Synthesis of (−)-Malyngolide from D-Lactose

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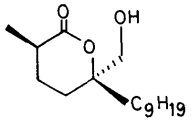
(−)-Malyngolide, which is an antibiotic isolated from a marine blue-green alga, was synthesized starting from D-lactose.

Keywords (−)-malyngolide; antibiotic; D-lactose; tributyltin hydride; radical reaction; Wittig reaction

In the course of our synthetic studies on biologically active natural products,[1] we planned to synthesize frontalin,[2] malyngolide,[3] and vertinolide[4] starting from the common chiral substance, D-lactose. It is available in large quantities and is very cheap. We have already reported the synthesis of (−)-frontalin starting from D-lactose.[5] This paper deals with the synthesis of optically active malyngolide ((−)-**1**)[3] starting from the same chiral substance.

Malyngolide ((−)-**1**) is the major antibiotic in the lipid extract of the shallow water variety of blue-green alga *Lyngbya majuscula Gommont*, and is active against *Mycobacterium smegmatis* and *Streptococcus pyogenes*.[3] Synthesis of malyngolide (**1**) in racemic or optically active forms has already been reported by many research groups.[6] We reported the synthesis of racemic malyngolide (**1**) in 1981.[1a,b]

The synthetic route to malyngolide ((−)-**1**) starting from D-lactose is illustrated in Chart 1. An aldehyde (**2**) was prepared from D-lactose by the known procedure in good yield.[7,8] The absolute sterochemistry at C-2 of **2** is *S*,[8] as required for C-5 of malyngolide ((−)-**1**), when the formyl group is converted to a nonyl group.



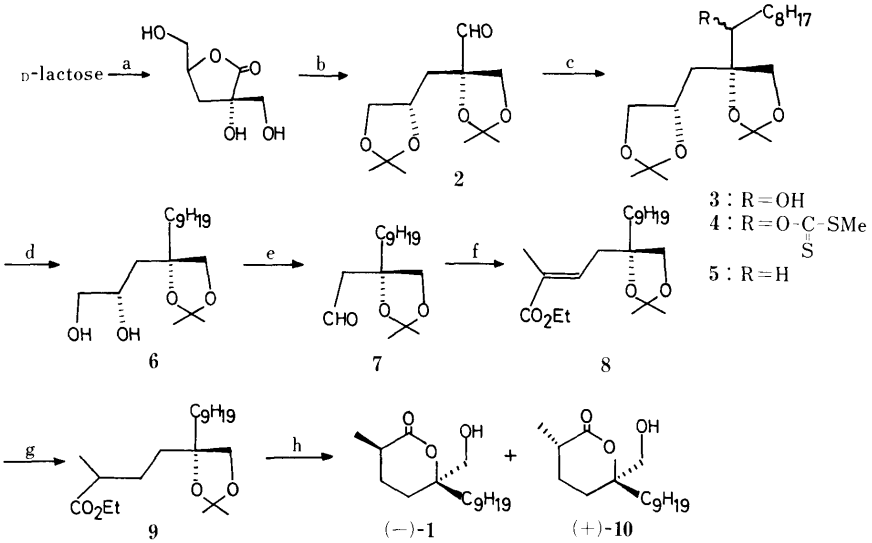
(−)-1

Fig. 1

In order to transform the formyl group of **2** to the nonyl group, Witting reaction of **2** with octylidene triphenylphosphorane, and hydrogenation of the product were thought to be suitable. But the Wittig reaction resulted in recovery of the starting aldehyde (**2**), probably because the formyl group is sterically hindered.

Alternatively, Grignard reaction of **2** with octylmagnesium bromide in ether proceeded smoothly to form an alcohol (**3**) in 88.5% yield. Dehydration of **3** including tosylation and successive base treatment did not give the desired olefin. Therefore, reductive removal of the hydroxy group was examined. The alcohol (**3**) was treated with carbon disulfide in the presence of sodium hydride (NaH) and then with iodomethane to give methyl xanthate (**4**) in 64.5% yield. Redical reaction of **4** with tributyltin hydride in benzene in the presence of 2,2′-azobisisobutyronitrile (AIBN) under reflux gave the desired reduced compound (**5**) in 98.8% yield. When this reaction was performed in toluene, **5** was obtained in 96.1% yield within 1 h.[9]

Selective hydrolysis of one of the acetal groups of **5** with acetic acid in a mixture of methanol and water furnished a glycol (**6**) in 51.5% yield. Oxidative cleavage of the glycol in **6** with sodium metaperiodate in a mixture of methanol and water gave an aldehyde (**7**) in quantitative yield, and



a) ref. 4, b) ref. 5, c) i) $C_8H_{17}MgBr$, ii) NaH, CS_2, MeI, iii) Bu_3SnH, AIBN, d) $AcOH-H_2O$, e) $NaIO_4$, f) $Ph_3P=C(Me)CO_2Et$, g) H_2, Ra-Ni, h) i) $KOH-H_2O$, ii) $HCl-H_2O$.

Chart 1

this was successively treated with ethyl 2-(triphenylphosphoranylidene)propanoate in acetonirile to form an unsaturated ester (**8**) in quantitative yield. Hydrogenation of the ester (**8**) was performed using Raney Ni as a catalyst to give a saturated ester (**9**) as a diastereomeric mixture in quantitative yield. Finally, hydrolysis of the ester group of **9** and successive acidic treatment of the carboxylic acid formed in acetonitrile gave a mixture of malyngolide ((−)-**1**) and 2-epimalyngolide ((+)-**10**), which was readily separated by SiO_2 column chromatography to give pure (−)-**1** and (+)-**10** in 40.1 and 35.7% yields, respectively. Spectral data for the synthesized (−)-**1** were identical with those of the natural (−)-**1**.[1a,b,3] The specific optical rotation of the synthesized (−)-**1** was $[\alpha]_D^{20}$ −12.9° (c=2.0, $CHCl_3$), which is in good accord with the reported value, $[\alpha]_D$ −13° (c=2, $CHCl_3$), for the natural product.[3] We have already reported that the base treatment of racemic 2-epimalyngolide (**10**) gave a recemic mixture of **1** and **10** in a ratio of 9:4.[1a] Therefore, the yield of (−)-**1** might be increased by the base treatment of (+)-**10**.

This synthetic route shows that the chiral aldehyde (**2**), which is easily derived from D-lactose, is a good synthon for the synthesis of optically active malyngolide ((−)-**1**).

Experimental

Melting points were determined on a Yanagimoto micro-melting point apparatus, model MP-S3, and are uncorrected. Infrared (IR) spectra were measured with a Hitachi 260-30 infrared spectrometer, and proton nuclear magnetic resonance (^{1}H-NMR) spectra were recorded on a JEOL JNM-FX200 (200 MHz) spectrometer using tetramethylsilane (TMS) as an internal standard. Mass spectra (MS) were measured with a JEOL JMS-HX 100 instrument. Optical rotations were taken on a JASCO DIP-370 digital polarimeter.

4-[(2,2-Dimethyl-1,3-dioxolan-4-yl)methyl]-α-octyl-2,2-dimethyl-1,3-dioxolane-4-methanol (3) An ether (5 ml) solution of the aldehyde (**2**) (4 g, 16.4 mmol) was added to an ether (100 ml) solution of octylmagnesium bromide [prepared from 1-bromooctane (11.68 g, 60.5 mmol) and Mg (0.982 g, 40.4 mg atom)] with stirring under a nitrogen atmosphere at room temperature. Stirring was continued overnight. Saturated NH_4Cl solution (50 ml) was added to the reaction mixture under ice-cooling, and the resulting solution was extracted with ether. The organic extract was washed with H_2O and saturated brine, and then dried over anhydrous Na_2SO_4. Removal of the solvent gave a pale yellow oil, which was purified by SiO_2 column chromatography (C_6H_6:AcOEt=3:1) to give an alcohol (**3**) (5.194 g, 88.5%) as a colorless oil, bp 174—180 °C (0.4 Torr) (oven temperature), $[\alpha]_D^{20}$ +2.9° (c=1.0, MeOH). IR ν_{max}^{neat} cm^{-1}: 3400, 1055. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, $CH_2C\underline{H}_3$), 1.28 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.35, 1.39, 1.40, 1.43 (each 3H, s, CH_3), 1.74 (1H, dd, J=14, 4 Hz, 3-CH_2), 1.95 (1H, dd, J=14, 4.5 Hz, 3-CH_2), 2.22 (1H, d, J=3 Hz, OH), 3.50 and 4.10 (each 1H, d, J=8 Hz, 4′-CH_2), 3.66 (1H, dd, J=9, 6.5 Hz, 1-CH_2), 4.09 (1H, m, 1-CH_2), 4.3 (1H, m, 2-CH), 4.43 (1H, m, 5-CH). MS Calcd for $C_{20}H_{38}O_5$ m/z: 358.2719. Found: 358.2706.

1,2,4,5-Tetrahydroxy-4-(hydroxymethyl)-5-*O*-[(methylthio)thiocarbonyl]-1,2:4,4′-di-*O*-isopropylidenetridecane (4) A 60% NaH dispersion (50 mg, 1.3 mmol) was added to an anhydrous tetrahydrofuran (THF) (5 ml) solution of the alcohol (**3**) (0.3 g, 0.8 mmol) with stirring under a N_2 atmosphere at room temperature, and the whole was stirred for 30 min. Then CS_2 (0.19 g, 2.5 mmol) was added and the whole was stirred for another 1.5 h. Iodomethane (0.36 g, 2.5 mmol) was added and the reaction mixture was stirred for 1 h. Acetic acid (0.1 ml) was added and the mixture was diluted with ether (30 ml), then washed successively with saturated $NaHCO_3$ solution, H_2O and saturated brine, dried over anhydrous Na_2SO_4, and evaporated to give a yellow oil, which crystallized on standing. Recrystallization from hexane furnished **4** (0.24 g, 64.5%) as a diastereomeric mixture (2:1), mp 50—61 °C. IR ν_{max}^{neat} cm^{-1}: 1220, 1060. ^{1}H-NMR ($CDCl_3$) (major isomer) δ: 0.88 (3H, t, J=7 Hz, $CH_2C\underline{H}_3$), 1.25 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.36—1.42 (12H, m, 4×$C\underline{H}_3$), 1.82 (1H, dd, J=14, 7 Hz, 3-CH_2), 2.11 (1H, dd, J=14, 6 Hz, 3-CH_2), 2.58 (3H, s, SCH_3), 3.5 and 4.12 (each 1H, m, 1-CH_2), 3.80 and 4.10 (each 1H, d, J=9 Hz, 4′-CH_2), 4.36 (1H, m, 2-CH), 6.25 (1H, dd, J=10, 2 Hz, 5-CH). *Anal.* Calcd for $C_{22}H_{40}O_5S$: C, 58.89; H, 8.99. Found: C, 58.61; H, 8.85.

2,2-Dimethyl-4-[(2,2-dimethyl-1,3-dioxolan-4-yl)methyl]-4-nonyl-1,3-dioxolane (5) A solution of the xanthate (**4**) (3.50 g, 7.8 mmol), tributyltin hydride (2.78 g, 9.5 mmol) and AIBN (70 mg) in dry benzene (105 ml) was refluxed for 3 h. After addition of tributyltin hydride (1.39 g, 4.8 mmol) and AIBN (35 mg), the reaction mixture was refluxed for another 30 min. Evaporation of the solvent under reduced pressure gave a pale yellow oil, which was purified by SiO_2 column chromatography (hexane:AcOEt=10:1) to furnish **5** (2.64 g, 98.8%) as a colorless oil, $[\alpha]_D^{18}$ −8.0° (c=1.0, MeOH). IR ν_{max}^{neat} cm^{-1}: 1370, 1250, 1210, 1050. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, $CH_2C\underline{H}_3$), 1.28 (16H, s, $(C\underline{H}_2)_8CH_3$), 1.35 and 1.39 (each 3H, s, CH_3), 1.37 (6H, s, 2×CH_3), 1.80 (1H, dd, J=14, 5.5 Hz, 3-CH_2), 1.91 (1H, dd, J=14, 7 Hz, 3-CH_2), 3.53 (1H, t, J=7.5 Hz, 1-CH_2), 3.80 (2H, s, 4′-CH_2), 4.10 (1H, dd, J=7.5, 6 Hz, 1-CH_2), 4.17 (1H, m, 2-CH). MS Calcd for $C_{19}H_{35}O_4$ (M^+-CH_3) m/z: 327.2535. Found: 327.2527.

β-Hydroxy-2,2-dimethyl-4-nonyl-1,3-dioxolane-4-propanol (6) Water (50 ml) and AcOH (100 ml) were added to a solution of **5** (2.5 g, 7.3 mmol) in MeOH (100 ml), and the whole was stirred at room temperature for 17 h. After addition of 4 N NaOH solution under cooling, the mixture was extracted with AcOEt. The organic layer was washed with saturated brine and dried over anhydrous Na_2SO_4. Removal of the solvent gave a pale yellow oil, which was purified by SiO_2 column chromatography (hexane:AcOEt=1:1) to furnish a diol (**6**) (1.14 g) (51.5%) and recovered **5** (0.74 g). IR ν_{max}^{neat} cm^{-1}: 3400, 1250, 1210, 1050. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, $CH_2C\underline{H}_3$), 1.27 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.42 and 1.43 (each 3H, CH_3), 1.60 (1H, dd, J=14.5, 2.5 Hz, 3-CH_2), 1.68 (2H, m, 5-CH_2), 1.86 (1H, dd, J=14.5, 9.5 Hz, 3-CH_2), 2.26 and 3.32 (each 1H, br s, OH), 3.50 (1H, dd, J=11, 7 Hz, 1-CH_2), 3.64 (1H, dd, J=11, 4 Hz, 1-CH_2), 3.83 (2H, s, 4′-CH_2), 3.94 (1H, m, 2-CH).

2,2-Dimethyl-4-nonyl-1,3-dioxolane-4-acetaldehyde (7) A solution of $NaIO_4$ (0.18 g, 0.8 mmol) in H_2O (3 ml) was added a solution of **6** (0.25 g, 0.8 mmol) in MeOH (3 ml), and the whole was stirred at room temperature for 30 min. The obtained suspension was made alkaline (pH 8) with aqueous $NaHCO_3$ solution and extracted with AcOEt. The organic layer was washed with saturated brine and dried over anhydrous Na_2SO_4. Removal of the solvent left a colorless oil, which was purified by SiO_2 column chromatography (benzene:AcOEt=50:1) to give **7** (0.23 g, quant.) as a colorless oil, bp 130—135 °C (0.7 Torr) (oven temperature), $[\alpha]_D^{19}$ −20.8° (c=1.0, MeOH). IR ν_{max}^{neat} cm^{-1}: 1720, 1250, 1210, 1060. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, $CH_2C\underline{H}_3$), 1.27 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.39 and 1.42 (each 3H, s, 2×CH_3), 1.65 (2H, m, 4-CH_2), 2.61 (1H, dd, J=16, 3 Hz, 2-CH_2), 2.73 (1H, dd, J=16, 2.5 Hz, 2-CH_2), 3.87 (2H, s, 3′-CH_2), 9.82 (1H, t, J=2.5 Hz, CHO). *Anal.* Calcd for $C_{16}H_{30}O_3$: C, 71.07; H, 11.18. Found: C, 71.11; H, 11.32.

Ethyl 2-Methyl-4-(2,2-dimethyl-4-nonyl-1,3-dioxolan-4-yl)-2-butenoate (8) A solution of **7** (0.8 g, 3.0 mmol) and ethyl 2-triphenylphosphoranylidenepropanoate (1.61 g, 4.4 mmol) in CH_3CN (20 ml) was refluxed for 1 h. Removal of the solvent gave a yellow oil, to which ether (20 ml) was added, and the whole was stirred for 1 h. The precipitate was filtered off, and the filtrate was concentrated under reduced pressure to give a yellow oil. Purification of the crude product by SiO_2 column chromatography (benzene:AcOEt=5:1) gave **8** (1.02 g, quant.) as a colorless oil. IR ν_{max}^{neat} cm^{-1}: 1705, 1250, 1060. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, $CH_2CH_2C\underline{H}_3$), 1.26 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.30 (3H, t, J=7 Hz, $OCH_2C\underline{H}_3$), 1.39 and 1.41 (each 3H, s, CCH_3), 1.57 (2H, m, 6-CH_2), 1.88 (3H, d, J=1 Hz, $=CCH_3$), 2.46 (2H, d, J=7 Hz, 4-CH_2), 3.78 (2H, s, 5′-CH_2), 4.20 (2H, q, J=7 Hz, $OC\underline{H}_2CH_3$), 6.80 (1H, dt, J=7, 1 Hz, 3-CH). MS Calcd for $C_{20}H_{35}O_4$ (M^+-CH_3) m/z: 339.2539. Found: 339.2539.

Ethyl 2-Methyl-4-(2,2-dimethyl-4-nonyl-1,3-dioxolan-4-yl)butanoate (9) Raney-Ni (prepared from 2 g of Al-Ni alloy) was added to a solution of **8** (0.80 g, 2.3 mmol) in EtOH (12 ml), and the whole was stirred under a H_2 atmosphere overnight. After removal of the catalyst by filtration, the filtrate was concentrated under reduced pressure to give **9** (0.81 g, quant.) as a colorless oil, $[\alpha]_D^{18}$ −2.0° (c=1.0, MeOH), bp 161—172°C (0.5 Torr) (oven temperature). IR ν_{max}^{neat} cm^{-1}: 1730, 1250, 1210, 1190, 1150, 1060. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J=7 Hz, $CH_2CH_2C\underline{H}_3$), 1.16 (3H, d, J=7 Hz, $CHC\underline{H}_3$), 1.26 (3H, t, J=7 Hz, $OCH_2C\underline{H}_3$), 1.27 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.37 (6H, s, 2×CH_3), 1.4—1.7 (6H, m, 3-, 4- and 6-CH_2), 2.40 (1H, m, 2-CH), 3.73 (2H, s, 5′-CH_2), 4.13 (2H, q, J=7 Hz, $OC\underline{H}_2CH_3$). *Anal.* Calcd for $C_{21}H_{40}O_4$: C, 70.74; H, 11.31. Found: C, 70.51; H, 11.18.

Malyngolide ((−)-1) and 2-Epimalyngolide ((+)-10) A solution of KOH (3.86 g, 68.9 mmol) in H_2O (14 ml) was added to a solution of **9** (0.50 g, 1.5 mmol) in EtOH (14 ml), and the whole was stirred at room

temperature overnight. The solution was acidified with 6 N HCl (pH 3) under ice-cooling and stirred for 1 h. After addition of NaCl, the whole was extracted with AcOEt and the organic layer was washed with saturated brine. The organic extracts were dried over anhydrous Na_2SO_4 and the solvent was removed under reduced pressure to give an oil, which was diluted with CH_3CN (15 ml). Amberlyst 15 (0.1 g) was added and the whole was stirred at room temperature for 1 h and then filtered. The filtrate was concentrated under reduced pressure to leave a pale yellow oil, which was diluted with ether (20 ml). The solution was washed successively with saturated $NaHCO_3$ solution and saturated brine, dried over anhydrous Na_2SO_4, and concentrated to furnish a pale yellow oil (0.377 g). The oil (0.327 g) was purified by SiO_2 column chromatography ($CHCl_3$: acetone = 4 : 1) to give (+)-**10** (0.122 g, 35.7%) and (−)-**1** (0.136 g, 40.1%) as colorless oils in that order of elution.

(−)-**1**: $[\alpha]_D^{20}$ −12.9° (c = 2.0, $CHCl_3$) (lit.[3] $[\alpha]_D$ −13°, c = 2, $CHCl_3$). IR ν_{max}^{neat} cm^{-1}: 3400, 1720, 1705, 1690, 1245, 1210, 1060. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J = 7 Hz, $CH_2C\underline{H}_3$), 1.26 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.28 (3H, J = 7 Hz, $CHC\underline{H}_3$), 1.51—1.84 (4H, m, 3- and 4-CH_2), 1.95 (2H, m, $CC\underline{H}_2CH_2$), 2.4 (1H, m, OH), 2.45 (1H, m, $C\underline{H}CH_3$), 3.47 (1H, dd, J = 12, 7 Hz, $C\underline{H}_2OH$), 3.66 (1H, dd, J = 12, 6 Hz, $C\underline{H}_2OH$). MS Calcd for $C_{15}H_{27}O_2$ ($M^+ - CH_2OH$) m/z: 239.2011. Found; 239.1995.[3]

(+)-**10**: $[\alpha]_D^{20}$ +18.5° (c = 2.0, $CHCl_3$). IR ν_{max}^{neat} cm^{-1}: 3400, 1725, 1710, 1220, 1090. ^{1}H-NMR ($CDCl_3$) δ: 0.88 (3H, t, J = 7 Hz, $CH_2C\underline{H}_3$), 1.28 (14H, s, $(C\underline{H}_2)_7CH_3$), 1.29 (3H, d, 7 Hz, $CHC\underline{H}_3$), 1.7 (4H, m, 3- and 4-CH_2), 1.95 (3H, m, $CC\underline{H}_2CH_2$ and OH), 2.45 (1H, m, $C\underline{H}CH_3$), 3.61 (2H, d, J = 6.5 Hz, $C\underline{H}_2OH$).

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