Reactions of 2-ethoxycarbonyl(carboxy)-5,6,7,8-tetrafluorochromones with N-nucleophiles

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The direction of reactions of 2-ethoxycarbonyl-5,6,7,8-tetrafluoro- and 2-carboxy-5,6,7,8-tetrafluorochromones with ammonia, methylamine, hexylamine, and aniline depends on the inductive effect of the substituent at position C-2 of the chromone and on the basicity of the amine. Nucleophilic aromatic substitution of a fluorine atom takes place in the interaction of 2-ethoxycarbonyl-5,6,7,8-tetrafluorochromone with secondary amines (morpholine, *N*-methylpiperazine) and ethylenediamine.

Key words: 2-ethoxycarbonyl-5,6,7,8-tetrafluorochromone, 2-carboxy-5,6,7,8-tetrafluorochromone; primary and secondary amines, ethylenediamine; nucleophilic replacement; α,β-unsaturated ketones; nucleophilic addition at activated double bond.

It has been mentioned in the literature that 3-ethoxycarbonyl-5,6,7,8-tetrafluoro-2-methylchromone reacts with benzyl- and hydroxylamines.[1] However, no detailed information has been reported regarding the effect of substituents in molecules of 5,6,7,8-tetrafluorochromone derivatives on the reactions with nucleophilic reagents.

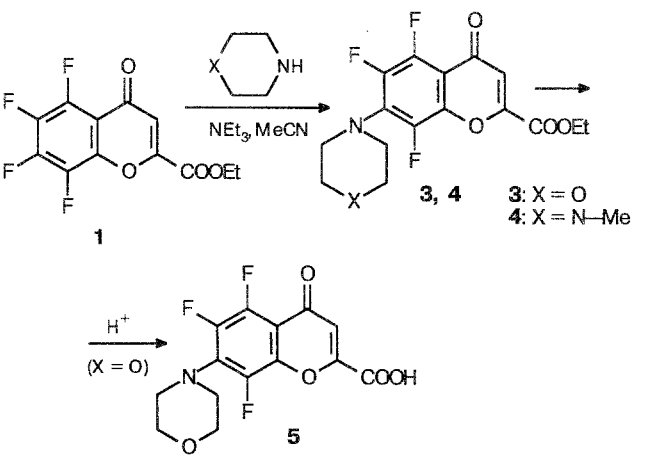
Previously[2] we have obtained 2(3)-ethoxycarbonyl- and 2(3)-carboxy-5,6,7,8-tetrafluorochromones. The analysis of their ^{1}H NMR spectra has shown that the F atoms do not affect significantly the ring current of the heterocycle. In the present work, reactions of 2-ethoxycarbonyl-5,6,7,8-tetrafluorochromone (**1**) and 2-carboxy-5,6,7,8-tetrafluorochromone (**2**) with ammonia, primary amines (methylamine, hexylamine, and aniline), secondary amines (*N*-methylpiperazine and morpholine), and ethylenediamine were studied.

The reactions of compound **1** with morpholine and *N*-methylpiperazine do not involve the heterocycle (which is typical of reactions of nonfluorinated chromones with secondary amines[3]) but give 7-aminochromone derivatives **3** and **4** in 52 % and 47 % yields, respectively (Scheme 1). Compound **3** undergoes hydrolysis on boiling with HCl to give carboxylic acid **5**.

Depending on the solvent, the reaction of chromone **1** with ethylenediamine (pK_a = 10.075, 6.985)[4] affords either piperazine-2-one derivative **6** (in a CH_2Cl_2—MeOH mixture or in MeCN) or an aromatic substitution product **7** (in DMSO) (Scheme 2). One can explain the formation of these compounds by assuming that the electron-withdrawing nature of the polyfluoroaryl ring (and, hence, the ability of a F atom to undergo nucleophilic substitution) increases significantly on passage from protic solvents (alcohol) to dipolar aprotic solvents (DMSO).[5] However, compound **6** also forms from compound **1** and ethylenediamine in acetonitrile. Probably, MeCN (ε = 36.2),[4] unlike DMSO (ε = 49),[4] cannot hinder the formation of an ionic pair of ethylenediamine and the chromone at the electrophilic carbon atom of the COOEt group or at the C-2 atom.

The behavior of chromone **1** toward primary amines is not typical of the known chromone structures.[3,6] For example, compound **1** does not react either with aniline (pK_a = 5.63)[4] in boiling benzene, or even with ammonia (pK_a = 9.247)[4] in MeCN or in boiling toluene. The treatment of compound **1** with methylamine (pK_a = 10.657)[4] gives chromone derivative **8** in a 41 % yield (Scheme 3).

Scheme 1



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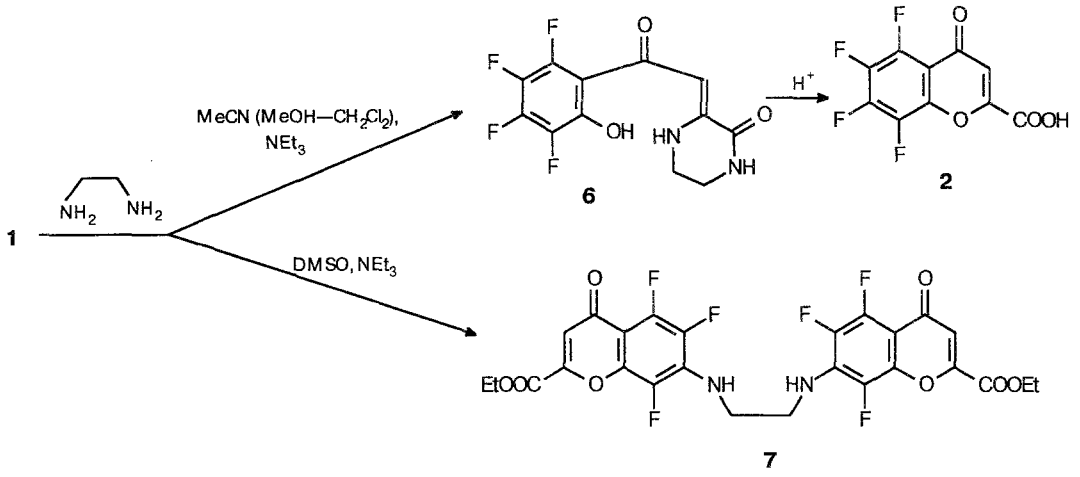
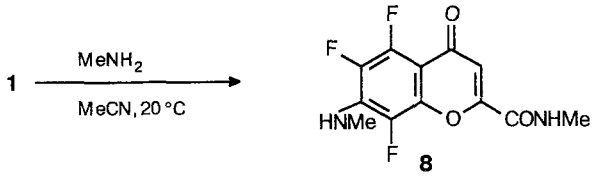
Scheme 2



Scheme 3



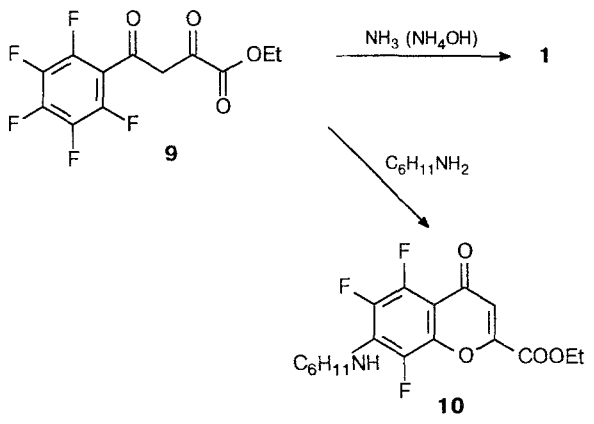
The ester group ($-I$ effect) decreases the reactivity of the chromone, which is in fact an α,β-unsaturated ketone,[3,6] probably by affecting the polarization of the carbonyl vinylog.[7] This also decreases the reactivity of the COOEt group, probably due to the compensation of the partial positive charge at the carbonyl C atom. Therefore, the basicity of the reacting amine is of great importance.

The formation of chromone **1** in a quantitative yield from ethyl pentafluorobenzoylpyruvate (**9**) and NH_3 (Scheme 4) also demonstrates the unprecedented stability of **1** toward the action of ammonia. The reaction of ester **9** with cyclohexylamine ($pK_a = 10.64$)[8] also results in a chromone structure **10** (yield 63 %).

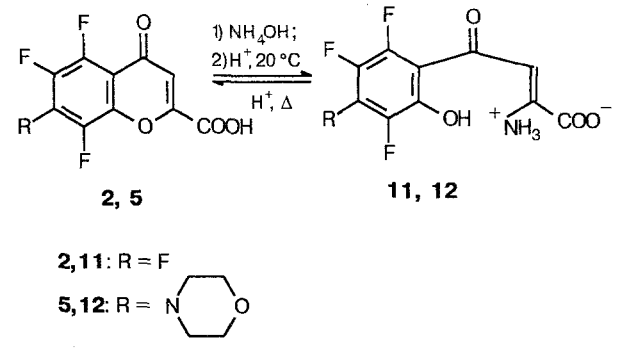
In contrast to ester **1**, chromonecarboxylic acids **2** and **5** readily undergo heterocycle opening when treated with an excess of NH_4OH, which is typical of chromones.[3,6] This reaction gave the corresponding amino acids **11** and **12**, which were isolated by acidifying the reaction mixture. The acids **11** and **12** can undergo recyclization on boiling in an acid medium to give the original compounds (Scheme 5).

Chromone **2** reacts with hexylamine ($pK_a = 10.64$)[8] to give a γ-chromanol salt **13** (Scheme 6), acidification of which affords chromanone **14** (yield 81 %). The ^1H NMR spectrum of compound **14** does not contain the signal of a CH= group but displays a triplet-degenerate AB-system of the protons of the CH_2 group in the ring. According to Scheme 6, both nucleophilic addition at the activated C=C bond and interaction at the carbonyl group of the chromone occur, which allows us to characterize chromone **2** as a typical carbonylic vinylog. This is also demonstrated by reactions of chromone **2** with the weakly basic aniline (see Scheme 6) involving the carbonyl C atom to give salt **15**.

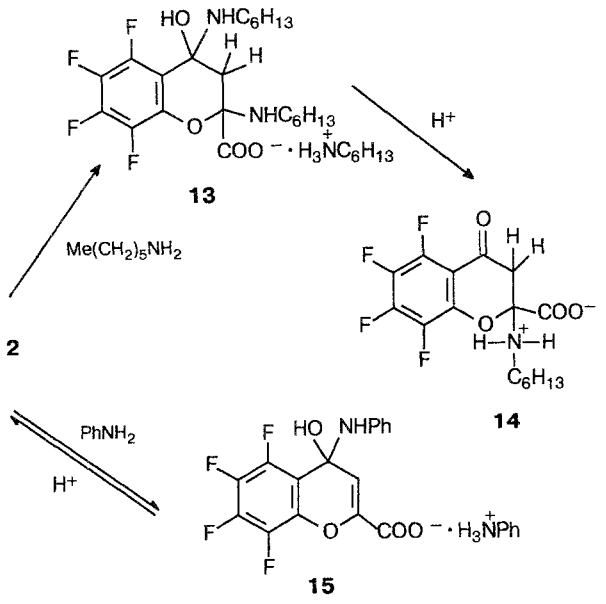
Scheme 4



Scheme 5



Scheme 6



Compound **2** reacts with methylamine to give a mixture of products that is difficult to separate. Heating this mixture in an acidic medium gives the original chromone **2**. This allows us to assume that the mixture does not contain any product of addition at the C=C bond similar to salt **13**. Most likely, opening of the chromone ring occurs (as in the reaction with NH_4OH) accompanied by the formation of 4-hydroxychrominyl, which is unstable in acidic media and analogous to compound **15**.

The ease of the reactions of 2-carboxychromone **2** with amines is probably caused by activation of the electrophilic reaction centers of the heterocycle due to the strong $+I$ effect of the carboxylate anion formed under these conditions.

Experimental

IR spectra were recorded on a Specord-75 IR spectrophotometer in the range 400—4000 cm^{-1} in vaseline oil suspensions. ^1H NMR spectra were recorded on a Tesla BS-567 A spectrometer (100 MHz) in acetone-d_6 relative to Me_4Si. ^{19}F NMR spectra were obtained on a Tesla BS-587 spectrometer (^{19}F, 75 MHz) in acetone-d_6 relative to $CFCl_3$; the digital resolution was 0.5 Hz per point, which corresponded to the accuracy of chemical shift measurement of 0.01 ppm. Mass spectra were obtained on a Varian MAT-311a mass spectrometer in the standard mode.

Ethyl pentafluorobenzoylpyruvate (9) was synthesized according to the reported procedure.[2]

2-Ethoxycarbonyl-5,6,8-trifluoro-7-morpholinochromone (3). A solution of chromone **1** (15 g, 51.7 mmol), morpholine (4.5 g, 51.7 mmol), and triethylamine (20 mL) in MeCN (50 mL) was kept for 5 h at 20 °C. The precipitate was filtered off and washed with MeCN to give 9.5 g (52 %) of compound **3**, m.p. 160—161 °C. IR, ν/cm^{-1}: 1720 (C=O, ester); 1660 (C=O, chromone); 1615 (C=C). ^1H NMR, δ (J/Hz): 1.41 (t, 3 H, $CH_2\underline{CH_3}$, J = 7); 3.40—3.58 (m, 4 H, CH_2NCH_2); 3.79 (t, 4 H, CH_2OCH_2, J = 5); 4.45 (q, 2 H, $\underline{CH_2}CH_3$, J = 7); 6.80 (s, 1 H, CH). ^{19}F NMR, δ: −149.42 (dm, 1 F); −147.14 (dm, 1 F); −145.59 (dd, 1 F). Found (%): C, 53.60; H, 3.38; F, 16.17; N, 4.07. $C_{15}H_{14}F_3NO_5$. Calculated (%): C, 53.79; H, 3.95; F, 15.95; N, 3.92.

2-Ethoxycarbonyl-5,6,8-trifluoro-7-(*N*'-methylpiperazino)-chromone (4). Chromone **4** was obtained in a similar way from chromone **1** (10 g, 34.5 mmol), *N*-methylpiperazine (3.5 g, 35.0 mmol), and NEt_3 (15 mL) in MeCN (35 mL). Recrystallization from MeCN gave 6.0 g (47 %) of compound **4**, m.p. 145 °C. IR, ν/cm^{-1}: 1720 (C=O, ester); 1650 (C=O, chromone). ^1H NMR ($CDCl_3$), δ (J/Hz): 1.44 (t, 3 H, $CH_2\underline{CH_3}$, J = 7); 2.38 (br.s, 8 H, 2 CH_2CH_2N); 3.50 (br.s, 3 H, CH_3N); 4.46 (q, 2 H, $\underline{CH_2}CH_3$, J = 7); 6.92 (s, 1 H, CH). ^{19}F NMR ($CDCl_3$), δ: −149.88 (dd, 1 F); −148.63 (dd, 1 F); −145.90 (dd, 1 F). Found (%): C, 55.34; H, 4.91; F, 15.34; N, 7.39. $C_{17}H_{17}F_3N_2O_4$. Calculated (%): C, 55.14; H, 4.63; F, 15.39; N, 7.57.

2-Carboxy-5,6,8-trifluoro-7-morpholinochromone (5). A solution of compound **3** (7 g, 19.6 mmol) in MeCOOH (150 mL) and conc. HCl (20 mL) was boiled for 6 h. The solvent was distilled off until the residue crystallized. The precipitate was filtered off and washed with water to give 4.5 g (70 %) of chromone **5**, m.p. 227—229 °C. IR, ν/cm^{-1}: 1730 (C=O, COOH); 1630 (C=O, chromone); 2600, 3150 (OH). ^1H NMR, δ (J/Hz): 3.57 (m, 4 H, CH_2NCH_2); 3.80 (t, 4 H, CH_2OCH_2, J = 4.8); 6.83 (s, 1 H, CH). ^{19}F NMR, δ: −149.53 (dm, 1 F); −147.15 (dm, 1 F); −145.70 (dd, 1 F). Found (%): C, 51.02; H, 3.35; F, 17.61; N, 4.20. $C_{14}H_{10}F_3NO_5$. Calculated (%): C, 51.07; H, 3.06; F, 17.31; N, 4.26.

3-(3,4,5,6-Tetrafluoro-2-hydroxybenzoylmethylene)piperazin-2-one (6). ***A.*** A mixture of chromone **1** (2.9 g, 10 mmol) and ethylenediamine (0.5 g, 8.32 mmol) in CH_2Cl_2 (20 mL) and MeOH (30 mL) was kept for 4 h at 20 °C. The precipitate was filtered off and washed with MeOH and with CH_2Cl_2 to give 2 g (79 %) of piperazinone **6**, m.p. 270 °C (subl.). IR, ν/cm^{-1}: 1680 (C=O, amide); 1640 (C=O); 1595 (C=C); 2600 (OH); 3170 (NH); 1550 (δNH). ^1H NMR (DMF-d_7), δ (J/Hz): 3.67 (s, 4 H, CH_2CH_2); 6.55 (d, 1 H, CH, $J_{H,F}$ = 1.2). ^{19}F NMR, (DMF-d_7), δ: −172.41 (dd, 1 F); −164.75 (ddd, 1 F); −153.05 (ddd, 1 F); −138.70 (ddd, 1 F). MS, m/z (I_{rel} (%)): 304 $[M]^+$ (54.0), 247 $[M-CONHCH_3]^+$ (100), 193 $[HOC_6F_4CO]^+$ (43.6), 111 $[M-HOC_6F_4CO]^+$ (13.4). Found (%): C, 47.03; H, 2.52; F, 24.90; N, 8.77. $C_{12}H_8F_4N_2O_3$. Calculated (%): C, 47.38; H, 2.65; F, 24.98; N, 9.21.

B. In a similar way, 1.27 g (50 %) of compound **6**, m.p. 270 °C (subl.), was obtained from chromone **1** (2.9 g, 10 mmol) and ethylenediamine in a mixture of MeCN (35 mL) and triethylamine (15 mL).

Hydrolysis of 3-(3,4,5,6-tetrafluoro-2-hydroxybenzoylmethylene)piperazin-2-one (6). A solution of compound **6** (4.2 g, 13.8 mmol) in MeCOOH (60 mL) and conc. HCl (10 mL) was boiled for 15 h. The solvent (~2/3 of the initial volume) was distilled off. A solution of conc. HCl (10 mL) in H_2O (30 mL) was added to the hot residue. After 2—3 h the precipitate was filtered off and dried in air to give 2.0 g (55 %) of chromone **2**, m.p. 209—210 °C (subl.). The physicochemical constants were in agreement with the literature data.[2]

***N,N'*-Ethylenebis(7-amino-2-ethoxycarbonyl-5,6,8-trifluorochromone) (7).** Compound **1** (5.6 g, 19.3 mmol) was dissolved in DMSO (100 mL) at 50 °C. Triethylamine (10 mL)

and ethylenediamine (0.5 g, 8.32 mmol) in DMSO (5 mL) were added, and the mixture was stirred for 1 h at 20 °C. The precipitate was filtered off, washed with MeCN, recrystallized from DMF, and dried *in vacuo* at 100—120 °C to give 2.7 g (54 %) of chromone **7**, m.p. 258 °C. IR, ν/cm^{-1}: 1725 (C=O, ester); 1635 (C=O, chromone); 1610 (C=C); 3380, 3320 (NH). ^{1}H NMR (CF_3COOH—DMF-d_7), δ (*J*/Hz): 1.16 (t, 6 H, 2 CH_2CH_3, *J* = 7); 3.73 (m, 4 H, CH_2CH_2); 4.28 (q, 2 H, CH_2CH_3, *J* = 7); 7.05 (s, 2 H, 2 CH). ^{19}F NMR (CF_3COOH—DMF-d_7), δ: −157.53 (t, 1 F); −154.42 (dd, 1 F); −141.96 (dd, 1 F). MS, *m/z* (I_{rel} (%)): 600 $[M]^+$ (11.6), 301 (100), 300 $[1/2\ M]^+$ (38.5), 272 $[1/2\ M-CO]^+$ (53.0). Found (%): C, 52.13; H, 3.19; F, 18.84; N, 4.77. $C_{26}H_{18}F_6N_2O_8$. Calculated (%): C, 52.01; H, 3.02; F, 18.99; N, 4.66.

5,6,8-Trifluoro-7-methylaminochromone-2-(*N*-methyl)carboxamide (8). A stream of methylamine was passed through a solution of chromone **1** (1.0 g, 3.45 mmol) in MeCN (15 mL) until the exothermic reaction ceased. The precipitate was filtered off and reprecipitated with water from conc. HCl to give 0.4 g (41 %) of amide **8**, m.p. 260 °C (subl.). IR, ν/cm^{-1}: 1655 (C=O, amide); 1640 (C=O, chromone); 1620 (C=C); 1550, 1580, 3320 (NH). ^{1}H NMR (CF_3COOH—CD_3COOD), δ (*J*/Hz): 2.88 (t, 3 H, $CONHCH_3$, *J* = 2.8); 3.09 (dd, 3 H, CH_3NH, *J* = 2.5); 7.09 (s, 1 H, CH). ^{19}F NMR (DMF-d_7), δ: −147.72 (dd, 1 F); −159.42 (m, 1 F); −160.28 (m, 1 F). Found (%): C, 50.52; H, 3.49; F, 19.84; N, 9.70. $C_{12}H_9F_3N_2O_3$. Calculated (%): C, 50.36; H, 3.17; F, 19.92; N, 9.79.

Interaction of ethyl pentafluorobenzoylpyruvate (9) with ammonia. Ester **9** (1 g, 3.22 mmol) was shaken with 10 % NH_4OH (10 mL) at 20 °C for several minutes, and the precipitate was filtered off to give 0.93 g (100 %) of chromone **1**, m.p. 125 °C. The physicochemical constants were in agreement with the literature data.[2]

7-Cyclohexylamino-2-ethoxycarbonyl-5,6,8-trifluorochromone (10). Cyclohexylamine (0.5 g, 5.04 mmol) in DMSO (20 mL) was added to a solution of compound **9** (0.8 g, 2.58 mmol) in CCl_4 (20 mL), then the CCl_4 was distilled off from the reaction mixture. When the temperature of the residue reached 130 °C, the Liebig condenser was replaced by a reflux condenser, and heating was continued at this temperature for 10 min. The product was precipitated with water and twice recrystallized from *n*-octane to give 0.6 g (63 %) of chromone **10**, m.p. 120—121 °C. IR, ν/cm^{-1}: 1735 (C=O, ester); 1655 (C=O, chromone); 1610 (C=C); 1540 (δNH); 3310 (NH). ^{1}H NMR (CD_3CN), δ (*J*/Hz): 1.38 (t, 3 H, CH_2CH_3, *J* = 7); 1.1—2.1 (m, 10 H, 5 CH_2); 3.68 (br.s, 1 H, CHN); 4.40 (q, 2 H, CH_2CH_3, *J* = 7); 5.0 (br.s, 1 H, NH); 6.72 (s, 1 H, CH). ^{19}F NMR, δ (*J*/Hz): −147.04 (dd, 1 F, F-5); −157.84 (m, 2 F, F-6, F-8, $J_{F\text{-}5,F\text{-}8}$ = 12.7, $J_{F\text{-}5,F\text{-}6}$ = 20.2). Found (%): C, 58.33; H, 4.92; F, 15.47; N, 3.84. $C_{18}H_{18}F_3NO_4$. Calculated (%): C, 58.54; H, 4.91; F, 15.43; N, 3.79.

2-Amino-3-(3,4,5,6-tetrafluoro-2-hydroxybenzoyl)acrylic acid (11). Acid **2** (3.1 g, 11.8 mmol) was kept in 10 % NH_4OH (75 mL) at 20 °C for 24 h. The solution was acidified to pH 3. The precipitate was filtered off and reprecipitated with water from MeOH and then with hexane from ethyl acetate to give 1.6 g (49 %) of acid **11**, m.p. 225 °C. IR, ν/cm^{-1}: 1710 (C=O, COOH); 1640 (C=O); 1610 (C=C); 2650, 2500 (OH, NH_3^+); 1575 (COO^-); 3315, 3455 (NH). ^{1}H NMR, δ (*J*/Hz): 6.63 (d, 1 H, CH, $J_{H,F}$ = 1.1); 7.92 (br.s, 1 H, NH); 9.6 (br.s, 1 H, NH); 11.1 (br.s, 2 H, 2 OH). ^{19}F NMR, δ: −137.49 (ddd, 1 F); −151.22 (ddd, 1 F); −164.31 (ddd, 1 F); −172.13 (ddd, 1 F). Found (%): C, 43.09; H, 2.02; F, 26.93; N, 5.09. $C_{10}H_5F_4NO_4$. Calculated (%): C, 43.03; H, 1.81; F, 27.23; N, 5.02.

2-Amino-3-(3,5,6-trifluoro-2-hydroxy-4-morpholinobenzoyl)acrylic acid (12) was synthesized similarly to acid **11** from compound **5** (2.0 g, 6.08 mmol) and 10 % NH_4OH (150 mL). The precipitate was washed with water to give 1.2 g (57 %) of acid **12**, m.p. 197—199 °C. IR, ν/cm^{-1}: 1705 (C=O, COOH); 1640 sh (C=O); 1610 (C=C); 2650, 2500 (OH, NH_3^+); 1575 (COO^-); 3170, 3250, 3370 (NH); 1535 (δNH). ^{1}H NMR, δ: 3.39 (m, 4 H, CH_2NCH_2); 3.76 (m, 4 H, CH_2OCH_2); 6.66 (s, 1 H, CH); 7.6 (br.s, 1 H, NH); 8.7 (br.s, 2 H, OH); 9.4 (br.s, 1 H, NH). ^{19}F NMR, δ: −139.41 (dd, 1 F); −152.45 (dd, 1 F); −159.76 (d, 1 F). Found (%): C, 48.58; H, 3.50; F, 16.87; N, 8.28. $C_{14}H_{13}F_3N_2O_5$. Calculated (%): C, 48.56; H, 3.78; F, 16.46; N, 8.28.

Hexylammonium 5,6,7,8-tetrafluoro-2,4-bishexylamino-4-hydroxychromane-2-carboxylate (13). Chromone **2** (4 g, 15.3 mmol) in water (40 mL) and $CHCl_3$ (40 mL), and hexylamine (48.4 mmol) were stirred for 3 h at 30—35 °C then cooled to 0 °C. After 1 h the salt (yellow plates) was filtered off to give 6.8 g (78 %) of salt **13**, m.p. 152—154 °C. IR, ν/cm^{-1}: 1565 (COO^-); 1645, 2190, 2610, 2680 (NH_2^+, OH); 3125 sh (NH). Found (%): C, 59.55; H, 8.56; F, 13.24; N, 7.66. $C_{28}H_{47}F_4N_3O_4$. Calculated (%): C, 59.45; H, 8.38; F, 13.43; N, 7.43.

2-Carboxy-5,6,7,8-tetrafluoro-2-hexylaminochroman-4-one (14). A solution of salt **13** (6.7 g, 11.8 mmol) in water (200 mL) and conc. HCl (20 mL) was stirred for 20 min at 20 °C. The precipitate was filtered off, washed with water, and dried *in vacuo* at 40 °C to give 3.5 g (81 %) of compound **14**, m.p. 131—133 °C. IR, ν/cm^{-1}: 1710 (C=O, COOH); 1650 (C=O, chromane); 1590, 2660 (NH_2^+); 1560 (COO^-); 3160, 3210 (NH). ^{1}H NMR, δ (*J*/Hz): 0.9 (m, 3 H, CH_3); 1.2—1.9 (m, 10 H, 5 CH_2O); 3.65 (center of an AB-system, 2 H, CH_2 in the cycle, ΔAB = 0.6, *J* = 6.8). ^{19}F NMR, δ: −137.72 (ddd, 1 F); −152.32 (ddd, 1 F); −164.62 (ddd, 1 F); −172.52 (ddd, 1 F). Found (%): C, 53.35; H, 5.02; F, 20.94; N, 3.82. $C_{16}H_{17}F_4NO_4$. Calculated (%): C, 52.90; H, 4.72; F, 20.92; N, 3.85.

Anilinium 4-anilino-5,6,7,8-tetrafluoro-4-hydroxychromane-2-carboxylate (15). Aniline (12.0 g, 129.0 mmol) was added at 35 °C to a solution of acid **2** (5.0 g, 19.0 mmol) in a mixture of acetonitrile (50 mL) and water (25 mL). The reaction mixture was kept for 30 min at 35—40 °C and cooled to 10 °C. After 2 h yellow crystals were filtered off and washed with acetonitrile to give 3.1 g (36 %) of salt **15**, m.p. 158 °C (dec.). IR, ν/cm^{-1}: 1600 (C=C); 1560 (COO^-); 2000, 2500, 2650 (OH, NH_2^+); 1530, 3400 (NH). ^{1}H NMR, δ: 6.26 (s, 1 H, CH); 6.7—7.5 (m, 10 H, 2 C_6H_5). Found (%): C, 58.84; H, 3.91; F, 16.87; N, 5.90. $C_{22}H_{16}F_4N_2O_4$. Calculated (%): C, 58.93; H, 3.60; F, 16.87; N, 5.90.

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