SAKURAI, T., TSUBOYAMA, S. & TSUBOYAMA, K. (1980). *Acta Cryst.* B**36**, 1797–1801.
TSUBOYAMA, S., SAKURAI, T. & TSUBOYAMA, K. (1987). *J. Chem. Soc. Dalton Trans.* pp. 721–727.
TSUBOYAMA, S., TAKISHIMA, T., SAKURAI, T. & TSUBOYAMA, K. (1987). *Nippon Kagaku Kaishi*, pp. 313–317.
YASHIRO, M., AJIOKA, M., YANO, S., TORIUMI, K., ITO, T. & YOSHIKAWA, S. (1986). *Inorg. Chem.* **25**, 1709–1715.

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Structure of Barium Bromate Monohydrate

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Abstract. $Ba(BrO_3)_2.H_2O$, $M_r = 411{\cdot}17$, monoclinic, $I2/c$, $a = 9{\cdot}0696\,(10)$, $b = 7{\cdot}8952\,(9)$, $c = 9{\cdot}6295\,(8)$ Å, $\beta = 93{\cdot}26\,(1)°$, $V = 688{\cdot}4\,(2)$ Å^3, $Z = 4$, $D_m = 3{\cdot}950$, $D_x = 3{\cdot}966\,(1)$ g cm^{-3}, Mo $K\alpha_1$, $\lambda = 0{\cdot}70930$ Å, $\mu = 172{\cdot}5$ cm^{-1}, $F(000) = 736$, $T = 298$ K, $R = 0{\cdot}016$, $wR = 0{\cdot}020$ for 576 independent reflections with $I > \sigma(I)$. Each Ba atom has eleven O neighbors at distances 2·702 (6) to 3·057 (2) Å; the average Br—O bond length is 1·652 (6) Å, or 1·661 (5) Å if corrected for thermal motion; O···O = 2·832 (4) Å in a hydrogen bond.

Experimental. Crystals were prepared by mixing hot aqueous solutions of barium nitrate and sodium bromate and allowing the mixture to cool. The measured density is quoted from Heydweiller (1920). A crystal 0·043 × 0·055 × 0·25 mm (9 faces, elongated on **c**) was glued to a glass fiber and mounted on an Enraf–Nonius CAD-4 diffractometer equipped with a graphite monochromator. Cell dimensions were derived from 24 reflections in the range $12{\cdot}8 < \theta < 28{\cdot}0°$. Intensities were measured for θ up to 25° at two azimuthal angles [h 0 to 10, k −9 to 0, l −11 to 11] and at one azimuth [h −10 to −1, k −9 to 2, l −11 to 11]. Of 2047 reflections permitted by the space group, 119 were rejected as too weak to measure. After analytical correction for absorption $(2{\cdot}11 < A < 12{\cdot}1)$ reflections which were equivalent were averaged giving 582 unique ones with $F^2 > \sigma(F^2)$; $R_{\rm int} = 0{\cdot}031$. Measurements of standard reflections (800, 006, $0\bar{2}0$) every 3600 s of X-ray exposure were constant within about 1%; no correction was made. Refinement by least squares (on F) with weights $w = [\sigma(F)]^{-2}$, derived from $\sigma(F^2) = \{[\sigma_{\rm cs}(F^2)]^2 + (0{\cdot}03F^2)^2 + 41^2\}^{1/2}$, was started with coordinates of Kartha (1953). Anisotropic thermal parameters were assigned to 6 atoms. The H-atom coordinates and isotropic thermal parameter were refined; the starting position in the hydrogen bond between O2 and O4 was calculated. The 3 strongest reflections (reduced in intensity *ca* 25% by extinction) and 3 others which agreed poorly with the calculated values for unknown reasons were rejected. An empirical isotropic correction for extinction increased the largest remaining F by 12%. The maximum final shift was $0{\cdot}03\sigma$; extremes of ΔF synthesis were +0·56 and −0·58 e Å^{-3}; GOF = 1·10. Atomic scattering factors, including dispersion terms, for Ba^{2+}, Br, O (for O4), $O^{1/3-}$ (for O1, O2, O3) and H were taken from *International Tables for X-ray Crystallography* (1974). Calculations were made with unpublished local programs. Atomic parameters are listed in Table 1 and

Table 1. *Fractional atomic coordinates and isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{\rm eq} = \textstyle\sum(B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j)/3.$$

	x	y	z	$B/B_{\rm eq}^*$
Ba	0	0·39194 (3)	0·250	1·03*
O4	0	0·0497 (7)	0·250	3·73*
Br	0·25101 (3)	0·27704 (5)	0·55741 (3)	1·11*
O1	0·07699 (27)	0·3401 (4)	0·56027 (25)	1·64*
O2	0·2566 (3)	0·0980 (3)	0·64701 (28)	1·87*
O3	0·2629 (3)	0·2181 (3)	0·39456 (25)	1·94*
H	0·051 (6)	0·011 (7)	0·215 (5)	2·2 (11)

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

Ba—O1, O1$^{\rm i}$	3·057 (2)	Ba—O3, O3$^{\rm i}$	3·022 (3)
—O1$^{\rm iv}$, O1$^{\rm v}$	2·906 (6)	—O3$^{\rm vi}$, O3$^{\rm vii}$	2·767 (6)
—O2$^{\rm ii}$, O2$^{\rm iii}$	2·874 (6)	—O4	2·702 (6)
Br—O1	1·657 (2), 1·663*	O1—Br—O2	104·4 (1)
—O2	1·655 (3), 1·664*	O1—Br—O3	102·4 (1)
—O3	1·645 (2), 1·655*	O2—Br—O3	104·7 (1)
O4—H, H$^{\rm i}$	0·67 (5)	H—O4—H$^{\rm i}$	125 (9)
O4···O2$^{\rm viii}$, O2$^{\rm ix}$	2·832 (4)	H—O4···O2$^{\rm viii}$	12 (5)

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

* Corrected for thermal motion by riding model.

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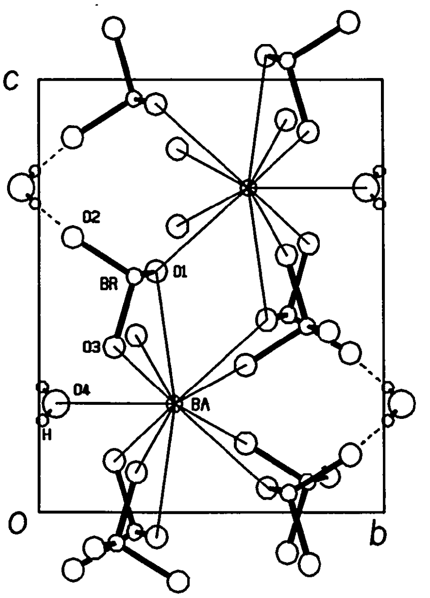

Fig. 1. Projection of part of the structure down [100]. Broken lines indicate hydrogen bonds.

interatomic distances and angles in Table 2.* Fig. 1 shows a view of the structure (Johnson, 1976).

Related literature. The bromate ion exhibits X-ray birefringence and novel diffraction effects near the Br *K* absorption edge (Templeton & Templeton, 1985, 1987). Crystals of barium bromate monohydrate were prepared to study these phenomena in another space-group symmetry. Kartha (1953) determined this crystal structure from photographic data ($R = 0{\cdot}20$). It was redetermined because analysis of the planned experiments requires more accurate structural parameters. Kartha (1952) showed that $Ba(ClO_3)_2.H_2O$ has the same structure. Other salt monohydrates which are isomorphous include lead chlorate, lead bromate, strontium bromate and barium iodate (Groth, 1908). Structures of $NaBrO_3$ and $KBrO_3$ (Abrahams & Bernstein, 1977; Templeton & Templeton, 1985) show bromate-ion dimensions similar to those reported here, other than the O1—Br—O3 angle. That angle may be smaller as a result of the bidentate coordination to Ba.

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* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51555 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Abrahams, S. C. & Bernstein, J. L. (1977). *Acta Cryst.* **B33**, 3601–3604.
Groth, P. (1908). *Chemische Krystallographie,* Vol. II, pp. 111–116. Leipzig: Engelmann.
Heydweiller, A. (1920). *Z. Phys.* **3**, 308–317.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Kartha, G. (1952). *Acta Cryst.* **5**, 845–846.
Kartha, G. (1953). *Proc. Indian Acad. Sci.* **A38**, 1–12; *Struct. Rep.* **17**, 527–528.
Templeton, D. H. & Templeton, L. K. (1985). *Acta Cryst.* **A41**, 133–142.
Templeton, D. H. & Templeton, L. K. (1987). *Acta Cryst.* **A43**, 573–574.

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Structure of $KMoP_2O_7$, a Molybdenum Pyrophosphate Containing Isolated Mo^{3+} Cations

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Abstract. Molybdenum potassium pyrophosphate, $KMoP_2O_7$, $M_r = 308{\cdot}985$, monoclinic, $P2_1/c$, $a = 7{\cdot}376\,(1)$, $b = 10{\cdot}355\,(1)$, $c = 8{\cdot}350\,(4)$ Å, $\beta = 106{\cdot}83\,(3)°$, $V = 610{\cdot}44$ Å^3, $Z = 4$, $D_x = 3{\cdot}362$ g cm^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}7093$ Å, $\mu = 3{\cdot}28$ mm^{-1}, $F(000) = 588$, $T = 296$ K, $R = 0{\cdot}020$ and $wR = 0{\cdot}025$ for 1363 reflections with $I > 2{\cdot}5\sigma(I)$. The compound, which forms a tunnel structure along the *c* axis, is

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