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A practical and efficient preparation of (−)-(4a*S*,5*R*)-4,4a,5,6,7,8-hexahydro-4a,5-dimethyl-2(3*H*)-naphthalenone: a key intermediate in the synthesis of (−)-dehydrofukinone

Rossana A. Schenato,[b] Éverton M. dos Santos,[a] Beatriz S. M. Tenius,[a,*] Paulo R. R. Costa,[b]
Ignez Caracelli[c] and Julio Zukerman-Schpector[c]

[a]*Instituto de Química, Universidade Federal do Rio Grande do Sul, Av. Bento Gonçalves, 9500-9150-970 Porto Alegre, Brazil*
[b]*NPPN, Universidade Federal do Rio de Janeiro, Rio de Janeiro, RJ, Brazil*
[c]*Departamento de Química, Universidade Federal de São Carlos, SP and Instituto de Química, USP, SP, Brazil*

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Abstract—A novel diastereoselective route to octalone (−)-**1** has been developed. The key step involves an asymmetric Michael addition of the corresponding chiral secondary enamines derived from (*S*)-(−)-1-phenylethylamine and (3*R*)-2,3-dimethylcyclohexanone to methyl vinyl ketone. This enone was successfully transformed into the eremophilane-type sesquiterpenoid (−)-dehydrofukinone. © 2001 Elsevier Science Ltd. All rights reserved.

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1. Introduction

In connection with our ongoing programme of synthesis, we have recently been concerned with the development of viable procedures for the preparation of (−)-(4a*S*,5*R*)-4,4a,5,6,7,8-hexahydro-4a,5-dimethyl-2(3*H*)-naphthalenone **1**. A survey of the literature revealed few syntheses of (±)-**1**, most of which were non-stereoselective and led to a mixture of the two diastereoisomers, which meant the synthesis was low yielding. Additionally, the diastereoisomers were difficult to separate.[1] Recently, two multistep syntheses of (+)-**1**[2,3] and one of (−)-**1**[4] have also been presented. In the synthesis of (−)-**1**, the target molecule was synthesised in five steps from commercially available (*R*)-3-methylcyclohexanone, with an overall yield of 28%. Herein, we report a shorter, straightforward and highly diastereoselective method for the preparation of (−)-**1**, starting from (+)-pulegona. We also demonstrated the utility of this methodology by the synthesis of (−)-dehydrofukinone **2**, the enantiomer of the natural eremophilane-type sesquiterpenoid, isolated from leaves of *Arctium lappa* L.[5] (Fig. 1).

The enantioselective version of the Robinson annulation using chiral imines, developed by d'Angelo and Pfau[6] (deracemising alkylation protocol), has become one of the most powerful tools available for the construction of homochiral octalone units of the type **1**. In a previous study,[7] we used this strategy to synthesise octalones from (−)-dihydrocarvone. We showed that although the resident stereogenic centre in the cyclohexanone ring and the incoming electrophile have a 1,4-relationship in the transition state, the stereogenic centre still influences the stereoselectivity of the conjugate addition step. While a matched situation was observed when (*S*)-phenylethylamine [(*S*)-PEA] was used as a chiral auxiliary, leading to the corresponding adduct in excellent enantiomeric purity, a mismatched situation occurred with (*R*)-phenylethyl amine [(*R*)-PEA] (Scheme 1).

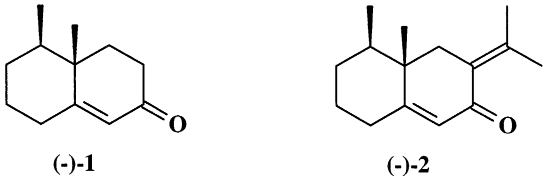

Figure 1.

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[*] Corresponding author. Fax: 00 55 51 319 14 99; e-mail: tenius@if.ufrgs.br

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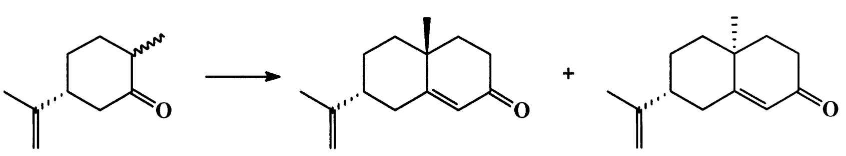
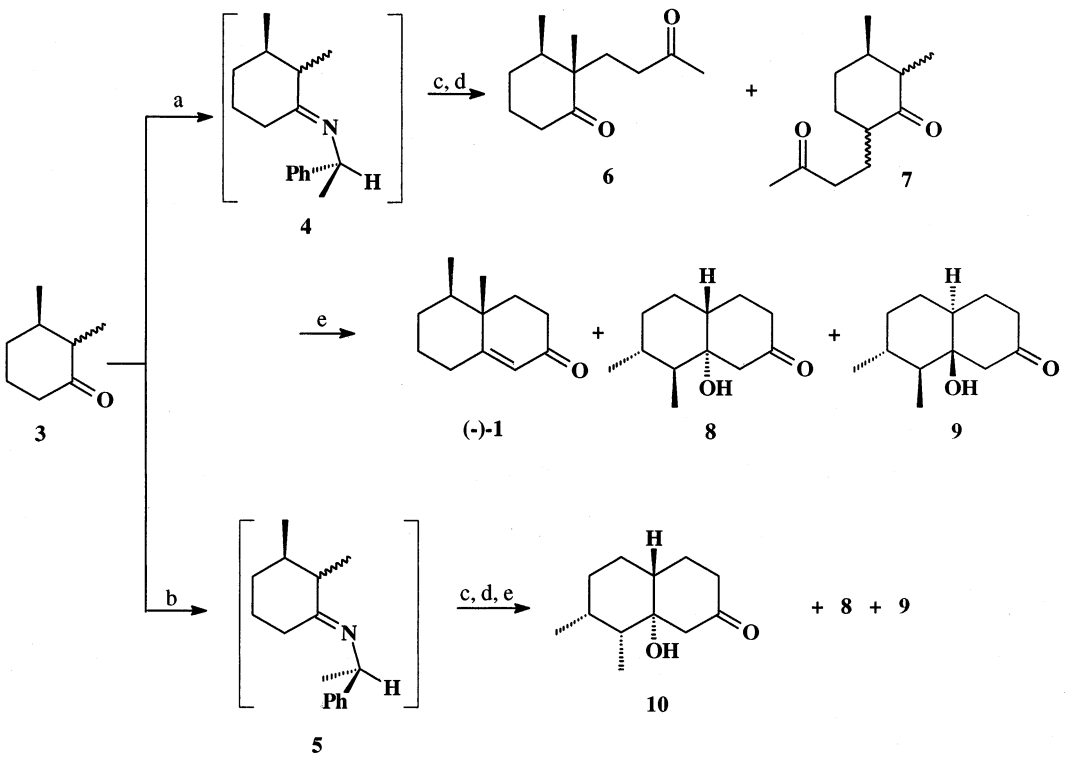


(S)-PEA > 98 : 1
(R)-PEA 1 : 4

Scheme 1.



Scheme 2. (a) (*S*)-PEA, *p*-TsOH, benzene, reflux, 5 h; (b) (*R*)-PEA, *p*-TsOH, benzene, reflux, 5 h; (c) MVK, THF, 3 days, rt; (d) 10% AcOH:H_2O, 0°C, 1.5 h, rt, 69%; (e) 0.05 M NaOMe, THF/MeOH 8:1 (v/v), 0°C.

Herein, we describe the use of (3*R*)-2,3-dimethylcyclohexanone as a starting material in the deracemising alkylation protocol. A multigram-scale enantioselective synthesis of (−)-**1**, and its transformation in the sesquiterpenoid (−)-dehydrofukinone, is also described.

2. Results and discussion

The requisite (3*R*)-2,3-dimethyl cyclohexanone **3** was prepared from (*R*)-(+)-pulegone, according to an alkylation and retro-aldolisation[8] process, in 68% yield. The addition of the chiral imine derivative **4** prepared from [(*S*)-PEA], to methyl vinyl ketone (MVK) led, after hydrolysis, to diketones **6** and **7**, in a ratio of 3.4:1, as determined by GC analysis. The diketones were subjected to base-catalysed aldol condensation, carried out in a 0.05 M solution of NaOMe in THF/MeOH 8:1 (v/v) at 0°C. Under these conditions, the intermediate ketols do not have the same behaviour, dehydration being easier for the aldol product from diketone **6**, to give the bicyclic enone (−)-**1** (in 43% yield from **6**/**7** and 30% from **3**) and the β-hydroxyketones **8** and **9** (derived from the cyclisation of **7**), easily separated by flash chromatography, together with 6% of the corresponding inseparable octalone (Scheme 2).

When the same protocol was applied to [(*R*)-PEA], the corresponding enamine derived from ketone **3** on reaction with MVK gave, after hydrolysis, only regioisomeric adducts that, under base-catalysed conditions, underwent aldol reaction leading to the corresponding β-hydroxyketones (Scheme 2).

Optical rotations were measured and the products were further characterised according to the NMR data, which were assigned unequivocally by 2D NMR experiments (COSY, HETCOR, HMQC), in conjunction

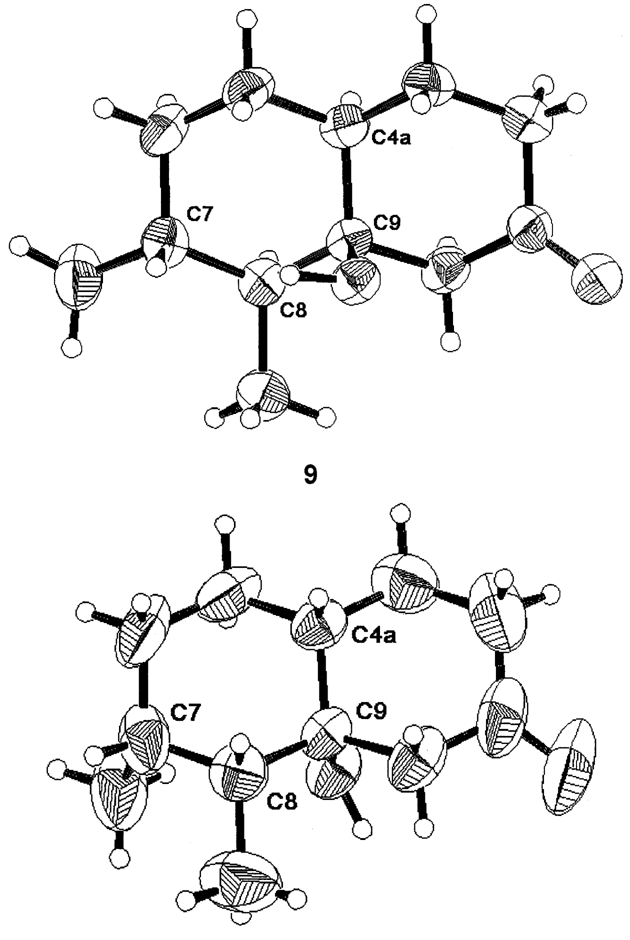
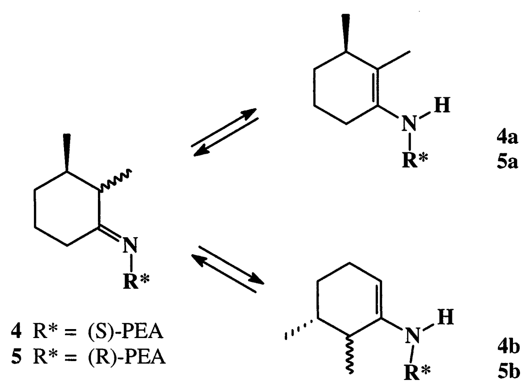


Figure 2. X-ray crystal structures of ketols **9** and **12**

with lanthanide shift methodology using Eu(fod)$_3$. The absolute configuration of β-hydroxyketones **9** and **10** was determined as C(4a)-(*S*); C(7)-(*R*); C(8)-(*S*); C(9)-(*R*) and C(4a)-(*R*); C(7)-(*R*); C(8)-(*R*); C(9)-(*S*), respectively, by single-crystal X-ray crystallographic analysis. Fig. 2 shows ORTEP drawings of the X-ray crystal structures of compounds **9** and **10**.

This protocol, when applied to (3*R*)-2,3-dimethylcyclohexanone although highly diastereoselective, is of low regioselectivity. This unexpected result[9] can be rationalised in terms of loss of regiocontrol of imines **4** and **5** in which the tautomeric equilibrium towards the more substituted secondary enamines **4a** and **5a** is disfavoured on steric grounds (arising from the 2,3-substituents) (Scheme 3). The electronically favoured axial attack of the incoming electrophile to the more stable conformer of the tautomeric enamine **4a** accounts for the high stereoselectivity observed in the formation of diketone **6** (Fig. 3), bearing in mind that the regioselectivities observed in the Michael additions of these imines do not reflect necessarily the populations of these regioisomers in the ground state (Curtin–Hammet principle). On the other hand, enamines **4b** and **5b** support the formation of ketols **8**–**10**.



Scheme 3.

4a **5a**

Figure 3.

Houk's model for the conformational transmission of chirality[10] provides a rationalisation for the different degree of loss of regiocontrol when using the two antipodal amines as chiral auxiliaries in this process: the preferred conformation for **4a** and **5a** being diastereomer half-chairs, could not have the same stability leading, in the case of **5**, to a predominance of the least substituted enamine **5b**, through an equilibration process, favouring the formation of regioisomeric products (Fig. 3).

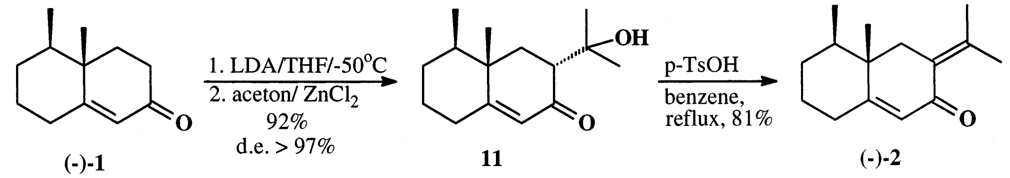
Enolisation of octalone (−)-**1** with LDA/THF/−50°C gave the corresponding enolate that, on addition to acetone/ZnCl$_2$, led to the aldol adduct **11**, in 88% yield, with >97% diastereoselectivity. Alcohol **11** was dehydrated to (−)-dehydrofukinone **2** with *p*-toluenesulfonic acid in refluxing benzene[11] (Scheme 4).

In summary, we have developed an efficient and stereoselective route toward enone (−)-**1**, a versatile building block for the synthesis of sesqui- and diterpenes, starting from commercially available (*R*)-pulegone and using a chiral version of the Robinson annulation reaction. This three-step synthesis includes a selective base-catalysed aldol condensation that can selectively afford the β-hydroxy ketone, or its dehydration products, according to the stability of the ketols. From enone (−)-**1** we have prepared (−)-dehydrofukinone in two steps and 71% overall yield.

3. Experimental

3.1. General

^{1}H and ^{13}C NMR spectra were recorded on a Varian VXR-200 or INOVA-300 spectrometer and signals are expressed downfield from the internal standard tetra-



Scheme 4.

methylsilane. Chemical shifts (δ) are reported in ppm relative to $CDCl_3$ and coupling constants (J) in hertz. ^1H NMR data are reported in the following order: chemical shift, multiplicity (s, singlet; d, doublet; t, triplet; m, multiplet) and number of hydrogens. ^{13}C NMR spectra were interpreted with aid of APT experiments. The complete assignment of hydrogens and carbons atoms of all ketols was made from lanthanide displacement shift methodology, using tris(1,1,1,2,2,3,3-heptafuoro-7,7-dimethyl-4,6-octadionate)europium(III), $Eu(fod)_3$. Optical rotations were measured on a Perkin–Elmer 341 polarimeter at the sodium D line. Infrared spectra were determined with a Mattson Galaxy series FT-3000 spectrophotometer and melting points were determined with an Electrothermal IA 9000 series digital melting point apparatus. Silica gel 60 F_{254} plates were used for TLC; 230–400 mesh silica gel was used for column chromatography. All chemicals and solvents were of analytical grade and were used without further purification. THF was distilled from sodium/benzophenone under nitrogen immediately before use. Reactions were carried out under argon when necessary. Organic extracts were dried over $MgSO_4$. Gas chromatography analysis were carried out with a Shimadzu GC-174 chromatograph DB-1 column.

3.2. Preparation of diketones 6 and 7

To a solution of **3** (5.0 g, 40.36 mmol) in benzene (26.8 mL) in a 50 mL round-bottomed flask equipped with a Dean–Stark apparatus was added (*S*)-(−)-PEA (7.0 mL, 6.22 g, 51.44 mmol), followed by a catalytic amount of *p*-toluenesulfonic acid. The reaction mixture was refluxed for 5 h with azeotropic removal of water. After cooling, the mixture was concentrated under reduced pressure and then distilled (0.01 mmHg, 120°C) to afford the corresponding imine (7.53 g, 34.7 mmol, 82%). To a solution of this imine in dry THF (13 mL) was added dropwise freshly distilled MVK (3.81 mL, 45.86 mmol), and the reaction mixture was stirred at room temperature for 72 h. Aqueous acetic acid solution (10%, 13.5 mL) was added and the mixture was stirred for 1.5 h. The solvents were removed under reduced pressure, and aqueous HCl (1N, 10 mL) was added to the residual oil. The mixture was then extracted with ether. The ethereal extract was washed with brine and concentrated in vacuo to afford 5.49 g of crude material. Flash chromatography on silica gel (hexane:EtOAc, 85:15) gave of a mixture of diketones **6** and **7** (4.68 g, 23.9 mmol, 69%, a mixture of **6**:**7** = 3.4:1 as determined by capillary GC on a DB-1 column) (oven parameters: initial temperature 50°C; final temperature 250°C; rate 10°C min^{-1}; temperature detection 250°C, carrier gas H_2).

Compound **6**: oil; FT-IR: 1709, 1647 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.92 (d, 3H, $J = 6.7$ Hz, Me-3), 1.02 (s, 3H, Me-2), 1.49–2.11 (m, 7H, H-3, H-4, H-5, H-1′), 2.15 (s, 3H, H-4′), 2.20–2.58 (m, 4H, H-2′, H-6); ^{13}C NMR (50 MHz, $CDCl_3$): δ 15.29 (Me-2), 18.50 (Me-3), 24.40 (C-5), 28.86 (C-4), 29.14 (C-4′), 29.78 (C-1′), 38.12 (C-2′), 38.58 (C-6), 38.63 (C-3), 51.16 (C-2), 208.88 (C-3′), 215.39 (C-1).

3.3. Cyclisation of diketones 6 and 7

To diketones **6** and **7** (1.80 g, 9.21 mmol) was added cold (0°C) NaOMe (0.05 M, 22.1 mL, 2.14 mmol) in THF/MeOH 8:1 (v/v, prepared by dissolving 0.489 g of Na in 42.7 mL of MeOH, followed by addition of 30 mL of dry THF). The resulting solution was stirred for 4 h at 0°C and then 10% aqueous AcOH (1.0 mL) was added. MeOH was removed under reduced pressure and the mixture was extracted with ethyl ether (3×20 mL). The organic layers were treated with brine, dried over $MgSO_4$ and concentrated in vacuo to afford 1.63 g of crude material. Flash chromatography on silica gel (hexane:AcOEt, 95:5) gave enone (−)-**1** (0.704 g, 43%), and with hexane:AcOEt (90:10) as eluent, gave ketol **8** (0.039 g, 2.2%) and ketol **9** (0.160 g, 9%).

Compound **1**: $[\alpha]_D^{20} = -185.0$ (*c* 1.63, CH_2Cl_2); oil; FT-IR: 1670, 1616 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.92 (d, 3H, $J = 5.86$ Hz, Me-5), 1.11 (s, 3H, Me-4a), 1.35–1.64 (m, 4H, H-6, H-7), 1.72 (dd, 1H, $J = 13.7$, 5.1 Hz, H-4_{ax}), 1.81–1.91 (m, 1H, H-5), 2.04 (ddd, 1H, $J = 13.2$, 5.1, 3.4 Hz, H-4_{eq}), 2.18–2.58 (m, 4H, H-3, H-8), 5.73 (s, 1H, H-1); ^{13}C NMR (50 MHz, $CDCl_3$): δ 15.00 (Me-5), 15.78 (Me-4a), 26.31 (C-7), 30.24 (C-6), 33.14 (C-8), 33.77 (C-3), 35.30 (C-4), 38.77 (C-4a), 42.94 (C-5), 123.79 (C-1), 171.17 (C-8a), 199.40 (C-2).

Compound **8**: mp 119–120°C; FT-IR: 3398, 1709 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.87 (d, 3H, $J = 7.3$ Hz, Me-8), 0.89 (d, 3H, $J = 6.8$ Hz, Me-7), 1.10–1.66 (m, 6H, 2(H-4), 2(H-5), H-8, OH), 1.67–1.98 (m, 3H, H-4a, 2(H-6)), 2.03–2.22 (*n* absorptions, 2H, H-1, H-7), 2.28–2.47 (m, 2H, 2(H-3)), 2.68 (d, 1H, $J = 13.5$ Hz, H-1); ^{13}C NMR (50 MHz, $CDCl_3$): δ 8.92 (Me-8), 19.54 (Me-7), 27.55 (C-5), 28.21 (C-4), 28.68 (C-6), 28.89 (C-7), 35.88 (C-4a), 40.97 (C-3), 45.72 (C-8), 53.02 (C-1), 78.17 (C-8a), 212.01 (C-2).

Compound **9**: mp 63–65°C; $[\alpha]_D^{20}=+5.0$ (*c* 2.0, CH_2Cl_2); FT-IR: 3460, 1693 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.90 (d, 3H, $J=6.1$ Hz, Me-7), 0.91 (d, 3H, $J=5.0$ Hz, Me-8), 1.03–1.28 (m, 2H, H-6, H-8), 1.29–1.63 (m, 4H, 2(H-5), H-7, OH), 1.63–2.04 (m, 4H, 2(H-4), H-4a, H-6), 2.17 (d, 1H, $J=14.0$ Hz, H-1), 2.26–2.53 (m, 2H, 2(H-3)), 2.67 (d, 1H, $J=14.3$ Hz, H-1); ^{13}C NMR (50 MHz, $CDCl_3$): δ 10.70 (Me-8), 20.35 (Me-7), 27.64 (C-5), 28.47 (C-4), 32.78 (C-7), 34.85 (C-6), 40.95 (C-3), 43.50 (C-4a), 47.46 (C-8), 52.06 (C-1), 76.15 (C-8a), 211.29 (C-2).

Compound **10** (obtained from **5**): mp 118°C; $[\alpha]_D^{20}=-5.0$ (*c* 0.18, CH_2Cl_2); FT-IR: 3506, 1709 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.95 (d, 3H, $J=7.1$ Hz, Me-8), 1.00 (d, 3H, $J=7.3$ Hz, Me-7), 1.25–1.54 (m, 2H, H-5, OH), 1.55–2.07 (m, 8H, 2(H-4), H-4a, H-5, 2(H-6), H-7, H-8), 2.09–2.50 (*n* absorptions, 3H, H-1, 2(H-3)), 2.56 (d, 1H, $J=15.6$ Hz, H-1); ^{13}C NMR (50 MHz, $CDCl_3$): δ 12.48 (Me-8), 14.17 (Me-7), 22.94 (C-5), 28.45 (C-4), 32.78 (C-6), 33.41 (C-7), 41.40 (C-3), 42.33 (C-8), 44.21 (C-4a), 52.91 (C-1), 77.10 (C-8a), 210.98 (C-2).

3.4. Preparation of (4a*S*,5*R*)-4,4a,5,6,7,8-hexahydro-3-(1-hydroxy-1-methyl)-4a,5-dimethylnaphthalen-2(3*H*)-one 11

To a solution of lithium diisopropylamide (0.19 mL, 0.131 g, 1.30 mmol) in anhydrous THF (1.5 mL) and hexane (0.86 mL) was added dropwise a solution of enone **1** (0.206 g, 1.12 mmol) at −50°C under argon, and the resulting solution was stirred at −50 to −36°C for 30 min. A solution of zinc chloride (0.236 g, 1.74 mmol) in THF (1.5 mL) was added and, after stirring at −36°C for 5 min, a solution of acetone (0.11 mL, 0.09 g, 1.55 mmol) in THF (0.7 mL) was added. The mixture was stirred at −36°C for 5 min and was then allowed to warm to 0°C and stirred for 2 h. Aqueous ammonium chloride was added and the product was extracted with ether. The combined ethereal extracts were washed with water and brine, and the ether was removed. The organic residue was flash chromatographed on silica gel (hexane:AcOEt, 96:4) to furnish alcohol **11** (0.233 g, 88%).

Compound **11**: oil; FT-IR: 3468, 1651 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.96 (d, 3H, $J=6.3$ Hz, Me-5), 0.97 (s, 3H, Me-4a), 1.32–1.64 (m, 7H), 1.85 (s, 3H, Me(2′)), 2.09 (s, 3H, Me-1′), 2.15–2.37 (m, 3H), 2.88 (d, 1H, $J=13.2$ Hz, H-4_{eq}), 5.74 (s, 1H, H-1); ^{13}C NMR (50 MHz, $CDCl_3$): δ 15.43 (Me-5), 16.01 (Me-4a), 22.00 (Me(2′)), 22.54 (Me-1′), 26.47 (C-7), 30.51 (C-8), 32.51 (C-6), 41.01 (C-4), 41.86 (C-4a), 42.48 (C-5), 126.02 (C-1), 128.12 (C-3), 142.14 (C-1′), 168.66 (C-8a), 192.24 (C-2).

3.5. (4a*S*,5*R*)-4,4a,5,6,7,8-Hexahydro-4a,5-dimethyl-3-(1-methylethylidene)-naphthalen-2-(3*H*)-one (−)-2

A solution of alcohol **11** (0.03 g, 0.127 mmol) in anhydrous benzene (3 mL), containing a catalytic amount of fused *p*-toluenesulfonic acid, was heated under reflux for 2.5 h using a Dean–Stark water separator. The reaction mixture was poured into a cold solution of sodium hydrogen carbonate, and the organic layer was washed with water and brine, and evaporated to dryness. The organic residue was purified by flash chromatography on silica gel (hexane:AcOEt, 99:1) to furnish dehydrofukinone **2** (0.023 g, 81%).

Compound **2**: oil; $[\alpha]_D^{20}=-169.0$ (*c* 1.05, $CHCl_3$); FT-IR: 1659, 1620 cm^{-1}; ^1H NMR (200 MHz, $CDCl_3$): δ 0.96 (d, 3H, $J=6.3$ Hz, Me-5), 0.97 (s, 3H, Me-4a), 1.32–1.64 (m, 7H), 1.85 (s, 3H, Me(2′)), 2.09 (s, 3H, Me-1′), 2.15–2.37 (m, 3H), 2.88 (d, 1H, $J=13.2$ Hz, H-4_{eq}), 5.74 (s, 1H, H-1); ^{13}C NMR (50 MHz, $CDCl_3$): δ 15.43 (Me-5), 16.01 (Me-4a), 22.00 (Me(2′)), 22.54 (Me-1′), 26.47 (C-7), 30.51 (C-8), 32.51 (C-6), 41.01 (C-4), 41.86 (C-4a), 42.48 (C-5), 126.02 (C-1), 128.12 (C-3), 142.14 (C-1′), 168.66 (C-8a), 192.24 (C-2).

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