

Redetermination of the Tutton's salt Cs₂[Cu(H₂O)₆](SO₄)₂

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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{S-O}) = 0.003$ Å; R factor = 0.034; wR factor = 0.080; data-to-parameter ratio = 29.4.

The crystal structure of dicaesium hexaquaacopper(II) bis-[sulfate(VI)], Cs₂[Cu(H₂O)₆](SO₄)₂, has been redetermined from single-crystal X-ray diffraction data. In comparison with the previous refinement based on single-crystal neutron data [Shields & Kennard (1972). *Cryst. Struct. Commun.* **1**, 189–191], the results show an improved precision and benefit from inclusion of anisotropic displacement parameters for the non-H atoms. The structure is characterized by a mean Cu–O bond length of 2.094 Å. The [Cu(H₂O)₆] octahedron ($\bar{1}$ symmetry) is strongly distorted because of the Jahn–Teller effect, with a calculated distortion parameter Δ of 0.0054. The Cs⁺ cation is ninefold coordinated by seven O atoms and two water molecules, with a mean Cs–O bond length of 3.238 Å.

Related literature

For related structures of other $M^I_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$ salts, see: Cotton *et al.* (1993) ($M = \text{NH}_4$); Simmons *et al.* (2006) ($M = \text{K}$); Ballirano & Belardi (2007) ($M = \text{Rb}$). The distortion of the [Cu(H₂O)₆] octahedron was calculated according to Brown & Shannon (1973). A reasonable hydrogen-bonding geometry was obtained after value normalization following the procedure described by Jeffrey & Lewis (1978) and Taylor & Kennard (1983).

Experimental

Crystal data

Cs ₂ [Cu(H ₂ O) ₆](SO ₄) ₂	$V = 730.14$ (11) Å ³
$M_r = 629.61$	$Z = 2$
Monoclinic, $P2_1/a$	Mo $K\alpha$ radiation
$a = 9.4383$ (6) Å	$\mu = 6.76$ mm ⁻¹
$b = 12.7605$ (12) Å	$T = 293$ (2) K
$c = 6.3130$ (6) Å	$0.30 \times 0.20 \times 0.20$ mm
$\beta = 106.199$ (7)°	

Data collection

Siemens P4 diffractometer	3208 independent reflections
Absorption correction: ψ scan	2728 reflections with $I > 2\sigma(I)$
(North <i>et al.</i> , 1968)	$R_{\text{int}} = 0.031$
$T_{\text{min}} = 0.226$, $T_{\text{max}} = 0.584$	3 standard reflections
(expected range = 0.100–0.259)	every 47 reflections
3436 measured reflections	intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$	109 parameters
$wR(F^2) = 0.081$	Only H-atom coordinates refined
$S = 1.09$	$\Delta\rho_{\text{max}} = 1.53$ e Å ⁻³
3208 reflections	$\Delta\rho_{\text{min}} = -2.57$ e Å ⁻³

Table 1

Selected geometric parameters (Å, °).

Cu–OW3	1.965 (2)	Cs–O2 ⁱⁱ	3.179 (3)
Cu–OW1	2.005 (2)	Cs–O1 ⁱⁱⁱ	3.182 (2)
Cu–OW2	2.311 (2)	Cs–OW2 ^{iv}	3.233 (3)
Cs–O1	3.098 (2)	Cs–O2 ⁱⁱⁱ	3.239 (3)
Cs–O4 ⁱ	3.114 (2)	Cs–OW1	3.422 (3)
Cs–O3 ⁱⁱ	3.151 (3)	Cs–O2 ⁱ	3.528 (3)

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
OW1–H11 \cdots O3	0.77 (7)	1.97 (7)	2.714 (2)	166 (7)
OW1–H12 \cdots O4 ^v	0.75 (7)	1.99 (7)	2.738 (4)	173 (7)
OW2–H21 \cdots O2 ^{vi}	0.87 (6)	1.91 (7)	2.777 (3)	174 (7)
OW2–H22 \cdots O4 ⁱ	0.74 (6)	2.04 (6)	2.779 (4)	171 (6)
OW3–H31 \cdots O3 ^{viii}	0.74 (7)	2.02 (7)	2.725 (3)	155 (7)
OW3–H32 \cdots O1 ^{viii}	0.71 (7)	1.99 (6)	2.693 (3)	171 (6)

Symmetry codes: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, z - 1$; (v) $x, y, z - 1$; (vi) $x - 1, y, z - 1$; (vii) $-x, -y, -z + 1$; (viii) $-x + \frac{1}{2}, y - \frac{1}{2}, -z + 1$.

Data collection: XSCANS (Siemens, 1994); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ATOMS (Dowty, 2003); software used to prepare material for publication: PARST (Nardelli, 1982) and publCIF (Westrip, 2007).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2127).

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supplementary materials

Acta Cryst. (2007). E63, i164-i165 [doi:10.1107/S1600536807029790]

Redetermination of the Tutton's salt $\text{Cs}_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$

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Comment

The title compound $\text{Cs}_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$ is a member of the isotypic series known as Tutton's salts. Their general formula is $M^I_2[M^{II}(\text{H}_2\text{O})_6](\text{XO}_4)_2$, where M^I and M^{II} are, respectively, a monovalent and a divalent cation and X is hexavalent S, Se or Cr. The structure of Tutton's salts consists of isolated $[M^{II}(\text{H}_2\text{O})_6]$ octahedra and XO_4^{2-} tetrahedra connected by the M^I cation (Fig. 1). As a result sheets parallel to (100) are formed, interconnected *via* medium to weak hydrogen bonds originating from the aqua ligands of the M^{II} cations to the acceptors of the sulfate groups.

The structure of the title compound has been previously determined by Shields & Kennard (1972) from single-crystal neutron data. However, the original data were characterized by large standard uncertainties of cell parameters and fractional coordinates. Moreover, no displacement parameters were reported. Because of the requirements of accurate structural data to rationalize the crystal chemistry of Tutton's salts, we decided to carry out a redetermination of the crystal structure of $\text{Cs}_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$ based on single-crystal X-ray data. The refined cell parameters and the cell volume differ significantly from the reference data [$P2_1/a$, $a = 9.42$ (1), $b = 12.785$ (9), $c = 6.28$ (1) Å, $\beta = 105.9$ (1)°, (Shields & Kennard, 1972)]. The sulfate group is slightly distorted and, similarly to all Tutton's salts, the S—O2 distance is smaller (1.463 (2) Å) than the remaining three S—O distances (1.474 (2)–1.484 (2) Å) because of bond-valence requirements. The mean Cu—O distance of the $[\text{Cu}(\text{H}_2\text{O})_6]$ octahedron is 2.094 Å. This value compares favourably with the range between 2.085 and 2.098 Å as reported for other $M^I_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$ salts (NH₄: Cotton *et al.*, 1993; K: Simmons *et al.*, 2006; Rb: Ballirano & Belardi, 2007). The $[\text{Cu}(\text{H}_2\text{O})_6]$ octahedron is strongly distorted as a result of the Jahn-Teller effect. The calculated distortion parameter Δ , as defined by Brown & Shannon (1973), amounts to 0.0054. The Cs^+ cation is coordinated by seven O atoms and two water molecules with a mean Cs—O distance of 3.238 Å. The refined hydrogen atom positions are not defined with high accuracy. However, after value normalization following the procedure given by Jeffrey & Lewis (1978) and Taylor & Kennard (1983), reasonable H...O distances in the range between 1.765 and 1.850 Å were obtained. Both O1 and O2 atoms are acceptors of a single hydrogen bond, whereas O3 and O4 are acceptors of two hydrogen bonds. As a result, a shortening of the distances H32...O1 and of S—O2 is observed.

Experimental

The title compound has been prepared by dissolving Cs_2SO_4 (Carlo Erba, P.P.A.) and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ (Carlo Erba, P.P.A.) in double deionized water and subsequent slow evaporation of the solvent at 295 K. The obtained crystals, in equilibrium with the mother solution, were removed by filtration and dried at room temperature. The product consisted of platy blue crystals of $\text{Cs}_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$.

Refinement

The structure was refined in the non-standard setting $P2_1/a$ of space group $P2_1/c$ for a better comparison with other Tutton's salts. After a few refinement cycles using neutral scattering factors, fully ionized for Cu and half ionized for O and neutral scattering factors for the remaining atoms were finally selected, leading to the lowest R agreement indices. The coordinates of the hydrogen atoms were refined freely using a common U_{iso} parameter of 0.05 \AA^2 . The deepest hole and the highest peak are, respectively, 0.69 and 0.75 \AA from atom Cs.

Figures

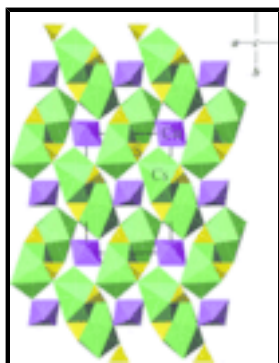


Fig. 1. The crystal structure of $\text{Cs}_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$ in polyhedral representation, projected along $[001]$.

Dicaesium hexaaquacopper(II) bis[sulfate(VI)]

Crystal data

$\text{Cs}_2[\text{Cu}(\text{H}_2\text{O})_6](\text{SO}_4)_2$

$M_r = 629.61$

Monoclinic, $P2_1/a$

Hall symbol: $-P 2yab$

$a = 9.4383 (6) \text{ \AA}$

$b = 12.7605 (12) \text{ \AA}$

$c = 6.3130 (6) \text{ \AA}$

$\beta = 106.199 (7)^\circ$

$V = 730.14 (11) \text{ \AA}^3$

$Z = 2$

$F_{000} = 613.8$

Non-standard setting of space group $P2_1/c$ to adhere to reference data of other Tutton's salts

$D_x = 2.864 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 58 reflections

$\theta = 4.0\text{--}22.5^\circ$

$\mu = 6.76 \text{ mm}^{-1}$

$T = 293 (2) \text{ K}$

Plate, blue

$0.30 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Siemens P4 diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293(2) \text{ K}$

ω scans

$R_{\text{int}} = 0.031$

$\theta_{\text{max}} = 35.0^\circ$

$\theta_{\text{min}} = 2.8^\circ$

$h = -15 \rightarrow 14$

$k = 0 \rightarrow 20$

Absorption correction: ψ scan
(North *et al.*, 1968) $l = 0 \rightarrow 10$
 $T_{\min} = 0.226$, $T_{\max} = 0.584$ 3 standard reflections
 3436 measured reflections every 47 reflections
 3208 independent reflections intensity decay: 1%
 2728 reflections with $I > 2\sigma(I)$

Refinement

Refinement on F^2 Only H-atom coordinates refined
 Least-squares matrix: full $w = 1/[\sigma^2(F_o^2) + (0.0466P)^2 + 0.5369P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $R[F^2 > 2\sigma(F^2)] = 0.034$ $(\Delta/\sigma)_{\max} = 0.001$
 $wR(F^2) = 0.081$ $\Delta\rho_{\max} = 1.53 \text{ e } \text{\AA}^{-3}$
 $S = 1.09$ $\Delta\rho_{\min} = -2.57 \text{ e } \text{\AA}^{-3}$
 3208 reflections Extinction correction: SHELXL97,
 $F_c^* = kFc[1 + 0.001x Fc^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 109 parameters Extinction coefficient: 0.080 (2)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cu	0.0000	0.0000	0.0000	0.01692 (10)
Cs	0.11407 (2)	0.354561 (15)	0.35562 (3)	0.02654 (8)
S	0.37975 (7)	0.14584 (5)	0.73909 (10)	0.01703 (11)
O1	0.3992 (3)	0.24027 (18)	0.6156 (4)	0.0273 (4)
O2	0.5138 (3)	0.0821 (2)	0.7893 (5)	0.0314 (5)
O3	0.2560 (2)	0.08353 (19)	0.6000 (3)	0.0250 (4)
O4	0.3439 (3)	0.1767 (2)	0.9448 (4)	0.0280 (4)
OW1	0.1450 (3)	0.1085 (2)	0.1577 (4)	0.0252 (4)
OW2	-0.1908 (3)	0.1109 (2)	0.0139 (4)	0.0297 (5)
OW3	-0.0002 (2)	-0.06342 (18)	0.2836 (3)	0.0205 (3)
H11	0.184 (7)	0.093 (5)	0.277 (11)	0.050*
H12	0.199 (7)	0.122 (5)	0.094 (11)	0.050*
H21	-0.281 (7)	0.099 (5)	-0.063 (10)	0.050*
H22	-0.181 (7)	0.166 (5)	-0.017 (11)	0.050*

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H31	-0.071 (7)	-0.051 (5)	0.310 (11)	0.050*
H32	0.034 (7)	-0.113 (5)	0.308 (10)	0.050*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu	0.01750 (19)	0.0183 (2)	0.01490 (17)	-0.00407 (15)	0.00436 (13)	0.00055 (14)
Cs	0.02807 (11)	0.02663 (11)	0.02645 (10)	0.00196 (6)	0.01012 (7)	0.00193 (6)
S	0.0157 (2)	0.0182 (2)	0.0169 (2)	-0.00196 (19)	0.00399 (18)	0.00030 (19)
O1	0.0344 (11)	0.0214 (9)	0.0283 (10)	-0.0026 (8)	0.0124 (9)	0.0051 (8)
O2	0.0188 (9)	0.0314 (12)	0.0407 (12)	0.0036 (8)	0.0031 (8)	0.0038 (10)
O3	0.0203 (8)	0.0315 (11)	0.0219 (8)	-0.0090 (8)	0.0039 (7)	-0.0048 (8)
O4	0.0334 (11)	0.0330 (11)	0.0197 (8)	-0.0064 (9)	0.0112 (8)	-0.0049 (8)
OW1	0.0259 (10)	0.0295 (11)	0.0197 (8)	-0.0102 (8)	0.0055 (7)	-0.0012 (7)
OW2	0.0231 (10)	0.0280 (11)	0.0390 (12)	0.0030 (9)	0.0101 (9)	0.0051 (10)
OW3	0.0218 (9)	0.0220 (9)	0.0197 (8)	0.0005 (7)	0.0089 (7)	0.0030 (7)

Geometric parameters (\AA , $^\circ$)

Cu—OW3	1.965 (2)	S—O1	1.474 (2)
Cu—OW3 ⁱ	1.965 (2)	S—O3	1.482 (2)
Cu—OW1	2.005 (2)	S—O4	1.484 (2)
Cu—OW1 ⁱ	2.005 (2)	S—Cs ^v	3.7066 (7)
Cu—OW2 ⁱ	2.311 (2)	S—Cs ^{vi}	3.7677 (7)
Cu—OW2	2.311 (2)	S—Cs ^{vii}	3.9035 (8)
Cs—O1	3.098 (2)	O1—Cs ^v	3.182 (2)
Cs—O4 ⁱⁱ	3.114 (2)	O2—Cs ^{vi}	3.179 (3)
Cs—O3 ⁱⁱⁱ	3.151 (3)	O2—Cs ^v	3.239 (3)
Cs—O2 ⁱⁱⁱ	3.179 (3)	O2—Cs ^{vii}	3.528 (3)
Cs—O1 ^{iv}	3.182 (2)	O3—Cs ^{vi}	3.151 (3)
Cs—OW2 ^v	3.233 (3)	O4—Cs ^{vii}	3.114 (2)
Cs—O2 ^{iv}	3.239 (3)	OW1—H11	0.77 (7)
Cs—OW1	3.422 (3)	OW1—H12	0.76 (7)
Cs—O2 ⁱⁱ	3.528 (3)	OW2—Cs ^{iv}	3.233 (3)
Cs—S ^{iv}	3.7066 (7)	OW2—H21	0.87 (7)
Cs—S ⁱⁱⁱ	3.7677 (7)	OW2—H22	0.74 (6)
Cs—S ⁱⁱ	3.9035 (8)	OW3—H31	0.75 (7)
Cs—H11	3.47 (6)	OW3—H32	0.70 (7)
S—O2	1.463 (2)		
OW3—Cu—OW3 ⁱ	180.00 (17)	O2 ^{iv} —Cs—OW1	127.78 (6)
OW3—Cu—OW1	90.34 (9)	O1—Cs—O2 ⁱⁱ	126.34 (6)
OW3 ⁱ —Cu—OW1	89.66 (9)	O4 ⁱⁱ —Cs—O2 ⁱⁱ	42.23 (6)
OW3—Cu—OW1 ⁱ	89.66 (9)	O3 ⁱⁱⁱ —Cs—O2 ⁱⁱ	82.25 (6)
OW3 ⁱ —Cu—OW1 ⁱ	90.34 (9)	O2 ⁱⁱⁱ —Cs—O2 ⁱⁱ	61.70 (8)

OW1—Cu—OW1 ⁱ	180.00 (16)	O1 ^{iv} —Cs—O2 ⁱⁱ	125.49 (6)
OW3—Cu—OW2 ⁱ	88.94 (9)	OW2 ^v —Cs—O2 ⁱⁱ	48.26 (6)
OW3 ⁱ —Cu—OW2 ⁱ	91.06 (9)	O2 ^{iv} —Cs—O2 ⁱⁱ	137.77 (8)
OW1—Cu—OW2 ⁱ	90.60 (10)	OW1—Cs—O2 ⁱⁱ	82.32 (6)
OW1 ⁱ —Cu—OW2 ⁱ	89.40 (10)	O2—S—O1	109.96 (15)
OW3—Cu—OW2	91.06 (9)	O2—S—O3	108.73 (15)
OW3 ⁱ —Cu—OW2	88.94 (9)	O1—S—O3	108.61 (14)
OW1—Cu—OW2	89.40 (10)	O2—S—O4	110.66 (16)
OW1 ⁱ —Cu—OW2	90.60 (10)	O1—S—O4	109.77 (14)
OW2 ⁱ —Cu—OW2	180.00 (17)	O3—S—O4	109.07 (13)
O1—Cs—O4 ⁱⁱ	140.29 (7)	S—O1—Cs	116.54 (12)
O1—Cs—O3 ⁱⁱⁱ	97.49 (6)	S—O1—Cs ^v	98.82 (11)
O4 ⁱⁱ —Cs—O3 ⁱⁱⁱ	113.63 (6)	Cs—O1—Cs ^v	119.34 (7)
O1—Cs—O2 ⁱⁱⁱ	141.84 (6)	S—O2—Cs ^{vi}	102.10 (12)
O4 ⁱⁱ —Cs—O2 ⁱⁱⁱ	73.76 (6)	S—O2—Cs ^v	96.71 (12)
O3 ⁱⁱⁱ —Cs—O2 ⁱⁱⁱ	44.43 (6)	Cs ^{vi} —O2—Cs ^v	99.31 (8)
O1—Cs—O1 ^{iv}	97.59 (5)	S—O2—Cs ^{vii}	93.62 (13)
O4 ⁱⁱ —Cs—O1 ^{iv}	83.64 (6)	Cs ^{vi} —O2—Cs ^{vii}	118.30 (8)
O3 ⁱⁱⁱ —Cs—O1 ^{iv}	126.51 (6)	Cs ^v —O2—Cs ^{vii}	137.77 (8)
O2 ⁱⁱⁱ —Cs—O1 ^{iv}	104.49 (6)	S—O3—Cs ^{vi}	102.83 (11)
O1—Cs—OW2 ^v	81.11 (6)	S—O4—Cs ^{vii}	111.17 (13)
O4 ⁱⁱ —Cs—OW2 ^v	87.04 (6)	Cu—OW1—Cs	134.36 (11)
O3 ⁱⁱⁱ —Cs—OW2 ^v	69.69 (6)	Cu—OW1—H11	113 (5)
O2 ⁱⁱⁱ —Cs—OW2 ^v	85.68 (7)	Cs—OW1—H11	87 (5)
O1 ^{iv} —Cs—OW2 ^v	163.61 (6)	Cu—OW1—H12	112 (5)
O1—Cs—O2 ^{iv}	94.88 (6)	Cs—OW1—H12	97 (5)
O4 ⁱⁱ —Cs—O2 ^{iv}	111.81 (6)	H11—OW1—H12	112 (6)
O3 ⁱⁱⁱ —Cs—O2 ^{iv}	83.77 (6)	Cu—OW2—Cs ^{iv}	134.73 (10)
O2 ⁱⁱⁱ —Cs—O2 ^{iv}	80.69 (8)	Cu—OW2—H21	122 (4)
O1 ^{iv} —Cs—O2 ^{iv}	43.99 (6)	Cs ^{iv} —OW2—H21	75 (4)
OW2 ^v —Cs—O2 ^{iv}	152.24 (6)	Cu—OW2—H22	114 (5)
O1—Cs—OW1	67.15 (6)	Cs ^{iv} —OW2—H22	101 (5)
O4 ⁱⁱ —Cs—OW1	73.24 (6)	H21—OW2—H22	101 (6)
O3 ⁱⁱⁱ —Cs—OW1	144.36 (5)	Cu—OW3—H31	110 (5)
O2 ⁱⁱⁱ —Cs—OW1	142.85 (6)	Cu—OW3—H32	117 (5)
O1 ^{iv} —Cs—OW1	88.33 (6)	H31—OW3—H32	122 (7)
OW2 ^v —Cs—OW1	76.06 (6)		

Symmetry codes: (i) $-x, -y, -z$; (ii) $x-1/2, -y+1/2, z-1$; (iii) $-x+1/2, y+1/2, -z+1$; (iv) $x-1/2, -y+1/2, z$; (v) $x+1/2, -y+1/2, z$; (vi) $-x+1/2, y-1/2, -z+1$; (vii) $x+1/2, -y+1/2, z+1$.

supplementary materials

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
OW1—H11 \cdots O3	0.77 (7)	1.97 (7)	2.714 (2)	166 (7)
OW1—H12 \cdots O4 ^{viii}	0.75 (7)	1.99 (7)	2.738 (4)	173 (7)
OW2—H21 \cdots O2 ^{ix}	0.87 (6)	1.91 (7)	2.777 (3)	174 (7)
OW2—H22 \cdots O4 ⁱⁱ	0.74 (6)	2.04 (6)	2.779 (4)	171 (6)
OW3—H31 \cdots O3 ^x	0.74 (7)	2.02 (7)	2.725 (3)	155 (7)
OW3—H32 \cdots O1 ^{vi}	0.71 (7)	1.99 (6)	2.693 (3)	171 (6)

Symmetry codes: (viii) $x, y, z-1$; (ix) $x-1, y, z-1$; (ii) $x-1/2, -y+1/2, z-1$; (x) $-x, -y, -z+1$; (vi) $-x+1/2, y-1/2, -z+1$.

Fig. 1

