

In common with other septanoside derivatives that have been subjected to X-ray diffraction [see Choong, McConnell, Stephenson & Stevens (1980) for references], bond angles within the seven-membered ring are all greater than the tetrahedral angle. This is consistent with the results of a theoretical study on the 'parent' heterocycle, oxepane (Bocian & Strauss, 1977).

As with other polyhydroxy compounds (Jeffrey, 1973), there is extensive intermolecular hydrogen bonding involving the hydroxyl groups in the title compound (Table 3). All hydroxyl groups are both donors and acceptors. A symmetrical bifurcated hydrogen bond is formed between O(1), O(2) and H(O5) related translationally in the $-\mathbf{b}$ direction. This bond and the O(2)—H(O2)···O(5ⁱ) bond are responsible for the formation of zigzag chains parallel to \mathbf{b} . Each molecule is hydrogen bonded to six neighbouring molecules: four in the same chain, and two in a chain lying adjacent in the $-\mathbf{a}$ direction. The C(2) hydroxyl group is involved in bonding with three different molecules, accepting from O(3) in an adjacent chain and being both donor and acceptor to molecules in the same chain. This group and those attached to C(3) and C(4) interbond adjacent chains to form 'puckered' sheets lying parallel to the ab plane. All $D\cdots A$ distances are greater than 2.67 Å and are categorized as 'weak'

Table 3. Hydrogen-bond distances (Å) and angles ($^{\circ}$) with *e.s.d.*'s in parentheses

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O(2) H(O2) O(5 ⁱ)	0.87 (4)	1.87 (4)	2.724 (2)	168 (4)
O(3) H(O3) O(4)	0.81 (5)	2.22 (4)	2.673 (2)	116 (4)
O(3) H(O3) O(2 ⁱⁱ)	0.81 (5)	2.09 (4)	2.844 (2)	153 (4)
O(4) H(O4) O(3 ⁱⁱ)	0.91 (4)	1.90 (4)	2.795 (2)	167 (4)
O(5) H(O5) O(1 ⁱⁱⁱ)	0.74 (5)	2.28 (5)	2.913 (2)	143 (4)
O(5) H(O5) O(2 ⁱⁱⁱ)	0.74 (5)	2.30 (5)	2.913 (2)	140 (4)

The superscripts refer to atoms related to those in Table 1 by the operations: (i) $1-x, -\frac{1}{2}+y, 1-z$; (ii) $-x, \frac{1}{2}+y, 1-z$; (iii) $x, 1+y, z$.

(Brown, 1976), although the cooperative aspect of the bonding and the formation of chains of bonds implies a stable hydrogen-bonding scheme (Jeffrey & Takagi, 1978). The non-hydroxylic O(6) is not involved in hydrogen bonding. The hydroxylic H(O3) is involved in two interactions, one of which is a weak intramolecular hydrogen bond. Attention has been drawn elsewhere (Newton, Jeffrey & Takagi, 1979) to weak hydrogen bonds being part of a bifurcated hydrogen-bond system.

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Acta Cryst. (1983). **C39**, 612–614

The Structure of 2-(2-Hydroxy-5-methylphenyl)-1-propenyl Acetate, $C_{12}H_{14}O_3$ *

BY N. N. DHANESHWAR AND S. S. TAVALE

National Chemical Laboratory, Pune 411008, India

(Received 25 October 1982; accepted 12 January 1983)

Abstract. $M_r = 206.2$, monoclinic, $P2_1/c$, $a = 6.52$ (1), $b = 24.95$ (3), $c = 7.23$ (1) Å, $\beta = 112.0$ (1) $^{\circ}$, $V = 1090$ Å³, $D_x = 1.257$ Mg m⁻³ for $Z = 4$, $F(000) = 440$. Final R is 0.09 for 953 observed reflections. The structure was solved by direct methods. Two molecules

combine to form a dimer through O—H···O hydrogen bonds.

Introduction. Enol esters are naturally occurring compounds (Bohlmann, Niedballa & Schulz, 1969). The synthesis of related enol esters was undertaken in this laboratory by Dhekne & Rao (1980a,b). They

* NCL communication No. 3126.

showed that oxiranes on thermal rearrangement furnish enol esters. The formula of 2-(2-hydroxy-5-methylphenyl)-1-propenyl acetate is shown in Fig. 1. The X-ray crystal structure analysis of this compound was undertaken to establish the stereochemistry at the ethylenic linkage based on NMR data. While this compound exists as a solid at room temperature, its diacetate is a liquid which suggests the possibility of hydrogen bonding in the title compound.

Experimental. Needle-shaped crystal 0.6×0.8 mm cross-section, three-dimensional intensity data ($0kl-5kl$) for 953 observed reflections (2971 unique, 1190 unobserved), unfiltered Cu radiation, multiple-film equi-inclination Weissenberg technique; intensities, estimated visually, were corrected for spot extension; layerwise scaling of the intensities by Wilson's method; cell dimensions from zero-layer Weissenberg photographs with crystals mounted along the three axes; film mounted in the Straumanis arrangement; systematic absences $0k0$ for k odd and $h0l$ for l odd; direct methods [*MULTAN*71 (Germain, Main & Woolfson, 1971)], isotropic and then anisotropic full-matrix least-squares refinement, function minimized $\sum w|F_o - |F_c||^2$; aromatic H atoms were fixed by stereochemical considerations while remaining H atoms were located by difference syntheses; the positional parameters of the H atoms were refined by isotropic block-diagonal least-squares refinement and then held constant during the full-matrix refinement of the C and O atoms; Cruickshank's weighting scheme was used with $a = 4.0$ and $c = 0.012$; ratio of maximum least-squares shift to error in the final refinement is 0.1 while the average ratio is 0.024; $R = 0.09$; least-squares refinement by program *LALS* (Gantzel, Sparks & Trueblood, 1961), bond lengths, angles and intermolecular distances by *NRC-12* (Pippy & Ahmed, 1978), least-squares plane by *NRC-22* (Pippy & Ahmed, 1978); atomic scattering factors from *International Tables for X-ray Crystallography*, 1962.*

Discussion. Table 1 gives atomic coordinates and B_{eq} values, Table 2 bond lengths and angles.

Fig. 2 shows the molecular packing in the (100) projection. In the crystal lattice two molecules combine to form a dimer through O—H...O hydrogen bonds around a centre of symmetry. This dimer formation seems to be responsible for the compound existing as a solid at room temperature. The hydrogen bond is linear.

The ethylenic linkage has rotated around the C'(1)—C(2) bond in such a way that the bulky methyl group is

* Lists of structure factors, anisotropic thermal parameters, H atom coordinates and least-squares-plane data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38330 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

away from the hydroxy group of the phenyl ring thus confirming the NMR data and chemical considerations.

The phenolic ring can be compared with *p*-cresol (Bois, 1970). Some similarity is seen in the bond lengths. The side chain may be compared with tiglic acid (Porte & Robertson, 1959). A parallel trend in bond lengths is observed in both cases.

We are grateful to Dr A. S. Rao for suggesting this problem, supplying the crystals and for useful discussions.

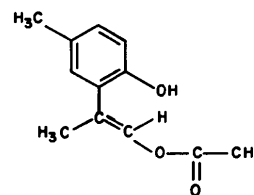


Fig. 1. Molecular formula.

Table 1. Fractional atomic coordinates ($\times 10^4$) with their standard deviations in parentheses and equivalent values of the anisotropic temperature-factor coefficients ($\text{\AA}^2 \times 10^2$)

$$b_{eq} = \frac{1}{3}(b_{11} + b_{22} + b_{33} + b_{13})$$

	x	y	z	b_{eq}
C(1)	755 (9)	4464 (2)	7435 (9)	2.3
C(2)	692 (10)	3957 (2)	6766 (9)	2.4
C(3)	-1383 (11)	3754 (3)	5215 (12)	2.9
C(4)	-1058 (11)	5284 (2)	7484 (10)	2.7
C(5)	-3157 (11)	5542 (2)	6754 (11)	3.1
O(1)	-1123 (6)	4775 (2)	6740 (7)	2.7
O(2)	638 (8)	5467 (2)	8598 (8)	3.2
C'(1)	2546 (10)	3598 (2)	7532 (9)	2.4
C'(2)	4715 (10)	3752 (2)	8514 (10)	2.7
C'(3)	6391 (10)	3384 (3)	9145 (11)	2.7
C'(4)	5936 (12)	2838 (3)	8896 (12)	3.2
C'(5)	3816 (12)	2667 (2)	7978 (10)	3.4
C'(6)	2150 (11)	3034 (2)	7323 (9)	2.9
C'(7)	3312 (13)	2065 (3)	7744 (11)	4.3
O'(1)	5196 (7)	4294 (2)	8801 (8)	3.2

Table 2. Intramolecular bond lengths (\AA) and angles ($^\circ$) and their standard deviations (in parentheses)

C'(1)—C'(2)	1.378 (10)	C'(1)—C'(6)	1.428 (7)
C'(1)—C(2)	1.438 (9)	C'(2)—C'(3)	1.368 (10)
C'(2)—O'(1)	1.386 (7)	C'(3)—C'(4)	1.391 (11)
C'(4)—C'(5)	1.359 (11)	C'(5)—C'(6)	1.363 (9)
C'(5)—C'(7)	1.533 (9)	C(2)—C(3)	1.486 (10)
C(2)—C(1)	1.349 (7)	C(1)—O(1)	1.376 (8)
O(1)—C(4)	1.374 (7)	C(4)—C(5)	1.423 (10)
C(4)—O(2)	1.189 (9)		
C(2)—C'(1)—C'(6)	116.0 (6)	C(2)—C'(1)—C(2)	125.2 (6)
C'(6)—C'(1)—C(2)	118.7 (5)	C'(1)—C'(2)—C'(3)	121.5 (6)
C'(1)—C'(2)—O'(1)	118.7 (6)	C'(3)—C'(2)—O'(1)	119.8 (6)
C(2)—C'(3)—C'(4)	120.6 (7)	C'(3)—C'(4)—C'(5)	120.0 (7)
C'(4)—C'(5)—C'(6)	119.4 (7)	C'(4)—C'(5)—C'(7)	119.9 (7)
C'(6)—C'(5)—C'(7)	120.7 (6)	C'(1)—C'(6)—C'(5)	122.4 (6)
C(1)—C(2)—C(1)	122.5 (6)	C'(1)—C(2)—C(3)	118.3 (6)
C(2)—C(1)—O(1)	119.6 (5)	C(3)—C(2)—C(1)	119.1 (6)
O(1)—C(4)—C(5)	112.9 (6)	C(1)—O(1)—C(4)	119.1 (5)
O(2)—C(4)—C(5)	126.8 (6)	O(1)—C(4)—O(2)	120.3 (6)

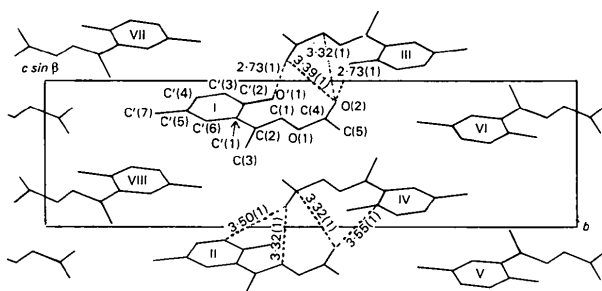


Fig. 2. (100) projection of the structure. Symmetry code: (I) x, y, z ; (II) $x, y, z-1$; (III) $\bar{x}, 1-y, 2-z$; (IV) $\bar{x}, 1-y, 1-z$; (V) $\bar{x}, \frac{1}{2}+y, \frac{1}{2}-z$; (VI) $\bar{x}, \frac{1}{2}+y, \frac{3}{2}-z$; (VII) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (VIII) $x, \frac{1}{2}-y, z-\frac{1}{2}$.

Acta Cryst. (1983). **C39**, 614–616

(2R,4S,5R)-2-Anilino-3,4-dimethyl-5-phenyl-1,3,2-oxazaphospholidine 2-Sulphide,
C₁₆H₁₉N₂OPS*

BY TADEUSZ J. BARTCZAK† AND ZDZISŁAW GALDECKI

Institute of General Chemistry, Technical University of Łódź, Żwirki 36, 90–924 Łódź, Poland

AND MARIA RUTKOWSKA

Regional Laboratory of Physicochemical Analysis and Structural Research, Jagiellonian University, Karasia 3, 30–360 Kraków, Poland

(Received 21 April 1982; accepted 13 January 1983)

Abstract. $M_r = 318.4$, orthorhombic, $P2_12_12_1$, $a = 10.935(1)$, $b = 16.757(5)$, $c = 9.192(1)$ Å, $V = 1684.3$ Å³, $Z = 4$, $D_c = 2.084$ Mg m⁻³, graphite-monochromated Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 0.242$ mm⁻¹, $F(000) = 672$, room temperature, $R = 0.0682$ for 1324 observed reflections. The five-membered oxazaphospholidine ring exists in the crystal in an envelope conformation with P deviating by $-0.339(2)$ Å downwards from the least-squares plane N(1), C(4), C(5), O.

Introduction. The compound was synthesized by reaction of (2*S*,4*S*,5*R*)-2-chloro-3,4-dimethyl-5-phenyl-1,3,2-oxazaphospholidine 2-sulphide (Lesiak & Stec, 1978; Bartczak & Galdecki, 1983) with lithium anilide.

Experimental. Irregular plate, approximately $0.4 \times 0.5 \times 0.6$ mm, CAD-4 automated four-circle diffrac-

tometer, 1778 unique reflections, $1 \leq 2\theta \leq 54^\circ$, 1324 with $F_o \geq 3\sigma(F_o)$, no absorption correction, maximum $hkl = 13,21,11$, noncentrosymmetric direct methods (SHELX 76, Sheldrick, 1976), standard least-squares methods and difference electron density syntheses, then blocked full-matrix technique. All H atoms, except those belonging to methyl groups C(6) and C(7), were located in subsequent difference Fourier syntheses based on reflections with $\sin \theta/\lambda \leq 0.3$ Å⁻¹. C(4) and C(6) and some C atoms of the phenyl rings were resolved with some difficulty in the initial stages of the structure solution, so the phenyl rings were converted into regular hexagons and refined as rigid groups. The constraints were released when passing to anisotropic refinements. Some final thermal parameters of C(4), C(6), C(22), C(23), C(24), C(25) and C(12) have rather high values which may indicate some degree of conformational disorder. However, a difference Fourier synthesis calculated without C(4) and C(6) did not reveal significantly extended regions of electron density. Methyl H atoms were generated in the final stages of refinement with the methyl groups C(6) and C(7)

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* Conformation of the 1,3,2-Oxazaphospholidine Ring. III.

† To whom correspondence should be addressed.