

A Convenient Synthesis of 4-(Perfluoroalkyl)pyrimidines and 4-(Perfluoroalkyl)tetrahydropyrimidines

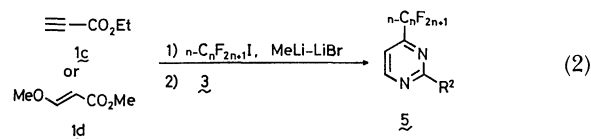
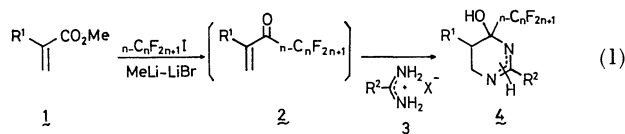
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Synopsis. Vinyl perfluoroalkyl ketones formed in situ from the reactions of perfluoroalkyllithiums with ethyl acrylate can easily be trapped with thiouronium and amidinium salts to give 4-(perfluoroalkyl)tetrahydropyrimidines in moderate to good yields. Similarly, ethynyl perfluoroalkyl ketones generated in situ can be converted to 4-(perfluoroalkyl)pyrimidines, although the yields are low.

The introduction of a perfluoroalkyl moiety into a molecular frame often brings about a drastic change in the physical properties of the molecule, which should have an effect on its biological activities. Thus, considerable pharmacological attention has been paid to perfluoroalkylated compounds of which fluorine-free parts are bioactive. Perfluoroalkylated pyrimidines are such compounds, of which both bioactivities and syntheses have been the subject of extensive studies.¹⁾ We have recently reported a new route to 6-(perfluoroalkyl)uracils based on a direct introduction of the perfluoroalkyl group into pyrimidine rings.²⁾ In this note we discuss an alternative facile route to (perfluoroalkyl)tetrahydropyrimidines and (perfluoroalkyl)pyrimidines involving the in situ trapping of unstable vinyl and ethynyl perfluoroalkyl ketones, either with thiouronium salts or with amidinium salts.



Perfluoroalkyllithiums generated from perfluoroalkyl iodides and methyllithium react with methyl acrylate to give the hemiacetals of the corresponding perfluoroalkyl vinyl ketones.³⁾ Perfluoroalkyl vinyl ketones are generally unstable and readily dimerize to dihydropyrans,⁴⁾ or undergo the addition of a nucleophile present in the reaction system.^{4,5)} Thus, using the thiouronium salt **3** as bidentate nucleophiles, perfluoroalkyl vinyl ketones **2** are efficiently converted to tetrahydropyrimidines **4** in good yields (Eq. 1; Table 1). Amidine hydrochlorides **3** may also be used similarly to give 2-alkyltetrahydropyrimidines **4** in comparative yields. In the case of 2-methylacrylate **1b**, diastereomeric mixtures of tetrahydropyrimidines **4i**–**4j** are obtained in respective yields of 76 and 66%. The diastereomeric ratios determined by the ¹⁹F NMR spectra are ca. 6:1 for **4i** and ca. 17:1 for **4j**, although a structural assignment of each isomer has not yet been made.

The treatment of ethyl propiolate (**1c**) with perfluoroalkyllithium followed by thiouronium salts affords 4-(perfluoroalkyl)pyrimidines **5** in less satisfactory yields (Eq. 2). By a similar procedure, pyrimidine **5** is also obtained from 3-methoxyacrylate **1d** in low yield.

Table 1. Preparation of 4-Perfluoroalkylpyrimidines

1	C _n F _{2n+1} I	3		Product	Yield/% ^{a)}
		R ²	X		
1a (R ¹ =H)	2	CH ₂ =CHCH ₂ S	I	(3a) 4a	77
1a	4	3a		4b	90
1a	6	3a		4c	93
1a	8	3a		4d	98
1a	8	CH ₂ =CHCH ₂ CH ₂ S	I	(3b) 4e	72
1a	8	EtS	I	(3c) 4f	76
1a	4	Me	Cl	(3d) 4g	61
1a	4	Ph	Cl	(3e) 4h	77
1b (R ¹ =Me)	4	3c		4i	76
1b	4	3d		4j	66
1c	6	3b		5a	27
1c	6	3c		5b	30
1d	6	3b		5a	9

a) Yields refer to the isolated compounds.

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In conclusion, the trapping of unstable vinyl and ethynyl perfluoroalkyl ketones with thiouronium and amidinium salts described here offers a new entry to 4-(perfluoroalkyl)tetrahydropyrimidines and 4-(perfluoroalkyl)pyrimidines, which are compounds of interest as the starting materials for pharmaceutical and agrochemical syntheses.

Experimental

Melting points were measured with a Yanagimoto micromelting point apparatus and are uncorrected. All boiling points refer to the Kugelrohr bath temperatures. All NMR spectra were observed with a JEOL GSX-270 spectrometer by using tetramethylsilane as an internal standard for ^1H and ^{13}C , and CFCl_3 for ^{19}F . Mass spectra were measured with a Hitachi M80LCAPI spectrometer under the following conditions: EI (20 eV) and CI (70 eV, methane as CI gas). IR spectra were recorded on a Hitachi 270-30 spectrophotometer.

General Procedure: To an ethereal solution of **1** and perfluoroalkyl iodide (1.2 equiv) is added an ethereal solution of MeLi-LiBr (1.1 equiv) with stirring at -78°C . After 1 h a methanolic solution of thiouronium salt or amidine hydrochloride **3** (1.2–5 equiv) is added and the resulting mixture is allowed to gradually warm up to room temperature. The reaction mixture is diluted with saturated aqueous NaHCO_3 . The organic phase is separated, and the aqueous phase is extracted with ether. The combined ethereal extracts are washed with brine, dried over Na_2SO_4 , and evaporated. The residual solids (for **4**) are recrystallized from CHCl_3 to give analytically pure **4**. An additional crop is obtained from the mother liquor by column chromatography on silica gel (CH_2Cl_2). The oil (for **5**) is chromatographed on silica gel (hexane– CH_2Cl_2) and then distilled with a Kugelrohr apparatus to give **5**.

2-(Allylthio)-4-(pentafluoroethyl)-4-hydroxy-1(3),4,5,6-tetrahydropyrimidine (4a): Mp $86\text{--}88^\circ\text{C}$; ^1H NMR (acetone- d_6) $\delta=1.78$ (1H, ddd, $J=13.1$, 11.9, and 6.4 Hz, H^5), 1.93 (1H, dm, $J=13.1$ Hz, H^5), 3.40 (2H, m, H^6), 3.59 (1H, ddt, $J=13.7$, 7.0, and 1.0 Hz, allylic H), 3.66 (1H, ddt, $J=13.7$, 7.0, and 1.0 Hz, allylic H), 4.99 (1H, ddt, $J=17.1$, 1.5, and 1.0 Hz, $=\text{CH}_2$), 5.08 (1H, br s, OH or NH), 5.17 (1H, ddt, $J=17.1$, 1.5, and 1.0 Hz, $=\text{CH}_2$), 5.90 (1H, ddt, $J=17.1$, 10.0, and 7.0 Hz, $-\text{CH}=\text{}$), and 6.86 (1H, br s, NH or OH); ^{19}F NMR (acetone- d_6) $\delta=-77.21$ (3F, s), -125.52 (1F, d, $J=271$ Hz), and -125.69 (1F, d, $J=271$ Hz); IR (KBr) 3312 (s), 3000 (m), 1642 (m), 1596 (vs), 1338 (s), 1268 (s), 1224 (vs), 1190 (vs), 1124 (vs), 1022 (s), and 988 cm^{-1} (s).

2-(Allylthio)-4-(nonafluorobutyl)-4-hydroxy-1(3),4,5,6-tetrahydropyrimidine (4b): Mp 94°C ; ^1H NMR (acetone- d_6) $\delta=1.81$ (1H, ddd, $J=13.1$, 12.5, and 6.1 Hz), 1.98 (1H, dm, $J=13.1$ Hz), 3.42 (2H, m), 3.60 (1H, dd, $J=14.0$ and 7.3 Hz), 3.66 (1H, dd, $J=14.0$ and 7.0 Hz), 5.00 (1H, dm, $J=10.0$ Hz), 5.11 (1H, br s), 5.17 (1H, dm, $J=17.1$ Hz), 5.91 (1H, ddt, $J=17.1$, 10.0, and 7.0 Hz), and 6.87 (1H, br s); ^{19}F NMR (acetone- d_6) $\delta=-80.57$ (3F, tt, $J=10$ and 3 Hz), -118.91 (1F, dm, $J=297$ Hz), -119.62 (1F, dm, $J=297$ Hz), -120.99 (1F, dm, $J=275$ Hz), -121.63 (1F, dm, $J=275$ Hz), and -125.57 (2F, m); IR (KBr) 3348 (s), 3100 (br s), 1642 (m), 1600 (vs), 1512 (s), 1342 (s), 1224 (vs), 1122 (vs), and 984 cm^{-1} (s); MS (CI) m/z (rel intensity) 391 (M^++1 , 38), 373 (48), 371 (10), 351 (8), 317 (11), and 275 (100). Found: C, 33.83; H, 2.79; N, 7.20%. Calcd for $\text{C}_{11}\text{H}_{11}\text{N}_2\text{F}_9\text{OS}$: C, 33.85; H, 2.84; N, 7.18%.

2-(Allylthio)-4-(tridecafluorohexyl)-4-hydroxy-1(3),4,5,6-tetrahydropyrimidine (4c): Mp $112\text{--}114^\circ\text{C}$; ^1H NMR (acetone- d_6) $\delta=1.81$ (1H, ddd, $J=13.1$, 12.5, and 6.0 Hz), 1.98 (1H, dm, $J=13.1$ Hz), 3.41 (2H, m), 3.60 (1H, dd, $J=14.0$ and

7.3 Hz), 3.66 (1H, dd, $J=14.0$ and 7.0 Hz), 4.99 (1H, dm, $J=10.0$ Hz), 5.11 (1H, br s), 5.17 (1H, dm, $J=17.1$ Hz), 5.91 (1H, ddt, $J=17.1$, 10.0, and 7.0 Hz), and 6.87 (1H, br s); ^{19}F NMR (acetone- d_6) $\delta=-80.63$ (3F, tt, $J=10$ and 2 Hz), -117.83 (1F, dm, $J=301$ Hz), -118.72 (1F, dm, $J=301$ Hz), -120.9 (1F, m), -121.4 (3F, m), -122.17 (2F, m), and -125.73 (2F, m); IR (KBr) 3320 (s), 3025 (m), 1660 (m), 1570 (vs), 1508 (s), 1350 (s), 1232 (vs), 1206 (vs), 1146 (vs), 1128 (vs), and 984 cm^{-1} (s); MS (CI) m/z (rel intensity) 491 (M^++1 , 24), 473 (37), 471 (8), 417 (9), and 375 (100). Found: C, 31.66; H, 2.21; N, 5.63%. Calcd for $\text{C}_{13}\text{H}_{11}\text{N}_2\text{F}_{13}\text{OS}$: C, 31.85; H, 2.26; N, 5.71%.

2-(Allylthio)-4-(heptadecafluorooctyl)-4-hydroxy-1(3),4,5,6-tetrahydropyrimidine (4d): Mp $124\text{--}125^\circ\text{C}$; ^1H NMR (acetone- d_6) $\delta=1.81$ (1H, td, $J=12.2$ and 6.4 Hz), 1.98 (1H, dm, $J=12.2$ Hz), 3.41 (2H, m), 3.59 (1H, dd, $J=13.6$ and 7.0 Hz), 3.66 (1H, dd, $J=13.6$ and 7.0 Hz), 4.99 (1H, dm, $J=10.0$ Hz), 5.11 (1H, br s), 5.16 (1H, d, $J=17.1$ Hz), 5.91 (1H, ddt, $J=17.1$, 10.0, and 7.0 Hz), and 6.87 (1H, br s); ^{19}F NMR (acetone- d_6) $\delta=-80.60$ (3F, tt, $J=10$ and 2 Hz), -117.82 (1F, dm, $J=307$ Hz), -118.66 (1F, dm, $J=307$ Hz), $-120.5\text{--}121.5$ (8F, m), -122.24 (2F, m), and -125.70 (2F, m); MS (CI) m/z (rel intensity) 591 (M^++1 , 22), 573 (29), 571 (6), 533 (5), 517 (6), and 475 (100); IR (KBr) 3328 (s), 3020 (m), 1650 (m), 1572 (vs), 1506 (s), 1348 (s), 1212 (vs), 1204 (vs), 1152 (vs), 1130 (s), 986 cm^{-1} (s). Found: C, 30.47; H, 1.83; N, 4.76%. Calcd for $\text{C}_{15}\text{H}_{11}\text{N}_2\text{F}_{17}\text{OS}$: C, 30.52; H, 1.88; N, 4.75%.

2-(3-Butenylthio)-4-(heptadecafluorooctyl)-4-hydroxy-1(3),4,5,6-tetrahydropyrimidine (4e): Mp $122\text{--}124^\circ\text{C}$; ^1H NMR (acetone- d_6) $\delta=1.81$ (1H, td, $J=12.5$ and 5.8 Hz), 1.98 (1H, dm, $J=12.5$ Hz), 2.35 (2H, m), 2.85–3.10 (2H, m), 3.40 (2H, m), 4.65 (1H, br s), 4.96 (1H, ddt, $J=10.1$, 2.1, and 1.2 Hz), 5.03 (1H, dq, $J=17.1$ and 2.1 Hz), 5.79 (1H, ddt, $J=17.1$, 10.1, and 6.7 Hz), and 6.75 (1H, br s); ^{19}F NMR (acetone- d_6) $\delta=-80.81$ (3F, tt, $J=10$ and 2 Hz), -117.88 (1F, dm, $J=295$ Hz), -118.74 (1F, dm, $J=295$ Hz), -120.9 (1F, m), $-121.2\text{--}121.6$ (7F, m), -122.41 (2F, m), and -125.89 (2F, m); ^{13}C NMR (acetone- d_6) $\delta=26.71$, 29.96, 35.38, 36.55, 84.15 (t, $J=25$ Hz), 105–125 (8C), 116.39, 138.10, and 159.77; IR (KBr) 3332 (s), 3098 (m), 1646 (m), 1572 (vs), 1506 (s), 1352 (s), 1222 (vs), 1154 (vs), 1130 (vs), and 986 cm^{-1} (s). Found: C, 31.53; H, 2.18; N, 4.78%. Calcd for $\text{C}_{16}\text{H}_{13}\text{N}_2\text{F}_{17}\text{OS}$: C, 31.80; H, 2.17; N, 4.63%.

2-(Ethylthio)-4-(heptadecafluorooctyl)-4-hydroxy-1(3),4,5,6-tetrahydropyrimidine (4f): Mp 127°C ; ^1H NMR (acetone- d_6) $\delta=1.23$ (3H, t, $J=7.3$ Hz), 1.82 (1H, td, $J=12.5$ and 6.1 Hz), 1.97 (1H, dm, $J=12.5$ Hz), 2.80–3.05 (2H, m), 3.41 (2H, m), 5.04 (1H, br s), and 6.97 (1H, br s); ^{19}F NMR (acetone- d_6) $\delta=-80.63$ (3F, tt, $J=10$ and 2 Hz), -117.82 (1F, dm, $J=298$ Hz), -118.80 (1F, dm, $J=298$ Hz), $-120.5\text{--}121.7$ (8F, m), -122.27 (2F, m), and -125.74 (2F, m); IR (KBr) 3336 (s), 3000 (br s), 1572 (vs), 1506 (s), 1350 (s), 1202 (vs), 1152 (vs), and 1130 cm^{-1} (s); MS (CI) m/z (rel intensity) 519 (M^++1 , 24), 561 (43), 559 (9), 541 (8), 539 (5), 517 (6), 475 (100), and 157 (34). Found: C, 28.91; H, 1.82; N, 4.64%. Calcd for $\text{C}_{14}\text{H}_{11}\text{N}_2\text{F}_{17}\text{OS}$: C, 29.08; H, 1.92; N, 4.84%.

4-(Nonafluorobutyl)-4-hydroxy-2-methyl-1(3),4,5,6-tetrahydropyrimidine (4g): Mp 174°C (decomp); ^1H NMR (DMSO- d_6) $\delta=1.58$ (1H, m), 1.74 (1H, m), 1.77 (3H, s), 3.15 (2H, m), 5.79 (1H, br s), and 7.18 (1H, br s); ^{19}F NMR (DMSO- d_6) $\delta=-80.20$ (3F, tt, $J=10$ and 3 Hz), -119.20 (2F, m), -121.37 (2F, m), and -125.09 (2F, t, $J=16$ Hz); ^{13}C NMR (DMSO- d_6) $\delta=21.89$, 24.62, 33.28, 81.41 (t, $J=24$ Hz), 100–125 (4C), and 156.57; IR (KBr) 3316 (s), 3100 (m), 1602 (vs), 1560 (s), 1442 (s), 1356 (s), 1342 (s), 1240 (vs), 1228 (vs), 1200 (vs), 1134 (vs), 812 (s), and 726 cm^{-1} (s); MS (CI) m/z (rel intensity) 333 (M^++1 , 75), 315 (100), 313 (25), 293 (16), 275 (28), and 113 (66). Found: C, 32.54; H, 2.71; N, 8.76%. Calcd

for $C_9H_9N_2F_9O$: C, 32.54; H, 2.73; N, 8.43%.

4-(Nonafluorobutyl)-4-hydroxy-2-phenyl-1(3),4,5,6-tetrahydropyrimidine (4h): Mp 163 °C; 1H NMR (acetone- d_6) δ =1.77 (1H, m), 1.96 (1H, m), 3.47 (2H, m), 5.02 (1H, br s), 7.32 (1H, m), 7.38 (3H, m), and 7.82 (2H, m); ^{19}F NMR (acetone- d_6) δ =-80.56 (3F, tt, J =10 and 3 Hz), -118.37 (1F, dm, J =300 Hz), -120.03 (1F, dm, J =300 Hz), -120.68 (1F, dm, J =277 Hz), -122.23 (1F, dm, J =277 Hz), and -125.49 (2F, m); IR (KBr) 3332 (s), 3072 (m), 2756 (m), 1600 (vs), 1572 (s), 1544 (vs), 1356 (vs), 1220 (vs), 1180 (vs), 1132 (vs), 720 (s), and 710 cm^{-1} (s); MS (EI) m/z (rel intensity) 394 (M^+ , 1), 375 (4), 348 (1), 175 (17), 120 (26), 104 (46), 77 (23), 69 (20), and 55 (100). Found: C, 42.49; H, 2.80; N, 7.39%. Calcd for $C_{14}H_{11}N_2F_9O$: C, 42.65; H, 2.81; N, 7.11%.

2-(Ethylthio)-4-(nonafluorobutyl)-4-hydroxy-5-methyl-1(3),4,5,6-tetrahydropyrimidine (4i): Mp 90–91 °C; (major isomer) 1H NMR ($CDCl_3$) δ =1.10 (3H, dd, J =6.7 and 1.5 Hz), 1.26 (3H, t, J =7.3 Hz), 2.28 (1H, m), 2.48 (1H, br s), 2.93 (1H, m), 2.99 (1H, m), 3.15 (2H, m), and 5.01 (1H, br s); ^{19}F NMR ($CDCl_3$) δ =-81.21 (3F, tt, J =10 Hz and 3 Hz), -117.53 (1F, dm, J =296 Hz), -117.73 (1F, dm, J =296 Hz), -119.78 (1F, dm, J =296 Hz), -122.67 (1F, dm, J =296 Hz), -125.31 (1F, dm, J =292 Hz), and -127.75 (1F, dm, J =292 Hz); IR (KBr) 3340 (s), 2996 (m), 1574 (vs), 1506 (m), 1342 (m), 1226 (vs), 1198 (vs), 1130 (s), 1112 (m), 874 (s), and 746 cm^{-1} (m); MS (EI) m/z (rel intensity) 392 (M^+ , 16), 363 (17), 173 (98), 131 (19), 88 (18), 69 (100), 62 (36), 60 (41), and 55 (67). Found: C, 33.80; H, 3.42; N, 7.51%. Calcd for $C_{11}H_{13}N_2F_9OS$: C, 33.68; H, 3.34; N, 7.14%.

4-(Nonafluorobutyl)-4-hydroxy-2,5-dimethyl-1(3),4,5,6-tetrahydropyrimidine (4j): Mp 158 °C (decomp); (major isomer) 1H NMR (DMSO- d_6) δ =0.93 (3H, d, J =6.1 Hz), 1.76 (3H, s), 1.90 (1H, m), 2.86 (1H, m), 2.99 (1H, m), 5.56 (1H, br s), and 7.17 (1H, br s); ^{19}F NMR (DMSO- d_6) δ =-80.24 (3F, tt, J =10 and 3 Hz), -116.28 (1F, dm, J =277 Hz), -117.75 (1F, dm, J =294 Hz), -118.75 (1F, dm, J =277 Hz), -120.21 (1F, dm, J =294 Hz), -124.11 (1F, dm, J =289 Hz), and -125.72 (1F, dm, J =289 Hz); ^{13}C NMR (DMSO- d_6) δ =12.59, 21.46, 28.80, 40.94, 83.24 (t, J =27 Hz), 100–125 (4C), and 156.00; IR (KBr) 3308 (s), 1606 (vs), 1562 (m), 1354 (m), 1296 (m), 1244 (vs), 1220 (s), 1198 (vs), 1170 (s), 1130 (vs), 806 (m), and 730 cm^{-1} (m); MS (CI) m/z (rel intensity) 347 (M^++1 , 77), 329 (100), 327 (27), 307 (17), and 127 (51). Found: C, 34.64; H, 3.20; N, 8.40%. Calcd for $C_{10}H_{11}N_2F_9O$: C, 34.69; H, 3.20; N,

8.09%.

2-(3-Butenylthio)-4-(tridecafluorohexyl)pyrimidine (5a): Bp 140–143 °C/21 mmHg[#]; 1H NMR ($CDCl_3$) δ =2.52 (2H, m), 3.23 (2H, t, J =7.3 Hz), 5.04–5.17 (2H, m), 5.87 (1H, ddt, J =17.1, 10.4, and 6.7 Hz), 7.28 (1H, d, J =4.9 Hz), and 8.74 (1H, d, J =4.9 Hz); ^{19}F NMR ($CDCl_3$) δ =-81.33 (3F, tt, J =10 and 2 Hz), -116.50 (2F, m), -121.89 (2F, m), -122.21 (2F, m), -123.23 (2F, m), and -126.63 (2F, m); ^{13}C NMR ($CDCl_3$) δ =30.47, 33.13, 100–125 (6C), 113.40 (t, J =4 Hz), 116.48, 136.02, 156.09 (t, J =27 Hz), 159.05, and 174.04. Found: C, 34.45; H, 1.86; N, 5.47%. Calcd for $C_{14}H_9N_2F_{13}S$: C, 34.72; H, 1.87; N, 5.78%.

2-(Ethylthio)-4-(tridecafluorohexyl)pyrimidine (5b): Bp 82–83 °C/0.2 mmHg; 1H NMR ($CDCl_3$) δ =1.41 (3H, t, J =7.3 Hz), 3.18 (2H, q, J =7.3 Hz), 7.28 (1H, d, J =4.9 Hz), and 8.75 (1H, d, J =4.9 Hz); ^{19}F NMR δ =-81.41 (3F, tt, J =10 and 3 Hz), -116.20 (2F, m), -121.64 (2F, m), -122.01 (2F, m), -123.04 (2F, m), and -126.39 (2F, m, J =14.5 Hz); IR (NaCl) 2980 (m), 2936 (m), 1564 (s), 1430 (m), 1352 (s), 1298 (s), 1240 (s), 1210 (s), 1148 (s), 1056 (m), 1030 (m), 840 (m), 712 (m), 662 (m), and 466 cm^{-1} (br s); MS (EI) m/z (rel intensity) 458 (M^+ , 100), 443 (17), 430 (10), 425 (34), 189 (28), 156 (11), 111 (37), 79 (15), 61 (12), 59 (10), and 52 (13). Found: C, 31.39; H, 1.60; N, 6.18%. Calcd for $C_{12}H_7N_2F_{13}S$: C, 31.45; H, 1.54; N, 6.11%.

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[#] 1 mmHg $\approx$ 133.322 Pa.