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A convenient three-component reaction for the construction of multifunctional 6-fluoroalkyl-1,2,3,4-tetrahydropyrimidines

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Abstract

β-Fluoroalkylenaminoketones were prepared and their reaction with primary amines and formaldehyde were studied. Multifunctional 6-fluoroalkyl-1,2,3,4-tetrahydropyrimidines were synthesized conveniently from the three-component reaction in good yields under mild conditions.
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Keywords: β-Fluoroalkylenaminoketone; Fluoroalkylated 1,2,3,4-tetrahydropyrimidine; Primary amine; Formaldehyde

1. Introduction

The ability of fluorine atom to enhance biological and therapeutic activities of certain organic compounds has led to widespread interest in selective introduction of fluorine atom and fluoroalkyl groups into organic molecules [1–5], especially those heterocyclic compounds which have potential biological activities. 1,2,3,4-Tetrahydropyrimidine ring is an important moiety in both natural and synthetic organic compounds, and many compounds containing it show interesting biological activities [6–12]. Accordingly many methods have been developed for the synthesis of various 1,2,3,4-tetrahydropyrimidines [13]. However, the synthesis of fluoroalkylated 1,2,3,4-tetrahydropyrimidines is less studied [14] and their biological activities remain unexplored. Therefore, it is our interest to develop synthetic methodologies for those fluorine-containing heterocycles which might have unique properties by the introduction of fluoroalkyl groups.

Recently, it was found in our laboratory that the reaction of fluorine-containing enaminoketones or 3-fluoroalkylanilinoacrylic acid esters with primary amines and formaldehyde could afford multifunctional 1,2,3,4-tetrahydropyrimidines under mild conditions [14]. To explore the scope of this procedure further, β-fluoroalkylenaminoketones were prepared and their reaction with primary amines and formaldehyde were investigated. The results of this three-component reaction are reported in this paper.

2. Results and discussion

β-Fluoroalkylenaminoketones (**1**) were prepared from commercial available fluoroalkyl iodides as shown in Scheme 1 [15].

The reaction of **1b** with methylamine and formaldehyde was first carried out in refluxing acetonitrile with a mole ratio of 1:2:4. It was found that the reaction was very slow and only trace product was formed after 24 h. Then the amount of methylamine and formaldehyde was increased. With a ratio of 1:8:18 (**1**/methylamine/formaldehyde), the reaction was completed in 10 h. After workup, a light yellow solid was obtained. Spectral and elemental analysis showed that it was the expected product, 6-chlorodifluoromethyl-5-*tert*-butylcarbonyl-3-methyl-1-phenyl-1,2,3,4-tetrahydropyrimidine (**3ba**) (Scheme 2).

To find suitable conditions, the reaction of **1b**, methylamine and formaldehyde under various conditions was investigated. As shown in Table 1, the yield of **3ba** was influenced by the ratio of three reactants and better result was obtained when more methylamine and formaldehyde were used. Solvent and temperature also played important roles in the reaction. When the reaction was carried out in DMF at 110 °C, the reaction time was fantastically shortened, and much less methylamine and formaldehyde were needed for a satisfactory conversion of **1b**. Increasing temperature further made the reaction more

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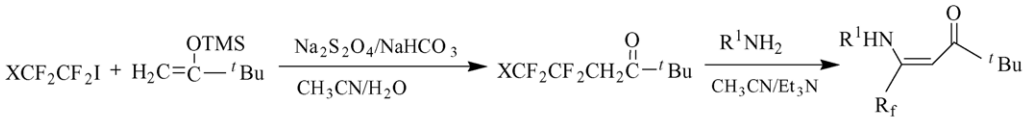
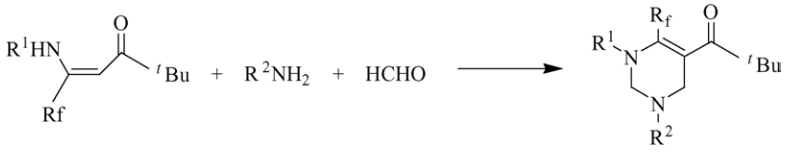


1

1a: $R_f = CF_3$, $R^1 = C_6H_5$
1b: $R_f = CF_2Cl$, $R^1 = C_6H_5$
1c: $R_f = CF_2Cl$, $R^1 = p\text{-}CH_3OC_6H_4$
1d: $R_f = CF_2Cl$, $R^1 = p\text{-}BrC_6H_4$

Scheme 1.



1 **2** **3**

Scheme 2.

complex. Therefore, an optimized condition was chosen for this three-component reaction as follows: in DMF at 110 °C with a mole ratio of 1:3:6.5 (**1b**:CH_3NH_2:HCHO).

Using the optimized conditions, various β-fluoroalkylenaminoketones and primary amines were examined. As shown in Table 2, aliphatic primary amines reacted readily with **1** and formaldehyde to give the desired product in good yields. The length and bulkiness of primary amine had little influence on the reaction. However, the reaction was very complicated when aromatic amines such as aniline and *para*-methoxy aniline were used and no expected cyclic products were obtained. The electron charge of the aromatic group in enaminoketones had little influence on the reaction. Enaminoketones containing either electron-rich or electron-deficient aromatic ring gave satisfactory results.

In conclusion, the three-component reaction of β-fluoroalkylenaminoketone, primary amine and formaldehyde was achieved under mild conditions, providing a convenient method for the synthesis of fluoroalkylated multifunctional 1,2,3,4-tetrahydropyrimidines. Using this method, a series of novel multifunctional 6-fluoroalkyl-1,2,3,4-tetrahydropyrimidines were synthesized from easily available starting materials for the first time.

Table 1
The reaction of **1b** with methylamine and formaldehyde under different conditions[a]

Entry	Solvent	**1b**:CH_3NH_2:HCHO	Temperature (°C)	Time (h)	Yield (%)[b]
1	CH_3CN	1:2:4	Reflux	24	Trace
2	CH_3CN	1:4:9	Reflux	17	78
3	CH_3CN	1:8:18	Reflux	10	87
4	DMF	1:8:18	80	9	90
5	DMF	1:4:9	80	15	81
6	DMF	1:4:9	110	2.5	90
7	DMF	1:3:6.5	110	3	88
8	DMF	1:2:4	110	10	50
9	DMF	1:3:6.5	140	2	Complex

[a] All reactions were carried out with **1** (1 mmol) in solvent (5 mL).
[b] Determined by ^{19}F NMR.

3. Experimental

3.1. General

Melting points were uncorrected. IR spectra were taken on a Perkin-Elmer Jeol 983 spectrophotometer. ^1H NMR spectra were measured on a Bruker AM300 (300 MHz) spectrometer using TMS as internal standard. ^{19}F NMR spectra were taken on a Bruker AM300 (282 MHz) spectrometer using $CFCl_3$ as external standard. Mass spectra were obtained on a Hewlett–Packard HP-5989A spectrometer. Column chromatography was performed on silica gel H, particle size 10–40 μm.

Table 2
The reaction of **1** with primary amines and formaldehyde[a]

Entry	1	Time (h)	R_f	R^1	R^2	Product	Yield (%)[b]
1	**1a**	2	CF_3	Ph	Me	**3aa**	85
2	**1a**	3	CF_3	Ph	Et	**3ab**	83
3	**1a**	3	CF_3	Ph	*n*-Pr	**3ac**	80
4	**1a**	3	CF_3	Ph	*t*-Bu	**3ad**	84
5	**1b**	3	CF_2Cl	Ph	Me	**3ba**	85
6	**1b**	4	CF_2Cl	Ph	Et	**3bb**	80
7	**1b**	4	CF_2Cl	Ph	*n*-Pr	**3bc**	80
8	**1b**	5	CF_2Cl	Ph	*t*-Bu	**3bd**	80
9	**1c**	3	CF_2Cl	*p*-OMe-Ph	Me	**3ca**	82
10	**1c**	3	CF_2Cl	*p*-OMe-Ph	Et	**3cb**	80
11	**1c**	4	CF_2Cl	*p*-OMe-Ph	*n*-Pr	**3cc**	78
12	**1c**	5	CF_2Cl	*p*-OMe-Ph	*t*-Bu	**3cd**	80
13	**1d**	4	CF_2Cl	*p*-Br-Ph	Me	**3da**	79

[a] All reactions were carried out with **1** (1 mmol) in DMF (5 mL).
[b] Isolated yields based on **1**.

3.2. Typical procedure for the synthesis of 6-fluoroalkyl-1,2,3,4-tetrahydropyrimidines (**3**)

To a solution of **1** (1 mmol) in DMF (5 mL), were added primary amines **2** (3 mmol) and 37% formaldehyde (6.5 mmol). The mixture was stirred at 110 °C for 2–5 h (monitored by TLC or ^{19}F NMR). After completion of the reaction, the mixture was cooled to room temperature, diluted with water and extracted with Et_2O. The organic layer was washed with saturated NaCl solution and dried over anhydrous Na_2SO_4. The residue obtained after evaporation of the solvent was purified by flash chromatography on silica gel eluting with petroleum-ethyl acetate to give **3**.

3.2.1. 5-tert-Butylcarbonyl-3-methyl-1-phenyl-6-trifluoromethyl-1,2,3,4-tetrahydropyrimidine (**3aa**)

Light yellow liquid. IR (film): ν 3062, 2975, 1695, 1646, 1596, 1492, 1370, 1183, 1158, 1135, 865, 767, 699 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.28–7.33 (2H, m), 7.14–7.17 (3H, m), 3.92 (2H, s), 3.35 (2H, s), 2.29 (3H, s), 1.26 (9H, s); ^{19}F NMR (282 MHz, $CDCl_3$): δ −60.71 (s); EIMS (70 eV) *m/z* (%): 326 [M^+] (23), 269 (53), 57 (100). Anal. Calcd. for $C_{17}H_{21}F_3N_2O$: C, 62.56; H, 6.49; N, 8.58. Found: C, 62.53; H, 6.59; N, 8.34.

3.2.2. 5-tert-Butylcarbonyl-3-ethyl-1-phenyl-6-trifluoromethyl-1,2,3,4-tetrahydropyrimidine (**3ab**)

Light yellow liquid. IR (film): ν 3062, 2974, 1694, 1645, 1597, 1493, 1369, 1159, 1135, 1071, 990, 763, 699 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.28–7.33 (2H, m), 7.14–7.17 (3H, m), 4.04 (2H, s), 3.43 (2H, s), 2.50 (2H, q, J = 7.2 Hz), 1.29 (9H, s), 0.95 (3H, t, J = 7.2 Hz); ^{19}F NMR (282 MHz, $CDCl_3$): δ −60.44 (s). EIMS (70 eV) *m/z* (%): 340 [M^+] (25), 283 (100), 227 (79), 57 (44). Anal. Calcd. for $C_{18}H_{23}F_3N_2O$: C, 63.51; H, 6.81; N, 8.23. Found: C, 63.43; H, 6.83; N, 8.03.

3.2.3. 5-tert-Butylcarbonyl-1-phenyl-3-propyl-6-trifluoromethyl-1,2,3,4-tetrahydropyrimidine (**3ac**)

Light yellow liquid. IR (film): ν 3061, 2968, 1693, 1647, 1597, 1493, 1479, 1465, 1367, 1297, 1180, 1158, 1136, 927, 699 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.26–7.32 (2H, m), 7.11–7.17 (3H, m), 4.04 (2H, s), 3.42 (2H, s), 2.38 (2H, t, J = 7.5 Hz), 1.27–1.37 (2H, m), 1.26 (9H, s), 0.74 (3H, t, J = 7.2 Hz,); ^{19}F NMR (282 MHz, $CDCl_3$): δ −60.42 (s); EIMS (70 eV) *m/z* (%): 354 [M^+] (16), 297 (44), 226 (55), 57 (100). Anal. Calcd. for $C_{19}H_{25}F_3N_2O$: C, 64.39; H, 7.11; N, 7.90. Found: C, 4.28; H, 7.08; N, 7.86.

3.2.4. 5-tert-Butylcarbonyl-1-phenyl-3-tert-butyl-6-trifluoromethyl-1,2,3,4-tetrahydropyrimidine (**3ad**)

Light yellow solid: mp 54–55 °C; IR (KBr): ν 3061, 2955, 1693, 1644, 1597, 1492, 1360, 1199, 1181, 1164, 1128, 917, 753, 698 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.26–7.32 (2H, m), 7.11–7.17 (3H, m), 3.94 (2H, s), 3.46 (2H, s), 1.27 (9H, s), 0.92 (9H, s); ^{19}F NMR (282 MHz, $CDCl_3$): δ −61.61 (s); EIMS (70 eV) *m/z* (%): 330 [M^+] (3), 368 (13), 226 (100), 283 (60). Anal. Calcd. for $C_{20}H_{27}F_3N_2O$: C, 65.20; H 7.39, N, 7.60. Found: C, 65.08; H, 7.45; N, 7.49.

3.2.5. 5-tert-Butylcarbonyl-6-chlorodifluoromethyl-3-methyl-1-phenyl-1,2,3,4-tetrahydropyrimidine (**3ba**)

Light yellow solid: mp 55–57 °C; IR (KBr): ν 3062, 3040, 2976, 2849, 1687, 1639, 1595, 1495, 1181, 1154, 1136, 1114, 896, 699 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.27–7.31 (2H, m), 7.15–7.19 (3H, m), 3.90 (2H, s), 3.35 (2H, s), 2.29 (3H, s), 1.29 (9H, s); ^{19}F NMR (282 MHz, $CDCl_3$): δ −48.11 (s). EIMS (70 eV) *m/z* (%): 342 [M^+] (5), 214 (100), 57 (21). Anal. Calcd. for $C_{17}H_{21}ClF_2N_2O$: C, 59.66; H, 6.17; N, 8.17. Found: C, 59.61; H, 6.25; N, 7.98.

3.2.6. 5-tert-Butylcarbonyl-6-chlorodifluoromethyl-3-ethyl-1-phenyl-1,2,3,4-tetrahydropyrimidine (**3bb**)

Light yellow liquid. IR (film): ν 3064, 2974, 1695, 1638, 1597, 1494, 1180, 1153, 1129, 896, 761, 699 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.26–7.36 (2H, m), 7.14–7.20 (3H, m) 3.99 (2H, s), 3.41 (2H, s), 2.49 (2H, q, J = 7.2 Hz), 1.29 (9H, s), 0.95 (3H, t, J = 7.2 Hz); ^{19}F NMR (282 MHz, $CDCl_3$): δ −47.68 (s); EIMS (70 eV) *m/z* (%): 356 [M^+] (4), 299 (18), 214 (100). Anal. Calcd. for $C_{18}H_{23}ClF_2N_2O$: C, 60.59; H, 6.50; N, 7.85. Found: C, 60.56; H, 6.28; N, 7.82.

3.2.7. 5-tert-Butylcarbonyl-6-chlorodifluoromethy-1-phenyl-3-propyl-1,2,3,4-tetrahydropyrimidine (**3bc**)

Light yellow liquid. IR (film): ν 3064, 2968, 1694, 1638, 1597, 1494, 1479, 1193, 1133, 912, 699 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.26–7.32 (2H, m), 7.14–7.20 (3H, m), 3.97 (2H, s), 3.41 (2H, s), 2.38 (2H, t, J = 7.2 Hz), 1.30–1.36 (2H, m), 1.29 (9H, s), 0.75 (3H, t, J = 7.2 Hz); ^{19}F NMR (282 MHz, $CDCl_3$): δ −47.72 (s); EIMS (70 eV) *m/z* (%): 370 [M^+] (10), 214 (100), 57 (72). Anal. Calcd. for $C_{19}H_{25}ClF_2N_2O$: C, 61.53; H, 6.79; N, 7.55. Found: C, 61.56; H, 6.99; N, 7.35.

3.2.8. 3-tert-Butyl-5-tert-butylcarbonyl-6-chlorodifluoromethyl-1-phenyl-1,2,3,4-tetrahydropyrimidine (**3bd**)

Light yellow solid: mp 64–66 °C; IR (KBr): ν 3062, 2974, 1684, 1630, 1595, 1493, 1479, 1366, 1289, 1181, 1133, 1124, 887, 696 cm^{-1}; ^{1}H NMR (300 MHz,$CDCl_3$): δ 7.26–7.29 (2H, m), 7.16–7.18 (3H, m), 3.95 (2H, s), 3.47 (2H, s), 1.27 (9H, s), 0.93 (9H, s); ^{19}F NMR (282 MHz, $CDCl_3$): δ −48.53 (s); EIMS (70 eV) *m/z* (%): 384 [M^+] (4), 214 (100), 57 (38). Anal. Calcd. for $C_{20}H_{27}ClF_2N_2O$: C, 62.41; H, 7.07; N, 7.28. Found: C, 62.62; H, 7.30; N, 7.00.

3.2.9. 5-tert-Butylcarbonyl-6-chlorodifluoromethyl-3-methyl-1-(4-methoxyphenyl)-1,2,3,4-tetrahydropyrimidine (**3ca**)

Light yellow liquid. IR (film): ν 3041, 2972, 1693, 1637, 1584, 1509, 1464, 1183, 1153, 1125, 1068, 902, 836, 794 cm^{-1}; ^{1}H NMR (300 MHz,$CDCl_3$): δ 7.12–7.16 (2H, m), 6.82–6.86 (2H, m), 3.78–3.81 (5H, m), 3.31 (2H, s), 2.30 (3H, s), 1.28 (9H, s); ^{19}F NMR (282 MHz, $CDCl_3$): δ −47.30 (s); EIMS (70eV) *m/z* (%): 372 [M^+] (6), 244 (100), 57 (84). Anal. Calcd. for

$C_{18}H_{23}ClF_2N_2O_2$: C, 57.99; H, 6.22; N, 7.51. Found: C, 57.85; H, 6.31; N, 7.48.

3.2.10. 5-tert-Butylcarbonyl-6-chlorodifluoromethyl-3-ethyl-1-(4-methoxyphenyl)-1,2,3,4-tetrahydropyrimidine (**3cb**)

Light yellow liquid. IR (film): ν 3041, 2972, 1693, 1637, 1584, 1508, 1494, 1479, 1199, 1180, 1127, 920, 899, 836, 791 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.16 (2H, d, J = 9.0 Hz), 6.83 (2H, d, J = 9.0 Hz), 3. 88 (2H, s), 3.78 (3H, s), 3.37 (2H, s), 2.48 (2H, q, J = 7.2 Hz), 1.27 (9H, s), 0.97 (3H, t, J = 7.2 Hz); ^{19}F NMR (282 MHz, $CDCl_3$): δ −46.98 (s); EIMS (70 eV) m/z (%): 386 [M^+] (7), 329 (36), 244 (100). Anal. Calcd. for $C_{19}H_{25}ClF_2N_2O_2$: C, 58.99; H, 6.51; N, 7.24; Found: C, 59.17; H, 6.62; N, 7.20.

3.2.11. 5-tert-Butylcarbonyl-6-chlorodifluoromethyl -1-(4-methoxyphenyl)-3-propyl-1,2,3,4-tetrahydropyrimidine (**3cc**)

Light yellow liquid. IR (film): ν 3041, 2966, 1693, 1637, 1584, 1509, 1475, 1465, 1246, 1193, 1180, 1153, 1127, 1034, 1008, 904, 836, 796 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.16 (2H, d, J = 8.7 Hz), 6.83 (2H, d, J = 8.7Hz), 3.84 (2H, s), 3.78 (3H, s), 3.35 (2H, s), 2.37 (2H, t, J = 7.2 Hz), 1.33–1.40 (2H, m), 1.26 (9H, s), 0.78 (3H, t, J = 7.2 Hz); ^{19}F NMR (282 MHz, $CDCl_3$): δ −47.90 (s); EIMS (70 eV) m/z (%): 400 [M^+] (7), 329 (40), 244 (100), 57 (53). Anal. Calcd. for $C_{20}H_{27}ClF_2N_2O_2$: C, 59.92; H, 6.79; N, 6.99. Found: C, 59.83; H, 6.80; N, 6.80.

3.2.12. 6-Chlorodifluoromethyl-1-(4-methoxyphenyl)-3-tert-butyl-5-tert-butylcarbonyl-1,2,3,4-tetrahydropyrimidine (**3cd**)

Light yellow liquid. IR (film): ν 2972, 1693, 1637, 1610, 1584, 1508, 1478, 1465, 1290, 1247, 1199, 1183, 1129, 1034, 921, 895, 836 cm^{-1}; ^1H NMR (300 MHz,$CDCl_3$): δ 7.14–7.16 (2H, m), 6.79–6.83 (2H, m), 3.85 (2H, s), 3.78 (3H, s), 3.44 (2H, s), 1.26 (9H, s), 0.94 (9H, s); ^{19}F NMR (282 MHz, $CDCl_3$): δ 48.16 (s); EIMS (70 eV) m/z (%): 414 [M^+] (7), 329 (37), 244 (100), 57 (14). Anal. Calcd. for $C_{21}H_{29}ClF_2N_2O_2$: C, 60.79; H, 7.04; N, 6.75. Found: C, 60.62; H, 7.02; N, 6.63.

3.2.13. 1-(4-Bromophenyl)-5-tert-butylcarbonyl-6-chlorodifluoromethyl-3-methyl-1,2,3,4-tetrahydropyrimidine (**3da**)

Light yellow liquid. IR (film): ν 2974, 1693, 1638, 1588, 1487, 1181, 1152, 1139, 1066, 1008, 900, 852, 768 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$): δ 7.44 (2H, d, J = 9.0 Hz), 7.05 (2H, d, J = 9.0Hz), 3.85 (2H, s), 3.33 (2H, s), 2.28 (3H, s), 1.28 (9H, s); ^{19}F NMR (282 MHz, $CDCl_3$): δ 48.46 (s). EIMS (70 eV) m/z (%): 422 [M^+] (8), 379 (27), 292 (73), 57 (100). Anal. Calcd. for $C_{18}H_{23}ClF_2N_2O_2$: C, 48.42; H, 4.78; N, 6.64. Found: C, 48.64; H, 4.97; N, 6.50.

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