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Facile access to stereodefined dienoates and cyclopropylenoates containing a trifluoromethyl group

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Abstract

Ethyl (2*E*,4*E*)-3-trifluoromethyl-2,4-dienoates **1a–e** and ethyl (*E*)-3-*trans*-alkylcyclopropyl-4,4,4-trifluoro-2-butenoates **2a–e** were prepared from the *trans*-alkenylboronic acids **3a–e** and the *trans*-cyclopropylboronic acids **4a–e** with ethyl (*Z*)-3-iodo-4,4,4-trifluoro-2-butenoate (**5**) by the Suzuki cross-coupling reaction in high yields (88–95%). The configurations of both **3a–e** or **4a–e** and **5** were retained in the reaction.

Keywords: Dienoate; Cyclopropylenoate; Trifluoromethyl; Stereodefined; Suzuki cross-coupling

1. Introduction

Stereodefined dienoates and cyclopropylenoates are so attractive to chemists, not only because of their unique biological and physiochemical properties [1], but also their many versatile synthetic utilities [2]. Therefore, the exploration of practical and efficient method for their preparation is still significant. Trifluoromethylated compounds, especially, the stereodefined dienoates and cyclopropylenoates bearing a trifluoromethyl group exhibit biological properties in the area of pharmaceuticals, agrochemicals and materials science [3], the efficacy of many pharmaceuticals and agrochemicals is often enhanced by or are dependent on the presence of a trifluoromethyl group in the molecular structure [4]. Among the various approaches for the introduction of the trifluoromethyl group into a wide variety of organic compounds, halogen exchange reaction [5] and trifluoromethylation [6] are possible, but these suffer from somewhat low reactivity, selectivity, even hazards (toxic or explosive), and lose favor upon the application. A convenient strategy for producing trifluoromethylated compounds is the preparation and application of trifluoromethylated building blocks [7]. Jeong et al. [8a] reported the preparation of trifluoromethylated vinylstannanes reagents and their cross-coupling with aryl iodies and acyl chlorides for producing trifluoromethylated enone derivatives, but the toxic organotin reagents were involved; Qing and Zhang [8b] described a new method for the synthesis of ethyl (*Z*)-3-iodo-4,4,4-trifluoro-2-butenoate (**5**) and its alkynylation by the Sonogashira reaction; Gildas et al. [8c] reported a stereoselective access to functional dienes containing the trifluoromethyl group via the Still cross-coupling of the compound **5**; we also demonstrated a novel method for constructing aryl substituted α,β-unsaturated esters bearing a trifluoromethyl group in high yields by arylation of the compound **5** [8d]. As a continuous development of our previous work, herein, we wish to describe a facile access to stereodefined dienoates and cyclopropylenoates containing a trifluoromethyl group by alkenylation and cyclopropanation of the compound **5** through the Suzuki cross-coupling reaction.

2. Results and discussion

2.1. Preparation of ethyl (Z)-3-iodo-4,4,4-trifluoro-2-butenoate (5)

Hydroiodination of ethyl 4,4,4-trifluoro-2-butynoate [9] using 57% HI dropwise at −5 °C and stirring for 8 h [8b],

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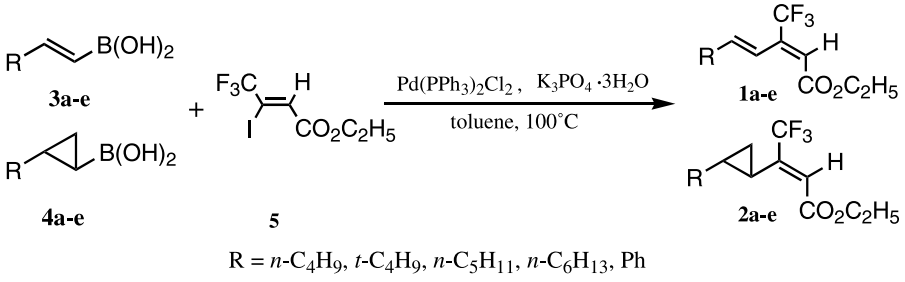


R = *n*-C_4H_9, *t*-C_4H_9, *n*-C_5H_{11}, *n*-C_6H_{13}, Ph

Scheme 1. The Suzuki cross-coupling of **3** or **4** with the compound **5**.

or in the presence of NaI and HOAc under nitrogen and refluxing for 72 h [8c], respectively, compound **5** was achieved in good yields (80–85%) as a colorless oil without additional purification. It was found that both the methods provided the sole isomer, which was proved by the data of ^{19}F NMR, HPLC and GC to be the compound **5**. The single peak of the CF_3 group in **5** ($\delta = -10.4$, for $CF_3CO_2C_2H_5$ utilized as an external standard, upfield being positive) at $\delta = -10.0$ showed that the CF_3 and $CO_2C_2H_5$ groups were *trans*-oriented [10a].

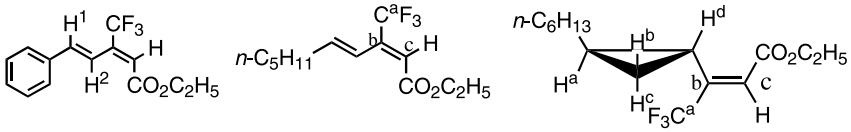
2.2. The Suzuki cross-coupling of trans-alkenyboronic acids *3a–e* or trans-cyclopropylboronic acids *4a–e* with the compound *5*

The *trans*-alkenylbornic acids **3a–e** are easily available and handled, because of their stability to moisture and air. The *trans*-cyclopropylboronic acids **4a–e** are achieved by the cyclopropanation of the *trans*-alkenylboronic acids **3a–e** through the Simmon–Smith reaction [11]. The Suzuki cross-coupling of **3a–e** or **4a–e** with the compound **5** were investigated (Scheme 1).

It was found that using $Pd(PPh_3)_2Cl_2$ as a catalyst and $K_3PO_4{\cdot}3H_2O$ as a base in toluene, the cross-coupling **3a–e** or **4a–e** with the compound **5** takes place at 100 °C to produce the corresponding cross-coupling products **1a–e** or **2a–e** in high yields (88–95%). The results are shown in Table 1. The stereo hindrance was observed in the cross-coupling of **4b** with **5** (entry 7, R = *t*-Bu, 33%). The data at Table 1 showed that all of the cross-coupling products **1a–e** or **2a–e** were obtained as the sole isomer, because a single peak of the CF_3 group was observed in the ^{19}F NMR spectrum [10a,10b].

The ^1H NMR spectra indicate that the H–H coupling constant ($^3J_{H-H} = 15$–17 Hz) of the products **1a–e** is bigger than *cis* H–H coupling constant ($^3J_{H-H} = 10$–11 Hz). For example, the coupling constant of the olefinic protons H^1 and H^2 in the product **1e** (entry 5, R = Ph) is 16.8 Hz (Scheme 2), this is a typical coupling constant of *trans*-olefinic protons [12]. Thus, the configuration of the alkenyl function in **1e** is 4*E*-isomer. The ^{13}C NMR spectra exhibit that the coupling constant $^3J^c_{F-C}$ is beyond 5.0 Hz (Scheme 2), typically, $^3J^c_{F-C}$ is 6.3 Hz for **1c** and 6.4 Hz for **2d**, these strongly support that the CF_3 and $CO_2C_2H_5$ groups in **1c** or **2d** are arranged to be the different side of the double bonds [10c]. The configurations of the cyclopropyl group in **2** are determined by the 2D $^1H-^1H$ NOESY spectra (Scheme 2). One of the cyclopropyl protons H^a ($\delta = 1.15$–1.50) in the product **2d** (entry 9, R = *n*-C_6H_{13}) showed a very strong NOE interaction with H^c ($\delta = 1.05$–1.12) and no NOE interaction with the other two protons H^b ($\delta = 0.80$–0.95) and H^d ($\delta = 2.20$–2.26), therefore the configuration of the cyclopropyl group in **2d** is the sole *trans*-isomer [13]. All these facts suggest the configurations of both the *trans*-alkenylboronic acids **3a–e** or the *trans*-cyclopropylboronic acids **4a–e** and the compound **5** are retained in the cross-coupling [14].

In conclusion, we have provided a facile access to stereodefined dienoates and cyclopropylenoates containing a trifluoromethyl group. This method has many attractive features: the easily availability of staring materials, mild conditions, high yields and effective stereoselectivity. The further research on synthesis of optically active cyclopropylenoates bearing a trifluoromethyl group is in the continuous study.

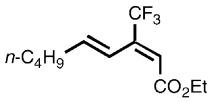
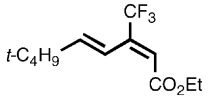
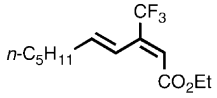
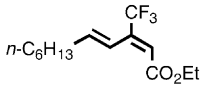
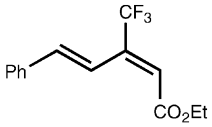
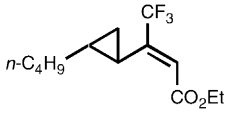
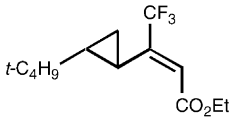
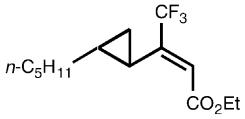
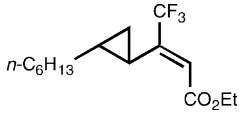
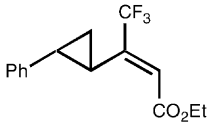


1e **1c** **2d**

$^3J_{H^1\text{-}H^2} = 16.8$Hz $\quad$ $^3J_{F\text{-}C^c} = 6.3$Hz $\quad$ $^3J_{F\text{-}C^c} = 6.4$Hz

Scheme 2. H–H and F–C coupling constants.

Table 1
The Suzuki cross-coupling of **3** or **4** with **5**

Entry	R		Products		δ_F (ppm)[a]	Yields (%)[b]
1	n-C_4H_9	**3a**		**1a**	−13.8	93
2	t-C_4H_9	**3b**		**1b**	−14.4	90
3	n-C_5H_{11}	**3c**		**1c**	−13.8	92
4	n-C_6H_{13}	**3d**		**1d**	−14.0	95
5	Ph	**3e**		**1e**	−14.2	93
6	n-C_4H_9	**4a**		**2a**	−12.4	91
7	t-C_4H_9	**4b**		**2b**	−12.6	33
8	n-C_5H_{11}	**4c**		**2c**	−12.6	89
9	n-C_6H_{13}	**4d**		**2d**	−12.6	90
10	Ph	**4e**		**2e**	−12.6	88

[a] CF_3CO_2H as an external standard, upfield being positive.
[b] Isolated yields, based on the compound **5**.

3. Experimental

3.1. General

All experiments were carried out under nitrogen atmosphere. The compound **5** was prepared according to the Qing and Gildas methods, respectively [8b,8c]. ^{1}H NMR and ^{13}C NMR spectra were recorded on a AM300 (300 MHz) spectrometer using $CDCl_3$ as solvent with TMS as an internal standard. ^{19}F NMR spectra were obtained on a Varian-360L (60 MHz) spectrometer with CF_3CO_2H as an external standard and upfield shift was designated as positive. Infrared spectra were taken on a PE-938G Spectrometer using KBr disc. Mass spectra were taken on a HP5989A Mass spectrometer using EI (70 eV). Element analyses were determined on a Foss-Heraeus Vario EL instrument.

3.2. Typical procedure for preparation of *1a–e* and *2a–e*

To a solution of **5** (0.5 mmol) in toluene (3 ml), the *trans*-alkenylboronic acid **3** or the *trans*-cyclopropylboronic acid **4** (0.55 mmol), $Pd(PPh_3)_2Cl_2$ (11.0 mg, 0.015 mmol), and $K_3PO_4{\cdot}3H_2O$ (400.0 mg, 1.5 mmol) were added under a nitrogen atmosphere. The reaction mixture was stirred at 100 °C for 8 h. The mixture was allowed to cool to room temperature, water (10 ml) was added, and the mixture was

extracted with ether (3 × 5 ml). The combined organic layer was washed with brine (3 × 5 ml) and dried over $MgSO_4$. After removal of the solvent on a rotary evaporator, the residue was purified by silica gel flash column chromatography (petroleum ether:ethyl acetate = 30:1 (v/v) as the elutant) to yield **1a**–**e** or **2a**–**e** as light yellow oils (88–95%).

Ethyl (2*E*,4*E*)-3-trifluoromethyl-2,4-nonadienoate (**1a**) ^{1}H NMR ($CDCl_3$) δ 7.42–7.00 (m, 1H), 6.46–6.16 (m, 1H), 6.04 (s, 1H), 4.16 (q, 2H), 2.50–2.00 (m, 2H), 1.70–0.65 (m, 10H); ^{19}F NMR ($CDCl_3$) δ −13.8 (s, CF_3); MS, *m*/*z* (rel. int.): 250 (M^+, 2.9), 222 (22.6), 205 (2.1), 177 (1.9), 135 (100.0), 91 (71.9); IR (KBr) 2960–2860, 1725, 1640, 1275–1130, 1060, 960, 880 cm^{-1}; Anal. Calcd. for $C_{12}H_{17}F_3O_2$: C, 57.60; H, 6.85. Found: C, 57.44; H, 6.66.

Ethyl (2*E*,4*E*)-3-trifluoromethyl-6,6-dimethyl-2,4-heptdienoate (**1b**) ^{1}H NMR ($CDCl_3$) δ 7.24–7.00 (m, 1H), 6.46–6.16 (m, 1H), 6.04 (s, 1H), 4.16 (q, 2H), 2.50–2.00 (m, 2H), 1.70–0.65 (m, 10H); ^{19}F NMR ($CDCl_3$) δ −13.8 (s, CF_3); MS, *m*/*z* (rel. int.): 249 ($M^+ - 1$, 7.4), 222 (10.4), 174 (21.3), 152 (29.8), 128 (31.4), 91 (73.8); IR (KBr) 2960–2860, 1725, 1640, 1275–1160, 1040, 965, 850 cm^{-1}; Anal. Calcd. for $C_{12}H_{17}F_3O_2$: C, 57.60; H, 6.85. Found: C, 57.37; H, 6.76.

Ethyl (2*E*,4*E*)-3-trifluoromethyl-2,4-decadienoate (**1c**) ^{1}H NMR ($CDCl_3$) δ 7.55–7.15 (m, 1H), 6.56–6.20 (m, 1H), 6.06 (s, 1H), 4.16 (q, 2H), 2.46–2.02 (m, 2H), 1.85–0.65 (m, 12H); ^{13}C NMR ($CDCl_3$) δ 163.90, 141.88, 139.66 (q, C^b, J = 28.7 Hz), 121.67 (q, C^a J = 276.4 Hz), 117.35 (q, C^c, J = 6.3 Hz), 59.79, 33.04, 30.50, 27.72, 21.55, 13.08, 12.93; ^{19}F NMR ($CDCl_3$) δ −13.8 (s, CF_3); MS, *m*/*z* (rel. int.): 250 (M^+, 2.9), 222 (22.6), 205 (2.1), 177 (1.9), 135 (100.0), 91 (71.9); IR (KBr) 2960–2860, 1725, 1640, 1275–1130, 1060, 960, 880 cm^{-1}; Anal. Calcd. for $C_{13}H_{19}F_3O_2$: C, 59.08; H, 7.24. Found: C, 59.24; H, 7.25.

Ethyl (2*E*,4*E*)-3-trifluoromethyl-2,4-undecadienoate (**1d**) ^{1}H NMR ($CDCl_3$) δ 7.42–7.04 (m, 1H), 6.52–6.16 (m, 1H), 6.10 (s, 1H), 4.20 (q, 2H), 2.50–2.00 (m, 2H), 1.70–0.60 (m, 14H); ^{19}F NMR ($CDCl_3$) δ −14.0 (s, CF_3); MS, *m*/*z* (rel. int.): 279 ($M^+ + 1$, 49.7), 249 (4.1), 233 (21.7), 193 (100.0), 165 (73.9), 115 (18.5); IR (KBr) 2760–2750, 1690, 1600, 1240–1100, 990, 940, 840 cm^{-1}; Anal. Calcd. for $C_{14}H_{21}F_3O_2$: C, 60.43; H, 7.55. Found: C, 60.56; H, 7.70.

Ethyl (2*E*,4*E*)-3-trifluoromethyl-5-phenyl-2,4-pentdienoate (**1e**) ^{1}H NMR ($CDCl_3$) ^{1}H NMR ($CDCl_3$) δ 8.10–8.0 (dd, J = 16.8 Hz, 1H), 7.56–7.26 (m, 5H), 7.06 (dd, J = 16.8 Hz, 1H), 6.22 (s, 1H), 4.20 (q, 2H), 1.32 (t, 3H); ^{19}F NMR ($CDCl_3$) δ −14.2 (s, CF_3); MS, *m*/*z* (rel. int.): 270 (M^+, 38.9), 241 (1.6), 225 (27.2), 197 (24.1), 177 (100.0), 146 (5.4) 128 (23.69); IR (KBr) 3120–3020, 1730, 1640, 1240–1120, 980, 885, 760, 700 cm^{-1}; Anal. Calcd. for $C_{14}H_{13}F_3O_2$: C, 62.22; H, 4.85. Found: C, 62.58; H, 4.96.

Ethyl (*E*)-3-*trans*-*n*-butylcyclopropyl-4,4,4-trifluoro-2-butenoate (**2a**) ^{1}H NMR ($CDCl_3$) δ 6.34 (s, 1H), 4.24 (q, 2H), 2.50–2.20 (m, 1H), 2.0–1.70 (m, 1H) 1.60–0.60 (m, 14H); ^{19}F NMR ($CDCl_3$) δ −12.5 (s, CF_3); MS, *m*/*z* (rel. int.): 265 ($M^+ + 1$, 100.0), 237 (11.9), 219 (41.7), 193 (28.8), 165 (27.5), 127 (27.9); IR (KBr) 3040–2860, 1726, 1640, 1285–1130, 1030, 880, 805 cm^{-1}; Anal. Calcd. for $C_{13}H_{19}F_3O_2$: C, 59.08; H, 7.25. Found: C, 58.84; H, 7.54.

Ethyl (*E*)-3-*trans*-*t*-butylcyclopropyl-4,4,4-trifluoro-2-butenoate (**2b**) ^{1}H NMR ($CDCl_3$) δ 6.25 (s, 1H), 4.18 (q, 2H), 2.70–2.36 (m, 1H), 1.28 (t, 3H), 0.9 (s, 9H), 0.60–0.30 (m, 2H); ^{19}F NMR ($CDCl_3$) δ −12.6 (s, CF_3); MS, *m*/*z* (rel. int.): 265 ($M^+ + 1$,, 28.9), 247 (1.9), 219 (9.7), 191 (11.3), 149 (13.5), 70 (100.0); IR (KBr) 3030–2860, 1726, 1640, 1285–1130, 1030, 885, 805 cm^{-1}; Anal. Calcd. for $C_{13}H_{19}F_3O_2$: C, 59.08; H, 7.25. Found: C, 58.86; H, 7.34.

Ethyl (*E*)-3-*trans*-*n*-pentylcyclopropyl-4,4,4-trifluoro-2-butenoate (**2c**) ^{1}H NMR ($CDCl_3$) δ 6.30 (s, 1H), 6.46–6.16 (m, 1H), 4.24 (q, 2H), 1.50–1.23 (m, 12H), 1.10–1.0 (m, 1H), 0.80 (t, 3H), 0.77–0.70 (m, 1H); ^{19}F NMR ($CDCl_3$) δ −12.6 (s, CF_3); MS, *m*/*z* (rel. int.): 279 ($M^+ + 1$, 3.7), 253 (2.2), 233 (2.5), 205 (4.5), 167 (18.5), 149 (55.3), 57 (100.0); IR (KBr) 3020–2850, 1740, 1635, 1285–1120, 1040, 880 cm^{-1}; Anal. Calcd. for $C_{14}H_{21}F_3O_2$: C, 60.43; H, 7.55. Found: C, 60.77; H, 7.35.

Ethyl (*E*)-3-*trans*-*n*-hexylcyclopropyl-4,4,4-trifluoro-2-butenoate (**2d**) ^{1}H NMR ($CDCl_3$) δ 6.30 (s, 1H), 4.15 (q, 2H), 2.26–2.20 (m, 1H), 1.50–1.15 (m, 14H), 1.12–1.05 (m, 1H), 0.95–0.85 (t, 3H), 0.76–0.62 (m, 1H); ^{13}C NMR ($CDCl_3$) δ 164.23, 144.74 (q, C^b, J = 27.5 Hz), 121.85 (q, C^a, J = 276.2 Hz), 121.20 (q, C^c, J = 6.5 Hz), 59.80, 33.36, 30.83, 28.04, 27.92, 21.61, 20.95, 16.07, 13.88, 13.13, 13.03; ^{19}F NMR ($CDCl_3$) δ −12.6 (s, CF_3); MS, *m*/*z* (rel. int.): 293 ($M^+ + 1$, 100.0), 265 (5.9), 247 (13.5), 217 (3.8), 193 (45.7), 165 (46.9); IR (KBr) 3020–2860, 1736, 1640, 1285–1120, 1030, 880, 805 cm^{-1}; Anal. Calcd. for $C_{15}H_{23}F_3O_2$: C, 61.63; H, 7.93. Found: C, 61.41; H, 7.53.

Ethyl (*E*)-3-*trans*-phenylclopropyl-4,4,4-trifluoro-2-butenoate (**2e**) ^{1}H NMR ($CDCl_3$) δ 7.50–7.0 (m, 5H), 6.40 (s, 1H), 4.16 (q, 2H), 3.0–2.74 (m, 2H), 2.68–2.16 (m, 1H), 1.74–1.16 (m, 5H); ^{19}F NMR ($CDCl_3$) δ −12.6 (s, CF_3); MS, *m*/*z* (rel. int.): 285 ($M^+ + 1$, 100.0), 284 (M^+, 3.8), 239 (25.8), 210 (74.4), 191 (100.0), 165 (74.3), 115 (56.0); IR (KBr) 3025–2860, 1740, 1630, 1290–1120, 1040, 896, 750, 700 cm^{-1}; Anal. Calcd. for $C_{15}H_{15}F_3O_2$: C, 63.38; H, 5.32. Found: C, 63.47; H, 5.16.

Acknowledgements

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References

[1] (a) S.D. Rychnocsky, Chem. Rev. 95 (1995) 2021–2040;
(b) B.E. John, Chem. Rev. 103 (2003) 1197–1212;
(c) F. Gnad, O. Reiser, Chem. Rev. 103 (2003) 1603–1624.
[2] K.C. Nicolau, E.J. Sorensen, Classics in Total Synthesis Targets, Strategies and Methods, Washington, DC, 1996, pp. 137–151, 265–284.

[3] (a) T. Hiyama, Organofluorine Compounds, Springer-Verlag, Berlin, 2000;
(b) R. Filler, Y. Kobayashi, L.M. Yagupolskii, Organofluorine Compounds in Medicinal Chemistry and Biomedical Applications, Elsevier, Amsterdam, 2000;
(c) R.E. Bank, B.E. Smart, J.C. Tatlow, Organofluorine Chemistry: Principle and Commercial Applications, Plenum Press, New York, 1994.
[4] (a) C. Elema, M. Manuel, J. Med. Chem. 36 (1993) 2984–2988;
(b) M.D. Jullia, C.D. Poulter, Tetrahedron 52 (1996) 119–121;
(c) R.W. Dolbier Jr., M.A. Battiste, Chem. Rev. 103 (2003) 1071–1098.
[5] (a) S.T. Purington, T.S. Evett, C.L. Bungarler, Tetrahedron Lett. 25 (1984) 1329–1332;
(b) O.D. Gupta, J.M. Shreeve, Tetrahedron Lett. 44 (2003) 2799–2801.
[6] (a) D.W. Wiemers, D.J. Burton, J. Am. Chem. Soc. 108 (1986) 832–834;
(b) T. Umemoto, A. Ando, Bull. Chem. Soc. Jpn. 59 (1986) 447–452;
(c) G.K.S. Prakash, R. Krishnamuiti, G.A. Olah, J. Am. Chem. Soc. 111 (1989) 393–395;
(d) Z.Y. Long, J.X. Duan, Y.B. Lin, Q.Y. Chen, J. Fluorine Chem. 78 (1996) 177–181.
[7] (a) I.H. Jeong, Y.S. Min, Y.S. Kim, B.T. Kim, K.Y. Cho, Tetrahedron Lett. 35 (1994) 7783–7784;
(b) G.Q. Shi, Y.Y. Xu, Chem. Commun. (1989) 607–608;
(c) B. Jiang, Y.Y. Xu, J. Org. Chem. 56 (1991) 7336–7340;
(d) B. Jiang, Y.Y. Xu, Tetrahedron Lett. 33 (1992) 511–514;
(e) T. Konno, H. Umetani, T. Kitazume, J. Org. Chem. 62 (1997) 137–150.
[8] (a) I.H. Jeong, Y.S. Park, M.S. Kim, Y.S. Song, J. Fluorine Chem. 120 (2003) 195–209;
(b) F.L. Qing, Y.M. Zhang, Tetrahedron Lett. 38 (1997) 6729–6732;
(c) P. Gildas, T. Jerome, A. Mohamed, D. Alain, P. Jean-Luc, Tetrahedron Lett. 40 (1999) 3151–3154;
(d) R.Q. Pan, P.A. Wang, M.Z. Deng, Heteroatom Chem. 13 (2002) 287–290.
[9] B.C. Hamper, Org. Synth. 70 (1991) 246–250.
[10] (a) F. Camps, R. Canela, J. Coll, A. Messeguer, A. Roca, Tetrahedron 34 (1978) 2179–2182;
(b) G.Q. Shi, X.H. Huang, F. Hong, J. Org. Chem. 61 (1996) 3200–3204;
(c) J.P. Bégue, D. Bonnet-Delpon, D. Mesureur, M. Ourévitch, Magn. Reson. Chem. 29 (1991) 675–682.
[11] X.Z. Wang, M.Z. Deng, J. Chem. Soc., Perkin Trans. I (1996) 2663–2664.
[12] Y.C. Ning, Structural Identification of Organic Compounds and Organic Spectroscopy, Science Press, Beijing, 2000, pp. 48–50, 98–141.
[13] (a) S.M. Zhou, M.Z. Deng, L.J. Xia, M.H. Tang, Angew. Chem. Int. Ed. Eng. 37 (1998) 2845–2847;
(b) M.L. Yao, M.Z. Deng, Tetrahedron Lett. 41 (2000) 9083–9087;
(c) H. Chen, M.Z. Deng, Org. Lett. 2 (2000) 1649–1651.
[14] (a) N. Miyaura, T. Yanagi, A. Suzuki, Syn. Commun. 11 (1981) 513–518;
(b) N. Miyaura, A. Suzuki, Chem. Rev. 95 (1995) 2457–2483;
(c) A. Suzuki, J. Organomet. Chem. 576 (1999) 147–168.