HETEROCYCLES, Vol. 97, No. 2, 2018, pp. 1019 - 1027. © 2018 The Japan Institute of Heterocyclic Chemistry
Received, 2nd March, 2018, Accepted, 22nd March, 2018, Published online, 12th April, 2018
DOI: 10.3987/COM-18-S(T)84

TOTAL SYNTHESIS OF LISSOCLINOLIDE BY ACID-INDUCED LACTONIZATION OF AN (*E*)-α-BROMO-γ,δ-EPOXY ACRYLATE DERIVATIVE

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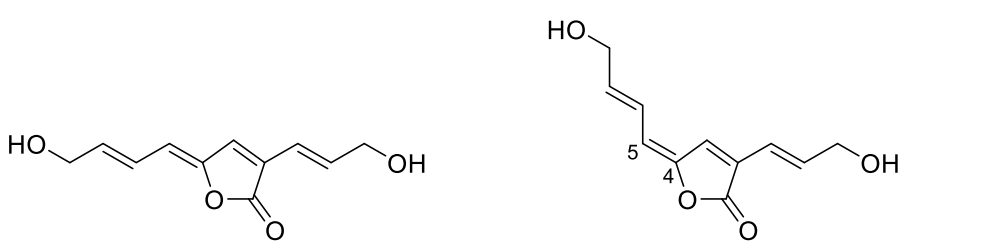
This paper is dedicated to Professor Kiyoshi Tomioka on the occasion of his 70th birthday.

Abstract – The stereoselective total synthesis of lissoclinolide, a naturally occurring antibiotic and cytotoxic butenolide, was achieved in 10 steps including a highly *E*-selective Still–Gennari-type olefination and an acid-induced lactonization of an (*E*)-α-bromo-γ,δ-epoxy acrylate derivative. The key regioselective 5-*exo* lactonization could be regulated by using AcOH under kinetically controlled conditions.

INTRODUCTION

Lissoclinolide (**1**) was first isolated from *Lissoclinum patella* and structurally elucidated by Ireland and co-workers in 1990 and was reported to exhibit slight activity against the Gram-negative bacterium



lissoclinolide (**1**) tetrenolin (**2**, originally proposed structure)

Figure 1. Structures of lissoclinolide (**1**) and that originally proposed for tetrenolin (**2**)

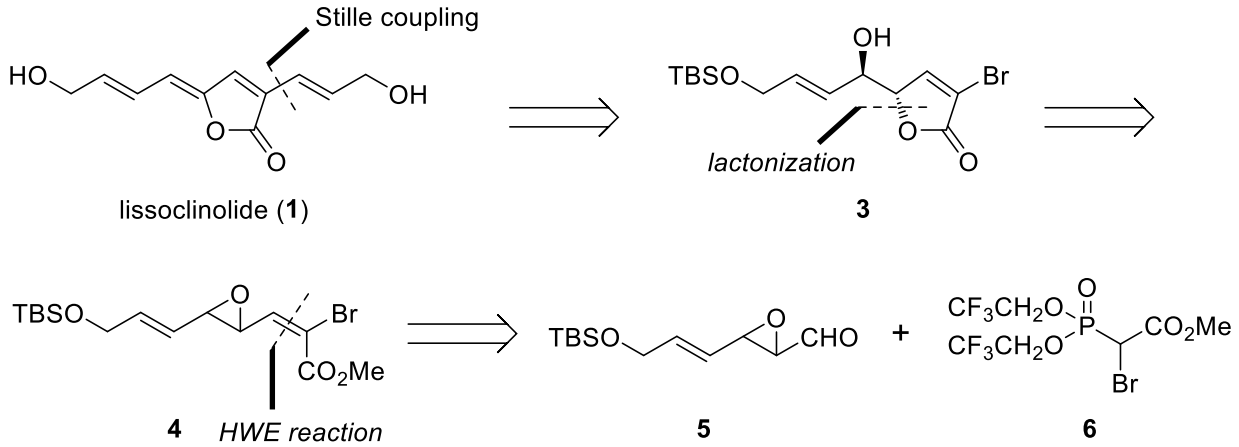
Escherichia coli.[1] In 2005, Görth and Brückner concluded that tetrenolin (**2**), originally assigned as a geometric isomer ($\Delta^{4,5}$) of lissoclinolide, was actually the same compound, and they also biologically re-evaluated **1**, revealing that it possesses activity against not only Gram-negative but also Gram-positive bacteria (Figure 1).

The simple and unique structure of **1**, which possesses a tetraene moiety incorporating a lactone ring, renders this biologically promising natural product an attractive synthetic target. To date, several successful total syntheses of **1** have been reported in the literature, including those by Rossi and Bellina in 1998,[3] Negishi in 1999,[4] and Brückner in 1999 and 2005.[2,5] In addition, recent studies by Ireland and co-workers demonstrated the inhibitory effect of **1** on tumor cell growth for potential anticancer therapy,[6] thereby generating further interest from the synthetic and biological communities.

Captivated by both the interesting structure and promising biological activities of **1**, we embarked upon its total synthesis using our original Still–Gennari-type phosphonate reagent[7] to produce the key substrate for the characteristic butenolide portion. Herein, we report on the stereoselective total synthesis of **1** via the regioselective lactonization of an intermediate (*E*)-α-bromo-γ,δ-epoxy acrylate derivative.

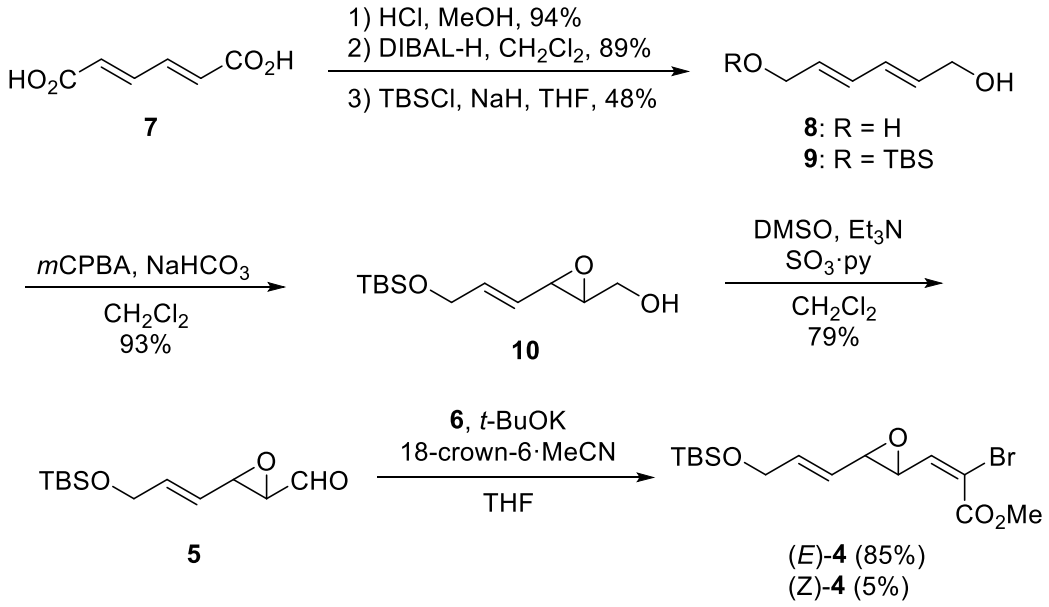
RESULTS AND DISCUSSION

Our retrosynthetic analysis of **1** is depicted in Scheme 1. According to previous reports,[2,3,5] **1** can be obtained from α-bromobutenolide **3** by Stille coupling with a known (alkenyl)tributylstannane.[8] The construction of the butenolide portion of **3** was expected to be achievable through the regioselective 5-*exo* cyclization[9–12] of the (*E*)-α-bromo-γ,δ-epoxy acrylate derivative **4**, which was retrosynthetically transformed to the known epoxy aldehyde **5**[13] by an *E*-selective Horner–Wadsworth–Emmons (HWE) reaction with our original Still–Gennari-type phosphonate **6**.[7]



Scheme 1. Retrosynthetic analysis of lissoclinolide (**1**)

Our synthesis commenced with the preparation of epoxy aldehyde **5** via the previously reported protocol[13] with slight modifications, involving methyl esterification of the commercially available *trans*,*trans*-muconic acid (**7**) (94%), DIBAL reduction (**8**, 89%), mono-TBS protection (**9**, 48%), epoxidation with *m*CPBA (**10**, 93%), and Parikh–Doering oxidation (**5**, 79%), as presented in Scheme 2. Based on our previous reports,[7] subsequent HWE olefination of aldehyde **5** with the bromine-containing phosphonate reagent **6**[7] was carried out under the optimized conditions using *t*-BuOK and 18-crown-6 to stereoselectively give the required unsaturated ester (*E*)-**4** in 85% yield alongside its isomer (*Z*)-**4** in 5% yield. The configurations of (*E*)- and (*Z*)-**4** were determined by ^{1}H NMR analysis.

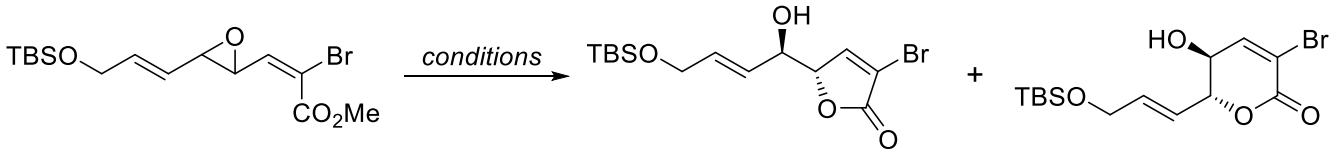


Scheme 2. Preparation of (*E*)-α-bromo-γ,δ-epoxy acrylate derivative (*E*)-**4**

The crucial acid-induced lactonization of (*E*)-**4** was next investigated and the results are summarized in Table 1. Both protic acids, such as $HClO_4$ (entry 1),[9] H_2SO_4 (entry 2),[10] and TFA (entry 3),[11] and the Lewis acid TMSOTf (entry 4) afforded complex mixtures of products. These results were presumably attributable to the decomposition of the starting material (*E*)-**4** and/or the products owing to the strong acids used. Thus, weak acids were next evaluated (entries 5–10). To our delight, excess HCO_2H in CH_2Cl_2 preferentially gave rise to the desired butenolide **3** via the expected 5-*exo* cyclization over 6-*endo* cyclization (entry 5),[14] and the use of AcOH in CH_2Cl_2 led to significantly improved 5-*exo*/6-*endo* selectivity (entry 6). With this good result in hand, AcOH was employed as the acid in other solvents such as toluene (entry 7) and THF (entry 8), but both the yield and product ratio were found to decrease. The use of AcOH in the absence of solvent clearly diminished the regioselectivity (entry 9), whereas the use of excess AcOH and one equivalent of H_2O led to a significantly improved yield at the expense of a less

desirable product ratio (entry 10). Corrêa et al. recently demonstrated a successful 5-*exo* cyclization using montmorillonite K10 (MK-10) as an acid catalyst in EtOH;[12] however, applying this method to our substrate (*E*)-**4** unfortunately afforded a modest yield of the products **3** and **11** in a ratio of 1.5:1 (entry 11). As we were intrigued by the 5-*exo* versus 6-*endo* cyclization, the six-membered lactone **11** was treated with AcOH to examine the reaction pathway. The starting lactone was not converted to the five-membered lactone **3** during the reaction, which implies that the cyclization reaction in the presence of AcOH was kinetically controlled to preferentially provide butenolide **3**.

Table 1. Acid-induced lactonization



(*E*)-**4** **3** **11**

Entry	Conditions	Result	**3**:**11**[b]
1	$HClO_4$, 1,4-dioxane	complex mixture	–
2	H_2SO_4, acetone	complex mixture	–
3	TFA, CH_2Cl_2	complex mixture	–
4	TMSOTf, toluene	complex mixture	–
5	HCO_2H (12 eq), CH_2Cl_2	44%[a]	4:1
6	AcOH (12 eq), CH_2Cl_2	51%[a]	8.5:1
7	AcOH (12 eq), toluene	35%[a]	7.7:1
8	AcOH (12 eq), THF	36%[a]	5.1:1
9	AcOH	46%[a]	3.5:1
10	AcOH, H_2O (1 eq)	93%[a,c]	4.2:1
11	MK-10, EtOH	32%[a]	1.5:1

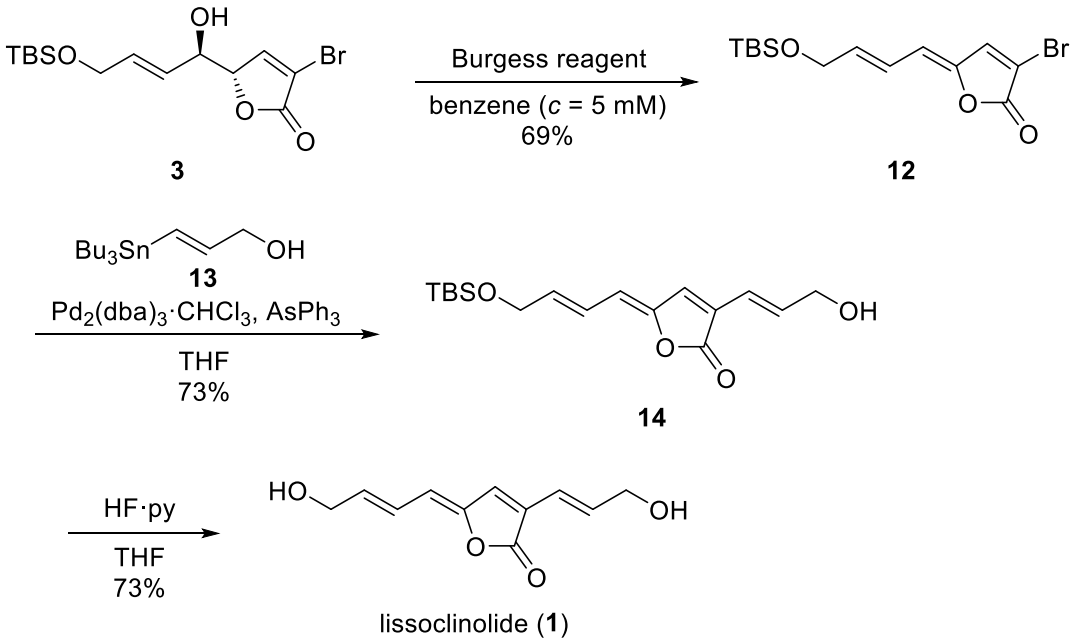
[a]Combined yield of **3** and **11**.

[b]The ratio was determined by ^{1}H NMR analysis of a mixture of **3** and **11**.

[c]Compounds **3** and **11** were obtained in yields of 75% and 18%, respectively, after separation by column chromatography.

Following the stereoselective preparation of the requisite α-bromobutenolide **3**, the total synthesis of **1** was completed as follows (Scheme 3). The stereoselective dehydration of **3** was achieved using Burgess reagent[15] in benzene under highly dilute conditions (5 mM)[16] to yield the γ-alkylidenebutenolide **12** in good yield, despite the fact that initial attempts using $POCl_3$/pyridine[17] and Martin sulfurane[18] led to low

product yield and poor *E*/*Z* selectivity. Finally, Stille coupling between bromide **12** and *E*-configured alkenyl stannane (**13**) (73%) and subsequent deprotection of the TBS group in **14** using HF·py furnished the target compound lissoclinolide (**1**) (73%). All of the spectral data for the synthetic compound were fully identical to those of natural lissoclinolide.[1]



Scheme 3. Completion of the total synthesis of lissoclinolide (**1**)

In conclusion, we have accomplished the total synthesis of the butenolide-containing natural product lissoclinolide (**1**) in ten steps, including a highly *E*-selective Still–Gennari-type olefination and 5-*exo* cyclization promoted by AcOH. The noteworthy 5-*exo* and 6-*endo* cyclizations could be applicable to the synthesis of natural products containing five- or six-membered lactone rings. Synthetic studies toward these natural products, including biverlactones A–D,[19] are ongoing projects in our laboratory and the results will be reported in due course.

EXPERIMENTAL

General: ^{1}H NMR spectra were measured in $CDCl_3$ or CD_3OD solution on JEOL JNM-AL300 (300 MHz), JEOL JNM-AL500 (500 MHz), or JEOL ECX-400 (400 MHz) spectrometers. The ^{1}H chemical shifts are expressed in ppm relative to tetramethylsilane (δ = 0.00); this was either added as an internal standard or the residual $CHCl_3$ peak (δ = 7.26) was used as a reference. The splitting patterns are indicated as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad peak. ^{13}C NMR spectra were measured at 125 MHz or 100 MHz. The ^{13}C chemical shifts are reported in ppm relative to tetramethylsilane (δ = 0.0) or the central line of the $CDCl_3$ triplet at 77.0 ppm. Infrared spectra (IR) were

measured on a JASCO VALOR-III spectrophotometer and are reported as wavenumbers (cm^{-1}). High-resolution mass spectra (HRMS) were obtained using a JEOL JMS-700 double-focusing mass spectrometer in either the electron ionization or fast atom bombardment mode using a direct inlet system for sample introduction. Column chromatography was performed on silica gel (40–100 mesh). Analytical TLC was performed using 0.25 mm silica gel 60F plates.

Methyl (*E*)-2-bromo-3-((2*R,3*R**)-3-((*E*)-3-((*tert*-butyldimethylsilyl)oxy)prop-1-en-1-yl)oxiran-2yl)-acrylate [(*E*)-4] and (*Z*)-2-bromo-3-((2*R**,3*R**)-3-((*E*)-3-((*tert*-butyldimethylsilyl)oxy)prop-1-en-1-yl)oxiran-2-yl)acrylate [(*Z*)-4]**

Phosphonate reagent **6** (1.11 g, 2.80 mmol) and 18-crown-6·MeCN (977 mg, 3.20 mmol) were dissolved in THF (20 mL) and the solution was stirred at −78 °C. *t*-BuOK (1.0 M solution in THF, 2.80 mL, 2.80 mmol) was slowly added into the solution over 5 min. After stirring for 40 min, aldehyde **5** (494 mg, 2.04 mmol) in THF (10 mL) was added dropwise over 20 min. After stirring for 1 h at the same temperature, the reaction was quenched by the addition of saturated aqueous NH_4Cl solution, and the resulting mixture was extracted three times with EtOAc. The combined organic layers were washed with water and brine, dried over Na_2SO_4, filtered, and concentrated in vacuo. The crude residue was purified by flash chromatography on silica gel (19:1→9:1 hexane–EtOAc) to afford (*E*)-**4** (655 mg, 85% yield) and (*Z*)-**4** (41 mg, 5% yield) as colorless oils.

(*E*)-4: IR ($CHCl_3$) 2932, 1719, 1257, 1124, 838 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.07 (6H, s), 0.91 (9H, s), 3.38 (1H, dd, *J* = 7.7, 1.8 Hz), 3.86 (3H, s), 4.19 (1H, d, *J* = 2.0 Hz), 4.21 (1H, d, *J* = 2.6 Hz), 4.22 (1H, d, *J* = 2.0 Hz), 5.52 (1H, ddt, *J* = 15.4, 7.7, 1.8 Hz), 6.05 (1H, dt, *J* = 15.4, 4.4 Hz), 6.31 (1H, d, *J* = 8.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.3, –5.3, 18.4, 25.9 (3C), 53.3, 57.0, 59.1, 62.7, 114.9, 125.0, 136.1, 145.8, 162.8; HRMS (EI-DFMS) *m/z* $[M]^+$ Calcd for $C_{15}H_{25}BrO_4Si$ 376.0705, found 376.0708.

(*Z*)-4: IR (neat) 2928, 1736, 1263, 1106, 837 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.08 (6H, s), 0.92 (9H, s), 3.49 (1H, dd, *J* = 7.4, 1.4 Hz), 3.73 (1H, dd, *J* = 8.0, 1.6 Hz), 3.85 (3H, s), 4.23 (2H, dd, *J* = 4.4, 2.0 Hz), 5.55 (1H, ddt, *J* = 15.6, 7.4, 2.0 Hz), 6.07 (1H, dt, *J* = 15.6, 4.4 Hz), 6.94 (1H, d, *J* = 8.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.3, –5.3, 18.4, 25.9 (3C), 53.6, 58.7, 59.3 62.7, 118.3, 124.8, 136.3, 142.0, 162.1; HRMS (EI-DFMS) *m/z* $[M]^+$ Calcd for $C_{15}H_{25}BrO_4Si$ 376.0705, found 376.0709.

(*S)-3-Bromo-5-((*R**,*E*)-4-((*tert*-butyldimethylsilyl)oxy)-1-hydroxybut-2-en-1-yl)furan-2(5*H*)-one (3) and (5*S**,6*R**)-3-Bromo-6-((*E*)-3-((*tert*-butyldimethylsilyl)oxy)prop-1-en-1-yl)-5-hydroxy-5,6-dihydro-2*H*-pyran-2-one (11)**

H_2O (1.0 M in AcOH, 0.13 mL, 0.13 mmol) and AcOH (1.2 mL) were successively added to (*E*)-**4** (51 mg, 0.13 mmol). The resulting solution was stirred at room temperature for 2 h, diluted with toluene, and concentrated in vacuo. The crude residue was purified by flash chromatography on silica gel (12:1 toluene–EtOAc) to afford **3** (36 mg, 75% yield) and **11** (8.6 mg, 18% yield) as colorless oils.

3: IR ($CHCl_3$) 3604, 2931, 2858, 1777, 1471, 1258, 1130, 838 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.08 (6H, s), 0.92 (9H, s), 1.94 (1H, d, *J* = 4.8 Hz), 4.23 (2H, dt, *J* = 4.0, 1.8 Hz), 4.46-4.53 (1H, m), 4.94 (1H, dd, *J* = 4.6, 1.8 Hz), 5.78 (1H, ddt, *J* = 15.4, 5.9, 1.8 Hz), 6.00 (1H, dtd, *J* = 15.3, 4.0, 1.3 Hz), 7.53 (1H, d, *J* = 1.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.3, –5.3, 18.4, 25.9 (3C), 62.6, 71.3, 84.8, 114.2, 124.9, 134.3, 142.7, 168.3; HRMS (FAB-DFMS) *m/z* $[M+H]^+$ Calcd for $C_{14}H_{24}BrO_4Si$ 362.0627, found 363.0625.

11: IR (neat) 3446, 2929, 2856, 1741, 1471, 1256, 1104, 837 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 0.08 (6H, s), 0.91 (9H, s), 2.23 (1H, d, *J* = 5.4 Hz), 4.22-4.26 (2H, m), 4.38 (1H, ddd, *J* = 8.3, 5.4, 2.9 Hz), 4.82 (1H, t, *J* = 7.6 Hz), 5.82 (1H, ddt, *J* = 15.1, 7.6, 2.0 Hz), 6.05 (1H, dt, *J* = 15.1, 3.9 Hz), 7.22 (1H, d, *J* = 2.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.4, –5.3, 18.4, 25.9 (3C), 62.3, 68.0, 83.3, 114.6, 122.5, 137.3, 147.3, 158.5; HRMS (FAB-DFMS) *m/z* $[M+H]^+$ Calcd for $C_{14}H_{24}BrO_4Si$ 362.0627, found 363.0629.

(*Z*)-3-Bromo-5-((*E*)-4-((*tert*-butyldimethylsilyl)oxy)but-2-en-1-ylidene)furan-2(5*H*)-one (12)

In a glove box, a solution of Burgess reagent (10.1 mg, 0.0425 mmol) in benzene (3.0 mL) was added to a solution of **3** (10.3 mg, 0.0283 mmol) in benzene (2.5 mL). The resulting solution was stirred at room temperature for 1 h and then heated to 50 °C and stirred for an additional 15 h. The reaction was then quenched by the addition of water. The organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo. The crude residue was purified by flash chromatography on silica gel (10:1 hexane–EtOAc) to afford **12** (6.8 mg, 69% yield) as a yellow oil. IR ($CHCl_3$) 2957, 2931, 2858, 1767, 1471, 1259, 1127, 838, cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.09 (6H, s), 0.93 (9H, s), 4.31 (2H, dd, *J* = 4.8, 1.6 Hz), 5.90 (1H, d, *J* = 11.2 Hz), 6.18 (1H, dt, *J* =15.2, 4.8 Hz), 6.76 (1H, ddt, *J* = 15.6, 11.6, 2.0 Hz), 7.47 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.3, –5.3, 18.4, 25.9 (3C), 63.6, 111.6, 114.8, 121.6, 141.1, 141.4, 146.6, 165.1; HRMS (FAB-DFMS) *m/z* $[M+Na]^+$ Calcd for $C_{14}H_{21}BrNaO_3Si$ 367.0341, found 363.0340.

(*Z*)-5-((*E*)-4-((*tert*-Butyldimethylsilyl)oxy)but-2-en-1-ylidene)-3-((*E*)-3-hydroxyprop-1-en-1-yl)furan-2(5*H*)-one (14)

To a stirred solution of $AsPh_3$ (3.2 mg, 0.010 mmol) and $Pd_2(dba)_3 \cdot CHCl_3$ (1.5 mg, 0.0014 mmol) in THF (0.2 mL) were added a solution of **12** (24.0 mg, 0.070 mmol) in THF (0.5 mL) and a solution of

(*E*)-3-(tributylstannyl)prop-2-en-1-ol (**13**) (29.3 mg, 0.084 mmol) in THF (0.4 mL) at room temperature. After stirring for 4 h at the same temperature, water (5 mL) was added to the reaction mixture and the aqueous layer was extracted four times with Et_2O. To the combined organic layers were added activated carbon (10.6 mg) and Na_2SO_4, and the resulting mixture was filtered through celite and concentrated in vacuo. The crude residue was purified by flash chromatography on silica gel (7:3→13:7→3:2 hexane–EtOAc) to afford **14** (16.4 mg, 73% yield) as a yellow oil. IR ($CHCl_3$) 2957, 2931, 2857, 1753, 1471, 1258, 838 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.09 (6H, s), 0.93 (9H, s), 4.32 (2H, dd, *J* = 5.2, 1.6 Hz), 4.36 (2H, dd, *J* = 4.4, 0.8 Hz), 5.81 (1H, d, *J* = 11.6 Hz), 6.07 (1H, dt, *J* = 15.6, 5.2 Hz), 6.47 (1H, dt, *J* = 15.6, 2.0 Hz), 6.78 (1H, ddt, *J* = 15.6, 11.6, 2.0 Hz), 6.85 (1H, dt, *J* = 16.0, 4.8 Hz), 7.07 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ –5.2, –5.2, 18.4, 25.3 (3C), 63.1, 63.5, 113.0, 118.4, 122.7, 127.5, 134.6, 137.0, 139.2, 147.4, 168.2; HRMS (EI-DFMS) *m/z* $[M]^+$ Calcd for $C_{17}H_{26}O_4Si$ 322.1600, found 322.1604.

Lissoclinolide (1)

To a stirred solution of **14** (14.8 mg, 0.046 mmol) in THF (0.6 mL) was added HF·pyridine [HF ≥ 70%, pyridine ≤ 30% in THF (1:4), 0.1 mL] at 0 °C. After stirring for 24 h, the reaction was poured into a saturated aqueous solution of $NaHCO_3$ (1.5 mL). After stirring for 30 min, the whole mixture was extracted six times with $CHCl_3$. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated in vacuo. The crude residue was purified by flash chromatography on silica gel (23:2→9:1 $CHCl_3$–MeOH) to afford **1** (7.0 mg, 73% yield) as a yellow solid (mp 121.5–124.5 °C). IR (neat) 3256, 2921, 2860, 1747, 1329, 1300, 1049 cm^{-1}; ^1H NMR (400 MHz, CD_3OD) δ 4.21 (2H, dd, *J* = 5.4, 1.5 Hz), 4.23 (2H, dd, *J* = 4.9, 1.5 Hz), 6.00 (1H, d, *J* = 11.2 Hz), 6.18 (1H, dt, *J* = 15.4, 5.4 Hz), 6.46 (1H, dt, *J* = 16.1, 2.0 Hz), 6.77 (1H, ddt, *J* = 15.1, 11.2, 1.5 Hz), 6.92 (1H, dt, *J* = 16.1, 4.9 Hz), 7.38 (1H, s); ^{13}C NMR (100 MHz, CD_3OD) δ 61.1, 63.2, 114.7, 119.2, 123.9, 128.7, 136.4, 138.7, 140.5, 149.1, 169.9; HRMS (EI-DFMS) *m/z* $[M]^+$ Calcd for $C_{11}H_{12}O_4$ 208.0736, found 208.0735.

ACKNOWLEDGEMENTS

This work was supported by JSPS KAKENHI Grant Number 17K08227 and partially by a grant from the Dementia Drug Resource Development Center, Project S1511016, the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

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