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The First Aminimide Synthesis from Benzoyl Azide and Pyridine

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Irradiation of benzoyl azide **1** at 254 nm in the presence of some amines produces aminimides: in the presence of pyridine the aminimide **13** can be isolated in 41% yield; in the presence of *N*,*N*-dimethylaniline a CH insertion product **7** is obtained via an intermediate aminimide **12**. This is the first reported synthesis of aminimides from a benzoyl azide.

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Introduction

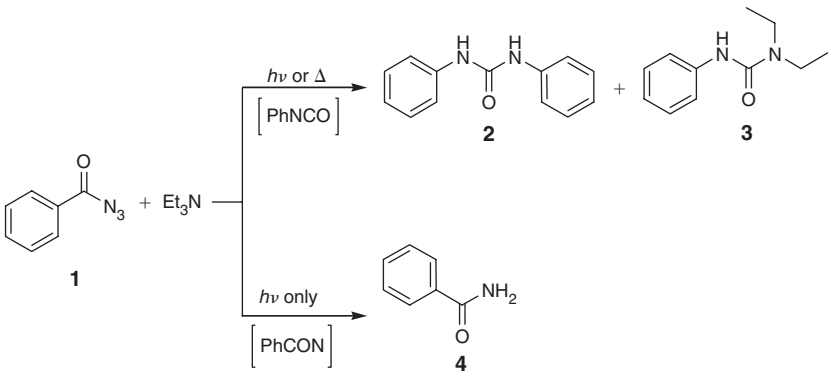
Aminimides are a functional group possessing pharmaceutically desirable physicochemical properties as a result of their zwitterionic character. Recently, there have been reports describing the use of the aminimide functionality in antimycotics[1] as peptidomimetic inhibitors of elastase[2] and HIV-1 protease,[3] as initiators in the polymerization of epoxides,[4] drag-reducing surfactants,[5] for the extraction of heavy and transition metal cations,[6–9] and as intermediates in the total synthesis of alkaloids and indazoles.[10,11]

The chemistry of aminimides has been the subject of various review articles, and these compounds can be synthesized by several methods.[12–14] Most commonly, aminimides are prepared by alkylation of hydrazine derivatives[15–19] and acylation of hydrazinium salts.[20] There are reports of aminimide synthesis from azides and amines; however, these relate to sulfonyl azides[21,22] and azidoformates.[23–28] The synthesis of aminimides from azides and amines has been rationalized as proceeding via singlet nitrenes.[21,22] Benzoyl azides have been reported to give isocyanates upon thermolysis.[29,30] To date, there has been no report in the literature of the synthesis of an aminimide from a benzoyl azide. Here, we report the first aminimide synthesis from benzoyl azide and an amine.

In efforts to develop a synthesis of aminimides from tertiary amines, we have investigated the photochemical and thermolytic reactions of benzoyl azide with a variety of tertiary amines. Most of the past efforts have concentrated on the thermolysis of acyl azides, which leads to products derived from the Curtius rearrangement. However, singlet benzoyl nitrene is thought to be the primary species generated by photolysis of benzoyl azide.[31] Thus, the photolytic decomposition of benzoyl azide in the presence of a tertiary amine might serve as a possible synthesis for aminimides from tertiary amines. To test this proposal, the decomposition of benzoyl azide was carried out under both photolytic and thermolytic conditions in the presence of triethylamine, *N*,*N*-dimethylaniline, and pyridine.

Results and Discussion

The photolysis and thermolysis of benzoyl azide **1** in the presence of triethylamine led to the isolation of several products (Scheme 1). Three products were isolated from the photolytic reaction mixture and were identified as *N*,*N*′-diphenylurea



Scheme 1.

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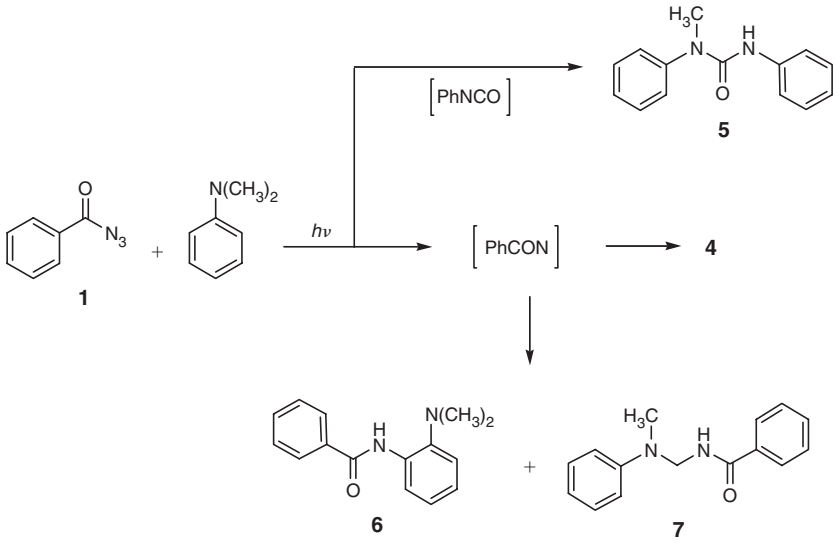


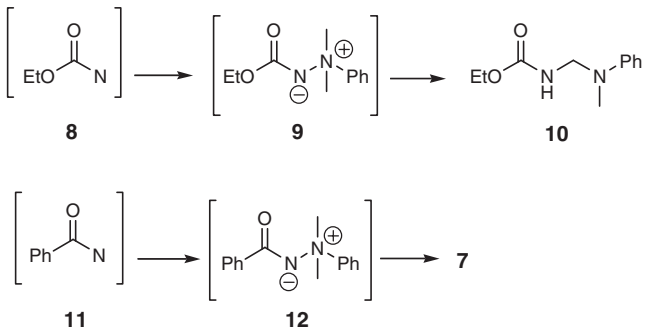
Scheme 2.

2 (7%), *N*′,*N*′-diethyl-*N*-phenylurea **3** (27%), and benzamide **4** (32%). The thermolytic reaction gave rise only to **2** (90%) and **3** (5%). As has been previously reported,[23,30,32–34] thermolysis and photolysis of benzoyl azide gave rise to products via the Curtius rearrangement; in this instance the ureas **2** and **3** were formed. The urea **2** has also been reported as a photolytic degradation product of trialkylammonio-*N*-benzoylimides;[35–37] however it is unclear whether these ureas are formed directly or as a rearrangement product of the aminimide. Benzamide **4** was isolated only from the photolytic reaction, a product of hydrogen abstraction by benzoyl nitrene in the triplet state.

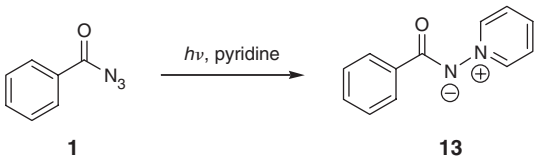
The photolytic reaction of benzoyl azide and *N*,*N*-dimethylaniline gave a product resulting from the Curtius rearrangement **5** (14%), the triplet nitrene product **4** (40%), and two CH insertion products, **7** (17%) and **6** (9%) (Scheme 2). Thermolysis of **1** in the presence of *N*,*N*-dimethylaniline gave only *N*,*N*′-diphenylurea **2** (82%).

Hafner[23] has previously reported a product **10** of CH insertion into the methyl group of *N*,*N*′-dimethylaniline involving ethoxycarbonyl nitrene **8** (Scheme 3). The insertion product was proposed to arise from the rearrangement of a short-lived aminimide intermediate **9**. The CH insertion product **7** could be explained by a similar intermediate **12** derived from the reaction of *N*,*N*′-dimethylaniline with singlet benzoyl nitrene **11**.

Thermolysis of benzoyl azide **1** in the presence of pyridine gave rise only to the Curtius product **2** (94%). In contrast, photolysis of **1** in the presence of pyridine gave the pyridine-1-benzenecarbonylimide **13** (41%) together with recovered **1** (25%) (Scheme 4). The aminimide **13** was identified by comparison with a literature melting point and the characteristic aminimide infrared absorption (1590 cm^{-1}). Although this product is known, this is the first reported synthesis of an aminimide from a benzoyl azide and an amine.



Scheme 3.



Scheme 4.

Conclusions

These studies suggest that the decomposition of benzoyl azide follows the nitrene path only under photolytic degradation conditions, while thermolysis gives rise only to products of the Curtius rearrangement. Thus, irradiation of benzoyl azide in the presence of pyridine affords an aminimide upon singlet nitrene generation. Products that can be explained by an aminimide intermediate arise upon irradiation of benzoyl azide in the presence of *N*,*N*-dimethylaniline. Products of the photo-Curtius rearrangement and amides, resulting from hydrogen abstraction by triplet benzoyl nitrene, were also isolated from the photolytic degradation of benzoyl azide in the presence of tertiary amines. Thermolysis of benzoyl azide in the presence of tertiary amines gave only products derived from the Curtius rearrangement.

Experimental

General Procedures

Melting points were determined on a Reichert Micro-melting point apparatus and are uncorrected. Only principal, sharply defined IR peaks are reported. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-300 spectrometer at 300.13 and 75.5 MHz, respectively. Electrospray ionization (ESI) mass spectra were recorded in the positive-ion mode using a Micromass platform II mass spectrometer at the indicated cone voltage.

Fast atom bombardment (FAB) mass spectra were obtained using a JEOL JMS-DX300 mass spectrometer using the matrix indicated (TG/G, thioglycerol/glycerol; 3NBA, 3-nitrobenzyl alcohol). Thin-layer chromatography (TLC) was performed on silica gel 60 F_{254} pre-coated plates (Merck), and spots were visualized by irradiation with ultraviolet light (254 nm) or by iodine vapour. Flash chromatography was performed using Merck Kieselgel 60 (230–400) mesh.

Benzoyl Azide **1**

To an ice-cooled solution of sodium azide (0.56 g, 8.5 mmol) in water (10 mL) was added dropwise a solution of benzoyl chloride (830 μL, 1.0 g, 7.1 mmol) in anhydrous acetone (30 mL). The mixture was vigorously stirred in ice for 2.5 h. The acetone was removed under vacuum, the mixture extracted with dichloromethane (2 × 50 mL), dried over sodium sulfate, and concentrated. The resulting colourless oil solidified on standing and was recrystallized from dichloromethane–hexane to afford benzoyl azide **1** as colourless needles (0.55 g, 52%). mp 29–31°C (lit.[38] 32°C). ν_{max} (KBr)/cm^{-1} 2132, 2100, 1692, 1600. δ_H ($CDCl_3$) 7.45 (2H, br app. t, *J* 7.5, H3, H5), 7.61 (1H, br t, *J* 7.5, H4), 8.03 (2H, dd, *J* 7.5, 1, H2, H6). δ_C ($CDCl_3$) 172.4 (CO), 134.3 (C4), 130.7 (C1), 129.4 (C3, C5), 128.6 (C2, C6).

Photochemistry: General Procedure

A solution of **1** in the anhydrous amine was degassed under vacuum, fitted with a cold finger of cooling water (10–15°C), and then irradiated at 254 nm using a Photochemical Reactors 6 W low-pressure mercury lamp. The excess amine was removed and the reaction mixture analyzed.

Photolytic Reaction of Benzoyl Azide **1** and Triethylamine

A solution of **1** (162 mg, 1.1 mmol) in anhydrous triethylamine (50 mL) was irradiated for 3 h. The solvent was removed under vacuum to afford a yellow oil which, on standing, was collected as a white precipitate. Recrystallization from ethyl acetate–hexane gave *N*,*N*′-diphenylurea **2** as colourless needles (15 mg, 7%). mp 239–242°C (lit.[39] 239–241°C). ν_{max} (KBr)/cm^{-1} 3348, 3304, 1648. δ_H (d_6-DMSO) 6.96 (2H, br t, *J* 7.5, H4, H4′), 7.27 (4H, br app. t, *J* 7.5, H3, H3′, H5, H5′), 7.46 (4H, dd, *J* 7.5, 1, H2, H2′, H6, H6′), 8.63 (2H, s, 2 × NH). δ_C ($CDCl_3$) 152.5 (CO), 139.6 (C1), 128.7 (C3, C3′, C5, C5′), 121.7 (C4, C4′), 118.2 (C2, C2′, C6, C6′). *m/z* (FAB, 3NBA) 213 (100%, $[M + H]^+$).

The filtrate was subjected to purification by flash chromatography (9:1 dichloromethane/ethyl acetate) resulting in the isolation of a further two products. The least polar fraction (R_F 0.37) crystallized from dichloromethane–hexane to give *N*′,*N*′-diethyl-*N*-phenylurea **3** as colourless rosettes (58 mg, 27%). mp 81–83°C (lit.[40] 84–85°C). ν_{max} (KBr)/cm^{-1} 3320, 1638. δ_H ($CDCl_3$) 1.22 (6H, t, *J* 7, 2 × CH_2CH_3), 3.37 (4H, q, *J* 7, 2 × CH_2CH_3), 6.42 (1H, br s, NH), 7.01 (1H, br t, *J* 7.5, H4), 7.27 (2H, br app. t, *J* 7.5, H3, H5), 7.40 (2H, br d, *J* 7.5, H2, H6). δ_C ($CDCl_3$) 154.7 (CO), 139.4 (C1), 128.8 (C3, C5), 122.8 (C4), 119.9 (C2, C6), 41.6 (2 × CH_2CH_3), 13.9 (2 × CH_2CH_3). *m/z* (FAB, TG/G) 193 (100%, $[M + H]^+$).

The more polar fraction (R_F 0.1) gave benzamide **4** which was crystallized from dichloromethane as colourless needles (43 mg, 32%) mp 128–129°C (lit.[41] 130°C). ν_{max} (KBr)/cm^{-1} 3388, 1656. δ_H ($CDCl_3$) 6.30 (2H, br s, NH_2), 7.44 (2H, br app. t, *J* 7, H3, H5), 7.53 (1H, br t, *J* 7, H4), 7.83 (2H, br d, *J* 7.5, H2, H6). δ_C ($CDCl_3$) 165.3 (CO), 133.6 (C1), 132.0 (C4), 128.8 (C3, C5), 127.6 (C2, C6). *m/z* (FAB, TG/G) 122 (100%, $[M + H]^+$).

Photolytic Reaction of Benzoyl Azide **1** and N,N-Dimethylaniline

A solution of **1** (141 mg, 0.96 mmol) in anhydrous *N*,*N*-dimethylaniline (50 mL) was irradiated (254 nm) for 3 h. The excess amine was removed under vacuum and the resulting dark brown oil purified by flash chromatography (10:1 hexane/ethyl acetate) resulting in the isolation of four products.

The least polar fraction (R_F 0.34) gave a tan solid, which recrystallized from hexane to give *N*-1-[2-(dimethylamino)-phenyl]benzamide **6** as colourless needles (20 mg, 9%). mp 51–52°C (lit.[42] 53°C). ν_{max} (KBr)/cm^{-1} 3360, 1672. δ_H ($CDCl_3$) 2.71 (6H, s, 2 × CH_3N), 7.09 (1H, app. td, *J* 7.5, 1.5, H5′), 7.18 (1H, app. td, *J* 7.5, 1.5, H4′), 7.23 (1H, dd, *J* 8, 1.5, H6′), 7.49–7.56 (3H, m, H3, H4, H5), 7.92 (2H, dd, *J* 8, 2, H2, H6), 8.54 (1H, dd, *J* 8, 1.5, H3′), 9.34 (1H, br s, NH). δ_C ($CDCl_3$) 165.3 (CO), 143.5 (C1′), 135.8 (C2′), 134.0 (C1), 132.0 (C4), 129.1 (C3, C5), 127.4 (C2, C6), 125.6 (C4′), 124.2 (C5′), 120.3 (C6′), 120.0 (C3′), 45.3 (2 × NCH_3). *m/z* (ESI, positive ion, 30 V) 241 (100%, $[M + H]^+$).

The next fraction (R_F 0.23) gave *N*-methyl-*N*,*N*′-diphenylurea **5** which recrystallized from dichloromethane–hexane as colourless needles (31 mg, 14%). mp 102–105°C (lit.[43] 106°C). ν_{max} (KBr)/cm^{-1} 3296, 1700. δ_H ($CDCl_3$) 3.32 (3H, s, CH_3N), 6.35 (1H, br s, NH), 6.97 (1H, br t, *J* 7.5, H4′), 7.21 (2H, br app. t, *J* 7.5, H3′, H5′), 7.28–7.32 (4H, m, H2, H2′, H6, H6′), 7.35 (1H, t, *J* 7.5, H4), 7.46 (2H, app. t, *J* 7.5, H3, H5). δ_C ($CDCl_3$) 154.0 (CO), 142.7 (C1), 138.6 (C1′), 129.9 (C3′, C5′), 128.4 (C3, C5), 127.4 (C4), 127.0 (C2, C6), 122.5 (C4′), 119.0 (C2′, C6′), 36.9 (CH_3N). *m/z* (ESI, positive ion, 30 V) 227 (100%, $[M + H]^+$).

Fraction three (R_F 0.18) gave a colourless solid which recrystallized from hexane to afford *N*-[(methylanilino)methyl]benzamide **7** as colourless needles (38 mg, 17%). mp 121–123°C (lit.[44] 120–125°C). δ_H (d_6-DMSO) 3.29 (3H, s, NCH_3), 4.89 (2H, d, *J* 5, $HNCH_2$), 6.66 (1H, t, *J* 7.5, H4′), 6.89 (2H, d, *J* 8, H2′, H6′), 7.17 (2H, app. t, *J* 7.5, H3′, H5′), 7.44 (2H, app. t, *J* 7.5, H3, H5), 7.52 (1H, t, *J* 7, H4), 7.84 (2H, d, *J* 8, H2, H6), 8.92 (1H, br app. t, *J* 5, NH). δ_C ($CDCl_3$) 168.2 (CO), 148.1 (C1′), 134.3 (C1), 131.8 (C4), 129.6 (C3′, C5′), 128.7 (C3, C5), 127.2 (C2, C6), 118.8 (C4′), 113.8 (C2′, C6′), 58.3 (CH_2), 38.1 (CH_3N). *m/z* (ESI, positive ion, 30 V) 263 (25%, $[M + Na]^+$), 240 (90, $[M + H]^+$), 120 (100).

The most polar fraction (R_F 0.1) recrystallized from dichloromethane as colourless needles (46 mg, 40%), and was identified as benzamide **4**.

Photolytic Reaction of Benzoyl Azide **1** and Pyridine

A solution of **1** (141 mg, 0.96 mmol) in anhydrous pyridine (50 mL) was irradiated (254 nm) for 18.5 h. The excess pyridine was removed under vacuum and the resulting brown oil purified by flash chromatography (4:1 chloroform/diethyl ether) to afford unchanged starting material **1** (37 mg, 25%) and pyridine-1-benzenecarbonylimide **13**. The crude product recrystallized from dichloromethane–hexane as orange prisms (78 mg, 41%). mp 175–177°C (lit.[45] 177.5°C). ν_{max} (KBr)/cm^{-1} 1590. δ_H (d_6-DMSO) 7.36–7.43 (3H, m, H3′, H4′, H5′), 7.90 (2H, app. t, *J* 7, H3, H5), 8.05 (2H, dd, *J* 7.5, 1.5, H2′, H6′), 8.17 (1H, t, *J* 7.5, H4), 8.85 (2H, br d, *J* 6, H2, H6). δ_C (d_6-DMSO) 175.2 (CO), 143.7 (C4), 139.0 (C1′), 137.1 (C2, C6), 130.6 (C4′), 128.3 (C3, C5), 128.2 (C3′, C5′), 126.3 (C2′, C6′). *m/z* (FAB, 3NBA) 199 (100%, $[M + H]^+$).

Thermolysis: General Procedure

A solution of **1** in the anhydrous amine was degassed under vacuum and heated at reflux for a period of time. The excess amine was removed and the reaction mixture analyzed.

*Thermolysis of Benzoyl Azide **1** in the Presence of Triethylamine*

A solution of **1** (164 mg, 1.12 mmol) in anhydrous triethylamine (10 mL) was heated at reflux for 1 h under an atmosphere of nitrogen. A white precipitate was collected by filtration and recrystallized from ethyl acetate–hexane to afford a white solid (75 mg, 70%), identified as **2**. The filtrate was concentrated and purified by flash chromatography (10:1 hexane/dichloromethane) to afford additional **2** (30 mg, 20%) and **3** as a white solid (10 mg, 5%).

*Thermolysis of Benzoyl Azide **1** in the Presence of N,N-Dimethylaniline*

A solution of **1** (154 mg, 1.05 mmol) in *N*,*N*-dimethylaniline (10 mL) was heated at reflux for 16 h under an atmosphere of nitrogen. The reaction mixture was concentrated and purified by flash chromatography (10:1 hexane/dichloromethane) to afford a tan solid (91 mg, 82%), which was identified as **2**.

*Thermolysis of Benzoyl Azide **1** in the Presence of Pyridine*

A solution of **1** (161 mg, 1.10 mmol) in anhydrous pyridine (10 mL) was refluxed under an atmosphere of nitrogen for 15 h. The reaction mixture was cooled, concentrated, and resuspended in ethyl acetate. A white solid precipitated on the addition of hexane to afford **1** as the major product (85 mg, 73%). The filtrate was purified by flash chromatography (9:1 dichloromethane/ethyl acetate) to afford additional **2** (25 mg, 21%).

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