

Reactions of Some Pyranylidene Esters with Amines

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The reactions of 4-(2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2,6-diphenyl-4*H*-pyran (**1**) with primary amines gave the corresponding 1-substituted 1,4-dihydropyridine derivatives. The related benzo derivative of **1** (**12**) and primary amines gave 3-substituted 3,4-dihydro-2-phenyl-5*H*-[1]benzopyrano[3,4-*c*]pyridine-4,5-dione derivatives. With secondary amines, **12** gave 2-phenyl-4*H*,5*H*-pyrano[3,4-*c*] [1]benzopyrane-4,5-dione, and with isopropylamine, *N,N*-dimethylhydrazine, and methanolic potassium hydroxide, **12** gave 4-phenacylcoumarin. Some reaction intermediates were isolated which indicate probable reaction paths. The reactions with amines were extended to a naphtho derivative of **1** (**19**) and to a thia homolog of **12** (**24**).

The reactions of 4-dicyanomethylene-4*H*-pyran with amines and certain other nucleophiles have been described (1). The present paper reports our results for similar reactions with the pyranylidene derivatives **1**, **12**, and **19**, and the thiopyranylidene derivative **24**.

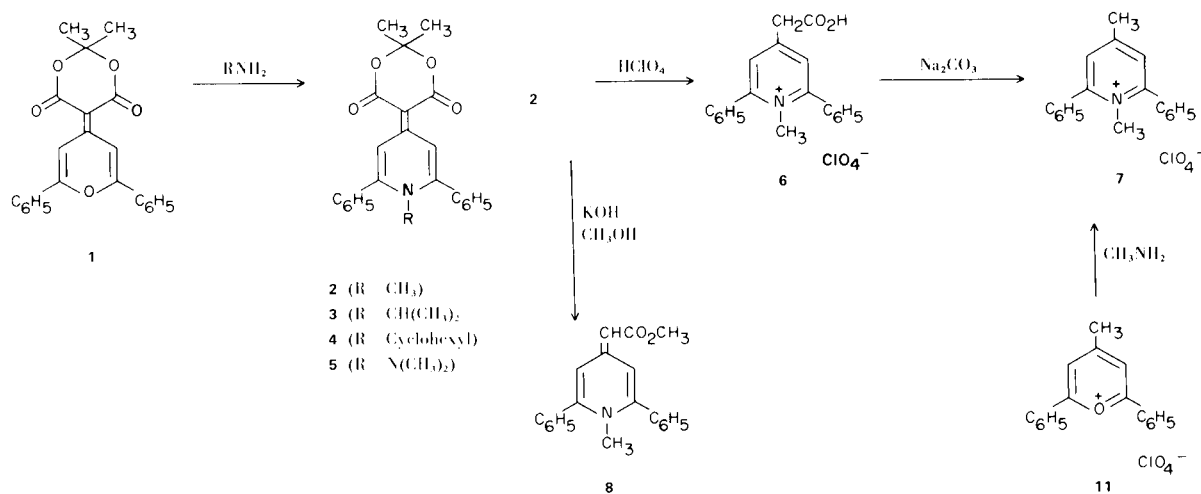
Compound **1** reacted with methylamine, isopropylamine, cyclohexylamine, and *N,N*-dimethylhydrazine to give the 1,4-dihydropyridine derivatives **2-5** (Scheme I). With perchloric acid, **2** gave the pyridinium salt **6** which on treatment with sodium carbonate yielded 1,4-dimethyl-2,6-diphenylpyridinium perchlorate (**7**). Compound **7** was also prepared from 4-methyl-2,6-diphenylpyrylium perchlorate and methylamine. This sequence of reactions establishes the structure of **2**, and since the electronic spectra of **3-5** are similar to that of **2**, the relationship

of these compounds is demonstrated. Treatment of **2** with methanolic potassium hydroxide gave the ester **8**. Eiden and Peter have described reactions which are similar to the conversion of **1** to **2** (**2**).

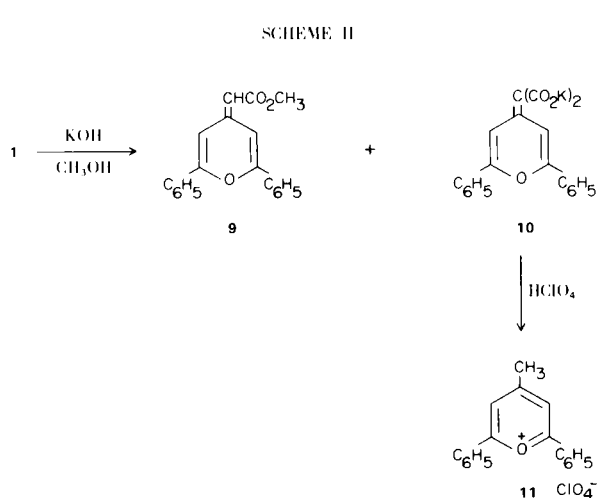
Compound **1** reacted with methanolic potassium hydroxide to give a mixture of **9** and **10**, due to attack on the dioxane ring by methoxide and hydroxide ions (Scheme II). Perchloric acid converted **10** to 4-methyl-2,6-diphenylpyrylium perchlorate (**11**).

The benzopyranylidene derivative **12** reacted with methylamine and benzylamine to give the monolactones **13** and **14** (Scheme III). With the secondary amines, dimethylamine, piperidine or pyrrolidine **12** gave the dilactone **15**. No reaction occurred between **12** and triethylamine. Isopropylamine, *N,N*-dimethylhydrazine and

SCHEME I

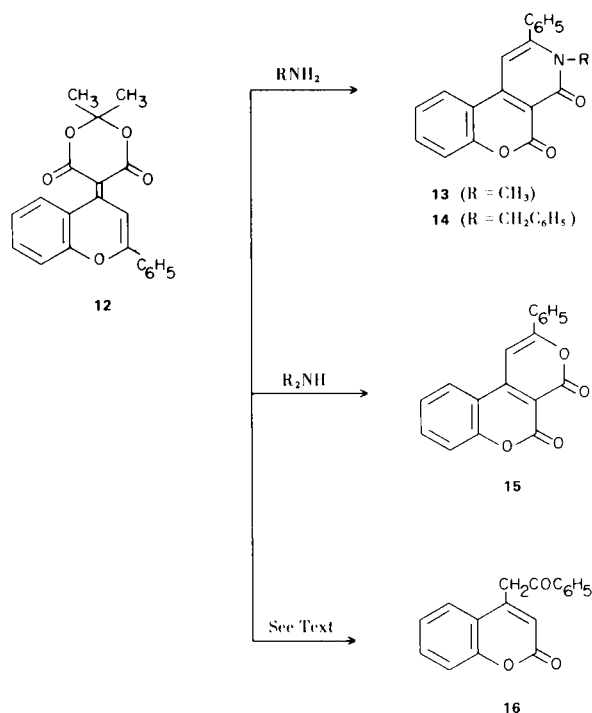


SCHEME II

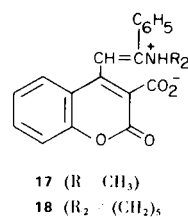


methanolic potassium hydroxide reacted with **12** to give 4-phenacylcoumarin (**16**). In the case of dimethylhydrazine, the dimethylhydrazone of **16** was isolated as an

SCHEME III

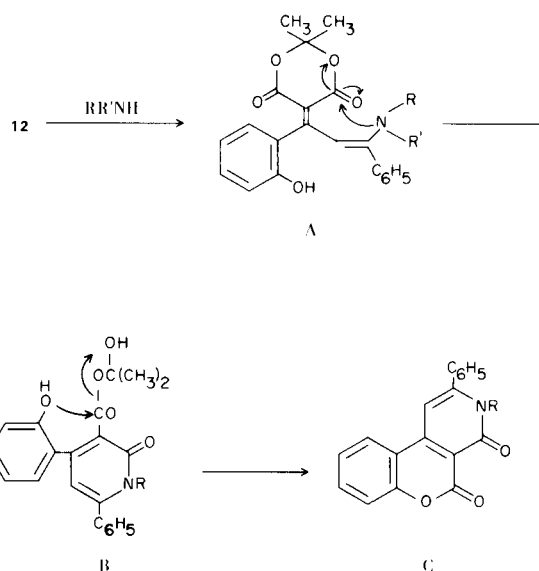


intermediate. Compound **12** reacted with dimethylamine and piperidine to give intermediates which we believe to have the structures **17** and **18**. These intermediates were readily converted to **15** by acetic acid, recrystallization from alcohol, or heat. Addition of some of the secondary amine permitted the crystallization of **17** and **18** from alcohol without change.



The isolation of these intermediates offered a clue to the mechanism of the reaction of **12** with amines. The following rationale (Scheme IV) is suggested for the sequence of reactions involved. For all amines, the primary

SCHEME IV

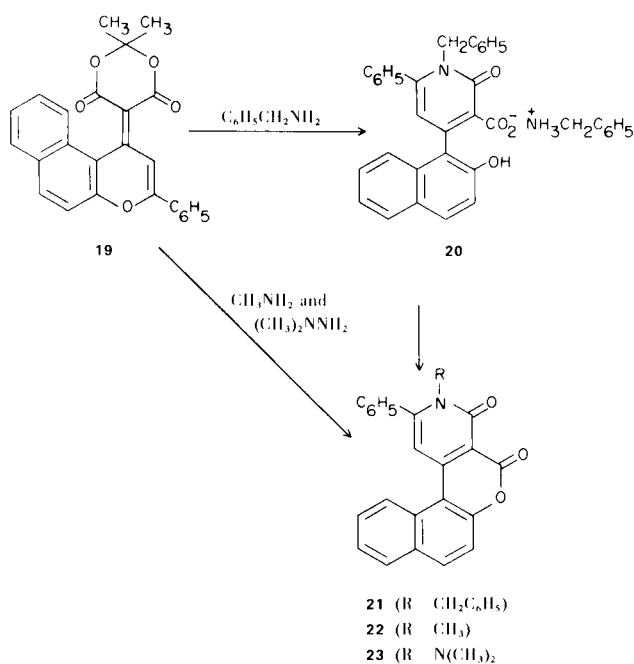


step was the addition of the amine to the 2-position of the pyran to give A. When R' was hydrogen (*i.e.*, when the nucleophile was a primary amine), cyclization occurred to give B and then C. For the cases with secondary amines, cyclization of the adduct A through the amino function was not possible, and ring closure occurred *via* the phenolic oxygen to give intermediates such as **17** and **18**. On warming, **17** and **18** underwent internal nucleophilic displacement of the amine groups by the carboxylate oxygen to give a dilactone. We believe that sterically hindered amines (isopropylamine and dimethylhydrazine) formed compounds similar to **17** and **18**, but the cyclization of the carboxylate ion was so retarded that competing decarboxylation and hydrolysis of the enamine took place giving **16**.

The reactions of the naphthopyranylidene derivative **19** were similar to those described for **12** (Scheme V). Thus **19** and benzylamine gave the intermediate **20**, which was readily converted to **21** with heat or acid. Methylamine

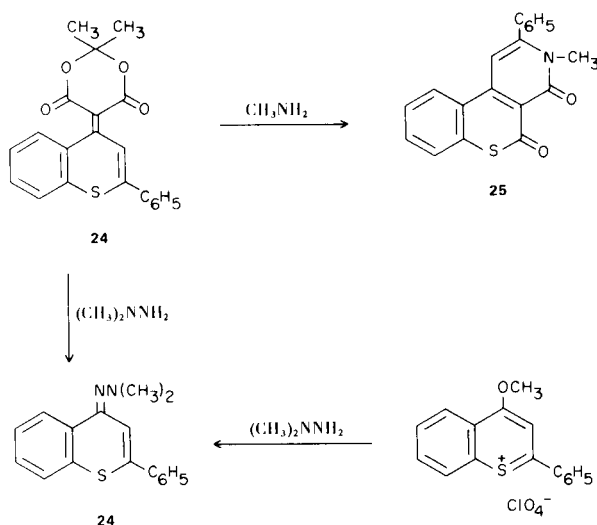
and dimethylhydrazine reacted with **19** to give **22** and **23** directly. We are unable to account for the different products that were obtained from the reaction of dimethylhydrazine with **12** and **19**.

SCHEME V



The thiopyranlydene derivative **24** reacted with methylamine in a similar fashion to the corresponding pyranlydene derivative **12** to give **25** (Scheme VI). However, **24** reacted with *N,N*-dimethylhydrazine to give the hydrazone **26**, which was also prepared from dimethylhydrazine and 4-methoxythioflavylium perchlorate.

SCHEME VI



EXPERIMENTAL (3)

4-(2,2-Dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2,6-diphenyl-4H-pyran (**1**).

A mixture of 0.01 mole of 4-methoxy-2,6-diphenylpyrylium perchlorate, 1.5 g. of 2,2-dimethyl-1,3-dioxane-4,6-dione (Mel-drum's acid), 2 ml. of diisopropylethylamine, and 100 ml. of alcohol was heated to boiling and the solution was cooled. The solid was collected and crystallized from alcohol yielding 3.3 g. of **1**, m.p. 247-248°.

Anal. Calcd. for $\text{C}_{23}\text{H}_{18}\text{O}_5$: C, 73.8; H, 4.9. Found: C, 73.8; H, 5.0.

1,4-Dihydro-1-methyl-4-(2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2,6-diphenylpyridine (**2**).

A solution of 0.01 mole of **1**, 5 ml. of 40% aqueous methylamine, and 15 ml. of alcohol was refluxed for 1 hour and then poured into dilute acetic acid. The solid was collected and crystallized from alcohol giving 3 g. of **2**, m.p. 259-260°. The λ_{max} ($\epsilon \times 10^{-3}$) are 245 (22.4), ~270 (11.7) and 366 nm (38.2). Some important *m/e* for **2** are: 387 (M) [53%], 330 (M- CH_3COCH_2) [3%], 285 (M-(CH_3) $_2\text{CO}_2\text{CO}$) [100%], and 139 [6%].

Anal. Calcd. for $\text{C}_{24}\text{H}_{21}\text{NO}_4$: C, 74.4; H, 5.5; N, 3.6. Found: C, 74.2; H, 5.6; N, 3.6.

1,4-Dihydro-1-isopropyl-4-(2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2,6-diphenylpyridine (**3**).

A mixture of 0.01 mole of **1** and 40 ml. of isopropylamine was refluxed for 8 hours, chilled, and the solid was collected and crystallized from butyl alcohol giving 1.7 g. of **3**, m.p. 232-233°. The λ_{max} ($\epsilon \times 10^{-3}$) are 245 (21.2), ~270 (8.7), and 366 nm (39.4). Some *m/e* are: 415 (M) [61%], 373 (M- C_3H_6) [2.2%], 316 (373- CH_3COCH_2) [8.3%], 313 (M-(CH_3) $_2\text{CO}_2\text{CO}$) [33%], 271 (313- C_3H_6) [100%], and 139 [25%].

Anal. Calcd. for $\text{C}_{26}\text{H}_{25}\text{NO}_4$: C, 75.2; H, 6.1; N, 3.4. Found: C, 75.5; H, 6.3; N, 3.5.

1-Cyclohexyl-1,4-dihydro-4-(2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2,6-diphenylpyridine (**4**).

Compound **4** was prepared by the procedure described for **3** using cyclohexylamine. The yield of product was 22%; m.p. 284-285° from alcohol. The λ_{max} ($\epsilon \times 10^{-3}$) are: 245 (22.0), ~270 (8.0), and 365 nm (44.0). Some *m/e* are: 455 (M) [19%], 373 (M- C_6H_{10}) [10%], 315 (*m/e* 372- CH_3COCH_2) [11%], 353 (M-(CH_3) $_2\text{CO}_2\text{CO}$) [6%], 271 (*m/e* 353- C_6H_{10}) [100%], and 139 [11%].

Anal. Calcd. for $\text{C}_{29}\text{H}_{29}\text{NO}_4$: C, 76.5; H, 6.4; N, 3.1. Found: C, 76.7; H, 6.5; N, 3.3.

1-Dimethylamino-1,4-dihydro-4-(2,2-dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2,6-diphenylpyridine (**5**).

A mixture of 0.01 mole of **1**, 5 ml. of *N,N*-dimethylhydrazine, and 15 ml. of alcohol was refluxed for 7 hours and worked up as described for **2** giving 2.8 g. of **5** (from alcohol), m.p. 216-217°. The λ_{max} ($\epsilon \times 10^{-3}$) are 245 (21.8), ~274 (10.0), and 368 nm (40.0). The mass spectral cracking pattern is analogous to that of compounds **2-4**.

Anal. Calcd. for $\text{C}_{25}\text{H}_{24}\text{N}_2\text{O}_4$: C, 72.1; H, 5.8; N, 6.7. Found: C, 71.9; H, 5.7; N, 6.8.

4-Carboxymethyl-2,6-diphenyl-1-methylpyridinium Perchlorate (**6**).

A solution of 0.01 mole of **2**, 8 ml. of 70% perchloric acid,

and 30 ml. of methyl alcohol was refluxed for 10 minutes, cooled, diluted with ether, and the solid was collected, yielding 1.7 g. of **6**, m.p. 105-107°.

Anal. Calcd. for $C_{20}H_{18}ClNO_6$: C, 59.5; H, 4.5; Cl, 8.8. Found: C, 59.8; H, 4.7; Cl, 8.6.

1,4-Dimethyl-2,6-diphenylpyridinium Perchlorate (7).

(A) A solution of 1 g. of **6** in 20 ml. of 5% sodium carbonate was heated to boiling and the solid that separated was collected and crystallized from ethoxyethanol giving 0.5 g. of **7**, m.p. 230-231°.

(B) A mixture of 2 g. of 4-methyl-2,6-diphenylpyridium perchlorate, 5 ml. of 40% aqueous methylamine, and 10 ml. of alcohol was refluxed on a steam bath for 1 hour, cooled, and the solid was collected and crystallized from ethoxyethanol giving 1.8 g. of **7**, m.p. 230-231°, which shows its absorption identical with that of the product obtained by procedure (A).

Anal. Calcd. for $C_{19}H_{18}ClNO_4$: C, 63.4; H, 5.0; N, 3.9. Found: C, 63.5; H, 5.0; N, 3.9.

4-Carbomethoxymethylene-1,4-dihydro-1-methyl-2,6-diphenylpyridine (8).

A mixture of 2 g. of **2** and 25 ml. of 10% methanolic potassium hydroxide was refluxed for 4 hours, diluted with aqueous acetic acid, and the solution treated with Norite and filtered. The filtrate was made basic with sodium carbonate, and the yellow solid was collected and crystallized from alcohol giving 1.6 g. of **8**, m.p. 177-178°. Some m/e are: 317 (M+) [86%], 286 (M-OCH₃) [86%] and 259 (M-CO₂CH₂) [100%].

Anal. Calcd. for $C_{21}H_{19}NO_2$: C, 79.5; H, 6.0; N, 4.4. Found: C, 79.5; H, 5.9; N, 4.6.

4-Carbomethoxymethylene-2,6-diphenyl-4H-pyran (9).

A mixture of 3 g. of **1** and 80 ml. of 10% methanolic potassium hydroxide was heated on a steam bath for 0.5 hour, cooled, and the solid (**10**) was collected. The filtrate was added to dilute acetic acid and after chilling, the solid was collected and crystallized from alcohol yielding 1.5 g. of **9**, m.p. 113-114°. Some m/e are: 304 (M+), 273 (M-OCH₃), and 246 (M-CO₂CH₂). The λ max ($\epsilon \times 10^{-3}$) are: 253 (18.4), 260 (19.1) and 352 nm (28.1).

Anal. Calcd. for $C_{20}H_{16}O_3$: C, 78.9; H, 5.3. Found: C, 78.9; H, 5.3.

Dipotassium Salt of 4-Dicarboxymethylene-2,6-diphenyl-4H-pyran (10).

The solid that was isolated from the reaction mixture during the preparation of **9** was **10**; yield 0.6 g., m.p. above 300°.

Anal. Calcd. for $C_{20}H_{12}K_2O_5 \cdot 2H_2O$: C, 53.8; H, 3.6; K, 17.5. Found: C, 54.1; H, 3.9; K, 18.0.

4-Methyl-2,6-diphenylpyridium Perchlorate (11).

To a solution of 0.5 g. of **10** in 10 ml. of water was added 2 ml. of 70% perchloric acid; the solid was collected and crystallized from acetonitrile giving 0.2 g. of **11**, m.p. 271-273°. The m.p. and infrared spectrum of this compound were identical with those of a sample which was prepared as described previously (4).

4-(2,2-Dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2-phenyl-4H-[1]-benzopyran (12).

A mixture of 3.5 g. of 4-methoxyflavylium perchlorate, 4 g. of Meldrum's acid, 10 ml. of diisopropylethylamine, and 200 ml. of alcohol was refluxed for 0.5 hour, chilled, and the solid was collected and crystallized from ethoxyethanol giving 3.3 g. of **12**, m.p. 260-261°.

Anal. Calcd. for $C_{21}H_{16}O_5$: C, 72.4; H, 4.6. Found: C, 72.2; H, 4.8.

3,4-Dihydro-3-methyl-2-phenyl-5H-[1]benzopyrano[3,4-c]pyridine-4,5-dione (13).

A mixture of 3.5 g. of **12**, 10 ml. of 40% aqueous methylamine, and 25 ml. of alcohol was refluxed for 2 hours and diluted with aqueous acetic acid. The solid was collected and crystallized from alcohol yielding 3 g. of **13**, m.p. 265-266°. Some m/e are: 303 (M+) [50%], 302 (M-H) [100%], 274 (M-HCO) [20%] and 118 (C₆H₅C-NCH₃) [10%]. The λ max ($\epsilon \times 10^{-3}$) are 258 (22.6), 286 (8.3), and 370 nm (14.9).

Anal. Calcd. for $C_{19}H_{13}NO_3$: C, 75.2; H, 4.3; N, 4.6. Found: C, 74.9; H, 4.0; N, 4.8.

3-Benzyl-3,4-dihydro-2-phenyl-5H-[1]benzopyrano[3,4-c]pyridine-4,5-dione (14).

A solution of 3.5 g. of **12** and 25 ml. of benzylamine was boiled until the red color was nearly gone, and after cooling, the solid was collected and crystallized from ethoxyethanol giving 3.1 g. of **14**, m.p. 214-215°. Some m/e are: 379 (M+) [37%], 378 (M-H) [100%], 302 (M-HCO) [1%], 118 (C₆H₅C-NCH₃) [11%], and 91 (C₆H₅CH₂) [37%]. The λ max ($\epsilon \times 10^{-3}$) are 255 (23.0), 288 (10.0), and 350-370 nm (14.0).

Anal. Calcd. for $C_{25}H_{17}NO_3$: C, 79.1; H, 4.5; N, 3.7. Found: C, 78.8; H, 4.5; N, 3.8.

2-Phenyl-4H,5H-pyrano[3,4-c][1]benzopyran-4,5-dione (15).

A mixture of 3.5 g. of **12**, 3 ml. of either dimethylamine or piperidine, and 25 ml. of alcohol was refluxed for 1 hour, chilled, and the solid collected and crystallized from alcohol yielding 2.3 g. of **15**, m.p. 304-305°. Some m/e are: 290 (M+) [100%], 262 (M-HCO) [43%], and 105 (C₆H₅CO) [100%]. The λ max ($\epsilon \times 10^{-3}$) are 262 (5.7), 305 (2.7), and 355-370 nm (4.8).

Anal. Calcd. for $C_{18}H_{10}O_4$: C, 74.5; H, 3.5. Found: C, 74.3; H, 3.6.

4-Phenacylcoumarin (16).

(A) A mixture of 3.5 g. of **12**, 4 ml. of dimethylhydrazine, and 20 ml. of alcohol was refluxed for 2 hours, added to dilute acetic acid, and heated for 5 minutes. The solid was collected and crystallized from dimethylformamide giving 2.4 g. of **16**, m.p. 185-186°.

(B) The reaction was carried out as described in A except the dilute acetic acid was not added to the reaction mixtures. The hot solution was diluted with water, chilled and the solid was collected and crystallized from alcohol yielding 2.8 g. of the *N,N*-dimethylhydrazone of **16**, m.p. 110-111°.

Anal. Calcd. for $C_{19}H_{18}N_2O_2$: C, 74.5; H, 5.9; N, 9.1. Found: C, 74.6; H, 6.2; N, 9.0.

A sample of the hydrazone was recrystallized from aqueous acetic acid and gave **16**.

(C) The reaction was carried out as described in (A) using 4 ml. of isopropylamine. The yield of **16** was 2.2 g., m.p. 185-186°. When the method described in B was used, an oil was obtained which was hydrolyzed by aqueous acetic acid to give **16**.

(D) A mixture of 3.5 g. of **12** and 80 ml. of 10% methanolic potassium hydroxide was allowed to stand for 1 hour and then refluxed for 0.5 hour. The solution was poured into dilute acetic acid and the solid was collected and crystallized giving 2.1 g. of **16**, m.p. 186-187°. Some m/e are 264 (M+), 236 (M-CO), 131, 115, and 105 (C₆H₅CO). The nmr spectrum in deuteriochloroform shows ArCOCH₂ at δ 4.84 (s, 2H), proton in 3-position of

coumarin at δ 6.53 (S, 1H), and aromatic protons at δ 7.48-8.25 ppm (M, 9H).

Anal. Calcd. for $C_{17}H_{12}O_3$: C, 77.3; H, 4.6. Found: C, 77.4; H, 4.6.

3-Carboxy-4-(α -dimethylaminostyryl)-2H-[1]benzopyran-2-one (17).

The reaction was carried out by the procedure described for the preparation of **15** using dimethylamine, except that the product was crystallized from alcohol containing 2 ml. of the secondary amine. The yield of **17** was 1.6 g., m.p. 304-305°.

Anal. Calcd. for $C_{20}H_{17}NO_4$: C, 71.6; H, 5.1; N, 4.2. Found: C, 71.7; H, 5.1; N, 4.2.

3-Carboxy-4-(α -piperidinostyryl)-2H-[1]benzopyran-2-one (18).

Compound **18** was prepared as described for the synthesis of **17**, and the yield was 1.8 g., m.p. 301-302°.

Anal. Calcd. for $C_{23}H_{21}NO_4$: C, 73.6; H, 5.7; N, 3.7. Found: C, 73.7; H, 5.8; N, 3.7.

4-(2,2-Dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2-phenyl-4H-naphtho-[2,1-*b*]pyran.

A mixture of 0.1 mole of 1-methoxy-3-phenylnaphtho[2,1-*b*]pyrylium perchlorate, 11 g. of Meldrum's acid, 20 ml. of diisopropylethylamine, and 150 ml. of acetonitrile was refluxed for 0.5 hour, chilled, and the solid collected and crystallized from dimethylformamide yielding 2.2 g. of **19**, m.p. 224-225°.

Anal. Calcd. for $C_{25}H_{18}O_5$: C, 75.4; H, 4.6. Found: C, 75.1; H, 4.7.

Benzylamine Salt of 1-Benzyl-1,2-dihydro-4-(2-hydroxy-1-naphthyl)-2-oxo-6-phenylpyridine-3-carboxylic Acid (20).

A mixture of 2 g. of **19** and 5 ml. of benzylamine was heated to boiling for 5 minutes, cooled, diluted with alcohol, and the solid was collected giving 1.5 g. of **20**, m.p. 192-193°. The mass spectrum did not show the parent peak but did show peaks for **21** and benzylamine. The λ max ($\epsilon \times 10^{-3}$) are 275 (6.8), 290 (6.1), and 335 nm (14.9).

Anal. Calcd. for $C_{36}H_{30}N_2O_4$: C, 77.9; H, 5.5; N, 5.1. Found: C, 77.9; H, 5.4; N, 5.1.

3-Benzyl-4,5-dihydro-2-phenylnaphtho[1',2':5,6]pyrano[3,4-*c*]pyridine-4,5-dione (21).

A mixture of 2 g. of **19**, 4 ml. of benzylamine, and 20 ml. of alcohol was refluxed for 15 minutes, chilled, and the solid was collected and crystallized from alcohol yielding 1.1 g. of **21**, m.p. 152-153°. The λ max ($\epsilon \times 10^{-3}$) are 228 (59.5), 270 (7.8), 310 (18.6), and 335 nm (10.0).

Anal. Calcd. for $C_{29}H_{19}NO_3$: C, 81.1; H, 4.5; N, 3.3. Found: C, 81.4; H, 4.2; N, 3.3.

4,5-Dihydro-3-methyl-2-phenylnaphtho[1',2':5,6]pyrano[3,4-*c*]pyridine-4,5-dione (22).

Compound **22** was prepared by the procedure described for the preparation of **21** using methylamine. The product was crystallized from dimethylformamide and was obtained in 57% yield, m.p. 305-306°. The λ max ($\epsilon \times 10^{-3}$) are 232 (22.3), 256 (5.8), 274 (5.0) and 370 nm (8.9).

Anal. Calcd. for $C_{23}H_{15}NO_3$: C, 78.2; H, 4.3; N, 4.0. Found: C, 78.4; H, 4.4; N, 4.0.

3-Dimethylamino-3,4-dihydro-2-phenylnaphtho[1',2':5,6]pyrano-

[3,4-*c*]pyridine-4,5-dione (23).

Compound **23** was prepared from **19** and dimethylhydrazine by the method described for **21**, and was obtained in 78% yield, m.p. 319-320° (from dimethylformamide).

Anal. Calcd. for $C_{24}H_{18}N_2O_3$: C, 75.4; H, 4.8; N, 7.3. Found: C, 75.2; H, 4.7; N, 7.4.

4-(2,2-Dimethyl-4,6-dioxo-1,3-dioxan-5-ylidene)-2-phenyl-4H-[1]benzothiopyran (24).

A mixture of 0.01 mole of 4-methoxythioflavylum perchlorate, 2 g. of Meldrum's acid, 3 ml. of diisopropylethylamine, and 25 ml. of alcohol was refluxed for 0.5 hour, cooled, and the solid was crystallized from ethoxyethanol yielding 2.8 g. of **24**, m.p. 200-201°.

Anal. Calcd. for $C_{21}H_{16}SO_4$: C, 69.2; H, 4.4; S, 8.8. Found: C, 69.0; H, 4.4; S, 9.1.

3,4-Dihydro-3-methyl-2-phenyl-5H-[1]benzothiopyrano[3,4-*c*]pyridine-4,5-dione (25).

A mixture of 3 g. of **24**, 10 ml. of 40% aqueous methylamine, and 40 ml. of alcohol was refluxed for 5 hours, poured into dilute aqueous acetic acid, and the solid was collected and crystallized from alcohol giving 1.1 g. of **25**, m.p. 259-260°.

Anal. Calcd. for $C_{19}H_{13}NO_2S$: C, 71.5; H, 4.1; N, 4.4. Found: C, 71.6; H, 4.2; N, 4.5.

4-Thioflavone *N,N*-dimethylhydrazone (26).

(A) Compound **26** was prepared by the procedure described for **25** using *N,N*-dimethylhydrazine. The yield was 90% of product melting at 111-113° (from butyl alcohol).

Anal. Calcd. for $C_{17}H_{16}N_2S$: C, 72.8; H, 5.8; N, 10.0. Found: C, 72.8; H, 6.0; N, 9.7.

(B) A mixture of 0.5 g. of 4-methoxythioflavylum perchlorate, 2 ml. of *N,N*-dimethylhydrazine and 10 ml. of alcohol was refluxed for 1 hour, poured into dilute acetic acid, and the solid was collected and crystallized from ethoxyethanol giving 0.5 g. of solid, m.p. 111-113°; its ir spectrum was identical with that of the product obtained by method (A). The mass spectrum of **26** shows M^+ at 280, m/e at 265 ($M-CH_3$), m/e at 250 ($265-CH_3$), m/e at 222 ($M-N_2(CH_3)_2$), as well as peaks at 221, 140, 120, 103, and 102. The nmr spectrum in deuteriochloroform at 60 MHz shows a singlet at δ 2.73 ppm (6H) for the methyl groups and a multiplet at δ 7.32-7.9 ppm (10H) for the other protons.

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