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Note

An unexpected rearrangement of pent-4-enofuranosides to cyclopentanones upon hydrogenolysis of the anomeric benzyl group

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ABSTRACT

During our synthesis toward the unique nucleoside antibiotic A201A, we were surprised to find that a benzyl arabino-pent-4-enofuranoside underwent a Ferrier II-like rearrangement readily to provide the corresponding cyclopentanone derivative in high yield and stereoselectivity upon hydrogenolysis of the anomeric benzyl group.

1. Introduction

Polyoxygenated cyclopentanoids are the essential scaffolds of many bioactive molecules of pharmaceutical interest, including carbocyclic nucleosides [1], glycosidase inhibitors [2], prostaglandin [3,4], pentenomycin [5], and caryose [6,7]. Over the past few decades, numerous approaches to the construction of the cyclopentanoid skeleton have been developed [2,8–11]. A few of these approaches employ carbohydrates as starting materials to furnish polyoxygenated cyclopentanoids, however, in multiple steps and unsatisfactory efficiency [12–15].

On the other hand, carbohydrates have been extensively used in the construction of polyoxygenated cyclohexanoids [16–22], that could be mainly attributed to the classical Ferrier II rearrangement of hex-5-enopyranosides promoted by a stoichiometric or catalytic amount of Lewis acids such as Hg(II) or Pd(II) salts (Scheme 1) [17,23–28]. This rearrangement involves a cleavage of the acetal bond and a subsequent aldol-like intramolecular cyclization. However, despite its reliable performance and widespread application in synthesizing cyclohexanone derivatives [27,29–35], the Ferrier II rearrangement has rarely been applied successfully in converting pent-4-enofuranosides into the corresponding cyclopentanones [16]. Thus, Gallos et al. reported a nitrile oxide-assisted rearrangement to prepare five-membered carbocycles in two steps, involving a nitrile oxide cycloaddition and subsequent reductive cleavage of the spiroisoxazoline intermediate to promote the key aldol-like cyclization [14,15]. Other examples are limited only on specific substrates, such as spiro dienones [36–38], vinyl alkylidenes [39,40], and γ-enollactones [41]. Rearrangements of pyranosides to construct cyclopentanoids with zirconium, samarium and palladium were also reported [42–47]. It is noteworthy that all the resultant cyclopentanones in these Ferrier II-like reactions remain redundant substituents, which are difficult to remove afterwards.

Very recently, during our synthesis toward a unique nucleoside antibiotic A201A [48], we were surprised to find a cyclopentanone product resulting from the rearrangement of a pent-4-enofuranoside upon hydrogenolysis of the anomeric benzyl group. Herein, we report this unexpected discovery and a brief examination of the reaction scope.

2. Results and discussion

Disaccharide **1** bearing an arabino-pent-4-enofuranoside moiety was envisioned as a key intermediate in our synthetic studies toward nucleoside antibiotic A201A (Scheme 2) [48]. Thus, D-arabinose was converted into 5-aldehyde-furanoside **2** in 9 steps

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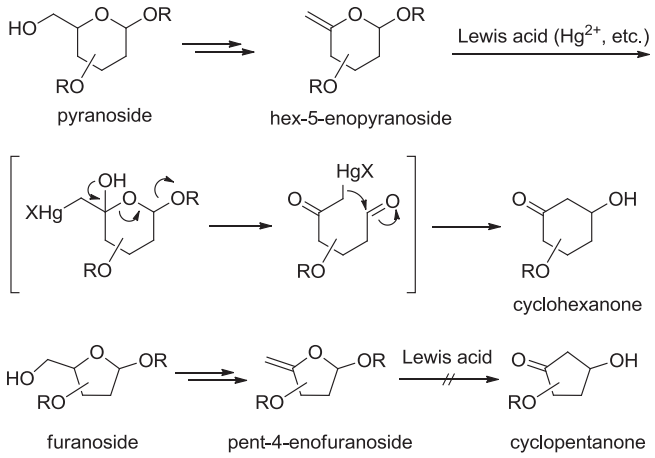
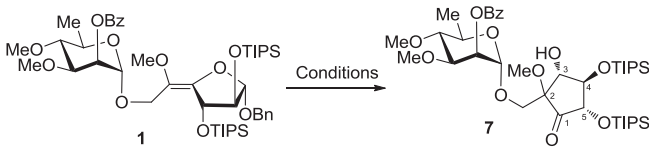
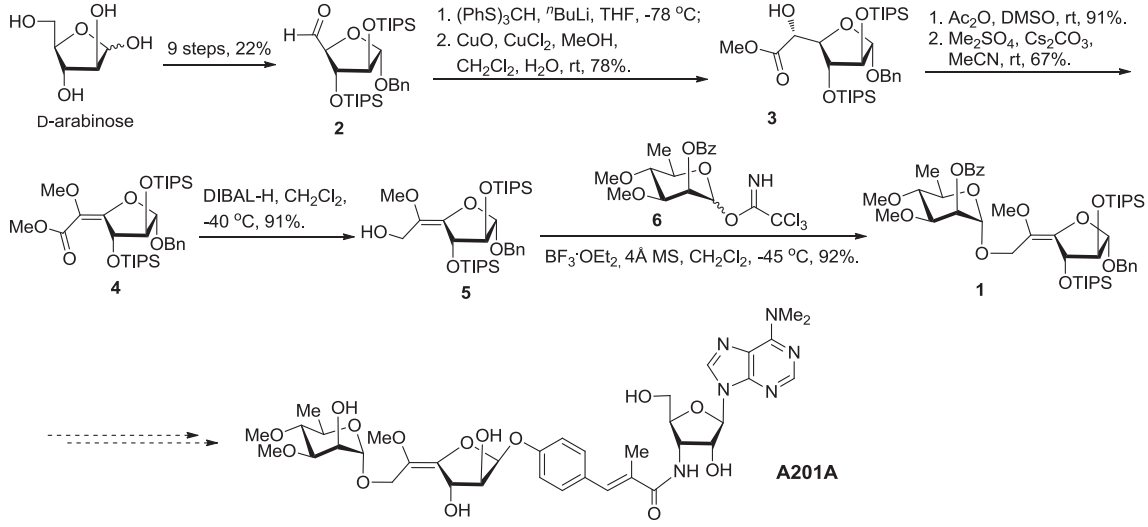


Scheme 1. Ferrier II rearrangement.

Table 1
Attempts to deprotect the benzyl group and the unexpected rearrangement of disaccharide **1**.



Entry	Conditions	Results
1	LDBB, THF, −78 °C.	Complex
2	$FeCl_3$, dry CH_2Cl_2, rt.	Complex
3	Pd/C, H_2(1 atm), ethyl acetate, Et_3N, rt.	No reaction
4	Raney Ni, EtOH, rt.	**7**, 90%

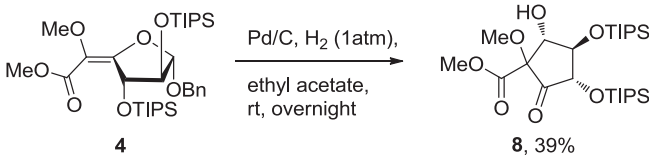


Scheme 2. The preparation of disaccharide **1** toward the synthesis of A201A.

with an overall yield of 22% (see SI for detailed information) [49–52]. Addition of aldehyde **2** with tris-(phenylthio)methyllithium at −78 °C resulted in an unstable phenylthioorthoester, which was treated with CuO and $CuCl_2$ to give α-OH methyl ester **3** in good yield. Oxidation of the nascent α-OH on **3** and subsequent enolization in the presence of Cs_2CO_3 and Me_2SO_4 led to enol methyl ester **4**, which was then reduced with DIBAL-H to give alcohol **5**. Glycosyaltion of **5** with imidate **6** (prepared from D-mannose according to the reported procedure) [48] promoted with $BF_3{\cdot}OEt_2$ at −45 °C furnished the desired disaccharide **1** in 92% yield.

To deprotect the anomeric benzyl group on **1** for further synthetic transformations toward A201A, several debenzylation conditions were examined, wherein the reactive exocyclic enol ether moiety should remain intact. As depicted in Table 1, either Lewis acid $FeCl_3$ or radical reagent LiDBB (lithium di-tert-butylbiphenyl) made disaccharide **1** fully decomposed and led to complex mixtures (entry 1, 2) [53,54]. On the other hand, no reaction was observed in Pd/C-catalyzed hydrogenolysis in the presence of Et_3N (entry 3). Surprisingly, when disaccharide **1** was treated with a catalytic amount of Raney Ni in EtOH at rt, cyclopentanone **7** resulting from a rearrangement was afforded in 90% yield. The structure of cyclopentanone **7** was elaborated with a set of NMR spectra (see SI for more details): The signal at 208 ppm in ^{13}C NMR spectrum revealed the generation of carbonyl group at C1; the nascent 3-OH was confirmed by its active hydrogen signal at 2.61 ppm in ^1H NMR spectrum; the configuration of C3 was presumed by the absence of NOE correlation between H3 (δ 4.03 ppm) and H4 (δ 4.21 ppm). Although the NMR spectra could not provide enough information to determine the configuration of C2, the rearrangement turned out to be highly stereoselective with only trace epimers being observed.

Further study was then conducted on monosaccharide **4** bearing a conjugated exocyclic enol ether which was much less reactive (Scheme 3). As expected, the rearrangement was much slower upon treatment of Raney Ni in EtOH, and most of **4** remained intact



Scheme 3. Rearrangement of conjugated enol ether furanoside **4** under Pd/C-catalyzed hydrogenolysis.

after 12 h at rt. A better conversion was obtained under the conditions of Pd/C-catalyzed hydrogenolysis under H_2 in ethyl acetate at rt, leading to the desired cyclopentanone **8** in 39% yield and recovered **4** in 40% yield.

To briefly check the scope of this rearrangement, three benzyl pent-4-enofuranosides **9**–**11** were prepared from either D-arabinose or D-ribose (see SI for the preparation). These substrates were then examined under the conditions of Raney Ni in EtOH at rt. As depicted in Table 2, TIPS-protected arabino-enofuranoside **9**, which was similar to the furanosyl unit of disaccharide **1**, could be converted smoothly into the desired cyclopentanone **12** in 86% yield and excellent stereoselectivity (entry 1). The mechanism was hypothesized to involve the cleavage of C1–O5 bond and subsequent attack of the resultant aldehyde by enol [18,19,41,55]. However, replacing TIPS with Ac groups led to full hydrolysis of **10** under these conditions (entry 2). This result implied that the bulkiness of the TIPS groups are important for the rearrangement. However, TIPS-protected ribo-enofuranoside **11** turned out to be stable upon treatment of Raney Ni in EtOH even at 90 °C. This outcome could be attributed to the configuration of C2 being critical to the hydrogenolysis of the anomeric benzyl group.

3. Conclusion

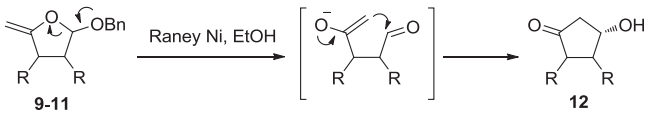
In conclusion, we have discovered a Ferrier II-like rearrangement upon hydrogenolysis of the anomeric benzyl group on pent-4-enofuranosides, however, with a different mechanism. Although the highly efficient rearrangements are only observed herein on arabinofuranoside substrates with bulky TIPS protecting groups, it is, to the best of our knowledge, the first rearrangement of enofuranosides to cyclopentanones resulting from hydrogenolysis conditions. Further studies on the scope of this reaction and its application in the synthesis are still in progress.

4. Experimental

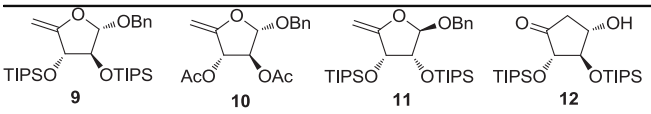
4.1. General methods

All reactions were carried out under nitrogen or argon with anhydrous solvents in flame-dried glassware, unless otherwise noted. All glycosylation reactions were performed in the presence of 4 Å or 5 Å molecular sieves, which were flame-dried immediately before use in the reaction under high vacuum. Glycosylation solvents were dried using a solvent purification system and used directly without further drying. The chemicals used were reagent grade as supplied, except where noted. Analytical thin-layer chromatography was performed using silica gel 60 F254 glass plates. Compound spots were visualized by UV light (254 nm) and by heating with a solution with 10% H_2SO_4 in ethanol. Flash column chromatography was performed on silica gel. NMR spectra were referenced using Me_4Si (0 ppm), residual $CHCl_3$ (^{1}H NMR δ = 7.26 ppm, ^{13}C NMR δ = 77.16 ppm), CD_3OD (^{1}H NMR δ = 3.30 ppm, ^{13}C NMR δ = 49.00 ppm), or D_2O (^{1}H NMR δ = 4.67 ppm). Peak and coupling constant assignments are based on ^{1}H NMR, ^{1}H–^{1}H COSY, and ^{1}H–^{13}C HMQC experiments. Splitting patterns are indicated as s (singlet), d (doublet), t (triplet), q (quartet), and brs (broad singlet) for ^{1}H NMR data. ESI-MS and MALDI-MS were run on an IonSpec Ultra instrument using HP5989A or VG Quattro MS. Optical rotations were measured using a Perkin-Elmer 241 polarimeter.

Table 2
Examination of the rearrangement of benzyl pent-4-enofuranosides **9**–**11**.



Entry	Substrates	Results
1	**9**	**12**, 86%
2	**10**	Hydrolysis
3	**11**	No reaction



4.2. Methyl (benzyl 2,3-di-O-triisopropylsilyl-α-D-altrofuranosid) uronate **3**

To a solution of tris(phenylthio)methane (311 mg, 0.92 mmol) in freshly distilled THF (3.5 mL) was added dropwise ^{n}BuLi (1.6 M in hexanes, 0.54 mL, 1.26 mmol) at −78 °C. The mixture was stirred at that temperature for 2 h, and aldehyde **2** (419 mg, 0.74 mmol) in THF (3.5 mL) was added dropwise over 10 min. The mixture was stirred at that temperature for additional 3 h, and was then quenched with saturated aqueous NH_4Cl. The mixture was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was instable and thus was used in the next reaction without further purification.

To a solution of the residue above in CH_2Cl_2 (1.2 mL) and MeOH (13.1 mL) were added CuO (86 mg, 1.1 mmol), $CuCl_2$ (313 mg, 2.33 mmol) and H_2O (1.2 mL) at rt. The resulting mixture was stirred overnight, and was then filtered and washed with CH_2Cl_2. The filtrates were concentrated in vacuo. The residue was diluted with CH_2Cl_2 and water, and was then extracted with CH_2Cl_2. The organic layer was washed with saturated aqueous $NaHCO_3$ and brine, dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 100:1 to 30:1 to 10:1) to afford **3** (361 mg, 78%) as a colorless syrup: $[\alpha]_D^{28} = +58.3$ (*c* 0.7, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.24 (m, 5H), 5.08 (s, 1H), 4.78 (d, J = 12.1 Hz, 1H), 4.50 (d, J = 12.1 Hz, 1H), 4.46–4.39 (m, 2H), 4.38 (s, 1H), 4.25 (s, 1H), 3.77 (s, 3H), 3.55 (d, J = 5.7 Hz, 1H), 1.04 (s, 42H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.8, 138.1, 128.3, 128.0, 127.5, 108.3, 89.7, 82.8, 79.0, 71.9, 69.0, 52.4, 18.1, 18.02, 17.97, 12.5, 12.4; HRMS (MALDI-FTMS) calcd for $C_{32}H_{58}O_7Si_2Na$ $[M+Na]^+$: 633.3613, found: 633.3609.

4.3. Methyl (benzyl (Z)-2,3-di-O-triisopropylsilyl-5-O-methyl-α-D-altro-hex-4-enofuranosid)uronate **4**

To a solution of alcohol **3** (76 mg, 0.12 mmol) in DMSO (1.2 mL) was added Ac_2O (0.79 mL) at rt. The mixture was stirred at rt overnight before it was quenched with MeOH. The mixture was diluted with H_2O, and extracted with EtOAc. The organic layer was washed with brine, dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 12:1) to afford an α-keto-ester (69 mg, 91%) as a colorless syrup: $[\alpha]_D^{28} = +56.8$ (*c* 1.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36–7.25 (m, 6H), 5.18 (s, 1H), 5.05 (s, 1H), 4.84 (d, J = 12.0 Hz, 1H), 4.60 (s, 1H), 4.54 (d, J = 12.0 Hz, 1H), 4.20 (s, 1H), 3.85 (s, 3H), 1.10–0.98 (m, 42H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.6, 162.9, 138.0, 128.2, 127.9, 127.5, 109.8, 90.5, 81.5, 80.3, 69.7, 52.7, 18.10, 18.08, 17.94, 17.87, 12.4, 12.2; HRMS (MALDI-FTMS) calcd for $C_{32}H_{56}O_7Si_2Na$ $[M+Na]^+$: 631.3457, found: 631.3476.

To a solution of the α-keto-ester above (53 mg, 0.08 mmol) in

MeCN (3.6 mL) were added Me_2SO_4 (0.077 mL, 0.81 mmol) and Cs_2CO_3 (53 mg, 0.16 mmol) at rt. The mixture was stirred overnight, and was then quenched with 1 M aqueous NaOH over 10 min. The mixture was extracted with CH_2Cl_2. The organic layer was dried over Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 60:1 to 40:1) to afford **4** (36 mg, 67%) as colorless syrup: $[\alpha]_D^{26}$ = +125.3 (*c* 0.73, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43–7.22 (m, 5H), 5.32 (s, 2H), 4.87 (d, *J* = 12.0 Hz, 1H), 4.65 (d, *J* = 12.0 Hz, 1H), 4.27 (s, 1H), 3.77 (s, 3H), 3.64 (s, 3H), 1.15–0.93 (m, 42H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.2, 162.2, 137.3, 128.3, 128.2, 128.0, 127.8, 111.3, 80.0, 76.6, 71.0, 60.4, 51.7, 18.2, 18.0, 12.7, 12.4; HRMS (MALDI-FTMS) calcd for $C_{33}H_{58}O_7Si_2Na$ $[M+Na]^+$: 645.3613, found: 645.3632.

4.4. Benzyl (Z)-2,3-di-O-triisopropylsilyl-5-O-methyl-α-D-altro-hex-4-enofuranoside **5**

To a solution of ester **4** (67 mg, 0.11 mmol) in CH_2Cl_2 (1.2 mL) was added DIBAL-H (0.4 mL, 0.4 mmol) at −40 °C. Another portion of DIBAL-H (0.4 mL, 0.4 mmol) was added after 30 min. The reaction was quenched with saturated potassium sodium tartrate. The mixture was extracted with CH_2Cl_2, dried over Na_2SO_4, and concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 12:1) to afford **5** (58 mg, 91%) as a colorless syrup: $[\alpha]_D^{28}$ = +80.0 (*c* 0.9, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39–7.24 (m, 5H), 5.18 (s, 1H), 4.84 (d, *J* = 12.1 Hz, 1H), 4.63 (s, 1H), 4.59 (d, *J* = 12.0 Hz, 1H), 4.25 (s, 1H), 4.20 (s, 2H), 3.75 (s, 3H), 1.06–1.00 (m, 42H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 145.6, 137.7, 134.8, 128.3, 128.1, 127.7, 109.3, 80.6, 76.7, 70.1, 60.1, 58.7, 18.3, 18.2, 18.04, 17.99, 13.0, 12.4; HRMS (MALDI-FTMS) calcd for $C_{32}H_{58}O_6Si_2Na$ $[M+Na]^+$: 617.3664, found: 617.3665.

4.5. Benzyl (2-O-benzoyl-3,4-di-O-methyl-α-D-rhamnopyranosyl)-(1 → 6)-(Z)-2,3-di-O-triisopropylsilyl-5-O-methyl-α-D-altro-hex-4-enofuranoside **1**

$BF_3{\cdot}OEt_2$ (20 μL) was diluted with CH_2Cl_2 (2.26 mL) to give a 0.07 M solution. To a solution of imidate **6** (31 mg, 0.071 mmol) and alcohol **5** (14 mg, 0.024 mmol) in CH_2Cl_2 (2 mL) was added activated 4 Å MS (100 mg) under argon atmosphere. The mixture was stirred at rt for 2 h, and then cooled to −45 °C. $BF_3{\cdot}OEt_2$ (0.1 mL, 0.07 M, 0.007 mmol) was added dropwise. The reaction was stirred for 40 min, and then quenched with Et_3N and filtered. The filtrate was concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 20:1) to afford **1** (19 mg, 92%) as a colorless syrup: $[\alpha]_D^{27}$ = +74.7 (*c* 1.1, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.3 Hz, 2H), 7.57 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.38–7.24 (m, 5H), 5.54 (d, *J* = 1.8 Hz, 1H), 5.20 (s, 1H), 4.90 (s, 1H), 4.85 (d, *J* = 12.0 Hz, 1H), 4.61–4.58 (m, 2H), 4.32 (d, *J* = 11.9 Hz, 1H), 4.24 (s, 1H), 4.02 (d, *J* = 11.9 Hz, 1H), 3.82–3.75 (m, 1H), 3.75 (s, 3H), 3.65 (dd, *J* = 9.3, 3.2 Hz, 1H), 3.55 (s, 3H), 3.42 (s, 3H), 3.17 (t, *J* = 9.4 Hz, 1H), 1.35 (d, *J* = 6.2 Hz, 3H), 1.10–0.95 (m, 42H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.8, 147.7, 137.7, 133.2, 132.4, 130.2, 130.0, 128.5, 128.3, 128.2, 127.7, 109.7, 97.8, 82.4, 80.6, 79.8, 76.4, 70.3, 69.2, 68.0, 65.5, 61.0, 59.1, 57.5, 18.3, 18.2, 18.1, 18.0, 13.0, 12.4; HRMS (MALDI-FTMS) calcd for $C_{47}H_{76}O_{11}Si_2Na$ $[M+Na]^+$: 895.4818, found: 895.4832.

4.6. Cyclopentanone **7**

To a solution of **1** (10 mg, 0.011 mmol) in EtOH (2 mL) was added Raney Ni (*ca.* 30 mg, wet). The mixture was stirred at rt until the starting material was fully consumed as indicated by TLC. The mixture was filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 10:1) to afford **7** (8 mg, 90%) as a colorless syrup: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 5.47 (s, 1H), 4.81 (s, 1H), 4.22–4.19 (m, 1H), 4.14 (d, *J* = 7.5 Hz, 1H), 4.04 (brs, 1H), 4.00 (d, *J* = 9.8 Hz, 1H), 3.60 (d, *J* = 9.8 Hz, 1H), 3.58–3.54 (m, 1H), 3.53 (s, 3H), 3.47–3.44 (m, 4H), 3.39 (s, 3H), 3.15 (t, *J* = 9.4 Hz, 1H), 2.61 (d, *J* = 5.8 Hz, 1H), 1.35 (d, *J* = 6.2 Hz, 3H), 1.24–1.08 (m, 42H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.1, 165.8, 133.4, 130.1, 130.0, 128.6, 98.3, 82.0, 81.6, 80.2, 79.7, 78.6, 77.4, 74.7, 69.0, 68.5, 64.0, 60.8, 57.7, 53.1, 18.40, 18.36, 18.3, 18.2, 13.2, 13.0; HRMS (MALDI-FTMS) calcd for $C_{40}H_{70}O_{11}Si_2Na$ $[M+Na]^+$: 805.4349, found: 805.4362.

4.7. (3S,4R,5S)-2-methoxycarbonyl-2-O-methyl-4,5-di-O-triisopropylsilyl-3-hydroxy-cyclopentanone **8**

To a solution of **4** (15 mg, 0.024 mmol) in EtOAc (3 mL) was added 10% Pd/C (12 mg). The mixture was stirred overnight under 1 atm hydrogen atmosphere. The mixture was filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 30:1) to afford **8** (5 mg, 39%) as a syrup: ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.38 (d, *J* = 8.0 Hz, 1H), 4.16–4.09 (m, 2H), 3.80 (s, 3H), 3.60 (s, 3H), 2.72 (d, *J* = 8.6 Hz, 1H), 1.22–1.07 (m, 42H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.4, 168.5, 82.1, 81.6, 80.0, 77.4, 55.4, 52.9, 18.34, 18.28, 18.2, 13.2, 13.0; ESI-MS calcd for $C_{26}H_{52}O_7Si_2Na$ $[M+Na]^+$: 555.3, found: 555.7.

4.8. (2S,3R,4S)-2,3-di-O-triisopropylsilyl-4-hydroxy-cyclopentanone **12**

To a solution of **9** (35 mg, 0.065 mmol) in EtOH (3 mL) and THF (0.3 mL) was added Raney Ni (*ca.* 70 mg, wet). The mixture was stirred at rt until the starting material was fully consumed as indicated by TLC. The mixture was filtered and concentrated in vacuo. The residue was purified by silica gel column chromatography (petroleum ether-CH_2Cl_2, 2:1) to afford **12** (25 mg, 86%) as a colorless syrup: $[\alpha]_D^{28}$ = −27.0 (*c* 2.3, $CHCl_3$); ^{1}H NMR (400 MHz, $CDCl_3$) δ 4.29–4.24 (m, 1H), 4.16 (s, 1H), 3.82 (s, 1H), 3.19 (d, *J* = 11.4 Hz, 1H), 2.70 (dd, *J* = 19.5, 6.3 Hz, 1H), 2.45 (d, *J* = 19.5 Hz, 1H), 1.25–1.04 (m, 42H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 213.1, 78.4, 77.5, 75.3, 43.4, 18.04, 18.02, 17.97, 17.88, 12.3, 12.2; HRMS (ESI-FTMS) calcd for $C_{23}H_{48}O_4Si_2Na$ $[M+Na]^+$: 467.2983, found: 467.2990.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.carres.2016.06.006.

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