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Phenylphosphinic acid

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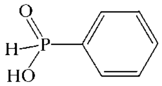
The crystal structure of the title compound, $C_6H_7O_2P$, shows continuous hydrogen-bonding chains in the *x* direction, with a P—O···O═P distance of 2.513 (3) Å.

Comment

Phosphinic acids possess the ability to form strong hydrogen bonds in the solid state by virtue of the very electronegative O atoms bound to an electropositive P atom. Simple phosphinic acids in the gas phase show almost twice the dimerization enthalpy of the analogous carboxylic acids (Denisov & Tokhadze, 1994; González *et al.*, 1998; Colominas *et al.*, 1998). The title compound, (I), was investigated as part of our research into the effects of stong hydrogen bonding in phosphinic acid derivatives.



(I)

The title compound crystallizes in the non-centrosymmetric space group $Pna2_1$, but shows racemic twinning; the Flack *x* parameter refined to 0.60 (14). Selected bond lengths and angles are given in Table 1. Molecules of phenylphosphinic acid related by the a glide plane perpendicular to the *c* axis form infinite hydrogen-bonded chains of the type OH···O═P—OH···O═P. The very short P—O···O═P distance of 2.513 (3) Å indicates a strong hydrogen bond. This arrangement is the more commonly found in the crystalline state for phosphinic or phosphonic acids; a dimeric form, forming an eight-membered ring, is also seen (Druyan *et al.*, 1976; Reis *et al.*, 1976). The P═O and P—O distances are normal compared with averages found for related compounds, *i.e.* 1.489 and 1.560 Å, respectively (Allen *et al.*, 1987). The phenyl groups pack in a herring-bone-type arrangement along *ab* planes, with the planes of the phenyl groups tilted 59.44 (6)° with respect to each other. A weak interaction is noted between atoms H15 and O2 and is shown in the supplementary material. Table 2 lists the hydrogen-bonding interactions.

Experimental

Phenylphosphinic acid was prepared according to the procedure of Voight & Gallais (1953), using $P(C_6H_5)Cl_2$ and $CHCl_3$ in place of PCl_3 and CCl_4. The yield was quantitative, giving pure product as checked with ^1H and ^{31}P NMR spectroscopy. Crystals suitable for X-ray diffraction were grown by slow evaporation of a solution of the acid in acetone in a desiccator containing silica desiccant.

Crystal data

$C_6H_7O_2P$
$M_r = 142.09$
Orthorhombic, $Pna2_1$
$a = 7.6624$ (6) Å
$b = 5.5762$ (4) Å
$c = 15.2489$ (11) Å
$V = 651.54$ (8) Å^3
$Z = 4$
$D_x = 1.449$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4387 reflections
$\theta = 2.67$–27.48°
$\mu = 0.337$ mm^{-1}
$T = 100$ (2) K
Block, colourless
0.30 × 0.30 × 0.26 mm

Data collection

Nonius KappaCCD diffractometer
ψ and ω scans with κ offsets
Absorption correction: multi-scan (Otwinowski & Minor, 1997)
$T_{\min} = 0.906$, $T_{\max} = 0.918$
4387 measured reflections
1364 independent reflections
1162 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.054$
$\theta_{\max} = 27.48°$
$h = -9 \rightarrow 9$
$k = -7 \rightarrow 7$
$l = -19 \rightarrow 19$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.089$
$S = 1.064$
1364 reflections
93 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0474P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.006$
$\Delta\rho_{\max} = 0.22$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.002 (4)
Absolute structure: Flack (1983), 593 Friedel pairs
Flack parameter = 0.60 (14)

Table 1
Selected geometric parameters (Å, °).

P—O1	1.4933 (19)	P—H1	1.40 (3)
P—O2	1.556 (2)	O2—H2	0.88 (4)
O1—P—O2	115.82 (11)	O2—P—H1	97.5 (11)
O1—P—H1	119.3 (11)	P—O2—H2	107 (2)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1$^{\rm i}$	0.88 (4)	1.64 (4)	2.513 (3)	168 (3)
C15—H15···O2$^{\rm ii}$	0.95	2.66	3.383 (3)	133

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

The H atom bound to the P atom and the H atom of the hydroxyl group were found in the difference Fourier map and allowed to refine with isotropic displacement parameters. The H atoms on the phenyl ring were constrained to positions bisecting the C—C—C angles and the C—H distance fixed to the default value for the program, 0.95 Å. The isotropic displacement parameters were set to be 20% larger than the those of the atoms to which the H atoms were bonded.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement and data reduction: *DENZO–SMN* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure andprepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–19.

Colominas, C., Teixidó, J., Cemelí, J., Luque, F. J. & Orozco, M. (1998). *J. Phys. Chem. B*, **102**, 2269–2276.

Denisov, G. S. & Tokhadze, K. G. (1994). *Dokl. Akad. Nauk.* **337**, 54–56.

Druyan, M. E., Reis, A. H. Jr, Gebert, E., Peterson, S. W., Mason, G. W. & Peppard, D. F. (1976). *J. Am. Chem. Soc.* **98**, 4801–4805.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

González, L., Mó, O., Yáñez, M. & Elguero, J. (1998). *J. Chem. Phys.* **109**, 2685–2693.

Nonius (1997). *KappaCCD Server Software*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods Enzymol.* **276**, 307–326.

Reis, A. H. Jr, Peterson, S. W., Druyan, M. E., Gebert, E., Mason, G. W. & Peppard, D. F. (1976). *Inorg. Chem.* **15**, 2748–2752.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.

Voight, D. & Gallais, F. (1953). *Inorg. Synth.* **4**, 55–58.

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