

SYNTHESIS AND PROPERTIES OF DEOXYPEGAN-1-ONE

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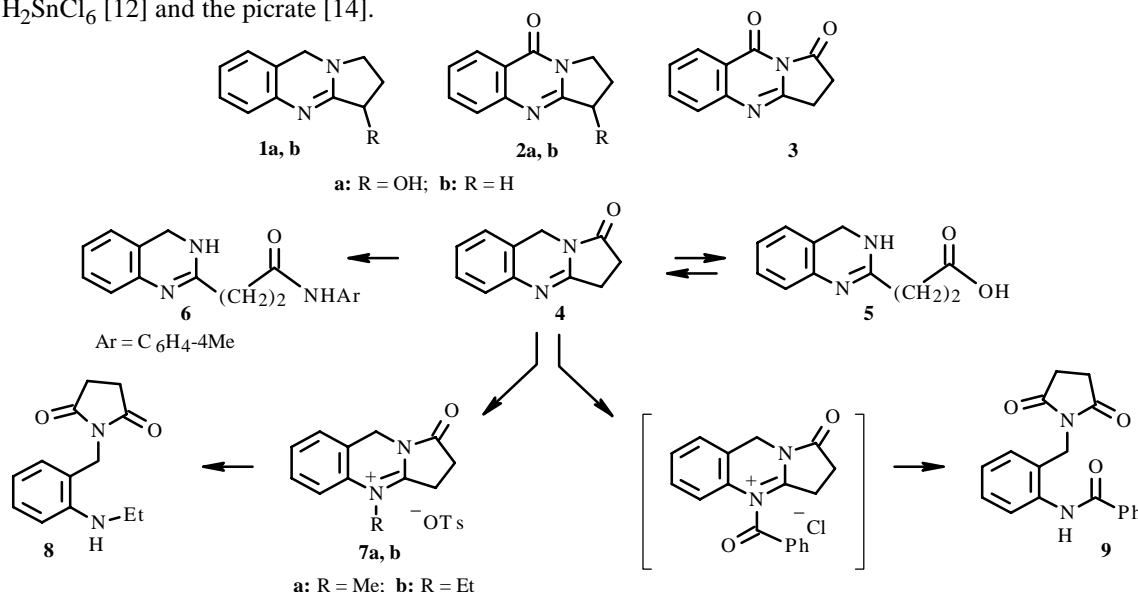
UDC 547.856.1+547.75

Quinazoly-2-propionic acid hydrochloride (**5**) was synthesized by reduction of *N*-(*o*-nitrobenzyl)succinimide with tin chloride. A pyrroloquinazolin-1-one (**4**, 3,9-dihydropyrrolo[2,1-*b*]quinazolin-1(2*H*)-one) was prepared in 68% yield by heating **5** in Ac_2O and subsequent treatment with Et_3N . Compound **4** was obtained in 71% yield in one step by reduction of *N*-(*o*-nitrobenzyl)succinimide with Fe in the presence of HCl. Compound **4** was protonated, alkylated, and acylated on the $\text{N}_{(4)}$ atom. Derivatives of quinazoly-2-propionic acid and 1-(2-aminobenzyl)succinimide were prepared by reaction of derivatives of **4** with nucleophilic reagents.

Key words: alkylation, acylation, 3,9-dihydropyrrolo[2,1-*b*]quinazolin-1(2*H*)-one, quinazoly-2-propionic acid, 1-(2-aminobenzyl)succinimide.

The search for new synthetic pathways and the study of the properties of pyrrolo[2,1-*b*]quinazoline derivatives have recently acquired increasing importance. 1,2,3,9-Tetrahydro- and 9-oxo-dihydro derivatives of this heterocyclic system [peganine (**1a**), deoxypeganine (**1b**), vasicinone (**2a**), and deoxyvasicinone (**2b**)] are the principal alkaloids of *Peganum harmala* and *P. nigellastrum* and possess distinct cholinotropic activity [1, 2]. For this reason, they have been used pharmacologically to treat various types of dependency (nicotine, alcohol, drug) and the depressive states, Alzheimer disease, and respiratory malfunctions associated with them [3-7].

Other derivatives of pyrrolo[2,1-*b*]quinazoline are also known. These include 2,3-dihydropyrrolo[2,1-*b*]quinazolin-1,9-dione (**3**) [8-11] and 3,9-dihydropyrrolo[2,1-*b*]quinazolin-1(2*H*)-one (deoxypegan-1-one, **4**), which was prepared for the first time by Gabriel at the beginning of the last century [12]. However, the properties of **3** and **4** have practically not been studied because of their scarcity. Currently known chemical transformations of **4** include only reduction [12-15] and opening of the pyrrole ring upon reaction with nucleophiles [12-14]. The only reactions with electrophiles are formation of the protonated salts **4**-HI and **4**- H_2SnCl_6 [12] and the picrate [14].



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Herein we present results from a study of the synthesis of **4** and its reactions with certain electrophiles.

Methods for preparing **4** rely on reduction of *N*-(*o*-nitrobenzyl)succinimide [12, 13, 15, 16] or succinic acid (*o*-nitrobenzyl)amide [13, 14]. In all instances, the final yield of product was <40%. We used the literature method [13] to prepare **4**. This consisted of reduction of *N*-(*o*-nitrobenzyl)succinimide by tin chloride. In the original method, the "acid chloride" formed in the final step was treated with silver carbonate solution and afforded **4** (40%). These same researchers found that the N_(9a)-C₍₁₎ bond in **4** was readily cleaved by weak bases (dilute NaOH solution or barite water) to give quinazoly-2-propionic acid (**5**). This explained the production of a small quantity (<10%) of **5** during the isolation of pyrroloquinazolin-1-one.

Our spectral investigations of the intermediate (acid chloride) showed that it was a mixture consisting of 85% quinazoly-2-propionic acid chloride (**5**·HCl). Multiple recrystallizations of the mixture from water produced pure **5**·HCl in 50% yield.

It was previously found [12] that **5** readily loses water upon vacuum distillation and cyclizes into **4**. We found that this transformation also occurs on boiling **5**·HCl in acetic anhydride and forms **4**·HCl, which reacts with Et₃N to give free base **4** in quantitative yield. Use of the acid chloride in these reactions enabled the final yield of **4** to be increased to 68%. Heating **5**·HCl in the presence of bases [12] or vacuum distillation in the presence of KOH produced **4** in yields of <40%.

A characteristic feature of the spectra of **5**·HCl salt was the presence of signals for OH and NH groups. The PMR spectrum contained singlets at 12.47 and 10.88 ppm that exchanged with D₂O. The IR spectra had a broad band for ν_{NH,OH} vibrations at 3150 cm⁻¹. Aromatic protons and methylenes C₍₂₎H₂ and C₍₃₎H₂ in the PMR spectrum of **5**·HCl resonated at weaker field compared with the spectrum of **4**·HCl. The splitting of signals in **4**·HCl and **5**·HCl corresponded to AA'XX' spin systems. The chemical shifts of the cyclic product **4**·HCl differed much more than those for **5**·HCl.

We found that **4** was also formed upon reduction of *N*-(*o*-nitrobenzyl)succinimide by Fe in the presence of HCl. In contrast with the previously developed methods of Zn or Sn reduction in acidic medium that form primarily *N*-(*o*-aminobenzyl)succinimide or quinazoly-2-propionic acid, the proposed method can produce **4** in one step in good yield (71%).

Compound **4** was hydrolytically unstable in aqueous acidic and basic solutions. Depending on the concentration and the reaction temperature, not only the N_(9a)-C₍₁₎ amide bond could cleave but also the N₍₄₎=C_(3a) imino bond. This reaction is typical of 5-imino-2-pyrrolidinone derivatives [17, 18]. Furthermore, according to the literature on the oxidation of peganine and deoxypeganine [19, 20], oxidation of the methylene at the 9-position should readily occur under these same conditions. Therefore, complicated mixtures of products are formed in most instances upon reaction of **4** with nucleophiles. However, heating **4**·HCl with *p*-toluidine produced in good yield (70%) 3-(3,4-dihydro-2-quinazolinyl)-4-(4-methylphenyl)propanamide (**6**). This same compound was easily formed by the reaction with **5**·HCl.

The simplest example of a reaction with electrophiles is the formation of protonated salts. For **4**, this can only be carried out in anhydrous media. Passing gaseous HCl through a solution of **4** in absolute EtOH produced **4**·HCl; fusion with *p*-toluenesulfonic acid, **4**·HOTs. The PMR spectra of **4**·HX salts had different positions for the signal of the aromatic proton with *o*-SSCC (8.0 Hz) assigned to the resonance of H-5. For **4**·HCl, this was an isolated doublet at 7.46 ppm; for **4**·HOTs, 7.24 ppm. Obviously, the signal for H-5 behaved this way because of shielding by the bulky anion TsO⁻ in **4**·HOTs, which was positioned near the cationic center, protonated N₍₄₎.

Pyrroloquinazolin-1-one (**4**) also forms readily quaternary salts upon fusion with alkyltosylates. Signals for protons of C₍₂₎H₂ (2.85 ppm, m), C₍₃₎H₂ (3.47 ppm, m), and H-5 (7.58 ppm, d, ³J = 8 Hz) in the PMR spectra of 4-alkyl-1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazolin-4-ium tosylates (**7a** and **b**) appeared at weaker field compared with the corresponding signals in starting **4**. Performing the reaction in solutions (acetonitrile, acetone, methylethylketone, propan-2-ol) with alkyltosylates, alkyl iodides, and benzylchloride formed **4**·HX salts. However, complicated mixtures of hydrolysis products formed in the presence of bases (K₂CO₃, *i*-PrONa, Et₃N). Quaternary salts **7a** and **b**, like protonated salts **4**·HX (X = Cl, OTs, I), are hydrolytically unstable in the presence of bases. However, in contrast with **4**·HX, the main course of the reaction with Na₂CO₃ and Et₃N is cleavage of the N₍₄₎=C_(3a) bond to produce 1-(2-aminobenzyl)succinimide (**8**), which was demonstrated using 4-ethylpyrroloquinazolin-4-ium tosylate (**7b**) as an example. Acylation products of **4** were even less stable hydrolytically. Thus, heating in anhydrous pyridine with benzoylchloride produced only the cleavage product of 4-benzoyl-1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazolin-4-ium chloride, *N*-{2-[(2,5-dioxo-1-pyrrolidinyl)methyl]phenyl}benzamide (**9**). The presence of signals for NH groups in the PMR spectra of **8** and **9** (5.26 ppm for **8** and 10.11 ppm for **9**) and in the IR spectra (3320-3420 cm⁻¹) indicated that they had formed. The methylene protons of the succinimide were equivalent and observed as a 4H singlet at 2.68-2.70 ppm.

EXPERIMENTAL

Melting points were determined on a Boetius heating stage and are uncorrected. IR spectra of compounds in KBr disks were recorded on a Pye Unicam SP3-300 instrument. PMR spectra of compounds in DMSO- d_6 solutions were obtained on a Mercury 400 (Varian, 400 MHz) instrument with TMS internal standard. Chemical shifts are given on the δ scale. The course of reactions and purity of products were monitored using TLC on Silufol UV-254 plates. Elemental analyses of all compounds agreed with those calculated.

3,9-Dihydropyrrolo[2,1-*b*]quinazolin-1(2H)-one (4). Method A. A mixture of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ (15 g, 0.07 mol) and HCl (20 mL, $\rho = 1.192$) was stirred, treated in portions with *o*-nitrobenzylsuccinimide (5 g, 0.02 mol), and heated for 1 h on a water bath at 70°C. The precipitate that formed on cooling was filtered off, washed with HCl solution ($\rho = 1.192$), and dried to afford a colorless crystalline compound (4.38 g) that was dissolved in distilled H_2O (50 mL). H_2S was passed through the solution for 15 min. The resulting yellow precipitate was filtered off. The bulk of the water remaining in the filtrate was evaporated in vacuo (water aspirator, temperature <80°C). The colorless crystalline precipitate that formed on cooling was a mixture that contained quinazolyl-2-propionic acid hydrochloride (**5**·HCl, 85%), which was filtered off and washed with alcohol.

Then the mixture (2 g) obtained from reduction of *o*-nitrobenzylsuccinimide by $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ was heated in acetic anhydride (5 mL) for 2 h. The precipitate that formed on cooling was filtered off and washed with acetone to afford colorless crystalline 3,9-dihydropyrrolo[2,1-*b*]quinazolin-1(2H)-one (**4**·HCl), which was heated with Et_3N (5 mL) for 1 h. The hot solution was decanted and concentrated in vacuo. The colorless crystalline precipitate that formed on cooling was 3,9-dihydropyrrolo[2,1-*b*]quinazolin-1(2H)-one (**4**, 1.36 g, 68%), mp 189-191°C (H_2O , lit. [15] 192°C).

Method B. A mixture of *N*-(*o*-nitrobenzyl)succinimide (1 g, 0.004 mol) and Fe (dust, 2.5 g) was heated in EtOH (10 mL) and HCl (0.5 mL, $\rho = 1.192$) for 7 h. The hot solution was filtered. Solvent was evaporated at reduced pressure. The solid was crystallized from EtOH. Yield 0.56 g (71%), mp 190-192°C (EtOH).

IR spectrum (KBr, ν , cm^{-1}): 1730 (C=O), 1630 (C=N), 1610, 1410, 1210, 1200, 810, 770. PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 7.16 (1H, m, H-7), 7.08 (2H, m, H-5, H-6), 7.03 (1H, d, J = 7.8, H-8), 4.72 (2H, s, C_9H_2), 2.82 (2H, m, C_2H_2), 2.57 (2H, m, C_3H_2). $\text{C}_{11}\text{H}_{10}\text{N}_2\text{O}$.

3,9-Dihydropyrrolo[2,1-*b*]quinazolin-1(2H)-one Hydrochloride (4·HCl). Gaseous HCl was passed through a cooled suspension of **4** (1 g, 0.005 mol) in 5 mL of absolute alcohol for 1 h. The solution was concentrated in vacuo. The precipitate that formed was filtered off, washed with acetone, and recrystallized from water to afford a colorless crystalline compound. Yield 0.88 g (74%), mp 186-189°C (EtOH). IR spectrum (KBr, ν , cm^{-1}): 3100 (N-H), 1745 (C=O), 1670 (C=N), 1640, 1590, 1420, 1200, 1180, 800, 760.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 7.45 (1H, d, J = 8.0, H-5), 7.37-7.27 (3H, m, H-6—H-8), 4.85 (2H, s, C_9H_2), 3.24 (2H, m, C_2H_2), 2.77 (2H, m, C_3H_2). $\text{C}_{11}\text{H}_{11}\text{ClN}_2\text{O}$.

3,9-Dihydropyrrolo[2,1-*b*]quinazolin-1(2H)-one Tosylate (4·HOTs). A mixture of **4**·HCl (1 g, 0.005 mol) and *p*-toluenesulfonic acid (0.77 g, 0.005 mol) was ground in a mortar, fused in an oil bath at 170°C for 1.5 h, cooled, and treated with propan-2-ol (10 mL). The resulting precipitate was filtered off, washed with propan-2-ol, and recrystallized from propan-2-ol to afford colorless crystals. Yield 1.1 g (70%), mp 176-179°C (dioxane). IR spectrum (KBr, ν , cm^{-1}): 3460 (N-H), 1790 (C=O), 1675 (C=N), 1620, 1590, 1420, 1210, 1195, 815, 725.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 7.48 (2H, d, J = 8.0, H-2', H-6'), 7.35-7.30 (3H, m, H-6—H-8), 7.24 (1H, d, J = 7.8, H-5), 7.08 (2H, d, J = 8.0, H-3', H-5'), 4.85 (2H, s, C_9H_2), 3.24 (2H, m, C_2H_2), 2.78 (2H, m, C_3H_2), 2.32 (3H, s, CH_3 -4'), $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_3\text{S}$.

3,9-Dihydropyrrolo[2,1-*b*]quinazolin-1(2H)-one Iodide (4·HI). A boiling suspension of **4** (1 g, 0.005 mol) in CH_3CN (5 mL) was treated with methyl iodide (0.8 mL, 0.008 mol), boiled for 4 h, and cooled to afford colorless crystals. Yield 1.17 g (70%), mp 245-246°C (dec., dioxane). $\text{C}_{11}\text{H}_{11}\text{IN}_2\text{O}$.

The PMR (DMSO- d_6) and IR spectra corresponded with those of **4**·HCl.

Quinazolyl-2-propanoic Acid Hydrochloride (5·HCl). Repeated recrystallization of **4**·HCl (1 g, 0.005 mol) from water afforded **5**·HCl. Yield 0.55 g (50%), mp 196-198°C (H_2O), lit. [12] mp 202°C. IR spectrum (KBr, ν , cm^{-1}): 3150 (N-H, O-H), 1730 (C=O), 1660 (C=N), 1630, 1585, 1450, 1200, 1175, 830, 770.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 12.40 (1H, s, OH), 10.82 (1H, s, NH), 7.24 (2H, m, H-5', H-7'), 7.16 (1H, t, J = 8.0, H-6'), 7.12 (1H, d, J = 8.0, H-8'), 4.72 (2H, s, $\text{C}_4'\text{H}_2$), 2.93-2.76 (4H, m, C_2H_2 , C_3H_2).

3-(3,4-Dihydro-2-quinazolinyl)-4-(4-methylphenyl)propanamide (6). **Method A.** A mixture of **4**-HCl (1 g, 0.005 mol) and *p*-toluidine (0.5 g, 0.005 mol) was ground in a mortar, fused in an oil bath at 150°C for 2 h, cooled, and treated with dioxane (10 mL). The precipitate that formed was filtered off, washed with dioxane, and recrystallized from dioxane to afford light brown crystals. Yield 0.92 g (70%).

Method B. A mixture of **5**-HCl (2 g, 0.008 mol) and *p*-toluidine (0.9 g, 0.008 mol) was ground in a mortar, fused in an oil bath at 150°C for 2 h, cooled, and treated with dioxane (10 mL). The precipitate that formed was filtered off, washed with dioxane, and recrystallized from dioxane to afford light beige crystals. Yield 1.7 g (70%), mp 202–205°C (dioxane). IR spectrum (KBr, ν , cm^{-1}): 3320 (N–H), 1670 (C=O), 1640 (C=N), 1590, 1545, 1450, 1200, 1120, 820, 770.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 10.21 (1H, s, N(1')H), 7.48 (2H, d, J = 7.6, H-2'', H-6''), 7.25–7.12 (4H, m, H-5'—H-8'), 7.02 (2H, d, J = 7.6, H-3'', H-5''), 4.74 (2H, s, C(4')H₂), 2.96 (2H, m, C(2)H₂), 2.87 (2H, m, C(3)H₂), 2.27 (3H, s, CH-4''). C₁₈H₁₉N₃O.

4-Methyl-1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazolin-4-ium Tosylate (7a). A mixture of **4** (1 g, 0.005 mol) and methyltosylate (1.1 g, 0.005 mol) was ground in a mortar, fused in an oil bath at 170°C for 2.5 h, cooled, and treated with propan-2-ol (10 mL). The precipitate that formed was filtered off, washed with propan-2-ol, and recrystallized from propan-2-ol to afford colorless crystals. Yield 1.25 g (60%), mp 130–134°C (propan-2-ol). IR spectrum (KBr, ν , cm^{-1}): 1790 (C=O), 1665 (C=N), 1595, 1220, 1120, 820, 685.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 7.54 (1H, d, J = 8.0, H-5), 7.40–7.20 (5H, m, H-6—H-8, H-2', H-6'), 7.04 (2H, d, J = 8.0, H-3', H-5'), 4.87 (2H, s, C(9)H₂), 3.65 (3H, s, NCH₃), 3.46 (2H, m, C(2)H₂), 2.85 (2H, m, C(3)H₂), 2.31 (3H, s, CH₃-4'). C₁₉H₂₀N₂O₃S.

4-Ethyl-1,2,3,9-tetrahydropyrrolo[2,1-*b*]quinazolin-4-ium tosylate (7b) was prepared by the method given for **7a** using ethyltosylate (1.1 g, 0.005 mol). Yield 1.4 g (65%), mp 160–163°C (propan-2-ol). IR spectrum (KBr, ν , cm^{-1}): 1800 (C=O), 1640 (C=N), 1595, 1200, 1130, 815, 675.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 7.58 (1H, d, J = 8.0, H-5), 7.47–7.41 (5H, m, H-6—H-8, H-2', H-6'), 7.03 (2H, d, J = 8.0, H-3', H-5'), 4.86 (2H, s, C(9)H₂), 4.20 (2H, q, ³J = 7.0, NCH₂CH₃), 3.47 (2H, m, C(2)H₂), 2.85 (2H, m, C(3)H₂), 2.31 (3H, s, CH₃-4'), 1.39 (3H, t, ³J = 7.0, NCH₂CH₃). C₂₀H₂₂N₂O₃S.

1-[2-(Ethylamino)benzyl]-2,5-pyrrolidinedione (8). **7b** (1 g, 0.003 mol) was boiled with Et₃N (5 mL) for 1 h. The solution was concentrated in vacuo. The oil that formed was treated with water. The aqueous solution was extracted three times with CCl₄ (30 mL each). The extract was evaporated to afford a light brown oil. Yield 0.34 g (50%). IR spectrum (KBr, ν , cm^{-1}): 3440 (N–H), 1700 (C=O), 1640 (C=N), 1545, 1180, 1030, 825, 770.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 7.12 (1H, d, J = 7.2, H-6), 7.07 (1H, t, J = 8.0, H-4), 6.52 (2H, m, H-3, H-5), 5.26 (1H, br.s, NH), 4.42 (2H, s, ArCH₂), 3.08 (2H, q, ³J = 7.0, NCH₂CH₃), 2.68 (4H, s, –C(O)CH₂CH₂CO–), 1.29 (3H, t, ³J = 7.0, NCH₂CH₃). C₁₃H₁₆N₂O₂.

N-{2-[(2,5-Dioxo-1-pyrrolidinyl)methyl]phenyl}benzamide (9). A boiling suspension of **4** (1 g, 5 mmol) in pyridine (5 mL) was treated with benzoyl chloride (2.2 mL, 5 mmol), boiled for 14 h, cooled, and treated with water (20 mL). The precipitate that formed was filtered off, washed with water, and recrystallized from propan-2-ol to afford colorless crystals of **9**. Yield 0.88 g (57%), mp 186–190°C (propan-2-ol). IR spectrum (KBr, ν , cm^{-1}): 3320 (N–H), 1700 (C=O), 1675 (C=O), 1530, 1410, 1350, 1310.

PMR spectrum (400 MHz, DMSO- d_6 , δ , ppm, J/Hz): 10.11 (1H, s, NH), 8.05 (2H, d, J = 8.0, H-2, H-6), 7.59–7.48 (4H, m, H-3—H-5, H-6'), 7.31 (2H, m, H-3', H-4'), 7.16 (1H, t, J = 8.0, H-5'), 4.59 (2H, s, ArCH₂), 2.71 (4H, s, –C(O)CH₂CH₂CO–). C₁₈H₁₆N₂O₃.

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