310. 4,5,6,7-*Tetrahydroisatin and Related Compounds. Part II.*[1] *Tetrahydroisatins from 2-Oxocyclohexylglyoxylic Acid Lactones*

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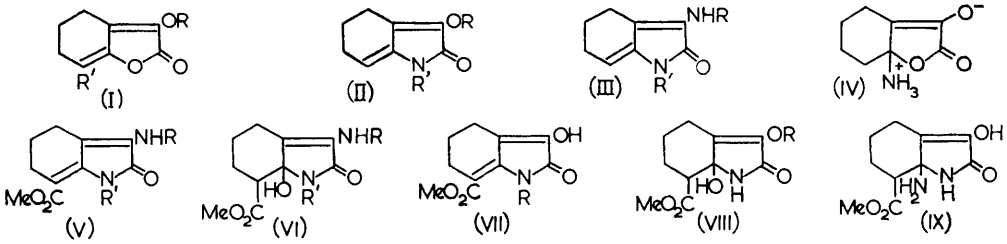
The two γ-lactones, 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran (I; R = H, R′ = CO_2Me) and 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran (I; R = Ac, R′ = H), have been converted, by the action of ammonia or amines, into derivatives of 4,5,6,7-tetrahydroisatin (II; R = R′ = H) and of 3-amino-2,4,5,6-tetrahydro-2-oxoindole (III; R = R′ = H). In several reactions, intermediate compounds have been isolated and the structures of these are discussed.

Claisen condensation of methyl 2-oxocyclohexanecarboxylate and methyl oxalate gives directly the γ-lactone, 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran (I; R = H, R′ = CO_2Me),[2] whilst 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran (I; R = Ac, R′ = H)[3] is readily prepared from 2-oxocyclohexylglyoxylic acid. In continuation of studies on the preparation of tetrahydroisatins,[1] we have investigated the reactions of these lactones with ammonia and with amines.

3-Acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran reacted smoothly with aniline to give 4,5,6,7-tetrahydro-1-phenylisatin 3-anil (3-anilino-2,4,5,6-tetrahydro-2-oxo-1-phenylindole). Reaction of the lactone with ammonia, in refluxing ethanol, gave both 4,5,6,7-tetrahydroisatin (II; R = R′ = H) and the related amino-compound, 3-amino-2,4,5,6-tetrahydro-2-oxoindole (III; R = R′ = H), but with ammonia at 0°, the lactone was converted into an unstable crystalline product, $C_8H_{11}NO_3$ (" compound A "), the structure of which has not been definitely established. This sparingly soluble product has a complex infrared absorption spectrum and is thermally unstable, giving 4,5,6,7-tetrahydroisatin when

[1] Part I, R. J. S. Beer and J. Hollowood, *J.*, 1964, 991.
[2] W. E. Bachmann, G. I. Fugimoto, and L. B. Wick, *J. Amer. Chem. Soc.*, 1950, **72**, 1995.
[3] P. A. Plattner and L. M. Jampolsky, *Helv. Chim. Acta*, 1943, **26**, 687.

heated alone or with solvents; with acetic anhydride and pyridine, it regenerated 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran. Treatment of " compound A " with dilute aqueous acid produced an unstable nitrogen-free compound which was not purified but which had the spectroscopic and chemical properties expected of 3-hydroxy-2,4,5,6-tetrahydro-2-oxobenzofuran; its ultraviolet spectrum showed a maximum at 286 mμ compared with the



maximum at 277 mμ exhibited by 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran, and the presence of a hydroxyl group was indicated by a broad peak at 3220 cm.$^{-1}$ in the infrared spectrum; the compound gave an intense ferric reaction, reacted with ammonia to give " compound A " in almost quantitative yield, and, on acetylation, produced 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran. Of the various structures considered for " compound A," the most probable seems to be 8-amino-2,4,5,6,7,8-hexahydro-3-hydroxy-2-oxobenzofuran as the zwitterion (IV), formed by addition of ammonia to 2,3,4,5,6,7-hexahydro-2,3-dioxobenzofuran.

1-Ethyl-4,5,6,7-tetrahydroisatin (II; R = H, R′ = Et), was obtained by the action of ethylamine on 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran; like tetrahydroisatin,[1] it reacted with nitrous acid to give an intensely coloured 7-hydroxyimino-derivative.

Two products were isolated from the reaction of 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran with aniline in refluxing ethanol, the expected 3-anilino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxo-1-phenylindole (V; R = R′ = Ph) and a hydrated derivative, probably (VI; R = R′ = Ph), which readily gave the foregoing 3-anilino-compound on treatment with warm methanolic hydrochloric acid. 3-Anilino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxo-1-phenylindole resembled the tetrahydro-1-phenylisatin 3-anil prepared by Horwitz [4] in its hydrolysis to the corresponding tetrahydroisatin (VII; R = Ph), which formed normal derivatives.

The reaction of ammonia with 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran proved to be unexpectedly complicated and markedly temperature-dependent. The product obtained at low temperatures is formulated as 2,4,5,6,7,8-hexahydro-3,8-dihydroxy-7-methoxycarbonyl-2-oxoindole (VIII; R = H) since treatment with cold aqueous acid converted it into 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxoindole (VII; R = H). The structure of the latter compound is indicated by its spectroscopic and other properties and by the formation of derivatives analogous to those prepared from 4,5,6,7-tetrahydroisatin.[1] The 7-methoxycarbonyltetrahydroisatin (VII; R = H) differs from 4,5,6,7-tetrahydroisatin in reacting with ammonia to give an adduct, $C_{10}H_{14}N_2O_4$. This product probably has structure (IX); it reverts to the tetrahydroisatin either on being heated or on treatment with aqueous acid.

The dihydroxy-compound (VIII; R = H) yielded a monoacetate which did not give an immediate ferric reaction and is thus the enol-acetate (VIII; R = Ac), a conclusion supported by the infrared data; the enol acetate was rapidly decomposed by cold aqueous acid, giving 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxoindole. In alkaline solution, the dihydroxy-compound (VIII; R = H) was slowly converted into 4,5,6,7-tetrahydroisatin (II; R = R′ = H), loss of the methoxycarbonyl group accompanying dehydration.

[4] L. Horwitz, *J. Amer. Chem. Soc.*, **1953**, **75**, 4060.

Three closely related products were isolated from the reaction of 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran with ammonia in hot ethanol. One of these is clearly 3-amino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole (V R = R′ = H); the same product was obtained in excellent yield by a Bucherer reaction [1] with 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxoindole. The other two products (referred to as compounds B and C in the Experimental section) are isomeric hydrated derivatives of the 3-amino-compound (V; R = R′ = H), which is readily formed from them by the action of acids. Compounds B and C differed in their behaviour on acetylation, C giving directly 3-acetamido-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole (V; R = Ac, R′ = H), whilst B gave a normal acetyl-derivative, which was dehydrated by subsequent treatment with aqueous acids to (V; R = Ac, R′ = H). These results suggest that B and C are stereoisomers of structure (VI; R = R′ = H), C having a hydrogen atom at the 7-position *trans* to the hydroxyl group at position 8.

Experimental

Reactions of 3-Acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran.—3-Acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran [3] (2·0 g.) and aniline (5·0 g.), heated under reflux in methanol (20 ml.) for 2 hr., gave 4,5,6,7-tetrahydro-1-phenylisatin 3-anil which crystallised from methanol in yellow prisms (2·1 g.), m. p. 194°, identical with a sample prepared by Horwitz's [4] method.

When ammonia was passed into a solution of 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran (4·0 g.) in ice-cold ethanol (100 ml.), a pale yellow crystalline solid separated. After being washed with ethanol and then with ether, this *product* (" compound A ") (3·2 g.), had m. p. 98—100° (decomp.) (Found: C, 56·8; H, 6·6; N, 8·0. $C_8H_{11}NO_3$ requires C, 56·8; H, 6·55; N, 8·3%). In a similar reaction in refluxing ethanol, the acetoxy-lactone (2·0 g.) gave an orange solid (after evaporation of solvent) which was triturated with 2N-aqueous hydrochloric acid. The non-basic residue, crystallised from acetone, gave 4,5,6,7-tetrahydroisatin as prisms (0·38 g.), m. p. 205°; addition of alkali to the acid solution precipitated 3-amino-2,4,5,6-tetrahydro-2-oxoindole, which formed yellow prisms (0·55 g.), m. p. 154—155°, from benzene. These products were identical with samples previously prepared.[1]

The acetoxy-lactone (2·5 g.) was heated in methanol (25 ml.) with ethylamine (5 ml. of 33% solution in methanol) for 1 hr. Removal of methanol left a brown oil which was solidified by treatment with 2N-aqueous hydrochloric acid. Recrystallisation from aqueous ethanol gave 1-*ethyl*-4,5,6,7-*tetrahydroisatin* as prisms (1·0 g.), m. p. 128° (Found: C, 67·1; H, 7·55; N, 7·7. $C_{10}H_{13}NO_2$ requires C, 67·0; H, 7·3; N, 7·8%). This product (0·75 g.), suspended in ice-cold dilute hydrochloric acid (10 ml.), reacted with sodium nitrite (0·29 g.) to give 1-*ethyl*-4,5,6,7-*tetrahydro*-7-*hydroxyiminoisatin*, which crystallised from benzene in very dark red prisms (0·45 g.), m. p. 196—197° (sublimes) (Found: C, 57·7; H, 5·6; N, 13·9. $C_{10}H_{12}N_2O_3$ requires C, 57·7; H, 5·8; N, 13·6%).

Reactions of " Compound A."—Attempts to recrystallise this product failed; in hot solvents it was converted smoothly into 4,5,6,7-tetrahydroisatin. This intermediate (1·0 g.) reacted with acetic anhydride (2 ml.) and pyridine (5 ml.) at room temperature to give 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran (0·78 g.), m. p. 86—87°, identical with an authentic specimen.

Compound A (10·0 g.) was decomposed by treatment with dilute hydrochloric acid; the resulting golden yellow oil (2,4,5,6-tetrahydro-3-hydroxy-2-oxobenzofuran?) (8·9 g.), which could not be distilled without decomposition, gave an intense black ferric reaction, afforded 3-acetoxy-2,4,5,6-tetrahydro-2-oxobenzofuran (25% yield) with acetic anhydride and pyridine, and reverted to " compound A " with ammonia in ice-cold ethanol. The yellow oil (2·0 g.) gave 2-oxocyclohexylglyoxylic acid (0·82 g.), m. p. 124—126°, when heated on the steam-bath with dilute hydrochloric acid for 15 min.

3-*Anilino*-2,4,5,6-*tetrahydro*-7-*methoxycarbonyl*-2-*oxo*-1-*phenylindole.*—The crystalline product obtained by heating 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran [2] (6·0 g.) with aniline (8·0 g.) in ethanol (60 ml.) for 3 hr. was recrystallised from methanol, giving 3-*anilino*-2,4,5,6-*tetrahydro*-7-*methoxycarbonyl*-2-*oxo*-1-*phenylindole* as pale yellow prisms (1·5 g.), m. p. 245—246° (Found: C, 73·05; H, 5·6; N, 7·85; OMe, 9·2. $C_{22}H_{20}N_2O_3$ requires C, 73·3; H, 5·6; N, 7·8; OMe, 8·7). The concentrated mother-liquor, kept at 0° overnight, yielded a

second *product*, which crystallised from benzene in prisms (4·0 g.), m. p. 154—155° (Found: C, 72·0; H, 6·1; N, 6·6%). This product was converted in high yield, by the action of warm dilute methanolic hydrochloric acid, into 3-anilino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxo-1-phenylindole.

2,4,5,6-*Tetrahydro*-3-*hydroxy*-7-*methoxycarbonyl*-2-*oxo*-1-*phenylindole*.—3-Anilino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxo-1-phenylindole (1·5 g.) was heated with concentrated hydrochloric acid (6 ml.) in propan-2-ol (15 ml.) for 2 hr. After removal of solvent *in vacuo*, addition of water (5 ml.) precipitated 2,4,5,6-*tetrahydro*-3-*hydroxy*-7-*methoxycarbonyl*-2-*oxo*-1-*phenylindole*, which crystallised from aqueous ethanol in pale yellow needles (0·8 g.), m. p. 178—179° (sublimes) (Found: C, 67·25; H, 5·4; OMe, 10·8. $C_{16}H_{15}NO_4$ requires C, 67·3; H, 5·3; OMe, 10·9%); $\lambda_{max.}$ 300 mμ (ε 7480); $\nu_{max.}$ 3250 (OH), 1720 (ester C=O), 1690 cm.$^{-1}$ (lactam C=O); *quinoxaline derivative*, pale yellow needles, m. p. 210—212° (sublimes) (Found: C, 73·6; H, 5·2; N, 11·7; OMe, 8·7. $C_{22}H_{19}N_3O_2$ requires C, 73·9; H, 5·4; N, 11·8; OMe, 8·7%); 3-*acetoxy*-2,4,5,6-*tetrahydro*-7-*methoxycarbonyl*-2-*oxo*-1-*phenylindole* formed prisms, m. p. 152—153° (Found: C, 66·15; H, 5·3; N, 4·45. $C_{18}H_{17}NO_5$ requires C, 66·05; H, 5·2; N, 4·3%); $\lambda_{max.}$ 288 mμ (ε 10,500); $\nu_{max.}$ 1780 cm.$^{-1}$ (enol acetate C=O).

When heated under reflux with aniline (0·4 g.) in n-butanol (30 ml.) containing a trace of aniline hydrochloride, 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxo-1-phenylindole (0·5 g.) reverted to the 3-anilino-compound (0·42 g.), m. p. and mixed m. p. 246°.

2,4,5,6,7,8-*Hexahydro*-3,8-*dihydroxy*-7-*methoxycarbonyl*-2-*oxoindole*.—Passage of ammonia for 1 hr. into an ice-cold solution of 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran (1·0 g.) in dry ethanol (25 ml.) gave 2,4,5,6,7,8-*hexahydro*-3,8-*dihydroxy*-7-*methoxycarbonyl*-2-*oxoindole* which crystallised from methanol in prisms (0·65 g.), m. p. 149—150° (decomp.) (Found: C, 52·85; H, 5·75; N, 6·5. $C_{10}H_{13}NO_5$ requires C, 52·9; H, 5·8; N, 6·2%); $\lambda_{max.}$ 251 mμ (ε 3300); $\nu_{max.}$ 3240 (OH), 3450 (NH), 1720 (ester C=O), and 1690 cm.$^{-1}$ (lactam C=O). This product (1·0 g.), warmed on the steam-bath for 1 hr. with acetic anhydride (2 ml.) and pyridine (10 ml.), gave 3-*acetoxy*-2,4,5,6,7,8-*hexahydro*-8-*hydroxy*-7-*methoxycarbonyl*-2-*oxoindole* forming prisms (0·5 g.), m. p. 163—165° (from methanol) (Found: C, 53·6; H, 5·7; N, 5·25; OMe, 11·65. $C_{12}H_{15}NO_6$ requires C, 53·5; H, 5·6; N, 5·2; OMe, 11·5%); $\nu_{max.}$ 3225 (OH), 3430 (NH), and 1775 cm.$^{-1}$ (enol acetate C=O).

A solution of the hexahydro-3,8-dihydroxyindole (0·5 g.) in N-aqueous sodium hydroxide (10 ml.) was kept at room temperature for 16 hr. and then acidified, giving 4,5,6,7-tetrahydroisatin (0·18 g.), m. p. 204°.

2,4,5,6-*Tetrahydro*-3-*hydroxy*-7-*methoxycarbonyl*-2-*oxoindole*.—When the foregoing hexahydro-3,8-dihydroxyindole (5·9 g.) was stirred with 2N-aqueous hydrochloric acid (30 ml.) and water (20 ml.), it slowly dissolved and was replaced by a pale yellow solid. Crystallisation from aqueous methanol gave 2,4,5,6-*tetrahydro*-3-*hydroxy*-7-*methoxycarbonyl*-2-*oxoindole* as yellow prisms (4·2 g.), m. p. 202—203° (Found: C, 57·6; H, 5·2; N, 7·1; O, 31·0. $C_{10}H_{11}NO_4$ requires C, 57·4; H, 5·3; N, 6·7; O, 30·6%); $\lambda_{max.}$ 312 mμ (ε 24,800); $\nu_{max.}$ 3360, 3230 (NH, OH), 1715 (ester C=O), and 1680 cm.$^{-1}$ (lactam C=O). The compound gave an intense green ferric reaction.

The same product (0·11 g.) was obtained by the action of cold dilute hydrochloric acid on 3-acetoxy-2,4,5,6,7,8-hexahydro-8-hydroxy-7-methoxycarbonyl-2-oxoindole (0·2 g.).

Derivatives of 2,4,5,6-*Tetrahydro*-3-*hydroxy*-7-*methoxycarbonyl*-2-*oxoindole*.—The following derivatives were prepared by methods analogous to those used [1] in the tetrahydroisatin series: 2,4,5,6-*Tetrahydro*-3-*methoxy*-7-*methoxycarbonyl*-2-*oxoindole*, prisms, m. p. 172—174° (sublimes), from methanol (Found: C, 59·2; H, 5·7; N, 6·45. $C_{11}H_{13}NO_4$ requires C, 59·2; H, 5·8; N, 6·3%), $\lambda_{max.}$ 312 mμ (ε 25,300); *quinoxaline derivative*, pale yellow prisms, m. p. 204—205° (from methanol) (Found: C, 67·8; H, 5·2; N, 14·5. $C_{16}H_{15}N_3O_2$ requires C, 68·3; H, 5·4; N, 14·9%), $\nu_{max.}$ 3340 (NH) and 1710 cm.$^{-1}$ (ester C=O). 3-*Acetoxy*-2,4,5,6-*tetrahydro*-7-*methoxycarbonyl*-2-*oxoindole*, needles, m. p. 144—145° (from methanol) (Found: C, 57·2; H, 5·3; N, 5·7; OMe, 12·6. $C_{12}H_{13}NO_5$ requires C, 57·4; H, 5·2; N, 5·6; OMe, 12·3%), $\lambda_{max.}$ 300 mμ (ε 18,030).

Ammonia was passed for 1 hr. into a solution of 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxoindole (1·2 g.) in refluxing ethanol (35 ml.). Crystallisation of the product from methanol gave 8-*amino*-2,4,5,6,7,8-*hexahydro*-3-*hydroxy*-7-*methoxycarbonyl*-2-*oxoindole* as yellow needles (0·8 g.), m. p. 140—150° (decomp.) (Found: C, 53·4; H, 6·35; N, 12·0. $C_{10}H_{14}N_2O_4$ requires C, 53·1; H, 6·2; N, 12·4%). This product reacted immediately with dilute hydrochloric acid, giving 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxoindole, m. p. 203°.

3-*Amino*-2,4,5,6-*tetrahydro*-7-*methoxycarbonyl*-2-*oxoindole*.—Ammonia was passed for 1 hr. into a solution of 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxobenzofuran (1·0 g.) in refluxing ethanol (30 ml.). " *Compound B* " separated from the cooled solution and, after recrystallisation from methanol, formed prisms (0·32 g.), m. p. 188—189° (sublimes) (Found: C, 53·5; H, 6·2; N, 12·3. $C_{10}H_{14}N_2O_4$ requires C, 53·1; H, 6·2; N, 12·4%); $\lambda_{max.}$ 273 mμ (ε 3800); $\nu_{max.}$ 3410, 3300, 3230, 1715 (ester C=O), and 1690 cm.$^{-1}$ (lactam C=O). The filtered reaction mixture was evaporated to dryness and the yellow residue extracted with boiling benzene. The benzene-insoluble residue was crystallised from ethyl acetate, giving " *compound C* " as plates (0·23 g.), m. p. 100—140° (with decomposition to 3-amino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole—see below) (Found: C, 52·8; H, 6·1; N, 12·3. $C_{10}H_{14}N_2O_4$ requires C, 53·1; H, 6·2; N, 12·4%); $\lambda_{max.}$ 272 mμ (ε 2800); $\nu_{max.}$ 3330, 3270, 3190, 1730 (ester C=O), and 1700 cm.$^{-1}$ (lactam C=O). The benzene extract was concentrated and chromatographed on neutral alumina; elution with benzene–ethyl acetate (50 : 50) gave 3-*amino*-2,4,5,6-*tetrahydro*-7-*methoxycarbonyl*-2-*oxoindole* (0·15 g.) as yellow prisms, m. p. 146—147° (Found: C, 58·0; H, 6·0; N, 13·3. $C_{10}H_{12}N_2O_3$ requires C, 57·7; H, 5·8; N, 13·5%); $\lambda_{max.}$ 318 mμ (ε 11,400); $\nu_{max.}$ 3330, 3410, 1710 (ester C=O), and 1680 cm.$^{-1}$ (lactam C=O).

Both " compound B " and " compound C " were rapidly converted in high yield into 3-amino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole by the action of cold aqueous 2N-hydrochloric acid.

When heated on the steam-bath for 1 hr. with acetic anhydride (1 ml.) and pyridine (3 ml.), 3-amino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole (0·15 g.) gave the 3-*acetamido-derivative* which formed needles (0·10 g.), m. p. 189—190° (sublimes), from methanol (Found: C, 57·9; H, 5·6; N, 10·9. $C_{12}H_{14}N_2O_4$ requires C, 57 6; H, 5 6; N, 11·2%), $\lambda_{max.}$ 319 mμ (ε 8050).

The solution obtained by heating 3-amino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole (0·26 g.) with propan-2-ol (10 ml.) and concentrated hydrochloric acid (2 ml.) for 1½ hr. was concentrated to small volume, and left overnight. Crystallisation of the product from aqueous methanol gave 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxoindole (0·16 g.), m. p. 204°.

Acetylation of " Compound B " and " Compound C."—With warm acetic anhydride–pyridine, " compound B " (0·25 g.) yielded a *mono acetyl derivative*, prisms (0·14 g.), m. p. 202—203°, from ethyl acetate (Found: C, 53·5; H, 6·1; N, 10·5. $C_{12}H_{16}N_2O_5$ requires C, 53·7; H, 6·0; N, 10·4%), converted in 60% yield by the action of cold aqueous hydrochloric acid into 3-acetamido-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole, m. p. 189—190°.

Acetylation of " compound C " under similar conditions gave directly 3-acetamido-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole.

Conversion of 2,4,5,6-*Tetrahydro*-3-*hydroxy*-7-*methoxycarbonyl*-2-*oxoindole into* 3-*Amino*-2,4,5,6-*tetrahydro*-7-*methoxycarbonyl*-2-*oxoindole*.—Sulphur dioxide was passed into concentrated aqueous ammonia (*d* 0·88; 20 ml.) at 0° until the increase in weight was 5·2 g., a little water was added to dissolve the precipitate, and the resulting solution was heated with 2,4,5,6-tetrahydro-3-hydroxy-7-methoxycarbonyl-2-oxoindole (1·0 g.) in a sealed tube at 120° for 24 hr. Crystallisation of the solid from benzene gave 3-amino-2,4,5,6-tetrahydro-7-methoxycarbonyl-2-oxoindole (0·78 g.), m. p. 147°.

Infrared spectra are reported for Nujol mulls. Ultraviolet spectra were determined in ethanol.

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