Supporting Information

SmI₂-mediated Reduction of γ,γ -Difluoro- α,β -enoates with Application to the Synthesis of (Z)-Fluoroalkene-type Dipeptide Isosteres

Akira Otaka,^{*,‡} Junko Watanabe,[‡] Akira Yukimasa,[‡] Yoshikazu Sasaki,[‡] Hideaki Watanabe,[‡] Takayoshi Kinoshita,[§] Shinya Oishi,[‡] Hirokazu Tamamura[‡] and Nobutaka Fujii[‡]

Graduate School of Pharmaceutical Sciences, Kyoto University, Sakyo-ku, Kyoto 606-8501, Japan and Fujisawa Pharmaceutical Co., Ltd., Kashima, Yodogawa-ku, Osaka 532-8514, Japan

aotaka@pharm.kyoto-u.ac.jp

Experimental procedures for compounds in b-series of Scheme 1 and 2: S2-S6

ORTEP diagram for **31**: S7

ORTEP diagram for **36**: S8

¹H NMR charts for **16**: S9-S10

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Ethyl 4-Benzyloxy-2,2-difluoro-3-hydroxybutanoate (8b). By use of procedure similar to that described for the preparation of the ester **8a**, the aldehyde **7b** (10.0 g, 66.6 mmol) was converted into the title compound **8b** (6.6 g, 36% yield) as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ 1.29 (t, $J = 7.2$ Hz, 3H), 2.82 (d, $J = 6.4$ Hz, 1H), 3.68-3.74 (m, 2H), 4.22-4.30 (m, 1H), 4.25 (q, $J = 7.1$ Hz, 2H), 4.55 (s, 2H), 7.30-7.38 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ 13.8, 63.0, 67.6, 70.6, 73.6, 113.4, 127.5, 127.7, 128.1, 136.8, 162.7; HRMS (FAB), m/z calcd for $\text{C}_{13}\text{H}_{17}\text{F}_2\text{O}_4$ (MH^+), 275.1095, found: 275.1090.

Ethyl 4-Benzyloxy-3-[(*tert*-butyl)dimethylsiloxy]-2,2-difluorobutanoate (9b). By use of procedure similar to that described for the preparation of the ester **9a**, the ester **8b** (2.8 g, 10.2 mmol) was converted into the title compound **9b** (3.0 g, 76% yield) as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ 0.11 (s, 6H), 0.88 (s, 9H), 1.30 (t, $J = 7.2$ Hz, 3H), 3.53-3.59 (m, 1H), 3.74 (dd, $J = 9.9, 4.5$ Hz, 1H), 4.18-4.27 (m, 2H), 4.28-4.38 (m, 1H), 4.51 (dd, $J = 14.4, 11.7$ Hz, 2H), 7.29-7.38 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ -5.1, -4.5, 14.0, 18.2, 25.7, 62.7, 70.0, 72.3, 73.5, 114.0, 127.6, 128.2, 137.4, 163.1; HRMS (FAB), m/z calcd for $\text{C}_{19}\text{H}_{30}\text{F}_2\text{O}_4\text{Si}$ (MH^+), 389.1960, found: 389.1952.

Ethyl (2E)-6-Benzyloxy-5-[(*tert*-Butyl)dimethylsiloxy]-4,4-difluorohex-2-enoate (10b). By use of procedure similar to that described for the preparation of the enoate **10a**, the ester **9b** (3.0 g, 7.72 mmol) was converted into the title compound **10b** (2.3 g, 73% yield) as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ 0.08 (d, $J = 1.5$ Hz, 6H), 0.88 (s, 9H), 1.30 (t, $J = 7.1$ Hz, 3H), 3.44 (ddd, $J = 10.2, 6.7, 1.3$ Hz, 1H), 3.63 (dq, $J = 10.1, 1.7$ Hz, 1H), 4.04-4.13 (m, 1H), 4.23 (q, $J = 7.2$ Hz, 2H), 4.51 (s, 2H), 6.26 (dt, $J = 15.8, 2.2$ Hz, 1H), 6.86 (ddd, $J = 15.9, 13.5, 10.9$ Hz, 1H), 7.24-7.36 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ -5.1, -4.6, 14.1, 18.1, 25.6, 60.2, 60.9, 73.4, 73.9, 118.7, 125.5, 127.3, 128.0, 136.9, 137.4, 164.5; HRMS (FAB), m/z calcd for $\text{C}_{21}\text{H}_{33}\text{F}_2\text{O}_4\text{Si}$ (MH^+), 415.2116, found: 415.2126.

N-(4-Methoxyphenyl)-4-benzyloxy-2,2-difluoro-3-hydroxybutanamide (11b). By a procedure identical with that described for the preparation of the amide **11a**, the

ester **8b** (4.0 g, 14.6 mmol) was converted into the title compound **11b** (2.3 g, 45% yield) as a colorless powder: mp 80-82 °C; ¹H NMR (400 MHz, CDCl₃) δ 3.13 (d, *J* = 5.6 Hz, 1H), 3.75-3.84 (m, 2H), 3.80 (s, 3H), 4.36-4.46 (m 1H), 4.58 (s, 2H), 6.87 (d, *J* = 9.0 Hz, 2H), 7.27-7.33 (m, 5H), 7.43 (d, *J* = 8.8 Hz, 2H), 7.95 (br, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 55.5, 68.0, 70.5, 73.8, 114.2, 114.8, 122.0, 127.7, 127.8, 128.4, 128.8, 137.0, 157.1, 160.7; Anal. Calcd. for C₁₈H₁₉F₂NO₄: C, 61.53; H, 5.45; N, 3.99. Found: C, 61.33; H, 5.43; N, 4.00.

***N*-(4-Methoxyphenyl)-3-benzyloxymethyl-2,2-difluoropropano-3-lactam (12b).** By use of procedure similar to that described for the preparation of the lactam **12a**, the amide **11b** (2.3 g, 6.61 mmol) was converted into the title compound **12b** (2.2 g, 99% yield) as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ 3.79-3.84 (m, 1H), 3.81 (s, 3H), 3.90 (dd, *J* = 11.0, 4.6 Hz, 1H), 4.55-4.60 (m, 1H), 4.56 (s, 2H), 6.89 (d, *J* = 9.0 Hz, 2H), 7.24-7.37 (m, 5H), 7.49 (d, *J* = 9.0 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 55.6, 65.5, 66.0, 74.0, 114.4, 119.4, 119.6, 127.6, 128.0, 128.4, 129.0, 136.6, 156.9, 157.2; HRMS (FAB), *m/z* calcd for C₁₈H₁₇F₂NO₃ (M⁺), 333.1176, found: 333.1172.

Methyl 4-Benzyloxy-2,2-difluoro-3-[*N*-(4-methoxyphenyl)amino]butanoate (13b). By use of procedure similar to that described for the preparation of the ester **13a**, the lactam **12b** (2.2 g, 6.60 mmol) was converted into the title compound **13b** (1.8 g, 78% yield) as a pale yellow oil: ¹H NMR (400 MHz, CDCl₃) δ 3.70-3.73 (m, 2H), 3.71 (s, 3H), 3.75 (s, 3H), 4.10-4.20 (m, 1H), 4.49 (s, 2H), 6.66-6.70 (m, 2H), 6.75-6.80 (m, 2H), 7.25-7.37 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 53.3, 55.7, 57.6, 66.9, 73.6, 114.7, 115.9, 127.5, 127.7, 128.3, 137.2, 139.9, 153.0, 163.8; HRMS (FAB), *m/z* calcd for C₁₉H₂₁F₂NO₄ (M⁺), 365.1439, found: 365.1450.

Methyl 4-Benzyloxy-3-[*N*-(*tert*-butoxycarbonyl)amino]-2,2-difluorobutanoate (14b). By use of procedure similar to that described for the preparation of the ester **14a**, the ester **13b** (1.8 g, 5.12 mmol) was converted into the title compound **14b** (1.1 g, 61% yield) as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ 1.45 (s, 9H), 3.60-3.65 (m, 1H), 3.66-3.75 (m, 1H), 3.70 (s, 3H), 4.44-4.52 (m, 1H), 4.48 (s, 2H), 5.12 (d, *J* = 10.0 Hz,

1H), 7.26-7.38 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 28.3, 52.7, 53.4, 66.9, 73.6, 80.5, 113.5, 127.7, 127.8, 128.3, 137.0, 154.9, 163.3; HRMS (FAB), *m/z* calcd for C₁₇H₂₄F₂NO₅ (MH⁺), 360.1622, found: 360.1616.

Ethyl (2*E*)-6-Benzyloxy-5-[*N*-(*tert*-butoxycarbonyl)amino]-4,4-difluorohex-2-enoate (15b). By use of procedure similar to that described for the preparation of the enoate **15a**, the ester **14b** (500 mg, 1.52 mmol) was converted into the title compound **15b** (457 mg, 75% yield) as a colorless oil: ¹H NMR (400 MHz, CDCl₃) δ 1.29 (t, *J* = 7.1 Hz, 3H), 1.44 (s, 9H), 3.65 (dd, *J* = 10.4, 2.8 Hz, 1H), 3.72 (dd, *J* = 10.0, 3.7 Hz, 1H), 4.22 (q, *J* = 7.2 Hz, 2H), 4.28 (br, 1H), 4.50 (d, *J* = 12.0 Hz, 1H), 4.54 (d, *J* = 11.8 Hz, 1H), 5.02 (d = 9.0 Hz, 1H), 6.28 (dt, *J* = 15.9, 2.2 Hz, 1H), 6.86 (dt, *J* = 15.9, 12.2 Hz, 1H), 7.27-7.38 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 14.2, 28.3, 54.1, 61.1, 66.9, 73.3, 80.2, 119.0, 125.7, 127.5, 127.7, 128.2, 136.9, 137.2, 154.9, 164.5; HRMS (FAB), *m/z* calcd for C₂₀H₂₇F₂NO₅ (MH⁺), 400.1935, found: 400.1948.

Ethyl (3*Z*)-6-Benzyloxy-5-[(*tert*-butyl)dimethylsiloxy]-4-fluorohex-3-enoate (16b) (Table 1, entry 2). By use of procedure similar to that described for the preparation of the enoate **16a** (Table 1, entry 1), the enoate **10b** (100 mg, 0.241 mmol) was converted into the title compound **16b** (34 mg, 36% yield) and a byproduct **17b** (20 mg, 31%).

Compound **16b**: colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 0.09 (s, 3H), 0.10 (s, 3H), 0.90 (s, 9H), 1.26 (t, *J* = 7.2 Hz, 3H), 3.09-3.22 (m, 2H), 3.50 (dd, *J* = 10.2, 6.6 Hz, 1H), 3.57-3.65 (m, 1H), 4.15 (q, *J* = 7.4 Hz, 2H), 4.32 (m, 1H), 4.56 (s, 2H), 5.16 (dt, *J* = 36.4, 7.3 Hz, 1H), 7.24-7.36 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ -5.1, -4.5, 14.1, 18.1, 25.7, 27.8, 60.2, 70.7, 73.4, 73.7, 99.5, 127.3, 128.0, 137.5, 158.1, 170.7; HRMS (FAB), *m/z* calcd for C₂₁H₃₄FO₄Si (MH⁺), 397.2210, found: 397.2201.

Compound **17b**: colorless oil; ¹H NMR (400 MHz, CDCl₃) δ 1.32 (t, *J* = 7.1 Hz, 3H), 4.17-4.28 (m, 4H), 4.53 (s, 2H), 5.42 (dt, *J* = 34.7, 7.0 Hz, 1H), 6.19 (d, *J* = 15.4 Hz, 1H), 7.02 (dd, *J* = 26.6, 15.5 Hz, 1H), 7.24-7.40 (m, 5H); LRMS (FAB), *m/z* 265 (MH⁺), 91 (PhCH₃⁺, base peak), 73 (CH₂=CH-CF=CH₂ + H⁺).

Ethyl 6-Benzyloxy-5-[*N*-(*tert*-butoxycarbonyl)amino]-4,4-difluorohexanoate

(18b) (Table 1, entry 5). By use of procedure identical to that employed in Table 1, entry 4, the enoate **10b** was converted to a mixture of **16b** and **18b** (**16b:18b** = 78:15, 93% combined yield).

Compound **18b**: colorless oil; ^1H NMR (400 MHz, CDCl_3) δ 0.10 (s, 6H), 0.89 (s, 9H), 1.26 (t, J = 7.1 Hz, 3H), 2.08-2.41 (m, 2H), 2.50-2.56 (m, 2H), 3.45-3.53 (m, 1H), 3.63-3.69 (m, 1H), 3.96-4.04 (m, 1H), 4.15 (q, J = 7.2 Hz, 2H), 4.53 (s, 2H), 7.26-7.38 (m, 5H); HRMS (FAB), m/z calcd for $\text{C}_{21}\text{H}_{35}\text{F}_2\text{O}_4\text{Si}$ (MH^+), 417.2273, found: 417.2283.

Ethyl (3Z)-6-Benzoyloxy-5-[N-(tert-butoxycarbonyl)amino]-4-fluorohex-3-enoate (23b) (Table 2, entry 4). By use of procedure similar to that described for the preparation of the enoate **23a** (Table 2, entry 3), the enoate **15b** (40 mg, 0.100 mmol) was converted into the title compound **23b** (36 mg, 94% yield) as a colorless oil: ^1H NMR (400 MHz, CDCl_3) δ 1.25 (t, J = 7.1 Hz, 3H), 1.45 (s, 9H), 3.10-3.23 (m, 2H), 3.58 (dd, J = 9.8, 4.6 Hz, 1H), 3.64-3.69 (m, 1H), 4.14 (q, J = 7.1 Hz, 2H), 4.44 (br, 1H), 4.54 (s, 2H), 5.06 (br, 1H), 5.12 (dt, J = 36.4, 7.1 Hz, 1H), 7.27-7.36 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3) δ 14.3, 28.4, 29.4, 51.1, 60.9, 69.0, 73.2, 80.0, 99.3, 127.5, 127.6, 128.3, 137.4, 154.9, 157.7, 170.8; HRMS (FAB), m/z calcd for $\text{C}_{20}\text{H}_{29}\text{FNO}_5$ (MH^+), 382.2030, found: 382.2039.

Ethyl (3R)-2,2-Difluoro-3-{N-[(1R)-(2-methoxy-1-phenylethyl)]amino}-4-phenylbutanoate (32). By use of procedure similar to that described for the preparation of the ester **27**, the aldehyde **7a** (500 mg, 4.16 mmol) was converted into the title compound **32** (490 mg, 31% yield) as a colorless oil: $[\alpha]_D^{20}$ -41.0 (c 1.00, CHCl_3); ^1H NMR (600 MHz, CDCl_3) δ 1.37 (t, J = 7.1 Hz, 3H), 2.53 (dd, J = 13.8, 10.1 Hz, 1H), 2.91 (dd, J = 13.8, 3.7 Hz, 1H), 3.23-3.31 (m, 3H), 3.30 (s, 3H), 4.07 (dd, J = 7.5, 4.8 Hz, 1H), 4.27-4.37 (m, 2H), 6.77 (d, J = 7.1 Hz, 2H), 7.07 (t, J = 7.5 Hz, 4H), 7.12-7.16 (m, 1H), 7.23-7.26 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 14.1, 34.5, 58.6, 59.8, 61.5, 62.8, 77.8, 114.2, 126.3, 127.1, 127.5, 128.8, 129.0, 129.2, 137.4, 139.6, 164.0; HRMS (FAB), m/z calcd for $\text{C}_{21}\text{H}_{26}\text{F}_2\text{NO}_3$ (MH^+), 378.1881, found: 378.1875.

Ethyl (3R)-3-[N-(tert-Butoxycarbonyl)amino]-2,2-difluoro-4-phenylbutanoate

(33). By use of procedure similar to that described for the preparation of the ester **28**, the ester **32** (450 mg, 1.19 mmol) was converted into the title compound **33** (209 mg, 51% yield) as colorless crystals: mp 74-76 °C; $[\alpha]_D^{21}$ -36.6 (*c* 1.01, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ 1.28 (s, 9H), 1.32 (t, *J* = 7.1 Hz, 3H), 2.69 (dd, *J* = 14.2, 10.0 Hz, 1H), 3.14 (d, *J* = 14.9, 1H), 4.21-4.33 (m, 2H), 4.55-4.65 (m, 2H), 7.20-7.31 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 14.1, 28.0, 33.9, 53.6, 63.1, 80.1, 114.1, 126.6, 128.2, 128.9, 135.4, 154.4, 163.2; Anal. Calcd. for C₁₇H₂₃F₂NO₄: C, 59.46; H, 6.75; N, 4.08. Found: C, 59.43; H, 6.75; N, 3.85.

Ethyl (5*R*,2*E*)-5-[*N*-(*tert*-Butoxycarbonyl)amino]-4,4-difluoro-6-phenylhex-2-enoate (34). By use of procedure similar to that described for the preparation of the enoate **15a**, the ester **33** (2.5 g, 7.37 mmol) was converted into the title compound **34** (1.6 g, 60% yield) as colorless crystals: mp 93-95 °C; $[\alpha]_D^{23}$ -18.3 (*c* 0.99, CHCl₃); ¹H NMR (400 MHz, CDCl₃) δ 1.28 (s, 9H), 1.30 (t, *J* = 7.1 Hz, 3H), 2.67 (dd, *J* = 13.9, 10.8 Hz, 1H), 3.18 (dd, *J* = 14.5, 3.8 Hz, 1H), 4.23 (q, *J* = 7.2 Hz, 2H), 4.34 (br, 1H), 4.51 (d, *J* = 9.3 Hz, 1H), 6.33 (d, *J* = 15.9 Hz, 1H), 6.85 (dt, *J* = 15.6, 11.9 Hz, 1H), 7.18-7.32 (m, 5H); ¹³C NMR (100 MHz, CDCl₃) δ 14.1, 28.0, 34.2, 55.1, 61.0, 80.0, 119.3, 126.1, 126.5, 128.2, 128.8, 135.9, 137.0, 154.6, 164.3; Anal. Calcd. for C₁₉H₂₅F₂NO₄: C, 61.78; H, 6.82; N, 3.79. Found: C, 61.50; H, 6.86; N, 3.71.

***N*-(1*R*)-1-Phenylethyl)-(5*R*,2*E*)-5-[*N*-(*tert*-butoxycarbonyl)amino]-4,4-difluoro-6-phenylhex-2-enamide (36).** By use of procedure similar to that described for the preparation of the enamide **31**, the enoate **34** (100 mg, 0.271 mmol) was converted into the title compound **36** (72 mg, 60% yield) as colorless crystals: mp 209-211 °C; $[\alpha]_D^{21}$ -74.0 (*c* 0.91, CHCl₃); ¹H NMR (600 MHz, CDCl₃) δ 1.27 (s, 9H), 1.53 (d, *J* = 6.8 Hz, 3H), 2.65 (t, *J* = 12.4 Hz, 1H), 3.17 (dd, *J* = 14.3, 3.2 Hz, 1H), 4.32 (br, 1H), 4.52 (d, *J* = 9.7 Hz, 1H), 5.17-5.22 (m, 1H), 5.93 (d, *J* = 7.1 Hz, 1H), 6.32 (d, *J* = 15.5 Hz, 1H), 6.75 (dd, *J* = 26.2, 11.9 Hz, 1H), 7.17-7.37 (m, 10H); Anal. Calcd. for C₂₅H₃₀F₂N₂O₃: C, 67.55; H, 6.80; N, 6.30. Found: C, 67.43; H, 6.78; N, 6.30.

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ORTEP Diagram for **36**

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RGAIN 15

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Scan : WL

Band width :

2.0 nm

Sensitivity :

100 mdeg

Response :

1 sec

Wavelength range :

300.0 - 180.0nm

Step resolution :

0.1nm/data

Scan speed :

100nm/min

Accumulation : 10