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Location and Anisotropic Refinement of Hydrogen Atoms in Disodium Dihydrogen-silicate Tetrahydrate, Na₂H₂SiO₄.4H₂O, by Neutron Diffraction; Hydrogen Bonding at 173 K

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Abstract. $M_r = 212 \cdot 14$, triclinic, $P\bar{1}$, $a = 7 \cdot 916$ (2), $b = 9 \cdot 558$ (2), $c = 6 \cdot 643$ (1) Å, $\alpha = 70 \cdot 13$ (2), $\beta = 104 \cdot 83$ (2), $\gamma = 122 \cdot 56$ (2)°, $V = 396 \cdot 71$ Å³, $Z = 2$, $D_m = 1 \cdot 74$, $D_x = 1 \cdot 776$ Mg m⁻³, $\lambda = 0 \cdot 7017$ Å, $\mu(n) = 0 \cdot 204$ mm⁻¹, $F(000) = 4 \cdot 09$, $T = 173$ K, $R = 0 \cdot 028$ for 1498 independent reflections. Anisotropic structure refinement including absorption and secondary-extinction correction. The crystal structure of Na₂H₂SiO₄.4H₂O consists of linear chains of hydrogen-bonded [H₂SiO₄]²⁻ tetrahedra running along [110] and columns of edge-linked Na—O polyhedra running parallel to the c axis. The [H₂SiO₄]²⁻ tetrahedron shows the smallest HO—Si—OH angle observed in the series of Na₂H₂SiO₄.nH₂O ($n = 8, 7, 5, 4$) hydrates which is due to the arrangement of the hydroxyl H atoms. All Na atoms have coordination number (CN) 5 with short Na—O bonds [mean Na—O = $2 \cdot 37$ (2) Å]. CN = 4 with tetrahedral geometry is observed for the coordination polyhedra of the O atoms of all hydrate water molecules.

Introduction. Two series of crystalline hydrate phases exist in the alkali-rich section of the system Na₂O–SiO₂–H₂O: Na₃HSiO₄.nH₂O ($n = 5,2,1,0$) with an Na₂O:SiO₂ ratio of 3:2; and Na₂H₂SiO₄.nH₂O ($n = 8, 7, 5, 4$) with an Na₂O:SiO₂ ratio of 1:1. Members of the first series are built up of isolated [HSiO₄]³⁻ anions, as shown by the X-ray single-crystal structure analyses for Na₃HSiO₄.5H₂O (Smolin, Shepelev & Butikova, 1973)

and Na₃HSiO₄.2H₂O (Schmid, Huttner & Felsche, 1979; Schmid, Szolnay, Felsche & Huttner, 1981). The members of the second series are built up of isolated [H₂SiO₄]²⁻ anions as has been shown by X-ray and neutron single-crystal structure analyses: Na₂H₂SiO₄.8H₂O [Jamieson & Dent-Glasser, 1966a,b (X-ray); Schmid, Felsche & McIntyre, 1984 (neutron)], Na₂H₂SiO₄.7H₂O [Dent-Glasser & Jamieson, 1976 (X-ray)], Na₂H₂SiO₄.5H₂O [Jamieson & Dent-Glasser, 1967 (X-ray); Williams & Dent-Glasser, 1971 (neutron)] and Na₂H₂SiO₄.4H₂O [Jost & Hilmer, 1966 (X-ray)].

Na₂H₂SiO₄.4H₂O shows the most pronounced thermal stability within the 1:1 series of sodium silicate hydrates which all melt congruently at temperatures between 313 and 343 K (Felsche, Ketterer & Schmid, 1984). A distinct tendency to form supercooled melts has been observed for all four hydrate phases existing within this series. Glass-points at temperatures between 223 and 273 K have been observed by means of differential scanning calorimetry (Felsche, Ketterer & Schmid, 1984). In order to provide a better understanding of these thermal properties which are unique in oxosalt–hydrate crystal chemistry we will elaborate correlations with hydrogen bonding in the structures of all four hydrate phases based on neutron diffraction.

In this paper we will report on a single-crystal neutron diffraction study of Na₂H₂SiO₄.4H₂O at 173 K. The results of a 296 K experiment will be published separately.

Experimental. Single crystals of $Na_2H_2SiO_4.4H_2O$ were synthezised from aqueous solutions containing 45% Na_2O, 10% SiO_2 and 45% H_2O (by weight) at 296 K by isothermal crystal growth. The single-crystal quality was checked by polarizing microscopy. D_m was measured by the flotation method.

Neutron integrated intensities were measured on the D9 four-circle diffractometer at the ILL, Grenoble. A constant temperature of 173 K was maintained by an Air Products Displex cryorefrigerator (Allibon, Filhol, Lehmann, Mason & Simms, 1981). Data reduction was carried out using the program *COLL5N* (Lehmann & Wilson, 1982) which uses the step-scan profile-analysis algorithm of Lehmann & Larsen (1974). Program for structure analysis and refinement *SHELX*76 (Sheldrick, 1976); TR 440 computer; $w = m/[\sigma^2(F_o) + kF_o^2]$, from Poisson counting statistics, $m = 1.563$, $k = 0.0006$. Secondary-extinction correction applied using $F_o^* = F_o(1-0.0001gF_o^2/\sin\theta)$, where g was refined to 3.5; absorption correction applied. Two standard reflections ($\overline{7}43$ and $\overline{2}82$) measured every 50 observations, intensity variation 1%. Lattice parameters refined on the basis of 16 reflections. Neutron wavelength 0.7017 Å; Cu(200) monochromator. Total of 1997 reflections measured in the range $2\theta = 31-65°$; $(\sin\theta/\lambda)_{max} = 0.60$ Å^{-1}; min. h, k and l -9, -11 and 0; max. h, k and l 9, 11 and 8. Total number of unique reflections observed 1642; total number of observations used in the refinement 1498, with 144 unobserved $[I < 3\sigma(I)]$; $R_{int} = 0.0102$. Least-squares refinement based on F magnitudes. Total number of parameters refined 192; final $R = 0.0282$ and $wR = 0.0328$. Max. Δ/σ in the last cycle of refinement 0.001.

The H-atom positions were obtained by Fourier methods starting from phases calculated with the heavy-atom coordinates found by Jost & Hilmer (1966). All atomic parameters were then refined by full-matrix least squares with free site-occupation factors in the first isotropic refinement procedure. The site-occupation factors were held fixed in the final iso- and anisotropic refinement runs. Isotropic refinement gave a final R of 0.061. Scattering lengths for Si, Na, O and H were taken from the compilation of Koester & Rauch (1981).

Discussion. The final atomic parameters are listed in Table 1;† the bond lengths and angles observed in the hydrogen-bonding system and the H_2O and Na coordination polyhedra are listed in Table 2.

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† Lists of structure factors and anisotropic thermal parameters and a figure showing the hydrogen bonding around the $[H_2SiO_4]^{2-}$ anion have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39939 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Final atomic parameters for* $Na_2H_2SiO_4.$-$4H_2O$ at 173 K

The numbers in parentheses give the e.s.d.'s corresponding to the last significant digit. The equivalent U's are given as follows: $U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$ after diagonalization of the U tensor.

	x	y	z	$U_{eq}(Å^2)$
Na(1)	0.6080 (2)	0.1506 (2)	0.1865 (2)	0.0156 (7)
Na(2)	0.6513 (2)	0.3384 (2)	0.6315 (2)	0.0147 (7)
Si	0.2688 (2)	0.2330 (1)	0.9701 (2)	0.0068 (5)
O(1)	0.2594 (1)	0.0776 (1)	0.1721 (1)	0.0095 (4)
O(2)	0.2098 (1)	0.3567 (1)	0.0107 (1)	0.0107 (4)
O(3)	0.1281 (1)	0.1508 (1)	0.7618 (1)	0.0097 (4)
O(4)	0.4967 (1)	0.3434 (1)	0.8866 (1)	0.0117 (4)
O(5)	0.7899 (1)	0.3877 (1)	0.3282 (1)	0.0141 (5)
O(6)	0.1339 (1)	0.3739 (1)	0.3683 (2)	0.0152 (5)
O(7)	0.3538 (1)	0.0789 (1)	0.5828 (1)	0.0139 (4)
O(8)	0.7923 (1)	0.1957 (1)	0.9214 (1)	0.0154 (5)
H(31)	−0.0161 (3)	0.0627 (2)	0.7836 (3)	0.0193 (8)
H(41)	0.5919 (3)	0.4556 (2)	0.9211 (3)	0.0218 (8)
H(51)	0.7986 (3)	0.4880 (2)	0.2103 (3)	0.0248 (8)
H(52)	0.9196 (3)	0.3941 (2)	0.3470 (3)	0.0268 (9)
H(61)	0.1566 (3)	0.3477 (2)	0.2495 (3)	0.0256 (8)
H(62)	0.1323 (3)	0.2892 (2)	0.5013 (3)	0.0255 (9)
H(71)	0.3103 (3)	0.0795 (2)	0.4278 (3)	0.0232 (8)
H(72)	0.2580 (3)	0.0874 (2)	0.6368 (3)	0.0291 (9)
H(81)	0.7742 (3)	0.0945 (2)	0.8878 (3)	0.0257 (8)
H(82)	0.9379 (3)	0.2600 (2)	0.9576 (3)	0.0260 (8)

The crystal structure of $Na_2H_2SiO_4.4H_2O$ consists of linear chains of hydrogen-bonded $[H_2SiO_4]^{2-}$ tetrahedra running parallel to [110] and columns of edge-linked Na—O polyhedra with CN = 5 running parallel to the c axis (see Fig. 1). Fig. 2 shows the typical chain-like arrangement of the hydrogen-bonded $[H_2SiO_4]^{2-}$ tetrahedra.

All O atoms in the crystal structure of $Na_2H_2SiO_4.$-$4H_2O$ are involved in hydrogen bonding. As in the structure of $Na_2D_2SiO_4.8D_2O$ (Schmid, Felsche & McIntyre, 1984) all the hydrogen bonding is concentrated around the $[H_2SiO_4]^{2-}$ anion (see Table 2). All hydrogen-bonding data show normal behaviour, *i.e.* they fit well into the bond-length–bond-strength correlation schemes of Brown & Shannon (1973) and Donnay & Donnay (1973). Significant examples are: the $O(7)\cdots O(1)$ contact, the shortest and strongest in the structure, of 2.640 (2) Å corresponding to the long $O(7)—H(71)$ bond of 0.997 (2) Å and the longest $O\cdots O$ contact of 2.836 (2) Å between O(7) and O(3) with a corresponding short O—H bond of 0.968 (2) Å between O(7) and H(72). The hydrogen bonds in $Na_2H_2SiO_4.4H_2O$ also show normal behaviour with respect to their O—H$\cdots$O angular dimensions. The values measured range between 160 and 180° and correlate well with those compiled by Baur (1965), Ferraris & Franchini-Angela (1972), Chiari & Ferraris (1982) and Ferraris & Ivaldi (1984).

All O atoms of the hydrate water molecules in $Na_2H_2SiO_4.4H_2O$ have CN = 4 and almost tetrahedral coordination, angles ranging from 90 to 120° (see Table 2). With the exception of O(6), which forms only one Na—O contact but also an additional O$\cdots$H bond

Table 2. *Bond lengths (Å) and angles (°) in* Na$_2$H$_2$SiO$_4$.4H$_2$O, *with e.s.d.'s in parentheses corresponding to the last significant digit*

(*a*) Hydrogen-bonding system
The [H$_2$SiO$_4$]$^{2-}$ tetrahedron

H(31)—O(3)	1·000 (2)		
H(31)···O(1)	1·684 (2)		
O(3)···O(1)	2·683 (2)	O(3)—H(31)···O(1)	176·3 (2)
H(41)—O(4)	0·990 (2)		
H(41)···O(2)	1·745 (2)		
O(4)···O(2)	2·726 (2)	O(4)—H(41)···O(2)	170·5 (2)

The H$_2$O molecules

O(5)—H(52)	0·972 (2)		
O(5)—H(51)	0·991 (2)		
H(52)···O(6)	1·774 (2)		
O(5)···O(6)	2·738 (2)	O(5)—H(52)···O(6)	170·7 (2)
H(51)···O(2)	1·714 (2)		
O(5)···O(2)	2·701 (2)	O(5)—H(51)···O(2)	173·0 (2)
O(6)—H(62)	0·971 (2)		
O(6)—H(61)	0·987 (2)		
H(62)···O(3)	1·781 (2)		
O(6)···O(3)	2·747 (2)	O(6)—H(62)···O(3)	172·7 (2)
H(61)···O(2)	1·707 (2)		
O(6)···O(2)	2·671 (2)	O(6)—H(61)···O(2)	164·8 (2)
O(7)—H(72)	0·968 (2)		
O(7)—H(71)	0·997 (2)		
H(72)···O(3)	1·888 (2)		
O(7)···O(3)	2·836 (2)	O(7)—H(72)···O(3)	166·0 (2)
H(71)···O(1)	1·647 (2)		
O(7)···O(1)	2·640 (2)	O(7)—H(71)···O(1)	173·6 (2)
O(8)—H(82)	0·980 (2)		
O(8)—H(81)	0·993 (2)		
H(82)···O(2)	1·817 (2)		
O(8)···O(2)	2·790 (2)	O(8)—H(82)···O(2)	171·3 (2)
H(81)···O(1)	1·689 (2)		
O(8)···O(1)	2·683 (2)	O(8)—H(81)···O(1)	179·1 (2)

(*b*) Angles in the coordination polyhedra of the H$_2$O oxygen atoms (CN = 4)

H(52)—O(5)—H(51)	106·4 (2)	H(72)—O(7)—H(71)	107·2 (2)
H(52)—O(5)—Na(1)	96·6 (1)	H(72)—O(7)—Na(1)	108·6 (1)
H(52)—O(5)—Na(2)	118·5 (1)	H(72)—O(7)—Na(2)	100·0 (1)
H(51)—O(5)—Na(1)	107·3 (1)	H(71)—O(7)—Na(1)	118·8 (1)
H(51)—O(5)—Na(2)	115·6 (1)	H(71)—O(7)—Na(2)	112·1 (1)
Na(1)—O(5)—Na(2)	110·4 (1)	Na(1)—O(7)—Na(2)	108·4 (1)
H(62)—O(6)—H(61)	109·7 (2)	H(82)—O(8)—H(81)	102·9 (2)
H(62)—O(6)—H(52)	107·4 (1)	H(82)—O(8)—Na(1)	112·2 (1)
H(62)—O(6)—Na(2)	117·5 (1)	H(82)—O(8)—Na(2)	116·8 (1)
H(61)—O(6)—H(52)	119·6 (1)	H(81)—O(8)—Na(1)	118·8 (1)
H(61)—O(6)—Na(2)	110·7 (1)	H(81)—O(8)—Na(2)	110·2 (1)
H(52)—O(6)—Na(2)	91·3 (1)	Na(1)—O(8)—Na(2)	96·8 (1)

(*c*) Bond lengths in the Na—O polyhedra (CN = 5)

Na(1)—O(5)	2·311 (2)	Na(2)—O(5)	2·327 (2)
Na(1)—O(7)	2·333 (2)	Na(2)—O(6)	2·329 (2)
Na(1)—O(8)	2·353 (2)	Na(2)—O(4)	2·356 (2)
Na(1)—O(1)	2·439 (2)	Na(2)—O(8)	2·360 (2)
Na(1)—O(4)	2·548 (2)	Na(2)—O(7)	2·381 (2)

of 1·774 (2) Å to H(52), all O atoms of the H$_2$O molecules have two neighbouring Na atoms. The coordination types are class 2 *G* for O(6) and class 2 *A* for O(5), O(7) and O(8) after the classification of Ferraris & Franchini-Angela (1972). This almost tetrahedral coordination with two Na atoms or one Na and one H atom along the lone-pair orbitals of the O atom is one significant difference between Na$_2$H$_2$SiO$_4$.-

4H$_2$O and the other members of the 1:1 series which show mostly CN = 5 for their H$_2$O molecules. Although all the H$_2$O molecules in Na$_2$H$_2$SiO$_4$.4H$_2$O are similar with respect to their overall tetrahedral geometry, they differ significantly with respect to geometry and angles. It is O(6) with its 2 *G* type geometry which shows the largest H—O—H angle of 109·7 (2)° observed in this structure; H—O—H angles of O(5), O(7) and O(8) which show values of 106·4 (2), 107·2 (2) and 102·9 (2)°, respectively, are closer to the angle of 105·5° observed in the free H$_2$O molecule.
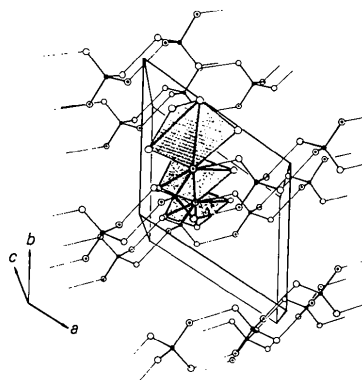

Fig. 1. The unit cell of Na$_2$H$_2$SiO$_4$.4H$_2$O (*ORTEP*; Johnson, 1965). The figure shows the chains of hydrogen-bonded [H$_2$SiO$_4$]$^{2-}$ anions running parallel to [110] and one column of Na(1)—O polyhedra which are edge linked and running parallel to [001]. The H atoms as well as the Na(2)—O polyhedra are omitted for clarity.
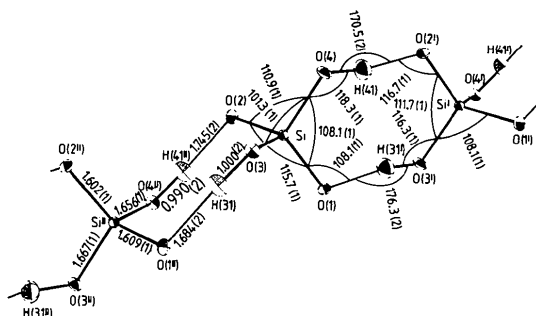
Fig. 2. Graphic representation of the [H$_2$SiO$_4$]$^{2-}$ chain in Na$_2$H$_2$SiO$_4$.4H$_2$O, showing the thermal ellipsoids of the Si, O and H atoms and the interatomic distances (Å) and angles (°) in the [H$_2$SiO$_4$]$^{2-}$ tetrahedron and the hydrogen bonds. The values are given with e.s.d.'s in parentheses.

The Na—O polyhedra in $Na_2H_2SiO_4.4H_2O$ show almost trigonal-bipyramidal geometry (CN = 5) which is a striking difference between this structure and those of the higher hydrates of the 1:1 series, all of which show octahedral coordination (CN = 6) for their Na—O polyhedra (Williams & Dent-Glasser, 1971; Jamieson & Dent-Glasser, 1967; Dent-Glasser & Jamieson, 1976; Schmid, Felsche & McIntyre, 1984). Taking into account bond-length–bond-strength considerations, bond-valence sums give 1 for both Na atoms. In this context it is interesting to see that in the Na(1)—O polyhedron one relatively long Na—O distance of 2·548 (2) Å [Na(1)—O(4)] is well compensated by the short Na(1)—O(5) bond of 2·311 (2) Å.

Bond-length–bond-strength correlations can also be found in the $[H_2SiO_4]^{2-}$ tetrahedron. The Si—OH bond lengths correlate well with calculated bond valences for these Si—O distances, i.e. taking into account the contributions to the valence sums of O(3) and O(4), the hydroxyl oxygens, from the corresponding bond valences of the O—H, O···H and O—Na contacts, thus yielding valence sums of nearly 2 for both O atoms. Hence, it is evident that the Si—O(4) bond of 1·656 (1) Å must be shorter than the Si—O(3) bond of 1·667 (1) Å taking into consideration the fact that O(3) forms two additional hydrogen bonds with O(6) and O(7) of two of the four crystallographically independent water molecules in that structure. In contrast, atom O(4) forms only one hydrogen bond with O(2) of one neighbouring tetrahedron (see Table 2). The non-hydroxyl O atoms, O(1) and O(2), also fit well into that scheme considering the contributions of all neighbouring atoms, i.e. the bond valences of the O···H and O—Na bonds, to the O(1) and O(2) valence sums calculated from universal and individual parameters (Brown & Shannon, 1973). The O—Si—O angles in the $[H_2SiO_4]^{2-}$ anion of $Na_2H_2SiO_4.4H_2O$ are in good agreement with those listed by Dent-Glasser & Jamieson (1976) for the whole series of 1:1 sodium silicate hydrates. The angular decrease in the sequence O—Si—O, O—Si—OH and HO—Si—OH is of the same magnitude, even though the HO—Si—OH angle of 101·3 (1)° is the smallest observed in the whole series – a fact which has already been mentioned by Jost & Hilmer (1966). This very low angle is due to electrostatic and geometrical reasons and the very special arrangement of the hydroxyl H atoms within the chain. The first reason is evident; the longer a given Si—O bond, the smaller the corresponding angles will be at a given constant O—O force of repulsion. However, this geometrical effect and the lowering of the force of repulsion by the H atoms of the hydroxyl groups, an effect which will certainly intensify the decrease of the HO—Si—OH angle, cannot sufficiently explain that distortion in the $[H_2SiO_4]^{2-}$ tetrahedron. The more important reason is evident from Fig. 2. Both of the hydroxyl H atoms, H(31) and H(41), point in opposite

directions in their general [110] arrangement along the chain. The H atoms are therefore perfectly separated, and thus the tetrahedra, by means of parallel hydrogen bonds. This arrangement is almost ideal because of the pairs of alternating donor and acceptor O atoms forming these parallel hydrogen bonds. This fact reduces the force of repulsion between the O atoms to a minimum which accounts for the low HO—Si—OH angle of 101·3 (1)°. In $Na_2H_2SiO_4.8H_2O$ with its isolated $[H_2SiO_4]^{2-}$ tetrahedron (Schmid, Felsche & McIntyre, 1984), bonded to neighbouring ones by means of hydrogen bonds from the hydroxyl groups which are in this case arranged in a neighbouring position (Williams & Dent-Glasser, 1971), this force of repulsion between both OH groups is higher and results in a larger HO—Si—OH angle.

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