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Barium D-Glucose 6-Phosphate Heptahydrate: New Diffractometer Data

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Abstract. $Ba^{2+}.C_6H_{11}O_9P^{2-}.7H_2O$, $M_r = 521{\cdot}6$, monoclinic, $P2_1$, $a = 11{\cdot}881$ (6), $b = 8{\cdot}621$ (4), $c = 8{\cdot}345$ (5) Å, $\beta = 102{\cdot}88$ (4)°, $V = 833{\cdot}2$ (8) Å^3, $Z = 2$, $D_x = 2{\cdot}079$ (3) g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 26{\cdot}3$ cm^{-1}, $F(000) = 520$, $T = 289$ (1) K, final $R = 0{\cdot}0248$ for 5521 observed data including both hkl and $\bar{h}k\bar{l}$ reflections. The two endocyclic C—O bonds in the sugar ring C(5)—O(5) [1·432 (4) Å] and C(1)—O(5) [1·437 (4) Å] have similar bond lengths. The phosphate ester bond, P—O(6), is 1·615 (3) Å. The Cremer–Pople puckering parameters are: $\theta = 5{\cdot}2$ (4)°, $Q = 0{\cdot}595$ (4) Å and $\varphi = 271$ (4)°. There is an intramolecular hydrogen bond between the sugar hydroxyl O(4) and phosphate O(8) atoms of length 2·664 (4) Å.

Experimental. The structure of barium D-glucose 6-phosphate heptahydrate was determined by Katti, Seshadri & Viswamitra (1982) on the basis of 1603 diffractometer data and final $R = 0{\cdot}068$. They found that, of the two endocyclic C—O bonds in the glucose ring, C(5)—O(5) [1·463 (23) Å] is longer than C(1)—O(5) [1·395 (23) Å]. This was in contrast with the situation observed in monosodium (Lis, 1985; Narendra & Viswamitra, 1985) and bis(cyclohexylammonium) (Lis, 1990) salts of D-glucose 6-phosphate. Furthermore, since the oxygen-bonded H atoms were not located, the H-bonding scheme was not accurately described. Therefore, it was decided to reinvestigate this structure. The compound was obtained by adding a $BaCl_2$ water solution to a disodium D-glucose 6-phosphate water solution. The crystals precipitated as long needles. A fragment crystal 0·07 × 0·3 × 0·2 mm was cut from a larger one and mounted on a $P2_1$ diffractometer. Mo $K\alpha$ radiation and graphite monochromator were used for lattice parameters (15 reflections in the range $21 < 2\theta < 29°$) and the intensity measurements. 7030 reflections were measured below $2\theta = 65°$ ($-17 \le h \le -12$ and $0 \le h \le 17$, $-13 \le k \le 13$, $-12 \le l \le 12$) operating in $2\theta/\theta$ scan technique. After each group of 50 reflections two standards were measured; variation ± 4%. Scattering factors for Ba^{2+}, P, O, C and H were from *International Tables for X-ray Crystallography* (1974, Vol. IV); real and imaginary dispersion corrections included for all non-H atoms. The refinement was started with the published coordinates of Katti, Seshadri & Viswamitra (1982). The C-bonded H atoms were included in geometrically calculated positions with d(C—H) = 1·08 Å. The remaining H atoms were found from difference Fourier synthesis and refined with constraints that d(O—H) = 0·97 Å. An absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) was applied; min. and max. absorption corrections: 0·855 and 1·113. Symmetry-related reflections were averaged after *DIFABS* to give 5521 data (Friedel pairs were not averaged) with $I > 3\sigma(I)$; $R_{int} = 0{\cdot}0166$. Final refinement was performed (on F) with *SHELX*76 (Sheldrick, 1976), using anisotropic thermal parameters, isotropic for H atoms. $\sum w(|F_o| - |F_c|)^2$ minimized, $w = 1/\sigma^2(F_o)$. Final $R = 0{\cdot}0248$, $wR = 0{\cdot}0235$ for 5521 reflections and 290 refined parameters; $(\Delta/\sigma)_{max} = 0{\cdot}3$, minimum and maximum heights in difference Fourier map 3·1 and −1·5 e Å^{-3}.

Table 1. *Final atomic parameters for barium* D-*glucose* 6-*phosphate heptahydrate*

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

	x	y	z	U_{eq}(Å^2)
Ba	0·06613 (1)	0·27†	0·43134 (2)	0·0193 (1)
P	0·23316 (7)	0·50031 (10)	0·13488 (10)	0·0200 (2)
O(1)	0·4375 (2)	1·1932 (3)	0·3297 (4)	0·0333 (6)
O(2)	0·6684 (2)	1·0858 (3)	0·4353 (4)	0·0267 (5)
O(3)	0·68976 (17)	0·7523 (4)	0·4309 (3)	0·0271 (5)
O(4)	0·5365 (2)	0·6019 (3)	0·1698 (4)	0·0329 (6)
O(5)	0·3869 (2)	0·9713 (3)	0·1866 (4)	0·0287 (5)
O(6)	0·2529 (2)	0·6856 (3)	0·1321 (4)	0·0283 (5)
O(7)	0·1896 (2)	0·4806 (4)	0·2909 (4)	0·0255 (5)
O(8)	0·3500 (2)	0·4235 (3)	0·1413 (4)	0·0367 (6)
O(9)	0·1446 (3)	0·4569 (4)	−0·0183 (3)	0·0371 (6)
C(1)	0·4815 (3)	1·0727 (4)	0·2548 (4)	0·0258 (6)
C(2)	0·5714 (3)	0·9858 (4)	0·3834 (4)	0·0224 (5)
C(3)	0·6078 (2)	0·8367 (4)	0·3107 (4)	0·0217 (5)
C(4)	0·5016 (2)	0·7392 (3)	0·2387 (4)	0·0233 (6)
C(5)	0·4211 (3)	0·8370 (4)	0·1083 (4)	0·0229 (6)
C(6)	0·3132 (2)	0·7521 (5)	0·0182 (4)	0·0261 (6)
W(1)	0·8898 (3)	0·4959 (4)	0·3705 (4)	0·0288 (6)
W(2)	0·9431 (2)	0·2542 (5)	0·6725 (4)	0·0421 (6)
W(3)	0·9786 (2)	0·2858 (6)	0·0982 (3)	0·0452 (7)
W(4)	0·1967 (3)	0·1384 (4)	0·9336 (5)	0·0519 (7)
W(5)	0·1635 (3)	0·0393 (4)	0·2624 (5)	0·0374 (7)
W(6)	0·6808 (3)	0·4082 (4)	0·4253 (6)	0·0542 (9)
W(7)	0·8846 (3)	0·0350 (4)	0·3380 (4)	0·0281 (6)

† Fixed.

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Table 2. *Bond lengths* (Å), *bond angles* (°), *torsion angles* (°) *and barium coordination distances* (Å) *in barium* D-*glucose 6-phosphate heptahydrate*

O(1)—C(1)	1·375 (4)	C(2)—C(3)	1·525 (4)
O(2)—C(2)	1·427 (4)	C(3)—C(4)	1·524 (4)
O(3)—C(3)	1·431 (4)	C(4)—C(5)	1·531 (5)
O(4)—C(4)	1·418 (4)	C(5)—C(6)	1·522 (5)
O(5)—C(5)	1·432 (4)	P—O(6)	1·615 (3)
O(5)—C(1)	1·437 (4)	P—O(7)	1·514 (3)
O(6)—C(6)	1·431 (4)	P—O(8)	1·528 (3)
C(1)—C(2)	1·530 (5)	P—O(9)	1·510 (3)
O(6)—P—O(7)	101·6 (2)	O(2)—C(2)—C(3)	110·4 (3)
O(6)—P—O(8)	107·2 (2)	C(1)—C(2)—C(3)	110·4 (3)
O(6)—P—O(9)	108·0 (2)	O(3)—C(3)—C(2)	111·1 (3)
O(7)—P—O(8)	113·8 (2)	O(3)—C(3)—C(4)	111·7 (3)
O(7)—P—O(9)	113·1 (2)	C(2)—C(3)—C(4)	109·8 (3)
O(8)—P—O(9)	112·2 (2)	O(4)—C(4)—C(3)	109·2 (3)
P—O(6)—C(6)	120·1 (3)	O(4)—C(4)—C(5)	111·3 (3)
C(1)—O(5)—C(5)	113·3 (3)	C(3)—C(4)—C(5)	107·9 (3)
O(1)—C(1)—O(5)	107·2 (3)	O(5)—C(5)—C(4)	108·4 (3)
O(1)—C(1)—C(2)	109·1 (3)	O(5)—C(5)—C(6)	108·7 (3)
O(5)—C(1)—C(2)	110·0 (3)	C(4)—C(5)—C(6)	114·2 (3)
O(2)—C(2)—C(1)	107·9 (3)	O(6)—C(6)—C(5)	110·9 (3)
O(7)—P—O(6)—C(6)	169·2 (3)	P—O(6)—C(6)—C(5)	−119·6 (5)
O(8)—P—O(6)—C(6)	49·6 (4)	O(6)—C(6)—C(5)—O(5)	−68·2 (5)
O(9)—P—O(6)—C(6)	−71·6 (4)	O(6)—C(6)—C(5)—C(4)	53·0 (5)
Ba—O(3^{i})	2·876 (3)	Ba—O(7)	2·754 (3)
W(1^{ii})	2·823 (4)	*W*(1^{i})	2·865 (4)
W(2^{ii})	2·742 (3)	*W*(3^{iii})	2·746 (3)
W(5)	2·830 (4)	*W*(7^{iii})	2·934 (3)
W(7^{iii})	2·961 (3)		

Symmetry code: (i) $1-x$, $y-0{\cdot}5$, $1-z$; (ii) $x-1$, y, z; (iii) $1-x$, $y+0{\cdot}5$, $1-z$.

Final atom parameters are summarized in Table 1.* The overall molecular configuration and the atom-numbering scheme of the dianion are shown in

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and hydrogen-bond data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53488 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

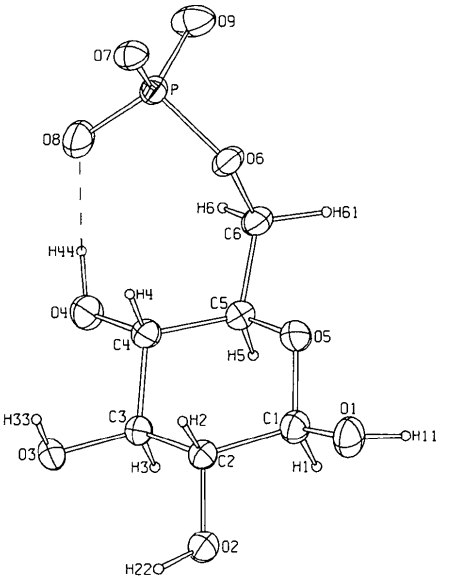

Fig. 1. The structure of the D-glucose 6-phosphate dianion, showing the numbering of the atoms in the barium salt.

Fig. 1. Principal interatomic distances, bond and torsion angles are given in Table 2.

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Structure of (μ-Sulfur dioxide)bis(dicarbonylcyclopentadienyliron) Hydrate

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Abstract. $[Fe_2(CO)_4(SO_2)(C_5H_5)_2].H_2O$, $M_r = 436{\cdot}0$, monoclinic, $P2_1/n$, $a = 10{\cdot}884$ (2), $b = 12{\cdot}424$ (1), $c = 12{\cdot}658$ (5) Å, $\beta = 103{\cdot}35$ (3)°, $V = 1665{\cdot}3$ (13) Å^3, $Z = 4$, $D_x = 1{\cdot}74$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 18{\cdot}9$ cm^{-1}, $F(000) = 880$, $T = 293$ K, $R = 0{\cdot}0355$ for 1455 reflections with $F_o^2 > 3\sigma(F_o^2)$. Hydrogen bonds

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