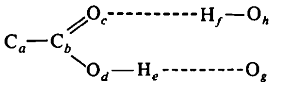
Table 4. *Representative distances and angles in intermolecularly hydrogen-bonded carboxyl moieties*

Values are uncorrected for thermal motion.



	–C=O (Å)	>C–OH (Å)	C–C=O (°)	C–C–OH (°)	O=C–OH (°)
Planar*	1·22	1·29	123	115	122
*R*COOH†	1·244	1·277	118·7	117·6	123·6
Twisted‡	1·245	1·279	119	117	124

* Dunitz & Strickler (1968); Derissen & Smit (1974).
† This work.
‡ Trimesic acid; Duchamp & Marsh (1969).

trimesic acid which are approximately coplanar with their benzene rings and which, consequently, also form twisted hydrogen-bonded moieties. On the other hand, the carboxyl groups in trimesic acid which are rotated out of the planes of the benzene rings and do not form twisted hydrogen-bonded moieties have interatomic distances and angles which are similar to those reported for other nearly planar hydrogen-bonded moieties. Since the differences between the twisted and planar structures are small, a systematic investigation of these systems is needed to reveal if the 'twist' of the hydrogen-bonded moiety has a significant effect on the hydrogen-bond parameters which are usually reported and to determine if there is a relationship between the C_α–C_β–C=O torsion angle and the hydrogen-bond moiety twist.

From the rigid-body analysis it was found that in *R*COOH the root-mean-square (r.m.s.) amplitudes of libration about the three principal axes are 4·54, 3·90 and 2·61°, while the r.m.s. amplitudes of translation are 0·164, 0·161 and 0·138 Å. These amplitudes of libration are considerably smaller than those observed in other plastic-crystal precursors, such as pentaerythritol (Eilerman & Rudman, 1979) and 2-hydroxymethyl-2-methyl-1,3-propanediol (Eilerman & Rudman, 1980). However, these other materials form layered structures with strong hydrogen bonding found only within the layers, while in *R*COOH the librational motion is restricted because the hydrogen-bonded network is three-dimensional.

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Structure of Obtusol Acetate, a Halogenated Chamigrene-Type Sesquiterpene

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Abstract. $C_{17}H_{25}Br_2ClO_2$, monoclinic, $P2_1$, $Z = 2$, $a = 8{\cdot}916\,(1)$, $b = 14{\cdot}622\,(3)$, $c = 7{\cdot}703\,(1)$ Å, $\beta = 104{\cdot}23\,(1)°$, $V = 973{\cdot}4\,(2)$ Å^3, $D_x = 1{\cdot}547$ Mg m^{-3}, μ(Mo $K\alpha$) = 45·2 mm^{-1}. The structure was refined to $R = 0{\cdot}064$ for 1080 observed Friedel pairs. The molecule consists of two six-carbon-atom rings, both in

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chair conformations and forming a (−)-β-chamigrene-type skeleton. Halogen substituents are all equatorial while the acetate group is axial. The geometry of the carbon skeleton with its substituents and the absolute configuration are compared with those of other known chamigrene-type sesquiterpenes.

Introduction. Red alga *Laurencia obtusa* forms several sesquiterpenes of both (+)-β-chamigrene and (−)-β-chamigrene types (Gonzalez, Darias, Diaz, Fourneron, Martin & Pérez, 1976). Previously, two crystal structure determinations of these sesquiterpenes have been performed: one was of the (+)-β-chamigrene type, elatol (Sims, Lin & Wing, 1974), the other of the (−)-β-chamigrene type, isoobtusol (to be published). Crystals of obtusol acetate, another (−)-β-chamigrene sesquiterpene of *L. obtusa*, were provided by A. G. Gonzalez of the Instituto de Productos Naturales Orgánicos, Tenerife, Spain. These three molecules are shown in Fig. 1. A colourless single crystal of section 0·2 mm supplied intensities for 1778 independent Friedel pairs in the range $2 < \theta < 25°$ (Mo $K\alpha$ radiation). A fully automated diffractometer with a graphite monochromator, the ω–2θ scan and a scan speed of 0·04° s^{-1} were used for data collection. An intensity decay of 18% was observed during the 80 h of experiment at room temperature. After Lorentz and polarization correction, 1080 Friedel pairs with $I > 2\sigma(I)$ were considered observed. Intensities were corrected for absorption with factors ranging from 1·6 to 2·9. The crystal structure was solved by the heavy-atom method and refined by anisotropic full-matrix least squares. The H atoms were found in a difference map, except for those of methyl groups which were assumed to be disordered between two alternative positions. Fixed positional and isotropic thermal parameters of H atoms and anomalous-dispersion factors for Br and Cl were included in the last cycle of the correct enantiomorph. All observed Friedel pairs were used in that refinement, after a weighting scheme to prevent bias of ΔF *vs* F_o and $\sin\theta/\lambda$. The final $R = 0{\cdot}064$ and $R_w = 0{\cdot}059$. The absolute configuration of the molecule, shown in Figs. 1(*c*) and 2, was decided after a check of the 29 most relevant Bijvoet pairs, those with $F_o > 10\sigma(F_o)$, $|\Delta|F_c|| > 3{\cdot}0$ and $|\Delta|F_o|| > 1{\cdot}0$, which gave the following discrepancy indices: averaged Bijvoet difference for the right enantiomorph of 0·45 *vs* 8·74 for the wrong one, and averaged

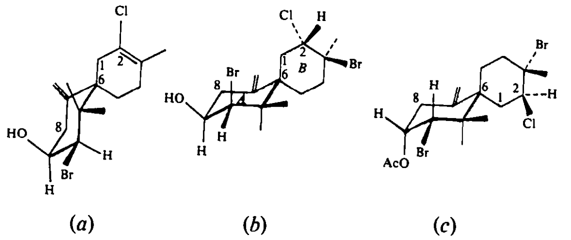

Fig. 1. Stereochemistry of (*a*) elatol, (*b*) isoobtusol and (*c*) obtusol acetate.

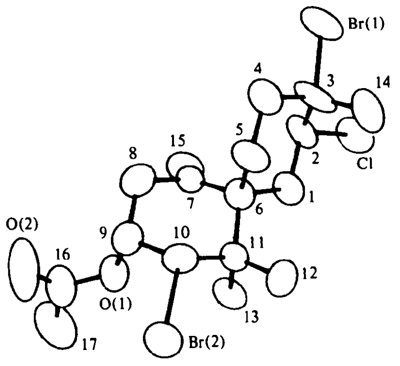

Fig. 2. X-ray model of obtusol acetate. C atoms are designated by numbers only.

Table 1. *Fractional atomic coordinates* (×10^4) *with e.s.d.'s in parentheses*

	x	*y*	*z*
Br(1)	−1158 (1)	5000	7355 (2)
Br(2)	4222 (1)	8395 (1)	16493 (1)
Cl	1356 (4)	3753 (2)	10627 (4)
O(1)	5908 (8)	7934 (4)	13572 (10)
O(2)	6679 (11)	9297 (6)	12892 (17)
C(1)	2337 (12)	5363 (6)	12216 (15)
C(2)	1416 (10)	4994 (6)	10405 (14)
C(3)	−203 (13)	5404 (8)	9859 (18)
C(4)	26 (13)	6416 (7)	9736 (18)
C(5)	872 (11)	6835 (8)	11568 (16)
C(6)	2532 (12)	6421 (6)	12248 (13)
C(7)	3585 (12)	6729 (7)	11149 (13)
C(8)	3748 (14)	7772 (8)	11035 (15)
C(9)	4330 (13)	8189 (7)	12912 (15)
C(10)	3398 (12)	7812 (7)	14101 (13)
C(11)	3246 (12)	6742 (6)	14262 (13)
C(12)	2198 (14)	6568 (8)	15499 (15)
C(13)	4827 (11)	6269 (7)	15056 (15)
C(14)	−1292 (14)	5113 (10)	10983 (19)
C(15)	4420 (13)	6228 (9)	10337 (16)
C(16)	6974 (16)	8568 (8)	13528 (17)
C(17)	8573 (14)	8220 (12)	14403 (21)

Table 2. *Bond lengths* (Å)

The averaged e.s.d. is 0·01 Å

C(1)–C(2)	1·53	C(7)–C(8)	1·56
–C(6)	1·56	–C(15)	1·31
C(2)–Cl	1·82	C(8)–C(9)	1·54
–C(3)	1·53	C(9)–O(1)	1·42
C(3)–Br(1)	2·00	–C(10)	1·49
–C(4)	1·50	C(10)–Br(2)	2·00
–C(14)	1·51	–C(11)	1·58
C(4)–C(5)	1·55	C(11)–C(12)	1·51
C(5)–C(6)	1·57	–C(13)	1·55
C(6)–C(7)	1·48	C(16)–O(1)	1·33
–C(11)	1·60	–O(2)	1·18
		–C(17)	1·51

Bijvoet ratio of 0·05 *vs* 0·69. Atomic coordinates, bond lengths and angles for non-H atoms are in Tables 1, 2 and 3.*

Discussion. As shown in Fig. 1, both obtusol acetate and isoobtusol have the same (–)-β-chamigrene-type skeleton in contrast to the (+)-β-chamigrene of elatol. If one compares the corresponding torsion angles (Table 4), the chamigrene skeletons of both obtusol acetate and isoobtusol are almost equal. From Table 4 it can also be seen that both rings have a slightly flattened chair conformation. The bond lengths and angles of both compounds are also comparable with no differences $>3\sigma$. A common feature of the three molecules in Fig. 1 is that the Cl atom is substituted on C(2) in contrast to other *L. obtusa* sesquiterpenes like caespitol and isocaespitol (Gonzalez, Darias, Martin & Pérez, 1974) which are substituted on C(3). The only difference between isoobtusol and obtusol acetate consists in the positions of the substituents: while in the former the halogens are all axial, in the latter they are all equatorial. Also, with respect to ring *A*, C(1) is axial for isoobtusol but equatorial for obtusol acetate. Only substituents at C(4) reverse that situation. As is usually found, the acetyl group is *cis* with respect to O(2), the torsion angle C(9)–O(1)–C(16)–O(2) being 4 (1)°. The torsion angle of 18° for H(9)–C(9)–O(1)–C(16) probably indicates a repulsion of Br(2). Finally, the vinyl C(15) groups for both isoobtusol and obtusol acetate form opened bond angles C(15)=C(7)–C(6) of 125 (1) and 128 (1)°, respectively, probably due to the repulsion of ring *B*.

Table 3. *Bond angles* (°)

The averaged e.s.d. is 0·8°.

C(2)–C(1)–C(6)	113·4	C(6)–C(7)–C(8)	114·6
Cl–C(2)–C(1)	106·6	C(6)–C(7)–C(15)	128·3
Cl–C(2)–C(3)	111·7	C(8)–C(7)–C(15)	117·1
C(1)–C(2)–C(3)	111·8	C(7)–C(8)–C(9)	110·8
Br(1)–C(3)–C(2)	107·4	C(8)–C(9)–C(10)	108·8
Br(1)–C(3)–C(4)	105·3	C(8)–C(9)–O(1)	107·9
Br(1)–C(3)–C(14)	106·8	C(10)–C(9)–O(1)	109·6
C(2)–C(3)–C(4)	105·7	C(9)–C(10)–Br(2)	106·0
C(2)–C(3)–C(14)	115·5	C(9)–C(10)–C(11)	119·2
C(4)–C(3)–C(14)	115·5	C(11)–C(10)–Br(2)	111·7
C(3)–C(4)–C(5)	112·0	C(6)–C(11)–C(10)	103·7
C(4)–C(5)–C(6)	111·1	C(6)–C(11)–C(12)	113·8
C(1)–C(6)–C(7)	112·2	C(6)–C(11)–C(13)	110·9
C(1)–C(6)–C(5)	106·6	C(10)–C(11)–C(12)	107·2
C(1)–C(6)–C(11)	108·9	C(10)–C(11)–C(13)	112·5
C(5)–C(6)–C(7)	111·8	C(12)–C(11)–C(13)	108·6
C(5)–C(6)–C(11)	109·3	C(9)–O(1)–C(16)	117·2
C(7)–C(6)–C(11)	108·0	O(1)–C(16)–O(2)	123·6
		O(1)–C(16)–C(17)	110·8
		O(2)–C(16)–C(17)	125·6

Table 4. *Torsion angles* (°) *of the rings in obtusol acetate*

In square brackets are given those torsion angles corresponding to isoobtusol by superposition of this molecule over obtusol acetate. Averaged e.s.d.'s are 1° for obtusol acetate and 2° for isoobtusol.

C(6)–C(1)–C(2)–C(3)	59 [56]
C(1)–C(2)–C(3)–C(4)	–60 [–50]
C(2)–C(3)–C(4)–C(5)	62 [49]
C(3)–C(4)–C(5)–C(6)	–62 [–54]
C(4)–C(5)–C(6)–C(1)	53 [50]
C(5)–C(6)–C(1)–C(2)	–53 [–52]
C(11)–C(6)–C(7)–C(8)	63 [58]
C(6)–C(7)–C(8)–C(9)	–58 [–63]
C(7)–C(8)–C(9)–C(10)	48 [53]
C(8)–C(9)–C(10)–C(11)	–53 [–52]
C(9)–C(10)–C(11)–C(6)	57 [49]
C(10)–C(11)–C(6)–C(7)	–57 [–49]

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34619 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.