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Efficient synthesis of tertiary acyclic amides by the Chapman rearrangement of aryl benzimidates in ionic liquids

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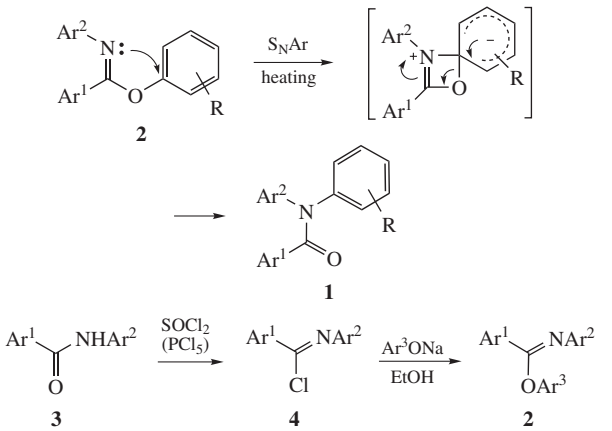
The Chapman rearrangement of aryl *N*-arylbenzimidates to tertiary acyclic amides is accelerated in ionic liquids and proceeds at lower temperatures as 120–190 °C.

Tertiary acyclic amides **1** possess biological activity[1] and are used as intermediates in organic synthesis, in particular as precursors for diarylamines[2] that are widely applied as anti-aging agents for rubbers, antioxidant additives to vulcanizates and polymerization inhibitors.[3] Effective methods for the preparation of tertiary acyclic amides **1**, based on the transition metal-catalyzed (Pd, Cu) C–N cross-coupling of haloarenes with acyclic secondary amides, have been developed over the past years.[4] The most attractive and low cost cross-coupling $Ar^1C(O)NHAr^2 + Ar^3Hal$ requires heating in organic solvents at 100–130 °C for 20–24 h in the presence of a catalytic amount of $CuBr_2$ or CuI, ligands and an excess of inorganic bases.[4(*b*)–(*d*)]

Apart from the cross-coupling, the Chapman rearrangement can also be used for the synthesis of tertiary acyclic amides **1** (Scheme 1). The Chapman rearrangement discovered back in 1915 consists of the irreversible thermal isomerization (220–300 °C) of aryl imidates **2** to compounds **1**.[5] The reaction is monomolecular, proceeds through the four-membered transition state and represents an option of the 1,3-shift. Initial reactants are usually prepared by the transformation of acyclic secondary amides **3** to imidoyl chlorides **4** under the action of $SOCl_2$ or PCl_5. Next, compounds **4** are treated with the appropriate sodium phenoxide in dry EtOH. Yields of tertiary acyclic amides **1** accessed by the Chapman rearrangement approach 70–80%, however, the reaction is hardly practicable owing to a very high temperature and, in some cases, compounds **1** cannot be prepared since initial imidate **2** would decompose before the rearrangement.[5(*b*)]



Scheme 1

Ionic liquids (ILs) are extensively utilized as reaction media or catalysts to promote different reactions, especially heterolytic ones. ILs have become inherent in the modern 'green' chemistry due to their unique physicochemical properties (non-flammability, low vapor pressure, possible recovery, *etc.*). ILs are comprised of non-coordinated ions and form an ideal environment for polar intermediates, which may bring about an unprecedented increase in the rate and selectivity of different processes.[6] Our research team has gained a successful experience in the performance of various reactions in Ils,[7] which seemed a challenge to study the Chapman rearrangement of imidates **2** in their media.

Initially, imidate **2a** ($Ar^1 = Ar^2 = Ar^3 = Ph$) was selected as a model substrate in search of optimal conditions for its rearrangement (Table 1).† According to literature, heating at 250 °C for 13 h[5(*f*)] or at 276 °C for 4 h[5(*g*)] was necessary to complete this reaction. To find the optimum conditions in IL media, tem-

† IR spectra were measured on a UR-20 spectrometer. ^1H and ^{13}C NMR spectra were recorded on a Bruker AM300 (300 MHz for ^1H and 75.5 MHz for ^{13}C) spectrometer ($CDCl_3$ was used as an internal standard) at 30 °C. Mass spectra were measured on a Finnigan MAT INCOS-50 instrument. TLC was conducted on silica gel plates (Silufol UV-254). Melting points were measured on a Sanyo Gallenkamp instrument. Benzanilide **3a** is a commercial product. Arylbenzamides **3b–e** were prepared by known procedures.[8–11]

Aryl N-*arylbenzimidates* **2a–g** *(general procedure)*. Thionyl chloride (5.5 ml, 75 mmol) was added to 15 mmol of *N*-arylbenzamide **3a–e** and the mixture was refluxed for ~5 h until consumption of the initial compounds **3a–e** (TLC monitoring). Then an excess of $SOCl_2$ was evaporated *in vacuo* and imidoyl chlorides **4a–e** formed were used without additional purification. Sodium metal (0.35 g, 15.2 g-atom) in small pieces was dissolved in 20 ml of absolute ethanol and the corresponding phenol (22.1 mmol) was added. After 30 min, the corresponding imidoyl chloride (15.2 mmol) dissolved in Et_2O (10 ml) or DMF was added to this solution and the reaction mixture was left overnight at room temperature. Next day the mixture was poured into water (75 ml), the resulted solid was filtered off, washed with water and minimal Et_2O and dried in air.

Phenyl N-*phenylbenzimidate* **2a**: mp 103–104 °C (MeOH) (lit.,[5(*g*)] 104–105 °C). ^{13}C NMR, δ: 123.4, 124.7, 126.0, 126.8, 128.5, 129.7, 130.4, 130.9, 133.8, 135.7, 145.6, 148.9. IR (KBr, ν/cm^{-1}): 1717, 1657, 1589, 1486, 1267, 1210 (PhO), 1165. MS, *m*/*z* (%): 273 [M$^+$] (14), 180 [M$^+$ – PhOH + 1] (100), 77 Ph (53).

4-Nitrophenyl N-*phenylbenzimidate* **2b**: mp 76–77 °C (EtOH) (lit.,[5(*b*)] 76–77 °C). ^1H NMR, δ: 7.0–8.10 (m, 14H). ^{13}C NMR, δ: 126.0, 126.8, 127.9, 129.1, 129.6, 130.3, 130.6, 131.5, 136.5, 142.2, 145.4. IR (KBr, ν/cm^{-1}): 1655, 1586, 1511, 1353, 1226 (ArO).

Table 1 Screening of the conditions of the rearrangement of imidate **2a** into *N*,*N*-diphenylbenzamide **1a**.

Entry	IL	*T*/°C	*t*/h	Isolated yield (%)
1	[bmim]BF_4	190	2.0	88
2	[bmim]PF_6	190	1.5	90
3	[bdmim]BF_4	190	2.0	78
4	[emim]CF_3SO_3	180	1.7	30[a]
5	[bmpyrr]CF_3SO_3	180	2.0	38[a]
6	[emim]HSO_4	180	2.0	0[b]
7	[bmim]BF_4	150	14.0	85
8	[bmim]BF_4	120	24.0	90
9	[bmim]PF_6	150	12.0	87
10	[bmim]PF_6	120	22.0	85
11[c]	[bmim]BF_4	190	2.0	90
12[c]	[bmim]BF_4	190	2.0	90

[a] And the products of decomposition. [b] Only products of decomposition. [c] Recovered IL.

perature and reaction time were varied. A temperature of 190 °C [60–86 °C lower than in refs. 5(*f*),(*g*)] was selected to begin with. The reaction termination was TLC controlled. Among the tested ILs (entries 1–6, ILs are substrate-specific media), [bmim]BF_4 and [bmim]PF_6 appeared the best for the processing at this temperature – the reaction was complete within 1.5–2.0 h (entries 1, 2). Ionic liquid [bdmim]BF_4 is also suitable, however the yield of the final product was lower (entry 3). The other ILs proved to be inefficient. In ILs with $CF_3SO_3^-$ anion (entries 4, 5) reactant **2a** partially gives the target product and the remaining part is decomposed. The total decomposition of imidate **2a** is observed in acidic IL [emim]HSO_4 (entry 6). Lowering temperatures to 150 and even to 120 °C did not stop the reaction, however, longer time, 12–14 and 22–24 h, respectively, was required (entries 7–10). This is the first instance of the Chapman rearrangement at 120 °C. After the final product isolation, IL was recovered and recycled in the same reactions at least twice without a decrease in the yield of **1a** (entries 11, 12).

The optimal conditions were applied to the Chapman rearrangement of other imidates **2b**–**g** (Table 2). Imidates **2**, except for **2d**, are known compounds, however, the methods for their preparation had some distinctions and the most part of them were insufficiently characterized. We have synthesized these compounds according to the general procedure and added new physicochemical characteristics.‡

First of all, imidate **2b** that was reported[5(*b*)] to fail to undergo the rearrangement and decompose at 150 °C, was successfully transformed into product **1b** in ILs at the same temperature 150 °C in 60–63% yield (Table 2, entries 1, 3), while only a small fraction of reactant **2b** decomposed. Moreover, at a temperature drop to 120 °C compound **1b** was obtained in 85% yield, however, the reaction duration increased (entries 2, 4). Evidently, in the ongoing rearrangement, ILs stabilize the generation of dipolar intermediates, which promotes their subsequent transformation to final products **1**. Since the rearrangement at 120–150 °C proceeds over a long time, other imidates **2c**–**f** were subjected to the rearrangement at 180–190 °C, *i.e.*, under considerably milder conditions than those known from literature (see Table 2). The yields of all final tertiary acyclic amides **1** exceeded 80%.

Structures of the synthesized compounds were established by a comparison of their melting points and IR, ^{1}H, ^{13}C NMR and mass spectra with published data. Since the elemental compositions of the initial imidates **2** do not change in the rearrangement process the most important information about the structure of compounds **1** was obtained from IR, ^{13}C NMR (appearance of CO group) and mass spectra. In particular, the molecular ion of the final products **1** did not differ from that of initial compounds **2**, whereas the fragment ions in mass spectra were different, namely, in compounds **2** fragment ions ArO^+ were present, while in compounds **1** fragment ions $ArCO^+$ appeared.

In conclusion, we have developed a new metal-free approach for the synthesis of the tertiary acyclic amides by the Chapman rearrangement of imidates in ILs [bmim]BF_4(PF_6). The use of ILs as reaction media promoted acceleration of the rearrangement,

4-Methylphenyl N-*phenylbenzimidate* **2c**: mp 58–59 °C (EtOH) (lit.,[5(*e*)] 58.8–59.8 °C). ^{1}H NMR, δ: 2.33 (s, 3H, Me), 6.90–7.85 (m, 14H, Ar). IR (KBr, ν/cm^{-1}): 1659, 1594, 1506, 1263, 1236 (ArO), 1199, 1163. MS, *m*/*z*, (%): 288 [M^+ + 1] (3), 180 [M^+ – MeC_6H_4OH + 1] (95), 105 [PhCO] (12), 77 [Ph] (100).

Phenyl N-*(4-nitrophenyl)benzimidate* **2d**: mp 80–81 °C (EtOH). ^{1}H NMR, δ: 6.95–7.30 (m, 10H, Ar), 7.55–7.62 (m, 2H, Ar), 8.02–8.15 (m, 2H, Ar). ^{13}C NMR, δ: 125.5, 126.3, 127.0, 127.8, 128.8, 129.8, 130.5, 131.0, 132.2, 136.3, 142.6, 145.6. IR (KBr, ν/cm^{-1}): 1663, 1600, 1512, 1268, 1244, 1206 (PhO), 1169. MS, *m*/*z* (%): 318 [M^+] (8), 272 [M^+ – NO_2] (2), 225 [M^+ – PhOH + 1] (100), 195 [M^+ – $PhNO_2$] (17), 101 [M^+ – PhOH – – $PhNO_2$] (29).

Phenyl N-*(4-methoxyphenyl)benzimidate* **2e**: mp 66–67 °C (MeOH) (lit.,[1] 66–67 °C). Other spectral data (^{1}H NMR, ^{13}C NMR, IR, MS) are consistent with reported ones.[12]

Phenyl 4-methoxy-N-*phenylbenzimidate* **2f**: mp 84–87 °C (EtOH) (lit.,[13] 84–86 °C). ^{1}H NMR, δ: 3.67 (s, 3H, Me), 7.10–7.85 (m, 14H, Ar). IR (KBr, ν/cm^{-1}): 1657, 1596, 1510, 1260, 1233, 1207 (PhO), 1165. MS, *m*/*z* (%): 287 [M^+] (6), 194 [M^+ – PhOH + 1] (100), 91 [MeC_6H_4] (41).

Phenyl 4-nitro-N-*phenylbenzimidate* **2g**: mp 101–102 °C (EtOH) (lit.,[5(*b*)] 101–102.5 °C). ^{1}H NMR, δ: 6.92–8.15 (m, 14H, Ar). ^{13}C NMR, δ: 126.3, 126.9, 127.9, 129.3, 129.8, 130.1, 130.9, 131.3, 136.5, 143.4, 145.8. IR (KBr, ν/cm^{-1}): 1660, 1588, 1512, 1351, 1223 (PhO). MS, *m*/*z* (%): 318 [M^+] (65), 272 [M^+ – NO_2] (16), 224 [M^+ – PhOH] (100), 122 [$PhNO_2$] (38).

N,N-*Diarylbenzamides* **1a**–**g** *(general procedure)*. The solution or suspension of the imidate **2** (2 mmol) in 2 g of corresponding IL was heated at corresponding temperature (see Table 2) and stirred until disappearance of the reactant (TLC monitoring, eluent CH_2Cl_2). Then the reaction mixture was cooled to room temperature, CH_2Cl_2 (1.5 ml) was added and the obtained *N*,*N*-diarylbenzamide **1** was extracted with Et_2O (5×10 ml). The ethereal extract was stirred with 1 g of absorbent carbon, filtered, Et_2O was evaporated from the filtrate *in vacuo* and the residue was crystallized from corresponding solvent. The IL was dried *in vacuo* from the solvents remainders and repeatedly used several time in the next syntheses of the same product without lowering the yield.

N,N-*Diphenylbenzamide* **1a**: mp 177–178 °C (EtOH) (lit.,[4(*b*)] 179–180 °C). ^{13}C NMR, δ: 120.4, 121.8, 124.6, 127.1, 128.9, 131.9, 135.1, 138.1 (Ar), 165.9 (C=O). IR (KBr, ν/cm^{-1}): 1656 (C=O), 1600, 1536, 1493, 1439, 1323, 1261, 751, 691. MS, *m*/*z* (%): 273 [M^+] (20), 180 (5), 168 [M^+ – PhCO] (21), 105 [PhCO] (100), 77 [Ph] (40).

N-*(4-Nitrophenyl)*-N-*phenylbenzamide* **1b** (prepared from imidates **2b** and **2d**): mp 123–124 °C (EtOH) (lit.,[4(*b*)] 124–125 °C). IR (KBr, ν/cm^{-1}): 1656 (C=O), 1600, 1535, 1439, 1322, 1261, 750, 691. MS, *m*/*z* (%): 318 [M^+] (54), 272 [M^+ – NO_2] (8), 197 [M^+ – $PhCONH_2$] (35), 180 [M^+ – – $NO_2C_6H_4NH_2$] (52), 139 [$NO_2C_6H_4NH_2$ + 1] (26), 105 [PhCO] (100), 77 [Ph] (76).

N-*(4-Methylphenyl)*-N-*phenylbenzamide* **1c**: mp 91–92 °C (EtOH) (lit.,[4(*b*),5(*g*)] 89–90 °C). IR (KBr, ν/cm^{-1}): 1656 (C=O), 1599, 1513, 1439, 1265, 750, 691. MS, *m*/*z* (%): 212 [MeC_6H_4NH – COPh + 1] (66), 197 [PhNH – COPh] (28), 105 [PhCO] (62), 77 [Ph] (100).

N-*(4-Methoxyphenyl)*-N-*phenylbenzamide* **1e**: mp 119 °C (EtOH) (lit.,[4(*b*)] 119–120 °C). IR (KBr, ν/cm^{-1}): 1653 (C=O), 1593, 1521, 1436, 1267, 742, 693. MS, *m*/*z* (%): 303 [M^+] (22), 198 [M^+ – PhCO] (35), 105 [PhCO] (100), 77 [Ph] (42).

4-Methoxy-N,N-*diphenylbenzamide* **1f**: mp 139–140 °C (EtOH) (lit.,[4(*b*)] 140–141 °C). IR (KBr, ν/cm^{-1}): 1658 (C=O), 1600, 1515, 1436, 1265, 753, 690. MS, *m*/*z* (%): 303 [M^+] (14), 184 [M^+ – 4-$MeOC_6H_4CO$] (27), 135 [4-$MeOC_6H_4CO$] (100), 107 (37).

4-Nitro-N,N-*diphenylbenzamide* **1g**: mp 155–156 °C (EtOH) (lit.,[14] 156–157 °C). IR (KBr, ν/cm^{-1}): 1660 (C=O), 1591, 1532, 1435, 1322, 1264, 752. MS, *m*/*z* (%): 318 [M^+] (81), 272 [M^+ – NO_2] (19), 224 [M^+ – – PhOH] (47), 150 [4-$NO_2C_6H_4CO$] (100), 122 [$PhNO_2$] (38).

Table 2 Synthesis of tertiary acyclic amides **1** by the Chapman rearrangement of imidates **2** in ionic liquids.

Entry	Imidate	Ar^1	Ar^2	Ar^3	IL	*T*/°C	*t*/h	Product **1**	Isolated yield (%)	Literature data
1	**2b**	Ph	Ph	4-$O_2NC_6H_4$	[bmim]BF_4	150	3	**1b**	60	Decomposition at 150 °C[5(b)]
2	**2b**	Ph	Ph	4-$O_2NC_6H_4$	[bmim]BF_4	120	18	**1b**	85	
3	**2b**	Ph	Ph	4-$O_2NC_6H_4$	[bmim]PF_6	150	2	**1b**	63	
4	**2b**	Ph	Ph	4-$O_2NC_6H_4$	[bmim]PF_6	120	18	**1b**	85	
5	**2c**	Ph	Ph	4-MeC_6H_4	[bmim]BF_4	180	10	**1c**	86	4 h at 276 °C[5(g)]
6	**2c**	Ph	Ph	4-MeC_6H_4	[bmim]PF_6	180	12	**1c**	81	
7	**2d**	Ph	4-$O_2NC_6H_4$	Ph	[bmim]BF_4	190	7	**1b**	82	**2d** was not previously subjected to the Chapman rearrangement
8	**2d**	Ph	4-$O_2NC_6H_4$	Ph	[bmim]PF_6	190	7	**1b**	83	
9	**2e**	Ph	4-$MeOC_6H_4$	Ph	[bmim]BF_4	190	5	**1e**	83	Conversion is 80% for 1.5 h at 270 °C[5(b)]
10	**2e**	Ph	4-$MeOC_6H_4$	Ph	[bmim]PF_6	190	7	**1e**	86	
11	**2f**	4-$MeOC_6H_4$	Ph	Ph	[bmim]BF_4	190	2	**1f**	87	Conversion is 84% for 1.5 h at 270 °C[5(b)]
12	**2f**	4-$MeOC_6H_4$	Ph	Ph	[bmim]PF_6	190	2	**1f**	85	
13	**2g**	4-$O_2NC_6H_4$	Ph	Ph	[bmim]BF_4	190	2.2	**1g**	88	Conversion is 49% for 1.5 h at 270 °C[5(b)]
14	**2g**	4-$O_2NC_6H_4$	Ph	Ph	[bmim]PF_6	190	2.4	**1g**	85	

that allowed us to reduce the reaction temperature in some cases by 90–120 °C and to process such imidates which under conventional conditions would decompose prior to the rearrangement. The found approach makes it possible to extend a list of known methods for the synthesis of tertiary acyclic amides.

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