Table 2. *Selected interatomic distances (Å) and angles (°)*

Pt—I	2·626 (2)	Pt—P	2·292 (6)
P—C(11)	1·823 (25)	P—C(21)	1·834 (23)
P—C(31)	1·836 (23)	Cl(1)—C	1·658 (39)
Cl(2)—C	1·828 (35)		

	Range	Mean
C—F	1·309 (26)–1·365 (29)	1·340 (7)
C—C	1·332 (36)–1·431 (34)	1·380 (8)

I—Pt—P	85·1 (2)	Pt—P—C(11)	118·0 (8)
Pt—P—C(21)	119·6 (8)	Pt—P—C(31)	106·8 (8)
C(11)—P—C(21)	98·0 (11)	C(11)—P—C(31)	108·5 (12)
C(21)—P—C(31)	104·9 (11)	P—C(11)—C(12)	124·4 (17)
P—C(11)—C(16)	119·9 (19)	C(12)—C(11)—C(16)	115·4 (21)
P—C(21)—C(22)	116·4 (16)	P—C(21)—C(26)	130·9 (18)
C(22)—C(21)—C(26)	112·7 (20)	P—C(31)—C(32)	116·1 (18)
P—C(31)—C(36)	126·6 (18)	C(32)—C(31)—C(36)	116·7 (21)
Cl(1)—C—Cl(2)	110·6 (19)		

C—C—F angles are in the range 116·1 (21)–123·0 (22)°. C—C—C angles not given above are in the range 117·4 (21)–124·6 (24)°.

References

ALCOCK, N. W. & LEVISTON, P. G. (1974). *J. Chem. Soc. Dalton Trans.* pp. 1834–1836.
ALYEA, E. C., DIAS, S. A., FERGUSON, G. & ROBERTS, P. J. (1979). *J. Chem. Soc. Dalton Trans.* pp. 948–951.
DOCHERTY, J. B., RYCROFT, D. S., SHARP, D. W. A. & WEBB, G. A. (1979). *J. Chem. Soc. Chem. Commun.* pp. 336–337.
HITCHCOCK, P. B., JACOBSON, B. & PIDCOCK, A. (1977). *J. Chem. Soc. Dalton Trans.* pp. 2038–2042.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
KEMMITT, R. D. W., NICHOLS, D. I. & PEACOCK, R. D. (1967). *J. Chem. Soc. Chem. Commun.* pp. 599–601.
MANOJLOVIĆ-MUIR, LJ. (1978). *Izvj. Jugosl. Cent. Krist. (Zagreb)*, 13, 21–31.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SOLOMUN, T. (1979). PhD Thesis, Univ. of Glasgow.
TOLMAN, C. A. (1977). *Chem. Rev.* 77, 313–348.

Structure of Disodium D-Fructose 6-Phosphate Heptahydrate

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Abstract. $2Na^+.C_6H_{11}O_9P^{2-}.7H_2O$, $M_r = 430·2$, orthorhombic, $P2_12_12_1$, $a = 9·856$ (6), $b = 11·007$ (6), $c = 16·361$ (8) Å, $V = 1775$ (3) Å³, $Z = 4$, $D_m = 1·62$, $D_x = 1·61$ (1) Mg m⁻³, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu = 0·29$ mm⁻¹, $F(000) = 904$, $T = 294$ (1) K, final $R = 0·040$ for 2500 non-zero reflexions. The length of the phosphate ester bond is 1·609 (2) Å. The furanose ring is in the ^2_3T conformation. The torsion angles O(6)—C(6)—C(5)—O(5) and O(6)—C(6)—C(5)—C(4) are 65·6 (5) and −174·5 (5)° and the conformation about the C(5)—C(6) bond is therefore *gauche–trans*. The crystal structure is stabilized by a three-dimensional hydrogen-bonding network.

Introduction. Different D-fructose phosphates are known to play important roles in living organisms. However, of four known fructose phosphates: fructose 1,6-diphosphate (F1,6-dp), fructose 6-phosphate (F6P), fructose 1-phosphate (F1P) and fructose 2,6-diphosphate (F2,6-dp) only the crystal structure of trisodium D-fructose 1,6-diphosphate octahydrate has been published (Narendra, Seshadri & Viswamitra, 1985). This paper presents the structure of $Na_2(F6P).7H_2O$. This investigation follows the author's earlier studies on sodium salts of D-glucose 6-phosphate and D-2-phosphoglycerate (Lis, 1985a,b).

Experimental. Crystals of disodium D-fructose 6-phosphate heptahydrate were obtained by slow concentration in a refrigerator ($\sim$273 K) of an aqueous solution of the commercially available compound (Boehringer). The compound crystallized as colorless multi-faceted, often rounded, crystals. D_m by flotation in 1,2-dibromoethane/CCl₄; preliminary examination by rotation and Weissenberg photographs. An irregular specimen, edge length 0·4 to 0·5 mm, was cut from a large crystal. Syntex $P2_1$ diffractometer, Mo $K\alpha$ radiation for lattice parameters (15 reflexions in the range $18 < 2\theta < 28°$) and intensity measurements; ω–2θ scan technique. 2919 reflexions measured below $\theta = 30°$; $h\ 0\rightarrow13$, $k\ 0\rightarrow15$, $l\ 0\rightarrow23$; after every 50 reflexions two standards measured, variation 5%; most calculations performed on a Nova 1200 computer with locally modified *XTL/XTLE* programs (Syntex, 1976); neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); real and imaginary components of anomalous dispersion included for all non-H atoms. Structure solved by direct methods; H atoms from difference synthesis; refinement based on F values; Na, P, O, C anisotropic, H isotropic; 2500 reflexions with $I > 2\sigma(I)$, $w = 1/\sigma^2(F_o)$ gave $R = 0·040$ and $wR = 0·038$; max. $\Delta/\sigma\ 0·14$. No absorption and extinction corrections. Residual electron

Table 1. *Positional and equivalent isotropic thermal parameters* (Å^2) *for non-H atoms with e.s.d.'s in parentheses*

$$B_{eq} = \frac{1}{3}(B_{11} + B_{22} + B_{33}).$$

	x	y	z	B_{eq}
P	0·95562 (8)	0·51017 (7)	0·54745 (6)	1·48 (4)
Na(1)	0·3231 (2)	0·5784 (2)	0·30421 (9)	2·56 (10)
Na(2)	0·3829 (2)	0·2779 (2)	0·68291 (9)	3·13 (12)
C(1)	0·3070 (4)	0·7107 (3)	0·5381 (3)	1·63 (20)
C(2)	0·3983 (4)	0·6165 (3)	0·4995 (2)	1·43 (19)
C(3)	0·3357 (3)	0·4875 (3)	0·4944 (2)	1·31 (18)
C(4)	0·4587 (4)	0·4042 (3)	0·4965 (2)	1·52 (19)
C(5)	0·5644 (3)	0·4776 (3)	0·5433 (3)	1·46 (18)
C(6)	0·7014 (4)	0·4769 (4)	0·5014 (3)	1·66 (20)
O(1)	0·2617 (3)	0·6763 (3)	0·6174 (2)	1·85 (16)
O(2)	0·4373 (3)	0·6527 (3)	0·4202 (2)	2·11 (17)
O(3)	0·2534 (3)	0·4692 (3)	0·4244 (2)	1·71 (15)
O(4)	0·4295 (3)	0·2930 (3)	0·5373 (2)	2·10 (17)
O(5)	0·5132 (3)	0·6000 (2)	0·5506 (2)	1·75 (14)
O(6)	0·7957 (3)	0·5378 (3)	0·5538 (2)	1·87 (15)
O(7)	1·0013 (3)	0·5299 (3)	0·4597 (2)	2·11 (16)
O(8)	0·9784 (3)	0·3806 (3)	0·5752 (2)	2·52 (19)
O(9)	1·0129 (3)	0·6047 (3)	0·6056 (2)	1·91 (16)
W(1)	0·1378 (4)	0·5080 (3)	0·2194 (2)	2·72 (20)
W(2)	0·3586 (4)	0·7178 (4)	0·1932 (3)	2·90 (22)
W(3)	0·5162 (4)	0·4493 (4)	0·2696 (2)	3·32 (25)
W(4)	0·1073 (5)	0·7146 (4)	0·3341 (3)	5·03 (34)
W(5)	0·4208 (4)	0·0690 (3)	0·6815 (2)	2·88 (22)
W(6)	0·2497 (5)	0·2314 (4)	0·3574 (3)	4·61 (32)
W(7)	0·2363 (5)	0·8845 (3)	0·7150 (3)	4·06 (27)

density in final difference Fourier map within -0.26 and 0.33 e Å^{-3}. H(W4) has $B_{iso} = -2.1$ (4) Å^2 and is the only H 'atom' not utilized in hydrogen bonding. Therefore, the W(4) water molecule may be partially disordered and the 'H(W4)' site partially occupied by the O atom.

The final atomic parameters are given in Table 1.*

Discussion. The crystals of the title compound are composed of Na$^+$ cations, D-fructose 6-phosphate dianions and water of hydration. The principal bond lengths, bond angles and torsion angles are summarized in Table 2. The molecular configuration and the atom-numbering scheme of the anion are illustrated in Fig. 1.

The furanose ring has the ^2_3T conformation which differs from the ^3E conformation found in the D-fructose 1,6-diphosphate trianion (Narendra *et al.*, 1985). The Cremer & Pople (1975) puckering parameters are $Q_2 = 0.329$ (4) Å and $\varphi_2 = 236.0$ (6)°. The torsion angles O(6)—C(6)—C(5)—O(5) and O(6)—C(6)—C(5)—C(4) are 65·6 (5) and -174.5 (5)° and the conformation about the exocyclic bond C(5)—C(6) is therefore *gauche–trans* and is similar to that in the F 1,6-dp

trianion. O(1) is *trans* to O(2) and *gauche* to O(5) and C(3) (Table 2) – as in the F 1,6-dp trianion. The lengths of the two endocyclic C—O bonds C(2)—O(5) and C(5)—O(5) [1·420 (4) and 1·443 (5) Å] are not the same. The C(2)—C(3) bond length of 1·550 (4) Å is longer than the other C—C bond lengths which range from 1·511 (5) to 1·525 (4) Å. The C—OH bonds vary from 1·410 (4) to 1·423 (4) Å.

Table 2. *Principal bond lengths* (Å), *bond angles* (°) *and torsion angles* (°)

C(1)—O(1)	1·423 (4)	C(1)—C(2)	1·511 (5)
C(2)—O(2)	1·410 (4)	C(2)—C(3)	1·550 (4)
C(3)—O(3)	1·418 (4)	C(3)—C(4)	1·520 (4)
C(4)—O(4)	1·423 (4)	C(4)—C(5)	1·525 (4)
C(5)—O(5)	1·443 (4)	C(5)—C(6)	1·515 (4)
C(2)—O(5)	1·420 (4)	C(6)—O(6)	1·431 (4)
P—O(6)	1·609 (2)	P—O(7)	1·521 (3)
P—O(8)	1·513 (3)	P—O(9)	1·518 (3)

O(1)—C(1)—C(2)	112·7 (3)	C(1)—C(2)—C(3)	114·4 (3)
C(1)—C(2)—O(5)	108·5 (3)	C(1)—C(2)—O(2)	110·7 (3)
C(3)—C(2)—O(5)	103·4 (3)	C(3)—C(2)—O(2)	108·5 (3)
O(5)—C(2)—O(2)	111·1 (3)	C(2)—C(3)—C(4)	103·5 (3)
C(2)—C(3)—O(3)	113·6 (3)	C(4)—C(3)—O(3)	112·9 (3)
C(3)—C(4)—C(5)	103·7 (3)	C(3)—C(4)—O(4)	111·7 (3)
C(5)—C(4)—O(4)	111·0 (3)	C(4)—C(5)—C(6)	112·3 (3)
C(4)—C(5)—O(5)	107·3 (3)	C(6)—C(5)—O(5)	110·7 (3)
C(2)—O(5)—C(5)	110·4 (3)	C(5)—C(6)—O(6)	107·8 (3)
C(6)—O(6)—P	120·6 (3)	O(6)—P—O(7)	108·9 (2)
O(6)—P—O(8)	107·7 (2)	O(6)—P—O(9)	101·2 (2)
O(7)—P—O(8)	112·0 (2)	O(7)—P—O(9)	112·6 (2)
O(8)—P—O(9)	113·7 (2)		

O(1)—C(1)—C(2)—O(2)	178·3 (5)	O(1)—C(1)—C(2)—O(5)	−59·6 (5)
O(1)—C(1)—C(2)—C(3)	55·3 (5)	C(1)—C(2)—C(3)—O(3)	85·1 (5)
C(1)—C(2)—C(3)—C(4)	−152·1 (5)	C(1)—C(2)—O(5)—C(5)	150·3 (5)
O(2)—C(2)—C(3)—O(3)	−39·1 (5)	O(2)—C(2)—C(3)—C(4)	83·7 (4)
O(2)—C(2)—O(5)—C(5)	−87·8 (5)	C(2)—C(3)—C(4)—O(4)	146·9 (5)
C(2)—C(3)—C(4)—C(5)	27·3 (5)	O(3)—C(3)—C(4)—O(4)	−89·8 (5)
O(3)—C(3)—C(4)—C(5)	150·6 (5)	O(3)—C(3)—C(2)—O(5)	−157·2 (5)
C(3)—C(4)—C(5)—C(6)	−133·1 (5)	C(3)—C(4)—C(5)—O(5)	−11·2 (5)
O(4)—C(4)—C(5)—O(5)	−131·2 (5)	O(4)—C(4)—C(5)—C(6)	106·9 (5)
C(4)—C(3)—C(2)—O(5)	−34·4 (5)	C(4)—C(5)—O(5)—C(2)	−11·3 (5)
C(4)—C(5)—C(6)—O(6)	−174·5 (5)	C(6)—C(5)—O(5)—C(2)	111·5 (6)
O(5)—C(5)—C(6)—O(6)	65·6 (5)	C(5)—O(5)—C(2)—C(3)	28·5 (5)
C(6)—O(6)—P—O(7)	54·6 (7)	C(6)—O(6)—P—O(8)	−67·0 (7)
C(6)—O(6)—P—O(9)	173·4 (6)	C(5)—C(6)—O(6)—P	156·0 (5)

Fig. 1. The molecular configuration and the atom-numbering scheme for the D-fructose 6-phosphate dianion.

The P—O(6) ester bond of 1·609 (2) Å in F6P and 1·622 (3) Å in F 1,6-dp do not differ significantly. The orientation of the phosphate group at position 6 relative to the sugar residue is similar in the two crystals, which is shown by the torsion angles P—O(6)—C(6)—C(5) of 156·0 (5) and 138·2 (3)° and C—O—P—O_t of 173·4 (6) and −173·9 (3)° for F6P and F 1,6-dp, respectively. (O_t denotes the terminal *trans* O atom with respect to the C—O—P—O torsion angle.) In the geometry of the —OPO_3^{2-} group in both compounds, the same characteristic features that can be found in almost all esterified doubly ionized phosphates are visible (Starynowicz & Lis, 1985), *e.g.* the significant decrease of the O(ester)—P—O_t bond angles compared with the other O(ester)—P—O angles.

The packing of the crystal is shown in Fig. 2. The D-fructose 6-phosphate dianions are situated with their long molecular axis approximately parallel to the *c* axis. Except for the H(W4) atom (see *Experimental*) all other H atoms bonded to O atoms are utilized in hydrogen bonds. The hydrogen-bond data are summarized in Table 3. There is no intramolecular hydrogen bonding and the ester O(6) atom is not engaged in a hydrogen bond. The total effect of the arrangement is to produce a three-dimensional network of hydrogen bonds throughout the crystal.
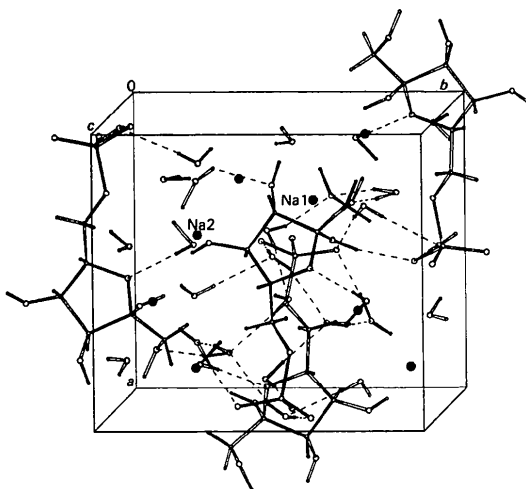
Table 3. *Geometry of the hydrogen bonds (distances in Å, angles in °)*

O—H···O	O···O	O—H	H···O	O—H···O
O(1)—H(11)···O(9^i)	2·584 (3)	0·98 (5)	1·62 (5)	166 (5)
O(2)—H(22)···O(9^{ii})	2·805 (3)	0·85 (4)	1·97 (4)	167 (4)
O(3)—H(33)···O(7^i)	2·637 (3)	0·80 (5)	1·86 (5)	164 (5)
O(4)—H(44)···O(8^{iii})	2·697 (4)	0·78 (5)	1·98 (5)	152 (5)
W(1)—H(W1)···O(1^{iv})	2·807 (4)	0·75 (7)	2·08 (7)	162 (7)
W(1)—H(W11)···W(5^{iii})	2·815 (5)	0·74 (5)	2·09 (5)	169 (6)
W(2)—H(W2)···O(8^v)	2·735 (4)	0·89 (5)	1·86 (6)	167 (5)
W(2)—H(W21)···W(3^{vi})	2·895 (5)	0·71 (5)	2·20 (5)	170 (6)
W(3)—H(W3)···O(9^v)	2·763 (4)	0·90 (6)	1·86 (6)	179 (6)
W(3)—H(W31)···W(7^{vii})	2·849 (5)	0·70 (6)	2·20 (6)	154 (6)
W(4)—H(W41)···O(5^{ii})	2·930 (4)	0·94 (7)	2·03 (7)	162 (6)
W(5)—H(W5)···W(7^{viii})	2·780 (5)	0·84 (5)	1·97 (5)	162 (5)
W(5)—H(W51)···O(7^{iii})	2·674 (4)	0·86 (5)	1·82 (5)	173 (5)
W(6)—H(W6)···O(3)	2·839 (5)	0·79 (7)	2·23 (7)	134 (7)
W(6)—H(W61)···O(8^{iii})	2·795 (5)	1·03 (7)	1·77 (7)	174 (6)
W(7)—H(W7)···W(6^{ix})	2·659 (5)	0·97 (6)	1·71 (6)	165 (5)
W(7)—H(W71)···O(1)	2·804 (4)	1·09 (7)	1·75 (7)	161 (6)

Symmetry code: (i) $x-1, y, z$; (ii) $x-\frac{1}{2}, \frac{3}{2}-y, 1-z$; (iii) $x-\frac{1}{2}, \frac{1}{2}-y, 1-z$; (iv) $\frac{1}{2}-x, 1-y, z-\frac{1}{2}$; (v) $\frac{3}{2}-x, 1-y, z-\frac{1}{2}$; (vi) $1-x, \frac{1}{2}+y, \frac{1}{2}-z$; (vii) $\frac{1}{2}+x, \frac{3}{2}-y, 1-z$; (viii) $x, y-1, z$; (ix) $\frac{1}{2}-x, 1-y, \frac{1}{2}+z$.

There are two kinds of Na$^+$ ions in the structure. Na(1) is coordinated by four water molecules and two hydroxyl O atoms at distances of 2·353 (3)–2·648 (5) Å, forming a distorted octahedron. Na(2) is five coordinated in the form of a somewhat distorted tetragonal pyramid by four water molecules and O(2) (hydroxyl) at distances of 2·330 (3)–2·476 (4) Å.

References

CREMER, D. & POPLE, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
LIS, T. (1985a). *Carbohydr. Res.* **135**, 187–194.
LIS, T. (1985b). *Acta Cryst.* C**41**, 1578–1580.
NARENDRA, N., SESHADRI, T. P. & VISWAMITRA, M. A. (1985). *Acta Cryst.* C**41**, 31–34.
STARYNOWICZ, P. & LIS, T. (1985). Proc. of School–Symposium on Inorganic Biochemistry and Molecular Biophysics, Wrocław-Karpacz, Poland, pp. 228–229.
SYNTEX (1976). *XTL/XTLE Structure Determination System.* Syntex Analytical Instruments, Cupertino, California.

Fig. 2. The packing arrangement. Dashed lines show the hydrogen bonds.