

Table 3 (*cont.*)

Libration		<i>p</i> -Terphenyl	
Quaterphenyl			
Ω_{11}	1.43 (0.16) ($^{\circ}$) ² ($I_x = 12.8 \cdot 10^{-44}$ kg m ²)	Ω_{11}	3.58 (0.20) ($^{\circ}$) ² ($I_x = 5.5 \cdot 10^{-44}$ kg m ²)
Ω_{22}	178.3 (4.7) (central ring)	Ω_{22}	260.3 (7.5) (central ring)
Ω_{22}	71.5 (4.6) (outer ring)	Ω_{22}	68.9 (3.8) (outer ring)
Ω_{33}	1.59 (0.16) ($I_z = 12.0 \cdot 10^{-44}$ kg m ²)	Ω_{33}	2.74 (0.27) ($I_z = 5.0 \cdot 10^{-44}$ kg m ²)

are not rigid because of the important part of torsional movements of rings. In *p*-terphenyl the very large value of Ω_{22} for the central ring cannot be interpreted as resulting from usual harmonic motion even when taking into account low-frequency internal modes (Baudour *et al.*, 1974). Only a dynamic disorder can explain this result. The central ring moves in a double potential well whose minima are located at about 15° on either side of the mean position determined by X-ray diffraction. In quaterphenyl the value of Ω_{22} for the inner ring, 178 ($^{\circ}$)², although smaller than that in *p*-terphenyl is, however, large and shows unusual librational behaviour. Because of the similarity between the two molecular configurations we can interpret this as resulting from a dynamic disorder similar to that of *p*-terphenyl. Consequently for the two quaterphenyl central rings it can be shown that the minima of the double potential well are at about 11° on either side of the mean position determined by X-ray diffraction.

Structural transition in quaterphenyl

Oscillation and Weissenberg photographs obtained at low temperature (110 K) with the Renaud-Fourme apparatus (Renaud & Fourme, 1967) revealed a pseudomonoclinic superstructure. Neglecting the thermal contraction, this supercell corresponds to a

doubling of the *a* and *b* high-temperature cell parameters. This structural transition is comparable to that existing in *p*-terphenyl (Baudour *et al.*, 1975). Three-dimensional measurements are now in progress.

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Sodium Silicate Hydrates. V. The Crystal Structure of $\text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot 8\text{H}_2\text{O}$

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(Received 11 July 1975; accepted 7 August 1975)

The title compound is monoclinic, $P2_1/c$, $a = 8.96$, $b = 13.54$, $c = 9.99$ Å, $\beta = 119.6^\circ$, $Z = 4$. X-ray analysis shows that its constitutional formula is $\text{Na}_2(\text{H}_2\text{SiO}_4) \cdot 7\text{H}_2\text{O}$; it thus resembles other members of the series $\text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot n\text{H}_2\text{O}$ in containing isolated $\text{H}_2\text{SiO}_4^{2-}$ groups. Each Na is surrounded, roughly octahedrally, by six O atoms, mostly of water molecules. The geometries of the $\text{H}_2\text{SiO}_4^{2-}$ groups and of the Na–O frameworks of all the known members of the series are compared.

Introduction

The structures of all except one of the series of sodium silicate hydrates $\text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot n\text{H}_2\text{O}$ have been deter-

mined and the constitutional formulae shown to be $\text{Na}_2(\text{H}_2\text{SiO}_4) \cdot x\text{H}_2\text{O}$, where $x = n - 1$ (for $x = 4$ see Jost & Hilmer, 1966; $x = 5$, Jamieson & Dent Glasser, 1967, and Williams & Dent Glasser, 1971; $x = 8$, Jamieson & Dent Glasser, 1966a). The present work completes our knowledge of the structures of this series, and enables them to be compared.

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Experimental

The $\text{Na}_2\text{O} \cdot \text{SiO}_2 \cdot 8\text{H}_2\text{O}$ crystals used were prepared by crystallization from aqueous solution as described in part I (Jamieson & Dent Glasser, 1966*b*). Of all the hydrates in the series, this one has the narrowest field of stability, and is the most difficult to prepare. Crystal data are given in Table 1.

The crystal selected for intensity measurement was about $0.1 \times 0.2 \times 0.3$ mm. It was mounted about b , and intensities were measured over one half of reciprocal space on a Hilger and Watts Y190 Automatic Linear Diffractometer with $\text{Mo } K\alpha$ radiation. After equivalent reflexions had been averaged, and those rejected for which agreement was poor, just over 1000 independent reflexions remained. Intensities not significantly above

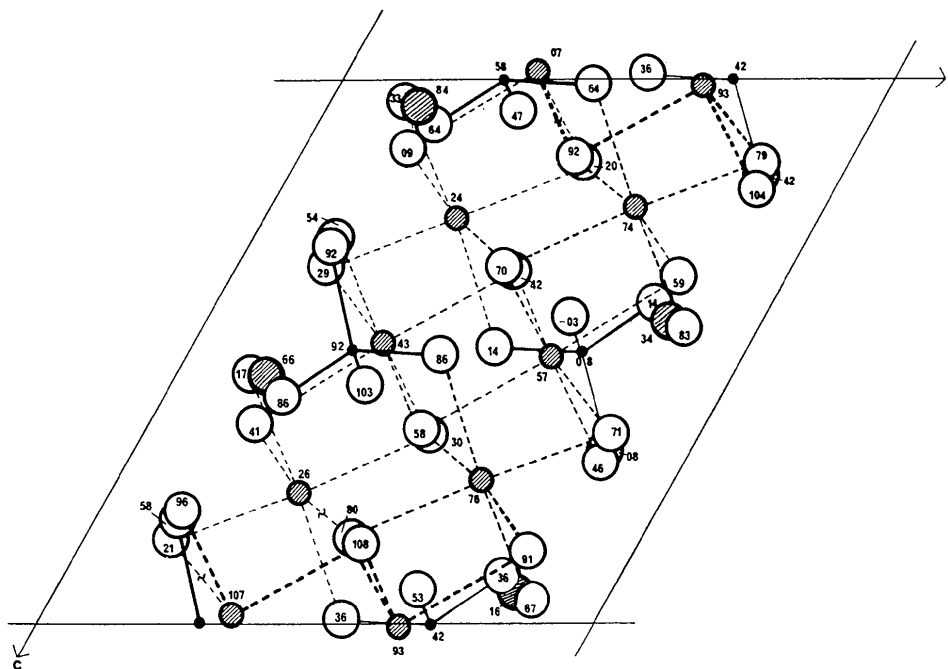


Fig. 1. Overall view of the structure; heights are given in $b/100$. Si are shown as small black circles; Na as medium cross-hatched circles. Large open circles represent O atoms or water molecules bonded to Na; the large cross-hatched circles are water molecules not bonded to Na. Si-O and Na-O bonds are represented as full and broken lines respectively, their thicknesses giving a rough indication of height in the cell. No hydrogen bonding is shown.

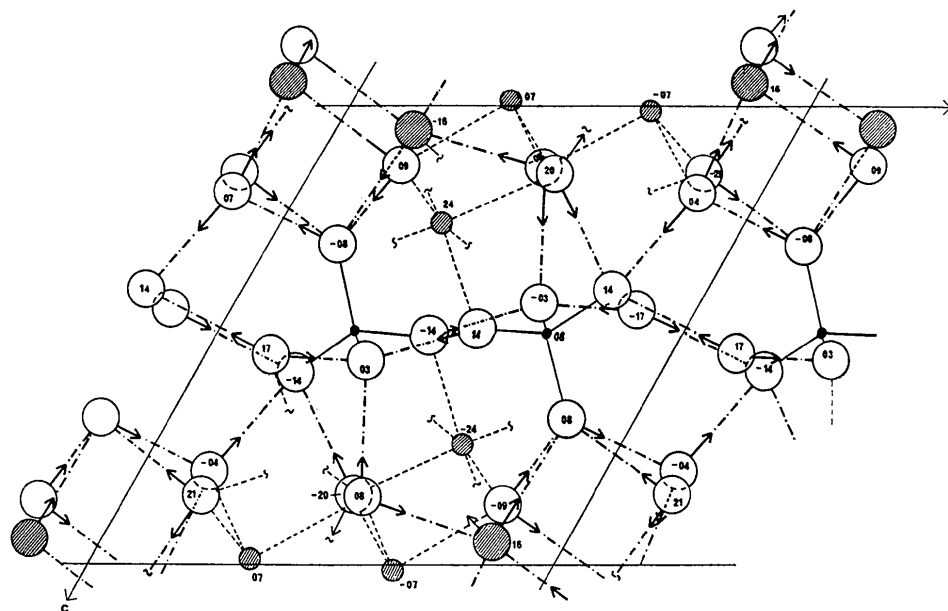


Fig. 2. Detail of a slab of the structure lying between $y = -\frac{1}{4}$ and $y = +\frac{1}{4}$. Hydrogen bonds are shown as dot-dashed lines, and the heads of the arrows represent the approximate position of the H atoms. Other symbols are as in Fig. 1.

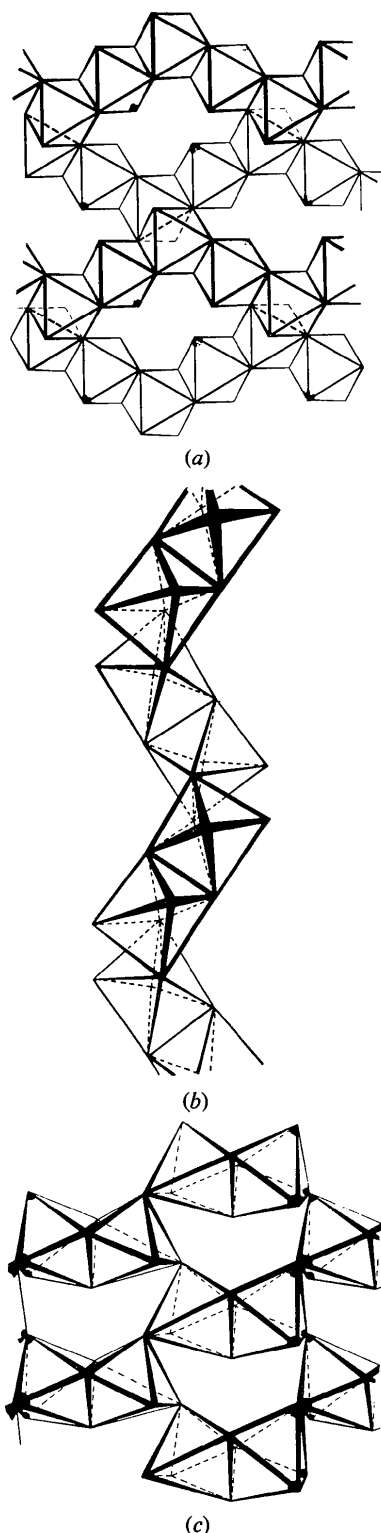


Fig. 3. (a) The Na-O framework in $\text{Na}_2\text{H}_2\text{SiO}_4 \cdot 7\text{H}_2\text{O}$ viewed perpendicular to (100). Shaded corners are hydroxyl ions and represent the points of attachment of H_2SiO_4 groups. (b) The chains of $\text{Na}(\text{H}_2\text{O})_6$ octahedra in $\text{Na}_2\text{H}_2\text{SiO}_4 \cdot 8\text{H}_2\text{O}$. (c) The Na-O framework in $\text{Na}_2\text{H}_2\text{SiO}_4 \cdot 5\text{H}_2\text{O}$. Shaded corners have the same significance as in (a).

Table 1. *Crystal data for* $\text{Na}_2\text{H}_2\text{SiO}_4 \cdot 7\text{H}_2\text{O}$

$a = 8.96 \text{ \AA}$	$d_m^* = 1.67 \text{ g cm}^{-3}$
$b = 13.54$	$Z = 4$
$c = 9.99$	$d_x = 1.67 \text{ g cm}^{-3}$
$\beta = 119.6^\circ$	

* From Baker, Woodward & Pabst (1933).

background were retained and treated as 'observed'; this provided a convenient way of handling very weak intensities. No corrections were made for absorption or extinction.

Initial processing of data was done on an Elliott 803 computer, mainly with programs of Daly, Stephens & Wheatley (1963). Later calculations were made on an ICL 4/70 computer, with programs based on those supplied by Dr F. R. Ahmed of the National Research Council of Canada and adapted by Mr J. S. Knowles of the University of Aberdeen Computing Centre. Scattering factors for Na, Si, O and H were taken from *International Tables for X-ray Crystallography* (1962).

Table 2. *Final atomic parameters for* $\text{Na}_2\text{H}_2\text{SiO}_4 \cdot 7\text{H}_2\text{O}$

Figures in brackets give the estimated standard deviation corresponding to the least significant digit.

	Coordinates (fractions of cell edge)			Isotropic temperature factor
	x	y	z	B
Na(1)	0.3447 (4)	0.2372 (4)	0.2618 (4)	2.77 (7)
Na(2)	0.3443 (4)	0.0742 (4)	0.9893 (4)	2.61 (6)
Si	0.7073 (3)	-0.0779 (2)	0.5014 (2)	1.50 (3)
O(1)	0.6502 (7)	0.0320 (6)	0.4397 (7)	2.4 (1)
O(2)	0.7905 (7)	0.1392 (6)	0.4168 (6)	2.0 (1)
O(3)	0.8496 (7)	0.0751 (6)	0.6916 (6)	2.0 (1)
O(4)	0.5447 (7)	0.1394 (6)	0.4926 (6)	1.9 (1)
O(5)	0.1594 (8)	0.2093 (6)	0.8458 (7)	2.6 (1)
O(6)	0.5025 (7)	0.0783 (6)	0.8552 (6)	2.4 (1)
O(7)	0.1909 (8)	0.0884 (7)	0.1317 (7)	3.2 (1)
O(8)	0.1366 (7)	0.1692 (6)	0.5434 (7)	2.4 (1)
O(9)	0.5205 (7)	0.1992 (6)	0.1532 (6)	2.1 (1)
O(10)	0.8531 (7)	0.0442 (6)	0.2052 (7)	2.5 (1)
O(11)	0.8382 (8)	0.1619 (6)	0.9487 (7)	2.6 (1)
H(31)	0.92 (1)	0.05 (1)	0.71 (1)	2.8*
H(41)	0.46 (1)	0.10 (1)	0.49 (1)	
H(51)	0.08 (1)	0.16 (1)	0.79 (1)	
H(52)	0.15 (1)	0.26 (1)	0.92 (1)	
H(61)	0.46 (1)	0.03 (1)	0.76 (1)	
H(62)	0.63 (1)	0.08 (1)	0.89 (1)	
H(71)	0.20 (1)	0.03 (1)	0.13 (1)	
H(72)	0.13 (1)	0.08 (1)	0.07 (1)	
H(81)	0.21 (1)	0.11 (1)	0.55 (1)	
H(82)	0.04 (1)	0.16 (1)	0.51 (1)	
H(91)	0.64 (1)	0.17 (1)	0.25 (1)	
H(92)	0.52 (1)	0.26 (1)	0.13 (1)	
H(101)	0.83 (1)	0.08 (1)	0.27 (1)	
H(102)	0.85 (1)	0.11 (1)	0.15 (1)	
H(111)	0.82 (1)	0.23 (1)	0.95 (1)	
H(112)	0.84 (1)	0.13 (1)	0.87 (1)	

* Estimated value, not refined.

H(31) and H(41) are attached to O(3) and O(4) which are therefore hydroxyl groups. A similar system is used for numbering the hydrogens attached to O(5)-O(11), which are the oxygen atoms of water molecules.

Structure determination

The structure was solved by direct methods. An initial set of signs was obtained by graphical application of the 'coincidence method' (Woolfson, 1961) and extended by repeated use of the basic sign relationship. A Fourier synthesis with 149 signs determined in this

way revealed the structure, and the positions of the non-hydrogen atoms were refined by electron density and difference maps, and block-diagonal least squares. A difference map calculated when $R=0.09$ gave plausible positions for the H atoms. Although inclusion of these did not significantly improve the agreement, they were retained in the refinement, which finally

Table 3. Summary of bond lengths (Å) and angles (°)

Figures in brackets give the estimated standard deviation corresponding to the least significant digit; bond lengths in Å, angles in degrees.

(a) Silicate tetrahedron

	Bond lengths		Coordination of O		
			Cation	H bonds to	H bonds from
Si-O(1)	1.595 (8)	oxygen atom	-	-	O(4), O(6), O(8)
O(2)	1.607 (7)	ditto	-	-	O(8), O(9), O(10), O(11)
O(3)	1.686 (6)	hydroxyl group	-	O(10)	O(5), O(7), O(11)
O(4)	1.643 (8)	ditto	Na(1)	O(1)	O(9), O(10)

(b) Sodium-oxygen octahedra

Bond lengths			
Na(1)-O(4)	2.490 (7)	Na(2)-O(5)	2.408 (9)
O(7)	2.425 (10)	O(6)	2.384 (8)
O(9)	2.369 (8)	O(7')	2.425 (9)
O(5)	2.316 (9)	O(9')	2.342 (8)
O(6)	2.795 (9)	O(6')	2.540 (9)
O(8)	2.413 (8)	O(10)	2.466 (8)

O-Na-O angles, subtended by adjacent oxygen atoms

Range	77.9-98.8 (3)	82.4-100.7 (3)
Mean	89.9	90.1

(c) Hydrogen bonding

			Angle at water molecule
O(3)...	O(10)	2.84 (1)	
O(4)...	O(1)	2.61 (1)	
O(5)...	O(3)	3.03 (1)	111.3 (3)
	O(8)	2.66 (1)	
O(6)...	O(1)	2.64 (1)	109.6 (3)
	O(11)	2.90 (1)	
O(7)...	O(3)	2.96 (1)	102.3 (3)
	O(11)	2.94 (1)	
O(8)...	O(1)	2.61 (1)	123.4 (3)
	O(2)	2.74 (1)	
O(9)...	O(2)	2.68 (1)	121.1 (3)
	O(4)	2.78 (1)	
O(10)...	O(2)	2.76 (1)	118.2 (3)
	O(4)	2.96 (1)	
O(11)...	O(2)	2.72 (1)	111.9 (3)
	O(3)	2.87 (1)	

(d) Environment of water molecules

Angle subtended at water (all e.s.d.'s 0.3°) given below coordinating atoms; omitted if given in (c).

(i) Roughly tetrahedral

						Mean*	Class†
O(5)	Na(1)Na(2)	Na(1)O(3)	Na(1)O(8)	Na(2)O(3)	Na(2)O(8)	108.1	2A
		92.8	130.2	114.1	92.0		
O(7)	Na(1)Na(2)	Na(1)O(3)	Na(1)O(11)	Na(2)O(3)	Na(2)O(11)	109.2	2A
		92.5	120.4	101.8	124.2		
O(8)	Na(1)O(1)	Na(1)O(2)	Na(1)O(5)	O(5)O(1)	O(5)O(2)	108.0	2H
		95.8	133.5	92.2	99.6		
O(9)	Na(1)Na(2)	Na(1)O(2)	Na(1)O(4)	Na(2)O(2)	Na(2)O(4)	108.7	2A
		96.1	95.6	110.7	116.0		
O(10)	Na(2)O(2)	Na(2)O(3)	Na(2)O(11)	O(3)O(2)	O(3)O(11)	108.4	2H
		124.7	95.5	87.7	119.8		

(ii) Roughly trigonal bipyramidal

	Angles to apices			Mean	Equatorial angles		Mean*	Class†
O(6)	Na(1)Na(2)	Na(1)O(1)	Na(1)O(11)	86.9	Na(2)O(1)	Na(2)O(11)	119.7	3O
		82.2	86.7					
O(11)	Na(2')Na(2)	Na(2')O(1)	Na(2')O(11)	94.0	119.8	129.6	120.0	3T
		86.0	108.4					
O(6)O(2)	O(6)O(2)	O(6)O(3)	O(6)O(10)	89.5	O(10)O(2)	O(10)O(3)	124.8	123.2
		105.7	92.4					
O(7)O(2)	O(7)O(2)	O(7)O(3)	O(7)O(10)	91.9	124.8	123.2	120.0	3T
		117.8	85.1					

* Including angles listed in (c).

† See text.

Table 4. *The geometry of the H₂SiO₄ group in Na₂H₂SiO₄·xH₂O*

	Bond distances (Å)			Bond angles (°)		
	Si-O	Si-OH	O-Si-O	O-Si-OH	OH-Si-OH	
	Mean	Mean		Mean		
x=8	1.591	1.672	116.9	107.8 } 108.0 }	107.9	105.9
x=7	1.595 } 1.607 }	1.686 } 1.643 }	115.0	109.5 } 110.2 } 109.8 }	109.5	103.2
x=5 X-ray	1.597 } 1.612 }	1.691 } 1.657 }	116.1	107.4 } 106.7 } 110.1 }	108.2	107.7
Neutron	1.599 } 1.591 }	1.698 } 1.672 }	116.6	108.0 } 107.4 } 110.2 }	108.3	106.8
x=4	1.61 } 1.61 }	1.70 } 1.64 }	116.3	108.2 } 107.6 } 110.4 }	109.4	101.7

converged with $R=0.0712$ for 935 reflexions; the poor agreements were all weak and were mainly confined to a few scattered measurements at relatively high angles.

Final positional and isotropic thermal parameters for the non-hydrogen atoms are given in Table 2. The approximate positional parameters of the H atoms are also given. In the final cycle the temperature factors of the H atoms, hitherto given an arbitrary value of about 2.8, were allowed to refine; none rose above 3.9 \AA^2 , although one became negative, so it was assumed that the positional parameters were indeed roughly correct.*

Description of the structure

Fig. 1 shows an overall view of the structure. Na-O octahedra, sharing edges, interlink with H₂SiO₄ tetrahedra to form layers parallel to (100). Fig. 2 gives a more detailed picture of the arrangement between $y = \pm \frac{1}{4}$, and shows how these layers are linked together by hydrogen bonding roughly perpendicular to (100). There is also extensive hydrogen bonding within the layers, which also contain an 'odd' water molecule, O(11), that is not connected to Na; this is distinguished by cross-hatching in Figs. 1 and 2. In Fig. 2 the heads of the arrows correspond, very roughly, to the positions of the H atoms. Bond lengths and angles are summarized in Table 3.

Fig. 3(a) shows the framework of Na-O octahedra (somewhat idealized) viewed along the perpendicular to (100). The arrangement is unusual in that the water molecule O(6) is coordinated by three Na atoms and two water molecules forming a rough trigonal bipyramid. In the classification of Ferraris & Franchini-Angela (1972) (which is based on and extended from that of Chidambaram, Sequiera & Sikka, 1964) this is type 3O environment; to the best of our knowledge this has been found only once before, in sodium perxenate octahydrate (Ibers, Hamilton & MacKenzie, 1964). The 'odd' water molecule that is not attached to

Na at all, O(11), also lies at the centre of a trigonal bipyramid, which consists of an O atom O(2) and a hydroxyl group O(3) attached to Si and three water molecules including O(6). This is the environment called 3T in the above classification, and seems also to be unusual: no example of it was given by Ferraris & Franchini-Angela. One is tempted to speculate that the combination of these two odd coordination features is reflected in the narrow stability field of the compound.

The remaining water molecules are all roughly tetrahedrally coordinated; the lone pairs point sometimes to two Na atoms (class 2A) and sometimes to one Na atom and to an H atom of a water molecule or hydroxyl group (2H).

Comparison with other members of the series

Table 4 summarizes the bond lengths and angles found in the H₂SiO₄ groups for the series Na₂H₂SiO₄·xH₂O. In the compound with $x=8$, the Si lies on a twofold axis, so the two Si-O and the two Si-OH bonds are related by symmetry. This is not true of the other three compounds, and it is found that, although in each case the two Si-O distances are not significantly different, the two Si-OH distances are (although barely so for the structure refined from neutron diffraction data), and moreover they are remarkably consistent for the three compounds. An explanation for the occurrence of two different Si-OH distances in the same silicate group was sought in the environment of the hydroxyl group: it has been suggested that the significant factor might be the strength of the hydrogen bond formed by the H of the hydroxyl group (Beagley, 1975). Table 5 analyses this; if the strength of the hydrogen bond formed is assumed to be related to the O...O distance, the observed Si-OH distances in the compounds with $x=7$ and $x=5$ are explained. Unfortunately this is not true when $x=4$. Nor can the differences be correlated with the total bonding to the O in the question, as a study of Table 5 will show.

The Na-O arrangements in the four structures may also be compared. When $x=8$, Na(H₂O)₆ octahedra share edges to give infinite chains [Fig. 3(b)] of composition [Na(H₂O)₄]_x. In the present ($x=7$) compound infinite chains of rather different conformation are

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31309 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 5. *Environment of the two different -OH groups in Na₂H₂SiO₄.xH₂O*

Values for x=5 are estimated 'best' values for X-ray and neutron refinements

x=7	←1.69→ ←2.84→ Si-----O-H···O	←1.64→ ←2.61→ Si-----O-H···O
	.	.
	.	.
	4H ₂ O at 2.84, 2.87, 2.96, 3.03	Na at 2.49 H ₂ O at 2.78
x=5	←1.68→ ←2.88→ Si-----O-H···O	←1.65→ ←2.61→ Si-----O-H···O
	.	.
	.	.
	2Na at 2.53, 2.53	Na at 2.30 H ₂ O at 3.16
x=4	←1.70→ ←2.72→ Si-----O-H···O	←1.64→ ←2.75→ Si-----O-H···O
	.	.
	.	.
	2H ₂ O at 2.75, 2.82	2Na at 2.38, 2.59

further cross-linked to form the 'net' or puckered sheet shown in Fig. 3(a). This relatively orderly behaviour does not extend to the compound with x=5; although this also contains octahedra linked into sheets, the linkages here are not through edges but

through faces and corners [Fig. 3(c)]. Finally, when x=4, (not illustrated) the coordination number of Na drops to five.

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The Structure of Hydrated 8'-Hydroxyzearealene, C₁₈H₂₂O₆.H₂O. An Estrogenic Syndrome-Producing Microtoxin*

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(Received 22 May 1975; accepted 7 July 1975)

8'-Hydroxyzearealene, C₁₈H₂₂O₆, is a microtoxin produced by the fungus *Gibberella zeare* (*Fusarium graminearum*). The material crystallizes in space group *P*2₁2₁2₁ with cell dimensions *a*=12.81 (4), *b*=16.13 (5) and *c*=8.61 (3) Å with *Z*=4. Counter techniques were used to collect 1611 independent reflections of which 1061 had intensities greater than 3σ(*I*). After renormalization of each parity group, the direct methods program *MULTAN* was used to calculate phases for 445 reflections with *E* values greater than 1.15. All 25 nonhydrogen atoms were located in the *E* map. The structure was refined by full-matrix least-squares techniques to a final *R* of 0.065 for 1061 reflections. The structure consists of a 14-membered lactone ring fused to a benzene ring. Some of the chemistry associated with the 14-membered ring can be rationalized in terms of the conformation. The structure is extensively hydrogen bonded.

Introduction

Zearealene is a microtoxin produced by *Gibberella zeare* (*Fusarium graminearum*) when the fungus is allowed to grow on maize under proper conditions of moisture and temperature. Infected grain fed to swine or isolates injected into laboratory animals produce

the estrogenic syndrome which involves primarily the genital system; this appears as vulva hypertrophy, occasional vaginal eversion in the female, preputial enlargement in the castrated male and prominent mammary gland enlargement in both sexes (Stob, Baldwin, Tuite, Andrews & Gillette, 1962; Christensen, Nelson & Mirocha, 1965; Mirocha & Christensen, 1971).

The structure of zearealene (I) was determined by

* FASTBIOS contribution No. 23.