ISSN 1070-4280, Russian Journal of Organic Chemistry, 2006, Vol. 42, No. 12, pp. 1838–1844. © Pleiades Publishing, Inc., 2006.
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Reactions of 5,6,7,8-Tetrafluoro-4-hydroxy-2*H*-chromen-2-ones with Methylamine

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Received July 20, 2005

Abstract—5,6,7,8-Tetrafluoro-4-hydroxy-2*H*-chromen-2-one reacts with methylamine to give methylammonium 5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olate, regardless of the solvent. The reaction of 3-acetyl-5,6,7,8-tetrafluoro-4-hydroxy-2*H*-chromen-2-one with the same amine in ethanol or acetonitrile leads to the formation of methylammonium 3-acetyl-5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olate, while in dimethyl sulfoxide 5,6,8-trifluoro-7-methylamino-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione is formed. The latter is also formed in the reaction of 5,6,7,8-tetrafluoro-4-hydroxy-3-(1-iminoethyl)-2*H*-chromen-2-one with methylamine in DMSO, whereas in ethanol and acetonitrile 5,6,7,8-tetrafluoro-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione is obtained. 5,6,7,8-Tetrafluoro-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione reacts with methylamine, yielding 7-mono- or 5,7-bis(methylamino)-substituted derivatives.

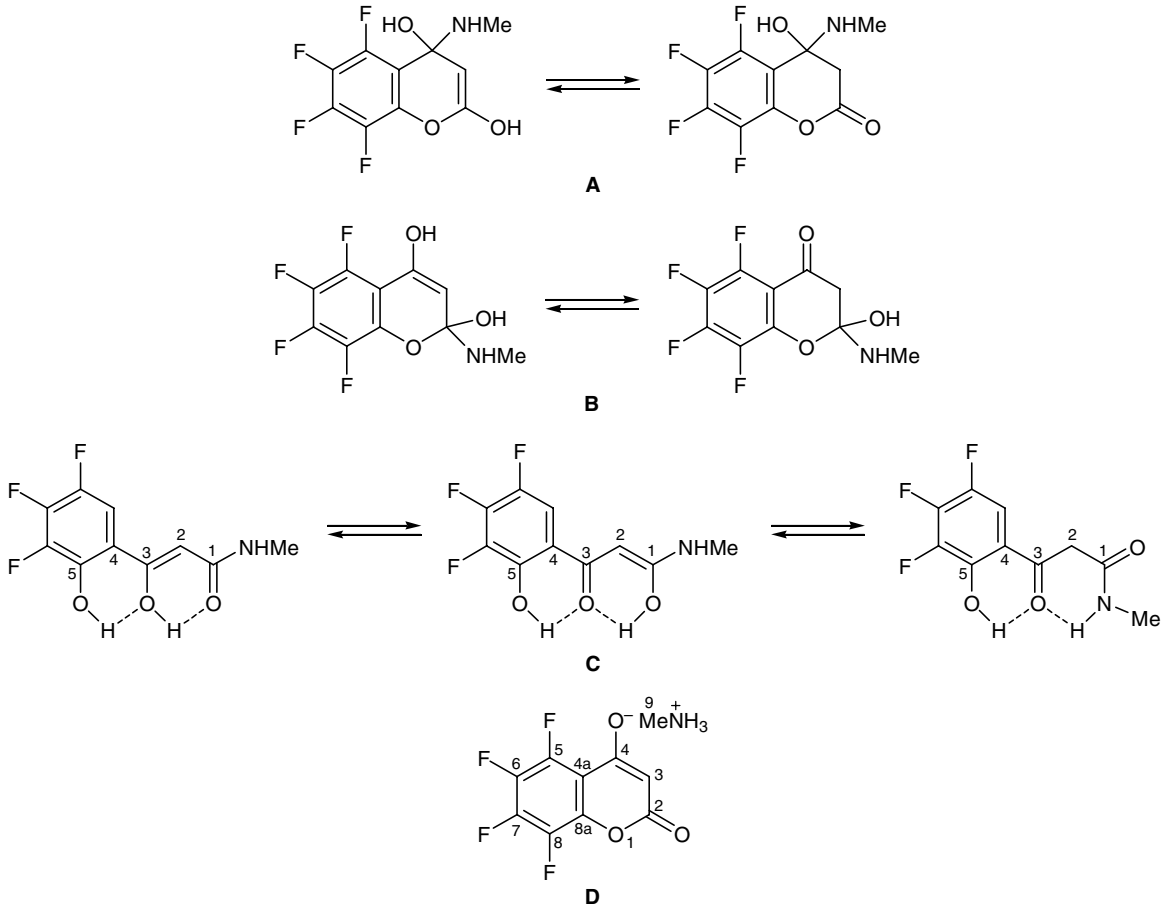
DOI: 10.1134/S107042800612013X

Coumarin derivatives, both synthetic and isolated from natural sources, are used as fluorescent and luminescent markers, dyes, medicines (anticoagulants, anticarcinogenic, and antiallergic), and rodenticides [1]. However, fluorine-containing coumarin derivatives have been studied very poorly. Unsubstituted and 4-halogen-substituted coumarins were reported to react with ammonia and primary amines at the double bond in the pyran ring to give the corresponding addition products [2] and/or at the lactone moiety with subsequent ring opening to give *o*-hydroxycinnamic acid amides [3]. 4-Hydroxycoumarins react with primary amines with formation of condensation product at the hydroxy group, but cleavage of the α-pyrone ring is also possible [4]. Introduction into the 3-position of 4-hydroxycoumarin of a substituent having its own electrophilic center, e.g., acetyl group, makes the nucleophilic reaction pattern more complex [5]. We previously described reactions of 5,6,7,8-tetrafluoro-4-hydroxycoumarin derivatives with ammonia and morpholine, which followed mainly the aromatic nucleophilic substitution scheme (the fluorine atom on C^7 was replaced) [6].

In the present work we examined reactions of 4-hydroxy-, 3-acetyl-4-hydroxy-, and 3-acetimidoyl-4-hydroxy-5,6,7,8-tetrafluoro-2*H*-chromen-2-ones **I**–**III** with methylamine. Molecules of coumarins **I**–**III** possess four nonequivalent electrophilic centers which may undergo attack by the nucleophile. These are (1) the C^2 atom [the reaction should be accompanied by opening of the pyran ring to give 3-oxo-3-(3,4,5,6-tetrafluoro-2-hydroxyphenyl)prop-2-enamide derivatives], (2) C^4 atom attached to the hydroxy group, and (3, 4) activated C^7 and C^5 atoms in the fluoroaromatic ring. Compounds **II** and **III** contain an additional electrophilic center, the carbonyl (imidoyl) carbon atom (C^9).

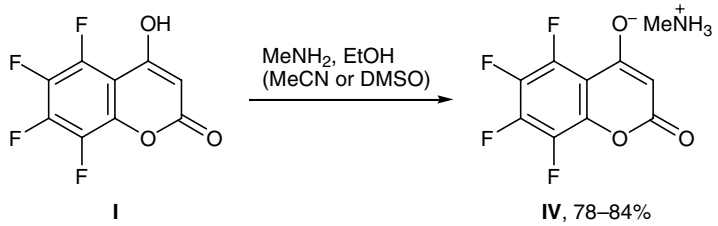
We found that 4-hydroxycoumarin **I** reacts with methylamine in acetonitrile, ethanol, or DMSO to give compound **IV**. In keeping with its elemental composition, compound **IV** can be assigned four alternative structures: 5,6,7,8-tetrafluoro-4-methylamino-4*H*-chromene-2,4-diol (**A**; methylamine addition at the double C^3=C^4 bond), 5,6,7,8-tetrafluoro-2-methylamino-2*H*-chromene-2,4-diol (**B**; methylamine addition at the C^2=O carbonyl group), 3-hydroxy-*N*-methyl-3-(3,4,5,6-tetrafluoro-2-hydroxyphenyl)prop-2-enamide (**C**; opening of the pyran ring at the C^2–O bond), and methylammonium salt **D**. Structures **A**–**C** could give rise to different tautomeric forms (Scheme 1). The

Scheme 1.

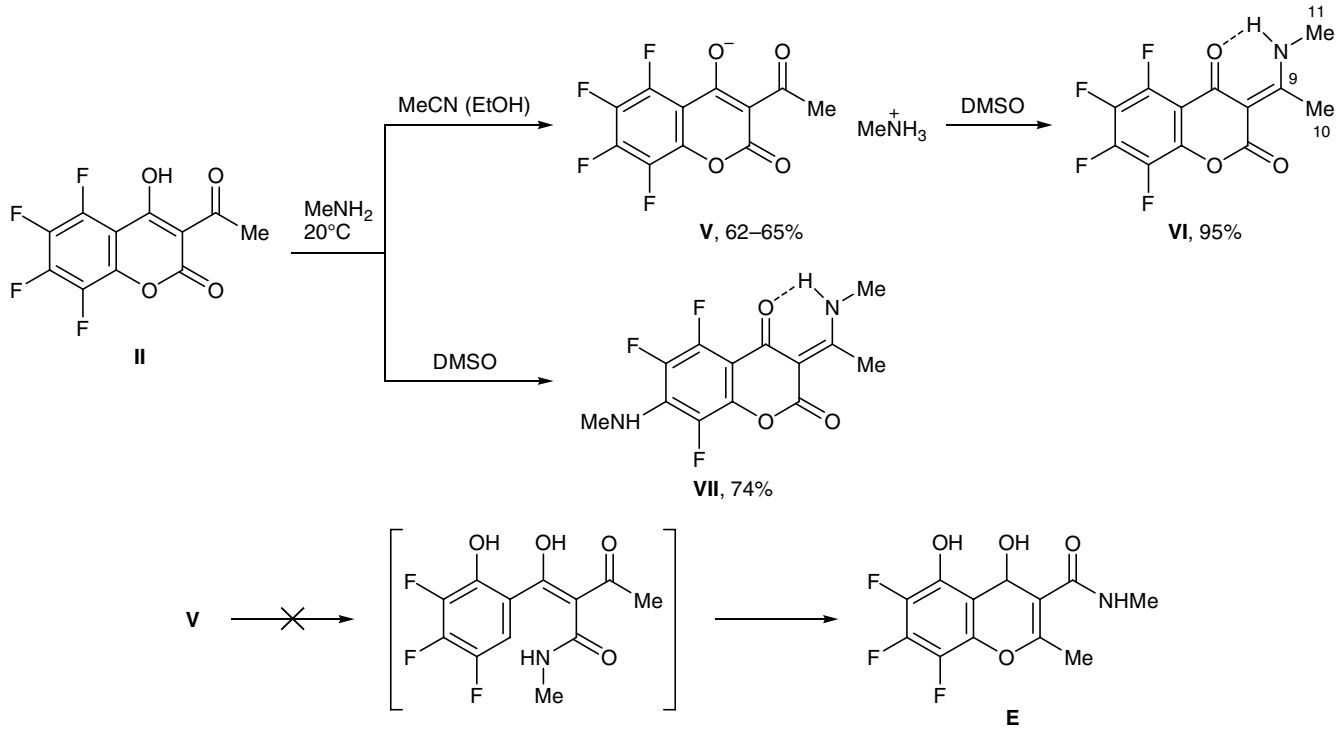


product structure was determined on the basis of the IR and ^{1}H and ^{13}C NMR data. The IR spectrum of **IV** contained a high-frequency absorption band at 1708 cm^{-1} which is most likely to result from stretching vibrations of the lactone carbonyl group in coumarins **A** or **D** rather than from the carbonyl group in the oxo tautomers of **B** and **C**. Only one set of signals was observed in the ^{1}H and ^{19}F NMR spectra of the product in DMSO-d_6, indicating that compound **IV** exists as a single tautomer. The ^{1}H NMR spectrum lacked two-proton signal from methylene protons, which should appear in the spectra of oxo tautomers of **A**–**C**; no downfield signals were observed from protons in the hydroxy and amino groups involved in intramolecular hydrogen bond with the carbonyl group; such signals are typical of the enol tautomers of **C**. The spectrum contained a one-proton singlet at δ ~4.49 ppm (CH) and a broadened three-proton singlet at δ ~7.66 ppm; the latter could be attributed to protons of the hydroxy and amino groups in the enol forms of **A** and **B** and salt **D**. It is quite probable that compound **IV** in the crystalline state exists mainly as oxo tautomer, and in DMSO solution, as enol tautomer. However, the ^{13}C NMR spectrum of a solution of **IV** in DMSO-d_6 revealed the presence of two downfield signals (see Experimental) assignable to C^2 and C^4 in

Scheme 2.



Scheme 3.



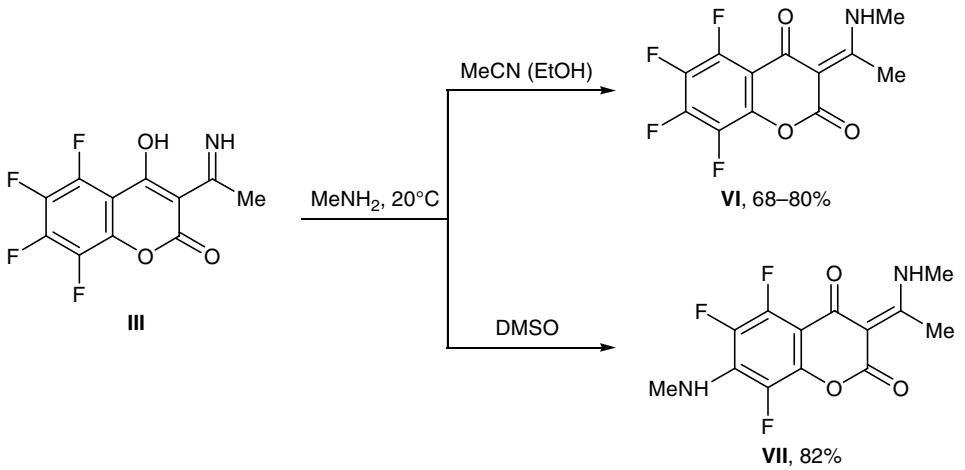
ammonium salt **D**. Coumarins **A** and **B** each should give rise to only one downfield signal belonging to C^2 or C^4, respectively, while in the spectrum of amide **C** three downfield signals (from C^1, C^3, and C^5) should appear. Therefore, compound **IV** was assigned structure **D** (Scheme 2). We failed to involve salt **IV** in further transformations; it remained unchanged under mild conditions, while under more severe conditions (heating in boiling toluene) an intractable mixture of products was formed.

The direction of the reaction of 3-acetyl-4-hydroxycoumarin **II** with methylamine was found to depend on the solvent nature. Compound **II** reacted with methylamine in acetonitrile and ethanol at room temperature to give methylammonium salt **V** at the 4-hydroxy group (Scheme 3), i.e., in a way similar to the transformation of 4-hydroxycoumarin **I** (Scheme 2). The structure of **V** was assigned on the basis of the same considerations as those given above for compound **IV**. The IR spectrum of **V** contained a high-frequency carbonyl absorption band (1715 cm^{-1}) typical of lactone group, and only one set of signals was present in the ^1H NMR spectrum recorded in DMSO-d_6. The ^1H NMR spectrum contained signals from the methyl group protons and a three-proton singlet belonging to the MeN^+H_3 cation (see Experimental). No molecular ion peak was found in the mass spectrum of **V**, and the base peak and the fragmentation pattern corresponded to the spectrum of initial coumarin **II**.

Unlike salt **IV**, compound **V** turned out to be unstable in DMSO; on storage at room temperature it was converted into 5,6,7,8-tetrafluoro-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione (**VI**). In keeping with the analytical and ^1H NMR data, an alternative structure of 5,6,7,8-tetrafluoro-*N*,2-dimethyl-4-oxo-4*H*-chromene-3-carboxamide (**E**) is also possible; structure **E** could be formed via cleavage of the pyran ring as a result of attack by methylamine on C^2 and subsequent recyclization. The choice between structures **VI** and **E** was made on the basis of the IR and mass spectra. The mass spectrum of the product contained the molecular ion peak with *m*/*z* 289 (I_{rel} 100%) and strong peaks corresponding to fragmentation of dihydrochromene-2,4-dione (see Experimental). In the IR spectrum of **VI** we observed a high-frequency carbonyl absorption band at 1710 cm^{-1}, typical of lactone fragment; no such band could appear in the spectrum of 4-oxochromene-3-carboxamide **E**.

In the reaction of coumarin **II** with methylamine in DMSO we isolated 5,6,8-trifluoro-7-methylamino-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione (**VII**) (Scheme 3); compound **VII** was formed as a result of two concurrent processes: condensation of one methylamine molecule at the acetyl

Scheme 4.



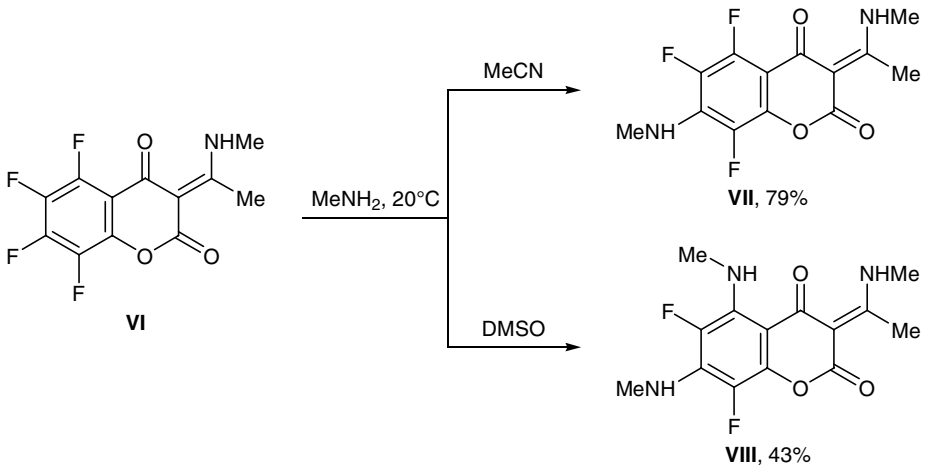
fragment (C^9) and replacement of the fluorine atom on C^7 by the second amine molecule. Analogous reaction pattern was observed previously [5] in the reaction with coumarin **III**.

By treatment of 3-acetimidoyl-5,6,7,8-tetrafluoro-4-hydroxy-2*H*-chromen-2-one (**III**) with methylamine in acetonitrile or ethanol at room temperature we obtained chromenedione **VI** (Scheme 4) due to transamination with more basic methylamine (pK_b 2.34 and 4.2 for methylamine and aqueous ammonia, respectively [7]). The same reaction in DMSO gave 5,6,8-trifluoro-7-methylamino-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione (**VII**) (Scheme 4). We also tried to effect more profound transformations of 4-hydroxycoumarins by the action of methylamine. For this purpose, gaseous methylamine was bubbled for a long time through a solution of chromenedione **VI**. Depending on the solvent, we isolated 7-mono- and 5,7-bis(methylamino)-substituted chromenediones **VII** and **VIII** (Scheme 5). The formation of compound **VIII** was accompanied by strong tarring, and the yield of **VIII** was poor.

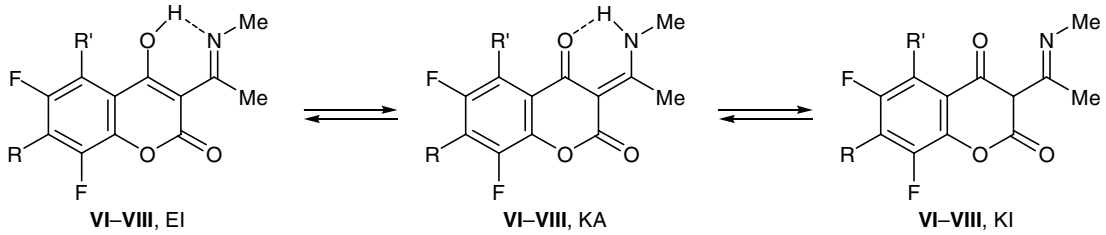
Chromene-2,4-diones **VI–VIII** could give rise to keto–enol and amine–enimine tautomerism; therefore, they can exist as one, two, or three tautomers (Scheme 6). The ^1H, ^{19}F, and ^{13}C NMR spectra of compounds **VI–VIII** in DMSO-d_6 contained only one set of signals corresponding to ketoenamine tautomer KA. The signal from the methyl protons on the nitrogen atom appeared in the ^1H NMR spectrum as a doublet (J = 5.0 Hz) due to coupling with the NH proton; the signal from the latter was located in a weak field (δ ~13 ppm) due to formation of intramolecular hydrogen bond with the carbonyl group.

Thus we have shown that 5,6,7,8-tetrafluoro-4-hydroxycoumarins **I–III** react with methylamine in different ways, depending on the conditions and substrate structure. Unlike analogous transformations of

Scheme 5.



Scheme 6.



non-fluorinated 4-hydroxycoumarins [4], neither pyran ring opening nor replacement of the hydroxy group occurred in reactions with compound **I**–**III**. In the case of 5,6,7,8-tetrafluoro-4-hydroxy-2*H*-chromen-2-one (**I**), the main process is formation of the corresponding ammonium salt, regardless of the solvent used. Presumably, the acidity of the hydroxy proton increases due to the presence of electron-withdrawing fluorine atoms in molecule **I**. 3-Acetyl-5,6,7,8-tetrafluoro-4-hydroxy-2*H*-chromen-2-one (**II**) with methylamine gives salt **V** only in acetonitrile and ethanol, while in DMSO condensation of methylamine at the acetyl fragment and replacement of the 7-fluorine atoms by the second methylamine molecule occur, leading to chromene-2,4-dione (**VII**). Likewise, 3-acetimidoyl-5,6,7,8-tetrafluoro-4-hydroxy-2*H*-chromene-2-one (**III**) reacts with methylamine in DMSO to give compound **VII**. The formation of 7-methylamino derivative **VII** in DMSO can be rationalized taking into account that the electron-withdrawing power of the fluorinated benzene ring and the ability of fluorine atom to undergo nucleophilic replacement considerably depend on the solvent polarity (ε = 49, 36.2, and 24.3 for DMSO, acetonitrile, and ethanol, respectively [8]). Presumably, the formation of coumarin **VII** involves initial condensation of methylamine at the acyl fragment, and replacement of the fluorine atom occurs only in the next step, for salt **V** in DMSO is transformed into chromenedione **VI**. Furthermore, just compound **VI** was obtained from coumarin **III** in ethanol or acetonitrile, while fluorine replacement product was not isolated at all.

The fact that no salt like **IV** or **V** was obtained from 3-acetimidoylcoumarin **III** may be explained assuming that the latter, unlike compounds **I** and **II**, in solution exists as ketoenamine tautomer (cf. **VI**–**VIII**). Therefore, it cannot form a salt at the hydroxy group but readily undergoes transamination by the action of methylamine.

Our results led us to conclude that reactions of fluorine-containing 4-hydroxycoumarins **I**–**III** with methylamine, depending on the substrate structure and solvent nature, involve concurrent salt formation at the hydroxy group, condensation at the acyl fragment, and nucleophilic aromatic substitution of fluorine atom.

EXPERIMENTAL

The IR spectra were recorded on a Perkin–Elmer Spectrum I spectrometer from samples dispersed in mineral oil. The ^{1}H, ^{13}C, and ^{19}F NMR spectra were measured from solutions in DMSO-d_6 on a Bruker DRX-400 spectrometer at 400, 100.6, and 376 MHz, respectively, using TMS (^{1}H and ^{13}C) and C_6F_6 (^{19}F) as internal references. The elemental compositions were determined on a Perkin–Elmer PE-2400 Series II CHNS-O analyzer. The mass spectra were obtained on a Varian MAT-311A instrument.

Initial coumarins **I**–**III** were synthesized according to the procedures described in [9].

Methylammonium 5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olate (IV). Excess gaseous methylamine was bubbled through a solution of 234 mg (1 mmol) of coumarin **I** in 20 ml of DMSO, acetonitrile, or ethanol at 20°C. When the initial compound disappeared (TLC), the solution was evaporated, and the solid residue was recrystallized from chloroform. Yield 207 mg (78%, DMSO), 215 mg (81%, acetonitrile), 223 mg (84%, ethanol), pale yellow powder, mp 172–173°C. IR spectrum, ν, cm^{-1}: 3205, 3061, 2769, 2583, 1614 (NH^+); 1708 (C=O); 1654 (C=C); 1554, 1520 (C=C_{arom}); 1021 (C–F_{arom}). ^{1}H NMR spectrum, δ, ppm: 2.41 s (3H, CH_3), 4.49 s (1H, =CH), 7.66 br.s (3H, NH_3^+). ^{19}F NMR spectrum, δ_F, ppm: –5.15 m (1F), 1.22 m (1F), 5.99 m (1F), 15.32 m (1F). ^{13}C NMR spectrum, δ_C, ppm: 24.48 s (C^9), 85.1 s (C^3), 109.9 m (C^{4a}), 135.06 d.m (C^6, C^7, J_{CF} = 244.7 Hz), 139.8 m (C^{8a}), 140.37 d.m (C^5, J_{CF} = 249.2 Hz), 144.09 (C^8, J_{CF} = 255.6 Hz), 162.0 br.m (C^4), 172.2 br.m (C^2). Found, %: C 45.27; H 2.59; F 28.90; N 5.51. $C_{10}H_7F_4NO_3$. Calculated, %: C 45.30; H 2.66; F 28.66; N 5.28.

Methylammonium 3-acetyl-5,6,7,8-tetrafluoro-2-oxo-2*H*-chromen-4-olate (V) was obtained in a simi-

lar way from 276 mg (1 mmol) of coumarin **II**. The product was recrystallized from hexane. Yield 190 mg (62%, acetonitrile), 200 mg (65%, ethanol), pale yellow powder, mp 175–176°C. IR spectrum, ν, cm^{-1}: 3200, 2870, 1629 (NH); 1715 (C^2=O); 1650 (C^9=O); 1566, 1520, 1510 (C=C); 1028 (C–F_{arom}). ^{1}H NMR spectrum, δ, ppm: 2.31 s (3H, CH_3), 2.40 s (3H, CH_3), 7.61 br.s (3H, NH_3). ^{19}F NMR spectrum, δ_F, ppm: –5.61 m (1F), 0.52 m (1F), 6.91 m (1F), 14.62 m (1F). Mass spectrum, *m/z* (I_{rel}, %): 276 (64) $[M - NH_2Me]^+$, 261 (23) $[M - NH_2Me - Me]^+$, 234 (49) $[M - NH_2Me - COMe]^+$, 193 (41) $[HOC_6F_4C{=}O]^+$, 192 (100) $[OC_6F_4C{=}O]^+$, 164 (17) $[C_6F_4O]^+$, 136 (14) $[C_5F_4]^+$, 117 (10) $[C_5F_3]^+$, 69 (24) $[HOC{=}C–C{=}O]^+$. Found, %: C 47.16; H 2.99; F 24.87; N 4.41. $C_{12}H_9F_4NO_4$. Calculated, %: C 46.92; H 2.95; F 24.74; N 4.56.

5,6,7,8-Tetrafluoro-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione (VI). *a*. The procedure was the same as in the synthesis of compound **IV**; 275 mg (1 mmol) of coumarin **III** was used, and the product was recrystallized from ethanol. Yield 231 mg (80%, acetonitrile), 197 mg (68%, ethanol), pale yellow powder, mp 174–175°C. IR spectrum, ν, cm^{-1}: 3194, 1594 (N–H); 1710 (C^2=O); 1654 (C^4=O); 1617, 1531, 1491 (C=C); 987 (C–F_{arom}). ^{1}H NMR spectrum, δ, ppm: 2.61 s (3H, CH_3), 3.23 d (3H, NHC**H**$_3$, $^3J_{HH}$ = 5.0 Hz), 13.40 br.s (1H, NH). ^{19}F NMR spectrum, δ_F, ppm: –2.82 m (1F), 1.89 m (1F), 11.51 m (1F), 16.74 m (1F). ^{13}C NMR spectrum, δ_C, ppm: 18.5 s (C^{10}), 31.2 s (C^{11}), 95.7 s (C^3), 107.2 m (C^{4a}), 134.99 d.m (C^6, J_{CF} = 248.4 Hz), 136.17 d.m (C^7, J_{CF} = 247.0 Hz), 138.3 m (C^{8a}), 142.54 d.m (C^5, J_{CF} = 249.8 Hz), 144.88 d.m (C^8, J_{CF} = 254.0 Hz), 160.4 d (C^2, $^4J_{CF}$ = 1.0 Hz), 176.5 br.m (C^4), 176.9 m (C^9). Mass spectrum, *m/z* (I_{rel}, %): 289 (100) M^+, 274 (42) $[M - Me]^+$, 272 (22) $[M - OH]^+$, 97 (20) $[HOC{=}C–C(=NMe)Me]^+$, 82 (20) $[HOC{=}C–C(=N)Me]^+$, 69 (30) $[HOC{=}C–C{=}O]^+$, 56 (76) $[C(=NMe)Me]^+$. Found, %: C 49.63; H 2.62; F 26.51; N 4.65. $C_{12}H_7F_4NO_3$. Calculated, %: C 49.84; H 2.44; F 26.28; N 4.84. *M* 289.19.

b. A mixture of 307 mg (1 mmol) of salt **V** and 10 ml of DMSO was stirred for 8 h. Recrystallization from ethanol gave 275 mg (95%) of compound **VI** with mp 174–175°C.

5,6,8-Trifluoro-7-methylamino-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione (VII) was synthesized as described above in *a* from 276 mg (1 mmol) of coumarin **II** in DMSO. The product was recrystallized from hexane. Yield 222 mg (74%), white powder, mp 238–239°C. IR spectrum, ν, cm^{-1}: 3434, 3404, 3318, 1582 (NH); 1711 (C^2=O); 1647 (C=N); 1607 (C=C); 1549, 1504 (C=C_{arom}); 1019 (C–F_{arom}). ^{1}H NMR spectrum, δ, ppm: 2.57 s (3H, CH_3), 3.02 d.t (3H, 7-NHC**H**$_3$, $^3J_{HH}$ = 4.1, $^5J_{HF}$ = 3.0 Hz), 3.17 d (3H, NHC**H**$_3$, J_{HH} = 5.1 Hz), 6.57 br.m (1H, 7-NH), 13.24 br.s (1H, NH). ^{19}F NMR spectrum, δ_F, ppm: –1.20 d.m (1F, 6-F, $^3J_{FF}$ = 18 Hz), 2.04 m (1F, 8-F), 15.33 d.d.m (1F, 5-F, $^3J_{FF}$ = 18, $^5J_{FF}$ = 10 Hz). Found, %: C 51.96; H 3.62; F 19.06; N 9.37. $C_{13}H_{11}F_3N_2O_3$. Calculated, %: C 52.01; H 3.69; F 18.98; N 9.33.

Following an analogous procedure, from 275 mg (1 mmol) of coumarin **III** in DMSO we obtained 246 mg (82%) of compound **VII** with mp 238–239°C.

Following an analogous procedure, from 289 mg (1 mmol) of compound **VI** in acetonitrile we obtained 237 mg (79%) of compound **VII** with mp 238–239°C.

6,8-Difluoro-5,7-bis(methylamino)-3-(1-methylaminoethylidene)-3,4-dihydro-2*H*-chromene-2,4-dione (VIII) was synthesized in a similar way from 289 mg (1 mmol) of compound **VI** in DMSO. The product was isolated by column chromatography on silica gel (40–100 μm) using chloroform–acetone (10:1) as eluent. Yield 134 mg (43%), pale yellow powder, mp 234–237°C. IR spectrum, ν, cm^{-1}: 3328, 3226, 1586 (N–H); 1687 (C^2=O), 1644 (C^4=O); 1600 (C=C); 1541, 1515 (C=C_{arom}); 960 (C–F_{arom}). ^{1}H NMR spectrum, δ, ppm: 2.58 s (3H, 10-CH_3), 2.96 d.d (3H, 5-NHC**H**$_3$, $^3J_{HH}$ = 5.5, $^5J_{HF}$ = 6.9 Hz), 2.58 m (3H, 7-NHC**H**$_3$), 3.15 d (3H, 9-NHC**H**$_3$, $^3J_{HH}$ = 5.1 Hz), 6.05 br.s (1H, NH), 8.56 br.s (1H, NH), 12.89 br.s (1H, NH). ^{19}F NMR spectrum, δ, ppm: –7.70 m (1F), 3.36 m (1F). Found, %: C 55.57; H 4.98; F 13.24; N 12.15. $C_{14}H_{15}F_2N_3O_3$. Calculated, %: C 54.02; H 4.86; F 12.21; N 13.50.

This study was performed under financial support by the Russian Foundation for Basic Research (project no. 03-03-33118), by the Russian Foundation for Support of Russian Science, and by the Ural Division, Russian Academy of Sciences (a grant for young scientists and post-graduate students).

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