Cycloaddition Reactions of Diphenylketene with Carbon-Nitrogen Double Bonds of Heterocycles

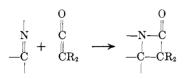
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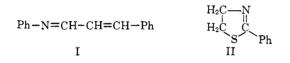
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The cycloaddition of diphenylketene to certain oxazoles, thiazoles, and imidazoles has been studied. The reaction of two molecules of diphenylketene and one of the heterocycle produces a piperidinedione. These sixmembered ring products are generally observed in the reaction of ketenes with carbon-nitrogen double bonds, except when both the carbon and the nitrogen of the double bond are attached to aromatic groups.

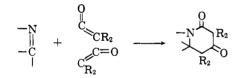
The special case of β -lactam formation from the cycloaddition of ketenes to carbon-nitrogen double bonds has been extensively investigated.^{1,2} In most of



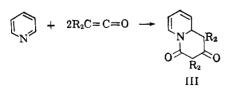
these cases, both the carbon and the nitrogen of the double bond are attached to aromatic groups; however, exceptions are known, such as the reaction of I^1 and $II.^3$



The cycloaddition reaction of two molecules of a ketene with a carbon-nitrogen double bond to form a six-membered ring, a piperidinedione, is more general.



With the above exceptions, this reaction occurs unless both the carbon and the nitrogen of the double bond hold an aromatic group. The most common examples of piperidinedione formation are with open chain compounds containing carbon-nitrogen double bonds, Schiff bases from aliphatic amines, and/or aliphatic aldehydes or ketones.^{3,4} Another example of this type of cycloaddition reaction is the reaction of two molecules of a ketene with pyridine or quinoline.^{4,5} Here the carbon-nitrogen double bond is part of the aromatic system.



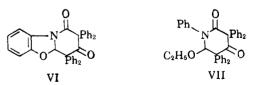
⁽¹⁾ H. Staudinger, Ann., 356, 51 (1970).

Cycloaddition reactions of ketenes with the carbonnitrogen double bonds of other heterocyclic systems have not been studied extensively. Cycloaddition reactions of several thiazoles with ketenes were investigated in the work on penicillin.³ In addition to the reaction of II with diphenylketene to form a β -lactam, both 2-methylthiazoline and benzothiazole were found to react with two molecules of diphenylketene to form IV and V, respectively. These reactions leading to IV and V were repeated and confirmed in the present study. Good yields were obtained even in the presence of excess 2-methylthiazoline or benzothiazole.



The present paper describes the addition of diphenylketene to benzoxazole, benzimidazole, N-methylbenzimidazole, and related compounds. In all the reactions studied, the products formed contained new six-membered rings, showing that the heterocyclic carbon-nitrogen double bond had added two molecules of diphenylketene. The structures were supported by analyses, infrared spectra, and analogy to known reactions.

Two molecules of diphenylketene were found to react with one of benzoxazole to produce the adduct VI in 88% yield. Compound VI was obtained in nearly as high a yield based on the diphenylketene when benzoxazole was in excess. Similar results were obtained in the reaction of diphenylketene with ethyl *N*-phenylformimidate (like the oxazoles, the oxygen of this compound is attached to the carbon-nitrogen double bond); the product VII was isolated in 60% yield.



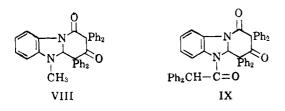
Two molecules of diphenylketene reacted exothermically with N-methylbenzimidazole to give the adduct VIII. Benzimidazole reacted with three molecules of diphenylketene to produce IX. The reaction of benzimidazole with one molecule of diphenylketene gave the amide (X), but the yield was low (30%). The infrared spectrum showed that the crude reaction mixture contained unreacted benzimidazole, IX, and perhaps XI. The presence of IX and unchanged benzimid-

⁽²⁾ J. C. Sheehan and E. J. Corey, Org. Reactions, 9, 395 (1957).

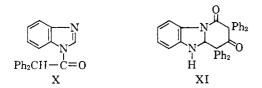
⁽³⁾ H. T. Clark, J. R. Johnson, and Sir R. Robinson, "The Chemistry of Penicillin," Princeton University Press, Princeton, N. J., 1949, pp. 975– 991.

⁽⁴⁾ H. Staudinger, H. W. Klever, and P. Kober, Ann., 374, 1 (1910).

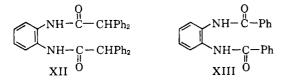
⁽⁵⁾ H. Staudinger, Ber., 40, 1145 (1907); J. A. Berson and W. M. Jones, J. Am. Chem. Soc., 78, 1625 (1956).



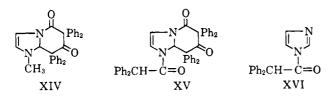
azole indicates that the rate of the cycloaddition forming the six-membered ring is comparable to that of amide formation with benzimidazole, which is not the case with imidazole (see below). The amide (X) was converted into IX by treatment with two molecules of diphenylketene.



The amide (X) was prepared also from benzimidazole and diphenylacetyl chloride, but the product was contaminated with appreciable amounts of XII. The identity of XII was established by comparison with the product of the reaction of excess diphenylacetyl chloride with *o*-phenylenediamine in pyridine. Acylation of benzimidazole with benzoyl chloride reveals a similar side reaction yielding XIII.⁶



The reaction of diphenylketene with N-methylimidazole gave XIV. As expected, imidazole reacted with three molecules of diphenylketene to give XV, and, if only one mole of diphenylketene were used, the expected amide (XVI) was formed in good yield (71%). Here the infrared spectrum of the crude product was identical with that of the purified product, indicating that only one reaction had occurred, amide formation, and that the rate of amide formation was so fast that cycloaddition was not a competing reaction. The amide (XVI) was converted into XV by the addition of two molecules of diphenylketene.

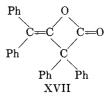


The amides (X and XVI) could not be synthesized by the action of dicyclohexylcarbodiimide on a tetrahydrofuran solution of the imidazole and diphenylacetic acid.

The N-(diphenylacetyl)imidazole (XVI) was an active acetylating agent.⁷ It gave diphenylacetanilide with aniline at 100° and ethyl diphenylacetate with ethanol at 80° . In moist air it was hydrolyzed to the

salt, imidazolium diphenylacetate, which was also obtained from imidazole and diphenylacetic acid.

The cycloaddition of ketenes to enamines is known to proceed readily.⁸ In an attempt to obtain cycloaddition at both double bonds of the N-methylimidazole, 5 moles of diphenylketene was mixed with 1 mole of N-methylimidazole. An exothermic reaction occurred, but the desired cycloaddition at both double bonds of the N-methylimidazole was not obtained. In addition to the adduct XIV, another compound was isolated that was identified as 2,2,4,4-tetraphenyl-3-hydroxy-3-butenoic lactone (XVII), the β -lactone dimer of diphenylketene.^{9,10} That no cycloaddition of diphenylketene occurs at the carbon-carbon double bond of Nmethylimidazole is less surprising in view of the fact that no further addition has been reported to occur at the carbon-carbon double bonds in the adducts of a ketene and pyridine (III).



In 2-phenylbenzothiazole (XVIII) and 1-methyl-2phenylbenzimidazole (XIX) both the carbon and nitrogen of the carbon-nitrogen double bond are attached to aromatic groups. If reaction occurred, such compounds would be expected to react with one molecule of diphenylketene to give a β -lactam. No products could be isolated from the reaction of either XVIII of XIX with diphenylketene, even when the substances were heated in a sealed tube at 100° for 5 days. The recovery of XVIII was 86%. The recovery of XIX was 63%, and here the diphenylketene was converted to XVII, isolated in 67% yield.



Very little is known about the mechanism of the type of cycloaddition reaction forming the piperidinediones, but the high yield with an excess of the compound containing the carbon-nitrogen double bond is an indication that the reaction does not have the β -lactam as a precursor.

Experimental

All analyses were by Galbraith Laboratories, Inc., Knoxville, Tenn. Melting points are uncorrected. Infrared spectra were measured on a Perkin-Elmer Infracord.

Diphenylketene, b.p. 115-125° at 1 mm., was freshly distilled before use."

N-Methylbenzimidazole was prepared by a modification of the method of Fischer.¹² A sealed tube containing 9.4 ml. of methyl iodide and 17.7 g. of benzimidazole dissolved in 100 ml. of methanol was heated at 100° for an hour. After the cooled tube was opened, the solvent was evaporated and 75 ml. of chloroform was added to the residue. The insoluble material

⁽⁶⁾ B. Oddo and F. Ingraffia, Gazz. chim. ital., 62, 1092 (1932); Chem. Abstr., 27, 2687 (1933).

⁽⁷⁾ E. R. Stadtman and F. H. White, Jr., J. Am. Chem. Soc., 75, 2022 (1953); T. Wieland and G. Schneider, Ann., 580, 159 (1953); H. A. Staab, Chem. Ber., 89, 1927 (1956).

 $^{(8)\} R.$ H. Hasek and J. C. Martin, J. Org. Chem., $\mathbf{28},\ 1468\ (1963),\ and$ references given there.

⁽⁹⁾ R. D. Kimbrough, Jr., ibid., 29, 1246 (1964).

⁽¹⁰⁾ R. Anet, Chem. Ind. (London), 1313 (1961).

⁽¹¹⁾ L. I. Smith and H. H. Hoehn, Org. Syn., 20, 47 (1940).

⁽¹²⁾ O. Fisher and H. Veiel, Ber., 38, 321 (1905).

was removed, the chloroform was evaporated, and the residue was distilled at 0.2-0.3 mm. A fraction of 8 ml., collected 108-123°, was redistilled to obtain a fraction boiling at 102-106° at 0.2 mm., which solidified in the receiver. After two recrystallizations from pentane, the yield was 2.1 g. (11%), m.p. 61-62° (lit.¹² m.p. 61°); infrared (CHCl₃): 3.00, 3.36, 4.00, 6.18, 6.69, 6.88, 7.07, 7.15, 7.53, 7.69, 8.25, 8.70, 9.51, 9.95, 11.24, and 11.56 μ .

Diphenylketene and Benzimidazole (1:1).—A tetrahydrofuran solution of 2.0 g. of diphenylketene and 1.2 g. of benzimidazole was kept under nitrogen at 25° overnight. The solvent was evaporated and the residual oil was treated with boiling pentane. The hot solution was filtered and crystals separated from the pentane on cooling. After another such recrystallization the yield of N-(diphenylacetyl)benzimidazole was 0.96 g. (30%), m.p. 72–73°; infrared (CHCl₃): 2.90, 3.32, 5.78, 6.22, 6.68, 6.89, 7.32, 7.66, 7.81, 8.69, 9.10, 10.26 and 11.28 μ .

Anal. Calcd. for $C_{21}H_{16}N_2O$: C, 80.8; H, 5.1; N, 9.0. Found: C, 80.7; H, 4.9; N, 9.4.

Diphenylacetyl Chloride and Benzimidazole.—When 1.0 g. of diphenylacetyl chloride¹³ was added to a solution of 1.0 g. of benzimidazole in 10 ml. of dry tetrahydrofuran, the mixture warmed and a precipitate formed. The reaction mixture was kept overnight at 25°. The solid benzimidazole hydrochloride was separated and the filtrate was evaporated. The residual oil was boiled with 50 ml. of pentane and the pentane was decanted. The residue was about 0.1 g. of N,N'-o-phenylenebis-(diphenylacetamide), m.p. 185–190°. Its melting point was not depressed by mixture with an authentic sample (below). The pentane solution was cooled in ice and the solid that formed was impure N-(diphenylacetyl)benzimidazole, 0.52 g., m.p. 55–65°. The cold pentane solution was evaporated, yielding 0.10 g. (8%) of product with m.p. and m.m.p. 70–72°. N,N'-o-Phenylenebis(diphenylacetamide) (XII).—Diphenyl-

 N, \bar{N}' -o-Phenylenebis(diphenylacetamide) (XII).—Diphenylacetyl chloride,¹³ 1.0 g., was added to a solution of o-phenylenediamine in 10 ml. of benzene containing 5 ml. of pyridine. The mixture was heated on a steam bath for 20 min. and poured into 75 ml. of water. The benzene layer was separated, washed with 20 ml. of 5% sodium carbonate, dried (MgSO₄), filtered, and evaporated. The crystalline residue was dissolved in 10 ml. of benzene and 20 ml. of pentane was added. The solid was collected on a filter, yielding 0.47 g. (88.%) of product, m.p. 187-192°. Recrystallization from 15 ml. of ethanol yielded 0.33 g. (62%) of product, m.p. 192-193°; infrared (CHCl₃): 2.84, 2.96, 3.19, 5.96, 6.24, 6.70, 6.90, 7.70, 8.62, 9.29, and 9.73 μ .

Anal. Calcd. for $C_{34}H_{28}N_2O_2$: C, 82.2; H, 5.7; N, 5.6. Found: C, 82.2; H, 6.0; N, 5.3.

N-(Diphenylacetyl)imidazole from Diphenylketene and Imidazole (1:1).—To a solution of 1.36 g. of imidazole in 10 g. of dry tetrahydrofuran was added 3.88 g. of diphenylketene. The reaction mixture warmed and the yellow color of the diphenylketene disappeared instantly. Solvent was removed and the solid residue was recrystallized from 15 ml. of benzene. The yield was 4.1 g. (71%), m.p. 127–129. The melting point was not depressed on mixture with the product of diphenylacetyl chloride and imidazole.

N-(Diphenylacetyl)imidazole from Imidazole and Diphenylacetyl Chloride.—A mixture, 4.60 g. of diphenylacetyl chloride¹³ and 1.36 g. of imidazole, was heated on the steam bath for an hour. A solution of the product in 50 ml. of chloroform was extracted with 50 ml. of 5% sodium bicarbonate and was dried (MgSO₄). The filtered chloroform solution was evaporated and the residue crystallized. The solid was recrystallized twice from 15 ml. of benzene. The yield was 3.3 g. (65%) of product, m.p. 127-129°; infrared (CHCl₃): 2.92, 3.29, 5.73, 6.22, 6.79, 7.28, 7.66, 9.03, 9.29, and 10.31 μ .

Anal. Caled. for $C_{17}H_{14}N_2O$: C, 77.8; H, 5.4; N, 10.7. Found: C, 77.8; H, 5.1; N, 10.7.

N-(Diphenylacetyl)imidazole and Ethanol.—A solution of 1.0 g. of N-(diphenylacetyl)imidazole in 5 ml. of ethanol was refluxed for 15 min. To the cooled solution was added 5 ml. of water. After 10 hr. the ethyl diphenylacetate was collected, yielding 0.65 g. (71%) of product, m.p. and m.m.p. 56–58°, lit.¹⁴ m.p. 58–59°.

N-(Diphenylacetyl)imidazole and Aniline.—A solution of 1.0 g. of N-(diphenylacetyl)imidazole in 2 ml. of aniline was heated on the steam bath for an hour. The brown reaction mixture was

poured into 10 ml. of 5% hydrochloric acid. The precipitate of diphenylacetic anilide was 1.1 g. (100%), m.p. 174–178°, lit.¹⁶ m.p. 180°. The melting point was not depressed by mixture with authentic diphenylacetic anilide.

Hydrolysis of N-(Diphenylacetyl)imidazole to Give Imidazolium Diphenylacetate.—A very finely powdered sample of 1.0 g. of N-(diphenylacetyl)imidazole was kept in the air for 2 days during warm humid weather. The melting point of this crude material was $125-135^{\circ}$. Recrystallization from benzene gave m.p. $137-139^{\circ}$. The yield of imidazolium diphenylacetate was 0.60 g. (56%). The melting point of this material was not depressed on mixture with authentic imidazolium diphenylacetate.

Imidazolium Diphenylacetate.—A solution of 0.68 g. of imidazole in 3 ml. of tetrahydrofuran was added to 2.12 g. of diphenylacetic acid. The solvent was evaporated, and the salt crystallized. The yield was 2.8 g. (100%) of product, m.p. 130–136°. Recrystallization from 10 ml. of benzene raised the melting point to 138–139°; infrared (CHCl₃): 3.31, 3.85, 5.12, 6.26, 6.39, 6.70, 6.90, and 7.29 μ .

Anal. Caled. for $C_{17}H_{16}N_2O_2$: C, 72.9; H, 5.7; N, 10.0. Found: C, 72.7; H, 5.7; N, 10.0.

1-Methyl-2-phenylbenzimidazole.¹⁶—A solution containing 11.64 g. of 2-phenylbenzimidazole, 2.4 g. of sodium hydroxide, and 3.72 ml. of methyl iodide in 75 ml. of methanol was kept for 4 days at 25°. The solvent was evaporated, 75 ml. of chloroform was added, and the insoluble material (sodium iodide and 2-phenylbenzimidazole) was removed by filtration. The filtrate was chromatographed over 100 ml. of neutral alumina in a column with an inside diameter of 2.0 cm. The column was eluted with chloroform and 15-ml. fractions were taken. Fractions 5 and 6 each contained 2.5 g. of product, m.p. 92–95°. These fractions were combined and recrystallized from hexane. The yield of 1-methyl-2-phenylbenzimidazole was 4.25 g. (34%), m.p. 94–96°, lit.¹⁷ m.p. 98°; infrared (CHCl₃): 2.94, 3.32, 3.94, 6.20, 6.85, 6.96, 7.25, 7.55, 7.83, 8.69, 8.89, 9.80, 9.95, and 12.22 μ .

Diphenylketene and 1-Methyl-2-phenylbenzimidazole.—A mixture of 0.60 g. of 1-methyl-2-phenylbenzimidazole and 0.60 g. of diphenylketene was sealed in a tube under nitrogen and was heated at 100° for 4 days. The contents of the tube were added to 15 ml. of ethanol, and the solid that formed was collected on a filter. This was XVII, the β -lactone dimer of diphenylketene,^{9,10} 0.40 g. (67%), m.p. and m.m.p. 146–148°. The filtrate was evaporated and the residue was triturated with 10 ml. of hexane. The solid was collected on a filter, yielding 0.38 g. (63% recovery) of 1-methyl-2-phenylbenzimidazole, m.p. 85–89°.

Diphenylketene and 2-Phenylbenzothiazole.—A sealed tube containing 2.0 g. of diphenylketene and 2.1 g. of 2-phenylbenzo-thiazole under nitrogen was heated at 100° for 4 days. The reaction mixture was treated with pentane and 1.8 g. (86% recovery) of 2-phenylbenzothiazole was obtained with m.p. and m.m.p. $109-112^{\circ}$.

Cycloaddition Reactions of Diphenylketone.—A reaction, typical of those outlined in Table I, is described for the preparation of VI.

2,2,4,4-Tetraphenyl-1,2,3,4-tetrahydro-4aH-pyrido[2,1-b] benzoxazole-1,3-dione (VI).—A mixture of 2.0 g. of diphenylketene (0.010 mole) and 0.60 g. of benzoxazole (0.005 mole) was kept under nitrogen at 25° for a week, then was made to crystallize by treatment with 5 ml. of ethanol. The solid was rinsed with 5 ml. of ethanol to yield 2.3 g. (88%) of product, m.p. 207-209°. Recrystallization from a mixture of ethyl acetate and ethanol gave a product with m.p. 209-210°; infrared (CHCl₃): 3.21, 3.37, 5.60, 6.02, 6.24, 6.76, 6.91, 7.48, 7.93, 9.14, 9.97, 10.99, and 11.60 μ .

Anal. Caled. for $C_{35}H_{28}NO_3$: C, 82.8; H, 4.9; N, 2.8. Found: C, 82.4; H, 5.2; N, 2.8.

6,6,8,8-Tetraphenyl-8a-methyl-2,3,5,6,7,8-hexahydro-8aHthiazolo[3,2-a]pyridine-1,3-dione (IV).—The infrared spectrum in chloroform showed bands at 3.24, 5.65, 6.10, 6.70, 6.95, 7.66, 8.71, 9.41, 9.81, 10.04, and 10.35 μ .

2,2,4,4-Tetraphenyl-1,2,3,4-tetrahydro-4aH-pyrido[2,1-b]benzothiazole-1,3-dione (V).—The infrared spectrum in chloro-

⁽¹³⁾ H. Staudinger, Ber., 44, 1619 (1919).

⁽¹⁴⁾ R. S. Yost and C. R. Hauser, J. Am. Chem. Soc., 69, 2325 (1947).

⁽¹⁵⁾ F. Klingemann, Ann., 275, 84 (1893).

⁽¹⁶⁾ The only reference to this compound in Beilstein is O. Fischer, Ber., 25, 2842 (1892). Here the melting point is given as 170-171°. This has been corrected by R. Weidenhagen,¹⁷ who gives the melting point as 98°.
(17) R. Weidenhagen and J. Train, *ibid.*, 75, 1936 (1942).

TABLE I

Cycloaddition Reactions of Diphenylketene ^a										
		Solvent of	Yield,				Anal., %			
Other reactant	Conditions	crystallization	Product	%	M.p., °C.	Formula		C	Η	Ν
2-Methylthiazoline	No solvent 25° for 1 wk.	Ethyl acetate– ethanol	IV	73	167-167.5 ^b	$\mathrm{C_{32}H_{25}NO_{2}S}$	Calcd. Found		5.5 5.5	
Benzothiazole	No solvent 25° for 1 day	Ethyl acetate- ethanol	V	86	163–164°	$\mathrm{C}_{35}\mathrm{H}_{25}\mathrm{NO}_{2}\mathrm{S}$	Calcd. Found	-	$\frac{4.8}{5.4}$	$egin{array}{c} 2.7 \\ 2.5 \end{array}$
Benzoxazole	No solvent 25° for 1 wk.	Ethyl acetate- ethanol	VI	88	209-210	$\mathrm{C}_{35}\mathrm{H}_{25}\mathrm{NO}_{3}$	Caled. Found		$\begin{array}{c} 4.9 \\ 5.2 \end{array}$	$\frac{2.8}{2.8}$
Ethyl N-phenyl- formimidate ^d	No solvent 25° for 1 wk.	Ethanol	VII	60	163-164	$\mathrm{C}_{37}\mathrm{H}_{31}\mathrm{NO}_3$	Caled. Found		5.8 6.1	$egin{array}{c} 2.6 \\ 2.6 \end{array}$
N-Methylbenz- imidazole	In eth er 25° for 1 day	Ethyl acetate	VIII	85	206-208	${ m C_{36}H_{28}N_2O_2}$	Calcd. Found		5.4 5.4	5.4 5.2
Benzimidazole	No solvent 100° for 1 hr.	Triturated with ethyl acetate	IX	83	152-153	$\mathrm{C_{49}H_{36}N_2O_3}$	Calcd. Found		5.1 5.2	$\begin{array}{c} 4.0\\ 4.1 \end{array}$
N-(Diphenyl- acetyl)benz- imidazole	No solvent 100° for 1 hr.	Triturated with ethyl acetate	IX	86	152-153					
N-Methylimidazole	No solvent 25° for 1 day	Ethanol	XIV	64	157-158	${ m C}_{32}{ m H}_{26}{ m N}_2{ m O}_2$	Calcd. Found		5.6	5.9 5.8
Imidazole	No solvent 100° for 1 hr.	Triturated with benzene	XV	19	132 dec.	${ m C_{45}H_{34}N_2O_3}$	Calcd. Found		5.2 5.1	$\begin{array}{c} 4.3 \\ 4.2 \end{array}$
N-(Diphenyl- acetyl)imidazole	In tetrahydro- furan, 25° for 1 day	Triturated with benzene	XV	56	132 dec.					

^a All reactions were run under nitrogen. ^b Lit.² m.p. 160-161°. ^c Lit.³ m.p. 163-164°. ^d R. M. Roberts and P. J. Vogt, Org. Syn., **35**, 65 (1955).

form showed bands at 3.26, 5.66, 6.10, 6.26, 6.34, 6.71, 6.84, 7.27, 8.10, 8.39, 9.16, 9.45, 9.93, 10.21, 11.05 μ .

1,3,3,5,5-Pentaphenyl-6-ethoxypiperidine-2,4-dione (VII).— The infrared spectrum in chloroform showed bands at 3.27, 5.64, 6.13, 6.26, 6.69, 6.91, 7.10, 7.53, 7.77, 8.10, 8.91, 9.40, and 11.65 μ .

2,2,4,4-Tetraphenyl-5-methyl-1,2,3,4,4a,5-hexahydropyrido-[1,2-a] benzimidazole-2,4-dione (VIII).—The infrared spectrum in chloroform showed bands at 3.02, 3.32, 3.39, 4.00, 5.70, 6.24, 6.69, 6.88, 7.20, 7.55, 7.80, 8.54, 8.91, and 9.43 μ .

2,2,4,4-Tetraphenyl-5-(diphenylacetyl)-1,2,3,4,4a,5-hexahydropyrido[1,2-a]benzimidazole-2,4-dione (IX).—The infrared spectrum in chloroform showed bands at 2.26, 5.66, 5.98, 6.29, 6.76, 6.94, 7.23, 7.53, 8.65, 8.78, 9.24, 9.85, 9.98, and 11.03 μ . 1-Methyl-6,6,8,8-tetraphenyl-1,5,6,7,8,8a-hexahydroimidazo-[1,2-a]pyridine-5,7-dione (XIV).—The infrared spectrum in chloroform showed bands at 2.98, 3.35, 3.98, 5.73, 6.25, 6.70, 6.91, 7.82, 8.85, 9.29, 9.73, 9.99, 10.35, 10.92, and 11.40 μ .

1-(Diphenylacetyl)-6,6,8,8-tetraphenyl-1,5,6,7,8,8a-hexahydroimidazo[1,2-a]pyridine-5,7-dione (XV).—The infrared spectrum in chloroform showed bands at 2.95, 3.25, 5.64, 6.01, 6.25, $6.70, 6.91, 7.20, 8.87, and 9.26 \mu$.

Acknowledgment.—The author gratefully acknowledges the assistance of Mr. N. Burke, Mr. J. R. Carlile, and Mr. D. F. Hill.