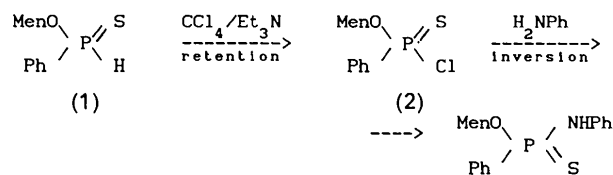


ratio test (Hamilton, 1965) gave theoretical $\mathcal{R}_{1,3677,0.005} = 1.00108$ while the value obtained for this structure was $\mathcal{R} = wR(+)/wR(-) = 1.0654$. Hence, because $\mathcal{R} > \mathcal{R}_{1,3677,0.005}$, the probability that the opposite enantiomer is correct can be accepted at the 99.5% confidence level. The final full-matrix least-squares refinement of the correct enantiomer ($-x, -y, -z$) gave $R = 0.0690$ and $wR = 0.0644$ for 3860 observed reflections [$F_o > 3\sigma(F_o)$]. Function minimized was $\sum w(|F_o| - |F_c|)^2$. The extinction parameter, x , refined to $1.3(4) \times 10^{-7}$ where $F_{\text{corr}} = F(1 - xF^2/\sin\theta)$. Final $(\Delta/\sigma)_{\text{max}}$ was 0.001 and residual electron density $-0.45 < \Delta\rho < 0.71 \text{ e } \text{\AA}^{-3}$, the maximum was near to the P atom. Calculations were carried out using the *SHELXS86* (Sheldrick, 1986) and *SHELXL76* (Sheldrick, 1976) systems. Final atomic parameters for the correct enantiomer are given in Table 1.* Bond lengths and angles are listed in Table 2. The conformation and configuration of the molecule, and atomic numbering are shown in Fig. 1. The absolute configuration of the P atom is *S* while the menthyl group has the configuration 1*R*,2*S*,5*R*, which is consistent with that established for (–)-menthol (Ohrt & Parthasarathy, 1975). The intermolecular N—H...S hydrogen bond [N...S = 3.393(4), H...S = 2.43 Å, $\angle\text{N—H...S} = 176^\circ$] links the molecules to form a chain parallel to the shortest of the crystallographic axes, *a*.

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54511 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The title compound is a product of stereospecific reactions investigated as part of the stereochemical studies on thiophosphonates (Łuczak, Łopusiński & Michalski, 1991).



Knowledge of the absolute configuration of the chiral P atom of *O*-menthoxy-*N*-anilinothiophosphonate enables the determination of the configurations of its precursors (1) and (2).

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References

- HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.
 JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 ŁUCZAK, L., ŁOPUSIŃSKI, A. & MICHALSKI, J. (1991). In preparation.
 OHRT, J. M. & PARTHASARATHY, R. (1975). *J. Cryst. Mol. Struct.* **5**, 359–376.
 ROGERS, D. (1981). *Acta Cryst.* **A37**, 734–741.
 SHELDRIK, G. M. (1976). *SHELXL76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1986). *SHELXS86*. *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRUGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

Acta Cryst. (1992). **C48**, 400–402

Structure of 4,4-Diphenylcyclohex-2-en-1-one (Monoclinic Form)

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Abstract. C₁₈H₁₆O, $M_r = 248.33$, monoclinic, $P2_1/c$, $a = 7.139(6)$, $b = 15.823(2)$, $c = 12.472(5)$ Å, $\beta = 103.12(9)^\circ$, $V = 1372(1)$ Å³, $Z = 4$, $D_x = 1.202 \text{ g cm}^{-3}$, $\text{Mo } K\alpha_1$, $\lambda = 0.70930$ Å, $\mu = 0.7 \text{ cm}^{-1}$, and $\text{Cu } K\alpha_1$, $\lambda = 1.54056$ Å, $\mu = 5.3 \text{ cm}^{-1}$, $F(000) = 528$, $T = 295 \text{ K}$, $R = 0.052$ for 1245 reflections (Mo), 0.070 for 2164 reflections (Cu). The structure is disordered with two

(equivalent) molecular conformations occupying the same site (50:50). The cyclohexene ring has a C(5)-sofa conformation.

Experimental. Crystals from ethanol, colourless prisms, $0.25 \times 0.25 \times 0.4$ mm. Nonius CAD4-F diffractometer, monochromatized Mo $K\alpha$ and Cu $K\alpha$ radiations were used. Solution of the structure with a

Table 1. Positional (fractional $\times 10^4$) and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$) (Cu data)

	$U_{eq} = \frac{1}{3} \times (\text{trace of diagonalized } U \text{ tensor}).$			
	x	y	z	U_{eq}
O(1)	10581 (3)	910 (2)	4335 (2)	118
C(1)	9005 (3)	704 (2)	3802 (2)	75
C(2)*	7357 (17)	1051 (6)	4039 (8)	81
C(2)*	7161 (14)	721 (6)	4240 (8)	69
C(3)	5520 (4)	516 (2)	3654 (2)	62
C(4)	5158 (3)	270 (1)	2440 (1)	50
C(5)	7036 (3)	-102 (2)	2243 (2)	62
C(6)*	8762 (12)	93 (5)	2865 (7)	74
C(6)*	8759 (11)	480 (5)	2620 (8)	76
C(7)	4510 (3)	1049 (1)	1728 (2)	54
C(8)	5204 (4)	1220 (2)	790 (2)	74
C(9)	4571 (5)	1905 (2)	129 (2)	91
C(10)	3259 (5)	2445 (2)	374 (3)	95
C(11)	2522 (4)	2290 (2)	1286 (3)	91
C(12)	3141 (4)	1596 (2)	1964 (2)	69
C(13)	3629 (3)	-435 (1)	2172 (2)	51
C(14)	3690 (4)	-1108 (1)	2884 (2)	75
C(15)	2400 (5)	-1770 (2)	2648 (2)	90
C(16)	1019 (4)	-1779 (2)	1681 (2)	80
C(17)	942 (3)	-1121 (2)	954 (2)	72
C(18)	2230 (3)	-453 (1)	1199 (2)	62

* Occupancy = 0.5.

data set collected with Mo radiation (maximum $\theta = 21^\circ$, maximum $\sin\theta/\lambda = 0.50 \text{ \AA}^{-1}$) revealed large displacement parameters for some atoms, probably resulting from disorder. It seemed advantageous to attempt to resolve the disordered atoms by collecting a second data set to higher resolution; since the high-angle data with Mo radiation were quite weak, Cu radiation was used for the second data set (maximum $\theta = 65^\circ$, maximum $\sin\theta/\lambda = 0.59 \text{ \AA}^{-1}$). Lattice parameters from 25 reflections with $\theta = 26\text{--}48^\circ$ (Cu). Intensities for $\theta \leq 21^\circ$ (Mo) [$\leq 65^\circ$ (Cu)], hkl : -7 to 7, 0 to 15, 0 to 12 [-8 to 8, 0 to 19, 0 to 15], ω -2 θ scan, scan width ($a + b \tan\theta$) $^\circ$ ($a = 1.10$ [1.20], $b = 0.35$ [0.14]) at 2.2-10 [1.8-6.7] $^\circ \text{ min}^{-1}$, extended 25% on each side for background measurement, three standard reflections showed no significant variation, Lp but no absorption corrections. 1430 (Mo) and 2481 (Cu) unique reflections measured, 1245 (Mo) and 2164 (Cu) with $F > 3\sigma(F)$. Structure by direct methods, refined by full-matrix least-squares procedures. Refinement with the Mo data resulted in large thermal parameters and unusual molecular dimensions for C(2) and C(6); the analysis was repeated with new Cu data, and these atoms were each resolved into two disordered sites. Refinement on F , with $w = 1/\sigma^2(F)$, where $\sigma^2(I) = S + 4(B_1 + B_2) + (0.04I)^2$, $S = \text{scan}$, B_1 and $B_2 = \text{background counts}$, locally written programs and *SHELX76* (Sheldrick, 1976); anisotropic thermal parameters for non-H atoms, H atoms from difference maps, refined with isotropic thermal parameters. Mo data: final $R = 0.052$, $wR = 0.047$ for 1245 reflections with $F > 3\sigma(F)$, $R = 0.061$ for all 1430 reflections [with single sites for C(2) and C(6); split-atom sites are unlikely

Table 2. Bond lengths (\AA) and angles ($^\circ$) (Cu data)

C(1)—O(1)	1.214 (3)	C(9)—C(10)	1.352 (5)
C(1)—C(2)	1.389 (13)	C(10)—C(11)	1.380 (6)
C(1)—C(2')	1.535 (11)	C(11)—C(12)	1.395 (4)
C(2)—C(3)	1.543 (11)	C(7)—C(12)	1.387 (4)
C(2')—C(3)	1.274 (9)	C(4)—C(13)	1.544 (3)
C(3)—C(4)	1.528 (3)	C(13)—C(14)	1.381 (3)
C(4)—C(5)	1.533 (3)	C(14)—C(15)	1.382 (4)
C(5)—C(6)	1.334 (8)	C(15)—C(16)	1.374 (4)
C(5)—C(6')	1.523 (8)	C(16)—C(17)	1.373 (4)
C(1)—C(6)	1.497 (9)	C(17)—C(18)	1.389 (3)
C(1)—C(6')	1.487 (11)	C(13)—C(18)	1.386 (3)
C(4)—C(7)	1.528 (3)	*C(2)—C(2')	0.608 (15)
C(7)—C(8)	1.397 (4)	*C(6)—C(6')	0.685 (12)
C(8)—C(9)	1.375 (4)		
C(2)—C(1)—O(1)	120.2 (5)	C(6')—C(5)—C(4)	113.1 (4)
C(2')—C(1)—O(1)	124.3 (4)	C(5)—C(6)—C(1)	122.0 (6)
C(6)—C(1)—O(1)	121.9 (4)	C(5)—C(6')—C(1)	110.8 (6)
C(6')—C(1)—O(1)	119.5 (4)	C(8)—C(7)—C(4)	121.7 (2)
C(6)—C(1)—C(2)	117.9 (6)	C(12)—C(7)—C(4)	121.1 (2)
C(6')—C(1)—C(2)	109.4 (5)	C(12)—C(7)—C(8)	117.2 (2)
C(6)—C(1)—C(2')	112.9 (6)	C(9)—C(8)—C(7)	121.6 (3)
C(6')—C(1)—C(2')	116.0 (5)	C(10)—C(9)—C(8)	120.9 (3)
C(3)—C(2)—C(1)	114.7 (7)	C(11)—C(10)—C(9)	119.2 (3)
C(3)—C(2)—C(1')	122.9 (7)	C(12)—C(11)—C(10)	120.8 (3)
C(4)—C(3)—C(2)	112.9 (4)	C(11)—C(12)—C(7)	120.3 (3)
C(4)—C(3)—C(2')	124.1 (5)	C(14)—C(13)—C(4)	120.0 (2)
C(7)—C(4)—C(3)	109.3 (2)	C(18)—C(13)—C(4)	122.6 (2)
C(13)—C(4)—C(3)	110.5 (2)	C(18)—C(13)—C(14)	117.3 (2)
C(13)—C(4)—C(7)	110.6 (2)	C(15)—C(14)—C(13)	121.7 (2)
C(5)—C(4)—C(3)	107.4 (2)	C(16)—C(15)—C(14)	120.4 (3)
C(7)—C(4)—C(5)	112.1 (2)	C(17)—C(16)—C(15)	119.1 (3)
C(13)—C(4)—C(5)	106.8 (2)	C(18)—C(17)—C(16)	120.4 (2)
C(6)—C(5)—C(4)	123.0 (4)	C(17)—C(18)—C(13)	121.2 (2)

* Separation between disordered atoms.

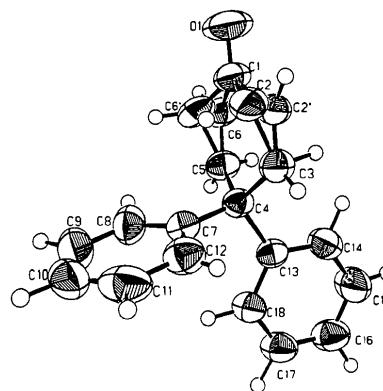


Fig. 1. View of the diphenylcyclohexenone molecule, showing the disorder.

to reduce these R factors (Nowell, Rettig & Trotter, 1972)]; Cu data: final $R = 0.070$, $wR = 0.073$ for 2164 reflections with $F > 3\sigma(F)$, $R = 0.076$ for all 2481 reflections [split-atom model; high values of R are a result of difficulty in modelling the disordered structure; atoms other than C(2) and C(6), particularly C(1), undoubtedly also have slightly split positions], maximum $\Delta/\sigma = 0.07$ [0.23], $\Delta\rho = -0.18$ to $+0.18$ [-0.28 to $+0.20$] e \AA^{-3} . Atomic scattering factors were those from *SHELX76*. Positional parameters

are in Table 1, bond lengths and angles in Table 2 and a view of the molecule is in Fig. 1.*

Related literature. The structure has been determined as part of a study of the photochemistry of cyclohexenones (Zimmerman, Rieke & Scheffer, 1967; Schuster, 1980; Chen, Hwang, Scheffer & Trotter, 1991). An orthorhombic form is obtained from hexane solvent (Zimmerman & Zuraw, 1989).

* Lists of positional parameters, bond lengths and angles, a stereoview of the molecule (Mo data), anisotropic thermal parameters, H-atom positions, torsion angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54502 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0362]

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References

- CHEN, J., HWANG, C., SCHEFFER, J. R. & TROTTER, J. (1991). *Acta Cryst.* **C47**, 2417–2419.
 NOWELL, I. W., RETTIG, S. & TROTTER, J. (1972). *J. Chem. Soc. Dalton Trans.* pp. 2381–2388.
 SCHUSTER, D. I. (1980). *Photochemical Rearrangements of Enones, Rearrangements in Ground and Excited States*, Vol. 3, edited by P. DE MAYO, pp. 187–193. New York: Academic Press.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 ZIMMERMAN, H. E., RIEKE, R. D. & SCHEFFER, J. R. (1967). *J. Am. Chem. Soc.* **89**, 2033–2047.
 ZIMMERMAN, H. E. & ZURAW, M. J. (1989). *J. Am. Chem. Soc.* **111**, 7974–7989.

Structure of [(1*S*)-1-[(1'*S*)-3'-Oxocyclohexyl]-3,3-dimethoxy-1-propyl] [(1*S*,2*S*,3*R*)-1,3,7,7-Tetramethyl-2-hydroxy-3-bicyclo[2.2.1]heptyl] Sulfone

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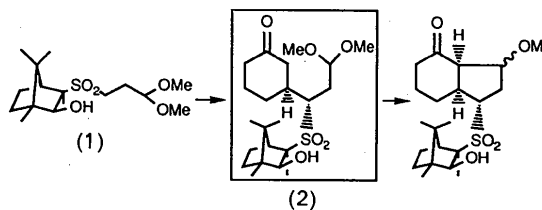
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(Received 10 April 1991; accepted 2 August 1991)

Abstract. $C_{22}H_{38}O_6S$, $M_r = 430.60$, monoclinic, $P2_1$, $a = 6.922$ (2), $b = 16.733$ (2), $c = 10.070$ (3) Å, $\beta = 96.88$ (2)°, $V = 1158.0$ (4) Å³, $Z = 2$, $D_x = 1.24$ g cm⁻³, $Cu K\alpha$, $\lambda = 1.54178$ Å, $\mu = 14.9$ cm⁻¹, $F(000) = 468$, $T = 291$ K, $R = 0.050$ for 3558 observed reflections. The development of methods for the construction of five-membered carbocycles is a topic of current interest as a result of the ubiquitous occurrence of such rings in many biologically active products. We have recently developed [De Lombaert, Nemery, Roekens, Carretero, Kimmel & Ghosez (1986). *Tetrahedron Lett.* **27**, 5099–5102] a novel two-step (3 + 2) annulation yielding highly functionalized fused cyclopentanones. We are now developing an enantioselective version of this sequence. In this context we have studied the asymmetric Michael addition of a chiral homoenolate to cyclohexenone from which the title compound was obtained in an optically pure crystalline form. The

present X-ray crystallographic study establishes unambiguously the absolute stereochemistry of the title compound and the precursor homoenolate.

Experimental. Title compound prepared as shown below.



D_m not measured. Parallelepiped crystal with dimensions $0.35 \times 0.4 \times 0.2$ mm. Lattice parameters refined using 18 reflections in the range $10 \leq 2\theta \leq 49^\circ$. Huber four-circle diffractometer and Rigaku rotating