The Chemistry of Tetrafluoroallene: One-pot Synthesis of Trifluoromethylindolizines from 1,3-Diiodo-1,1,3,3-tetrafluoropropane by 1,3-Dipolar Cycloaddition†

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Heating a mixture of 1, 3-diiodo-1, 1, 3, 3-tetrafluoropropane (2), K_2CO_3, pyridinium bromides (3) in CH_3CN at 65 ℃ for 10 h gives the corresponding trifluoromethylindolizines.

Keywords tetrafluoroallene, trifluoromethylindolizine, 1,3-diiodo-1,1,3,3-tetrafluoropropane, 1,3-dipolar cycloaddition, one-pot synthesis

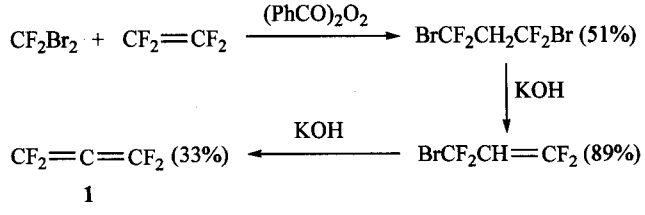
Introduction

Fluoroallene and 1,1-difluoroallene, as allene derivatives, are useful compounds which can undergo a quite variety of reactions, especially *e.g.* cycloadditions with alkenes,[1] dienes,[1a,2] nitrones[3] and diazo compounds.[4] However, their analog, tetrafluoroallene (**1**) was less investigated. Near 40 years ago, Banks *et al.*[5] first carried out the ionic reactions of **1** with hydrogen halides, HX (X = Br, Cl, F) and fluoride ion to give $CF_2XCH=CF_2$ and $CF_3CH=CF_2$ respectively. **1** readily dimerized to perfluoro(1,2-dimethylene cyclobutane) and homopolymerized to $[CF_2C(=CF_2)]_n$.[5,6] The reaction of tetrafluoroallene dimmer with trifluoronitrosomethane was also described.[5] The cycloaddition of **1** to CF_3NO[7] and $CF_3C\equiv CCF_3$[8] gave the corresponding (2 + 2) adducts. Some interesting heterocyclic compounds were obtained through 1,3-dipolar cycloaddition of **1** to *N*-phenylsydnone,[9] phenylazide,[9] nitrone,[10] diazophenylmethane[10] and diazodiphenylmethane.[10] Apparently, the chemistry of **1** has not been significantly developed due to its difficult preparation.

Tetrafluoroallene, **1**, was previously synthesized by three-step procedure starting from CF_2Br_2 shown as follows[5] (Scheme 1).

We improved the yield of **1** by using 1,3-diiodo-1,1,3,3-tetrafluoropropane ($ICF_2CH_2CF_2I$, **2**) as its precursor which can be smoothly synthesized by the reaction of CF_2I_2 with $CF_2=CH_2$ in the presence of $Pb(OAc)_4$.[11] Elimination of hydrogen iodide from **2** with K_2CO_3 in CH_3CN affords **1** in good yield.[11] Thus our new synthetic method for **1** makes it possible to further expand the investigation of **1**.

Scheme 1



On the other hand, we recently were interested in synthesizing fluorinated heterocyclic compounds from fluorinated olefins or their equivalents. For example, fluorinated indolizines could be prepared through 1,3-dipolar cycloaddition of fluoroalkenes,[12a] CF_3CH_2Cl (HFC-133a) or CF_3CH_2F (HFC-134a) to *N*-ylides.[12b] Because **1** is particularly sensitive to nucleophilic attack,[13] we envisioned that **1** would undergo the same reaction to give the indolizine derivatives. Herein, the results reported.

Results and discussion

Direct treatment of **1** with reactant under normal conditions is not convenient because **1** is a gas (b.p. −37.6 ℃). Thus we tried to carry out the 1,3-dipolar cycloaddition of *N*-phenacylpyridinium bromide (**3a**) to **2** in CH_3CN in the presence of K_2CO_3, provided **1** was generated *in situ*. The desired indolizine **4a** was indeed obtained in 61% yield. The control reaction, *i.e.*, the reaction from gas **1** instead of **2**, gave a comparable yield (68%) of the same product although the purification seemed somewhat easy (Scheme 2). Similarly, the other indolizine derivatives were synthesized by this one-pot pro-

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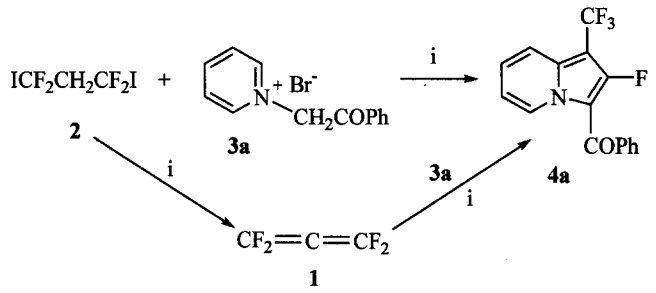
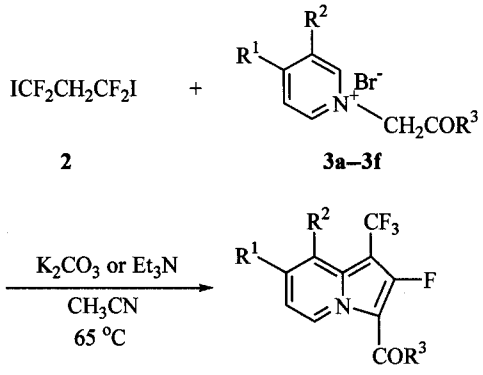
cedure (Scheme 3). The structures of the products (**4a**—**4f**) were established by their spectra data (MS, NMR, IR) and elemental analyses. The spectral data of **4a** and **4b** are in consistent with the reports in literature.[14]

Scheme 2



Scheme 3



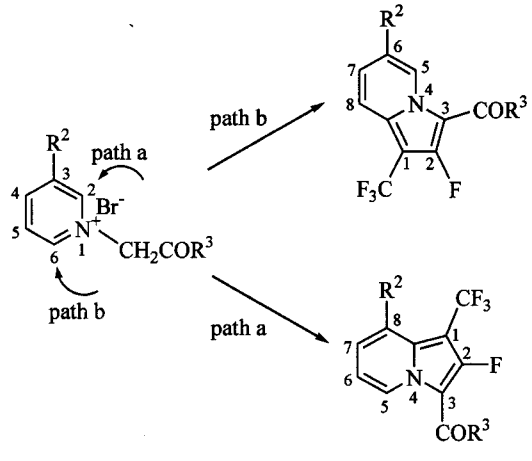
a: $R^1 = H$; $R^2 = H$; $R^3 = Ph$ **b**: $R^1 = H$; $R^2 = H$; $R^3 = OC_2H_5$

c: $R^1 = CH_3$; $R^2 = H$; $R^3 = Ph$ **d**: $R^1 = CH_3$; $R^2 = H$; $R^3 = OC_2H_5$

e: $R^1 = H$; $R^2 = Br$; $R^3 = Ph$ **f**: $R^1 = H$; $R^2 = Br$; $R^3 = OC_2H_5$

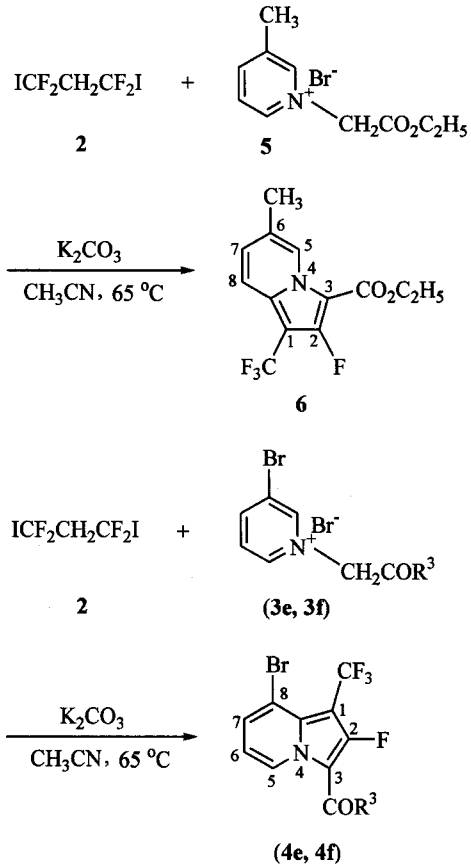
In the case of 3-substituted pyridinium *N*-ylide, it is possible to obtain either 6- or 8-substituted indolizines (Scheme 4).

Scheme 4



But in fact when 3-methyl-pyrimidinium bromide (**5**) was used, the 6-methyl indolizine (**6**) was only isolated. This was in contrast to the results when 3-bromopyridinium bromide (**3e**, **3f**) was employed, which solely gave 8-bromo-indolizine derivatives (**4e**, **4f**) (Scheme 5). ^{1}H NMR spectrum of compound **6** showed resonance at δ 9.19 (s, 1H, C^5-H), which is the characteristic signal of this kind of structure. The two doublet signals at δ 7.51 (d, $^3J_{HH}$ = 9 Hz, 1H) and 7.27 (d, $^3J_{HH}$ = 9 Hz, 1H) were assigned to C^8-H and C^7-H, respectively. But in the case of 8-bromoindolizine **4f**, the doublet signal at δ 9.69 (d, $^3J_{HH}$ = 1.8 Hz, 1H, ArH) was assigned to C^5-H; the doublet signal at δ 7.55 and the quartet signal at δ 7.38 to C^7-H and C^6-H, respectively.

Scheme 5

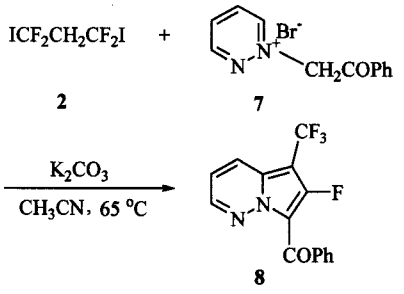


N-Phenacylpyridazinium bromide (**7**) reacted also in a similar way with $ICF_2CH_2CF_2I$ (**2**) to give the corresponding pyrrolo[1, 2-*b*]pyridazine derivatives (**8**) (Scheme 6).

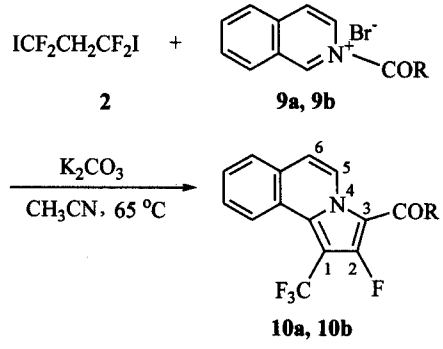
In the case of isoquinolinium *N*-ylides (**9a**, **9b**) (Scheme 7), cycloaddition took place at its 1-position, which was characterized by its ^{1}H NMR spectra data. Taking **10b** as an example, the doublet signals at δ 9.41 (d, $^3J_{HH}$ = 7.6 Hz, 1H) and 7.55 (d, $^3J_{HH}$ = 7.5 Hz, 1H) were assigned to C^5-H and C^6-H, respectively.

A possible mechanism for this reaction is shown in Scheme 8. 1,3-Dipolar cycloaddition of *N*-ylide generated from *N*-phenacylpyridinium bromide after HBr elimination

Scheme 6

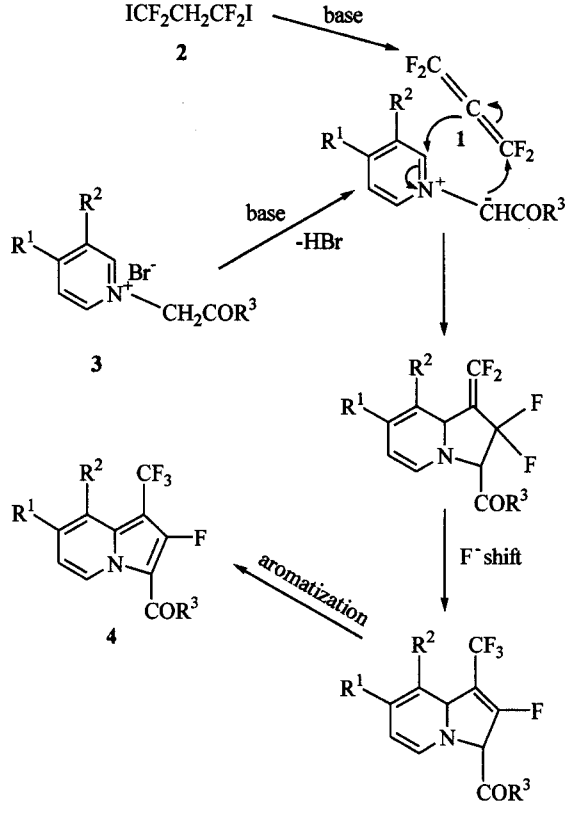


Scheme 7



a: R = Ph; **b**: R = OC_2H_5

Scheme 8



to **1** produced *in situ* from **2** in the presence of K_2CO_3 takes place first. The resultant five-member ring intermediate then undergoes fluoride ion migration. After aromatization, the desired trifluoromethylindolizine derivatives are obtained. The similar anionotropic rearrangement and aromatization have been reported previously by Taylor.[9,10]

The yields of all these cycloadducts are summarized in Table 1.

In summary, a convenient one-pot method for synthesizing trifluoromethylated indolizines from tetrafluoroallene precursor ($ICF_2CH_2CF_2I$, **2**) through 1,3-dipolar cycloaddition is described.

Experimental

Melting points were uncorrected. ^1H NMR and ^{19}F NMR spectra were recorded on a Varian-360L instrument or Bruker AM-300 spectrometer for solution of $CDCl_3$ or CD_3COCD_3 with TMS and $CFCl_3$ as the internal and external standards respectively, and the upfields are negative. IR spectra were obtained with a Perkin Elmer 983G spectro-photometer on KBr disks. Lower resolution mass spectra (LRMS) and higher resolution mass spectra (HRMS) were obtained on a HP-5989a and Finnigan MAT-8430 instruments, respectively. Organic solvents were dried by standard methods when necessary. Commercially obtained reagents were used without further purification. Flash column chromatography was carried out using 300—400 mesh silica gel.

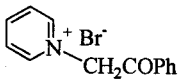
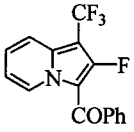
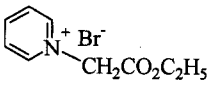
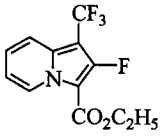
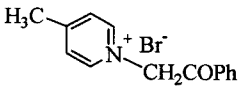
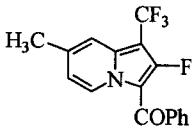
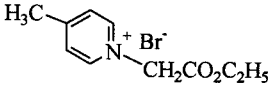
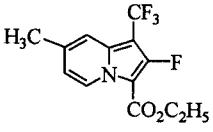
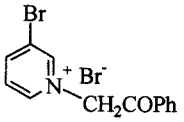
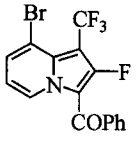
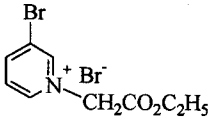
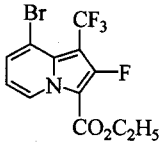
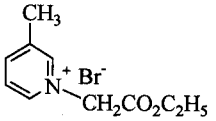
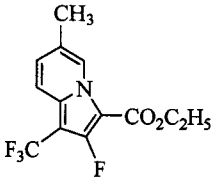
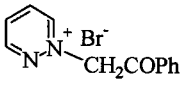
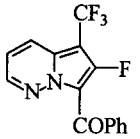
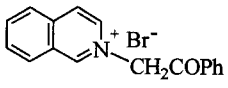
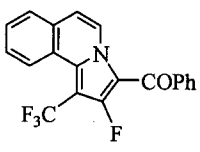
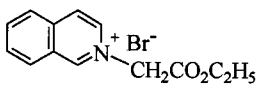
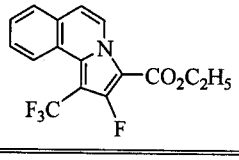
General procedure A: one-pot method

A 6 mL pyrex tube was placed $ICF_2CH_2CF_2I$ (**2**) (5 mmol, 1.839 g), pyridinium salt (3 mmol) and anhydrous acetonitrile (3 mL). After addition of K_2CO_3 (10 mmol, 1.382 g), the tube was immediately sealed and then heated in an oil bath at 65 ℃ for 10 h. The tube was cooled to −50 ℃ for 5 min, opened and then warmed again to room temperature. The mixture was poured into H_2O (40 mL) and extracted with Et_2O (3 × 15 mL). Then the combined organic layer was washed with brine (3 × 20 mL). The organic layer was dried over anhydrous sodium sulfate. The solvent was removed under reduced pressure and the crude product was purified by flash column chromatography to give the product.

General procedure B: two-step method

Tetrafluoroallene (**1**) was prepared according to literature.[11] Into a 6 mL pyrex tube containing pyridinium salt (3 mmol), K_2CO_3 (4 mmol, 0.553 g), anhydrous CH_3CN (3 mL) was condensed $CF_2=C=CF_2$ (0.448 g 4 mmol). The tube was sealed and allowed to warm to room temperature and then heated in an oil bath at 65 ℃ for 8 h. The subsequent operation was the same as procedure A.

Table 1 Yields (isolated material) of 1,3-dipolar cycloadducts (based on dipoles)

Entry	Substrate		Product		Yield (%)
1		**3a**		**4a**	61[a](68[b])
2		**3b**		**4b**	71[a]
3		**3c**		**4c**	75[a]
4		**3d**		**4d**	30[a]
5		**3e**		**4e**	81[a,c]
6		**3f**		**4f**	32[a](41[b])
7		**5**		**6**	55[a](63[b])
8		**7**		**8**	20[a]
9		**9a**		**10a**	70[a]
10		**9b**		**10b**	80[a](87[b])

[a] One-pot yields; [b] two-step yields; [c] Et_3N was used as the base.

(*2-Fluoro-1-trifluoromethyl-indolizin-3-yl*)-*phenyl-methanone* (**4a**) Colorless solid; m.p. 99—101 ℃; ^{1}H NMR ($CDCl_3$) δ: 9.80 (d, J = 6.9 Hz, 1H, ArH), 7.08—7.78 (m, 8H, ArH); ^{19}F NMR (CD_3COCD_3) δ: −54.7 (d, J = 11.1 Hz, 3F, CF_3), −134.3 (q, J = 11.0 Hz, 1F, CF); IR (KBr) ν: 3064, 1616, 1554, 1410, 1256, 1226, 1108, 958 cm^{-1}; MS m/z (%): 307 (M^+, 100.00), 288 (35.5), 238 (16.7), 230 (83.3), 105 (76.37), 77 (64.60). Anal. calcd for $C_{16}H_9F_4NO$: C 66.55, H 2.95, N 4.56, F 24.73; found C 62.90, H 3.32, N 4.62, F 24.46.

2-Fluoro-1-trifluoromethyl-indolizine-3-carboxylic acid ethyl ester (**4b**) Colorless solid; m.p. 101—103 ℃; ^{1}H NMR (CD_3COCD_3) δ: 9.53 (d, J = 6.9 Hz, 1H, ArH), 7.24—7.80 (m, 3H, ArH), 4.44 (q, J = 7.1 Hz, 2H, CH_2), 1.41 (t, J = 7.1 Hz, 3H, CH_3); ^{19}F NMR (CD_3COCD_3) δ: −55.60 (d, J = 11.3 Hz, 3F, CF_3), −141.34 (q, J = 9.9 Hz, 1F, CF); IR (KBr) ν: 3123, 3001, 1701, 1643, 1558, 1486, 1435, 1261, 1214 cm^{-1}; MS m/z (%): 275 (M^+, 86.63), 256 (16.15), 230 (73.22), 203 (100.00), 184 (27.96).

(*2-Fluoro-7-methyl-1-trifluoromethyl-indolizin-3-yl*)-*phenyl-methanone* (**4c**) Colorless solid; m.p. 116—118 ℃; ^{1}H NMR (CD_3COCD_3) δ: 9.66 (d, J = 7.2 Hz, 1H, ArH), 7.19—7.82 (m, 7H, ArH), 2.54 (s, 3H, CH_3); ^{19}F NMR (CD_3COCD_3) δ: −55.66 (d, J = 7.7 Hz, 3F, CF_3), −137.88 (q, J = 10.5 Hz, 1F, CF); IR (KBr) ν: 3068, 1652, 1615, 1558, 1471, 1408, 1257, 1231, 1110 cm^{-1}; MS m/z (%): 321 (M^+, 3.85), 262 (100.00), 183 (91.69), 105 (13.15); HRMS (EI) calcd for $C_{17}H_{11}F_4NO$ 321.07768, found 321.07545.

2-Fluoro-7-methyl-1-trifluoromethyl-indolizine-3-carboxylic acid ethyl ester (**4d**) Colorless solid; m.p. 73—75 ℃; ^{1}H NMR ($CDCl_3$) δ: 9.36 (d, J = 7.4 Hz, 1H, ArH), 7.41 (s, 1H, ArH), 6.83 (d, J = 7.4 Hz, 1H, ArH), 4.41 (q, J = 6.9 Hz, 2H, CH_2), 2.43 (s, 3H, CH_3), 1.41 (t, J = 7.1 Hz, 3H, CH_3); ^{19}F NMR ($CDCl_3$) δ: −54.98 (d, J = 15.7 Hz, 3F, CF_3), −138.34 (q, J = 16.6 Hz, 1F, CF); IR (KBr) ν: 3142, 3001, 2926, 1697, 1651, 1562, 1487, 1471, 1426, 1267, 1220, 1108, 1029, 800 cm^{-1}; MS m/z (%): 289 (M^+, 63.85), 270 (10.52), 217 (100), 198 (12.49); HRMS (EI) calcd for $C_{13}H_{11}F_4NO_2$ 289.07259, found 289.07325.

(*8-Bromo-2-fluoro-1-trifluoromethyl-indolizin-3-yl*)-*phenyl-methanone* (**4e**) Pale yellow solid; m.p. 130—132 ℃; ^{1}H NMR (CD_3COCD_3) δ: 9.80 (d, J = 6.9 Hz, 1H, ArH), 7.23—7.97 (m, 7H, ArH); ^{19}F NMR (CD_3COCD_3) δ: −44.90 (d, J = 7.6 Hz, 3F, CF_3), −129.73 (q, J = 10.5 Hz, 1F, CF); IR (KBr) ν: 3126, 3062, 1614, 1601, 1540, 1419, 1395, 1257, 1120, 1109 cm^{-1}; MS m/z (%): 385 (M^+, 46.48), 387 (46.89), 307 (25.50), 105 (100.00), 77 (81.75); HRMS (EI) calcd for $C_{16}H_8BrF_4NO$ 384.97254, found 384.97359.

8-Bromo-2-fluoro-1-trifluoromethyl-indolizine-3-carboxylic acid ethyl ester (**4f**) Colorless solid; m.p. 90—92 ℃; ^{1}H NMR ($CDCl_3$) δ: 9.69 (d, J = 1.8 Hz, 1H, ArH), 7.55 (d, J = 10.3 Hz, 1H, ArH), 7.38 (dd, J = 1.7, 10.4 Hz, 1H, ArH), 4.43 (q, 2H, J = 7.2 Hz, CH_2), 1.42 (t, J = 7.2 Hz, 3H, CH_3); ^{19}F NMR ($CDCl_3$) δ: −54.92 (d, J = 10.8 Hz, 3F, CF_3), −137.20 (q, J = 10.8 Hz, 1F, CF); IR (KBr) ν: 3134, 2987, 1689, 1552, 1472, 1428, 1253, 1210, 1164, 954 cm^{-1}; MS m/z (%): 353 (M^+, 99.85), 355 (97.29), 325 (67.67), 308 (56.28), 281 (100.00), 201 (34.51); HRMS (EI) calcd for $C_{12}H_8BrF_4NO_2$ 352.96745, found 352.96571.

2-Fluoro-6-methyl-1-trifluoromethyl-indolizine-3-carboxylic acid ethyl ester (**6**) Pale yellow solid; m.p. 106—108 ℃; ^{1}H NMR (CD_3COCD_3) δ: 9.19 (s, 1H, ArH), 7.51 (d, J = 9 Hz, 1H, ArH), 7.27 (d, J = 9 Hz, 1H, ArH), 4.42 (q, J = 7.2 Hz, 2H, CH_2), 2.43 (s, 3H, CH_3), 1.40 (t, J = 7.2 Hz, 3H, CH_3); ^{19}F NMR (CD_3COCD_3) δ: −55.57 (d, J = 6.1 Hz, 3F, CF_3), −141.76 (q, J = 10.6 Hz, 1F, CF); IR (KBr) ν: 3127, 2997, 1698, 1629, 1555, 1432, 1247, 1226, 1147, 1096, 951 cm^{-1}; MS m/z (%): 289 (M^+, 73), 270 (8.70), 261 (68.99), 244 (55.70), 217 (100.00), 198 (12.30); HRMS (EI) calcd for $C_{13}H_{11}F_4NO_2$ 289.07259, found 289.07262.

(*6-Fluoro-5-trifluoromethyl-pyrrolo*[*1,2-b*]*pyridazin-7-yl*)-*phenyl-methanone* (**8**) Viscous oil; ^{1}H NMR (CD_3COCD_3) δ: 7.38—8.54 (m, ArH); ^{19}F NMR (CD_3COCD_3) δ: −55.28 (d, J = 8.8 Hz, 3F, CF_3), −146.98 (q, J = 8.7 Hz, 1F, CF); IR (film) ν: 3067, 1651, 1629, 1548, 1467, 1417, 1251, 1105, 943 cm^{-1}; MS m/z (%): 308 (M^+, 100), 289 (16.25), 279 (50.42), 239 (3.64), 231 (57.84), 105 (63.81), 77 (90.06); HRMS (EI) calcd for $C_{15}H_8F_4N_2O$ 308.05728, found 308.05334.

(*2-Fluoro-1-trifluoromethyl-pyrrolo*[*2,1-a*]*-isoquinolin-3-yl*)-*phenyl-methanone* (**10a**) Colorless solid; m.p. 182—184 ℃; ^{1}H NMR (CD_3COCD_3) δ: 9.36 (d, J = 7.6 Hz, 1H, ArH), 8.45 (d, J = 9.0 Hz, 1H, ArH), 7.55—8.05 (m, 9H, ArH); ^{19}F NMR (CD_3COCD_3) δ: −54.72 (d, J = 22.0 Hz, 3F, CF_3), −139.25 (q, J = 23.3 Hz, 1F, CF); IR (KBr) ν: 3147, 1616, 1557, 1457, 1420, 1386, 1279, 1207, 1106 cm^{-1}; MS m/z (%): 357 (M^+, 100.00), 329 (9.32), 280 (30.23), 252 (18.44), 105 (29.39), 77 (37.49). Anal. calcd for $C_{20}H_{11}F_4NO$: C 67.23, H 3.10, N 3.92, F 21.27; found C 66.92, H 3.28, N 3.74, F 21.07.

2-Fluoro-1-trifluoromethyl-pyrrolo[*2,1-a*]*-isoquinolin-3-carboxylic acid ethyl ester* (**10b**) Colorless solid; m.p. 156—158 ℃; ^{1}H NMR (CD_3COCD_3) δ: 9.41 (d, J = 7.6 Hz, 1H, ArH), 7.76—8.42 (m, 4H, ArH), 7.55 (d, J = 7.5 Hz, 1H, ArH), 4.45 (q, J = 7.2 Hz, 2H, CH_2), 1.41 (t, J = 7.2 Hz, 3H, CH_3); ^{19}F NMR (CD_3COCD_3) δ: −54.74 (d, J = 21.2 Hz, 3F, CF_3), −141.41 (q, J = 23.2 Hz, 1F, CF); IR (KBr) ν:

3138, 2921, 1701, 1644, 1568, 1524, 1458, 1259, 1201, 1111, 1068, 798 cm^{-1}; MS *m/z* (%): 325 (M^+, 59.16), 297 (42.50), 280 (43.37), 253 (76.41), 69 (36.16), 43 (100); HRMS (EI) calcd for $C_{16}H_{11}F_4NO_2$ 325.07259, found 325.07363.

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