Discussion. Final atomic parameters are given in Table 1, bond lengths and angles in Tables 2 and 3, and equations for the least-squares planes in Table 4. The atom numbering is shown in Fig. 1.

Table 4. *Equations for the least-squares planes*

Plane

(A) C(1) to C(10):
$$0.0164x + 0.1217y + 0.9924z = 1.8598$$

(B) O(1), C(11) and C(12):
$$0.0625x + 0.0478y + 0.9975z = 1.9606$$

(C) C(1) to C(12) and O(1):
$$0.0191x + 0.1013y + 0.9947z = 1.7721$$

(D) C(1) to C(13) and O(1):
$$0.0368x + 0.1091y + 0.9934z = 1.8601$$

(E) C(1) to C(10) and C(13):
$$0.0258x + 0.1300y + 0.9912z = 1.9264$$

(F) C(13), C(14) and O(2):
$$0.2668x + 0.9510y - 0.1591z = 1.7203$$

The direction cosines of the normals to various planes

Normal to plane	l	m	n
(A)	0.0164	0.1217	0.9924
(B)	0.0625	0.0478	0.9975
(C)	0.0191	0.1013	0.9947
(D)	0.0368	0.1091	0.9934
(E)	0.0258	0.1300	0.9912
(F)	0.2668	0.9510	0.1591

The dihedral angle between the planes (A) and (B) is 1° and between planes (A) and (F) is 86.8°.

The deviations of the atoms in the central aromatic ring are all within experimental error and the ring may be assumed to be planar. The atoms of the ethoxy group are nearly coplanar with the ring atoms, the largest deviation being for C(12) (0.13 Å). C(13), C(14) and O(2) of the ethyl methyl ether group are in a plane nearly at right angles to the plane of the aromatic ring.

In the crystal, the molecules are arranged in layers (Fig. 2), the interlayer separation being approximately 3.00 Å; they are held together loosely by van der Waals forces, the contacts ranging from 3.04 to 3.98 Å. The coefficient of molecular packing (Kitaigorodsky, 1955) is 0.72 with a coordination number of six.

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Hydrogen-Bonded Dimers in Tin(II) Hydrogen Phosphate

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Abstract. SnHPO$_4$, monoclinic, $P2_1/c$, $a = 4.608$ (2), $b = 13.603$ (4), $c = 5.823$ (2) Å, $\beta = 98.76$ (1)°, $Z = 4$. This neutron diffraction study has shown that the HPO$_4^{2-}$ ions are linked together by two asymmetric hydrogen bonds [$d(\text{O}\cdots\text{O}) = 2.560$, $d(\text{O–H}) = 1.017$ Å, $\angle$O–H$\cdots$O $= 178.1°$] to form dimers. Tin–phosphate–oxygen coordination takes place primarily in layers with the hydrogen bonds occurring between these layers.

Introduction. The structure of SnHPO$_4$ was determined by Berndt & Lamberg (1971) and further refined by McDonald (1975). Both studies used X-ray diffraction. T. H. Jordan prepared colorless crystals of SnHPO$_4$ by the reaction of SnCl$_2$ and H$_3$PO$_4$ at $pH \simeq 2$. The cell dimensions in our study were obtained by least squares

from the setting angles for 30 reflections measured on a four-circle diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. The crystal used for the collection of neutron data was a diamond-shaped (010) plate with a volume ~ 3.6 mm³. 1724 reflections within a hemisphere defined by a neutron wavelength of 1.232 Å and a limiting 2θ of 100° were measured on a four-circle diffractometer according to the procedure described by Prince (1972).

The heavy-atom parameters as given by Berndt & Lamberg (1971) were used to phase an F_o nuclear density synthesis that revealed the H position. The scattering lengths (in units of 10^{-12} cm) used were Sn:0.61, P:0.51, O:0.580 and H: -0.374 (Bacon, 1972). Refinement of atomic parameters was accomplished with the program *RFINE*4 (Finger & Prince, 1975); the sum $\sum w(|F_o| - |F_c|)^2$, where $w = \sigma^{-2}$, was minimized. The variance of $|F_o|$, σ^2, was taken as $\sigma^2_{count} + 0.0004F_o^2$.

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Equivalent reflections were not averaged and so all reflections in the hemisphere with $|F_o| > 2\sigma$ were used in the refinement. The value of R ($=\sum||F_o|-|F_c||/\sum|F_o|$) for these 1473 reflections was 0·079 at convergence. A normal probability plot of the residuals, $(|F_o|-|F_c|)/\sigma$, against the expected quantiles showed some non-linearity due to outliers. The interquartile range (quantiles $-0·667$ to $+0·667$) was doubled to give an approximate value of Δ_{max} to generate new weights (Nicholson & Prince, 1977) $w' = w(1 - 3x^4 + 2x^6)$ where $x = (|F_o| - |F_c|)/\sigma\Delta_{max}$ and $w' = 0$ if $|x| > 1$. Two cycles using w' reduced R to 0·073 for 1415 reflections and yielded residuals whose normal probability plot was quite linear. The subsequent interquartile range was multiplied by 3·5 to yield a value of Δ_{max} that changes the weights of 97% of normally distributed data by less than 9%. Two additional cycles of refinement with the new $\Delta_{max} = 16·5$ rejected only 1% of the data and gave $R = 0·075$ for 1460 reflections.† The normal probability plot of the residuals was essentially linear. Some thermal parameters changed by 2σ and the approximate extinction parameter $Tr*$ by 4σ when weights w' were used. The differences between the positional parameters common to this study and a recent study by McDonald (1975) are insignificant. The final atomic parameters are given in Table 1. Selected interatomic distances and angles are given in Table 2.

† A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31914 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Discussion. This investigation was undertaken to elucidate the nature of the hydrogen bonding in order to provide a structural basis for the interpretation of some Raman and IR data obtained on SnHPO$_4$. In turn, the spectroscopic characterization of HPO$_4^{2-}$ dimers provides a means of looking for such entities in various situations. For example, the state of HPO$_4^{2-}$ ions in tooth enamel is of interest (Davidson & Hoekstra, 1975).

Fig. 1 shows the structure as viewed along the c axis. The HPO$_4^{2-}$ dimers lie across centers of symmetry and in sheets parallel to the (100) plane while the hydrogen bonds are all in planes parallel to (010). Thus, layers of strong interactions, Sn$\cdots$O bonds, alternate with weak interactions, the hydrogen bonds.

Table 1. *Positional and thermal parameters for* SnHPO$_4$

The form of the thermal factor is $\exp[-2\pi^2(\sum_{ij}U_{ij}h_ih_ja_i^*a_j^*)]$. The figures in parentheses are standard deviations as estimated by the last cycle of least-squares refinement. All values are $\times 10^4$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn	511 (2)	1575 (1)	1093 (2)	158 (6)	183 (6)	212 (7)	21 (3)	−2 (4)	−21 (4)
P	5714 (2)	1511 (1)	5640 (2)	117 (6)	150 (6)	138 (7)	−5 (4)	18 (5)	−2 (4)
O(1)	3027 (2)	2129 (1)	4566 (2)	126 (6)	239 (7)	185 (7)	29 (4)	6 (5)	−1 (4)
O(2)	4807 (3)	822 (1)	7565 (2)	275 (7)	207 (7)	188 (7)	−48 (5)	65 (5)	11 (5)
O(3)	6828 (3)	913 (1)	3763 (2)	242 (7)	183 (6)	215 (7)	−16 (4)	87 (5)	−50 (5)
O(4)	8042 (2)	2181 (1)	6989 (2)	134 (6)	197 (7)	174 (7)	−31 (3)	9 (4)	−9 (4)
H	4120 (5)	4861 (2)	2021 (5)	341 (12)	310 (13)	342 (13)	26 (9)	83 (10)	−34 (10)

Table 2. *Selected interatomic distances and angles with estimated standard deviations in parentheses*

i	j	k	d_{ij} (Å)	d_{jk} (Å)	d_{ik} (Å)	$\angle ijk$ (°)
O(2)	H	O(3)	1·017 (2)	1·544 (3)	2·560 (2)	178·1 (2)
P	O(2)	H	1·566 (2)	1·017 (2)	2·203 (3)	115·4 (2)
O(1)	P	O(2)	1·548 (2)	1·566 (2)	2·539 (2)	109·3 (1)
O(1)	P	O(3)	1·548 (2)	1·514 (2)	2·505 (2)	109·8 (1)
O(1)	P	O(4)	1·548 (2)	1·530 (2)	2·519 (2)	109·8 (1)
O(2)	P	O(3)	1·566 (2)	1·514 (2)	2·532 (2)	110·6 (1)
O(2)	P	O(4)	1·566 (2)	1·530 (2)	2·431 (1)	103·4 (1)
O(3)	P	O(4)	1·514 (2)	1·530 (2)	2·549 (2)	113·7 (1)
O(1)	Sn	O(1^i)*	2·300 (2)	2·357 (2)	3·082 (1)	82·85 (5)
O(1)	Sn	O(4^i)	2·300 (2)	2·146 (2)	2·713 (2)	75·11 (6)
O(1^i)	Sn	O(4^i)	2·357 (2)	2·146 (2)	2·874 (2)	79·15 (6)

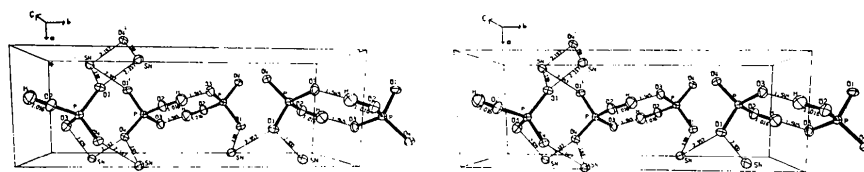* Atom O(n^i) is related to O(n) by the symmetry operation $x, \frac{1}{2} - y, \frac{1}{2} + z$.

Fig. 1. A stereoscopic pair showing the crystal structure of SnHPO$_4$ as viewed along the c axis. The star marks the origin of the coordinate system.

The observed O–H distance is consistent with the curve given by Pimentel & McClellan (1971) relating O–H distance as a function of $O \cdots O$ distance. In addition, the O–H and $O \cdots O$ distances are very close to recent neutron results presented (Fig. 1.4) by Speakman (1972).

A neutron study of $CaHPO_4.2H_2O$ (Curry & Jones, 1971) showed that HPO_4^{2-} ions are linked together by hydrogen bonds forming a chain $\cdots HOPO_3^- \cdots HOPO_3^- \cdots$. The $O \cdots O$ distance in this chain is 2·678 Å, which is significantly longer than the distance of 2·560 Å for the HPO_4^{2-} dimers in $SnHPO_4$.

Some characteristics of the HPO_4^{2-} ion have been tabulated by Baur (1974). These were obtained from nine published structures in which the dimeric configuration does not occur. Baur gives an average value of 1·590 Å for the P–O(H) distance compared to our value of 1·566 Å. This is to be expected because the H atom in $SnHPO_4$ is participating in a moderately short, strong hydrogen bond. Baur's data show that P–O(H) distances shorten even more (1·516 Å) when the H is involved in a symmetrical hydrogen bond. Thus, the $O \cdots O$ distance of 2·560 Å, the $O–H \cdots O$ angle of 178·1°, and the P–O(H) distance of 1·566 Å in $SnHPO_4$ all indicate that the asymmetric hydrogen bond in dimeric $(HPO_4^{2-})_2$ is quite strong.

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Single-Crystal X-ray Study of β-Uranium Pentafluoride. The Eight Coordination of U^V

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Abstract. β-UF_5, tetragonal, $I\bar{4}2d$; $a = 11·456$ (2), $c = 5·195$ (1) Å; $Z = 8$. The structure of β-UF_5 has been solved by a three-dimensional X-ray diffraction study, with counter techniques. Rather than seven-coordinated as originally proposed on the basis of powder data, the U is found to be eight-coordinated, with coordination geometry intermediate between a dodecahedron and a square antiprism.

Introduction. Isolated seven-coordinated fluoride anions of the d transition elements are well established in NbF_7^{2-} and TaF_7^{2-} by X-ray (Hoard, 1939) and neutron (Brown & Walker, 1966) diffraction. In these K_2MF_7 compounds the anion coordination polyhedron is that of a capped trigonal prism [but distorted towards a capped octahedron (Penneman, Ryan & Rosenzweig, 1973)]. In $(NH_4)_3ZrF_7$ there is evidence for a

pentagonal bipyramid coordination unit (Hurst & Taylor, 1970), first proposed for this compound and for K_3UF_7 by Zachariasen (1954a,b). In both these latter structures there is disorder.

It is interesting to note that there are several actinide elements with radii relatively larger than the d elements mentioned, but whose structures are thought to contain coordination polyhedra of both the above types (see Penneman *et al.*, 1973). However, to date there are no single-crystal studies to substantiate such geometry. Thus, when crystals of β-UF_5 were prepared the opportunity of establishing one of these, *i.e.* the pentagonal bipyramid geometry for an actinide bound only to fluorine, arose. The dioxopentafluorouranyl case with five equatorial fluorines is well established (Zachariasen, 1954a). The structure of β-UF_5 had been deduced by Zachariasen (1949) and has remained a