ISSN 1070-4280, Russian Journal of Organic Chemistry, 2014, Vol. 50, No. 1, pp. 66–71. © Pleiades Publishing, Ltd., 2014.
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Synthesis and Anticorrosive Properties of Alkylammonium Polyfluoro-3-(ethoxycarbonyl)-2-oxo-2*H*-chromen-4-olates

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Received May 28, 2013

Abstract—Reactions of ethyl polyfluoro-4-hydroxy-2-oxo-2*H*-chromene-3-carboxylates with amines under mild conditions afforded alkylammonium polyfluoro-3-(ethoxycarbonyl)-2-oxo-2*H*-chromen-4-olates which efficiently inhibited hydrochloric acid corrosion of mild steel at low concentrations (10^{-4}–10^{-5} M).

DOI: 10.1134/S1070428014010138

Chemical compounds used as metal corrosion inhibitors should contain appropriate structural fragments such as heteroatoms (P, S, N, O) in both open chains and cyclic units and π-bonds favoring stronger fixing of organic molecules on the metal surface [1]. While developing new metal corrosion inhibitors, increasing attention is now given to ecological aspects implying reduction of their negative toxic impact on the environment. In this connection, the recent review by Gese [2] demonstrated prospects in searching for metal corrosion inhibitors among medicines of natural and synthetic origin, which possess necessary structural elements. As a rule, most medicines undergo biodegradation in the environment [3].

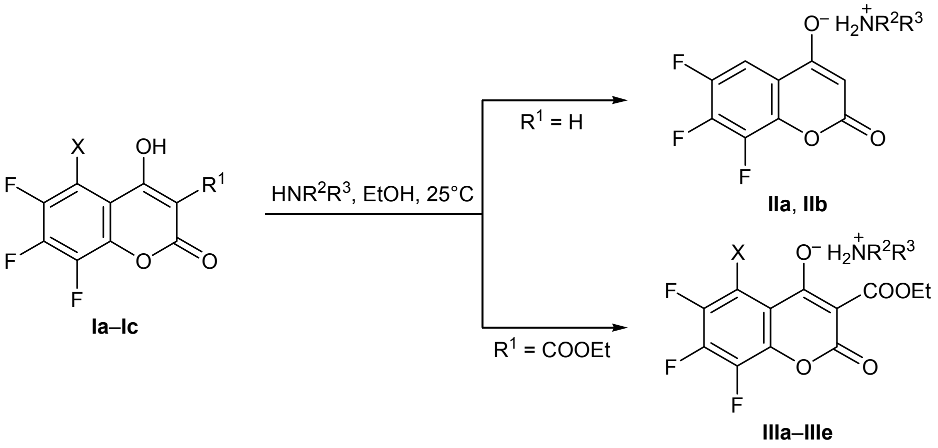
Data on anticorrosive properties of a broad spectrum of drugs belonging to various chemical classes have been collected in [2]; in particular, antibiotics (β-lactams, quinolinones, fluoroquinolinones, tetracyclins, macrolides, aminoglycosides, etc.), sulfamides, fungicides, antihelminthics, and many others have been covered. However, there are no published data on anticorrosive effect of coumarin derivatives, though coumarin fragment constitutes many natural and synthetic drugs. For example, 4-hydroxycoumarin derivatives exhibit anticoagulant activity [4], but their use as corrosion inhibitors in aqueous media is practically impossible, for coumarin derivatives are insoluble in aqueous media and are unstable in alkaline solution [5].

We previously found [6] that, unlike fluorine-free analog, 4-hydroxy-5,6,7,8-tetrafluorocoumarin (**Ia**) reacts with primary amines under mild conditions to give water-soluble salts **IIa** and **IIb** (Scheme 1). The goal of the present work was to synthesize ammonium salts of fluorine-containing 4-hydroxycoumarins and test them as inhibitors of hydrochloric acid corrosion of mild steel.

New alkylammonium polyfluoro-3-(ethoxycarbonyl)-2-oxo-2*H*-chromen-4-olates **IIIa**–**IIIe** were obtained by reacting ethyl polyfluoro-4-hydroxy-2-oxo-2*H*-chromene-3-carboxylates **Ib** and **Ic** with various amines (Scheme 1). Salts were formed preferably by strongly basic amines. As amine component we tried primary monoamines (methylamine, pK_a 10.62; benzylamine, pK_a 9.34), secondary amine (1-methylpiperazine, pK_a 9.14), and diamine (*o*-phenylenediamine, pK_a 4.47) [7, 8]. Except for *o*-phenylenediamine, weakly basic amines (e.g., aniline, pK_a 4.58 [7]) did not form stable salts with coumarins **Ib** and **Ic**. The use of different amines should ensure diversity of counterions in the resulting salts. The reactions were carried out in ethanol with equimolar amounts of the reactants; only in the synthesis of methylammonium salt **Ia** gaseous methylamine was taken in excess.

Although the salt structure of compounds **IIa** and **IIb** synthesized previously from coumarin **Ia** [9] (Scheme 1) was proved by a set of spectral methods, four alternative structures may be proposed theoretically for salts **IIIa**–**IIIf** (Scheme 2). Chroman structure **A** results from addition of amine to the C^3=C^4 bond of coumarin **Ib** or **Ic**; chroman **B** is the product of amine addition to the lactone carbonyl group (C^2=O); pro-

Scheme 1.

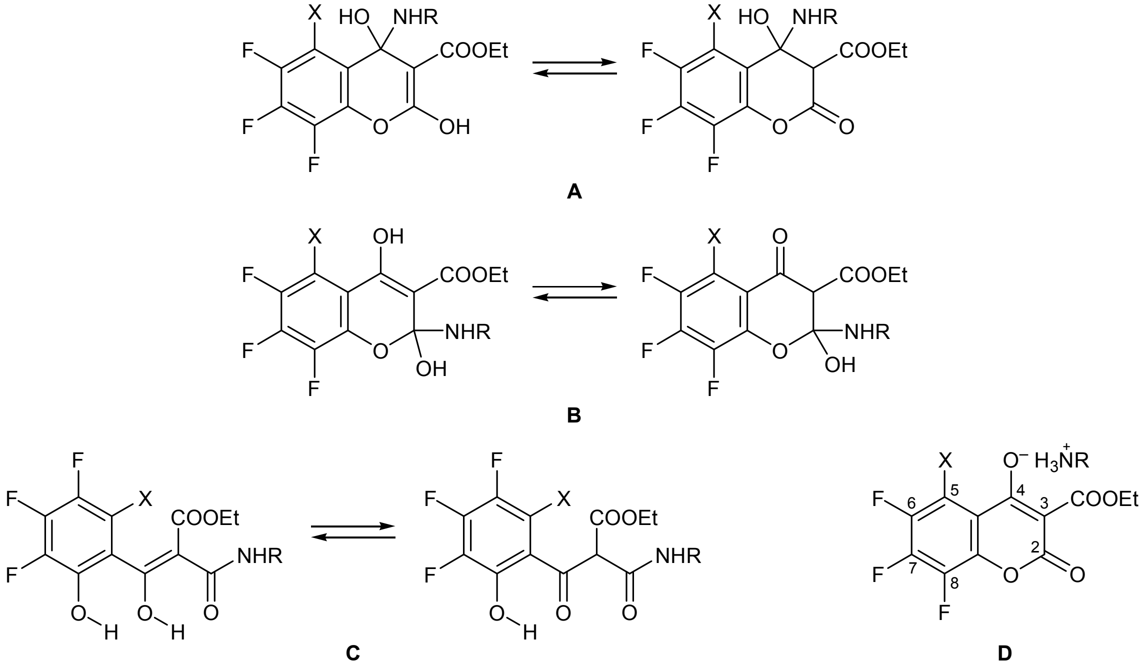


I, X = F, R^1 = H (**a**), COOEt (**b**); X = H, R^1 = COOEt (**c**); **II**, R^2 = H, R^3 = Me (**a**), $PhCH_2$ (**b**); **III**, X = H, R^2 = H, R^3 = Me (**a**), $PhCH_2$ (**b**), 2-$H_2NC_6H_4$ (**c**); R^2R^3N = 4-methylpiperazin-1-yl (**d**); X = F, R^2 = H, R^3 = $PhCH_2$ (**e**).

penoate **C** could be formed via opening of the α-pyranone ring; and structure **D** corresponds to the target ammonium salt. Several tautomeric forms are possible for structures **A**–**C** due to keto–enol transformation. The probability for the formation of structure **C** from coumarins **Ib** and **Ic** increases due to the presence of electron-withdrawing ester group on C^3, which raises partial positive charge on the neighboring lactone carbonyl carbon atom (C^2). Therefore, reliable determination of the structure of **IIIa**–**IIIe** is necessary.

Compounds **IIIa**–**IIIe** were assigned salt structure **D** on the basis of their IR and ^{1}H NMR spectra which were compared with the corresponding data for previously described salts **IIa** and **IIb**. The IR spectra of **IIIa**–**IIIe** contained absorption bands typical of stretching vibrations of lactone (1694–1726 cm^{-1}) and ester carbonyl groups (1620–1677 cm^{-1}), while the most characteristic were bands due to stretching vibrations of H_3N^+ (H_2N^+) group in the region 2609–2731 cm^{-1}. In the ^{1}H NMR spectra of **IIIa**–**IIIe**, protons in the H_3N^+ (H_2N^+) group appeared as a broadened three- or two-proton singlet at δ 7.66–8.45 ppm. Salt **IIIc** displayed in the ^{13}C NMR spectrum (DMSO-d_6) only one set of signals, including three

Scheme 2.



downfield signals at δ_C 159.89, 167.04, and 170.19 ppm (C^2, C^4, and 3-C=O, respectively, in structure **D**). Structures **A** and **B** each should give rise to two downfield signals, whereas four downfield signals should be observed from propenoate **C**.

Thus, the formation of salts **II** and **III** in the reactions of fluorinated 4-hydroxycoumarins **Ia**–**Ic** with strongly basic amines under mild conditions is the predominant process regardless of their structure. Obviously, the presence of three or four electron-withdrawing fluorine atoms in the aromatic moiety of coumarins **Ia**–**Ic** is the main factor determining high acidity of the 4-hydroxy group. This makes the behavior of **Ia**–**Ic** different from the transformations of their fluorine-free analogs.

Next, we examined anticorrosive properties of both previously synthesized salts **IIa** and **IIb** and salts **IIIa**–**IIId** obtained in the present work. Preliminarily, it was found that salts **IIa**, **IIb**, **IIIa**, **IIIc**, and **IIId** are poorly soluble in water; therefore, their use as corrosion inhibitors is limited to low concentration range. Benzylammonium salts **IIIb** and **IIIe** turned out to be almost insoluble in water, and their anticorrosive effect was not studied.

The inhibitory effect of salts **IIa**, **IIb**, **IIIa**, **IIIc**, and **IIId** is based on electrostatic interaction between the positively charged ammonium nitrogen atom and negatively charged electrode [10]. Analysis of the K_n values revealed fairly similar anticorrosive properties of the examined compounds. They reduced the rate of corrosion of mild steel (St3) in hydrochloric acid medium by a factor of 2–3 at a low concentration. The inhibitory effect decreases in the series **IIIc** > **IIa** ≈ **IIId** > **IIb** > **IIIa** (Fig. 1).

The corrosion protection factor *Z* was calculated by the formula

$$Z = (K_{n1} - K_{n2})/K_{n1} \times 100\%,$$

where K_{n1} and K_{n2} are, respectively, the rates of uniform corrosion in the absence and in the presence of inhibitor; the values of *Z* for the compounds tested are as follows: **IIIa**, 21%; **IIb**, 37%; **IIa**, 49%; **IIId**, 53%; **IIIc**, 66%.

Analysis of the counterion structure in salts **IIa**, **IIb**, **IIIa**, **IIIc**, and **IIId** shows that 2-aminophenylammonium cation in **IIIc** is the most promising from the viewpoint of physical adsorption on the electrode surface. The presence therein of π-electrons favors stronger coordination to *d*-metal [1], while the free amino group possessing a lone electron pair favors electron density redistribution upon coordination at the metal surface. Furthermore, possible protonation of the free amino group in aqueous HCl medium gives rise to an additional center which may be involved in the formation of an electrical double layer at the electrode surface. In any case, these competing processes lead to a larger reduction of K_n as compared to other cations in salts **IIa**, **IIb**, **IIIa**, and **IIId**.

Among salts **IIIa**, **IIIc**, and **IIId** having the same anion but different cations, the lowest anticorrosive

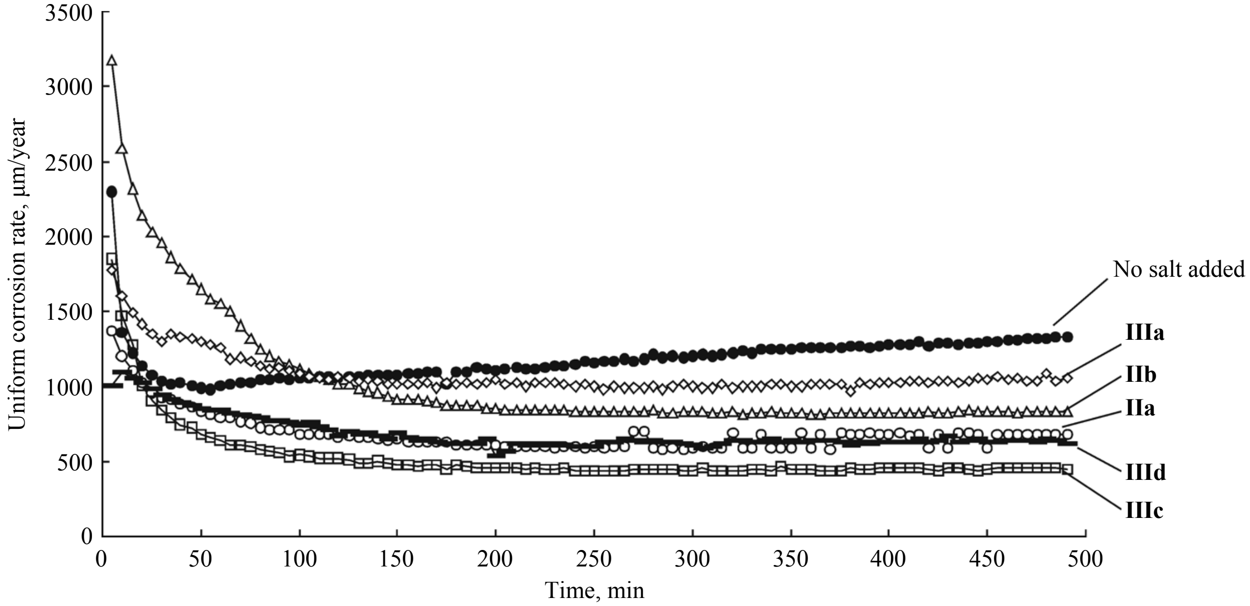


Fig. 1. Rate of uniform corrosion of mild steel (St3) in 1 M aqueous HCl; concentration of salts **II** and **III** 1×10^{-5} M.

effect was observed for salt **IIIa** with methylammonium cation. In order to rationalize higher efficiency of 1-methylpiperazinium salt **IIId** we made use of the data of [11], where adsorption of some piperazine derivatives was studied by semiempirical quantum chemical calculations. It was shown that under acid corrosion conditions most piperazine derivatives are initially protonated at the nitrogen atom (N^1–Me). The subsequent physical adsorption on the metal surface may involve either one or both amino nitrogen atoms. In the first case, the piperazine fragment is fixed orthogonally to the surface through the N^4 atom possessing a larger positive charge (vertical adsorption), while in the second the piperazine ring in a *chair* conformation is oriented parallel to the surface (flat adsorption). In both cases, the adsorption is followed by deprotonation.

The data for alkylammonium 5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olates **IIa** and **IIb** revealed appreciably higher inhibitory effect of the methylammonium salt as compared to benzylammonium. The reduction of K_n in going from **IIa** to **IIb** may be rationalized taking into account steric factor [12]. In the case of methylammonium salt **IIa** a larger number of relatively small coordinating cations per unit surface area could fit the electrode. In the bulkier benzylammonium cation of salt **IIb** conjugation with the π-electron system is weakened due to the presence of methylene spacer, so that its inhibitory effect is lower.

Comparison of the anticorrosive properties of salts **IIa** and **IIIa** having the same cation but different anions shows a stronger inhibitory effect of salt **IIa** with 5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olate anion. This may result from combined action of several factors. Presumably, electrolytic dissociation of **IIa** in aqueous HCl eventually leads to orientation of the cations and anions at the cathode and anode regions, respectively, and the system reaches an equilibrium state. In analogous process with compound **IIIa** a part of anions are likely to act as chelating agent (by means of the $^{-}O–C^4=C^3–C=O$ fragment) toward Fe^{2+} ions [13] which are transferred from the electrode to acid medium. As a result, compensation processes at the anode slow down, while polarization resistance of the system increases, which reduces the inhibitory effect of salt **IIIa** as compared to **IIa**.

Raising the concentration to 10^{-4} M considerably increases the inhibitory effect of salt **IIb** ($Z = 73\%$, Fig. 2) and, to a lesser extent, the effect of **IIIc** ($Z = 78\%$, Fig. 2).

In summary, we have found that the use of readily accessible alkylammonium polyfluoro-2-oxo-2*H*-chromen-4-olates **II** and **III** as metal corrosion inhibitors is limited by their poor solubility in aqueous media. The examined compounds at a relatively low concentration (10^{-4}–10^{-5} M) considerably reduce the rate of uniform corrosion of mild steel in aqueous HCl solution, which favors development of resource-saving technologies.

EXPERIMENTAL

The melting points were measured in open capillaries on a Stuart SMP3 melting point apparatus and are uncorrected. The IR spectra were recorded on

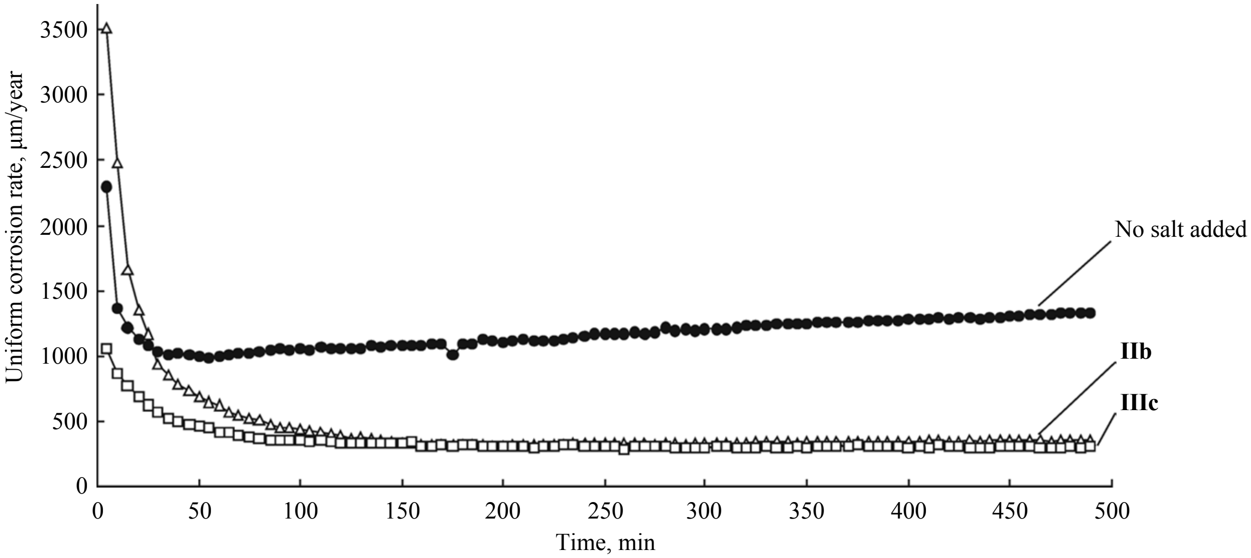


Fig. 2. Rate of uniform corrosion of mild steel (St3) in 1 M aqueous HCl; concentration of salts **IIb** and **IIIc** 1×10^{-4} M.

a Perkin Elmer Spectrum One spectrometer from samples dispersed in mineral oil. The ^{1}H, ^{13}C, and ^{19}F NMR spectra were measured in DMSO-d_6 on a Bruker DRX-400 spectrometer at 400, 100, and 376.4 MHz, respectively, using tetramethylsilane (^{1}H, ^{13}C) or C_6F_6 (^{19}F) as reference. The elemental compositions were determined on Perkin Elmer PE 2400 Series II and Thermo Scientific EA 1108 analyzers.

The rates of uniform corrosion (K_n) of mild steel (St3) and corrosion inhibition efficiencies of compounds **IIa**, **IIb**, **IIIa**, **IIIc**, and **IIId** were determined at room temperature by measuring polarization resistance with the aid of an Ekspert-004 universal automatic corrosion meter and a two-electrode probe (*Ekoniks-Ekspert* Ltd., Moscow). The electrodes, each of 0.00078 m^2 surface area, were made of St3 mild steel (Cu, 0.05–0.15%; Mn, 0.40–0.65%; P ≤ 0.04%, Cr ≤ 0.30%, S ≤ 0.05). Each K_n value given in Figs. 1 and 2 was determined as average of four parallel measurements, the mean-square deviation being no more than 3%.

Initial coumarins **Ia**–**Ic** were synthesized according to the procedures reported in [14], and salts **IIa** and **IIb** were prepared as described in [6].

Ammonium polyfluoro-3-(ethoxycarbonyl)-2-oxo-2*H*-chromen-4-olates IIIa–IIIe (*general procedure*). Coumarin **Ia**–**Ic**, 1 mmol, was dissolved in 20 mL of ethanol, 1 mmol of the corresponding amine was added, and the mixture was stirred for 25°C until the reaction was complete (TLC). The solvent was removed, and the solid residue was recrystallized from appropriate solvent.

Methylammonium 3-(ethoxycarbonyl)-6,7,8-trifluoro-2-oxo-2*H*-chromen-4-olate (IIIa). Yield 92%, yellow powder, mp >350°C (from Et_2O). IR spectrum, ν, cm^{-1}: 3084, 2985 (CH, H_3N^+), 1707 (C^2=O), 1664, 1647 (C=O, ester), 1575, 1521, 1449, 1412 (C=C), 1050, 1030, 996 (C–F). ^{1}H NMR spectrum, δ, ppm: 1.20 t (3H, $CH_2C\mathbf{H}_3$, J = 7.1 Hz), 2.38 s (3H, NCH_3), 4.07 q (2H, $C\mathbf{H}_2CH_3$, J = 7.1 Hz), 7.50 d.d.d (1H, 5-H, J = 10.7, 8.6, 2.2 Hz). ^{19}F NMR spectrum, δ_F, ppm: 5.54 m, 7.79 m, 19.47 m (1F each). Found, %: C 49.75; H 4.10; F 17.74; N 4.09. $C_{13}H_{12}F_3NO_5$. Calculated, %: C 48.91; H 3.79; F 17.85; N 4.39.

Benzylammonium 3-(ethoxycarbonyl)-6,7,8-trifluoro-2-oxo-2*H*-chromen-4-olate (IIIb). Yield 81%, white powder, mp 169°C (from CCl_4). IR spectrum, ν, cm^{-1}: 3098, 2986, 2902 2731 (C–H, H_3N^+), 1700 (C^2=O), 1637 (C=O, ester), 1574, 1519, 1486 (C=C), 1033, 999 (C–F). ^{1}H NMR spectrum, δ, ppm: 1.20 t (3H, $CH_2C\mathbf{H}_3$, J = 7.1 Hz), 4.05 s (2H, $C\mathbf{H}_2Ph$), 4.06 q (2H, $C\mathbf{H}_2CH_3$, J = 7.1 Hz), 7.38–7.52 m (6H, Ph, 5-H), 8.15 br.s (3H, H_3N^+). ^{19}F NMR spectrum, δ_F, ppm: 5.47 m, 7.78 m, 19.45 m (1F each). Found, %: C 57.77; H 4.18; F 14.27; N 3.69. $C_{19}H_{16}F_3NO_5$. Calculated, %: C 57.72; H 4.08; F 14.42; N 3.54.

2-Aminoanilinium 3-(ethoxycarbonyl)-5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olate (IIIc). Yield 64%, white powder, mp 174°C (from EtOH). IR spectrum, ν, cm^{-1}: 3422, 3352, 3248 (NH_2, C–H), 3024, 2611 (H_3N^+), 1720 (C^2=O), 1677, 1662, 1632 (C=O, ester), 1540, 1522, 1500, 1476 (δNH, C=C), 1047, 1014 (C–F). ^{1}H NMR spectrum, δ, ppm: 1.20 t (3H, $CH_2C\mathbf{H}_3$, J = 7.1 Hz), 4.08 q (2H, $C\mathbf{H}_2CH_3$, J = 7.1 Hz), 6.84–7.00 m (4H, C_6H_4), 7.51 d.d.d (1H, 5-H, J = 10.6, 8.5, 2.3 Hz), 7.66 br.s (5H, H_2N, H_3N^+). ^{13}C NMR spectrum, δ_C, ppm: 14.27 s ($CH_2\mathbf{C}H_3$), 58.79 s ($\mathbf{C}H_2CH_3$), 93.58 s (C^3), 106.03 d.d (C^{4a}, J = 19.0, 3.4 Hz), 118.73 br.s ($C^{1'}$), 119.98 s ($C^{3'}$), 122.55 s ($C^{4'}$), 129.55 br.s ($C^{2'}$), 138.11 d.d.d (C^5, J = 250.0, 12.5, 3.1 Hz), 139.15–139.81 m (C^{8a}, C^8), 140.78 d.d.d (C^6, J = 250.8, 17.5, 12.8 Hz), 145.33 d.d.d (C^7, J = 243.0, 10.6, 1.3 Hz), 159.89 s (C^2), 167.04 s (C^4), 170.19 s (3-CO). ^{19}F NMR spectrum, δ_F, ppm: 5.89 m, 7.90 m, 19.71 m (1F each). Found, %: C 54.51; H 3.97; F 14.27; N 6.92. $C_{18}H_{15}F_3N_2O_5$. Calculated, %: C 54.55; H 3.82; F 14.38; N 7.07.

1-Methylpiperazinium 3-(ethoxycarbonyl)-6,7,8-trifluoro-2-oxo-2*H*-chromen-4-olate (IIId). Yield 69%, white powder, mp 141°C (from Et_2O). IR spectrum, ν, cm^{-1}: 3085, 2980, 2907 2806 (C–H, NCH_3, H_2N^+), 1726 (C^2=O), 1677, 1646 (C=O, ester), 1554, 1512, 1478 (C=C), 1046, 1032 (C–F). ^{1}H NMR spectrum, δ, ppm: 1.21 t (3H, $CH_2C\mathbf{H}_3$, J = 7.1 Hz), 2.22 s (3H, CH_3), 2.80 m (8H, NCH_2CH_2N), 4.08 q (2H, $C\mathbf{H}_2CH_3$, J = 7.1 Hz), 7.52 d.d.d (1H, 5-H, J = 10.7, 8.6, 2.3 Hz), 8.45 br.s (2H, H_2N^+). ^{19}F NMR spectrum, δ_F, ppm: 5.62 m, 7.81 m, 19.53 m (1F each). Found, %: C 52.75; H 5.10; F 14.74; N 7.09. $C_{17}H_{19}F_3N_2O_5$. Calculated, %: C 52.58; H 4.93; F 14.68; N 7.21.

Benzylammonium 3-(ethoxycarbonyl)-5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olate (IIIe). Yield 68%, white powder, mp 176°C (from CCl_4). IR spectrum, ν, cm^{-1}: 3035, 2609 (C–H, H_3N^+), 1694 (C^2=O), 1647, 1620 (C=O, ester), 1580, 1518, 1488 (C=C), 1028, 996 (C–F). ^{1}H NMR spectrum, δ, ppm: 1.20 t (3H, $CH_2C\mathbf{H}_3$, J = 7.1 Hz), 4.05 s (2H, $C\mathbf{H}_2Ph$), 4.06 q (2H, $C\mathbf{H}_2CH_3$, J = 7.1 Hz), 7.37–7.47 m (5H, Ph), 8.16 br.s (3H, H_3N^+). ^{19}F NMR spectrum, δ_F, ppm:

–5.32 m, 0.96 m, 6.56 m, 14.20 m (1F each). Found, %: C 55.24; H 3.65; F 18.29; N 3.13. $C_{19}H_{15}F_4NO_5$. Calculated, %: C 55.21; H 3.66; F 18.39; N 3.39.

This study was performed under financial support by the Council for Grants at the President of the Russian Federation (project no. NSh-3656.2014.3) and by the Ural Branch of the Russian Academy of Sciences (project no. 12-M-123-2045).

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