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## Preparation of *gem*-Difluorinated $\beta$ -Phenylthio Substituted Allylic Bromides and Their Reactions

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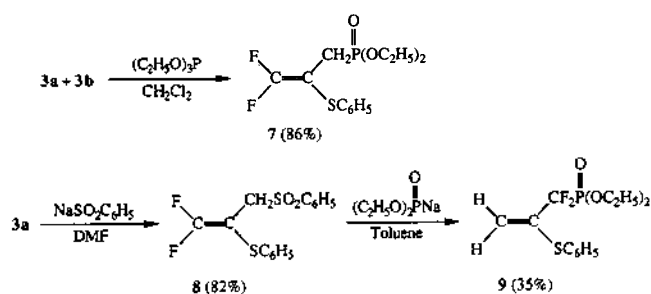
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The introduction of difluoromethylene (CF<sub>2</sub>) unit into organic molecules has recently been received much attention because of enhancement of biological properties of pharmaceuticals and agrochemicals.<sup>1,2</sup> A variety of biologically interesting compounds that contain the difluoromethylene group, such as the antitumor nucleoside Gemcitabine<sup>3</sup> and  $\alpha,\alpha$ -difluoroalkylphosphonate-based mimics,<sup>4</sup> have been discovered in recent work. Although there have been various methods for the introduction of difluoromethylene functionality, the synthetic methods which the difluoromethylene phosphonate group is directly attached to an vinyl carbon atom have been quite limited.<sup>5-8</sup> *gem*-Difluoroallylation is one of valuable methods for the construction of difluoromethylene frameworks because of a wide range of functional group transformations of alkene group. The most potential reagent for *gem*-difluoroallylation is *gem*-difluoroallylic bromide. The synthetic method for the 3-bromo-3,3-difluoropropene as a *gem*-difluoroallylic bromide has been well known.<sup>9</sup> However, we are interested in the preparation of *gem*-difluorinated  $\beta$ -phenylthio substituted allylic bro-

mides because the presence of phenylthio group at the vinyl carbon could provide more versatility for the functional group transformation than in the case of the presence of alkene group only. Unfortunately, there has been no methodology for the preparation of *gem*-difluorinated  $\beta$ -phenylthio substituted allylic bromides. In this paper, we wish to describe a new synthetic method for the preparation of *gem*-difluorinated  $\beta$ -phenylthio substituted allylic bromides and their reactions.

Perfluorinated dithioketals which we have developed<sup>10</sup> are promising reagents to approach *gem*-difluorinated  $\beta$ -phenylthio substituted allylic bromides. Thus, the starting materials, 1,1,1-trifluoro-2,2-bis(phenylthio)propane (**1a**) and 1,1,1-trifluoro-2,2-bis(phenylthio)butane (**1b**), were prepared in 82% and 79% isolated yields, respectively, from the reaction of 1,1,1-trifluoro-2-propanone and 1,1,1-trifluoro-2-butanone with thiophenol in the presence of AlCl<sub>3</sub> at -78 °C for 20 hours. The treatment of **1a** and **1b** with a mixture of 2 equiv. of TiCl<sub>4</sub> and 3 equiv. of LiAlH<sub>4</sub> in THF at reflux temperature for 3 hours resulted in the formation of 1,1-difluoro-2-





### Experimental

**General.**  $^1\text{H}$  NMR spectra were recorded on a 100 MHz Bruker AC-100F NMR Spectrometer with tetramethylsilane (TMS) as an internal standard.  $^{19}\text{F}$  NMR spectra were recorded on a 100 MHz Bruker AC-100F NMR spectrometer.  $\text{CFCl}_3$  was used as an internal standard and chemical shifts are reported in parts per million. Infrared spectra were determined on a Mattson Genesis series FT High Resolution Spectrophotometer. Mass spectra were obtained Hewlett-Packard 5890 GC/5970B MSD (EI, 70 eV).

Commercially available reagents were purchased from Aldrich, PCR and Tokyo Kasei. All solvents were dried by general purification methods.

**Preparation of 2,2-Bis(phenylthio)-1,1,1-trifluoropropane (1a).** A mixture of 1,1,1-trifluoroacetone (2.24 g, 0.02 mol), thiophenol (4.40 g, 0.04 mol) and 200 mL of dry  $\text{CH}_2\text{Cl}_2$  was cooled to  $-78^\circ\text{C}$  by using dry-ice/isopropanol slush and then  $\text{AlCl}_3$  was added in several portions via a solid addition tube. After stirring at  $-78^\circ\text{C}$  for 20 hours, the reaction mixture was quenched with water at  $-78^\circ\text{C}$ . The mixture was poured into 150 mL of water, extracted with  $\text{CH}_2\text{Cl}_2$  (300 mL  $\times$  2). After washing with saturated NaCl water solution,  $\text{CH}_2\text{Cl}_2$  layer was dried with anhydrous  $\text{Na}_2\text{CO}_3$ . Column chromatography (hexane) provided 5.15 g (82% yield) of **1a**: oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.69-7.27 (m, 10H), 1.38 (s, 3H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -71.60 (s, 3F); MS,  $m/z$  (relative intensity) 314 ( $M^+$ , 5), 205(100), 165(37), 109(25), 77(12), 51(7); IR (neat) 3060, 2994, 1574, 1476, 1449, 1384, 1253, 1181, 756,  $690\text{ cm}^{-1}$ .

**Preparation of 2,2-Bis(phenylthio)-1,1,1-trifluorobutane (1b).** After the reaction of a solution of 1,1,1-trifluoro-2-butanone (2.52 g, 0.02 mol), thiophenol (4.40 g, 0.04 mol) and 200 mL of dry  $\text{CH}_2\text{Cl}_2$  with  $\text{AlCl}_3$  (2.67 g, 0.02 mol) according to the procedure for the preparation of **1a**, column chromatography (hexane) provided 5.18 g (79% yield) of **1b**: oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.68-7.24 (m, 10H), 1.86 (q,  $J=7.4$  Hz, 2H), 1.24 (t,  $J=7.4$  Hz, 3H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -66.40 (s, 3F); MS,  $m/z$  (relative intensity) 328 ( $M^+$ , 7), 219(85), 179(7), 141(5), 109 (100), 77(5), 65(11); IR (neat) 3060, 2980, 2940, 1582, 1473, 1439, 1249, 1253, 1181, 1102, 750,  $690\text{ cm}^{-1}$ .

**Preparation of 1,1-difluoro-2-phenylthiopropene (2a).** A mixture of  $\text{TiCl}_4$  (3.80 g, 20 mmol) and  $\text{LiAlH}_4$  (1.14 g, 30 mmol) in dry THF (100 mL) was stirred at room temperature for 1 hour and then heated to boiling. 2,2-Bis(phenylthio)-1,1,1-trifluoropropane (3.14 g, 10 mmol) in THF (5 mL) was added under reflux and the reaction mixture was kept boiling for further 3 hours. After cooling,

the reaction mixture was poured on ice water, neutralized with conc. HCl and extracted with ether. The ether solution was dried and then chromatographed (hexane) to provide 1.45 g (78% yield) of **2a**: oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.33-7.17 (m, 5H), 1.83 (t,  $J=3.5$  Hz, 3H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -82.63 (d,  $J=28.5$  Hz, 1F), -83.06 (d,  $J=28.5$  Hz, 1F); MS,  $m/z$  (relative intensity) 186 ( $M^+$ , 100), 165(30), 127(65), 121(16), 109(24), 77(35), 65(26), 59(81), 51(49), 29(16); IR (neat) 3067, 2929, 1718, 1587, 1483, 1443, 1267, 1109, 1031, 913, 743,  $691\text{ cm}^{-1}$ .

**Preparation of 1,1-difluoro-2-phenylthio-1-butene (2b).** After the reaction of 2,2-bis(phenylthio)-1,1,1-trifluorobutane (3.28 g, 10 mmol) with a mixture of  $\text{TiCl}_4$  (3.80 g, 20 mmol) and  $\text{LiAlH}_4$  (1.14 g, 30 mmol) in dry THF (100 mL) according to the procedure for the preparation of **2a**, column chromatography (hexane) provided 1.50 g (75% yield) of **2b**: oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.33-7.17 (m, 5H), 2.33-2.06 (m, 2H), 1.06 (t,  $J=7.4$  Hz, 3H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -82.31 (d,  $J=28.7$  Hz, 1F), -82.88 (d,  $J=28.7$  Hz, 1F); MS,  $m/z$  (relative intensity) 200 ( $M^+$ , 97), 171(12), 165(27), 127(19), 110(23), 91(13), 73(100), 65(13), 51(16); IR (neat) 3075, 2973, 2934, 1711, 1583, 1477, 1440, 1259, 1117, 964, 739,  $690\text{ cm}^{-1}$ .

**Preparation of 3-bromo-3,3-difluoro-2-phenylthiopropene (3a) and 3-bromo-1,1-difluoro-2-phenylthiopropene (3b).** A mixture of 1,1-difluoro-2-phenylthiopropene (0.93 g, 5 mmol), N-bromosuccinimide (1.07 g, 6 mmol), 10 mol% AIBN and  $\text{CH}_3\text{CN}$  (50 mL) was heated to reflux for 3 hours. After cooling and evaporation of solvent, the reaction mixture was chromatographed (hexane) to provide 1.09 g (82% yield) of **3a** and **3b**: oil. **3a**:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.58-7.26 (m, 5H), 6.00 (m, 1H), 5.24 (m, 1H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -46.54 (s, 2F); MS,  $m/z$  (relative intensity) 266 ( $M^++2$ , 21), 264 ( $M^+$ , 21), 185(44), 165(100), 135(22), 109(40), 91(23), 77(35), 65(38), 51(24); IR (neat) 3062, 1702, 1606, 1583, 1478, 1384, 1228, 1085, 920, 783, 747,  $691\text{ cm}^{-1}$ . **3b**:  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.82-7.24 (m, 5H), 4.06-3.99 (m, 2H);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -43.31 (s, 1F), -43.79 (s, 1F); MS,  $m/z$  (relative intensity) 266 ( $M^++2$ , 31), 264 ( $M^+$ , 30), 185(54), 165(100), 135(18), 109(34), 91(16), 65(20); IR (neat) 3059, 1700, 1578, 1476, 1439, 1286, 1247, 1153, 1067, 967, 867, 742,  $689\text{ cm}^{-1}$ .

**Preparation of 1-bromo-1,1-difluoro-2-phenylthio-2-butene (3c).** A mixture of 1,1-difluoro-2-phenylthio-1-butene (1.00 g, 5 mmol), N-bromosuccinimide (1.07 g, 6 mmol), 10 mol% AIBN and  $\text{CH}_3\text{CN}$  (50 mL) was heated to reflux for 3 hours. After cooling and evaporation of solvent, the reaction mixture was chromatographed (hexane) to provide 1.12 g (86% yield) of **3c** ( $E:Z=4:1$ ): oil;  $^1\text{H}$  NMR ( $\text{CDCl}_3$ )  $\delta$  7.33-7.15 (m, 10H,  $E$  and  $Z$  isomer), 7.04 (q,  $J=6.6$  Hz, 1H,  $E$ -isomer), 6.44 (q,  $J=7.6$  Hz, 1H,  $Z$ -isomer), 2.11-1.84 (m, 6H,  $E$  and  $Z$  isomer);  $^{19}\text{F}$  NMR ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -39.58 (s, 2F,  $Z$  isomer), -45.72 (s, 2F,  $E$  isomer); MS,  $m/z$  (relative intensity) 280 ( $M^++2$ , 45), 278 ( $M^+$ , 43), 199(38), 179(100), 146(28), 109(34), 77(9), 65(15); IR (neat) 3075, 2923, 2847, 1626, 1583, 1479, 1440, 1216, 1136, 1094, 992, 944, 799, 739,  $689\text{ cm}^{-1}$ .

**Preparation of 3,3-difluoro-3-trimethylsilyl-2-phenylthiopropene (5).**  $n\text{-BuLi}$  (2.5 M solution in hexane) (1.2 mL, 3.0 mmol) was added to a solution of a mix-

ture of 3-bromo-3,3-difluoro-2-phenylthiopropene and 3-bromo-1,1-difluoro-2-phenylthiopropene (0.264 g, 1.0 mmol),  $(\text{CH}_3)_2\text{SiCl}_2$  (0.162 g, 1.5 mmol) and TMEDA (0.116 g, 1.0 mmol) in 10 mL of mixed solvent (pentane:ether:THF=1:1:4) at  $-90^\circ\text{C}$  dropwise and then the reaction mixture was stirred at  $-90^\circ\text{C}$  for 3 hours. After quenching with saturated NaCl solution, extraction with ether and drying with anhy.  $\text{MgSO}_4$ , the reaction mixture was chromatographed (hexane) to provide 0.106 g (41% yield) of **5**: oil;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$  7.43-7.16 (m, 5H), 5.43 (m, 1H), 4.82 (s, 1H), 0.20 (s, 9H);  $^{19}\text{F NMR}$  ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -109.36 (s, 2F); MS,  $m/z$  (relative intensity) 258 ( $M^+$ , 9), 182(30), 165(34), 147(13), 73(100), 45(16); IR (neat) 3076, 2960, 1644, 1583, 1478, 1440, 1263, 1081, 1015, 944, 799, 741, 691  $\text{cm}^{-1}$ .

**Preparation of 2,2-difluoro-1-phenyl-2-phenylthio-3-buten-1-ol (6).** A solution of a mixture of 3-bromo-3,3-difluoro-2-phenylthiopropene and 3-bromo-1,1-difluoro-2-phenylthiopropene (0.264 g, 1.0 mmol),  $\text{Zn}(\text{Cu})$  (0.26 g, 4.0 mmol) and benzaldehyde (0.127 g, 1.2 mmol) in dry THF (5 mL) was heated to reflux for 3 hours. After cooling and quenching with 5% HCl solution, extraction with ether and drying with anhy.  $\text{MgSO}_4$ , the reaction mixture was chromatographed (ethyl acetate:n-hexane=1:4) to provide 0.190 g (65% yield) of **6**: oil;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$  7.42-7.18 (m, 5H), 5.57 (s, 1H), 5.26-5.05 (m, 2H), 2.47 (d,  $J=4.1$  Hz, 1H);  $^{19}\text{F NMR}$  ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -107.75 (d,  $J=10.4$  Hz, 1F), -108.05 (d,  $J=11.4$  Hz, 1F); MS,  $m/z$  (relative intensity) 292 ( $M^+$ , 34), 186(48), 107(100), 79(65), 77(39); IR (neat) 3442, 3076, 1715, 1593, 1480, 1454, 1445, 1393, 1262, 1087, 799, 741, 691  $\text{cm}^{-1}$ .

**Preparation of 3-diethylphosphonyl-1,1-difluoro-2-phenylthiopropene (7).** A solution of a mixture of 3-bromo-3,3-difluoro-2-phenylthiopropene and 3-bromo-1,1-difluoro-2-phenylthiopropene (0.264 g, 1.0 mmol) and triethylphosphite (0.249 g, 1.5 mmol) in dry  $\text{CH}_2\text{Cl}_2$  (5 mL) was stirred at room temperature for 50 hours. After quenching with saturated NaCl solution, extraction with  $\text{CH}_2\text{Cl}_2$  and drying with anhy.  $\text{MgSO}_4$ , the reaction mixture was chromatographed (ethyl acetate:n-hexane=1:1) to provide 0.277 g (86% yield) of **7**: oil;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$  7.32-7.22 (m, 5H), 4.12 (dq,  $J=7.4, 7.2$  Hz, 4H), 2.70 (dt,  $J=20.0, 1.8$  Hz, 2H), 1.32 (t,  $J=7.2$  Hz, 6H);  $^{19}\text{F NMR}$  ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -77.97 (t,  $J=13.8$  Hz, 1F), -78.70 (t,  $J=16.3$  Hz, 1F); MS,  $m/z$  (relative intensity) 322 ( $M^+$ , 50), 182(26), 165(100), 135(32), 109(38), 81(32), 65(26), 29(21); IR (neat) 3058, 2983, 2908, 1711, 1583, 1478, 1440, 1392, 1271, 1028, 823, 744, 691  $\text{cm}^{-1}$ .

**Preparation of 1,1-difluoro-3-phenylsulfonyl-2-phenylthiopropene (8).** A solution of 3-bromo-3,3-difluoro-2-phenylthiopropene (0.264 g, 1.0 mmol) and sodium benzenesulfinate (0.246 g, 1.5 mmol) in dry DMF (15 mL) was stirred at room temperature for 12 hours. After quenching with saturated NaCl solution, extraction with ether and drying with anhy.  $\text{MgSO}_4$ , the reaction mixture was chromatographed (ethyl acetate:n-hexane=1:4) to provide 0.267 g (82% yield) of **8**: oil;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$  7.94-7.20 (m, 10H), 3.85 (t,  $J=2.1$  Hz, 2H);  $^{19}\text{F NMR}$  ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -74.24 (s, 1F), -74.47 (s, 1F); MS,  $m/z$

(relative intensity) 326 ( $M^+$ , 40), 182(54), 165(100), 135(87), 109(26), 77(25); IR (neat) 3064, 2927, 1704, 1584, 1478, 1328, 1289, 1167, 1086, 1011, 689  $\text{cm}^{-1}$ .

**Preparation of 3-diethylphosphonyl-3,3-difluoro-2-phenylthiopropene (9).** Diethyl phosphite (0.138 g, 1.0 mmol) was added to a solution of sodium (0.034 g, 1.5 mmol) in toluene (3 mL) and then the reaction mixture was stirred at room temperature for 0.5 hour. 1,1-Difluoro-3-phenylsulfonyl-2-phenylthiopropene (0.228 g, 0.7 mmol) in toluene (1 mL) was added to the reaction mixture and then the reaction mixture was stirred at room temperature for 24 hours. After quenching with saturated NaCl solution, extraction with  $\text{CH}_2\text{Cl}_2$  and drying with anhy.  $\text{MgSO}_4$ , the reaction mixture was chromatographed (ethyl acetate:n-hexane=1:1) to provide 0.079 g (35% yield) of **9**: oil;  $^1\text{H NMR}$  ( $\text{CDCl}_3$ )  $\delta$  7.51-7.30 (m, 5H), 5.96 (m, 1H), 5.26 (m, 1H), 4.31 (dq,  $J=7.4, 7.2$  Hz, 4H), 1.31 (t,  $J=7.2$  Hz, 6H);  $^{19}\text{F NMR}$  ( $\text{CDCl}_3$ , internal standard  $\text{CFCl}_3$ )  $\delta$  -105.62 (d,  $J=111.4$  Hz, 2F); MS,  $m/z$  (relative intensity) 322 ( $M^+$ , 87), 273(12), 246(18), 184(100), 165(67), 134(14), 109(61), 91(30), 81(39), 77(31), 29(15).

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