C(5)—N(1)—N(2)—C(6)	−14.2 (4)	C(2)—C(3)—C(4)—N(1)	14.6 (6)
C(4)—N(1)—N(2)—C(1)	61.2 (4)	N(2)—N(1)—C(5)—N(3)	11.7 (4)
N(1)—N(2)—C(1)—C(2)	−43.9 (4)	C(6)—N(3)—C(5)—N(1)	−5.4 (4)
N(2)—C(1)—C(2)—C(3)	15.8 (5)	N(1)—N(2)—C(6)—N(3)	10.4 (4)
C(1)—C(2)—C(3)—C(4)	−2.9 (6)	C(5)—N(3)—C(6)—N(2)	−3.2 (4)
N(2)—N(1)—C(4)—C(3)	−42.1 (4)		

Table 2. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O(50)—H(50*B*)···O(1^{i})	0.96	2.71 (5)	2.91 (2)	93 (3)
O(50)—H(50*B*)···N(3^{ii})	0.96	3.36 (4)	4.22 (4)	148 (4)

Symmetry codes: (i) $x, \frac{1}{2} - y, z - \frac{1}{2}$; (ii) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$.

From the first steps of the refinement, a residual electron density appeared. An unexpected water molecule could be modelled into the residual electron density. The water O atom was disordered [U_{eq} for the O atom = 0.575 (13) Å^2] and the H atoms must be geometrically placed. All non-H atoms in the asymmetric unit were anisotropically refined, and all H atoms except for the water molecule were located by a difference Fourier synthesis. A common displacement parameter was refined for all the H atoms. The final difference Fourier map showed no peaks higher than 0.29 e Å^{-3} and no deeper than −0.30 e Å^{-3}.

Data were collected by profile analysis over all reflections (Lehman & Larsen, 1974; Grant & Gabe, 1978) using Nonius CAD-4 diffractometer software. Cell refinement: *CRYSDA* (Beurskens *et al.*, 1992). Data reduction: *THE REFLEX* (Aguirre-Pérez, Gutiérrez-Rodríguez & García-Granda, 1997). The structure was solved using direct methods (*SHELXS*86; Sheldrick, 1985) and refined by anisotropic least-squares techniques using *SHELXL*93 (Sheldrick, 1993). Geometrical calculations were made with *PARST* (Nardelli, 1983) and molecular graphics incorporated *EUCLID* (Spek, 1982). *SHELXL*93 was used for preparing the material for publication and all calculations were made at the University of Oviedo on the Scientific Computer Centre and X-ray group VAX/AXP computers.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1480). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1943–1945

Cyclopropanation of a β-Himachalene Monoepoxide Derivative

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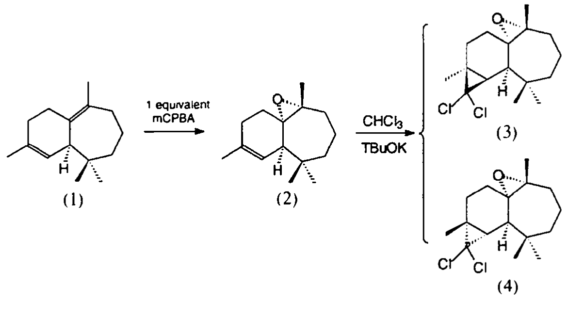
Abstract

The stereochemistry of the title compound, (1*S*,2*R*,7*S*,8*S*,10*R*)-9,9-dichloro-1,2-epoxy-2,6,6,10-tetramethyltricyclo[5.5.0.0.^{8,10}]dodecane, $C_{16}H_{24}Cl_2O$, a derivative of β-himachalene, has been established. The epoxide bridge located at positions 1 and 2 on the seven-membered ring adopts a *cis* conformation with respect to the cyclopropane bridge located at positions 8 and 10 on the six-membered ring.

Comment

β-Himachalene, (1), is the main constituent of Atlas Cedar essential oils (Plattier & Teisseire, 1974). An early study (Joseph & Dev, 1968*a*) performed on Himalaya Cedar showed that (1) is a major component in its oils. The reactivity of this sesquiterpene has been widely investigated (Joseph & Dev, 1968*b*,*c*; Plattier & Teisseire, 1974) and this laboratory has studied its epoxidation and the reactivity of its derivatives.



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The treatment of β-himachalene, (1), with one equivalent of *meta*-chloroperbenzoic acid (*m*CPBA) leads, with a quantitative yield, to the monoepoxide with an α configuration at positions 1 and 2, *i.e.* (2) (Chiaroni *et al.*, 1991). The reactivity of compound (2) has been reported in the literature (Teisseire & Plattier, 1974; Narula & Dev, 1977; Benharref, Chekroun & Lavergne, 1991; Chiaroni, Riche, Benharref, Lassaba & Baouid, 1996). The action of dichlorocarbene (produced from ^{t}BuOK and $CHCl_3$) on (2) leads to compounds (3) and (4) in the ratio 25:75. The absolute structure of the himachalene core has been confirmed previously by circular dichroism and chemical correlations (Joseph & Dev, 1968*b*) and by the crystal structure determinations of epoxide derivatives (Chiaroni *et al.*, 1991). The structure determination of compound (4) now allows us to assign the stereochemistry of the cyclopropane bridge in positions 8 and 10 for (3) and (4).

The bond lengths and angles in (4) are very similar to those found in related molecules (Chiaroni, Riche, Benharref, Chekroun & Lavergne, 1992; Benharref, El Jamili, Lassaba, Giorgi & Pierrot, 1995). The core of the molecule consists of a six- and a seven-membered fused ring system. The torsion angles reported in Table 1 show that the six-membered ring adopts a conformation approaching that of a cyclohexene half chair, with atom C12 located 0.685 (2) Å out of the mean plane composed of atoms C1, C7, C8, C10 and C11.

The seven-membered ring adopts a conformation closer to an envelope, while in a similar compound (Benharref, El Jamili, Lassaba, Giorgi & Pierrot, 1995), it has been described as a chair conformation. Atoms C4, C5 and C6 are 1.336 (3), 2.088 (2) and 1.380 (2) Å, respectively, out of the plane composed of atoms C7, C1, C2 and C3.

The epoxide bridge shares a common atom, C1, with the two rings of the molecule and the cyclopropane bridge, located at positions 8 and 10 on the six-membered ring, is in a *cis* conformation with respect to the epoxide (Fig. 1).

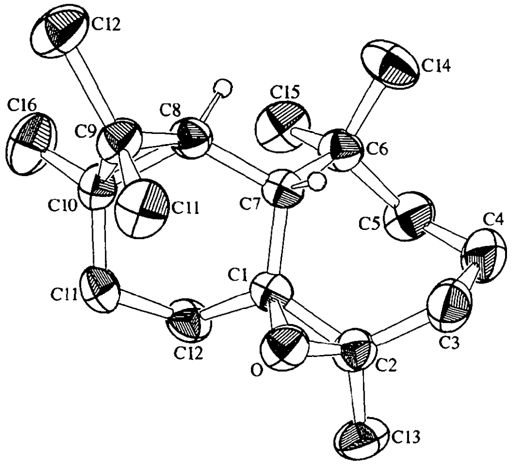

Fig. 1. An *ORTEP*II (Johnson, 1976) drawing of the title molecule showing the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

Experimental

Compound (4) was crystallized by evaporation of a pentane solution.

Crystal data

$C_{16}H_{24}Cl_2O$
$M_r = 303.28$
Orthorhombic
$P2_12_12_1$
$a = 14.208\ (4)$ Å
$b = 16.484\ (6)$ Å
$c = 6.663\ (2)$ Å
$V = 1560.5\ (9)$ Å^3
$Z = 4$
$D_x = 1.29$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11-19°$
$\mu = 0.407$ mm^{-1}
$T = 294$ K
Prism
0.7 × 0.5 × 0.5 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1594 measured reflections
1594 independent reflections
1515 reflections with $I > 0.5\sigma(I)$

$\theta_{\max} = 25°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 19$
2 standard reflections frequency: 120 min intensity decay: −1.03%

Refinement

Refinement on F
$R = 0.036$
$wR = 0.048$
$S = 1.840$
1515 reflections
173 parameters
H atoms included but not refined
$w = 4F_o^2/[\sigma^2(F_o^2) + 0.0016F_o^4]$
$(\Delta/\sigma)_{\max} = 0.059$

$\Delta\rho_{\max} = 0.3106$ e Å^{-3}
$\Delta\rho_{\min} = -0.2214$ e Å^{-3}
Extinction correction: isotropic (Zachariasen, 1963)
Extinction coefficient: 0.21×10^{-5}
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

O—C1	1.446 (3)	C8—C9	1.502 (3)
O—C2	1.445 (3)	C8—C10	1.537 (4)
C1—C2	1.469 (4)	C9—C10	1.508 (4)
C1—O—C2	61.1 (2)	C9—C8—C10	59.5 (2)
O—C1—C2	59.4 (2)	C8—C9—C10	61.4 (2)
O—C2—C1	59.5 (2)	C8—C10—C9	59.1 (2)
C7—C1—C2—C3	3.7 (3)	C4—C5—C6—C7	−56.6 (3)
C2—C1—C7—C6	67.0 (2)	C5—C6—C7—C1	−29.0 (3)
C12—C1—C7—C8	35.2 (2)	C1—C7—C8—C10	−7.3 (3)
C7—C1—C12—C11	−62.4 (2)	C7—C8—C10—C11	6.7 (3)
C1—C2—C3—C4	−74.4 (3)	C8—C10—C11—C12	−32.4 (3)
C2—C3—C4—C5	33.8 (3)	C10—C11—C12—C1	59.5 (2)
C3—C4—C5—C6	55.1 (3)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *BEGIN* in *SDP-Plus* (Frenz, 1985). Program(s) used to solve structure: direct methods *MULTAN*80 (Main *et al.*, 1980). Program(s) used to refine structure: *LSFM* in *SDP-Plus*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *CIF VAX* in *MolEN* (Fair, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: LN1007). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). **C53**, 1945–1947

3,3-Dichloro-4-(4-chlorophenyl)-1-phenylazetidin-2-one

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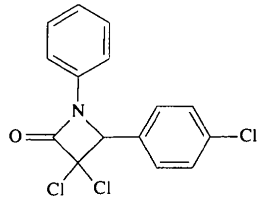
Abstract

In molecules of $C_{15}H_{10}Cl_3NO$, the four-membered lactam ring is nearly planar, with a C—N bond length of 1.367 (4) Å in the amide group and C—C distances of 1.535 (5) and 1.566 (5) Å. The displacement of the amide N atom from the plane through the atoms attached to it is 0.056 (3) Å, indicating some pyramidal character which is correlated to the biological activity in related compounds. The phenyl rings are nearly perpendicular to one another [dihedral angle 79.4 (1)°]. There are intramolecular and intermolecular hydrogen bonds in the structure.

Comment

As a result of their reported antibiotic and antifungal activity (Chambers & Doedens, 1980), structural studies of monocyclic β-lactams have been of interest to this laboratory (Ercan, Ülkü & Güner, 1996*a*,*b*). The four lactam ring atoms are coplanar to within experimental error in other monocyclic β-lactams (Paulus, Kobelt & Jensen, 1969; Kartha & Ambady, 1973; Colens, Declercq, Germain, Putzeys & Van Meerssche, 1974). However, in the title compound, (I), the lactam ring N atom is displaced by 0.056 (3) Å from the C2/C4/C11 plane which indicates that it adopts a flat pyramidal coordination. Sweet & Dahl (1970) reported a correlation between the pyramidal character of the amide N atom, which possibly arises from intramolecular repulsions and the amide C—N bond lengths. It has been suggested that the antibiotic activity of β-lactams depends on the pyramidal character of the lactam N atoms. There also seems to be a correlation between increased activity and decreased electron delocalization in the amide group as shown by a lengthening of the C—N bond and a shortening of the C═O bond (Lee *et al*, 1990; Lee *et al*, 1990).



(I)

A comparison of the activity-related structural parameters of (I) with those reported for biologically active and inactive compounds, indicates that its parameters are closer to those of the active compounds. For instance, the C—N bond length of 1.367 (4) Å is closer to the corresponding C—N bond length [1.392 (4) Å; Domiano, Nardelli, Blasmo, Macchia & Macchia, 1979] reported for an active penicillin derivative, than to the corresponding C—N bond lengths observed in some structurally related but biologically inactive compounds, such as azetidone derivatives [1.342 (4)–1.337 (4) Å; Lee *et al.*, 1990; Lee *et al*, 1990] and Δ^2-cephalosporin (1.339 Å; Sweet & Dahl, 1970). The displacement of the lactam ring N atom is also notably larger than in the inactive compound mentioned above. Whether these

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