Epitaondiol: The First Polycyclic Meroditerpenoid Containing Two Fused Six-Membered Rings Forced into the Twist-Boat Conformation

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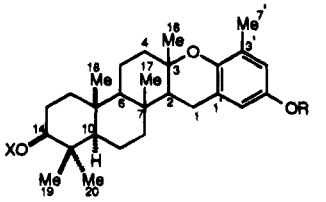
Marine organisms are a rich source of secondary metabolites, often displaying both unusual structures and interesting biological activities.[1] In particular, certain brown algae (*Phaeophyta*), brown seaweeds (*Dictyotaceae*), and marine sponges (*Dictyoceratidae*) have been found to contain polycyclic meroterpenoids biogenetically derived from prenylated hydroquinones,[2–10] sometimes displaying pronounced cytotoxic[3,10] and/or ichthyotoxic biological activities.

About twenty years ago, reports appeared describing the isolation of taondiol (**1**) from the Atlantic alga *Taonia atomaria*,[2] and the biomimetic synthesis[11] of **1** and its stereoisomer isotaondiol (**2**), formed from **1** under base-catalyzed rearrangement.[12] In more recent years, new ichthyotoxic taondiol stereoisomers were found in the Caribbean alga *Stypopodium zonale*, which contains epitaondiol,[13] and in the Pacific alga *Stypopodium flabelliforme*, which contains isoepitaondiol.[14] In light of the indirect evidence available at the time, epitaondiol was assigned structure **3**,[13] while isoepitaondiol was assigned structure **4**.[14]

We now report that the results of a complete NMR examination of epitaondiol, its methyl ether, and its monoacetate, using standard 1D and 2D techniques such as those recently described for marine sponge sesterterpenes,[15] could only be accommodated by structures **5**–**7**, in which the *trans-syn-trans-syn-trans* A/B/C/D ring fusion forces both the B and C rings into the twist-boat conformation. Repeated attempts to confirm this unprecedented steric arrangement by means of X-ray diffraction of crystalline **5**, recrystallized **6**, its derived *N*-(4-bromophenyl)carbamate **8** or the corresponding allophanate **9** have so far been unsuccessful (more than half a dozen samples tried over the span of more than one year).



1 (2α,3β,6α,7β); R = X = H
2 (2α,3α,6α,7β); R = X = H
3 (2β,3β,6α,7β); R = X = H
4 (2β,3α,6α,7β); R = X = H
5 (2α,3β,6β,7α); R = X = H
6 (2α,3β,6β,7α); R = Me; X = H
7 (2α,3β,6β,7α); R = Ac; X = H
8 (2α,3β,6β,7α); R = Me; X = 4-Br-C_6H_4-NH-CO-
9 (2α,3β,6β,7α); R = Me; X = 4-Br-C_6H_4-N(CO-C_6H_4-4-Br)CO

Figure 1. Structure and numbering of epitaondiol stereoisomers and derivatives.

Epitaondiol (**5**) was isolated from *S. flabelliforme* as previously described.[14] Epitaondiol methyl ether (**6**) was prepared using standard methods (see Experimental Section) and the known derivative epitaondiol monoacetate (**7**) was prepared as previously described.[14] Carbamate **8** and allophanate **9** were prepared from **6** by reaction with 4-bromophenyl isocyanate (see Experimental Section).

The 400 MHz ^{1}H NMR spectrum of epitaondiol (**5**) in $CDCl_3$ showed well resolved singlets for all six *C*-methyl groups (see Figure 1 for numbering), at δ_H 0.80 (H-19), 0.92 (H-18), 1.00 (H-20), 1.19 (H-16), 1.22 (H-17) and 2.08 (H-7′) ppm. Other well resolved signals were found for the aromatic protons at 6.45 (d, 2 Hz, H-4′) and 6.38 (d, 2 Hz, H-6′), for the secondary carbinol proton at 3.29 (dd, 10 Hz and 4 Hz, H-14), and for the benzylic protons at 2.68 (dd, 15 Hz and 14 Hz, H-1β) and 2.47 (dd, 15 Hz and 5 Hz, H-1α).[16] Selected NOE difference data were clearly incompatible with the published structure **3**. Thus, for structure **3** the H-17 angular methyl should be in the β face of the molecule, in a *syn*-1,3-diaxial relationship with methyls H-16 and H-18. However, irradiation of H-17 gave a 4.9% NOE on the well identified benzylic proton H-1α, and on two methine protons (later identified as H-2 and H-10) most probably also located in the α face of the molecule.[17] On the other hand, irradiation of H-16 gave a 3.5% NOE enhancement on the well identified benzylic proton H-1β. We therefore suspected that the H-17 methyl was indeed α rather than β. In addition, both inspection of models and preliminary MM2 calculations revealed that in structure **3** methyls H-16 and H-17

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(16) A broad multiplet, partly overlapped with OH signals, centered at 2.14 ppm, was later rationalized as H-4α.
(17) In an effort to check experimentally the behavior of compounds displaying structures similar to **3**, with *trans-anti-trans* A/B/C ring fusion, we carefully measured methyl–methyl NOEs on a sample of oleanolic acid in the same solvent, using the same spectrometer and settings. On that sample, irradiation of the β angular methyl linked to C-8 (steroid numbering) resulted in a clear enhancement (2.5%) of the *syn*-1,3-diaxial angular methyl linked to C-10 (steroid numbering).

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are too far away from the benzylic protons (over 4.0 Å) to produce these significant NOE enhancements. Methyl ether **6** and acetate **7** showed a similar behavior for the angular methyl H-17.

The far more soluble methyl ether **6** was used for the remaining NMR determinations, including the measurement of the ^{13}C NMR spectrum and, in particular, the use of the HETNOE technique.[18,19] This consists in a selective heteronuclear $^{13}C\{^1H\}$ NOE measurement, in which low power, selective CW irradiation of a given proton signal results in quite large NOE enhancements of the spatially closest quaternary carbon signals in the ^{13}C NMR spectrum.[18,19] This was necessary at this stage to assign unambiguously the pair of methyls numbered 16 and 17. One more reason to prefer using methyl ether **6** rather than free phenol **5** was to avoid the possible epimerization of the latter, in a process similar to the known conversion of **1** to **2** under basic conditions.[12]

Both the ^1H and the ^{13}C NMR spectra of epitaondiol methyl ether **6** could be completely analyzed and assigned unambiguously using an array of 1D (NOEDIFF, HETNOE) and 2D (COSY-45, DQF-COSY, CHCORR, HMQC, HMBC) methods. The assignments, together with the relevant spectral evidence, are given in Tables 1 and 2.

Again, the results of the 1D interproton NOE measurements (Figure 2) ruled out the *trans-anti-trans-syn-cis* A/B/C/D ring fusion previously proposed for epitaondiol[13] and fully supported the unprecedented *trans-syn-trans-syn-trans* arrangement, which forces rings B and C into the twist boat conformation. A phase-sensitive 2D ROESY spectrum of **6** fully confirmed the 1D NOEDIFF results, in particular the presence of ROE peaks correlating the methine proton H-6 with both methyls H-16 and H-18.

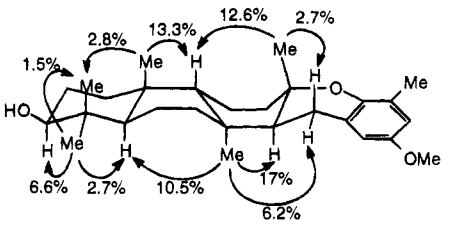

Figure 2. Selected NOE data for epitaondiol methyl ether (**6**).

The unambiguous assignment of the pair of methyls appearing at δ_H 1.20 and 1.24 ppm (δ_C 24.9 and 28.0 ppm, respectively) as H-16 and H-17 was absolutely necessary as crucial evidence in favor of structure **6**. Two independent methods, namely HETNOE[18,19] and HMBC, confirmed this assignment. Thus, low power selective irradiation of the proton singlet at 1.20 ppm resulted in a 31.3% heteronuclear NOE enhancement of the quaternary carbon C-3, appearing at 76.5 ppm, while irradiation at δ_H 1.24 resulted in a 33.9% increase of the quaternary carbon C-7 at δ_C 34.9 ppm. In addition, the HMBC spectrum of **6** showed very intense two-bond correlations for the pairs H-16/C-3 and H-17/C-7.

Similar spectroscopic evidence obtained for the other epitaondiol derivatives examined, i.e., acetate **7**, carbamate **8**, and allophanate **9**, always supported the *trans-syn-trans-syn-trans* A/B/C/D ring fusion, with rings B and C conformationally blocked into the boat (or twist-boat) conformation.

There is ample precedent of terpenoid metabolites featuring a twist-boat B ring as a consequence of the *trans-syn-trans* A/B/C ring fusion. One case of special biosynthetic significance is the protosterol family, for which both the biosynthesis and the chemical synthesis have recently received much attention.[20-23] Another example, now from marine origin, is the triterpenoid

Table 1. 400 MHz ^1H NMR Spectrum of Epitaondiol Methyl Ether 6 ($CDCl_3$)

H	δ (m)	couplings (Hz)	COSY (δ, H)[a]	NOEs from (δ, H, %)[b]
$H_{1\alpha}$	2.54 dd	$J_{1\alpha,1\beta}$ = 16.7	2.73 ($H_{1\beta}$)	1.24 (H_{17}), 6.2%
		$J_{1\alpha,2}$ = 4.9	1.70 (H_2)	6.44 ($H_{6'}$), 3.7%
$H_{1\beta}$	2.73 dd	$J_{1\beta,2}$ = 13.5	2.54 ($H_{1\alpha}$)	1.20 (H_{16}), 2.7%
		$J_{1\alpha,1\beta}$ = 16.7	1.70 (H_2)	
H_2	1.70 dd	$J_{1\alpha,2}$ = 4.9	2.54 ($H_{1\alpha}$)	1.24 (H_{17}), 17.0%
		$J_{1\beta,2}$ = 13.5	2.73 ($H_{1\beta}$)	
$H_{4\alpha}$	2.14 m		1.88 ($H_{4\beta}$)	
			1.49 ($H_{5\alpha+5\beta}$)	
			1.20 (H_{16})	
$H_{4\beta}$	1.88 dt	$J_{4\alpha,4\beta}$ = 13.5	2.14 ($H_{4\alpha}$)	1.20 (H_{16}), 5.7%
		$J_{4\beta,5\alpha}$ = 9.9	1.49 ($H_{5\alpha+5\beta}$)	
		$J_{4\beta,5\beta}$ = 9.9		
$H_{5\alpha+\beta}$	1.49 m		2.14 ($H_{4\alpha}$)	1.24 (H_{17}), 2.0%
			1.88 ($H_{4\beta}$)	0.94 (H_{18}), 5.0%
			1.30 (H_6)	
H_6	1.30 dd	$J_{5\alpha,6}$ = 7.8	1.49 ($H_{5\alpha+5\beta}$)	1.20 (H_{16}), 12.6%
		$J_{5\beta,6}$ = 6.1		0.94 (H_{18}), 13.3%
$H_{8\alpha}$	1.32 m		1.81 ($H_{8\beta}$)	1.24 (H_{17}), <1.0%
			1.67 ($H_{9\alpha}$)	
$H_{8\beta}$	1.81 m		1.32 ($H_{8\alpha}$)	1.20 (H_{16}), <1.0%
			1.35 ($H_{9\beta}$)	
$H_{9\alpha}$	1.67 m		1.32 ($H_{8\alpha}$)	1.00 (H_{20}), 12.5%
			1.35 ($H_{9\beta}$)	
$H_{9\beta}$	1.35 m		1.81 ($H_{8\beta}$)	0.80 (H_{19}), <1.0%
			1.67 ($H_{9\alpha}$)	
			1.75 (H_{10})	
H_{10}	1.75 d	$J_{9\beta,10}$ = 12.4	1.35 ($H_{9\beta}$)	1.24 (H_{17}), 10.5%
				1.00 (H_{20}), 2.7%
$H_{12\alpha\beta}$	1.43 m		1.62 ($H_{13\beta}$)	1.24 (H_{17}), 2.0%
			1.75 ($H_{13\alpha}$)	
			0.94 (H_{18})	
$H_{13\alpha}$	1.75 m		3.25 (H_{14})	
			1.43 ($H_{12\alpha\beta}$)	
$H_{13\beta}$	1.62 m		3.25 (H_{14})	0.94 (H_{18}), 10.1%
			1.43 ($H_{12\alpha}$)	
H_{14}	3.25 dd	$J_{13\alpha,14}$ = 5.1	1.75 ($H_{13\alpha}$)	1.00 (H_{20}), 6.6%
		$J_{13\beta,14}$ = 11.6	1.62 ($H_{13\beta}$)	
H_{16}	1.20 s		2.14 ($H_{4\alpha}$)	2.12 ($H_{7'}$), <1.0%
H_{17}	1.24 s		1.81 ($H_{8\beta}$)	
H_{18}	0.94 s		1.43 ($H_{12\alpha}$)	0.80 (H_{19}), 3.5%
H_{19}	0.80 s		1.00 (H_{20})	0.94 (H_{18}), 2.8%
				1.00 (H_{20}), 1.5%
H_{20}	1.00 s		0.80 (H_{19})	0.80 (H_{19}), 1.1%
$H_{4'}$	6.54 d	$J_{4',6'}$ = 3.0	nm[c]	3.72 (OMe), 10.0%
				2.12 ($H_{7'}$), 16.2%
$H_{6'}$	6.44 d	$J_{4',6'}$ = 3.0	nm[c]	3.72 (OMe), 8.8%
$H_{7'}$	2.12 s			6.54 ($H_{4'}$), 2.8%
OMe	3.72 s			6.54 ($H_{4'}$), 1.8%
				6.44 ($H_{6'}$), 2.5%

[a] Cross peaks seen in the COSY-45 and/or DQF-COSY spectra. [b] Percent NOEs seen on the proton of each entry upon irradiation at the frequencies shown. [c] Not measured. The spectral window of the COSY spectrum did not cover this chemical shift.

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Table 2. 100 MHz ^{13}C NMR Spectrum of Epitaondiol Methyl Ether 6 ($CDCl_3$)

C	δ (m)	CHCORR cross peaks (δ_H)	HETNOE enhancements from (δ_H, %)[a]
C_1	23.3 t	2.54 ($H_{1\alpha}$); 2.73 ($H_{1\beta}$)	
C_2	48.5 d	1.70 (H_2)	
C_3	76.5 s		1.24 (H_{17}, 12%); 1.20 (H_{16}, 31%)
C_4	40.1 t	1.88 ($H_{4\beta}$); 2.14 ($H_{4\alpha}$)	
C_5	16.9 t	1.49 ($H_{5\alpha}$ + $H_{5\beta}$)	
C_6	47.9 d	1.30 (H_6)	
C_7	34.9 s		1.24 (H_{17}, 34%); 1.20 (H_{16}, 15%)
C_8	31.4 t	1.81 ($H_{8\beta}$); 1.32 ($H_{8\alpha}$)	
C_9	17.0 t	1.67 ($H_{9\alpha}$); 1.35 ($H_{9\beta}$)	
C_{10}	45.2 d	1.75 (H_{10})	
C_{11}	36.9 s		0.80 (H_{19}, 9%); 0.94 (H_{18}, 35%); 1.00 (H_{20}, 5%)
C_{12}	31.8 t	1.43 ($H_{12\alpha}$ + $H_{12\beta}$)	
C_{13}	29.0 t	1.75 ($H_{13\alpha}$); 1.62 ($H_{13\beta}$)[b]	
C_{14}	79.3 d	3.25 (H_{14})	
C_{15}	39.0 s		0.80 (H_{19}, 28%); 0.94 (H_{18}, 10%); 1.00 (H_{20}, 35%)
C_{16}	24.9 q	1.20 (H_{16})	
C_{17}	28.0 q	1.24 (H_{17})	
C_{18}	21.6 q	0.94 (H_{18})	
C_{19}	15.9 q	0.80 (H_{19})	
C_{20}	29.1 q	1.00 (H_{20})	
$C_{1'}$	121.9 s		
$C_{2'}$	145.2 s		2.12 ($H_{7'}$, 9%); 1.20 (H_{16}, 11%)
$C_{3'}$	126.8 s		2.12 ($H_{7'}$, 23%)
$C_{4'}$	114.5 d	6.54 ($H_{4'}$)	
$C_{5'}$	152.1 s		
$C_{6'}$	111.0 d	6.44 ($H_{6'}$)	
$C_{7'}$	16.2 q	2.12 ($H_{7'}$)	
OMe	55.5 q	nm	

[a] HETNOE enhancements measured after 15 s irradiation at the proton frequencies shown at very low power. See refs 18 and 19 for details of the HETNOE method. [b] No cross peak was found in the CHCORR spectrum for this carbon. These two correlations were found on the HMQC spectrum.

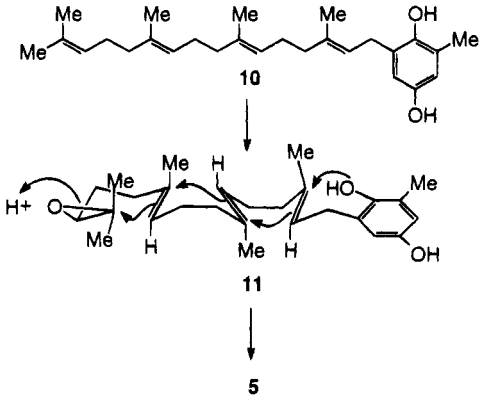

Figure 3. Required folding of epitaondiol precursor **11**.

boehmerol.[24] However, to the best of our knowledge, a pair of twist-boat B/C rings, as found in **5**, is unprecedented. Thus, the formation of epitaondiol from the presumed[25] precursor 6-methyl-2-(geranylgeranyl)-*p*-hydroquinone (**10**) (which has been shown[13] to cooccur with **5** in *Stypopodium zonale*) requires a rather unusual folding of the terminal epoxide **11** as shown in Figure 3.

In contrast, the now well known enzymic cyclization/rearrangement of squalene 2,3-oxide (**12**) to lanosterol *via* protosterol intermediates requires the regular folding depicted in Figure 4b.[21,22] The presence of the highlighted methyls in **12** has been described[23] as crucial for

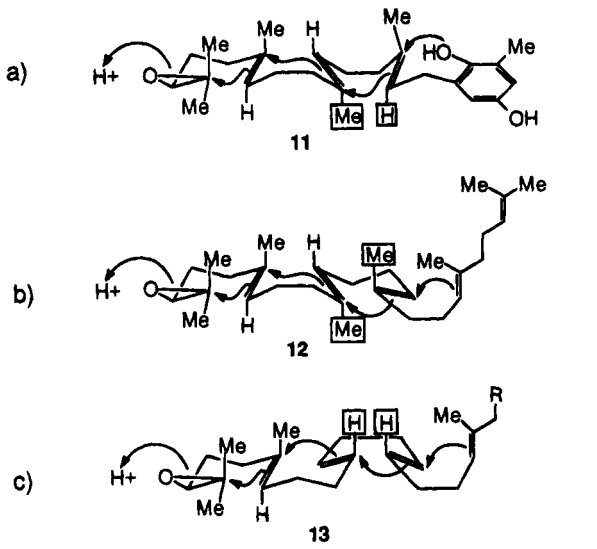

Figure 4. Steric course of several terpenoid polyene epoxide cyclizations: (a) proposed folding of precursor **11** to give epitaondiol **5**; (b) established[21,22] folding of squalene 2,3-oxide (**12**) to give lanosterol via protosterols; (c) established[23] folding of a squalene 2,3-oxide analog **13**.

the correct folding of squalene 2,3-oxide (**12**). Thus, when the hog liver enzyme which converts **12** into lanosterol is used on an analog substrate **13** lacking these two methyls, the cyclization follows a different stereochemical course (Figure 4c), through a folding, in which ring B is now in a pro-chair conformation, while ring C changes to a pro-boat conformation.[23]

It remains to be seen whether or not the enzyme responsible for the formation of epitaondiol **5** from **11** behaves like this hog liver enzyme. Thus, the presence of the highlighted methyl (see structure **11**) could prevent pro-chair folding of the B ring, while the absence of a second methyl group at the position of the highlighted hydrogen (see structure **11**) could result in pro-boat folding of the C ring. Alternatively, the cyclization of **11** to **5** could perhaps proceed stepwise *via* a series of cyclic carbenium ions, although in this case the preference for two consecutive twist boat rings, in spite of their large steric strain,[26] would remain unexplained.

Experimental Section

S. flabelliforme was collected intertidally in December 1990, by hand, using SCUBA at −5 to −10 m depth, near Vaihu, Easter Island, Chile, and frozen immediately for transportation. The previously reported[14] extraction procedure was used with no modifications. Mps (uncorr) were determined on a Kofler hot stage apparatus. Optical rotations were measured with a Perkin-Elmer Model 241 polarimeter using a 10 cm cell. HREIMS were taken on a VG Micromass ZAB -2F spectrometer. Elemental analyses were performed by "Centro de Instrumentación, Facultad de Química, Pontificia Universidad Católica de Chile". NMR spectra were measured using 250 and 400 MHz spectrometers.

Epitaondiol (5). Starting from 1.2 kg of dry algae, 110 mg of crystalline **5** was isolated as colorless needles (from Et_2O), unsuitable for X-ray determination. Mp 149−150 °C (lit.,[13] 149−152 °C); $[\alpha]_D$ +40.1 (lit.,[13] +43.1); HREIMS (*m/z* 412.2972; Δ −0.5 mmu); IR (KBr) ν_{max} 3430, 2940, 1610, 1460, 1370, 1220, 1140, 1020 cm^{-1}; ^1H NMR ($CDCl_3$) δ 6.45 (1H, d, *J* = 2 Hz), 6.38 (1H, d, *J* = 2 Hz), 3.29 (1H, dd, *J* = 10, 4 Hz), 2.68 (1H, dd, *J* = 15, 14 Hz), 2.47 (1H, dd, *J* = 15, 5 Hz), 2.08 (3H, s), 1.22 (3H, s),

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(26) Preliminary results from MM2 calculations show that structure **5** is 18.5 kcal/mol^{-1} less stable than structure **1**.

1.19 (3H, s), 1.00 (3H, s), 0.92 (3H, s), 0.80 (3H, s); ^{13}C NMR ($CDCl_3$) δ 148.5, 144.4, 126.5, 122.1, 115.3, 112.4, 79.2, 76.4, 49.8, 48.4, 45.1, 40.0, 38.9, 36.7, 34.7, 31.6, 31.3, 28.9, 28.5, 27.8, 24.6, 23.0, 21.4, 16.9, 16.8, 15.8, 15.7. Anal. Calcd for $C_{27}H_{40}O_3$: C, 78.59%; H, 9.77%. Found: C, 78.65%; H, 9.68%.

5′-*O*-Methylepitaondiol (6). Epitaondiol (**5**) (100 mg) in THF (30 mL) was treated with NaH (10 mg) and MeI (22 mg) at room temp. After 1 h, the mixture was poured into water and extracted with Et_2O. The extract was dried (anhyd $MgSO_4$) and filtered, and after solvent removal the residue was crystallized from hot acetone with a few drops of water, yielding 100 mg of **6** as fine colorless needles, unsuitable for X ray determination, mp 178–180 °C; $[\alpha]_D$ +49.9 (*c* 2.0, $CHCl_3$); IR (KBr) ν_{max} 3557, 3480, 2993, 2941, 1483, 1439, 1227, 1150, 1060 cm^{-1}; ^1H NMR see Table 1; ^{13}C NMR see Table 2. Anal. Calcd for $C_{28}H_{42}O_3$: C, 78.83%; H, 9.92%. Found: C, 78.46%; H, 9.89%.

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Supplementary Material Available: Copies of 1D and 2D NMR spectra of compounds **5**–**9**, methyl–methyl NOEs in oleanolic acid, table of assigned 400 MHz ^1H NMR spectrum of acetate **7**, preparation of carbamate **8** and allophanate **9** (15 pages). This material is contained in libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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Additions and Corrections

Vol. 57, 1992

Hisao Nemoto, J. Gerald Wilson, Hiroyuki Nakamura, and Yoshinori Yamamoto*. Polyols of a Cascade Type as a Water-Solubilizing Element of Carborane Derivatives for Boron Neutron Capture Therapy.

Page 435, Summary and column 2, the last paragraph. The water solubility of the tetrol **6a** should be 5.44 mmol/L and that of the diol **5a** should be 0.67 mmol/L, although the solubility was reported to be 5.44 mol/L and 0.67 mol/L, respectively. We thank Professor M. F. Hawthorne for this clarification.

JO944011K

Vol. 59, 1994

Seiichi Takano,* Takehiko Yoshimitsu, and Kunio Ogasawara. Asymmetric Dihydroxylation of a *Meso*-Symmetric Cyclic Diene Using AD-Mix Reagents: A New Enantiocontrolled Route to Conduritol E.

Page 54. Since we overlooked the correction of the absolute configuration of (+)-conduritol E (**7**) made by the original authors (ref 5, see: Hudlicky, T.; Luna, H.; Olivo, H. F.; Andersen, Nugent, T.; Price, J. D. *J. Chem. Soc., Perkins Trans. 1* **1991**, 2907), all the absolute configurations shown in our paper (compounds **4**–**12** and **6**–*ent*-**12**) were depicted in inverted forms. They, therefore, should be read in opposite configurations. We greatly appreciate Professor K. B. Sharpless, The Scripps Research Institute, for pointing out our oversight.

JO9440135

Haruyoshi Masuda, Kiyoshi Takase, Masahiro Nishio, Akira Hasegawa, Yutaka Nishiyama, and Yasutaka Ishii*. A New Synthetic Method of Preparing Iodohydrin and Bromohydrin Derivatives through *in Situ* Generation of Hypohalous Acids from H_5IO_6 and $NaBrO_3$ in the Presence of $NaHSO_3$.

Page 5550. Reference 2 should have two additional references: Rodriguez, J.; Dulcère, J.-P. *Synthesis* **1993**, 1177. Bonini, C.; Righi, G. *Synthesis* **1994**, 225.

JO944012C

Xing-Chung Cheng, Mustafa Varoglu, Leif Abrell, Phillip Crews,* Emil Lobkovsky, and Jon Clardy*. Chloriolins A–C, Chlorinated Sesquiterpenes Produced by Fungal Cultures Separated from a *Jaspis* Marine Sponge.

Page 6345, Figure 1. The arrow legends were assigned incorrectly. Figure 1 should be as shown below.

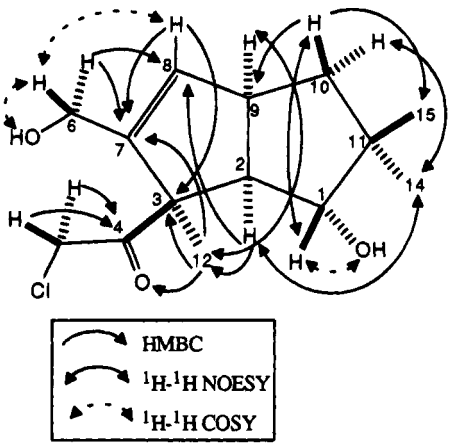

Figure 1.

JO944010S

Francis Beaulieu and Victor Snieckus*. Directed Metalation of Diaryl Sulfone 2-Amides and 2-*O*-Carbamates. Regiospecific General Route to Thioxanthen-9-one 10,10-Dioxides via Anionic Friedel–Crafts and Remote Fries Rearrangement Equivalents.

Page 6509, column 1, line 4 should read "Quenching after 5 min and 1 h resulted in recovery of intractable mixtures and starting material in both cases."

Page 6509, column 2, line 1, should read "starting material (47%), thioxanthenone dioxide (43%), and 4-(trimethylsilyl)thioxanthenone dioxide ...."

JO944009T