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Fluorine-Containing Heterocycles: VIII.* Transformations of 2-Polyfluorobenzoylacrylates Having a Thiosemicarbazide Fragment**

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Abstract—Depending on the conditions, 3-(4-R-thiosemicarbazido)-2-polyfluorobenzoylacrylates can be converted into the corresponding potassium salts, [1,3,4]thiadiazino[6,5,4-*ij*]quinolines, and pyrazole or 1,3,4-thiadiazole derivatives.

We previously showed that 3-hydrazino-2-polyfluorobenzoylacrylates are convenient synthons for the preparation of [*ij*]-fused fluoroquinolinones and other fluorinated nitrogen-containing heterocycles [2, 3]. We also reported that heating of 3-(4-R-thiosemicarbazido)-2-polyfluorobenzoylacrylates **I** in benzene or toluene leads to formation of tricyclic fluoroquinolinones, 9,10-difluoro-7*H*-[1,3,4]thiadiazino[6,5,4-*ij*]quinolin-7-one derivatives **III** [4]. The reaction is relatively fast, and it requires no catalyst to occur [5]. We failed to isolate bicyclic intermediates like fluoroquinolinones **II**.

The goal of the present work was to study intramolecular cyclizations of acrylates **I** with participation of the carbonyl groups, as well as to synthesize fluoroquinolinones **II**. Heating of ethyl 3-(4-cyclohexylthiosemicarbazido)-2-pentafluorobenzoylacrylate (**Ie**) in ethanol gave a product which was assigned structure **IIe** on the basis of its ^{1}H and ^{19}F NMR spectra. The ^{1}H NMR spectrum of **IIe** contained signals from the 2-H proton, protons of the ethyl and cyclohexyl groups, and two NH protons (Table 1). In the ^{19}F NMR spectrum of **IIe** we observed signals from four fluorine atoms. Absorption bands belonging to ester and ketone carbonyl groups were present in the IR spectrum. By heating in boiling toluene for 1 h, compound **IIe** was converted into tricyclic thiadiazinoquinoline **III**.

Acrylates **I** are also capable of undergoing intramolecular cyclizations with participation of the C=O groups. When acrylate **Ia** was heated in boiling toluene, apart from compound **IIIa**, we isolated 70% of a product with a lower melting point. The product is readily soluble in toluene, and its ^{1}H and ^{19}F NMR spectra correspond to the structure of 1,4,5-trisubstituted pyrazole **IV**. The ^{1}H NMR spectrum of **IV** contained signals from protons of the ethyl group and morpholine, tetrafluorobenzoyl, and pyrazole fragments (Table 1). Signals from four fluorine nuclei were present in the ^{19}F NMR spectrum. Compound **IV** showed in the IR spectrum only one carbonyl absorption band located at a lower frequency, as compared with acrylate **Ia**. Taking into account published data [6], this band was assigned to ketone carbonyl. In the mass spectrum of **IV** we observed only fragment ion peaks, the most abundant being that with *m*/*z* 177. It corresponds to elimination of the tetrafluorobenzoyl fragment from the molecular ion (Table 2). Pyrazole **IV** was also obtained in 40% yield (in a mixture with products **III** and **VIII**) by heating of acrylate **Ia** in boiling toluene in the presence of KF. Heating of **Ia** in ethanol also yields product **IV**. According to our previous data [2], pyrazoles like **IV** are characterized

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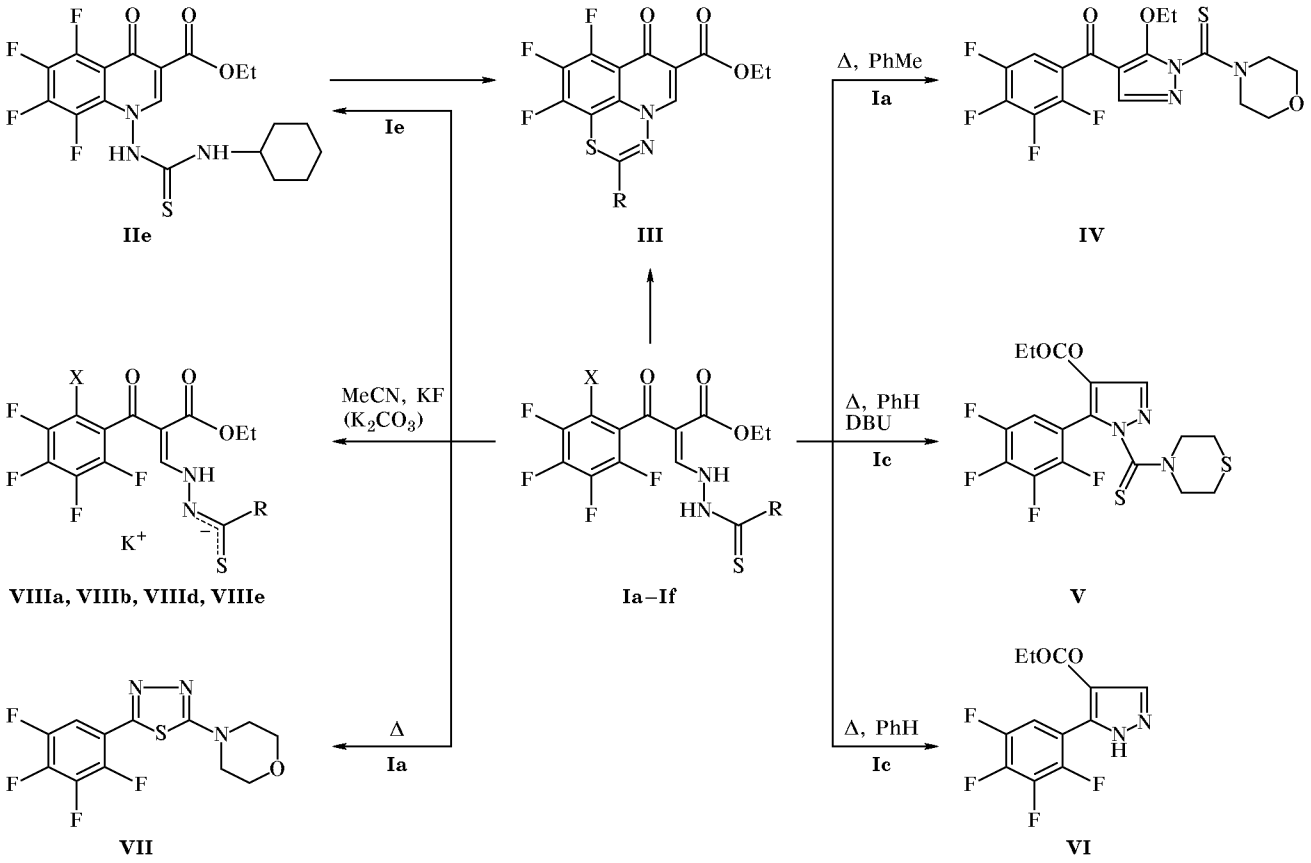
Scheme 1.



X = H, R = morpholino (**a**); X = F, R = morpholino (**b**); X = H, R = thiomorpholino (**c**); X = H, R = perhydroazepin-1-yl (**d**); X = F, R = cyclohexylamino (**e**); X = H, R = cyclohexylamino (**f**).

by a more downfield signal from 3-H, as compared to pyrazoles like **V** and **VI**. In fact, an analogous pattern was observed in the present work (Table 1).

Heating of compound **Ic** for 1.5 h in boiling benzene in the presence of a catalytic amount of such a strong base as 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) afforded 1,4,5-trisubstituted pyrazole **V** as a result of cyclization involving the ketone carbonyl group. The ^{1}H NMR spectrum of **V** contain signals from all protons present therein, and four fluorine signals were observed in the ^{19}F NMR spectrum (Table 1). Protons of the $(CH_2)_2N$ fragment in the thiomorpholino group appear in the ^{1}H NMR spectrum as two multiplets, and one fluorine signal is an unresolved multiplet. This pattern suggests coupling between hydrogen and fluorine nuclei through 8 bonds or through space, which is possible in the structure with α,β-arrangement of the tetrafluorophenyl and thiomorpholino groups. The IR spectrum of **V** contains only one high-frequency carbonyl absorption band due to the ester group. Compound **V** shows in the mass spectrum the molecular ion peak and also fragment ion peaks corresponding to elimination of OEt, COOEt, and thiomorpholino groups (minus one or two hydrogen atoms); the latter peaks are the most intense in the spectrum (Table 2).

4,5-Disubstituted pyrazole **VI** was obtained by heating of acrylate **Ic** in benzene containing traces of water; obviously, compound **VI** is formed from pyrazole **V** as a result of hydrolysis at the thioamide group. Unlike compound **V**, pyrazole **VI** has a higher melting point and is characterized by a lower solubility in organic solvents. The IR spectrum of **VI**, as well as of **V**, contains a high-frequency ester carbonyl absorption band. In the mass spectrum of **VI** we observed the molecular ion peak and those resulting from elimination of ethoxy group (base peak) and nitrogen molecule (Table 2). The fragmentation patterns of pyrazoles **V** and **VI** under electron impact differ from that observed for compound **IV**. Pyrazole **VI** was synthesized previously from 3-hydrazinopyridyl-2-tetrafluorobenzoylacrylates [3].

Table 1. IR, UV, and ^1H and ^{19}F NMR spectra of compounds **I**–**VIII**

Compound no.	^1H NMR spectrum, δ, ppm, *J*, Hz					
	3-H	NH	CH_3, t	OCH_2, q	6-H, m	R
Ia	8.33 d, ^3J = 11.9 Hz	12.94 d, ^3J = 12.2 Hz, 11.43 br.d	1.04	4.00	7.43	3.32 m [4H, $N(CH_2)_2$], 3.74 m [4H, $O(CH_2)_2$]
IIe	8.10 s	8.4 br.s, 10.5 br.s	1.33	4.25	–	1.2–1.4 m [6H, $(CH_2)_3$], 1.6–1.9 m [4H, $(CH_2)_2$], 4.1 br. s (1H, CHNH)
IV	8.82 s	–	1.18	4.12	7.84	3.60 m [4H, $N(CH_2)_2$], 3.70 m [4H, $O(CH_2)_2$]
V	8.12 s	–	1.21	4.17	7.40	2.83 m [4H, $N(CH_2)_2$], 3.72 m (2H, NCH_2), 4.32 m (2H, NCH_2)
VI	8.38 s	13.71 br.s	1.16	4.15	7.55	–
VII	–	–	–	–	8.06	3.43 m [4H, $N(CH_2)_2$], 3.76 m [4H, $O(CH_2)_2$]
VIIIa	8.18 d, ^3J = 14.1 Hz	13.62 d, ^3J = 12.7 Hz	1.01	3.92	7.25	3.55 m [4H, $N(CH_2)_2$], 3.71 m [4H, $O(CH_2)_2$]
VIIIb	8.33 d, ^3J = 12.7 Hz	13.11 d, ^3J = 12.7 Hz	1.06	4.02	–	3.68 m [4H, $N(CH_2)_2$], 3.84 m [4H, $O(CH_2)_2$]
VIIIc	8.15 d, ^3J = 13.7 Hz	13.6 d, ^3J = 12.8 Hz	1.07	3.93	7.15	2.71 m [4H, $S(CH_2)_2$], 4.16 m [4H, $N(CH_2)_2$]
VIIId	8.21 d, ^3J = 13.6 Hz	13.23 d, ^3J 11.9 Hz	1.01	3.95	7.30	1.53 m [4H, $(CH_2)_2$], 1.69 m [4H, $(CH_2)_2$], 3.76 m [4H, $N(CH_2)_2$]

Compound no.	IR spectrum, ν(C=O), cm^{-1}	UV spectrum, λ_{max}, nm	^{19}F NMR spectrum, δ_F, ppm
Ia	1700, 1620	345	140.5 m (1F), 142.5 m (1F), 156.8 m (1F), 158.0 m (1F)
IIe	1710, 1630	312	143.7 m (1F), 150.5 m (1F), 155,2 m (1F), 162,1 m (1F)
IV	1630	270	137.0 m (1F), 138.5 m (1F), 148.5 m (1F), 155.0 m (1F)
V	1720	250, 298	138.0 m (1F), 140.3 m (1F), 154.1 m (1F), 157.1 m (1F)
VI	1720	264	134.5 m (1F), 141.1 m (1F), 157.0 m (1F), 157.9 m (1F)
VII	–	322	140.0 m (1F), 140.7 m (1F), 155.8 m (1F), 156.5 m (1F)
VIIIa	1630, 1680	384	142.2 m (1F), 143.4 m (1F), 159.3 m (1F), 160.2 m (1F)
VIIIb	1710, 1630	384	145.2 m (2F), 156.0 m (1F), 163.1 m (1F)
VIIIc	1680, 1620	384	
VIIId	1630, 1670	384	141.1 m (1F), 143.1 m (1F), 158.3 m (1F), 158.6 m (1F)

2,5-Disubstituted thiadiazole **VII** was obtained in 55% yield by heating of acrylate **Ia** in boiling benzene in the presence of a catalytic amount of DBU. Compound **VII** is characterized by a strong UV band with its maximum at λ 322 nm. The IR spectrum of **VII** lacks carbonyl absorption band. Its ^1H NMR spectrum contains signals from morpholine protons and 6-H of the aromatic fragment, and four fluorine signals are present in the ^{19}F NMR spectrum. Thiadiazole **VII** shows the molecular ion peak in the mass spectrum. Compound **VII** was also obtained (together with salt **VIIIa**) by heating of acrylate **Ia** in boiling toluene in the presence of K_2CO_3. It should be noted that the fragmentation pattern of **Ia** at 120°C resembles that of pyrazole **IV**, whereas at 200°C it is similar to that of thiadiazole **VII**.

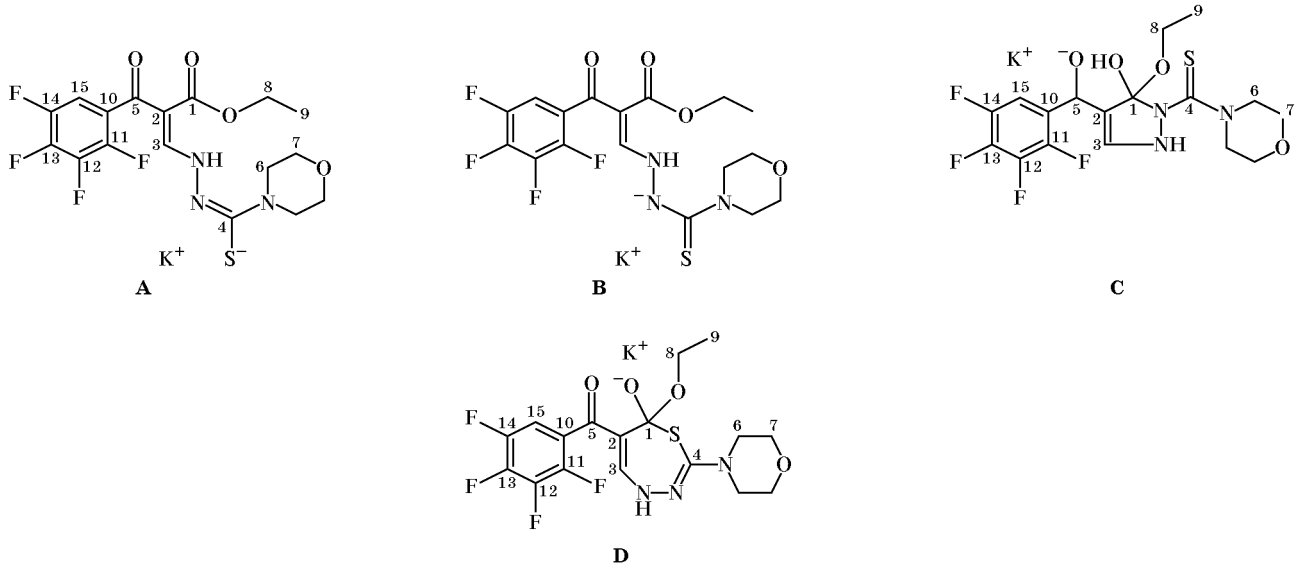
Table 2. Melting points, mass spectra, and elemental analyses of compounds **I**–**VIII**

Comp. no.	mp, °C	Mass spectrum, *m*/*z* (I_{rel}, %)	Found, %			Formula	Calculated, %		
			C	H	N		C	H	N
Ia	104–106	120°C: 264 (11), 219 (7), 177 (100), 149 (14) 220°C: 318 (74), 261 (62), 192 (100), 174 (23)	46.8	3.9	10.4	$C_{17}H_{17}F_4N_3O_4S$	46.90	3.91	9.66
IIe	195–197	–	55.4	4.5	10.0	$C_{19}H_{19}F_4N_3O_3S$	55.21	4.63	10.17
IV	55–57	50°C: 265 (11), 264 (77), 219 (57), 218 (66), 177 (100), 149 (36)	48.5	3.9	10.3	$C_{17}H_{15}F_4N_3O_3S$	48.92	3.62	10.07
V	78–80	90°C: 433 (24, M^+), 386 (12), 289 (8), 243 (14), 200 (10), 146 (49), 145 (100), 144 (99)	46.8	3.2	9.4	$C_{17}H_{15}F_3N_3O_2S_2$	47.11	3.49	9.70
VI	140–142	90°C: 288 (76, M^+), 260 (30), 244 (63), 243 (100), 240 (76), 216 (41)	50.4	3.2	9.9	$C_{12}H_8F_4N_2O_2$	50.09	2.80	9.72
VII	158–160	120°C: 319 (100, M^+), 262 (98), 193 (100), 175 (40)	45.3	3.0	13.0	$C_{12}H_9F_4N_3OS$	45.14	2.82	13.16
VIIIa	225–227	120°C: 417 (41), 264 (51), 219 (42), 177 (100), 149 (47)	43.4	3.3	9.1	$C_{17}H_{16}F_4KN_3O_4S$	43.15	3.29	8.90
VIIIb	214–216	–	41.8	3.4	8.7	$C_{17}H_{15}F_5KN_3O_4S$	41.54	3.05	8.50
VIIIc	202–204	–	41.5	3.6	8.8	$C_{17}H_{17}F_4KN_3O_3S_2$	41.62	3.49	8.57
VIIId	178–180	–	46.6	4.2	8.5	$C_{19}H_{20}F_4KN_3O_3S$	47.00	4.15	8.65

Table 3. Experimental ^{13}C chemical shifts of compounds **Ia** and **VIIIa** and calculated chemical shifts of structures **A**–**D**

Atom no.	^{13}C NMR spectrum, δ_C, ppm		Calculated chemical shifts δ_C			
	Ia	**VIIIa**	**A**	**B**	**C**	**D**
C^1	165.20	179.11	165.0	165.0	87.0	58.0
C^2	110.50	110.74	111.6	111.6	118.6	118.6
C^3	151.96	145.61	158.3	158.3	142.5	142.5
C^4	183.98	167.25	185.0	154.0	184	154
C^5	178.29	181.16	187.0	187.0	187.0	187.0
C^6	42.22	48.03	58	53	57.6	49.6
C^7	65.52	67.23	71	72	71.5	71.2
C^8	59.38	59.11	59.6	59.6	59	60
C^9	13.92	14.31	13.7	13.7	15	14
C^{10}	127.50	130.61	122.5	122.5	122.5	122.5
C^{11}	140.82	138.77	147.5	147.5	147.5	147.5
C^{12}	142.68	143.14	138.2	138.2	138.2	138.2
C^{13}	143.91	147.97	143.5	143.5	143.5	143.5
C^{14}	141.56	141.14	146.8	146.8	146.8	146.8
C^{15}	119.92	129.26	115.5	115.5	115.5	115.5

Scheme 2.



By heating of acrylate **Ia** in acetonitrile in the presence of KF or K_2CO_3 we obtained high-melting water-soluble product **VIIIa** which showed in the electron absorption spectrum a strong band with its maximum at λ 384 nm, i.e., at a longer wavelength than that observed for acrylate **Ia**. In the IR spectrum of **VIIIa**, the ketone and ester carbonyl absorption bands were located closer to each other (1630 and 1600 cm^{-1}), as compared to the corresponding bands of acrylate **Ia** (1700 and 1620 cm^{-1}; Table 1). Its ^1H NMR spectrum contained signals from protons of morpholino group and 6-H in tetrafluorobenzoyl fragment, and signals from the CH= and NH protons appeared as doublets (as well as in the ^1H NMR spectrum of **Ia**). Four fluorine signals were present in the ^{19}F NMR spectrum. Product **VIIIa** dissolved on addition of acetic acid, but after a time a solid precipitated from the solution. All spectral parameters of the latter were identical to those of acrylate **Ia**. We compared the ^{13}C NMR spectrum of **VIIIa** with the calculated spectra of several hypothetical structures **A**–**D** (Scheme 2; the calculations were performed with the use of ChemDraw Ultra 6.0.1 program; Table 3).

The upfield region of the ^{13}C spectrum of **VIIIa** contains no other signals belonging to tetrahedral carbon atoms than those from ethoxy group and morpholine residue. Therefore, compound **VIIIa** cannot be a product of intramolecular addition of the NH or SH moiety of **Ia** to the carbonyl group. Thus structures **C** and **D** can be ruled out. The data corresponding to a superposition of structures **A** and **B** turned out to be the closest to the experimental spectrum of **VIIIa** (Table 3). The mass spectrum of **VIIIa** contained ion peaks with *m*/*z* 264, 219, 177, and 149, which were also typical of acrylate **Ia** (at 120°C) and pyrazole **IV** (Table 2).

The above data led us to presume the structure of potassium salt **VIIIa** in which the negative charge is delocalized mainly over the S–C–N fragment. This structure is consistent with the chemical shift of C^4 in the experimental ^{13}C NMR spectrum. Similar salts were obtained from acrylates **Ib**, **Id**, and **Ie** by heating in acetonitrile in the presence of KF. Salts **VIII** are fairly stable. For example, salt **VIIIa** remains unchanged on heating in toluene for 2 h. Using UV spectroscopy, we detected formation of salts of acrylates **I** with triethylamine. However, these salts turned out to be less stable than **VIII**.

We previously showed [2, 3] that heating of 3-R-hydrazino-2-polyfluorobenzoylacrylates (R = aroyl, benzazolyl) in acetonitrile in the presence of KF gives rise to pyrazoles together with penta- and tricyclic quinolinone derivatives. No salt formation was observed in these reaction. Presumably, increased acidity of the SH group in the thiol form of acrylates **I** favors formation of stable salts. The formation of stable potassium salts from symmetric polyfluorinated β-diketones was reported in [7].

EXPERIMENTAL

The ^1H and ^{19}F NMR spectra were recorded on Bruker WP-250 and Bruker WP-80SY spectrometers (250.135 MHz for ^1H and 75.38 MHz for ^{19}F) from

solutions in DMSO-d_6. Tetramethylsilane was used as internal reference for ^{1}H NMR spectra, and ^{19}F chemical shifts were measured relative to hexafluorobenzene. The ^{13}C NMR spectra were obtained on a Bruker WP-500 instrument at 125 MHz using $CDCl_3$ as solvent and reference. The mass spectra were recorded on a Varian MAT 311A spectrometer (accelerating voltage 3 kV, cathode emission current 300 μA, direct sample admission into the ion source). The UV spectra were measured on a Specord UV-Vis spectrophotometer in ethanol, and the IR spectra were obtained on a Specord 75IR instrument from samples pelleted with KBr. The solvents (96% ethanol, benzene, toluene, and acetonitrile) were preliminarily dried over sodium sulfate and distilled. 3-(4-R-Thiosemicarbazido)-2-polyfluorobenzoylacrylates **I** were synthesized by the procedure reported in [4].

Ethyl 1-cyclohexylaminothiocarbonylamino-5,6,7,8-tetrafluoro-4-oxo-1,4-dihydroquinoline-3-carboxylate (IIe). A solution of 0.4 g (0.92 mmol) of compound **Ie** in 12 ml of ethanol was heated for 1 h at 80°C. It was then cooled and evaporated, and the residue was recrystallized from ethanol. Yield 0.3 g (79%).

Ethyl 2-cyclohexylamino-8,9,10-trifluoro-7-oxo-7*H*-[1,3,4]oxadiazino[6,5,4-*ij*]quinoline-6-carboxylate (III). A solution of 0.1 g (0.24 mmol) of ester **IIe** in 8 ml of toluene was heated for 1.5 h under reflux. The mixture was cooled, and the precipitate was filtered off and recrystallized from ethanol. Yield 0.07 g (74%).

5-Ethoxy-2-morpholinothiocarbonyl-4-(2,3,4,5-tetrafluorobenzoyl)pyrazole (IV). *a*. A solution of 1.0 g (2.3 mmol) of ester **Ia** in 12 ml of toluene was heated for 2 h under reflux. The mixture was cooled, and the precipitate of quinolinone **IIa**, 0.1 g (11%), was filtered off. The filtrate was evaporated, and the residue was recrystallized from ethanol. Yield of pyrazole **IV** 0.6 g (63%).

b. Potassium fluoride, 0.6 g (9.2 mmol), was added to a solution of 2.0 g (4.6 mmol) of ester **Ia** in 18 ml of toluene. The mixture was heated for 2 h under reflux and cooled, and the precipitate was filtered off and washed with water to obtain 0.5 g (23%) of compound **VIIIa**. The toluene solution was evaporated, and the residue was recrystallized from ethanol. Yield of **IV** 1.3 g (68%).

c. A solution of 0.5 g (1.15 mmol) of ester **Ia** in 15 ml of ethanol was heated for 1 h under reflux. The mixture was evaporated, and the residue was recrystallized from ethanol. Yield 0.35 g (73%).

Ethyl 5-(2,3,4,5-tetrafluorophenyl)-1-thiomorpholinothiocarbonylpyrazole-4-carboxylate (V). 1,8-Diazabicyclo[5.4.0]undec-7-ene, 10 drops, was added to a suspension of 0.75 g (1.65 mmol) of ester **Ic** in 15 ml of benzene. The mixture was stirred for 1.5 h at 80° and evaporated almost to dryness, 4 drops of acetic acid was added, and the precipitate was filtered off. Recrystallization from ethanol gave 0.5 g (62%) of pyrazole **V**.

Ethyl 5-(2,3,4,5-tetrafluorophenyl)-1*H*-pyrazole-4-carboxylate (VI). *a*. A solution of 2.0 g (4.4 mmol) of ester **Ic** in 30 ml of wet benzene was heated for 2 h at 80°C. The mixture was cooled, and the precipitate was filtered off and recrystallized from ethanol. Yield 1.3 g (38%).

b. A solution of 0.18 g (0.4 mmol) of ester **If** in 8 ml of ethanol was heated for 2 h at 80°C. The mixture was evaporated, and the residue was recrystallized from ethanol. Yield 0.09 g (78%).

2-Morpholino-5-(2,3,4,5-tetrafluorophenyl-1,3,4-thiadiazole (VII). *a*. A 0.1-ml portion of DBU was added to a suspension of 0.5 g (1.15 mmol) of ester **Ia** in 10 ml of dry benzene, and the mixture was heated for 2 h at 80°C and evaporated. The residue was recrystallized from ethanol. Yield 0.2 g (55%).

b. Potassium carbonate, 1.0 g (7.25 mmol), was added to a suspension of 1.6 g (4.0 mmol) of ester **Ia** in 15 ml of toluene, and the mixture was heated for 2 h under reflux and filtered while hot. The precipitate was washed with water and recrystallized from acetone to obtain 0.4 g (21%) of potassium salt **VIIIa**. The toluene solution was evaporated, and the residue was recrystallized from ethanol to obtain 0.9 g (71%) of thiadiazole **VII**.

Ethyl 3-[2-(R-thiocarbonyl)hydrazino]-2-tetra-(penta)fluorobenzoylacrylate potassium salts VIII. *a*. Potassium fluoride, 0.3 g (4.6 mmol), was added to a solution of 1.0 g (2.3 mmol) of ester **Ia** in 15 ml of acetonitrile, and the mixture was heated for 1 h under reflux. It was then cooled, and the precipitate of salt **VIIIa** was filtered off and recrystallized from acetone. Yield 0.45 g (41%). Salts **VIIIb** and **VIIId** were synthesized in a similar way.

b. Potassium carbonate, 0.64 g (4.6 mmol), was added to a solution of 1.0 g (2.3 mmol) of ester **Ia** in 12 ml of acetonitrile. The mixture was heated for 2 h under reflux and cooled, and the precipitate of salt **VIIIa** was filtered off and recrystallized from acetonitrile. Yield 0.5 g (46%). Salt **VIIIb** was obtained in a similar way.

c. Ethyl 3-ethoxy-2-(2,3,4,5-tetrafluorobenzoyl)-acrylate, 1.4 g (4.3 mmol), and potassium fluoride,

0.5 g (8.0 mmol), were added to a suspension of 0.6 g (4 mmol) of 4-morpholinothiosemicarbazide in 10 ml of acetonitrile. The mixture was heated for 1 h under reflux and evaporated, 10 ml of acetone was added to the residue, and the precipitate of salt **VIIIa** was filtered off and recrystallized from acetone. Yield 0.9 g (47%).

d. Potassium carbonate, 0.5 g (3.6 mmol), was added to a suspension of 0.8 g (1.8 mmol) of ester **Ic** in 10 ml of toluene. The mixture was heated for 2 h under reflux and cooled, and the precipitate of salt **VIIIc** was filtered off and recrystallized from ethanol. Yield 0.55 g (61%).

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