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Preparations and reactions of 2-trifluoromethylketenimines

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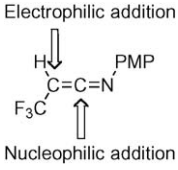
ABSTRACT

Preparations and reactions of a series of 2-trifluoromethylketenimines are described. Trifluoromethylketenimines were prepared from trifluoropropanoic acids via corresponding imidoyl chlorides in good yields. 2-Trifluoromethylketenimine was functionalized at its β-position by electrophilic addition of halide, followed by dehydrohalogenation. Addition of nucleophile at α-position gave trifluoroethylated β-amino acid derivative via 1,3-proton shift.

1. Introduction

Fluorinated building blocks are of importance in preparation of biologically active organic molecules [1]. Among them, building blocks with unsaturated bonds gain much interest for their electronic structures of the π-system, thus their unique reactivities by the strong electron withdrawing effect of the fluorine atom(s) and/or fluoroalkyl group(s) [1,2]. Similar to the fluoroalkylated imines [1,3], fluoroalkylated ketenimines would be highly talented class of compounds in the field of synthetic organic chemistry. They could undertake nucleophilic additions at sp carbon (α-position) and electrophilic additions at sp^2 carbon (β-position) [4]. However, they are less explored than the fluoroalkylated imines, because of their low availability.

To date, a few reports have described preparations of fluoroalkylated ketenimines. Bis(trifluoromethyl)ketenimines have been prepared by perfluoroisobutene or its analogs [5]. More complicated ketenimine has been prepared by the reaction of perfluoro(3,4-dimethylhex-3-en-2-one) with *tert*-butylamine [6], the reaction of perfluoro(2-methyl-2-pentene) with amines [7], and the reaction of 3,4-dicyano-1,1,1,4,4,4-hexafluoro-2-butene with thiadiazoline [8]. These ketenimines react with nucleophiles to give aldimines [9], heterocyclic compounds [5b,6,10], and metal complex [11]. To date, no electrophilic addition at sp^2 carbon of fluoroalkylated ketenimine has been reported.



Here, we report a facile preparation of β-trifluoromethylketenimines from the corresponding commercially available carboxylic acids via imidoyl halide [12]. We also report modifications of β-position (sp^2 ketenimine carbon) by electrophilic addition – dehydrohalogenation, as well as a nucleophilic addition at α-position to give trifluoroethylated β-amino acid derivative (dimethyl 2-{1-(*p*-anisyl)amino-3,3,3-trifluoropropylidene}malonate) via 1,3-proton shift.

2. Results and discussion

2.1. Preparations of ketenimines

Conventionally, fluorinated ketenimines were prepared by nucleophilic substitution (addition-defluorination) of olefinic fluorine of perfluoroisobutene derivatives with amines [3]. Although imidoyl halides have been frequently used as starting materials for non-fluorinated ketenimines [12], these compounds have not been used as intermediates of trifluoromethylketenimines, yet. The preparation of trifluoromethylated ketenimine from imidoyl halide would experience a tentative generation of α-trifluoromethylated carbanion, which is well known for its instability to leave β-fluoride [13]. Previously, we reported a

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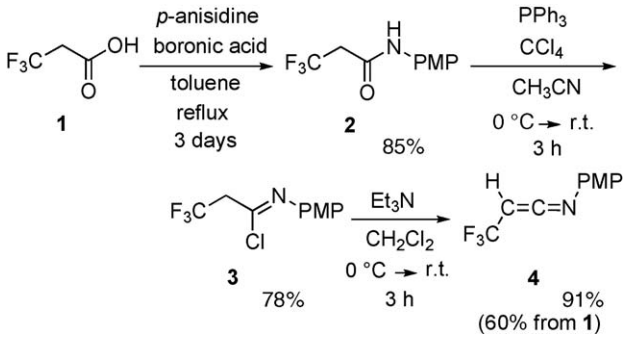
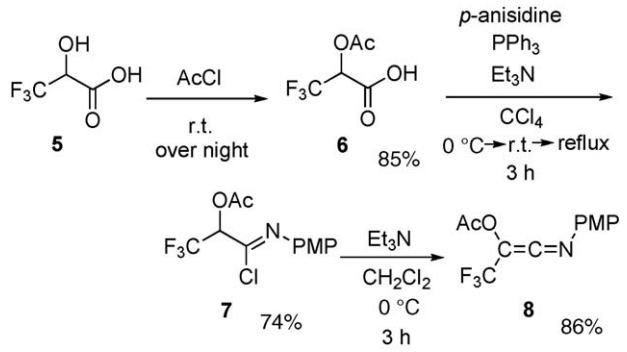


Scheme 1.



Scheme 2.

racemization of imidoyl halide of the trifluorolactate [3b]. These racemizations would also undergo via tentative α-trifluoromethylated carbanion intermediates or via β-trifluoromethylketenes. These observations encouraged us to prepare the β-trifluoromethylated ketenimines from the corresponding β-trifluoropropanoic acids via imidoyl chlorides.

The trifluoropropioimidoyl halide **3** was prepared from corresponding trifluoropropanoic amide **2** by one pot reaction with triphenylphosphine and carbon tetrachloride, as previously reported [14]. Then dehydrochlorination of the imidoyl chloride **3** with triethylamine gave the aimed ketenimine **4** in over all 60% yield from the commercially available trifluoropropanoic acid **1** (Scheme 1).

2-Acetoxy-2-trifluoromethylketenimine, **8** was prepared from commercially available trifluorolactic acid **5** in 54% yield [15]. The carboxylic acid **6** was directly converted to imidoyl chloride **7**, followed by dehydrochlorination to the ketenimine **8** (Scheme 2) [3b,14].

2.2. Electrophilic modification of the β-position

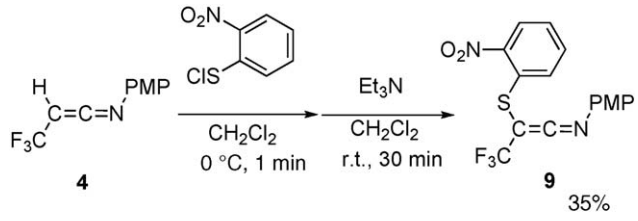
Electrophilic addition of halides to the ketenimine **4**, followed by dehydrohalogenations with triethylamine, gave β-substituted-β-trifluoromethylketenimines (**9** and **10** in Scheme 3).

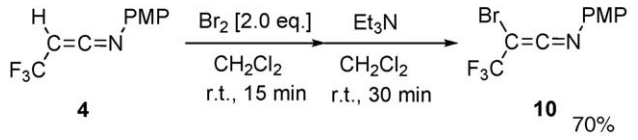
Similarly, addition of TMS-OTf to the ketenimine **4**, followed by detriflation gave β-trimethylsilyl-β-trifluoromethylketenimine **11** (Scheme 4).

As shown in these examples, these electrophilic modifications of the ketenimine **4** would give a variety of β-substituted-β-trifluoromethylketenimines.

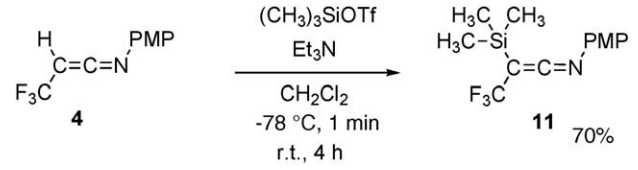
2.3. Nucleophilic addition to the α-position

Nucleophilic C–C bond formation of the ketenimine **4** with enolates would give β-lactams via following amide formation.

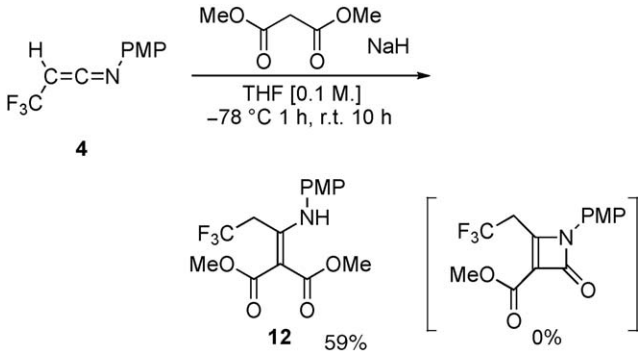




Scheme 3.



Scheme 4.



Scheme 5.

However, the reaction of malonate resulted in formation of α,β-unsaturated carboxylate **12** as a sole product, without formation of the aimed β-lactam (Scheme 5).

It is noteworthy that, a 1,3-shift of the hydrogen of active methylene to α-position of trifluoromethyl group seemed to be undertaken in the course of the reaction [16]. The double bond would put carbonyl group too far to attack by nitrogen nucleophile. Moreover, the larger strain energy of β-lactam with double bond would make the β-lactam too reactive to isolate.

3. Summary

In summary, we prepared a β-trifluoromethylketenimine **4** via corresponding imidoyl halide. Electrophilic addition of halides to the ketenimine **4** gave β-substituted-β-trifluoromethylketenimines, also via corresponding imidoyl halides or its synthon. Nucleophilic addition of active methylene compound unexpectedly produced α,β-unsaturated carboxylate **12** which could be a precursor of β-amino acids. Further studies, especially for the reaction of the α,β-unsaturated carboxylate **12** for preparations of β-amino acids, would enable to prepare a variety of trifluoroethylated β-amino acids.

4. Experimental

4.1. General

4.1.1. Spectroscopic measurements

NMR spectra: All NMR spectra were recorded as $CDCl_3$ solutions. ^1H and ^{19}F NMR spectra were recorded at 300 and 282 MHz respectively with Varian MERCURY 300 instrument. The chemical shifts are reported in δ (ppm) related to the $CHCl_3$ (7.26 ppm for ^1H NMR) and C_6F_6 (0 ppm for ^{19}F NMR: The relative chemical shift of C_6F_6 to $CFCl_3$ is −162.2 ppm.). Coupling constants (J) are reported in hertz (Hz).

IR spectra: Infrared spectra were recorded on a Hitachi 270-30 spectrometer. Only selected absorbances are reported (ν in cm^{-1}).

MS analysis: MS analyses were performed on a Shimadzu GCMS-QP5050A.

Elemental analysis: A Perkin-Elmer series II CHNS/O Analyzer 2400 was employed for elemental analysis.

4.1.2. Chemicals

CH_2Cl_2, triethylamine were distilled from CaH_2 and stored under an argon atmosphere. Acetonitrile was dried over P_2O_5, distilled, and stored under an argon atmosphere. Other reagents and solvents were employed without further purification.

3,3,3-Trifluoro-*N*-(4-methoxyphenyl)propanamide **2** was prepared from 3,3,3-trifluoropropanoic acid and anisidine with boronic acid catalyst. 2-Acetoxy-3,3,3-trifluoropropanoic acid **6** was prepared by an acetylation of 3,3,3-trifluorolactic acid [17].

4.2. Preparation of *N*-*p*-methoxyphenyl-3,3,3-trifluoropropanimidoyl chloride (Scheme 1, compound 3)

To a mixture of *N*-*p*-methoxyphenyl-3,3,3-trifluoropropanamide (**2**) (1.16 g, 5.0 mmol) and PPh_3 (1.57 g, 6.0 mmol) in CH_3CN (10 ml) was added CCl_4 (0.577 ml, 6.0 mmol) at 0 °C under an argon atmosphere, then was stirred for 3 h at room temperature. The solvent was removed under reduced pressure from reaction mixture. The residue was immediately purified by Kugelrohr distillation at 130 °C/0.2 Torr. The colorless oil of *N*-*p*-methoxyphenyl-3,3,3-trifluoropropanimidoyl chloride (**3**) (0.978 g, 3.9 mmol, 78%) was obtained.

^{19}F NMR δ 99.2 (t, J = 9 Hz, 3F) ppm; ^1H NMR δ 2.94 (q, J = 9 Hz, 2H), 3.26 (s, 3H), 6.67 (d, J = 9 Hz, 2H), 6.90 (d, J = 9 Hz, 2H) ppm; IR (neat) 3010, 2950, 1690, 1610 cm^{-1}; MS m/z (GC-EI) 253 (M^+, tr.), 251 (10), 215 (100), 200 (58), 152 (12), 77 (53); elemental analysis: Calcd for $C_{10}H_9ClF_3NO$: C, 47.73; H, 3.61; N, 5.57. Found: C, 47.49; H, 3.93; N, 5.26.

4.3. Preparation of *N*-*p*-methoxyphenyl trifluorometylketenimine (Scheme 1, compound 4)

To a solution of *N*-*p*-methoxyphenyl-3,3,3-trifluoropropanimidoyl chloride (**3**) (0.854 g, 3.4 mmol) in CH_2Cl_2 (5 ml) was added Et_3N (0.71 ml, 5.1 mmol) at 0 °C under an argon atmosphere, then was stirred for 3 h at room temperature. The reaction mixture was filtered and concentrated under reduced pressure. The crude product was immediately purified by Kugelrohr distillation apparatus at 100 °C/0.2 Torr. The colorless oil of *N*-*p*-methoxyphenyl trifluorometylketenimine (**4**) (0.66 g, 3.1 mmol, 91%) was obtained.

^{19}F NMR δ 109.8 (d, J = 6 Hz, 3F) ppm; ^1H NMR δ 3.18 (s, 3H), 3.98 (q, J = 6 Hz, 1H), 6.40–6.55 (m, 2H), 6.93–7.08 (m, 2H) ppm; IR (neat) 2060, 1610 cm^{-1}; MS m/z (GC-EI) 216 (M^++1, 60), 215 (M^+, 100), 200 (79), 152 (20); elemental analysis: Calcd for $C_{10}H_{18}F_3NO$: C, 55.82; H, 3.75; N, 6.51. Found: C, 55.71; H, 3.66; N, 6.50.

4.4. Preparation of *N*-*p*-methoxyphenyl-2-acetoxy-3,3,3-trifluoropropanimidoyl chloride (Scheme 2, compound 7)

Two necked round-bottomed flask was charged with PPh_3 (4.4 g, 16.8 mmol), triethylamine (2.3 ml, 17 mmol), CCl_4 (42 ml), and *p*-anisidine (2.06 g, 16.7 mmol). Then, 2-acetoxy-3,3,3-trifluoropropionic acid (**6**) (2.58 g, 13.9 mmol) was added to the mixture. After stirring at 0 °C for 10 min, the reacting solution was refluxed for 3 h. Solvents were removed under a reduced pressure. The residue was immediately purified by Kugelrohr distillation apparatus at 120 °C/0.05 Torr. The pale yellow oil of *N*-*p*-methoxyphenyl-2-acetoxy-3,3,3-trifluoropropanimidoyl chloride (**7**) (3.19 g, 10.3 mmol, 74%) was obtained.

^{19}F NMR δ 88.4 (d, J = 9 Hz, 3F) ppm; ^1H NMR δ 2.28 (s, 3H), 3.82 (s, 3H), 5.84 (q, J = 6 Hz, 1H), 6.92 (dd, J = 9, 2 Hz, 2H), 7.09 (dd, J = 9, 2 Hz, 2H) ppm; IR (neat) 1770, 1510 cm^{-1}; MS m/z (GC-EI) 309 (M^+, 11), 267 (17), 134 (80), 92 (14), 77 (22), 63 (14), 43 (100); elemental analysis: Calcd for $C_{12}H_{10}ClF_3NO_3$: C, 46.54; H, 3.58; N, 4.61. Found: C, 46.51; H, 3.73; N, 4.61.

4.5. Preparation of *N*-*p*-methoxyphenyl-2-acetoxy-2-trifluorometylketenimine (Scheme 2, compound 8)

To a solution of *N*-*p*-methoxyphenyl-2-acetoxy-3,3,3-trifluoropropanimidoyl chloride (**7**) (1.84 g, 6.0 mmol) in CH_2Cl_2 (8.21 ml) was added Et_3N (8.3 ml, 59 mmol) at 0 °C under an argon atmosphere. The reaction mixture was stirred for 3 h at this temperature. The reaction mixture was filtered and concentrated under a reduced pressure. The crude product was immediately purified by Kugelrohr distillation apparatus at 120 °C/0.03 Torr. The pale yellow oil of *N*-*p*-methoxyphenyl-2-acetoxy-2-trifluorometylketenimine (**8**) (1.39 g, 5.1 mmol, 86%, purity >98%) was obtained.

^{19}F NMR δ 96.8 (s, 3F) ppm; ^1H NMR δ 2.26 (s, 3H), 3.84 (s, 3H), 6.94 (d, J = 9 Hz, 2H), 7.46 (d, J = 9 Hz, 2H) ppm; IR (neat) 2040, 1770 cm^{-1}; MS m/z (GC-EI) 273 (M^+, 29), 232 (15), 231 (100) 134 (55); elemental analysis: Calcd for $C_{12}H_{10}F_3NO_3$: C, 52.75; H, 3.69; N, 5.13. Found: C, 52.58; H, 3.61; N, 5.20.

4.6. Preparation of 2-(2-nitrophenyl)sulfenyl-2-trifluorometylketenimine (Scheme 3, compound 9)

Two necked round-bottomed flask was charged with 2-nitrophenylsulfenyl chloride (0.39 g, 2.1 mmol) in 2 ml of CH_2Cl_2 under an argon atmosphere. Trifluoromethylketenimine (**4**) (0.44 g, 2.04 mmol) was added dropwise and the reaction mixture was stirred for 1 min at 0 °C. Triethylamine (0.38 ml, 2.7 mmol) was added and stirred for 10 min at 0 °C. The reaction mixture was concentrated under reduced pressure. The crude product was purified by triethylamine treated silica gel column chromatography with hexane/ethyl acetate eluent (6/1). The orange oil of 2-(2-nitrophenyl)sulfenyl-2-trifluoromethylketenimine (**9**) (0.265 g, 0.72 mmol, 35%) was obtained.

^{19}F NMR δ 104.3 (s, 3F) ppm; ^1H NMR δ 3.84 (s, 3H), 6.93 (d, J = 9 Hz, 2H), 7.28 (d, J = 9 Hz, 2H), 7.36 (ddd, J = 2, 7, 8 Hz, 1H), 7.69 (ddd, J = 2, 7, 8 Hz, 1H), 7.81 (d, J = 8 Hz, 1H), 8.29 (dd, J = 2, 8 Hz, 1H) ppm; IR (neat) 2050 cm^{-1}; MS m/z (DI) 368 (M^+, trace), 230 (22), 149 (100), 138 (36), 77 (88), 69 (67); elemental analysis: Calcd for $C_{16}H_{11}F_3N_2O_3S$: C, 52.17; H, 3.01; N, 7.61. Found: C, 51.95; H, 2.88; N, 7.55.

4.7. Preparation of 2-bromo-2-trifluoromethylketenimine (Scheme 3, compound 10)

Two necked round-bottomed flask was charged with trifluoromethylketenimine (**4**) (0.086 g, 0.40 mmol) in 0.5 ml of CH_2Cl_2

under an argon atmosphere. Bromine (0.041 ml, 0.79 mmol) was added dropwise and the reaction mixture was stirred for 1 min at 0 °C. Water bath was removed and then stirred for another 15 min at room temperature. Then, triethylamine (0.14 ml, 1.0 mmol) was added and stirred for 30 min at 0 °C. The reaction mixture was filtered and concentrated under a reduced pressure. The crude product was immediately purified by Kugelrohr distillation apparatus at 100 °C/0.05 Torr. The orange oil of 2-bromo-2-trifluoromethylketenimine (**10**) (0.082 g, 0.28 mmol, 70%) was obtained.

^{19}F NMR δ 101.5 (s, 3F) ppm; ^{1}H NMR δ 3.85 (s, 3H), 6.94 (d, J = 9 Hz, 2H), 7.32 (d, J = 9 Hz, 2H) ppm; IR (neat) 2030 cm^{-1}; MS m/z (GC-EI) 295 (M^+, 70), 293 (68), 280 (10), 278 (8), 214 (100), 107 (25), 92 (68), 77 (88); elemental analysis: Calcd for $C_{10}H_7BrF_3NO$: C, 40.84; H, 2.40; N, 4.76. Found: C, 40.67; H, 2.26; N, 4.87.

4.8. Preparation of 2-trimethylsilyl-2-trifluoromethylketenimine (Scheme 4, compound 11)

Two necked round-bottomed flask was charged with trifluoromethylketenimine (**4**) (0.10 g, 0.47 mmol) in 0.5 ml of CH_2Cl_2. Then, triethylamine (0.25 ml, 1.8 mmol) was added under an argon atmosphere and flask was cooled to −78 °C. Trimethylsilyl trifluoromethanesulfonate (0.25 ml, 1.4 mmol) was added dropwise and the reaction mixture was stirred for 1 h at −78 °C. An ethanol bath was put off and the reaction mixture was continuously stirred for 5 h at room temperature. The reaction mixture was concentrated under a reduced pressure. The crude product was immediately purified by Kugelrohr distillation apparatus at 100 °C/0.05 Torr. The colorless oil of 2-trimethylsilyl-2-trifluoromethylketenimine (**11**) (0.095 g, 0.33 mmol, 70%) was obtained.

^{19}F NMR δ 113.3 (s, 3F) ppm; ^{1}H NMR δ 0.25 (s, 9H), 3.82 (s, 3H), 6.90 (d, J = 9 Hz, 2H), 7.20 (d, J = 9 Hz, 2H) ppm; IR (neat) 2040 cm^{-1}; MS m/z (GC-EI) 287 (M^+, 67), 272 (95), 176 (80), 77 (100); elemental analysis: Calcd for $C_{13}H_{16}F_3NOSi$: C, 54.34; H, 5.61; N, 4.87. Found: C, 54.26; H, 5.68; N, 4.91.

4.9. Reaction of ketenimine 4 with enol from malonate: dimethyl 2-(3,3,3-trifluoro-1-(4-methoxyphenylamino)propylidene)malonate (Scheme 5, compound 12)

Two necked round-bottomed flask was charged with NaH (58% in oil, 0.48 g, 1.16 mmol) in 2.0 ml of THF and cooled to −78 °C. To the solution dimethyl malonate (0.136 g, 1.03 mmol) was added and reaction mixture was stirred for 20 min at −78 °C, ketenimine **4** (0.147 g, 0.68 mmol) dissolved in 3 ml of THF were added dropwise (30 min). The reaction mixture was stirred for 1 h at −78 °C, then stirred 10 h at room temperature. Reaction mixture was treated with silica gel. Short column chromatography gave dimethyl 2-(3,3,3-trifluoro-1-(4-methoxyphenylamino)propylidene)malonate **12** (0.133 g, 0.38 mmol, 59%) as a yellowish solid.

^{19}F NMR δ 99.3 (t, J = 10 Hz, 3F) ppm; ^{1}H NMR δ 3.62 (q, J = 10 Hz, 2H), 3.77 (s, 3H), 3.77 (s, 3H), 3.83 (s, 3H), 6.91 (d, J = 9 Hz, 2H), 7.06 (d, J = 9 Hz, 2H) ppm; IR (neat) 1700, 1600 cm^{-1}; MS m/z (GC-EI) 295 (M^+, 70), 293 (68), 280 (10), 278 (8), 214 (100), 107 (25), 92 (68), 77 (88); elemental analysis: Calcd for $C_{15}H_{16}F_3NO_5$: C, 51.88; H, 4.64; N, 4.03. Found: C, 52.07; H, 4.55; N, 4.03.

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