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## Structure of an Enantiomerically Pure Tetrahydropyranyl Ether

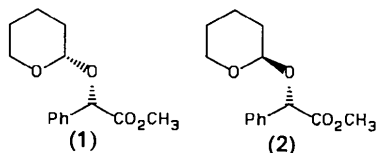
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**Abstract.** (2*S*)-Methyl 2-phenyl-2-[(2*S*)-tetrahydro-2(2*H*)-pyran-2-yl]acetate, C<sub>14</sub>H<sub>18</sub>O<sub>4</sub>, *M<sub>r</sub>* = 250.29, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 7.850 (1), *b* = 8.211 (1), *c* = 21.123 (3) Å, *V* = 1361.5 (3) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.22 g cm<sup>-3</sup>, Mo *Kα*, λ = 0.71073 Å, μ = 0.8 cm<sup>-1</sup>, *F*(000) = 536, *T* = 296 K, *R* = 0.039 for 973 unique reflections with *F<sub>o</sub>*<sup>2</sup> > 3σ(*F<sub>o</sub>*<sup>2</sup>). In this diastereomer the anomeric alkoxy substituent is axially oriented. The absolute stereochemistry at the anomeric carbon is assigned as *S* based upon the known stereochemistry of the mandelate ester.

**Experimental.** The title compound (1) crystallized as white plates by slow evaporation from ether. It was prepared by acid-catalyzed tetrahydropyran-2-yl-ation of (*S*)-(+)-methyl mandelate using 3,4-dihydro-2*H*-pyran, followed by chromatographic separation of the resulting pyranoside diastereomers (1) and (2).



The title compound was the less polar of the two (Mash, Arterburn, Fryling & Mitchell, 1991). Diastereomer (2) could not be induced to crystallize. The data crystal had dimensions 0.50 × 0.50 × 0.30 mm. A Syntex *P*2<sub>1</sub> diffractometer using Mo *Kα* radiation (λ = 0.71073 Å) and equipped with a graphite monochromator was used to collect the data. The lattice parameters were obtained from the least-squares refinement of 25 reflections with 20 < 2θ < 30°. The data were collected from the *hkl* octant using the θ–2θ scan technique to a maximum 2θ of 50.0° (0 < *h* < 10, 0 < *k* < 10, 0 < *l* < 26). The ratio of peak counting time to background counting time was 2:1.

As a check on crystal and electronic stability, two representative reflections were measured after every 98 reflections. The total loss in intensity was 7.8%; a linear decay correction was applied. Lorentz and polarization corrections were also applied to the data, but no absorption correction was made. Of 1442 reflections measured, 1419 were unique and not systematically absent. Reflections having *F<sub>o</sub>*<sup>2</sup> < 3σ(*F<sub>o</sub>*<sup>2</sup>) were considered unobserved (446 reflections). The structure was solved by direct methods and refined by full-matrix least squares where the function minimized was Σ*w*(*F<sub>o</sub>* – |*F<sub>c</sub>*|)<sup>2</sup>. H atoms were added at idealized positions and included in the refinement but restrained to ride on the atom to which they were bonded. All non-H atoms were refined anisotropically. In all, 163 parameters were refined. The refinements converged with *R* = 0.039, *wR* = 0.047, the e.s.d. of an observation of unit weight = 0.44, and a final shift to e.s.d. ratio of 0.00. The weights for each reflection were calculated using the counter weighting scheme *w* = 4(*F<sub>o</sub>*<sup>2</sup>)/σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>). The minimum and maximum peaks in the final Δ*F* map were –0.14 and 0.18 e Å<sup>-3</sup>, respectively, and the maximum |Δ/σ| was 0.03σ. Scattering factors were taken from Cromer & Waber (1974). Anomalous-dispersion effects were included in *F<sub>c</sub>* (Ibers & Hamilton, 1964) and the values for *f*' and *f*'' were those of Cromer (1974). All calculations were performed on a MicroVAX computer using *SDP/VAX* (Frenz, 1978). Atomic coordinates are listed in Table 1, while bond lengths and angles are listed in Table 2.\* The atom-labeling scheme is shown in Fig. 1.

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54422 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Positional parameters with e.s.d.'s in parentheses

$$B_{eq} = (8\pi^2/3) \sum_i \sum_j U_i \mu_i^* a_j^* a_i a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
O1	0.5670 (3)	0.3476 (3)	-0.0204 (1)	4.41 (5)
O2	0.5848 (3)	0.4782 (3)	0.0784 (1)	3.74 (4)
O3	0.6600 (4)	0.0844 (3)	0.1333 (1)	5.21 (6)
O4	0.4695 (4)	0.2573 (4)	0.1725 (1)	6.40 (7)
C1	0.6645 (5)	0.4639 (5)	-0.0561 (2)	4.70 (8)
C2	0.5524 (5)	0.5977 (5)	-0.0826 (2)	5.21 (9)
C3	0.4550 (5)	0.6809 (5)	-0.0293 (2)	5.15 (9)
C4	0.3597 (5)	0.5544 (5)	0.0095 (2)	4.60 (8)
C5	0.4750 (5)	0.4169 (4)	0.0299 (2)	4.00 (7)
C6	0.6945 (4)	0.3575 (4)	0.1041 (1)	3.34 (6)
C7	0.5927 (5)	0.2308 (4)	0.1406 (2)	3.82 (7)
C8	0.5978 (7)	-0.0496 (5)	0.1670 (2)	7.1 (1)
C9	0.8199 (4)	0.4429 (4)	0.1480 (1)	3.15 (6)
C10	0.7595 (5)	0.5343 (5)	0.1987 (1)	4.39 (8)
C11	0.8721 (6)	0.6135 (5)	0.2379 (2)	5.35 (9)
C12	1.0448 (5)	0.6024 (5)	0.2277 (2)	5.06 (9)
C13	1.1054 (5)	0.5110 (5)	0.1773 (2)	4.64 (8)
C14	0.9925 (4)	0.4320 (4)	0.1374 (2)	3.80 (7)

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

O1—C1	1.438 (7)	C4—C5	1.511 (8)
O1—C5	1.405 (7)	C6—C7	1.522 (8)
O2—C5	1.430 (7)	C6—C9	1.523 (8)
O2—C6	1.421 (6)	C9—C10	1.392 (7)
O3—C7	1.322 (7)	C9—C14	1.376 (8)
O3—C8	1.454 (8)	C10—C11	1.375 (9)
O4—C7	1.198 (7)	C11—C12	1.38 (1)
C1—C2	1.514 (9)	C12—C13	1.386 (9)
C2—C3	1.523 (9)	C13—C14	1.384 (8)
C3—C4	1.519 (9)		
C1—O1—C5	113.7 (4)	C7—C6—C9	110.3 (4)
C5—O2—C6	113.2 (4)	O3—C7—O4	123.7 (6)
C7—O3—C8	117.2 (5)	O3—C7—C6	110.6 (5)
O1—C1—C2	111.5 (5)	O4—C7—C6	125.7 (6)
C1—C2—C3	110.2 (5)	C6—C9—C10	119.8 (6)
C2—C3—C4	109.8 (5)	C6—C9—C14	120.5 (5)
C3—C4—C5	111.7 (5)	C10—C9—C14	119.7 (6)
O1—C5—O2	111.9 (5)	C9—C10—C11	120.0 (7)
O1—C5—C4	113.2 (5)	C10—C11—C12	120.4 (6)
O2—C5—C4	107.6 (5)	C11—C12—C13	119.7 (7)
O2—C6—C7	110.6 (5)	C12—C13—C14	120.1 (6)
O2—C6—C9	107.6 (4)	C9—C14—C13	120.0 (6)

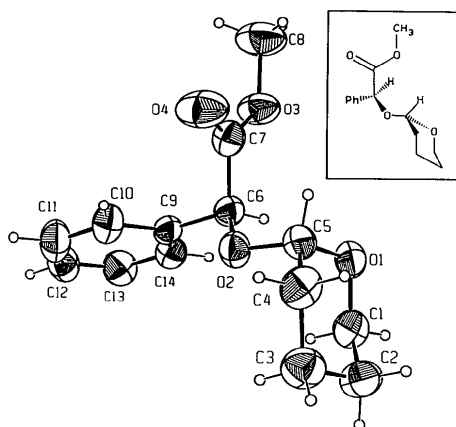


Fig. 1. ORTEP (Johnson, 1965) view of the title compound.

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Structure of  $\epsilon$ -Ammoniohexanoic Acid Dihydrogenphosphate

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**Abstract.** (5-Carboxypentyl)ammonium dihydrogenphosphate,  $\text{C}_6\text{H}_{14}\text{NO}_2^+ \cdot \text{H}_2\text{PO}_4^-$ ,  $M_r = 229.17$ , monoclinic,  $I2/a \pm (x, y, z; \frac{1}{2}-x, y, -z)$ ,  $a = 15.108 (3)$ ,  $b = 4.636 (1)$ ,  $c = 28.819 (3) \text{\AA}$ ,  $\beta = 98.62 (1)^\circ$ ,  $V =$

$1996 (4) \text{\AA}^3$ ,  $Z = 8$ ,  $D_x = 1.526 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71073 \text{\AA}$ ,  $\mu = 2.7 \text{ cm}^{-1}$ ,  $F(000) = 976$ ,  $T = 292 \text{ K}$ ,  $R = 0.039$  for 1682 observed reflections with  $I > 3\sigma(I)$ . The protonated  $\epsilon$ -aminohexanoic acid and the