

Fluorine-containing Heterocycles: X.* Acetoacetamides in the Synthesis of Fluorine-containing Chromone

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Abstract—Fluorine-containing chromone-3-carboxamides were synthesized by reaction of tetra(penta)fluorobenzoyl chlorides with acetoacetamides in dichloromethane in the presence of triethylamine. Nucleophilic substitution of fluorine atoms by amine rests in compounds synthesized was investigated.

Interest grew recently to polycyclic derivatives of fluorine-containing azaheterocycles possessing antibacterial, tuberculocidal, and other types of biological activity [2–6]. In extension of our research on the synthesis of fluoroazaheterocycles by reaction of polyfluorobenzoyl chlorides **I** with bifunctional nucleophiles [1, 7, 8] we deemed promising to apply acetoacetamides (**II**). As distinct from alkyl acetoacetates (classic C,O-dinucleophiles) the acetoacetamides can undergo N-acylation and function as C,N- or C,O-dinucleophiles [9–11]. Therefore the condensation of reagents **I** and **II** might afford either N-aryl-substituted isoquinolones, chromones with an amide moiety in position 3, or coumarines.

The reaction of acyl chlorides **I** with amides **IIa–d** in dichloromethane in the presence of triethylamine at room temperature within 3 h gave rise to chromones **III** in 63–76% yield, and we failed to isolate under given conditions the intermediate products of C-acylation (Scheme 1).

The structure of chromones **III** was derived using ¹H and ¹⁹F NMR and mass spectra (Table 1). In the ¹H NMR spectra of compounds **III** appear broadened singlets from protons of NH groups, singlets from methyl groups in the α -position with respect to oxygen, and also resonances from the aryl substituent. The ¹H NMR spectra of chromones **IIIa–d** possess a characteristic doublet of doublets of doublets signal from H⁵ proton.

Selectively recorded ¹⁹F NMR spectra of compounds **IIIa, f** contained the same number of signals as the number of fluorine atoms in the compound. The molecular ion peaks in the mass spectra of compounds **IIIa–d** are sufficiently intensive (38–56%) but the most abundant is the peak of ion with m/z 241 arising on elimination of the anilide fragment.

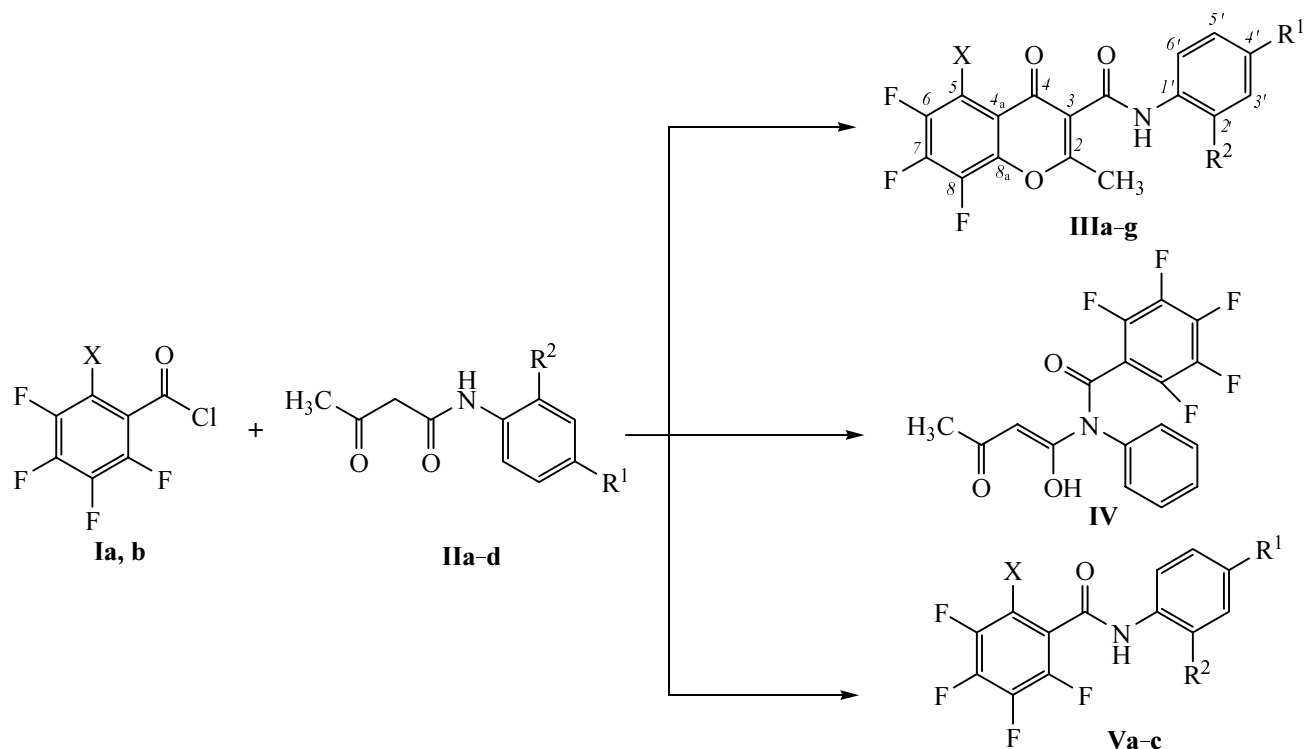
The structure of cyclocondensation products is supported by the ¹³C NMR spectrum registered for compound **IIIa** where in the coupling with fluorine are involved atoms C⁵–C⁸, nodal carbons C^{4a} and C^{8a}, and also the C⁴ atom of the carbonyl group. These data permit rejection of isoquinolone and coumarine structures and confirm the choice of the chromone system **III**.

Chromones were formerly synthesized by reaction of esters of aceto-, benzoyl-, and pentafluorobenzoylacetic acids with pentafluorobenzoyl chloride in the presence of magnesium ethoxide [12–14] but the preparation of chromone-3-carboxylic acid benzylamide from the corresponding ethyl ester was mentioned in a single publication [15].

According to ¹H NMR spectra the reaction between pentafluorobenzoyl chloride (**Ib**) and acetoacetanilide (**IIa**) in dichloromethane in the presence of triethylamine gave rise to a mixture of N-acyl derivative **IV** and chromone **IIIg** in 1:1 ratio. The presence of a broadened singlet downfield from the signal of NH group proton in the ¹H NMR spectrum of the mixture (see EXPERIMENTAL) suggests that the resonance belongs to the hydroxy group of enol **IV**. The downfield shift

* For communication IX see [1].

Scheme 1.



I, X = H (**a**), F (**b**); **II**, R¹ = R² = H (**a**); R¹ = CH₃, R² = H (**b**); R¹ = H, R² = CH₃ (**c**); R¹ = OCH₃, R² = H (**d**); **III**, X = H, R¹ = R² = H (**a**); R¹ = CH₃, R² = H (**b**); R¹ = H, R² = CH₃ (**c**); R¹ = OCH₃, R² = H (**d**); X = F, R¹ = CH₃, R² = H (**e**); R¹ = H, R² = CH₃ (**f**); R¹ = R² = H (**g**); **V**, X = H, R¹ = R² = H (**a**); R¹ = OCH₃, R² = H (**b**); X = F, R¹ = OCH₃, R² = H (**c**).

may originate from a hydrogen bond formation; the existence of enol form is confirmed by a singlet from CH group at 6.0 ppm.

In reaction of pentafluorobenzoyl chloride (**Ib**) with *p*-acetoacetanilide (**IId**) a product of transacylation **Vc** was isolated. Amides of similar structure **Va**, **b** were obtained by boiling tetrafluorobenzoyl chloride (**Ia**) with anilides **IIa**, **d** in toluene; their structure was confirmed by ¹H NMR spectra (see EXPERIMENTAL).

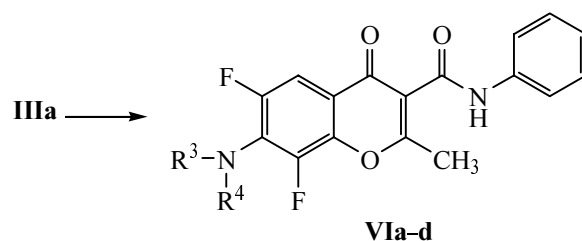
A heating of chromone **IIIa** with morpholine, 2,6-dimethylmorpholine, ethoxycarbonylpiperazine, or isopropylamine in acetonitrile for 3 h yielded products of atom F⁷ replacement **VIa-d** (Scheme 2). In the ¹H NMR spectra of compounds **VI** characteristic signal of proton H⁵ appears as a doublet of doublets, also are present a broadened singlet of NH group in the 10.6–11.0 ppm region, a singlet of the α-methyl group at 2.6–2.7 ppm, and the resonances of the phenyl substituent and the corresponding amine rest (Table 2). The values of proton–fluorine coupling constants ³J 11.5–12.0 and ⁵J 1.5–2.5 Hz for H⁵ permit an unambiguous conclusion on substitution of the F⁷ atom. Mass spectrum of compound **VIa** is consistent with its assumed structure.

Thus in this study conditions were established for preparation of fluorine-containing derivatives of chromone-3-carboxamide (**III**).

EXPERIMENTAL

¹H NMR spectra were registered on spectrometers Bruker WM-250 and Bruker DRX-400 at operating frequencies 250.14 and 400.13 MHz respectively, ¹⁹F NMR spectra were measured on spectrometer Bruker DRX-500 at operating frequency 376.45 MHz. As internal references were used TMS (¹H) and hexafluoro-

Scheme 2.



IV, NR³R⁴ = morpholinO (**a**), 4-ethoxycarbonylpiperazino (**b**), 2,6-dimethylmorpholinO (**c**), NHCH(CH₃)₂ (**d**).

Table 1. ^1H , ^{19}F NMR, and mass spectra of compounds **IIIa–f**

Compd. no.	X	R ¹	R ²	^1H NMR spectrum (DMSO- <i>d</i> ₆), δ , ppm (<i>J</i> , Hz)				^{19}F NMR spectrum (DMSO- <i>d</i> ₆), δ , ppm	Mass spectrum, <i>m/z</i> (<i>I</i> _{rel} , %)
				H ⁵	CH ₃	NH	Ar		
IIIa^a	H	H	H	7.83 d.d.d (³ <i>J</i> 9.5, ⁴ <i>J</i> 7.7, ⁵ <i>J</i> 2.4)	3.04 s	11.32 br.s	7.13 t.t (1H, H ^{4'} , ³ <i>J</i> 7.5, ⁴ <i>J</i> 1.2), 7.35 m (2H, H ^{3'} , H ^{5'} , ³ <i>J</i> 8.5, ³ <i>J</i> 7.5), 7.65 m (2H, H ^{2'} , H ^{6'} , ³ <i>J</i> 8.5, ⁴ <i>J</i> 1.2)	151.86 d.d.d (1F, F ⁸ , ³ <i>J</i> _{FF} 19.8, ⁴ <i>J</i> _{FF} 3.7, ⁵ <i>J</i> _{FH} 2.6), 150.37 d.d.d (1F, F ⁷ , ³ <i>J</i> _{FF} 22.6, ³ <i>J</i> _{FF} 19.8, ⁴ <i>J</i> _{FH} 7.9), 36.78 d.d.d (1F, F ⁶ , ³ <i>J</i> _{FF} 22.6, ³ <i>J</i> _{FH} 9.9, ⁴ <i>J</i> _{FF} 3.7)	333 (48) [<i>M</i>] ⁺ , 316 (10), 242 (12), 241 (100), 175 (10), 93 (42), 67 (99)
IIIb	H	CH ₃	H	7.83 d.d.d (³ <i>J</i> 10.3, ⁴ <i>J</i> 8.3, ⁵ <i>J</i> 2.5)	2.61 s	10.38 br.s	2.31 s (3H, SH ₃), 7.11 d (2H, H ^{2'} , H ^{6'}), 7.52 d (2H, H ^{3'} , H ^{5'}), ³ <i>J</i> 8.4		347 (51) [<i>M</i>] ⁺ , 330 (24), 242 (12), 241 (100), 175 (10), 107 (51), 106 (11), 67 (92)
IIIc	H	H	CH ₃	7.90 d.d.d (³ <i>J</i> 10.0, ⁴ <i>J</i> 8.0, ⁵ <i>J</i> 2.5)	2.71 s	10.30 br.s	2.32 s (3H, SH ₃), 7.08 m (1H), 7.21 m (1H), 7.73 m (1H)		347 (38) [<i>M</i>] ⁺ , 332 (15), 242 (12), 241 (100), 175 (10), 107 (32), 106 (39), 67 (99)
III^d	H	OC H ₃	H	7.83 d.d.d (³ <i>J</i> 10.0, ⁴ <i>J</i> 8.0, ⁵ <i>J</i> 2.5)	2.60 s	10.32 br.s	3.76 s (3H, OSH ₃), 6.86 d (2H, H ^{3'} , H ^{5'}), 7.56 d (2H, H ^{2'} , H ^{6'}), ³ <i>J</i> 8.8		363 (56) [<i>M</i>] ⁺ , 346 (18), 241 (93), 331 (7), 175 (10), 123 (44), 108 (15), 67 (100)
IIIe	F	CH ₃	H	–	2.54 c	10.29 br.s	2.30 s (3H, SH ₃), 7.11 d (2H, H ^{2'} , H ^{6'}), 7.52 d (2H, H ^{3'} , H ^{5'}), ³ <i>J</i> 8.0		
III^f	F	H	CH ₃	–	2.67 c	10.15 br.s	2.32 s (3H, SH ₃), 7.07 m (1H), 7.20 m (1H), 7.70 m (1H)	161.87 m (1F), 159.05 m (1F), 148.53 m (1F), 144.73 m (1F)	

^a Spectra recorded in CDCl₃.**Table 2.** ^1H NMR and mass spectra of compounds **VI**

Compd. no.	NR ³ R ⁴	^1H NMR spectrum (DMSO- <i>d</i> ₆), δ , ppm					Mass spectrum, <i>m/z</i> (<i>I</i> _{rel} , %)
		H ⁵	SH ₃	NH	Ar	NR ³ R ⁴	
VIa	Morpholino	7.52 d.d (³ <i>J</i> 11.9, ⁵ <i>J</i> 2.0 Hz)	2.60 s	10.60 br.s	7.10 m (1H, H ^{4'}), 7.31 m (2H, H ^{2'} , H ^{6'}), 7.65 m (2H, H ^{3'} , H ^{5'})	3.35 m [4H, N(CH ₂) ₂], 3.74 m [4H, O(CH ₂) ₂]	400 (42) [<i>M</i>] ⁺ , 383 (17), 309 (18), 308 (100), 307 (12), 242 (30), 93 (20), 67 (41)
VIb	4-ethoxy- carbonyl- piperazino	7.53 d.d (³ <i>J</i> 11.5, ⁵ <i>J</i> 2.0 Hz)	2.62 s	10.63 br.s	7.07 m (1H, H ^{4'}), 7.31 m (2H, H ^{2'} , H ^{6'}), 7.65 m (2H, H ^{3'} , H ^{5'})	1.25 t (3H, SH ₃), 4.09 κ (2H, OSH ₂), 3.32 m [4H, N(CH ₂) ₂], 3.54 m [4H, N(CH ₂) ₂]	
VIc	2,6-dimethyl- morpholino	7.51 d.d (³ <i>J</i> 12.0, ⁵ <i>J</i> 2.5 Hz)	2.61 s	10.64 br.s	7.09 m (1H, H ^{4'}), 7.30 m (2H, H ^{2'} , H ^{6'}), 7.65 m (2H, H ^{3'} , H ^{5'})	1.15 s (3H, SH ₃), 1.17 s (3H, SH ₃), 2.90 m (2H, NCH ₂), 3.33 m (2H, NCH ₂), 3.73 m (2H, 2SH)	
VI^d	HNCH(CH ₃) ₂	7.43 d.d (³ <i>J</i> 12.0, ⁵ <i>J</i> 1.5 Hz)	2.67 s	10.93 br.s	7.05 m (1H, H ^{4'}), 7.29 m (2H, H ^{2'} , H ^{6'}), 7.65 m (2H, H ^{3'} , H ^{5'})	1.25 s (3H, SH ₃), 1.27 s (3H, SH ₃), 4.08 m (1H, SH), 5.76 m (1H, NH)	

benzene (^{19}F). ^{13}C NMR spectra were obtained on spectrometer Bruker DRX-400 at operating frequency 100.61 MHz. Mass spectra were measured on Varian MAT 311A instrument in the following conditions: accelerating voltage 3kV, catode emission current 300 μA , ionizing electrons energy 70 eV, direct sample admission into the ion source.

Yields, melting points, and elemental analyses of compounds synthesized are given in Table 3.

2-Methyl-5-X-6,7,8-trifluoro-4-oxo-4H-chromene-3-carboxamides (IIIa–f). To a solution of 0.7 g (4.0 mmol) of acetoacetanilide (**IIa**) in 6 ml of anhydrous dichloromethane was added 1.2 ml (8 mmol) of triethylamine and then dropwise 1.7 ml (4 mmol) of tetrafluorobenzoyl chloride solution in toluene. The reaction mixture was left standing at room temperature for 24 h, the separated precipitate of chromone **IIIa** was filtered off, washed with water, and recrystallized from ethanol. Yield 0.9 g (69%), mp 202–204°C. ^{13}C NMR spectrum (CDCl_3), δ , ppm: 22.48 d (CH_3 , 1J 132.1 Hz), 107.35 d.d.d [C^5 , $^1J(\text{C}^5, \text{H}^5)$ 172.9, $^2J(\text{C}^5, \text{F}^6)$ 19.5, $^3J(\text{C}^5, \text{F}^7)$ 3.8 Hz], 114.48 d [C^3 , $^4J(\text{C}^3, \text{CH}_3)$ 2.0 Hz], 119.27 d.d.d [C^{4a} , $^3J(\text{C}^{4a}, \text{F}^6)$ 7.4, $^3J(\text{C}^{4a}, \text{H}^5)$ 2.4, $^4J(\text{C}^{4a}, \text{F}^7)$ 2.8 Hz], 120.67 d.d.d.d [$\text{C}^{2,6}$, 1J 162.6, 2J 4.1, 3J 7.3, 4J 3.5 Hz], 124.49 d [C^4 , 1J 160.6 Hz], 128.98 d.d.d [$\text{C}^{3,5}$, 1J 157.6, 2J 3.0, 3J 8.5 Hz], 138.01 d.d [C^1 , 2J 1.7, 3J 9.3 Hz], 140.28 d.d.d.d [C^8 , $^1J(\text{C}^8, \text{F}^8)$ 260.8, $^2J(\text{C}^8, \text{F}^7)$ 12.8, $^3J(\text{C}^8, \text{F}^6)$ 3.1, $^4J(\text{C}^8, \text{H}^5)$ 1.7 Hz], 141.28 d.d.d.d [C^{8a} , $^2J(\text{C}^{8a}, \text{F}^8)$ 8.7, $^3J(\text{C}^{8a}, \text{H}^5)$ 9.6, $^3J(\text{C}^{8a}, \text{F}^7)$ 2.8, $^4J(\text{C}^{8a}, \text{F}^6)$ 2.8 Hz], 144.01 d.d.d.d [C^7 , $^1J(\text{C}^7, \text{F}^7)$ 261.8, $^2J(\text{C}^7, \text{F}^6)$ 17.6, $^2J(\text{C}^7, \text{F}^8)$ 11.6, $^3J(\text{C}^7, \text{H}^5)$ 1.9 Hz], 149.07 d.d.d.d [C^6 , $^1J(\text{C}^6, \text{F}^6)$ 253.7, $^2J(\text{C}^6, \text{F}^7)$ 11.0, $^3J(\text{C}^6,$

$\text{F}^8)$ 1.4, $^2J(\text{C}^6, \text{H}^5)$ 6.0 Hz], 161.14 C (N–CO), 175.50 d.d.d.d [C^4 , $^4J(\text{C}^4, \text{F}^6)$ 2.6, $^5J(\text{C}^4, \text{F}^7)$ 2.6, $^6J(\text{C}^4, \text{F}^8)$ 1.2, $^3J(\text{C}^4, \text{H}^5)$ 3.7 Hz], 176.10 d [C^2 , $^2J(\text{C}^2, \text{CH}_3)$ 6.7 Hz].

Compounds **IIIb–f** were prepared in a similar way.

2-Methyl-5,6,7,8-tetrafluoro-4-oxo-4H-chromene-3-carboxanilide (IIIg) and N-pentafluorobenzoyl-N-phenylacetamide (IV). To 0.7 g (4.0 mmol) of acetoacetanilide (**IIa**) in 6 ml of anhydrous dichloromethane was added 1.2 ml (8 mmol) of triethylamine and then by small portions 1.4 ml (4 mmol) of pentafluorobenzoyl chloride (**IIb**) solution in toluene. The reaction mixture was left standing at room temperature for 24 h, the separated mixed precipitate of compounds **IIIg** and **IV** was filtered off, washed with water, and dried. ^1H NMR spectrum ($\text{DMSO-}d_6$), δ , ppm: 1.94 s [3H, CH_3 , (**IV**)], 2.60 s [3H, CH_3 , (**IIIg**)], 6.02 s [1H, CH, (**IV**)], 7.13 t.t [1H, H^d , $^3J_{4,3}$ 7.5, $^4J_{4,2}$ 1.2 Hz (**IIIg**)], 7.21 m [2H, H^2 , H^6 (**IV**)], 7.35 d.d [2H, H^3 , H^5 , $^3J_{3,4}$ 7.5, $^3J_{3,2}$ 8.5 Hz (**IIIg**)], 7.45–7.55 m [3H, H^3 – H^5 (**IV**)], 7.65 d.d [2H, H^2 , H^6 , $^3J_{2,3}$ 8.5, $^4J_{2,4}$ 1.2 Hz (**IIIg**)], 10.37 br.s [1H, NH (**IIIg**)], 15.69 br.s [1H, OH (**IV**)]. The ratio of compounds **IIIg** and **IV** was 1:1.

2,3,4,5-Tetrafluoro-6-X-benzamides (Va–c). (a) To a solution of 0.7 g (3.4 mmol) of *N*-acetoacetyl-*p*-anisidine in 6 ml of anhydrous dichloromethane was added 1.1 ml (7 mmol) of triethylamine and then by small portions 1.2 ml (3.4 mmol) of pentafluorobenzoyl chloride (**IIb**) solution in toluene. The reaction mixture was left standing at room temperature for 3 h, the separated precipitate of amide **Vc** was filtered off, washed with water, and recrystallized from ethanol. Yield 0.71 g (66%), mp 188–190°C. ^1H NMR spectrum ($\text{DMSO-}d_6$),

Table 3. Yields, melting points, and elemental analyses of compounds synthesized

Compd. no.	mp°C	Yield, %	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
IIIa	202–204	69	61.24	3.05	4.32	$\text{C}_{17}\text{H}_{10}\text{F}_3\text{NO}_3$	61.27	3.02	4.20
IIIb	216–218	64	62.27	3.50	4.23	$\text{C}_{18}\text{H}_{12}\text{F}_3\text{NO}_3$	62.25	3.48	4.03
IIIc	202–204	67	61.84	3.54	4.04	$\text{C}_{18}\text{H}_{12}\text{F}_3\text{NO}_3$	62.25	3.48	4.03
III d	205–207	76	59.42	3.37	3.92	$\text{C}_{18}\text{H}_{12}\text{F}_3\text{NO}_4$	59.51	3.33	3.86
III e	198–200	63	59.04	3.11	3.69	$\text{C}_{18}\text{H}_{11}\text{F}_4\text{NO}_3$	59.18	3.06	3.84
III f	224–226	68	59.05	3.10	3.81	$\text{C}_{18}\text{H}_{11}\text{F}_4\text{NO}_3$	59.18	3.06	3.84
Va	125–127	82	58.08	2.49	5.33	$\text{C}_{13}\text{H}_7\text{F}_4\text{NO}$	57.99	2.60	5.20
Vb	131–133	79	56.27	2.95	4.79	$\text{C}_{14}\text{H}_9\text{F}_4\text{NO}_2$	56.19	3.01	4.68
Vc	188–190	66	53.09	2.39	4.56	$\text{C}_{14}\text{H}_8\text{F}_5\text{NO}_2$	53.00	2.52	4.42
VIa	200–202	81	62.80	4.53	7.24	$\text{C}_{21}\text{H}_{18}\text{F}_2\text{N}_2\text{O}_4$	63.00	4.53	7.00
VIb	184–186	72	60.96	4.93	8.80	$\text{C}_{24}\text{H}_{23}\text{F}_2\text{N}_3\text{O}_5$	61.14	4.92	8.91
VIc	175–177	76	64.03	5.30	6.58	$\text{C}_{23}\text{H}_{22}\text{F}_2\text{N}_2\text{O}_4$	64.48	5.17	6.54
VI d	146–148	73	64.22	4.98	7.55	$\text{C}_{20}\text{H}_{18}\text{F}_2\text{N}_2\text{O}_3$	64.51	4.87	7.52

δ , ppm: 3.76 s (3H, OCH₃), 6.88 d (2H, H^{3'}, H^{5'}, ³J 8.8 Hz), 7.54 d (2H, H^{2'}, H^{6'}, ³J 8.8 Hz), 10.59 br.s (1H, NH).

(b) To a dispersion of 0.7 g (4 mmol) of acetoacetanilide in 10 ml of anhydrous toluene was added 1.6 ml (11 mmol) of tetrafluorobenzoyl chloride solution in toluene. The reaction mixture was heated at reflux for 1.5 h, and then evaporated. The residue was washed with ethanol and recrystallized from ethanol to obtain compound **Va**. Yield 0.97 g (82%), mp 125–127°C. ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 7.15 m (1H, H^d), 7.38 m (2H, H^{2'}, H^{6'}), 7.69 m (2H, H^{3'}, H^{5'}), 7.78 m (1H, H^{6'}), 10.60 br.s (1H, NH).

Likewise was prepared compound **Vb**. ¹H NMR spectrum (DMSO-*d*₆), δ , ppm: 3.76 C (3H, OCH₃), 6.87 d (2H, H^{3'}, H^{5'}, ³J 8.9 Hz), 7.57 d (2H, H^{2'}, H^{6'}, ³J 8.9 Hz), 7.57 m (1H, H^{6'}), 10.21 br.s (1H, NH).

2-Methyl-6,8-difluoro-7-NR³R⁴-4-oxo-4H-chromene-3-carboxanilides (VIa–f). To a dispersion of 0.4 g (1.2 mmol) of compound **IIIa** in 8 ml of acetonitrile was added 0.5 ml (6 mmol) of morpholine. The reaction mixture was heated to 80°C for 3 h. On cooling the precipitate of derivative **VIa** was filtered off and recrystallized from acetonitrile. Yield 0.39 g (81%), mp 200–202°C.

Compounds **VIb–d** were prepared in a similar way.

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