

except the disordered methyl protons, were located on a difference Fourier map and refined using isotropic thermal parameters. Final number of parameters refined = 273, $R = 0.021$, $wR = 0.029$, $S = 1.446$; secondary-extinction coefficient (Zachariassen, 1963) was included and refined to 2.548×10^{-7} . Maximum shift/e.s.d. on final cycle = 0.05; largest peak on final difference Fourier map = $0.74 \text{ e } \text{Å}^{-3}$. Atomic scattering factors and anomalous corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV).

A drawing of the title compound with the atom-numbering scheme is shown in Fig. 1. Atomic coordinates and final equivalent isotropic thermal parameters of all non-H atoms included in the refinement are given in Table 1.* Selected interatomic distances and angles are listed in Table 2.

* Lists of structure factors, anisotropic thermal parameters, least-squares-planes data, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53442 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Monosodium Phosphoenolpyruvate Hydrate: New Diffractometer Data

BY T. LIS

Instytut Chemii, Uniwersytet, 50-383 Wrocław, Poland

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Abstract. $\text{Na}^+\cdot\text{C}_3\text{H}_4\text{O}_6\text{P}^-\cdot\text{H}_2\text{O}$, $M_r = 208.04$, monoclinic, Cc , $a = 11.426$ (7), $b = 23.274$ (22), $c = 6.620$ (5) Å, $\beta = 123.66$ (5)°, $V = 1465$ (3) Å³, $Z = 8$, $D_x = 1.886$ (3) g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 4.44$ cm⁻¹, $F(000) = 848$, $T = 303$ (2) K, final $R = 0.034$ for 3357 observed data including both hkl and $\bar{h}\bar{k}\bar{l}$ reflections. The two crystallographically independent anions have slightly different configurations with P—O(ester) bond lengths of 1.606 (3) and 1.618 (3) Å.

Experimental. The structure of sodium phosphoenolpyruvate hydrate was determined by Katti, Hosur & Viswamitra (1981) from 1697 diffractometer data with final $R = 0.063$. A fragment of $0.25 \times 0.2 \times 0.5$ mm was cut from a larger crystal and mounted on a Syntex $P2_1$ diffractometer. Mo $K\alpha$ radiation and graphite monochromator were used for lattice parameters (15 reflections in the range $24 < 2\theta < 27^\circ$) and intensity measurements. 5984 intensities were

The fulvalene ligand is essentially planar with only a 6° twist about the bond connecting C1 and C6. The metals adopt the *trans* orientation found in all fulvalene complexes in which there is no $M-M$ bond, to minimize steric interactions between the respective ligand coordination spheres.

This crystal structure was solved at the X-ray Crystallographic Facility (CHEXRAY) at the Department of Chemistry, University of California, Berkeley, as part of the requirements of a graduate chemistry course on structure analysis by X-ray diffraction. We thank Mr Tim Karpishin, Dr F. Hollander and Professor K. N. Raymond for their guidance.

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measured, $2\theta \leq 65^\circ$, $h - 10 \rightarrow 17$, $k 0 \rightarrow 35$, $l - 9 \rightarrow 9$, from $\omega/2\theta$ scans. After each group of 50 reflections two standards were measured; variation $\pm 5\%$. Scattering factors for Na⁺, P, C, O and H were from *International Tables for X-ray Crystallography* (1974, Vol. IV); real and imaginary dispersion corrections included for all non-H atoms. No absorption or extinction corrections. The refinement was started with the published coordinates of Katti *et al.* (1981). Merging in point group m yielded 3871 reflections, 3357 with $I > 3\sigma(I)$ (including 1212 Friedel pairs) were used for refinement; $R_{\text{merge}} = 0.017$. Full-matrix refinement on F (Na, P, C, O anisotropic, H isotropic) with $w = 1/\sigma^2(F_o)$ using *SHELX76* (Sheldrick, 1976) gave final $R = 0.0332$ and $wR = 0.0307$ (refinement of the inverted structure gave $R = 0.0337$ and $wR = 0.0311$); max $\Delta/\sigma = 0.05$. The positional parameters of the H atoms were refined with constraints $d(\text{C—H}) = 1.08$ and $d(\text{O—H}) = 0.97$ Å. Residual electron density in final difference map

Table 1. Final atomic parameters for sodium phosphoenolpyruvate hydrate

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}/U_{iso}(\text{\AA}^2)$
Na(1)	0.23725 (16)	0.49590 (6)	0.37171 (29)	0.0346 (3)
Na(2)	0.43542 (16)	0.04172 (6)	0.35119 (31)	0.0400 (4)
P(1)	0.24 (fixed)	0.37468 (3)	0.09739 (18)	0.0205 (2)
P(2)	0.01283 (10)	-0.39169 (3)	0.8726 (fixed)	0.0213 (2)
C(11)	0.59735 (31)	0.36264 (13)	0.66136 (52)	0.0252 (6)
C(12)	0.45135 (31)	0.33875 (12)	0.53107 (53)	0.0236 (6)
C(13)	0.41948 (36)	0.28694 (15)	0.56364 (67)	0.0376 (8)
C(21)	-0.33942 (29)	-0.36235 (12)	0.72277 (54)	0.0227 (6)
C(22)	-0.19046 (28)	-0.34299 (12)	0.89407 (53)	0.0223 (6)
C(23)	-0.15309 (35)	-0.29100 (14)	0.98631 (71)	0.0369 (8)
O(11)	0.13817 (23)	0.32414 (9)	0.05696 (42)	0.0305 (5)
O(12)	0.16731 (21)	0.43119 (9)	0.03169 (40)	0.0256 (5)
O(13)	0.31276 (22)	0.35752 (10)	-0.02462 (42)	0.0306 (6)
O(14)	0.35294 (23)	0.38038 (8)	0.38466 (39)	0.0271 (5)
O(15)	0.62338 (25)	0.41013 (10)	0.62201 (44)	0.0375 (6)
O(16)	0.69339 (23)	0.32656 (10)	0.82224 (41)	0.0311 (5)
O(21)	0.12170 (21)	-0.34197 (9)	1.01015 (40)	0.0272 (5)
O(22)	0.07490 (23)	-0.45010 (9)	0.94676 (43)	0.0304 (5)
O(23)	-0.06308 (22)	-0.37631 (10)	0.60785 (40)	0.0288 (5)
O(24)	-0.09533 (21)	-0.38810 (8)	0.95744 (41)	0.0256 (5)
O(25)	-0.36793 (23)	-0.40997 (9)	0.63279 (43)	0.0320 (5)
O(26)	-0.43190 (22)	-0.32310 (9)	0.68022 (44)	0.0312 (6)
O(7)	0.42607 (25)	0.50758 (10)	0.31348 (44)	0.0352 (6)
O(8)	0.78900 (25)	0.49982 (10)	0.96970 (45)	0.0339 (6)
H(13)	0.310 (2)	0.275 (2)	0.471 (7)	0.050 (12)
H(131)	0.504 (3)	0.260 (2)	0.699 (5)	0.038 (10)
H(23)	-0.047 (2)	-0.277 (2)	1.117 (5)	0.050 (12)
H(231)	-0.236 (3)	-0.261 (2)	0.940 (8)	0.060 (13)
H(11)	0.058 (5)	0.337 (3)	0.059 (13)	0.158 (30)
H(16)	0.780 (3)	0.348 (2)	0.925 (7)	0.065 (14)
H(21)	0.198 (3)	-0.346 (2)	1.179 (2)	0.046 (12)
H(26)	-0.529 (2)	-0.335 (2)	0.600 (7)	0.055 (13)
H(7)	0.477 (4)	0.540 (1)	0.419 (6)	0.057 (13)
H(71)	0.481 (4)	0.473 (1)	0.383 (7)	0.069 (15)
H(8)	0.733 (4)	0.525 (2)	0.830 (5)	0.061 (14)
H(81)	0.740 (4)	0.464 (1)	0.943 (6)	0.061 (14)

Table 2. Principal bond lengths (Å), bond angles (°), torsion angles (°), sodium coordination (Å) and H-bonding data in sodium phosphoenolpyruvate hydrate

P(1)—O(11)	1.570 (3)	P(2)—O(21)	1.568 (3)
P(1)—O(12)	1.486 (3)	P(2)—O(22)	1.485 (3)
P(1)—O(13)	1.499 (3)	P(2)—O(23)	1.506 (3)
P(1)—O(14)	1.606 (3)	P(2)—O(24)	1.618 (3)
O(14)—C(12)	1.391 (4)	O(24)—C(22)	1.398 (4)
C(11)—C(12)	1.497 (5)	C(21)—C(22)	1.499 (5)
C(12)—C(13)	1.312 (5)	C(22)—C(23)	1.315 (4)
C(11)—O(15)	1.210 (4)	C(21)—O(25)	1.214 (4)
C(11)—O(16)	1.322 (4)	C(21)—O(26)	1.304 (4)
O(11)—P(1)—O(12)	111.8 (2)	O(21)—P(2)—O(22)	113.8 (2)
O(11)—P(1)—O(13)	106.8 (2)	O(21)—P(2)—O(23)	105.1 (2)
O(11)—P(1)—O(14)	107.3 (2)	O(21)—P(2)—O(24)	105.8 (2)
O(12)—P(1)—O(13)	117.7 (2)	O(22)—P(2)—O(23)	117.6 (2)
O(12)—P(1)—O(14)	103.0 (2)	O(22)—P(2)—O(24)	104.4 (2)
O(13)—P(1)—O(14)	109.8 (2)	O(23)—P(2)—O(24)	109.5 (2)
P(1)—O(14)—C(12)	125.2 (3)	P(2)—O(24)—C(22)	123.4 (3)
O(14)—C(12)—C(11)	110.8 (3)	O(24)—C(22)—C(21)	111.7 (3)
O(14)—C(12)—C(13)	124.3 (4)	O(24)—C(22)—C(23)	123.6 (4)
C(11)—C(12)—C(13)	124.7 (4)	C(21)—C(22)—C(23)	124.6 (4)
C(12)—C(11)—O(15)	122.9 (4)	C(22)—C(21)—O(25)	121.9 (3)
C(12)—C(11)—O(16)	113.0 (3)	C(22)—C(21)—O(26)	113.4 (3)
O(15)—C(11)—O(16)	124.1 (4)	O(25)—C(21)—O(26)	124.6 (4)
P(1)—O(11)—H(11)	113 (5)	P(2)—O(21)—H(21)	121.3 (18)
C(11)—O(16)—H(16)	108.4 (22)	C(21)—O(26)—H(26)	117.0 (21)
C(13)—C(12)—C(11)—O(15)	177.9 (9)	C(23)—C(22)—C(21)—O(25)	176.0 (9)
C(13)—C(12)—C(11)—O(16)	-2.1 (9)	C(23)—C(22)—C(21)—O(26)	-3.1 (8)
C(13)—C(12)—O(14)—P(1)	-61.6 (9)	C(23)—C(22)—O(24)—P(2)	-72.4 (9)
C(11)—C(12)—O(14)—P(1)	123.0 (8)	C(21)—C(22)—O(24)—P(2)	111.5 (8)
C(12)—O(14)—P(1)—O(11)	64.3 (7)	C(22)—O(24)—P(2)—O(21)	68.4 (7)
C(12)—O(14)—P(1)—O(12)	-177.5 (6)	C(22)—O(24)—P(2)—O(22)	-171.2 (6)
C(12)—O(14)—P(1)—O(13)	-51.3 (7)	C(22)—O(24)—P(2)—O(23)	-44.4 (7)
O(15)—C(11)—C(12)—O(14)	-6.7 (8)	O(25)—C(21)—C(22)—O(24)	-7.9 (7)
O(16)—C(11)—C(12)—O(14)	173.2 (7)	O(26)—C(21)—C(22)—O(24)	173.0 (7)
O(14)—P(1)—O(11)—H(11)	89 (6)	O(24)—P(2)—O(21)—H(21)	78 (2)
C(12)—C(11)—O(16)—H(16)	-167 (3)	C(22)—C(21)—O(26)—H(26)	-167 (3)

Table 2 (cont.)

Na(1)—O(12)	2.445 (3)	Na(2)—O(12 ^{iv})	2.310 (3)
O(12)	2.362 (3)	O(22 ^v)	2.524 (3)
O(14)	2.976 (3)	O(23 ^{vi})	2.548 (3)
O(22 ^{iv})	2.674 (3)	O(24 ^{vii})	2.928 (3)
O(22 ⁱⁱⁱ)	2.418 (3)	O(8 ^{viii})	2.333 (3)
O(7)	2.408 (4)	O(8 ^{ix})	2.409 (4)
O(7')	2.494 (3)		

Symmetry code: (i) $x, 1-y, 0.5+z$; (ii) $x, 1+y, z-1$; (iii) $x, -y, z-0.5$; (iv) $0.5+x, 0.5-y, 0.5+z$; (v) $0.5+x, -0.5-y, z-0.5$; (vi) $0.5+x, 0.5+y, z$; (vii) $0.5+x, 0.5+y, z-1$; (viii) $x-0.5, y-0.5, z-1$; (ix) $x-0.5, 0.5-y, z-0.5$.

O—H...O	O...O	H...O	O—H...O
O(11)—H(11)...O(23 ⁱ)	2.781 (4)	1.83 (7)	165 (7)
O(16)—H(16)...O(23 ⁱⁱ)	2.613 (4)	1.65 (4)	174 (4)
O(21)—H(21)...O(13 ⁱⁱⁱ)	2.632 (4)	1.67 (2)	172 (3)
O(26)—H(26)...O(13 ^{iv})	2.570 (4)	1.60 (4)	178 (4)
O(7)—H(7)...O(25 ^v)	2.859 (4)	1.93 (3)	159 (3)
O(7)—H(71)...O(15)	3.049 (4)	2.10 (3)	164 (3)
O(8)—H(8)...O(25 ^v)	2.857 (4)	1.92 (3)	162 (3)
O(8)—H(81)...O(15)	2.900 (4)	2.17 (3)	131 (3)

Symmetry code: (i) $x, -y, z-0.5$; (ii) $1+x, -y, z+0.5$; (iii) $x, -y, 1.5+z$; (iv) $x-1, -y, z+0.5$; (v) $x+1, y+1, z$.

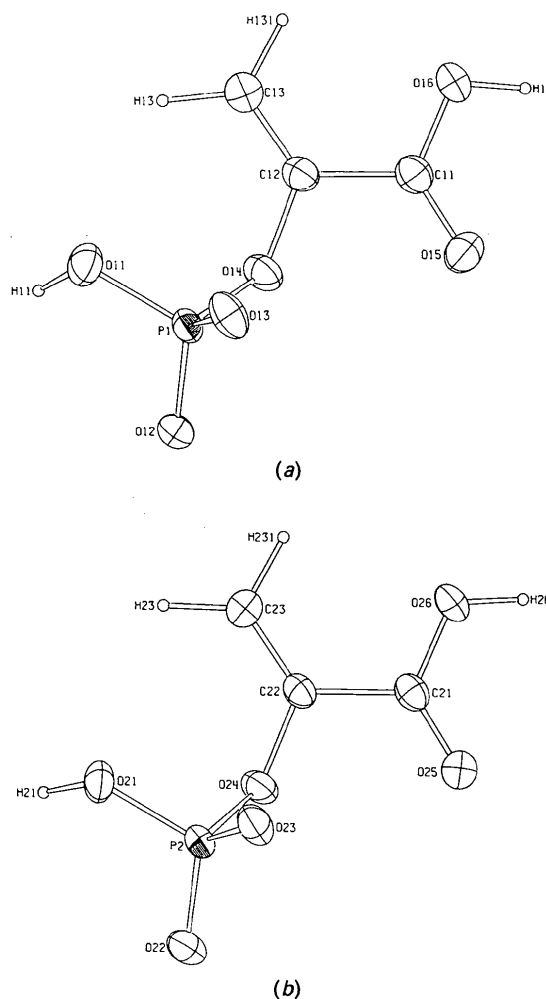


Fig. 1. View of two crystallographically independent monoanions perpendicular to the pyruvate group.

between -0.50 and $0.76 \text{ e} \text{ \AA}^{-3}$. The final atomic parameters are given in Table 1.* The principal interatomic distances and angles are given in Table 2. The view showing the structure of two crystallographically independent anions is shown in Fig. 1 and the packing of the crystal in Fig. 2.

Related literature. This redetermination has yielded e.s.d.'s about half those achieved by Katti *et al.* (1981). The structures of phosphoenolpyruvic acid (PEP) and of the anions PEP^- , PEP^{2-} and PEP^{3-} are therefore now known to similar precision since the structures of two polymorphic forms of cyclohexylammonium phosphoenolpyruvate were given by Weichsel & Lis (1989a) and that of the potassium salt by Lis (1987) while phosphoenolpyruvic acid itself has been described by Weichsel & Lis (1989b).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53393 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

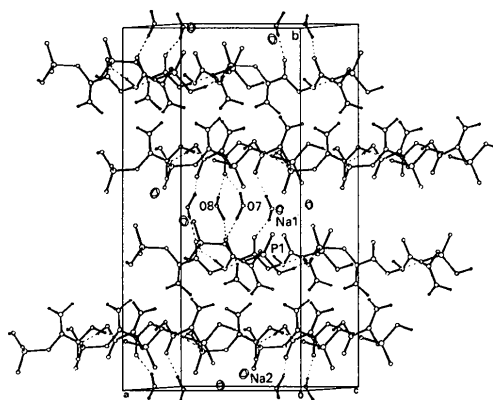


Fig. 2. Packing arrangement and hydrogen bonds in sodium phosphoenolpyruvate hydrate.

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Structure of (2,3,7,8,12,13,17,18-Octaethylporphinato)copper(II)

BY ROGER PAK AND W. ROBERT SCHEIDT*

Department of Chemistry and Biochemistry, University of Notre Dame, Notre Dame, Indiana 46556, USA

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Abstract. $[\text{Cu}(\text{C}_{36}\text{H}_{44}\text{N}_4)]$, $M_r = 596.3$, triclinic, $P\bar{1}$, $a = 13.314(5)$, $b = 13.392(5)$, $c = 4.805(3) \text{ \AA}$, $\alpha = 92.42(4)^\circ$, $\beta = 93.38(4)^\circ$, $\gamma = 113.08(1)^\circ$, $V = 784.8 \text{ \AA}^3$, $Z = 1$, $D_x = 1.26 \text{ g cm}^{-3}$, $D_m = 1.25 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$, $\mu = 7.25 \text{ cm}^{-1}$, $F(000) = 317$, $T = 293 \text{ K}$, $R = 0.055$ for 2166 unique observed reflections.

Experimental. Crystals of the title compound were prepared by slow evaporation of a chloroform solution. D_m was measured by flotation in $\text{CCl}_4/\text{hexane}$. A purple, air-stable crystal with approximate dimensions of $0.41 \times 0.05 \times 0.04 \text{ mm}$ was mounted on the end of a glass fiber. All measurements were per-

formed with graphite-monochromated Mo $K\alpha$ radiation on an Enraf–Nonius CAD-4 diffractometer. Intensity data were measured by θ – 2θ scans at scan rates of 2 – $16^\circ \text{ min}^{-1}$. Data were collected to a maximum 2θ of 58.7° and standard θ – 2θ CAD-4 moving-crystal moving-counter background measurements were used. 25 reflections used for measuring lattice parameters $9.23 < \theta < 12.17^\circ$. Range of hkl : $-16 \rightarrow 16$, $-16 \rightarrow 16$, $-6 \rightarrow 6$, 9147 reflections measured, 4305 unique, 2166 with $I > 3\sigma(I)$ considered observed. Merging $R = 0.031$ for 4297 duplicates. Four standard reflections, 9.7% intensity decrease. The data were corrected for background (profile analysis), Lorentz, polarization, crystal decay and absorption effects using the Blessing (1987) suite for data reduction. Empirical absorption correction was

* To whom correspondence should be addressed.