Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* ($\AA^2$)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Br	0.12507 (7)	0.09735 (14)	0.22300 (3)	0.0535 (2)
O(1)	0.0517 (4)	0.4842 (8)	0.3010 (2)	0.0503 (14)
O(2)	−0.3398 (4)	0.8589 (8)	0.0114 (2)	0.056 (2)
O(3)	−0.5606 (6)	0.3775 (10)	0.1275 (2)	0.077 (2)
O(4)	−0.6736 (5)	0.6782 (8)	0.0764 (2)	0.0519 (13)
N	−0.4553 (5)	0.8022 (9)	0.0366 (2)	0.0406 (13)
C(1)	−0.2189 (6)	0.4748 (11)	0.1373 (3)	0.037 (2)
C(2)	−0.2284 (6)	0.6501 (10)	0.1785 (3)	0.038 (2)
C(3)	−0.1408 (5)	0.6569 (10)	0.2332 (3)	0.034 (2)
C(4)	−0.0373 (6)	0.4903 (10)	0.2470 (3)	0.0306 (14)
C(5)	−0.0235 (5)	0.3150 (11)	0.2060 (3)	0.035 (2)
C(6)	−0.1133 (6)	0.3053 (11)	0.1517 (3)	0.036 (2)
C(7)	−0.3142 (6)	0.4515 (12)	0.0774 (3)	0.052 (2)
C(8)	−0.4383 (6)	0.6113 (13)	0.0677 (2)	0.041 (2)
C(9)	−0.5631 (7)	0.5409 (12)	0.0949 (3)	0.047 (2)
C(10)	−0.7993 (7)	0.6248 (16)	0.1004 (3)	0.071 (3)
C(11)	−0.9136 (8)	0.7703 (18)	0.0678 (4)	0.099 (4)

Table 2. *Selected geometric parameters* ($\AA$, °)

Br—C(5)	1.885 (6)	C(1)—C(7)	1.513 (7)
O(1)—C(4)	1.375 (6)	C(2)—C(3)	1.381 (7)
O(2)—N	1.384 (6)	C(3)—C(4)	1.374 (7)
O(3)—C(9)	1.184 (7)	C(4)—C(5)	1.384 (7)
O(4)—C(9)	1.332 (7)	C(5)—C(6)	1.383 (7)
O(4)—C(10)	1.454 (7)	C(7)—C(8)	1.492 (8)
N—C(8)	1.286 (7)	C(8)—C(9)	1.509 (9)
C(1)—C(2)	1.379 (7)	C(10)—C(11)	1.474 (9)
C(1)—C(6)	1.401 (7)		
C(9)—O(4)—C(10)	116.3 (6)	C(6)—C(5)—Br	119.9 (5)
C(8)—N—O(2)	112.8 (5)	C(4)—C(5)—Br	119.4 (4)
C(2)—C(1)—C(6)	117.8 (5)	C(5)—C(6)—C(1)	120.2 (6)
C(2)—C(1)—C(7)	124.9 (6)	C(8)—C(7)—C(1)	115.5 (5)
C(6)—C(1)—C(7)	117.3 (6)	N—C(8)—C(7)	127.8 (6)
C(1)—C(2)—C(3)	122.0 (6)	N—C(8)—C(9)	114.4 (6)
C(4)—C(3)—C(2)	119.8 (6)	C(7)—C(8)—C(9)	117.8 (6)
C(3)—C(4)—O(1)	122.8 (5)	O(3)—C(9)—O(4)	125.6 (7)
C(3)—C(4)—C(5)	119.5 (5)	O(3)—C(9)—C(8)	122.3 (7)
O(1)—C(4)—C(5)	117.7 (5)	O(4)—C(9)—C(8)	112.1 (6)
C(6)—C(5)—C(4)	120.6 (5)	O(4)—C(10)—C(11)	107.7 (6)

The structure was solved by Patterson methods and refined successfully in the monoclinic space group $P2_1/n$. Full-matrix least-squares refinement was carried out by minimizing $w(F_o^2 - F_c^2)^2$. The non-H atoms were refined anisotropically to convergence. Hydroxy H atoms were refined isotropically whereas the remaining H atoms were refined using an appropriate riding model.

Data collection: *P3/PC Diffractometer Program* (Siemens, 1989a). Cell refinement: *P3/PC Diffractometer Program*. Data reduction: *XDISK* (Siemens, 1989b). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1994). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXTL-Plus*.

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1,3-Diphenylpropan-1-one

Nenad Judaš, Branko Kaitner* and Ernest Meštrović

Chemistry Department, Laboratory of General and Inorganic Chemistry, Faculty of Science, University of Zagreb, ul. Kralja Zvonimira 8, 41000 Zagreb, Croatia

Abstract

The stereochemistry of the title compound, $C_{15}H_{14}O$, is similar to that of the analogous compounds 2-phenylacetophenone and substituted and non-substituted phenyl benzoate, although the middle part of the title molecule contains an additional methylene group. The phenyl rings are twisted with respect to both each

other and the central propanone moiety. The molecular interactions are based on weak van der Waals attractions only.

Comment

Small molecules containing two phenyl rings integrated by —C(=O)—O— or —C(=O)—CH₂— groups [phenyl benzoate (PB) and 2-phenylaceto-phenone (PAP)] are interesting simple systems characterized by specific orientation of the terminal rings (Adams & Morsi, 1976; Rieker et al., 1993). Structural investigations carried out on fluoro- and chloro-substituted PB showed that the position and type of the polar substituent could control the molecular arrangement (Shibakami & Sekiya, 1995). On the other hand, the PAP systems can be correlated with typical thermotropic liquid-crystal molecules composed of a rigid central core with two flexible aliphatic chains attached at either end. In an attempt to prepare 3-benzyl-1,3-diphenylpropane-1,3-dione, the title compound, 1,3-diphenylpropanone, DPP, was obtained as the main cleavage product (Abell, 1912).

O
‖
DPP
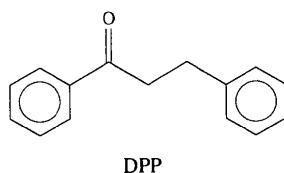
The central part of the title molecule is extended by a methylene group, resulting in one extra atom in the chain compared to either PB or PAP. On this basis, we accounted for the possible structural influence of an additional CH_2 group on the stereochemistry of the molecule. The molecule can be described in terms of three planes. Atoms O1, C1, C2 and C3 are coplanar within estimated error. The dihedral angle between the two phenyl ring planes is $67.0(1)°$; corresponding angles in PAP and PB are 115.8 and $123.8(3)°$, respectively. It seems that it is not only the F or Cl pendants that 'steer' the molecular structure; the effect of an additional methylene group in the central moiety on the stereochemistry of the molecule is significant, as can be seen from the values of other interplanar angles. The dihedral angles between the central plane (O1, C1, C2 and C3) and the planes defined by the C4–C9 and C10–C15 phenyl ring atoms are $112.3(2)$ and $2.7(1)°$, respectively. The latter value is somewhat smaller than those found in PAP [$8.2(4)°$] and PB [$9.9(2)°$]. This discrepancy is probably caused by different crystal packing which deforms the molecule, resulting in an intramolecular C11—H···O1 close contact [C11···O1 $2.769(3)$ and H11···O1 2.472 Å]. Certain similarities are observed between other dihedral angles in DPP, PAP and both substituted and unsubstituted PB. The

valence bond lengths in the central C_{ar}—C_{sp^3}—C_{sp^3}—C_{sp^2}(=O)—C_{ar} part of the molecule, as well as the average C_{ar}—C_{ar} bond distance [$1.377(4)$ Å], agree very well with those published elsewhere (Allen et al., 1987). Distances between discrete molecules of the order of very weak van der Waals attractions are observed: C14—H···O1($\frac{1}{2}+x$, $\frac{1}{2}-y$, $-z$) 3.443 Å (Steiner & Saenger, 1993). There are no other intermolecular contacts less than 3.5 Å involving non-H atoms.
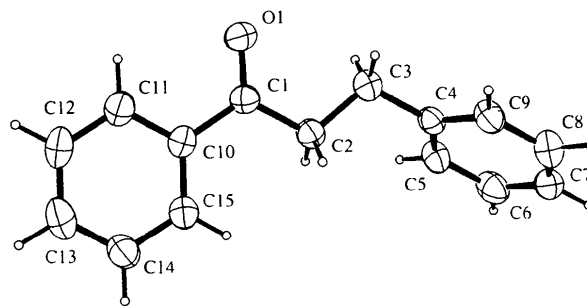

Fig. 1. An ORTEPII (Johnson, 1976) drawing of the title molecule illustrating the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level.

Experimental

Crystals of 1,3-diphenylpropan-1-one suitable for X-ray analysis were prepared by slow evaporation from an ether solution. An IR spectrum recorded on a Perkin-Elmer 1600 FTIR spectrometer and a sharp melting point at 325 K confirmed the identity of the substance. The density D_m was measured by flotation.

Crystal data

$C_{15}H_{14}O$	Mo $K\alpha$ radiation
$M_r = 210.27$	$\lambda = 0.71073$ Å
Orthorhombic	Cell parameters from 22
$Pbca$	reflections
$a = 8.854(4)$ Å	$\theta = 8.5–12.5°$
$b = 9.832(4)$ Å	$\mu = 0.08$ mm^{-1}
$c = 27.110(8)$ Å	$T = 293$ K
$V = 2360(2)$ Å^3	Irregular block
$Z = 8$	$0.70 \times 0.54 \times 0.47$ mm
$D_x = 1.184$ Mg m^{-3}	Pale yellow
$D_m = 1.181$ Mg m^{-3}	

Data collection

Phillips PW1100 diffractom-eter	$R_{int} = 0.002$
	$\theta_{max} = 27.0°$
ω scans	$h = 0 \rightarrow 11$
Absorption correction:	$k = 0 \rightarrow 12$
none	$l = 0 \rightarrow 34$
2903 measured reflections	3 standard reflections
2073 independent reflections	frequency: 90 min
975 observed reflections	intensity decay: 3.5%
[$I > \sigma(I)$]	

Refinement

Refinement on F	$w = 1/[\sigma^2(F) + 0.0020F^2]$
$R = 0.037$	$(\Delta/\sigma)_{max} = 0.003$
$wR = 0.061$	$\Delta\rho_{max} = 0.18$ e Å^{-3}
$S = 1.21$	$\Delta\rho_{min} = -0.09$ e Å^{-3}
975 reflections	Atomic scattering factors
145 parameters	from *International Tables*
H atoms riding on parent	*for X-ray Crystallography*
atoms, $U_{iso}(H_i) = 1.2 \times$	(1974, Vol. IV, Table
$U_{eq}(C_i)$	2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.7695 (3)	0.0298 (2)	0.05955 (7)	0.082 (1)
C1	0.8479 (3)	0.1223 (3)	0.04401 (9)	0.056 (1)
C2	0.9239 (3)	0.2185 (3)	0.07865 (8)	0.059 (1)
C3	0.8936 (3)	0.1893 (3)	0.13258 (9)	0.067 (2)
C4	0.9708 (3)	0.2871 (3)	0.16671 (8)	0.055 (1)
C5	0.9276 (3)	0.4219 (3)	0.16872 (9)	0.068 (2)
C6	0.9990 (4)	0.5118 (3)	0.2004 (1)	0.080 (2)
C7	1.1136 (4)	0.4665 (3)	0.2307 (1)	0.080 (2)
C8	1.1571 (4)	0.3342 (4)	0.2290 (1)	0.084 (2)
C9	1.0876 (4)	0.2449 (3)	0.19712 (9)	0.068 (2)
C10	0.8674 (3)	0.1398 (3)	−0.01035 (8)	0.056 (1)
C11	0.7906 (3)	0.0538 (3)	−0.0420 (1)	0.070 (2)
C12	0.8039 (4)	0.0707 (4)	−0.0926 (1)	0.081 (2)
C13	0.8921 (4)	0.1713 (4)	−0.1116 (1)	0.086 (2)
C14	0.9679 (4)	0.2571 (3)	−0.0808 (1)	0.077 (2)
C15	0.9555 (3)	0.2412 (3)	−0.02996 (9)	0.067 (2)

Table 2. *Selected geometric parameters* ($\text{Å}, \degree$)

O1—C1	1.219 (3)	C2—C3	1.514 (3)
C1—C2	1.493 (4)	C3—C4	1.499 (4)
C1—C10	1.494 (3)	C_{ar}—C_{ar} (mean)	1.377 (4)
O1—C1—C2	120.8 (2)	C3—C4—C5	120.9 (2)
O1—C1—C10	119.5 (2)	C3—C4—C9	121.0 (2)
C2—C1—C10	119.7 (2)	C1—C10—C11	119.0 (2)
C1—C2—C3	114.0 (2)	C1—C10—C15	122.1 (2)
C2—C3—C4	113.2 (2)	C_{ar}—C_{ar}—C_{ar} (mean)	120.0 (3)

Data collection: *DIF4* (Stoe & Cie, 1992*a*). Cell refinement: *DIF4*. Data reduction: *REDU4* (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *NRCVAX SOLVER* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *NRCVAX TABLES*.

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2β,3β-Epoxy-α-*trans*-himachalene and 2β,3β,11α,15α-Diepoxy-*trans*-himachalane Derivatives

A. Benharref, H. El Jamili and E. Lassaba

Laboratoire des Substances Naturelles et des Hétérocycles, Faculté des Sciences, Université Cadi Ayyad, BP S15, Semlia-Marrakech, Morocco

M. Giorgi and M. Pierrot

LBS-URA 1409, Centre Scientifique Saint Jerome, 13397 Marseille, France

Abstract

The stereochemistry of the major isomers resulting from the double epoxidation of α-*trans*-himachalene has been established. The title compounds, decahydro-2,9,9-trimethyl-5-methylene-2,3-epoxy-1*H*-benzocyclo-heptene, $C_{15}H_{24}O$, and 2,9,9-trimethylspiro(decahydro-2,3-epoxy-1*H*-benzocycloheptene-5-oxirane), $C_{15}H_{24}O_2$, are isostructural, and in both compounds the seven-membered ring adopts a chair conformation while the six-membered ring conformation is closer to an envelope than a half-chair.

Comment

The preparation of dichlorohimachalane from *cis*-himachalenes, the main constituents of *Atlas cedar* (*cedrus atlantica*) essential oils is described by several authors (Joseph & Dev, 1968*a*; Narula & Dev, 1977; Nambudiry & Rao, 1974). The dehydrogenation of dihydromachalane leads to the *trans*-himachalenes and particularly to an α-*trans*-himachalene (1) (Joseph