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Lithium phosphoenolpyruvate monohydrate at 85 K

Tomasz Zych,[a] Ilona Turowska-Tyrk[a]* and Tadeusz Lis[b]

[a]Department of Chemistry, Wrocław University of Technology, 27 Wybrzeże Wyspiańskiego, 50-370 Wrocław, Poland, and [b]Department of Chemistry, University of Wrocław, 14 F. Joliot-Curie, 50-383 Wrocław, Poland
Correspondence e-mail: ilona@kchf.ch.pwr.wroc.pl

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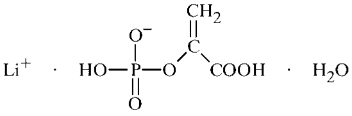
The crystal structure of the title compound, lithium (1-carboxyethenyloxy)phosphonate monohydrate, $Li^+ \cdot C_3H_4O_6P^- \cdot H_2O$, is governed by lithium–oxygen interactions and hydrogen bonds. The Li^+ cation is tetrahedrally coordinated by phosphate and water O atoms. The phosphoenolpyruvate monoanions form carboxyl-to-carboxyl and phosphate-to-water hydrogen bonds.

Comment

The phosphoenolpyruvate (PEP) plays an important biological role (Stryer, 1995). The geometrical details of PEP derivatives, as well as the effect of the protonation level and chemical environment on the PEP geometry, have been discussed previously by Weichsel & Lis (1994) and Souhassou *et al.* (1996). We performed the X-ray structure determination of lithium phosphoenolpyruvate monohydrate, $LiPEP \cdot H_2O$, (I), as part of diffraction studies of charge density in crystals of phosphoenolpyruvate derivatives.



$$\mathrm{Li^+ \cdot HO{-}P(=O)(O^-){-}O{-}C(=CH_2){-}COOH \cdot H_2O}$$

(I)

The crystals of (I) are composed of tetracoordinated Li^+ cations, phosphoenolpyruvate monoanions and water of hydration. An *ORTEP*-3 (Johnson *et al.*, 1997) view of the PEP fragment in the title monoanion is presented in Fig. 1. The enolpyruvate fragment is nearly planar and the enolic O4—C2 bond exhibits partial double-bond character, as was also observed in other monoionized PEP systems (Weichsel & Lis, 1994). The length of the 'high-energy' phosphate ester bond P—O4 is 1.6200 (3) Å, which is typical for monoionized PEP derivatives (Souhassou *et al.*, 1996).

The phosphate and water O atoms form tetrahedral coordination around the Li^+ cation. The Li···O contacts range from 1.900 (1) to 2.021 (1) Å. Each O3 atom joins two centrosymmetrically related Li^+ cations, forming in this way an Li_2O_2 unit. A short distance between the Li^+ cations in the four-membered ring [2.661 (2) Å] is also observed in other lithium derivatives. Furthermore, the O2 and O3 atoms of the phosphate group bridge two other centrosymmetrically related Li atoms, forming an eight-membered ring with an Li···Li distance of 3.805 (2) Å. In this way, the polymeric structure along the *z* axis is formed (Fig. 2).

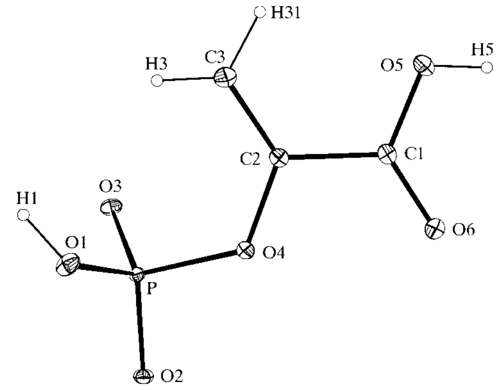


Figure 1
An *ORTEP*-3 (Johnson *et al.*, 1997) view of the PEP fragment with the atomic labelling scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level. Displacement parameters of H atoms were artificially diminished for clarity.

The geometry of the hydrogen bonds is given in Table 2. Each carboxyl group participates in a hydrogen-bonded cyclic dimer around an inversion centre, creating in this way a two-dimensional network. The phosphate O1—H1 group takes part in a bond with the O7 water atom, and the H71 water atom is utilized in bonding with the O2 atom from an adjacent anion, forming cyclic centrosymmetric systems of hydrogen bonds, so generating a three-dimensional structure.

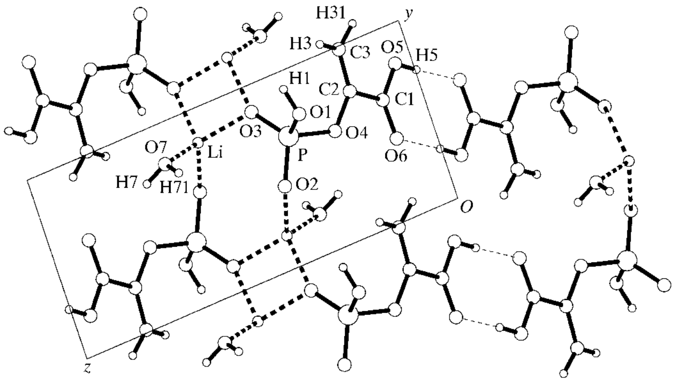


Figure 2
The packing diagram viewed down the *x* axis showing part of the (100) net.

Experimental

Lithium phosphoenolpyruvate was prepared by the reaction of phosphoenolpyruvic acid with lithium carbonate in an aqueous environment. Crystals were obtained by slow evaporation from water at 277 K and afterwards ball-shaped by the solvent.

Crystal data

$\mathrm{Li^+\cdot C_3H_4O_6P^-\cdot H_2O}$
$M_r = 191.99$
Triclinic, $P\overline{1}$
$a = 5.2281$ (7) Å
$b = 5.5511$ (7) Å
$c = 12.009$ (1) Å
$\alpha = 94.59$ (1)°
$\beta = 90.36$ (1)°
$\gamma = 94.91$ (1)°
$V = 346.10$ (7) Å^3

$Z = 2$
$D_x = 1.842$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 53 reflections
$\theta = 7.5$–17.0°
$\mu = 0.392$ mm^{-1}
$T = 85$ (2) K
Ball, colourless
0.55 × 0.55 × 0.55 mm

Data collection

Kuma KM-4 diffractometer
$\omega/2\theta$ scans
8521 measured reflections
8272 independent reflections
6788 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.017$
$\theta_{\rm max} = 55.16°$

$h = -12 \rightarrow 0$
$k = -12 \rightarrow 12$
$l = -27 \rightarrow 27$
3 standard reflections every 100 reflections intensity decay: 6.2%

Table 1
Selected geometric parameters (Å, °).

P—O1	1.5657 (4)	C2—C1	1.4880 (5)
P—O2	1.4903 (3)	C2—C3	1.3327 (5)
P—O3	1.4916 (3)	C1—O5	1.3085 (5)
P—O4	1.6200 (3)	C1—O6	1.2292 (5)
O4—C2	1.3685 (5)		
O1—P—O2	108.51 (2)	O4—C2—C1	110.68 (3)
O1—P—O3	111.59 (2)	O4—C2—C3	127.08 (3)
O1—P—O4	104.28 (2)	C3—C2—C1	122.22 (3)
O2—P—O3	118.43 (2)	C2—C1—O5	114.00 (3)
O2—P—O4	103.19 (2)	C2—C1—O6	121.40 (3)
O3—P—O4	109.63 (2)	O5—C1—O6	124.59 (4)
P—O4—C2	124.28 (3)		
O1—P—O4—C2	−74.39 (3)	O4—C2—C1—O5	173.81 (3)
O2—P—O4—C2	172.28 (3)	O4—C2—C1—O6	−7.11 (5)
O3—P—O4—C2	45.21 (4)	C3—C2—C1—O5	−7.91 (6)
P—O4—C2—C1	−158.78 (3)	C3—C2—C1—O6	171.17 (4)
P—O4—C2—C3	23.04 (6)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O5—H5⋯O6$^{\rm i}$	0.85 (1)	1.78 (1)	2.634 (1)	177 (1)
O1—H1⋯O7$^{\rm ii}$	0.89 (2)	1.78 (2)	2.655 (1)	171 (1)
O7—H71⋯O2$^{\rm iii}$	0.87 (1)	1.84 (1)	2.699 (1)	172 (1)

Symmetry codes: (i) $-1 - x, 1 - y, -z$; (ii) $1 - x, 2 - y, 1 - z$; (iii) $1 - x, 1 - y, 1 - z$.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.079$
$S = 1.20$
8272 reflections
133 parameters
All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.0319P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.058$
$\Delta\rho_{\rm max} = 0.72$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.78$ e Å^{-3}

The refined C—H distances are 0.919 (13) and 0.983 (13) Å.

Data collection: *Kuma KM*-4 *Software* (Kuma Diffraction, 1989); cell refinement: *Kuma KM*-4 *Software*; data reduction: *Kuma KM*-4 *Software*; program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*-3 (Johnson *et al.*, 1997); software used to prepare material for publication: *SHELXL*93.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD1096). Services for accessing these data are described at the back of the journal.

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