Er2—O3	2.35 (2)	Er5—O59	2.44 (2)
Er2—O45	2.41 (2)	Er5—O8	2.46 (2)
Er2—O4	2.43 (2)	Er6—O2	2.21 (3)
Er2—O44	2.43 (3)	Er6—O53	2.25 (2)
Er2—O40	2.45 (2)	Er6—O6	2.34 (2)
Er3—O16	2.24 (3)	Er6—O14	2.37 (2)
Er3—O1	2.27 (2)	Er6—O27	2.40 (3)
Er3—O4	2.33 (2)	Er6—O28	2.42 (3)
Er3—O11	2.37 (2)	Er6—O7	2.45 (2)
Er3—O64	2.38 (3)	Er6—O21	2.47 (1)

During the refinement, U_{iso} for Na9 converged to a value which was too large (0.27 Å^2), so the site occupancy was reduced to 0.5. The resulting value of U_{iso}, 0.12 (1) Å^2, is reasonable for an Na$^+$ cation. The site occupancies of the O138 and O149 atoms, both of which are from waters of crystallization, were fixed at 0.5 because of the short distance (1.6 Å) between them. Although the distance between Nb22 and O75 [1.68 (4) Å] is rather short for a terminal Nb—O bond, the same [1.68 (4) Å] and shorter [1.62 (2) Å] distances have been reported for Na_7H_{19}-[$\{Eu_3O(OH)_3(H_2O)_3\}_2Al_2(Nb_6O_{19})_5$].47H$_2$O (Ozeki, Yamase, Naruke & Sasaki, 1994) and AgLaNb$_2$O$_7$ (Sato, Watanabe & Uematsu, 1993), respectively. The maximum Fourier peak (3.56 e Å^{-3}) is 2.20, 2.20 and 2.21 Å from O110, O146 and O151, respectively, all of which are from waters of crystallization. These distances are too short for Na$\cdots$O or O$\cdots$O hydrogen-bond distances. Moreover, since U_{iso} for O110 and O146 converged to moderate values [0.12 (1) and 0.20 (2) Å^2, respectively] with site occupancies of 1.0, disorder among O110, O146 and the peak site is not likely to occur. Therefore, the maximum Fourier peak was not identified as an atom. The minimum Fourier peak (-3.13 e Å^{-3}) is a very short distance from Nb28 (0.39 Å), suggesting that it is a ghost of the Nb atom. The high R value of 0.076 is probably due to the rather poor quality of the crystal, which is strongly efflorescent.

Program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1992). Program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1989).

Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: OA1001). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Besecker, C. J., Day, V. W., Klemperer, W. G. & Thompson, M. R. (1984). *J. Am. Chem. Soc.* **106**, 4125–4136.

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., García-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Brown, I. D. & Altermatt, D. (1985). *Acta Cryst.* B41, 244–247.

Flynn, C. M. Jr & Stucky, G. D. (1969). *Inorg. Chem.* 8, 335–344.

Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1989). *TEXSAN. Single Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

Naruke, H., Ozeki, T. & Yamase, T. (1991). *Acta Cryst.* C47, 489–492.

Ozeki, T., Yamase, T., Naruke, H. & Sasaki, Y. (1994). *Inorg. Chem.* **33**, 409–410.

Sato, M., Watanabe, J. & Uematsu, K. (1993). *J. Solid State Chem.* **107**, 460–470.

Walker, N. & Stuart, D. (1983). *Acta Cryst.* A39, 158–166.

Yamase, T., Naruke, H. & Sasaki, Y. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1687–1696.

The First Beryllium Periodate: Be(H₄IO₆)₂.4H₂O

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Abstract

The title compound, beryllium tetrahydrogenhexaoxoiodate(VII) tetrahydrate, is built up from slightly deformed Be(OH₂)₄ tetrahedra (*a,b*) and IO₂(OH)₄ octahedra (*A,B*) with *cis* configuration of the oxo ligands. They are arranged along [001] forming layers with stacking *aBAbABaBABAbAB*. The [H₄IO₆]⁻ and Be(OH₂)₄²⁺ ions are held together by a network of eight independent hydrogen bonds of different strength. Two of the H atoms of [H₄IO₆]⁻ are disordered over O—H↔H—O split positions.

Comment

The title compound, which is very easily decomposed by exposure to X-rays, was first described as Be(IO₄)₂.8H₂O (Biber, Neiman & Bragina, 1941; Maneva, Georgiev & Pavlova, 1988). This was indicated by IR experiments, but resulted from the strong reaction with KBr used as the matrix. Raman spectroscopic and more careful IR experiments, however, revealed that the compound under study was an orthoperiodate, Be(H₄IO₆)₂.4H₂O, rather than a metaperiodate (Maneva, Georgiev, Lange & Lutz, 1991). This has been confirmed by the single-crystal X-ray studies reported in this work.

The crystal structure, which represents a hitherto unknown structure, is built up of layers of Be(OH₂)₄ tetrahedra (*a,b*) and double layers of H₄IO₆ octahedra

(*A,B*) parallel to (001) (see Fig. 1). They are held together by different strong hydrogen bonds formed by the two crystallographically different types of water molecule and the four different OH groups of the periodate ion with O···O distances as short as 2.513 (9) Å. The hydrogen bonds drawn in Fig. 2, however, are

Fig. 1. Unit cell and packing of Be(H$_4$IO$_6$)$_2$.4H$_2$O. H atoms are omitted for clarity.

Fig. 2. An H$_4$IO$_6$ octahedron. Dashed lines represent hydrogen bonds donated from the IOH groups, dotted lines represent hydrogen bonds accepted by the O atoms and empty circles represent H atoms on split positions. Displacement ellipsoids are drawn at the 82% probability level for all non-H atoms.

in part only tentative because the H-atom positions are not fully known. The IO and IOH groups can be distinguished by the different I—O bond lengths 1.799 (4) Å (O3), 1.844 (4) and 1.850 (4) Å (O4 and O5), and 1.899 (4)–1.915 (5) Å (O6–O8) (see Table 2). In contrast to LiH$_4$IO$_6$.H$_2$O, the only other tetra-hydrogenorthoperiodate with a known crystal structure (Kraft & Jansen, 1994), the oxo ligands display a *cis* configuration. Two of the H atoms are disordered over O—H↔H—O split positions (O4, O5) with symmetric double minimum potentials, as also established for BaH$_3$IO$_6$ (Sasaki, Yarita & Sato, 1995).
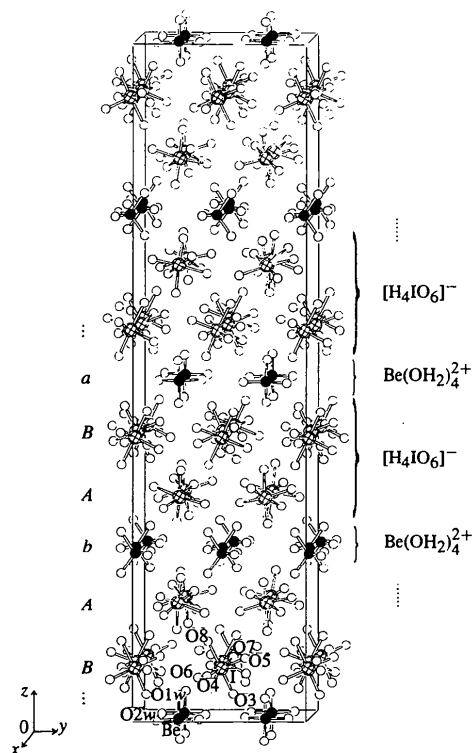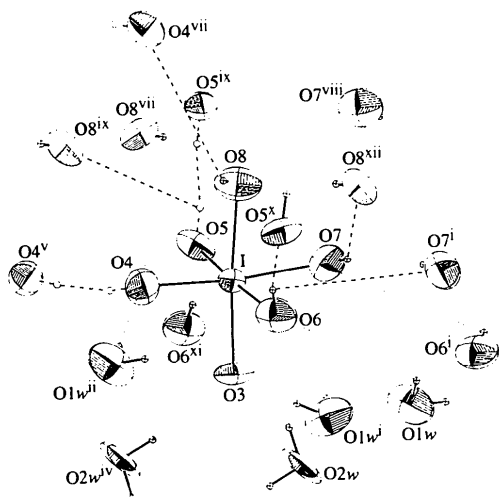
Experimental

Crystal data

Be(H$_4$IO$_6$)$_2$.4H$_2$O	Mo $K\alpha$ radiation
$M_r = 534.94$	$\lambda = 0.7107$ Å
Tetragonal	Cell parameters from 61
$I4_1/acd$	reflections
$a = 10.8997$ (7) Å	$\theta = 4.11$–$39.90°$
$c = 42.817$ (4) Å	$\mu = 5.031$ mm^{-1}
$V = 5086.8$ (7) Å^3	$T = 293$ (2) K
$Z = 16$	Prism
$D_x = 2.794$ Mg m^{-3}	$0.3 \times 0.2 \times 0.1$ mm
$D_m = 2.75$ Mg m^{-3}	Colourless
D_m measured by pycnometry	

Data collection

Enraf–Nonius CAD-4	778 independent reflections
diffractometer	708 observed reflections
ω–2θ scans [width (0.9	$[I > 2\sigma(I)]$
+ 0.35$\tan\theta$)°]	$\theta_{max} = 21.93°$
Absorption correction:	$h = 0 \rightarrow 11$
ψ scans (Gabe, Le Page,	$k = 0 \rightarrow 8$
Charland, Lee & White,	$l = 0 \rightarrow 44$
1989)	3 standard reflections
$T_{min} = 0.38$, $T_{max} =$	monitored every 160
0.60	reflections
778 measured reflections	intensity decay: none

Refinement

Refinement on F^2	$\Delta\rho_{max} = 0.649$ e Å^{-3}
$R[F^2 > 2\sigma(F^2)] = 0.0286$	$\Delta\rho_{min} = -0.892$ e Å^{-3}
$wR(F^2) = 0.0870$	Extinction correction:
$S = 1.344$	SHELXL93 (Sheldrick,
778 reflections	1993)
115 parameters	Extinction coefficient:
Only H-atom coordinates	0.00007 (3)
refined	Atomic scattering factors
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2$	from *International Tables*
$+ 35.7863P]$	*for Crystallography* (1992,
where $P = (F_o^2 + 2F_c^2)/3$	Vol. C, Tables 4.2.6.8 and
$(\Delta/\sigma)_{max} = -0.001$	6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Be	3/4	0.2344 (13)	0	0.026 (4)
I	0.48630 (5)	−0.00410 (3)	0.078806 (11)	0.0144 (3)
O1W	0.7523 (5)	0.3161 (6)	0.03115 (12)	0.0320 (14)
O2W	0.6253 (5)	0.1548 (5)	0.00081 (11)	0.0306 (14)
O3	0.4194 (4)	0.0176 (4)	0.04080 (9)	0.0208 (11)
O4	0.5605 (4)	−0.1519 (4)	0.06973 (10)	0.0221 (11)
O5	0.3457 (4)	−0.0746 (4)	0.09501 (9)	0.0206 (11)
O6	0.6263 (4)	0.0805 (4)	0.06349 (10)	0.0238 (12)
O7	0.4277 (5)	0.1518 (4)	0.09268 (13)	0.0276 (13)
O8	0.5634 (5)	−0.0132 (5)	0.11893 (13)	0.0277 (13)

Table 2. *Selected geometric parameters (Å, °)*

IO₆ octahedron

I—O3	1.799 (4)	I—O6	1.899 (4)
I—O4	1.844 (4)	I—O7	1.910 (5)
I—O5	1.850 (4)	I—O8	1.915 (5)
O3—I—O4	95.9 (2)	O5—I—O7	88.6 (2)
O3—I—O5	93.3 (2)	O6—I—O7	86.8 (2)
O4—I—O5	94.6 (2)	O3—I—O8	175.0 (2)
O3—I—O6	87.1 (2)	O4—I—O8	87.2 (2)
O4—I—O6	89.9 (2)	O5—I—O8	90.3 (2)
O5—I—O6	175.4 (2)	O6—I—O8	89.0 (2)
O3—I—O7	91.6 (2)	O7—I—O8	85.1 (2)
O4—I—O7	171.7 (2)		

BeO₄ tetrahedron

Be—O1W	1.604 (9)	Be—O2W^{iii}	1.613 (10)
Be—O1W^{iii}	1.604 (9)	Be—O2W^{iii}	1.613 (10)
O1W—Be—O1W^{iii}	112.5 (10)	O1W—Be—O2W	107.1 (3)
O1W—Be—O2W^{iii}	107.6 (2)	O1W^{iii}—Be—O2W	107.7 (2)
O1W^{iii}—Be—O2W^{iii}	107.1 (3)	O2W^{iii}—Be—O2W	115.0 (9)

Possible hydrogen bonds [d(O⋯O) < 3.3 Å]

O1W⋯O3^i	2.638 (7)	O5⋯O8^{ix}	3.157 (7)
O1W⋯O6	3.226 (8)	O6⋯O5^x	2.747 (6)
O1W⋯O4^{vi}	2.648 (6)	O6⋯O7^i	3.229 (7)
O2W⋯O3^{iv}	2.636 (6)	O7⋯O7^i	2.659 (9)
O2W⋯O6	2.804 (6)	O7⋯O8^{xii}	2.679 (8)
O2W⋯O3	3.194 (7)	O8⋯O8^{vii}	2.722 (9)
O4⋯O4^v	2.513 (9)	O8⋯O4^{vii}	3.276 (7)
O5⋯O5^{ix}	2.588 (8)		

Symmetry codes: (i) $1-x, \frac{1}{2}-y, z$; (ii) $\frac{3}{2}-x, y-\frac{1}{2}, z$; (iii) $\frac{3}{2}-x, y, -z$; (iv) $1-x, -y, -z$; (v) $1-x, -\frac{1}{2}-y, z$; (vi) $\frac{3}{2}-x, \frac{1}{2}+y, z$; (vii) $\frac{3}{4}+y, x-\frac{3}{4}, \frac{1}{4}-z$; (viii) $\frac{3}{4}-y, x-\frac{1}{4}, \frac{1}{4}-z$; (ix) $\frac{1}{4}-y, \frac{1}{4}-x, \frac{1}{4}-z$; (x) $x+\frac{1}{2}, -y, z$; (xi) $x-\frac{1}{2}, -y, z$; (xii) $\frac{1}{4}+y, \frac{3}{4}-x, \frac{1}{4}-z$.

Polycrystalline samples of the compound were characterized by X-ray analysis using the Guinier technique with Cu $K\alpha$ radiation and quartz as internal standard. The unit-cell dimensions were calculated by least-squares methods (*LSUCR*: Least Squares Unit Cell Refinement, Programmbibliothek für die Chemie, HRZ Köln, Germany). The crystal studied was mounted in a sealed glass capillary. The space group was inferred from precession photographs of $hk0$, $hk1$, $hk2$, $0kl$, $1kl$ and $2kl$ layers taken using a Buerger precession camera (Firma Huber, Rimsting) and powder data. The data were corrected for Lorentz and polarization effects. Empirical absorption corrections were applied *via* ψ scans of nine reflections in the range $3.86 < \theta < 24.72°$. The standard reflections were $10,0,0$, $0,10,0$, and $0,0,40$. The positions of the H atoms were deduced from difference Fourier maps. H atoms were refined with a common isotropic displacement parameter (U_{iso} = 0.05 Å²).

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *LSUCR*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *NRCVAX*. Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) and *SCHAKAL92* (Keller, 1992).

References

Biber, V. A., Neiman, I. A. & Bragina, A. A. (1941). *Zh. Obshch. Khim.* **11**, 861–868.

Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.

Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Keller, E. (1992). *SCHAKAL92. Program for the Graphic Representation of Molecular and Crystallographic Models*. University of Freiburg, Germany.

Kraft, Th. & Jansen, M. (1994). *Z. Anorg. Allg. Chem.* **620**, 805–808.

Maneva, M., Georgiev, M., Lange, N. & Lutz, H. D. (1991). *Z. Naturforsch. Teil B*, **46**, 795–799.

Maneva, M., Georgiev, M. & Pavlova, A. (1988). *J. Therm. Anal.* **33**, 589–595.

Sasaki, M., Yarita, T. & Sato, S. (1995). *Acta Cryst.* C**51**, 1968–1970.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

K₂Be₂Si₃O₉, a Potassium Beryllium Silicate Framework Built from Edge-Sharing Be—O Tetrahedra and Three-Ring Silicate Tetrahedra

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Abstract

Dipotassium diberyllium trisilicon nonaoxide, K₂Be₂Si₃O₉, was synthesized at high temperature and high pressure and has a three-dimensional framework structure containing six-ring channels along the b axis. The framework is built from cyclic trimers of silicate tetrahedra and edge-sharing Be—O tetrahedra.

Comment

Zeolite structures based on two types of tetrahedral atoms which have a combined formal charge of +7 or