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SYNTHESIS OF FUNCTIONALLY SUBSTITUTED BENZO[c]PYRYLIUM SALTS

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Benzo[c]pyrylium salts that contain an acetyl, cyano, or ethoxycarbonyl group in the 4 position of the pyrylium ring were synthesized by the reaction of 3-(3,4-dimethoxyphenyl)pentane-2,4-dione and α-(3,4-dimethoxyphenyl)acetoacetonitrile and ethyl α-(3,4-dimethoxyphenyl)acetoacetate, as well as ethyl α-(3,4-dimethoxyphenyl)benzoylacetate, with acyl perchlorates. It is shown that α-(3,4-dimethoxyphenyl)acetoacetonitrile ethyleneketal is converted to 3-amino-4-acetylbenzo(c)pyrylium salts under these conditions.

The advances recently made in the development of methods for obtaining functionally substituted pyrylium salts have made it possible to significantly expand the synthetic possibilities of this interesting class of heteroaromatic cations. Thus unknown (in the pyrylium series) ring-contraction reactions with the formation of furan, thiophene, and benzofuran dereivatives have been accomplished on the basis of 4-chloromethylpyrylium and 4-chloromethylbenzo[b]pyrylium salts [1-5]. An unusual recycliation leading to the one-step synthesis of derivatives of the tricyclic pyrazolo[5,4-c]isoquinoline system was observed in the reaction of 4-cyano-3-phenylbenzo[c]pyrylium salt with hydrazine [6]. In our opinion, benzo[c]pyrylium salts are of greatest interest for such syntheses in view of the ease of their conversion to isoquinoline derivatives, which are promising from the point of view of the search for new biologically active compounds.

In the present paper we report the preparation of the previously unknown functionally substituted benzo[c]pyrylium salts by the acid-catalyzed acylation of β-oxoalkyl derivatives of benzene [7].

As starting compounds we used 3-(3,4-dimethoxyphenyl)-pentane-2,4-dione (I) and α-(3,4-dimethoxyphenyl)acetoacetonitrile (IIa) and ethyl α-(3,4-dimethoxyphenyl)acetoacete (IIIa), as well as ethyl α-(3,4-dimethoxyphenyl)benzoylacetate (IIIb).

β-Diketone I was obtained by the reaction of arenediazonium salts with acetylacetone, which proceeds via a free-radical mechanism in the presence of copper compounds as catalysts [8].

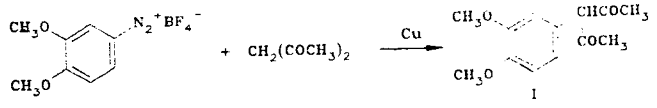

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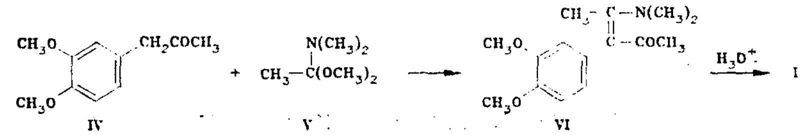
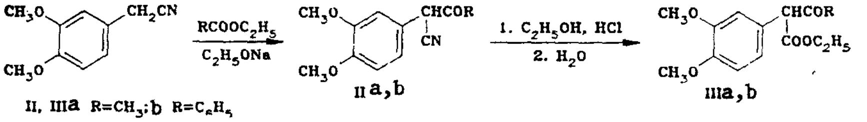
TABLE 1. Characteristics of the Synthesized Compounds

Compound	Empirical formula	mp, °C	Yield, %
VIIa	$C_{15}H_{17}ClO_8$	201...203	51
VIIb	$C_{16}H_{19}ClO_8$	197...199	53
VIIc	$C_{17}H_{21}ClO_8$	190...192	42
IXa	$C_{15}H_{18}ClNO_8$	183...184	20
IXb	$C_{16}H_{20}ClNO_8$	192...193	18
Xa	$C_{14}H_{14}ClNO_7$	210...211	60
Xb	$C_{15}H_{16}ClNO_7$	205...206	35
Xc	$C_{16}H_{18}ClNO_7$	185...187	30
XIa	$C_{14}H_{16}ClNO_8$	203...205	60
XIb	$C_{15}H_{18}ClNO_8$	176...178	42
XIc	$C_{16}H_{20}ClNO_8$	225...227	25
XVIa	$C_{16}H_{19}ClO_9$	212...214	64
XVIb	$C_{17}H_{21}ClO_9$	203...205	59
XVIc	$C_{18}H_{23}ClO_9$	194...196	28
XVId	$C_{21}H_{21}ClO_9$	237...239	32
XVIe	$C_{22}H_{23}ClO_9$	228...230	35
XVIf	$C_{23}H_{25}ClO_9$	220...221	34

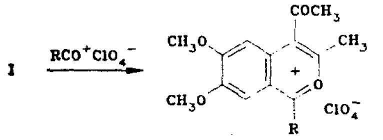
Because of the low yield of reaction product I (21%) we attempted to synthesize this compound by condensation of 3,4-dimethoxyphenylacetone (IV) with dimethylacetamide dimethylacetal (V) with subsequent acidic hydrolysis of β-enamino ketone VI. However, the yield of diketone I did not exceed 35% in this case also.



β-Keto esters IIIa,b were obtained by alcoholysis of the corresponding β-keto nitriles IIa,b, which are formed, in turn, by Claisen acylation of 3,4-dimethoxyphenylacetonitrile.



The synthesis of the pyrylium salts was accomplished by treatment of I-III with acyl perchlorates generated by mixing the carboxylic acid anhydrides with 70% perchloric acid; β-diketone I is converted to 40-50% yield to 4-acetyl-substituted benzo[c]pyrylium salts VIIa-c (Table 1).



VIIa $R=CH_3$, b $R=C_2H_5$; c $R=C_3H_7$

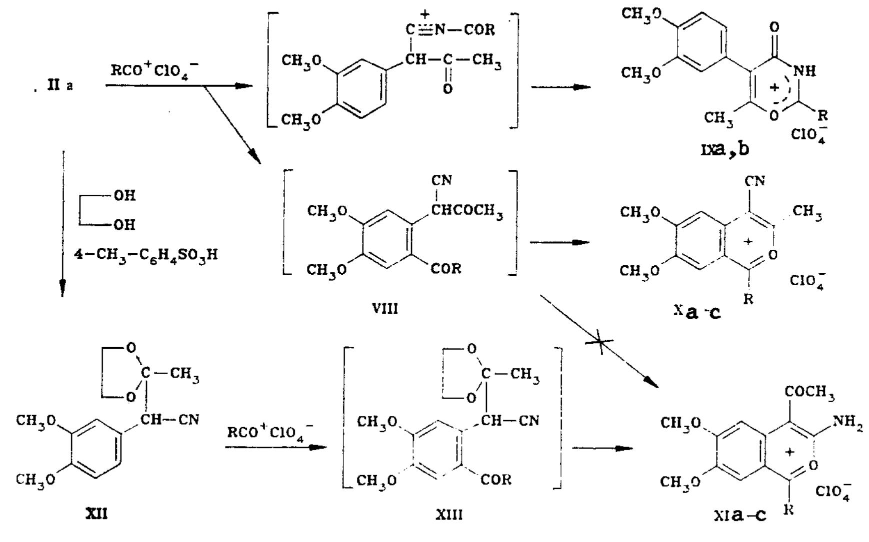
The IR spectra of perchlorates VIIa-c are characterized by absorption bands of an acetyl group (1700-1720 cm^{-1}), a pyrylium cation (1620-1630 cm^{-1}), and the ClO_4^- anion (1100 cm^{-1}). Data from the PMR spectra are presented in Table 2.

In the case of β-keto nitrile IIa the presence in its molecule of two nucleophilic centers that are capable of reacting with acylium ions — the aromatic ring and the nitrogen atom of the cyano group — and the existence of two alternative pathways for heterocyclization of intermediate VII [9, 10] make it possible to assume that either individual salts IX and X or XI or mixtures of them may be products of the reaction under consideration.

It was found that the only product isolated from the reaction mixture in the acylation of β-keto nitrile IIa with a mixture of acetic anhydride and perchloric acid is 1,3-dimethyl-4-cyano-6,7-dimethoxybenzo[c]pyrylium perchlorate (Xa). Absorption bands of a cyano group (2240 cm^{-1}), a pyrylium ring (1630 cm^{-1}), and the ClO_4^- anion (1100 cm^{-1}) are observed in its IR spectrum. The PMR spectrum contains two singlets at 3.05 and 3.30 ppm, which belong to methyl groups in the 1 and 3 positions, two singlets of protons of methoxy groups at 4.15 and 4.35 ppm, and signals of two protons of an aromatic ring at 7.15 and 7.35 ppm.

TABLE 2. PMR spectra of Perchlorates VII, IX-XI, and XVI

Compound	PMR spectrum (CF_3COOH), δ, ppm
VIIa	2,75 (3H, s, CH_3); 2,78 (3H, s, CH_3); 3,23, (3H, s, CH_3); 4,10 (3H, s, OCH_3); 4,15 (3H, s, OCH_3); 7,17 (1H, s, H arom); 7,67 (1H, s, H arom)
VIIb	1,51 (3H, t, CH_3); 2,67 (3H, s, CH_3); 2,72 (3H, s, CH_3); 3,45 (2H, q, CH_2); 3,97 (3H, s, OCH_3); 4,02 (3H, s, OCH_3); 6,97 (1H, s, H arom); 7,50 (1H, s, H arom)
VIIc	1,01 (3H, t, CH_3); 1,89 (2H, m, CH_2); 2,60 (3H, s, CH_3); 2,67 (3H, s, CH_3); 3,38 (2H, t, CH_2); 3,90 (3H, s, OCH_3); 3,98 (3H, s, OCH_3); 6,92 (1H, s, H arom.); 7,40 (1H, s, H arom)
IXa	1,56 (3H, t, CH_3); 2,53 (3H, s, CH_3); 3,33 (2H, q, CH_2); 3,96 (3H, s, OCH_3); 4,0 (3H, s, OCH_3); 7,06 (3H, s, H arom)
IXb	0,97 (3H, t, CH_3); 1,83 (2H, m, CH_2); 2,33 (3H, s, CH_3); 3,07 (2H, t, CH_2); 3,73 (3H, s, OCH_3); 3,80 (3H, s, OCH_3); 6,83 (3H, s, H arom)
Xa	3,05 (3H, s, CH_3); 3,30 (3H, s, CH_3); 4,15 (3H, s, OCH_3); 4,35 (3H, s, OCH_3); 7,15 (1H, s, H arom.); 7,35 (1H, s, H arom.)
Xb	1,53 (3H, t, CH_3); 3,03 (3H, s, CH_3); 3,63 (2H, q, CH_2); 4,12 (3H, s, OCH_3); 4,32 (3H, s, OCH_3); 7,53 (1H, s, H arom.); 7,70 (1H, s, H arom)
Xc	1,13 (3H, t, CH_3); 2,05 (2H, m, CH_2); 3,03 (3H, s, CH_3); 3,60 (2H, t, CH_2); 4,13 (3H, s, OCH_3); 4,33 (3H, s OCH_3); 7,57 (1H, s, H arom.); 7,73 (1H, s, H arom)
XIa	2,83 (3H, s, CH_3); 3,17 (3H, s, CH_3); 4,07 (3H, s, OCH_3); 4,17 (3H, s, OCH_3); 7,40 (1H, s, H arom.); 7,63 (1H, s, H arom); 7,87 (2H, s, NH_2)
XIb	1,60 (3H, t, CH_3); 2,87 (3H, s, CH_3); 3,60 (2H, q, CH_2); 4,10 (3H, s, OCH_3); 4,20 (3H, s, OCH_3); 7,43 (1H, s, H arom.); 7,67 (1H, s, H arom); 7,90 (2H, s, NH_2)
XIc	1,23 (3H, t, CH_3); 2,10 (2H, m, CH_2); 3,00 (3H, s, CH_3); 3,63 (2H, t, CH_2); 4,20 (3H, s, OCH_3); 4,33 (3H, s, OCH_3); 7,53 (1H, s, H arom); 7,77 (1H, s, H arom); 8,00 (2H, s, NH_2)
XVIa	1,55 (3H, t, CH_3); 2,97 (3H, s, CH_3); 3,33 (3H, s, CH_3); 4,20 (3H, s, OCH_3); 4,30 (3H, s, OCH_3); 4,81 (2H, q, CH_2); 7,87 (1H, s, H arom); 7,93 (1H, c, H arom)
XVIb	1,61 (6H, t, CH_3); 2,93 (3H, s, CH_3); 3,75 (2H, q, CH_2); 4,17 (3H, s, OCH_3); 4,27 (3H, s, OCH_3); 4,78 (2H, q, CH_2); 7,90 (2H, s H arom),
XVIc	1,15 (3H, t, CH_3); 1,55 (3H, t, CH_3); 2,09 (2H, m CH_2); 3,00 (3H, s, CH_3); 3,68 (2H, t, CH_2); 4,23 (3H, s, OCH_3); 4,33 (3H, s, OCH_3); 4,83 (2H, q, CH_2); 7,90 (2H, s, H arom)
XVId	1,13 (3H, t, CH_3); 3,40 (3H, s, CH_3); 4,23 (3H, s, OCH_3); 4,30 (3H, s, OCH_3); 4,63 (2H, q, CH_2); 7,80...7,93 (7H, m, H arom.)
XVIe	1,16 (3H, t, CH_3); 1,70 (3H, t, CH_3); 3,85 (2H, q, CH_2); 4,30 (3H, s, OCH_3); 4,37 (3H, s, OCH_3); 4,63 (2H, q, CH_2); 7,80...8,00 (7H, m, H arom)
XVIf	1,14 (3H, t, CH_3); 2,27 (2H, m, CH_2); 3,76 (2H, t, CH_2); 4,23 (3H, s, OCH_3); 4,33 (3H, s, OCH_3); 4,63 (2H, q, CH_2); 7,80...7,97 (7H, m, H arom)



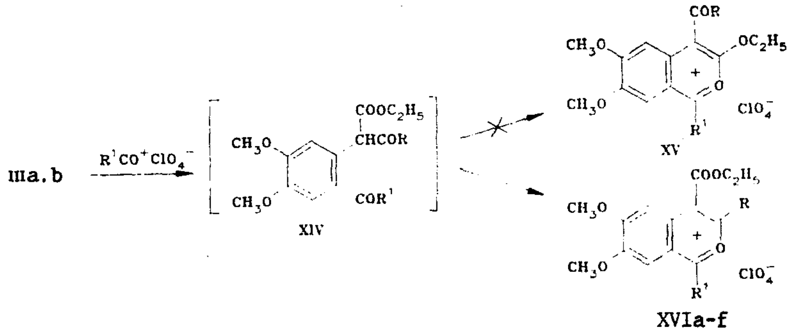
IX a R=C_2H_5, b R=C_3H_7; X, XIa R=CH_3, b R=C_2H_5, c R=C_3H_7

The use of propionic and butyric acid anhydrides as acylating agents led to mixtures of 4-cyanobenzo[c]pyrylium (Xb,c) and 4-oxo-1,3-oxazinium (IXa,b) salts, which are separable by crystallization from acetic acid. The IR spectra of perchlorates IXa,b contain all of the bands that characterize the 4-oxo-1,3-oxazinium molecule [11]: 1745-1750 (C=O), 1665 (C=C), 1100 cm^{-1} (ClO_4^-). The PMR spectra also correspond to structure IX. For example, the spectrum of 6-methyl-1-ethyl-5-(3,4-dimethoxyphenyl)-4-oxo-1,3-oxazinium perchlorate

(IXa) contains a singlet of protons of a methyl group in the 6 position of the heteroring (2.53 ppm), a quartet (3.33 ppm) and a triplet (1.56 ppm), which belong to an ethyl group, two singlets of protons of methoxy groups (3.96 and 4.0 ppm), and a singlet of aromatic protons of a benzene ring (7.06 ppm).

The occurrence of the reaction via a third possible pathway [10] with the formation of 3-amino-4-acetylbenzo[c]pyrylium salts XI was not observed in any of the examined examples. One cannot, however, exclude the possibility that the composition of the reaction products isolated may differ from that presented above when the conditions that we selected for carrying out this reaction (see the experimental section), viz., the temperature conditions, the order of mixing of the reagents, etc., are changed.

Benzo[c]pyrylium salts XIa-c were nevertheless obtained when β-keto nitrile ethyleneketal XII was used as the starting compound [12]. The presence of a protective group makes cyclization of intermediate XIII with the participation of the carbon atom of the cyano group possible. Absorption bands that are characteristic for acetyl and amino groups are present in the IR spectra of perchlorates XIa-c: two bands of stretching vibrations of the NH_2 group at 3170-3430 cm^{-1} and several intense bands at 1670-1690 cm^{-1}, which correspond to deformation vibrations of the amino group and vibrations of a carbonyl function. A characteristic feature of the PMR spectra of salts XI in trifluoroacetic acid is a broad singlet of protons of the NH_2 group, which is observed at 7.9-8.0 ppm.



XVI a $R=R^1=CH_3$; b $R=CH_3$, $R^1=C_2H_5$; c $R=CH_3$, $R^1=C_3H_7$; d $R=C_6H_5$, $R^1=CH_3$; e $R=C_6H_5$, $R^1=C_2H_5$; f $R=C_6H_5$, $R^1=C_3H_7$

In contrast to β-keto nitrile IIa, β-keto esters IIIa,b react unequivocally on treatment with acyl perchlorates to give 4-carbethoxy-substituted salts XIIa-f. The theoretically possible variant of cyclization of intermediate XIV with the participation of the ester group [10], which leads to 3-ethoxy-4-acylbenzo[c]pyrylium salts XV, is not realized.

Evidence that the compounds obtained have salt structure XVI was obtained by studying the transformations that they undergo under the influence of nucleophilic reagents. Information regarding these transformations and the reactions of salts X and XI will be presented in one of our future communications.

EXPERIMENTAL

The IR spectra of suspensions of the compounds in mineral oil were recorded with a UR-20 spectrometer. The PMR spectra were obtained with a Tesla BS-467 spectrometer (60 MHz) with tetramethylsilane (TMS) as the internal standard.

The characteristics and spectral data for perchlorates VII, IX-XI, and XVI are presented in Tables 1 and 2. The results of elementary analysis for C, H, Cl, and N were in agreement with the calculated values.

3-(3,4-Dimethoxyphenyl)pentane-2,4-dione (I, $C_{13}H_{16}O_4$. A) A 39.9-g (0.135 mole) sample of 3,4-dimethoxybenzenediazonium tetrafluoroborate was added to a stirred suspension of 4.3 g (0.067 mole) of freshly prepared copper powder, 17.7 g (0.067 mole) of copper acetylacetonate,and 135 ml (1.35 moles) of acetylacetone, and the mixture was heated for 2 h at 60°C. It was then cooled and treated with 200 ml of ether, and the mixture was stirred for 15 min and filtered. The precipitate was washed thoroughy on the filter with ether, the filtrate was shaken with water, and the organic layer was separated. The ether and excess acetylacetone were evaporated in vacuo, the residue was dissolved in 150 ml of ether, and the ether solution was treated with a solution of 25 g (0.137 mole) of copper acetate in 100 ml of water. The resulting copper salt of diketone I was removed by filtration and crystallized from toluene. A suspension of the salt in 200 ml of ether and 250 ml of 10% sulfuric acid was shaken until it had dissolved completely, after which the organic layer was separated, washed with water, and dried. The ether was evaporated, and the residue was crystallized from pentane to give 10.4 g (21%)) of a product with mp 57-59°C. PMR spectrum (d_6-DMSO): 1.78 (6H, s, CH_3), 3.65 (6H, s, OCH_3), 6.58-6.95 (3H, m, aromatic H), 16.71 ppm (1H, s, enol OH)

B) A mixture of 11.6 g (0.06 mole) of 3,4-dimethoxyphenylacetone [13] and 29.9 g (0.18 mole) of dimethylacetamide dimethylacetal [14] was refluxed for 22 h, after which it was evaporated in vacuo on a boiling-water bath. The residue was dissolved in 20 ml of methanol, and the solution was treated with 30 ml of concentrated hydrochloric acid, which caused warming up of the solution. The mixture was cooled and diluted with 50 ml of water, and the resulting precipitate was removed by filtration, washed with water, dried, and crystallized from pentane to give 5 g (35%) of a product with mp 57-59°C.

α-(3,4-Dimethoxyphenyl)acetoacetonitrile (IIa) was obtained by the method in [15].

α-(3,4-Dimethoxyphenyl)benzoylacetonitrile (IIb), $C_{17}H_{15}NO_3$. A solution of 71 ml (0.5 mole) of ethyl benzoate and 58.4 g (0.33 mole) of 3,4-dimethoxyphenylacetonitrile in 50 ml of toluene was added to a stirred suspension of 34 g (0.5 mole) of sodium ethoxide in 250 ml of toluene, and the mixture was stirred for 2 h with gentle refluxing of the reaction mass. It was then cooled and poured into water, and the aqueous layer was separated and acidified with hydrochloric acid. The resulting precipitate was removed by filtration, washed with water, squeezed, and crystallized from isopropyl alcohol–water to give 60 g (65%) of a product with mp 110-112°C. IR spectrum: 3210 (enol OH), 2210 (C≡N), 1620 cm^{-1} (C=C). PMR spectrum [$(CD_3)_2CO$]: 3.67 (6H, s, OCH_3), 6.13 (1H, s, CH), 6.87-8.07 ppm (8H, m, aromatic H).

Ethyl α-(3,4-Dimethoxyphenyl)acetoacetate (IIIa), $C_{14}H_{18}O_5$. A strong stream of hydrogen chloride was passed through a suspension of 29.8 g (0.136 mole) of β-keto nitrile IIa in 55 ml of 95% ethanol until the solid material had dissolved completely (∿5 h), after which the solution was allowed to stand overnight. It was then stirred for 1 h at 40°C after which 100 ml of water was added, and the aqueous mixture was stirred for another 2 h. The mixture was then poured into 500 ml of water, and the aqueous mixture was extracted with ether. The ether extracts were washed with water and dried with anhydrous Na_2SO_4, and the solvent was evaporated. The residue was crystallized from pentane to give 19.6 g (54%) of a product with mp 58-60°C. IR spectrum: 1760 (C=O), 1730 cm^{-1} ($COOC_2H_5$). PMR spectrum [$(CD_3)_2 \cdot CO$]: 1.13 (3H, t, CH_3), 2.07 (3H, s, CH_3), 3.70 (6H, s, OCH_3), 4.10 (2H, q, CH_2), 4.73 (1H, s, CH), 6.83-6.97 ppm (3H, m, aromatic H).

Ethyl α-(3,4-Dimethoxyphenyl)benzoylacetate (IIIb). This compound was obtained in 70% yield from β-keto nitrile IIb under the conditions of the method in [16]., The product was an uncrystallized yellow oil. IR spectrum: 1760 (C=O), 1710 cm^{-1} ($COOC_2H_5$). PMR spectrum (d_7-DMF): 1.20 (3H, t, CH_3), 3.73 (6H, s, OCH_3), 4.20 (2H, s, CH_2), 6.07 (1H, s, CH), 6.97-8.23 ppm (8H, m, aromatic H).

1-R-3-Methyl-4-acetyl-6,7-dimethoxybenzo[c]pyrylium Perchlorates VIIa-c. A 0.01-mole sample of β-diketone I was added in small portions to a mixture prepared from 0.07 mole of the aliphatic carboxylic acid anhydride and 0.8 ml of 70% $HClO_4$, and the mixture was allowed to stand for 10-15 h. It was then diluted with ether, and the resulting precipitate was removed by filtration, washed with ether, dried, and crystallized from glacial acetic acid.

1-R-3-Methyl-4-cyano-6,7-dimethoxybenzo[c]pyrylium Perchlorates Xa-c and 6-Methyl-2-R-5-(3,4-dimethoxyphenyl)-4-oxo-1,3-oxazinium Perchlorates IXa,b. A 0.01-mole sample of β-ketonitrile IIa was added in small portions to a mixture prepared from 0.1 mole of the aliphatic carboxylic acid anhydride and 0.8 ml of 70% $HClO_4$, and the mixture was allowed to stand overnight. Ether was added to the reaction mixture, and the resulting precipitate was removed by filtration, washed with ether, and dried. The mixtures of salts Xb,c and IXa,b formed when propionic and butyric anhydrides were used were dissolved by heating in the minimum amount of glacial acetic acid. The perchlorates IXa,b that precipitated after cooling to 20°C were removed by filtration, and the filtrates were diluted with ether to give salts Xb, c.

1-Methyl-3-amino-4-acetyl-6,7-dimethoxybenzo[c]pyrylium Perchlorate (XIa). A 2.6-g (0.01 mole) sample of ethyleneketal XII [12] was added gradually to a mixture of 9.5 ml (0.1 mole) of acetic anhydride and 0.8 ml of 70% $HClO_4$, and the reaction mixture was shaken until the solid material had dissolved. The mixture was diluted with ether, and the oily precipitate was separated by decantation and triturated under a layer of ether. The solid material was removed by filtration, washed with ether, and dried.

1-Ethyl-3-amino-4-acetyl-6,7-dimethoxybenzo[c]pyrylium Perchlorate (XIb). A 1.3-g (0.005 mole) sample of ethyleneketal XII was added to a mixture of 6.5 ml (0.05 mole) of propionic anhydride and 0.4 ml of 70% $HClO_4$, and the mixture was allowed to stand overnight. Dilution of the mixture with ether (50 ml) gave an oily precipitate that crystallized on standing under ether for 24 h. The precipitate was removed by filtration, washed successively with a small amount of acetic acid and ether, and dried.

1-Propyl-3-amino-4-acetyl-6,7-dimethoxybenzo[c]pyrylium Perchlorate (XIc). This compound was similarly obtained from ethyleneketal XII and butyric anhydride.

1-R^1-3-R-4-Ethoxycarbonyl-6,7-dimethoxybenzo[c]pyrylium Perchlorates XVIa-f. These compounds were obtained from β-keto esters IIIa,b in the same way as VIIa-f.

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