Synthesis of an Isomer of the Renieramycin Skeleton from L-Tyrosine

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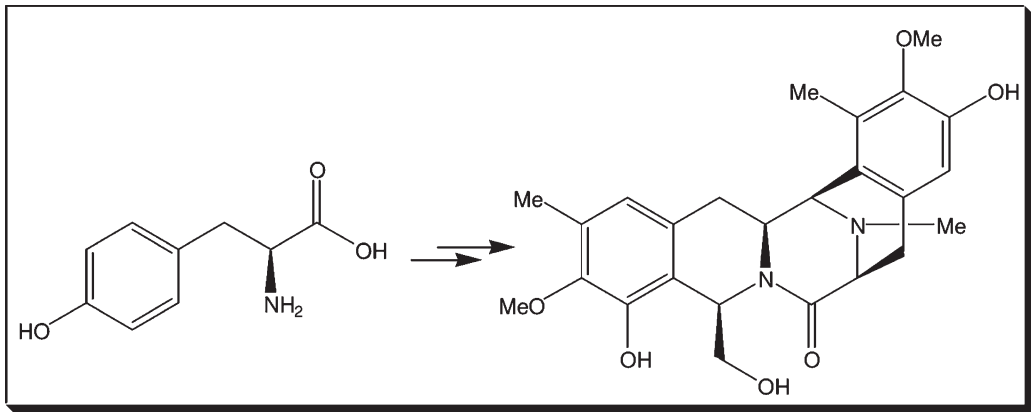

A new approach that tried to obviate the use of bromine protection groups was studied to synthesize (−)-renieramycin G from L-tyrosine. It was found that the first intermolecular Pictet–Spengler reaction proceeded successfully to give the correct tetrahydroisoquinoline precursor **6**. However, the second intramolecular Pictet–Spengler cyclization step failed to give the desired product, and an isomer of the skeleton of the renieramycins was obtained *via* 12 steps starting from L-tyrosine.

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INTRODUCTION

Members of the tetrahydroisoquinoline family of alkaloids including saframycins, ecteinascidins, renieramycins, quinocarcins, and lemonomycin display a wide range of biological properties such as antitumor and antimicrobial activities [1]. Renieramycins are marine natural products that are structurally and biologically related to the isoquinoline natural products. Renieramycin G (Fig. 1) was isolated in 1992 by Davidson [2] from the Fijian sponge *Xestospongia caycedoi*. Several studies on the total synthesis of the renieramycin natural products have been reported [3–5].

The stereospecific intramolecular Pictet–Spengler cyclization is one of the key steps for the construction of the pentacyclic skeleton of the bistetrahydroisoquinoline alkaloids. Organic acids such as HCOOH, $MeSO_3H$, CF_3SO_3H, and CF_3COOH have been used to realize the cyclization [6–23]. Previous research in our group mainly focused on the study of new approaches to construct the pentacyclic skeleton and the discovery of simplified derivatives of the bistetrahydroisoquinoline alkaloids [24]. Recently, our group reported a new approach for the total synthesis of (−)-renieramycin G using L-tyrosine as the chiral starting material [25]. Now as a continuation of this research, we investigated a more efficient synthetic route of (−)-renieramycin G, which tried to avoid the use of the bromine protection groups in the two benzene rings.

RESULTS AND DISCUSSIONS

The synthesis of amino acid **4** (Scheme 1) and the key 1,2,3,4-tetrahydroisoquinoline precursor **6** (Scheme 2) basically followed our published procedures. The difference was that the use of the bromine protecting groups on the benzene rings was obviated. The Baeyer–Villiger oxidation of **1** using *m*-chloroperoxybenzoic acid (*m*-CPBA) in chloroform at room temperature, followed by hydrolysis of the resulting formate intermediate, provided phenol **2**. The N-acetyl group of **2** was removed with $SOCl_2$ in methanol, and the resulting free amine was reprotected as the corresponding Boc carbamate to afford compound **4**. Finally, hydrolysis of the methyl ester with LiOH provided amino acid **4**. Amino ester **2** was reduced to the corresponding alcohol by $LiBH_4$ in 91% yield. The N-acetyl group was removed

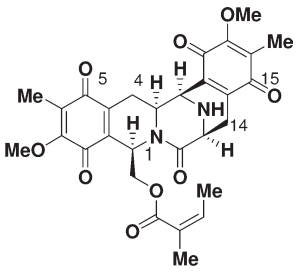


Figure 1. Structure of (−)-renieramycin G.

with 6 N aqueous HCl in CH_3OH to give amino alcohol **5** in 90% yield. The highly diastereoselective Pictet–Spengler cyclization reaction between amino alcohol **5** and benzyloxyacetaldehyde at −10°C regioselectively provided (1*R*,3*S*)-1,2,3,4-tetrahydroisoquinoline **6** in 87% yield. No product from the cylization occurring para to the hydroxyl group on the benzene ring was found. The stereochemistry of compound **6** was verified through the analysis of the stereochemistry of (−)-MY 336a, which was obtained from the hydrogenation of compound **6** [26]. Thus it proved that the intermolecular Pictet–Spengler reaction was successful without the bromine blocking group.

Next, 1,2,3,4-tetrahydroisoquinoline **6** was coupled with **4** through the action of bis(2-oxo-3-oxazolidinyl) phosphinic chloride (BOPCl) to afford amide **7**. Then, silylation of compound **7** with *tert*-butyldimethylsilyl chloride (TBSCl) and subsequent cleavage of TBS group selectively provided the primary alcohol **8**. Oxidation of compound **8** with Dess–Martin periodinane provided hemiaminal as a single diastereomer. Cleavage of the aryl TBS ether using tetrabutylammonium fluoride (TBAF) afforded compound **9** (Scheme 3).

With **9** in hand, we then investigated the key intramolecular Pictet–Spengler reaction to construct the pentacyclic skeleton. However, the pentacyclic product **10** was not obtained from the treatment of **9** with HCOOH, CF_3SO_3H, or $MeSO_3H$. Finally, treatment of compound **9** with trifluoroacetic acid (TFA) at room temperature provided pentacyclic compound **10** with the Boc group having been removed simultaneously. Without purification, crude compound **10** was N-methylated to give product **11** through the reductive methylation. The characteristic nuclear Overhauser effects (NOEs) between 5-H and 6-CH_3, and between 15-H and 16-OH in compound **11** confirmed that 15-H was ortho to 16-OH (Scheme 3). Thus it was confirmed that the intramolecular Pictet–Spengler cyclization occurred para instead of ortho to the hydroxyl group of the right benzene ring. It is supposed that the relatively strong condition of this reaction (CF_3CO_2H/r.t.) other than the mild one of the first intermolecular Pictet–Spengler reaction (acetic acid/−10°C) failed to give the desired product.

CONCLUSION

In conclusion, we studied a new approach for the total synthesis of (−)-renieramycin G without the use of the bromine protection groups. The first intermolecular Pictet–Spengler reaction proceeded smoothly to give the desired tetrahydroisoquinoline product. However, the second intramolecular Pictet–Spengler reaction did not give the correct cyclization product, and an isomer of the skeleton of renieramycin natural products was obtained through 12 steps for the longest linear route.

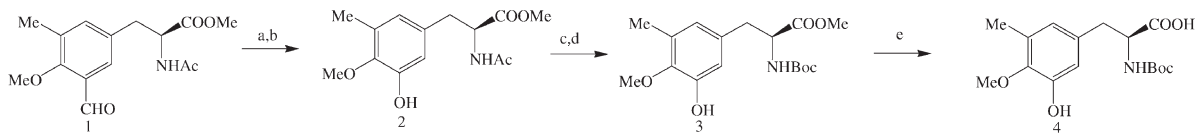
EXPERIMENTAL

General. ^{1}H-NMR spectra were recorded on a Bruker AM 600 or 300 instrument (Bruker BioSpin Corporation, MA) at 24°C in the indicated solvent and are reported in parts per million relative to tetramethylsilane and referenced internally to the residually protonated solvent. ^{13}C-NMR spectra were recorded at 150 or 75 MHz spectrometer at 24°C in the solvent indicated and are reported in parts per million relative to tetramethylsilane and referenced internally to the residually protonated solvent. HRMS were carried out by Agilent LC/MSD TOF (Agilent Technologies, CA). Optical rotations were measured on a Perkin Elmer Polarimeter 341LC (PerkinElmer Incorporation, MA) using 10-cm cells and the sodium D line (589 nm) at 20°C and concentration was indicated. All reagents were obtained from commercial suppliers unless otherwise stated.

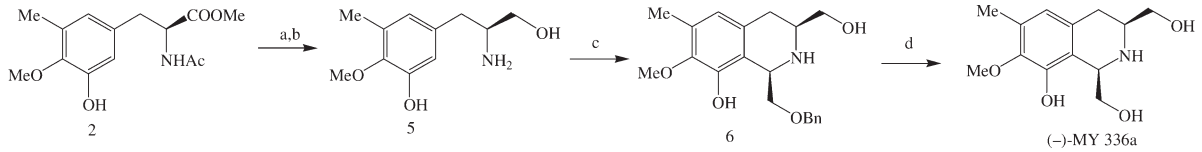
Synthesis of compound 4. To a solution of **3** (1.56 g, 4.6 mmol) in CH_3OH (10 mL), a solution of lithium hydroxide (0.44 g, 18.4 mmol, 4 equiv.) in water (5 mL) was added. The solution was stirred at room temperature for 4 h. The methanol was removed *in vacuo*, and the aqueous phase was acidified to pH 1.5. The aqueous phase was extracted with EtOAc (50 mL × 2), and the combined organic extracts were washed with brine and dried over Na_2SO_4. The organic phase was concentrated, and the residue was purified by flash column chromatography (EtOAc) to afford **4** (1.39 g, 93%) as a white solid. m.p.: 77–80°C.

^{1}H-NMR (300 MHz, dimethyl sulfoxide-d_6): σ 12.53 (s, 1H), 9.04 (s, 1H), 7.03 (d, J = 9.7 Hz, 1H), 6.55 (s, 1H), 6.46 (s, 1H), 4.02 (m, 1H), 3.63 (s, 3H), 2.85 (dd, J = 13.8, 4.2 Hz, 1H), 2.68 (dd, J = 13.2, 9.9 Hz, 1H), 2.13 (s, 3H), 1.34 (s, 9H).

Scheme 1. Reagents and conditions. (a) *m*-chloroperoxybenzoic acid, $CHCl_3$, rt, 6 h; (b) 12 N aq HCl, CH_3OH, 10 h, 91%; (c) $SOCl_2$, CH_3OH, reflux, 24 h, 94%; (d) Boc_2O, Et_3N, CH_2Cl_2, 91%; and (e) LiOH, CH_3OH–H_2O, 93%.



Scheme 2. Reagents and conditions. (a) $LiBH_4$, THF, 24 h, 91%; (b) 6 N aq HCl, CH_3OH, reflux, 6 h, 87%; (c) $BnCH_2CHO$, HOAc, 4 Å molecular sieves, CH_2Cl_2–CF_3CH_2OH, −10°C, 87%; and (d) H_2 (50 psi), $Pd(OH)_2$, CH_3OH, 12 h, 86%.



Synthesis of compound 6. To a solution of compound **5** (0.63 g, 3.0 mmol), acetic acid (0.45 g, 0.44 mL, 7.5 mmol, 2.5 equiv.), and the 4 Å molecular sieves (0.6 g) in CH_2Cl_2–CF_3CH_2OH (7:1, v/v, 12 mL), a solution of benzyloxyacetaldehyde (495 mg, 3.3 mmol, 1.1 equiv.) in dichloromethane (4 mL) was added slowly *via* syringe over 60 min at −10°C. After being stirred at −10°C for 8 h, the reaction mixture was diluted with dichloromethane and filtered. The filtrate was concentrated under reduced pressure, and the residue was purified by column chromatography ($CHCl_3$:CH_3OH:NEt_3 = 100:1:0.2) to afford compound **6** (0.89 g, 87%) as a white solid. $[\alpha]_D^{20}$: −115.2 (c 0.5, CH_3OH). m.p: 109–111°C. HRMS calcd. for $C_{20}H_{26}NO_4$ ($M + H^+$) 344.1856 Da, Found 344.1885 Da.

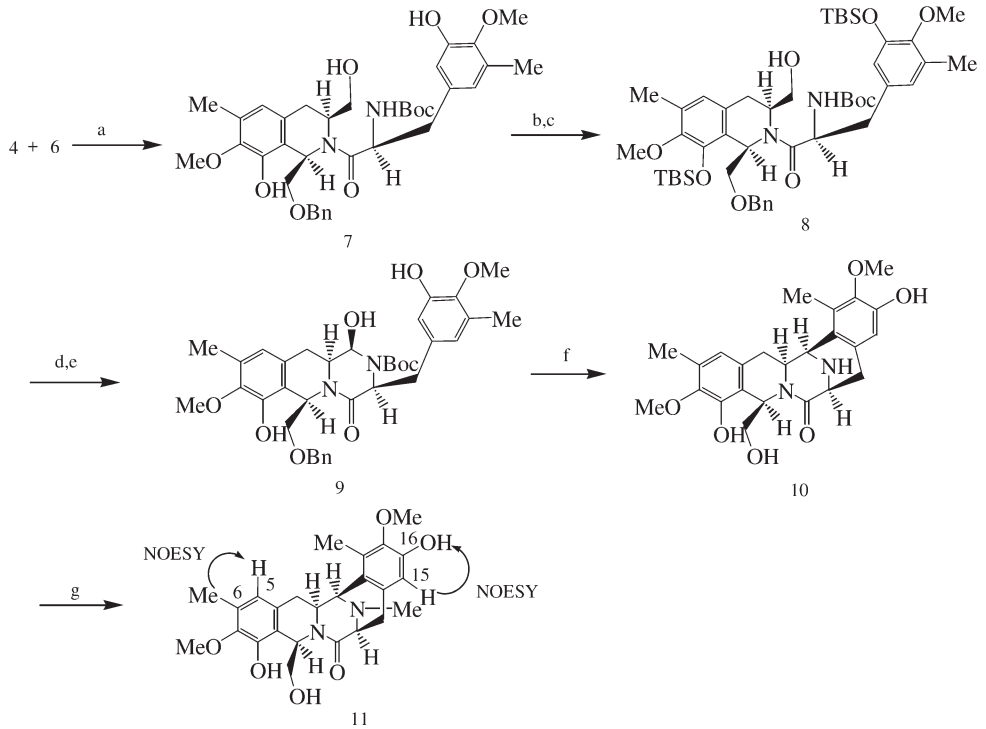
^{1}H-NMR (300 MHz, dimethyl sulfoxide-d_6): δ 8.65 (s, 1H), 7.32 (m, 5H), 6.37 (s, 1H), 4.70 (t, 1H), 4.49 (dd, J = 17.1, 12.6 Hz, 2H), 4.31 (brd, 1H), 4.13 (dd, J = 8.7, 2.7 Hz, 1H), 3.59 (s, 3H), 3.46 (m, 1H), 3.42 (d, J = 8.4 Hz, 1H), 3.36 (m, 1H), 3.33 (s, 1H), 2.68 (m, 1H), 2.42 (brd, J = 15 Hz, 1H), 2.28 (dd, J = 14.1, 11.1 Hz, 1H), 2.14 (s, 3H). ^{13}C-NMR (75 MHz, dimethyl sulfoxide-d_6): δ 146.7, 143.8, 138.7, 132.6, 128.1, 128.0, 127.3, 127.2, 120.9, 73.8, 72.0, 65.2, 59.9, 54.9, 53.9, 53.0, 33.0, 15.3.

Synthesis of compound 7. To a solution of tetrahydroisoquinoline **6** (1.16 g, 3.37 mmol) and triethylamine (1.17 mL, 8.42 mmol, 2.5 equiv.) in CH_2Cl_2 (70 mL) at 0°C, *N*-Boc amino acid **4** (1.20 g, 3.71 mmol, 1.1 equiv.) was added followed by BOPCl (0.94 g, 3.71 mmol, 1.1 equiv.) in portions. The mixture was aged for 6 h at room temperature. Water (40 mL) and 2*M* HCl were added to pH 1.5, and the organic phase was separated. The organic phase was washed with saturated aqueous $NaHCO_3$, brine, and dried over Na_2SO_4, and concentrated by rotary evaporation. The residue was purified by column chromatography ($CHCl_3$) to provide **7** (1.64 g, 75%) as a white solid. HRMS calcd. for $C_{36}H_{46}N_2O_9$ ($M + H^+$) 651.3281 Da, Found 651.3282 Da.

^{1}H-NMR (300 MHz, $CDCl_3$): δ 7.81 (s, 1H), 7.29 (m, 5H), 6.67–6.26 (m, 3H), 6.18 (m, 1H), 5.93 (d, J = 5.1 Hz, 1H), 5.56 (d, J = 7.2 Hz, 1H), 5.28 (d, J = 7.2 Hz, 1H), 5.93 (dd, J = 7.5 Hz, 1H), 4.98 (m, 1H), 4.67 (m, 1H), 4.48 (m, 1H), 4.43 (m, 1H), 3.94 (m, 1H), 3.88 (m, 1H), 3.75 (s, 3H), 3.61 (s, 3H), 3.54 (m, 1H), 3.45(m, 1H), 3.10–2.74 (m, 4H), 2.23 (s, 3H), 2.22 (s, 3H), 1.42–1.34(s, 9H).

Synthesis of compound 8. To a solution of compound **7** (0.786 g, 1.21 mmol) in CH_2Cl_2 (45 mL), TBSCl (1.002 g, 5.4

Scheme 3. Reagents and conditions. (a) BOPCl, Et_3N, CH_2Cl_2, 88%; (b) TBSCl, Et_3N, DMAP, CH_2Cl_2, rt, 88%; (c) HCOOH, THF, H_2O, 92%; (d) Dess–Martin periodinane, CH_2Cl_2, 94%; (e) TBAF, THF, 2 h, 90%; (f) CF_3COOH, 82%; and (g) HCHO, $NaBH_3CN$, HOAc, CH_3OH, 83%.



mmol, 4 equiv.), triethylamine (1.9 mL, 7.26 mmol, 6 equiv.), and 4-dimethylamino pyridine (DMAP) (111 mg, 0.6 mmol, 0.5 equiv.) were added. The solution was stirred for 24 h and quenched with saturated aqueous NH_4Cl (30 mL). The phases were separated, and the aqueous phase was extracted with CH_2Cl_2 (30 mL × 2). The combined organic phase was washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. The residue was purified by column chromatography (5% EtOAc in *n*-hexane) to provide a yellow oil (694 mg, 88%).

The yellow oil (546 mg, 0.55 mmol) was dissolved in THF–HCO_2H–H_2O (6:3:1; 20 mL), the solution was stirred for 2 h at room temperature. The solution was concentrated by rotary evaporation, and the residue was dissolved in EtOAc (50 mL). Then, the organic phase was washed with saturated aqueous $NaHCO_3$, brine, and dried over Na_2SO_4. After concentration of the solution by rotary evaporation, the residue was purified by column chromatography (10% EtOAc in *n*-hexanes) to provide **8** (445 mg, 92%) as a white solid.

^{1}H-NMR (300 MHz, dimethyl sulfoxide-d_6): δ 7.23 (m, 5H), 6.63 (s, 1H), 6.55 (s, 1H), 6.47 (s, 1H), 6.07 (m, 1H), 4.87 (brs, 1H), 4.65 (brd, J = 5.7 Hz, 1H), 4.50 (d, J = 12.6 Hz, 1H), 4.46 (d, J = 10.5 Hz, 1H), 4.35 (brs, 1H), 3.88 (brs, 1H), 3.61 (m, 2H), 3.57 (s, 3H), 3.54 (m, 2H), 3.50 (s, 3H), 2.86 (m, 2H), 2.74 (d, J = 4.8 Hz, 2H), 2.17 (s, 3H), 2.15 (s, 3H), 1.32 (s, 9H), 0.96 (s, 9H), 0.94 (s, 9H), 0.12 (s, 6H), 0.11(s, 6H).

Synthesis of compound 9. To a solution of compound **8** (298 mg, 0.34 mmol) in CH_2Cl_2 (25 mL) at 0°C, Dess–Martin periodinane (228 mg, 0.51 mmol, 1.5 equiv.) was added, and the solution was stirred for 2 h at room temperature. The reaction was quenched with two drops of 2-propanol. The solution was washed with saturated aqueous $NaHCO_3$, brine, and dried over Na_2SO_4, and concentrated by rotary evaporation. The residue was purified by column chromatography (5% EtOAc in *n*-hexanes) to provide **12** (271 mg, 91%) as a white solid.

The white solid (245 mg, 0.28 mmol) was dissolved in THF (25 mL). To this solution, tetrabutylammonium fluoride (TBAF) was added (1.0*M* in THF, 0.84 mL, 0.84 mmol, 3.0 equiv.) at 0°C, and the solution was stirred for 1 h. The reaction was quenched with saturated aqueous NH_4Cl (20 mL) and extracted with EtOAc (30 mL × 3). The combined organic phase was washed with saturated brine, dried over Na_2SO_4, and concentrated by rotary evaporation. The residue was purified by column chromatography (1% CH_3OH in CH_2Cl_2) to provide **9** (181 mg, 90%) as a white solid. HRMS calcd. For $C_{36}H_{45}N_2O_9$ ($M + H^+$) 649.3125 Da, Found 649.3148 Da.

^{1}H-NMR (300 MHz, dimethyl sulfoxide-d_6): δ 8.94 (s, 3H), 7.24 (m, 5H), 6.64 (s, 1H), 6.60 (d, J = 4.8 Hz, 1H), 6.56 (s, 1H), 6.52 (s, 1H), 5.77 (m, 1H), 5.66 (m, 1H), 4.49 (d, J = 12.6 Hz, 2H), 4.38 (d, J = 12.6 Hz, 1H), 3.75 (dd, J = 9.9, 5.4 Hz, 1H), 3.64 (s, 3H), 3.62 (s, 3H), 3.59 (m, 1H), 3.12 (d, J = 12.6, 1H), 3.04 (d, J = 9.6 Hz, 2H), 2.62 (d, J = 12.3, 1H), 2.19 (s, 3H), 2.14 (s, 3H), 1.07 (s, 9H).

Synthesis of compound 11. To trifluoroacetic acid (TFA) (2 mL, 23 mmol), compound **9** was added (20 mg, 0.23 mmol) in one portion, and the mixture was stirred for 1 h at room temperature under argon atmosphere. Then, the reaction mixture was poured into 3 mL of ice-water, basified with saturated aqueous $NaHCO_3$ with stirring, and the whole mixture was extracted with EtOAc (20 mL × 3). The combined organic phase was washed with brine, dried over Na_2SO_4, and evaporated under reduced pressure. The residue was dissolved in MeOH (3 mL). To the solution, HCHO (0.22 mL, 37%), $NaBH_3CN$ (25 mg, 0.40 mmol), and CH_3COOH (0.4 mL) were added, and the mixture was stirred at room temperature for 2 h and concentrated. The residue was dissolved in EtOAc (20 mL), and saturated aqueous $NaHCO_3$ was added. Then the organic layer was separated and washed with saturated aqueous NaCl and dried over anhydrous Na_2SO_4 for 10 h. The organic layer was concentrated under reduced pressure, and the resultant residue was purified by flash column chromatography (EtOAc:-CH_3OH:Et_3N = 100:2:0.2) to afford compound **11** (8 mg, 47.7%). $[\alpha]_D^{20}$: −115.4 (c 4.8, CH_3OH). HRMS calcd. for 15 ($M + H^+$) 545.2646 Da, Found 545.2668 Da.

^{1}H-NMR (600 MHz, dimethyl sulfoxide-d_6): δ 9.01 (s, 1H, 16-OH), 8.86 (s, 1H, 8-OH), 7.20 (dd, 2H, Bn-H, J = 7.2), 7.16 (t, 1H, Bn-H, J = 7.2), 6.81 (d, 2H, Bn-H, J = 7.2), 6.49 (s, 1H, 5-H), 6.49 (s, 1H, 15-H), 5.55 (dd, 1H, 1-H, J = 3.6), 4.30 (d, 1H, 11-H, J = 3.0), 3.848 (m, 1H, 3-H), 3.82 (brs, 2H, 22-H), 3.625 (d, 1H, 21-H, J = 7.2), 3.59 (s, 3H, 17-H), 3.57 (s, 3H, 7-H), 3.39 (dd, 1H, 21-H, J = 10.2, 3.6), 3.30 (m, 1H, 14-H), 3.03 (dd, 1H, 14-H, J = 16.8, 7.2), 2.79 (m, 1H, 13-H), 2.79 (m, 1H, 4-H), 2.37 (dd, 1H, 4-H, J = 13.8), 2.21 (s, 3H, N-CH_3), 2.15 (s, 3H, 6-CH_3), 2.07 (s, 3H, 16-CH_3); ^{13}C-NMR (150 MHz, dimethyl sulfoxide-d_6): δ 70.61, 146.54, 145.85, 144.25, 144.16, 138.58, 132.89, 130.09, 129.11, 128.80, 127.97, 126.89, 126.68, 123.44, 119.83, 119.28, 114.12, 72.36, 71.08, 61.81, 59.90, 59.04, 53.18, 50.02, 48.84, 34.46, 31.99, 30.67, 15.44, 13.43.

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