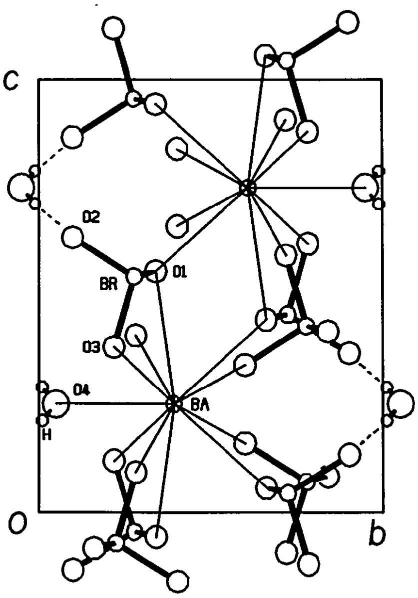

Fig. 1. Projection of part of the structure down [100]. Broken lines indicate hydrogen bonds.

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.

This research was supported by National Science Foundation Grant CHE-8515298. It used facilities of the Lawrence Berkeley Laboratory, supported by Department of Energy Contract DE-AC03-76-SF00098, and the UC Berkeley X-ray Crystallographic Facility (CHEXRAY).

---

* 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.* B**33**, 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.* A**38**, 1–12; *Struct. Rep.* **17**, 527–528.
Templeton, D. H. & Templeton, L. K. (1985). *Acta Cryst.* A**41**, 133–142.
Templeton, D. H. & Templeton, L. K. (1987). *Acta Cryst.* A**43**, 573–574.

Acta Cryst. (1989). C**45**, 673–675

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

By J. J. Chen, C. C. Wang and K. H. Lii*

Institute of Chemistry, Academia Sinica, Nankang, Taipei, Taiwan

(*Received* 9 *June* 1988; *accepted* 14 *October* 1988)

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

---

* To whom correspondence should be addressed.

0108-2701/89/040673-03$03.00

Table 1. *Positional parameters and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{4}{3}\textstyle\sum_i\sum_j \beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
K	0·8227 (1)	0·1766 (1)	0·9431 (1)	1·61 (3)
Mo	0·7663 (1)	−0·3998 (1)	0·7399 (1)	0·36 (1)
P1	0·8668 (1)	−0·0990 (1)	0·6738 (1)	0·49 (3)
P2	0·5552 (1)	−0·3602 (1)	0·3056 (1)	0·50 (3)
O1	0·9244 (3)	−0·2309 (2)	0·7521 (3)	0·86 (9)
O2	0·6783 (4)	−0·3923 (3)	0·4781 (3)	1·27 (10)
O3	0·6422 (3)	−0·5836 (2)	0·7361 (3)	0·94 (9)
O4	0·8657 (4)	−0·4060 (3)	0·9956 (3)	1·29 (10)
O5	0·5405 (3)	−0·2822 (2)	0·7612 (3)	0·85 (9)
O6	1·0087 (3)	−0·4967 (2)	0·7178 (3)	0·67 (8)
O7	0·6560 (3)	−0·4298 (2)	0·1813 (3)	0·77 (8)

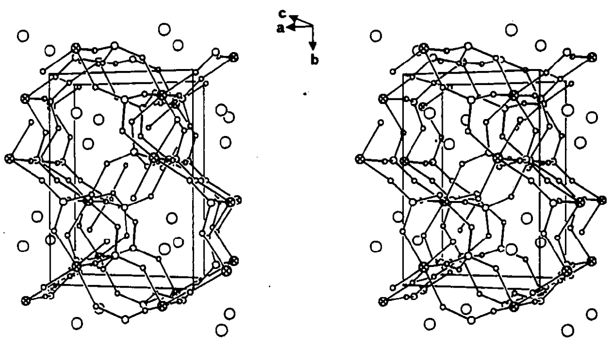

Fig. 1. A stereoscopic view of the structure of $KMoP_2O_7$ in a direction approximately parallel to the *c* axis. The K, Mo, P and O atoms are represented by large open circles, cross-hatched circles and medium and small open circles, respectively.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

Mo—O(4)	2·048 (3)	K—O(5^{viii})	2·761 (3)
Mo—O(1)	2·088 (2)	K—O(6^{iv})	2·746 (3)
Mo—O(2)	2·093 (3)	K—O(6^{vi})	2·965 (3)
Mo—O(6)	2·105 (2)	P(1)—O(4^{ix})	1·486 (3)
Mo—O(3)	2·108 (2)	P(1)—O(6^{iv})	1·517 (2)
Mo—O(5)	2·112 (2)	P(1)—O(1)	1·521 (3)
K—O(1^{i})	2·743 (3)	P(1)—O(7^{vi})	1·602 (3)
K—O(1^{iv})	2·969 (3)	P(2)—O(2)	1·501 (3)
K—O(2^{vi})	3·174 (3)	P(2)—O(3^{x})	1·512 (3)
K—O(3^{vii})	3·097 (3)	P(2)—O(5^{ix})	1·517 (3)
K—O(3^{vi})	3·252 (3)	P(2)—O(7)	1·611 (3)
K—O(4^{i})	3·241 (3)		

O(1)—Mo—O(2)	91·1 (1)	P(1)—O(7)—P(2)	125·2 (2)
O(1)—Mo—O(3)	172·2 (1)	O(1)—P(1)—O(7^{vi})	107·6 (1)
O(1)—Mo—O(4)	87·1 (1)	O(1)—P(1)—O(6^{iv})	109·0 (1)
O(1)—Mo—O(5)	87·5 (1)	O(1)—P(1)—O(4^{ix})	112·4 (2)
O(1)—Mo—O(6)	85·8 (1)	O(4^{ix})—P(1)—O(6^{iv})	113·6 (1)
O(2)—Mo—O(3)	90·9 (1)	O(4^{ix})—P(1)—O(7^{vi})	108·0 (2)
O(2)—Mo—O(4)	177·2 (1)	O(6^{iv})—P(1)—O(7^{vi})	105·9 (1)
O(2)—Mo—O(5)	93·0 (1)	O(2)—P(2)—O(3^{x})	114·2 (2)
O(2)—Mo—O(6)	86·6 (1)	O(2)—P(2)—O(5^{ix})	115·5 (2)
O(3)—Mo—O(4)	90·6 (1)	O(2)—P(2)—O(7)	104·8 (2)
O(3)—Mo—O(5)	99·9 (1)	O(3^{x})—P(2)—O(5^{ix})	108·8 (1)
O(3)—Mo—O(6)	86·8 (1)	O(3^{x})—P(2)—O(7)	105·8 (1)
O(4)—Mo—O(5)	89·0 (1)	O(5^{ix})—P(2)—O(7)	106·9 (1)
O(4)—Mo—O(6)	91·1 (1)		
O(5)—Mo—O(6)	173·3 (1)		

Symmetry code: (i) $2-x$, $-y$, $2-z$; (ii) x, $0·5-y$, $0·5+z$; (iii) x, $0·5-y$, $-0·5+z$; (iv) $2-x$, $0·5+y$, $1·5-z$; (v) $1-x$, $-y$, $1-z$; (vi) x, $-0·5-y$, $0·5+z$; (vii) x, $1+y$, z; (viii) $1-x$, $0·5+y$, $1·5-z$; (ix) x, $-0·5-y$, $-0·5+z$; (x) $1-x$, $-1-y$, $1-z$.

isostructural with $CsMoP_2O_7$. The counter cations are located in the tunnels formed by pyrophosphate groups and unusual $Mo^{3+}O_6$ octahedra. Trivalent molybdenum appears as a rare species especially in oxides.

Experimental. Yellowish green crystals of $KMoP_2O_7$ were prepared by heating a mixture of K_2MoO_4, Mo and P_2O_5 (mole ratio of 1:1:2) at 1423 K in an evacuated quartz ampoule for several days. Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\bar{\alpha}$ radiation; ω–2θ scan technique. Cell parameters of crystal 0·26 × 0·15 × 0·20 mm from least-squares procedure on 25 reflections ($9·5° < \theta < 12°$). Analytical absorption correction applied; max./min. transmission factors 0·9983/0·8678. Systematic absences: $0k0$, $k = 2n$; $h0l$, $l = 2n$. Total of 1405 reflections measured with $(\sin\theta/\lambda)_{\max} = 0·651$ Å^{-1} and in the range $-9 \le h \le 9$, $0 \le k \le 13$, $0 \le l \le 10$. No significant variation in intensities of three standards ($\bar{2}2\bar{3}$, $03\bar{4}$, 034) monitored every 2 h. Scan width of $0·80° + 0·35°\tan\theta$ and scan speed 5° min^{-1}. 1363 unique structure amplitudes with $I > 2·5\sigma(I)$. The structure was solved by direct methods and refined by full-matrix least-squares refinement based on F values. All of the atoms were refined anisotropically. At convergence $R = 0·020$, $wR = 0·025$, $w = [\sigma^2(F)]^{-1}$, $\sigma^2(F)$ based on counting statistics, $(\Delta/\sigma)_{\max} = 0·01$, $S = 1·32$, $(\Delta\rho)_{\max} = 0·56$ e Å^{-3}, $(\Delta\rho)_{\min} = -0·77$ e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a VAX 11/780 computer system using NRC VAX program (Larson, Lee, Le Page & Gabe, 1986). Atomic parameters are given in Table 1, bond distances and angles in Table 2.* A stereoscopic view of the structure of $KMoP_2O_7$ along the *c* axis is shown in Fig. 1.

Related literature. The $KMoP_2O_7$ compound is isostructural with $CsMoP_2O_7$ (Lii & Haushalter, 1987) and contains Mo^{3+} cations in the oxygen array.

Support for this study from the National Science Council and the Institute of Chemistry Academia Sinica are gratefully acknowledged.

---

* Lists of structure factors and anisotropic thermal parameters and full lists of bond distances and bond angles, have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51533 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

LARSON, A. C., LEE, F. L., LE PAGE, Y. & GABE, E. J. (1986). *The NRC VAX Crystal Structure System*. National Research Council of Canada, Ottawa, Canada.

LII, K. H. & HAUSHALTER, R. C. (1987). *Acta Cryst.* **C43**, 2036–2038.

Acta Cryst. (1989). C**45**, 675–676

Structure of Rubidium Hydrogen (+)-Tartrate

BY LIESELOTTE K. TEMPLETON AND DAVID H. TEMPLETON

Department of Chemistry, University of California, Berkeley, CA 94720, *USA*

(*Received* 16 *September* 1988; *accepted* 31 *October* 1988)

Abstract. $[RbH(C_4H_4O_6)]$, $M_r = 234{\cdot}55$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}9233\,(8)$, $b = 10{\cdot}9883\,(9)$, $c = 7{\cdot}6527\,(22)$ Å, $V = 666{\cdot}3\,(2)$ Å^3, $Z = 4$, $D_m = 2{\cdot}29$, $D_x = 2{\cdot}338\,(1)$ g cm^{-3}, Mo $K\alpha$, $\lambda(\alpha_1) = 0{\cdot}70930$ Å, $\mu = 72{\cdot}1$ cm^{-1}, $F(000) = 456$, $T = 299$ K, $R = 0{\cdot}014$, $wR = 0{\cdot}015$ for 1100 independent reflections with $I > \sigma(I)$. The structure is nearly the same as that of caesium hydrogen tartrate except for cation–oxygen distances shorter by 0·15 Å on average. The anomalous scattering term f'' for rubidium measured at 0·71 Å is 2·90 (4).

Experimental. A mixture of RbCl, (+)-tartaric acid and water was heated and allowed to cool. Two crystals were studied. The first, with diameters 0·2 to 0·3 mm, was described by 17 faces. Absorption corrections did not agree well with intensities measured at different azimuths, and R could not be reduced below 0·042 for 978 independent reflections. Another crystal 0·021 × 0·060 × 0·37 mm (seven 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 19 reflections in the range $16{\cdot}4 < \theta < 27{\cdot}7°$. Intensities were measured (θ–2θ scan) for θ up to 25° for h −9 to 9, k −12 to 0, l −9 to 9. Of 2443 reflections permitted by the space group, 196 were rejected as too weak to measure ($I < \sigma$). After analytical correction for absorption ($1{\cdot}16 < A < 1{\cdot}51$) reflections which were equivalent in point group 222 were averaged giving 1102 unique ones with $F^2 > \sigma(F^2)$; $R_{\rm int} = 0{\cdot}019$. Measurements of standard reflections (400, $0\bar{6}0$, $00\bar{6}$) every 7200 s of X-ray exposure were constant within about 2%; no correction was made. Refinement by least squares (on F) with weights $w = [\sigma(F)]^{-2}$, derived from $\sigma(F^2) = \{[\sigma_{\rm cs}(F^2)]^2 + (0{\cdot}017F^2)^2\}^{1/2}$, was started with parameters for the caesium isomorph (Templeton & Templeton, 1978). Anisotropic thermal parameters were assigned to 11 atoms and isotropic ones to five hydrogen atoms. The dispersion correction f'' for rubidium was included as a variable. Two reflections were rejected because of poor agreement with calculations: 130, the strongest one, and 020, another low-angle one. An empirical isotropic correction for extinction increased F by 1% for the strongest remaining reflection. The maximum final shift was $0{\cdot}005\,\sigma$; extremes of a ΔF synthesis were +0·19 and −0·21 e Å^{-3}; $S = 1{\cdot}12$. Atomic scattering factors, including dispersion terms, for Rb^+, C, O (for O1 to O4), $O^{1/2-}$ (for O5, O6) 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 interatomic distances and angles in Table 2, with atoms numbered as in Templeton & Templeton (1978).[*] Positions for non-hydrogen atoms found in the unsatisfactory first experiment differ by 0·02 Å or less. The measured density is quoted from van Bommel & Bijvoet (1958).

Related literature. Potassium, rubidium, caesium, ammonium and thallium(I) hydrogen (+)-tartrates are all isomorphous (Groth, 1910). Crystals of the ammonium, potassium and rubidium salts were studied with X-rays by van Bommel & Bijvoet (1958). They refined the parameters (including hydrogen-atom coordinates) for the ammonium salt and used data from the rubidium salt to verify the absolute configuration of the tartrate ion. The parameters for the caesium compound were determined by Templeton & Templeton (1978) and used to measure anomalous scattering of synchrotron radiation by caesium near the L absorption edges

---

[*] Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51554 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/89/040675-02$03.00