Structures of Six New Eremophilenolides from the Rhizomes of *Petasites japonicus* MAXIM.[1]

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Six new eremophilenolides, 3β-hydroxyeremophil-7(11)-en-12,8β-olide (1), 3β-hydroxy-6β-methoxyeremophil-7(11)-en-12,8β-olide (2), 3β-hydroxy-6β,8α-dimethoxyeremophil-7(11)-en-12,8β-olide (3), the mixture of 3β,8α-dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8β-olide (4) and 3β,8β-dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8α-olide (5), and 6β-angeloyloxy-8β-hydroxy-3-oxoeremophil-7(11)-en-12,8α-olide (6), were isolated from the dried rhizomes of *Petasites japonicus* MAXIM. (Compositae). The structures of these compounds were elucidated on the basis of spectroscopic evidence.

Keywords *Petasites japonicus*; Compositae; sesquiterpenoid; eremophilenolide

The rhizomes of *Petasites japonicus* MAXIM. (Compositae) have been used for the treatment of tonsillitis, contusion and poisonous snake bite in China.[2] In previous papers, we reported on the structure elucidation of eremophilenolides,[3] triterpenoids,[4] anthraquinones,[4] and phenolic compounds.[5] The present paper describes the further isolation and structure elucidation of six new eremophilenolides: 3β-hydroxyeremophil-7(11)-en-12,8β-olide (**1**), 3β-hydroxy-6β-methoxyeremophil-7(11)-en-12,8β-olide (**2**), 3β-hydroxy-6β,8α-dimethoxyeremophil-7(11)-12,8β-olide (**3**), the mixture of 3β,8α-dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8β-olide (**4**) and 3β,8β-dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8α-olide (**5**), and 6β-angeloyloxy-8β-hydroxy-3-oxoeremophil-7(11)-en-12,8α-olide (**6**). Extraction and isolation were carried out as described in the Experimental section.

Compound **1**, $C_{15}H_{22}O_3$, $[\alpha]_D$ −136.0°, was isolated as colorless needles, mp 168—169 °C. The IR spectrum suggested the presence of a hydroxyl group (3471 cm^{-1}), an α,β-unsaturated-γ-lactone (1741 cm^{-1}) and a double bond (1684 cm^{-1}). The UV spectrum also suggested the presence of an α,β-unsaturated-γ-lactone (λ_{max}: 223 nm). The ^{1}H- (Table I) and ^{13}C-NMR (Table II) spectra were similar to those of the eremophilenolides previously isolated from the rhizomes of *P. japonicus*[3] and showed signals due to a tertiary methyl group [δ_H 0.85 (s, H-15), δ_C 25.0 (C-15)], a secondary methyl group [δ_H 0.98 (d, J=7.3 Hz, H-14), δ_C 7.5 (C-14)], an olefinic methyl group [δ_H 1.80 (dd, J=1.5, 1.5 Hz, H-13), δ_C 8.1 (C-13)], an AB-type methylene [δ_H 2.20 (d, J=13.9 Hz, H-6β), 2.75 (dd, J=13.9, 1.5 Hz, H-6α), δ_C 33.7 (C-6)], a hydroxy-bearing methine [δ_H 4.13 (ddd, J=11.4, 4.4, 4.4 Hz, H-3α), δ_C 68.9 (C-3)], an oxygenated methine [δ_H 4.80 (m, H-8α), δ_C 77.7 (C-8)] and α,β-unsaturated-γ-lactone [δ_C 122.3 (C-11), 161.8 (C-7), 174.7 (C-12)], establishing that the lactone belongs to a sesquiterpene of the eremophilane-type. Naya *et al.*[6] reported that for 8α-methoxyeremophilenolide derivatives, the chemical shifts due to the secondary methyl group (H-14) are downfield from those due to the tertiary methyl group (H-15), whereas this relationship is reversed in the 8β-series. These variations in the chemical shifts may be explained similarly in terms of the effect due to the alteration in geometry of the skeleton observed in the steroid field: by bending rings away from the angular methyl group, or by blocking the angular methyl's view over the remaining skeleton the

TABLE I. ^{1}H-NMR Chemical Shifts ($CDCl_3$, 400 MHz)

Proton	1	2	3	4a	5a	6
3α	4.13 ddd (J=11.4, 4.4, 4.4)	4.07 ddd (J=11.4, 4.4, 4.4)	4.01 ddd (J=11.4, 4.4, 4.4)	4.97 ddd (J=11.7, 4.4, 4.4)	4.99 ddd (J=3.0, 3.0, 2.9)	
6α	2.75 dd (J=13.9, 1.5)	4.35 d (J=1.5)	4.28 d (J=1.5)	6.19 d (J=1.5)	5.76 s	5.61 s
6β	2.20 d (J=13.9)					
8α	4.80 m	4.77 m				
9α	2.17 ddd (J=12.8, 6.6, 2.2)	2.17 ddd (J=12.8, 6.6, 2.2)				
13	1.80 dd (J=1.5, 1.5)	1.97 dd (J=1.5, 1.5)	2.01 d (J=1.5)	1.83 d (J=1.5)	2.03 s	2.02 s
14	0.98 d (J=7.3)	0.95 d (J=7.3)	0.94 d (J=7.3)	0.96 d (J=7.3)	0.95 d (J=7.0)	1.02 d (J=6.6)
15	0.85 s	0.83 s	0.83 s	0.97 s	1.26 s	1.00 s
3′				6.97 qq (J=7.0, 1.5)	6.84 qq (J=7.0, 1.5)	6.20 qq (J=7.3, 1.5)
4′				1.87 dq (J=7.0, 1.1)	1.81 dq (J=7.0, 1.1)	2.04 dq (J=7.3, 1.5)
5′				1.93 dq (J=1.5, 1.1)	1.84 dq (J=1.5, 1.1)	1.92 dq (J=1.5, 1.5)
6-OCH_3		3.49 s	3.47 s			
8-OCH_3			3.20 s			
$COCH_3$				2.00 s	1.89 s	
$COCH_3$				2.10 s	2.08 s	

Coupling constants (J in Hz) are given in parentheses.

TABLE II. ^{13}C-NMR Chemical Shifts ($CDCl_3$, 100 MHz)

Carbon	**1**	**2**	**3**	**4a**	**5a**	**6**
1	27.3	27.3	26.9	26.3	21.1	26.3
2	29.3	28.9	28.7	25.3	24.6	38.7
3	68.9	68.7	68.8	71.5	73.1	209.9
4	44.7	38.2	38.8	35.5[a)]	32.3	45.1
5	41.0	46.6	47.2	45.6	41.9	46.6
6	33.7	80.5	79.8	70.2	70.2	70.6
7	161.8	160.9	157.2	154.2	149.7	151.1
8	77.7	77.4	106.9	104.6	103.4	103.5
9	34.6	35.3	38.1	35.6[a)]	38.0	36.7
10	35.2	35.1	34.9	35.1	34.6	34.7
11	122.3	121.9	126.2	126.2	129.8	129.4
12	174.7	174.6	171.4	170.8	170.3[c)]	170.5
13	8.1	8.5	8.4	8.5[b)]	8.9	9.0
14	7.5	7.4	7.6	8.6[b)]	12.3	8.0
15	25.0	19.5	18.9	19.7	18.3	18.9
1'				166.4	166.5	166.8
2'				127.9	127.9	126.7
3'				139.0	137.5	141.1
4'				14.7	14.4	16.0
5'				12.2	12.1	20.6
6-OCH_3		60.0	59.6			
8-OCH_3			50.1			
CO$\underline{C}H_3$				21.3	21.3	
CO$\underline{C}H_3$				22.2	21.6	
$\underline{C}OCH_3$				168.1	168.3	
$\underline{C}OCH_3$				170.0	170.3[c)]	

a—b) Assignments may be reversed. *c)* Signals were overlapped.

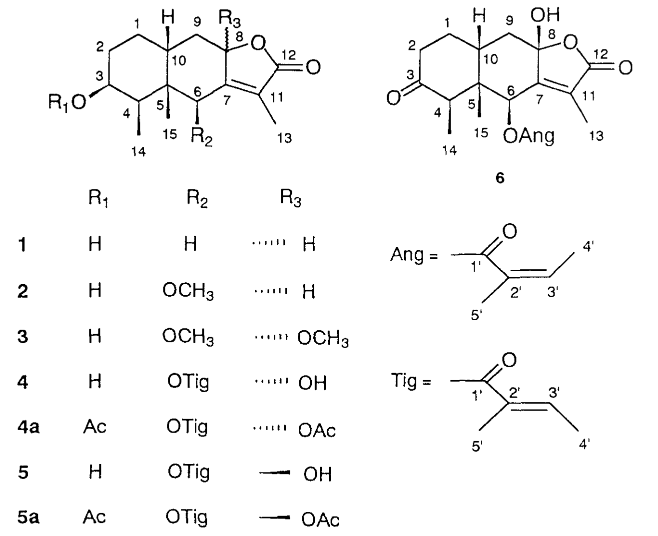

Fig. 1

methyl signal may cause a downfield shift.[6)] Naya and his colleagues also reported that the homoallylic coupling (J=1.0—1.5 Hz) between the olefinic methyl group (H-13) and H-6α found in the 8α-series, is absent in the 8β-series. The value of the optical rotation of the 8β-series, which had a steroidal conformation, was positive, and that of the 8α-series, which had a non-steroidal conformation, was negative.[6)] The ^{1}H-NMR spectrum of **1** showed a singlet of the tertiary methyl group (H-15) at δ 0.85 and a doublet of the secondary methyl group (H-14) at δ 0.98 (J=7.3 Hz), as well as the homoallylic coupling (J=1.5 Hz) of the olefinic methyl group (H-13) with H-6α. Furthermore, the value of the optical rotation was negative. These data indicated that **1** exists in a non-steroidal conformation. The position of the hydroxyl group was determined to be at the C-3β by comparing the chemical shift, coupling pattern and constants of the hydroxy-bearing methine proton of **1** with those of 3β-hydroxy-6β-acyleremophil-7(11)-en-12,8β-olides.[3)] On the basis of the above evidence, the structure of **1** was determined to be 3β-hydroxy-eremophil-7(11)-en-12,8β-olide. Compound **1** was isolated from a natural source for the first time, although **1** has already been synthesized by Naya *et al.*[7)]

Compound **2**, $C_{16}H_{24}O_4$, $[\alpha]_D$ −189.6°, was isolated as colorless needles, mp 180—181 °C. The IR spectrum suggested the presence of a hydroxyl group (3474 cm^{-1}), an α,β-unsaturated-γ-lactone (1746 cm^{-1}) and a double bond (1678 cm^{-1}). The UV spectrum also suggested the presence of an α,β-unsaturated-γ-lactone (λ_{max}: 219 nm). The ^{1}H- and ^{13}C-NMR spectra of **2** were virtually identical to those of **1** except for the presence of a methoxyl group [δ_H 3.49 (s), δ_C 60.0]. The position of the methoxyl group was determined by ^{1}H-detected heteronuclear multiple bond correlation (HMBC) and nuclear Overhauser effect correlation spectroscopy (NOESY). In the HMBC spectrum, a cross peak was observed between the methoxyl group at δ 3.49 and the C-6 at δ 80.5, so that the methoxyl group is attached at the C-6. In the NOESY spectrum, each signal of H-6α and H-8α showed a correlation, so that the methoxyl group is β-oriented. On the basis of this evidence, the structure of **2** was determined to be 3β-hydroxy-6β-methoxyeremophil-7(11)-en-12,8β-olide.

Compound **3**, $C_{17}H_{26}O_5$, $[\alpha]_D$ −86.7°, was isolated as colorless oil. The IR spectrum suggested the presence of a hydroxyl group (3509 cm^{-1}), an α,β-unsaturated-γ-lactone (1757 cm^{-1}) and a double bond (1680 cm^{-1}). The UV spectrum also suggested the presence of an α,β-unsaturated-γ-lactone (λ_{max}: 225 nm). The ^{1}H- and ^{13}C-NMR spectra of **3** were virtually identical to those of **2** except for the presence of one more methoxyl group [δ_H 3.20 (s), δ_C 50.1], the position of which was determined as follows. In the HMBC spectrum, a cross peak was observed between the methoxyl group at δ 3.20 and the C-8 at δ 106.9, so that this methoxyl group is attached at the C-8. In the NOESY spectrum, each signal of the methoxyl proton and H-6α showed a correlation, so that this methoxyl group is α-oriented. Based on this evidence, the structure of **3** was determined to be 3β-hydroxy-6β,8α-dimethoxyeremophil-7(11)-en-12,8β-olide.

A mixture of compounds **4** and **5** was obtained as colorless oil. These compounds could not be separated by silica gel column chromatography or HPLC. The ^{1}H-NMR spectrum was virtually identical to that of C-8-epimers of 6β-angeloyloxy-3β,8-dihydroxyeremophilenolides[8)] except for the presence of a tigloyloxyl group in place of an angeloyloxyl group. The mixture was treated with acetic anhydride-pyridine to afford an epimeric mixture of diacetate (**4a** and **5a**), which was then separated by silica gel column chromatography. Compound **4a**, $C_{24}H_{32}O_8$, $[\alpha]_D$ −66.7°, was isolated as a coloress oil. The IR spectrum suggested the presence of an α,β-unsaturated-γ-lactone (1776 cm^{-1}), an α,β-unsaturated ester (1729 cm^{-1}) and a double bond (1650 cm^{-1}). The UV spectrum also suggested the presence of an α,β-

unsaturated-γ-lactone (λ_{max}: 222 nm). The ^{1}H- and ^{13}C-NMR spectra of **4a** were virtually identical to those of 3β,8α-diacetoxy-6β-angeloyloxyeremophilenolide[8] except for the presence of a tigloyloxy group [δ_H 1.87 (dq, J=7.0, 1.1 Hz, H-4′), 1.93 (dq, J=1.5, 1.1 Hz, H-5′), 6.97 (qq, J=7.0, 1.5 Hz, H-3′), δ_C 12.2 (C-5′), 14.7 (C-4′), 127.9 (C-2′), 139.0 (C-3′), 166.4 (C-1′)][3] in place of an angeloyloxyl group. The position of the tigloyloxyl group was confirmed by the HMBC spectrum, in which a cross peak was observed between the H-6α at δ 6.19 and the C-1′ at δ 166.4, confirming that a tigloyloxyl group was attached at the C-6β. The structure of **4a** was then determined to be 3β,8α-diacetoxy-6β-tigloyloxyeremophil-7(11)-en-12,8β-olide. Thus, the structure of **4** is 3β,8α-dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8β-olide.

Compound **5a**, $C_{24}H_{32}O_8$, $[\alpha]_D$ +55.6°, was isolated as colorless oil. The IR spectrum suggested the presence of an α,β-unsaturated-γ-lactone (1775 cm^{-1}), an α,β-unsaturated ester (1733 cm^{-1}) and a double bond (1650 cm^{-1}). The UV spectrum also suggested the presence of an α,β-unsaturated-γ-lactone (λ_{max}: 225 nm). The ^{1}H- and ^{13}C-NMR spectra of **5a** were virtually identical to those of 3β,8β-diacetoxy-6β-angeloyloxyeremophilenolide[8] except for the presence of a tigloyloxyl group [δ_H 1.81 (dq, J=7.0, 1.1 Hz, H-4′), 1.84 (dq, J=1.5, 1.1 Hz, H-5′), 6.84 (qq, J=7.0, 1.5 Hz, H-3′), δ_C 12.1 (C-5′), 14.4 (C-4′), 127.9 (C-2′), 137.5 (C-3′), 166.5 (C-1′)][3] in place of an angeloyloxyl group. The position of a tigloyloxyl group was confirmed by the HMBC spectrum, in which a cross peak was observed between the H-6α at δ 5.76 and the C-1′ at δ 166.5, showing that a tigloyloxyl group was attached at the C-6β. The structure of **5a** was then determined to be 3β,8β-diacetoxy-6β-tigloyloxyeremophil-7(11)-en-12,8α-olide. Thus, the structure of **5** is 3β,8β-dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8α-olide.

Compound **6**, $C_{20}H_{26}O_6$, $[\alpha]_D$ +38.1°, was isolated as colorless oil. The IR spectrum suggested the presence of a hydroxyl group (3600—3200 cm^{-1}), an α,β-unsaturated-γ-lactone (1759 cm^{-1}), a six-membered ring ketone and an α,β-unsaturated ester (1713 cm^{-1}), and a double bond (1648 cm^{-1}). The UV spectrum also suggested the presence of an α,β-unsaturated-γ-lactone (λ_{max}: 220 nm). The ^{1}H- and ^{13}C-NMR spectra showed signals due to a tertiary methyl group [δ_H 1.00 (s, H-15), δ_C 18.9 (C-15)], a secondary methyl group [δ_H 1.02 (d, J=6.6 Hz, H-14), δ_C 8.0 (C-14)], an olefinic methyl group [δ_H 2.02 (s, H-13), δ_C 9.0 (C-13)], an angeloyloxyl group [δ_H 1.92 (dq, J=1.5, 1.5 Hz, H-5′), 2.04 (dq, J=7.3, 1.5 Hz, H-4′), 6.20 (qq, J=7.3, 1.5 Hz, H-3′), δ_C 16.0 (C-4′), 20.6 (C-5′), 126.7 (C-2′), 141.1 (C-3′), 166.8 (C-1′)],[3] an angeloyloxy-bearing methine [δ_H 5.61 (s, H-6α), δ_C 70.6 (C-6)], an α,β-unsaturated-γ-lactone [δ_C 129.4 (C-11), 151.1 (C-7), 170.5 (C-12)], a hemi-ketal carbon [δ_C 103.5 (C-8)] and a carbonyl carbon [δ_C 209.9 (C-3)], which is correlated with the secondary methyl group (C-14) at δ 1.02 in the HMBC spectrum. Thus, the structure of **6** was estimated to be 6β-angeloyloxy-8(α or β)-hydroxy-3-oxoeremophilenolide. In the ^{1}H-NMR spectrum, the homoallylic coupling between the olefinic methyl group (H-13) and H-6α is lacking. In the NOESY spectrum, each signal of the secondary methyl group (H-14) and H-6α showed a correlation. These data indicated that **6** exists in a steroidal conformation. On the basis of the above evidence, the structure of **6** was determined to be 6β-angeloyloxy-8β-hydroxy-3-oxoeremophil-7(11)-en-12,8α-olide.

Experimental

General Procedures Melting points were determined with a Yanagimoto micromelting apparatus and are uncorrected. Optical rotations were determined with a JASCO DIP-360 digital polarimeter. IR spectra were recorded with a Perkin-Elmer FT-IR 1725X infrared spectrophotometer and UV spectra with a Beckman DU-64 spectrophotometer. ^{1}H- and ^{13}C-NMR spectra were recorded with a JEOL JNM-GSX 400 (400 and 100 MHz, respectively) spectrometer. Chemical shifts were given on a δ (ppm) scale with tetramethylsilane as an internal standard (s, singlet; d, doublet; dd, double doublet; ddd, double double doublet; dq, double quartet; qq, quartet quartet; m, multiplet). The electron ionization mass spectrum (EI-MS) and high resolution mass spectrum (HR-MS) were recorded on a JEOL JMS-DX 303 mass spectrometer. Column chromatography was carried out on Kieselgel 60 (Merck; 230—400 mesh). Preparative HPLC was carried out on a Tosoh HPLC system (pump, CCPD; detector, UV-8011) using a TSK gel ODS-120T column (Tosoh) and TSK gel OH-120 column (Tosoh).

Plant Material The dried rhizomes of *Petasites japonicus* were purchased from Tochimoto Tenkaido Co., Ltd. in 1990.

Extraction and Isolation The dried rhizomes of *Petasites japonicus* (3.0 kg) were extracted three times with MeOH at room temperature for 2 weeks. The MeOH extract was concentrated under reduced pressure and the residue was suspended in a small excess of water. This suspension was extracted, successively, with $CHCl_3$, Et_2O, AcOEt and *n*-BuOH. The $CHCl_3$-soluble fraction was concentrated under reduced pressure to afford a residue (112.5 g). This residue (60.0 g) was chromatographed on a silica gel column using benzene–AcOEt (9 : 1, 8 : 2, 7 : 3) and $CHCl_3$–MeOH (8 : 2), and the eluate was separated into 4 fractions (frs. 1—4). Fraction 4 was rechromatographed on a silica gel column using benzene–AcOEt (6 : 4, 5 : 5, 4 : 6, 3 : 7) and $CHCl_3$–MeOH (9 : 1, 8 : 2), and the eluate was separated into 4 fractions (frs. 1′—4′). Fraction 2′ was rechromatographed on a silica gel column using *n*-hexane–acetone (5 : 4, 5 : 5, 4 : 5, 3 : 6) and acetone, and the eluate was separated into 8 fractions (frs. 1″—8″). Fraction 3″ was rechromatographed on a silica gel column using benzene–AcOEt (3 : 2, 2 : 2), and the eluate was separated into 7 fractions (frs. 1‴—7‴). Fraction 6‴ was purified by preparative HPLC (Column, TSK gel ODS-120T, 21.5 mm i.d. × 30 cm; mobile phase, MeOH–H_2O (1 : 1); flow rate, 3.0 ml/min; UV detector, 220 nm) to give **3** (20.5 mg), a mixture of **4** and **5** (16.8 mg) and **6** (5.3 mg). Fraction 7‴ was separated by preparative HPLC (Column, TSK gel OH-120, 7.8 mm i.d. × 30 cm; mobile phase, *n*-hexane–EtOH (5 : 1); flow rate, 0.85 ml/min; UV detector, 220 nm) to give a mixture of **1** and **2**. The mixture of **1** and **2** was purified by preparative HPLC (Column, TSK gel ODS-120T, 7.8 mm i.d. × 30 cm; mobile phase, MeOH–H_2O (1 : 1); flow rate, 1.2 ml/min; UV detector, 220 nm) to give **1** (2.7 mg) and **2** (8.9 mg).

3β-Hydroxyeremophil-7(11)-en-12,8β-olide (1) Colorless needles (from Et_2O–AcOEt), mp 168—169 °C. $[\alpha]_D^{22}$ −136.0° (c=0.3, $CHCl_3$). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 3471, 1741, 1684. UV λ_{max}^{MeOH} nm (log ε): 223 (4.2). HR-MS m/z: 250.1577 (M^+, Calcd for $C_{15}H_{22}O_3$; 250.1569). ^{1}H-NMR: see Table I. ^{13}C-NMR: see Table II.

3β-Hydroxy-6β-methoxyeremophil-7(11)-en-12,8β-olide (2) Colorless needles (from Et_2O–AcOEt), mp 180—181 °C $[\alpha]_D^{23}$ −189.6° (c=0.9, $CHCl_3$). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 3474, 1746, 1678. UV λ_{max}^{MeOH} nm (log ε): 219 (4.2). HR-MS m/z: 280.1662 (M^+, Calcd for $C_{16}H_{24}O_4$; 280.1675). ^{1}H-NMR: see Table I. ^{13}C-NMR: see Table II.

3β-Hydroxy-6β,8α-dimethoxyeremophil-7(11)-en-12,8β-olide (3) Colorless oil. $[\alpha]_D^{26}$ −86.7° (c=1.1, $CHCl_3$). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 3509, 1757, 1680. UV λ_{max}^{MeOH} nm (log ε): 225 (3.8). HR-MS m/z: 310.1807 (M^+, Calcd for $C_{17}H_{26}O_5$; 310.1780). ^{1}H-NMR: see Table I. ^{13}C-NMR: see Table II.

The Mixture of 3β,8α-Dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8β-olide (4) and 3β,8β-dihydroxy-6β-tigloyloxyeremophil-7(11)-en-12,8α-olide (5) Colorless oil. ^{1}H-NMR ($CDCl_3$) **4** δ: 0.97 (3H, d, J=7.3 Hz, H-14), 0.97 (3H, s, H-15), 7.00 (1H, qq, J=7.3, 1.3 Hz, H-3′). **5** δ: 1.06 (3H, d, J=7.0 Hz, H-14), 1.32 (3H, s, H-15), 6.91 (1H, qq, J=7.3, 1.3 Hz, H-3′).

Acetylation of the Mixture of 4 and 5 Acetylation of 11 mg of the mixture with acetic anhydride–pyridine for 2 d at room temperature followed by the usual work up and purification by silica gel column chromatography [benzene–AcOEt (19:1)] gave two diacetates [**4a** (5.4 mg) and **5a** (0.9 mg)].

3β,8α-Diacetoxy-6β-tigloyloxyeremophil-7(11)-en-12,8β-olide (4a) Colorless oil. $[\alpha]_D^{20}$ −66.7° (c=0.5, $CHCl_3$). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1776, 1729, 1650. UV λ_{max}^{MeOH} nm (log ε): 222 (4.2). HR-MS m/z: 448.2169 (M^+, Calcd for $C_{24}H_{32}O_8$; 448.2148). ^{1}H-NMR: see Table I. ^{13}C-NMR: see Table II.

3β,8β-Diacetoxy-6β-tigloyloxyeremophil-7(11)-en-12,8α-olide (5a) Colorless oil. $[\alpha]_D^{21}$ +55.6° (c=0.1, $CHCl_3$). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1775, 1733, 1650. UV λ_{max}^{MeOH} nm (log ε): 225 (4.9). HR-MS m/z: 448.2133 (M^+, Calcd for $C_{24}H_{32}O_8$; 448.2148). ^{1}H-NMR: see Table I. ^{13}C-NMR: see Table II.

6β-Angeloyloxy-8β-hydroxy-3-oxoeremophil-7(11)-en-12,8α-olide (6) Coloress oil. $[\alpha]_D^{19}$ +38.1° (c=0.5, $CHCl_3$). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 3600—3200, 1759, 1713, 1648. UV λ_{max}^{MeOH} nm (log ε): 220 (4.3). HR-MS m/z: 362.1706 (M^+, Calcd for $C_{20}H_{26}O_6$; 362.1729). ^{1}H-NMR: see Table I. ^{13}C-NMR: see Table II.

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References and Notes

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