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Rerefinement of sodium thiosulfate pentahydrate

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The crystal structure of sodium thiosulfate pentahydrate, $Na_2S_2O_3.5H_2O$, has been refined to an R value of 0.022 for 1621 observed reflections. All the water-H atoms were located.

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(S–O) = 0.001 Å
R factor = 0.022
wR factor = 0.060
Data-to-parameter ratio = 10.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

The crystal structure of the title compound was determined by Taylor & Beevers (1952) using two-dimensional X-ray diffraction data. El Saffar (1968) reported the positional parameters of H atoms deduced from NMR studies. Padmanabhan *et al.* (1971) refined the structure with two-dimensional neutron diffraction data which confirmed the H-bonding scheme proposed by Taylor & Beevers (1952) with the exception of atom H9. In all these works, the role of the H atom H9 remained inconclusive.

Up to now, the highest precision refinement of the structure of sodium thiosulfate pentahydrate was that reported by Uraz & Armagan (1977) who refined the structure with densitometer-measured photographic data to an R value of 0.089 for 1359 observed reflections. The s.u.'s of the bond lengths for non-H atoms ranged from 0.003 for S1—S2 to 0.007 Å for Na1—O4 and Na1—O7 and those of bond angles from 0.2 to 0.3°. The present work reports the results of a much higher precision refinement of the structure with an R value of 0.022 for 1615 reflections. The s.u.'s for bond lengths range from 0.0005 Å for S1—S2 to 0.0013 Å for Na1—O4 and Na1—O7. while those of bond angles are 0.02–0.06°.

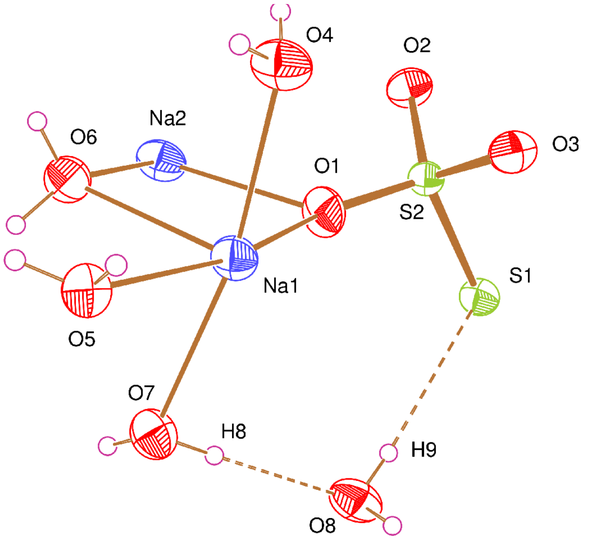

Figure 1
An *ORTEP*-3 (Farrugia, 1997) plot of the atoms in the asymmetric unit with 50% probability displacement ellipsoids for non-hydrogen atoms.

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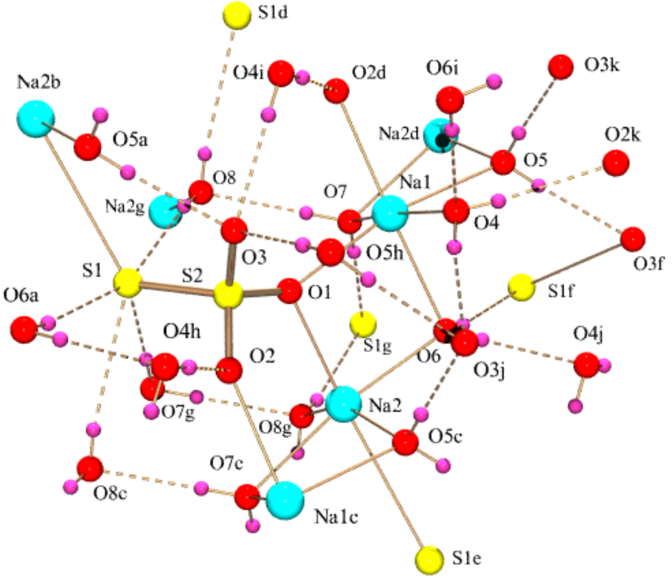

Figure 2
Diagram showing atoms of the asymmetric unit and their intra- and intermolecular contacts, including the hydrogen bonds, and the coordination around the Na atom. Symmetry codes: (*a*) $x-1, y, z-1$; (*b*) $x, y, 1+z$; (*c*) $x-1, y, z$; (*d*) $1+x, y, z$; (*e*) $x, y, 1+z$; (*f*) $1+x, y, 1+z$; (*g*) $1-x, -y, 1-z$; (*h*) $x-1, \frac{1}{2}-y, z-\frac{1}{2}$; (*i*) $x, \frac{1}{2}-y, z-\frac{1}{2}$; (*j*) $x, \frac{1}{2}-y, \frac{1}{2}-z$; (*k*) $1+x, \frac{1}{2}-y, \frac{1}{2}-z$.

The hydrogen-bonding scheme suggested by Uraz & Armagan (1977) is confirmed. However, the O—H distances, which vary from 0.75 (2) to 0.83 (2) Å, are much shorter than those obtained by Uraz & Armagan (1977). H—O—H angles are close to the tetrahedral value, varying from 96 (2) to 106 (2)°.

Experimental

Crystal data

$Na_2S_2O_3 \cdot 5H_2O$
$M_r = 248.2$
Monoclinic, $P2_1/c$
$a = 5.947$ (1) Å
$b = 21.574$ (2) Å
$c = 7.526$ (1) Å
$\beta = 103.82$ (1)°
$V = 937.6$ (2) Å^3
$Z = 4$
$D_x = 1.758$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 9.2$–$15.4°$
$\mu = 0.67$ mm^{-1}
$T = 293$ (2) K
Hexagonal prism, colourless
0.65 × 0.35 × 0.30 mm

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
Profile data from ω scans
Absorption correction: none
1637 measured reflections
1615 independent reflections
1571 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\rm max} = 25.0°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 25$
$l = -8 \rightarrow 8$
3 standard reflections every 50 reflections intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.060$
$S = 1.09$
1615 reflections
150 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0424P)^2 + 0.2426P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.48$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.34$ e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.0413 (25)

Table 1
Selected geometric parameters (Å, °).

S1—S2	2.0159 (5)	S2—O2	1.4653 (10)
S1—Na2$^{\rm i}$	3.3192 (8)	S2—O3	1.4759 (10)
S2—O1	1.4581 (11)		
O1—S2—O2	110.93 (7)	H1—O4—H2	105 (4)
O1—S2—O3	111.25 (6)	H3—O5—H4	106 (3)
O2—S2—O3	109.56 (6)	H5—O6—H6	97 (3)
O1—S2—S1	108.71 (5)	H7—O7—H8	106 (3)
O2—S2—S1	109.10 (5)	H9—O8—H10	102 (4)
O3—S2—S1	107.19 (4)		

Symmetry code: (i) $x, y, z-1$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O4—H1···O2$^{\rm i}$	0.76 (2)	2.10 (2)	2.847 (2)	169 (2)
O4—H2···O3$^{\rm ii}$	0.77 (2)	2.06 (2)	2.826 (2)	171 (2)
O5—H3···O3$^{\rm iii}$	0.81 (2)	2.10 (2)	2.900 (2)	169 (2)
O5—H4···O3$^{\rm i}$	0.80 (2)	2.00 (2)	2.803 (2)	175 (2)
O6—H5···S1$^{\rm iii}$	0.82 (2)	2.54 (2)	3.353 (3)	173 (2)
O6—H6···O4$^{\rm ii}$	0.83 (2)	2.03 (2)	2.857 (2)	178 (2)
O7—H7···S1$^{\rm iv}$	0.76 (2)	2.59 (2)	3.340 (2)	170 (2)
O7—H8···O8	0.83 (2)	2.01 (2)	2.843 (2)	178 (2)
O8—H9···S1	0.75 (2)	2.62 (2)	3.360 (2)	170 (2)
O8—H10···S1$^{\rm v}$	0.82 (2)	2.55 (2)	3.337 (2)	159 (2)

Symmetry codes: (i) $1+x, \frac{1}{2}-y, \frac{1}{2}+z$; (ii) $x, \frac{1}{2}-y, \frac{1}{2}+z$; (iii) $1+x, y, 1+z$; (iv) $1-x, -y, 1-z$; (v) $1+x, y, z$.

The X-ray diffraction data of the crystal were collected by mistake as it was supposed to be a crystal of a calcium complex of aspartic acid. When all attempts to solve the structure failed, the structure was refined blindfold, *i.e.* all atoms were assumed to be of the same type, namely oxygen, with all significant peaks obtained in the *E* map computed with the phase set with the highest combined figure of merit, varying the occupany factors of all the atoms except one. The structure refined rapidly and was then identified as that of sodium thiosulfate pentahydrate. This is an excellent demonstration of the power of direct methods in revealing structures when nothing is known about the crystal composition.

All H atoms belonging to the five water molecules were located from a difference map and refined.

Data collection: *CAD*-4 *Software* (Enraf–Nonius,1989); cell refinement: *CAD*-4 *Software*; data reduction: *CAD*-4 *Software*; program(s) used to solve structure: *MULTAN*80 (Main *et al.*, 1980); program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *ORTEP*-3 (Farrugia, 1997).

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References

El Saffar, Z. M. (1968). *Acta Cryst.* B**24**, 1131–1133.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN*80. Universities of York, England, and Louvain, Belgium.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Padmanabhan, V. M., Yadava, V. S., Navarro, Q. O., Garcia, A., Karsono, L., Suh, I.-H. & Chien, L. S. (1971). *Acta Cryst.* B**27**, 253–257.

Taylor, P. G. & Beevers, C. A. (1952). *Acta Cryst.* **5**, 341–344.

Sheldrick, G. M. (1993). *SHELXL*93. University of Göttingen, Germany.

Uraz, A. A. & Armagan, N. (1977). *Acta Cryst.* B**33**, 1396–1399.