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Ethyl [(2-hydroxyethylaminio)(2-hydroxyphenyl)methyl]phosphonate dihydrate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.047
wR factor = 0.112
Data-to-parameter ratio = 13.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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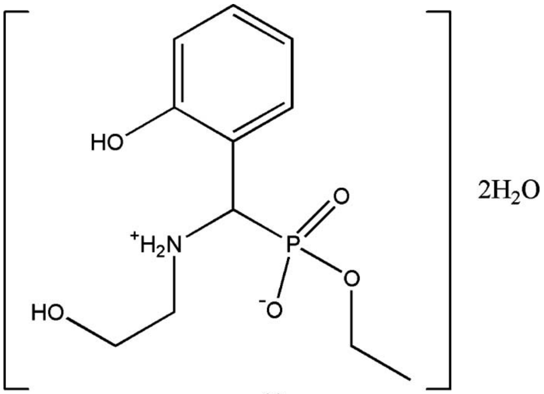
The organic moiety of the title compound, $C_{11}H_{18}NO_5P\cdot 2H_2O$, is zwitterionic. The crystal packing is stabilized by N—H···O and O—H···O hydrogen bonds.

Comment

Aminophosphonic acids and their derivatives have received much attention because they are phosphorus analogs of naturally occurring aminocarboxylic acids (Palacios *et al.*, 2000). Indeed, some potentially very powerful chelating agents, antibiotics, and pharmacological agents arise from aminophosphonic acids and their derivatives (Kurzak *et al.*, 2000; Xu *et al.*, 2000). A number of synthetic methods for the preparation of 1-aminoalkylphosphonates have been described (Xu *et al.*, 2000; Xu & Fu, 2001). One of the routes involves the addition of dialkyl phosphonates to Schiff bases (Namza *et al.*, 1999). In this paper, we report the one-pot reaction of the synthesis of the title compound, (I), an α-*N*-ethylaminomethylphosphonic acid monoethyl ester starting from salicylaldehyde, primary amine and dialkyl phosphonate.



(I)

The molecular conformation and geometric parameters of (I) are shown in Fig. 1 and Table 1. The structural parameters for (I) are similar to those of the previously reported hemihydrate compound of the same organic moiety (Namza *et al.*, 1999). Both crystallize as zwitterionic organic molecules containing $—NH_2^+—$ and $—PO_2^-—$ ionic groups, *i.e.* nominal proton transfer from hydrogenphosphate to amine.

The difference arises in unit-cell content, *viz*. (I) having two water molecules of hydration instead of half an equivalent of water in the compound previously described (Namza *et al.*, 1999). As a result of this, (I) contains more intermolecular hydrogen bonds (Table 2) to stabilize the crystal packing, including N—H···O and O—H···O interactions. An intramolecular N1—H1*A*···O5 bond is also present (Fig. 1).

Experimental

All chemicals were of reagent grade and commercially available, and were used without further purification. A mixture of salicylaldehyde (0.02 mol) and ethanolamine (0.02 mol) in ethanol (30 ml) was refluxed for 2–4 h. An ethanol solution (10 ml) of diethyl phosphonate (0.02 mol) was then added dropwise. The resulting solution was refluxed until a solid appeared. The solid product was filtered off, washed with ethanol and recrystallized from water to give (I) in 45% yield.

Crystal data

$C_{11}H_{18}NO_5P\cdot2H_2O$
$M_r = 311.27$
Monoclinic, $P2_1/c$
$a = 7.356$ (2) Å
$b = 8.707$ (5) Å
$c = 23.417$ (8) Å
$\beta = 102.80$ (3)°
$V = 1462.6$ (11) Å^3
$Z = 4$
$D_x = 1.414$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1375 reflections
$\theta = 1.8$–27.5°
$\mu = 0.22$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.20 × 0.15 × 0.15 mm

Data collection

SMART 1K CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)
$T_{\min} = 0.958$, $T_{\max} = 0.968$
5972 measured reflections
2498 independent reflections
1718 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.075$
$\theta_{\max} = 25.0$°
$h = -8 \rightarrow 1$
$k = -9 \rightarrow 9$
$l = -24 \rightarrow 26$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.047$
$wR(F^2) = 0.112$
$S = 1.03$
2498 reflections
184 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0442P)^2 + 0.189P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

Table 1
Selected bond lengths (Å).

P1—C7	1.837 (3)	P1—O3	1.489 (2)
P1—O4	1.480 (2)	P1—O5	1.596 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O5	0.90	2.52	3.010 (3)	115
N1—H1*A*···O6$^{\rm i}$	0.90	2.14	2.901 (3)	142
N1—H1*B*···O7$^{\rm ii}$	0.90	1.95	2.825 (3)	162
O1—H1···O3$^{\rm iii}$	0.82	1.82	2.639 (3)	172
O2—H2···O3$^{\rm i}$	0.82	1.91	2.716 (3)	167
O6—H61···O4	0.82	2.05	2.866 (3)	169
O6—H62···O4$^{\rm ii}$	0.82	1.94	2.714 (3)	159
O7—H72···O5$^{\rm iv}$	0.82	2.44	3.073 (3)	135
O7—H71···O6	0.82	2.07	2.867 (4)	163

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

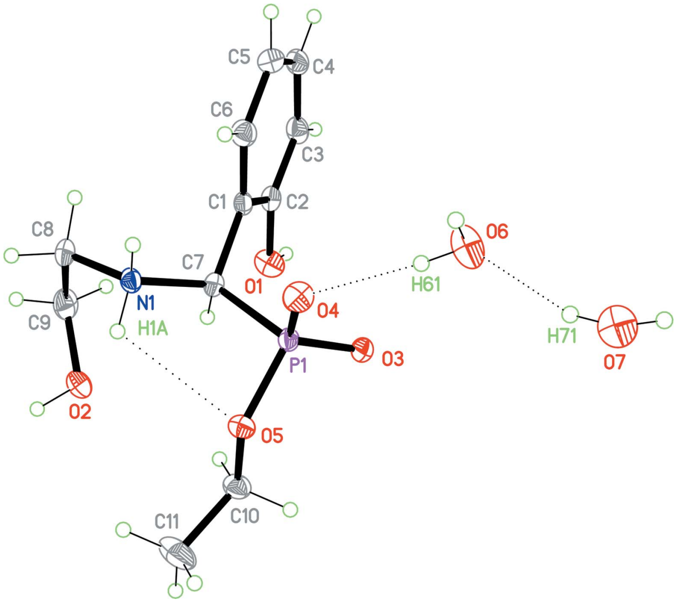


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 30% probability level for non-H atoms. Dashed lines indicate hydrogen bonds.

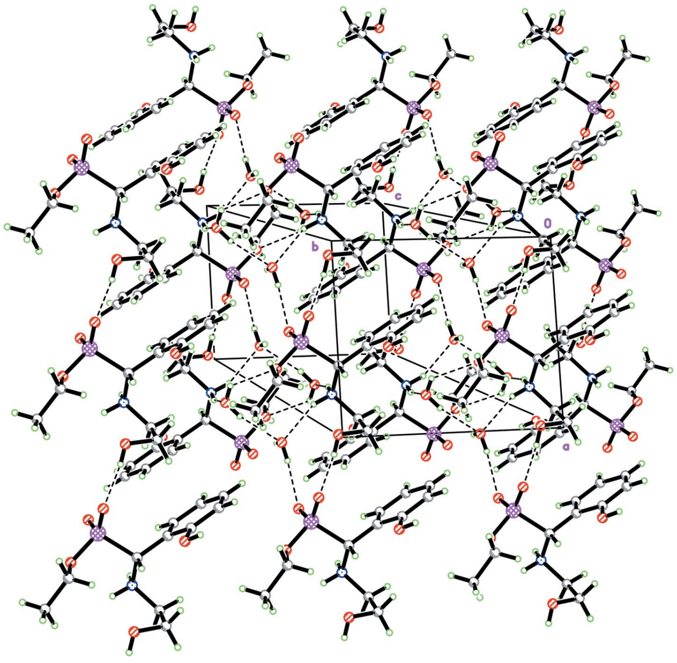


Figure 2
A packing diagram for (I). Colour key: O red, C grey, P pink, H green and N blue. Dashed lines indicate hydrogen bonds.

H atoms attached to C, N and O atoms (except for water) of (I) were placed in geometrically idealized positions, with Csp^2—H = 0.93 Å, ethyl Csp^3—H = 0.97 Å, methyl Csp^3—H = 0.96 Å, N—H = 0.86 Å and O—H = 0.82 Å, and refined as riding, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$ or $1.5U_{\rm eq}(O,C_{\rm methyl})$. H atoms attached to water O atoms were located in difference Fourier maps and refined with a global $U_{\rm iso}$ value. The O—H distances in water are in the range 0.820–0.824 Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

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