2-[(4-Chlorophenyl)hydroxymethyl]-5,5-dimethyl-4-phenyl-1,3,2-dioxaphosphorinan-2-one

In the title compound, C_{18}H_{20}ClO_{4}P, the P atom is in a distorted tetrahedral configuration. In the crystal structure, molecules are linked by intermolecular C–H⋯O and O–H⋯O hydrogen bonds.

**Comment**

Some hydroxyphosphonates have shown good biological and pharmaceutical activities (Chen *et al.*, 1995). In continuation of our work on the synthesis of phosphonic acid derivatives with good biological activities (He *et al.*, 2005), a number of hydroxyphosphonates have been synthesized, and we report here the crystal structure of the title compound, (I).

The dioxaphosphorinane ring in the molecule adopts a chair conformation (Fig. 1). Selected bond lengths and angles are listed in Table 1. The P1=O3, P1–O1 and P1–O2 bond lengths are similar to those observed in a related structure, viz. 2-benzylamino-5,5-dimethyl-4-phenyl-1,3,2-dioxaphosphorinan-2-oxide [1.4650 (13), 1.5814 (11) and 1.5888 (12) Å, respectively; Wan *et al.*, 2005]. The bond angles around P range from 106.1 (1) to 112.3 (1)°, indicating a distorted tetrahedral configuration for the P atom.

The crystal packing is stabilized by C–H⋯O and O–H⋯O hydrogen bonds (Table 2 and Fig. 2).
Experimental

5,5-Dimethyl-4-phenyl-1,3,2-dioxaphosphorinan-2-one was prepared according to a literature procedure (Ten Hoeve & Wynberg, 1985). To a mixture of 5,5-dimethyl-4-phenyl-1,3,2-dioxaphosphorinan-2-one (5 mmol), 4-chlorobenzaldehyde (5 mmol) and toluene (10 ml) was added triethylamine (5 mmol). The flask was cooled in an ice-bath, and the mixture was stirred for 1 h at room temperature. The white solid was filtered, washed with toluene (5 ml) and recrystallized (CH₂Cl₂/toluene, 1:1) to give the title compound. Suitable crystals were obtained by vapor diffusion of ethanol into a dimethylformamide solution at room temperature (m.p. 499 K).

Crystal data

\[ \text{C}_9\text{H}_{16}\text{ClO}_4\text{P} \]

\[ M_e = 366.76 \]

Monoclinic, \( P2_1 \)

\( a = 10.5609 \) (14) Å

\( b = 6.4450 \) (9) Å

\( c = 14.1161 \) (19) Å

\( \beta = 110.475 \) (2)

\( V = 900.1 \) (2) Å³

Data collection

Bruker SMART APEX CCD area-detector diffractometer

\( \varphi \) and \( \omega \) scans

Absorption correction: none

3837 independent reflections

2940 reflections with \( I > 2\sigma(I) \)

\( R_{\text{int}} = 0.032 \)

\( \theta_{\text{max}} = 27.0 \)

Table 1

Selected geometric parameters (Å, °).

<table>
<thead>
<tr>
<th>C12—P1</th>
<th>1.817 (3)</th>
<th>O2—P1</th>
<th>1.568 (2)</th>
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</thead>
<tbody>
<tr>
<td>O1—P1</td>
<td>1.564 (2)</td>
<td>O3—P1</td>
<td>1.466 (2)</td>
</tr>
<tr>
<td>O3—P1—O1</td>
<td>111.83 (13)</td>
<td>O3—P1—C12</td>
<td>112.29 (14)</td>
</tr>
<tr>
<td>O3—P1—O2</td>
<td>111.64 (13)</td>
<td>O3—P1—C12</td>
<td>107.05 (13)</td>
</tr>
<tr>
<td>O1—P1—O2</td>
<td>106.09 (11)</td>
<td>O1—P1—C12</td>
<td>107.80 (13)</td>
</tr>
</tbody>
</table>

Table 2

Hydrogen-bond geometry (Å, °).

<table>
<thead>
<tr>
<th>D—H—A</th>
<th>D—H</th>
<th>H···A</th>
<th>D···A</th>
<th>D—H···A</th>
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</thead>
<tbody>
<tr>
<td>C11—H11A—O1</td>
<td>0.97</td>
<td>2.57</td>
<td>3.290 (4)</td>
<td>131</td>
</tr>
<tr>
<td>C12—H12—O4w</td>
<td>0.98</td>
<td>2.29</td>
<td>3.126 (4)</td>
<td>143</td>
</tr>
<tr>
<td>C9—H9A—O3w</td>
<td>0.96</td>
<td>2.52</td>
<td>3.439 (4)</td>
<td>159</td>
</tr>
<tr>
<td>O4—H4A—O3w</td>
<td>0.814 (10)</td>
<td>1.913 (16)</td>
<td>2.696 (5)</td>
<td>161 (4)</td>
</tr>
</tbody>
</table>

Symmetry codes: (i) \(-x+2, y-\frac{1}{2}, -z\); (ii) \(-x+1, y-\frac{1}{2}, z\); (iii) \(x, y, 1-z\).

Figure 2

The molecular structure of (I), showing the C—H···O and O—H···O hydrogen bonds (dashed lines). H atoms not involved in these interactions have been omitted.

The hydroxyl H atom was located in a difference map and refined with the restraint O—H = 0.82 (1) Å; \( U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O}) \). All the carbon-bound H atoms were positioned geometrically and treated as riding on their parent atoms; C—H = 0.93–0.98 Å; \( U_{\text{iso}}(\text{H}) = x U_{\text{eq}}(\text{C}) \), where \( x = 1.5 \) for methyl H atoms and 1.2 for the remaining H atoms.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELX97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 1997); software used to prepare material for publication: SHELXTL.

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References


