consistent with Laue group $6/mmm$. On the basis of systematic absences (hhl, l = odd), three space groups were allowed, of which two were noncentrosymmetric ($P6_3mc$ and $P\bar{6}2c$) and one was centrosymmetric ($P6_3/mmc$). Since the structure of lithium perchlorate trihydrate had been assigned in $P6_3mc$ (Chomnilpan *et al.*, 1977, and earlier references cited therein), $P6_3mc$ (No. 186) was given initial preference; since refinement proceeded well it was adopted. Subsequent attempts to refine a structure in the potential alternative space groups were not successful. The *TEXSAN* (Molecular Structure Corporation, 1989) structure analysis package was used to generate a Patterson map from which the initial Br position was assigned. Difference Fourier methods were then used to locate the remaining atoms. The non-H atoms, all of which were assigned at special positions, were refined anisotropically, the H atom isotropically, using full-matrix least squares. An extinction coefficient (Zachariasen, 1963, 1968) was included in the least-squares refinement; the maximum effect of extinction was 27.4% of F_o for $00\bar{2}$. Following the extinction correction, 13 reflections had $\Delta F/\sigma_F$ greater than 5.0. The enantiomorphic structure was rejected since its agreement was inferior (R = 0.061 and wR = 0.083).

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Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71005 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1039]

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Structure of Tetraaquacalcium Perbromate

ANTHONY C. BLACKBURN AND ROGER E. GERKIN*

Department of Chemistry, The Ohio State University, Columbus, OH 43210, USA

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Abstract

The structure of the title compound consists of columns of Ca ions surrounded by four columns of perbromate ions and four columns of water molecules alternating in pairs. The CaO_8 complex was found to be dimensionally similar to corresponding complexes in other simple tetrahydrated calcium salts. The eight coordinating O atoms form a polyhedron which departs only slightly from a (distorted) square antiprism. Each of the crystallographically inequivalent perbromate ions was found to conform to rigid-body behavior while the CaO_8 complex did not. Although the perbromate ions depart moderately from regular tetrahedral geometry, the mean Br—O bond length agrees well with previously determined values. Hydrogen bonds in this structure involve water-oxygen acceptors as well as perbromate-oxygen acceptors.

Comment

This structural study of $[Ca(H_2O)_4](BrO_4)_2$ is one of a series of such studies of simple salts of perbromic acid. Refinement of the H atoms to permit a detailed description of the hydrogen bonding was a matter of particular interest. This is the first report on the structure of tetraaquacalcium perbromate.

Final atomic coordinates and isotropic and equivalent isotropic displacement parameters and their e.s.d.'s are given in Table 1. A unit cell is shown in Fig. 1.

In this structure, Ca is coordinated by four water O atoms at a mean distance of 2.37 (3) Å and by four anion O atoms at the slightly greater mean distance of 2.57 (4) Å, the overall mean distance being 2.47 (10) Å (Table 2). The same pattern of distances is exhibited by Ca in other simple hydrated salts, for example, in $[Ca(H_2O)_4](NO_3)_2$, for which the corresponding distances are 2.44 (1), 2.52 (3) and 2.48 (5) Å (Leclaire & Monier, 1977), and in $[Ca(H_2O)_4]Cd(NO_2)_4$, for which the corresponding distances are 2.41 (1), 2.58 (1) and 2.49 (9) Å (Aoyama, Ohba & Saito, 1988). In the title salt, the eight coordinating O atoms form a polyhedron which departs only slightly from a (distorted) square

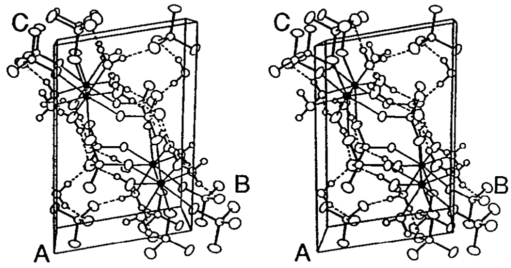

Fig. 1. A stereoview of a unit cell of tetraaquacalcium perbromate drawn using *ORTEP*II (Johnson, 1976). Displacement ellipsoids are drawn for 50% probability for all atoms except H, for which they have been set artificially small. The eight most significant hydrogen bonds are shown as dashed lines, the calcium ions as filled symbols.

antiprism: the dihedral angles between faces defined by vertices O(3), O(4), O(11$^{\text{iii}}$) and O(3), O(4), O(23) and between the faces defined by vertices O(1), O(2), O(14$^{\text{iv}}$) and O(1), O(2), O(22$^{\text{ii}}$) are approximately 11 and 15°, respectively; if these angles were 0°, the figure would be a (distorted) square antiprism.

There are no symmetry constraints on either of the two perbromate ions, which depart somewhat from regular tetrahedral geometry with respect to both distances and angles. For anion (I), the mean observed bond distance is 1.605 (3) Å (1.622 Å rigid-body corrected) and the mean angle is 109.5 (1.2)°, while for anion (II), the corresponding values are 1.608 (7) Å (1.624 Å rigid-body corrected) and 109.5 (0.9)°. Thus, while one ion shows a greater dispersion of distances, the other shows a greater dispersion of angles. The overall mean Br—O distance is 1.607 (7) Å (1.623 Å, rigid-body corrected), which agrees well with mean values previously reported (Blackburn, Gallucci, Gerkin & Reppart, 1992).

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic thermal parameters* (Å^2)

H refined isotropically; $B_{\text{eq}} = \frac{8\pi^2}{3}\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for Br, Ca, O.

	x	y	z	$B_{\text{iso}}/B_{\text{eq}}$
Br(1)	0.25767 (5)	0.17081 (4)	0.05633 (2)	1.64 (1)
Br(2)	0.16532 (6)	0.32122 (4)	0.41698 (3)	1.83 (1)
Ca	0.01567 (11)	0.23131 (8)	0.74262 (5)	1.78 (3)
O(1)	−0.2335 (5)	0.3804 (4)	0.8603 (2)	2.4 (1)
O(2)	−0.2764 (6)	0.1298 (4)	0.6332 (3)	3.3 (1)
O(3)	0.2534 (5)	−0.0134 (3)	0.7347 (3)	2.3 (1)
O(4)	0.2999 (6)	0.4408 (4)	0.7540 (3)	3.3 (2)
O(11)	0.2335 (5)	0.1439 (3)	−0.0729 (2)	3.1 (1)
O(12)	0.0522 (4)	0.2950 (3)	0.0889 (2)	3.1 (1)
O(13)	0.5158 (4)	0.2486 (3)	0.0730 (2)	2.8 (1)
O(14)	0.2418 (5)	−0.0103 (3)	0.1355 (2)	2.9 (1)
O(21)	−0.1007 (4)	0.2578 (3)	0.3956 (2)	2.9 (1)
O(22)	0.1692 (5)	0.5254 (3)	0.3931 (2)	2.7 (1)
O(23)	0.2364 (4)	0.2553 (3)	0.5459 (2)	2.9 (1)
O(24)	0.3552 (5)	0.2522 (3)	0.3352 (2)	3.7 (1)
H(1)	−0.272 (8)	0.346 (5)	0.912 (4)	2.8 (11)
H(2)	−0.180 (7)	0.470 (5)	0.878 (3)	2.9 (9)
H(3)	−0.351 (12)	0.071 (8)	0.651 (6)	8.8 (22)
H(4)	−0.312 (11)	0.184 (7)	0.554 (5)	9.5 (18)
H(5)	0.217 (8)	−0.075 (5)	0.700 (4)	3.2 (11)
H(6)	0.293 (8)	−0.056 (5)	0.787 (4)	3.8 (12)
H(7)	0.286 (8)	0.500 (5)	0.722 (4)	2.0 (11)
H(8)	0.448 (9)	0.419 (5)	0.793 (4)	5.1 (11)

Table 2. *Selected interatomic distances* (Å) *and angles* (°) *in tetraaquacalcium perbromate at* 296 K

CaO$_8$ polyhedron				
			Maximum and minimum angles for each polyhedral O atom	
Ca—O(1)	2.419 (3)		O(1)—Ca—O(3)	145.79 (10)
Ca—O(2)	2.345 (3)		O(1)—Ca—O(14$^{\text{iv}}$)	73.01 (9)
Ca—O(3)	2.381 (3)		O(2)—Ca—O(4)	146.70 (11)
Ca—O(4)	2.343 (3)		O(2)—Ca—O(22$^{\text{ii}}$)	69.39 (9)
Ca—O(11$^{\text{iii}}$)	2.540 (2)		O(3)—Ca—O(11$^{\text{iii}}$)	69.37 (9)
Ca—O(14$^{\text{iv}}$)	2.546 (2)		O(4)—Ca—O(11$^{\text{iii}}$)	73.10 (11)
Ca—O(22$^{\text{ii}}$)	2.554 (2)		O(11$^{\text{iii}}$)—Ca—O(22$^{\text{ii}}$)	146.34 (8)
Ca—O(23)	2.628 (3)		O(14$^{\text{iv}}$)—Ca—O(4)	142.22 (11)
			O(14$^{\text{iv}}$)—Ca—O(2)	69.49 (10)
			O(23)—Ca—O(1)	144.41 (9)
			O(23)—Ca—O(3)	69.46 (9)
Perbromate ion (I) Rigid body I				
		Rigid-body corrected		
Br(1)—O(11)	1.606 (2)	1.623	O(11)—Br(1)—O(12)	110.43 (13)
Br(1)—O(12)	1.606 (2)	1.621	O(11)—Br(1)—O(13)	108.97 (13)
Br(1)—O(13)	1.609 (2)	1.625	O(11)—Br(1)—O(14)	108.45 (13)
Br(1)—O(14)	1.601 (2)	1.619	O(12)—Br(1)—O(13)	110.37 (13)
			O(12)—Br(1)—O(14)	110.86 (13)
Mean for ion (I)	1.605 (3)	1.622	O(13)—Br(1)—O(14)	107.68 (13)
Perbromate ion (II) Rigid body II				
		Rigid-body corrected		
Br(2)—O(21)	1.619 (2)	1.634	O(21)—Br(2)—O(22)	108.71 (13)
Br(2)—O(22)	1.613 (2)	1.628	O(21)—Br(2)—O(23)	109.42 (13)
Br(2)—O(23)	1.606 (2)	1.622	O(21)—Br(2)—O(24)	111.37 (14)
Br(2)—O(24)	1.596 (3)	1.611	O(22)—Br(2)—O(23)	109.35 (13)
			O(22)—Br(2)—O(24)	108.84 (13)
Mean for ion (II)	1.608 (7)	1.624	O(23)—Br(2)—O(24)	109.12 (14)
Overall ion mean	1.607 (7)	1.623		
Water molecules				
O(1)—H(1)	0.68 (4)		H(1)—O(1)—H(2)	97 (4)
O(1)—H(2)	0.84 (4)			
O(2)—H(3)	0.64 (7)		H(3)—O(2)—H(4)	111 (6)
O(2)—H(4)	1.00 (6)			
O(3)—H(5)	0.73 (4)		H(5)—O(3)—H(6)	109 (5)
O(3)—H(6)	0.70 (4)			
O(4)—H(7)	0.57 (4)		H(7)—O(4)—H(8)	121 (5)
O(4)—H(8)	0.96 (5)			

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

Since among inequivalent atoms there are eight H atoms, four water O atoms and eight perbromate O atoms, the hydrogen bonding is rather complex. The four shortest hydrogen bonds have H···O distances in the range 1.87–1.99 Å with O—H···O angles in the range 167–178°, and involve H atoms 1, 2, 5 and 8 with two acceptors from anion (I), one from anion (II) and one from a water molecule (Table 3); the next-shortest have H···O distances in the range 2.08–2.25 Å with O—H···O angles in the range 136–162°, and involve H atoms 3, 4, 6 and 7. These eight hydrogen bonds involve each of the eight H atoms once, anion O atoms O(21) (thrice), O(13) (twice), O(12) and water O atoms O(1) and O(3) (once each).

Table 3. *Hydrogen-bond parameters* (Å, °)

Water oxygen–acceptor oxygen	Distance	Proton–acceptor oxygen	Observed distance	Neutron-adjusted distance*	Observed O—H—O angle
O(1)—O(13^{i})	2.940 (4)	H(1)—O(13^{i})	2.28 (4)	1.99	167 (5)
O(1)—O(12^{ii})	2.953 (4)	H(2)—O(12^{ii})	2.12 (4)	1.98	176 (4)
O(1)—O(12^{iii})	3.174 (4)†	H(1)—O(12^{iii})	2.79 (4)	2.66	119 (4)
		H(2)—O(12^{iii})	3.00 (4)	2.99	94 (3)
O(1)—O(14^{iv})	2.955 (4)	H(1)—O(14^{iv})	2.84 (4)	2.84	93 (4)
O(2)—O(3^{v})	3.044 (4)	H(3)—O(3^{v})	2.48 (7)	2.21	149 (8)
O(2)—O(21)	3.020 (4)	H(4)—O(21)	2.22 (6)	2.25	136 (5)
O(2)—O(24^{iv})	3.054 (4)	H(3)—O(24^{iv})	2.57 (6)	2.35	135 (7)
O(2)—O(23^{v})	3.054 (4)†	H(3)—O(23^{v})	2.93 (7)	2.92	95 (6)
		H(4)—O(23^{v})	2.60 (6)	2.62	107 (4)
O(2)—O(14^{iv})	2.792 (4)	H(3)—O(14^{iv})	2.61 (6)	2.57	100 (6)
O(2)—O(22^{ii})	2.794 (4)	H(4)—O(22^{ii})	2.64 (6)	2.64	88 (4)
O(3)—O(21^{iv})	2.838 (4)	H(5)—O(21^{iv})	2.11 (4)	1.87	178 (5)
O(3)—O(13^{vi})	3.033 (4)	H(6)—O(13^{vi})	2.35 (4)	2.10	163 (5)
O(3)—O(11^{iii})	2.804 (4)	H(6)—O(11^{iii})	2.52 (4)	2.45	107 (4)
O(3)—O(24^{vi})	3.103 (4)†	H(5)—O(24^{vi})	2.85 (4)	2.80	104 (4)
		H(6)—O(24^{vi})	3.03 (4)	3.04	90 (4)
O(3)—O(14^{vi})	3.278 (4)	H(6)—O(14^{vi})	2.87 (5)	2.74	120 (4)
O(3)—O(12^{iv})	3.299 (4)	H(6)—O(12^{v})	2.94 (4)	2.84	115 (4)
O(4)—O(21^{ii})	2.986 (4)	H(7)—O(21^{ii})	2.44 (4)	2.08	162 (6)
O(4)—O(1^{vii})	2.938 (4)	H(8)—O(1^{vii})	1.98 (5)	1.98	175 (4)
O(4)—O(24^{viii})	3.173 (4)†	H(7)—O(24^{viii})	2.83 (4)	2.64	123 (5)
		H(8)—O(24^{viii})	3.03 (4)	3.03	90 (3)
O(4)—O(11^{iii})	2.912 (4)	H(8)—O(11^{iii})	2.78 (4)	2.78	88 (3)
O(4)—O(22^{ii})	3.180 (4)	H(7)—O(22^{ii})	2.95 (4)	2.85	109 (5)

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

* See text for details.

† Bifurcated hydrogen bonds.

This involvement of water O atoms as acceptors in relatively strong hydrogen bonds is unusual among the salts we have investigated. Each of the four water molecules is also involved in a set of even weaker bifurcated hydrogen bonds. Water O atoms O(2) and O(4) are not involved in hydrogen bonding as acceptors, and are of hydrate class D; water O atoms O(1) and O(3) act as hydrogen-bond acceptors, and are of hydrate class H. O(2) and O(4) have B_{eq} values approximately 40% greater than those of O(1) and O(3), as well as substantially more eccentric displacement ellipsoids (Fig. 1), presumably as a result of their lesser involvement in hydrogen bonding.

This structure consists of columns of Ca ions along x surrounded by four columns of perbromate ions slightly canted with respect to x and occurring in pairs alternating with four columns of water molecules also occurring in pairs. These arrays, in turn, interact both by sharing perbromate groups approximately along [0$\bar{1}$1] and by hydrogen bonding along the columns and approximately along [011].

Experimental

Crystal data

$[Ca(H_2O)_4](BrO_4)_2$
M_r = 399.945
Triclinic
$P\bar{1}$
a = 5.6218 (9) Å
b = 8.0058 (7) Å
c = 11.9924 (16) Å
α = 80.643 (9)°
β = 89.160 (12)°
γ = 89.750 (10)°
V = 532.50 (12) Å^3
Z = 2
D_x = 2.494 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 14.2–15.0°
μ = 8.06 mm^{-1}
T = 296 K
Rod
0.31 × 0.19 × 0.15 mm
Colorless
Crystal source: aqueous solution

Data collection

Rigaku AFC-5S diffractometer
ω-2θ scans
Absorption correction: empirical (six ψ-scans)
T_{min} = 0.709, T_{max} = 1.000
2697 measured reflections
2447 independent reflections
1979 observed reflections [$I > 3\sigma(I)$]

R_{int} = 0.009
θ_{max} = 27.5°
$h = 0 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 15$
6 standard reflections monitored every 150 reflections intensity variation: ±2.2%

Refinement

Refinement on F
Final R = 0.021
wR = 0.027
S = 1.18
1979 reflections
169 parameters
All H-atom parameters refined
$w = (\sigma_F)^{-2}$
$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 0.49 e Å^{-3} [0.8 Å from Br(1)]

$\Delta\rho_{min}$ = -0.50 e Å^{-3} [0.8 Å from Br(2)]
Extinction correction: Zachariasen (1963, 1968)
Extinction coefficient: 1.43 × 10^{-6}
Atomic scattering factors for Ca, O, Br from Cromer & Waber (1974); for H from Stewart, Davidson & Simpson (1965)

Solid reagent-grade calcium hydroxide (Jenneile Chemical Company, Cincinnati, Ohio) was reacted with a slight excess of approximately 0.7 *M* perbromic acid prepared using a modification of Appelman's (1972) procedure. The resulting solution was evaporated at room temperature to produce crystalline tetraaquacalcium perbromate; the experimental sample grew upon further evaporation of the saturated mother liquor decanted from the initial growth. The sample was mounted with epoxy cement and was coated with Apiezon L grease to retard potential loss or gain of water.

Data were collected using scan widths of $(1.55 + 0.35\tan\theta)^\circ$ in ω, and a background/scan time ratio of 0.5. No decay correction was applied but the data were corrected for Lorentz-polarization effects and for absorption. For the latter, ψ-scan data were obtained just prior to the conclusion of intensity-data collection; the correction method was that of North, Phillips & Mathews (1968).

The crystal system was found to be triclinic. The initial space-group choice $P\bar{1}$ (No. 2) was confirmed by structure refinement. The direct-methods program *SHELXS*86 (Sheldrick, 1985) in the *TEXSAN* structure analysis package (Molecular Structure Corporation, 1989) was used to generate an *E* map from which the initial positions of the Ca atom and the two Br atoms were assigned. Subsequently, difference Fourier maps provided initial positions of the remaining 20 atoms. The non-H atoms were refined anisotropically, the H atoms isotropically, using full-matrix least squares. An extinction coefficient (Zachariasen, 1963, 1968) was included in the least-squares refinement; the maximum effect of extinction was 5.5% of F_o for 110.

As analyzed by the program *THMA*11 (1986) based on the work of Schomaker & Trueblood (1968), the polyhedron did not conform to rigid-body behavior, but the average magnitudes of the differences in the mean-square displacement amplitudes along the interatomic vectors for the ten unique atom pairs of the Br(1) and the Br(2) perbromate ions were both $17\,(18) \times 10^{-4}$ Å. Thus, the Hirshfeld (1976) rigid-bond test, extended as described by Rosenfield, Trueblood & Dunitz (1978), is satisfied by both anions and corrections for the rigid-body motion have been made (Table 2).

In the water molecules, the O—H bond lengths ranged from 0.57 to 1.00 Å. The molecular H—O—H angles were held fixed at the X-ray determined values; the O—H distances were adjusted to the mean values as determined by neutron diffraction for the appropriate hydrate class (Chiari & Ferraris, 1982; Ferraris & Franchini-Angela, 1972). O(1) and O (3) were assigned to hydrate class H, for which the mean O—H distance is 0.972 Å; O(2) and O (4) were assigned to class D, for which the mean distance is 0.956 Å. The neutron-adjusted distances involving the water H atoms and O acceptors are taken to be the hydrogen-bond lengths (Table 3).

Lists of structure factors, anisotropic displacement parameters and material relating to the rigid-body analysis have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71101 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1042]

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A New Modification of Sr_5Sb_3

A. Rehr and S. M. Kauzlarich

Department of Chemistry, University of California, Davis, California 95616, USA

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Abstract

Single crystals of Sr_5Sb_3 in the orthorhombic form (β-Yb_5Sb_3 structure type) were obtained as a minor product of a reaction of the elements and Mn in a welded Nb container. To date only the hexagonal form (Mn_5Si_3 structure type) of Sr_5Sb_3 has been reported. The orthorhombic phase of Sr_5Sb_3 has