It seems reasonable to conclude, therefore, that the rearrangement occurs at the stage in which the enamine I is subjected to the rather strenuous condition of being refluxed in ethylene glycol. The rearrangement can be pictured as a dissociation of I to methyl acrylate and VI followed by a Michael addition of the acrylate to carbon 1 of VI and cyclization. A simpler preparation of V than those discussed thus far consists of heating an ethylene glycol solution of 2-tetralone with methylamine followed by the addition of methyl acrylate. The reaction mixture is composed of at least six compounds, but the product V can be isolated easily.

Experimental

1-(β-Carbethoxyethyl)-2-tetralone (IV).—To a stirred solution of 7 g. of ethyl acrylate and 10 g. of 2-tetralone in 40 ml. of absolute ethanol was added 10 drops of a solution prepared from 500 mg. of sodium and 5 ml. of ethanol. The mixture was cooled to maintain a temperature below 30°, and after being stirred at room temperature for 2 hr. the mixture was poured into cold water and acidified with hydrochloric acid; the product was extracted with ether. The ether extract was washed with sodium bicarbonate solution and water, dried, and distilled giving 4.8 g. of IV, b.p. 165–166° at 4 mm. [lit.[10] b.p. 162.5° at 4 mm.].

1-(β-Carbethoxyethyl)-2-tetralone Oxime.—A mixture of 1.0 g. of the keto ester IV, 0.35 g. of hydroxylamine hydrochloride, 0.41 g. of anhydrous sodium acetate, and 20 ml. of absolute ethanol was refluxed for 6 hr. The reaction mixture was concentrated, diluted with water, and extracted with ether. The ether extract was washed with saturated sodium bicarbonate solution and water, dried, and concentrated. Recrystallization of the residue from aqueous ethanol gave 0.8 g. (78%) of the oxime, m.p. 101–102° raised to 101–103° on further recrystallizations from aqueous ethanol or cyclohexane, $\nu_{max}^{CHCl_3}$ 3546 and 3300 (O–H stretching) and 1718 cm.$^{-1}$ (ester carbonyl).

Anal. Calcd. for $C_{15}H_{19}NO_3 \cdot 0.5H_2O$: C, 66.64; H, 7.46; N, 5.18. Found: C, 66.52, 66.55; H, 7.27, 7.39; N, 5.13, 5.24.

1,2,4a,5,6,10b-Hexahydrobenzo[*f*]quinolin-3(4H)-one (VIIa).—1-(β-Carbethoxyethyl)-2-tetralone oxime (0.4 g.) in 8 ml. of absolute ethanol was shaken under hydrogen in the presence of 0.08 g. of platinum oxide until the theoretical uptake of hydrogen had occurred. Removal of the catalyst and concentration of the filtrate gave a residue which was recrystallized from benzene giving 0.22 g. (70%) of product, m.p. 188–190°. A recrystallized analytical sample had m.p. 191–192°, $\nu_{max}^{CHCl_3}$ 3385 and 3210 (amide N–H) and 1648 cm.$^{-1}$ (amide carbonyl).

Anal. Calcd. for $C_{13}H_{15}NO$: C, 77.58; H, 7.51; N, 6.96. Found: C, 77.66; H, 7.33; N, 7.00.

4-Methyl-1,2,4a,5,6,10b-hexahydrobenzo[*f*]quinolin-3(4H)-one (VIIb). A. **From the Lactam V.**—A solution of 400 mg. (0.0019 mole) of 4-methyl-1,2,5,6-tetrahydrobenzo[*f*]quinolin-3(4H)-one[2] in 5 ml. of anhydrous ethanol and 5 ml. of anhydrous dioxane was hydrogenated over 400 mg. of 10% palladium charcoal at 35° and under atmospheric pressure. After 12 hr. the theoretical quantity of hydrogen was consumed. The catalyst was filtered and the solvent was removed by distillation *in vacuo*. The residual solid was recrystallized from ether to yield 270 mg. (68%) of VIIb as colorless crystals, m.p. 135–136°, $\nu_{max}^{CHCl_3}$ 1620 (for a 3% solution) and 1645 cm.$^{-1}$ (for a 0.23% solution, CCl_4).

Anal. Calcd. for $C_{14}H_{17}NO$: C, 78.10; H, 7.96; N, 6.51. Found: C, 77.92; H, 8.07; N, 6.33.

B. **From the Lactam VIIa.**—A solution of 0.59 g. of the lactam VIIa in 160 ml. of dry toluene was concentrated to 100 ml. by distillation, then 180 mg. of a 54% suspension of sodium hydride in oil was added. The mixture was refluxed in a nitrogen atmosphere for 2 hr. and cooled, and 9 ml. of methyl iodide was added. The mixture was refluxed for 2 hr., cooled, diluted with water, and extracted with chloroform. The organic extract was washed with water, dried, and concentrated, and the residue was recrystallized two times from ether giving 0.45 g. (72%) of material with m.p. 126–128°. Two additional recrystallizations of the product from ether gave an analytically pure sample of VIIb, m.p. 135–136°. The mixture melting point of this material with that described in part A was not depressed and the two samples had the same infrared spectra and identical behavior on thin layer and gas chromatography.

4-Methyl-1,2,5,6-tetrahydrobenzo[*f*]quinolin-3(4H)-one (V). A. **From 1-(β-Carbethoxyethyl)-2-tetralone.**—A mixture of 1 g. of the keto ester IV and 10 ml. of a 30% solution of methylamine in benzene was kept at room temperature in a sealed flask for 19 hr. Removal of the solvent and trituration of the residue with ether gave 300 mg. of colorless crystals which were recrystallized from aqueous ethanol to give 250 mg. (28%) of V, m.p. 106–107°. Chromatography of the residues on alumina using benzene as eluent afforded an additional 250 mg. of V, m.p. 106–107°. This material did not depress the melting point of the cyclization product derived from I[2] and the infrared spectra of the two samples are identical.

In a different experiment, treatment of the keto ester IV with methylamine as above, but for 84 hr., gave a total of 77% of V, m.p. 105–107°.

B. **From 2-Tetralone.**—A solution of 14.6 g. of 2-tetralone in 175 ml. of ethylene glycol was heated to boiling over a 20-min. period while passing in a stream of gaseous methylamine. When the boiling point was reached, the gas flow was stopped and 50 ml. of distillate was collected. The mixture was cooled somewhat, 8.8 ml. of methyl acrylate was added, and the resulting mixture was refluxed for 8 hr. The solution was cooled, diluted with water, and extracted with ether. The organic extract was washed with water, dried, and concentrated giving 7.1 g. of V, m.p. 103–105°, mixture melting point with authentic V not depressed. The infrared spectra of the two samples are identical. A thin layer chromatogram of the residues using silica gel and 5% methanol in chloroform as developer showed the presence of five other components as well as additional product V.

C. **From 2-Tetralone.**—A mixture of 21.9 g. of 2-tetralone and 300 ml. of toluene was refluxed under a water trap and nitrogen atmosphere while passing in gaseous methylamine for 3 hr. The gas flow was stopped and the solution was refluxed for an additional 2 hr. before collecting 75 ml. of distillate. The mixture was cooled, 13.2 ml. of methyl acrylate was added, and the mixture was refluxed for 4 hr. Removal of the solvent at 80° (0.1 mm.) gave a residue, λ_{infl}^{EtOH} 228 mμ (ε 7,100) and λ_{max}^{EtOH} 307 mμ (ε 10,400). The infrared spectrum ($CHCl_3$) of the residue showed carbonyl absorption bands at 1735, 1720, 1670, and 1640 cm.$^{-1}$ indicating a complex mixture; however, the infrared spectrum and a thin layer chromatogram (when compared with authentic V) clearly established the presence of 30–40% cyclization product V. The residue was refluxed with 200 ml. of ethylene glycol for 8 hr., and work-up of the product as in part B above gave 10.25 g. of V, m.p. 101–104°.

(10) N. P. Shusherina, R. Ya. Levina, and V. I. Zdanovich, *Zh. Obshch. Khim.*, **26**, 2847 (1956).

Some Facile Intramolecular Amide–Lactam–Lactone Interconversions[1]

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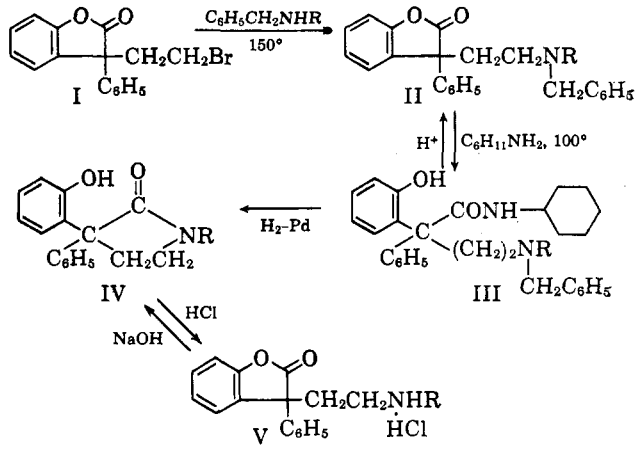
Received April 23, 1964

When 3-(β-bromoethyl)-3-phenyl-2-benzofuranone (I) was treated with N-benzylcyclohexylamine *at 150°*, the main product formed (*i.e.*, II, R = cyclohexyl, 77% yield) resulted from direct halide displacement.[2] Further treatment of the two amino lactones (II, R = *n*-butyl and cyclohexyl) with cyclohexylamine *at only 100°* effected aminolysis to the corresponding

(1) Part XI: "Neighboring Group Reactions." For Part X see ref. 9.

(2) Compare H. E. Zaugg, F. E. Chadde, and R. J. Michaels, *J. Am. Chem. Soc.*, **84**, 4567 (1962), for alternate routes of reactions of I with secondary amines under various conditions.

hydroxyamides (III).[3] Attempts to isolate them in the form of their hydrochlorides were not successful because contact with acid caused recyclization to the lactones (II).



Catalytic debenzylation of the two bases (III, R = *n*-butyl and cyclohexyl) was accompanied by spontaneous cyclization to the lactams (IV)[4]; and treatment of the latter with ethereal hydrogen chloride caused isomerization to the amino lactone hydrochlorides (V), which in their basic form again reverted spontaneously to IV.[7]

Compound IV where R = *n*-butyl has been reported to have the amino lactone structure (*i.e.*, the base corresponding to V).[10] The present work serves to correct this error. Documentation of all structural assignments is given in the Experimental section.

Experimental

3-(β-N-Benzylcyclohexylaminoethyl)-3-phenyl-2-benzofuranone Hydrochloride (II, R = Cyclohexyl).—A mixture of 23.4 g. (0.074 mole) of 3-(β-bromoethyl)-3-phenyl-2-benzofuranone (I) and 28 g. (0.148 mole) of N-benzylcyclohexylamine was heated in an oil bath at 150° overnight. The cooled solid mass was triturated with dry ether (200 ml.) and allowed to stand overnight at room temperature. Filtration gave 17.6 g. (88%) of N-benzylcyclohexylamine hydrobromide, m.p. 284–286° dec. The filtrate was treated with excess ethereal hydrogen chloride and the precipitated salt (31.7 g., m.p. 200°) was collected, dried, and suspended in water. After standing 3 hr. at room temperature, it was once more collected at the filter and dried to give 27.3 g. (77%) of product, m.p. 214–216°. Recrystallization of a sample from an ethanol–ether mixture gave the pure hydrochloride of II (R = cyclohexyl), m.p. 217–219°, $\lambda_{max}^{CHCl_3}$ 5.54 μ (lactone carbonyl).

Anal. Calcd. for $C_{29}H_{32}ClNO_2$: C, 75.39; H, 6.98; N, 3.03; Cl, 7.67. Found: C, 75.37; H, 7.17; N, 3.17; Cl, 7.68.

4-(N-Benzyl-*n*-butylamino)-2-(*o*-hydroxyphenyl)-2-phenyl-N-cyclohexylbutyramide (III, R = *n*-C_4H_9).—A mixture of 10 g. (0.023 mole) of the hydrochloride of II (R = *n*-C_4H_9)[10] and 40 ml. of cyclohexylamine was heated on the steam bath over a weekend. Precipitated cyclohexylamine hydrochloride (3.9 g.) was removed from the cooled mixture by filtration. Excess cyclohexylamine was removed from the filtrate by vacuum distillation using the steam bath as a source of heat. The residual dark glass (13 g.) was taken up in ether, washed with water to neutrality, and dried over anhydrous magnesium sulfate. Filtration and removal of the ether by distillation gave a glass (9.4 g.) which was caused to crystallize by treatment with an ether–hexane mixture. Filtration and drying gave 4.6 g. (40%), m.p. 112–114°. Recrystallization from cyclohexane yielded pure III (R = *n*-C_4H_9), m.p. 115–116°; $\lambda_{max}^{CHCl_3}$ 2.90–2.95 (NH), 3.5–4.0 (bonded OH), 6.15 (amide I), 6.64 (amide II), no lactone carbonyl absorption at 5.55 μ.

Anal. Calcd. for $C_{33}H_{42}N_2O_2$: C, 79.47; H, 8.49; N, 5.62. Found: C, 79.40; H, 8.82; N, 5.62.

Application of the foregoing procedure to the aminobenzofuranone (II, R = cyclohexyl) resulted in a 91% yield of a glassy product that could not be crystallized. However, its infrared spectrum indicated that it was mainly III (R = cyclohexyl); $\lambda_{max}^{CHCl_3}$ 2.90 (NH), 3.5–4.0 (bonded OH), 6.11 (amide I), 6.59 (amide II), no lactone carbonyl absorption at 5.55 μ.

1-Cyclohexyl-3-(*o*-hydroxyphenyl)-3-phenyl-2-pyrrolidinone (IV, R = Cyclohexyl).—A solution of 9.0 g. (0.0207 mole) of the glassy III (R = cyclohexyl) in 100 ml. of 95% ethanol was shaken at room temperature with hydrogen at 2-atm. pressure in the presence of 5% palladium–charcoal (1.8 g.). Uptake was complete in less than 2 hr. The catalyst was removed by filtration and the filtrate was concentrated to dryness. The residual glass (6.2 g.) was triturated with an ether–hexane mixture to induce crystallization. Filtration and drying gave 2.0 g., m.p. 131–133°. Recrystallization from cyclohexane yielded pure IV (R = cyclohexyl), m.p. 133–134°; $\lambda_{max}^{CHCl_3}$ 3.8 (bonded OH), 6.05 μ (amide I), no NH absorption in the near infrared.

Anal. Calcd. for $C_{22}H_{25}NO_2$: C, 78.78; H, 7.51; N, 4.18. Found: C, 79.89; H, 7.38; N, 4.20.

1-*n*-Butyl-3-(*o*-hydroxyphenyl)-3-phenyl-2-pyrrolidinone (IV, R = *n*-C_4H_9).—Application of the foregoing procedure to 1.2 g. (0.0024 mole) of III (R = *n*-C_4H_9), m.p. 115–116°, yielded 0.60 g. (81%) of IV (R = *n*-C_4H_9), m.p. 102–104°; $\lambda_{max}^{CHCl_3}$ 3.5–4.0 (bonded OH), 6.05 μ (amide I); no NH absorption in the near infrared. It was identical (mixture melting point and infrared spectrum) with the "base" previously reported[10] under code no. AP-138.

3-(β-Cyclohexylaminoethyl)-3-phenyl-2-benzofuranone Hydrochloride (V, R = Cyclohexyl).—A solution of 0.40 g. of IV (R = cyclohexyl) in dry ether was treated with an excess of ethereal hydrogen chloride. After 15 min. salt precipitated began and after 3 hr. appeared to be complete. It was collected at the filter and dried to give 0.30 g. of V (R = cyclohexyl), m.p. 170–171°; $\lambda_{max}^{CHCl_3}$ 5.56 μ (lactone carbonyl), no lactam carbonyl absorption.

Anal. Calcd. for $C_{22}H_{26}ClNO_2$: C, 71.06; H, 7.05; N, 3.77. Found: C, 71.11; H, 6.93; N, 3.79.

Treatment of this salt with aqueous alkali reconverted it to the lactam IV (R = cyclohexyl) as the only identifiable product.

Likewise, the action of excess ethereal hydrogen chloride on the lactam IV (R = *n*-butyl, 0.40 g.) produced 0.40 g. of the hydrochloride V (R = *n*-butyl), m.p. 133–135°, identical with material reported previously[10]; $\lambda_{max}^{CHCl_3}$ 5.56 μ (lactone carbonyl), no lactam carbonyl absorption.

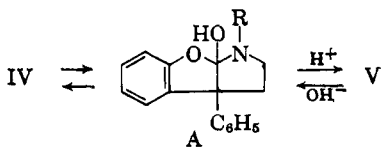
(3) G. Cramer, *Ber.*, **31**, 2813 (1898), showed that a number of 3-phenyl-2-benzofuranone derivatives readily formed the corresponding hydroxy amides on treatment with ammonia. The first two reactions of the present work show that this aminolysis reaction can be extended to primary but not to secondary amines.

(4) Either or both of two factors may be involved in this facile cleavage of an amide linkage. Possibly because of their *gem* disubstitution, α,α-diphenyl-γ-aminobutyric acid derivatives tend to produce 2-pyrrolidinones with extreme ease.[5] Also, suitably located hydroxyl groups are known to provide marked neighboring group assistance to the acid-catalyzed hydrolysis of amide linkages.[6] The phenolic hydroxyl group in III may function in this capacity.

(5) J. H. Gardner, N. R. Easton, and J. R. Stevens, *J. Am. Chem. Soc.*, **70**, 2906 (1948); R. L. Clarke, A. Mooradian, and P. Lucas, *ibid.*, **71**, 2821 (1949); D. J. Dupré, J. Elks, B. A. Hems, K. N. Speyer, and R. M. Evans, *J. Chem. Soc.*, 500 (1949); E. Walton, P. Ofner, and R. H. Thorp, *ibid.*, 648 (1949).

(6) L. Zurn, *Ann.*, **631**, 56 (1960); T. C. Bruice and F.-H. Marquardt, *J. Am. Chem. Soc.* **84**, 365 (1962).

(7) The facile interconversions IV ⇌ V are very possibly mediated by the cyclol derivatives A, which are structurally analogous to the tetrahedral intermediates recently isolated from the isomerization reactions of certain N-hydroxyacyllactams.[8] Tetrahedral intermediates related to A in which the nitrogen function (instead of oxygen) is exocyclic also have been isolated.[2,9]



(8) R. G. Griot and A. J. Frey, *Tetrahedron*, **19**, 1661 (1963); H. Ott, A. J. Frey, and A. Hofmann, *ibid.*, **19**, 1675 (1963); V. K. Antonov, A. M. Shkrob, and M. M. Shemyakin, *Tetrahedron Letters*, **No. 7**, 439 (1963).

(9) H. E. Zaugg, V. Papendick, and R. J. Michaels, *J. Am. Chem. Soc.*, **86**, 1399 (1964).

(10) A. W. Weston and W. B. Brownell, *ibid.*, **74**, 653 (1952).

Acknowledgment.—We are indebted to Mr. Wm. Washburn for the infrared spectra, Mr. E. F. Shelberg and Mr. O. Kolsto for the microanalyses, and to Mr. M. Freifelder for the catalytic hydrogenations.

The Bischler–Napieralski Cyclization of an Imide

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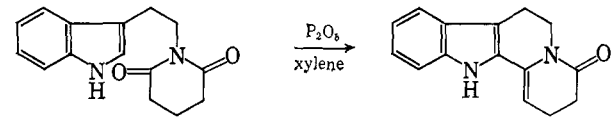
Received May 27, 1964

The cyclodehydration of amides derived from β-phenethylamines was first reported by Bischler and Napieralski[1] in 1893. Since that time many variations[2] of this reaction have been described.

In connection with one of our synthetic programs, we wished to be able to cyclize a substituted glutarimide of tryptamine. An examination of the literature pointed up the wide variety of results in examples of this type of cyclization. Wenkert, *et al.*,[3] were unable to cyclize N-(2-indol-3-ylethyl)succinimide. Haworth, *et al.*,[4] obtained a chloro intermediate from the treatment of 1,2,3,4-tetrahydro-2-piperonylmethyl-1,3-isoquinolinedione with phosphorus oxychloride. Křepelka and Štefec[5] were able to cyclize N,N′-(2,2′-biphenylylene)diphthalimide with aluminum chloride, but the product underwent cleavage to an amino acid. Kametani and Yanase[6] cyclized N-(3,4-dimethoxyphenethyl)-glutarimide with phosphorous oxychloride, but obtained a product which had suffered oxidation. Jost[7] claimed a 5–20% yield of a cyclization product from the treatment of *trans*-hexahydro-2-(2-indol-3-ylethyl)-1,3-(2H,4H)-isoquinolinedione with phosphorus oxychloride, but gave no direct experimental procedure or structure proof. Schlittler and Speitel[8] cyclized 1,2,-3,4-tetrahydro-2-(2-indol-3-ylethyl)-5-methyl-1,3-isoquinolinedione to ketoyobyrin with phosphorus oxychloride, but did not report the yield of the purified product.

In view of the lack of an example in which the experimental procedure, yield, and structure proof of the product were described, and the variation in the results of the different investigators, we decided to study a simple model for this reaction, namely, the cyclization of N-(2-indol-3-ylethyl)glutarimide (**1**). The synthesis of the required imide was accomplished by the condensation of tryptamine with diethyl glutarate to give an esteramide which was converted to the imide by refluxing in xylene with *p*-toluenesulfonic acid.

First, we attempted the cyclization of the imide **1** with phosphorus oxychloride which had been the catalyst used by most of the other workers. This gave only starting material and products which could not be characterized. However, when phosphorus pentoxide in xylene was utilized, the desired lactam **2** was obtained in 31% yield.



1 **2**

Melting point, ultraviolet absorption maxima, and infrared carbonyl absorption frequency of our material corresponds to the values previously reported for **2** by Vogt,[9] who obtained this compound by oxidation of 1,2,6,7,12,12b-hexahydroindolo[2,3-*a*]quinolizin-4-(3H)-one with N-bromosuccinimide. As a proof of structure, the lactam **2** was reduced with lithium aluminum hydride to the known 2,3,4,6,7,12-hexahydroindolo[2,3-*a*]quinolizine,[10] which was in turn reduced to 1,2,3,4,6,7,12,12b-octahydroindolo[2,3-*a*]quinolizine and shown to be identical with an authentic sample prepared according to Reckhow and Tarbell.[11]

Experimental[12]

Ethyl N-(2-Indol-3-lyethyl)glutaramate.—A mixture of 20.0 g. of tryptamine and 26.0 g. of diethyl glutarate was heated at 175° for 18 hr. The reaction mixture was digested with 2.5 l. of chloroform, cooled to room temperature, and filtered. Removal of the solvent and crystallization of the residue from benzene gave a solid which on recrystallization from methylene chloride–petroleum ether (b.p. 30–60°) gave 32.8 g. (47%) of a crystalline solid, m.p. 101–102°.

Anal. Calcd. for $C_{17}H_{22}N_2O_3$: C, 67.52; H, 7.33; N, 9.27. Found: C, 67.67; H, 7.54; N, 9.47.

N-(2-Indol-3-ylethyl)glutarimide (1).—A mixture of 31.7 g. of ethyl N-(2-indol-3-ylethyl)glutaramate, 9.6 g. of *p*-toluenesulfonic acid and 1200 ml. of xylene was refluxed for 9 hr. while the water was continuously removed in a Dean–Stark tube. The reaction mixture was filtered and diluted with 1800 ml. of petroleum ether. On standing, there was deposited 15.0 g. (53%) of a crystalline solid, m.p. 170–172°. Recrystallization from methanol gave an analytical sample, m.p. 173–175°.

Anal. Calcd. for $C_{15}H_{16}N_2O_2$: C, 70.29; H, 6.29; N, 10.93. Found: C, 70.01; H, 6.29; N, 10.95.

2,3,4,6,7,12-Hexahydroindolo[2,3-*a*]quinolizin-4-one (2).—To a solution of 3.0 g. of N-(2-indol-3-ylethyl)glutarimide in 410 ml. of refluxing xylene was added three 15-g. portions of phosphorus pentoxide with stirring over a 45-min. interval. Then the mixture was refluxed for 5 hr. The reaction mixture was filtered and the solid was added to 1 l. of ice–water, made basic with 40% potassium hydroxide solution, and extracted with chloroform. The chloroform layer was washed with water and dried over sodium sulfate, and the solvent was removed. One recrystallization from ethanol gave 0.94 g. (31%) of a crystalline solid, m.p. 234–235°. Further recrystallization gave an analytical sample, m.p. 237–238° (lit.[9] m.p. 232–233°); $\nu_{max}^{CHCl_3}$, cm.$^{-1}$, 3460 (NH), 1645 (C=O), 1665 (C=O) [lit.[9] 3450 (NH), 1640 (C=O), 1600 (C=O)]; λ_{max}^{EtOH}, mμ (ε), 220 sh (27,000), 232 (30,000), 308 (22,200) 319 (20,500) [lit.[9] λ_{max}^{MeOH}, mμ (ε), 222 (27,100), 308 (18,700), 318 (16,000)]; $\lambda_{max}^{0.1\ N\ HCl}$, mμ (ε), 245 (8400), 395 (33,900).

Anal. Calcd. for $C_{15}H_{14}N_2O$: C, 75.60; H, 5.92; N, 11.76. Found: C, 75.71; H, 5.90; N, 11.50.

1,2,3,4,6,7-Hexahydro-12H-indolo[2,3-*a*]quinolizinium Perchlorate.—To a suspension of 4.8 g. of lithium aluminum hydride in 750 ml. of ether was added, over a 1-hr. interval, a solution of 1.5 g. of 2,3,4,6,7,12-hexahydroindolo[2,3-*a*]quinolizin-4-one in

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(2) W. Whaley and T. Govindachari, *Org. Reactions*, **6**, 74 (1951).
(3) E. Wenkert, S. Garratt, and K. Dave, *Can. J. Chem.*, **42**, 489 (1964).
(4) R. Haworth, W. Perkin, and H. Pink, *J. Chem. Soc.*, **127**, 1709 (1925).
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(7) J. Jost, *Helv. Chim. Acta*, **32**, 1297 (1949).
(8) E. Schlittler and R. Speitel, *ibid.*, **31**, 1199 (1948).
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(11) W. A. Reckhow and D. S. Tarbell, *ibid.*, **74**, 4960 (1952).
(12) Melting points are corrected. The authors are indebted to Mr. R. Puchalski for the spectral data and Mrs. U. Zeek for analytical determinations.