ELSEVIER

Journal of Fluorine Chemistry 111 (2001) 241–246

JOURNAL OF FLUORINE CHEMISTRY

www.elsevier.com/locate/jfluchem

A novel resin-bound CF_3-containing building block: application to the solid-phase synthesis of CF_3-containing acrylates and 4-isoxazolecarboxylates

Hong-Jun Wang, Wen Ling, Long Lu*

Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, 354 Fenglin Lu, Shanghai 200032, PR China

Received 24 April 2001; accepted 28 June 2001

Abstract

A novel resin-bound CF_3-containing building block (**3**) was prepared. Stereospecific Suzuki cross-coupling of compound **3** with a variety of aryl boronic acids gave the corresponding resin-bound coupling products (**4**), which can be cleaved with sodium methoxide to afford methyl (*E*)-3-aryl-4,4,4-trifluoro-2-butenoate (**5**). 1,3-Dipolar cycloaddition of compound **3** with aryl nitrile oxides followed by cleavage with TFA led to methyl 3-aryl-5-trifluoromethyl-4-isoxazolecarboxylate regioselectively, which was determined by ^{19}F NMR analysis.

Keywords: Trifluoromethyl; Solid-phase synthesis; 1,3-Dipolar cycloaddition

1. Introduction

Solid-phase organic synthesis (SPOS) has attracted much attention over the past decade [1–4], as a powerful tool in the rapid synthesis of small molecule libraries for drug discovery [5–10]. It is known that introduction of fluorine into an organic molecule often leads to changes in biological activities [11,12]. Recently, great efforts have been made in the search for practical and efficient methods for the synthesis of selectively fluorinated organic compounds [13,14]. Due to our continuing interest in organofluorine chemistry [15–18], we tried to investigate the preparation and application of polymer-supported fluoro-containing building blocks. Herein we report the preliminary results for the preparation of a novel resin-bound CF_3-containing building block and its palladium-catalyzed cross-coupling reactions with aryl boronic acids and 1,3-dipolar cyclo-addition reaction with aryl nitrile oxides.

2. Results and discussion

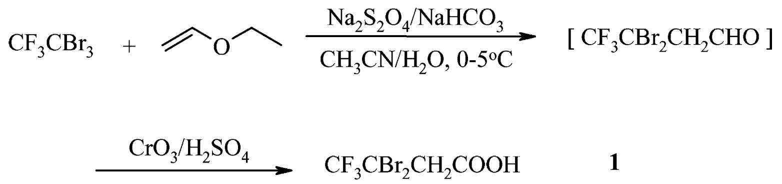
3,3-Dibromo-4,4,4-trifluorobutyrid acid (**1**) was prepared previously as an intermediate through the sodium dithionite-initiated addition on reaction of 1,1,1-tribromo-2,2,2-trifluoroethane with vinyl ethyl ether followed by Jones' oxidation [19], and is considered as a suitable CF_3-containing building block (Scheme 1).

For attaching 3,3-dibromo-4,4,4-trifluorobutyric acid (**1**) onto Wang resin (Scheme 1), initial attempts using 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrchloride, 1-hydroxybenzotriazole hydrate and diisopropylethylamine (EDC-HOBt-DIPEA) or Mitsunobu reaction conditions have been made but gave very poor results. So we activated Wang resin by using Hanessian's procedure [20], namely treatment of Wang resin with trichloroacetonitrile in CH_2Cl_2 in the presence of 1,8-diazabicyclo [5.4.0]undec-7-ene (DBU) to give Wang trichloroacetimidate resin **2**, which reacted with 3,3-dibromo-4,4,4-trifluorobutyric acid (**1**) in CH_2Cl_2 followed by elimination with triethylamine to afford the corresponding resin-bound CF_3-containing building block **3**. The reaction of Wang trichloroacetimidate resin **2** with acid **1** was monitored by FT-IR spectroscopy which showed the disappearance of bands (3340, 1664 cm^{-1}) associated with trichloroacetimidate and appearance of a strong band at 1749 cm^{-1}. Similarly, ^{19}F NMR indicated that the configuration of the double bond was *Z* form [19] after the cleavage from the resin. The structure and loading of the resin-bound CF_3-containing building block **3** was confirmed by cleavage with MeONa in MeOH/THF (1:4) [21] to give the corresponding methyl ester [19] (Scheme 2).

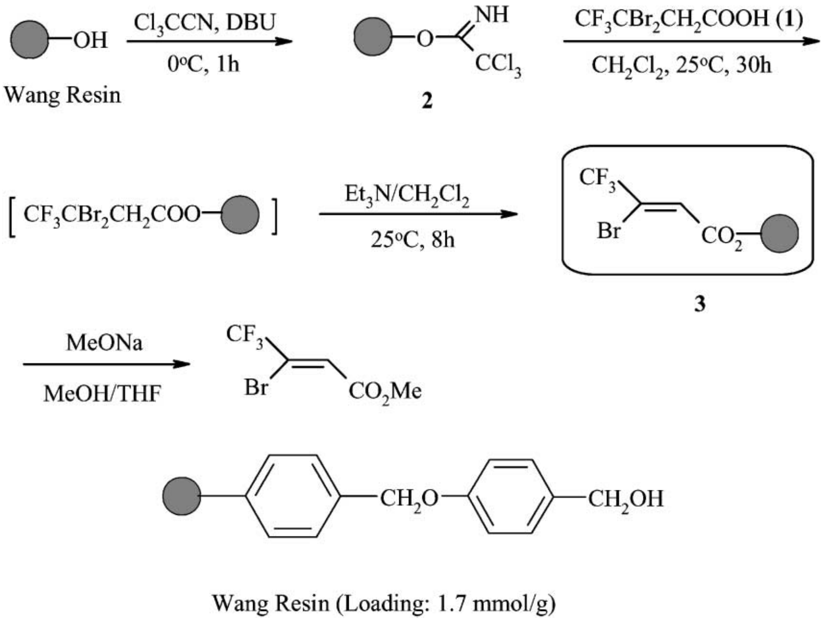
---

* Corresponding author. Tel.: +86-21-64163300;
fax: +86-21-64166128.
E-mail address: lulong@pub.sioc.ac.cn (L. Lu).

0022-1139/01/$ – see front matter © 2001 Elsevier Science B.V. All rights reserved.
PII: S0022-1139(01)00461-4



Scheme 1.

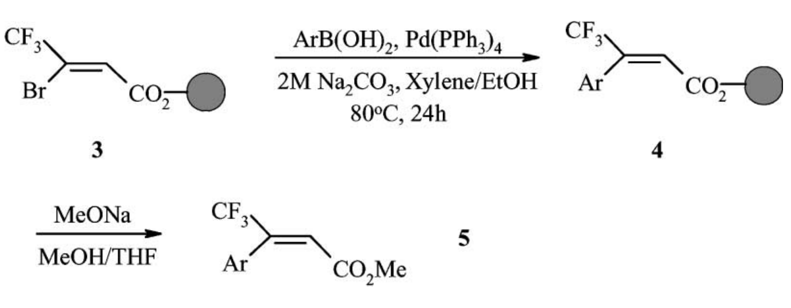


Scheme 2.

Suzuki coupling reaction of the resin-bound CF_3-containing building block **3** with aryl boronic acids was first investigated. Recently, solid-phase Suzuki coupling reactions have been widely investigated [22–28]. Under normal Suzuki conditions, the coupling reaction with a variety of aryl boronic acids proceeded smoothly, affording the corresponding resin-bound coupling products **4** in good yields. Treatment of resin-bound coupling products with MeONa in MeOH/THF (1:4) followed by the purification on a silica gel column led to the corresponding methyl esters **5**, which are characterized by MS, ^1H NMR, ^{19}F NMR and HRMS, etc. (Scheme 3). The detailed results of Suzuki coupling reactions were summarized in Table 1.

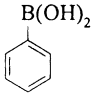
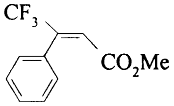
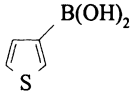
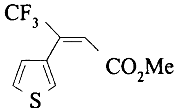
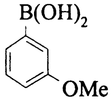
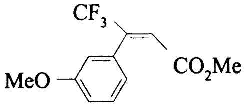
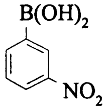
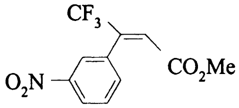
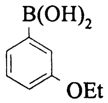
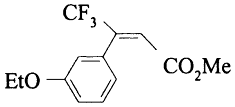
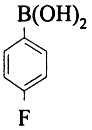
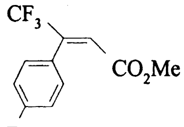
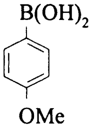
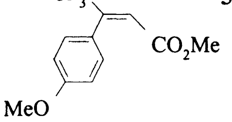
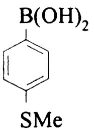
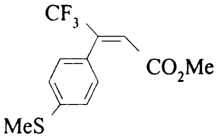
For compound **5d**, a byproduct was also isolated in yield of 26% based on the initial loading of the Wang resin (1.7 mmol/g) and proved to be the Michael addition product (**6**) of **5d** with methanol (Scheme 4).

Solid-phase 1,3-dipolar cycloaddition reaction has been well studied recently [29–38] as a very powerful method to synthesize a variety of heterocycle compounds such as isoxazolines, isoxazoles, pyrrolidines, tetrahydrofurans and so on. For the preparation of CF_3-containing heterocycles, the 1,3-dipolar cycloaddition reaction of CF_3-containing building block **3** with aryl nitrile oxides was studied in detail. A variety of oximes **7**, which are the precursors of nitrile oxides in the presence of base, were



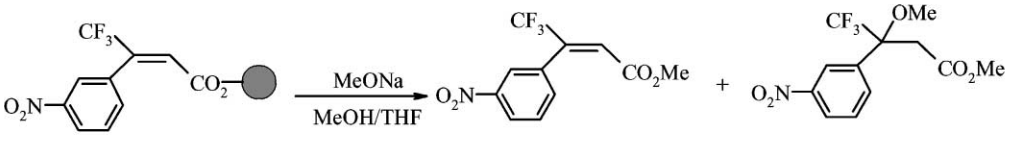
Scheme 3.

Table 1
Suzuki coupling reactions of resin-bound CF_3-containing building block **3**

Entry	$ArB(OH)_2$	Final coupling products	MS (EI) (M^+)	Yield[a] (%)
1		 **5a**	230	43
2		 **5b**	236	27
3		 **5c**	260	35
4		 **5d**	275	49
5		 **5e**	274	36
6		 **5f**	248	33
7		 **5g**	260	42
8		 **5h**	276	49

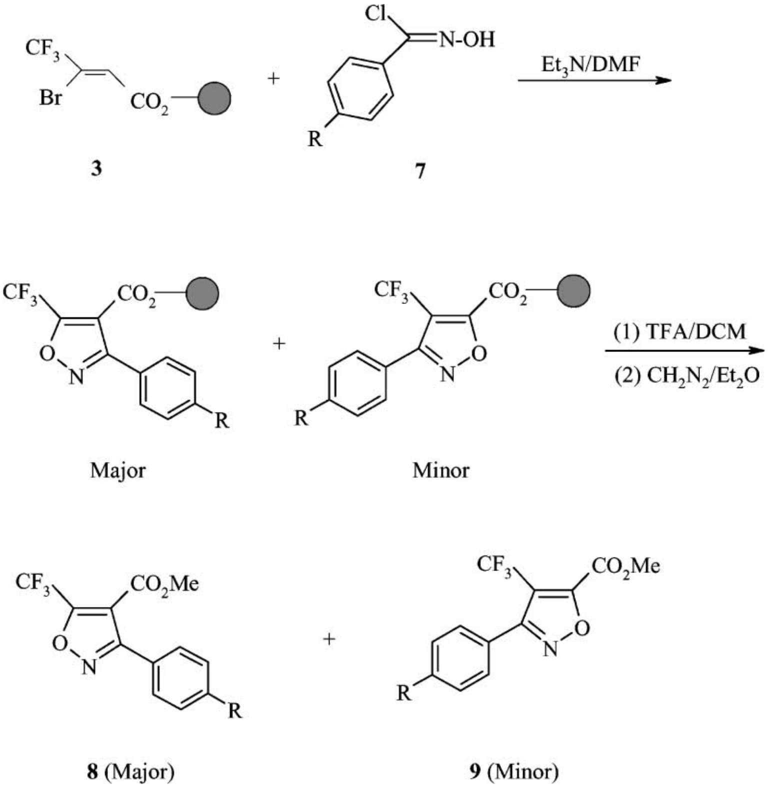
[a] Isolated yields were based on the initial loading of Wang resin (1.7 mmol/g).

prepared according to the literature procedures [39]. The CF_3-containing building block **3** reacted with oximes in the presence of Et_3N in DMF, affording a mixture of 1,3-dipolar cycloaddition products (**8** and **9**) after cleavage with 10% TFA/DCM solution followed by the treatment with diazomethane in Et_2O (Scheme 5). The ratio of the two isomers was determined by ^{19}F NMR analysis and the detailed result summarized in Table 2. The major isomers, 3-aryl-5-trifluoromethyl-4-isoxazolecarboxylate (**8**), were isolated by chromatography on a silica gel column, and characterized by MS (EI), HRMS, ^1H and ^{19}F NMR [40].



5d (Major) **6** (minor)

Scheme 4.



Scheme 5.

Table 2
1,3-Dipolar cycloaddition reaction of CF$_3$-containing building block **3** with oximes

Entry	R–	δ_{CF_3} of **8** (ppm)	δ_{CF_3} of **9** (ppm)	Ratio of **8/9**[a]	Yield[b] (%)
1	MeO	21.9	14.0	13:1	48 (**8a**)
2	EtO	21.9	14.0	13:1	43 (**8b**)
3	Me	22.0	14.0	14:1	32 (**8c**)
4	H	22.0	14.1	8:1	27 (**8d**)
5	Cl	22.0	14.1	18:1	21 (**8e**)
6	PhO	22.0	14.0	14:1	38 (**8f**)
7	BnO	21.9	14.0	13:1	34 (**8g**)

[a] The ratio of **8/9** was determined by ^{19}F NMR analysis of the crude methyl ester mixture.

[b] Isolated yields based on initial loading of Wang resin (1.7 mmol/g).

3. Conclusions

3,3-Dibromo-4,4,4-trifluorobutyric acid, which was isolated for the first time, reacted with Wang resin through trichloroacetonitrile activation to afford a novel resin-bound CF$_3$-containing building block (*Z*)-Wang 3-bromo-4,4,4-trifluoro-2-butenoate. The resin-bound CF$_3$-containing building block underwent Suzuki coupling reactions with a variety of aryl boronic acids to give methyl (*E*)-3-aryl-4,4,4-trifluoro-2-butenoates in good yield. Furthermore, a series of methyl 3-aryl-5-trifluoromethyl-4-isoxazolecarboxylates were prepared by 1,3-dipolar cycloaddition reactions of (*Z*)-Wang 3-bromo-4,4,4-trifluoro-2-butenoate with the precursors of aryl nitrile oxides. Construction of fluorinated compound libraries by using resin-bound fluorinated building blocks is on the way.

4. Experimental

4.1. General

Wang resin (1.7 mmol/g) was obtained from Polymer Laboratories Inc. ^{1}H and ^{19}F NMR spectra were recorded at 300 and 282 MHz, respectively, in CDCl$_3$ solutions with a Bruker AM300 instrument. Chemical shifts are expressed in ppm downfield from internal tetramethylsilane (for ^{1}H NMR) or trifluoroacetic acid (for ^{19}F NMR). Mass spectra were obtained on a HP5989A analytical spectrometer. Infrared spectra were recorded on a Bio-Rad FTS-185 spectrometer.

4.1.1. Preparation of resin-bound CF_3-containing building block (3)

Resin **2** was prepared from Wang resin according to Hanessian's method [20]. For high loaded Wang resin (1.70 mmol/g), the reaction should be repeated. Theoretical loading for resin **2** was 1.31 mmol/g. The CF_3-containing acid was prepared according to the literature [19].

To a suspension of resin **2** (3.0 g, 3.93 mmol) in CH_2Cl_2 (30 ml), acid **1** (3.54 g, 11.79 mmol) was added. The resulting mixture was shaken for 30 h at 25°C. The resin was filtered, washed successively with CH_2Cl_2, DMSO, THF and CH_2Cl_2 (three times for each solvent) and dried in vacuum. To a suspension of this resin intermediate in CH_2Cl_2 (30 ml), Et_3N (1.64 ml, 11.79 mmol) was added. The mixture was shaken for 8 h at 25°C. The resin was filtered, washed successively with CH_2Cl_2, DMSO, THF and CH_2Cl_2 (three times for each solvent) and dried in vacuum to give to give resin-bound CF_3-containing building block (**3**) (3.19 g). Theoretical loading for resin **3** was 1.23 mmol/g.

4.1.2. General procedure for the Suzuki coupling

To a degassed suspension of resin-bound CF_3-containing building block (**3**, 200 mg, 0.246 mmol) in xylene (5.0 ml), $Pd(PPh_3)_4$ (29 mg, 0.025 mmol), 2 M Na_2CO_3 (0.31 ml, 0.615 mmol), EtOH (0.2 ml) and aryl boronic acid (0.984 mmol, 4.0 eq.) were added. The resulting mixture was stirred under argon for 24 h at 80°C. After cooling to room temperature, the resin was filtered, washed successively with DMF, DMF/H_2O (1:1), THF, MeOH and CH_2Cl_2 (three times for each solvent) and dried in vacuum to give resin **4**. Resin **4** was treated with a solution of MeONa (0.05 mmol) in MeOH/THF (1:4, 4.0 ml) for 5 h, removed by filtration and washed with THF (5.0 ml) and CH_2Cl_2 (5.0 ml). The combined filtrates were concentrated and purified by flash chromatography to give methyl (*E*)-3-aryl-4,4,4-trifluoro-2-butenoates.

Methyl (*E*)-3-phenyl-4,4,4-trifluoro-2-butenoate (**5a**): oil, yield: 43%. ^{1}H NMR δ 7.42 (m, 3H), 7.34 (m, 2H), 6.61 (dd, $J = 2.6$ Hz, 1.4 Hz, 1H), 3.61 (s, 3H) ppm; ^{19}F NMR δ 9.4 (s, 3F) ppm; MS (EI) *m/z* 230 (M^+). HRMS (EI) $C_{11}H_9F_3O_2$, calcd.: 230.0555; found: 230.0561.

Methyl (*E*)-3-(3-thienyl)-4,4,4-trifluoro-2-butenoate (**5b**): oil, yield: 27%. ^{1}H NMR δ 7.49 (d, $J = 2.7$ Hz, 1H), 7.36 (m, 1H), 7.12 (d, $J = 5.0$ Hz, 1H), 6.57 (d, $J = 1.4$ Hz, 1H), 3.70 (s, 3H) ppm; ^{19}F NMR δ 9.7 (s, 3F) ppm; MS (EI) *m/z* 236 (M^+). HRMS (EI) $C_9H_7F_3O_2S$, calcd.: 236.0119; found: 236.0110.

Methyl (*E*)-3-(3-methoxyphenyl)-4,4,4-trifluoro-2-butenoate (**5c**): oil, yield: 35%. ^{1}H NMR δ 7.32 (t, $J = 8.0$ Hz, 1H), 6.97 (m, 1H), 6.85 (m, 2H), 6.60 (d, $J = 1.5$ Hz, 1H), 3.82 (s, 3H), 3.62 (s, 3H) ppm; ^{19}F NMR δ 9.5 (s, 3F) ppm; MS (EI) *m/z* 260 (M^+). HRMS (EI) $C_{12}H_{11}F_3O_3$, calcd.: 260.0660; found: 260.0661.

Methyl (*E*)-3-(3-nitrophenyl)-4,4,4-trifluoro-2-butenoate (**5d**): oil, yield: 49%. ^{1}H NMR δ 8.32 (m, 1H), 8.18 (s, 1H), 7.64 (m, 2H), 6.73 (d, $J = 1.5$ Hz, 1H), 3.65 (s, 3H) ppm; ^{19}F NMR δ 9.4 (s, 3F) ppm; MS (EI) *m/z* 275 (M^+). HRMS (EI) $C_{11}H_8NF_3O_4$, calcd.: 275.0405; found: 275.0410.

Methyl (*E*)-3-(3-ethoxyphenyl)-4,4,4-trifluoro-2-butenoate (**5e**): oil, yield: 36%. ^{1}H NMR δ 7.30 (m, 1H), 6.95 (dd, $J = 8.2$ Hz, 2.3 Hz, 1H), 6.83 (m, 2H), 6.59 (d, $J = 1.4$ Hz, 1H), 4.03 (q, $J = 7.0$ Hz, 2H), 3.62 (s, 3H), 1.41 (t, $J = 7.0$ Hz, 3H) ppm; ^{19}F NMR δ 9.5 (s, 3F) ppm; MS (EI) *m/z* 274 (M^+).

Methyl (*E*)-3-(4-fluorophenyl)-4,4,4-trifluoro-2-butenoate (**5f**): oil, yield: 33%. ^{1}H NMR δ 7.28 (m, 2H), 7.11 (m, 2H), 6.62 (d, $J = 1.4$ Hz, 1H), 3.63 (s, 3H) ppm; ^{19}F NMR δ 9.3 (s, 3F), −34.4 (m, 1F) ppm; MS (EI) *m/z* 248 (M^+). HRMS (EI) $C_{11}H_8F_4O_2$, calcd.: 248.0460; found: 248.0463.

Methyl (*E*)-3-(4-methoxyphenyl)-4,4,4-trifluoro-2-butenoate (**5g**): oil, yield: 42%. ^{1}H NMR δ 7.24 (d, $J = 8.7$ Hz, 2H), 6.93 (m, 2H), 6.57 (d, $J = 1.0$ Hz, 1H), 3.84 (s, 3H), 3.64 (s, 3H) ppm; ^{19}F NMR δ 9.6 (s, 3F) ppm; MS (EI) *m/z* 260 (M^+).

Methyl (*E*)-3-(4-methylthiophenyl)-4,4,4-trifluoro-2-butenoate (**5h**): oil, yield: 49%. ^{1}H NMR δ 7.24 (m, 4H), 6.60 (d, $J = 1.3$ Hz, 1H), 3.64 (s, 3H), 2.50 (s, 3H) ppm; ^{19}F NMR δ 9.6 (s, 3F) ppm; MS (EI) *m/z* 276 (M^+). HRMS (EI) $C_{12}H_{11}F_3O_2S$, calcd.: 276.0432; found: 276.0627.

Methyl 3-methoxy-3-(3-nitrophenyl)-4,4,4-trifluorobutyrate (**6**): oil, yield: 28%. ^{1}H NMR δ 8.46 (s, 1H), 8.26 (dd, $J = 8.2$ Hz, 2.0 Hz, 1H), 7.90 (d, $J = 8.2$ Hz, 1H), 3.59 (s, 3H), 3.60 (s, 3H), 3.30 (dd, $J = 46.1$ Hz, 15.7 Hz, 2H) ppm; ^{19}F NMR δ 1.57 (s, 3F) ppm; MS (EI) *m/z* 307 (M^+). HRMS (EI) $C_{12}H_{12}NF_3O_5$, calcd.: 307.0667; found: 307.0660.

4.1.3. General procedure for the 1,3-dipolar cycloaddition reaction

To a suspension of resin-bound CF_3-containing building block **3** (200 mg, 0.246 mmol) in DMF, chloro oxime **7** (5.0 eq.) and Et_3N (0.51 ml, 3.69 mmol) were added. The mixture was shaken for 12 h. After that, the resin was filtered, washed successively with DMF, DMF/H_2O (1:1), THF, MeOH and CH_2Cl_2 (three times for each solvent), dried in vacuum. The resin was treated with 10% TFA/DCM for 3 h, removed by filtration and washed with CH_2Cl_2 (5.0 ml × 2). The combined filtrates were concentrated. The residue was then treated with CH_2N_2/Et_2O (>0.1 N, 2.0 ml) for 10 min. The solution was concentrated to give the crude ester mixture. The ratio was determined by ^{19}F NMR analysis. Chromatography of the crude ester mixture gave the major products, 3-aryl-5-trifluoromethyl-4-isoxazolecarboxylates (**8**).

Methyl 3-(4-methoxyphenyl)-5-trifluoromethyl-4-isoxazolecarboxylate (**8a**): oil, yield: 48%. ^{1}H NMR δ 7.64 (dd, $J = 7.0$ Hz, 2.0 Hz, 2H), 7.00 (dd, $J = 6.7$ Hz, 2.0 Hz, 2H), 3.89 (s, 3H), 3.87 (s, 3H) ppm; ^{19}F NMR δ

14.0 (s, 3F) ppm; MS (EI) *m/z* 301 (M^+). HRMS (EI) $C_{13}H_{10}NF_3O_4$, calcd.: 301.0562; found: 301.0570.

Methyl 3-(4-ethoxyphenyl)-5-trifluoromethyl-4-isoxazolecarboxylate (**8b**): oil, yield: 43%. ^{1}H NMR δ 7.63 (dd, J = 6.7 Hz, 2.0 Hz, 2H), 6.99 (dd, J = 7.0 Hz, 2.0 Hz, 2H), 4.1 (q, J = 7.0 Hz, 2H), 3.90 (s, 3H), 1.46 (t, J = 7.0 Hz, 3H) ppm; ^{19}F NMR δ 14.0 (s, 3F) ppm; MS (EI) *m/z* 315 (M^+).

Methyl 3-(4-methylphenyl)-5-trifluoromethyl-4-isoxazolecarboxylate (**8c**) [40]: oil, yield: 32%. ^{1}H NMR δ 7.57 (m, 2H), 7.30 (d, J = 8.6 Hz, 2H) 3.89 (s, 3H), 2.43 (s, 3H) ppm; ^{19}F NMR δ 14.0 (s, 3F) ppm; MS (EI): *m/z* 285 (M^+).

Methyl 3-phenyl-5-trifluoromethyl-4-isoxazolecarboxylate (**8d**) [40]: oil, yield: 27%. ^{1}H NMR δ 7.67 (m, 2H), 7.52 (m, 3H), 3.88 (s, 3H) ppm; ^{19}F NMR δ 14.1 (s, 3F) ppm; MS (EI): *m/z* 271 (M^+).

Methyl 3-(4-chlorophenyl)-5-trifluoromethyl-4-isoxazolecarboxylate (**8e**) [40]: oil, yield: 21%. ^{1}H NMR δ 7.64 (m, 2H), 7.48 (m, 2H), 3.90 (s, 3H) ppm; ^{19}F NMR δ 14.1 (s, 3F) ppm; MS (EI): *m/z* 307, 305 (M^+).

Methyl 3-(4-phenoxyphenyl)-5-trifluoromethyl-4-isoxazolecarboxylate (**8f**): oil, yield: 38%. ^{1}H NMR δ 7.66 (dd, J = 6.8 Hz, 2.0 Hz, 2H), 7.40 (m, 2H), 7.20 (m, 1H), 7.09 (m, 4H), 3.90 (s, 3H) ppm; ^{19}F NMR δ 14.0 (s, 3F) ppm; MS (EI) *m/z* 363 (M^+). HRMS (EI) $C_{18}H_{12}NF_3O_4$, calcd.: 363.0718; found: 363.0726.

Methyl 3-(4-benzyloxyphenyl)-5-trifluoromethyl-4-isoxazolecarboxylate (**8g**): oil, yield: 34%. ^{1}H NMR δ 7.65 (m, 2H), 7.42 (m, 5H), 7.09 (m, 2H), 5.14 (s, 2H), 3.90 (s, 3H) ppm; ^{19}F NMR δ 14.0 (s, 3F) ppm; MS (EI) *m/z* 377 (M^+). HRMS (EI) $C_{19}H_{14}NF_3O_4$, calcd.: 377.0875; found: 377.0882.

Acknowledgements

We thank the National Natural Science Foundation of China (Grant nos. 29825104 and 29632003) and the Chinese Academy of Sciences for financial support of this work.

References

[1] S. Booth, P.H.H. Hermkens, H.C.J. Ottenheijm, D. Rees, Tetrahedron 54 (1998) 15385.
[2] P.H.H. Hermkens, H.C.J. Ottenheijm, D. Rees, Tetrahedron 53 (1997) 5647.
[3] P.H.H. Hermkens, H.C.J. Ottenheijm, D. Rees, Tetrahedron 52 (1996) 4527.
[4] C.J. Andres, D.L. Whitehouse, M.S. Deshpande, Curr. Opin. Chem. Biol. 2 (1998) 353.
[5] R.G.J. Franze, Comb. Chem. 2 (2000) 195.
[6] R.E.J. Dolle, Comb. Chem. 2 (2000) 383.
[7] R.E. Dolle, K.H. Nelson Jr., J. Comb. Chem. 1 (1999) 235.
[8] A. Nefzi, J.M. Ostresh, R.A. Houghten, Chem. Rev. 97 (1997) 449.
[9] L.A. Thompson, J.A. Ellman, Chem. Rev. 96 (1996) 555.
[10] J.S. Fruchtel, G. Jung, Angew. Chem. Int. Ed. Engl. 35 (1996) 17.
[11] J.T. Welch, Tetrahedron 43 (1987) 3123.
[12] J.T. Welch, S. Eswarakrishnan, Fluorine in Bioorganic Chemistry, Wiley, New York, 1991.
[13] J.T. Welch, Selective Fluorination in Organic and Bioorganic Chemistry, American Chemical Society, Washington, DC, 1991.
[14] M. Hudlicky, Chemistry of Organic Fluorine Compounds, Ellis Horwood, Chichester, UK, 1976.
[15] Q.S. Zhang, L. Lu, Tetrahedron Lett. 41 (2000) 8545.
[16] L. Lu, D.J. Burton, Tetrahedron Lett. 38 (1997) 7673.
[17] L. Xue, L. Lu S. Pederson, Q. Liu, R. Narske, D.J. Burton, J. Org. Chem. 62 (1997) 1064.
[18] Y. Wang, L. Lu, to be published.
[19] W.Y. Huang, L. Lu, Chin. Chem. Lett. 2 (1991) 769.
[20] S. Hanessian, F. Xie, Tetrahedron Lett. 39 (1998) 733.
[21] R. Frenette, R.W. Friesen, Tetrahedron Lett. 35 (1994) 9177.
[22] H. Juteau, Y. Gareau, M. Labelle, S. Lamontagne, N. Tremblay, M.-C. Carriere, N. Sawyer, D. Denis, K.M. Metters, Bioorg. Med. Chem. Lett. 11 (2001) 747.
[23] C.A. Briehn, T. Kirschbaum, P. Bouerle, J. Org. Chem. 65 (2000) 352.
[24] M. Gravel, C.D. Berube, D.G. Hall, J. Comb. Chem. 2 (2000) 228.
[25] W. Li, K. Burgess, Tetrahedron Lett. 40 (1999) 6527.
[26] S.-E. Yoo, Y.-D. Gong, M.-Y. Choi, J.-S. Seo, K.Y. Yi, Tetrahedron Lett. 41 (2000) 6415.
[27] C.M. Huwe, H. Kunzer, Tetrahedron Lett. 40 (1999) 683.
[28] C.G. Blettner, W.A. Kinig, G. Ruhter, W. Stenzel, T. Schotten, Synlett. (1999) 307.
[29] W.J. Haap, D. Kaiser, T.B. Walk, G. Jung, Tetrahedron 54 (1998) 3705.
[30] J.-F. Cheng, A.M.M. Mjalli, Tetrahedron Lett. 39 (1998) 939.
[31] S. Kobayashi, R. Akiyama, Tetrahedron Lett. 39 (1998) 9211.
[32] B.B. Shankar, D.Y. Yang, S. Girton, A.K. Ganguly, Tetrahedron Lett. 39 (1998) 2447.
[33] E.J. Kantorowski, M.J. Kurth, J. Org. Chem. 62 (1997) 6797.
[34] D.L. Whitehouse, K.H. Nelson Jr., S.N. Savinov, D.J. Austin, Tetrahedron Lett. 38 (1997) 7139.
[35] M.R. Gowravaram, M.A. Gallop, Tetrahedron Lett. 38 (1997) 6973.
[36] B.C. Hamper, D.R. Dukesherer, M.S. South, Tetrahedron Lett. 37 (1996) 3671.
[37] A.M.M. Mjalli, S. Sarshar, T.J. Baiga, Tetrahedron Lett. 37 (1996) 2943.
[38] D.A. Goff, Tetrahedron Lett. 40 (1999) 8741.
[39] K.C. Liu, B.R. Shelton, R.K. Howe, J. Org. Chem. 45 (1980) 3916.
[40] Y.C. Shen, J.H. Zheng, Y.Z. Huang, Synthesis (1985) 970.