Chemical Conversion of Cyclic \(\alpha\)-Amino Acids to Cyclic \(\alpha\)-Aminophosphonic Acids

Mamoru KANAME, Hironori MASHIGE, and Shigeyuki YOSHIFUJI*

Faculty of Pharmaceutical Sciences of Hokuriku University, No.3, Kanagawa-machi, Kanazawa 920–1181, Japan.

Received October 19, 2000; accepted January 27, 2001

The oxidative decarboxylation of cyclic \(\alpha\)-amino acids having urethane-type \(N\)-protecting groups with lead tetraacetate \([\text{Pb(OAc)}_4]\) gave 2-hydroxy derivatives, which were transformed into the corresponding \(\alpha\)-aminophosphonic acid esters by treatment of trialkyl phosphites in the presence of Lewis acids. Deprotection and ester cleavage of the products in the usual manner afforded cyclic \(\alpha\)-aminophosphonic acids. The convenient chemical conversion of five- and six-membered cyclic \(\alpha\)-amino acids to the corresponding cyclic \(\alpha\)-aminophosphonic acids has been accomplished.

Key words \(\alpha\)-aminophosphonic acid; \(\alpha\)-amino acid; phosphorylation; acyliminium ion; Lewis acid; decarboxylation

\(\alpha\)-Aminophosphonic acids are believed to be phosphorus analogs of naturally occurring \(\alpha\)-amino acids and have found applications as potent active compounds with a wide range of biological activities as antibiotics,\(^1\) enzyme inhibitors,\(^2\) pharmacological agents,\(^2,3\) antiviral agents,\(^4\) and herbicides.\(^5\) Their negligible mammalian toxicity, and the fact that they bear a very close chemical resemblance to their amino carboxylic counterparts, make them remarkably important structural units of phosphonopeptides and peptidomimetics.\(^6\) During the last two decades, considerable efforts toward the synthesis of \(\alpha\)-aminophosphonic acids have been made.\(^7\) However, a simple and general synthetic method for cyclic amino-type of compounds, such as phosphonic acid analogs of 2-pyrrolidinecarboxylic acid (proline) and 2-piperidinecarboxylic acid, is not relatively known, and most of the reported methods are about acyclic compounds. The cyclic analogues can be viewed as useful tools for the elucidation of conformational requirements of a receptor, since the conformation is easily fixed. Many synthetic routes to \(\alpha\)-aminophosphonic acids and their derivatives include a key step of nucleophilic phosphorylation to either performed or \textit{in situ} generated imines (Schiff bases) or iminium ions.\(^8\) Shono\(^9\) and co-workers reported the phosphorylation of \(N\)-protected 2-methoxyamines using trialkyl phosphites in the presence of Lewis acids. Similar transformation of 2-benzotriazolylpyrrolidine derivatives into pyrrolidine-2-phosphonic acid esters was reported by Katritzky\(^10\) and co-workers. We wish to report herein the convenient conversion of cyclic \(\alpha\)-amino acids to cyclic \(\alpha\)-aminophosphonic acids through the corresponding \(N\)-protected 2-hydroxymamines, with which easy generation of acyliminium ions in the presence of Lewis acids would be expected. To our knowledge, only a few studies concerning the chemical conversion of naturally occurring cyclic \(\alpha\)-amino acid (proline or proline-containing dipeptide) into the corresponding phosphonic acid analogues have been published,\(^11,12\) and these are not systematic or detailed studies. In this paper, we describe a systematic study on a simple route to a series of phosphonic acid analogues from proline and 2-piperidinecarboxylic acid, as illustrated in Chart 1.

Initially, we examined the transformation of commercially available cyclic \(\alpha\)-amino acids (1, 2) to the key intermediates of cyclic \(N\)-protected 2-hydroxymamines (5, 6). Displacement of the carboxylic acid moiety by hydroxide group or its equivalents such as acetoxyl group had been realized by using two methods of oxidative decarboxylation: lead tetraacetate \([\text{Pb(OAc)}_4]\) oxidation of carboxylic acids and \(m\)-chloroperbenzoic acid (\(m\)-CPBA) oxidation of active esters. We applied these methods to \(N\)-protected amino acids (3a—e, 4a—c) and their active esters (3f, g), as summarized in Table 1.

Next, conversion of the 2-hydroxy compounds (5a—e) into the corresponding hydroxy compounds (5b) in satisfactory yields. In comparison with the \(\text{Pb(OAc)}_4\) method, however, this method seems to be disadvantageous for our purpose, because esterification of the starting amino acids is necessary. Therefore, oxidation of the 2-piperidinecarboxylic acid esters (4a—c) was achieved with \(\text{Pb(OAc)}_4\). \(N\)-Z derivative (4b) afforded only the hydroxy compound (6b) in high yield, while in the case of two substrates (4a, c) \(N\)-protected with trichloroethoxycarbonyl (Trocl) or \(p\)-nitrobenzoxycarbonyl (PNZ) group, the oxidation products, estimated as the hydroxy compounds, were very unstable and were completely transformed into the enecarbamates (12a, c) by elimination of \(\text{H}_2\text{O}\) during the purification. Therefore, these products were immediately used for the next step without further purification.

Next, conversion of the 2-hydroxy compounds (5, 6) into the phosphonic acid derivatives (7, 8) was studied. Conditions for the generation of the acyliminium ions and subsequent nucleophilic phosphorylation were examined with \(N\)-Troc-2-hydroxypyrrolidine (5a) as a model substrate. Upon treatment with trialkyl phosphites in the presence of 1 or 1.5 mol equivalent of various Lewis acids in \(\text{CH}_2\text{Cl}_2\) at room temperature, compound 5a was transformed into the corresponding 2-phosphonic acid esters (7aa—7ac) in variable

* To whom correspondence should be addressed. e-mail: s-yoshifuji@hokuriku-u.ac.jp © 2001 Pharmaceutical Society of Japan
yields. The results are summarized in Table 2. Production of 7aa by the reaction using a combination of trimethyl phosphite and trimethylsilyl trifluoromethanesulfonate (TMSOTf) as a Lewis acid gave the best yield of 87%. As bulkiness of the alkyl group in trialkyl phosphite increased, yield of the product was reduced. Use of the other substrates having different N-protecting groups did not improve for yield of the phosphonylation. Reaction of six-membered cyclic 2-hydroxy compounds (5a, b) with trialkyl phosphites proceeded smoothly under the same conditions, but moderate yields (46—66%) of the desired products (8a) were obtained.

All the N-protected cyclic α-amino phosphonic acid esters (7, 8) prepared above are new compounds, which were characterized on the basis of their spectral data, especially by observation of C–P spin couplings ($^1J_{CP} = 154.1—163.3$ Hz) of the C-2 carbon in $^{13}$C-NMR spectra. These N-protected derivatives would be very important for the synthesis of peptides and the related compounds containing these aminophosphonic acids. N-tert-Butoxycarbonyl (Boc) group is one of the most common amino protecting groups as well as N-Z group in the field of amino acid chemistry. However, it is impossible to use the N-Boc group in our phospho-
nlation under a Lewis acid catalyst. Therefore, we tried a conversion of the N-Z products into the N-Boc derivatives by the method developed for that of α-amino acids.16) The five and six-membered cyclic N-Z compounds (7ba, 8ba) were treated with Boc reagent (Boc2O) in methanol under hydrogenolytic conditions (Pd/C/H2) to furnish the expected N-Boc derivatives 7da and 8da in 97% and 74% yield, respectively (Chart 4).

Finally, the cyclic α-amino-phosphonic acids were obtained in a two-step sequence from N-protected α-amino-phosphonic acid methyl esters (7, 8) in good yields (Table 3). Deprotection of the N-protecting groups by the appropriate procedure utilizing in amino acid chemistry15,17) and hydrochloric acid-catalyzed hydrolysis of the methyl phosphonate moieties followed by desalting to the salt-free amino-phosphonic acids were successfully achieved. The structure of target compounds (9, 10) was supported by their analytical and spectral data, which were in accordance with literature data of the compounds (9, 10) synthesized from a different non-amino acids source.18,19)

Thus, the novel chemical conversion of five- and six-membered cyclic α-amino acids to cyclic α-amino-phosphonic acids and their N-protected derivatives has been established. The route described in this paper is potentially applicable to the preparation of optical active analogs of these compounds. N-Protected cyclic 2-hydroxamines were used in our approach for the formation of acyliminium ions. More recently, direct formation of the acyliminium ions from N-acylated cyclic α-amino acids by the novel oxidation with a combination of (diacetoxyiodo)benzene (DIB) and iodine has been reported by Boto20) and co-workers, who applied the acyliminium ions to C–C bond formation for alkaloid synthesis.

**Experimental**

All melting points were taken on a Yanagimoto micro melting point apparatus and are uncorrected. MS and HRMS were obtained on a JEOL JMS-DX300 or JMS-SX102A spectrometer. IR spectra were recorded on a Hitachi 270-30 spectrophotometer. 1H-NMR spectra were obtained at 23 °C on a JEOL PMX-60-SI, JNM-EX90A or JNM-GSX-400 spectrometer using tetramethylsilane (TMS, δ 0 ppm) or dioxane (δ 3.70 ppm from TMS) as an internal standard. 13C-NMR spectra were measured on a JEOL JNM-EX90A or JNM-GSX-400 spectrometer using TMS or dioxane (δ 67.4 ppm from TMS) as an internal standard. The following abbreviations are used: m = multiplet, q = quartet, t = triplet, d = doublet, s = singlet, and br = broad. 6J = doublet (H–P or C–P multiplicity). Column chromatography was carried out on silica gel (Kieselgel 60, 70—230 mesh, Merck) or alumina (aluminum oxide 90, 70—230 mesh, Merck).

**N-Protection of Cyclic α-Amino Acids**

All N-protected cyclic α-amino acids were prepared from commercially available amino acids (1, 2) by acylation with di-tert-butyl dicarbonate (Boc2O) or the corresponding acid chlorides under the Shotten-Baumann reaction conditions (dioxane–H2O, NaHCO3, 0 °C—room temperature).

1-(2,2,2-Trichloroethoxycarbonyl)piperidine-2-carboxylic Acid (8ba): Yield 88%.
1-(2,2,2-Trichloroethoxycarbonyl)pyrrolidine-2-carboxylic Acid (8da): Yield 84%.
1-Benzoylpyrrolidine-2-carboxylic Acid (3d): Yield 84%.
1-Benzoylpyrrolidine-2-carboxylic Acid (3e): Yield 84%.
1-(2,2,2-Trichloroethoxycarbonyl)piperidine-2-carboxylic Acid (4a): Yield 66%, colorless prisms, mp 117—119 °C (benzene–hexane). MS m/z: 303 (M+), 305 (M+ + 2), 307 (M+ + 4), 309 (M+ + 6). IR (KBr) cm−1: 3172 (OH), 1740, 1684 (C=O). 1H-NMR (CDC13, 60 MHz) δ: [1.15—2.01 (5H, m), 2.01—2.58 (1H, m), 2.75—3.53 (1H, m, C3-Ha), 3.88—4.40 (1H, m, C5-Ha), 4.79 (2H, s, OCH2CCl3), 4.85—5.16 (1H, m, C6-Hb)]. 13C-NMR (CDCl3, 60 MHz) δ: 21.7—25.3 (5C), 26.7—28.2 (2C), 30.7 (1C), 37.0—43.7 (5C), 62.2—67.9 (3C), 111.1 (1C), 126.0, 126.3 (2C), 171.2—174.9 (2C).

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<th>Substrate</th>
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<th>α-Aminophosphonic acid</th>
<th>Yield (%)</th>
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a) Yield of salt-free acid.

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**Table 3. Preparation of Cyclic α-Aminophosphonic Acids (9, 10)**

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a) Yield of salt-free acid.
1-Benzyl 2-(2,5-Dioxo-1-pyrrolidinyl) Pyrrolidine-1,2-dicarbamate (3g)\(^{30}\) A solution of N-Z-proline (3b) (9.63 g, 38.6 mmol) in DMF (50 ml) was stirred under cooling at 0 °C. N-Hydroxysuccinimide (4.4 g, 38.6 mmol) and DCC (8.77 g, 42.5 mmol) were added to the solution. The reaction mixture was allowed to warm to room temperature, and stirring was continued for 24 h. The white suspension of DCC was filtered off and washed with DMF (50 ml×2). The filtrate was concentrated under reduced pressure. The residue was diluted with isopropanol, the precipitate (DCC) was filtered off and washed with isopropanol (10 ml). The filtrate was diluted with benzene (350 ml), and washed with H₂O (100 ml×3), dieldro anhydrous Na₂SO₄ and concentrated under reduced pressure. The residue was purified by recrystallization. Yield 84%, colorless needles, mp 88—89.5° (benzene–hexane) (lit.\(^{18}\) mp 90°C). MS m/z: 346 (M⁺).

2-Benzyl 2-Hydroxypyrrolidine-1-carboxylate (5e) and (4-benzyloxy)butan-1-ol (11e): Pale yellow oil. MS m/z: 219 (M⁺). IR (neat) cm⁻¹: 1645 (OH), 1730 (C=O). 1H-NMR (CDCl₃) [60 MHz]: 6.45—6.56 (2H, m, C₃-H₂), 4.90—4.98 (1H, m, C₃-H). The reaction mixture was allowed to warm to room temperature and was stirred for a further 12 h. Water (5 ml) was added dropwise to the mixture under cooling after the reaction mixture was warmed to room temperature.

2-Azido-2-(2-azidovinyl)pyrrolidine-1-carboxylic acid (7b): Yield 71%, colorless oil. MS m/z: 381 (M⁺), 383 (M⁺+2), 385 (M⁺+4), 387 (M⁺+6). IR (neat) cm⁻¹: 1710 (C=O). 1H-NMR (CDCl₃) [60 MHz]: 6.15—6.24 (2H, m, C₃-H₂), 4.96—4.98 (1H, m, C₃-H). The reaction mixture was allowed to warm to room temperature and was stirred for a further 24 h. The white suspension of DCC was filtered off and washed with isopropanol (10 ml). The filtrate was concentrated under reduced pressure.

2-Azido-2-(2-azidovinyl)pyrrolidine-1-carboxylic acid (7b): Yield 71%, colorless oil. MS m/z: 381 (M⁺), 383 (M⁺+2), 385 (M⁺+4), 387 (M⁺+6). IR (neat) cm⁻¹: 1710 (C=O). 1H-NMR (CDCl₃) [60 MHz]: 6.15—6.24 (2H, m, C₃-H₂), 4.96—4.98 (1H, m, C₃-H). The reaction mixture was allowed to warm to room temperature and was stirred for a further 24 h. The white suspension of DCC was filtered off and washed with isopropanol (10 ml). The filtrate was concentrated under reduced pressure.

2-Azido-2-(2-azidovinyl)pyrrolidine-1-carboxylic acid (7b): Yield 71%, colorless oil. MS m/z: 381 (M⁺), 383 (M⁺+2), 385 (M⁺+4), 387 (M⁺+6). IR (neat) cm⁻¹: 1710 (C=O). 1H-NMR (CDCl₃) [60 MHz]: 6.15—6.24 (2H, m, C₃-H₂), 4.96—4.98 (1H, m, C₃-H). The reaction mixture was allowed to warm to room temperature and was stirred for a further 24 h. The white suspension of DCC was filtered off and washed with isopropanol (10 ml). The filtrate was concentrated under reduced pressure.
OCH_{3}(CH_{3})_1.190—2.40 (4H, m, C_{1}-H_{1}, C_{10}-H_{1}), 3.45—3.71 (2H, m, C_{2}-H_{2}, C_{3}-H_{3}), 4.04—4.27 (4H, m, 2\times OCH_{2}CH_{3}), 4.27—4.43 (1H, br, CH_{2}), [4.51 (0.3H, d, J=1.10 Hz), 4.70 (0.7H, d, J=11.7 Hz), OCH_{2}Cl], [4.82 (d, J=12.1 Hz), 5.07 (0.3H, d, J=11.7 Hz), OCH_{2}Cl].

13C-NMR (CDCl_{3}) [100 MHz]: δ 16.47 (q, CH_{3}), 23.50 and 23.49 (t, C_{1}), 26.81 and 27.50 (t, C_{2}), 46.39 and 47.13 (t, C_{3}, 53.91 (dd, J=163.3 Hz, C_{6}), [62.39 (td, J=0.26—1.65 Hz, OCH_{2}Cl), 75.68 (t, OCH_{2}Cl), 95.61 (s, OCH_{2}Cl), 153.29 (s, C=O). HRMS m/z was calculated for C_{12}H_{20}NO_{4}Cl_{3}P: 381.0067 (M'). Found: 381.0076. Calculated for C_{12}H_{20}NO_{4}Cl_{3}P: 243.9699 (M'—PO(OCH_{3})).

2,2,2-Trichloroethanol (H)-N-Propionyl(phenyl)pyrrolidine-1-carboxylate (7aa): Yield 95%, colorless oil. MS m/z: 279 (M'). IR (neat) cm⁻¹: 1700 (C=O).

1H-NMR (CDCl_{3}) [60 MHz]: δ 1.49 (9H, s, tert-butyl protons), 1.63—2.80 (4H, m, C_{2}-H_{2}, C_{3}-H_{3}), 3.30—3.60 (2H, m, C_{4}-H_{4}, C_{5}-H_{5}), 3.80 (6H, d, J=0.26—1.23 Hz, OCH_{2}Cl).

2,2,2-Trichloroethanol (H)-N-Propionyl(phenyl)pyrrolidine-1-carboxylate (7aa): Yield 97%, colorless oil. MS m/z: 293 (M'). IR (neat) cm⁻¹: 1696 (C=O).

H-NMR (CDCl_{3}) [60 MHz]: δ 1.49 (9H, s, tert-butyl protons), 1.50—2.23 (4H, m, C_{2}-H_{2}, C_{3}-H_{3}, C_{4}-H_{4}, C_{5}-H_{5}), 2.85—3.47 (1H, m, C_{6}-H_{a}), 3.78 (6H, d, J=0.26—1.65 Hz, OCH_{2}Cl), 10.70 (2× OCH, 4.28—4.43 (1H, m, C_{1}-H_{1}), 4.60 (1H, m, C_{2}-H_{2}).

Deprotection of N-Protected Derivatives (7, 8): Deprotection of N-protected derivatives (7, 8) was achieved as follows.

Deprotection of N-Troc Derivatives (7aa, 8aa): A solution of N-Troc compound (7aa, 8aa) (2 mmol) in acetic acid (12 ml). The suspension was stirred at room temperature for 3 h and then filtered. The filtered solution was washed with a little acetic acid. The filtrate and the washings were combined and concentrated in vacuo. The residue was used for the next step.

Deprotection of N-Z Derivatives (7, 8aa): A solution of N-Z compound (7, 8aa) (2 mmol) and 10% Pd-C (300 mg), under a hydrogen atmosphere (1 atm) in MeOH-2n HCl (5 ml—5 ml) was stirred at room temperature for 2 h. The reaction mixture was filtered off and concentrated under reduced pressure to leave a colorless oil of residue, which was used for the next step.

Deprotection of N-Boc Derivatives (7, 8aa): Deprotein D-N-Boc compound (5da, 6aa) (2 mmol) was done simultaneously with ester cleavage by acid hydrolysis as described below.

Hydrolysis of Esters and Preparation of Salt-free Cyclic α-Aminophosphonic Acids (9, 10): A solution of the above residue in 6n HCl (4 ml) was refluxed in an oil bath for 12 h. The reaction solution was concentrated under reduced pressure. The residue from 5-membered cyclic α-amino derivatives was dissolved in a small amount of H_{2}O. The aqueous solution was applied to a column of Dowex 50W×4 (50—100 mesh, H⁺ form), and eluted with H_{2}O. Concentration of the eluate to dryness afforded the crude salt-free product as a white solid. Recrystallization of the solid form H_{2}O gave the pure sample (9). The residue from 6-membered cyclic compounds was dissolved in a small amount of EtOH, and treated with propylene oxide (1 ml). Concentration of the treated solution to dryness afforded the crude salt-free amino phosphonic acid as a white solid. Recrystallization of the solid form EtOH gave the pure sample (10).

Total yields from n-protected derivatives (7, 8) are summarized in Table 3.

Pyridin-2-phosphonic Acid (9a, b): Colorless plasmids, mp 264—265°C (H_{2}O) (lit.,mp 266—267°C). MS (FAB) m/z: 152 (M⁺). IR (KBr) cm⁻¹: 3408 (OH), 2962 (NH), 1136 (P=O), 1068 (P=O).

1H-NMR (DMSO-D_{6}) [400 MHz]: δ [1.88—2.16 (3H, m), 2.16—2.32 (1H, m), C_{2}-H_{2}, C_{3}-H_{3}], 3.22—3.38 (2H, m, C_{4}-H_{4}, C_{5}-H_{5}), 3.52 (1H, dd, J=18.8—9.3 Hz, C_{H_{2}}). 13C-NMR (DMSO-D_{6}) [100 MHz]: δ [24.80 (dd, J=8.9—8.8 Hz, C_{H_{2}}), 27.23 (t, C_{3}), 47.67 (td, J=5.9 Hz, C_{3}), 56.64 (dd, J=143.8 Hz, C_{3}).

Anal. Calcd for C_{10}H_{16}O_{4}N_{3}P: C, 31.80; H, 6.67; N, 9.27.

Pyridin-2-phosphonic Acid (10a, b): Colorless needles, mp 268—270°C (ether-MeOH) (lit.,mp 269—271°C). IR (KBr) cm⁻¹: 3425 (OH), 2958 (NH), 1130 (P=O), 1076 (P=O). 1H-NMR (DMSO-D_{6}) [400 MHz]: δ [1.48—1.56 (1H, m), 1.56—1.78 (2H, m), 1.78—1.99 (2H, m), 1.99—2.50 (1H, m), C_{3}-H_{3}, C_{4}-H_{4}, C_{5}-H_{5}], [2.90—3.05 (1H, m), 3.05—3.20 (1H, m, C_{4}-H_{4}, C_{5}-H_{5}], J_{d_{1}}=0.26—1.23 Hz, C_{H_{2}}, J_{d_{1}}=12.5 Hz, C_{H_{2}}). 13C-NMR (DMSO-D_{6}) [100 MHz]: δ [22.47 (t, C_{3}), 22.62 (t, C_{3}), J=5.9 Hz, C_{3}), 46.61 (td, J=7.6 Hz, C_{3}). 55.28 (dd, J=141.9 Hz, C_{3}).

Anal. Calcd for C_{10}H_{16}O_{4}N_{3}P: C, 36.37; H, 7.32; N, 8.48.

References


