Synthesis of Perhydro-2(1*H*)-quinoxalinones and Perhydropyrrolo[1,2-*a*]quinoxalin-4(5*H*)-one Derivatives

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The novel trans-bicyclic-perhydro-2(1H)-quinoxalinones 4, 6 and 7 and the tricyclic-perhydropyrrolo[1,2-a]-quinoxalin-4(5H)-one derivatives 8 and 9 are prepared via a ring opening and spontaneous ring closing reaction of the aziridines 2 and 3 with the α -amino acids glycine, L-alanine, L-proline and L-phenylalanine. This methodology was used to prepare 5 and 10 which are novel rigid analogues of the kappa opioid compound 1. Treatment of aziridine 3 with methyl carbamate gave the cyclic urea 11.

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Research into the development of drugs for the alleviation of pain has focused recently on the identification of chemical structures that bind to kappa opioid receptors in the brain [1,2]. As part of a research programme in these laboratories aimed at producing novel kappa opioid structures it was decided to investigate the synthesis of rigid analogues of one of the most selective kappa opioid compounds, the 1,2-aminoamide 1 [3,4]. The target compounds were 5 and 10, where the two nitrogen atoms are incorporated into a piperazine ring to increase rigidity.

The synthetic route chosen to these novel heterocyclic moieties involved treating the aziridines 2 and 3 with the α -amino acids glycine, L-alanine, L-proline and L-phenylalanine (Scheme 1 and Scheme 2).

Aziridines have been reported previously to undergo ring cleavage reactions with a variety of nucleophilic reagents [5] including primary or secondary amines [6,7], hydrogen halides [6], alcohols [5,6] and inorganic azides [8]. Treatment of aziridines with nucleophiles possessing a latent electrophilic site is also known, this being a method of

constructing heterocyclic compounds [9]. For example ethyleneimine itself reacts with α -amino esters to give piperazinones [10], while α -mercapto ketones give thiazines [5,9] and malonate esters give substituted pyrrolidones [11].

In this paper the scope of the reaction of aziridine with α -amino esters is extended to include the substituted aziridines 2 and 3 with optically active amino acids. This provides a facile route to the 3-substituted decahydro-2(1H)-quinoxalinones 4, 6 and 7 and the dodecahydro-pyrrolo-[1,2-a]quinoxalin-4(5H)-ones 8 and 9.

When the aziridines 2 or 3 and the α -amino acid glycine are refluxed for 1-2 hours in water containing a catalytic

$$NR_1$$
 · NR_2 · NH_2 · N

NCH, R
$$\stackrel{\text{CH}_1}{\longrightarrow}$$
 COOH $\stackrel{\text{CH}_2}{\longrightarrow}$ COOH $\stackrel{\text{CH}_1}{\longrightarrow}$ COOH

Scheme 2

amount of ammonium chloride the bicyclic piperazinone 4a or 4b (respectively) is formed (Scheme 1). It is presumed that the mechanism involves a ring opening of the aziridine followed by a fast intramolecular condensation step. Compound 4b was converted into 5, a rigid analogue of compound 1 by reduction with aluminium lithium hydride, followed by acylation with 4-benzo[b]thiophenacetyl chloride.

When 3 was treated with L-alanine (R = CH₃) a 56% yield of an approximately 1:1 mixture of optically active diastereoisomers 6a and 7a was produced. These were separated by silica gel chromatography and converted into hydrochloride salts to form crystalline derivatives which were distinguished by proton nmr nOe experiments as follows. One of these diastereoisomers showed a strong nOe between the C(3)-methyl group and the C(4a)-axial proton (see Figure 1 and the Experimental). This would be expected for compound 7a but not for compound 6a. Several other nOe's were observed all of which are entirely consistent with the configuration shown for compound 7a in Figure 1. The other isomer was assigned structure 6a. A similar reaction with L-phenylalanine (R = CH₂Ph) also produced two optically active diastereoisomers 6b and 7b. in combined yield of 48%. One of these showed an nOe from the C(3)-H to the C(4a)-H axial proton indicating it has the configuration 6b shown in Figure 2. The other isomer was assigned structure 7b.

Figure 1

Figure 2

The ease of performing these transformations suggested that it should be possible to construct a saturated pyrroloquinoxaline skeleton using proline as the amino acid. Hence compound 3 was treated with L-proline and the expected diastereoisomeric mixture of optically active piperazinones 8b and 9b was obtained in a combined yield of 72% (Scheme 2). The aziridine 2 reacted analogously although in lower yield to give the piperazinones 8a and 9a. Reduction of the major isomer with aluminium lithium hydride and acylation of the resultant diamine as described above gave the dodecahydropyrrolo[1,2-a]quinoxaline derivative 10.

To investigate the scope of this annulation procedure for constructing smaller rings the aziridine 3 was treated with methyl carbamate. This gave the novel five membered cyclic urea 11 as a crystalline solid (28%).

EXPERIMENTAL

Melting points were determined using a Reichart Thermovar hot-stage apparatus and are uncorrected. Proton nmr spectra were recorded on a Bruker AM 300 spectrometer; chemical shifts were recorded in parts per million downfield from tetramethylsilane. The ir spectra were recorded using a Perkin-Elmer 1750 spectrophotometer. Silica gel used for chromatography was Kieselgel-60 (230-400 mesh). Mass spectra were recorded using electron impact on a Finnegan 4500 spectrometer.

trans-Decahydro-2(1H)-quinoxalinone (4a).

7-Azabicyclo[4.1.0]heptane 2 [6] (1.0 g, 10 mmoles), glycine (0.85 g, 11 mmoles), ammonium chloride (20 mg, 0.37 mmole) and water (3 ml) were

heated to reflux for 1.5 hours then cooled to 20°. During this cooling the title compound crystallised and was isolated by filtration, washed with cold water and dried to give 4a, (0.64 g, 40%), mp 201-201.5°; (lit [13] 199-199.5°); pmr (deuteriochloroform): 1.2-1.9 (8 H, m), 2.43 (1 H, m), 3.02 (1 H, m), 3.50 (3 H, s), 6.13 (1 H, br s); ir (nujol mull): 3469 (NH), 1677 (CO) cm⁻¹; ms: (m/e) 154 (M*, 100%).

Anal. Calcd. for C_eH₁₄N₂O: C, 62.3; H, 9.15; N, 18.2. Found: C, 62.2; H, 9.1; N, 18.0.

trans-3-Methyl-decahydro-2(1H)-quinoxalinone (4b).

7-Methyl-7-azabicyclo[4.1.0]heptane 3 [12], (5.5 g, 50 mmoles), glycine (2.2 g, 29 mmoles), ammonium chloride (20 mg, 0.37 mmole) and water (5 ml) were heated to reflux for 2 hours. The reaction mixture was poured into water (50 ml) and extracted with dichloromethane (2 × 50 ml). The organic layers were dried (magnesium sulphate) and distilled to give 4b (3.0 g, 62%) bp 126-128° (0.5 mbar); pmr (deuteriochlorform) 1.1-1.6 (5 H, m), 1.83 (3 H, m), 2.21 (1 H, m), 2.52 (1 H, m), 2.93 (3 H, s), 2.95 (1 H, m), 3.61 (2 H, m); ir (liquid film): 1641 (CO) cm⁻¹; ms: (m/e) 168 (M*100%).

Anal. Calcd. for C₉H₁₆N₂O·0.5H₂O: C, 61.0; H, 9.7; N, 15.8. Found: C, 61.2; H, 9.4; N, 16.0.

trans-1-Methyl-4(benzo[b]thien-4-ylacetyl)decahydroquinoxaline monohydrochloride (5).

Compound 4b (2.0 g, 12 mmoles) was dissolved in diethyl ether (5 ml) and added over 10 minutes to a stirred slurry of aluminium lithium hydride (0.90 g, 24 mmoles) in diethyl ether in an atmosphere of nitrogen gas at 0°. This mixture was warmed to 20° for 1 hour then propan-2-ol (3 ml) followed by water (100 ml) were added. The resulting suspension was extracted with dichloromethane (3 × 100 ml) to give an oil which was purified by silica gel chromatography using ethyl acetate-triethylamine (15:1) followed by ethyl acetate-methanol-aqueous ammonia (4:1:1) as eluant to give trans-1-methyldecahydroquinoxaline as an oil (0.80 g, 44%); pmr (deuteriochloroform): 2.98 (2 H, m), 2.80 (1 H, m), 2.50-2.15 (4 H, m), 2.27 (3 H, s), 2.05 (1 H, m), 1.72 (2 H, m), 1.53 (1 H, m), 1.4-1.0 (4 H, m); ir (liquid film): 3220 (NH); ms: (m/e) 154 (M* for C₉H₁₈N₂, 80%), 111 (100).

Benzo[b]thiophene-4-acetic acid (Aldrich Chemical Company) (137 mg, 0.71 mmole) was dissolved in thionyl chloride (2.0 ml) and heated to reflux for 40 minutes then concentrated in vacuo to give an oil which was dissolved in dichloromethane (5 ml) and added to the above amine (100 mg, 0.65 mmole). After 5 minutes at room temperature diethyl ether (15 ml) was added to give a white precipitate that was purified by silica gel chromatography using dichloromethane-methanol (5:1) as eluant to give compound 5, mp 126-132°; pmr (deuteriochloroform): 7.85 (1 H, d, J = 7), 7.55 (2 H, m), 7.20 (2 H, m), other signals very broad 4.3-1.2 (20 H, m); ir (liquid film): 3400 (NH), 1646 (CO) cm⁻¹; ms (m/e) 328 (M⁺, 40%), 124 (100).

Anal. Calcd. for C₁₉H₂₄N₂OS·HCl·H₂O: C, 59.6; H,7.1; N, 7.3. Found: C, 59.7; H, 7.0; N, 7.2.

trans-(3S,4aR,8aR)-1,3-Dimethyldecahydro-2(1H)-quinoxalinone Monohydrochloride (6a) and trans-(3S,4aS,8aS)-1,3-dimethyldecahydro-2(1H)-quinoxalinone Monohydrochloride (7a).

7-Methyl-7-azabicyclo[4.1.0]heptane 3 [12] (0.87 g, 7.8 mmoles), L-alanine (0.80 g, 9.0 mmoles), ammonium chloride (10 mg, 0.19 mmole) and water (10 ml) were heated together under reflux for 18 hours then the reaction mixture was poured into aqueous potassium carbonate (5%) and extracted with dichloromethane (3 × 40 ml). The combined organic layer was dried (potassium carbonate) and purified by silica gel chromatography using dichloromethane-methanol (10:1) as eluant to separate the diastereoisomers. These were converted into the crystalline monohydrochloride salts by dissolving in diethyl ether (10 ml) and adding an excess of dry ethereal hydrogen chloride solution to give compound 6a (460 mg, 27%) and compound 7a (440 mg, 26%). Compound 6a had mp 245-247°, [α] $_{\rm b}^{20}$ – 56° (C 1.0 in dichloromethane); pmr (deuteriochloroform): 10.45 (2 H, br s), 3.90 (1 H, br s), 3.80 (1 H, dt, J = 4, 12 Hz), 3.00 (1 H, m), 2.97 (3 H, s), 2.48 (1 H, m), 2.35 (1 H, m), 1.90 (3 H, s), 1.78 (3 H, d, J = 7 Hz),

1.31 (3 H, m); ir: 3401, 2655, 2510 (NH), 1652 cm⁻¹ (CO); ms: (m/e) 182 (M⁺, 100%), 139 (74), 111 (75).

Anal. Calcd. for $C_{10}H_{18}N_2O$ ·HCl: C, 54.9; H, 8.8; N, 12.8; Cl, 16.2. Found: C, 54.5; H, 8.7; N, 12.6; Cl, 16.0.

Compound 7a had mp 215-217.5°, $[\alpha]_0^{20} = 42^\circ$ (C = 1.0 in dichloromethane); pmr (deuteriochloroform): 10.5 (2 H, br s), 4.07 (1 H, q, J = 7 Hz, CHMe), 3.64 (1 H, dt, J = 3, 11 Hz C(8a)-Hax), 3.05 (1 H, m, C(4a)-Hax), 2.91 (3 H, s, N-Me), 2.46 (1 H, br d, J = 11 Hz, C(5)-Heq), 2.31 (1 H, br d, J = 10 Hz, C(8)-Heq), 1.88 (3 H, m, C(6)-Heq, C(7)-Heq, C(5)-Hax), 1.72 (3 H, d, J = 7 Hz, CHMe), 1.40 (2 H, m C(7)-Hax, C(6)-Hax), 1.22 (1 H, m, C(8)-Hax); configurational assignment was made because strong nOe was observed between resonances at 3.05 to 1.72, 2.91 to 3.64, 2.46 to 1.88 and 2.46 to 3.05, 2.31 to 1.22 and 2.31 to 2.91 and 2.31 to 3.64, 4.07 to 1.72; ir (liquid film): 3436, 2654, 2499, (NH), 1646 cm⁻¹ (CO); ms: (m/e) 182 (M*, 100%), 139 (74), 111 (75).

Anal. Calcd. for C₁₀H₁₈N₂O·HCl·0.25H₂O: C, 53.8; H, 8.8; N, 12.5; Cl, 15.9. Found: C, 54.1; H, 8.6; N, 12.6; Cl, 16.3.

trans-(3S,4aR,8aR)-1-Methyl-3-(phenylmethyl)-2(1H)-quinoxaline Monohydrochloride (6b) and trans-(3S,4aS,8aS)-1-methyl-3-(phenylmethyl)-2(1H)-quinoxalinone (7b).

7-Methyl-7-azabicyclo[4.1.0]heptane 3, [12] (0.60 g, 5.4 mmoles), L-phenylalanine (1.0 g, 6.1 mmoles), ammonium chloride (10 mg, 0.19 mmole) and water (10 ml) were heated together under reflux for 18 hours then poured into aqueous potassium carbonate (5%) and extracted with dichloromethane (3 × 40 ml). The combined organic layer was dried (potassium carbonate) and purified by silica gel chromatography using dichloromethane-methanol (20:1) as eluant to separate the diastereoisomers. The parent amine of one of these crystallised on standing at room temperature to give 7b (0.325 g, 23%), mp = 44.5-46.5°C; $[\alpha]_b^{20} = -40^\circ$ (C = 0.25 in dichloromethane); pmr (deuteriochloroform): 7.25 (5 H, m), 3.73 (1 H, dd, J = 7,2 Hz, CH₂Ph), 3.28 (1 H, dd, J = 7,2 Hz, CH₂Ph), 2.99 (1 H, m), 2.96 (3 H, s), 2.93 (1 H, m), 2.75 (1 H, m), 2.20 (1 H, m) 1.50 (8 H, m); ir (liquid film): 1641 cm⁻¹ (CO); ms: (m/e) 258 (M*, 2%), 167 (100%).

Anal. Calcd. for $C_{13}H_{22}N_2O \cdot 0.75H_2O$: C, 70.7; H, 8.7; N, 10.3. Found: C, 70.7; H, 8.4; N, 10.2.

The other product was dissolved in diethyl ether (10 ml) and treated with an excess of dry ethereal hydrogen chloride solution to form the crystalline derivative **6b** (0.40 g, 25%) mp 184-186°C; $[\alpha]_D^{20} = -90^\circ$ (C = 0.96 in dichloromethane); pmr (deuteriochloroform: 10.0 (2 H, br s), 7.43 (2 H, d, J = 6 Hz, σ -aromatic C-H), 7.21 (3 H, m- and p-aromatic C-H), 4.08 (1 H, t, J = 6 Hz, C(3)-H), 3.84 (1 H, dt, J = 3, 11 Hz, C(8a)-Hax), 3.51 (1H, dd, J = 14, 6.5 Hz, C H_2 Ph), 3.32 (1 H, dd, J = 14, 6.5 Hz, C H_2 Ph), 2.96 (1 H, t, part obscured, J = 9.5 Hz, C(4a)-Hax), 2.91 (3 H, s), 2.33 (2 H, m C(8)-Heq and C(5)-Heq), 1.55 (3 H, m), 1.25 (3 H, m), configurational assignment was made because strong nOe was observed between resonances at 2.91 to 3.84, 3.84 to 2.33, 4.08 to 2.96, 4.08 to 2.91, 4.08 to 3.51, 4.08 to 3.32; ir (liquid film) 3401 (NH), 1658 cm⁻¹ (CO); ms: (m/e) 258 (M*, 2%), 167 (100%).

Anal. Calcd. for $C_{16}H_{22}N_2O$ ·HCl·0.5 H_2O : C, 63.3; H, 8.0; N, 9.2; Cl, 11.7. Found: C, 63.6; H, 7.7; N, 9.3; Cl, 11.9.

(3aS,5aR-trans,9aR)Dodecahydropyrrolo[1,2-a]quinoxalin-4(5H)-one and (3aS,5aS-trans,9aS)Dodecahydropyrrolo[1,2-a]quinoxalin-4(5H)-one (8a and 9a).

7-Azabicyclo[4.1.0]heptane **2** [6] (2.5 g, 26 mmoles), L-proline (3.3 g, 29 mmoles), ammonium chloride (30 mg, 0.6 mmole) and water (6 ml) were heated together in an oil bath at 90-100° for 23 hours. The mixture was concentrated in vacuo and purified by silica gel chromatography using dichloromethane-methanol (8:1) as eluant to give **8a** and **9a**, combined yield = 1.30 g (26%). The major isomer (820 mg, 16%) has mp 149-155° (tetrahydrofuran), $\left[\alpha\right]_{0}^{20} = -34^{\circ}$, (C = 0.84 in dichloromethane); pmr (deuteriochloroform): 6.07 (1 H, br), 3.78 (1 H, t, J = 7 Hz, C(3a)-H), 3.17 (2 H, m), 2.65 (1 H, dt, J = 8, 10 Hz), 2.25-1.65 (9 H, m), 1.25 (4 H, m); ir (liquid film): 3180 (NH), 1670 cm⁻¹ (CO); ms: (m/e) 194 (M⁺, 37%), 138 (27).

Anal. Calcd. for C₁₁H₁₈N₂O·0.1H₂O: C, 67.4; H, 9.4; N, 14.3. Found: C, 67.6; H, 9.2; N, 14.3.

The minor isomer (480 mg, 10%) has mp 141-147°C (tetrahydrofuran), $[\alpha]_{\rm b}^{20} = -4^{\circ}$ (C = 1.0 in dichloromethane); pmr (deuteriochloroform): 6.00 (1 H, br), 3.33 (2 H, m), 2.87 (1 H, m), 2.50 (2 H, m), 2.24 (1 H, m), 2.1-1.6 (7 H, m), 1.5-1.2 (4 H, m); ir (liquid film): 3190 (NH), 1670 cm⁻¹ (CO); ms: (m/e) 194 (M⁺, 42%), 138 (33).

Anal. Calcd. for C₁₁H₁₈N₂O·0.1H₂O: C, 67.4; H, 9.4; N, 14.3. Found: C, 67.5; H, 9.2; N, 14.2.

(3aS,5a-trans)Dodecahydro-5-(benzo[b]thien-4-ylacetyl)pyrrolo[1,2-a]-quinoxaline (10).

The major isomer described above (0.50 g, 2.6 mmoles) was dissolved in tetrahydrofuran (45 ml) and added over 2 minutes to a stirred slurry of aluminium lithium hydride (94 mg, 2.5 mmoles) in diethyl ether (25 ml) in an atmosphere of nitrogen gas at 0°. The mixture was warmed to room temperature and after 3 hours more aluminium lithium hydride (100 mg, 2.7 mmoles) was added. After a total of 27 hours ethyl acetate (5 ml) followed by saturated aqueous ammonium chloride (50 ml) were added and the mixture was filtered and concentrated in vacuo. The resulting oil was purified by silica gel chromatography using dichloromethane-methanol-triethylamine (4:1:0 then 95:4:1) as eluant to give (3a.S,5a-trans)decahydropyrrolo[1,2-a]quinoxaline as an oil (100 mg); pmr (deuteriochloroform): 3.18 (2 H, m), 2.19 (1 H, br), 2.75 (1 H, dd, J = 10, 9 Hz), 2.40 (1 H, m), 2.2-1.5 (10 H, m); 1.4-1.1 (5 H, m); ir (liquid film): 3268 (NH); ms: (m/e) 180 (M*, 71%), 137 (52).

Benzo[b]thiophene-4-acetic acid (Aldrich Chemical Company) (118 mg, 0.61 mmole) was dissolved in thionyl chloride (2 ml), heated to reflux for 40 minutes and then concentrated in vacuo to give an oil which was dissolved in dichloromethane (5 ml) and added to the above amine (100 mg, 0.55 mmole). After 5 minutes at room temperature diethyl ether (10 ml) was added to give white crystalline monohydrochloride salt 10 (123 mg, 57%). An analytically pure sample obtained by silica gel chromatography using dichloromethane-methanol (5:1) as eluant had mp 42-45°, $[\alpha]_{D}^{20} = -23^{\circ}$ (C = 0.25 in dichloromethane); pmr (deuteriochloroform): 7.83 (1 H, d, J = 9 Hz), 7.25 (1 H, m part obscured), 4.05 (3 H, br m), 3.80 (1 H, br), 3.35 (2 H, br), 2.46 (2 H, m), 2.0-1.1 (14 H, m); ir (liquid film): 3415 (NH), 1646 cm⁻¹ (CO); ms: (m/e) 354 (M*).

Anal. Calcd. for C₂₁H₂₆N₂OS·HCl·H₂O: C, 61.7; H, 7.1; N, 6.8. Found: C, 61.45; H, 7.1; N, 7.0.

(3aS,5aR-trans,9aR)Dodecahydro-5-methylpyrrolo[1,2-a]quinoxalin-4(5H)-one Monohydrochloride and (3aS,5aS-trans,9aS)dodecahydro-5-methylpyrrolo[1,2-a]quinoxalin-4(5H)-one Monohydrochloride (8b and 9b).

7-Methyl-7-azabicyclo[4.1.0]heptane 3 [12] (5.7 g, 51 mmoles), L-proline (4.6 g, 40 mmoles), ammonium chloride (30 mg, 0.6 mmole) and water (5 ml) were heated together in an oil bath at 90-100° for 22 hours. The mixture was concentrated in vacuo and purified by silica gel chromatography using dichloromethane-methanol (10:1) as eluant to give **8b** (3.0 g, 36%), and **9b** (3.0 g, 36%), combined yield = 72%. Each of these was converted into monohydrochloride salts (procedure described for **6a** and **7a**) to give crystalline derivatives. One of these has mp 170-174°, $[\alpha]_p^{20} = +74^{\circ}$ (C = 0.77 in dichloromethane); pmr (deuteriochloroform): 13.5 (1 H, br), 4.44 (1 H, m), 3.60 (1 H, m), 3.46 (2 H, m), 3.08 (1 H, m), 2.99 (3 H, s), 2.7-1.8 (8 H, m), 1.40 (4 H, m); ir (liquid film): 3436 (NH), 1651 cm⁻¹ (CO); ms: (m/e) 208 (M⁺, 87%) 179 (36), 151 (57).

Anal. Calcd. for $C_{12}H_{20}N_2O$ ·HCl $0.8H_2O$: C, 55.6; H, 8.8; N, 10.8; Cl, 13.7. Found: C, 55.6: H, 8.7; N, 10.7; Cl, 13.8.

The other isomer has mp 188-191°, $[\alpha]_{D}^{20} = +2^{\circ}$ (C = 1.1 in dichloro-

methane); pmr (deuteriochloroform): 13.1 (1 H, br), 4.37 (1 H, m), 3.88 (1 H, m), 3.17 (1 H, m), 3.02 (3 H, s), 2.90 (2 H, m), 2.7-1.8 (9 H, m), 1.36 (3 H, m), ir (liquid film): 3416 (NH), 1651 cm⁻¹ (CO); ms (m/e) 208 (M⁺, 82%), 179 (33), 151 (56).

Anal. Calcd. for C₁₂H₂₀N₂O·HCl: C, 58.9; H, 8.6; N, 11.4; Cl, 14.4. Found: C, 58.8; H, 8.65; N, 11.2; Cl, 14.3.

trans-Octahydro-1-methyl-2H-benzimidazol-2-one (11).

7-Methyl-7-azabicyclo[4.1.0]heptane 3 [12] (6.3 g, 57 mmoles), methyl carbamate (4.7 g, 63 mmoles) and ammonium chloride (20 mg, 0.4 mmole) were heated together at $100\text{-}110^\circ$ for 18 hours. Then the mixture was poured into water (50 ml), extracted with dichloromethane (3 × 30 ml) and purified by silica gel chromatography using dichloromethanemethanol (10:1) as eluant to give the title compound (2.5 g, 28%) mp $102\text{-}105^\circ$ (hexane); pmr (deuteriochloroform): 4.82 (1 H, br), 3.02 (1 H, m), 2.74 (1 H, dt, part obscured, J = 12, 3 Hz), 2.71 (3 H, s), 2.01 (2 H, m), 1.85 (2 H, m), 1.47 (4 H, m); ir (liquid film): 3248 (NH), 1712 cm⁻¹ (CO); ms: (m/e) 154 (M⁺, 70%), 111 (100).

Anal. Calcd. for C₈H₁₄N₂O: C, 62.3; H, 9.1; N, 18.2. Found: C, 62.3; H, 9.1; N, 18.0.

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