

Redetermination of the hexagonal struvite analogue Cs[Mg(OH₂)₆](PO₄)

Matthias Weil

Institute for Chemical Technologies and Analytics, Division of Structural Chemistry, Vienna University of Technology, Getreidemarkt 9/164-SC, A-1060 Vienna, Austria
Correspondence e-mail: mweil@mail.zserv.tuwien.ac.at

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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{Mg}-\text{O}) = 0.004$ Å; R factor = 0.016; wR factor = 0.034; data-to-parameter ratio = 9.4.

The structure of the hexagonal modification of caesium hexaaquamagnesium phosphate has been redetermined from single-crystal X-ray data. The previous refinement from photographic data [Ferrari, Calvaca & Nardelli (1955). *Gazz. Chim. Ital.* **85**, 1232–1238] was basically confirmed, but with all H atoms located and with all non H-atoms refined with anisotropic displacement parameters. The structure can be derived from the NiAs structure type: the PO₄ tetrahedra ($3m$. symmetry) are on the Ni positions and the complex [Mg(OH₂)₆] octahedra ($3m$. symmetry) are on the As positions. The building units are connected to each other by hydrogen bonds. The Cs⁺ cations ($3m$. symmetry) are located in the voids of this arrangement and exhibit a distorted cuboctahedral 12-coordination by the O atoms of the water molecules.

Related literature

The crystal structure of struvite, NH₄[Mg(OH₂)₆](PO₄), was reported by Whitaker & Jeffery (1970*a,b*). Structure determinations of the hexagonal and cubic forms of Cs[Mg(OH₂)₆](PO₄) were performed by Ferrari *et al.* (1955) and Massa *et al.* (2003), respectively. Crystal growth of struvite-like compounds using the gel diffusion technique was reported by Banks *et al.* (1975). For general background, see: Flack (1983).

Experimental

Crystal data

Cs[Mg(H₂O)₆](PO₄)
 $M_r = 360.29$
Hexagonal, $P6_3mc$
 $a = 6.8827$ (8) Å
 $c = 11.9188$ (16) Å
 $V = 488.97$ (10) Å³

$Z = 2$
Mo $K\alpha$ radiation
 $\mu = 4.04$ mm⁻¹
 $T = 293$ (2) K
 $0.46 \times 0.38 \times 0.38$ mm

Data collection

Stoe IPDS diffractometer
Absorption correction: numerical
(*HABITUS*; Herrendorf, 1997)
 $T_{\min} = 0.213$, $T_{\max} = 0.327$
5412 measured reflections
368 independent reflections
367 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.016$
 $wR(F^2) = 0.034$
 $S = 1.29$
368 reflections
39 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.40$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³
Absolute structure: Flack (1983), with 164 Friedel pairs
Flack parameter: -0.01 (3)

Table 1

Selected bond lengths (Å).

Cs—O3 ⁱ	3.469 (5)	Mg—O4	2.082 (3)
Cs—O4 ⁱ	3.470 (4)	P—O1	1.536 (3)
Cs—O4 ⁱⁱ	3.742 (4)	P—O2	1.539 (6)
Mg—O3	2.054 (4)		

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H1 ⁱ ···O2	0.93 (4)	1.71 (4)	2.643 (5)	180 (6)
O3—H2 ⁱ ···O1 ⁱⁱⁱ	0.88 (4)	1.76 (5)	2.630 (5)	168 (7)
O4—H3 ⁱ ···O1 ^{iv}	0.83 (4)	1.85 (4)	2.672 (3)	177 (8)

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

Data collection: *EXPOSE* in *IPDS Software* (Stoe & Cie, 1998); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ATOMS* (Dowty, 2004); software used to prepare material for publication: *SHELXL97*.

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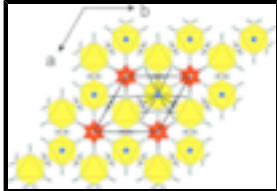
Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BR2080).

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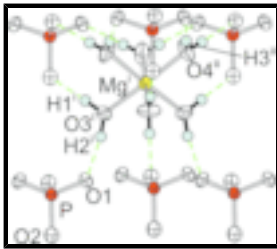
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