

Journal of Fluorine Chemistry 105 (2000) 169-173



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Unexpected regioselective Heck arylation of ethyl (*E*)-4,4,4-trifluorocrotonate

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Abstract

Palladium-catalyzed Heck reactions of ethyl (*E*)-4,4,4-trifluorocrotonate **1** with aryl bromides **2a–f** gave an unexpected α -arylated compound **4**, ethyl (*Z*)-2-aryl-4,4,4-trifluorocrotonate, mainly rather than normal β -arylated compound **3**, ethyl (*Z*)-3-aryl-4,4,4-trifluorocrotonate. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Heck reaction; Regioselective; Arylation; 4,4,4-trifluorocrotonate

1. Introduction

The palladium-catalyzed arylation (Heck reaction) is widely used to synthesize a variety of organic compounds [1,9–12]. This reaction produces regioselective arylation at the terminal β -position of electron-deficient olefins, such as α , β -unsaturated carbonyl compounds and at the α - or β position of electron-rich olefins, such as enol ethers, depending on the chelate control [2,13–16]. β -Arylation of ethyl acrylate with bromobenzene affords ethyl *trans*-3-phenylpropenoate, which on further arylation forms ethyl 3,3diphenyl propenoate in the presence of an excess amount of bromobenzene [3,17].

Recently, we have studied the effect of fluorine atoms on the bioactivity of indole-3-acetic acid (IAA), a famous plant hormone. A number of fluorinated derivatives of IAA have been prepared, of which 4,4,4-trifluoro-3-(indol-3-yl)butanoic acid is superior to IAA in the promotion of plant root growth [4,18]. This finding stimulated us to extend our research to a variety of plant growth regulators. As a part of our study, we undertook preparation of a trifluoromethylated analogue of cinnamic acid, a well-known bioactive compound [5], by the Heck reaction of ethyl (*E*)-4,4,4trifluorocrotonate **1** with bromoarenes. The unexpected α arylated product **4** was obtained mainly instead of the β arylated product **3**. We here report the palladium-catalyzed reaction of **1** with a number of aryl bromides **2a–f**.

2. Results and discussion

The reaction of ethyl (*E*)-4,4,4-trifluorocrotonate **1** and bromobenzene **2a** with a catalytic amount of palladium acetate as the pre-catalyst and triphenylphosphine as the ligand produced β -arylated product **3a** and α -arylated product **4a** (Scheme 1). The reaction was conducted in DMF at 100°C with *N*,*N*-diisopropylethylamine as the base. Some amounts of diarylation products **5** and **6**, characterized by their GC–MS and ¹H NMR spectra, also were produced, but the attempt to separate them was failed by silica gel column chromatography. For this reason, here the regioselective and stereospecific formation of monoarylated products are mainly discussed.

Palladium-catalyzed arylation of 1 with various aryl bromides 2b-e was also performed under the above conditions. Only a trace amount of the arylation product was detected by GC-MS for 1-bromonaphthalene 2c, and none for 2-bromothiophene 2e. In contrast, arylation products 3b, 3d and 4b, 4d were produced in moderate yields. Details of the reactions are given in Table 1 (runs 1–5). Furthermore, higher yields of 3 and 4 were obtained when a less sterically hindered bidentate ligand, 1,3-bis(diphenylphosphino)propane (DPPP), was used as the supporting ligand (Table 1, runs 6-11). On the other hand, the yields of 3a and 4a decreased when the molar ratio 2:1 changed from 2:1 to 1:1 (runs 7 and 6). The decreases in the yields of the monoarylated products also occurred when a large excess of aryl bromide was used owing to severe diarylation (run 8). In all cases, the formation of α -arylated compounds 4 was favored clearly much more than that of β -arylated compounds 3.

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Scheme 1

 Table 1

 Palladium-catalyzed arylation of ethyl 4.4.4-trifluorocrotonate^a

Run	ArBr ^b	2:1	Ligand	Conditions	3/4 [°]	4 (yields $\%$) ^d
1	2a	2:1	Ph ₃ P	100°C, 24 h	29/71	20
2	2b	2:1	Ph ₃ P	120°C, 36 h	43/57	26
3	2c	2:1	Ph ₃ P	120°C, 24 h	-	Trace
4	2d	2:1	Ph ₃ P	120°C, 24 h	41/59	30
5	2e	2:1	Ph ₃ P	120°C, 24 h	-	None
6	2a	1:1	DPPP	120°C, 24 h	32/68	26
7	2a	2:1	DPPP	120°C, 24 h	30/70	47
8	2a	5:1	DPPP	120°C, 24 h	33/67	23
9	2b	2:1	DPPP	120°C, 30 h	34/66	48
10	2d	2:1	DPPP	120°C, 30 h	33/67	43
11	2f	2:1	DPPP	120°C, 24 h	43/57	22

^a A mixture of 1 (1.0 mmol), 2 (2.0 mmol), *i*-Pr₂NEt (2.0 mmol), Pd(OAc)₂ (0.05 mmol), and ligand (0.12 mmol) was heated in DMF (4.5 ml) under argon.

^b **2a**: bromobenzene; **2b**: 4-bromoanisole; **2c**: 1-bromonaphthalene; **2d**: 2-bromonaphthalene; **2e**: 2-bromothiophene; **2f**: 4-bromochlorobenzene. ^c Calculated by ¹H NMR integration.

^d Isolated yields.

Products **3** and **4** were easily distinguished by the different splitting pattern of their olefinic protons. The peak of α -proton of **1** is split into doublet by one β -proton, and that of β -proton into doublet-quartet by one α -proton and three fluorine atoms¹. β -Arylation led to the removal of β -proton, thus, the α -proton of **3** shows singlet. Similarly, the β -proton of α -arylated product **4** shows a simple quartet (*J*=7.9 Hz).

In order to confirm the structure of α -arylated compound **4**, compound **4a** has been prepared according to the following way (Scheme 2). Firstly, ethyl 2-phenyl-4,4,4-trifluoroacetoacetate **7a**, prepared by the condensation of ethyl phenylacetate and ethyl trifluoroacetate, was reduced into the corresponding alcohol **8a**. The latter was then dehydrated to form (*Z*)-**4a** and (*E*)-**4a** in the ratio of about 1:9 as determined by ¹⁹F NMR. The β -protons of both (*Z*)-**4a** and (*E*)-**4a** are split into quartets with the same splitting constant (*J*_{HF}=7.9 Hz), but their chemical shifts (δ) are greatly different. The β -proton of (*E*)-**4a** has a higher δ value (6.87) compared with that of (*Z*)-**4a** (6.02) because of the strong deshielding effect of the ethoxycarbonyl group. Based on this spectroscopic analysis, we can believe (*Z*)-**4a** was formed stereospecifically in the palladium-catalyzed reac-

tion via a *syn*- β -hydride elimination (Scheme 3). This result is in agreement with experimental observations by Heck [6].

On the other hand, the geometric isomers of β -arylated product **3a** have been prepared via the Reformatsky reaction and the subsequent dehydration. Ethyl 3-hydroxy-3-phenyl-4,4,4-trifluorobutyrate (**9a**), obtained from the reaction of 2,2,2-trifluoroacetophenone, ethyl bromoacetate and zinc powder in toluene, was dehydrated to form (*E*)-**3a** and (*Z*)-**3a** in the ratio of about 5:1 (Scheme 4). They are easily distinguished by comparing the chemical shift of the fluorine atoms. The higher δ value (101.72) belongs to (*Z*)-**3a**, and the lower δ value (94.36) to (*E*)-**3a** owing to the obvious deshielding effect of the ethoxycarbonyl group. In addition, the α -olefinic protons of (*E*)-**3a** and (*Z*)-**3a** has a higher δ value (6.59) than (*Z*)-**3a** (6.33). Apparently, it is (*Z*)-**3a** that was formed in the palladium-catalyzed reaction (Scheme 3).

The regioselectivity of palladium-catalyzed arylation of the olefins was related only to the coordination-insertion pathway. Migration of the aryl group in the complex was markedly affected by the steric and electronic factors of the substituents. Usually migration favored the less substituted carbon with lower electron density, therefore only β -arylation occurred in the palladium-catalyzed arylation of ethyl acrylate, including ethyl cinnamate [3].

However, α -arylation (path b, Scheme 3) was greatly favored in the case of **1**, though only the β -addition took

¹ **1**: ¹H NMR (CDCl₃) δ: 6.85 (β-H, dq, J=16.0, 5.9 Hz), 6.47 (α-H, d, J=16.0 Hz), 4.28 (OCH₂, q, J=7.0 Hz), 1.33 (CH₃, t, J=7.0 Hz). ¹⁹F NMR (CDCl₃) δ: 96.27 (CF₃, d, J=5.9 Hz).



place in its reactions with various nucleophiles [7,19]. In fact, we realized that the trifluoromethyl group is much bulkier and stronger electron-inductive than the ethoxycarbonyl group from their Hammett substituent constant σ_p (0.53 for CF₃, 0.44 for COOEt), but its resonance contribution σ_R (0.08) is smaller (0.16 for CO₂Et) [8]. Therefore, we can consider that the steric hindrance and/or the inductive contribution of the substituent, play an important rule on regioselective migration of the aryl group in the palladium-catalyzed reactions, although the conjugative contribution usually acts as the main factor in other nucleophilic additions.

The biological activity of the above compounds (Z)-**3a**, (Z)-**4a**, (Z)-**3d** and (Z)-**4d** has been simply investigated in



promoting plant growth. In fact, the biological test was done using Chinese cabbage seeds by comparing their ability to enhance the root growth [18]. The parallel tests demonstrate that all of the four compounds are biologically active. Of the four compounds, (Z)-**3d** shows the best activity in root growth promotion, which is about 1.4 times higher compared to blank test. The activity of (Z)-**4d** is a little lower than that of (Z)-**3d**, but much higher than that of (Z)-**3a** and (Z)-**4a**. In addition, no detectable effect was observed on hypocotyls for the four compounds. The bioactivities are under further investigations using other plants and in the field.

3. Experimental details

3.1. General

¹H NMR spectra were recorded with tetramethylsilane (TMS) as an internal standard at 90 MHz on a Hitachi R-90H FT spectrometer. ¹⁹F NMR spectra were recorded with hexafluorobenzene as an internal standard at 84.7 MHz on the same spectrometer. Mass spectra (70 eV) were measured on a Hitachi M-80 instrument. High-resolution mass spectra were measured on a JEOL JMS-SX102A MS spectrometer.

3.2. *Ethyl* (Z)-3-phenyl-4,4,4-trifluorocrotonate (Z)-**3a** and ethyl (Z)-2-phenyl-4,4,4-trifluorocrotonate (Z)-**4a**

mixture of ethyl (E)-4,4,4-trifluorocrotonate А (1.0 mmol, 0.168 g), bromobenzene (2.0 mmol, 0.314 g), diisopropylethylamine (2.0 mmol, 0.258 g), palladium acetate (0.05 mmol, 0.0112 g), triphenylphophine (0.12 mmol, 0.0314 g) in anhydrous N,N-dimethylformamide (DMF, 4.5 ml) was degassed under a argon flow for 5 min, and then heated with continuous stirring at 100°C for 24 h. After cooled, the reaction mixture was poured into a saturated NH₄Cl aqueous solution (20 ml). The aqueous layer was extracted with ether $(3 \times 20 \text{ ml})$, and the combined organic phases were washed with saturated NH₄Cl (aq), dried over MgSO₄, filtered, and concentrated at reduced pressure. The residue was purified on a silica gel column with hexane/ethyl acetate (20:1, v/v) as the elution, to give 0.020 g of (Z)-3a (8.0%) and 0.048 g of (Z)-4a (19.7%).

For (*Z*)-**3a**: ¹H NMR (CDCl₃) δ : 7.41 (5H, s), 6.33 (1H, s), 4.31 (2H, q, *J*=7.2 Hz), 1.35 (3H, t, *J*=7.2 Hz). ¹⁹F NMR (CDCl₃) δ : 101.72 (3F, s). MS (*m/e*): 244 (M⁺, 60.2), 215 (49.6), 199 (100.0), 171 (15.0). HRMS Calc. 244.0711, Found: 244.0713.

For (*Z*)-**4a**: ¹H NMR (CDCl₃) δ : 7.42 (5H, s), 6.02 (1H, q, *J*=7.9 Hz), 4.37 (2H, q, *J*=7.1 Hz), 1.35 (3H, t, *J*=7.1 Hz). ¹⁹F NMR (CDCl₃) δ : 101.73 (3F, d, *J*=7.9 Hz). MS (*m/e*): 244 (M⁺, 100.0), 216 (24.9), 199 (31.3), 171 (95.8). HRMS Calc. 244.0711, Found: 244.0693.

3.3. Ethyl (Z)-3-(4-anisyl)-4,4,4-trifluorocrotonate (Z)-**3b** and ethyl (Z)-2-(4-anisyl)-4,4,4-trifluorocrotonate (Z)-**4b**

4-Bromoanisole (2.0 mmol, 0.374 g) was used instead of bromobenzene. The reaction was carried out at 120° C for 36 h, and worked-up similarly to give 0.054 g of (*Z*)-**3b** (19.6%) and 0.072 g of (*Z*)-**4b** (26.2%).

For (*Z*)-**3b**: ¹H NMR (CDCl₃) δ : 7.34 (2H, d, *J*=8.8), 6.90 (2H, d, *J*=8.8), 6.28 (1H, s), 4.29 (2H, q, *J*=7.0 Hz), 3.83 (3H, s), 1.34 (3H, t, *J*=7.0 Hz). ¹⁹F NMR (CDCl₃) δ : 101.72 (3F, s). MS (*m/e*): 274 (M⁺, 100.0), 229 (61.5), 201 (9.7). HRMS Calc. 274.0817, Found: 274.0818.

For (Z)-**4b**: ¹H NMR (CDCl₃) δ : 7.38 (2H, d, J=8.8), 6.90 (2H, d, J=8.8), 5.94 (1H, q, J=7.9 Hz), 4.36 (2H, q, J=7.3 Hz), 3.82 (3H, s), 1.35 (3H, t, J=7.3 Hz). ¹⁹F NMR (CDCl₃) δ : 102.14 (3F, d, J=7.9 Hz). MS (*m/e*): 274 (M⁺, 100.0), 229 (14.9), 201 (81.3). HRMS Calc. 274.0817, Found: 274.0828.

3.4. Ethyl (Z)-3-(2-naphthyl)-4,4,4-trifluorocrotonate (Z)-**3d** and ethyl (Z)-2-(2-naphthyl)-4,4,4-trifluorocrotonate (Z)-**4d**

A mixture of ethyl (*E*)-4,4,4-trifluorocrotonate (1.0 mmol, 0.168 g), 2-bromonaphthalene (2.0 mmol, 0.414 g), diisopropylethylamine (2.0 mmol, 0.258 g), palladium acetate (0.05 mmol, 0.0112 g), DPPP (0.12 mmol,

0.0494 g) in anhydrous *N*,*N*-DMF (4.5 ml) was degassed under a argon flow for 5 min, and then heated with continuous stirring at 120°C for 30 h. The residue was purified on a silica gel column to afford 0.062 g of (*Z*)-**3d** (21.1%) and 0.126 g of (*Z*)-**4d** (42.9%).

For (*Z*)-**3d**: ¹H NMR (CDCl₃) δ : 7.84 (4H, m), 7.54 (3H, m), 6.46 (1H, s), 4.33 (2H, q, *J*=7.3 Hz), 1.36 (3H, t, *J*= 7.3 Hz). ¹⁹F NMR (CDCl₃) δ : 102.07 (3F, s). MS (*m/e*): 294 (M⁺, 81.3), 266 (15.4), 249 (41.6), 248 (100.0). HRMS Calc. 294.0868, Found: 294.0857.

For (*Z*)-**4d**: ¹H NMR (CDCl₃) δ : 7.85 (4H, m), 7.53 (3H, m), 6.15 (1H, q, *J*=7.9 Hz), 4.42 (2H, q, *J*=7.3 Hz), 1.37 (3H, t, *J*=7.3 Hz). ¹⁹F NMR (CDCl₃) δ : 101.92 (3F, d, *J*=z 7.9 Hz). MS (*m/e*): 294 (M⁺, 100.0), 265 (12.1), 249 (18.6), 221 (23.8), 201 (33.5). HRMS Calc. 294.0868, Found: 294.0870.

3.5. *Ethyl* (*Z*)-3-(4-chlorophenyl)-4,4,4-trifluorocrotonate (*Z*)-**3f** and ethyl (*Z*)-2-(4-chlorophenyl)-4,4,4-trifluorocrotonate (*Z*)-**4f**

4-Bromochlorobenzene (2.0 mmol, 0.383 g) was used instead, and the reaction was done at 120° C for 24 h according to the above procedure. The residue was purified on a silica gel column to give 0.047 g of (*Z*)-**3f** (16.9%) and 0.062 g of (*Z*)-**4f** (22.3%).

For (*Z*)-**3f**: ¹H NMR (CDCl₃) δ : 7.36 (4H, s), 6.32 (1H, s), 4.32 (2H, q, *J*=7.0 Hz), 1.35 (3H, t, *J*=7.0 Hz). ¹⁹F NMR (CDCl₃) δ : 101.65 (3F, s). MS (*m*/*e*): 278 (M⁺, 46.8), 260 (92.5), 232 (100.0), 205 (78.3). HRMS Calc. 278.0321, Found: 278.0309.

For (*Z*)-**4f**: ¹H NMR (CDCl₃) δ : 7.38 (4H, s), 6.01 (1H, q, *J*=7.7 Hz), 4.36 (2H, q, *J*=7.3 Hz), 1.35 (3H, t, *J*=7.3 Hz). ¹⁹F NMR (CDCl₃) δ : 101.61 (3F, d, *J*=7.7 Hz). MS (*m/e*): 278 (M⁺, 95.4), 250 (41.9), 205 (100.0). HRMS Calc. 278.0321, Found: 278.0323.

3.6. Preparation of (E)-4a and (Z)-4a

In a flask, 20 ml of toluene and 1.2 g of clean sodium was placed. The sodium is melted by means of an oil bath and the stirrer is used to powder the sodium. After cooled, the toluene is decanted. To the sodium 8.2 g (50 mmol) of ethyl phenylacetate and 7.1 g (50 mmol) of ethyl trifluoroacetate are slowly added at $0-5^{\circ}$ C. The mixture is heated with vigorous stirring at $60-70^{\circ}$ C for 1 h, then chilled, acidified with 1 N HCl, extracted with ethyl acetate. The organic layer is dried over calcium chloride, filtered, the solvent removed, and the residue is distilled under reduced pressure to yield 2.9 g (22%) of ethyl 2-phenyl-4,4,4-trifluoroacetoacetate **7a**, bp 78–82°C/10 mmHg.

To a solution of 0.52 g of compound **7a** (2.0 mmol) in 10 ml ethanol, 0.11 g of sodium borohydride (2.9 mmol) is added in portion at room temperature. The mixture is stirred for 2 h, then acidified with 1 N HCl, extracted with ethyl acetate. The crude product is purified by silica gel

column chromatography (hexane/EtOAc 7:1) to give 0.48 g (92%) of ethyl 2-phenyl-3-hydroxy-4,4,4-trifluorobutyrate **8a**.

A mixture of compound **8a** (0.26 g, 1.0 mmol), sodium acetate (0.08 g) and acetic anhydride (0.20 g) in 2.0 ml tributylamine is refluxed with stirring for 4 h. The workup is done as mentioned above. The residue is passed through a silica gel column (hexane/EtOAc 15:1), and 0.16 g (66%) of the target compounds (*E*)-**4a** and (*Z*)-**4a** are collected.

For (*E*)-**4a**: ¹H NMR (CDCl₃) δ : 7.35 (5H, m), 6.87 (1H, q, *J*=7.9 Hz), 4.26 (2H, q, *J*=7.1 Hz), 1.27 (3H, t, *J*=7.1 Hz). ¹⁹F NMR (CDCl₃) δ : 104.10 (3F, d, *J*=7.9 Hz). MS (*m*/*e*): 244 (M⁺, 58.6), 216 (8.9), 199 (8.3), 171 (100.0), 151 (86.2).

3.7. Preparation of (E)-3a and (Z)-3a

To a flask containing a nitrogen atmosphere are added 0.65 g of fresh zinc powder, and 10 ml of dry toluene. The mixture is refluxed while a solution of 1.74 g (10 mmol) of 2,2,2-trifluoroacetophenone and 3.34 g (20 mmol) of ethyl bromoacetate in 10 ml of dry toluene is added dropwise with vigorous stirring. The addition is completed in about 15 min, and the mixture then refluxed for 2 h. When the solution has cooled to room temperature, 5 ml of 4 N HCl is added and the solution stirred for 15 min. The organic layer is separated and the aqueous layer extracted with ethyl acetate. The combined organic layer is dried over magnesium sulfate, evaporated under reduced pressure. The residue is passed through a silica gel column (hexane/EtOAc 7:1) to give 0.45 g (17%) of ethyl 3-hydroxy-3-phenyl-4,4,4-trifluorobutyrate **9a**.

Dehydration of compound 9a is carried out according to the same procedure with 8a, and gives the target compounds (*E*)-3a and (*Z*)-3a.

For (*E*)-**3a**: ¹H NMR (CDCl₃) δ : 7.35 (5H, m), 6.59 (1H, s), 4.10 (2H, q, *J*=7.2 Hz), 1.15 (3H, t, *J*=7.2 Hz). ¹⁹F NMR (CDCl₃) δ : 94.36 (3F, s). MS (*m*/*e*): 244 (M⁺, 81.6), 215 (28.3), 199 (100.0), 171 (16.2), 151 (78.9).

Acknowledgements

Y. Gong thanks for the Science and Technology Agency of Japan (STA) for the award of Fellowship Program, which is managed by the Research Development Corporation of Japan (JRDC) in cooperation with Japan International Science and Technology Exchange Center (JISTEC).

References

- [1] M. Shibasaki, E.M. Vogl, J. Organomet. Chem. 576 (1999) 1–2/1–15, and the other articles in this issue.
- [2] W. Cabri, I. Candiani, Acc. Chem. Res. 28 (1995) 2–7, and references cited therein.
- [3] M. Moreno-Manas, M. Perez, R. Pleixats, Tetrahedron Lett. 37 (1996) 7449–7452.
- [4] K. Kato, M. Katayama, S. Fujii, H. Kimoto, J. Ferment. Bioeng. 82 (1996) 355–360.
- [5] J. Van Overbeek, R. Blondeau, V. Horne, Am. J. Bot. 38 (1951) 589– 595.
- [6] R.F. Heck, J. Am. Chem. Soc. 91 (1969) 6707-6714.
- [7] L. Antolini, A. Forni, I. Moretti, F. Prati, E. Laurent, D. Gestmann, Tetrahedron: Asym. 7 (1996) 3309–3314.
- [8] J. March, Advanced Organic Chemistry, 3rd Edition, Wiley, New York, 1985, pages 244 and 247.
- [9] S. Brase, A. de Meijere, in: P.J. Stang, F. Diederich (Eds.), Metal-Catalyzed Cross-Coupling Reactions, Wiley, Weinheim, 1997, p. 99.
- [10] J. Tsuji, Palladium Reagents and Catalysts: Innovations in Organic Chemistry, Wiley, Chichester, 1995, p. 125.
- [11] R.F. Heck, in: B.M. Trost, I. Flemming (Eds.), Comprehensive Organic Synthesis, Vol. 4, Pergamon Press, Oxford, 1991, p. 833.
- [12] R.F. Heck, Org. React. 27 (1982) 345–389.
- [13] N. Garg, M. Larhed, A. Hallberg, J. Org. Chem. 63 (1998) 4158– 4162.
- [14] M. Beller, T.H. Riermeier, Tetrahedron Lett. 37 (1996) 6535-6538.
- [15] A. de Meijere, F.E. Meyer, Angew. Chem. Int. Ed. Engl. 33 (1994) 2379–2411.
- [16] G.D. Daves Jr., A. Hallberg, Chem. Rev. 89 (1989) 1433-1445.
- [17] R.F. Heck, Acc. Chem. Res. 12 (1979) 146–151.
- [18] M. Katayama, K. Kato, H. Kimoto, S. Fujii, Experientia 51 (1995) 721–724.
- [19] N. Shinohara, J. Haga, T. Yamazaki, T. Kitazume, S. Nakamura, J. Org. Chem. 60 (1995) 4363–4374.