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General Method for the Preparation of β,β-Difluoroacrylates Using BrF_3

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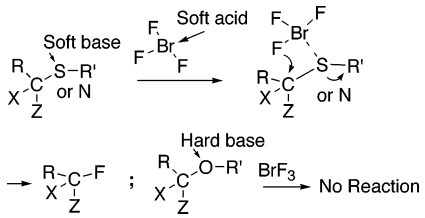
Various esters were reacted with base, carbon disulfide, and methyl iodide, producing 2-carboalkoxy-1,1-bis(methyl sulfide)-1-alkenes (**2**). The reaction of **2** with BrF_3, followed by oxidation with HOF·CH_3CN gave the bromodifluorosulfonyl derivatives **5**. Subsequent treatment with Raney nickel led to α-substituted β,β-difluoroacrylates **6** in overall yields of 50−80%.

Two families of compounds, acrylic esters and fluorine-containing molecules, command a high interest in organic chemistry and in material science. Combining these two subjects is of potential interest for making precursors for compounds used in coating, polymerization, special optical properties, and more.[1] However, the only known methods of preparing β,β-difluoroacrylates are the reactions of malonic esters with CF_2Br_2[2] and reactions of Grignard reagents with 2-(trifluoromethyl)propenoic acid.[3]

The incorporation of a CF_2 group by fluorodesulfuration using specialized dithianylium salts or various [BrF] reagents such as DBH/HF/Py has been recorded in the literature.[4] Bromine trifluoride, which is commercially available, has been known in the literature for more then 60 years, but for most of this period it was in a stage of hibernation where organic synthesis was concerned.[5] In the past decade though, we have started exploring its synthetic potential. It was revealed that it can serve as a valuable tool for constructing the CF_2[6] and the CF_3[7] groups attached to specific sites, for bromination of highly deactivated aromatic rings,[8] and more.[9] Most of these reactions proceeded through a complexation between the soft acidic electrophilic bromine of the reagent and soft basic heteroatoms such as sulfur or nitrogen of the reactant. Such complexation brings the nucleophilic fluorides to the vicinity of the electrophilic carbon, leading to the desired fluorinated products (Scheme 1).

SCHEME 1. Docking of BrF_3 with Soft Bases Resulting in Ionic Reactions



Recently, we prepared α-trifluoromethyl esters by reacting BrF_3 with 2-carboalkoxy-1,1-bis(methyl sulfide)-1-alkenes (**2**), easily obtained from carboxylic acids, CS_2, and MeI.[10] The reaction resulted immediately in a mixture of bromodifluorosulfides (**3**), sulfoxides (**4**), and traces of the sulfones (**5**), which was then oxidized in a fast and quantitative reaction by HOF·CH_3CN,[11] forming the bromodifluorosulfonyl derivatives **5**. We report now that these intermediates can serve as a good starting point for preparing the desired alkyl β,β-difluoroacrylates (**6**).

Results and Discussion

Reacting methyl heptanoate **1a** with LDA, carbon disulfide, and methyl iodide produced 2-carbomethoxy-

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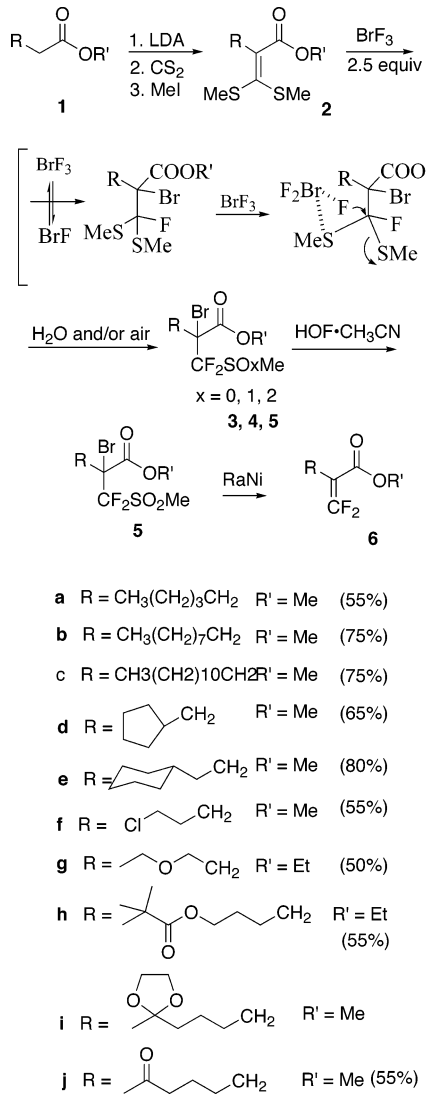
SCHEME 2. Preparation of β,β-Difluoroacrylates



1,1-bis(methyl sulfide)hept-1-ene (**2a**) in good yield. Bringing it in contact with 2.5 molar equiv of bromine trifluoride for a few seconds gave a mixture of the corresponding bromodifluoro sulfide **3a**, sulfoxide **4a**, and sulfone **5a**. The oxidation of this mixture with HOF·CH_3CN resulted in a single compound that proved to be the sulfone **5a**. Treating this sulfone with Raney nickel (RaNi) led to the desired methyl 2-pentyl-β,β-difluoroacrylate (**6a**) in 55% overall yield (Scheme 2). In general, longer alkyl chains are more sensitive to side reactions with BrF_3. However, when methyl undecanoate **1b** and methyl tetradecanoate **1c** served as starting materials, methyl 2-nonanyl-β,β-difluoroacrylate (**6b**) and methyl 2-dodecyl-β,β-difluoroacrylate (**6c**) were ultimately obtained in 75% overall yield each. Such relatively clean reaction indicates that the complexation of bromine trifluoride with the sulfur atoms is faster and more dominant then the indiscriminating radical destruction of organic molecules by a noncomplexed BrF_3 (see warning at the beginning of Experimental Section).

The fluorine atom in bromine trifluoride, can act, in certain cases, as an electrophile capable of replacing tertiary hydrogens[12] in a similar manner as F_2.[13] Again, however, we found that the bis(methyl sulfide) moiety reacts much faster and 2-carbomethoxy-3-cyclopentyl-1,1-bis(methyl sulfide)prop-1-ene (**2d**) and 2-carbomethoxy-4-cyclohexyl-1,1-bis(methyl sulfide)but-1-ene (**2e**), both of which possess tertiary hydrogens, gave after a short treatment with BrF_3, HOF·CH_3CN, and RaNi the desired methyl 2-cyclopentylmethylene-β,β-difluoroacrylate (**6d**) and methyl 4-cyclohexylethylene-β,β-difluoroacrylate (**6e**) in 65% and 80% overall yields, respectively.

Compounds with aromatic rings, which are not extremely deactivated toward electrophilic reactions, are not suitable substrates for this process because of their fast reaction with BrF_3.[8] However, other functional groups, such halogens, carbonyls, or hydroxyls, are tolerated. Thus, 2-carbomethoxy-5-chloro-1,1-bis(methyl sulfide)pent-1-ene (**2f**) was converted to methyl 2-(4-chlorobutyl)-β,β-difluoroacrylate **(6f)** in 55% yield despite the fact that in certain cases BrF_3 is known to substitute chlorine with fluorine.[5] Free alcohols are easily oxidized with BrF_3,[9a] but protecting them as either ethyl ether **2g** or pivaloyl ester **2h** led to ethyl 2-(2-ethoxyethyl)-β,β-difluoroacrylate (**6g**) and ethyl 2-(4-pivaloyloxybutyl)-β,β-difluoroacrylate (**6h**) in 50% and 55% overall yields. If an additional carbonyl group is present in the molecule as in methyl 2-oxo-octanoate (**1j**), it has to be protected as a ketal (**1i)** in order to prevent undesirable reactions by the strong base, CS_2 and MeI. After the formation of **2i** the acetal group was removed and the resulting 2-carbomethoxy-1,1-bis(methyl sulfide)oct-7-oxo-1-ene (**2j**) was reacted with BrF_3 followed by oxidation to produce **5j**. Treating the latter with RaNi furnished methyl 2-(hexa-5-one)-β,β-difluoroacrylate (**6j**) in 55% overall yield.

In conclusion, reactions with bromine trifluoride should be considered in many instances when the introduction of a fluorine atom in organic molecules is the issue. By using proper conditions it can be instrumental in turning easily available carboxylic acids into the potentially important and hard to prepare β,β-difluoroacrylates.

Experimental Section

^{1}H NMR spectra were recorded using a 200 MHz spectrometer with $CDCl_3$ as a solvent and Me_4Si as an internal standard. Only the relevant and characteristic peaks are reported. The ^{19}F NMR spectra were measured at 188.1 MHz and are reported upfield from $CFCl_3$, serving as an internal standard. The proton broad-band decoupled ^{13}C NMR spectra were recorded at 50.2 MHz. Here too, $CDCl_3$ served as a solvent and Me_4Si as an internal standard. IR spectra were recorded in $CHCl_3$ solution on a FTIR spectrophotometer. Silica gel 60H (Merck) and petrol ether/ethyl acetate were used in the flash chromatography when the purification of the product was desired.

Preparing and Handling of BrF_3. Although commercially available, we usually prepare our own BrF_3 by passing 0.6 mol of fluorine through 0.2 mol of bromine placed in a copper reactor and cooled to 0−10 °C. The product can be stored in Teflon containers indefinitely. *BrF_3 is a strong oxidizer and tends to react very exothermically with water and oxygenated organic solvents such as acetone or THF. Alkanes such as petrol ether cannot serve as solvents either, because they react quickly with BrF_3. Any work using BrF_3 should be conducted in a well-ventilated area, and caution and common sense should be exercised.*

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General Procedure for Preparation of 2-Carboalkoxy-1,1-bis(methyl sulfide)-1-alkenes (2).[10] A 10 mmol portion of an ester (**1**) was dissolved in 100 mL of dry THF and cooled to −78 °C, and 12.5 mmol of LDA (1.5 M in cyclohexane) was added. The reaction was stirred for 2 h with temperature maintained below −30 °C and cooled again to −78 °C, and carbon disulfide (about 40 mmol) was added. A brown solution formed and was stirred for an additional 1 h and again the reaction was not allowed to warm above −30 °C. It was cooled once more to −78 °C, and methyl iodide (about 40 mmol) was added and stirred for an additional 2 h. The reaction mixture was warmed to room temperature, poured into water, extracted with ether, and dried over $MgSO_4$, and the organic solvent was evaporated. The product was isolated by flash chromatography as a brown oil with yields of 85−90%.

General Procedure for Preparation of Alkyl 2-Alkyl-β,β-difluoroacrylates (6). The appropriate 2-carboalkoxy-1,1-bis(methyl sulfide)-1-alkene (**2**) derivative (usually 1 mmol) was dissolved in 10−15 mL of dry $CFCl_3$. About 2.5 mmol of BrF_3 was dissolved in 10 mL of the same solvent, cooled to 0 °C, and added dropwise for about 1 min. The reaction was quenched with saturated aqueous Na_2SO_3 and washed until colorless. The aqueous layer was extracted with CH_2Cl_2, and the organic layer was dried over $MgSO_4$. The oily crude that contained mainly the bromodifluoromethyl sulfide **3** and sulfoxide **4**[10] was dissolved in 15 mL of $CHCl_3$ and oxidized with a 3-fold excess of $HOF{\cdot}CH_3CN$[11] in 0 °C for 20 min. The reaction was terminated with $NaHCO_3$ solution, extracted with CH_2Cl_2, and dried over $MgSO_4$. After evaporation of the solvent, the reaction mixture contained mainly the corresponding bromodifluoromethylsulfonyl derivative (**5**), which if desired could be isolated by flash chromatography.[10] The crude **5** (300 mg) was dissolved in 15 mL of THF, and 3 g of RaNi (Aldrich, 22,167−8) was added. The reaction mixture was stirred for 30 min at room temperature and filtered through silica. The product **6** was isolated by flash chromatography in 50−80% overall yields. Compounds **6** possess coupling constant $^2J_{F,F}$ of 0−1 Hz, and the coupling constant represent the $^4J_{F,H}$. This was reported early[14] and we observed another small coupling constant (represented by - m), probably due to the other $^4J_{F,H}$ or the small $^2J_{F,F}$ constant.

Methyl 2-Pentyl-β,β-difluoroacrylate (6a). Prepared from **2a** as described above in 55% overall yield: oil; IR 1718 cm^{-1}; ^1H NMR 3.78 (3 H, s), 2.31−2.05 (2 H, m), 0.90 ppm (3 H, t, J = 7 Hz); ^{19}F NMR −70.1 (1 F, dm, J = 3 Hz), −75.0 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 167.3 (dd, J_1 = 13 Hz, J_2 = 6 Hz), 161.7 (dd, J_1 = 310 Hz, J_2 = 295 Hz), 90.6 (dd, J_1 = 23 Hz, J_2 = 5 Hz), 53.9, 33.1, 30.1, 26.3, 24.2, 15.8 ppm; MS (CI) (*m/z*) 193 $(MH)^+$. It should be noted that we were unable to run a microanalysis on this compound due to its volatility.

Methyl 2-Nonanyl-β,β-difluoroacrylate (6b). Prepared from **2b** as described above in 75% overall yield: oil; IR 1713 cm^{-1}; ^1H NMR 3.78 (3 H, s), 2.28−2.10 (2 H, m), 0.89 ppm (3 H, t, J = 7 Hz); ^{19}F NMR −70.1 (1 F, dm, J = 3 Hz), −75.0 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 165.3 (dd, J_1 = 14 Hz, J_2 = 7 Hz), 159.7 (dd, J_1 = 310 Hz, J_2 = 295 Hz), 88.6 (dd, J_1 = 24 Hz, J_2 = 5 Hz), 51.8, 31.7, 29.4, 29.2, 29.1, 28.9, 28.4, 24.4, 22.5, 13.9 ppm; HRMS (CI) (*m/z*) calcd for $C_{13}H_{23}F_2O_2$ 249.1666 $(MH)^+$, found 249.1665. Anal. Calcd for $C_{13}H_{22}F_2O_2$: C, 62.88; H, 8.93; F, 15.30. Found: C, 61.94; H, 8.88; F, 15.35.

Methyl 2-Dodecyl-β,β-difluoroacrylate (6c). Prepared from **2c** as described above in 75% overall yield: oil; IR 1715 cm^{-1}; ^1H NMR 3.78 (3 H, s), 2.28−2.15 (2 H, m), 0.90 ppm (3 H, t, J = 7 Hz); ^{19}F NMR −70.2 (1 F, dm, J = 3 Hz), −75.0 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 165.3 (dd, J_1 = 14 Hz, J_2 = 8 Hz), 159.7 (dd, J_1 = 310 Hz, J_2 = 295 Hz), 88.6 (dd, J_1 = 24 Hz, J_2 = 5 Hz), 51.8, 31.8, 29.5, 29.4, 29.3, 29.2, 28.9, 28.4, 24.9, 22.6, 13.9 ppm; HRMS (CI) (*m/z*) calcd for $C_{16}H_{29}F_2O_2$ 291.2135 $(MH)^+$, found 291.2138. Anal. Calcd for $C_{16}H_{28}F_2O_2$: C, 66.18; H, 9.72; F, 13.08. Found: C, 66.13; H, 9.69; F, 13.49.

Methyl 2-Cyclopentylmethylene-β,β-difluoroacrylate (6d). Prepared from **2d** as described above in 65% overall yield: oil; IR 1715 cm^{-1}; ^1H NMR 3.78 (3 H, s), 2.26−2.21 ppm (2 H, m); ^{19}F NMR −70.4 (1 F, dm, J = 3 Hz), −74.5 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 165.5 (dd, J_1 = 13 Hz, J_2 = 7 Hz), 159.9 (dd, J_1 = 310 Hz, J_2 = 295 Hz), 88.3 (dd, J_1 = 24 Hz, J_2 = 5 Hz), 51.9, 39.2, 31.9, 29.8, 24.6 ppm; HRMS (CI) (*m/z*) calcd for $C_{10}H_{15}F_2O_2$ 205.1040 $(MH)^+$, found 205.1036. Anal. Calcd for $C_{10}H_{14}F_2O_2$: C, 58.82; H, 6.91. Found: C, 58.42; H, 6.99.

Methyl 4-Cyclohexylethylene-β,β-difluoroacrylate (6e). Prepared from **2e** as described above in 80% overall yield: oil; IR 1713 cm^{-1}; ^1H NMR 3.78 (3 H, s), 2.28−2.16 ppm (2 H, m); ^{19}F NMR −70.2 (1 F, dm, J = 3 Hz), −75.2 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 165.3 (dd, J_1 = 14 Hz, J_2 = 7 Hz), 159.5 (dd, J_1 = 308 Hz, J_2 = 293 Hz), 89.0 (dd, J_1 = 24 Hz, J_2 = 5 Hz), 51.9, 37.1, 36.1, 32.9, 26.5, 26.1, 21.9 ppm; HRMS (CI) (*m/z*) calcd for $C_{12}H_{19}F_2O_2$ 233.1353 $(MH)^+$, found 233.1360. Anal. Calcd for $C_{12}H_{18}F_2O_2$: C, 62.05; H, 7.81. Found: C, 61.70; H, 7.92.

Methyl 2-(4-Chlorobutyl)-β,β-difluoroacrylate (6f). Prepared from **2f** as described above in 55% overall yield: oil; IR 1716 cm^{-1}; ^1H NMR 3.80 (3 H, s), 3.54 (2 H, t, J = 6 Hz), 2.51−2.36 ppm (2H, m); ^{19}F NMR −68.4 (1 F, dm, J = 3 Hz), −73.3 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 165.0 (dd, J_1 = 13 Hz, J_2 = 7 Hz), 160.0 (dd, J_1 = 310 Hz, J_2 = 296 Hz), 87.7 (dd, J_1 = 23 Hz, J_2 = 6 Hz), 54.1, 45.8, 33.2, 24.0 ppm; HRMS (CI) (*m/z*) calcd for $C_7H_{10}ClF_2O_2$ 199.0337 $(MH)^+$, found 199.0338. Anal. Calcd for $C_7H_9ClF_2O_2$: C, 42.33; H, 4.57; Cl,17.85. Found: C, 41.91; H, 4.40; Cl, 18.25.

Ethyl 2-(2-Ethoxyethyl)-β,β-difluoroacrylate (6g). Prepared from **2g** as described above in 50% overall yield: oil; IR 1718 cm^{-1}; ^1H NMR 4.24 (2 H, q, J = 7 Hz), 3.53−3.42 (4 H, m), 2.55−2.45 (2 H, m), 1.30 (3 H, t, J = 7 Hz), 1.17 ppm (3 H, t, J = 7 Hz); ^{19}F NMR −68.5 (1 F, dm, J = 3 Hz), −73.7 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 164.7 (dd, J_1 = 13 Hz, J_2 = 8 Hz), 160.2 (dd, J_1 = 310 Hz, J_2 = 295 Hz), 85.8 (dd, J_1 = 23 Hz, J_2 = 7 Hz), 68.2, 66.0, 61.0, 25.0, 15.0, 14.0 ppm; MS (CI) *m/z* 209 $(MH)^+$. It should be noted that we were unable to run a microanalysis on this compound due to its volatility.

Ethyl 2-(4-Pivaloyloxybutyl)-β,β-difluoroacrylate (6h). Prepared from **2h** as described above in 55% overall yield: oil; IR 1714 and 1728 cm^{-1}; ^1H NMR 4.23 (2 H, q, J = 7 Hz), 4.06 (2 H, t, J = 7 Hz), 2.31−2.11 (2 H, m), 1.29 (3 H, t, J = 7 Hz), 1.19 ppm (9 H, s); ^{19}F NMR −69.7 (1 F, dm, J = 3 Hz), −74.7 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 178.3, 164.4 (dd, J_1 = 14 Hz, J_2 = 8 Hz), 159.5 (dd, J_1 = 310 Hz, J_2 = 295 Hz), 88.4 (dd, J_1 = 23 Hz, J_2 = 5 Hz), 63.6, 60.8, 38.5, 27.3, 26.9, 24.7, 23.8, 13.9 ppm; HRMS (CI) (*m/z*) calcd for $C_{14}H_{23}F_2O_4$ 293.1564 $(MH)^+$, found 293.1563. Anal. Calcd for $C_{14}H_{22}F_2O_4$: C, 57.52; H, 7.59; F, 13.00. Found: C, 57.48; H, 7.66; F, 13.25.

Methyl 2-(Hexa-5-one)- β,β-difluoroacrylate (6j). Prepared from **2j** as described above in 55% overall yield: oil; IR 1714 and 1720 cm^{-1}; ^1H NMR 3.78 (3 H, s), 2.45 (2 H, t, J = 7 Hz), 2.30−2.19 (2 H, m), 2.14 ppm (3 H, s); ^{19}F NMR −69.4 (1 F, dm, J = 3 Hz), −74.3 ppm (1 F, dm, J = 3 Hz); ^{13}C NMR 208.5, 165.2 (dd, J_1 = 14 Hz, J_2 = 8 Hz), 159.8 (dd, J_1 = 310 Hz, J_2 = 295 Hz), 88.2 (dd, J_1 = 25 Hz, J_2 = 5 Hz), 52.0, 43.1, 29.8, 27.9, 24.2, 22.9 ppm; HRMS (CI) (*m/z*) calcd for $C_{10}H_{15}F_2O_3$ 221.0989 $(MH)^+$, found 221.0992. Anal. Calcd for $C_{10}H_{14}F_2O_3$: C, 54.54; H, 6.41. Found: C, 54.40; H, 6.42.

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