Studies toward the Total Synthesis of (−)-Dictyostatin: Stereoselective Preparation of the C1–C10 Fragment

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Abstract: The efficient synthesis of the C1–C10 fragment of (−)-dictyostatin has been achieved by Evans–Tischenko reduction of a β-hydroxy ketone, followed by cross metathesis and Z-olefination. The β-hydroxy ketone is easily synthesized diastereoselectively by allylation and dihydroxylation of an alcohol.

Key words: diastereoselectivity, Evans–Tischenko reduction, β-hydroxy ketones, Wittig olefination, cross metathesis

Dictyostatin (1; Figure 1) is a marine-derived macrolide, which displays powerful growth-inhibitory activity against a number of murine and human cancer cells at low concentration (<1 nm), including paclitaxel-resistant cancer cells that express active P-glycoprotein.² Initial studies demonstrated that dictyostatin (1) arrests the cell in the G2/M phase by potently inducing tubulin polymerization and suppressing microtubule dynamics, leading to apoptosis similar to that seen in cells exposed to paclitaxel.² Over the past decade, the search for microtubule stabilizers has yielded the epothilones,³ discodermolide,⁴ laulimalide,⁵ eleutherobins,⁶ sarcodictyins,⁷ and peloroside A,⁸ as well as synthetically prepared structural variants of these novel entities. First isolated by Pettit⁹ and more recently by Wright,² dictyostatin’s structure is defined by a 22-membered macrolactone, featuring eleven stereo-centers, two dienes, and a cis-1,2-disubstituted olefin (Figure 1).¹⁰ The in vitro testing of synthetic samples showed that dictyostatin (1) exhibits antiproliferative potencies comparable or superior to its open-chain cousin discodermolide.²,¹¹ Moreover, with the recent withdrawal of discodermolide from clinical development,¹² the importance of the dictyostatin family increases further. Its significant therapeutic potential and relationship to discodermolide has resulted in total syntheses from the groups of Paterson,¹³ Curran,¹⁴ Phillips,¹⁵ and Ramachandran.¹⁶

In considering an alternative strategy for the synthesis of (−)-dictyostatin and its structural analogues, e.g. (−)-16-normethyldictyostatin,¹⁷ we have developed a stereoselective synthesis of the C1–C10 fragment, which we wish to report here.¹⁸

Abstract: The efficient synthesis of the C1–C10 fragment of (−)-dictyostatin has been achieved by Evans–Tischenko reduction of a β-hydroxy ketone, followed by cross metathesis and Z-olefination. The β-hydroxy ketone is easily synthesized diastereoselectively by allylation and dihydroxylation of an alcohol.

Key words: diastereoselectivity, Evans–Tischenko reduction, β-hydroxy ketones, Wittig olefination, cross metathesis

The retrosynthetic analysis (Scheme 1) revealed that the C1–C10 fragment of dictyostatin could be assembled by a cross-metathesis approach between olefin ⁴ and crotonaldehyde, followed by Z-olefination. Olefin ⁴ was envisioned to be prepared by Evans–Tischchenko reduction of β-hydroxy ketone ⁵ followed by simple functional group transformations. β-Hydroxy ketone ⁵, in turn, could be obtained from alcohol ⁶ by Lewis acid mediated allylation followed by diastereoselective dihydroxylation (Scheme 1).

Accordingly, we started our synthesis with the conversion of ⁶ into ⁷ by known literature procedures (Scheme 2).¹⁹ The resulting allylic alcohol ⁷ was protected as its methoxymethyl ether ⁸, whose double bond was dihydroxylated to furnish diol ⁹ in good yield. Selective silylation of the primary hydroxyl group in ⁹ as its tert-butyldiphenylsilyl ether 10²⁰ and oxidation of the secondary alcohol by use of Dess–Martin periodinane²¹ yielded ¹¹ (Scheme 2). Deprotection of the methoxymethyl group of ¹¹ with zinc(II) bromide afforded β-hydroxy ketone ¹² in 89% yield (Scheme 2).

This set the stage for the anti-selective reduction of ¹² (Scheme 3), which was accomplished under Evans–Tischchenko conditions,²³ mediated by samarium(II) iodide and propionaldehyde, to provide 1,3-diol monoester ¹² in 90% yield and >97% diastereoselectivity. Saponification of ¹² with potassium carbonate/methanol/water afforded the 1,3-anti-diol ¹³, which was protected with 2,2-dimethoxypropane, resulting in the formation of dimethyl acetal ¹⁴ (Scheme 3). Benzyl-group deprotection of ¹⁴ followed by oxidation with Dess–Martin periodinane²¹ afforded aldehyde ¹⁶ in good yield; subsequent treatment of ¹⁶ with methyltriphenylphosphonium bromide gave cru-
cial intermediate 4 required for the cross metathesis (Scheme 3).

With the key intermediate 4 in hand, the cross-metathesis strategy was performed with crotonaldehyde in toluene at 60 °C in the presence of the Grubbs second-generation catalyst (15 mol%) (Scheme 4); this exclusively gave E-olefinic aldehyde 3 in 60% yield along with unchanged starting compound 4. The subsequent Z-olefination was achieved by reaction between Ando’s phosphonate and α,β-unsaturated aldehyde 3 in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene as base to provide a Z/E mixture (9:1) of ester 17 (Scheme 4). Separation of the two isomers by column chromatography provided pure Z-ester 17 in 70% yield as a single diastereomer. Finally, deprotection of the tert-butyldiphenylsilyl group with tetrabutylammonium fluoride afforded the required C1–C10 core fragment 2 in 78% yield with the required stereocenters (Scheme 4).

In summary, we have developed an efficient route for the synthesis of the C1–C10 fragment 2 of dictyostatin (1) from known alcohol 6. The route features a substrate-controlled Evans–Tischenko reaction, cross metathesis, and Z-olefination as the key steps. Studies toward the total synthesis of dictyostatin and its structural analogues for biological studies are currently underway in our laboratory.

Optical rotations were measured on a JASCO DIP-370 digital polarimeter at 20 °C, and concentrations (c) are quoted in g/100 mL. Infrared spectra were recorded on a Shimadzu IR Prestige 21 FT-IR spectrometer. §H (400 MHz) and 13C (50 MHz) NMR spectra of samples dissolved in CDCl3 were determined on a Perkin-Elmer API 3000 spectrometer. Column chromatography was carried out on silica gel (grade 60–120, 100–200 mesh). Reactions were monitored by TLC on silica gel plates (60 F254); visualization was achieved with UV light or I2 spray. Unless stated otherwise, reactions were performed under an argon atmosphere. All other reagents were purchased from Aldrich at the highest commercial quality and used without further purification.

Scheme 1 Retrosynthesis of the C1–C10 fragment 2

Scheme 2 Reagents and conditions: (a) MOMCl, DIPEA, CH2Cl2, r.t., 2 h, 85%; (b) OsO4, NMO, acetone–H2O–t-BuOH (1:1:1), r.t., 3 h, 92%; (c) TBDPSCl, Et3N, DMAP, CH2Cl2, 0 °C, 50 min, then r.t., 12 h, 98%; (d) DMP, CH2Cl2, r.t., 1 h, 88%; (e) ZnBr2, CH2Cl2, r.t., 4 h, 89%.
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Scheme 3  Reagents and conditions: (a) SmI₂, EtCHO, THF, –10 °C, 3.5 h, 90%; (b) K₂CO₃, MeOH–H₂O (10:1), r.t., 2 h, 93%; (c) (MeO)₂CMe₂, CSA, CH₂Cl₂, 0 °C, then r.t., 12 h, 65%; (d) H₂, Raney Ni, EtOH, r.t., 12 h, 94%; (e) DMP, CH₂Cl₂, r.t., 1 h, 90%; (f) MePPh₃Br, NaHMDS, THF, –78 °C to 0 °C, 2 h, 78%.

Scheme 4  Reagents and conditions: (a) crotonaldehyde, Grubbs second-generation catalyst (15 mol%), toluene, 60 °C, 6 h, 60%; (b) (PhO)₂P(O)CH₂CO₂Et, DBU, NaI, THF, –78 °C, 30 min, then 0 °C, 30 min, 70%; (c) TBAF, THF, r.t., 12 h, 78%.

(4R,5S)-6-(Benzyloxy)-4-(methoxymethoxy)-5-methylhexane-1,2-diol (9)
To a stirred soln of 8 (9.6 g, 36.36 mmol) and NMO (6.4 g, 54.54 mmol) in acetone–H₂O–t-BuOH (1:1:1, 108 mL) at r.t. was added 1% OsO₄ in t-BuOH (9.3 mL, 1 mol%). The mixture was stirred at r.t. for 3 h and then quenched with aq Na₂S₂O₆ (30 mL) and extracted with CHCl₃ (3 × 200 mL). The combined organic layers were dried (Na₂SO₄) and concentrated in vacuo. The residue was purified by column chromatography (EtOAc–hexane, 80:20); this gave diol 9 as a diastereomeric mixture.

Yield: 10 g (92%); colorless oil.

IR (neat): 3441, 2933, 2883, 1454, 1365, 1148, 1095, 1034 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 7.37–7.27 (m, 5 H), 4.72–4.61 (m, 2 H), 3.96–3.84 (m, 2 H), 3.63–3.57 (m, 1 H), 3.48–3.29 (m, 6 H), 2.26–2.20 (m, 1 H), 1.80 (br s, 2 H, OH) 0.95–0.86 (m, 3 H).

13C NMR (50 MHz, CDCl₃): δ = 138.18 (138.13), 128.25 (128.25), 127.69 (127.55), 96.97 (95.81), 78.50 (77.64), 77.01 (76.37), 72.96 (72.11), 68.65 (68.55), 66.83 (66.42), 55.83 (55.80), 37.45 (36.33), 33.89 (33.04), 12.54 (11.81).

ESI-MS: m/z = 321.1 [M + Na]+, 316.2, 267.1, 236.8.
(5R,4S)-6-(Benzyloxy)-1-(tert-butylidiphenylsiloxy)-4-(methoxy)methoxy)-5-methylhexan-2-one (10)

To a stirred solution of 9 (11.7 g, 39.26 mmol) in CH₂Cl₂ (230 mL) at 0 °C was added Et₃N (10.9 mL, 78.52 mmol), DMAP (0.72 g, 5.89 mmol), and TBBDPSCl (12.3 mL, 47.11 mmol). The mixture was stirred at 0 °C for 50 min and then at r.t. for 12 h. After dilution with CH₂Cl₂ (400 mL), the mixture was washed with sat. aq NH₄Cl (2 × 100 mL), sat. aq NaHCO₃ (2 × 100 mL), H₂O (1 × 100 mL), and brine (2 × 100 mL). The organic layer was dried (Na₂SO₄) and concentrated in vacuo. The residue was purified by column chromatography (EtOAc–hexane, 30:70); this gave 10 as a mixture of diastereomers.

Yield: 20 g (98%); colorless oil.
IR (neat): 3491, 3070, 2930, 1589, 1428, 1112, 1037, 701 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 7.67–7.64 (m, 4 H), 7.44–7.24 (m, 11 H), 4.65–4.58 (m, 2 H), 4.48–4.44 (m, 2 H), 3.92–3.78 (m, 2 H), 3.66–3.53 (m, 3 H), 3.42–3.29 (m, 5 H), 3.04–2.85 (br s, 1 H, 1H), 2.18–2.12 (m, 1 H), 1.61–1.48 (m, 2 H), 1.06 (s, 9 H), 0.95–0.89 (m, 3 H).

13C NMR (50 MHz, CDCl₃): δ = 138.45 (138.38), 135.52 (135.52), 129.69 (129.69), 128.26 (128.26), 127.68 (127.52), 127.47 (127.47), 127.44 (127.43), 119.96 (96.01), 75.82 (74.68), 37.41 (36.66), 34.25 (33.19), 26.83 (26.82), 19.20 (19.20), 12.54 (12.32).

ESI-MS: m/z = 559.3 [M + Na⁺], 513.5, 469.2, 321.2.
ESI-HRMS: m/z calcd for C₂₃H₂₀O₃NaSi: 559.2856; found: 559.2864.

(4S,5R)-6-(Benzyloxy)-1-(tert-butylidiphenylsiloxy)-4-(methoxy)methoxy)-5-methylhexan-2-one (11)

To a stirred solution of 10 (5.0 g, 9.33 mmol) in CH₂Cl₂ (125 mL) was added DMP (4.75 g, 11.19 mmol) at r.t. After stirring for 1 h, the mixture was treated with sat. aq Na₂SO₄ (100 mL) and sat. aq NaHCO₃ (200 mL). The organic layer was separated and the aqueous layer was extracted with CH₂Cl₂ (3 × 100 mL). The combined organic layers were dried (Na₂SO₄) and concentrated in vacuo. The residue was purified by column chromatography (EtOAc–hexane, 15:75).

Yield: 4.38 g (88%); colorless oil; [α]D²⁰ = -2.75 (c 2.00, CHCl₃).
IR (neat): 3070, 2957, 2932, 2889, 2857, 1743, 1427, 1361, 1193, 1112, 702 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 7.67–7.65 (m, 4 H), 7.45–7.31 (m, 11 H), 5.21–5.16 (m, 1 H), 4.47 (s, 2 H), 3.63–3.54 (m, 3 H), 3.46–3.42 (m, 1 H), 3.30–3.23 (m, 1 H), 2.84 (d, J = 3.6 Hz, 1 H, OH), 2.26 (q, J = 7.52 Hz, 2 H), 1.26–1.16 (m, 2 H), 1.12–1.05 (m, 12 H), 0.94 (d, J = 6.72 Hz, 3 H).

13C NMR (50 MHz, CDCl₃): δ = 174.63, 138.35, 135.52, 129.71, 128.28, 127.69, 127.55, 127.46, 73.02, 72.46, 68.23, 68.03, 37.47, 35.05, 27.68, 26.82, 19.20, 13.45, 9.21.

ESI-MS: m/z = 513.1 [M + Na⁺], 566.3, 471.2, 397.2.
ESI-HRMS: m/z calcd for C₂₃H₂₀O₃NaSn: 513.2876; found: 513.2889.

(2S,4S,5R)-6-(Benzyloxy)-1-(tert-butylidiphenylsiloxy)-4-hydroxy-5-methylhexane-2,4-diol (13)

To a stirred solution of ester 12 (4.54 g, 8.28 mmol) in MeOH (37.3 mL) and HzO (3.73 mL) was added K₂CO₃ (2.29 g, 16.57 mmol). After stirring for 2 h at r.t., the mixture was diluted with H₂O (15.0 mL) and extracted with CH₂Cl₂ (1 × 100 mL). The combined organic layers were dried (Na₂SO₄) and concentrated in vacuo. The residue was purified by column chromatography (EtOAc–hexane, 15:75); this gave 13 as an anti-diol.

Yield: 3.78% (93%); colorless oil; [α]D²⁰ = -9.8 (c 1.00, CHCl₃).
IR (neat): 3488, 2956, 2929, 2856, 1427, 1361, 1112, 823, 700 cm⁻¹.

1H NMR (400 MHz, CDCl₃): δ = 7.67–7.65 (m, 4 H), 7.45–7.26 (m, 11 H), 4.51 (s, 2 H), 4.11–4.04 (m, 1 H, OH), 3.88–3.82 (m, 1 H), 3.76–3.65 (m, 1 H), 3.64–3.46 (m, 4 H), 3.15 (d, J = 3.49 Hz, 1 H, OH), 2.03–1.87 (m, 1 H), 1.69–1.52 (m, 2 H), 1.06 (s, 9 H), 0.86 (d, J = 6.98 Hz, 3 H).

13C NMR (50 MHz, CDCl₃): δ = 137.65, 135.47, 133.25, 129.67, 128.38, 127.66, 127.58, 75.08, 73.38, 72.73, 69.15, 67.89, 38.39, 36.65, 26.79, 19.16, 13.66.
ESI-MS: $m/z = 515.2$ [M + Na]$^+$, 467.2, 437.2, 415.2.

ESI-HRMS: $m/z$ calcd for $C_{19}H_{20}O_{3}NaSi$: 515.2594; found: 515.2580.

[(4S,6S)-6-[(1R)-2-(Benzylxiloxy)-1-methylallyl]-2,2-dimethyl-[1,3]dioxan-4-ylmethoxy]-tert-butylphenylsilane (14)

To a stirred soln of 1,3-anti-dioli 13 (3.53 g, 7.17 mmol) in CH$_2$Cl$_2$ (21 mL) at 0 °C was added 2,2-dimethoxypropane (2.2 mL, 17.94 mmol) and CSA (0.116 g, 0.50 mmol). After the mixture had stirred for 12 h at r.t., sat. aq NaHCO$_3$ (30 mL) and CH$_2$Cl$_2$ (50 mL) were added, and the mixture was stirred for 10 min. The organic layer was separated and the aqueous layer was extracted with CH$_2$Cl$_2$ (3 × 100 mL). The combined organic layers were dried (Na$_2$SO$_4$) and concentrated in vacuo. The residue was purified by column chromatography (EtOAc–hexane, 10:90).

ESI-MS: $m/z = 463.6$ [M + Na]$^+$, 458.3, 419.2, 383.3.

tert-Butyl[(4S,6S)-2,2-dimethyl-6-[(1R)-1-methylallyl]-[1,3]dioxan-4-ylmethoxy]-[phenylsilane (4)

A 1.0 M soln of NaHMDS in THF (1.99 mL, 1.99 mmol) was added dropwise to a stirred soln of MePPh$_3$Br (0.81 g, 2.27 mmol) in THF (5 mL) at 0 °C. The resulting yellow suspension was stirred for 30 min at 0 °C. The mixture was then cooled to –78 °C and a soln of aldehyde 16 (0.25 g, 0.57 mmol) in THF (3 mL) was added by syringe. After addition, the mixture was gradually warmed to 0 °C over 2 h and stirred at this temperature for 30 min. The mixture was quenched by the addition of sat. aq NH$_4$Cl (20 mL) and extracted with Et$_2$O (3 × 50 mL). The combined organic extracts were washed with brine (2 × 50 mL), dried (Na$_2$SO$_4$) and concentrated in vacuo. The residue was purified by column chromatography (EtOAc–hexane, 1:96).

ESI-MS: $m/z = 0.195$ g (78%); gummy mass; $[\alpha]_D^{20} = 14.8$ (c 1.00, CHCl$_3$).

IR (neat): 3070, 2931, 2858, 1547, 1427, 1379, 1224, 1136, 1112, 702 cm$^{-1}$.

1H NMR (400 MHz, CDCl$_3$): δ = 7.70–7.64 (m, 4 H), 7.43–7.34 (m, 6 H), 5.86–5.78 (m, 1 H), 5.05–5.00 (m, 2 H), 3.91–3.88 (m, 1 H), 3.72–3.60 (m, 3 H), 2.23–2.17 (m, 1 H), 1.62–1.54 (m, 2 H), 1.32 (d, $J = 6.98$ Hz, 6 H), 1.05 (s, 9 H), 0.97 (d, $J = 6.72$ Hz, 3 H).

13C NMR (50 MHz, CDCl$_3$): $\delta$ = 140.77, 135.71, 133.88, 129.55, 127.54, 114.33, 100.18, 69.94, 68.77, 66.82, 42.03, 31.93, 26.80, 24.83, 19.27, 15.24.

Yield: 0.66 g (90%); colorless oil.

ESI-MS: $m/z = 461.2$ [M + Na]$^+$, 413.3, 341.2, 303.2.

ESI-HRMS: $m/z$ calcd for $C_{27}H_{40}O_{3}NaSi$: 461.2488; found: 461.2477.

(4R)-4-[(4S,6S)-6-[(1R)-2-(Benzylxiloxy)-1-methylallyl]-2,2-dimethyl-[1,3]dioxan-4-yl]pent-2-enal (3)

To a stirred soln of 4 (100 mg, 0.23 mmol) in toluene (1.0 mL) was added freshly distilled crotonaldehyde (95 mL, 1.14 mmol) and the Grubbs second-generation catalyst (29 mg, 0.034 mmol, 15 mol%). The stirred mixture was heated at 60 °C for 6 h, and then cooled to r.t. The volatiles were removed in vacuo, and the residue was purified by column chromatography (EtOAc–hexane, 8:92).

Yield: 64 mg (60%); colorless oil; $[\alpha]_D^{20} = 13$ (c 1.00, CHCl$_3$).

IR (neat): 2929, 2856, 1693, 1462, 1427, 1379, 1224, 1112, 702 cm$^{-1}$.

1H NMR (400 MHz, CDCl$_3$): $\delta$ = 7.70–7.64 (m, 4 H), 7.43–7.34 (m, 6 H), 5.86–5.78 (m, 1 H), 5.05–5.00 (m, 2 H), 3.91–3.88 (m, 1 H), 3.72–3.60 (m, 3 H), 2.23–2.17 (m, 1 H), 1.62–1.54 (m, 2 H), 1.32 (d, $J = 6.98$ Hz, 6 H), 1.05 (s, 9 H), 0.97 (d, $J = 6.72$ Hz, 3 H).

13C NMR (50 MHz, CDCl$_3$): $\delta$ = 140.77, 135.71, 133.88, 129.55, 127.54, 114.33, 100.18, 69.94, 68.77, 66.82, 42.03, 31.93, 26.80, 24.83, 19.27, 15.24.

ESI-MS: $m/z = 461.2$ [M + Na]$^+$, 413.3, 341.2, 303.2.

ESI-HRMS: $m/z$ calcd for $C_{27}H_{40}O_{3}NaSi$: 461.2488; found: 461.2477.

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Ethyl (6R)-6-[(45,6S)-6-[(tert-Butyldiphenylsiloxy)methyl]-2,2-dimethyl-1,3-dioxan-4-yl]hepta-2,4-diene (2)

To a stirred soln of (PhO)2P(O)CH2CO2Et (317 mg, 0.99 mmol) in THF (2 mL) was added NaI (178 mg, 1.19 mmol) and DBU (148 mg, 0.98 mmol) at 0 °C and the mixture was stirred for 10 min. It was then cooled to –78 °C and a soln of aldehyde 3 (370 mg, 0.79 mmol) in THF (2 mL) was added by syringe. The resulting mixture was stirred at –78 °C for 30 min and then at 0 °C for 30 min. The mixture was quenched by the addition of sat. aq NH4Cl (5 mL) and extracted with Et2O (3 × 20 mL). The combined organic extracts were washed with brine (2 × 10 mL), dried (Na2SO4), and concentrated in vacuo. The residue was purified by column chromatography (EtOAc–hexane, 4:96).

Yield: 299 mg (70%); colorless oil; [α]20<sub>D</sub> = –17.2 (c 1.00, CHCl3).

IR (neat): 2927, 2856, 1714, 1637, 1600, 1462, 1427, 1379, 1224, 1180, 1111, 821, 702 cm<sup>–1</sup>.

1H NMR (400 MHz, CDCl3): δ = 7.67–7.66 (m, 4 H), 7.43–7.34 (m, 7 H), 6.56 (t, J = 11.28 Hz, 1 H), 6.08 (dd, J = 15.49, 7.18 Hz, 1 H), 5.59 (d, J = 11.28 Hz, 1 H), 4.19 (q, J = 9.58 Hz, 2 H), 3.90–3.87 (m, 1 H), 3.71–3.59 (m, 3 H), 2.40–2.36 (m, 1 H), 1.60–1.56 (m, 2 H), 1.37–1.28 (m, 9 H), 1.05–1.03 (m, 12 H).

13C NMR (50 MHz, CDCl3): δ = 166.49, 149.44, 145.30, 135.68, 135.63, 129.55, 127.55, 127.53, 126.80, 100.25, 69.93, 67.82, 66.71, 59.79, 41.36, 32.14, 26.78, 24.61, 19.24, 15.49, 14.28.

ESI-MS: m/z = 559.3 [M + Na]+, 557.4, 547.4, 537.3.

ESI-HRMS: m/z calc for C₁₉H₂₈O₃NaSi: 559.2856; found: 559.2859.

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