sodium hydrogen sulfide[9] was added to the hot solution above. On cooling and addition of water N-(3-amino-4-picolylidene)-*p*-toluidine was precipitated and was collected and air-dried, giving 0.42 g. (50%) of material melting at 153–154°.[10] Normally the product was pale yellow in color and did not require further purification.

1,7-Naphthyridine-2-aldoxime (II). A solution of 1.28 g. (0.0060 mole) of N-(3-amino-4-picolylidene)-*p*-toluidine and 0.55 g. (0.0063 mole) of isonitrosoacetone in 12 ml. of ethanol and 3.0 ml. of 50% potassium hydroxide was heated for 6 hr. under reflux. The reaction mixture was steam distilled to remove the ethanol and *p*-toluidine. The solution was filtered and made acidic with acetic acid to precipitate the oxime. The crude, dried oxime, 0.89 g., m.p. 235–245°, was recrystallized from ethanol (charcoal) giving 0.5 g. (56%) of 1,7-naphthyridine-2-aldoxime, m.p. 245–246°, as a pale yellow powder.

Anal. Calcd. for $C_9H_7N_3O$: C, 62.42; H, 4.07; N, 24.27. Found: C, 62.50; H, 3.98; N, 24.31.

2,9-Diaza-6,8-dihydro-7,7-dimethyl-5-oxoanthracene (III). A mixture of 0.21 g. (0.001 mole) of N-(3-amino-4-picolylidene)-*p*-toluidine, 0.14 g. (0.001 mole) of dimethyldihydroresorcinol and three drops of piperidine was heated on the steam bath for 8 hr. After cooling, the brown mass was pulverized and extracted with ether. Evaporation of the ether gave 0.16 g. (71%) of crude product, m.p. 140–152°. After 4 recrystallizations from Skellysolve C,[11] 45 mg. (20%) of 2,9-diaza-6,8-dihydro-7,7-dimethyl-5-oxoanthracene, m.p. 152–155°, was obtained as colorless flakes.

Anal. Calcd. for $C_{14}H_{14}N_2O$: C, 74.31; H, 6.24; N, 12.38. Found: C, 74.23; H, 6.00; N, 12.15.

7,9-Diazabenz[f]indane (IV). A solution of 0.465 g. (0.0022 mole) of N-(3-amino-4-picolylidene)-*p*-toluidine and 0.185 g. (0.0022 mole) of cyclopentanone in 5 ml. of ethanol and 1.5 ml. of 2*N* sodium hydroxide solution was heated for 6 hr. on the steam bath. The ethanol and *p*-toluidine were removed by steam distillation. After cooling the solution, the naphthyridine was filtered off and air-dried, giving 0.345 g. (92%) of crude product, m.p. 80–84°. After 1 recrystallization from Skellysolve C[11] the colorless needles of 7,9-diazabenz[f]indane, 0.210 g. (56%), melted at 86–87°.

Anal. Calcd. for $C_{11}H_{10}N_2$: C, 77.62; H, 5.92; N, 16.46. Found: C, 77.92; H, 5.80; N, 16.62.

2,9-Diaza-5,6,7,8-tetrahydro-6-methylanthracene. (V). A solution of 0.53 g. (0.0025 mole) of N-(3-amino-4-picolylidene)-*p*-toluidine and 0.27 g. (0.0025 mole) of 4-methylcyclohexanone in 6 ml. of ethanol and 2 ml. of 2*N* sodium hydroxide solution was heated on the steam bath for 6 hr. The ethanol and *p*-toluidine were removed by steam distillation, and the naphthyridine was extracted from the residue with ether. Evaporation of the ether gave 0.36 g. (76%) of crude product, m.p. 82–86°. After 2 recrystallizations from Skellysolve C,[11] 0.275 g. (58%) of 2,8-diaza-5,6,7,8-tetrahydro-6-methylanthracene, m.p. 86–88°, was obtained as colorless needles.

Anal. Calcd. for $C_{13}H_{14}N_2$: C, 78.75; H, 7.12; N, 14.13. Found: C, 78.73; H, 7.11; N, 13.85.

2-Methyl-3-acetyl-1,7-naphthyridine (VI). A mixture of 0.211 g. (0.001 mole) of N-(3-amino-4-picolylidene)-*p*-toluidine, 0.100 g. (0.001 mole) of acetylacetone and 3 drops of piperidine was heated on the steam bath for 8 hr. After cooling, the brown mass was pulverized and extracted with ether. Evaporation of the ether gave 0.11 g. (59%) of crude product, m.p. 102–106°. After two recrystallizations from water (8 ml./0.1 g.), 0.03 g. (16%) of 2-methyl-3-acetyl-1,7-naphthyridine (colorless needles), m.p. 112–113.5°, was obtained.

Anal. Calcd. for $C_{11}H_{10}N_2O$: C, 70.95; H, 5.41; N, 15.05. Found: C, 71.41; H, 5.55; N, 15.31.

3-Nitro-2-phenyl-1,7-naphthyridine (VII). A solution of 0.211 g. (0.001 mole) of N-(3-amino-4-picolylidene)-*p*-toluidine and 0.166 g. (0.001 mole) of ω-nitroacetophenone in 2 ml. of absolute ethanol was heated under reflux for 2 hr. The cooled solution was diluted with water and extracted with ether. The ethereal solution was dried over magnesium sulfate, filtered, and evaporated under reduced pressure. The resultant red oil was recrystallized from Skellysolve C[11] to yield 0.025 g. (10%) of 3-nitro-2-phenyl-1,7-naphthyridine (yellow prisms), m.p. 120–121°.

Anal. Calcd. for $C_{14}H_9N_3O_2$: C, 66.93; H, 3.61; N, 16.72. Found: C, 67.13; H, 3.60; N, 16.31.

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(9) Hodgson and Ward, *J. Chem. Soc.*, 242 (1948).

(10) In the earlier paper[2] both the crude and purified materials were reported to melt at 146–148°. This was a typographical error on our part for the analytical sample actually melted at 152–153°.

(11) A hydrocarbon solvent, b.p. 88–98°.

[CONTRIBUTION FROM THE CONVERSE MEMORIAL LABORATORY, HARVARD UNIVERSITY]

Substituted Biphenyls by Action of Benzoyl Chloride on Some β-Aroylpropionic Acids

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The reaction of three β-aroylpropionic acids with benzoyl chloride is shown to give substituted phthalides in the biphenyl series. The ultraviolet and infrared spectra of these products and some related biphenyls are discussed.

The reaction of β-benzoylpropionic acid with benzoyl chloride was reported by Kugel[1] to yield an unidentified alkali-stable compound which had the empirical formula $C_{20}H_{14}O_3$. The structure of this product has now been investigated and the reaction extended to other β-aroylpropionic acids.

In our hands, this reaction produced a low yield of a product that had a melting point slightly higher than that of Kugel's compound. Combustion and molecular weight data required a revision of the earlier formula to $C_{27}H_{18}O_4$. The implicit stoichiometry, which can be represented as 2 $C_6H_5COCH_2CH_2COOH + C_6H_5COCl \rightarrow C_{27}H_{18}O_4 + HCl + 3H_2O$, was supported by the extension of the reaction to β-*p*-methoxybenzoyl- and β-*p*-chlorobenzoylpropionic acids to give similar condensation

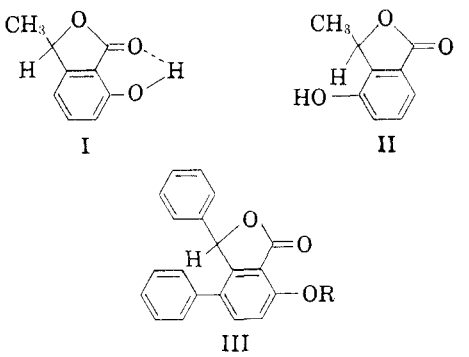
(1) Kugel, *Ann.*, 299, 61 (1898).

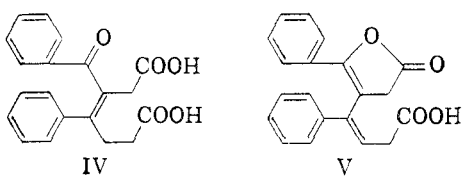
products. The appearance of a strong band at 5.66 μ and a shoulder near 5.71 μ in the infrared spectrum of each of these products gave evidence of the presence of two carbonyl groupings, at least one of which was very likely a five-membered lactone. These products displayed no olefinic character toward bromine in carbon tetrachloride or potassium permanganate in acetone.

The action of hot alcoholic potassium hydroxide on the above C_{27} compound, followed by acidification of the reaction mixture, led to the isolation of benzoic acid and a second compound whose analytical data suggested a $C_{20}H_{14}O_3$ formulation. This product exhibited hydroxyl and carbonyl absorption maxima in the infrared at 2.95 μ and 5.75 μ, respectively. The phenolic character of the hydroxyl group was easily recognized. The product gave a blue-green color with ferric chloride and readily reacted with diazomethane to give an ether which had no infrared maximum in the three-micron region but had a carbonyl band at 5.68 μ. The compound was inert toward chromium trioxide-pyridine, but readily formed a monoacetate which had a single carbonyl peak at 5.65 μ, characteristic of aryl acetates,[2] and likewise underwent benzoylation, regenerating the original C_{27} compound. The possibility that this substance contained an enolized ketone or other readily reducible group was unlikely since it was unaffected by 2,4-dinitrophenylhydrazine reagent or hydrogen over palladium on charcoal.

These results are consistent with the presence, in the C_{27} compound, of an aromatic benzoate ester and an alkali-stable five-membered lactone grouping. Of special significance is the observed decrease in wave length of the infrared carbonyl maximum of the C_{20} hydrolysis product when the phenolic hydroxyl is acylated or alkylated (*vide supra*). Such behavior would be expected of a phenolic lactone if the hydroxyl were suitably oriented to permit hydrogen-bonding to the carbonyl oxygen. For example, it has been shown[3] that on conversion of 7-hydroxy-3-methylphthalide (I) to its methyl ether the infrared carbonyl band is changed from 5.75 μ to 5.68 μ. In contrast, 4-hydroxy-3-methylphthalide (II), which is incapable of intramolecular hydrogen-bonding, and its methyl ether both absorb at 5.69 μ.

A consideration of the probable modes of condensation of two molecules of β-benzoylpropionic acid, viewed in the light of the above data, leads directly to structure III (R = C_6H_5CO—) for the C_{27} product. Although the exact sequence of steps is indeterminate, this product could arise through an initial acid-catalyzed aldol condensation and dehydration to yield IV. Cyclization to the enol-lactone V might then occur followed by cyclodehydration and aromatization to give III (R = H). Esterification of this phenol with benzoyl chloride would produce the postulated product (III, R = C_6H_5CO—).[4] This structure is compatible with all the observed properties of the compound. The extraordinary resistance of the phthalide toward alkaline hydrolysis is characteristic of 3-arylphthalides.[5] Moreover, the presence of the phenolate anion in the initial hydrolysis product (III, R=H) would further decrease the susceptibility of the carbonyl to nucleophilic attack.





To verify this structural conclusion, an ultraviolet spectral comparison with a known biphenyl was made. Since an appropriately disubstituted 4-hydroxybiphenyl was unavailable, it was necessary to employ one of the known 2- or 3-alkyl-4-hydroxybiphenyls as a model. Because of the sensitivity of the ultraviolet spectra of biphenyls to bulky 2-substituents, caused by steric interactions in the photoexcited state,[6] it was concluded that a member of the former group would be the more suitable model, provided its spectrum were compared with a derivative of III that lacked the carbonyl chromophore. The phthalide-ester III (R=C_6H_5CO—) was accordingly converted with lithium aluminum hydride to the triol VII, the identity of which was supported by an active hydrogen determination. The close similarity of the ultraviolet spectrum of this product with that of VIII (Fig. 1), obtained from lithium aluminum hydride reduction of 4-hydroxy-2-bi-

(2) Hartwell, Richards, and Thompson, *J. Chem. Soc.*, 1436 (1948).

(3) Hochstein and Pasternack, *J. Am. Chem. Soc.*, 74, 3905 (1952).

(4) Similar mechanisms can be envisaged for the condensations of β-*p*-methoxybenzoyl- and β-*p*-chlorobenzoylpropionic acids. The spectra of the products of these reactions are very similar to that of III (R = C_6H_5CO—), and in the latter case, hydrolysis of the product yielded a phenol with properties resembling those of III (R = H).

(5) Tasman, *Rec. trav. chim.*, 46, 653, 922 (1927).

(6) Beaven, Hall, Lesslie, and Turner, *J. Chem. Soc.*, 854 (1952), and references cited therein.

phenylcarboxylic acid,[7] provided convincing confirmation of the above structural arguments.

EXPERIMENTAL[8]

Condensation of β-benzoylpropionic acid was performed by heating 0.10 mole of the acid with 0.20 mole of benzoyl chloride on a steam bath for 15–18 hr. The resulting mixture was taken up in warm ethanol and allowed to stand at 0° for 2 days. The crystalline precipitate thus obtained was removed by filtration and recrystallized twice from 95% ethanol with the aid of charcoal to give nearly colorless needles of III (R = C_6H_5CO—), m.p. 194–195°. Yields averaged 7–10%.

Anal. Calcd. for $C_{20}H_{14}O_3$: C, 79.45; H, 4.67; mol. wt., 302. Calcd. for $C_{27}H_{18}O_4$: C, 79.79; H, 4.46; mol. wt., 406. Found: C, 80.14; H, 4.37; mol. wt. 413 (Rast).

The infrared spectrum showed major peaks at 5.65, (5.71), 6.23, 6.74, 6.89, 7.95, 8.53, 9.55, and 9.78 μ. The ultraviolet spectrum exhibited λ_{max} 228 and (285) mμ, log ε 4.58 and (3.70). The product was inert toward bromine in carbon tetrachloride or potassium permanganate in aqueous acetone.

Condensation of β-p-methoxybenzoylpropionic acid and benzoyl chloride, under the above conditions, produced a dark brown mass which, on reprecipitation from ethyl acetate-ethanol, was converted to a gummy powder. After trituration with ether-benzene, this product was recrystallized twice from benzene-ethyl acetate to give about a 5% yield of colorless flakes, m.p. 182–183°.

Anal. Calcd. for $C_{29}H_{22}O_6$: C, 74.67; H, 4.75. Found: C, 75.19; H, 5.12.

The principal infrared maxima were 5.67, (5.72), 6.22, 6.75, 8.06, 8.55, 9.29, 9.56, 9.79 μ; the spectrum was in general similar to that of III (R = C_6H_5CO—).

Condensation of β-p-chlorobenzoylpropionic acid with benzoyl chloride under similar conditions yielded a gum from which two products could be isolated. A solution of this mixture in ethyl acetate-ethanol gave, on standing, an amorphous, highly insoluble yellow powder, m.p. 245–248°.[9] The mother liquors, on longer standing, slowly deposited an almost equal quantity of a new substance which could be readily recrystallized from benzene-petroleum ether to give colorless needles, m.p. 191–192°, in 3% yield.

Anal. Calcd. for $C_{27}H_{16}O_4Cl_2$: C, 68.30; H, 3.40. Found: C, 68.73; H, 3.48.

The infrared spectrum showed maxima at 5.66, (5.72), 6.26, 6.79, 8.55, 9.20, 9.64, 9.79, and 9.87 μ. The ultraviolet absorption was very similar to that of III (R = C_6H_5CO—), with λ_{max} 229 mμ, log ε 4.63.

Hydrolysis of III (R = C_6H_5CO—) was carried out by refluxing 0.160 g. of the compound in a solution of 5% potassium hydroxide in 95% methanol for 6–10 hr. under a nitrogen atmosphere. Filtration of the cooled solution led to the isolation of a crystalline potassium salt (0.140 g.), which yielded a white curdy solid when stirred with dilute hydrochloric acid. An ether solution of this product was washed with water, dried over sodium sulfate, and evaporated to dryness. The powder so obtained was recrystallized from ether-petroleum ether to give colorless crystals (0.096 g., 82% yield), m.p. 139–140° (III, R = H).

Anal. Calcd. for $C_{20}H_{14}O_3$: C, 79.45; H, 4.67; mol. wt., 302. Found: C, 79.39; H, 4.84; mol. wt., 295 (Rast).

This substance gave a blue-green color with ferric chloride solution. It was insoluble in base and was unaffected by chromium trioxide–pyridine complex, 2,4-dinitrophenylhydrazine reagent or hydrogen over palladium on charcoal in ethanol. The infrared spectrum displayed peaks at 2.95, 5.75, 6.18, 6.73, 6.88, 7.40, 7.70, 8.55, 9.09, 9.40, 10.42, and 11.95 μ. The ultraviolet absorption showed λ_{max} (252) and 310 mμ, log ε (3.87) and 3.48.

Upon acidification and dilution of the original alkaline filtrate, a second substance was isolated. Sublimation of the crude material yielded benzoic acid, identified by mixed melting point with an authentic sample.

The methyl ether III (R = CH_3) crystallized directly in 85% yield from a saturated ethereal solution of the phenol containing excess diazomethane. Recrystallization of the product from ether-ethyl acetate gave colorless needles, m.p. 222–223°.

Anal. Calcd. for $C_{21}H_{16}O_3$: C, 79.73; H, 5.10. Found: C, 79.66; H, 5.07.

The methyl ether absorbed in the infrared at 5.68, 6.17, 6.29, 6.74, 7.80, 9.05, 9.30, and 9.60 μ. The ultraviolet spectrum had λ_{max} (252) and 307 mμ, log ε (3.97) and 3.62.

The acetate III (R = CH_3CO—) was formed upon acetylation of the phenol with acetic anhydride-pyridine at room temperature. Short needles were obtained (91% yield) from petroleum ether-ether, m.p. 191–192°.

Anal. Calcd. for $C_{22}H_{16}O_4$: C, 76.73; H, 4.68. Found: C, 77.16; H, 4.59.

The acetate had infrared absorption at 5.65, 6.24, 6.74, 6.90, 7.30, 7.50, 8.50, 9.12, 9.45, 9.80, 10.20, and 11.16 μ. The ultraviolet spectrum had λ_{max} (253) and 290 mμ, log ε (3.78) and 3.25.

The benzoate III (R = C_6H_5CO—) prepared with benzoyl chloride in pyridine and recrystallized from petroleum ether-ethyl acetate, was shown by mixed melting point and infrared and ultraviolet spectra to be identical with the β-benzoylpropionic acid-benzoyl chloride condensation product.

Hydrolysis of the $C_{27}H_{16}O_4Cl_2$ product (0.034 g., m.p. 191–192°) was performed in the same manner as the above hydrolysis of III (R = C_6H_5CO—). An insoluble potassium salt was also obtained and was purified in a like manner to yield a crude product (0.023 g., 87% yield) which, on crystallization from petroleum ether-ether, gave white needles, m.p. 186–187°.

Anal. Calcd. for $C_{20}H_{12}Cl_2O_3$: C, 64.71; H, 3.27. Found: C, 64.63; H, 3.66.

The infrared spectrum of this product showed absorption at 2.98, 5.76, 6.23, 6.79, 7.45, 8.61, 9.22, and 9.91 μ, and the ultraviolet had λ_{max} 259 and 309 mμ, log ε 4.06 and 3.66.

Reduction of III (R = C_6H_5CO—) was carried out by adding a saturated solution of the benzoate (0.40 g.) in dry benzene to a slurry of 0.40 g. lithium aluminum hydride in 30 ml. of freshly distilled tetrahydrofuran. The mixture was refluxed overnight, allowed to cool, and then treated with

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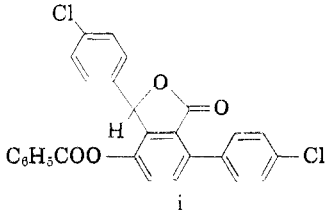
(7) Huntress and Seikel, *J. Am. Chem. Soc.*, 61, 817 (1939). We are indebted to Dr. Seikel (Wellesley College, Wellesley, Mass.) for providing a sample of this acid.

(8) Infrared spectra were of chloroform solutions unless otherwise stated, and were taken on a Perkin-Elmer 21B double beam spectrophotometer. Ultraviolet spectra were taken of 95% ethanol solutions on a Cary recording spectrophotometer. Spectral values in parentheses indicate shoulders. Analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, N. Y.

(9) The infrared maximum of this compound at 5.66 μ and the maxima of its amorphous hydrolysis product at 2.90 and 5.67 μ suggest that the former might be formulated as i. Such a product could arise by a mechanism similar to the proposed mode of formation of III (R = C_6H_5CO—).



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ethyl acetate to destroy the excess reducing agent. Addition of cold 5% sulfuric acid dissolved the precipitate. The resulting solution was extracted several times with ether. After washing with water and drying over magnesium sulfate, the extracts were evaporated to dryness to give a colorless oil (0.36 g.) which crystallized on trituration with benzene. Recrystallization from benzene-ethyl acetate yielded VII as a colorless product (0.26 g., 86% yield), m.p. 165–166°, which, after repeated recrystallizations, melted at 169–170°.

Anal. Calcd. for $C_{20}H_{18}O_3$: C, 78.41; H, 5.92. Found: C, 79.01; H, 5.77. An active hydrogen determination showed 0.84%, corresponding to 2.6 atoms.[10]

The infrared spectrum showed maxima at 3.00, 6.25, 6.82, 7.92, 8.57, and 8.85 μ. The ultraviolet spectrum is recorded in Fig. 1.

Reduction of 4-hydroxy-2-biphenylcarboxylic acid (0.080 g.) was performed essentially as described above. A crystalline white solid (0.045 g.) was obtained which was recrystallized several times from ether-benzene to yield VIII, m.p. 175–176°.

Anal. Calcd. for $C_{13}H_{12}O_2$: C, 77.98; H, 6.04. Found: C, 77.90; H, 5.87.

(10) Sample was incompletely soluble in the butyl ether.

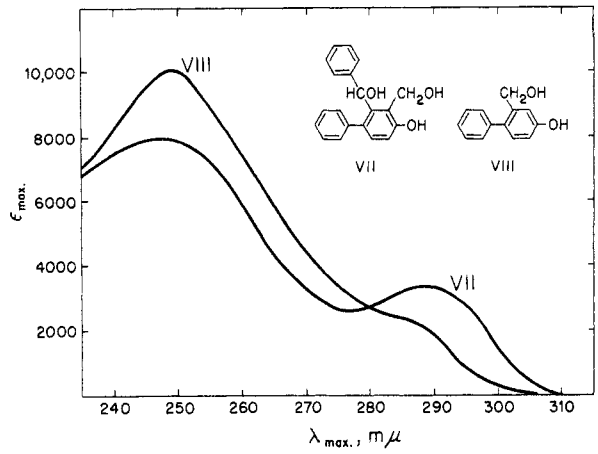

FIG. 1.—ULTRAVIOLET SPECTRA OF SUBSTITUTED 4-HYDROXYBIPHENYLS (IN 95% ETHANOL)

The ultraviolet spectrum is reproduced in Figure 1.

CAMBRIDGE, MASS.

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, UNIVERSITY OF VIRGINIA]

Some Pyridylnitroalkenes, Nitroalkanols, and Alkylamines

ALFRED BURGER, M. LUISA STEIN,[1] AND JOHN B. CLEMENTS

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Nicotinaldehyde condenses with nitroethane to 1-(3-pyridyl)-2-nitropropene which can be reduced stepwise to 3-pyridylacetoxime and 1-(3-pyridyl)-2-aminopropane. Pyridine-4-aldehyde and nitroethane furnish 1-(4-pyridyl)-2-nitropropanol-1, while isoquinoline-3-aldehyde and nitroethane give 1-(3-isoquinolyl)-2-nitropropene.

Considerable pharmacological interest has been attached to 2-aminoethyl and 2-aminopropyl derivatives of pyridine. 2-(2-Pyridyl)-ethylamine[2] and 1-(2-pyridyl)-2-aminopropane[3] resemble histamine in pharmacodynamic behavior whereas 1-(6-methyl-2-pyridyl)-2-aminopropane,[3] and especially 1-(5-ethyl-2-pyridyl)-2-aminopropane[3] produce marked analgesia in laboratory animals. 2-(3- or 4-Pyridyl)-ethylamines[2,4] are pressor amines, and 1-(3-pyridyl)-2-aminopropane[5] especially appears to have pronounced vasoconstrictor properties.[6] It became advisable to prepare this amine by a more rewarding route than described previously,[5] and the commercial availability of nicotinaldehyde invited a synthesis *via* 1-(3-pyridyl)-2-nitropropene. This compound was formed in 67% yield from nicotinaldehyde and nitroethane under the influence of *n*-butylamine at 90°, and could be reduced stepwise with lithium aluminum hydride. 3-Pyridylacetoxime[5] was obtained in high yields first. This reaction resembles the reduction of 1-phenyl-2-nitropropene which could be stopped at phenylacetoxime.[7] When four moles of the reducing agent was used in boiling ether for 10 hr., a mixture of 43% of 1-(3-pyridyl)-2-aminopropane and 40% of 3-pyridylacetoxime was formed. The latter could be separated and reduced in the same manner, but again only 48% of 1-(3-pyridyl)-2-aminopropane was obtained. No reduction of the pyridine ring was noted although this has been reported for certain other 3-substituted pyridine derivatives.[8,9]

Of the three pyridine aldehydes, only the 3-isomer reacts with nitromethane to give an α,β-unsaturated nitro derivative.[10] The 2- and 4-isomers furnish the corresponding 2-nitroethanols under analo-

(1) Consiglio Nazionale delle Ricerche Italia Fellow and Fulbright Grantee, 1955.

(2) L. A. Walter, W. H. Hunt, and R. J. Fosbinder, *J. Am. Chem. Soc.*, **63**, 2771 (1941).

(3) A. Burger and G. E. Ullyot, *J. Org. Chem.*, **12**, 342 (1947).

(4) C. O. Niemann and J. T. Hays, *J. Am. Chem. Soc.*, **64**, 2288 (1942).

(5) A. Burger and C. R. Walter, *J. Am. Chem. Soc.*, **72**, 1988 (1950).

(6) The pharmacological properties of the pyridylaminopropanes were studied by Smith, Kline and French Laboratories.

(7) R. T. Gilsdorf and F. F. Nord, *J. Am. Chem. Soc.*, **74**, 1837 (1952).

(8) V. M. Micovic and M. Lj. Mihailovic, *Rec. trav. chim.*, **71**, 970 (1952).

(9) F. Bohlmann and M. Bohlmann, *Chem. Ber.*, **86**, 1419 (1953).

(10) A. Dornow and F. Boberg, *Ann.*, **578**, 101 (1952).