H, 6.5; N, 7.8.

The isomeric purity of the tetramethyl ester of **4** was checked by hydrolysis of the ester to **4** and TLC analysis of the latter.[15]

Coproporphyrin I (1) (Tetramethyl Ester). The formyldipyrrylmethane **30** (100 mg) was dissolved in a mixture of 2 mL of glacial acetic acid and 0.4 mL of 40% hydrobromic acid in glacial acetic acid. The solution was kept at 5 °C during 15 min. It was then freeze-dried, the residue was dissolved in 60 mL of anhydrous methanol, and 100 mg of *p*-toluenesulfonic acid was added. The mixture was kept during 24 h at 20 °C in the dark. Methanol (10 mL) saturated with zinc acetate dihydrate was added to the solution, and the procedure described for the synthesis of **3** was followed to obtain **1** as its tetramethyl ester: 30 mg (43%); mp 245–247 °C (from methylene chloride–cyclohexane) (lit. mp 248–252[1] and 252–255 °C[3]); NMR (0.05 M $CDCl_3$) δ 3.25 (m, 8, CH_2CO_2R), 3.65, 3.7 (br, br, 24, OCH_3, CH_3), 4.4 (m, 8, $CH_2CH_2CO_2R$), 9.9 (br, 4, -CH=); MS *m/e* 710 (M^+, 35).

Anal. Calcd for $C_{40}H_{46}O_8N_4$: C, 67.6; H, 6.5; N, 7.9. Found: C, 67.4; H, 6.2; N, 7.8.

Hydrolysis of the ester (6 M hydrochloric acid) gave **1**, which was pure by TLC analysis.[8]

Coproporphyrin II (2) (tetramethyl ester) was obtained following the procedure described for **3** and **4**. Condensation of 200 mg of the diacid **32**[14] and 200 mg of the diformyldipyrrylmethane **5** afforded 68 mg (30%) of **2** (tetramethyl ester): mp 285–287 °C (lit.[3] mp 286–289 °C); NMR (0.05 M $CDCl_3$) δ 3.25 (m, 8, CH_2CO_2R), 3.55, 3.6 (br, br, 24, CH_3, OCH_3), 4.35 (m, 8, $CH_2CH_2CO_2R$), 10.0 (br, 4, CH=); MS *m/e* 710 (M^+, 100).

Anal. Calcd for $C_{40}H_{46}O_8N_4$: C, 67.6; H, 6.5; N, 7.9. Found: C, 67.5; H, 6.3; N, 7.8.

Hydrolysis of the ester (6 M hydrochloric acid) gave **2**, which was pure by TLC analysis.[8]

Acknowledgment. This work was made possible by a grant from the National Institutes of Health (GM-11973).

Registry No.—**1** (tetramethyl ester), 25767-20-8; **2** (tetramethyl ester), 865-16-7; **3** (tetramethyl ester), 5522-63-4; **4** (tetramethyl ester), 13306-30-4; **5**, 21211-64-3; **6**, 68781-31-7; **7**, 60204-96-8; **8**, 58684-20-1; **10**, 51741-18-5; **12**, 53700-88-2; **13**, 6122-77-6; **14**, 54278-18-1; **15**, 54278-16-9; **16**, 62562-72-5; **17**, 68781-32-8; **18**, 68781-33-9; **19**, 68781-34-0; **20**, 68781-35-1; **21**, 68781-36-2; **22**, 68781-37-3; **23**, 68781-38-4; **24**, 68813-11-6; **26**, 62562-80-5; **27**, 68781-39-5; **28**, 68781-40-8; **29**, 68813-12-7; **30**, 68781-41-9; **32**, 52091-10-8; *tert*-butyl acetoacetate, 1694-31-1; ethyl 4-acetyl-5-oxohexanoate, 2832-10-2.

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1,3-Diphenyldibenzo[*g,i*]thieno[3′,4′:3,4]pyrrolo[1,2-*a*]pyridine-2-S^{IV}, a New "Nonclassical" Thiophene System

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1,2-Dibenzoyldibenzo[*e,g*]pyrrolo[1,2-*a*]pyridine and P_4S_{10} at 140 °C in dry xylene gave 1,3-diphenyldibenzo[*g,i*]-thieno[3′,4′:3,4]pyrrolo[1,2-*a*]pyridine-2-S^{IV}, a "nonclassical" thiophene containing only three aromatic substituents attached to the thienopyrrole nucleus. Unstable to oxygen and light, it did not undergo cycloaddition with electron-deficient dipolarophiles. The analogous 1,3-diphenylbenzo[*g*]thieno[3′,4′:3,4]pyrrolo[1,2-*a*]pyridine-2-S^{IV} was prepared in a similar manner from 1,2-dibenzoylbenzo[*e*]pyrrolo[1,2-*a*]pyridine but was so unstable that it only had a transient existence. Other derivatives of the benzo- and dibenzopyrrolo[1,2-*a*]pyridine systems were prepared by cycloaddition reactions involving dimethyl acetylenedicarboxylate and intermediate anhydro-2-hydroxyoxazolium hydroxides derived from the corresponding 5-o..ophenanthridine and 2-oxoquinoline- and 1-oxoisoquinoline-*N*-acetic acids.

The "nonclassical" thiophenes, the thieno[3,4-*c*]thiophene (**1**), and the thieno[3,4-*c*]pyrrole (**2**) systems have only been isolated with aromatic substituents in the 1, 3, 4, and 6 positions.[2] It has been suggested that these aromatic substituents stabilize the system by electron delocalization or by a steric effect or by some combination of both, and in an effort to obtain experimental understanding of the role of these groups, we have attempted to synthesize representatives of the thieno[3,4-*c*]pyrrole system with fewer aromatic substituents. In an earlier study[3] attempted ring closure of 3,4-dibenzoyl-2-phenyl-1-methyl(or 1-phenyl)pyrrole to the corresponding thienopyrrole system was unsuccessful, the corresponding 3,4-bis(thiobenzoyl) derivatives of the pyrroles being obtained. In this present study our aim was to introduce additional conjugation involving one of the aromatic substituents to see whether this more extensive delocalization would allow the isolation of the fused-ring system with essentially three aromatic substituents. Suitable ring systems meeting these requirements would be those based on phenanthridine, quinoline, and isoquinoline and this publication describes our results in this area.

The most direc⁺ route to the desired system **7** involves ring

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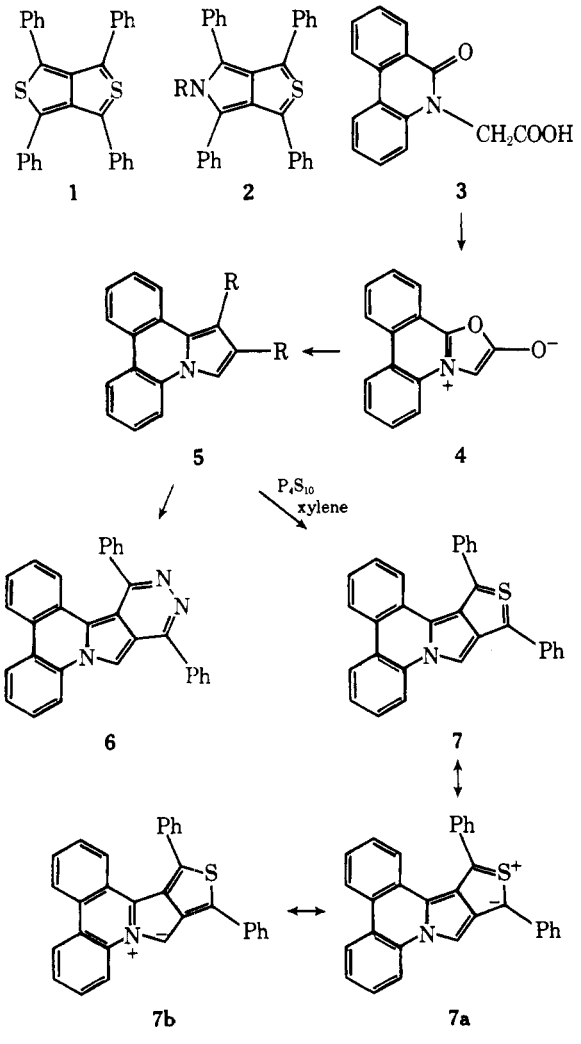
closure of the vicinal dibenzoyl compound **5** with P_4S_{10}, analogous to that used in our earlier work in the "nonclassical" thiophene area.

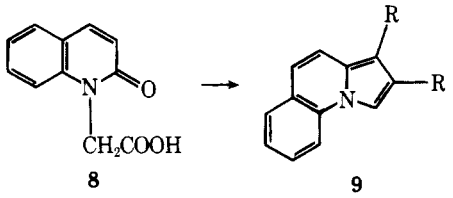
Reaction of 6-oxo-5(6*H*)-phenanthridineacetic acid (**3**) with *N,N'*-dicyclohexylcarbodiimide in hot benzene in the presence of dibenzoylacetylene gave 1,2-dibenzoyldibenzo[*e,g*]pyrrolo[1,2-*a*]pyridine (**5**, R = COPh), the product from the 1,3-dipolar cycloaddition[4] of the acetylene to the intermediate anhydro-2-hydroxyoxazolium hydroxide **4**. This diketone was readily characterized by conversion into the pyridazine **6** and, when heated with P_4S_{10} in refluxing xylene, yielded 1,3-diphenyldibenzo[*g,i*]thieno[3',4':3,4]pyrrolo[1,2-*a*]pyridine-2-S^{IV} (**7**) as purple microneedles in 94% yield. In addition to analytical and spectral data, **7** was characterized by its molecular ion, *m/e* 425 (100), and its doubly charged molecular ion, *m/e* 212.5 (4), characteristic of these systems.[3] This represents the first example of the thieno[3,4-*c*]pyrrole system **2** with the equivalent of only three aromatic substituents attached to the carbon atoms of the nucleus. As with 5-methyl-1,3,4,6-tetraphenylthieno[3,4-*c*]pyrrole-2-S^{IV} (**2**, R = CH_3), **7** was extremely sensitive to oxygen being rapidly bleached in solution and less rapidly in the solid state.

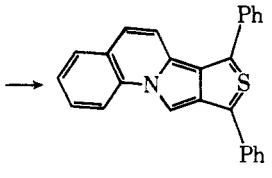
In contrast to the ready cycloaddition of **2** (R = CH_3) with various olefinic and acetylenic dipolarophiles,[3a] **7** did not undergo cycloaddition cleanly with *N*-phenylmaleimide, fumaronitrile, dimethyl acetylenedicarboxylate, or dibenzoylacetylene, multicomponent reaction mixtures being obtained. This no doubt reflects a major contribution from **7b** to the ground state and as a consequence **7** behaves as a phenanthridinium ylide. However, it was not sufficiently basic to undergo protonation or alkylation at the site of the negative charge in **7b**.



Trapping of the intermediate anhydro-2-hydroxyoxazolium hydroxide system **4** with acetylenic dipolarophiles is a convenient route to the ring system **5**. Reaction of **4** with dimethyl acetylenedicarboxylate gave **5** (R = $COOCH_3$) in 52% yield.

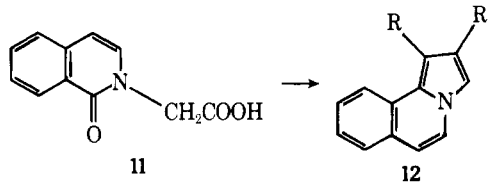
The effect of removing one of the benzene rings on the stability and reactions of **7** was of particular interest. 2(1*H*)-Quinolinone was converted into 2-oxo-1(2*H*)-quinolineacetic acid (**8**) which, with *N,N'*-dicyclohexylcarbodiimide in the presence of dibenzoylacetylene, gave the diketone **9** (R = COPh), characterized also as the pyridazine by reaction with hydrazine. Reaction of **9** (R = COPh) with P_4S_{10} gave 1,3-diphenylbenzo[*g*]thieno[3,4-*a*]indolizine (**10**) as small purple needles in 87% yield. This product was characterized by spectral data, especially $M^{+\cdot}$ 375 (100) and M^{2+} 187.5 (4), but it was too unstable to obtain satisfactory analytical data. On attempted cycloaddition with a variety of dipolarophiles in refluxing benzene, **10** also underwent deep-seated decomposition and no characterizable products were obtained. As would be anticipated, reaction of **8** with dimethyl acetylenedicarboxylate in the presence of DCC gave **9** (R = $COOCH_3$) in good yield. Attempts to introduce a phenyl group into the 4 position of **10** were unsuccessful as it was not possible to

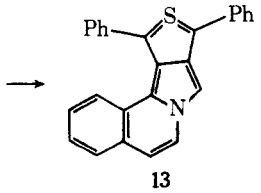




prepare the corresponding phenyl derivative of **8** by N-alkylation of 2(1*H*)-quinolinone with α-bromophenylacetic acid. This appears to be the results of a strong steric interaction with the 8 position of the quinoline system as an attempt in this laboratory to alkylate 2(1*H*)-benzothiazolone with α-bromophenylacetic acid was also unsuccessful.

A similar series of reactions to those used above should also provide access to 1,3-diphenylbenzo[*i*]thieno[3',4':3,4]pyrrolo[1,2-*a*]pyridine-2-S^{IV} (**13**). 1-Oxo-2(1*H*)-isoquinoline acetic acid (**11**) was prepared by alkylation of 1(2*H*)-quinolinone with bromoacetic acid, NaH, and DMF and underwent ready transformation into 1,2-dibenzoylpyrrolo[2,1-*a*]isoquinoline (**12**, R = COPh) on reaction with benzoylacetylene and DCC. The diketone, characterized as the pyridazine derivative, underwent reaction with P_4S_{10} in toluene as evidenced by the characteristic color of the reaction mixture but it was impossible to isolate a homogenous product from the





reaction. This apparently contained some amount of our desired product **13** as the mass spectrum showed $M^{+\cdot}$ 375 (100) and M^{2+} 187.5 (6), together with a relatively intense ion as *m/e* 406 (60). Reaction of **11** occurred readily with dimethyl acetylenedicarboxylate in the presence of DCC giving **12** (R = $COOCH_3$) and providing a convenient method for the annelation of a pyrrole ring to isoquinoline.

Experimental Section[5]

1,2-Dibenzoyldibenzo[*e,g*]pyrrolo[1,2-*a*]pyridine (5, R = COPh). 6-Oxo-5(6*H*)-phenanthridineacetic acid[6] (5.1 g, 0.02 mol), *N,N'*-dicyclohexylcarbodiimide (4.12 g, 0.02 mol), and dibenzoylacetylene (4.7 g, 0.02 mol) were refluxed in benzene (200 mL) for 3 h. *N,N'*-Dicyclohexylurea was filtered from the cooled solution and the solvent then evaporated under vacuum. The yellow residue was digested with methanol affording finely matted, pale yellow needles: 5.5 g (65%); mp 236–237 °C; IR (KBr) 1660, 1650 (CO) cm^{-1}; $M^{+\cdot}$ 425 (100), M^{2+} 212.5 (2).

Anal. Calcd for $C_{30}H_{19}NO_2$: C, 84.68; H, 4.50; N, 3.29. Found: C, 84.86; H, 4.59; N, 3.56.

Excess hydrazine hydrate was added dropwise to a stirred solution of the above ketone (0.1 g, 0.24 mmol) in pyridine (10 mL). After 15 min of reflux the reaction mixture was left overnight and then poured onto ice giving a yellow precipitate. Recrystallization from acetone afforded yellow prisms of **6**: 55 mg (56%); mp 282–283 °C; IR (KBr) 1525 (C=N) cm^{-1}; $M^{+\cdot}$ 421 (100), M^{2+} 210.5 (2).

Anal. Calcd for $C_{30}H_{19}N_3$: C, 85.48; H, 4.54; N, 9.97. Found: C, 85.42; H, 4.71; N, 10.08.

Use of dimethyl acetylenedicarboxylate in the above reaction gave methyl dibenzo[*e,g*]pyrrolo[1,2-*a*]pyridine-1,2-dicarboxylate (**5**, R = $COOCH_3$) in 52% yield, crystallizing from ethanol as colorless plates: mp 178–179 °C; IR (KBr) 1700 (CO) cm^{-1}; $M^{+\cdot}$ 333 (100), M^{2+} 166.5 (6).

Anal. Calcd for $C_{20}H_{15}NO_4$: C, 72.06; H, 4.54; N, 4.20. Found: C, 72.21; H, 4.73; N, 4.25.

1,3-Diphenyldibenzo[*g,i*]thieno[3′,4′:3,4]pyrrolo[1,2-*a*]pyridine-2-S^{IV} (7). A mixture of the diketone **5** (R = COPh) (425 mg, 1 mmol) and P_4S_{10} (222 mg, 1 mmol) was refluxed in dry xylene (10 mL) under N_2 for 2 h. Removal of all solvent under high vacuum left a golden residue which was treated with aqueous sodium hydroxide (20 mL, 10% solution) and the mixture was refluxed with stirring for 1 h. Purple microcrystals of **7** were collected on a sintered funnel and washed thoroughly with cold water: 400 mg (94%); mp 181–182 °C; IR (KBr) 1590 (C=N) cm^{-1}; $M^{+\cdot}$ 425 (100), M^{2+} 212.5 (4).

Anal. Calcd for $C_{30}H_{19}NS$: C, 84.68; H, 4.50; N, 3.29. Found: C, 84.73; H, 4.51; N, 3.39.

2-Oxo-1(2*H*)-quinolineacetic Acid (8). 2-(1*H*)-Quinolinone (1.5 g, 0.01 mol) was added in small portions to a suspension of NaH (0.9 g, 0.023 mol) in dry DMF (25 mL) and the mixture was stirred for 1 h. Sodium bromoacetate (1.6 g, 0.01 mol) was then added and the reaction mixture was stirred at room temperature for 2 h and then at 100 °C for 1 h. The cooled solution was poured into ice water and dilute HCl added. A colorless solid separated which was collected, washed with water, and air dried. Recrystallization from methanol afforded colorless needles: 1.5 g (74%); mp 284–285 °C (lit.[7] mp 282–283 °C); IR (KBr) 1720, 1625 (CO) cm^{-1}; $M^{+\cdot}$ 203 (43).

Anal. Calcd for $C_{11}H_9NO_3$: C, 65.02; H, 4.46; N, 6.89. Found: C, 64.77; H, 4.60; N, 6.91.

1,2-Dibenzoylbenzo[*e*]indolizine (9, R = COPh). A mixture of the acid (1.02 g, 5 mmol), *N,N'*-dicyclohexylcarbodiimide (1.03 g, 5 mmol), and dibenzoylacetylene (1.17 g, 5 mmol) was refluxed in benzene (60 mL) for 2.5 h. The separated *N,N'*-dicyclohexylurea was removed from the cooled solution and the solvent removed in vacuo. Recrystallization of the residue from acetone gave yellow prisms: 0.59 g (31%); mp 222–223 °C; IR (KBr) 1650, 1610 (CO) cm^{-1}; $M^{+\cdot}$ 375 (100), M^{2+} 187.5 (3).

Anal. Calcd for $C_{26}H_{17}NO_2$: C, 83.18; H, 4.56; N, 3.73. Found: C, 83.11; H, 4.52; N, 3.56.

Excess hydrazine hydrate was added to a solution of the above ketone (133 mg, 0.36 mol) in pyridine (10 mL). After 19 h of reflux the reaction mixture was cooled and poured into ice water and the greenish-yellow solid that separated collected and recrystallized from acetone giving microfine, green needles: 57 mg (44%); mp 228–229 °C; IR (KBr) 3050 (CH), 1600 (C=N); $M^{+\cdot}$ 371 (100), M^{2+} 185.5 (5).

Anal. Calcd for $C_{26}H_{17}N_3$: C, 84.07; H, 4.61; N, 11.31. Found: C, 84.09; H, 4.67; N, 11.01.

Use of dimethyl acetylenedicarboxylate in the above reaction gave methyl benzo[*e*]indolizine-1,2-dicarboxylate (**9**, R = $COOCH_3$) in 35% yield. It crystallized from methanol as yellow prisms: mp 134–135 °C; IR (KBr) 1720, 1680 (CO) cm^{-1}; $M^{+\cdot}$ 283 (100), M^{2+} 141.5 (2).

Anal. Calcd for $C_{16}H_{13}NO_4$: C, 67.84; H, 4.63; N, 4.95. Found: C, 67.63; H. 4.48; N, 4.86.

1-Oxo-2(1*H*)-isoquinolineacetic Acid[8] (11). 1(2*H*)-Isoquinolinone[9] (12.9 g, 0.02 mol) was added in small portions to a suspension of NaH (3.1 g, 0.06 mol) in dry DMF (50 mL) and stirred for 30 min. Sodium bromoacetate (3.22 g, 0.02 mol) was then added slowly to the cooled solution at such a rate that the temperature did not rise above 30 °C. After a further hour at room temperature, the reaction mixture was heated at 80 °C for 30 min and the cooled mixture then poured into ice water. After acidification with dilute HCl an oil separated which eventually crystallized: 2.13 g (52%). Crystallization from ethanol (charcoal) gave long, colorless needles: mp 245–246.5 °C; IR (KBr) 1740, 1645 (CO) cm^{-1}; $M^{+\cdot}$ 203 (67), M^{2+} 101.5 (1).

Anal. Calcd for $C_{11}H_9NO_3$: C, 65.02; H, 4.46; N, 6.89. Found: C, 65.08; H, 4.46; N, 7.02.

1,2-Dibenzoylbenzo[*g*]indolizine (12, R = COPh). 1-Oxo-2(1*H*)-isoquinolineacetic acid (1.02 g, 5 mmol), *N,N'*-dicyclohexylcarbodiimide (1.06 g, 5.1 mmol), and dibenzoylacetylene (1.17 g, 5 mmol) was refluxed in dry benzene (70 mL) for 2 h. The dicyclohexylurea was filtered from the orange solution which was then concentrated to give an orange oil which, on trituration with CH_3OH, gave yellow irregular prisms: 1.32 g (71%). Crystallization from benzene yielded bright-yellow needles: mp 195.5–196.5 °C; IR (KBr) 1630 (CO) cm^{-1}; $M^{+\cdot}$ 375 (100); M^{2+} 187.5 (8).

Anal. Calcd for $C_{26}H_{17}NO_2$: C, 83.18; H, 4.56; N, 3.73. Found: C, 83.04; H, 4.50; N, 3.54.

Conversion of **12** (R = COPh) into the pyridazine derivative was effected as above with hydrazine hydrate. It crystallized from $CHCl_3$:CH_3OH affording fine-yellow needles: mp 257.5–258.5 °C; IR (KBr) 1510 (C=N) cm^{-1}; $M^{+\cdot}$ 371 (100), M^{2+} 185.5 (3).

Anal. Calcd for $C_{26}H_{17}N_3$: C, 84.07; H. 4.61; N, 11.31. Found: C, 83.11; H. 4.41; N, 11.17.

Using dimethyl acetylenedicarboxylate in the above reaction gave methyl benzo[*g*]indolizine-1,2-dicarboxylate (**12**, R = $COOCH_3$) as colorless plates on recrystallization from methanol: 39%, mp 136–137 °C; IR (KBr) 1730, 1710 (CO) cm^{-1}; $M^{+\cdot}$ 283 (89), M^{2+} 141.5 (15).

Anal. Calcd for $C_{16}H_{13}NO_4$: C, 67.84; H. 4.63; N, 4.95. Found: C, 67.66; H, 4.57; N, 4.96.

Registry No.—**3,** 37046-34-7; **5** (R = COPh), 68949-98-4; **5** (R = $COOCH_3$), 68949-99-5; **6**, 68950-00-5; **7**, 68950-01-6; **8**, 55170-65-5; **9** (R = COPh), 68950-02-7; **9** (R = $COOCH_3$), 68950-03-8; **9** (R = COPh) pyridazine derivative, 68950-04-9; **10**, 68950-05-0; **11**, 59139-93-4; **12** (R = COPh), 68950-06-1; **12**, (R = $COOCH_3$), 68950-07-2; **12** (R =COPh) pyridazine derivative, 68950-08-3; **13**, 68950-09-4; dibenzoylacetylene, 1087-09-8; dimethyl acetylenedicarboxylate, 762-42-5; 2(1*H*)-quinolinone, 59-31-4; sodium bromoacetate, 1068-52-6; 1(2*H*)-isoquinolinone, 491-30-5.

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(4) This in situ technique has found many applications; see, e.g., ref 3; K. T. Potts, J. Baum, E. Houghton, D. N. Roy, and U. P. Singh, *J. Org. Chem.*, **39**, 3619 (1974); W. K. Anderson and P. F. Corey, *ibid.*, **42**, 559 (1977); F. M. Hershenson, *ibid.*, **37**, 3111 (1972).
(5) Melting points were determined in capillaries and evaporations were carried out under reduced pressure using a Buchi evaporator. Spectral characteristics were determined on the following instrumentation: infrared spectra, Perkin-Elmer Model 337 spectrophotometer; NMR spectra, Varian T-60 spectrometer using Me_4Si as internal standard; mass spectra, Hitachi Perkin-Elmer RMU-6E mass spectrometer, utilizing the direct inlet probe at a source temperature of ca. 160 °C. Microanalyses were performed by Instranal Laboratories, Inc., Rensselaer, N.Y.
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