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SYNTHESIS AND REACTIONS OF 1-(3-CHLOROPROPYL)-6,7-DIMETHOXY-3-METHYLBENZO[*c*]PYRYLIUM PERCHLORATE

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Acid-catalyzed acylation of 3,4-dimethoxyphenylacetone with γ-chlorobutyryl chloride gave 1-(3-chloropropyl)-6,7-dimethoxy-3-methylbenzo[c]pyrylium perchlorate. Recyclization of the product with nitrogen nucleophiles (ammonia, primary amines, hydrazine derivatives, hydroxylamine) led through the corresponding isoquinolinium salts to benzo[f]indolizinium, pyridazino[2,1-b]-isoquinolinium, and 1,2-oxazino[2,1-b]isoquinolinium salts.

Keywords: 3,4-dimethoxyphenylacetone, 1-(3-chloropropyl)-6,7-dimethoxy-3-methylbenzo[*c*]-pyrylium, γ-chlorobutyryl chloride, recyclization.

The variety of transformations found in pyrylium salts [1, 2] is due both to the presence of the heteroatom and to the nature of the substituents. In our opinion, one important aspect of the development of the chemistry of the pyrylium cation is the introduction of a functional substituent into its molecule. Of particular interest are cases where the substituent participates in recyclization reactions, since here new important transformations for pyrylium salts are observed [3-7].

In the development of this we synthesized pyrylium salts in which the functional group is in the α-alkyl substituent by using the chlorides of functionally substituted carboxylic acids in acid-catalyzed heterocyclization [8]. Thus, we have found that the acylation of mesityl oxide, 3,4-dimethoxyphenylacetone, and 3-indolylacetone with γ-butyryl chloride gives respectively mono-, di-, and tricyclic pyrylium cations, which undergo recyclization with ammonia to dihydroindolizinium derivatives by intramolecular quaternization of the intermediate pyridine base, i.e., the reaction takes place with reconstruction of the ring [9].

It seemed of interest to study the reaction of α-(γ-chloropropyl)pyrylium salts with various nitrogen nucleophiles. In the present work we describe the synthesis and some recyclization reactions of 1-(3-chloropropyl)-6,7-dimethoxy-3-methylbenzo[*c*]pyrylium perchlorate (**1**), produced by two procedures for the acylation of 3,4-dimethoxyphenylacetone (**2**) with γ-chlorobutyryl chloride, i.e., in the presence of perchloric acid and with the use of Lewis acids. The preferred method was the use of tin tetrachloride in methylene chloride with the intermediate isolation of 1-(3-chloropropyl)-6,7-dimethoxy-3-methylbenzo[*c*]pyrylium hexachlorostannate (**3**), the treatment of which with perchloric acid in methanol led to the corresponding perchlorate **1** with an overall yield of 80% (compared with the 63% obtained during acylation of the ketone **2** using perchloric acid).

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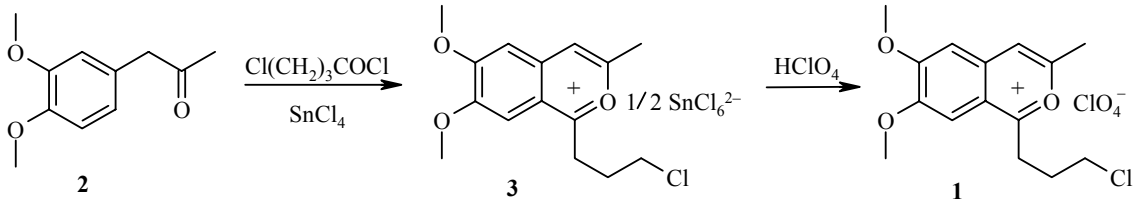
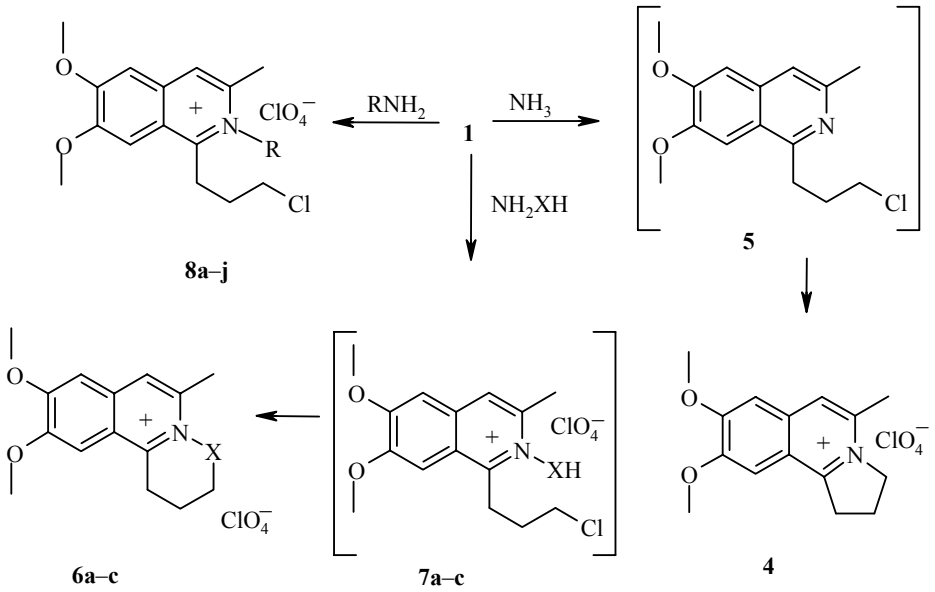


The following nitrogen nucleophiles were used to study the recyclization of the perchlorate **1**: Ammonia, primary amines, hydroxylamine, hydrazine and its derivatives.

It was established that the reaction of the perchlorate **1** with ammonia under various conditions (liquid ammonia, alcohol and water–alcohol solutions of ammonia, ammonium acetate in acetic acid) led with yields close to quantitative to 8,9-dimethoxy-5-methyl-8,9-dimethoxy-2,3-dihydrobenzo[*f*]indolizinium perchlorate (**4)**. The supposed intermediate γ-chloropropylisoquinoline **5** was not isolated even when liquid ammonia was used for the recyclization of the salt **1**. In our opinion the initially formed isoquinoline **5** is quaternized as a result of an intramolecular reaction, leading to the isolation of the tricyclic benzoindolizinium perchlorate **4**. Examples of the intramolecular quaternization of bases of the halogenoalkyl derivatives of pyridine known from the literature [10-13] demonstrate the high rate of this process.

As a rule the recyclization of 1,3-dialkyl-substituted benzo[*c*]pyrylium salts by the action of hydrazine and hydroxylamine leads to N-substituted isoquinolinium salts or N-oxides [14]. During recyclization of the perchlorate **1** by the action of hydrazine, its derivatives, and hydroxylamine we isolated the quaternary salts in the form of perchlorates. It was established on the basis of elemental analysis and spectral characteristics that the recyclization of the perchlorate **1** with hydrazine, methylhydrazine, and hydroxylamine leads to quaternary derivatives of isoquinoline with annelation of a new ring, i.e., 9,10-dimethoxy-6-methyl-1,2,3,4-tetrahydroisoquinolino[2,1-*b*]pyridazinium (**6a**), 9,10-dimethoxy-4,6-dimethyl-1,2,3,4-tetrahydroisoquinolino-[2,1-*b*]pyridazinium (**6b**), and 9,10-dimethoxy-6-methyl-1,2,3,4-tetrahydroisoquinolino[2,1-*b*]-1,2-oxazinium (**6c**) perchlorates respectively.



6, **7 a** X = NH, **b** X = NMe, **c** X = O; **8 a** R = Me, **b** R = CH_2Ph, **c** R = Ph, **d** R = *p*-MeC_6H_4, **e** R = *p*-$MeOC_6H_4$, **f** R = *o*-$H_2NC_6H_4$, **g** R = *o*-HOC_6H_4, **h** R = NHPh, **i** R = NHCOMe, **j** R = NHCOPh

As in the case of recyclization by the action of ammonia, the intermediate isoquinolinium derivatives **7a-c** were not isolated. This is fully explainable since the nucleophilic characteristics of the amino group in the intermediate N-aminoisoquinolinium cations **7a**,**b** and the oxygen atom of the N-oxide **7c** (in the deprotonated form) are very high, as confirmed earlier by alkylation and by the formation of derivatives with aldehydes (of the Schiff base type) [14].

The recyclization of the perchlorate **1** followed by spontaneous cyclization, observed under the influence of ammonia, hydrazine, methylhydrazine, and hydroxylamine, provides examples of tandem [15] reactions of cascade type. During the action of primary amines and aryl- and acylhydrazines, however, under the same conditions only the "normal" recyclization products, i.e., the N-substituted isoquinolinium salts **8a-j**, are isolated. This is probably due to the reduced basicity and nucleophilicity of the exocyclic amino group and also, possibly, steric hindrances.

The composition and structure of all the synthesized compounds were confirmed by elemental analysis and by IR and ^{1}H NMR spectroscopy. Thus, in the IR spectra of compounds **1**, **4**, **6a-c**, and **8a-j** there are characteristic absorption bands in the regions of 1100 (ClO_4) and 1620-1635 cm^{-1}, while in the spectra of the salts **6a** and **8f-j** there are in addition bands in the region of 3230-3275 (NH), 3375-3455, and 3440 cm^{-1} (OH). In the ^{1}H NMR spectra of the salts **4** and **6a**,**c** the triplet for the protons of the methylene groups adjacent to the heteroatom are substantially downfield from the triplet of the γ-methylene group in the initial salt **1**. In the

TABLE 1. The Characteristics of the Synthesized Compounds **1**, **3**, **4**, **6a-c**, and **8a-j**

Compound	Empirical formula	Found, % / Calculated, %				mp, °C	Yield, %
		C	H	Cl	N		
1	$C_{15}H_{18}Cl_2O_7$	47.5 / 47.3	4.8 / 4.7	18.4 / 18.6		214-217	91
3	$C_{15}H_{18}Cl_4O_3Sn_{1/2}$	40.2 / 40.3	4.0 / 4.1	32.1 / 31.7		217-219	92
4	$C_{15}H_{18}ClNO_6$	52.4 / 52.4	5.2 / 5.3	10.2 / 10.3	4.0 / 4.1	245-247	77-95
6a	$C_{15}H_{19}ClN_2O_6$	50.0 / 50.2	5.4 / 5.3	9.8 / 10.0	7.9 / 7.8	233-234	76
6b	$C_{16}H_{21}ClN_2O_6$	51.8 / 51.6	5.5 / 5.7	9.7 / 9.5	7.5 / 7.5	210-211	70
6c	$C_{15}H_{18}ClNO_7$	50.1 / 50.3	5.0 / 4.8	9.9 / 10.0	3.9 / 4.1	220-222	51
8a	$C_{16}H_{21}Cl_2NO_6$	48.8 / 48.7	5.1 / 5.4	17.9 / 18.0	3.4 / 3.6	211-213	68
8b	$C_{22}H_{25}Cl_2NO_6$	56.0 / 56.2	5.5 / 5.4	15.1 / 15.1	3.2 / 3.0	195-197	77
8c	$C_{21}H_{23}Cl_2NO_6$	55.3 / 55.3	5.1 / 5.2	15.5 / 15.3	3.1 / 3.3	254-255	54
8d	$C_{22}H_{25}Cl_2NO_6$	56.3 / 56.2	5.3 / 5.4	14.8 / 15.1	3.0 / 3.0	207-208	57
8e	$C_{22}H_{25}Cl_2NO_7$	54.1 / 54.3	5.0 / 5.2	14.8 / 14.6	2.7 / 2.9	221-223	97
8f	$C_{21}H_{24}Cl_2N_2O_6$	53.5 / 53.2	5.1 / 5.0	15.0 / 14.9	5.9 / 6.0	223-225	89
8g	$C_{21}H_{23}Cl_2NO_7$	53.4 / 53.2	4.9 / 5.1	15.0 / 15.2	3.0 / 2.9	202-203	94
8h	$C_{21}H_{24}Cl_2N_2O_6$	53.5 / 53.7	5.1 / 5.0	15.0 / 15.2	5.9 / 6.0	177-180	94
8i	$C_{17}H_{22}Cl_2N_2O_7$	46.7 / 46.6	5.1 / 5.0	16.2 / 16.2	6.4 / 6.3	228-230	92
8j	$C_{22}H_{24}Cl_2N_2O_7$	55.1 / 54.9	4.8 / 4.6	13.4 / 13.6	5.6 / 5.3	188-190	72

TABLE 2. The ^{1}H NMR Spectra of Compounds **1**, **3**, **4**, **6a-c**, and **8a-j**

Com-pound	Chemical shifts, δ, ppm (SSCC, *J*, Hz)
1	2.35-2.50 (2H, m, 1-β-CH_2); 2.76 (3H, s, 3-CH_3); 3.70 (2H, t, *J* = 6, 1-α-CH_2); 3.77 (2H, t, *J* = 6, 1-γ-CH_2); 4.07 and 4.16 (6H, 2 s, 6- and 7-OCH_3); 7.44 (1H, s, H-5); 7.62 (1H, s, H-8); 7.74 (1H, s, H-4)
3	2.37-2.51 (2H, m, 1-β-CH_2); 2.78 (3H, s, 3-CH_3); 3.70-3.83 (4H, m, 1-α- and γ-CH_2); 4.08 and 4.17 (6H, 2 s, 6- and 7-OCH_3); 7.55 (1H, s, H-5); 7.65 (1H, s, H-8); 7.88 (1H, s, H-4)
4	2.24-2.57 (2H, m, 2-CH_2); 2.73 (3H, s, 5-CH_3); 3.87 (2H, t, *J* = 6, 1-CH_2); 4.00 and 4.02 (6H, 2 s, 6- and 7-OCH_3); 4.78 (2H, t, *J* = 6, 3-CH_2), 7.52 (1H, s, H-7); 7.59 (1H, s, H-10); 7.96 (1H, s, H-6)
6a	2.05-2.18 (2H, m, 2-CH_2); 2.67 (3H, s, 6-CH_3); 3.41-3.51 (2H, m, 3-CH_2); 3.59 (2H, t, *J* = 7, 1-CH_2); 4.02 (6H, s, 9- and 10-OCH_3); 6.15 (1H, t, *J* = 7, H-4); 7.32 (1H, s, H-8); 7.43 (1H, s, H-11); 7.78 (1H, s, H-7)
6b	2.05-2.16 (2H, m, 2-CH_2); 2.77 (3H, s, 6-CH_3); 2.91 (3H, s, 4-CH_3); 3.30-3.55 (2H, m, 3-CH_2); 3.65-3.85 (2H, m, 1-CH_2); 4.02 and 4.03 (6H, 2 s, 9- and 10-OMe); 7.33 (1H, s, H-8); 7. 44 (1H, s, H-11); 7.81 (1H, s, H-7)
6c	2.33-2.47 (3H, m, 2-CH_2); 2.69 (3H, s, 6-CH_3); 3.63 (2H, t, *J* = 7, 1-CH_2); 4.03 (6H, s, 9- and 10-OCH_3); 4.66 (2H, t, *J* = 6, 3-CH_2), 7.38 (1H, s, H-8); 7.45 (1H, s, H-11); 7.82 (1H, s, H-7)
8a	2.17-2.25 (2H, m, 1-β-CH_2); 2.79 (3H, s, 3-CH_3); 3.68-3.77 (2H, t, *J* = 7, 1-α-CH_2); 3.93 (2H, t, *J* = 7, 1-γ-CH_2); 4.04 (6H, s, 6- and 7-OCH_3); 4.22 (3H, s, 2-CH_3); 7.54 (1H, s, H-5); 7.66 (1H, s, H-8); 8.06 (1H, s, H-4)
8b	2.15-2.25 (2H, m, 1-β-CH_2); 2.67 (3H, s, 3-CH_3); 3.45-3.55 (2H, m, 1-α-CH_2); 3.79 (2H, t, *J* = 7, 1-γ-CH_2); 4.04 and 4.08 (6H, s, 6- and 7-OCH_3); 5.84 (2H, s, 2-CH_2); 6.95-7.02 (2H, m, H-3' and H-5'); 7.33-7.41 (3H, m, H-2', H-4' and H-6'); 7.42 (1H, s, H-5); 7.56 (1H, s, H-8); 7.95 (1H, s, H-4)
8c	2.05-2.25 (2H, m, 1-β-CH_2); 2.52 (3H, s, 3-CH_3); 3.15-3.25 (2H, m, 1-α-CH_2); 3.69 (2H, t, *J* = 7, 1-γ-CH_2); 4.04 and 4.08 (6H, 2 s, 6- and 7-OCH_3); 6.95-7.02 (2H, m, H-3' and H-5'); 7.33-7.41 (3H, m, H-2', H-4' and H-6'); 7.42 (1H, s, H-5); 7.56 (1H, s, H-8); 7.95 (1H, s, H-4)
8d	2.03-2.16 (2H, m, 1-β-CH_2); 2.23 (3H, s, 4'-CH_3); 2.48 (3H, s, 3-CH_3); 3.22-3.32 (2H, m, 1-α-CH_2); 3.57 (2H, t, *J* = 6, 1-γ-CH_2); 4.06 and 4.10 (6H, 2 s, 6- and 7-OCH_3); 7.34 (2H, d, *J* = 8, H-3' and H-5'); 7.46 (1H, s, H-5); 7.53 (1H, d, *J* = 8, H-2' and H-6'); 7.56 (1H, s, H-8); 7.96 (1H, s, H-4)
8e	2.05-2.15 (2H, m, 1-β-CH_2); 2.30 (3H, s, 3-CH_3); 3.25-3.35 (2H, m, 1-α-CH_2); 3.59 (2H, t, *J* = 6, 1-γ-CH_2); 3.90 (3H, s, 4'-OCH_3); 4.05 and 4.09 (6H, s, 6- and 7-OCH_3); 7.20 (2H, d, *J* = 7, H-3' and H-5'); 7.39 (2H, d, *J* = 7, H-2' and H-6'); 7.41 (1H, s, H-5); 7.55 (1H, s, H-8); 7.97 (1H, s, H-4)
8f	2.03-2.16 (2H, m, 1-β-CH_2); 2.33 (3H, s, 3-CH_3); 3.05-3.22 (2H, m, 1-α-CH_2); 3.45-3.55 (2H, m, 1-α-CH_2); 3.57 (2H, t, *J* = 6, 1-γ-CH_2); 4.06 and 4.10 (6H, s, 6- and 7-OCH_3); 4.40 (2H, br. s, 2'-NH_2); 6.92-7.46 (4H, m, H-3', H-6'); 7.47 (1H, s, H-5); 7.59 (1H, s, H-8); 8.03 (1H, s, H-4)
8g	2.03-2.12 (2H, m, 1-β-CH_2); 2.30 (3H, s, 3-CH_3); 3.15-3.45 (2H, m, 1-α-CH_2); 3.57 (2H, t, *J* = 6, 1-γ-CH_2); 4.04 and 4.09 (6H, 2 s, 6- and 7-OCH_3); 7.10-7.60 (4H, m, H-3', H-6'); 7.44 (1H, s, H-5); 7.56 (1H, s, H-8); 7.97 (1H, s, H-4)
8h	2.15-2.26 (2H, m, 1-β-CH_2); 2.58 (3H, s, 3-CH_3); 3.35-3.55 (2H, m, 1-α-CH_2); 3.75 (2H, t, *J* = 7, 1-γ-CH_2); 4.06 and 4.10 (6H, s, 6- and 7-OCH_3); 6.65-7.25 (5H, m, H-2', H-6'); 7.48 (1H, s, H-5); 7.63 (1H, s, H-8); 8.02 (1H, s, H-4); 8.80 (1H, br. s, 2-NH)
8i	2.05-2.25 (2H, m, 1-β-CH_2); 2.30 (3H, s, 2-$NCOCH_3$); 2.58 (3H, s, 3-CH_3); 3.32-3.75 (2H, m, 1-α-CH_2); 3.79 (2H, t, *J* = 7, 1-γ-CH_2); 4.04 and 4.08 (6H, 2 s, 6- and 7-OCH_3); 7.41 (1H, s, H-5); 7.54 (1H, s, H-8); 7.93 (1H, s, H-4); 10.50 (1H, br. s, 2-NH)
8j	2.05-2.20 (2H, m, 1-β-CH_2); 2.46 (3H, s, 3-CH_3); 3.15-3.55 (2H, m, 1-α-CH_2); 3.67 (2H, t, *J* = 7, 1-γ-CH_2); 3.97 and 4.06 (6H, 2 s, 6- and 7-OCH_3); 7.10-7.55 (5H, m, H-2', H-6'); 7.46 (1H, s, H-5); 7.56 (1H, s, H-8); 7.96 (1H, s, H-4); 10.60 (1H, br. s, 2-NH)

^{1}H NMR spectrum of the pyridazinoisoquinolinium salt **6a** the signal for the protons of this methylene group is represented by a multiplet on account of spin–spin coupling with the proton at the nitrogen atom (H-4). The signal of this proton with a chemical shift depending on the temperature and the solvent is represented by a triplet with J = 6 Hz. In the ^{1}H NMR spectrum of the N-methyl salt **6b** the signals of all three methylene groups are multiplets on account, probably, of complications in the transformations of the conformations of the tetrahydropyridazine ring due to the presence of the methyl substituent.

Thus, our investigated recyclization reactions of the benzo[*c*]pyrylium salt **1** make it possible to obtain analogs of alkaloids widely found in nature [10-13, 16-18] and may in our opinion prove useful in the synthesis of new biologically active compounds.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Gemini-200 instrument (200 MHz) with MeCN-d_3 as solvent and HMDS (δ 0.055 ppm) as internal standard. The IR spectra were recorded on a Specord IR-75 instrument. After separation by filtration and washing with the solvent indicated in the specific method all the products obtained by the described procedures were washed with ether and dried. The characteristics and spectral data of the obtained compounds are given in Tables 1 and 2.

1-(3-Chloropropyl)-6,7-dimethoxy-3-methylbenzo[*c*]pyrylium Perchlorate (1). A. To a mixture of γ-chlorobutyryl chloride (5.6 ml, 0.05 mol) and 70% perchloric acid (0.8 ml, 0.01 mol), prepared with stirring and cooling, we added 3,4-dimethoxyphenylacetone **2** (1.94 g, 0.01 mol). The mixture was left at room temperature for 3 h.

B. To a solution of the hexachlorostannate **3** (27.74 g, 0.062 mol) in methanol (560 ml) we added 70% perchloric acid (10 ml, 0.124 mol). The mixture was boiled for 5 min and cooled to room temperature, and the precipitate was washed with isopropyl alcohol.

1-(3-Chloropropyl)-6,7-dimethoxy-3-methylbenzo[*c*]pyrylium Hexachlorostannate (3). To a mixture of the ketone **2** (3.88 g, 0.02 mol) and γ-chlorobutyryl chloride (6.2 g, 0.044 mol) in methylene chloride (10 ml) with stirring we added stannic chloride (2.6 g, 0.01 mol). The mixture was boiled for 3 h with a reflux condenser and with agitation. After cooling 10-15 ml of ether was added to the reaction mixture, and the precipitate was washed with methyl ethyl ketone.

8,9-Dimethoxy-5-methyl-2,3-dihydrobenzo[*f*]indolizinium Perchlorate (4). A. To a suspension of the perchlorate **1** (0.7 g) in alcohol (10 ml) we added aqueous ammonia (5 ml). After brief agitation the initial salt dissolved. After 5-10 min a colorless crystalline precipitate of **4** separated. It was washed with water and with alcohol.

B. A mixture of the salt **1** (0.76 g, 0.002 mol) and concentrated aqueous ammonia (0.5 ml) was boiled for 20 min in methanol (5 ml) and cooled. The precipitate was washed with isopropyl alcohol.

C. A mixture of the salt **1** (0.76 g, 0.002 mol), methanol (5 ml), and concentrated aqueous ammonia (0.5 ml) was stirred at room temperature for 10 h. The precipitate was washed with isopropyl alcohol.

To the salt **1** (0.76 g, 0.002 mol) we added liquid ammonia (5 ml). The mixture was kept until the solvent had completely evaporated, and the precipitate was washed with water and with methanol.

9,10-Dimethoxy-6-methyl-1,2,3,4-tetrahydroisoquinolino[2,1-*b*]pyridazinium Perchlorate (6a). To a solution of the salt **1** (0.76 g, 0.002 mol) in methanol (5 ml) we added 80% hydrazine hydrate (0.28 g, 0.0044 mol). The mixture was boiled for 30 min and was then cooled. The precipitate was washed with isopropyl alcohol.

9,10-Dimethoxy-4,6-dimethyl-1,2,3,4-tetrahydroisoquinolino[2,1-*b*]pyridazinium Perchlorate (6b). The compound was obtained similarly to the salt **6a**.

9,10-Dimethoxy-6-methyl-1,2,3,4-tetrahydroisoquinolino[2,1-*b*]-1,2-oxazinium Perchlorate (6c). To a solution of hydroxylammonium acetate, obtained from hydroxylamine hydrochloride (0.42 g, 0.006 mol) and an equimolar amount of sodium acetate by boiling in 6 ml of methanol, after filtration from the precipitate we added the perchlorate **1** (0.76 g, 0.002 mol). The mixture was boiled for 35 min and was then cooled, and the precipitate was washed with methanol.

1-(3-Chloropropyl)-6,7-dimethoxy-2,3-dimethylisoquinolinium Perchlorate (8a). To a solution of the salt **1** (0.76 g, 0.002 mol) in methanol (5 ml) we added aqueous methylamine (0.5 ml). The mixture was boiled for 2 h and cooled, and the precipitate was washed with isopropyl alcohol.

2-Benzyl-3-methyl-6,7-dimethoxy-1-(3-chloropropyl)isoquinolinium Perchlorate (8b). To a solution of the salt **1** (0.76 g, 0.002 mol) in methanol (5 ml) we added benzylamine (0.44 g, 0.0041 mol). The mixture was boiled for 2 h and diluted with water, and the product was washed with isopropyl alcohol.

The perchlorates **8c-j** were obtained similarly.

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