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CONFIGURATIONS AND CONFORMATIONS OF TORREYOL (δ-CADINOL), α-CADINOL, T-MUUROLOL AND T-CADINOL

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Abstract—The configurations and conformations of torreyol (**6**; "δ-cadinol", 10α-hydroxy-4-muurolene), α-cadinol (**7**; 10α-hydroxy-4-cadinene), T-muurolol (**8**, 10β-hydroxy-4-muurolene), and T-cadinol (**9**; 10β-hydroxy-4-cadinene) have been determined by an extensive PMR study using lanthanide-induced shifts. Characteristic mass spectrometric fragmentation patterns of muurolols and cadinols are briefly discussed.

Cadinenes and other related sesquiterpenes **1** frequently occur in Nature and have been further divided into four classes of compounds based on the nature of the ring fusion and the orientation of the isopropyl group at C(7). The four classes are the cadinanes **2**, the muurolanes **3**, the bulgaranes **4** and the amorphanes **5**. Due to the complex stereochemistry of the sesquiterpenes belonging to these classes, many of the previous structural and configurational assignments are uncertain. Thus, for example, no less than seven different structures have been proposed for torreyol (δ-cadinol).[1] In this paper we will present an extensive NMR investigation using the lanthanide-induced shift technique (LIS NMR) which firmly establishes the structures and configurations of this alcohol **6** and the three related epimers, α-cadinol **7**, T-muurolol **8** and T-cadinol **9**. Previous structural studies of these alcohols are also briefly reviewed.

Previous studies

Torreyol (**6**; *"δ-cadinol"*, 10α-*hydroxy*-4-*muurolene*). Torreyol has been isolated from various natural sources. It occurs in both the dextrorotatory and levorotatory forms (*cf* Ref. 2) and has been described under various names such as "albicaulol",[3-5] "pilgerol",[6] δ-cadinol,[7] and "sesquigoyol".[5,8] The name "torreyol" has been selected for this compound due to its priority of usage.[2]

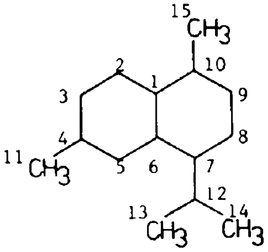
More recently (+)-torreyol was found to be produced as white crystalline needles when the fungi *Clitocybe illudens* was grown on an Agar medium.[9] Torreyol has also been identified by gas chromatography/mass spectrometry in the wings of the male butterfly (*Lysandra argyrognomon*; Lycaenidae, Lepidoptera), where it occurs together with other volatile compounds and plays an important role in the courtship behaviour of the insect.[10]

Several different structures have been proposed for torreyol. Structural investigations performed up to 1970 have been reviewed by Westfelt,[2] who also presented chemical proof for the *cis*-ring fusion in this alcohol. Thus torreyol is of the muurolane type **3**. On the basis of his results, Westfelt proposed structure **6** for (−)-torreyol. Lin *et al.*[1] interpreted some chemical and spectroscopic data and favour structure **10**. However, due to uncertainties in the assignment of the conformation of the compound their data do not provide conclusive evidence for the torreyol structure. Rezvuchin *et al.*[11] performed a LIS–NMR study and a conformational analysis of (+)-torreyol [(+)-δ-cadinol]. They propose that this alcohol is the enantiomer of structure **6**. A synthesis of (±)-torreyol has recently been accomplished,[12] providing additional information concerning the relative configurations of positions C(1), C(6) and C(7).

α-Cadinol (**7**, 10α-*hydroxy*-4-*cadinene*). α-Cadinol was first isolated in the crystalline state from the Japanese citronella oil by Semmler and Spornitz[13] and has since been isolated from several natural sources.[14] The structure of this alcohol **7** was assigned by Sorm *et al.*[7,15] and by Soffer *et al.*[16] The basic skeleton, and the positions of the double bond, and the OH group were determined by classical chemical methods. The assignment of the *trans* ring junction was based on the observation that (−)-α-cadinol is easily converted to (−)-cadinene dihydrobromide **11**, the structure of which has been settled by an X-ray diffraction analysis.[17] This assignment is only valid if it is assumed that no isomerisation occurs during the transformation. However, it has later become clear that such isomerisations frequently occur in similar reactions where muurolenes and muurolols are hydrochlorinated.[2,18]

The decalone **12a** has been prepared from dihydro-α-cadinol *via* an ozonolytic degradation of the exocyclic olefin **12b**.[19] The ORD-data of this ketone provides evidence for the configuration **12b** of the compound. The assignment of structure **7** to (−)-α-cadinol, based on its conversion to the decalone **12a**, is only valid under the assumption that no isomerisation has occurred in the reductive work-up (Zn/HOAc) of the ozonolysis product.

T-Muurolol (**8**; 10β-*hydroxy*-4-*muurolene*) *and T-cadinol* (**9**; 10β-*hydroxy*-4-*cadinene*). Erdtman and Vorbrüggen[14] isolated a sesquiterpene alcohol from



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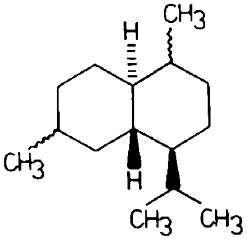
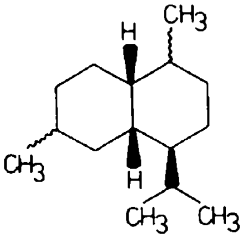
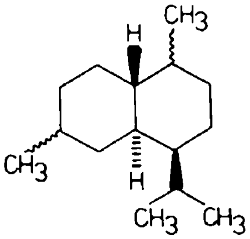


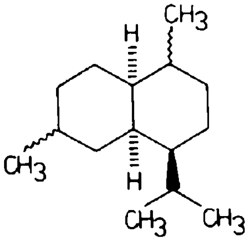
2
Cadinane



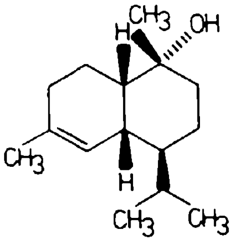
3
Muurolane



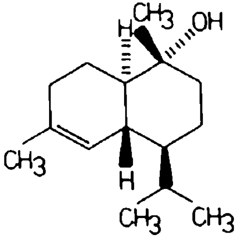
4
Bulgarane



5
Amorphane



6
(-)-Torreyol



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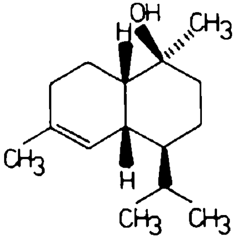
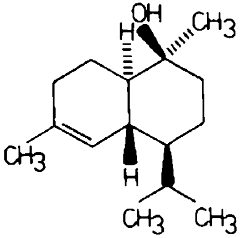
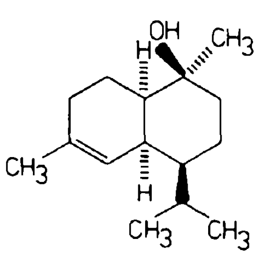
(-)-α-Cadinol



8
(-)-T-Muurolol



9
(+)-T-Cadinol



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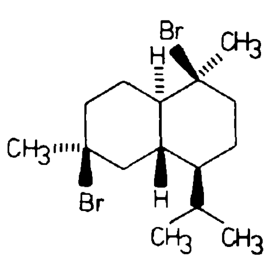

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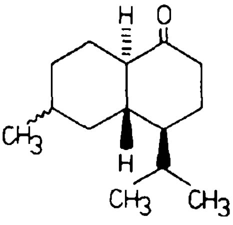

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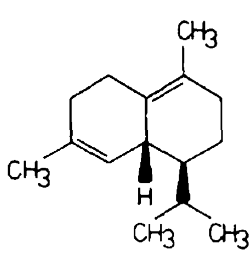

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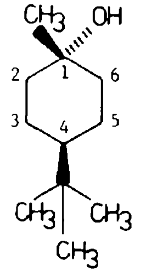
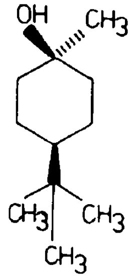


14



15

the heartwood of *Athrotaxis selaginoides* Don. They named this alcohol "α-cadinol". Westfelt and Wickberg[20] later found that this alcohol was a mixture of two new compounds which they named "α_1" and "α_2". Preliminary experiments indicated that "α_1" was a muurolol and "α_2" a cadinol. Cheng *et al.*[21] isolated two sesquiterpene alcohols from the wood of *Taiwania cryptomeroides* (Hayata), which they named T-muurolol and T-cadinol. They also reported that the previously reported α-cadinol of *A. selaginoides*[14] was a mixture of these two alcohols. Thus the alcohols α_1 and α_2 should be identical with T-muurolol and T-cadinol, respectively.

Conclusive evidence regarding the configuration of these two alcohols is not available. However, chemical and spectroscopic studies[21] indicate that T-muurolol and T-cadinol are best represented by the structures **8** and **9**, respectively. The structural assignment is supported by the partial synthesis of (+)-T-muurolol **8** and (+)-T-cadinol **9** from (+)-δ-cadinene **13**.[22]

RESULTS AND DISCUSSION

The ^{1}H-NMR spectra were recorded on a 100 MHz instrument using *ca* 20 mg samples dissolved in carbon tetrachloride (*ca* 450 mg). Weighed amounts of tris(dipivaloylmethanato)europium [Eu(dpm)$_3$] were added and a series of spectra containing different amounts of the Eu-complex were recorded. Assignments of the ^{1}H-NMR signals were based on chemical shifts, integrals and some characteristic coupling patterns verified by spin–spin decoupling. In order to make an unambiguous assignment of the various protons, the LIS-NMR spectra of the two model compounds **14** and **15** were also studied. The LIS NMR data are presented in the Tables 1 and 2. The observed coupling constants are given in Table 3.

The shifts were plotted against the amount of Eu(dpm)$_3$ added. The dependence of the chemical shifts of the various protons on the added amounts of Eu(dpm)$_3$ proved to be linear over a wide range. The slopes of the lines obtained were used as the relative shift values, DEu. The DEu value of the proton with the largest shift was chosen as the intramolecular standard of 100 relative units.

The configurations and conformations given in Fig. 1 were established by an iterative computer fitting of the pseudo-contact equation[23] to the experimental DEu values according to a previously described method.[24,25] The Cartesian coordinates of the various hydrogen atoms in an arbitrary coordinate system were determined by using Dreiding models in a mirror box as described by Stilbs.[27]

Shift data as well as coupling patterns show that the saturated B-ring of the four epimeric alcohols **6**–**9** must occupy a chair conformation with the isopropyl group in the equatorial position. For the computer fitting to the pseudo-contact equation, the unsaturated A-ring was assumed to exist in a twisted conformation (half-chair) as shown in Fig. 1.

The NMR data clearly differentiate between the two *cis* and *trans* ring-fused pairs. In the *cis*-fused compounds **6** and **8** there is a significant coupling between the olefinic proton and the bridgehead C(6)-proton, whereas in the *trans*-isomers **7** and **9** the olefinic proton appears as a slightly broadened singlet (*cf* also Ref. 21).

Of the two *cis*-fused isomers **6** and **8**, which differ in the C(10) configuration, torreyol **6** must have an equatorial OH group, whereas T-muurolol **8** has the OH group in an axial position. This is shown by the characteristic LIS of the vicinal protons and of the axial C(6) and C(8) protons. Similar shifts are observed in the model compounds **14** and **15**. The C(10) configuration of the two *trans*-fused isomers **7** and **9**

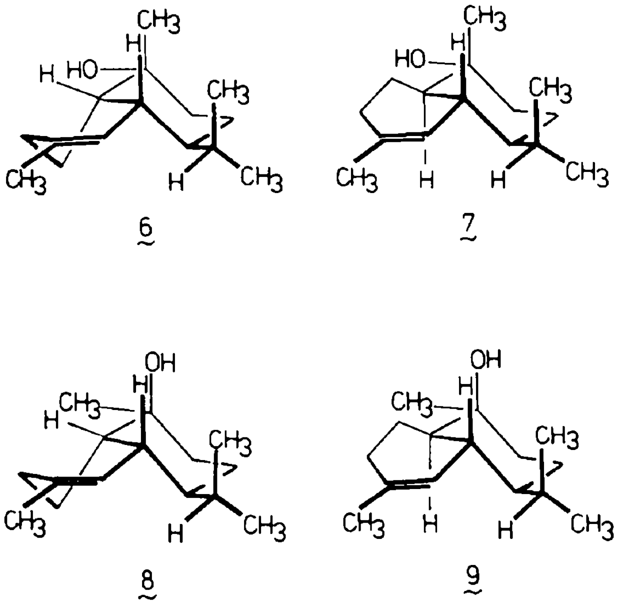


Fig. 1. Stereostructures of (−)-torreyol **6**, (−)-α-cadinol **7**, (−)-T-muurolol **8** and (+)-T-cadinol **9** in the favoured conformations.

Table 1. LIS NMR data of torreyol (**6**), α-cadinol (**7**), T-muurolol (**8**) and T-cadinol (**9**). The data are obtained by using Eu (dpm)$_3$ and the results are quoted as relative DEu values[24] (observed/calculated)

Proton[a]	DEu values Torreyol (6)	α-Cadinol (7)	T-Muurolol (8)	T-Cadinol (9)
1α	-	100.0/97.7	-	65.2/70.2
1β	87.3/89.8	-	100.0/94.2	-
2α	64.5/59.8	80.1/79.7	45.5/47.5	67.8/69.5
2β	96.9/94.2	46.5/48.9	46.0/49.2	81.1/74.2
5	17.5/16.5	16.8/15.8	16.3/12.3	22.9/16.4
6β	45.1/42.0	43.0/42.2	82.3/84.3	83.5/89.3
7α	35.8/37.8	30.1/35.6	42.2/40.3	42.2/42.1
8α	31.4/31.1	30.1/28.5	39.0/35.1	32.3/32.0
8β	44.3/40.8	41.5/43.5	80.2/81.7	78.2/77.5
9α	100.0/96.9	91.8/93.0	58.0/67.6	65.3/68.5
9β	82.0/84.6	91.2/89.5	96.4/92.0	100.0/93.2
11	5.4/6.5	6.5/3.1	6.6/8.4	10.8/15.9
13, 14	12.4/-; 15.7/-	9.5/-; 14.7/-	7.4/-; 12.3/-	7.8/-;11.0/-
15	87.3/-	82.5/-	92.3/-	99.8/-
Agreement factor R[b]	0.043	0.0400	0.069	0.071

[a] For the numbering of the atoms, see structure 1. The α and β notations refer to the face of the molecule according to steroid nomenclature.

[b] Agreement factor $R = \left[\frac{\Sigma(\Delta Eu_{obs} - \Delta Eu_{cal})^2}{\Sigma \Delta Eu_{obs}^2}\right]^{1/2}$ according to Davis and Willcott.[31]

Table 2. Calculated average structures of complexes between Eu(dpm)$_3$ and various alcohols (R = distance between the oxygen and the europeum atoms; θ = the angle C–O... Eu)

Compound	R,Å	θ°
Torreyol 6	2.0	159.5
α-Cadinol 7	2.3	172.1
T-Muurolol 8	2.3	172.2
T-Cadinol 9	2.7	165.9
Alcohol 14 equat.	1.9	165.9
Alcohol 15 axial	2.5	176.9

was assigned, based on the same arguments. The agreement between the observed and the calculated DEu values is convincing. It is interesting to note that the agreement is better for alcohols with equatorial hydroxyl groups (*cf* Table 1). The calculated positions of the europeum atom in the various complexes are given in Table 2. The data are in good agreement with similar examples reported in the literature.[25]

The present investigation clearly demonstrates the steric relationships between the alcohols **6**–**9** and also provides evidence for the preferred conformations of these naturally occurring alcohols.

The mass spectra (MS) of the four epimeric alcohols **6–9** are sufficiently different in order to allow the use of mass spectrometry for identification purposes. However, it is interesting to note the close similarities between the spectra of α-cadinol **7** and T-muurolol **8** (*Experimental*). There is a characteristic difference in the MS fragmentation patterns of some cadinenes and muurolenes.[28] It was hoped that similar differences could be observed in the spectra of the muurolols and the cadinols. However, this was not the case. Therefore, mass spectrometry does not seem to offer a simple way of differentiating between the cadinane **2** and the muurolane **3** classes of compounds.

EXPERIMENTAL

Mps were obtained on a Kofler micro hot stage and are not corrected. NMR spectra were recorded on a JEOL MN 100 instrument (100 MHz) using CCl_4 sols (*ca* 5%). Chemical shifts are given in δ-units, ppm relative to TMS (internal standard). The following abbreviations are used *s* = singlet; *d* = doublet; *tr* = triplet; *q* = quartet; *b* = broad; *u* = unresolved. Lanthanide-induced shift (LIS) data were obtained using tris(dipivaloylmethanato)europium, Eu(dpm)$_3$, as a shift reagent. Results are quoted as relative DEu values[24] (observed/calculated) and are presented in the Tables 1 and 2. Mass spectra (MS) were recorded on an LKB 2091 instrument (GC-MS system; 70 eV). The results are quoted as *m/e* (intensities in % of the most abundant fragment) and generally the ten most important fragments are quoted in order of decreasing intensities. A Varian Aerograph 1200 instrument was used for gas chromatography (GC). The GC separations were performed on a 2 m Carbowax 20 M TPA column (5%) on Chromosorb W.

(–)-*Torreyol* **6**. This alcohol was isolated from a commercial high boiling fraction of Swedish Sulphate Turpentine (derived from kraft pulping of *Pinus silvestris* L), which contained sesqui- and diterpenes. A similar fraction has previously been investigated in detail by Westfelt.[29] The crude fraction was distilled under reduced pressure (20 mm Hg) through a vacuum jacketed, packed column. The pot residue distilling above 130 was redistilled (10 mm Hg) and a fraction b.p. 140–160 was collected This fraction was carefully distilled under reduced pressure using a spinning band column. The fraction with b.p. 86 at 0.6 mm Hg crystallised. Recrystallisation from isopropyl ether followed by sublimation under reduced pressure (10 mm Hg) yielded pure (–)-torreyol **6**: m.p. 138–139 ; $[\alpha]_D$-100.4 ($CHCl_3$, *c* 1.2). NMR (see also Tables 1 3): 5.33 *d*, 1H, H(5), J 6.5 c/s; 1.65 *bs*, 3*H*, H_3(11); 1.24 *s*, 3*H*, H_3(15); 0.90 *d* and 0.81 *d*, each 3*H*, H_3(13) and H_3(14), J 6.9 c/s. MS: 222(M , 1), 161(100), 119(49), 204(46), 105(38), 43(28), 121(25), 93(19), 81(17), 41(15) and 79(13); other characteristic fragments at 58(9), 136(8), 189(8), 147(5) and 175(1).

Isolation of α-cadinol **7**, *T-muurolol* **8** and *T-cadinol* **9**. These alcohols were isolated from a crude sesquiterpene alcohol fraction from the wood of *Athrotaxis selaginoides* Don.[11] A fraction corresponding to that named "1g" by Erdtman and Vorbrüggen[14] was isolated The three alcohols were separated by chromatography on silica gel using ethyl acetate (20%) in light petroleum (b.p. 40–60). The alcohols were

Table 3. Coupling constants (Hz, the absolute values), as observed in the LIS NMR spectra of torreyol (**6**), α-cadinol (**7**), T-muurolol (**8**) and T-cadinol (**9**).

	Observed coupling constants,[J] Hz			
Coupling	Torreyol (6)	α-Cadinol (7)	T-Muurolol (8)	T-Cadinol (9)
1α, 2β	-	10	-	10
1α, 6β	-	12	-	12
1β, 2α	12	-	12	-
2α, 2β	14	13	n	14
2β, 3α	n	n	n	10
3β, 3α	n	n	n	16
5 , 6β	6.5	n	6.5	n
6β, 7α	n	n	n	12
7α, 8β	12	12	12	12
8α, 8β	14	14	14	14
8β, 9α	12	13	12	12
9β, 9α	14	14	15	14

n = the signal not properly resolved for the assignment and measurement of the coupling constants.

eluted in the order: T-cadinol **9**, T-muurolol **8** and α-cadinol **7**. Pure samples were obtained by sublimation under reduced pressure (10 mm Hg). The TLC (silica gel) R_f-values using ethyl acetate (20%) in light petroleum (b.p. 40 60) are 0.49 (T-cadinol), 0.43 (T-muurolol) and 0.33 (α-cadinol).

α-Cadinol **7** m.p 73 74 ; $[\alpha]_D$-37.1 ($CHCl_3$, *c* 0.9). NMR (see also Tables 1–3): 5.29 *bs*, 1*H*, H(5); 1.66 *bs*, 3*H*, $H_3(11)$; 1.05 *s*, 3*H*, $H_3(15)$: 0.91 *d* and 0.78 *d*, each 3*H*, $H_3(13)$ and $H_3(14)$, J 7.2 c/s. MS: 222(M^+, 15), 95(100), 121(85), 43(60), 161(53), 204(50), 164(46), 81(38), 109(37), 105(36), 71(30): other characteristic fragments at 137(25), 58(13), 189(11) and 179(10).

T-Muurolol **8** m.p. 80.5 81.5 : $[\alpha]_D$-114 ($CHCl_3$, *c* 1.1). NMR (see also Tables 1–3). 5.42 *d*, 1*H*, H(5), J 6.5 c/s; 1.62 *bs*, 3*H*, $H_3(11)$, 1.13 *s*, 3*H*, $H_3(15)$; 0.88 *d* and 0.83 *d*, each 3*H*, $H_3(13)$ and $H_3(14)$, J 7.2 c/s. MS: 222(M^+, 15), 95(100), 121(73), 161(68), 43(68), 204(61), 164(44), 105(38), 94(37), 109(35), 71(33), other characteristic fragments 58(18), 139(12), 189(10) and 179(8)

T-Cadinol **9**, m.p. 64.0-64.5 ; $[\alpha]_D$ + 3.4 ($CHCl_3$, *c* 1.2)† NMR (see also Tables 1–3): 5.42 *bs*, 1*H*, H(5): 1.61 *bs*, 3*H*, $H_3(11)$; 1.15 *s*, 3*H*, $H_3(15)$; 0.90 *d* and 0.78 *d*, each 3*H*, $H_3(13)$ and $H_3(14)$, J 6.9 c/s. MS: 222(M^+, 0), 161(100), 204(54), 105(19), 162(18), 43(17), 81(14), 134(14), 95(13), 121(13), 119(11); other characteristic fragments at 69(6) and 58(3).

cis-1-*Methyl-4-t-butylcyclohexanol* **14**.[30] NMR (for numbering of the carbon atoms see structural formula **14**): 1.23 *s*, 3*H*, $H_3(1)$; 0.88 *s*, 9*H*, 4-t-butyl-CH_3. LIS-NMR: *H*(2 and 6, α) 100.0/98.6, $J_{2(6)\alpha,2(6)\beta}$ 13 c/s, $J_{2(6)\alpha,3(5)\beta}$ 12 c/s; *H*(2 and 6, β) 88.1/89.6; *H*(3 and 5, β) 42.2/41.6, $J_{3(5)\beta,3(5)\alpha}$ 13 c/s, $J_{3(5)\beta,4\alpha}$ 12 c/s; *H*(3 and 5, α) 30.2/28.2: *H*(4α) 30.6/34.5: H_3(1-β-Me) 78.7/-: 3xH_3(4-β-t-Bu) 11.7/-

trans-1-*Methyl-4-t-butylcyclohexanol* **15**.[30] NMR (for numbering of the carbon atoms, see formula **14**): 1.21 *s*, $H_3(1)$; 0.88 *s*, 9*H*, 4-t-butyl-CH_3. LIS-NMR: *H*(2 and 6, β) 100.0/95.8, $J_{2(6)\beta,2(6)\alpha}$ 13 c/s, *H*(3 and 5, β) 82.5/83.8, $J_{3(5)\beta,2(6)\alpha}$ 12 c/s, $J_{3(5)\beta,3(5)\alpha}$ 13 c/s, $J_{3(5)\beta,4\alpha}$ 12 c/s; *H*(2 and 6, α) 63.0/69.4; *H*(3 and 5, α) 38.6/36.5: *H*(4α) 42.9/41.3; H_3(1-α-Me) 93.8/-, 3xH_3(4-β-t-Bu) 13.4/-

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† The optical rotation of (+)-T-cadinol **9** is small. Our sample exhibits positive rotation when recorded on a chloroform solution (*c* 1.2). Cheng *et al.*[21] report a small negative rotation $[\alpha]_D^{30}$ − 4.7 ($CHCl_3$, *c* 4.4), for T-cadinol of the same absolute configuration. However, since this alcohol exhibits a plane positive ORD curve ($[\alpha]_{436}$ + 5.9: $CHCl_3$, *c* 1.2) this enantiomer is quoted as the (+)-form.