_{18}O_3$: 234.1255. Found: 234.1256. Anal. Calcd for $C_{14}H_{18}O_3$: C, 71.77; H, 7.74. Found : C, 71,52; H, 7.58.

5-Oxo-5-(2,4,6-trimethylphenyl)pentanoic acid (1i)

Oily compound. IR (neat) ν : 2979, 1732, 1696 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.03 (q, *J* = 7.2 Hz, 2H), 2.16 (s, 6H), 2.26 (s, 3H), 2.42 (t, *J* = 7.2 Hz, 2H), 2.75 (t, *J* = 7.2 Hz, 2H), 6.81 (s, 2H). ^{13}CNMR ($CDCl_3$) δ : 17.1, 18.2, 20.2, 32.6, 42.9, 127.9, 131.7, 137.9, 173.0, 210.4. EI-MS: 234 (M^+), 147, 119. HRMS (EI) Calcd for $C_{14}H_{18}O_3$: 234.1255. Found: 234.1254. Anal. Calcd for $C_{14}H_{18}O_3$: C, 71.77; H, 7.74. Found : C, 71,57; H, 7.62.

Typical procedure for the preparation of 5-benzoyldihydro-2(3*H*)-furanones (2)

A mixture of 4-benzoylbutyric acid (0.20 g, 1 mmol), HDNIB (0.94 g, 2 mmol) and ionic liquid (2 mL) was stirred for 2 h at 90 ℃. The reaction mixture was extracted with Et_2O. The remaining ionic liquid suspension was filtered, and reused after drying in vacuum. The combined ethereal solution was evaporated under reduced pressure. The residue was chromatography on silica gel column eluting with $CHCl_3$-AcOEt (9:1) to give **2a**.

5-Benzoyldihydro-2(3*H*)-furanone (2a)

mp 77-78 ℃ ($CHCl_3$/hexane) (lit.,[22] 78-79 ℃). IR (neat) ν : 1780, 1687, 1245, 1060 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.44-2.62 (m, 4H), 5.79-5.82 (m, 1H), 7.50-7.55 (m, 2H), 7.63-7.67 (m, 1H), 7.98 (dd, *J* = 1.2, 8.8 Hz, 2H). ^{13}CNMR ($CDCl_3$) δ : 24.9, 26.6, 78.1, 128.5, 128.8, 133.3, 134.1, 176.3, 194.4. EI-MS: 105, 77.

5-(4-Methylbenzoyl)dihydro-2(3*H*)-furanone (2b)

mp 89-90 ℃ ($CHCl_3$/hexane) (lit.,[23] 90-91 ℃). IR (neat) ν : 1768, 1689, 1229, 1066 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.44 (s, 3H), 2.44-2.62 (m, 4H), 5.76-5.79 (m, 1H), 7.31 (d, *J* = 8.4 Hz, 2H), 7.88 (d, *J* = 8.4 Hz, 2H). ^{13}CNMR ($CDCl_3$) δ : 21.7, 25.0, 26.8, 78.1, 128.8, 128.9, 129.6, 129.7, 131.0, 145.4, 176.3, 193.9. EI-MS: 204 (M^+), 119, 91.

5-(4-Methoxybenzoyl)dihydro-2(3*H*)-furanone (2c)

mp 121-123 ℃ ($CHCl_3$/hexane) (lit.,[22] 122-124 ℃). IR (neat) ν : 1773, 1683, 1244, 1071 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.48-2.65 (m, 4H), 3.89 (s, 3H), 5.73-5.76 (m, 1H), 6.98 (d, *J* = 8.8 Hz, 2H), 7.97 (d, *J* = 8.8 Hz, 2H). ^{13}CNMR ($CDCl_3$) δ : 25.0, 26.9, 55.6, 78.1, 114.2, 126.6, 131.2, 164.4, 176.4, 192.7. EI-MS: 220 (M^+), 135, 77.

5-(4-Chlorobenzoyl)dihydro-2(3*H*)-furanone (2d)

mp 92-93 ℃ ($CHCl_3$/hexane) (lit.,[23] 93-94 ℃). IR (neat) ν : 1773, 1694, 1282, 1071 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.54-2.59 (m, 4H), 5.69-5.74 (m, 1H), 7.49 (dd, *J* = 2.0, 6.4 Hz, 2H), 7.95 (dd, *J* = 2.0, 6.8 Hz, 2H). ^{13}CNMR ($CDCl_3$) δ : 24.6, 26.8, 78.2, 129.3, 130.2, 131.9, 140.9, 175.9, 193.2. EI-MS: 141, 139.

5-(4-Bromobenzoyl)dihydro-2(3*H*)-furanone (2e)

mp 71-73 ℃ ($CHCl_3$/hexane) (lit.,[23] 70-72 ℃). IR (neat) ν : 1780, 1690, 1244, 1064 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.45-2.63 (m, 4H), 5.79-5.82 (m, 1H), 7.63-7.68 (m, 2H), 7.97-8.00 (m, 2H). ^{13}CNMR ($CDCl_3$) δ : 24.9, 26.7, 78.2, 128.7, 128.9, 130.2, 132.3, 133.4, 134.2, 176.3, 194.3. EI-MS: 271, 269 (M^+), 185, 183, 155, 77.

5-(2,4-Dimethylbenzoyl)dihydro-2(3*H*)-furanone (2f)

mp 145-146 ℃ ($CHCl_3$/hexane) (lit.,[23] 146 ℃). IR (neat) ν : 1783, 1688, 1245, 1063 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.33-2.62 (m, 4H), 2.38 (s, 3H), 2.52 (s, 3H), 5.69-5.72 (m, 1H), 7.11 (d, *J* = 8.0 Hz, 1H), 7.12 (s, 1H), 7.59 (d, *J* = 8.0 Hz, 1H). ^{13}CNMR ($CDCl_3$) δ : 21.4, 21.5, 25.2, 26.8, 78.9, 128.5, 129.4, 130.5, 133.3, 140.4, 143.5, 176.5, 196.8. EI-MS: 218 (M^+), 133, 105.

5-(2,5-Dimethylbenzoyl)dihydro-2(3*H*)-furanone (2g)

Oily compound. IR (neat) ν : 1783, 1692, 1244, 1063 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.38 (s, 3H), 2.47 (s, 3H), 2.49-2.64 (m, 4H), 5.68-5.72 (m, 1H), 7.18 (d, *J* = 8.0 Hz, 1H), 7.26-7.27 (m, 1H), 7.44 (s, 1H). ^{13}CNMR ($CDCl_3$) δ : 20.6, 20.7, 25.0, 26.7, 79.1, 129.3, 132.1, 133.2, 135.4, 136.5, 137.2, 176.4, 197.8. EI-MS: 218 (M^+), 133, 105. HRMS (EI) Calcd for $C_{13}H_{14}O_3$: 218.0943. Found: 218.0946. Anal. Calcd for $C_{13}H_{14}O_3$: C, 71.54; H, 6.47. Found: C, 71.42; H, 6.63

5-(4-Isopropylbenzoyl)dihydro-2(3*H*)-furanone (2h)

mp 81-83 ℃ ($CHCl_3$/hexane). IR (KBr) ν : 1770, 1687, 1601, 1225, 1072 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 1.28 (d, *J* = 6.8 Hz, 6H), 2.45-2.64 (m, 4H), 2.95-3.02 (m, 1H), 5.77-5.80 (m, 1H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.91 (d, *J* = 8.4 Hz, 2H). ^{13}CNMR ($CDCl_3$) δ : 23.4, 24.9, 26.7, 34.2, 78.1, 126.9, 128.9, 131.2, 176.2, 193.8. EI-MS: 147, 91. Anal. Calcd for $C_{14}H_{16}O_3$: C, 72.39; H, 6.94. Found: C, 72.43; H, 6.85.

5-(2,4,6-Trimethylbenzoyl)dihydro-2(3*H*)-furanone (2i)

Oily compound. IR (neat) ν : 1787, 1707, 1609, 1251, 1063 cm^{-1}. ^{1}HNMR ($CDCl_3$) δ : 2.17 (s, 6H), 2.24 (s, 3H), 2.16-2.61 (m, 4H), 5.23-5.26 (m, 1H), 6.78 (s, 2H). ^{13}CNMR ($CDCl_3$) δ : 19.5, 21.0, 24.5, 24.6, 26.9, 81.8, 128.8, 130.6, 134.0, 134.9, 137.3, 139.9, 176.0, 205.6. EI-MS: 232 (M^+), 149, 147, 121, 105. HRMS (EI) Calcd for $C_{14}H_{16}O_3$: 232.1099. Found: 232.1097. Anal. Calcd for $C_{14}H_{16}O_3$: C, 72.39; H, 6.94. Found: C, 72.52; H, 6.78

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