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

$$B_{eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
S	0.5400 (2)	0.2585 (1)	0.60652 (9)	5.29 (3)
N1	0.0693 (6)	−0.1333 (3)	0.9300 (3)	5.13 (9)
N2	−0.1170 (6)	−0.1338 (3)	0.5880 (3)	5.5 (1)
N3	0.1930 (6)	0.6103 (4)	0.5806 (3)	5.7 (1)
N4	0.3884 (6)	0.6327 (3)	0.9208 (3)	5.27 (9)
N5	0.6877 (5)	0.2407 (3)	0.8475 (3)	4.88 (8)
C1	0.0773 (5)	0.1177 (3)	0.7603 (3)	2.94 (8)
C2	0.0474 (5)	0.1815 (3)	0.6639 (3)	3.29 (8)
C3	0.0968 (5)	0.3034 (3)	0.6614 (3)	3.27 (8)
C4	0.1866 (5)	0.3731 (3)	0.7573 (3)	3.00 (8)
C5	0.2127 (5)	0.3096 (3)	0.8552 (3)	3.35 (8)
C6	0.1635 (6)	0.1879 (3)	0.8572 (3)	3.37 (8)
C7	0.0259 (5)	−0.0074 (3)	0.7616 (3)	3.25 (8)
C8	0.0507 (6)	−0.0761 (4)	0.8558 (3)	3.71 (9)
C9	−0.0543 (6)	−0.0781 (4)	0.6649 (3)	3.93 (9)
C10	0.2386 (5)	0.4976 (3)	0.7540 (3)	3.41 (8)
C11	0.2118 (6)	0.5606 (4)	0.6575 (3)	3.86 (9)
C12	0.3225 (6)	0.5719 (3)	0.8475 (3)	3.69 (9)
C13	0.6555 (6)	0.3747 (4)	0.6984 (3)	3.70 (9)
C14	0.6828 (6)	0.4921 (4)	0.6604 (4)	4.9 (1)
C15	0.7683 (7)	0.5889 (4)	0.7299 (4)	5.7 (1)
C16	0.8229 (7)	0.5698 (4)	0.8353 (4)	5.4 (1)
C17	0.7982 (6)	0.4551 (4)	0.8735 (3)	4.6 (1)
C18	0.7110 (6)	0.3552 (4)	0.8060 (3)	3.60 (8)
C20	0.6034 (6)	0.1331 (4)	0.7940 (3)	3.79 (9)
C21	0.5885 (6)	0.0256 (4)	0.8490 (4)	5.0 (1)
C22	0.5080 (6)	−0.0848 (4)	0.7960 (5)	5.9 (1)
C23	0.4372 (7)	−0.0873 (4)	0.6892 (4)	5.8 (1)
C24	0.4447 (6)	0.0190 (4)	0.6348 (4)	5.0 (1)
C25	0.5304 (6)	0.1296 (4)	0.6848 (3)	3.86 (9)
H′N5	0.734 (6)	0.235 (4)	0.916 (3)	

Table 2. *Selected geometric parameters* (Å, °)

S—C13	1.752 (4)	C7—C8	1.429 (6)
S—C25	1.750 (4)	C7—C9	1.442 (6)
N1—C8	1.145 (5)	C10—C11	1.423 (6)
N2—C9	1.144 (5)	C10—C12	1.436 (6)
N3—C11	1.132 (5)	C13—C14	1.390 (6)
N4—C12	1.140 (5)	C13—C18	1.391 (5)
N5—C18	1.376 (5)	C14—C15	1.388 (7)
N5—C20	1.378 (5)	C15—C16	1.364 (8)
N5—H′N5	0.88 (5)	C16—C17	1.360 (7)
C1—C2	1.425 (5)	C17—C18	1.401 (6)
C1—C6	1.451 (5)	C20—C21	1.388 (6)
C1—C7	1.380 (5)	C20—C25	1.399 (6)
C2—C3	1.345 (5)	C21—C22	1.392 (7)
C3—C4	1.444 (5)	C22—C23	1.368 (8)
C4—C5	1.443 (5)	C23—C24	1.365 (7)
C4—C10	1.378 (5)	C24—C25	1.391 (6)
C5—C6	1.343 (5)		
C13—S—C25	102.8 (2)	N3—C11—C10	179.1 (5)
C18—N5—C20	127.3 (4)	N4—C12—C10	178.8 (4)
C18—N5—H′N5	116 (3)	S—C13—C14	117.1 (3)
C20—N5—H′N5	116 (3)	S—C13—C18	122.9 (3)
C2—C1—C6	118.1 (3)	C14—C13—C18	120.0 (4)
C2—C1—C7	121.0 (3)	C13—C14—C15	119.7 (5)
C6—C1—C7	120.8 (3)	C14—C15—C16	120.3 (5)
C1—C2—C3	121.7 (4)	C15—C16—C17	120.6 (5)
C2—C3—C4	120.5 (4)	C16—C17—C18	120.8 (5)
C3—C4—C5	117.9 (3)	N5—C18—C13	122.2 (4)
C3—C4—C10	120.2 (3)	N5—C18—C17	119.2 (4)
C5—C4—C10	121.9 (3)	C13—C18—C17	118.6 (4)
C4—C5—C6	121.3 (4)	N5—C20—C21	119.7 (4)
C1—C6—C5	120.4 (4)	N5—C20—C25	121.5 (4)
C1—C7—C8	123.6 (3)	C21—C20—C25	118.8 (4)
C1—C7—C9	120.8 (3)	C20—C21—C22	120.6 (5)
C8—C7—C9	115.6 (3)	C21—C22—C23	120.2 (5)
N1—C8—C7	178.6 (4)	C22—C23—C24	119.8 (5)
N2—C9—C7	179.5 (4)	C23—C24—C25	121.5 (5)
C4—C10—C11	122.0 (4)	S—C25—C20	123.3 (3)
C4—C10—C12	122.2 (4)	S—C25—C24	117.6 (4)
C11—C10—C12	115.8 (3)	C20—C25—C24	119.1 (4)

All calculations were performed on a Digital MicroVAX 3100 computer with the *MolEN* package (Fair, 1990).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, including H-atom geometry, have been deposited with the IUCr (Reference: PA1062). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-6-Methoxycarbonyl-2,10-dioxa-1-phosphabicyclo[4.4.0]decane 1-Oxide

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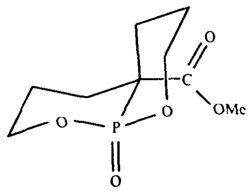
Abstract

The crystal structure of the title compound, $C_9H_{15}O_5P$, is described. The molecule consists of two *cis*-fused six-membered rings, both in the chair conformation.

Comment

The crystal structure of the title compound, (I), was investigated in order to establish the stereochemistry of the methoxycarbonyl group relative to the phosphoryl group. Since the two analogous model compounds, decalin and 2,10-dioxabicyclo[4.4.0]decane (Descotes, Lissac, Delmau & Duplan, 1968; Beaulieu, Dickinson & Deslongchamps, 1980) exist in both *cis* and *trans* isomeric forms, the goal was to synthesize both isomeric forms of this bicyclic phosphonate. For some as yet unknown reason only one isomer of the parent compound, 2,10-dioxa-1-phosphabicyclo[4.4.0]decane 1-oxide, (II), (Chérest & Whitham, 1976; Whitham, 1989; Bellard, Postle & Sheldrick, 1978) is produced regardless of the synthetic route employed (Rodriguez, Sommese & Cremer, 1993). It was hoped that the presence of the methoxycarbonyl substituent would lead to both the *cis* and *trans* isomers of (I). Surprisingly, as with (II), only the *cis* isomer of (I) has been observed so far (Rodriguez, Sommese & Cremer, 1993). The determination of the crystal structure was required in order to assign the *cis*-fused configuration unambiguously and therefore gain additional information about the apparent stability of the *cis* configuration (or instability of the *trans* configuration).



(I)

The two rings in (I), like the two rings in (II), are intrinsically non-equivalent in the solid state. Although the non-equivalence of the rings in (I) is not discernible from the P—O bond lengths [1.567 (2) and 1.573 (2) Å], which differ by only 0.006 (3) Å, *i.e.* 2σ, the bond angle at O2 [119.1 (2)°] is significantly wider than the angle at O3 [116.8 (2)°].

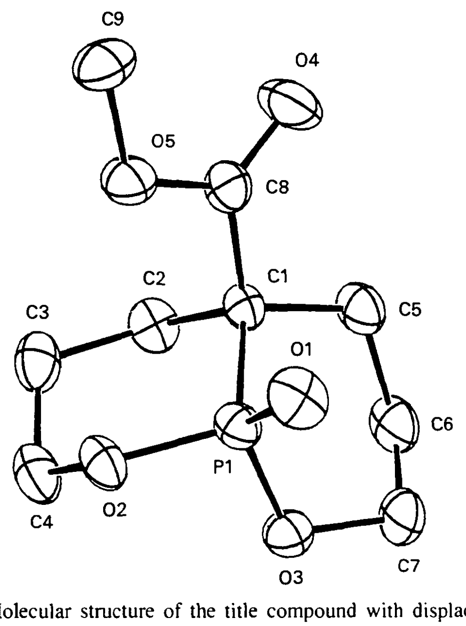


Fig. 1. Molecular structure of the title compound with displacement ellipsoids at the 35% probability level.

The P1—C1 bond [1.814 (3) Å] is, as expected, the longest bond in (I), and is even longer than the corresponding bond in (II) [1.783 (4) Å]. The P—C torsion angle in (I) [O2—P1—C1—C8 86.8 (2)°] is also larger than that in (II) [O1—P1—C4—H7 46.4°]. These differences may be the result of a greater steric and electronic interaction between the methoxycarbonyl group and the phosphoryl group in (I) than between the α-H atom and the phosphoryl group in (II).

Further studies aimed at determining whether the preferential formation of *cis*-(I) and *cis*-(II) during the synthesis of these compounds is directed by thermodynamic or kinetic control are underway.

Experimental

The title compound was synthesized by the procedure described by Rodriguez, Sommese & Cremer (1993). Crystals (m.p. 397–398 K) were grown from a methylene chloride solution layered with diethyl ether.

Crystal data

$C_9H_{15}O_5P$
$M_r = 234.12$
Orthorhombic
Pbca
$a = 26.377$ (4) Å
$b = 8.297$ (2) Å
$c = 9.994$ (2) Å
$V = 2187.2$ (8) Å^3
$Z = 8$
$D_x = 1.422$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 21 reflections
$\theta = 3.09$–$7.91°$
$\mu = 0.24$ mm^{-1}
$T = 298$ K
Plate
0.40 × 0.30 × 0.30 mm
Colorless

Data collection

Picker diffractometer
θ–2θ scans
Absorption correction: none
1690 measured reflections
1426 independent reflections
1021 observed reflections [$I > 2.0\sigma(I)$]

$\theta_{max} = 23.5°$
$h = 0 \rightarrow 28$
$k = 0 \rightarrow 8$
$l = 0 \rightarrow 10$
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R(F) = 0.034$
$wR(F^2) = 0.055$
$S = 2.55$
1021 reflections
138 parameters
H-atom parameters not refined
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max} = 0.304$

$\Delta\rho_{max} = 0.220$ e Å^{-3}
$\Delta\rho_{min} = -0.260$ e Å^{-3}
Extinction correction: $F_c^* = kF_c[1 + (0.001x \times F_c^2\lambda^3/\sin 2\theta)]^{-1/4}$
Extinction coefficient: $x = 0.0042$ (3)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å²)

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

	x	y	z	U_{eq}
P1	0.16207 (3)	0.43031 (10)	0.16173 (9)	0.041 (1)
O1	0.17235 (9)	0.5013 (3)	0.2921 (2)	0.057 (1)
O2	0.13699 (7)	0.2596 (2)	0.1722 (2)	0.048 (1)
O3	0.20979 (7)	0.3968 (3)	0.0715 (2)	0.054 (1)
O4	0.07147 (10)	0.7729 (3)	0.1417 (3)	0.082 (2)
O5	0.05538 (9)	0.52581 (9)	0.2121 (2)	0.058 (2)
C1	0.12217 (12)	0.5540 (4)	0.0553 (3)	0.038 (2)
C2	0.0979 (1)	0.4501 (4)	−0.0567 (3)	0.054 (2)
C3	0.0781 (1)	0.2851 (4)	−0.0141 (3)	0.060 (2)
C4	0.1181 (1)	0.1829 (4)	0.0515 (4)	0.060 (2)
C5	0.1552 (1)	0.6890 (4)	−0.0064 (3)	0.055 (2)
C6	0.2037 (1)	0.6279 (4)	−0.0740 (3)	0.065 (2)
C7	0.2371 (1)	0.5378 (4)	0.0206 (3)	0.066 (2)
C8	0.0809 (1)	0.6332 (4)	0.1394 (3)	0.045 (2)
C9	0.0180 (1)	0.5882 (4)	0.3036 (3)	0.062 (2)

Table 2. *Selected geometric parameters* (Å, °)

P1—O2	1.567 (2)	O2—C4	1.454 (4)
C8—C1	1.523 (5)	C2—C3	1.527 (5)
P1—O1	1.455 (2)	O5—C8	1.334 (4)
C8—O4	1.186 (5)	C3—C4	1.504 (5)
P1—O3	1.573 (2)	O5—C9	1.441 (4)
C1—C2	1.551 (5)	C5—C6	1.535 (5)
P1—C1	1.814 (3)	O3—C7	1.465 (4)
C1—C5	1.548 (5)	C7—C6	1.492 (5)
O2—P1—O1	112.65 (14)	P1—O2—C4	119.1 (2)
P1—C1—C8	109.6 (2)	C1—C2—C3	116.0 (3)
O2—P1—O3	102.50 (13)	C8—O5—C9	116.9 (3)
P1—C1—C2	110.4 (2)	C2—C3—C4	112.7 (3)
O2—P1—C1	107.79 (14)	P1—O3—C7	116.8 (2)
P1—C1—C5	108.4 (2)	C1—C5—C6	114.0 (3)
O1—P1—O3	115.81 (14)	O5—C8—C1	111.9 (3)
C8—C1—C2	110.2 (3)	O2—C4—C3	110.9 (3)
O1—P1—C1	113.81 (15)	O5—C8—O4	122.4 (3)
C8—C1—C5	108.0 (3)	O3—C7—C6	109.3 (3)
O3—P1—C1	103.19 (14)	C1—C8—O4	125.7 (3)
C2—C1—C5	110.2 (3)	C5—C6—C7	112.2 (3)
O1—P1—O2—C4	171.6 (3)	O3—P1—O2—C4	−63.2 (2)
C1—P1—O2—C4	45.2 (2)	O1—P1—C1—C8	−38.9 (2)
O1—P1—O3—C7	−68.0 (2)	O1—P1—C1—C5	78.8 (3)
O2—P1—C1—C8	86.8 (2)	O1—P1—C1—C2	−160.4 (3)
O3—P1—C1—C5	−47.5 (2)		

All H-atoms were allowed to ride on the heavy atom with isotropic displacement factors 1.5 times the equivalent isotropic displacement factor of the heavy atom. The methyl group was refined as a rigid rotor. The data were globally sorted and collected using *PCXTL* data collection software (Weinrach & Bennett, 1991). Cell refinement and data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976) and *PLUTO84* (Motherwell, 1984).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms and torsion angles have been deposited with the IUCr (Reference: BK1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(*S*)-1-Phenylethylammonium (*S*)-Mandelate–Mandelic Acid (1/2), $C_8H_{12}N^+.C_8H_7O_3^-.2C_8H_8O_3$

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Abstract

The asymmetric unit of the title compound contains one (*S*)-1-phenylethylammonium cation, one (*S*)-mandelate anion and two (*S*)-mandelic acid molecules. The entities are connected by an extensive hydrogen-bond system. The crystal structure is very similar to that of its diastereomeric less soluble compound (*R*)-1-phenylethylammonium (*S*)-mandelate–mandelic acid (1/2).

Comment

The title compound, (1), has been isolated as part of an investigation of salts formed between 1-phenylethylamine and mandelic acid. When racemic 1-phenylethylamine and (*S*)-mandelic acid are mixed in equimolar amounts in water, (*S*)-1-phenylethylammonium (*S*)-mandelate precipitates as the less soluble salt (Larsen & Lopez de Diego, 1993*a*). When racemic 1-phenylethylamine and (*S*)-mandelic acid are mixed in the molar ratio 1:3 in water, the less soluble compound