The glassy material showed an ultraviolet absorption maximum at 206 mμ (ε 6800).

(±)-*Retronecine* (III). A solution of 2.0 g. of the ester XIV in 50 ml. of anhydrous tetrahydrofuran (THF) was dropped slowly into a solution of 1.0 g. of lithium aluminum hydride in 100 ml. of THF. When the addition was complete, the solution was refluxed overnight and the excess reagent decomposed by the addition of 10 ml. of ethyl acetate and 20 ml. of water. The mixture was filtered through Celite and the filtrate evaporated to dryness. The residue was dried by the addition of benzene and distillation to dryness.

The dried residue was transferred to a Soxhlet thimble and extracted with chloroform. Removal of the chloroform left a brown oil which, when dissolved in a mixture of ethanol and acetone and cooled overnight, yielded 0.8 g. and 0.4 g. (second crop) of brownish crystals. The crude material was recrystallized twice from acetone, affording 0.95 g. of (±)-retronecine, m.p. 130–131°. The compound could also be purified by sublimation at 80°/0.01 mm.

The infrared spectrum of the (±)-base was similar, but not identical to that of natural (+)-retronecine when taken taken in KBr discs, but the infrared spectra in chloroform solution were identical.

Anal. Calcd. for $C_8H_{13}O_2N$: C, 61.91; H, 8.44; N, 9.03. Found: C, 62.00; H, 8.35; N, 9.21.

(+)-*Retronecine* (III). A solution of 174 mg. of (±) retronecine and 220 mg. of *d*-camphoric acid in 1.5 ml. of ethanol was warmed and diluted dropwise with ethyl acetate until it became cloudy. The solution was cleared with a few drops of ethanol and allowed to cool. The 210 mg. of crude crystalline product was recrystallized five times from ethanol–ethyl acetate, after which 46 mg. of material was obtained, m.p. 146–148°, $[\alpha]_D^{27}$ 35.5 (*c* 2, EtOH). Further recrystallization finally afforded 21 mg. with m.p. 151–152°, $[\alpha]_D^{27}$ 35.8° (*c* 1.37, EtOH). Natural (+)-retronecine gave a *d*-camphorate, m.p. 151–152°, $[\alpha]_D^{27}$ 35.4° (*c* 1.61, EtOH). A mixture of the natural and synthetic salts melted at 151–152°.

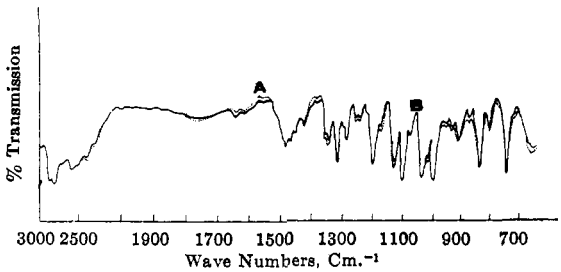


Fig. 1. Infrared absorption spectra of A) natural; B) synthetic (+)-retronecine. In KBr

The synthetic *d*-camphorate (13.7 mg.) was neutralized with aqueous sodium carbonate. A small amount of Celite was added and the slurry was dried on a steam bath in a current of air. The dry residue was extracted (Soxhlet) with chloroform for 24 hours. Removal of the chloroform left crude (+)-retronecine as a thick paste; this was dried in a vacuum desiccator, sublimed at 70°/0.1 mm., and the sublimate recrystallized from acetone. The 4.6 mg. of (+) retronecine that was obtained had m.p. 119–120° and did not depress the m.p. of natural (+)-retronecine (m.p. 120–121°). The infrared absorption spectra of the natural and synthetic materials (in KBr) were identical in every detail (Fig. 1).

In another preparation the (+)-retronecine had m.p. 119–20° and $[\alpha]_D^{26}$ +4.95° (*c* 0.58, ethanol); a sample of natural (+)-retronecine had $[\alpha]_D^{26}$ +50.2°.

Acknowledgment. The authors gratefully acknowledge the support of U.S. Public Health Service research grant RG-6457.

LOS ANGELES, CALIF.

[CONTRIBUTION FROM BATTELLE MEMORIAL INSTITUTE]

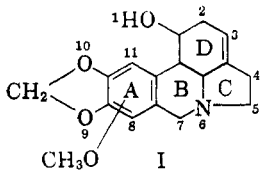
Contribution to the Structure of Falcatine. Synthesis of Isoanhydrofalcatine Lactam

F. BENINGTON AND R. D. MORIN

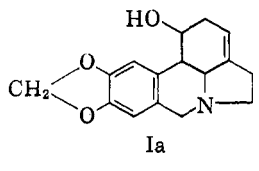
Received November 4, 1960

Previous investigations by others have shown that the methoxyl group in falcatine (I) is located at either C-8 or C-11. Oxidation of I gives anhydrofalcatine lactam which may possess either structure Ic or Id. An unambiguous synthesis of isoanhydrofalcatine lactam (Id), which has properties differing from anhydrofalcatine lactam, shows that the latter compound possesses structure Id and, accordingly, the methoxyl group in falcatine is located at C-11.

Fales and Wildman[1] have shown that the amaryllidacae alkaloid falcatine (I) undergoes ar-demethoxylation with sodium and *n*-amyl alcohol to produce caranine, whose structure has been established[2] as Ia. Thus, the only remaining problem concerning the structure of falcatine was whether the methoxyl group occupies the 8- or the 11-position in the A-ring. Oxidation of I with selenium dioxide followed by treatment with nitric acid afforded the intermediate quaternary salt, anhydrofalcatinium nitrate (Ib), which on further oxidation with potassium ferricyanide gave anhydrofalcatine lactam[1] (Ic or Id). Fales and Wildman have suggested that the synthesis of either 8- or 11-methoxy-4,5-



I

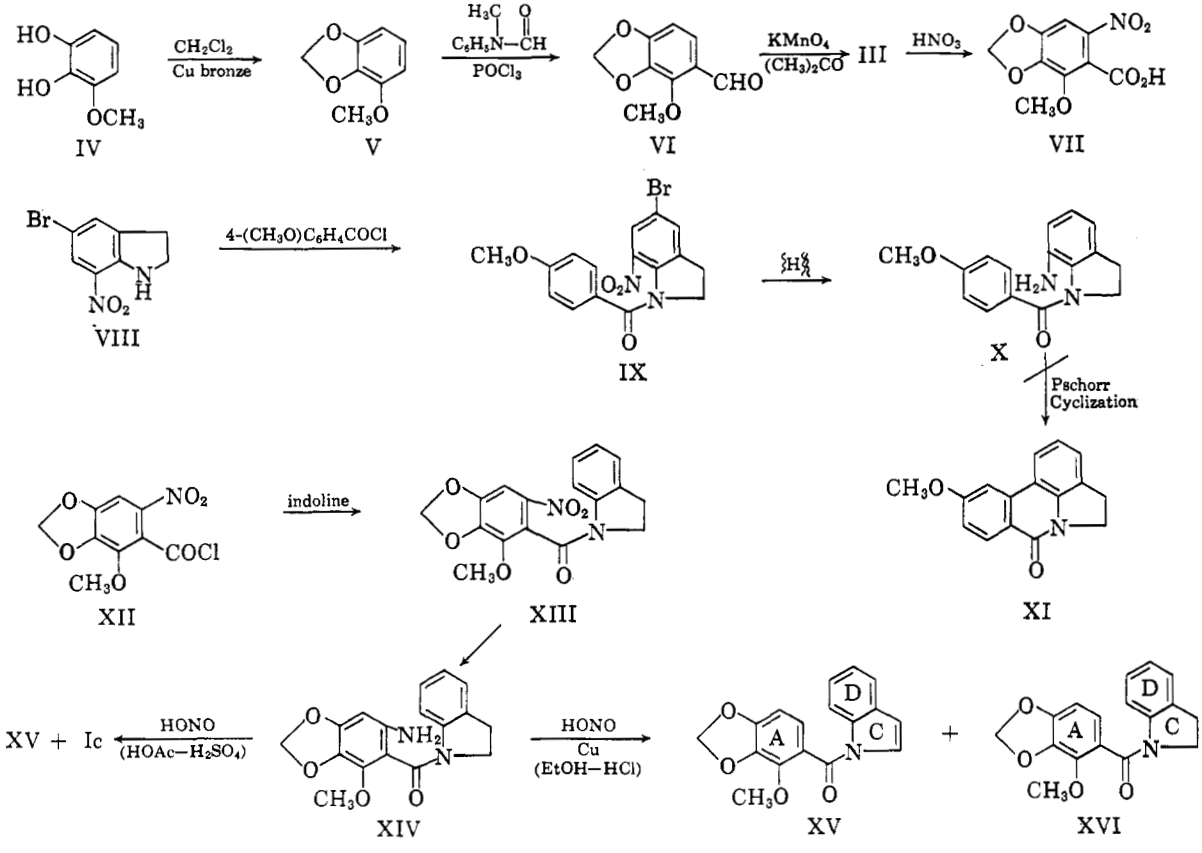


Ia

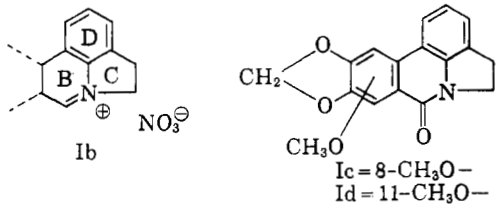
(1) H. M. Fales and W. C. Wildman, *J. Am. Chem. Soc.*, **80**, 4395 (1958).

(2) E. W. Warnhoff and W. C. Wildman, *J. Am. Chem. Soc.*, **79**, 2192 (1957).

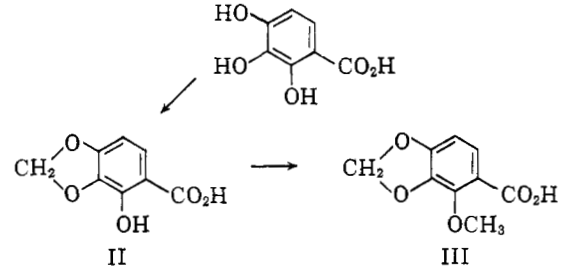
CHART I



dihydro-9,10-methylenedioxy-7-oxo-7H-dibenzo-[f,h,i]pyrrocoline (Ic or Id) would establish unequivocally the position of the methoxyl group in I.



We chose to undertake the synthesis of Ic because of the rather obvious difficulties associated with obtaining the desired orientation in the A-ring intermediate to be used for the synthesis of structure Id. The key intermediate in our approach to Ic was 2-methoxy-3,4-methylenedioxybenzoic acid (croweacic acid) (III). In our initial studies, we obtained this compound in exceedingly poor overall yield from pyrogallol via 2,3,4-trihydroxybenzoic acid,[3] which was converted to 2-hydroxy-3,4-methylenedioxybenzoic acid (II) by the action of methylene sulfate in acetone as a reaction medium.[4] Subsequent treatment of II with methyl sulfate and alkali afforded III.[5] A superior preparative route to



III started with the readily available *o*-vanillin in accordance with the scheme shown in Chart I. A Dakin oxidation afforded 3-methoxycatechol (IV),[6] which was converted to 3-methoxymethylenedioxybenzene (V) by the action of methylene chloride in methanolic potassium hydroxide solution in the presence of Tobin Bronze in a stirring autoclave at 100–110°.[7] Formylation of V with *N*-methylformanilide and phosphorus oxychloride gave chiefly 2-methoxy-3,4-methylenedioxybenzaldehyde (VI) (croweacin aldehyde) as reported by Brownell and Weston[8] rather than the mixture

(3) S. Kostanecki, *Ber.*, **18**, 3202 (1885).

(4) W. Baker and R. I. Savage, *J. Chem. Soc.*, 1602 (1938).

(5) W. Baker, A. R. Penfold, and J. L. Simonsen, *J. Chem. Soc.*, 439 (1939).

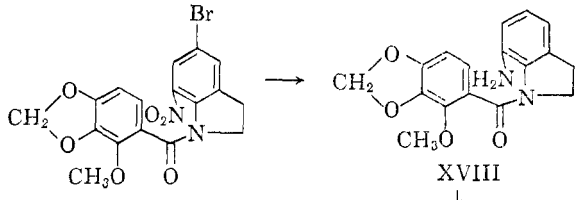
(6) A. R. Surrey, *Org. Syntheses*, **Coll. Vol. III**, 759 (1955).

(7) K. N. Campbell, P. F. Hopper, and B. K. Campbell, *J. Org. Chem.*, **16**, 1736 (1951).

(8) W. B. Brownell and A. W. Weston, *J. Am. Chem. Soc.*, **73**, 4971 (1951).

of isomers which Folkers *et al.* obtained.[9] Oxidation of VI with potassium permanganate in an aqueous acetone medium afforded III in 81% yield, and the over-all yield of III from *o*-vanillin was about 13%, which represented a distinct improvement over the route of Baker from pyrogallol.[4,5]

It was initially planned to treat the acid chloride of III with 5-bromo-7-nitroindoline (VIII), obtained by bromination and then nitration of 1-acetylindoline in accordance with the details reported by Boekelheide *et al.*[10] to obtain 1-(2-methoxy-3,4-methylenedioxybenzoyl)-5-bromo-7-nitroindoline (XVII). Hydrogenation of XVII in the presence of palladium-on-charcoal would be expected to give the dehalogenated 7-amino compound XVIII.[10] Subsequent Pschorr cyclization[11] of XVIII would then give the desired dihydrodibenzooxopyrrocoline, Ic.



In an attempted model cyclization experiment, the intermediate indoline (VIII) was first treated with *p*-anisoyl chloride to form 1-anisoyl-5-bromo-7-nitroindoline (IX). Reduction of IX afforded the 1-anisoyl-7-amino compound X in good yield. Although the amino compound X was smoothly diazotized in aqueous solution, heating with copper powder gave dark, alkali-soluble phenolic substances which were not characterized further, and none of the expected 4,5-dihydro-10-methoxy-7-oxo-7H-dibenzo[f,h,i]pyrrocoline (XI) was obtained. In a review of the Pschorr reaction,[11] it has been pointed out that *N*-benzoyl-*N*-alkylanilines, in which the amino group participating in the cyclization is located on the anilino moiety, do not usually yield the desired phenanthridones.

An alternative approach to the synthesis of Ic from III is via intermediates VII, XII, XIII, and XIV. When the nitration of III is carried out in concentrated nitric acid, 2-methoxy-3,4-methylenedioxy-6-nitrobenzoic acid (VII) is obtained as the reaction product.[9] The position of the entering nitro group was confirmed[9] by first decarboxylating VII and then reducing the resulting nitro compound to the known 3-methoxy-4,5-methylenedioxyaniline. In our hands, nitration of III occurred readily to give a 94% yield of VII, which was converted by treatment with thionyl chloride to the acid chloride XII which, in turn, was converted to the amide XIII by the action of excess indoline in ether solution. 1-(2-Methoxy-3,4-methylenedioxy-6-aminobenzoyl)indoline (XIV) was obtained by reduction of XIII in alcoholic solution over palladium-on-charcoal catalyst.

When the Pschorr cyclization of XIV was carried out in the same manner as that described for *N*-(6-amino-3,4-methylenedioxybenzoyl)-*p*-methylaminophenyl benzoate,[12] a complex mixture of products was obtained from which two pure substances were isolated by column chromatography. One of these (m.p. 132–133°) was shown to *N*-(2-methoxy-3,4-methylenedioxybenzoyl)indole (XV) on the basis of its ultraviolet absorption spectrum and the formation of III and indole upon saponification.

The second product was obtained as a nearly colorless crystalline solid, m.p. 155–156°, having an ultraviolet absorption spectrum which was different from that reported for anhydrofalcatine lactam.[1] The infrared absorption spectrum of this compound exhibited maxima at 6.05, 6.15, and 6.25 μ, as reported for Id. A nuclear magnetic resonance spectrum of this compound showed two broad absorptions and a nonequivalence quartet in the aromatic proton region. The two broad absorptions are consistent with what would be expected for four protons on ring D, while the remaining protons on ring A give rise to the observed nonequivalence quartet. Integration of the NMR curve against tetramethylsilane as an internal standard gave a total count of 15 protons, which is in agreement with structure XVI. Thus, *N*-(2-methoxy-3,4-methylenedioxybenzoyl)indoline (XVI) is formed via the deamination of XIV when the Pschorr cyclization of this compound is attempted using a copper powder catalyst.

Because the amine XIV was found to have limited solubility in the mineral acid media used for the diazotization step in the previously attempted Pschorr cyclizations, we repeated this step in an acetic–sulfuric acid mixture[13] without employing either ethanol or copper powder in the final cyclization step. Chromatographic separation of the reaction products, as described above, gave the indole (XV) (m.p. 132–133°) and a small quantity of a light yellow solid which melted at 258–260° after sublimation *in vacuo*. This compound exhibited an ultraviolet spectrum which was almost identical with anhydrofalcatine lactam. The NMR pattern, when compared with XVI, showed the modifications which would be expected upon introduction of the biphenyl linkage via the Pschorr cyclization. The low field resonance associated

(9) A. F. Wagner, E. Walton, A. N. Wilson, J. O. Rodin, F. W. Holly, N. G. Brink, and K. Folkers, *J. Am. Chem. Soc.*, **81**, 4983 (1959).

(10) W. G. Gall, B. D. Astill, and V. Boekelheide, *J. Org. Chem.*, **20**, 1538 (1955).

(11) R. Adams, *Org. Reactions*, 409 (1958).

(12) H. M. Fales, E. W. Warnhoff, and W. C. Wildman, *J. Am. Chem. Soc.*, **77**, 5885 (1955).

(13) Ref. 11, p. 434.

with the four protons on ring D (structure XVI) disappears and the nonequivalence quartet associated with the two protons on ring A is reduced to a single line, indicating a single proton attached to the benzene ring carrying the alkoxy groups. On the basis of these findings, it was concluded that this product has structure Ic. This compound, with the methoxyl in the 8-position, is therefore isoanhydrofalcatine lactam, as anhydrofalcatine lactam melts at 198–201°. Hence, the methoxyl group in falcatine is located in the 11-position of the A-ring. This conclusion is in agreement with the indirect evidence presented by Fales and Wildman.[1] They have suggested that, because caranine and 3,3a-dihydrocaranine both undergo the Oppenauer oxidation to the respective 1-keto compounds, whereas I remains unchanged in this reaction, the latter is sterically hindered by the presence of an 11-methoxy group.

EXPERIMENTAL[14]

5-Bromo-7-nitroindoline (VIII). N-Acetyl-5-bromo-7-nitroindoline was obtained in 65% over-all yield from indoline by acetylation, bromination, and nitration as described by Boekelheide.[10] A mixture of 29.3 g. of the acetyl compound, 50 ml. of alcohol, and 120 ml. of 6*N* hydrochloric acid was refluxed for 1.5 hr., cooled slightly, and poured into cold dilute ammonium hydroxide. The crude solid product was collected, washed thoroughly with dilute aqueous sodium hydroxide and water, and recrystallized from boiling methanol; yield, 17.6 g. (70%); m.p. 131–132°.

Anal. Calcd. for $C_8H_7BrN_2O_2$: C, 39.5; H, 2.9. Found: C, 39.5; H, 2.9.

N-(4-Methoxybenzoyl)-5-bromo-7-nitroindoline (IX). A mixture of 12.2 g. of VIII, 50 ml. of dry pyridine, and 12.7 g. of distilled anisoyl chloride was heated on a steam bath for 8 hr. under a reflux condenser protected with a drying tube. Upon cooling to room temperature and standing overnight, a mixture of crude product and long needles of pyridine hydrochloride crystallized. The solid was collected by filtration and washed with water, dilute hydrochloric acid, and water to remove all traces of pyridine; yield, 13.1 g. (70%) of a yellow crystalline solid, m.p. 197–198°. A sample recrystallized from alcohol–acetic acid for analysis melted at 198–199°.

Anal. Calcd. for $C_{16}H_{13}BrN_2O_4$: C, 51.0; H, 3.45. Found: C, 50.9; H, 3.50.

N-(4-Methoxybenzoyl)-7-aminoindoline (X). A suspension of 4.2 g. of IX in 200 ml. of boiling alcohol was shaken with hydrogen (3 atm.) in the presence of 2 g. of 10% palladium-charcoal catalyst for 0.5 hr. The catalyst was removed by filtration, and the filtrate was evaporated to dryness. The residue was dissolved in hot water and treated with 10% aqueous sodium carbonate, and the solid which separated was collected and recrystallized from alcohol; yield, 1.9 g. (64%), m.p. 161–162°.

Anal. Calcd. for $C_{16}H_{16}N_2O_2$: C, 71.6; H, 6.0. Found: C, 71.4; H, 5.8.

Attempted cyclization of X *to* XI. To a solution of 1.6 g. of X in 20 ml. of 2.5 *N* sulfuric acid and 10 ml. of methanol cooled to 0° was added gradually a solution of 0.5 g. of sodium nitrite in 3 ml. of water. The clear yellow solution of the diazonium salt was treated with 100 mg. of copper powder and heated on a steam bath for 35 min. The resulting dark solution was extracted with benzene, and the benzene extract was washed, dried, and concentrated. The residual tarry material would not crystallize, so it was chromatographed over alumina, eluting with benzene. No pure products were isolated from the eluates, and there was no evidence to indicate that the desired product, XI, had been formed.

N-(2-Methoxy-3,4-methylenedioxy-6-nitrobenzoyl)indoline (XIII). Croweacic acid (III) was prepared in about 1% over-all yield from pyrogallol by carbonation with aqueous sodium bicarbonate,[3] reaction of the resulting 2,3,4-trihydroxybenzoic acid with methylene sulfate and alkali in aqueous acetone,[4] and methylation with methyl sulfate and alkali.[5]

The following route to croweacic acid proved to be superior. To a solution of 28 g. of 3-methoxycatechol, obtained in 88% yield from *o*-vanillin,[6] in 30 ml. of methanol in a 300-ml. stirring autoclave was added 21.2 g. of methylene chloride, 6 g. of Tobin bronze turnings, 80 ml. of methanol, and a solution of 22 g. of potassium hydroxide in 30 ml. of water. The mixture was stirred and heated under pressure at 100 to 110° or 18 hr. The product, 3-methoxymethylenedioxybenzene (V), was isolated by steam distillation of the reaction mixture; yield, 11.5 g. (38%); m.p. 40–41° (reported, 41°).[7] To 67.5 g. of phosphorus oxychloride was added 60 g. of *N*-methylformanilide slowly and with cooling. After 30 min., 27 g. of V was added and the mixture was carefully warmed on a steam bath. When a vigorous exothermic reaction began, the mixture was removed from the steam bath. After this reaction had subsided, heating on a steam bath was resumed for 1.5 hr. The dark mixture was poured into ice and water, and the crude solid product which formed was collected after standing 0.5 hr. Recrystallization from 50% alcohol gave 16.4 g. (51%) of pure VI, m.p. 104–105° (reported,[8] 104°). To a stirred solution of 23 g. of VI in 325 ml. of acetone was added gradually a solution of 33 g. of potassium permanganate in 575 ml. of water. The mixture was then stirred and refluxed (68°) for 1.5 hr., made alkaline with 10% aqueous sodium hydroxide, and filtered from precipitated manganese dioxide; the acetone was evaporated under reduced pressure, and the residue acidified with hydrochloric acid to obtain croweacic acid (III), m.p. 153–154° (reported,[5] 153°); yield, 20.5 g. (81%). Nitration of croweacic acid as described[9] afforded 2-methoxy-3,4-methylene-dioxy-6-nitrobenzoic acid, m.p. 189–191°, (reported, 190–192°[9]) sufficiently pure for the next step, in 94% yield. To a cooled mixture of 95 ml. of dry reagent benzene, 25 ml. of pure thionyl chloride, and 2 drops of pyridine was added 31.5 g. of VII, and the mixture was stored at room temperature for 1 hr. After warming on a steam bath for 5 min. to complete the reaction, benzene and excess thionyl chloride were removed by evaporation under reduced pressure. The residual acid chloride was dissolved in 300 ml. of dry ether and added to a dry ether solution of 35 g. of purified indoline. After reaction was complete, the ether was evaporated and the residue was treated with water. The insoluble crude product was filtered, washed with water, and recrystallized from alcohol-water to give 35 g. (79%) of pure XI, m.p. 154–155°.

Anal. Calcd. for $C_{17}H_{14}N_2O_6$: C, 59.7; H, 4.1. Found: C, 59.5; H, 4.2.

N-(2-Methoxy-3,4-methylenedioxy-6-aminobenzoyl)indoline (XIV). A solution of 4 g. of XII in 200 ml. of boiling alcohol was shaken with hydrogen (3 atm.) in the presence of 1 g. of 10% palladium-charcoal catalyst until the quantity of hydrogen required to reduce the nitro group to amino had been absorbed (10 min.). The mixture was filtered to remove the catalyst, the filtrate was evaporated to dryness, and the residue was crystallized from alcohol–petroleum ether (b.p. 30–60°) to obtain 3.2 g. (87%) of XIV, m.p. 186–187°.

Anal. Calcd. for $C_{17}H_{16}N_2O_4$: C, 65.4; H, 5.1. Found: C, 65.6; H, 5.3.

4,5-Dihydro-8-methoxy-9,10-methylenedioxy-7-oxo-7H-dibenzo[f,h,i]pyrrocoline (Ic). A stirred suspension of 3.1 g. of XIV in 6.2 ml. of water containing 6.7 ml. of concd. hydrochloric acid and 10 ml. of ethanol was warmed to 50°

(14) Melting points are uncorrected.

and then cooled to 5–10° and treated with 5 ml. of 20% sodium nitrite solution. The resulting orange solution, which showed the presence of excess nitrous acid (starch–KI paper) was stirred in the cold for 15 min., then 600 mg. of urea was added to destroy the remaining nitrous acid. The mixture was immediately treated with 250 mg. of copper powder (Harshaw) (previously washed with concd. nitric acid followed by water) and then allowed to warm up to room temperature. When no further nitrogen evolution took place, the reaction mixture was found to be free of unreacted diazonium salt as indicated by the absence of a red color reaction when a drop of the solution was placed on a paper strip previously treated with an alkaline solution of β-naphthol.

The resulting dark brown oil which had formed was extracted from the aqueous phase with several portions of chloroform. The combined extracts were extracted 5 times with 10% aqueous sodium hydroxide to remove phenolic side-products and then dried over anhydrous magnesium sulfate. This extract was chromatographed through a 1.8 × 40 cm. column of neutral alumina (Woelm) and eluted with chloroform. Two crystalline fractions were obtained:

Fraction 1, m.p. 132–133°, 400 mg., was identified as *N-(2-methoxy-3,4-methylenedioxybenzoyl)indole*[15] (XV). The ultraviolet absorption spectrum had maxima at 246 mμ (log ε 4.18), 295 mμ (log ε 3.77), and 303 mμ (log ε 3.96).

Anal. Calcd. for $C_{17}H_{13}NO_4$: C, 69.1; H, 4.4. Found: C, 69.1; H, 4.3.

A 20 mg. sample of XV was refluxed with 10 ml. of alcohol and 1 ml. of 45% potassium hydroxide for 2 hr.; the alcohol was evaporated and the residue taken up in water and extracted with ether. The ether solution, on evaporation, gave an oil which solidified and was identified as indole. The alkaline aqueous layer was acidified and extracted with ether. Evaporation of the ether gave a solid, m.p. 153–154°, identical with croweacic acid.

Fraction 2, m.p. 136–142°, 950 mg., was a yellow crystalline solid which was again chromatographed on neutral alumina (Woelm). Elution with benzene, benzene-chloroform (1:1), and finally chloroform alone yielded 500 mg. of a pale yellow solid, m.p. 148–149°, in addition to a second fraction which still contained some XV. The entire 500 mg. sample was sublimed *in vacuo* (0.1 mm.) to afford XVI as a nearly colorless crystalline solid, m.p. 155–156°. The ultraviolet absorption spectrum exhibited maxima at 258 mμ (log ε 4.27), 284 mμ (log ε 4.12), and a shoulder at 292 mμ (log ε 4.09).

(15) The Pschorr cyclization of *N*-(1-amino-4,5-methylenedioxybenzoyl)indoline reported by L. G. Humber *et al.*, *J. Chem. Soc.*, 4622 (1954), yielded *N*-(4,5-methylenedioxybenzoyl)indole as well as the desired cyclization product 4,5-dihydro-9,10-methylenedioxy-7-oxo-7H-dibenzo[f,h,i]-pyrrocoline.

Anal. Calcd. for $C_{17}H_{15}NO_4$: C, 68.7; H, 5.05; N, 4.71. Found: C, 68.9; H, 5.1; N, 4.74.

In order to obtain a homogeneous system for the Pschorr reaction, an acetic acid-dilute sulfuric acid mixture was used. To a cooled (5 to 10°) solution of 2 g. of XIV in 150 ml. of glacial acetic acid and 15 ml. of 10% sulfuric acid was added a solution of 0.5 g. of sodium nitrite in 3 ml. of water. The mixture was allowed to warm to room temperature and then warmed on a steam bath for 4 hr. Most of the acetic acid was removed by evaporation under reduced pressure, and the residue was treated with 50 ml. of water and extracted thrice with chloroform. The dark chloroform extract of the crude product was washed with dilute aqueous sodium hydroxide and water, dried (anhydrous sodium sulfate), filtered, and chromatographed through alumina as before, using chloroform for elution. Two fractions were obtained: the first material to come off the column was identified as XV, m.p. 132–133°; then, a yellow band was eluted from the column which on evaporation, recrystallization from ethanol, and sublimation at 180–200°/0.05 mm. gave 20 mg. of a pale yellow solid, m.p. 258–260° (dec.).

Anal. Calcd. for $C_{17}H_{13}NO_4$: C, 69.1; H, 4.4. Found: C, 68.9; H, 4.4. The ultraviolet absorption spectrum exhibited maxima at 254 mμ (log ε 4.68), 273 mμ (log ε 4.29), 335 mμ (log ε 3.82), 350 mμ (log ε 3.85), and a shoulder at 300 mμ (log ε 3.98).[16]

Spectra. Ultraviolet spectra were determined on a Cary Model 14 recording spectrophotometer in ethanol solution. The nuclear magnetic resonance absorption spectra were determined on a Varian frequency R-F unit Model HR 60 with frequency of 60 megacycles and a field of 14,100 gauss. Deuterochloroform was the solvent and the chemical shifts were determined relative to tetramethylsilane as an internal standard. Infrared spectra were run with samples in potassium bromide disks on a Perkin-Elmer Model 221 spectrophotometer.

Acknowledgment. This research was supported by a Battelle Memorial Institute grant. We are indebted to Messrs. R. J. Jakobsen and T. F. Page, Jr., for measurement and interpretation of the NMR spectra. We also wish to thank Dr. W. C. Wildman for a reference sample of anhydrofalcatine lactam and for providing us with infrared and ultraviolet absorption curves for this compound.

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(16) The ultraviolet spectrum of anhydrofalcatine lactam is reported[1] to exhibit maxima at 256 mμ (log ε 4.69), 273 mμ (log ε 4.32), 332 mμ (log ε 3.79), 347 mμ (log ε 3.86), and a shoulder at 300 mμ (log ε 4.00).