

Ring Opening of 1-Azabicyclo[1.1.0]butanes with Hydrazoic Acid – a Facile Access to *N*-Unsubstituted Azetidin-3-Amines

by Grzegorz Mlostoń* and Małgorzata Celeda

University of Łódź, Department of Organic and Applied Chemistry, Narutowicza 68, PL-90-136 Łódź
(phone: + 48 42 635-57-61; fax: + 48 42 678-16-09; e-mail: gmloston@uni.lodz.pl)

Dedicated to Professor *Rolf Huisgen* on the occasion of his 85th birthday

Sterically congested 1-azabicyclo[1.1.0]butanes **1** add hydrazoic acid smoothly at 0–5°, giving 3-azidoazetidines **2** in good to excellent yields. After hydrogenolysis over Pd/C catalyst, compounds **2** were converted into *N*-unsubstituted azetidin-3-amines **4**. Attempted reduction of **2a** with *Raney*-Ni led to a mixture of the expected azetidin-3-amine **4a** and the ring-enlarged 2,5-dihydro-1*H*-imidazole derivative **5**.

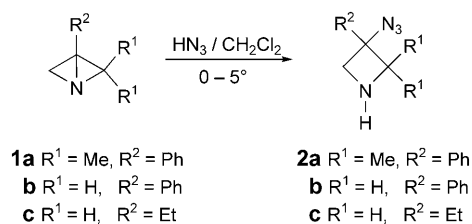
1. Introduction. – Azetidines are four-membered nitrogen heterocycles that are of great interest for fundamental research and practical applications [1]. These heterocycles show remarkable biological activity, and were found as a central structural moiety in some naturally occurring compounds [2]. Recently, a growing number of reports describing new applications of azetidine derivatives as antiviral and/or anticancer pharmaceuticals [3], plant-growth regulators [4], as well as energetic materials [5] were observed.

Azetidin-3-amines are less well-known for their practical applications, as there is no general method known for the preparation of these versatile building blocks [6]. However, one report deserves special attention. A few years ago, a French group described a multi-step procedure based on a modified *Strecker* reaction with Me₃SiCN and dibenzylamine, leading to 3-phenylazetidin-3-amine, which is required for the preparation of some antibacterial quinolones [7]. The isolated product could not be obtained in pure form, and the authors claimed to have prepared the hydrochloride in only *ca.* 95% purity.

In the last two decades, diverse conversions of strained 1-azabicyclo[1.1.0]butanes of the type **1** with electrophilic reagents such as HF [8], methyl azido- or chloroformate [9], thioacetic acid [10], as well as dichlorocarbene [11] have been described. In the present paper, we present a straightforward method for the smooth transformation of relatively easily available 1-azabicyclo[1.1.0]butanes **1a–d** into azetidin-3-amines **4** via the intermediate 3-azidoazetidines **2** (see *Scheme 1* below).

2. Results and Discussion. – Hydrazoic acid (HN₃) is well-known as a useful reagent that has been widely applied for the synthesis of amino compounds. Its reactions with small-ring heterocycles, however, were only scarcely studied, and the literature reports are limited to the ring-opening of some oxirane derivatives [12]. On the other hand, the ring-opening reactions of strained 1-azabicyclo[1.1.0]butanes with acidic agents, aimed

Scheme 1



at the preparation of azetidine derivatives, can be smoothly performed [13]. In known cases, strong acids of the type HX (X = F, Cl) or sulfuric acid were applied to complete the conversion [8][14][15].

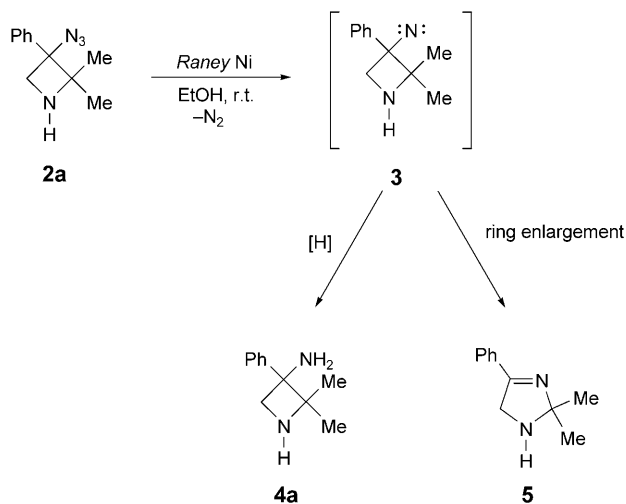
In a typical experiment, the addition of a diluted solution of **1** to a cooled solution of hydrazoic acid in CH_2Cl_2 or benzene resulted in instantaneous formation of 3-azidoazetidines **2** and, unlike reported reactions with strong acids [14], no formation of polymeric material was observed (*Scheme 1*). After typical aqueous workup, the oily products **2** were purified by chromatography on silica gel without noticeable decomposition. The IR spectra of the isolated products revealed the presence of N_3 and NH functional groups by absorption bands located at 2080 and *ca.* 3210 cm^{-1} , respectively. 3-Azidoazetidines **2** obtained in these reactions are fairly stable compounds, which could be stored in a refrigerator for several days. However, at room temperature, they decomposed after a few hours to a mixture of polymeric products.

The attempted reduction of **2a** with *Raney*-Ni in boiling EtOH resulted in the formation of a mixture of azetidin-3-amine **4a** and 2,5-dihydro-1*H*-imidazole **5**, which were separated by column chromatography (*Scheme 2*). Based on the ^1H -NMR spectrum of the crude mixture of products, a **4a/5** ratio of 55:45 was established. Apparently, the reduction of the azido group with *Raney*-Ni occurs *via* nitrene **3** as a reactive intermediate, which subsequently is either reduced to azetidin-3-amine **4a** or undergoes a competitive ring expansion to the isolated 2,5-dihydro-1*H*-imidazole **5**. It is noteworthy that the same ring expansion was observed in the gas phase during flash vacuum pyrolysis of **2a**, and, in this case, **5** was found as the major component of the pyrolysate collected on a cold finger [9].

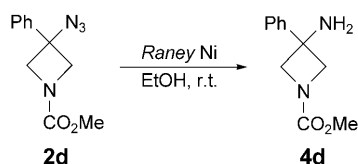
For comparison, reduction of the previously described 3-azidoazetidine-1-carboxylate **2d** [9] with *Raney*-Ni afforded 3-aminoazetidine-1-carboxylate **4d** as the sole product; in this case, ring enlargement was not observed (*Scheme 3*).

In order to avoid the undesirable reaction leading to 2,5-dihydro-1*H*-imidazoles of type **5** during reduction of **2a** with *Raney*-Ni, other reducing agents were tested (LiAlH_4 or H_2). Best results were achieved with H_2 over Pd/C. In this case, facile reactions led smoothly to azetidin-3-amines **4b–d** in good yields (*Scheme 4*). The structures of the products were established by means of spectroscopic methods. Thus, absorption bands characteristic for primary amino groups appeared in the IR spectra in the region of 3000–3300 cm^{-1} . On the other hand, the initially observed strong absorption of an N_3 group at 2080 cm^{-1} disappeared completely, which clearly indicated complete consumption of the starting material. In the ^1H -NMR spectrum of **4b**, the

Scheme 2



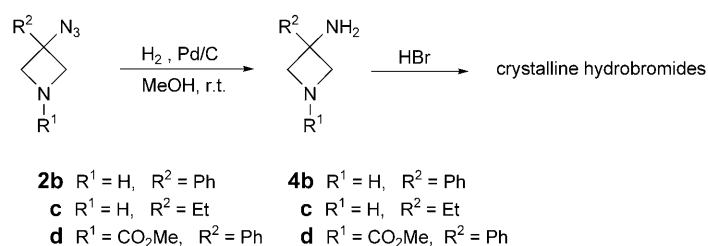
Scheme 3



signals of $\text{CH}_2(2)$ and $\text{CH}_2(4)$ appeared as an *AB*-system (3.68 and 3.94 ppm) shifted downfield in comparison with the starting **1b**.

Isolation of the pure 3-aminoazetidine-1-carboxylate **4d** was easily achieved by crystallization, but all attempts to purify the oily azetidin-3-amines **4b** and **4c** by means of chromatographic methods (column or plates) failed. For this reason, alcoholic solutions of **4b** and **4c** were treated with 2.5 mol-equiv. of HBr (*Scheme 4*). After cooling, crystalline dihydrobromides were obtained from EtOH diluted with Et_2O . The analogous treatment of **4d** with HBr afforded the monohydrobromide exclusively.

Scheme 4



3. Conclusions. – Strained 1-azabicyclo[1.1.0]butanes **1** react smoothly with hydrazoic acid at low temperature to 3-azidoazetidines **2** in basically quantitative yields. Upon hydrogenolysis with H₂ over Pd/C, these products can be cleanly converted into *N*-unsubstituted azetidin-3-amines **4**, which are valuable building blocks in organic synthesis. The presented method is based on the application of relatively easily available 1-azabicyclo[1.1.0]butanes **1**, and is more efficient than other procedures recommended for preparation of **4** [3][7]. Reduction of **2a** with Raney-Ni was proposed to occur *via* a nitrene intermediate **3**, and yielded a mixture of the expected 3-amino derivative **4a** and the ring expanded 2,5-dihydro-1*H*-imidazole **5**.

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Experimental Part

General. Melting points were determined in a capillary using a *MelTemp2* apparatus; uncorrected. IR Spectra were recorded on a *NEXUS FT-IR* spectrophotometer (KBr pellets or neat). ¹H- and ¹³C-NMR Spectra were recorded on a *Varian Gemini 200* spectrometer, and Me₄Si was used as an internal standard. ¹³C-NMR Assignments were made on the basis of DEPT experiments. Mass spectra (EI-MS) were recorded on a *MAT-112* spectrometer (15 or 70 eV). Elemental analyses were performed by the Microanalytical Laboratory of the Polish Academy of Science (CBMM Łódź).

Starting Materials. 2,2-Dimethyl-3-phenyl-1-azabicyclo[1.1.0]butane (**1a**), 3-phenyl-1-azabicyclo[1.1.0]butane (**1b**) [14], and 3-ethyl-1-azabicyclo[1.1.0]butane (**1c**) [16] were prepared according to described procedures. Hydrazoic acid (HN₃) was prepared in CH₂Cl₂ soln. according to [17], and the crude soln. was used without distillation. Raney-Ni was freshly prepared following the literature protocol [18]. Methyl 3-azido-3-phenylazetidine-1-carboxylate (**2d**) was prepared from **1b** and methyl azidoformate at r.t., according to [9].

Reactions of 1-Azabicyclo[1.1.0]butanes 1a–c with Hydrazoic Acid. The appropriate **1** (4 mmol) was dissolved in 1 ml of CH₂Cl₂ and cooled to 0–5° in a H₂O/ice bath. A cold soln. of HN₃ in CH₂Cl₂ was added dropwise with stirring, until TLC revealed consumption of the starting material. The mixture was extracted with a 1% soln. of NaOH, saturated with NaCl, before the org. layer was separated and dried (MgSO₄). The solvent was evaporated *in vacuo* without heating, and the residual oily products were identified by ¹H-NMR spectroscopy. Analytically pure products were obtained after PLC (SiO₂; CH₂Cl₂/MeOH 5 : 1): R_f 0.2 (**2a**), 0.5 (**2b**), and 0.6 (**2c**), resp.

3-Azido-2,2-dimethyl-3-phenylazetidine (2a). With **1a** (636.9 mg); yield 470 mg (58%, after chromatography). Colorless thick oil, decomposes slowly during storage at r.t. IR (film): 3210*m* (br., NH), 2920*s*, 2830*m*, 2100*vs* (N₃), 1500*m*, 1460*m*, 1360*m*, 1265*vs*, 1160*m*, 1050*m*, 910*m*, 770*s*, 730*s*, 705*vs*. ¹H-NMR (CDCl₃): 0.97 (*s*, Me); 1.45 (*s*, Me); 2.69 (*br. s*, NH); 3.68, 4.23 (*AB*, *J* = 9.2, CH₂(4)); 7.21–7.45 (*m*, 5 arom. H). ¹³C-NMR (CDCl₃): 25.4 (*q*, Me); 26.8 (*q*, Me); 49.9 (*t*, CH₂); 67.4 (*s*, C(2)); 73.0 (*s*, C(3)); 126.5, 128.2, 128.6 (3*d*, 5 arom. C); 137.6 (*s*, arom. C). EI-MS: 202 (0.3, *M*⁺), 173 (1), 145 (2), 117 (15), 77 (16), 43 (100). Anal. calc. for C₁₁H₁₄N₄ (202.26): C 65.32, H 6.98; found: C 65.12, H 7.12.

3-Azido-3-phenylazetidine (2b). With **1b** (524.7 mg); yield 530 mg (76%, after chromatography). Colorless liquid, changed to yellow on standing at r.t. IR (film) 3210*m* (NH), 2090*s*, 2020*s*, 2100*vs* (N₃), 1500*m*, 1450*m*, 1340*m*, 1260*s*, 770*s*, 715*vs*. ¹H-NMR (CDCl₃): 2.08 (*br. s*, NH); 3.97 (*br. s*, CH₂(2), CH₂(4)); 7.40 (*br. s*, 5 arom. H). ¹³C-NMR (CDCl₃): 58.2 (*t*, C(2), C(4)); 66.9 (*s*, C(3)); 125.4, 128.1, 128.8 (3*d*, 5 arom. C); 140.2 (*s*, arom. C). EI-MS: 174 (0.2, *M*⁺), 159 (3, [*M* – NH]⁺), 144 (3), 117 (8), 104 (17), 77 (12), 71 (100). Anal. calc. for C₉H₁₀N₄ (174.20): C 62.05, H 5.79; found: C 62.36, H 5.58.

3-Azido-3-ethylazetidine (2c). With **1c** (332.5 mg); yield 320 mg (63%, after chromatography). Colorless liquid, changed to yellow on standing at r.t. IR (film): 3210*m* (NH), 2090*s* (N₃), 1560*m*, 1400*s*, 1260*s*. ¹H-NMR (CDCl₃): 0.96 (*t*, *J* = 7.3, Me); 1.86 (*q*, *J* = 7.3, CH₂); 1.89 (*br. s*, NH); 3.49, 3.75 (*AB*, *J* = 9.5, CH₂(2), CH₂(4)). ¹³C-NMR (CDCl₃): 8.0 (*q*, Me); 30.0 (*t*, CH₂); 56.0 (*t*, C(2), C(4)); 65.0 (*s*, C(3)). EI-MS: 127 (12, [*M* + 1]⁺), 97 (7), 84 (21), 69 (36), 68 (32), 55 (19), 54 (98), 42 (100). Anal. calc. for C₅H₁₀N₄ (126.16): C 47.60, H 7.99; found: C 47.90, H 7.73.

Reduction of 2a with Raney-Nickel. To a stirred soln. of 260 mg (1.5 mmol) of **2a** in EtOH (10 ml) was added a suspension of freshly prepared *Raney-Ni* in EtOH in small portions at r.t. Each portion of the Ni initiated a vigorous, exothermic reaction, with evolution of N₂. When the gas evolution had ceased, the mixture was stirred for another 30 min, and the black suspension was filtered through paper to separate the black Ni precipitate. The colorless soln. was evaporated *in vacuo*, and the residual thick oil was analyzed by ¹³C- and ¹H-NMR spectroscopy. Products **4a** and **5** in a 55:45 ratio were the only components of the crude mixture. Attempted separation of both compounds using chromatography resulted in decomposition of **4a**, and only **5** could be obtained in analytically pure form.

2,2-Dimethyl-3-phenylazetidin-3-amine (4a). Identified spectroscopically as a crude product in the mixture with **5**; decomposed during attempted chromatographic workup. ¹H-NMR (CDCl₃): 0.93, 1.44 (2s, 2 Me); 2.42 (br. s, NH, NH₂); 3.36, 4.23 (AB, *J* = 9.2, CH₂(4)); 7.20–7.30 (*m*, 5 arom. H). ¹³C-NMR (CDCl₃): 24.0, 27.1 (2*q*, 2 Me); 54.7 (*s*, C(3)); 63.1 (*s*, C(2)); 67.2 (*t*, C(4)).

2,5-Dihydro-2,2-dimethyl-4-phenyl-1H-imidazole (5). Isolated after PLC (SiO₂; CH₂Cl₂/MeOH 9:1). Yield: 65 mg (30%). Pale yellow thick oil, which partially solidified at r.t. IR (neat): 3200 (br., NH), 1620s (C=N), 1460s, 1225s, 1170s, 1080*m*, 1020*m*, 850*m*, 780s, 705s. ¹H-NMR (CDCl₃): 1.48 (*s*, 2 Me); 2.45 (br. s, NH); 4.22 (*s*, CH₂(5)); 7.40–7.45 (*m*, 3 arom. H); 7.73–7.80 (*m*, 2 arom. H). ¹³C-NMR (CDCl₃): 28.0 (*q*, 2 Me); 54.8 (*t*, C(5)); 64.0 (*s*, C(2)); 91.1 (*t*, C(5)); 127.8, 128.6, 130.9 (3*d*, 5 arom. C); 132.6 (*s*, arom. C); 168.0 (*s*, C=N). EI-MS: 173 (2, [*M* – 1]⁺), 172 (18, [*M* – 2]⁺), 145 (95), 104 (92, [C₆H₅–C≡NH]⁺), 103 (25), 77 (28), 69 (100). Anal. calc. for C₁₁H₁₄N₂ (174.24): C 75.82, H 8.10, N 16.08; found: C 75.68, H 7.90, N 16.18.

Reductions of 3-Azidoazetidines 2 with H₂ over Pd/C. General Procedure. 3-Azidoazetidines **2b–d** (2.5 mmol) were dissolved in 10 ml of MeOH, and 200 mg of Pd/C were added to the soln. The black suspension was stirred vigorously in a closed vessel filled with H₂ under pressure. The progress of the reactions was monitored by TLC (SiO₂; CH₂Cl₂/MeOH 9:1), and the reactions were stopped when no more starting materials were detected in the mixture. The solns. were filtered through a paper filter, and evaporated *in vacuo* to give thick colorless oils. The crude products were identified by their ¹H-NMR and IR-spectra, and immediately converted into the corresponding hydrobromides by a treatment with a dilute soln. of HBr in MeOH. Only **4d** could be isolated and purified chromatographically. Compounds **4b** and **4c** decomposed on SiO₂ and could not be recovered from the PLC plates. When stored at r.t., **4b** and **4c** decomposed slowly to give a mixture of unidentified polymeric products.

3-Phenylazetidin-3-amine (4b). With **2b** (436.5 mg); yield of crude product: 351 mg (95%), reaction time 1 h. Colorless thick oil. IR (film): 3200vs (br., NH, NH₂). ¹H-NMR (CDCl₃): 2.50 (br. s, NH, NH₂); 3.68, 3.94 (AB, *J* = 8.0, CH₂(2), CH₂(4)); 7.16–7.53 (*m*, 5 arom. H). Crude **4b** was dissolved in MeOH and treated with a slight excess of aq. HBr diluted with MeOH. The solvent was evaporated, and the residue was triturated with a small amount of acetone to give 631 mg (82%) of pure, crystalline dihydrobromide **4b**·2 HBr. M.p. 179–182° (dec.). IR (KBr): 3300–2400vs (br.), 1480s, 1350*m*, 1305*m*, 950*m*, 910*m*, 780*m*, 705s. ¹H-NMR (D₂O): 5.25, 5.42 (2 br. s, CH₂(2), CH₂(4)); 9.80 (br. s, 5 arom. H). ¹³C-NMR (D₂O): 57.5 (*t*, C(2), C(4)); 58.0 (*s*, C(3)); 130.5, 134.1, 134.9 (3*d*, 5 arom. C); 138.5 (*s*, arom. C). Anal. calc. for C₉H₁₄Br₂N₂ (310.03): C 34.87, H 4.55, N 9.04; found: C 34.93, H 4.95, N 9.02.

3-Ethylazetidin-3-amine (4c). With **2c** (315.4 mg); yield of crude product 250 mg (99%), reaction time 1 h. Colorless thick oil. IR (neat): 3200vs (br., NH, NH₂). ¹H-NMR (CDCl₃): 0.92 (*t*, Me); 1.73 (*q*, CH₂); 3.30 (br. s, CH₂(2), CH₂(4)); 3.57 (br. s, NH, NH₂). Crude **4c** was dissolved in 2 ml of MeOH and treated with a slight excess of aq. HBr diluted with MeOH. Evaporation *in vacuo* afforded a thick oil, which was recrystallized from EtOH/Et₂O to give 478 mg (73%) of colorless crystals of dihydrobromide **4c**·2 HBr. M.p. 166–170° (dec.). IR (KBr): 3300–2400vs (br.). ¹H-NMR (D₂O): 1.62 (*t*, Me); 2.68 (*q*, CH₂); 4.83, 5.03 (AB, *J* = 12.0, CH₂(2), CH₂(4)). ¹³C-NMR (D₂O): 8.3 (*q*, Me); 30.4 (*t*, CH₂); 56.0 (*t*, C(2), C(4)); 57.2 (*s*, C(3)). Anal. calc. for C₅H₁₄Br₂N₂ (261.99): C 22.92, H 5.39, N 10.69; found: C 22.61, H 5.42, N 10.50.

Methyl 3-Amino-3-phenylazetidine-1-carboxylate (4d). With **2d** [9] (580.6 mg); reaction time 30 min, yield of crude product: 433 mg (84%), purified by crystallization from hexane. M.p. 67–69°. IR (KBr): 3250*m* (NH₂), 1700s (C=O), 1460s, 1390s, 1120*m*. ¹H-NMR (CDCl₃): 1.95 (br. s, NH₂); 3.72 (*s*, MeO); 4.08, 4.25 (AB, *J* = 9.0, CH₂(2), CH₂(4)); 7.25–7.58 (*m*, 5 arom. H). ¹³C-NMR (CDCl₃): 52.4 (*q*, MeO); 54.4 (*t*, C(2), C(4)); 64.8 (*s*, C(3)); 124.9, 126.5, 127.4 (3*d*, 5 arom. C); 144.5 (*s*, arom. C); 157.3 (*s*, C=O). Anal. calc. for C₁₁H₁₄N₂O₂ (206.24): C 64.06, H 6.84, N 13.58; found: C 63.87, H 6.96, N 13.52.

Methyl 3-Amino-3-phenylazetidine-1-carboxylate Hydrobromide (4d·HBr). From **4d**, after treatment with aq. HBr diluted with MeOH. Yield: 72%. M.p. 187–190° (dec.). Anal. calc. for C₁₁H₁₅BrN₂O₂ (287.15): C 46.01, H 5.27, N 9.76; found: C 46.06, H 5.45, N 9.64.

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