Acylimines of hexafluoroacetone and methyl trifluoropyruvate in cyclocondensation with 2-aminothiazolines

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Reactions of acylimines of hexafluoroacetone and methyl trifluoropyruvate with 2-aminothiazolines afforded fluorine-containing heterocycles of two structural types: 6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazines and 2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazoles.

Key words: acylimines, hexafluoroacetone, methyl trifluoropyruvate, 2-aminothiazolines, 6,7-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]triazines, 2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazoles, cyclocondensation.

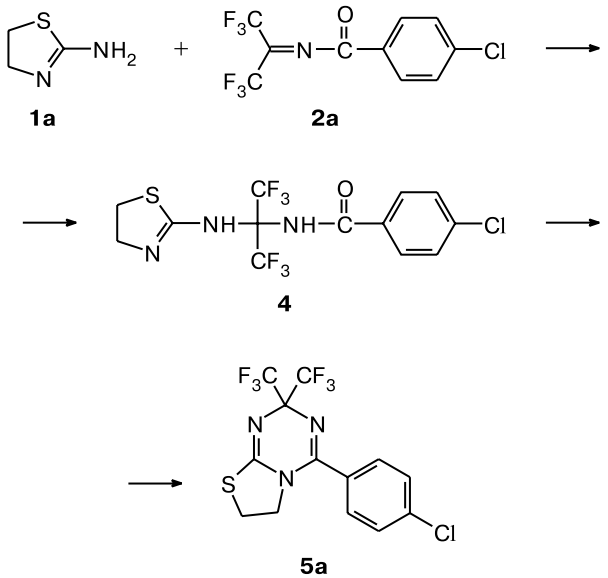
2-Aminothiazolines **1** are widely used in the synthesis of nitrogen-containing heterocyclic compounds, also *via* their cyclocondensation with various biselectrophilic reagents (*e.g.*, benzoyl isothiocyanate,[1] α-chloroacetophenones,[2] ethoxycarbonyl isothiocyanate,[3] and ethoxycarbonylimine of hexafluoroacetone[4]). It should also be noted that the aminothiazoline synthetic unit is successfully employed in the chemical design of pharmaceutical preparations.[5,6]

The present study was devoted to cyclocondensation of compound **1** with acylimines of hexafluoroacetone (HFA) **2** and methyl trifluoropyruvate (MTFP) **3**, which exhibit the properties of 1,3- and 1,2-biselectrophilic reagents.[4,7] The reaction afforded heterocycles containing an aminothiazoline fragment.

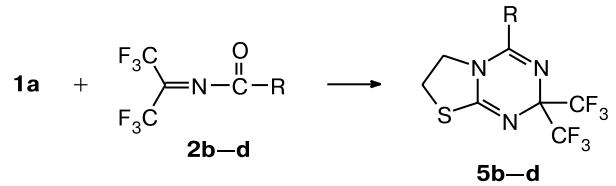
Like other compounds containing a primary amino group, 2-aminothiazoline **1a** exothermically reacted with imine **2a** to give stable acyclic adduct (at the amino group) **4**. The structure of the latter was proved by elemental analysis and NMR spectroscopy (Scheme 1).

Heating of compound **4** in DMF at 90—100 °C for 2 h in the presence of catalytic amounts of Et_3N yielded triazine **5a**. Cyclocondensation of 2-aminothiazoline **1a** with imines **2b—d** was carried out in one step without isolation of acyclic intermediates of the type **4** (Scheme 2).

Scheme 1



Scheme 2



2, 5: R = Et (**b**), 4-Me-C_6H_4 (**c**), 4-F-C_6H_4 (**d**)

In contrast to HFA acylimines **2**, MTFP acylimines **3** reacted with compound **1** as 1,2-biselectrophiles to give the corresponding tetrahydroimidazo[2,1-*b*][1,3]thiazoles **6**; in this case, no triazines of the type **5** were detected (Scheme 3).

5-Substituted 2-aminothiazoline hydrobromides **1b—d** can be successfully used in cyclocondensation with imines **2** and **3**; we carried out the reactions in the presence of equimolar amounts of Et_3N (Scheme 4).

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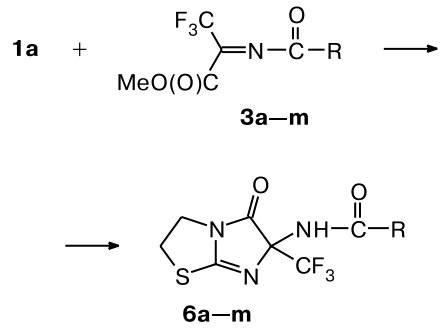
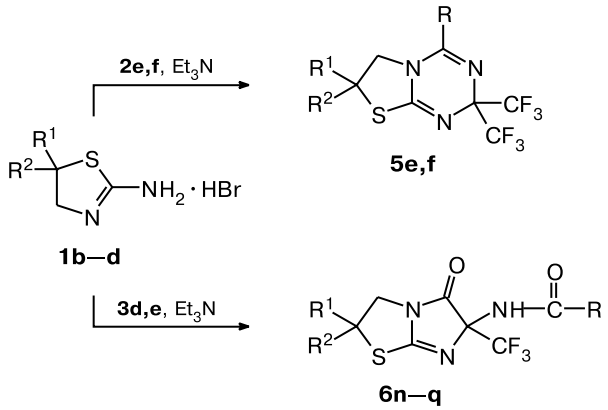
Scheme 3



3, 6: R = Bu^t (**a**), *cyclo*-C_3H_5 (**b**), CH_2Ph (**c**), Ph (**d**), 4-Me-C_6H_4 (**e**), 4-Bu^t-C_6H_4 (**f**), 4-MeO-C_6H_4 (**g**), 4-Cl-C_6H_4 (**h**), 2-F-C_6H_4 (**i**), 3-F-C_6H_4 (**j**), 4-F-C_6H_4 (**k**),  (**l**),  (**m**)

Scheme 4

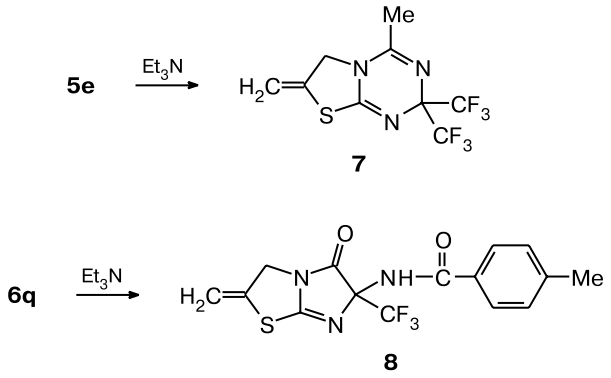


	R	R^1	R^2		R	R^1	R^2
1b	—	H	CH_2Br	**6n**	Ph	Me	Me
1c	—	Me	CH_2Br	**6o**	4-Me-C_6H_4	Me	Me
1d	—	Me	Me	**6p**	2-F-C_6H_4	Me	Me
2e, 5e	Me	H	CH_2Br	**6q**	4-Me-C_6H_4	H	CH_2Br
2f, 5f	Ph	Me	CH_2Br				

Triazines **5a**—**f** and imidazolinones **6a**—**q** are crystalline solids; their compositions and structures were confirmed by elemental analysis data and NMR spectra. The ^{19}F NMR spectra show characteristic signals at δ −1.2 to −0.7 for geminal trifluoromethyl groups in triazines **5a**—**f** and at δ 1.1—2.3 for a trifluoromethyl group in imidazolinones **6a**—**q**. The structures of compounds **5e** and **6q** were confirmed by chemical transformations. For instance, heating of compounds **5e** and **6q** in boiling benzene in the presence of equimolar amounts of Et_3N gave products **7** and **8** in 79 and 73% yields, respectively (Scheme 5).

Thus, the two-step cyclocondensation of 2-aminothiazolines (including 4-substituted ones) with acylimines of HFA and MTFP afforded earlier unknown fluorine-containing heterocycles of two structural types: 6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazines and 2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazoles.

Scheme 5



Experimental

^1H and ^{19}F NMR spectra were recorded on a Bruker DXP 200 spectrometer. Melting points were determined in glass capillaries. The starting 2-aminothiazoline **1a** (Aldrich) was used as purchased. 4-Substituted 2-aminothiazoline hydrobromides **1b**—**d** were prepared according to known procedures.[8,9] Acylimines of HFA (**2a**—**d**) and MTFP (**3a**—**e**) were prepared as described earlier.[10,11]

***N*-[1-(4,5-Dihydrothiazol-2-ylamino)-2,2,2-trifluoro-1-trifluoromethylethyl]-4-chlorobenzamide (4).** 2-Aminothiazoline **1a** (1.02 g, 0.01 mol) was added to a stirred solution of imine **2a** (3.04 g, 0.01 mol) in benzene (20 mL). After the exothermic reaction was completed, the solvent was removed and the residue was recrystallized from 50% EtOH to give benzamide **4** (3.8 g, 94%), m.p. 143—145 °C. Found (%): C, 38.32; H, 2.35; N, 10.16. $C_{13}H_{10}ClF_6N_3OS$. Calculated (%): C, 38.48; H, 2.48; N, 10.30. ^1H NMR (DMSO-d_6), δ: 3.27, 3.41 (both t, 2 H each, CH_2, J = 7.3 Hz); 7.36, 7.84 (both d, 2 H each, CH_{Ar}, J = 8.1 Hz); 8.02, 8.74 (both s, 1 H each, NH). ^{19}F NMR (DMSO-d_6), δ: 5.43 (s).

4-(4-Chlorophenyl)-2,2-bis(trifluoromethyl)-6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazine (5a). ***A.*** A mixture of benzamide **4** (2.03 g, 0.005 mol) and Et_3N (0.1 mL) in DMF (10 mL) was heated at 90—100 °C for 2 h and then poured into water (50 mL). The precipitate that formed was filtered off and recrystallized from 50% EtOH. The yield of compound **5a** was 1.5 g (77%).

B. 2-Aminothiazoline **1a** (3.04 g, 0.01 mol) was added to a stirred solution of imine **2a** (1.02 g, 0.01 mol) in DMF (20 mL). After the exothermic reaction was completed, Et_3N (0.1 mL) was added and the reaction mixture was heated at 90—100 °C for 2 h and poured into water (50 mL). The precipitate that formed was filtered off and recrystallized from 50% EtOH. The yield of compound **5a** was 2.6 g (67%).

4-Ethyl-2,2-bis(trifluoromethyl)-6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazine (5b), 4-(4-methylphenyl)-2,2-bis(tri-

fluoromethyl)-6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazine (5c), 4-(4-fluorophenyl)-2,2-bis(trifluoromethyl)-6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazine (5d), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)pivalamide (6a), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)cyclopropanecarboxamide (6b), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)phenylacetamide (6c), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)benzamide (6d), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-methylbenzamide (6e), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-*tert*-butylbenzamide (6f), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-methoxybenzamide (6g), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-chlorobenzamide (6h), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-2-fluorobenzamide (6i), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-3-fluorobenzamide (6j), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-fluorobenzamide (6k), *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)nicotinamide (6l), and *N*-(5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)thiophene-2-carboxamide (6m) were obtained analogously. The yields, melting points, and spectroscopic characteristics of compounds **5a—d** and **6a—m** are given in Tables 1 and 2.

7-Bromomethyl-4-methyl-2,2-bis(trifluoromethyl)-6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazine (5e). Triethylamine (1.1 g, 0.011 mol) and imine **2e** (2.1 g, 0.01 mol) were added to a stirred solution of thiazoline hydrobromide **1b** (3.1 g, 0.01 mol) in DMF (20 mL). After the exothermic reaction was completed, the reaction mixture was heated at 90—100 °C for 2 h and poured into water (50 mL). The precipitate that formed was filtered off and recrystallized from 50% EtOH. The yield of compound **5e** was 1.5 g (78%).

7-Bromomethyl-7-methyl-4-phenyl-2,2-bis(trifluoromethyl)-6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazine (5f), *N*-(2,2-dimethyl-5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)benzamide (6n), *N*-(2,2-dimethyl-5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-methylbenzamide (6o), *N*-(2,2-dimethyl-5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-2-fluorobenzamide (6h), and *N*-[2-(bromomethyl)-5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl]-4-methylbenzamide (6q) were obtained as described for compound **5e**. The yields, melting points, and spectroscopic characteristics of compounds **5e,f** and **6n—q** are given in Tables 1 and 2.

4-Methyl-7-methylene-2,2-bis(trifluoromethyl)-6,7-dihydro-2*H*-thiazolo[3,2-*a*][1,3,5]triazine (7). A solution of triazine **4e** (1.92 g, 0.005 mol) and Et_3N (0.5 g, 0.005 mol) in benzene (20 mL) was refluxed for 2 h. The precipitate that formed was filtered off, the solvent was concentrated, and the residue was recrystallized from 50% EtOH to give triazine **7** (1.2 g, 79%), m.p. 119—121 °C. Found (%): C, 35.81; H, 2.18; N, 13.71. $C_9H_7F_6N_3S$. Calculated (%): C, 35.65; H, 2.33; N, 13.86. ^{1}H NMR (DMSO-d_6), δ: 2.94 (s, 3 H, Me); 5.55 (t, 2 H, CH_2N, J = 2.1 Hz); 6.03, 6.18 (both q, 1 H each, CH_2=, J = 2.1 Hz). ^{19}F NMR (DMSO-d_6), δ: −1.12 (s).

Table 1. Yields, melting points, and elemental analysis data for compounds **5a—f** and **6a—q**

Compound	Yield (%)	M.p. /°C	Found / Calculated (%) C	H	N	Molecular formula
5a	77	126—128	40.42 / 40.27	2.19 / 2.08	10.68 / 10.84	$C_{13}H_8ClF_6N_3S$
5b	68	81—83	35.32 / 35.41	2.82 / 2.97	13.61 / 13.77	$C_9H_9F_6N_3S$
5c	62	92—94	45.61 / 45.78	2.93 / 3.02	11.59 / 11.44	$C_{14}H_{11}F_6N_3S$
5d	73	131—133	42.23 / 42.06	2.29 / 2.17	11.48 / 11.32	$C_{13}H_8F_7N_3S$
5e	78	96—98	28.26 / 28.14	2.28 / 2.10	11.12 / 10.94	$C_9H_8BrF_6N_3S$
5f	65	94—96	39.28 / 39.15	2.48 / 2.63	9.24 / 9.13	$C_{15}H_{12}BrF_6N_3S$
6a	78	215—217	42.83 / 42.71	4.42 / 4.56	13.73 / 13.59	$C_{11}H_{14}F_3N_3O_2S$
6b	74	241—243	40.79 / 40.96	3.58 / 3.44	14.42 / 14.33	$C_{10}H_{10}F_3N_3O_2S$
6c	70	251—252	48.81 / 48.98	3.35 / 3.52	12.07 / 12.24	$C_{14}H_{12}F_3N_3O_2S$
6d	66	212—214	47.27 / 47.42	3.21 / 3.06	12.92 / 12.76	$C_{13}H_{10}F_3N_3O_2S$
6e	69	238—240	48.81 / 48.98	3.69 / 3.52	12.37 / 12.24	$C_{14}H_{12}F_3N_3O_2S$
6f	64	235—242	52.76 / 52.98	4.58 / 4.71	11.03 / 10.90	$C_{17}H_{18}F_3N_3O_2S$
6g	78	239—241	45.65 / 46.80	3.48 / 3.37	11.81 / 11.69	$C_{13}H_{10}F_3N_3O_3S$
6h	69	253—255	42.77 / 42.93	2.61 / 2.49	11.72 / 11.55	$C_{13}H_9ClF_3N_3O_2S$
6i	64	216—218	44.79 / 44.96	2.73 / 2.61	12.21 / 12.10	$C_{13}H_9F_4N_3O_2S$
6j	65	216—218	44.85 / 44.96	2.78 / 2.61	12.25 / 12.10	$C_{13}H_9F_4N_3O_2S$
6k	74	240—242	44.78 / 44.96	2.49 / 2.61	12.27 / 12.10	$C_{13}H_9F_4N_3O_2S$
6l	71	216—218	43.49 / 43.64	2.91 / 2.75	17.13 / 16.96	$C_{12}H_9F_3N_4O_2S$
6m	63	280—282	39.22 / 39.40	2.53 / 2.40	12.69 / 12.53	$C_{11}H_8F_3N_3O_2S$
6n	72	242—244	50.26 / 50.42	4.11 / 3.95	11.58 / 11.76	$C_{15}H_{14}F_3N_3O_2S$
6o	63	270—272	51.62 / 51.75	4.17 / 4.34	11.18 / 11.31	$C_{16}H_{16}F_3N_3O_2S$
6p	68	252—254	48.16 / 48.00	3.32 / 3.49	11.03 / 11.19	$C_{15}H_{13}F_4N_3O_2S$
6q	66	202—204	41.43 / 41.30	2.87 / 3.00	9.79 / 9.63	$C_{15}H_{13}BrF_3N_3O_2S$

***N*-(2-Methylene-5-oxo-6-trifluoromethyl-2,3,5,6-tetrahydroimidazo[2,1-*b*]thiazol-6-yl)-4-methylbenzamide (8)** was obtained analogously. The yield of compound **8** was 1.3 g (73%), m.p. 225—227 °C. Found (%): C, 50.53; H, 3.52;

Table 2. ^{1}H and ^{19}F NMR spectra of compounds **5a—f** and **6a—q** in DMSO-d_6

Compound	NMR, δ (*J*/Hz)	
	^{1}H	^{19}F
5a	3.26, 3.98 (both t, 2 H each, CH_2, J = 7.6); 7.44, 7.69 (both d, 2 H each, CH_{Ar}, J = 8.1)	−1.12 (s)
5b	1.20 (t, 3 H, Me, J = 7.2); 2.52 (q, 2 H, MeC$\underline{H}_2$, J = 7.2); 3.32, 4.15 (both t, 2 H each, CH_2, J = 7.7)	−1.20 (s)
5c	2.38 (s, 3 H, Me); 3.61, 4.02 (both t, 2 H each, CH_2, J = 7.7); 7.32, 7.48 (both d, 2 H each, CH_{Ar}, J = 8.0)	−0.72 (s)
5d	3.33, 4.06 (both t, 2 H each, CH_2, J = 7.4); 7.24, 7.71 (both m, 2 H each, CH_{Ar})	−0.98 (s, 6 F, CF_3); −30.66 (m, 1 F)
5e	2.97 (s, 3 H, Me); 3.62—3.78 (m, 2 H, CH_2N); 4.08—4.25 (m, 3 H, CH_2Br + CHS)	−0.98, −1.15 (both m)
5f	1.72 (s, 3 H, Me); 3.69, 4.21 (both d, 1 H each, CH_2N, J = 11.2); 3.99 (s, 2 H, CH_2Br); 7.66 (m, 5 H, CH_{Ar})	−0.99 (s)
6a	0.91 (d, 6 H, Me, J = 7.2); 2.03 (m, 1 H, CH); 3.11 (m, 2 H, CH_2); 3.32 (m, 1 H, CH_2); 3.29 (m, 3 H, CH_2N + CH_2S); 9.42 (s, 1 H, NH)	1.16 (s)
6b	0.78 (m, 4 H, CH_2); 1.91 (m, 1 H, CH); 3.28 (m, 1 H, CH_2); 3.62—3.83 (m, 3 H, CH_2N + CH_2S); 9.89 (s, 1 H, NH)	1.14 (s)
6c	3.06 (s, 2 H, CH_2); 3.12 (m, 1 H, CH_2); 3.58—3.76 (m, 3 H, CH_2N + CH_2S); 7.23 (m, 5 H, CH_{Ar}); 9.42 (s, 1 H, NH)	1.25 (s)
6d	3.49 (m, 1 H, CH_2S); 3.78—3.96 (m, 3 H, CH_2N + CH_2S); 7.38—7.55 (m, 2 H, CH_{Ar}); 7.92 (d, 2 H, CH_{Ar}, J = 7.8); 9.83 (s, 1 H, NH)	2.35 (s)
6e	2.42 (s, 3 H, Me); 3.37 (m, 1 H, CH_2S); 3.64—3.85 (m, 3 H, CH_2N + CH_2S); 7.22, 7.83 (both d, 2 H each, CH_{Ar}, J = 7.7); 9.77 (s, 1 H, NH)	1.98 (s)
6f	1.36 (s, 9 H, Me); 3.37 (m, 1 H, CH_2S); 3.61—3.83 (m, 3 H, CH_2N + CH_2S); 7.23 (m, 5 H, CH_{Ar}); 7.41, 7.87 (both d, 2 H each, CH_{Ar}, J = 7.8); 9.76 (s, 1 H, NH)	1.89 (s)
6g	3.32 (m, 1 H, CH_2S); 3.66—3.79 (m, 3 H, CH_2N + CH_2S); 3.83 (s, 1 H, MeO); 7.22, 7.83 (both d, 2 H each, CH_{Ar}, J = 7.7); 9.77 (s, 1 H, NH)	2.07 (s)
6h	3.33—3.58 (m, 1 H, CH_2S); 3.79—3.96 (m, 3 H, CH_2N + CH_2S); 7.48, 7.97 (both d, 2 H each, CH_{Ar}, J = 7.8); 9.95 (s, 1 H, NH)	2.16 (s)
6i	3.49 (m, 1 H, CH_2S); 3.71—3.86 (m, 3 H, CH_2N + CH_2S); 7.12—7.27, 7.48—7.67 (both m, 2 H each, CH_{Ar}); 9.96 (s, 1 H, NH)	1.29 (s, 3 F, CF_3); −34.53 (m, 1 F)
6j	3.31—3.51 (m, 1 H, CH_2S); 3.63—3.91 (m, 3 H, CH_2N + CH_2S); 7.28, 7.47 (both m, 1 H each, CH_{Ar}); 7.76 (d, 2 H, CH_{Ar}, J = 7.8); 9.91 (s, 1 H, NH)	2.21 (s, 3 F, CF_3); −30.82 (m, 1 F)
6k	3.32—3.48 (m, 1 H, CH_2S); 3.64—3.85 (m, 3 H, CH_2N + CH_2S); 7.14 (m, 2 H, CH_{Ar}); 8.02 (m, 2 H, CH_{Ar}); 9.84 (s, 1 H, NH)	2.07 (s, 3 F, CF_3); −31.12 (m, 1 F)
6l	3.41 (m, 1 H, CH_2S); 3.72 (m, 3 H, CH_2N + CH_2S); 7.43 (t, 1 H, CH_{Ar}, J = 7.6); 8.23, 8.66 (both d, 1 H each, CH_{Ar}, J = 7.6); 9.04 (s, 1 H, CHAr); 10.08 (s, 1 H, NH)	2.11 (s)
?6m	3.43 (m, 1 H, CH_2S); 3.76 (m, 3 H, CH_2N + CH_2S); 7.21 (t, 1 H, CH, J = 7.8); 7.68, 8.12 (both d, 1 H each, CH, J = 7.8); 9.89 (s, 1 H, NH)	2.23 (s)
6n	1.66, 1.75 (both s, 3 H each, Me); 3.32, 3.59 (d, 1 H, CH_2, J = 10.2); 7.51, 7.96 (both m, 3 H each, CH_{Ar}); 9.85 (s, 1 H, NH)	1.89 (s)
6o	1.67, 1.75 (both s, 3 H each, Me); 2.53 (s, 3 H, CH_{Ar}); 3.27, 3.61 (both d, 1 H each, CH_2, J = 10.6); 7.29, 7.91 (both d, 2 H each, CH_{Ar}, J = 7.9); 9.86 (s, 1 H, NH)	1.92 (s)
6p	1.72, 1.76 (both s, 3 H each, Me); 3.38, 3.64 (both d, 1 H each, CH_2, J = 10.0); 7.29, 7.61 (both m, 2 H each, CH_{Ar}); 10.04 (s, 1 H, NH)	2.03 (s, 3 F, CF_3); −34.89 (m, 1 F)
6q	2.46 (s, 3 H, Me); 3.49 (m, 1 H, CH_2S); 3.66—3.94 (m, 3 H, CH_2S + CH_2N); 4.54 (m, 2 H, CH_2Br); 7.27, 7.86 (both d, 2 H each, CH_{Ar}, J = 8.1); 9.78, 9.82 (both s, 1 H, NH)	1.80, 2.32 (both s)

N, 11.68. $C_{15}H_{12}F_3N_3O_2S$. Calculated (%): C, 50.70; H, 3.40; N, 11.83. ^{1}H NMR (DMSO-d_6), δ: 2.51 (s, 3 H, Me); 4.24, 4.64 (both d, 1 H each, CH_2N, J = 13.8 Hz); 6.61 (br.s, 1 H, CH_2=); 6.68 (s, 1 H, CH_2=); 7.27, 7.84 (both d, 2 H each, CH_{Ar}, J = 8.2 Hz); 9.92 (s, 1 H, NH). ^{19}F NMR (DMSO-d_6), δ: 1.73 (s).

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