Table 3. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for* (5*b*) *with e.s.d.'s in parentheses*

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	2803 (4)	7301 (2)	1411 (2)	20 (1)
O(1)	5208 (3)	6964 (2)	1094 (1)	24 (1)
C(2)	1239 (4)	6594 (2)	1009 (2)	22 (1)
O(2)	1795 (3)	5382 (2)	1413 (1)	28 (1)
C(3)	1145 (4)	6930 (2)	−187 (2)	24 (1)
O(3)	−789 (3)	6362 (2)	−373 (2)	31 (1)
C(31)	3242 (4)	6467 (3)	−823 (2)	28 (1)
C(4)	685 (5)	8257 (2)	−562 (2)	29 (1)
C(5)	2451 (5)	8918 (2)	−241 (2)	29 (1)
C(6)	2320 (4)	8615 (2)	957 (2)	25 (1)
C(10)	2315 (4)	6983 (2)	2623 (2)	18 (1)
P	4063 (1)	7666 (1)	3278 (1)	18 (1)
O(10)	6540 (3)	7708 (2)	2772 (1)	24 (1)
C(11)	3752 (4)	6841 (2)	4635 (2)	19 (1)
C(12)	1927 (6)	6181 (4)	5102 (2)	60 (2)
C(13)	1801 (6)	5555 (4)	6144 (2)	59 (1)
C(14)	3496 (5)	5584 (2)	6732 (2)	28 (1)
C(15)	5299 (6)	6250 (3)	6285 (3)	59 (1)
C(16)	5437 (6)	6877 (3)	5244 (3)	56 (1)
C(21)	2782 (4)	9117 (2)	3305 (2)	21 (1)
C(22)	495 (5)	9345 (3)	3675 (3)	60 (2)
C(23)	−388 (6)	10472 (3)	3697 (3)	55 (1)
C(24)	975 (5)	11382 (3)	3342 (2)	36 (1)
C(25)	3245 (7)	11170 (3)	2964 (3)	64 (2)
C(26)	4157 (6)	10049 (3)	2941 (3)	45 (1)

[O(2)···O(3′) = 2.911 (3), H(2*a*)···O(3′) = 2.09 (3) Å, O(2)—H(2*a*)···O(3′) = 163 (2)°], and the O(3) H atom interacts with O(1) of an adjacent molecule [O(3)···O(1″) = 2.952 (3), H(3*a*)···O(1″) = 2.11 (3) Å; O(3)—H(3*a*)···O(1″) = 173 (2)°].

Table 4. *Selected bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

	(2*a*)	(3*a*)	(5*b*)
C(1)—O(1)	1.431 (5)	1.422 (3)	1.447 (3)
C(1)—C(2)	1.501 (6)	1.517 (4)	1.535 (4)
C(1)—C(10)	1.540 (6)	1.551 (4)	1.540 (3)
C(2)—O(2)		1.422 (4)	1.417 (3)
C(2)—C(3)	1.331 (6)	1.458 (4)	1.539 (3)
C(3)—O(2/3)		1.439 (4)	1.455 (4)
C(10)—P	1.804 (4)	1.818 (3)	1.804 (3)
P—O(10)	1.503 (3)	1.503 (2)	1.498 (2)
P—C(11)	1.807 (4)	1.805 (3)	1.804 (2)
P—C(21)	1.793 (4)	1.807 (3)	1.806 (3)
O(1)—C(1)—C(2)	110.6 (3)	110.5 (2)	108.9 (2)
O(1)—C(1)—C(10)	109.9 (3)	110.7 (2)	109.2 (2)
C(2)—C(1)—C(10)	113.2 (4)	110.7 (2)	107.5 (2)
C(1)—C(2)—O(2)		116.6 (3)	109.1 (2)
C(1)—C(2)—C(3)	122.6 (5)	121.7 (3)	116.0 (2)
O(2)—C(2)—C(3)		60.0 (2)	113.1 (2)
C(2)—O(2)—C(3)		61.3 (2)	
C(2)—C(3)—O(2/3)		58.8 (2)	107.2 (2)
C(1)—C(10)—P	114.2 (3)	114.9 (2)	115.4 (2)
C(10)—P—O(10)	111.4 (2)	111.6 (1)	112.3 (1)
C(10)—P—C(11)	106.8 (2)	106.2 (1)	105.6 (1)
O(10)—P—C(11)	112.1 (2)	112.7 (1)	113.2 (1)
C(10)—P—C(21)	108.4 (2)	107.9 (1)	109.3 (1)
O(10)—P—C(21)	111.6 (2)	111.9 (1)	111.0 (1)
C(11)—P—C(21)	106.3 (2)	106.1 (1)	105.2 (1)

In all three structures the geometry at the P atom is slightly distorted tetrahedral (Table 4), with C—P—C angles less than C—P—O angles, as has been observed in other diphenylphosphinoylmethyl structures (Bernardinelli & Gerdil, 1979; Tkachev, Bondarenko, Matrosov, Cvetkov, Atovmjan & Kabachnik, 1981*b*; Szlyk, Zhang, Palenik, Palenik & Colgate, 1989). Other bonding geometry is normal. The phenyl rings are all planar to within 0.006 Å with their mean planes mutually inclined at angles of 67.2 (4), 70.4 (3) and 78.7 (3)° for (2*a*), (3*a*) and (5*b*) respectively.

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Structure of Disodium β-D-Fructopyranose 1-Phosphate Pentahydrate

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Abstract. $2Na^+.C_6H_{11}O_9P^{2-}.5H_2O$, $M_r = 394.18$, monoclinic, $P2_1$, $a = 8.991$ (9), $b = 6.247$ (7), $c = 13.611$ (15) Å, $\beta = 92.53$ (6)°, $V = 763.7$ (15) Å^3, $Z = 2$, $D_m = 1.72$, $D_x = 1.714$ (4) g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 3.0$ cm^{-1}, $F(000) = 412$, $T = 302$ (3) K, final $R = 0.042$ for 2520 observed data including both hkl and $\bar{h}\bar{k}\bar{l}$ reflections. The β-D-fructopyranose ring adopts the $^2C_5$ conformation. The arrangement of the C(1)—O(1) bond with respect to the C(2)—O(6) and C(2)—C(3) ring bonds

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is *gauche–gauche*. The P—O(1)—C(1)—C(2) torsion angle is −118.3 (3)° and the phosphate ester bond, P—O(1), is 1.639 (3) Å. There is an intramolecular hydrogen bond between the sugar hydroxyl O(3) and phosphate O(9) [O···O = 2.764 (4) Å]. The phosphate O(7) and O(9) atoms form (as acceptors) four hydrogen bonds and are therefore formally pentacoordinated. The crystal structure is stabilized by three-dimensional hydrogen bonding utilizing all oxygen-bonded H atoms.

Introduction. Four naturally occurring fructose phosphates, fructose 6-phosphate (F6P), fructose 1,6-diphosphate (F1,6-dp), fructose 1-phosphate (F1P) and fructose 2,6-diphosphate (F2,6-dp), play important roles as intermediates and regulators of carbohydrate metabolism. Only two of these species, F6P and F1,6-dp, have been characterized by X-ray studies of $K_2(F6P).6H_2O$ (Narendra, Seshadri & Viswamitra, 1985*b*), $Na_2(F6P).7H_2O$ (Lis, 1986) and $Na_3H(F1,6\text{-dp}).8H_2O$ (Narendra, Seshadri & Viswamitra, 1985*a*; Cerrini, Coiro, Lamba & Bisso, 1986). In this paper the crystal structure of $Na_2(F1P).5H_2O$, the main product of fructose phosphorylation by liver fructokinase, is described. This report follows our earlier studies on the crystal structures of biologically important phosphate esters (Weichsel, Lis & Kuczek, 1989).

Experimental. Crystals of disodium *β*-D-fructopyranase 1-phosphate pentahydrate were obtained by slow concentration of an aqueous solution of the commercially available compound. The compound crystallized mostly as plates elongated along **b**, with **a** perpendicular to the plate. The crystals are unstable in air, probably because of dehydration. D_m measured by flotation in CCl_4/CH_2Br_2. Preliminary Weissenberg photographs indicated the space group $P2_1$. A fragment crystal 0.05 × 0.6 × 0.2 mm was cut from a larger one and sealed in a capillary. A Syntex $P2_1$ four-circle diffractometer, Mo *Kα* radiation and graphite monochromator were used for lattice parameters (15 reflections in the range $17 < 2\theta < 25°$) and intensity measurements. Almost full Ewald sphere up to $2\theta = 54°$ was measured by 2θ–ω-scan technique ($-11 \le h \le 11$, $-8 \le k \le 8$, $-17 \le l \le 17$). The intensities were corrected for Lorentz and polarization factors only. Averaging the 6516 intensity measurements in point group 2 yielded 3230 unique intensities. Of these 2520 with $I > 2\sigma(I)$, including 1093 Friedel pairs, were used subsequently. R_{merge} was 0.0239 for observed reflections. After each group of 50 reflections two standards were measured; variation ±4%. The structure was solved by direct methods and refined with *SHELX76* (Sheldrick, 1976) minimizing the function $\sum w(|F_o| - |F_c|)^2$, with $w = 1/\sigma^2(F_o)$. The known absolute configuration was assumed. The carbon-bonded H atoms were included in calculated positions with $d(\text{C—H}) = 1.08$ Å. All other H atoms were found from a difference Fourier synthesis and refined with the constraint $d(\text{O—H}) = 0.97$ Å. The refinement of 270 parameters, including U_{iso} of the H atoms, yielded a final R value of 0.0414 and a wR value of 0.0315; $(\Delta/\sigma)_{\max} = 0.3$, minimum and maximum heights in difference Fourier map were −0.55 and 0.56 e $Å^{-3}$. The atomic scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV); anomalous dispersion was included for P, Na^+, O and C atoms. The atomic positional parameters and equivalent temperature factors are listed in Table 1.* Bond lengths, angles, torsion angles and selected interatomic distances are given in Table 2.

Table 1. *Final atomic parameters*

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

	x	y	z	U_{eq} ($Å^2$)
P	0.20196 (10)	0.2†	0.25104 (7)	0.0191 (3)
O(1)	0.04277 (24)	0.0771 (5)	0.26289 (17)	0.0208 (6)
O(2)	−0.32853 (27)	0.3114 (5)	0.22761 (18)	0.0230 (7)
O(3)	−0.10746 (27)	0.5842 (5)	0.30381 (18)	0.0228 (6)
O(4)	−0.17351 (27)	0.5813 (5)	0.50179 (17)	0.0220 (7)
O(5)	−0.16686 (26)	0.1366 (5)	0.53508 (18)	0.0243 (7)
O(6)	−0.24491 (25)	0.0337 (5)	0.33285 (18)	0.0199 (6)
O(7)	0.24935 (26)	0.1525 (6)	0.14712 (17)	0.0287 (7)
O(8)	0.30006 (25)	0.0933 (5)	0.32955 (18)	0.0271 (7)
O(9)	0.17578 (30)	0.4375 (5)	0.27004 (19)	0.0287 (7)
C(1)	−0.08756 (37)	0.1526 (7)	0.20839 (25)	0.0212 (9)
C(2)	−0.20490 (36)	0.2244 (7)	0.28024 (25)	0.0180 (9)
C(3)	−0.14261 (39)	0.3904 (6)	0.35309 (25)	0.0163 (9)
C(4)	−0.24935 (41)	0.4380 (6)	0.43362 (25)	0.0183 (9)
C(5)	−0.29493 (37)	0.2283 (7)	0.48248 (25)	0.0210 (9)
C(6)	−0.35511 (37)	0.0770 (7)	0.40464 (26)	0.0225 (9)
Na(1)	−0.03812 (16)	0.8683 (4)	0.41732 (11)	0.0259 (4)
Na(2)	0.35905 (17)	0.1729 (4)	0.81728 (11)	0.0365 (4)
W(1)	0.55562 (28)	0.1996 (6)	0.70242 (19)	0.0336 (7)
W(2)	0.57638 (33)	0.0102 (6)	0.92411 (22)	0.0354 (8)
W(3)	0.85630 (36)	0.2183 (7)	0.89635 (23)	0.0474 (9)
W(4)	0.53408 (28)	0.0002 (5)	0.12103 (20)	0.0280 (7)
W(5)	0.17510 (32)	0.1824 (7)	0.93926 (20)	0.0471 (9)

† Fixed.

Discussion. The crystals of the title compound are composed of D-fructose 1-phosphate dianions, Na^+ cations and water of hydration. The molecular configuration and conformation and the atom-numbering scheme of the anion are illustrated in Fig. 1. The fructose moiety exists as *β*-D-fructopyranose. ^{13}C NMR studies (Koerner, Voll, Cary & Younathan, 1980) indicated that in aqueous solution several isomers of D-fructose 1-phosphate are present but the predominant form is *β*-D-fructopyranose, the isomer found in the crystal structure described here.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54607 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0275]

Table 2. *Bond lengths* (Å), *bond angles* (°), *torsion angles* (°) *and selected interatomic distances* (Å)

C(1)—C(2)	1.537 (5)	C(4)—O(4)	1.439 (4)
C(2)—C(3)	1.524 (5)	C(5)—O(5)	1.447 (4)
C(3)—C(4)	1.518 (5)	C(6)—O(6)	1.447 (4)
C(4)—C(5)	1.533 (5)	C(2)—O(6)	1.444 (5)
C(5)—C(6)	1.503 (5)	O(1)—P	1.639 (3)
C(1)—O(1)	1.438 (4)	O(7)—P	1.524 (3)
C(2)—O(2)	1.405 (4)	O(8)—P	1.510 (3)
C(2)—O(3)	1.426 (4)	O(9)—P	1.526 (3)

O(1)—C(1)—C(2)	109.5 (3)	O(5)—C(5)—C(4)	109.3 (3)
O(2)—C(2)—C(1)	109.8 (3)	O(5)—C(5)—C(6)	110.7 (3)
O(2)—C(2)—O(6)	111.3 (3)	C(4)—C(5)—C(6)	109.0 (3)
O(2)—C(2)—C(3)	109.2 (3)	O(6)—C(6)—C(5)	111.0 (3)
O(6)—C(2)—C(1)	105.5 (3)	C(2)—O(6)—C(6)	111.9 (3)
O(6)—C(2)—C(3)	109.3 (3)	C(1)—O(1)—P	119.4 (3)
C(1)—C(2)—C(3)	111.7 (3)	O(1)—P—O(7)	106.3 (2)
O(3)—C(3)—C(2)	110.7 (3)	O(1)—P—O(8)	102.1 (2)
O(3)—C(3)—C(4)	109.5 (3)	O(1)—P—O(9)	107.3 (2)
C(2)—C(3)—C(4)	112.2 (3)	O(7)—P—O(8)	113.1 (2)
O(4)—C(4)—C(3)	106.9 (3)	O(7)—P—O(9)	113.5 (2)
O(4)—C(4)—C(5)	112.3 (3)	O(8)—P—O(9)	113.5 (2)
C(3)—C(4)—C(5)	109.6 (3)		

O(7)—P—O(1)—C(1)	−69.1 (3)	O(1)—C(1)—C(2)—C(3)	54.9 (4)
O(8)—P—O(1)—C(1)	172.2 (3)	O(1)—C(1)—C(2)—O(6)	−63.8 (4)
O(9)—P—O(1)—C(1)	52.6 (3)	O(1)—C(1)—C(2)—O(2)	176.1 (4)
P—O(1)—C(1)—C(2)	−118.3 (4)		

Na(1)—O(1$^{\text{i}}$)	2.604 (3)	Na(2)—O(2$^{\text{iii}}$)	2.352 (4)
Na(1)—O(3)	2.417 (3)	Na(2)—O(3$^{\text{iii}}$)	2.795 (3)
Na(1)—O(4)	2.478 (3)	Na(2)—*W*(1)	2.417 (3)
Na(1)—O(4$^{\text{ii}}$)	2.533 (3)	Na(2)—*W*(2)	2.591 (3)
Na(1)—O(5$^{\text{i}}$)	2.623 (3)	Na(2)—*W*(4$^{\text{iv}}$)	2.396 (4)
Na(1)—O(5$^{\text{ii}}$)	2.409 (3)	Na(2)—*W*(5)	2.395 (3)
Na(1)—O(6$^{\text{i}}$)	2.378 (3)		

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

In crystals of F6P and F(1,6-dp) the β-D-fructofuranose form was also found. The sugar-ring conformation is a slightly distorted $^2C_5$ chair form, having Cremer & Pople (1975) puckering parameters of $Q = 0.577$ (4) Å, $\theta = 176.7$ (4) and $\varphi = 132$ (7)° so that C(1) is in an equatorial position; this geometry is energetically favoured for the β-D-fructopyranose moiety. The O(1) atom is *trans* to O(2) so that the conformation about the C(1)—C(2) bond is *gauche–gauche*. A similar conformation was found in crystals of both $Na_3H(F1,6\text{-dp}).8H_2O$ and β-D-fructopyranose (Takagi & Jeffrey, 1977). The conformation about the O(1)—C(1) bond is anticlinal [torsion angle P—O(1)—C(1)—C(2) is −118.3 (4)°] and departs considerably from that found in aqueous solution where *gauche*- and *trans*-periplanar rotamers make up 62 and 38% respectively of the molecules present (Koerner, Voll, Cary & Younathan, 1980). The energetically unfavourable conformation about the O(1)—C(1) bond in the solid state is probably a consequence of the intramolecular hydrogen bond between the phosphate oxygen O(9) and sugar oxygen O(3), though in $Na_3H(F1,6\text{-dp}).8H_2O$ crystals, where such a hydrogen bond is not present, a similar orientation of the phosphate group relative to the sugar residue is found. The C—C bond lengths range from 1.503 (5) to 1.537 (5) Å and the C—O bond lengths from 1.405 (4) to 1.447 (4) Å, close to those found in other carbohydrate molecules containing fructopyranosyl groups (Takagi & Jeffrey, 1977).

The phosphate ester bond, P—O(1), of 1.639 (3) Å is longer than the values of 1.597 (3) Å (Cerrini, Coiro, Lamba & Bisso, 1986) or 1.591 (3) Å (Narendra, Seshadri & Viswamitra, 1985*a*) in $Na_3H(F1,6\text{-dp}).8H_2O$ where the phosphate group is monoionized. This difference between singly and doubly ionized phosphate monoesters is expected (Starynowicz, 1986).

The conformation about the P—O(1) bond is that normally found in phosphate monoesters, *i.e.* one of the terminal phosphate O atoms [O(8)], which is *trans* with respect to C(1) [C(1)—O(1)—P—O(8) torsion angle is 172.2 (3)°], is involved in the smallest

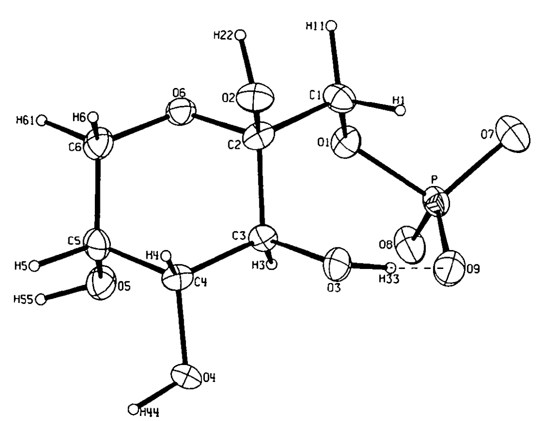

Fig. 1. The structure of the β-D-fructopyranose 1-phosphate dianion, showing the numbering of the atoms.

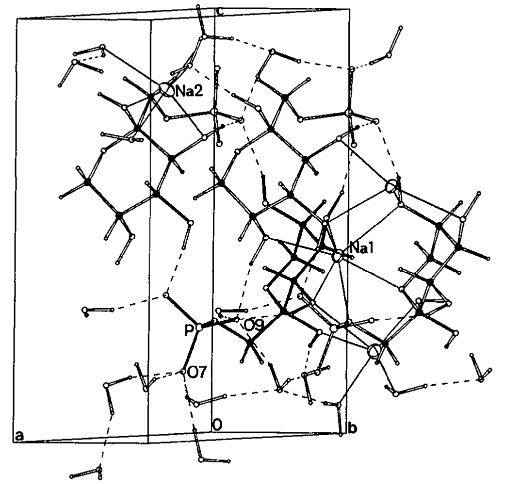

Fig. 2. The packing in the crystal of disodium β-D-fructopyranose 1-phosphate pentahydrate.

Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

X—H···O	*X*···O	H···O	*X*—H···O
O(2)—H(22)···*W*(4$^{\mathrm{i}}$)	2.693 (4)	1.74 (4)	167 (4)
O(3)—H(33)···O(9)	2.764 (4)	1.87 (3)	153 (3)
O(4)—H(44)···O(8$^{\mathrm{ii}}$)	2.608 (4)	1.67 (4)	161 (4)
O(5)—H(55)···O(9$^{\mathrm{iii}}$)	2.934 (4)	2.01 (3)	158 (4)
W(1)—H(*W*11)···O(9$^{\mathrm{iv}}$)	2.928 (4)	1.98 (3)	166 (4)
W(1)—H(*W*12)···O(8$^{\mathrm{v}}$)	2.823 (4)	1.87 (3)	165 (3)
W(2)—H(*W*21)···O(7$^{\mathrm{iv}}$)	2.919 (4)	1.98 (4)	163 (4)
W(2)—H(*W*22)···*W*(3)	2.872 (5)	2.00 (4)	149 (4)
W(3)—H(*W*31)···O(7$^{\mathrm{v}}$)	2.926 (5)	1.98 (3)	165 (3)
W(3)—H(*W*32)···O(9$^{\mathrm{iv}}$)	2.869 (4)	1.93 (3)	161 (3)
W(4)—H(*W*41)···O(7)	2.769 (4)	1.83 (3)	161 (4)
W(4)—H(*W*42)···*W*(2$^{\mathrm{vi}}$)	2.724 (4)	1.88 (3)	144 (4)
W(5)—H(*W*51)···*W*(3$^{\mathrm{i}}$)	2.908 (4)	1.99 (3)	156 (4)
W(5)—H(*W*52)···O(7$^{\mathrm{vii}}$)	2.884 (4)	1.92 (2)	175 (5)
C(6)—H(6)···O(8$^{\mathrm{i}}$)	3.223 (5)	2.29 (3)	143 (3)

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

O(ester)—P—O bond angle [O(1)—P—O(8) = 102.1 (2)°]. Two terminal phosphate O atoms [O(7) and O(9)] form (as acceptors) four hydrogen bonds and are therefore formally pentacoordinated (Table 3). Although this arrangement does not appear to have been observed previously in phosphate esters a few examples of formal pentacoordination of phosphate O atoms are already known (Weichsel & Lis, 1990).

All OH groups participate in hydrogen bonding (see Fig. 2 and Table 3). There is also an intermolecular hydrogen bond between terminal phosphate atoms O(8) and C(6) according to the criterion of Taylor & Kennard (1982).

There are two crystallographically independent Na^+ ions in the structure. The Na(1) atom (Table 2) is seven coordinate [Na—O distances of 2.378 (3)–2.623 (3) Å], binding to three independent fructose moieties. One of these is tridentate, binding to Na(1) through O(1$^{\mathrm{i}}$), O(5$^{\mathrm{i}}$) and O(6$^{\mathrm{i}}$), and two are bidentate, binding through O(3) and O(4) and through O(4$^{\mathrm{ii}}$) and O(5$^{\mathrm{ii}}$). The Na(2) atom has a distorted octahedral coordination formed by two sugar O atoms O(2$^{\mathrm{iii}}$) and O(3$^{\mathrm{iii}}$) and four water molecules at Na—O distances of 2.352 (4)–2.795 (3) Å. The phosphate O atoms do not participate in the coordination spheres of the Na^+ cations as observed previously in Na_2(F6P).$7H_2O$ (Lis, 1986) and Na_3H(F1,6-dp).$8H_2O$ (Narendra, Seshadri & Viswamitra, 1985*a*; Cerrini, Coiro, Lamba & Bisso, 1986).

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Structure of 1,3,4,5-Neopentanetetrayl Tetrakis[3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate]

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Abstract. $C_{73}H_{108}O_{12}$, $M_r = 1177.65$, monoclinic, Cc, $a = 25.442$ (8), $b = 10.051$ (3), $c = 27.062$ (10) Å, $\beta = 94.5$ (1)°, $V = 6898.5$ Å^3, $Z = 4$, $D_x = 1.1339$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 5.64$ cm^{-1}, $F(000) = 2568$, $T = 293$ K, $R = 0.094$ for 5060 observed reflections. The molecular structure

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