KELLER, R. & HÖLZAPFEL, W. B. (1977). *Rev. Sci. Instrum.* **48**, 517–523.

LARSON, A. C. (1970). *Crystallographic Computing*, p. 291. Copenhagen: Munksgaard.

LIEBENBERG, D. H. (1979). *Phys. Lett. A*, **73**, 74–77.

MERRILL, L. & BASSETT, W. A. (1974). *Rev. Sci. Instrum.* **45**, 290–294.

MILLS, R. L. & GRILLY, E. R. (1955). *Phys. Rev.* **99**, 480–486.

MILLS, R. L., LIEBENBERG, D. H., BRONSON, J. C. & SCHMIDT, L. C. (1980). *Rev. Sci. Instrum.* **51**, 891–895.

NICOL, M. (1980). Private communication to D. SCHIFERL.

NICOL, M., HIRSCH, K. R. & HOLZAPFEL, W. B. (1979). *Chem. Phys. Lett.* **68**, 49–52.

SCHIFERL, D. (1977). *High Temp. High Pressures*, **9**, 71–75.

SCHIFERL, D., CROMER, D. T. & MILLS, R. L. (1978). *High Temp. High Pressures*, **10**, 493–496.

SCHUCH, A. F. & MILLS, R. L. (1970). *J. Chem. Phys.* **52**, 6000–6008.

SCHWALBE, L. A., SCHIFERL, D., MILLS, R. L., JONES, L. H., EKBERG, S., CROMER, D. T., LeSAR, R. & SHANER, J. (1980). Proc. VII Int. AIRAPT Conf. Le Creusot, France. To be published.

STEVENSON, R. (1957). *J. Chem. Phys.* **27**, 673–675.

STEWART, J. W. (1959). *J. Phys. Chem. Solids*, **12**, 122–129.

SUTTON, L. E. (1958). *Tables of Interatomic Distances and Configuration in Molecules and Ions*. London: The Chemical Society.

Paraelectric–Paraelastic Rb₂KMoO₃F₃ Structure at 343 and 473 K

BY S. C. ABRAHAMS AND J. L. BERNSTEIN

Bell Laboratories, Murray Hill, New Jersey 07974, USA

AND J. RAVEZ

Laboratoire de Chimie du Solide du CNRS, Université de Bordeaux I, 33405 Talence CEDEX, France

Abstract

Rb₂KMoO₃F₃, FW 410·975, is cubic above T_c = 328 K with space group $Fm3m$ and $a(T_c)$ = 8·945 (5) Å, $V(T_c)$ = 715·72 Å³; D_m(298 K) = 3·81 (4), $D_x(T_c)$ = 3·814 Mg m⁻³, Z = 4, μ(Mo $K\alpha$) = 15·7 mm⁻¹, $F(000)$ = 744. The linear thermal expansion coefficient, α, is 30 (3) × 10⁻⁶ K⁻¹. All allowed reflections within a hemisphere (or quadrant) of reciprocal space with (sin θ)/λ ≤ 1·15 Å⁻¹ were measured at 343 K (or 473 K) using a CAD-4 diffractometer, resulting in 97 independent structure factors at 343 K, 81 at 473 K. Least-squares refinement gave R = 0·032 at 343 K, 0·056 at 473 K. The O and F atoms randomly occupy the same equivalent 24(*e*) positions, with anisotropic thermal vibrations that increase significantly between 343 and 473 K. Diffuse scattering is not observed above T_c. The crystal becomes ferroelastic and ferroelectric below T_c. The Mo—O/F distance in the regular $\mathrm{Mo(O/F)_6^{3-}}$ octahedron, on correction for thermal motion, is 1·900 (6) Å at 343 K, 1·926 (10) Å at 473 K.

Introduction

The atomic arrangement in crystals with chemical formula R_3MX_6 or R_2MX_6, which are also isostructural with $(NH_4)_3FeF_6$ (Pauling, 1924) or K_2PtCl_6 (Williams, Dillin & Milligan, 1973), may undergo slight distortions from cubic symmetry at a lower temperature and may thereby acquire interesting physical properties. One such family with composition $A_2BMO_3F_3$ has ten members in which A, B = K, Rb, Cs and M = Mo, W, each of which exhibits at least one phase transition above 78 K (Ravez, Peraudeau, Arend, Abrahams & Hagenmuller, 1980). The transition at T_c = 328 K for Rb₂KMoO₃F₃ is from rhombohedral to cubic symmetry: a dielectric anomaly is associated with the transition and the spontaneous polarization decreases to zero above 328 K. The crystal becomes ferroelastically twinned in the rhombohedral phase (Abrahams, Bernstein & Ravez, 1981). The structure in the cubic phase has been studied at two temperatures to give accurate prototype parameters preparatory to an investigation of a single-domain

crystal below the phase transition. The present results are compared with recently reported values for the position and thermal parameters of K_2SnCl_6, which is cubic at 300 K and is isostructural with K_2PtCl_6 (Ihringer, 1980).

Experimental

A sphere of radius 0·161 (2) mm was ground from a large crystal taken from a boule grown by the Bridgman technique (Chaminade, 1979) and mounted within a microfurnace (Lissalde, Abrahams & Bernstein, 1978). The regulating thermocouple was positioned within 0·5 mm of the crystal. A total of 4846 f.c.c. reflections are possible within a sphere of reciprocal space with $(\sin\theta)/\lambda \leq 1\cdot15$ Å^{-1}. All allowed and a sampling of forbidden reflections within a hemisphere at 343 K, and those within a quadrant at 473 K, were measured with an Enraf–Nonius CAD-4 diffractometer controlled by a PDP 11/40-8e computer using Enraf–Nonius (1979) software (see Table 1 for variables). Pyrolytic-graphite-monochromated Mo $K\alpha$ radiation was used to give the integrated intensities of 4150 reflections at 343 K and 2229 reflections at 473 K. Corrections were made for Lorentz, polarization and absorption effects. The integrated intensities of five standard reflections were remeasured hourly and the crystal orientation was checked after every 100 reflections. An averaged second-degree polynomial scaling function, derived from the average increase of 12·3% at 343 K and 11·1% at 473 K in the intensities of the five standards, was used to correct both sets of integrated intensities. Based on a counting-statistics estimate of σF^2meas, 426 reflections at 343 K and 341 at 473 K had F^2meas $> 3\sigma F^2$meas: the remainder, which included all the forbidden reflections, were regarded as unobserved and were not used in the following analysis. After averaging there resulted a total of 97 symmetry-independent Fmeas at 343 K, 81 at 473 K.* Final estimates of σFmeas, as used in least-squares refinement, are derived from the expression $(\sigma F^2\text{meas})^2 = V_1 + V_2(F\text{meas})^4 + $ larger of $[V_3(F\text{meas})^4$ or $V_4]$, where V_1 is the variance given by counting statistics, V_2 is the result of the first summation, V_3 is that of the second summation in equation (2) of Abrahams, Bernstein & Keve (1971), and V_4 is the variance given by differences among members of a form (see Table 1 for values of the constants).

The variation of the cubic lattice parameter (a) with temperature is shown in Fig. 1. Each value is based on

* The two sets of structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36008 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Variables used for structure factor measurement for cubic* $Rb_2KMoO_3F_3$

Transmission factors	0·041–0·100
Scan type	ω–2θ
Scan width	$0\cdot60° + 0\cdot35° \tan\theta$
Maximum counting time	120 s
2θ range	$5° \leq 2\theta \leq 110°$
V_2, V_3 (see text)	0·0043, 0·0038

measurement of the Bragg angle for 15 reflections with $13° \leq \theta \leq 30°$ followed by least-squares refinement. The solid line has been fitted by the method of least squares to the experimental values in Fig. 1, and corresponds to the lattice constant at T_c and linear expansion coefficient (α) given in the *Abstract*.

Initial coordinates above T_c were taken from the model found for the twinned ferroelectric–ferroelastic crystal previously studied at room temperature (Bernstein, Abrahams & Ravez, 1979). Least-squares refinement was made on the Honeywell 6080 computer using *ORFLS*-3 (Busing, Martin & Levy, 1973). Atomic scattering factors for neutral atoms were taken from *International Tables for X-ray Crystallography* (1974), as were the anomalous-dispersion corrections. Isotropic extinction corrections (Coppens & Hamilton, 1970) were made for both sets of structure factors, using *ORFLS*-3: the final value of $g = 9\cdot26$ (2·4) $\times$ 10^2 at 343 K, 2·7 (2·9) $\times$ 10^2 at 473 K. Refinement with isotropic temperature factors gave $R = 0\cdot0457$, $wR = 0\cdot0989$ and $S = 1\cdot315$ at 343 K; $R = 0\cdot0674$, $wR = 0\cdot1000$ and $S = 1\cdot479$ at 473 K [see Abrahams *et al.* (1971) for definitions]. The anisotropic model, with only one additional thermal parameter for the randomly occupied O and F atom site, gave $R = 0\cdot0318$, $wR = 0\cdot0689$ and $S = 0\cdot921$ at 343 K; $R = 0\cdot0564$,
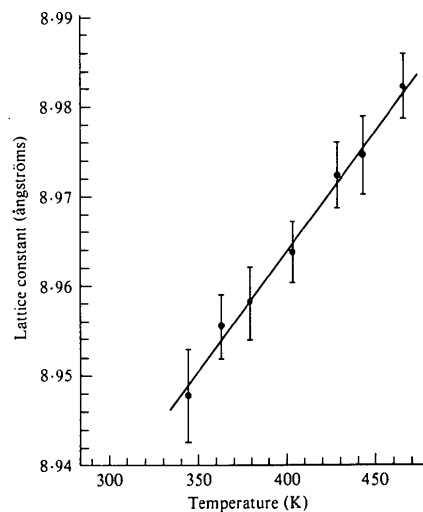
Fig. 1. Variation of the $Rb_2KMoO_3F_3$ lattice parameter with temperature in the cubic phase: the transition temperature $T_c = 328$ K. The length of the error bars represents 2 e.s.d.

Table 2. *Atomic positional and thermal parameters of* $Rb_2KMoO_3F_3$ *at* 343 *and* 473 K, *with* u_{ii} *in* Å

Rb in 8(c)	$(\frac{1}{4},\frac{1}{4},\frac{1}{4})$, $u_{11} = 0.195$ (2)
	$(\frac{1}{4},\frac{1}{4},\frac{1}{4})$, $u_{11} = 0.222$ (2)
K in 4(b)	$(\frac{1}{2},\frac{1}{2},\frac{1}{2})$, $u_{11} = 0.146$ (3)
	$(\frac{1}{2},\frac{1}{2},\frac{1}{2})$, $u_{11} = 0.165$ (3)
Mo in 4(a)	(000), $u_{11} = 0.186$ (2)
	(000), $u_{11} = 0.197$ (3)
O/F in 24(e)	[0.2105 (7), 0, 0], $u_{11} = 0.148$ (8)
	$u_{22} = 0.257$ (6)
	[0.2119 (11), 0, 0], $u_{11} = 0.187$ (11),
	$u_{22} = 0.287$ (10)

The upper line in each set of parameters corresponds to values at 343 K, the lower line to values at 473 K.

$wR = 0.0871$ and $S = 1.297$ at 473 K. In the final refinement cycle, the largest parameter shift was less than 10^{-3} of the corresponding estimated standard deviation. The final coordinates at 343 and 473 K are given in Table 2.

Independent evidence that the O and F atoms randomly occupy the same site is provided by the ease with which alkali-metal ions may be replaced by alkaline earths, or Mo by Fe, Nb or Ti, with corresponding change in the O:F ratio and variation in the transition temperature.

Amplitudes of vibration

The thermal vibrations of the O/F atoms in $Rb_2KMoO_3F_3$ are found, on the basis of Hamilton's (1965) $\mathscr{R}$ ratio, to be significantly anisotropic at 343 and 473 K: the value of wR(isotropic)/wR(anisotropic) = 1.435 at 343 K, 1.148 at 473 K whereas the theoretical value at the half-percent significance level is

1.051 at 343 K, 1.060 at 473 K. The heavy atoms are constrained by symmetry to vibrate isotropically, hence the anisotropy is entirely associated with the O/F atoms. Individual amplitudes of vibration increase very significantly for the cations between 343 and 473 K: the magnitude of the increase for the O/F atom is slightly larger but is of reduced significance (see Table 2).

The amplitudes reported for five crystals isostructural with cubic K_2PtCl_6, of formula K_2MX_6 with $X = Cl$ or Br, and also those for the related $Cs_2NaPrCl_6$ are collected in Table 3. The observed magnitudes, which are likely to be in greater error than indicated by the given estimated standard deviations (*cf.* Hamilton & Abrahams, 1970), are expected to be a function of T/T_c and hence not directly comparable. Nevertheless, it may be noted that the amplitude for the atom in position 8(c) is consistently close to the average value of the amplitudes for the atom in position 24(e) in all seven crystals. The amplitude found for Mo in $Rb_2KMoO_3F_3$ is higher than that reported for other atoms in the 4(a) position in Table 3, and probably results from the influence of the additional atoms in the 4(b) position. The low amplitude for Pr found in $Cs_2NaPrCl_6$ by neutron diffraction powder profile analysis (Fish, Rhyne, Lynn & Patterson, 1980) is highly correlated with the thermal parameter for Na (Fish, 1980) and does not differ significantly from the amplitudes of the other atoms in the 4(a) position as given in Table 3. Further study of $A_2BMX_3Y_3$ compounds is necessary to clarify the relationships among the observed amplitudes of vibration.

Interatomic distances and angles

Symmetry requirements in space group $Fm3m$ result in the O and F atoms occupying, on average, the same set

Table 3. *R.m.s. amplitudes of vibration* (Å) *in some cubic* $A_2BMX_3Y_3$ *compounds*

| Compound | Atomic position | | | | T (K)† | T_c (K) | Reference |
	4(a)	4(b)	8(c)	24(e)*			
K_2SnCl_6	0.144 (2)	—	0.232 (4)	0.144 (7)	300	111	Ihringer (1980)
				0.286 (7)			
K_2RuCl_6	0.120 (8)	—	0.194 (12)	0.124 (8)	295		Deloume, Faure &
				0.202 (7)			Thomas-David (1979)
K_2ReCl_6	0.132 (3)	—	0.210 (2)	0.144 (5)	300	111	Grundy & Brown (1970)
				0.230 (1)			
K_2ReBr_6	0.136 (4)	—	0.213 (10)	0.153 (9)	300		Grundy & Brown (1970)
				0.262 (3)			
K_2PtBr_6	0.116 (2)	—	0.207 (7)	0.135 (4)	300	$83 < T_c < 195$	Grundy & Brown (1970)
				0.241 (1)			
$Cs_2NaPrCl_6$	0.09 (4)	0.14 (5)	0.21 (1)	0.155 (8)	295	155	Fish *et al.* (1980)
				0.262 (4)			
$Rb_2KMoO_3F_3$	0.186 (2)	0.146 (3)	0.195 (2)	0.148 (8)	343	328	Present work
				0.257 (6)			

* The upper value is along the M–X/Y bond, the lower value is perpendicular to the bond.

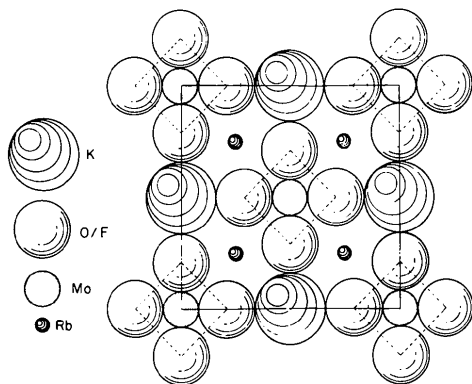† T is the measurement temperature, T_c is the phase-transition temperature.

Fig. 2. Arrangement of $MoO_3F_3^{3-}$ octahedra and K^+ ions in the (001) plane of one unit cell: the octahedra (dashed lines) at $z = 0$ are completed by pairs of O/F atoms at $0, 0, \pm z$ and $\frac{1}{2}, \frac{1}{2}, \pm z$ ($z \simeq 0\cdot21$). The Rb^+ ions, at $z = \pm\frac{1}{4}$, are represented in the (001) plane by vestigial circles. The Rb^+ ionic radius is about 12% greater than that of K^+.

of equivalent 24(e) positions. Part of the atomic arrangement is illustrated in Fig. 2. The Rb atom is 12-coordinated with Rb—O/F distance, based on the atomic coordinates in Table 2 and lattice constant taken from the *Abstract*, of 3·183 (1) Å at 343 K, 3·194 (1) Å at 473 K. Correction for thermal motion gives 3·185 (1) and 3·197 (1) Å respectively. The increase of 0·012 Å is significant in terms of the estimated standard deviation computed with *ORFFE*-3 (Busing, Johnson, Thiessen & Levy, 1973) from the least-squares variance–covariance matrix. The shortest Mo—O/F distance is 1·883 (6) Å at 343 K, 1·903 (10) Å at 473 K with the six O/F atoms forming a regular octahedron about the Mo atoms. The Mo—O/F distance on correction for thermal motion, 1·900 (6) Å at 343 K, 1·926 (10) Å at 473 K, is significantly shorter than that expected (Shannon, 1976) for a regular octahedral Mo—O distance of 1·935 Å, allowing for the smaller F^- radius. The six K—O/F distances of 2·591 (6) Å at 343 K, 2·588 (10) Å at 473 K [2·608 (6) and 2·609 (10) Å respectively, on correction for thermal motion] are also arranged as in a regular octahedron. Neither the Mo—O/F nor the K—O/F distance changes significantly with temperature in the range studied. The increase in Rb—O/F distance between 343 and 473 K is nearly identical to the increase of 0·015 Å in Mo—Rb or Rb—K distance, which is due entirely to thermal expansion.

$A_2BMX_3Y_3$ family: paraelectric–paraelastic prototype structure

Numerous materials with formula $A_2BMX_3Y_3$, in which A and B, also X and Y, may be identical or B may be absent, crystallize in space group $Fm3m$ with

atomic positions as given in Table 2. Atoms A and B are generally alkali metals or NH_4^+, M is usually a 3d, 4d or 5d transition metal, and X and Y are generally O^{2-}, F^-, Cl^-, Br^-, CN^- or NO_2^- ions. $Rb_2KMoO_3F_3$ is a typical member of this family. In the case that B is absent, the 4(b) symmetry positions in $Fm3m$ are unoccupied as shown in Table 3. As is found with many ABO_3 compounds that crystallize in the cubic perovskite structure and that undergo small distortions at lower temperatures, so small departures from the cubic prototype $A_2BMX_3Y_3$ structure have been detected. Thus four phase transitions have been reported in K_2ReCl_6 (Busey, Dearman & Bevan, 1962), two in K_2SnCl_6 (Boysen, Ihringer, Prandl & Yelon, 1976) and two each in $K_3MO_3F_3$, $Rb_3MO_3F_3$ and $Cs_3MO_3F_3$ (M = Mo, W) (Peraudeau, Ravez, Hagenmuller & Arend, 1978).

The transitions in K_2SnCl_6 are reported to be from space group $Fm3m$ to $P4/mnc$, followed by a lower-temperature transition to $P2_1/n$ (Boysen & Hewat, 1978): the structures of all three phases have been studied. K_2TeBr_6 is monoclinic at room temperature, space group $P2_1/n$ (Brown, 1964), as also is K_2SnBr_6 (Higashi, Syoyama & Osaki, 1979) and K_2TeCl_6 (Henke, 1980). The distorted K_2TeBr_6, K_2SnBr_6 and K_2TeCl_6 structures are expected to undergo transitions to the cubic K_2PtCl_6 type at higher temperatures: the ease with which twinning is initiated at room temperature indicates that these crystals are ferroelastic, as probably are all the distorted K_2PtCl_6 type structures.

The phase transition in $Rb_2KMoO_3F_3$ is from $Fm3m$ to $R3$: in the related structure $K_2PbCu(NO_2)_6$, the symmetry of the high-temperature phase is reported by Cullen & Lingafelter (1971) as $Fm3$, an intermediate phase as $Fmmm$ (Joesten, Takagi & Lenhert, 1977) and the low-temperature phase as triclinic (Noda, Mori & Yamada, 1978). The small distortions found at lower temperatures in these and other isostructural materials suggest that they may also be ferroelastic (see Abrahams, 1971). In the case that the lower-symmetry phases are in addition polar, the possibility arises that a spontaneous polarization may develop: the cubic to lower-symmetry phase transitions hence may be associated with the formation of a coupled spontaneous-strain–spontaneous-polarization.

The average entropy change observed (Ravez, Peraudeau, Arend, Abrahams & Hagenmuller, 1980) at the cubic to rhombohedral transition of six $A_3MO_3F_3$ compounds (A = K, Rb or Cs, M = Mo or W) is 3·9 (3) J mol^{-1} K^{-1}, which compares well with that expected for a change at the phase transition in the number of available orientations corresponding to $R \ln 3/2$, *i.e.* 3·4 J mol^{-1} K^{-1}. Busey *et al.* (1962) find an average entropy change of 5·1 J mol^{-1} K^{-1} at each of the three transitions in K_2ReCl_6, corresponding to $R \ln 2$: the symmetry progression in K_2ReCl_6 has been reported as cubic-to-tetragonal-to-monoclinic

for the three higher-temperature phases (O'Leary & Wheeler, 1970).

A network of diffuse scattering in cubic K$_2$SnCl$_6$ has been identified by Ihringer (1980) as caused by the in-phase rotations of the SnCl$_6^{2-}$ octahedra. Well-exposed precession photographs of Rb$_2$KMoO$_3$F$_3$ crystals at 343 and at 473 K, by contrast, show no evidence of diffuse scattering. Ihringer predicts a proportionality between temperature and the x coordinate of Cl in K$_2$SnCl$_6$: the x coordinate of O/F in Rb$_2$KMoO$_3$F$_3$ remains essentially constant with temperature in the interval measured, see Table 2. The phase transition in these two crystals is hence qualitatively different in character, as expected by the differing low-temperature symmetry. A consequence of the major dissimilarity between the two structures, namely the filling of the 4(b) sites in the $A_2BMO_3F_3$ crystals, is the occupation of all anionic octahedra by a cation, see Fig. 2, with resulting modification of the vibrational distribution in the crystal.

References

ABRAHAMS, S. C. (1971). *Mater. Res. Bull.* **6**, 881–890.

ABRAHAMS, S. C., BERNSTEIN, J. L. & KEVE, E. T. (1971). *J. Appl. Cryst.* **4**, 284–290.

ABRAHAMS, S. C., BERNSTEIN, J. L. & RAVEZ, J. (1981). To be published.

BERNSTEIN, J. L., ABRAHAMS, S. C. & RAVEZ, J. (1979). *Am. Crystallogr. Assoc. Prog. Abstr. Ser.* 2, **7**, 55.

BOYSEN, H. & HEWAT, A. W. (1978). *Acta Cryst.* **B34**, 1412–1418.

BOYSEN, H., IHRINGER, J., PRANDL, W. & YELON, W. (1976). *Solid State Commun.* **20**, 1019–1024.

BROWN, I. D. (1964). *Can. J. Chem.* **42**, 2758–2767.

BUSEY, R. H., DEARMAN, H. H. & BEVAN, R. B. (1962). *J. Phys. Chem.* **66**, 82–89.

BUSING, W. R., JOHNSON, C. K., THIESSEN, W. E. & LEVY, H. A. (1973). *J. Appl. Cryst.* **6**, 309–346.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1973). *J. Appl. Cryst.* **6**, 309–346.

CHAMINADE, J. P. (1979). Private communication.

COPPENS, P. & HAMILTON, W. C. (1970). *Acta Cryst.* A26, 71–83.

CULLEN, D. L. & LINGAFELTER, E. C. (1971). *Inorg. Chem.* **10**, 1264–1268.

DELOUME, J.-P., FAURE, R. & THOMAS-DAVID, G. (1979). *Acta Cryst.* B35, 558–561.

Enraf–Nonius (1979). *CAD-4 Operation Manual.* Delft.

FISH, G. E. (1980). Private communication.

FISH, G. E., RHYNE, J. J., LYNN, J. W. & PATTERSON, H. H. (1980). *Natl Bur. Stand. US Tech. Note* 1117.

GRUNDY, H. D. & BROWN, I. D. (1970). *Can. J. Chem.* **48**, 1151–1154.

HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.

HAMILTON, W. C. & ABRAHAMS, S. C. (1970). *Acta Cryst.* A26, 18–24.

HENKE, H. (1980). *J. Appl. Cryst.* **13**, 305–307.

HIGASHI, T., SYOYAMA, S. & OSAKI, K. (1979). *Acta Cryst.* B35, 144–146.

IHRINGER, J. (1980). *Acta Cryst.* A36, 89–96.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

JOESTEN, M. D., TAKAGI, S. & LENHERT, P. G. (1977). *Inorg. Chem.* **16**, 2680–2685.

LISSALDE, F., ABRAHAMS, S. C. & BERNSTEIN, J. L. (1978). *J. Appl. Cryst.* **11**, 31–34.

NODA, Y., MORI, M. & YAMADA, Y. (1978). *J. Phys. Soc. Jpn*, **45**, 954–966.

O'LEARY, G. P. & WHEELER, R. C. (1970). *Phys. Rev. B*,**1**, 4409–4439.

PAULING. L. (1924). *J. Am. Chem. Soc.* **46**, 2738–2751.

PERAUDEAU, G., RAVEZ, J., HAGENMULLER, P. & AREND, H. (1978). *Solid State Commun.* **27**, 591–593.

RAVEZ, J., PERAUDEAU, G., AREND, H., ABRAHAMS, S. C. & HAGENMULLER, P. (1980). *Ferroelectrics*, **26**, 767–769.

SHANNON, R. D. (1976). *Acta Cryst.* A32, 751–767.

WILLIAMS, R. J., DILLIN, D. R. & MILLIGAN, W. O. (1973). *Acta Cryst.* B29, 1369–1372.