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Synthesis of 6-Hydroxycarvone Derivatives and Their Oxidative Decyclization with Lead Tetraacetate

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Abstract—6-Hydroxy derivatives of R-(–)-carvone and 7,8-epoxycarvone were synthesized and subjected to oxidative ring cleavage by the action of lead tetraacetate. These reactions followed different patterns, depending on the substrate structure.

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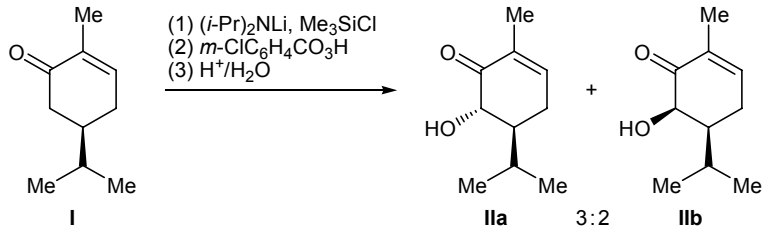
α-Hydroxy ketone moiety is a structural fragment of many natural compounds [1, 2]; it is necessary for cleavage of C–C bonds by the action of specific reagents, such as HIO_4, $Pb(OAc)_4$, $(NH_4)_2Ce(NO_3)_6$, etc. [3–5]. In this work we examined the possibility for oxygenation of (*R*)-(–)-carvone at C^6 according to Rubottom (transformation of ketone into α-hydroxy ketone through silyl enol ether, followed by epoxidation and rearrangement) [6, 7] and oxidative cleavage of the resulting hydroxy ketones with $Pb(OAc)_4$. α-Hydroxylation of ketones of the carvone series was reported previously, namely carvotanacetone (**I**) was oxidized to stereoisomeric 6-hydroxy derivatives **IIa** and **IIb** [8] (Scheme 1).

Analogous reaction sequence in the oxidation of (*R*)-(–)-carvone (**III**) led to the formation of 6β-hydroxy isomer **IVa** as the major product (yield 34%), the yield of isomer **IVb** did not exceed 6%, and 20% of initial carvone **III** was recovered from the reaction mixture. Unlike carvotanacetone (**I**) which gave rise to comparable amounts of stereoisomeric compounds **IIa** and **IIb**, the oxidation of (*R*)-(–)-carvone was characterized by good stereoselectivity (Scheme 2).

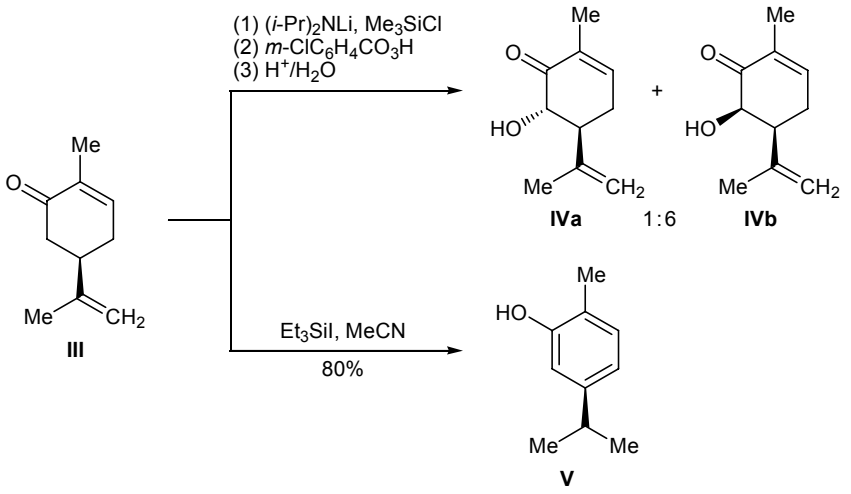
Moderate yields of isomers **IVa** and **IVb** are likely to be determined by considerable contribution of side processes. For example, carvone tends to undergo aromatization with formation of phenol (**V**, carvacrol) [9]. The same compound was obtained in high yield when enolsilylation of (*R*)-(–)-carvone was performed under conditions of thermodynamic control with triethylsilyl iodide generated *in situ*. Obviously, aromatization of carvone during the above process is facilitated by the presence of exocyclic double bond. Epoxidation of that bond made it possible to convert epoxide **VI** into isomeric hydroxy ketones **IXa** and **IXb** at a ratio of 10 : 1, the overall yield being 64% (Scheme 3).

α-Hydroxy ketones **IV** and **IX** were then subjected to oxidative cleavage by the action of lead tetraacetate [3]. Taking into account that the expected products, the corresponding carboxy enals, could be labile and difficultly identifiable, they were treated (without isolation) with $NaBH_4$ to obtain more stable hydroxy derivatives. As might be expected, oxidative cleavage of diastereoisomer mixture **IVa**/**IVb** by the action of $Pb(OAc)_4$ in benzene occurred at a high rate and led to

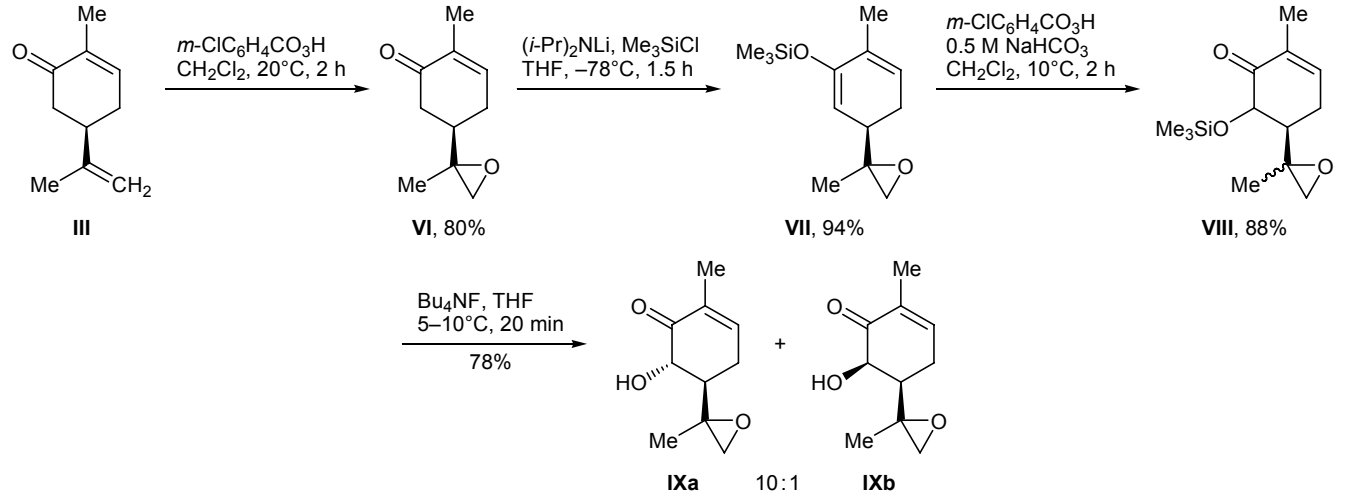
Scheme 1.



Scheme 2.



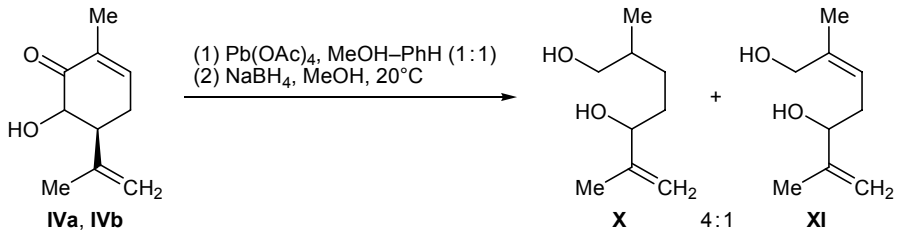
Scheme 3.



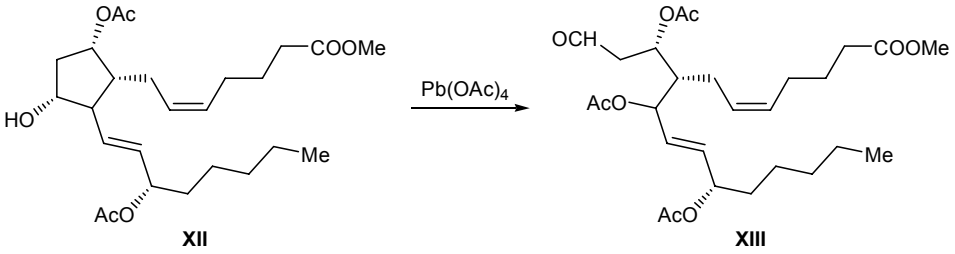
the formation of a mixture of compounds (according to the TLC data) which were treated with $NaBH_4$. Analysis of the reaction mixture by TLC revealed considerably smaller number of components, and chromatographic separation on a column charged with silica gel gave diols **X** and **XI** (Scheme 4). In keeping with the ^1H and ^{13}C NMR data, diol **X** was a mixture of diastereoisomers at a ratio of 2:1, and configuration of the chiral center in compound **XI** was not determined.

Structures **X** and **XI** suggest that oxidative cleavage of hydroxy ketone **IV** is accompanied by loss of one carbon atom. As concerns possible ways of formation of diols **X** and **XI**, the following should be noted. Analogous pattern was described in [10]: cleavage of the C^{11}–C^{12} bond in prostaglandin $F_{2\alpha}$ 9,15-diacetate (**XII**) by the action of $Pb(OAc)_4$ afforded compound **XIII** (Scheme 5). Presumably, ring opening in **IVa** and **IVb** follows a similar scheme with formation of

Scheme 4.

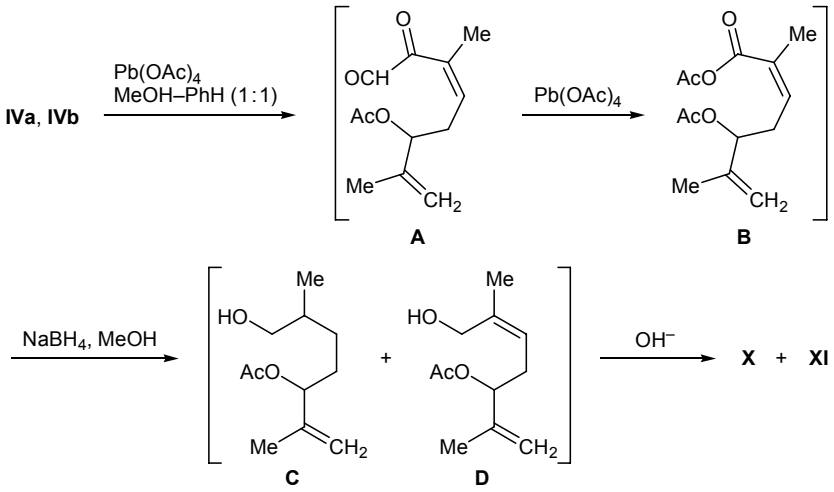


Scheme 5.



XII **XIII**

Scheme 6.



intermediate acetoxy derivative **A** (Scheme 6). The subsequent fragmentation of **A** gives anhydro derivative **B** as key intermediate in reductive transformations leading to compounds **X** and **XI**. Intermediate **B** contains an activated carboxy group, so that facile reduction of the conjugated double bond and subsequent reduction of the carboxy group become possible. Hydrolysis of the acetoxy groups in alcohols **C** and **D** is facilitated due to intramolecular assistance by the free OH group (Scheme 6).

Obviously, anomalous reaction course in the oxidation of **IV** with $Pb(OAc)_4$ is related to the presence of a substituted homoallylic alcohol fragment. If the latter is absent, oxidative cleavage of compounds **IXa** and **IXb** leads to hydroxy esters **XV** through intermediate acyclic aldehydo esters **XIV** (Scheme 7).

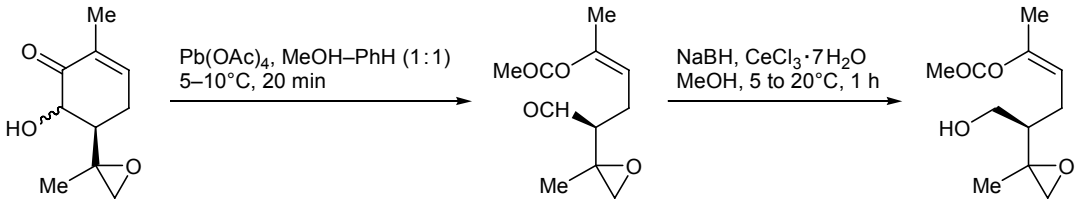
Thus 6-hydroxy derivatives **IVa**/**IVb** and **IXa**/**IXb** obtained, respectively, from (*R*)-(–)-carvone and epoxy carvone underwent oxidative cleavage of the endocyclic C–C bond by the action of $Pb(OAc)_4$ with formation of diols **X** and **XI** and hydroxy ester **XV**. The observed difference in the paths of oxidative decyclization of compounds **IVa**/**IVb** and **IXa**/**IXb** may be attributed to the presence of exocyclic double bond in the former.

EXPERIMENTAL

The IR spectra were recorded on a UR-20 spectrometer from samples prepared as thin films. The ^1H and ^{13}C NMR spectra were recorded from solutions in $CDCl_3$ on a Bruker AM-300 spectrometer operating at 300 and 75.47 MHz, respectively. Analysis by thin-layer chromatography was performed using Sorbfil plates; spots were detected by treatment with an acidified solution of 4-methoxybenzaldehyde. The optical rotations were measured on a Perkin–Elmer 241 MS instrument. The purity of the isolated compounds was checked by GLC on a Chrom-5 chromatograph equipped with a flame ionization detector (stationary phase SE-30; carrier gas helium, flow rate 60 ml/min, oven temperature 50–300°C). The mass spectra were obtained on Shimadzu LCMS-2010 (atmospheric pressure chemical ionization, 20 eV, positive and negative ion detection) and Thermo Finnigan MAT 95XP instruments (electron impact, 70 eV, ion source temperature 200°C).

(5*R*,6*R*)- and (5*R*,6*S*)-6-Hydroxy-5-isopropenyl-2-methylcyclohex-2-en-1-ones IVa and IVb were synthesized according to the procedure described in [8] and were isolated as oily liquids with an overall yield

Scheme 7.



IXa, **IXb** **XIV**, 89% **XV**, 93%

of 40%. Pure diastereoisomers **IVa** (34%) and **IVb** (6%) were isolated by column chromatography on silica gel using ethyl acetate–petroleum ether (1:10) as eluent.

Compound **IVa**. $[\alpha]_D^{20}$ = –23.3° (*c* = 1.63, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.81 t (6H, CH_3, *J* = 2.0 Hz), 2.30–2.50 m (2H, 4-H), 2.65 d.d (*J* = 5.2, 10.7 Hz), 2.69 d.d (1H, 5-H, *J* = 5.2, 10.7 Hz), 3.78 d (1H, OH, *J* = 1.9 Hz), 4.15 d.d (1H, 6-H, *J* = 1.9, 12.6 Hz), 4.89 m (2H, =CH_2), 6.72–6.74 m (1H, 3-H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 15.23 (CH_3), 18.65 (CH_3), 29.52 (C^4), 50.97 (C^5), 74.24 (C^6), 113.49 (=CH_2), 132.89 (C^2), 144.07 ($C^{2'}$), 145.60 (C^3), 200.48 (C^1).

Compound **IVb**. $[\alpha]_D^{20}$ = –83.0° (*c* = 1.72, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.69 s (3H, 2′-CH_3), 1.83 d (3H, 2-CH_3, *J* = 1.4 Hz), 2.49–2.57 m (1H, 4-H), 2.70–2.77 m (1H, 4-H, *J* = 2.7 Hz), 3.19 m (1H, 5-H), 3.59 d (1H, OH, *J* = 2.5 Hz), 4.43 d.d (1H, 6-H, *J* = 2.3, 5.8 Hz), 4.71 s (1H) and 4.86 t (1H, =CH_2, *J* = 1.4 Hz), 6.67 m (1H, 3-H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 15.21 (CH_3), 22.96 (CH_3), 29.55 (C^4), 45.84 (C^5), 74.51 (C^6), 113.66 (=CH_2), 133.61 (C^2), 142.99 ($C^{2'}$), 144.07 (C^3), 199.69 (C^1).

5-Isopropyl-2-methylphenol (V). A solution of 0.45 g (3.0 mmol) of sodium iodide in 5 ml of acetonitrile was added dropwise under stirring to a solution of 0.3 g (2.0 mmol) of carvone (**III**), 0.36 g (2.4 mmol) of chloro(triethyl)silane, and 0.19 ml (4.0 mmol) of triethylamine. The mixture was stirred for 4 h, 20 ml of chloroform and 20 ml of water were added, the aqueous phase was separated and extracted with chloroform, and the extract was combined with the organic phase, washed with a solution of $Na_2S_2O_3$ and a saturated solution of NaCl, and dried over $MgSO_4$. The residue was subjected to column chromatography on silica gel using ethyl acetate–petroleum ether (1:4) as eluent. Yield 0.2 g (67%), colorless oily liquid. ^{1}H NMR spectrum, δ, ppm: 1.29 d (6H, CH_3, *J* = 7.0 Hz), 2.29 s (3H, 2-CH_3), 2.88 m (1H, CH, *J* = 7.0 Hz), 5.44–5.49 m (1H, OH), 6.73 s (1H, 6-H), 6.80 d (1H, *J* = 7.6 Hz), 7.20 d (1H, 3-H, 4-H, *J* = 7.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 15.30 (CH_3), 23.90 (2-CH_3), 33.58 (CH), 45.99 (C^3); 113.01, 118.64, 121.07, 130.77, 148.27, 153.57 (C_{arom}).

(5*R*,2′*R*)- and (5*R*,2′*S*)-2-Methyl-5-(2-methyloxiran-2-yl)cyclohex-2-en-1-ones (VI) (a mixture of diastereoisomers at $C^{2'}$ at a ratio of 3:2**).** (*R*)-(–)-Carvone (**III**), 8.0 g (53.3 mmol), was dissolved in 50 ml of methylene chloride, 15.8 g (64.0 mmol) of 70% *m*-chloroperoxybenzoic acid was added, and the mixture was stirred for 1.5 h at room temperature (until the initial compound disappeared according to the TLC data). The mixture was then treated with a saturated solution of $Na_2S_2O_3$ and stirred for 15 min, the organic phase was separated, washed with a saturated solution of $NaHCO_3$, dried over $MgSO_4$, filtered, and evaporated, and the residue was distilled under reduced pressure. Yield 7.1 g (80%), colorless liquid, bp 120–124°C (7 mm). IR spectrum: ν 1676 cm^{-1}. ^{1}H NMR spectrum, δ, ppm: 1.28 s and 1.30 s (3H, 2′-CH_3), 1.74 s (3H, 2-CH_3), 2.04–2.09 m (1H, 5-H), 2.13–2.45 m (3H, 4-H, 6-H), 2.52–2.58 m (2H, 6-H, 3′-H), 2.64–2.69 m (1H, 3′-H), 6.69–6.72 m (1H, 3-H). ^{13}C NMR spectrum, δ_C, ppm: 15.44 (CH_3), 18.29 and 18.57 (CH_3), 27.57 and 27.76 (C^4), 39.68 and 40.01 (C^6), 40.70 and 41.00 (C^5), 52.23 and 52.44 ($C^{3'}$), 57.56 and 57.68 ($C^{2'}$), 135.19 (C^2), 143.81 and 144.01 (C^3), 198.38 (C^1).

(3*R*,2′*R*)- and (3*R*,2′*S*)-Trimethyl[6-methyl-3-(2-methyloxiran-2-yl)cyclohexa-1,5-dien-1-yloxy]-silanes (VII). A solution of 11.0 ml (78.3 mmol) of diisopropylamine in 20 ml of tetrahydrofuran was cooled to –78°C, 28.0 ml (78.3 mmol) of a 2.8 N solution of butyllithium in hexane was added dropwise under stirring, the mixture was stirred for 30 at –10°C and cooled to –78°C, and a solution of 6.5 g (39.2 mmol) of compound **VI** in 10 ml of THF was added dropwise. The mixture was allowed to warm up to –40°C, stirred for 40 min at that temperature, and cooled to –78°C, 9.9 ml (78.3 mmol) of chloro(trimethyl)silane was added dropwise, and the mixture

was stirred for 1 h at –70°C until the initial compound disappeared and treated with a saturated solution of sodium chloride. The organic phase was separated, the aqueous phase was extracted with methylene chloride (2×20 ml), and the extracts were combined with the organic phase, dried over $MgSO_4$, filtered, and evaporated. Yield 8.8 g (94%), yellow viscous liquid. The product was brought into further step without chromatographic purification.

(5*R*,2′*R*)- and (5*R*,2′*S*)-2-Methyl-5-(2-methyloxiran-2-yl)-6-trimethylsiloxycyclohex-2-en-1-ones (VIII). Crude compound **VII**, 8.8 g (36.9 mmol), was dissolved in 40 ml of methylene chloride, 37 ml of a 1 M solution of $NaHCO_3$ was added, and 11.8 g (48.0 mmol) of 70% *m*-chloroperoxybenzoic acid was added in three portions (through 15-min intervals) under stirring at 10°C. The mixture was stirred for 2 h at 10°C, a saturated solution of $Na_2S_2O_3$ was added, the mixture was stirred for 15 min, and the organic phase was separated, washed with a saturated solution of $NaHCO_3$, dried over $MgSO_4$, filtered, and evaporated. The residue was purified by column chromatography on silica gel using ethyl acetate–petroleum ether (1:6) as eluent. Yield 8.3 g (88%), light yellow liquid. IR spectrum, ν, cm^{-1}: 1690, 1248, 1165, 1049, 842. ^1H NMR spectrum, δ, ppm: 0.17 s and 0.18 s (9H, CH_3), 1.30 s and 1.38 s (3H, 2′-CH_3), 1.76 s (3H, 2-CH_3), 2.12–2.15 m (1H, 5-H), 2.35–2.50 m (2H, 4-H), 2.57–2.71 m (2H, 3′-H), 4.07–4.12 m (1H, 6-H), 6.61–6.62 m and 6.67–6.68 m (1H, 3-H). ^{13}C NMR spectrum, δ_C, ppm: 0.36 and 0.48 (CH_3); 15.45 and 16.21 (2-CH_3); 19.83 and 20.16 (2′-CH_3); 25.01, 26.57, and 27.23 (C^4); 47.72, 48.11, and 50.04 (C^5); 50.56 and 53.89 ($C^{3'}$); 55.77, 56.64, and 56.97 ($C^{2'}$); 73.81, 76.29, and 76.66 (C^6); 133.95 and 134.08 (C^2); 142.80, 143.27, and 143.92 (C^3); 198.76 (C^1).

(5*R*,6*R*,2′*RS*)- and (5*R*,6*S*,2′*RS*)-6-Hydroxy-2-methyl-5-(2-methyloxiran-2-yl)cyclohex-2-en-1-ones IXa and IXb (a mixture of diastereoisomers at C^6 at a ratio of 10:1**).** A solution of 8.3 g (32.6 mmol) of compound **VIII** in 20 ml of THF was cooled to 5°C, 32.6 ml (32.6 mmol) of a 1 M solution of tetrabutylammonium fluoride in THF was added dropwise under stirring, and the mixture was stirred for 20 min and treated with a saturated solution of sodium chloride. The organic phase was separated, the aqueous phase was extracted with methylene chloride (2×20 ml), the extracts were combined with the organic phase, dried over $MgSO_4$, filtered, and evaporated, and the residue was purified by column chromatography on silica gel using ethyl acetate–petroleum ether (1:2) as eluent. Yield 4.6 g (78%), light yellow liquid.

Compound **IXa**. IR spectrum, ν, cm^{-1}: 3468, 1676, 1140, 1038. ^1H NMR spectrum, δ, ppm: 1.34 s and 1.38 s (3H, 2′-CH_3), 1.76 s and 1.77 s (3H, 2-CH_3), 2.20–2.50 m (3H, 5-H, 4-H), 2.59 d (*J* = 4.6 Hz), 2.67 d (*J* = 1.3 Hz), 2.82 d (2H, 3′-H, *J* = 4.6 Hz), 3.94 d (*J* = 12.6 Hz), 4.02 d (1H, 6-H, *J* = 12.6 Hz), 6.67–6.69 m and 6.73–6.74 m (1H, 3-H). ^{13}C NMR spectrum, δ_C, ppm: 13.21 and 16.25 (CH_3), 17.34 and 19.46 (CH_3), 26.41 and 27.13 (C^4), 47.92 and 49.74 (C^5), 52.14 and 55.80 ($C^{3'}$), 56.73 and 60.23 ($C^{2'}$), 73.41 and 74.22 (C^6), 132.90 and 132.99 (C^2), 144.95 and 145.46 (C^3), 199.83 and 200.05 (C^1).

Compound **IXb**. ^1H NMR spectrum, δ, ppm: 1.34 s and 1.38 s (3H, 2′-CH_3), 1.76 s and 1.77 s (3H, 2-CH_3), 2.20–2.50 m (3H, 5-H, 4-H), 2.55 d (*J* = 2.6 Hz), 2.66 s and 2.69 d (2H, 3′-H, *J* = 2.6 Hz), 4.03 d (*J* = 2.4 Hz), 4.08 d (1H, 6-H, *J* = 2.4 Hz), 6.67–6.69 m and 6.73–6.74 m (1H, 3-H). ^{13}C NMR spectrum, δ_C, ppm: 13.21 and 16.25 (CH_3), 17.34 and 19.46 (CH_3), 29.16 and 29.50 (C^4), 46.50 and 49.09 (C^5), 52.53 and 56.13 ($C^{3'}$), 56.97 ($C^{2'}$), 73.09 and 74.49 (C^6), 132.90 and 132.99 (C^2), 143.99 and 146.12 (C^3), 199.83 and 200.05 (C^1).

Diols X and XI. Lead tetraacetate, 0.8 g (1.8 mmol), was added to a solution of 0.1 g (0.6 mmol) of compound **IVb** in 4 ml of a 1:1 methanol–benzene mixture, and the mixture was stirred for 8 h. Several drops of ethylene glycol and 10 ml of water were added, the mixture was treated with methylene chloride, and the extract was dried over $MgSO_4$ and evaporated. The residue was dissolved in 5 ml of methanol, 0.12 g (3.16 mmol) of sodium tetrahydridoborate was added, and the mixture was stirred for 8 h at room temperature. The mixture was then treated with a saturated solution of ammonium chloride and extracted with methylene chloride, and the extract was dried over $MgSO_4$ and evaporated. The residue was purified by chromatography on silica gel using ethyl acetate–petroleum ether (1:1) as eluent to isolate 0.04 g (42%) of compound **X** and 0.01 g (11%) of **XI** as light yellow liquids.

2,6-Dimethylhept-6-ene-1,5-diol (X). ^1H NMR spectrum ($CDCl_3$), δ, ppm: 0.91 d and 0.92 d (3H, 2-CH_3, *J* = 6.7 Hz), 1.25 m (1H, 3-H), 1.36–1.63 m (4H, 2-H, 3-H, 4-H), 1.72 s (3H, 6-CH_3), 2.15 br.s (2H, OH), 3.46 d.d (2H, 1-H, *J* = 2.2, 5.8 Hz), 4.04 t (1H, 5-H, *J* = 5.5, 6.7 Hz), 4.83 d (1H, 7-H, *J* = 1.2 Hz), 4.98 br.s (1H, 7-H). ^{13}C NMR spectrum, δ_C, ppm:

16.68 (CH_3), 17.43 and 17.63 (CH_3), 28.69 and 28.90 (C^3), 31.84 and 32.07 (C^4), 35.37 and 35.64 (C^2), 67.90 (C^1), 75.87 and 76.25 (C^5), 110.88 and 111.14 (C^7), 147.46 and 147.58 (C^6). Mass spectrum, *m/z* (I_{rel}, %): 158 (0.5) $[M]^+$, 140 (5) $[M - H_2O]^+$, 125 (10) $[M - H_2O - CH_3]^+$, 109 (3), 101 (4), 97 (5), 84 (9), 72 (11), 71 (100) $[CH_2{=}CMeC{=}OH]^+$, 70 (18), 57 (10), 55 (29).

(2*Z*)-2,6-Dimethylhepta-2,6-diene-1,5-diol (XI). $[\alpha]_D^{20} = -5.9°$ (*c* = 1.05, $CHCl_3$). ^{1}H NMR spectrum, δ, ppm: 1.75 s (3H, 6-CH_3), 1.85 s (3H, 2-CH_3), 2.28–2.40 m (2H, 4-H), 2.52 br.s (2H, OH), 3.95 d and 4.18 d (1H each, 1-H, *J* = 11.6 Hz), 4.06 d.d (1H, 5-H, *J* = 4.1, 7.8 Hz), 4.86 d and 4.98 d (1H each, 7-H, *J* = 0.87 Hz), 5.35 t (1H, 3-H, *J* = 7.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 18.24 (CH_3), 22.42 (CH_3), 33.57 (C^4), 61.18 (C^1), 74.33 (C^5), 110.75 (C^7), 123.96 (C^3), 138.61 (C^2), 147.18 (C^6).

Methyl (2*Z*,5*R*)-2-methyl-5-(2-methyloxiran-2-yl)-6-oxohex-2-enoate (XIV). Lead tetraacetate, 13.4 g (30.3 mmol), was added under stirring at 5°C to a solution of 4.6 g (25.2 mmol) of isomer mixture **IXa**/**IXb** in 30 ml of methanol–benzene (1 : 1), the mixture was stirred for 20 min at room temperature, 0.32 g (5.1 mmol) of ethylene glycol and 20 ml of water were added, and the mixture was stirred for 10 min and treated with ethyl acetate. The extract was dried over $MgSO_4$, filtered, and evaporated, and the residue was purified by column chromatography on silica gel using ethyl acetate–petroleum ether (1 : 3) as eluent. Yield 4.8 g (89%), yellow viscous liquid. IR spectrum, ν, cm^{-1}: 2951, 1719, 1703. ^{1}H NMR spectrum, δ, ppm: 1.26 and 1.29 s (3H, 2′-CH_3), 1.83 s and 1.84 s (3H, 2-CH_3), 2.28–2.39 m (1H, 5-H), 2.62–2.69 m (3H, 3′-H, 4-H), 2.72–2.80 m (1H, 4-H), 3.67 s and 3.69 s (3H, OCH_3), 5.87–5.95 m (1H, 3-H), 9.62 d and 9.75 d (1H, 6-H, *J* = 1.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 19.17 and 19.29 (2-CH_3), 20.52 (2′-CH_3), 25.47 and 25.76 (C^4), 51.34 (OCH_3), 52.31 and 52.48 ($C^{3'}$), 55.92 ($C^{2'}$), 57.85 and 58.32 (C^5), 128.95 and 129.01 (C^2), 138.87 and 138.96 (C^3), 167.77 and 167.86 (C^1), 201.01 and 202.07 (C^6).

Methyl (2*Z*,5*R*)-6-Hydroxy-2,5-dimethyl-5-(2-methyloxiran-2-yl)hex-2-enoate (XV). Compound **XIV**, 4.8 g (22.6 mmol), was dissolved in 30 ml of methanol, 8.4 g (22.6 mmol) of $CeCl_3 \cdot 7H_2O$ was added under stirring, the mixture was cooled to 5°C, 0.86 g (22.6 mmol) of $NaBH_4$ was added, and the mixture was stirred for 1 h at room temperature (until the initial compound disappeared according to the TLC data). The mixture was then treated with 60 ml of water, stirred for 15 min, saturated with sodium chloride, and extracted with ethyl acetate (3×30 ml). The combined extracts were dried over $MgSO_4$, filtered, and evaporated, and the residue was purified by column chromatography on silica gel using ethyl acetate–petroleum ether (1 : 2) as eluent. Yield 4.5 g (93%), light yellow liquid. IR spectrum, ν, cm^{-1}: 3449, 2951, 2927, 1715. ^{1}H NMR spectrum, δ, ppm: 1.36 s and 1.38 s (3H, 2′-CH_3), 1.70–1.75 m (1H, 5-H), 1.93 s (3H, 2-CH_3), 2.64–2.82 m (4H, 3′-H, 4-H), 3.53–3.57 m and 3.71–3.75 m (1H each, 6-H), 3.75 s (3H, OCH_3), 5.93–5.99 m (1H, 3-H). ^{13}C NMR spectrum, δ_C, ppm: 18.52 (2-CH_3), 19.94 and 20.66 (2′-CH_3), 27.68 and 28.12 (C^4), 46.86 (C^5), 51.59 (OCH_3), 53.08 ($C^{3'}$), 58.31 and 58.99 ($C^{2'}$), 62.65 and 62.74 (C^6), 128.61 and 128.77 (C^2), 140.59 and 140.80 (C^3), 168.78 (C^1).

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