



Crystal structure of $\text{CdSO}_4(\text{H}_2\text{O})$: a redetermination

Chatphorn Theppitak^a and Kittipong Chainok^{b*}

^aDepartment of Chemistry, Faculty of Science, Naresuan University, Muang, Phitsanulok, 65000, Thailand, and ^bDepartment of Physics, Faculty of Science and Technology, Thammasat University, Khlong Luang, Pathum Thani, 12120, Thailand.
*Correspondence e-mail: kc@tu.ac.th

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The crystal structure of the title compound, cadmium sulfate monohydrate or poly[(μ_2 -aqua)(μ_4 -sulfato)cadmium], was redetermined based on modern CMOS (complementary metal oxide silicon) data. In comparison with the previous study [Bregeault & Herpin (1970). *Bull. Soc. Fr. Mineral. Cristallogr.* **93**, 37–42], all non-H atoms were refined with anisotropic displacement parameters and the hydrogen-bonding pattern unambiguously established due to location of the hydrogen atoms. In addition, a significant improvement in terms of precision and accuracy was achieved. In the crystal, the Cd^{2+} cation is coordinated by four O atoms of four sulfate anions and two O atoms of water molecules, forming a distorted octahedral *trans*-[CdO_6] polyhedron. Each sulfate anion bridges four Cd^{2+} cations and each water molecule bridges two Cd^{2+} cations, leading to the formation of a three-dimensional framework, with $\text{Cd}\cdots\text{Cd}$ separations in the range 4.0757 (2)–6.4462 (3) Å. O–H \cdots O hydrogen-bonding interactions of medium strength between the coordinating water molecules and sulfate anions consolidate the crystal packing.

Keywords: crystal structure; redetermination; cadmium sulfate monohydrate; hydrothermal synthesis; hydrogen bonding.

CCDC reference: 1423357

1. Related literature

For the previous report on the structure of the title compound, see: Bregeault & Herpin (1970).

2. Experimental

2.1. Crystal data

$\text{CdSO}_4(\text{H}_2\text{O})$

$M_r = 226.48$

Monoclinic, $P2_1/c$
 $a = 7.6195$ (3) Å
 $b = 7.4517$ (3) Å
 $c = 8.1457$ (3) Å
 $\beta = 122.244$ (1)°
 $V = 391.17$ (3) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 6.01$ mm⁻¹
 $T = 296$ K
 $0.26 \times 0.22 \times 0.22$ mm

2.2. Data collection

Bruker APEXII D8 QUEST CMOS diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2014)
 $T_{\min} = 0.701$, $T_{\max} = 0.746$

17459 measured reflections
 1004 independent reflections
 958 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.023$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.012$
 $wR(F^2) = 0.026$
 $S = 1.18$
 1004 reflections
 72 parameters

2 restraints
 All H-atom parameters refined
 $\Delta\rho_{\text{max}} = 0.29$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O5}-\text{H5A}\cdots\text{O3}^{\text{i}}$	0.82 (2)	1.88 (2)	2.6958 (17)	170 (3)
$\text{O5}-\text{H5B}\cdots\text{O2}^{\text{ii}}$	0.86 (2)	1.90 (2)	2.7530 (17)	173 (2)

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

Table 2

Comparison of bond lengths (Å) in the current and the previous (Bregeault & Herpin, 1970) refinement of cadmium sulfate monohydrate.

For the previous refinement: $a = 7.607$, $b = 7.541$, $c = 8.186$ Å, $\beta = 121.86^\circ$ and reliability index $R = 0.12$.

Bond	Current refinement	Previous refinement
$\text{Cd1}-\text{O1}^{\text{i}}$	2.2417 (12)	2.21 (5)
$\text{Cd1}-\text{O2}^{\text{ii}}$	2.2530 (13)	2.27 (3)
$\text{Cd1}-\text{O3}$	2.2421 (12)	2.36 (5)
$\text{Cd1}-\text{O4}^{\text{iii}}$	2.3112 (12)	2.33 (3)
$\text{Cd1}-\text{O5}^{\text{i}}$	2.3210 (12)	2.24 (3)
$\text{Cd1}-\text{O5}$	2.4024 (12)	2.33 (3)
$\text{S1}-\text{O1}$	1.4703 (12)	1.50 (4)
$\text{S1}-\text{O2}$	1.4845 (12)	1.62 (6)
$\text{S1}-\text{O3}$	1.4831 (12)	1.45 (3)
$\text{S1}-\text{O4}$	1.4584 (12)	1.42 (4)

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINTE* (Bruker, 2014); data reduction: *SAINTE*; method used to solve structure: coordinates taken from previous refinement; program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2* and *pubCIF* (Westrip, 2010).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5211).

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supporting information

Acta Cryst. (2015). E71, i8–i9 [doi:10.1107/S2056989015016904]

Crystal structure of $\text{CdSO}_4(\text{H}_2\text{O})$: a redetermination

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S1. Synthesis and crystallization

The title compound was obtained serendipitously. $\text{CdSO}_4 \cdot 8/3\text{H}_2\text{O}$ (0.256 g, 1 mmol) and 3,6-di-2-pyridyl-1,2,4,5-tetrazine (0.236 g, 1 mmol) dissolved in water (5 ml) were added to a 23 ml Teflon-lined autoclave and heated at 356 K for 5 days. The product was collected by filtration, washed with water and air-dried. Colourless block-shaped crystals of the title compound suitable for X-ray analysis were isolated.

S2. Refinement

The same cell setting and atom numbering scheme as in the previous refinement (Bregeault & Herpin, 1970) were used. Starting coordinates for the atoms were also taken from the previous model. Hydrogen atoms of the water molecules were located from difference Fourier maps and were refined with an O—H distance restraint of 0.85 (2) Å.

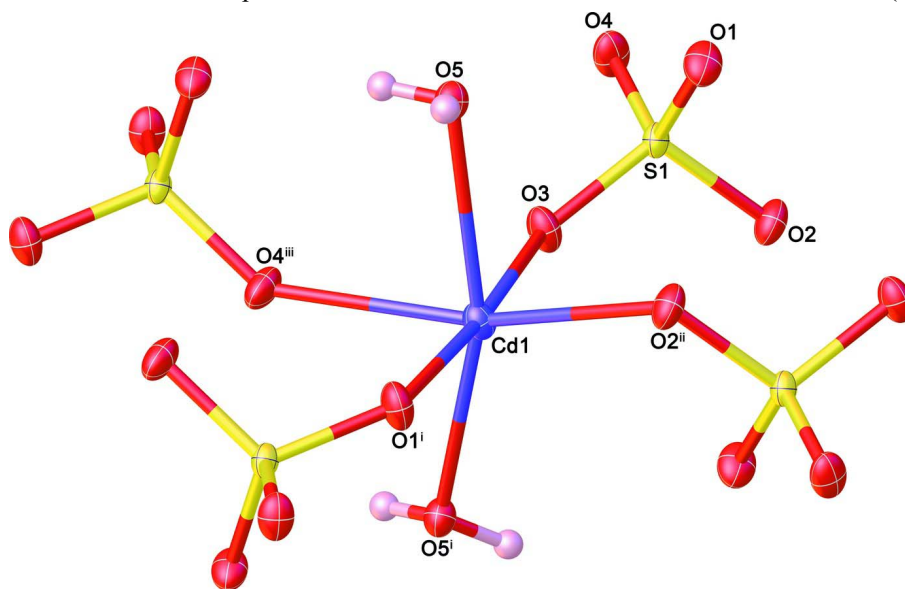
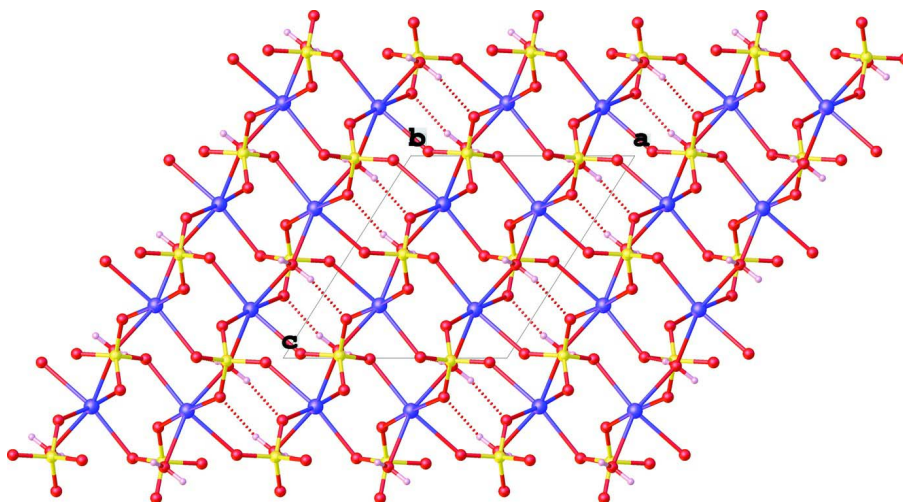


Figure 1

The coordination sphere around the Cd^{2+} cation with displacement ellipsoids drawn at the 50% probability level. [Symmetry codes: (i) $x, 1/2 - y, 1/2 + z$; (ii) $-x, 1 - y, -z$; (iii) $1 - x, -1/2 + y, 1/2 - z$].

**Figure 2**

The three-dimensional framework structure of the title compound in a view along the *b* axis. Dashed lines indicate intermolecular O—H...O hydrogen-bonding interactions.

Poly[(μ_2 -aqua)(μ_4 -sulfato)cadmium]

Crystal data

CdSO₄(H₂O)

$M_r = 226.48$

Monoclinic, $P2_1/c$

$a = 7.6195$ (3) Å

$b = 7.4517$ (3) Å

$c = 8.1457$ (3) Å

$\beta = 122.244$ (1)°

$V = 391.17$ (3) Å³

$Z = 4$

$F(000) = 424$

$D_x = 3.846$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9894 reflections

$\theta = 3.2$ – 30.5 °

$\mu = 6.01$ mm⁻¹

$T = 296$ K

Block, colourless

$0.26 \times 0.22 \times 0.22$ mm

Data collection

Bruker APEXII D8 QUEST CMOS
diffractometer

Radiation source: microfocus sealed x-ray tube,
Incoatec μ us

GraphiteDouble Bounce Multilayer Mirror
monochromator

Detector resolution: 10.5 pixels mm⁻¹

ω and φ scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2014)

$T_{\min} = 0.701$, $T_{\max} = 0.746$

17459 measured reflections

1004 independent reflections

958 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 28.7$ °, $\theta_{\min} = 3.2$ °

$h = -10 \rightarrow 10$

$k = -10 \rightarrow 10$

$l = -10 \rightarrow 10$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.012$

$wR(F^2) = 0.026$

$S = 1.18$

1004 reflections

72 parameters

2 restraints

Primary atom site location: structure-invariant
direct methods

Hydrogen site location: difference Fourier map

All H-atom parameters refined

$w = 1/[\sigma^2(F_o^2) + (0.0107P)^2 + 0.2955P]$

where $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.28 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. SADABS was used for absorption correction. wR2(int) was 0.0449 before and 0.0357 after correction. The Ratio of minimum to maximum transmission is 0.9396. The $\lambda/2$ correction factor is 0.00150.

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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Cd1	0.21887 (2)	0.26013 (2)	0.26053 (2)	0.01330 (5)
S1	0.25524 (6)	0.61729 (5)	0.01217 (5)	0.01018 (8)
O1	0.13754 (18)	0.50022 (16)	-0.15811 (17)	0.0183 (2)
O2	0.1103 (2)	0.75977 (15)	-0.00480 (19)	0.0169 (3)
O3	0.32756 (18)	0.51255 (16)	0.19211 (17)	0.0168 (2)
O4	0.43393 (18)	0.69973 (18)	0.02148 (18)	0.0184 (2)
O5	0.27603 (19)	0.09752 (16)	0.03844 (17)	0.0140 (2)
H5A	0.400 (3)	0.074 (4)	0.110 (3)	0.038 (7)*
H5B	0.216 (3)	-0.005 (2)	0.016 (3)	0.023 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cd1	0.01176 (7)	0.01373 (7)	0.01200 (7)	-0.00165 (4)	0.00472 (5)	0.00087 (4)
S1	0.00809 (16)	0.00898 (17)	0.01063 (17)	0.00001 (13)	0.00310 (14)	0.00041 (13)
O1	0.0156 (6)	0.0170 (6)	0.0158 (6)	-0.0004 (5)	0.0041 (5)	-0.0054 (5)
O2	0.0122 (6)	0.0134 (6)	0.0206 (6)	0.0027 (4)	0.0058 (5)	-0.0018 (4)
O3	0.0155 (6)	0.0153 (6)	0.0147 (5)	-0.0019 (5)	0.0048 (5)	0.0046 (4)
O4	0.0101 (5)	0.0224 (6)	0.0198 (6)	-0.0022 (5)	0.0061 (5)	0.0047 (5)
O5	0.0120 (5)	0.0133 (5)	0.0145 (5)	-0.0008 (4)	0.0055 (5)	-0.0001 (4)

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

Cd1—O1 ⁱ	2.2417 (12)	S1—O3	1.4831 (12)
Cd1—O2 ⁱⁱ	2.2530 (13)	S1—O4	1.4584 (12)
Cd1—O3	2.2421 (12)	O1—Cd1 ^{iv}	2.2417 (12)
Cd1—O4 ⁱⁱⁱ	2.3112 (12)	O2—Cd1 ⁱⁱ	2.2530 (13)
Cd1—O5 ⁱ	2.3210 (12)	O4—Cd1 ^v	2.3112 (12)
Cd1—O5	2.4024 (12)	O5—Cd1 ^{iv}	2.3211 (12)
S1—O1	1.4703 (12)	O5—H5A	0.822 (17)
S1—O2	1.4845 (12)	O5—H5B	0.859 (16)
O1 ⁱ —Cd1—O2 ⁱⁱ	82.50 (4)	O1—S1—O3	109.76 (7)
O1 ⁱ —Cd1—O3	175.24 (4)	O3—S1—O2	109.52 (8)
O1 ⁱ —Cd1—O4 ⁱⁱⁱ	89.31 (5)	O4—S1—O1	112.43 (8)

O1 ⁱ —Cd1—O5 ⁱ	92.57 (4)	O4—S1—O2	109.35 (7)
O1 ⁱ —Cd1—O5	88.62 (4)	O4—S1—O3	109.03 (7)
O2 ⁱⁱ —Cd1—O4 ⁱⁱⁱ	161.94 (4)	S1—O1—Cd1 ^{iv}	131.81 (7)
O2 ⁱⁱ —Cd1—O5	80.22 (4)	S1—O2—Cd1 ⁱⁱ	116.42 (7)
O2 ⁱⁱ —Cd1—O5 ⁱ	117.97 (4)	S1—O3—Cd1	134.06 (7)
O3—Cd1—O2 ⁱⁱ	101.65 (4)	S1—O4—Cd1 ^v	140.91 (8)
O3—Cd1—O4 ⁱⁱⁱ	86.05 (4)	Cd1 ^{iv} —O5—Cd1	119.27 (5)
O3—Cd1—O5	89.80 (4)	Cd1 ^{iv} —O5—H5A	109.7 (18)
O3—Cd1—O5 ⁱ	87.54 (4)	Cd1—O5—H5A	99.9 (18)
O4 ⁱⁱⁱ —Cd1—O5 ⁱ	78.31 (4)	Cd1 ^{iv} —O5—H5B	113.3 (14)
O4 ⁱⁱⁱ —Cd1—O5	83.53 (4)	Cd1—O5—H5B	108.6 (14)
O5 ⁱ —Cd1—O5	161.78 (6)	H5A—O5—H5B	104 (2)
O1—S1—O2	106.70 (7)		
O1—S1—O2—Cd1 ⁱⁱ	2.46 (10)	O3—S1—O1—Cd1 ^{iv}	-54.81 (11)
O1—S1—O3—Cd1	-23.82 (12)	O3—S1—O2—Cd1 ⁱⁱ	-116.29 (8)
O1—S1—O4—Cd1 ^v	-135.57 (12)	O3—S1—O4—Cd1 ^v	-13.61 (15)
O2—S1—O1—Cd1 ^{iv}	-173.39 (9)	O4—S1—O1—Cd1 ^{iv}	66.74 (12)
O2—S1—O3—Cd1	93.01 (11)	O4—S1—O2—Cd1 ⁱⁱ	124.30 (8)
O2—S1—O4—Cd1 ^v	106.10 (13)	O4—S1—O3—Cd1	-147.38 (10)

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

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5A \cdots O3 ⁱⁱⁱ	0.82 (2)	1.88 (2)	2.6958 (17)	170 (3)
O5—H5B \cdots O2 ^{vi}	0.86 (2)	1.90 (2)	2.7530 (17)	173 (2)

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