Reformatsky reaction promoted by an ionic liquid ([Bmim]Cl) in the synthesis of β-hydroxyl ketone derivatives bearing a coumarin unit

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A number of β-hydroxy-ketone derivatives bearing a coumarin unit have been synthesised by the Reformatsky reaction of formyl coumarin and α-bromoacetophenone in an ionic liquid (1-butyl-3-methylimidazolium chloride, [Bmim]Cl). The recovered ionic liquid can be reused at least five times without a significant reduction in yield. This procedure is green, mild and environmentally benign.

Keywords: Reformatsky reaction, β-hydroxyl ketone, coumarin, ionic liquid

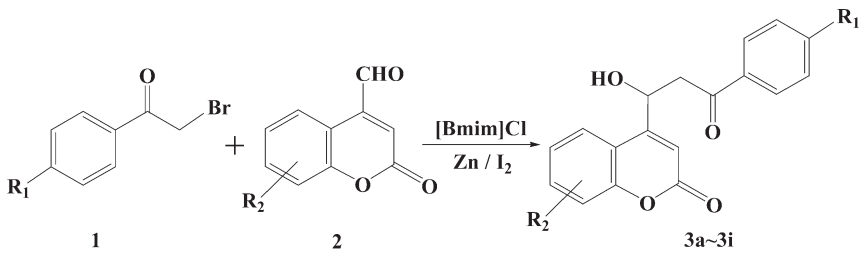
Since its discovery in 1887, the Reformatsky reaction has been one of the important reactions for carbon–carbon bond formation in organic chemistry.[1] With constant improvement, the Reformatsky reaction has been applied to the reaction of α-halo-ketones and aldehydes or ketones in which the corresponding products were β-hydroxy-ketone.[2–4] It offers the advantage of regioselective enolate formation under nearly neutral conditions.[5–7] However, the classic method employing zinc dust as a reactant can be plagued by extended reaction times and/or byproduct formation.[8]Many other approaches have been developed. to address these concerns, employing either alternative elemental reactants or low-valent organometallic species.[9–11] Unfortunately, several reagents have limitations of cost, availability, toxicity, and/or selectivity. Recently, many green approaches to Reformatsky reaction using aqueous media had been reported.[12–15]

In general, these reactions are carried out in organic solvents. Most organic solvents are flammable and may cause contamination of the environment. Accordingly, the use of green solvent in organic reactions is currently one of the most important issues in green chemistry, a field where new chemical methodologies are being developed to reduce the environmental impact of many chemical processes. Ionic liquids as solvent have been used in many organic reactions.[16–18] However, the Reformatsky reaction in ionic liquid is little known. In order to establish suitable conditions for the synthesis of β-hydroxy-ketone derivatives bearing coumarin unit, we investigated the Reformatsky reaction of formyl coumarin and α-bromoacetophenone in the ionic liquid of 1-butyl-3-methylimidazolium choloride ([Bmim]Cl) (Scheme 1).

Results and discussion

α-Bromoacetophenone derivatives (**1**) were prepared by the reaction of the acetophenones (acetophenone, 4-methyl acetophenone and 4-methoxy acetophenone) with bromine in diethyl ether in the presence of $AlCl_3$. Three 2-oxo-2H-chromene carbaldehydes (**2**) were synthesised by the oxidation of the corresponding methyl 2-oxo-2H-chromene and their structures were confirmed on the basis of their spectroscopic data and elemental analysis. The β-Hydroxy-ketone derivatives bearing coumarin unit (**3a–i**) that were synthesised in the ionic liquid ([Bmim]Cl) are shown in Scheme 1. α-Bromoacetophenones activated by zinc powder reacted with the formyl coumarins in ionic liquid to give the products. The whole procedure was very simple: A mixture of α-bromoacetophenone, formyl coumarin, zinc powder, trace iodine and anhydrous IL was stirred at 50–60 °C. At the end of the reaction, water was added to the mixture, and the crude product was extracted from the reaction medium with ethyl acetate. The pure product was isolated by the column chromatography.

To evaluate the effect of recycling on the efficiency of ionic liquid [Bmim]Cl, the ethyl acetate used to extract the residual water system was evaporated under vacuum and the residual ionic liquid was reused without any treatment for a third, fourth and fifth time. The recovered IL in mass had a little loss, but the activity did not decrease. Though the reaction time had slightly increased after a total of five cycles, the yield was still close to the first reaction, which indicated that the ionic liquid can be recycled and the reactivity of [Bmim]Cl was not decreased. The results of subsequent studies were shown in Table 1.



3a	R_1 = H,	R_2 = 6-CH_3
3b	R_1 = CH_3,	R_2 = 6-CH_3
3c	R_1 = OCH_3,	R_2 = 6-CH_3
3d	R_1 = H,	R_2 = 7-OCH_3
3e	R_1 = CH_3,	R_2 = 7-OCH_3
3f	R_1 = OCH_3,	R_2 = 7-OCH_3
3g	R_1 = H,	R_2 = 7, 8-benzo
3h	R_1 = CH_3,	R_2 = 7, 8-benzo
3i	R_1 = OCH_3,	R_2 = 7, 8-benzo

Scheme 1 Synthetic method of compounds **3a–i**.

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Table 1 Reuse of ionic liquid [Bmim]Cl in successive cycles of compound **3a** synthesis

Run[a]	Time /min	Yield/%[b]	IL Recovery/%[c]
1	200	76	97
2	200	74	96
3	240	74	96
4	240	70	95
5	240	70	95

[a]The conditions of all runs were consistent comparable to those in the experimental.
[b]Yields is the isolated product.
[c]Recovery percent of IL in mass is compared to the first reaction.

In conclusion, a green, solvent-free and environmentally benign method has been investigated for the synthesis of *β*-hydroxy-ketone derivatives bearing coumarin unit in ionic liquid. This reaction medium can be recycled five times without significant reduction in yield. These simple and mild conditions, moderate yields and multiple recycling of ionic liquid make this reaction useful and attractive from the environmental point of view.

Experimental

All reagents and solvents were of analytical grade and were obtained from commercial sources and used without further purification. Melting points were taken on a Stuart scientific melting point apparatus in open capillary tubes. ^{1}H-NMR spectra were recorded on a 400 MHz spectrometer in $CDCl_3$ with TMS as the internal standard. IR spectra were determined as KBr pellets on a Perkin-Elmer PE-683 IR spectrometer. Elemental analyses were performed on an Elementer Vario EL III elementary analysis instrument.

*Synthesis of α-bromoacetophenones **1**; general procedure*
Pure bromine (100 mL) was dropwise into the mixture of acetophenone (100 mmol) and anhydrous $AlCl_3$ (1.65 mmol) in anhydrous diethyl ether (20 mL) at 0 °C The crude product was precipitated, filtered and washed by the mixture of petroleum ether and wáter. It dried and recrystallised from the methanol to obtain the compounds **1**. The characterisation of compounds **1** were observed in the literature[19].

Synthesis of 2-oxo-chromene-4-carbaldehyde (**2**); *general procedure*
A mixture of 4-methyl-2H-chromen-2-one (20 mmol) and SeO_2 (32 mmol) in toluene (20 mL) was stirred and refluxed for 20 h. The reaction was monitored by TLC until it was complete. The deposit was separated by filtration and the filtrate was cooled to room temperature. A pure solid was formed and collected by filtration.

6-Methyl-2-oxo-2H-chromene-4-carbaldehyde: 72.9%; m.p. 165–167 °C. IR(KBr) υ: 3067 (ArH), 2923 (CH), 2873 (CH), 1738 (C=O), 1704 (C=O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 10.12 (s, 1H, CHO); 8.38 (s, 1H, ArH); 7.42 (d, 1H, *J* = 8.5 Hz, ArH); 7.29 (d, 1H, *J* = 8.5 Hz, ArH); 6.87 (s, 1H, CH=C); 2.44 (s, 3H, CH_3); Anal. Calcd for $C_{11}H_8O_3$: C, 70.21; H, 4.29. Found: C, 70.39; H, 4.10%.

7-Methoxy-2-oxo-2H-chromene-4-carbaldehyde: 59.6%; m.p. 200–202 °C. IR(KBr) υ: 3045 (ArH), 2972 (CH), 2857 (CH), 1712 (C=O), 1674 (C=O), 1211 (C-O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 10.07 (s, 1H, CHO); 8.50 (d, 1H, *J* = 9.0 Hz, ArH); 6.91 (d, 1H, *J* = 9.0 Hz, ArH); 6.87 (s, 1H, ArH); 6.71 (s, 1H, CH=C); 3.89 (s, 3H, CH_3); Anal. Calcd for $C_{11}H_8O_4$: C, 64.71; H, 3.95. Found: C, 64.59; H, 4.05%.

2-Oxo-2H-benzo[h]chromene-4-carbaldehyde: 65.9%; m.p. 202–203 °C; IR(KBr) υ: 3060 (ArH), 2928 (CH), 2844 (CH), 1758 (C=O), 1709 (C=O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 10.22 (s, 1H, CHO); 8.57 (d, 1H, *J* = 8.0 Hz, ArH); 8.54 (d, 1H, *J* = 8.0 Hz, ArH); 7.91 (d, 1H, *J* = 9.0 Hz, ArH); 7.76 (d, 1H, *J* = 9.0 Hz, ArH); 7.71–7.65 (m, 2H, ArH); 6.98 (s, 1H, CH=C); Anal. Calcd for $C_{14}H_8O_3$: C, 75.00; H, 3.60. Found: C, 75.12; H, 3.41%.

Synthesis of β-hydroxy-ketones (**3**); *general procedure*
The mixture of the 2-oxo-2H-chromene-4-carbaldehyde derivatives (4 mmol), *α*-bromoacetophenones (6 mmol), zinc powder (6 mmol) and a trace of iodine in ionic liquid [Bmim]Cl (3.0 g) were stirred at 55–60 °C for several hours. Water (15 mL) was added and the reaction mixture was extracted by ethyl acetate (10 mL × 3). The organic layer was dried and concentrated to obtain the crude product. Purification by column chromatography afforded the products in the moderate yield.

4-(1-Hydroxy-3-oxo-3-phenylpropyl)-6-methyl-2H-chromen-2-one (**3a**): 93%; m.p. 81–83 °C; IR(KBr) υ: 3433 (OH), 2916 (CH), 2844 (CH), 1715 (C=O), 1635 (C=O), 1167 (C–O) cm^{-1}; ^{1}H NMR($CDCl_3$, 400 MHz) δ: 8.05 (d, 2H, *J* = 8.0 Hz, ArH); 7.63 (t, 1H, *J* = 3.2 Hz, ArH); 7.54 (t, 2H, *J* = 7.5 Hz, ArH); 7.27 (d, 1H, *J* = 7.5 Hz, ArH); 7.25 (d, 1H, *J* = 7.5 Hz, ArH); 7.23 (s, 1H, ArH); 6.75 (s, 1H, CH=C); 5.72 (brs, 1H, OH); 3.76–3.62 (m, 1H, CH); 3.49 (dd, 1H, J_1 = 2.4, J_2 = 18.0 Hz, CH_2); 3.41 (dd, 1H, J_1 = 9.0, J_2 = 15.0 Hz, CH_2); 2.38 (s, 3H, CH_3); Anal. Calcd for $C_{19}H_{16}O_4$: C, 74.01; H, 5.23. Found: C, 74.11; H, 5.39%.

4-(1-Hydroxy-3-oxo-3-p-tolylpropyl)-6-methyl-2H-chromen-2-one (**3b**): 91%; m.p. 83–85 °C; IR(KBr) υ: 3402 (OH), 3066 (ArH), 2923 (CH), 1722 (C=O), 1653 (C=O), 1167 (C–O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.87 (d, 2H, *J* = 8.5 Hz, ArH); 7.34 (d, 2H, *J* = 8.5 Hz, ArH); 7.31 (d, 1H, *J* = 9.0 Hz, ArH); 7.29 (d, 1H, *J* = 9.0 Hz, ArH); 7.17 (s, 1H, ArH); 6.75 (s, 1H, CH=C); 5.72 (brs, 1H, OH); 3.76–3.73 (m, 1H, CH); 3.50 (dd, 1H, J_1 = 3.8, J_2 = 18.0 Hz, CH_2); 3.36 (dd, 1H, J_1 = 9.0, J_2 = 18.0 Hz, CH_2); 2.49 (s, 3H, CH_3); 2.39 (s, 3H, CH_3); Anal. Calcd for $C_{20}H_{18}O_4$: C, 74.52; H, 5.63. Found: C, 74.32; H, 5.71%.

4-(1-Hydroxy-3-(4-methoxyphenyl)-3-oxopropyl)-6-methyl-2H-chromen-2-one (**3c**): 91%; m.p. 166–169 °C; IR(KBr) υ: 3425 (OH), 2922 (CH), 1715 (C=O), 1689 (C=O), 1209 (C–O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.92 (d, 2H, *J* = 8.5 Hz, ArH); 7.34 (d, 2H, *J* = 8.5 Hz, ArH); 7.26 (d, 1H, *J* = 9.0 Hz, ArH); 7.24 (d, 1H, *J* = 9.0 Hz, ArH); 7.20 (s, 1H, ArH); 6.56 (s, 1H, CH=C); 5.74 (brs, 1H, OH); 3.98 (s, 3H, OCH_3); 3.74–3.70 (m, 1H, CH); 3.41 (dd, 1H, J_1 = 2.4, J_2 = 18.0 Hz, CH_2); 3.36 (dd, 1H, J_1 = 9.0, J_2 = 18.0 Hz, CH_2); 2.40 (s, 3H, CH_3); Anal. Calcd for $C_{20}H_{18}O_5$: C, 70.99; H, 5.36. Found: C, 71.15; H, 5.25%.

4-(1-Hydroxy-3-oxo-3-phenylpropyl)-7-methoxy-2H-chromen-2-one (**3d**): 91.%; m.p. 245–246 °C; IR(KBr) υ: 3425 (OH), 2922 (CH), 1715 (C=O), 1674 (C=O), 1209 (C–O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.96 (d, 2H, *J* = 8.0 Hz, ArH); 7.68 (t, 1H, *J* = 3.2 Hz, ArH); 7.60 (t, 2H, *J* = 3.2 Hz, ArH); 7.40 (d, 1H, *J* = 9.0 Hz, ArH); 6.95 (d, 1H, *J* = 9.0 Hz, ArH); 6.88 (s, 1H, ArH); 6.60 (s, 1H, CH=C); 5.73 (brs, 1H, OH); 3.92 (s, 3H, OCH_3); 3.64–3.63 (m, 1H, CH); 3.40 (dd, 2H, J_1 = 2.4, J_2 = 14.0 Hz, CH_2); Anal. Calcd for $C_{19}H_{16}O_5$: C, 70.36; H, 4.97. Found: C, 70.15; H, 5.04%.

4-(1-Hydroxy-3-oxo-3-p-tolylpropyl)-7-methoxy-2H-chromen-2-one (**3e**): 90.2%; m.p. 166–169 °C; IR(KBr) υ: 3730 (OH), 2958 (CH), 1718 (C=O), 1646 (C=O), 1209 (C–O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.84 (d, 2H, *J* = 8.5 Hz, ArH); 7.48 (d, 2H, *J* = 9.0 Hz, ArH); 7.35 (d, 1H, *J* = 9.0 Hz, ArH); 6.88 (s, 1H, ArH); 6.84 (d, 1H, *J* = 9.0 Hz, ArH); 6.59 (s, 1H, CH=C); 5.72 (brs, 1H, OH); 3.89 (s, 3H, OCH_3); 3.71–3.68 (m, 1H, CH); 3.46 (dd, 1H, J_1 = 2.4, J_2 = 18.0 Hz, CH_2); 3.38 (dd, 1H, J_1 = 9.0, J_2 = 18.0 Hz, CH_2); 2.40 (s, 3H, CH_3); Anal. Calcd for $C_{20}H_{18}O_5$: C, 70.99; H, 5.36;. Found: C, 70.87; H, 5.29%.

4-(1-Hydroxy-3-(4-methoxyphenyl)-3-oxopropyl)-7-methoxy-2H-chromen-2-one (**3f**): 92%; m.p. 172–175 °C; IR(KBr) υ: 3431 (OH), 2922 (CH), 1718 (C=O), 1682 (C=O), 1193 (C–O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 7.94 (d, 2H, *J* = 8.0 Hz, ArH); 7.44 (d, 2H, *J* = 8.0 Hz, ArH); 7.28 (d, 1H, *J* = 8.0 Hz, ArH); 6.97 (d, 1H, *J* = 8.0 Hz, ArH); 6.89 (s, 1H, ArH); 6.66 (s, 1H, CH=C); 5.74 (brs, 1H, OH); 3.99 (s, 3H, OCH_3); 3.96 (s, 3H, OCH_3); 3.68–3.65 (m, 1H, CH); 3.43 (dd, 1H, J_1 = 2.4, J_2 = 18.0 Hz, CH_2), 3.40 (dd, 1H, J_1 = 9.0, J_2 = 18.0 Hz, CH_2); Anal. Calcd for $C_{20}H_{18}O_6$: C, 67.79; H, 5.12;. Found: C, 67.51; H, 4.99%.

4-(1-Hydroxy-3-oxo-3-phenylpropyl)-2H-benzo[h]chromen-2-one (**3g**): 93.%; m.p. 158–160 °C; IR(KBr) υ: 3431 (OH), 2921 (CH), 1720 (C=O), 1679 (C=O), 1260 (C–O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.00 (d, 1H, *J* = 8.0 Hz, ArH); 7.86 (d, 2H, *J* = 8.5 Hz, ArH); 7.75 (d, 1H, *J* = 8.0 Hz, ArH); 7.71–7.65 (m, 2H, ArH); 7.42 (t, 2H, *J* = 8.5 Hz, ArH); 7.34 (t, 1H, *J* = 8.5 Hz, ArH); 7.28 (d, 1H, *J* = 7.5 Hz, ArH); 7.26 (d, 1H, *J* = 7.5 Hz, ArH); 6.40 (s, 1H, CH=C); 5.72 (brs, 1H, OH); 3.74–3.72 (m, 1H, CH); 3.53 (dd, 1H, J_1 = 2.4, J_2 = 18.0 Hz, CH_2), 3.49 (dd, 1H, J_1 = 9.0, J_2 = 18.0 Hz, CH_2); Anal. Calcd for $C_{22}H_{16}O_4$: C, 76.73; H, 4.68. Found: C, 76.59; H, 6.75%.

4-(1-Hydroxy-3-oxo-3-p-tolylpropyl)-2H-benzo[h]chromen-2-one (**3h**): 93%; m.p. 198–200 °C; IR(KBr) υ: 3445 (OH), 2921 (CH), 1710 (C=O), 1676 (C=O), 1263 (C–O) cm^{-1}; ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 8.02 (d, 1H, *J* = 8.0 Hz, ArH); 7.81 (d, 2H, *J* = 8.5 Hz, ArH); 7.75–7.71 (m, 2H, ArH); 7.68 (d, 1H, *J* = 8.0 Hz, ArH); 7.48 (d, 2H, *J* = 8.0 Hz, ArH); 7.35 (d, 1H, *J* = 8.0 Hz, ArH); 7.32 (d, 1H,

J = 8.0 Hz, ArH); 6.50 (s, 1H, CH=C); 5.73 (brs, 1H, OH); 3.68–3.65 (m, 1H, CH); 3.51 (dd, 1H, J_1 = 2.4, J_2 = 18.0 Hz, CH_2); 3.48 (dd, 1H, J_1 = 9.0, J_2 = 18.0 Hz, CH_2); 2.42 (s, 3H, CH_3); Anal. Calcd for $C_{23}H_{18}O_4$: C, 77.08; H, 5.06. Found: C, 76.95; H, 5.18%.

4-(1-Hydroxy-3-(4-methoxyphenyl)-3-oxopropyl)-2H-benzo[h]-chromen-2-one (**3i**): 93%; m.p. 179–182 °C; IR(KBr) υ: 3443 (OH), 2944 (CH), 1709 (C=O), 1678 (C=O), 1080 (C–O) cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 8.10 (d, 1H, J = 8.0 Hz, ArH), 7.92 (d, 1H, J = 8.0 Hz, ArH); 7.91 (d, 1H, J = 8.0 Hz, ArH); 7.72–7.66 (m, 2H, ArH); 7.54 (d, 2H, J = 8.0 Hz, ArH); 7.32 (d, 1H, J = 8.0 Hz, ArH); 7.30 (d, 1H, J = 8.0 Hz, ArH); 6.85 (s, 1H, CH=C); 5.72 (brs, 1H, OH); 3.97 (s, 3H, OCH_3); 3.87–3.85 (m, 1H, CH); 3.48 (dd, 1H, J_1 = 2.4, J_2 = 18.0 Hz, CH_2), 3.43 (dd, 1H, J_1 = 9.0, J_2 = 18.0 Hz, CH_2); Anal. Calcd for $C_{23}H_{18}O_5$: C, 73.79; H, 4.85. Found: C, 73.75; H, 4.96%.

Recycling of IL([Bmin]Cl)

The *β*-hydroxyl ketone derivatives were obtained from the organic layer of the reaction medium as above. Ethyl acetate extracted from the residual water layer and was concentrated, dried under vacuum and reused without any treatment.

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