Friedel–Crafts Alkylation of Nitrogen Heterocycles Using [Bmim][OTf] as a Catalyst and Reaction Medium

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Received 19 March 2008

Abstract: Friedel–Crafts alkylation of nitrogen heterocycles, such as indoles and pyrroles, can be carried out in ionic liquids under mild conditions to afford the corresponding alkylated product in moderate to good yields

Keywords: nitrogen heterocycle, ring opening, indole, pyrrole, ionic liquids, epoxide

Indole and pyrrole derivatives are important pharmacologically and biologically active compounds.[1] Alkylated pyrroles are also used for the development of functional organic materials.[2] Therefore a direct synthesis of their derivatives is desired.

Epoxides on the other hand are well-known carbon electrophiles, which can undergo ring-opening reactions with a number of nucleophiles to give rise to a vast array of synthetically important products.[3]

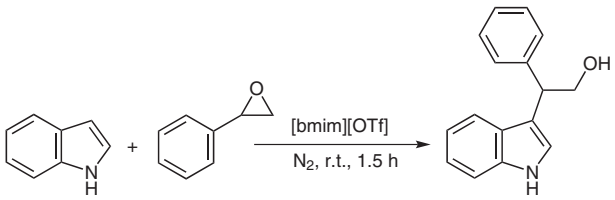
The general method for ring opening of epoxides with nitrogen heterocycles has been reported by using high-pressure conditions and acid catalysis.[4]

Recently, lanthanide triflates[5] and other Lewis acids such as $InBr_3$[6] and $InCl_3$[7] have also been reported as mild catalysts to facilitate this reaction. Enantioselective addition of indoles to aromatic epoxides, catalysed by chromium–salen complexes,[8] has also been reported. Although the Lewis acids initiate facile reactions under moderately mild conditions, they have the problem of waste disposal and high cost.

Industry favors catalytic processes induced by heterogeneous catalysts over homogeneous processes in view of the ease of handling, simple workup, and regenerability. Recently, our group reported the Friedel–Crafts alkylation of indoles by using nanocrystalline titanium(IV) oxide.[9] Bandgar et al.[10] have reported fluoroboric acid adsorbed on silica gel as a catalyst for ring opening of aryl epoxides with nitrogen heterocycles. Very recently, Das et al. have reported sulfated zirconia as a catalyst for these reactions.[11]

In the past decade, room-temperature ionic liquids (RTILs) have emerged as one of the most attractive ecobenevolent alternatives to volatile organic solvents and also as effective catalysts in many reactions.[12] The uniqueness of ionic liquids lies in their physical properties such as low vapor pressure, wide liquid range, high thermal stability, and highly conductive solvation ability for a variety of organic substrates and catalysts including Lewis acids and enzymes.[13] In addition, they can be recycled easily without any significant loss of activity. The use of these ionic liquids have been well demonstrated for various organic and biotransformations.[14,15]

In continuation of our interest to explore new reactions in ionic liquids, herein we report Friedel–Crafts alkylation of nitrogen heterocycles with indoles and pyrroles under ambient conditions in ionic liquids. This procedure allows a facile reaction, followed by a simple workup procedure to give the corresponding products in moderate to good yields (Scheme 1).



Scheme 1 Friedel–Crafts alkylation of indole with styrene oxide

A range of ionic liquids possessing different chemical properties was screened for the alkylation of indoles with styrene oxide. The ionic liquid [bmim][OTf] showed maximum efficiency (Table 1, entry 1).

Table 1 Screening of Different Ionic Liquids for Alkylation of Indoles[a]

Entry	Ionic liquid	Time (h)	Yield (%)[b]
1	[bmim][OTf]	1.5	85
2	[bmim][Br]	1.5	10
3	[bmim][PF_6]	1.5	–
4	[bmim][BF_4]	1.5	3
5	CH_2Cl_2	1.5	–

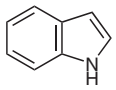
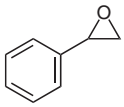
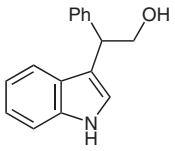
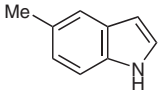
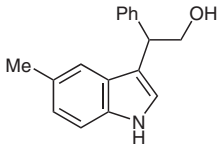
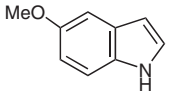
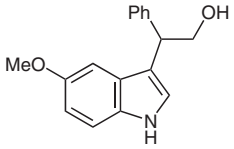
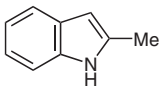
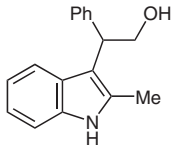
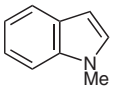
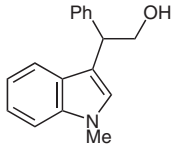
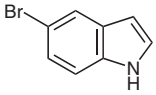
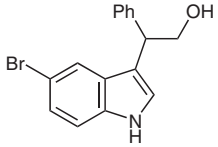
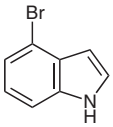
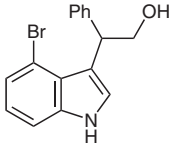
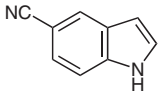
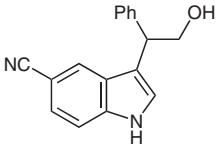
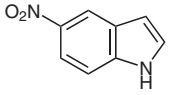
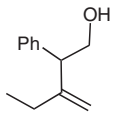
[a] Reaction conditions: indole (1.2 mmol), styrene oxide (1.0 mmol), ionic liquid (0.5 ml) stirred under N_2 atmosphere at r.t.
[b] Isolated yields.

SYNLETT 2008, No. 10, pp 1449–1454
Advanced online publication: 19.05.2008
DOI: 10.1055/s-2008-1078423; Art ID: D08908ST

The other ionic liquids gave very low yields of the product, whereas [bmim][PF_6] did not give any product at all. When the reaction was run in CH_2Cl_2, instead of ionic liquid, no product was formed even after prolonged time. The strong Lewis acid character of [bmim][OTf] may account for this increased activity. After choosing the ionic liquid [bmim][OTf] as the reaction medium, the substrates were added to the reaction in different molar ratios. The highest yields of the products were obtained when indole and epoxide reacted in the ratio 1.2:1, respectively.

To widen the scope of the [bmim][OTf]-promoted alkylation of indoles, a wide range of structurally varied indoles

Table 2 [Bmim][OTf]-Promoted Alkylation of Indoles with Styrene Oxide[a,16]

Entry	Nucleophile	Epoxide	Product	Time (h)	Yield (%)[b]
1				1.5	85, 82[c]
2				1.0	82
3				1.0	85
4				1.0	90
5				1.5	82
6				2.5	78
7				3.0	75
8				24	18
9				24	10

[a] Reaction conditions: indole (1.2 mmol), epoxide (1 mmol), [bmim][OTf] (0.5 mL), stirred under nitrogen atmosphere at r.t. for an appropriate time.
[b] Isolated yields.
[c] Yield after fourth cycle.

were subjected to undergo this reaction under the optimised reaction conditions affording the corresponding products in moderate to good yields (Table 2).

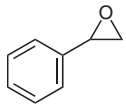
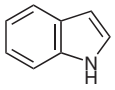
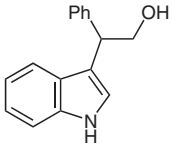
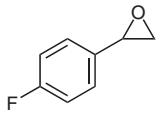
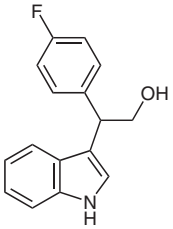
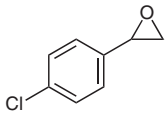
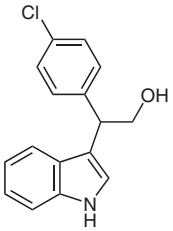
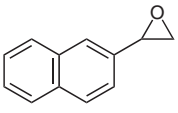
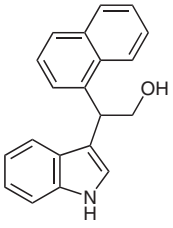
It was evident from the NMR spectral data that the aryl epoxides underwent cleavage by indoles with preferential attachment at the benzylic position, resulting in the formation of primary alcohols. Since the 3-position of indole is the preferred site for electrophilic substitution reactions, 3-alkyl indoles were obtained as the sole product in all reactions. In general, indoles bearing electron-donating groups furnished higher reaction rates, affording the alcohols in moderate to good yields (Table 2, entries 2–4). On the other hand, the presence of electron-withdrawing groups on the indole significantly decreased the rate of the reaction and a low yield of product was obtained even after prolonged reaction time. In the case of 5-nitro- and 5-cyanoindoles, very low yields of the product were observed (Table 2, entries 9 and 10). Significantly greater reactivity was observed in the case of *N*-methylindole, which gave a higher yield in comparison with indole (Table 2, entry 4). A variety of aromatic epoxides also underwent facile ring opening, resulting in the formation of the corresponding products in moderate to good yields (Table 3). The aromatic epoxides having electron-withdrawing groups reacted much faster (Table 3, entries 2 and 3). However, no product was formed by aliphatic epoxides.

In all cases, a single regioisomer was obtained and the structure was established by IR, ^{1}H NMR, and mass-spectrometric studies. The reactions were highly regioselective, affording good yields of products in a short period of time.

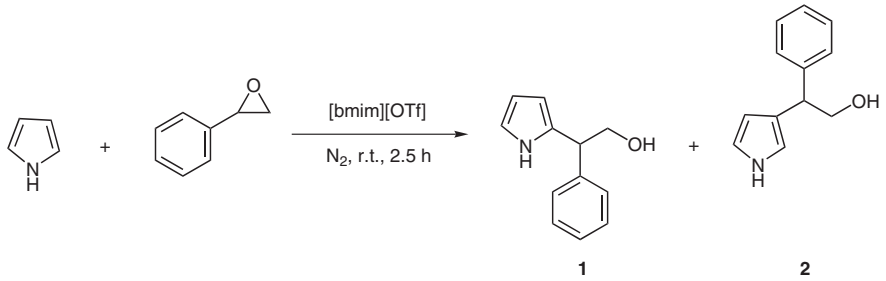
The methodology was further extended to the ring opening of aromatic epoxides with pyrroles (Scheme 2) to afford the corresponding products in moderate yields. The results are summarised in Table 4.

In all cases the reaction proceeded efficiently at ambient temperatures with high regioselectivity. It was observed that the epoxides also underwent cleavage selectively at the benzylic position to give the primary alcohol (Table 4). The products were obtained as a mixture of the 2-alkyl and 3-alkyl pyrroles, which were separated by column chromatography. Since 2-position of the pyrrole is the preferred site for the electrophilic substitution reaction, 2-alkyl pyrrole was obtained as a major product in all cases. These were characterised by IR, ^{1}H NMR, and mass-spectrometric studies. The presence of an electron-withdraw-

Table 3 [Bmim][OTf]-Promoted Ring Opening of Various Aromatic Epoxides with Indole[a,17]

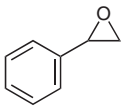
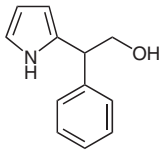
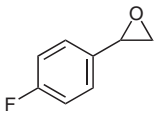
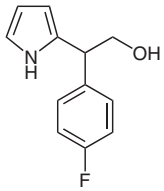
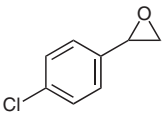
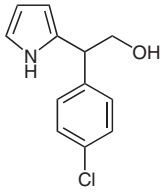
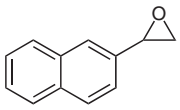
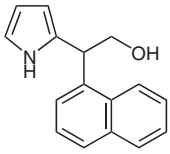
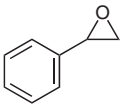
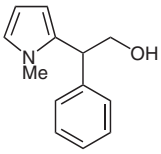
Entry	Nucleophile	Epoxide	Product	Time (h)	Yield (%)[b]
1				1.5	85
2				1.0	82
3				1.0	80
4				2.5	78

[a] Reaction conditions: indole (1.2 mmol), epoxide (1 mmol), [bmim][OTf] (0.5 mL), stirred under nitrogen atmosphere at r.t. for an appropriate time.
[b] Isolated yields.



Scheme 2 Friedel-Crafts alkylation of pyrrole with styrene oxide

Table 4 [Bmim][OTf]-Promoted Alkylation of Pyrroles with Aromatic Epoxides[a]

Entry	Nucleophile	Epoxide	Product	Time (h)	Yield (%)[b]
1				2.5	80
2				2.0	80
3				2.0	80
4				3.5	78
5				2.5	82

[a] Reaction conditions: pyrrole (1.2 mmol), epoxide (1 mmol), [bmim][OTf] (0.5 mL), stirred under nitrogen atmosphere at r.t. for an appropriate time.
[b] Isolated yields.

ing group in the epoxide gave a more facile reaction (Table 4, entries 2 and 3).

All reactions proceeded smoothly at ambient temperature with high regioselectivity, and good yields of the corresponding products were obtained in a short period. It is important to note that products arising from N-alkylation of indoles and pyrroles were not observed under these reaction conditions. Finally, upon completion of the reaction, the ionic liquid [bmim][OTf] was recovered almost quantitatively by simple extraction of the product with Et_2O. The recovered ionic liquid was reused for several cycles with minimal loss of catalytic activity.

In summary, a simple, efficient, and recyclable protocol for a facile alkylation of indoles and pyrroles with epoxides has been described. This occurs at ambient temperature, via ring opening of aromatic epoxides using ionic liquid [bmim][OTf] as an efficient catalyst and reaction medium. The notable features of this novel procedure are mild reaction conditions, high regio- and chemoselectivity, and simplicity in operation.

Acknowledgement

R.C. thanks the Council of Scientific and Industrial Research (CSIR), India, for research fellowship.

References and Notes

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(16) **General Procedure for the Friedel–Crafts Alkylation of N-Heterocycles Using [Bmim][OTf]**
To a stirred solution of indole (1.2 mmol, 142 mg) in ionic liquid (1.2 mmol, 0.5mL), styrene oxide (1.0 mmol, 120 mg) was added under nitrogen atmosphere and stirred for 1.5 h at r.t. After completion of the reaction, as monitored by TLC, the crude product was extracted with Et_2O (3 × 10 mL). The combined ether extracts were concentrated in vacuo and the resulting product was purified by column chromatography on silica gel (100-200 mesh) with EtOAc–*n*-hexane (1:4) as eluent to afford pure 2-(3-indolyl)-2-phenylethanol (Table 2, entry 1); yield 85%. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 1.61 (br, 1 H), 4.18–4.23 (m, 2 H), 4.50 (t, *J* = 6.7 Hz, 1 H), 7.03–7.48 (m, 10 H), 8.11 (br, 1 H). MS (EI): *m/z* = 237 (25), 206 (100), 178 (30), 128 (15), 102 (10), 77 (15), 63 (5), 51 (11). Other known products were identified by comparison with the data in the literature, see ref. 7 and 10. The ionic liquid was dried under vacuum and preserved for the next run.

(17) Spectroscopic and analytical data of new compounds.
2-(3-Indolyl)-2-(4-flourophenyl)ethanol (Table 3, Entry 2)
Yield 82%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.81 (1 H, br), 4.06–4.26 (2 H, m), 4.44 (1 H, t, *J* = 6.7 Hz), 6.90–7.37 (9 H, m), 8.05 (1 H, br). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 44.83, 66.36, 111.28, 115.23, 115.53, 115.84, 119.30, 119.63, 121.90, 122.42, 126.84, 129.69 (2), 136.53, 137.43, 163.33. MS (EI): *m/z* (rel. intensity) = 255 (13), 224 (100), 222 (14), 196 (10), 177 (7), 77 (12), 63 (13), 41 (14). IR (neat): 3578, 3419, 3069, 2885, 1623, 1556, 1501, 1462, 1412, 1351, 1250, 1106, 1070, 1017, 754 cm^{-1}. Anal. Calcd for $C_{16}H_{14}FNO$: C, 70.72; H, 5.19; N, 5.15. Found: C, 70.76; H, 5.24; N, 5.11.
2-(3-Indolyl)-2-(naphthyl)ethanol (Table 3, Entry 4)
Yield 78%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.62 (1 H, br), 4.17–4.25 (2 H, m), 4.46 (1 H, t, *J* = 6.7 Hz), 7.00–7.47 (12 H, m), 8.10 (1 H, br). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 44.96, 66.41, 111.09, 115.36, 119.53, 119.84, 121.86, 122.18, 126.68, 126.82, 126.91, 128.22, 128.53, 129.14, 129.60, 136.61, 137.03, 141.73. MS (EI): *m/z* (rel. intensity) = 287 (16), 283 (19), 218 (7), 185 (9), 171 (10), 155 (12), 144 (13). IR (neat): 3546, 3409, 3106, 2980, 1614, 1535, 1308, 1061, 1445, 1306, 1077, 1029,752 cm^{-1}. Anal. Calcd for $C_{20}H_{17}NO$: C, 83.59; H, 5.96; N, 4.87. Found: C, 83.62; H, 5.98; N, 4.80.
2-(2-Pyrrolyl)-2-(4-fluorophenyl)ethanol (Table 4, Entry 2)
Yield 80%. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.52 (1 H, br), 3.92–4.05 (2 H, m), 4.07–4.10 (1 H, m), 5.91–5.93 (1 H, m), 6.10 (1 H, dd, *J* = 6.0, 3.0 Hz), 6.70 (1 H, m), 7.18 (2 H, d, *J* = 8.8 Hz), 7.34 (2 H, d, *J* = 8.8 Hz), 8.16 (1 H, br). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 57.11, 67.78, 105.92, 108.20, 115.38, 117.41, 118.51, 129.66, 129.75, 129.83, 136.42, 161.76. MS (EI): *m/z* (rel. intensity) = 205 (15), 174 (100), 154 (5), 146 (17), 127 (14), 91 (27), 78 (18), 51 (16). IR

(neat): 3356, 3005, 1706, 1495, 1409, 1095, 1062, 1011, 831, 758 cm^{-1}. Anal. Calcd for $C_{12}H_{12}FNO$: C, 70.23; H, 5.89; N, 6.82. Found: C, 70.26; H, 5.91; N, 6.79.

2-(2-Pyrrolyl)-2-(4-chlorophenyl)ethanol (Table 4, Entry 3)

Yield 80%. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.52 (1 H, br), 3.90–4.04 (2 H, m), 4.05–4.12 (1 H, m), 5.91–5.94 (1 H, m), 6.08 (1 H, dd, *J* = 6.0, 3.0 Hz), 6.64 (1 H, m), 7.25 (2 H, d, *J* = 8.8 Hz), 7.29 (2 H, d, *J* = 8.8 Hz), 8.13 (1 H, br). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 56.28, 67.14, 107.0, 109.05, 115.16, 119.08, 130.62, 133.17, 133.24, 156.02. MS (EI): *m/z* (rel. intensity) = 221 (13), 190 (100), 154 (36), 141 (10), 127 (19), 97 (7), 73 (28), 57 (22), 43 (36). IR (neat): 3346, 2925, 1692, 1490, 1406, 1090, 1058, 1015, 826, 761 cm^{-1}. Anal. Calcd for $C_{12}H_{12}ClNO$: C, 65.02; H, 5.46; N, 6.32. Found: C, 65.06; H, 5.49; N, 6.29.

2-(2-Pyrrolyl)-2-(4-naphthyl)ethanol (Table 4, Entry 4)

Yield 78%. ^1H NMR (300 MHz, $CDCl_3$): δ = 1.52 (1 H, br), 4.15 (1 H, dd, *J* = 5.7, 10.6 Hz), 4.25 (1 H, dd, *J* = 6.6, 10.6 Hz), 4.36 (1 H, t, *J* = 7.5 Hz), 6.0 (1 H, dd, *J* = 6.0, 3.0 Hz), 6.18 (1 H, m), 6.71 (1 H, m), 7.34–7.82 (7 H, m), 8.38 (1 H, br). ^{13}C NMR (75 MHz, $CDCl_3$): δ = 47.16, 66.39, 105.95, 107.03, 117.36, 125.36, 125.82, 125.91, 126.26, 126.40, 127.57, 127.74, 128.56, 132.63, 137.84. MS (EI): *m/z* (rel. intensity) = 238 (75), 235 (28), 220 (28), 219 (15), 218 (9), 91 (27), 199 (6), 171 (12), 153 (11), 141 (19). IR (neat): 3498, 3349, 3040, 2995, 2925, 1692, 1490, 1241, 1090, 1058, 1015, 826, 711 cm^{-1}. Anal. Calcd for $C_{16}H_{15}NO$: C, 80.98; H, 6.37; N, 5.90. Found: C, 80.95; H, 6.35; N, 5.92.