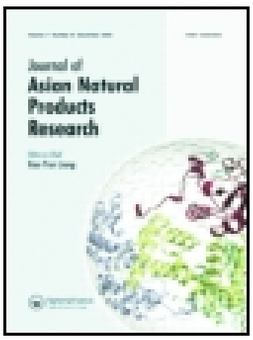

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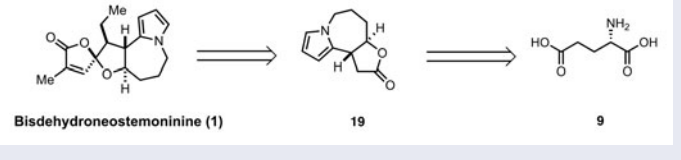
Formal enantioselective total synthesis of bisdehydroneostemoninine

Kai-Qing Ma[a], Hu-Bin Ren[a,b], Jian-Bin Chao[c] and Xue-Mei Qin[a]

[a]Modern Research Center for Traditional Chinese Medicine, Shanxi University, Taiyuan 030006, China;
[b]College of Chemistry and Chemical Engineering, Shanxi University, Taiyuan 030006, China;
[c]Scientific Instrument Center, Shanxi University, Taiyuan 030006, China

ABSTRACT

A formal enantioselective total synthesis of bisdehydroneostemoninine employing L-glutamic acid as the chiral pool is described. The key features of the synthesis include regioselective and enantioselective opening the chiral epoxide with dimethylsulfonium methylide and tandem Friedel–Crafts cyclization followed by lactonization to form the 5-7-5 tricyclic core of the target stemona alkaloids. The synthetic route provides opportunities to explore the biological behavior of enantiopure bisdehydroneostemoninine.



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1. Introduction

The Stemona alkaloids constitute a family of natural products featuring a characteristic pyrrolo[1, 2-a]azepine nucleus, which are categorized into eight different groups [1–3]. Within this family, rich sources of complex natural products have been shown to possess a variety of biological activity, such as antitumor, antitussive, anthelmintic, and insecticidal effects [4–7]. Bisdehydroneostemoninine (**1**) (Figure 1) was isolated from the roots of *Stemona tuberosa* in 2006 [8] and is a representative member of the stemoamide group.

In the previous study, we have completed the first total synthesis of bisdehydrostemoninine and bisdehydroneostemoninine in racemic forms employing palladium-catalyzed carbonylative spirolactonization as the key step [9]. In addition to the synthetic challenges posed by the oxaspirolactone moiety, constructing bisdehydroneostemoninine in enantiopure form is nontrivial as well and has not been reported

CONTACT Kai-Qing Ma makaiqing@sxu.edu.cn
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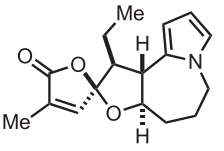


Bisdehydroneostemoninine (1)

Figure 1. The structure of bisdehydroneostemoninine (**1**).

so far. We envisioned that the chiral allylic alcohol **5** could serve as a common precursor for the enantioselective synthesis of bisdehydroneostemoninine and could be prepared from asymmetric reduction of the corresponding enone **4**. The existence of a highly nucleophilic enone moiety and the terminal alkene in the substrate **4** generates significant synthetic challenge in control of the stereochemistry. Herein, we report the formal enantioselective syntheses of bisdehydroneostemoninine employing L-glutamic acid as chiral pool.

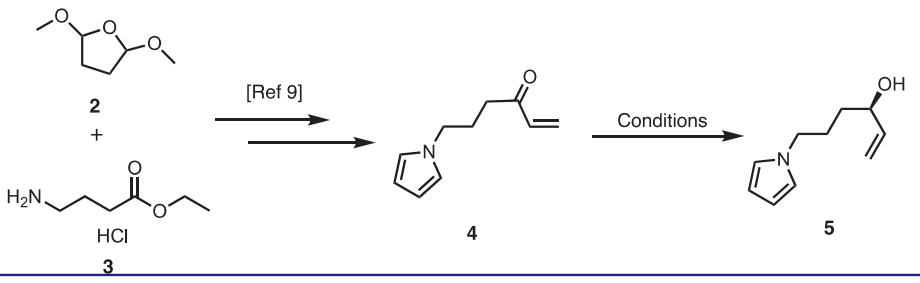
2. Result and discussion

Our initial attempt at the synthesis of the desired allylic alcohol **5** began with the enone compound **4**, which was prepared from 2, 5-dimethoxytetrahydrofuran (**2**) and ethyl 4-aminobutanoate hydrogen chloride salts (**3**) in three steps [9]. We assumed that the allylic alcohol could be accessed through Corey–Itsuno asymmetric reduction [10]. Unfortunately, the enone **4** treated with the (*S*)-2-Methyl-CBS-oxazaborolidine and borane dimethylsulfide under various conditions failed to afford any useful level of enantioselectivity (determined by ^{1}H-NMR analysis of the ester derived from the (*R*)-Mosher acid) (Scheme 1). These failures were presumably due to the alkene moiety, which could not provide enough steric interactions between the oxazaborolidine and the substrate for discrimination [11]. BH_3Et_2NPh, instead of borane dimethylsulfide, was also evaluated but led only to the 1, 4-reduction by-product [12]. We also investigated the possibility of employing (+)-diisopinocampheyl chloroborane as the reducing reagent [13]. Again, this method was not fruitful which was probably due to the instability of the enone **4** under the conditions. We also examined the feasibility of enzymatic kinetic resolution (EKR) of compound **5** employing *Candida antarctica* lipase B and vinyl acetate as an acylating agent. As a result, compound **5** was almost recovered.

When our synthetic endeavors toward compound **5** using the enone **4** failed, we then turned our attention to the intermediate **6**, which could be prepared from enone **4** in two steps (Scheme 2) [9]. We assumed that compound **7** with substitution on the double bond could potentially be the proper substrate for the Corey–Itsuno asymmetric reduction to obtain useful level of enantioselectivity. Unfortunately, after extensive screening of the oxidation conditions, compound **6** was consumed and desired product **7** could not be observed, which are presumably due to the instability of compound **7**.

In view of these results, a revised strategy was required for the enantioselective synthesis. We queried whether we might be able to achieve an enantioselective synthesis of the key intermediate **5** using a single, enantiomerically pure starting

Table 1. Conversion of enone 4 to the allylic alcohol 5.



Entry	Conditions(equiv)	T(°C)/time(h)	Yield	ee
1	(S)-CBS-Me (2 equ), BH_3-SMe_2 (5 equ), THF	–40 °C/1.5 h	64%	76%
2	(S)-CBS-Me (0.2 equ), BH_3-SMe_2 (5 equ), THF	–40 °C/7 h	62%	32%
3	(S)-CBS-Me (0.2 equ), BH_3-SMe_2 (2 equ), THF	−40 °C/7 h	60%	65%
4	(S)-CBS-Me (0.25 equ), BH_3-Et_2NPh (1 equ), THF	0 °C/3 h	NA	NA
5	(+)-DIP-Chloride (2 equ),THF	rt/1 h	NA	NA

material. Accordingly, a new approach was envisaged using the L-glutamic acid as the chiral pool (Scheme 3). Treated with HBr, $NaNO_2$, and KBr, the amino group of L-glutamic acid was converted to the corresponding bromide **10** with retention of configuration *via* a diazonium salt-mediated double inversion strategy [14]. Subsequently, two carboxylic acid groups were reduced with borane to provide a diol **11** in a 60% two-step yield. The diol was then subjected to NaH-promoted epoxidation and *in situ* esterification of remaining hydroxyl group with 4-toluenesulfonyl chloride (TsCl) to afford the epoxide **12**. The epoxide ring opening of compound **12** to the allyl secondary alcohol proves problematic [15]. Compound **12**, treated with dimethylsulfonium methylide, failed to furnish the desired allylic alcohol, which are presumably due to the toluenesulfonate moiety in the structure.

We then went back one step and protected the alcohol with tert-butyldimethylsilyl to afford the epoxide **14**. Gratifyingly, the epoxide ring of **14** was treated with the dimethylsulfonium methylide to give the allyl secondary alcohol **15** in 48% yield for two steps. The resulting secondary alcohol was protected with triisopropylsilyl chloride to afford the product **16**. The diprotected compound **16** was selectively deprotected to afford the primary alcohol, which reacted with TsCl to furnish the corresponding *p*-toluenesulfonate **17** in one pot. The pyrrole efficiently reacted with the toluenesulfonate **17**, followed by deprotection to give the key intermediate **5** in enantiopure form (ee $>$ 95%) [16]. The *ee* value was determined by ^{1}H-NMR analysis of the ester derived from the (*R*)-Mosher acid (see the supporting information). The α, β-unsaturated ester **8** was afforded in 91% yield via a cross-metathesis reaction between allylic alcohol **5** and methyl acrylate [17]. The azepine ring as well as the transfused γ-butyrolactone ring of compound **19** was constructed through a boron trifluoride etherate promoted tandem Friedel–Crafts cyclization and lactonization. Accordingly, preparation of enantiomerically pure **19** completes the formal enantioselective synthesis of **1**.

In summary, we have completed the formal enantioselective total synthesis of the stemona alkaloids bisdehydroneostemoninine (**1**), employing the L-glutamic acid as

the chiral pool. The key intermediate **19** was synthesized through nine steps, which could serve to access the enantiopure bisdehydroneostemoninine in five steps and other stemoamide group natural products. Further studies focusing on the biological evaluation of enantiopure bisdehydroneostemoninine and its derivatives are in progress and will be reported in due course.

3. Experimental

3.1. General experimental procedures

All reactions were carried out under an argon atmosphere with dry solvents. All reagents were obtained from commercial sources and used without further purification. NMR spectra were recorded on Bruker AV-600 spectrometers (Bruker Co. Billerica, USA) at ambient temperature, using TMS as an internal standard. High-resolution mass spectra (HRMS) were recorded at a Mass Spectrometry Laboratory using a Thermo Scientific Q Exactive (Thermo Fisher Scientific Inc. Waltham, USA). The value of optical rotation was recorded at AUTOPOL IV (Rudolph Research Analytical Inc. Hackettstown, USA).

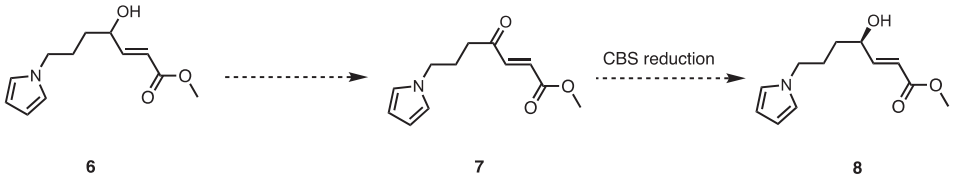
3.2. General procedures for the synthetic compounds

3.2.1. Synthesis of (S)-2-bromopentane-1, 5-diol (11)

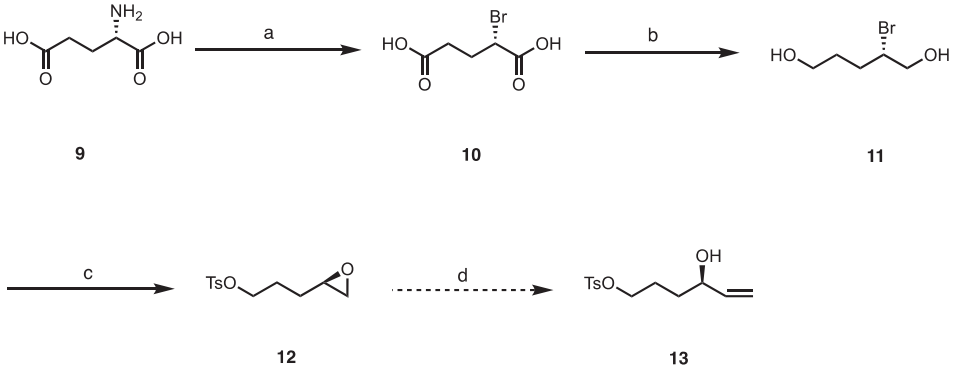
(*S*)-2-bromopentanedioic acid (10 g, 47.6 mmol) was dissolved in distilled tetrahydrofuran (THF) (125 mL) under argon atmosphere and cooled to −10 °C. Borane dimethylsulfide (71 mL, 142.8 mmol) was added dropwise at −10 °C. The mixture was warmed to rt and stirred for 10 h. The reaction was quenched with dropwise addition of MeOH (125 mL) at 0 °C and concentrated *in vacuo*. The crude product was purified by flash chromatography (dichloromethane(DCM)/acetone = 1:1) to give the title compound (7.2 g, two steps: 60%) as a colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 4.22 – 4.09 (m, 1H), 3.80 (qd, J= 12.2, 5.6 Hz, 2H), 3.69 (t, J= 6.2 Hz, 2H), 2.41 (s, 2H), 2.03 (tdd, J= 10.1, 5.8, 4.6 Hz, 1H), 1.97 – 1.76 (m, 2H), 1.76 – 1.63 (m, 1H) ^{13}C NMR (151 MHz, $CDCl_3$) δ 67.0, 62.0, 58.8, 31.2, 30.2. $[\alpha]^{20}_{D}$ -2.0 (c 0.09, MeOH); HRESIMS: *m/z* 183.0014 $[M+H]^+$ (calcd for $C_5H_{12}BrO_2$, 183.0015).

3.2.2. (2R)-2-[3-[[(1,1-Dimethylethyl)dimethylsilyl]oxy]propyl]oxir (14)

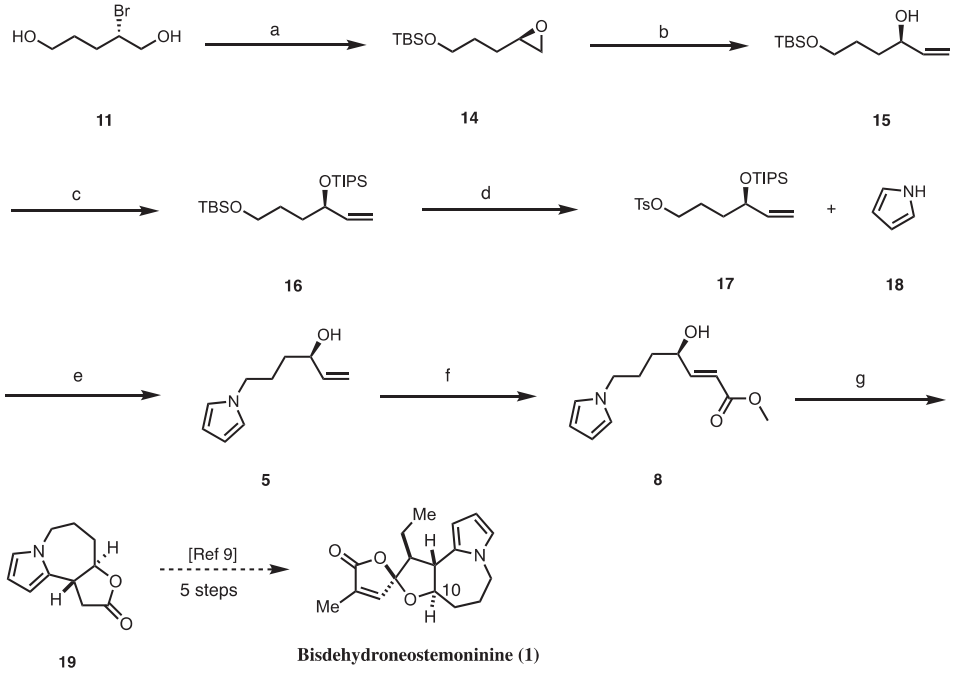
NaH (60% dispersion in oil, 280 mg, 7 mmol) was dissolved in distilled THF (5 mL) under argon atmosphere and cooled to 0 °C. Bromodiol (425 mg, 2.33 mmol) in distilled THF (2 mL) was added dropwise, and the reaction stirred for 30 min at 0 °C and tert-butyldimethylsilyl chloride (526.8 mg, 3.49 mol) was introduced. The reaction mixture was stirred for 3 h at room temperature, then quenched with saturated NH_4Cl, and extracted with Et_2O. The combined organic layers were dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography (petroleum ether (PE)/ethyl acetate (EtOAc)=8:1) to give the title compound (256 mg, 54%) as a yellow oil.



Scheme 1. The attempt to synthesize compound **8.**



Scheme 2. The synthesis of intermediate (**13**). Reagents and conditions: (a) HBr, KBr, $NaNO_2$, −15 °C to rt, 3 h. (b) BH_3 Me_2S, THF, MeOH, rt, 10 h, two steps: 60%. (c) NaH, THF, 0 °C, 0.5 h, then TsCl, py, 1 h, 52%. (d) Me_3SI, LHMDS, THF, –10 °C.



Scheme 3. The synthesis of intermediate (**19**). Reagents and conditions: (a) NaH, THF, 0 °C, 0.5 h, then TBSCl, 3 h, 54%. (b) Me_3SI, LHMDS, THF, –10 °C, room temperature, 2 h, 89%. (c) TIPSOTf, Et_3N, 0 °C, 1 h, 93%. (d) PPTS, THF, rt, 12 h and then TsCl, Et_3N, 4-DMAP, THF, rt, 12 h, 45%. (e) NaH, DMF, 2 h, and then TBAF, overnight, 93%. (f) Grubbs (2nd) (5%), methyl acrylate, DCM, 40 °C, 24 h, 92%. (g) BF_3 ether, DCM, 0 °C to rt, overnight, 57%.

3.2.3. (R)-6-((Tert-butyldimethylsilyl)oxy)hex-1-en-3-ol (15)

To a suspension of trimethylsulfonium iodide (72 mg, 0.35 mmol) in anhydrous THF (0.7 mL) was added lithium bis(trimethylsilyl)amide (0.7 mL, 0.7 mmol, 1 M in THF) at −10 °C. After 30 min, epoxide (65 mg, 0.32 mmol) in THF (0.4 mL) was introduced, producing a milky suspension. The reaction was allowed to warm to 0 °C about 30 min, followed by stirring for 2 h at room temperature. The reaction was quenched with water at 0 °C, extracted with EtOAc. The combined organic layers were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. The crude product was purified by flash chromatography (PE/EtOAc = 5:1) to give the title compound (65.8 mg, 89%) as a colorless oil; ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.97–5.82 (m, 1H), 5.26 (d, J= 17.2 Hz, 1H), 5.12 (d, J= 10.4 Hz, 1H), 4.15 (s, 1H), 3.68 (t, J= 5.2 Hz, 2H), 2.69 (s, 1H), 1.77 – 1.55 (m, 4H), 0.92 (s, 9H), 0.09 (s, 6H), ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.2, 114.3, 72.7, 63.4, 34.5, 28.8, 25.9, 18.3, 0.3. $[\alpha]^{20}_D$ -7.8 (c 0.03, MeOH); HRESIMS: m/z 231.1774 $[M+H]^+$ (calcd for $C_{12}H_{27}O_2Si$, 231.1775).

3.2.4. (R)-10,10-Diisopropyl-2, 2, 3, 3, 11-pentamethyl-8-vinyl-4, 9-dioxa-3, 10-disiladodecane (16)

To a solution of alcohol (200 mg, 0.87 mmol) in anhydrous methylene chloride (5 mL) were added triethylamine (0.6 mL, 4.35 mmol) and TIPSOTf (0.35 mL, 1.30 mmol) at −10 °C. After stirring 1 h at −10 °C, the reaction mixture was quenched with water. The organic layer was separated, and the aqueous layer was extracted with methylene chloride and washed with brine. The combined organic extracts were dried over $MgSO_4$, filtered, and concentrated *in vacuo*. Purification by flash column chromatography (PE/EtOAc = 20:1) gave title compound (336 mg, 93%) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 5.82 (ddd, J= 17.0, 10.4, 6.5 Hz, 1H), 5.16 (d, J= 17.2 Hz, 1H), 5.06 (d, J= 10.4 Hz, 1H), 4.26 (d, J= 5.2 Hz, 1H), 3.62 (dd, J= 11.0, 5.2 Hz, 2H), 1.71–1.49 (m, 4H), 1.08 (m, 21H), 0.91 (s, 9H), 0.12 (s, 6H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 141.6, 114.3, 73.7, 72.7, 63.4, 34.5, 28.8, 25.9, 18.3, 1.9, 0.3. $[\alpha]^{20}_D$ -2.0 (c 0.10, MeOH); HRESIMS: m/z 409.2933 $[M+Na]^+$ (calcd for $C_{21}H_{46}O_2Si_2Na$, 409.2929).

3.2.5. (R)-4-((Triisopropylsilyl)oxy)hex-5-en-1-yl-4-methylbenzenesulfonate (17)

To a solution of di-protected compound (380 mg, 0.98 mmol) in anhydrous THF (5 ml) were added pyridinium *p*-toluenesulfonate solution (50 mg, 0.20 mmol in 5 mL of anhydrous methanol) at rt and stirred overnight at room temperature. Triethylamine (0.4 mL, 2.94 mmol), 4-dimethylaminopyridine (12.2 mg, 0.1 mmol), TsCl (280 mg, 1.47 mmol) was added at 0 °C and then stirred for overnight at room temperature. The reaction mixture was quenched with water at 0 °C. The aqueous layer was extracted with ethyl acetate. The combined organic extracts were washed with brine, dried over Na_2SO_4, filtered, and concentrated in *vacuo*. Purification by flash column chromatography (PE/EtOAc = 8:1) gave compound (188 mg, 45%) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, J= 8.3 Hz, 2H), 7.36 (d, J= 8.0 Hz, 2H), 5.73 (ddd, J= 16.8, 10.4, 6.3 Hz, 1H), 5.16–5.09 (m, 1H), 5.07–5.03 (m, 1H), 4.25 (q, J= 5.9 Hz, 1H), 4.09–4.01 (m, 2H), 2.47 (s, 3H), 1.78–1.64 (m, 2H), 1.60–1.49 (m, 2H), 1.04 (m, 21H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 144.6, 140.9, 133.2,

129.8, 127.9, 114.5, 73.0, 70.9, 33.8, 23.9, 21.6, 18.1, 18.1, 12.3. $[\alpha]^{20}_{D}$ -7.2 (c 0.27, MeOH); HRESIMS: *m/z* 427.2328 $[M+H]^+$ (calcd for $C_{22}H_{40}O_4SSi$, 427.2333).

3.2.6. (R)-6-(1H-Pyrrol-1-yl)hex-1-en-3-ol (5)

Pyrrole (0.017 mL, 0.24 mmol) was added to a stirred suspension of NaH (10 mg, 0.24 mmol) in dry *N, N*-dimethylformamide (1 mL), and the resulting solution was starred at room temperature for 5 min. Then, a solution of tosylate (51.8 mg, 0.12 mmol) in DMF (0.5 mL) was added, and the flask containing the tosylate was rinsed with additional DMF (0.2 mL). The resulting mixture was stirred at room temperature for 2 h, and then, a solution of TBAF (0.5 mL, 0.5 mmol, 1 M in THF) was added and stirred at room temperature overnight. The reaction mixture was quenched with water, and the aqueous layer was extracted with ethyl acetate. The combined organic extracts were washed with brine, dried over Na_2SO_4, filtered, and concentrated in vacuo. Purification by flash column chromatography (PE/EtOAc = 4:1) gave title compound (18.6 mg, 93%) as a colorless oil. ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.66 (s, 2H), 6.14 (s, 2H), 5.84 (ddd, *J*= 17.2, 10.4, 6.2 Hz, 1H), 5.22 (d, *J*= 17.2 Hz, 1H), 5.12 (d, *J*= 10.4 Hz, 1H), 4.09 (q, *J*= 6.2 Hz, 1H), 3.91 (tt, *J*= 8.5, 4.2 Hz, 2H), 1.99–1.76 (m, 2H), 1.54–1.49 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 140.8, 120.5, 115.1, 107.9, 72.8, 49.5, 33.9, 27.4. $[\alpha]^{20}_{D}$ -2.5 (c 0.12, MeOH); HRESIMS: *m/z* 166.1221 $[M+H]^+$ (calcd for $C_{10}H_{16}NO$, 166.1226).

3.2.7. Methyl (R, E)-4-hydroxy-7-(1H-pyrrol-1-yl) hept-2-enoate (8)

To a solution of the alcohol (0.26 g, 1.6 mmol) in DCM (4 mL) were added methyl acrylate (1.4 g, 16 mmol) and the Grubbs second generation catalyst (66 mg, 0.082 mmol) at room temperature in sealed tube. The deep brown reaction mixture was raised to 40 °C and stirred for 24 h. The reaction mixture was concentrated to afford residue, which was purified by column chromatography (hexane: EtOAc = 4:1) to afford the desired product (0.32 g) as brown oil (Yield: 92%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.92 (dd, *J*= 15.7, 4.9 Hz, 1H), 6.66 (s, 2H), 6.16 (t, *J*= 2.1 Hz, 2H), 6.05 (dd, *J*= 15.7, 1.7 Hz, 1H), 4.34–4.25 (m, 1H), 3.97–3.88 (m, 2H), 3.77 (s, 3H), 1.98–1.81 (m, 2H), 1.54–1.62 (d, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 150.0, 120.5, 120.1, 108.1, 70.5, 51.7, 49.3, 33.5, 27.2. $[\alpha]^{20}_{D}$ -3.4 (c 0.27, MeOH).

3.2.8. (3aR, 10bR)-1, 3a, 4, 5, 6, 10b-Hexahydro-2H-furo[3, 2-c]pyrrolo[1, 2-a]azepin-2-one (19). To a solution of ester (25 mg, 0.11 mmol) in DCM (3.8 mL) was added the boron trifluoride diethyl etherate (0.017 ml, 0.14 mmol) at 0 °C. The reaction mixture was warmed to room temperature and stirred overnight. Triethylamine was added to the reaction mixture and stirred for 30 min. The organic layer was washed with H_2O and concentrated to afford residue, which was purified by column chromatography (PE: EtOAc = 4:1) to afford the desired product **19** (9.6 mg) as white solid (Yield: 57%). ^{1}H NMR (600 MHz, $CDCl_3$) δ 6.70–6.59 (m, 1H), 6.05 (dd, *J*= 3.4, 2.8 Hz, 1H), 5.99–5.92 (m, 1H), 4.14 (dd, *J*= 14.6, 5.4 Hz, 1H), 4.00 (ddd, *J*= 11.2, 10.1, 3.5 Hz, 1H), 3.91 (dd, *J*= 14.2, 11.8 Hz, 1H), 3.43 (dd, *J*= 15.4, 6.0 Hz, 1H), 2.98–2.87 (m, 2H), 2.60–2.50 (m, 1H), 2.20–2.10 (m, 1H), 1.88–1.80 (m, 1H),

1.77–1.69 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.2, 128.6, 122.8, 106.4, 105.5, 83.7, 49.1, 42.2, 34.0, 33.7, 26.1. $[\alpha]^{20}_{D}$ -4.7 (c 0.38, MeOH).

Disclosure statement

The authors declare no conflict of interest.

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