

Fig. 2 shows a view of the crystal structure along *c*. I(1) has nine nearest neighbours, all at the expected distance of about 4 Å. These comprise eight C and one O, belonging to four different molecules. I(2) also has nine nearest neighbours, at distances below 4 Å. These also consist of eight C and one O, belonging to six different molecules. These are indicated in Fig. 2 by broken lines. The coordination around both types of I⁻ ions seems to be determined by the requirements of close packing of nearly flat molecules. The shortest I⁻-N⁺ distances are: I(1)-N=4.64 (7) and I(2)-N=4.17 (7) Å, the two N atoms being from molecule 2 and molecule 1 respectively. The crystal structure consists of columns of molecules stacked along *c* (Fig. 2). The two shortest intermolecular distances [both 3.1 (1) Å] are rather short for normal van der Waals contacts. Both of these are between atoms of the same type: the first between O(16) of molecule 1 and C(7') of a neighbouring molecule 1, and the second between O(16) of molecule 2 and C(7') of another molecule 2.

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1-(2-Ethoxy-1-naphthyl)ethyl Methyl Ether

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Abstract. C₁₅O₂H₁₈, orthorhombic, *Pna*2₁; *a* = 15.66 (2), *b* = 11.25 (2), *c* = 7.24 (1) Å; *Z* = 4, *D_m* = 1.20, *D_c* = 1.21 g cm⁻³; μ(Cu Kα) = 6.28 cm⁻¹; *R* = 0.081. The central aromatic ring in the molecule is planar and the ethoxy group atoms are nearly coplanar with it. C(13), C(14), O(2) of the ethyl methyl ether group are in a plane nearly normal to the ring atoms.

Introduction. 580 independent X-ray reflexions were collected by single-crystal Weissenberg photography

around [001] and [010] with Cu K radiation; their intensities were estimated visually and brought to an absolute scale by statistical methods.

From the systematic absences (*0kl*, *k+l=2n+1*, *h0l*, *h=2n+1*) two space groups were possible: *Pna*2₁ and *Pnam*. Intensity statistics (Howells, Phillips & Rogers, 1950) indicated the non-centrosymmetric *Pna*2₁. Moreover, only four molecules are present in the unit cell and as there is no symmetry in the molecule, *Pnam* is ruled out.

The structure was solved from a [001] Patterson projection and packing considerations. Full-matrix least-squares analysis with *ORFLS* (Busing, Martin & Levy, 1962) with weights Δ*F* = *a* + *bF_o*, where *a* and *b* are constants for the observed reflexions, and anisotropic temperature factors for the heavy atoms reduced *R* to 0.102. An extinction correction was also applied in the last stages of refinement (Zachariasen, 1967). Fourteen H atoms were located from a difference map; the positions of the remaining four (marked by asterisks in Table 1) were assumed to have their theoretically expected values. The final *R* was 0.081.*

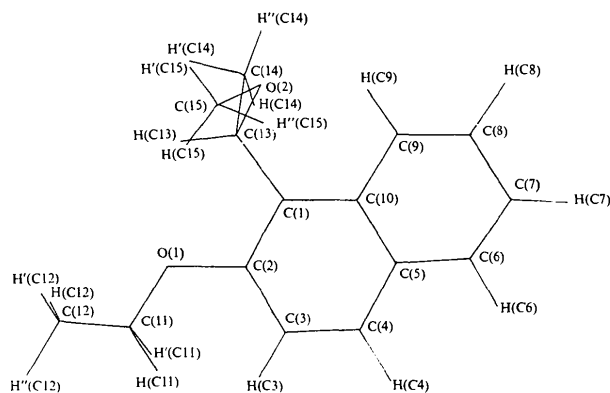


Fig. 1. Numbering scheme for C₁₅O₂H₁₈.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31923 (14 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

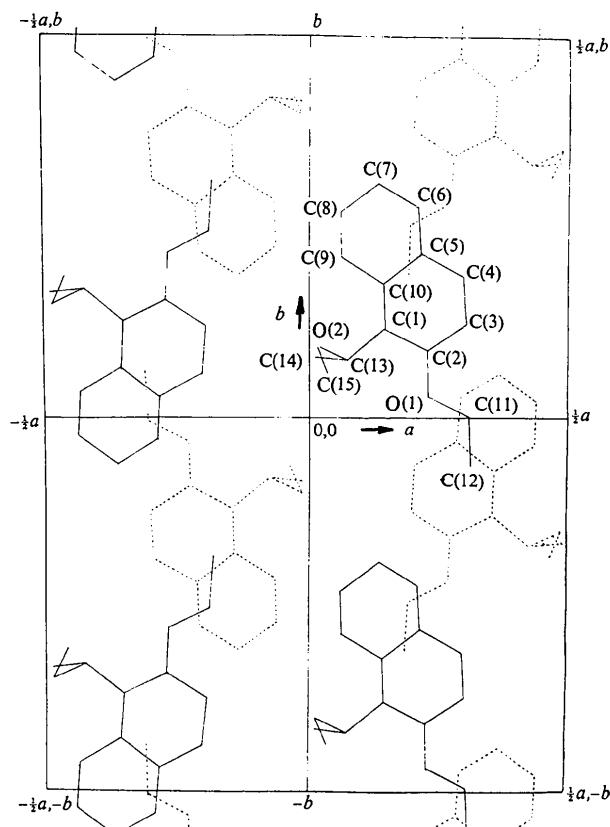


Fig. 2. The crystal structure viewed down [001].

Table 2. Bond lengths with *e.s.d.*'s in parentheses

C(1)—C(2)	1.43 (3) Å	C(3)—H(C3)	0.98 Å
C(1)—C(10)	1.36 (1)	C(4)—H(C4)	1.13
C(1)—C(13)	1.48 (2)	C(6)—H(C6)	0.93
C(2)—O(1)	1.36 (1)	C(7)—H(C7)	1.00
C(2)—C(3)	1.45 (2)	C(8)—H(C8)	0.99
C(3)—C(4)	1.38 (2)	C(9)—H(C9)	0.98
C(4)—C(5)	1.43 (3)	C(11)—H(C11)	1.12
C(5)—C(6)	1.44 (2)	C(11)—H'(C11)	1.23
C(5)—C(10)	1.41 (3)	C(12)—H(C12)	0.91
C(6)—C(7)	1.36 (3)	C(12)—H''(C12)	0.95
C(7)—C(8)	1.43 (2)	C(12)—H'''(C12)	1.22
C(8)—C(9)	1.36 (1)	C(13)—H(C13)	0.95
C(9)—C(10)	1.40 (2)	C(14)—H(C14)	0.95
C(11)—O(1)	1.37 (2)	C(14)—H'(C14)	1.02
C(11)—C(12)	1.55 (2)	C(14)—H''(C14)	1.16
C(13)—O(2)	1.27 (2)	C(15)—H(C15)	0.93
C(13)—C(14)	1.53 (2)	C(15)—H'(C15)	1.04
C(15)—O(2)	1.41 (2)	C(15)—H''(C15)	1.16

Table 3. Bond angles (°) with *e.s.d.*'s in parentheses

C(2)—C(1)—C(10)	119.4 (7)	C(6)—C(7)—C(8)	114.2 (15)
C(10)—C(1)—C(13)	127.2 (6)	C(7)—C(8)—C(9)	123.2 (8)
C(13)—C(1)—C(2)	113.4 (8)	C(8)—C(9)—C(10)	123.2 (8)
C(1)—C(2)—C(3)	119.2 (6)	C(9)—C(10)—C(5)	114.4 (7)
C(3)—C(2)—O(1)	120.3 (7)	C(5)—C(10)—C(1)	123.0 (7)
O(1)—C(2)—C(1)	120.2 (13)	C(9)—C(10)—C(1)	122.6 (7)
C(2)—C(3)—C(4)	119.7 (7)	O(1)—C(11)—C(12)	112.2 (10)
C(3)—C(4)—C(5)	121.3 (7)	C(1)—C(13)—O(2)	118.3 (7)
C(4)—C(5)—C(6)	120.8 (11)	O(2)—C(13)—C(14)	86.6 (15)
C(6)—C(5)—C(10)	121.4 (7)	C(14)—C(13)—C(1)	110.9 (11)
C(10)—C(5)—C(4)	117.8 (8)	C(13)—O(2)—C(15)	91.8 (14)
C(5)—C(6)—C(7)	123.0 (14)		

Table 1. Final atomic parameters and standard deviations (in parentheses)

The anisotropic temperature coefficients are in the form $T_i = \exp[-(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + 2b_{12}hk + 2b_{13}hl + 2b_{23}kl)]$. Fractional coordinates are $\times 10^4$ for the heavy atoms, and $\times 10^3$ for the H atoms. Anisotropic temperature coefficients are $\times 10^4$, isotropic *B*'s are $\times 10$.

	<i>x</i>	<i>y</i>	<i>z</i>	<i>b</i> ₁₁	<i>b</i> ₂₂	<i>b</i> ₃₃	<i>b</i> ₁₂	<i>b</i> ₁₃	<i>b</i> ₂₃
O(1)	2297 (4)	613 (7)	2365 (20)	20 (2)	81 (8)	314 (35)	3 (4)	-6 (9)	47 (16)
O(2)	106 (4)	1933 (9)	3393 (24)	10 (3)	112 (10)	752 (66)	-7 (4)	46 (11)	-41 (21)
C(1)	1448 (6)	2395 (9)	2103 (22)	31 (4)	65 (8)	31 (23)	2 (5)	-22 (11)	6 (2)
C(2)	2255 (6)	1807 (11)	2114 (29)	15 (3)	97 (11)	303 (44)	10 (5)	19 (15)	121 (25)
C(3)	3029 (5)	2510 (10)	2034 (29)	9 (3)	79 (10)	405 (57)	-3 (1)	18 (14)	101 (30)
C(4)	2967 (5)	3722 (9)	1775 (23)	4 (3)	93 (10)	131 (41)	-16 (5)	50 (9)	26 (20)
C(5)	2156 (7)	4303 (12)	1700 (40)	30 (5)	94 (13)	727 (106)	-9 (7)	-48 (22)	-156 (39)
C(6)	2099 (5)	5564 (11)	1394 (27)	3 (3)	99 (12)	378 (65)	-14 (5)	-37 (11)	-39 (25)
C(7)	1348 (10)	6166 (12)	1421 (38)	61 (7)	75 (11)	642 (103)	41 (8)	-94 (23)	-133 (32)
C(8)	609 (8)	5422 (11)	1500 (0)	46 (6)	65 (11)	792 (117)	-10 (7)	85 (25)	39 (37)
C(9)	648 (5)	4225 (8)	1763 (22)	14 (3)	58 (8)	227 (45)	4 (4)	24 (10)	-79 (16)
C(10)	1418 (6)	3594 (9)	1873 (18)	33 (4)	86 (9)	7 (18)	0 (5)	9 (10)	61 (17)
C(11)	3081 (6)	78 (9)	2283 (18)	30 (4)	56 (8)	86 (35)	1 (4)	-55 (10)	60 (16)
C(12)	3017 (15)	-1292 (15)	2383 (48)	180 (19)	95 (16)	1159 (160)	-14 (15)	419 (50)	-45 (48)
C(13)	719 (7)	1584 (10)	2386 (35)	34 (5)	52 (10)	548 (70)	6 (5)	-10 (19)	-14 (28)
C(14)	92 (7)	1654 (7)	764 (30)	57 (5)	5 (5)	269 (33)	-28 (6)	-130 (25)	37 (12)
C(15)	377 (9)	1216 (21)	4871 (37)	33 (6)	278 (32)	527 (95)	14 (12)	21 (20)	13 (45)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>		<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
H(C3)	360	215	200	28	H(C12)	280	-150	350	45
H(C4)	355	430	163	24	H'(C12)*	270	-160	138	45
H(C6)	360	600	135	33	H''(C12)	370	-180	255	45
H(C7)	133	704	115	35	H(C14)	35	180	-40	26
H(C8)	5	583	140	30	H'(C14)*	-50	190	121	26
H(C9)	15	370	168	21	H''(C14)*	-10	70	33	26
H(C13)	82	75	235	43	H(C15)	86	78	456	36
H(C11)	355	55	318	34	H'(C15)	-10	80	562	36
H'(C11)	341	42	83	34	H''(C15)*	55	200	585	36

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	Equation
(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	<i>l</i>	<i>m</i>	<i>n</i>
(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 [$d(\text{O}\cdots\text{O})=2.560$, $d(\text{O}-\text{H})=1.017$ Å, $\angle\text{O}-\text{H}\cdots\text{O}=178.1^\circ$] 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_3PO_4 at $\text{pH}\approx 2$. 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 *RFINE4* (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 $\sigma_{\text{count}}^2 + 0.0004F_o^2$.

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