

## Data collection

Siemens P4 diffractometer	$\theta_{\max} = 25^\circ$
$\omega$ scans	$h = -24 \rightarrow 24$
Absorption correction: none	$k = -11 \rightarrow 0$
3496 measured reflections	$l = 0 \rightarrow 10$
3269 independent reflections	3 standard reflections
2633 reflections with	every 97 reflections
$I > 2\sigma(I)$	intensity decay: 1%
$R_{\text{int}} = 0.015$	

## Refinement

Refinement on $F^2$	$(\Delta/\sigma)_{\max} = 0.003$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$\Delta\rho_{\max} = 0.186 \text{ e } \text{\AA}^{-3}$
$wR(F^2) = 0.085$	$\Delta\rho_{\min} = -0.305 \text{ e } \text{\AA}^{-3}$
$S = 1.062$	Extinction correction: none
3268 reflections	Scattering factors from
273 parameters	International Tables for
H atoms: see below	Crystallography (Vol. C)
$w = 1/[\sigma^2(F_o^2) + (0.048P)^2$	
$+ 0.616P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1. Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ )

D—H...A	D—H	H...A	D...A	D—H...A
O12—H12O...N11A	0.84	1.995	2.830 (2)	175
O15—H15O...N11B <sup>a</sup>	0.84	2.004	2.826 (2)	167
N10A—H10A...N8A <sup>ii</sup>	0.86	1.996	2.844 (2)	167
N10B—H10B...N8B <sup>iii</sup>	0.87	2.001	2.845 (2)	162

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

H atoms were placed in idealized positions and refined as riding.

Data collection: XSCANS (Fait, 1993). Cell refinement: XSCANS. Data reduction: XSCANS. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990a). Program(s) used to refine structure: SHELXL93 (Sheldrick, 1993). Molecular graphics: SHELXTL/PC (Sheldrick, 1990b). Software used to prepare material for publication: SHELXL93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1258). Services for accessing these data are described at the back of the journal.

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## Isoavarol diacetate

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## Abstract

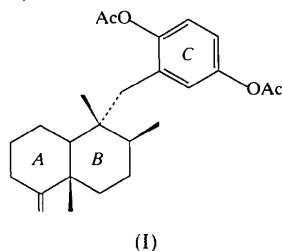
The crystal and molecular structures of isoavarol diacetate, (1*R*, 2*S*, 4*aS*, 8*aS*)-2-(1,2,4a-trimethyl-5-methylene-perhydro-1-naphthylmethyl)-*p*-phenylene diacetate, C<sub>25</sub>H<sub>34</sub>O<sub>4</sub>, have been determined. There are two molecules of this quinoid-containing sesquiterpene in the asymmetric unit. Each of these molecules participates in intermolecular C—H...O contacts between the aromatic C—H groups and the carbonyl-O atoms of the acetyl groups. Isoavarol differs from avarol in both the exocyclic position of the double bond at C4 and the chair conformation of ring A. The acetyl groups in each molecule of the title compound are oriented on one side of the phenylene ring, on opposite sides of the bicyclic system of the molecule.

## Comment

Isoavarol, a sesquiterpene hydroquinone isomer of the well known compound avarol (De Rosa *et al.*, 1976), has been isolated from the Pacific sponge *Dysidea sp.* and its structure, including its absolute configuration, has been studied using spectroscopic and chemical correlative methods (Shubina *et al.*, 1990). Simultaneously, the same compound was obtained from the Okinawan *Dysidea* (Iguchi *et al.*, 1990) and named as neoavarol. Current interest in structural investigations on isoavarol and its derivatives is stimulated by the high and diverse biological activities of avarol itself, as well as of some related prenylated sesquiterpene quinones (Müller *et al.*, 1987; De Giulio *et al.*, 1990; Pennings *et al.*, 1994).

Recently, crystal structures of avarol (Puliti *et al.*, 1994) and its diacetate (Puliti *et al.*, 1995) have been reported, but an X-ray analysis of isoavarol and its derivatives has not previously been reported. Certain long range effects are characteristic of natural products belonging to this series; for instance, a substitution of the hydroxyl group at C4 in illimaquinone results in the rotation of the aromatic residues around the C9—C15 bond (Ilyin *et al.*, 1991). To elucidate the spatial

structure of isoavarol, the X-ray analysis of isoavarol diacetate, (I), was carried out.



A perspective view of the final X-ray model of the two molecules of (I) is shown in Fig. 1. The two molecules are connected by C—H...O hydrogen bonds in an asymmetric unit. Ring A in (I) is in a chair conformation, and there is an exocyclic double bond. Ring B has a chair form, analogous to the conformation found in avarol diacetate, (II) (Puliti *et al.*, 1995): C9

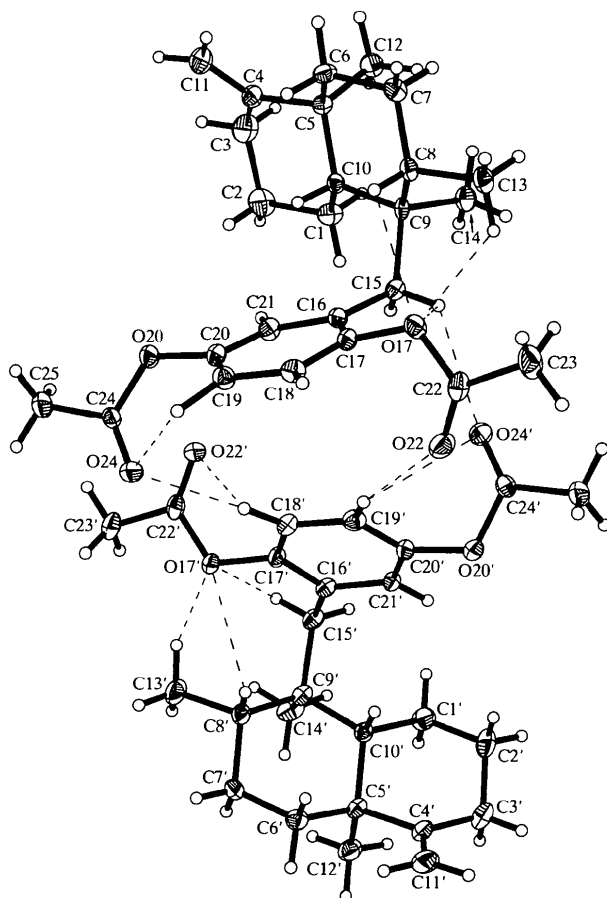


Fig. 1. A perspective view of the two molecules of (I), showing the C—H...O contacts together with the atom-numbering scheme for the non-H atoms. Displacement ellipsoids are plotted at the 30% probability level and H atoms are drawn as spheres of an arbitrary radius.

deviates by 0.568 (3) and C6 by  $-0.685$  (3) Å from the C7/C8/C5/C10 plane. The orientations of the aromatic rings relative to ring B are similar in (I) and (II). The C8'—C9'—C15'—C16' torsion angle has a value of  $59.8$  (2)° in (I), which is smaller than the  $63.3$  (3)° found in (II). The corresponding torsion angles around the C15—C16 and C15'—C16' bonds differ from each other more than they do from the value of  $-96.2$  (3)° in (II). In (II), the acetyl groups are oriented on opposite sides of the phenylene ring, whilst in each of the two molecules of (I) they are oriented on the same side of the ring, on opposite sides of the bicyclic system. The C2—C3 and C2'—C3' bond lengths differ by 0.035 Å.

The important intra- and intermolecular C—H...O contacts for both independent molecules of (I) are given in Table 2. The molecules form hydrogen bonds of the C—H...O type, giving infinite chains.

## Experimental

The title compound was obtained by acetylation of a mixture of avarol and isoavarol, isolated from the marine sponge *Dysidea sp.* This was followed by sequential separation. Slow evaporation from an ethanol solution yielded crystals suitable for X-ray analysis.

### Crystal data

C<sub>25</sub>H<sub>34</sub>O<sub>4</sub>  
 $M_r = 398.52$   
 Monoclinic  
 P2<sub>1</sub>  
 $a = 6.462$  (1) Å  
 $b = 33.014$  (7) Å  
 $c = 10.457$  (2) Å  
 $\beta = 93.30$  (3)°  
 $V = 2227.2$  (7) Å<sup>3</sup>  
 $Z = 4$   
 $D_x = 1.189$  Mg m<sup>-3</sup>  
 $D_m$  not measured

Mo K $\alpha$  radiation  
 $\lambda = 0.71069$  Å  
 Cell parameters from 24 reflections  
 $\theta = 11$ –13°  
 $\mu = 0.079$  mm<sup>-1</sup>  
 $T = 183$  (2) K  
 Prism  
 $0.42 \times 0.25 \times 0.10$  mm  
 Colourless

### Data collection

Syntex P2<sub>1</sub> diffractometer  
 $\theta/2\theta$  scans  
 Absorption correction: none  
 4113 measured reflections  
 3722 independent reflections  
 3195 reflections with  
 $I > 2\sigma(I)$   
 $R_{int} = 0.026$

$\theta_{max} = 27.06$ °  
 $h = 0 \rightarrow 7$   
 $k = 0 \rightarrow 42$   
 $l = -13 \rightarrow 13$   
 2 standard reflections  
 every 100 reflections  
 intensity decay: 2.8%

### Refinement

Refinement on  $F^2$   
 $R = 0.050$   
 $wR = 0.066$   
 $S = 3.385$   
 3722 reflections  
 539 parameters  
 H atoms: see below  
 $w = 1/[\sigma^2(F_o^2)]$

Extinction correction:  
 SHELXL93  
 (Sheldrick, 1993)  
 Extinction coefficient:  
 0.00468 (6)  
 Scattering factors from  
 International Tables for  
 Crystallography (Vol. C)

$$(\Delta/\sigma)_{\max} = 0.095$$

$$\Delta\rho_{\max} = 0.408 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.400 \text{ e } \text{\AA}^{-3}$$

Absolute structure: assigned  
according to Shubina *et al.* (1990)

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Table 1. Selected geometric parameters (Å, °)

C2—C3	1.537 (3)	C2'—C3'	1.502 (3)
C9—C10	1.584 (2)	C9'—C10'	1.574 (2)
C9—C15	1.573 (3)	C9'—C15'	1.580 (3)
C18—C17—C16	123.9 (2)	C18'—C17'—C16'	124.7 (2)
C18—C17—O17	117.6 (2)	C18'—C17'—O17'	119.1 (2)
C16—C17—O17	118.5 (2)	C16'—C17'—O17'	116.0 (2)
C24—O20—C20	119.1 (1)	C24'—O20'—C20'	116.3 (1)
C8—C9—C15—C16	56.0 (2)		
C9—C15—C16—C17	−99.8 (2)		
C18—C17—O17—C22	73.4 (2)		
C16—C17—O17—C22	−109.5 (2)		
C21—C20—O20—C24	128.3 (2)		
C19—C20—O20—C24	−54.3 (3)		
C8'—C9'—C15'—C16'	59.8 (2)		
C9'—C15'—C16'—C17'	−95.9 (2)		
C18'—C17'—O17'—C22'	61.2 (3)		
C16'—C17'—O17'—C22'	−122.7 (2)		
C21'—C20'—O20'—C24'	116.0 (2)		
C19'—C20'—O20'—C24'	−67.4 (2)		

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

D—H...A	D—H	H...A	D...A	D—H...A
C8—H8A...O17	0.96	2.84	3.343 (2)	114
C13—H13A...O17	0.96	2.46	3.124 (3)	126
C15—H15B...O17	0.96	2.49	2.891 (2)	105
C15—H15B...O24'	0.96	2.77	3.318 (2)	117
C19—H19...O24	1.15 (2)	2.48 (2)	2.939 (3)	102 (1)
C8'—H8B...O17'	0.96	2.65	3.193 (3)	116
C13'—H13E...O17'	0.96	2.37	3.049 (3)	128
C15'—H15C...O17'	0.96	2.48	2.858 (2)	103
C18'—H18'...O24	1.07 (2)	2.34 (2)	3.380 (2)	163 (1)
C18'—H18'...O22'	1.07 (2)	2.81 (2)	2.952 (2)	86.7 (9)
C19'—H19'...O22	0.95 (2)	2.47 (2)	3.345 (3)	153 (1)
C19'—H19'...O24'	0.95 (2)	3.00 (2)	3.029 (3)	83 (1)
C18—H18...O24 <sup>i</sup>	0.90 (2)	2.52 (2)	3.353 (2)	155 (1)
C19—H19...O22 <sup>i</sup>	1.15 (2)	2.23 (2)	3.320 (3)	158 (2)

Symmetry code: (i)  $x - 1, y, z$ .

The H atoms were located on the basis of geometrical considerations and difference Fourier map suggestions (for the methyl groups). Atoms H18, H19, H18' and H19' were included in the refinement with isotropic displacement parameters. All other H atoms were included as fixed atoms, with isotropic displacement parameters set equal to  $1.2U_{\text{eq}}$  ( $1.5U_{\text{eq}}$  for methyl groups) of the parent atoms. The absolute configuration was assigned in accordance with that established previously by Shubina *et al.* (1990).

Data collection: *P2<sub>1</sub> Diffractometer Program* (Syntax, 1975). Cell refinement: *P2<sub>1</sub> Diffractometer Program*. Data reduction: *XDISK (SHELXTL/PC)*; Sheldrick, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *XP* in *SHELXTL/PC*. Software used to prepare material for publication: *CIFTAB* in *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AV1024). Services for accessing these data are described at the back of the journal.

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## 2-Benzyl-2-hydroxycyclohexanone

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## Abstract

In the solid state, the title compound (C<sub>13</sub>H<sub>16</sub>O<sub>2</sub>) exists as dimers related by a centre of inversion and linked by two equivalent hydrogen bonds O1...H2(−*x*, 2−*y*, 1−*z*) [2.021 (3) Å]. The carbonyl- and hydroxyl-group O atoms attached to the cyclohexyl ring are almost eclipsed [O2—C2—C1—O1 18.5 (3)°].

## Comment

Treatment of 2-hydroxycyclohexanone with sodium hydride and benzyl bromide in tetrahydrofuran afforded the highly crystalline title compound, (1), rather than