Fig. 2. Stereoscopic drawing of the unit cell. The a axis runs across, the b axis into, the c axis up the page.

The coordination geometry around each Rh atom is essentially square planar with a slight tetrahedral distortion. The two 'RhC$_4$' (least-squares) planes are tipped by 40·2 (2)° (where 0° would be parallel), presumably due to the small 'bite' of the tmi ligand. The torsional blade angles, C(1)—Rh(1)—Rh(2)—C(8), are equal for the two tmh groups opposite each other: 16·6 (3)° for arm A and 16·4 (3)° for arm B; the corresponding angles for arms C and D are smaller, 12·0 (3) and 5·3 (3)°. The average Rh—C bond length of 1·95 (2) Å and the Rh—C≡N angle of 175 (3)° are consistent with the structural data of other Rh-bridge complexes (Mann et $al.$, 1980). The C—C and C—N

bond lengths, and associated angles, within the tmh rings are normal. The CN(1)—CN(6) bond length of 1·25 (1) Å implies a formal C—N double bond, and leads to the formulation of this arm as an aza-1-cyclohexen-2-ide ring. The bond distances and angles within the anion are normal.

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Structure of a Complex between rel-(6R,11S,17R,22S)-6,7,8,9,10,11,17,18,19,20,21,22-Dodecahydro-1,5,12,16,23,26,29-heptaoxa[7³,¹⁴][5.5]orthocyclophane and Magnesium Bis(perchlorate) Monohydrate, $C_{22}H_{38}O_7 \cdot Mg(ClO_4)_2 \cdot H_2O$

By JOHN D. OWEN

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Abstract. $M_r = 655·8$, monoclinic, $P2_1/c$, $a = 17·145$ (4), $b = 20·848$ (4), $c = 16·628$ (5) Å, $\beta = 92·31$ (2)°, $U = 5938$ Å^3, $Z = 8$, $D_m = 1·45$, $D_x = 1·47$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0·71069$ Å, $\mu = 3·07$ cm^{-1}, $F(000) = 2768$, m.p. > 520 K (decomp.), $R = 0·083$ for 2724 observations $[I > 2\sigma(I)]$. There are two molecules in the asymmetric unit, each containing one Mg coordinated to the seven O atoms of the macrobicycle, and to the O of a water molecule. Two perchlorates are H-bonded in different ways to the waters in the two molecules which are otherwise almost identical.
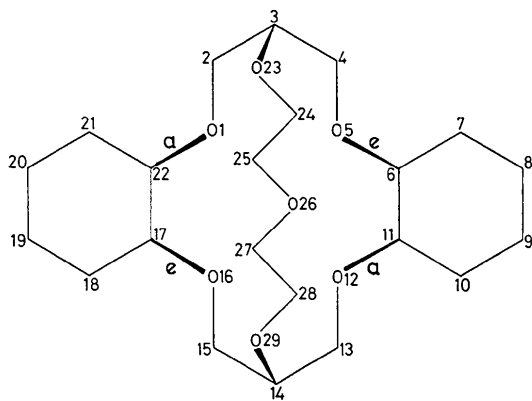
Fig. 1. Molecule of (I) showing the atomic numbering scheme.

Introduction. The macrobicyclic polyether (I) (Fig. 1) named in the title is formed by cyclization of the corresponding diol (Parsons, 1982). Model building indicated that the cavity was the correct size to accept smaller cations such as Li⁺ and Mg²⁺, but too small for the other cations of Groups Ia and IIa. Structure reports on magnesium–polyether complexes are relatively rare (Owen, 1978), so the present work was undertaken to determine the coordination to the cation, and to confirm the isomer of (I).

Experimental. Colourless crystal, $0.28 \times 0.36 \times 0.10$ mm, Enraf–Nonius CAD-4 diffractometer; 18 reflections with $9° < \theta < 14°$ used to refine the unit-cell parameters; 4268 reflections collected in the hkl and $hk\bar{l}$ octants to a maximum 2θ of 36°, ω–2θ scans; two control reflections measured after every 50 reflections, their intensities reduced by ~4% over the collection, and the data were corrected for this; the data were processed to give a total of 4078 unique reflections, of which 2724 had $I > 2\sigma(I)$, and were classed as observed; no absorption correction applied; structure solved using multisolution direct methods (Sheldrick, 1976), and developed by successive Fourier difference maps and least-squares refinements; the numbering scheme for (I) is shown in Fig. 1, and the H atoms were numbered according to the atom to which they are bonded; the H atoms were included in calculated positions (C—H = 0.98 Å) and given isotropic temperature factors 1.2 times that of the C to which they are bonded and allowed to 'ride' on the C atom; the water H atoms were located on a Fourier difference map and allowed to refine independently; with the Mg, water O and perchlorate atoms assigned anisotropic temperature factors, the number of parameters was large so a locally written block-diagonal least-squares program was used (Owen, 1982); the maximum shift/e.s.d. on the final cycle was 0.4, and the final $wR = 0.091$ where $w \propto 1/[\sigma²(F_o) + 0.00117 F_o²]$; the poor agreement is probably due to the disorder in the

perchlorate O atoms which took on unreasonable U_{ij} values; the final ΔF map showed a maximum peak height of 0.57 e Å⁻³ close to the O atoms of perchlorate 2; scattering factors were calculated using an analytical approximation (*International Tables for X-ray Crystallography*, 1974).

Discussion. The final atomic parameters are in Table 1,* and stereopairs (Johnson, 1971) of the two independent molecules are shown in Fig. 2. Bond lengths and torsion angles are given in Table 2; deviations from mean planes and bond angles have been deposited. The two independent molecules are essentially identical, as can be seen from Fig. 2. The Mg atoms are coordinated to eight O atoms, seven from the ligands with Mg—O between 2.12 and 2.58 Å, and the water O atom at 2.05 Å, significantly closer than the others. This spread of Mg—O distances is greater than that found in the Mg(SCN)₂ complex with benzo-15-crown-5 (Owen, 1978) where Mg is seven-coordinated. The longest distances are to O(12) and in

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* Lists of structure factors, anisotropic thermal parameters H-atom parameters, bond angles, deviations of atoms from the mean planes of the five O atoms and details of the H-bonding geometry have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38353 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
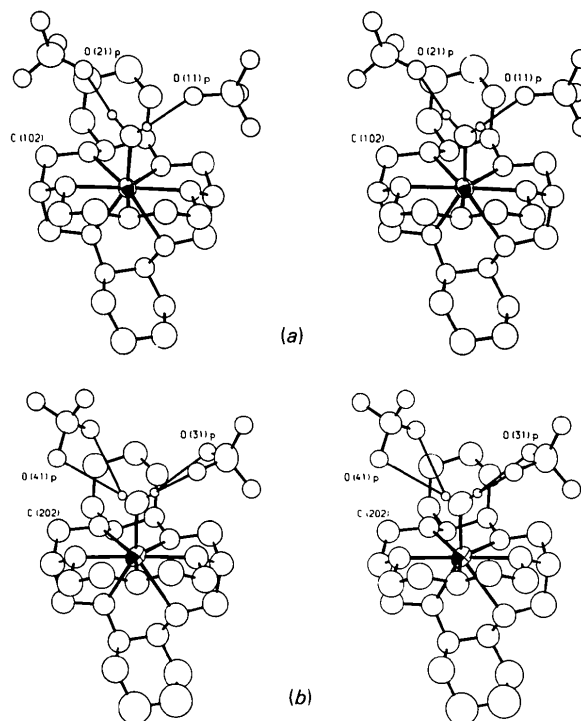

Fig. 2. (a) Molecule (1) and (b) molecule (2) of the complex. 50% thermal ellipsoids are shown, except for the water H and perchlorate O atoms which have radii of 0.1 and 0.2 Å respectively.

Table 1. *Fractional coordinates* ($\times 10^4$) *and* U_{iso}/U_{eq} *values* ($\text{Å}^2 \times 10^3$) *for the non-hydrogen atoms* (w *and* p *denote water and perchlorate, respectively*)

	x	y	z	U_{iso}/U_{eq}		x	y	z	U_{iso}/U_{eq}
Mg(1)	2602(2)	1338(2)	4959(2)	43(2)†	Mg(2)	2257(2)	6415(2)	4729(2)	51(2)†
O(101)	·2534(4)	2322(4)	4392(4)	49(2)	O(201)	1149(4)	5917(4)	4320(5)	52(2)
C(102)	1819(7)	2681(6)	4513(8)	56(4)	C(202)	1038(7)	5299(6)	4679(8)	64(4)
C(103)	1230(7)	2221(6)	4836(7)	53(4)	C(203)	1802(8)	5071(7)	5001(8)	74(4)
C(104)	934(7)	1739(6)	4238(8)	61(4)	C(204)	2385(8)	4926(6)	4378(8)	68(4)
O(105)	1525(4)	1255(3)	4157(4)	47(2)	O(205)	2675(4)	5528(4)	4111(5)	60(3)
C(106)	1268(7)	694(5)	3733(7)	48(4)	C(206)	3336(7)	5483(6)	3612(8)	63(4)
C(107)	828(8)	242(6)	4273(8)	71(4)	C(207)	4081(8)	5355(8)	4131(9)	91(5)
C(108)	668(8)	−399(7)	3843(9)	81(5)	C(208)	4799(11)	5367(9)	3601(11)	133(7)
C(109)	1389(8)	−706(7)	3562(9)	82(5)	C(209)	4820(12)	5996(9)	3147(12)	141(7)
C(110)	1816(7)	−244(6)	3037(7)	59(4)	C(210)	4089(9)	6098(8)	2630(10)	104(6)
C(111)	1982(6)	402(6)	3415(7)	47(4)	C(211)	3343(8)	6067(6)	3139(8)	68(4)
O(112)	2504(4)	348(4)	4121(5)	52(2)	O(212)	3341(5)	6579(4)	3713(5)	71(3)
C(113)	3246(7)	67(6)	3979(8)	70(4)	C(213)	3335(9)	7222(7)	3420(9)	87(5)
C(114)	3845(7)	402(6)	4550(7)	58(4)	C(214)	2797(8)	7622(7)	3906(8)	75(4)
C(115)	4161(7)	1003(6)	4192(8)	61(4)	C(215)	1958(8)	7566(7)	3612(9)	81(5)
O(116)	3493(4)	1425(4)	4114(4)	48(2)	O(216)	1757(4)	6897(4)	3707(5)	53(2)
C(117)	3648(7)	2022(5)	3721(7)	49(4)	C(217)	1015(7)	6708(6)	3349(8)	53(4)
C(118)	4232(8)	2428(6)	4192(8)	67(4)	C(218)	335(7)	7056(6)	3702(8)	65(4)
C(119)	4301(9)	3094(7)	3837(10)	95(5)	C(219)	−448(8)	6771(6)	3386(9)	75(5)
C(120)	3540(9)	3414(7)	3740(10)	94(5)	C(220)	−480(8)	6071(6)	3556(9)	81(5)
C(121)	2951(8)	3012(6)	3279(9)	82(5)	C(221)	188(7)	5727(6)	3190(8)	67(4)
C(122)	2871(7)	2322(6)	3606(7)	53(4)	C(222)	976(7)	5995(5)	3475(7)	47(4)
O(123)	1634(4)	1899(4)	5490(5)	53(2)	O(223)	2096(5)	5569(4)	5517(5)	62(3)
C(124)	1152(8)	1508(7)	5977(8)	72(4)	C(224)	2790(8)	5427(7)	6016(9)	84(5)
C(125)	1671(8)	1084(7)	6455(9)	78(5)	C(225)	3060(9)	6030(6)	6352(9)	82(5)
O(126)	2154(4)	751(4)	5941(5)	56(2)	O(226)	3204(5)	6443(4)	5678(5)	70(3)
C(127)	2737(7)	378(6)	6360(8)	67(4)	C(227)	3406(9)	7071(6)	5915(9)	84(5)
C(128)	3241(8)	73(6)	5759(8)	72(4)	C(228)	3519(8)	7460(7)	5185(8)	84(5)
O(129)	3490(4)	585(4)	5265(4)	52(2)	O(229)	2794(5)	7377(4)	4702(5)	68(3)
O(1)w	3243(4)	1797(4)	5859(5)	57(3)†	O(2)w	1568(5)	6851(4)	5542(5)	76(4)†
Cl(1)	5239(2)	1157(2)	6582(2)	80(2)†	Cl(3)	1665(3)	8592(2)	6023(2)	87(2)†
O(11)p	4602(8)	1503(9)	6706(10)	253(10)†	O(31)p	1157(10)	8258(8)	5515(11)	208(10)†
O(12)p	5443(9)	1238(8)	5775(7)	171(8)†	O(32)p	1924(11)	8126(8)	6550(8)	202(9)†
O(13)p	5059(9)	532(6)	6605(11)	194(9)†	O(33)p	2272(10)	8739(7)	5515(11)	197(9)†
O(14)p	5878(7)	1325(6)	7049(7)	143(6)†	O(34)p	1332(10)	9096(6)	6382(10)	194(9)†
Cl(2)	3029(2)	3576(2)	6416(2)	77(2)†	Cl(4)	−177(2)	6362(2)	6609(2)	69(1)†
O(21)p	3320(15)	3021(5)	6502(9)	262(13)†	O(41)p	473(9)	6002(8)	6648(10)	199(9)†
O(22)p	2312(9)	3599(13)	6245(18)	372(19)†	O(42)p	−177(10)	6570(11)	5871(9)	255(12)†
O(23)p	3350(15)	3800(8)	5734(7)	255(13)†	O(43)p	−267(14)	6846(9)	7077(12)	265(12)†
O(24)p	3245(6)	3983(5)	7054(6)	99(5)†	O(44)p	−812(11)	5998(8)	6725(14)	245(12)†

† $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses and selected torsion angles* (°) (*e.s.d.'s 1–2°*)

	n = 1	n = 2		n = 1	n = 2		n = 1	n = 2
Mg(n)−O(n01)	2·259(8)	2·246(9)	C(n09)−C(n10)	1·508(19)	1·506(25)	C(n27)−C(n28)	1·489(19)	1·479(20)
Mg(n)−O(n05)	2·241(8)	2·247(9)	C(n10)−C(n11)	1·509(17)	1·563(21)	C(n28)−O(n29)	1·423(15)	1·462(16)
Mg(n)−O(n12)	2·491(9)	2·583(10)	C(n11)−O(n12)	1·450(14)	1·432(16)	Cl(1)−O(11)p	1·331(16)	
Mg(n)−O(n16)	2·123(8)	2·125(9)	O(n12)−C(n13)	1·430(15)	1·426(16)	Cl(1)−O(12)p	1·411(13)	
Mg(n)−O(n23)	2·242(9)	2·222(9)	C(n13)−C(n14)	1·538(18)	1·503(20)	Cl(1)−O(13)p	1·340(14)	
Mg(n)−O(n26)	2·204(9)	2·218(9)	C(n14)−C(n15)	1·498(17)	1·505(19)	Cl(1)−O(14)p	1·362(12)	
Mg(n)−O(n29)	2·231(8)	2·208(10)	C(n14)−O(n29)	1·410(14)	1·419(16)	Cl(2)−O(21)p	1·266(15)	
Mg(n)−O(n)w	2·056(9)	2·045(10)	C(n15)−O(n16)	1·446(14)	1·447(16)	Cl(2)−O(22)p	1·251(17)	
O(n01)−C(n02)	1·458(14)	1·437(15)	O(n16)−C(n17)	1·436(13)	1·438(14)	Cl(2)−O(23)p	1·363(17)	
O(n01)−C(n22)	1·450(14)	1·435(14)	C(n17)−C(n18)	1·506(17)	1·511(17)	Cl(2)−O(24)p	1·396(10)	
C(n02)−C(n03)	1·508(17)	1·474(18)	C(n17)−C(n22)	1·477(16)	1·504(16)	Cl(3)−O(31)p		1·379(18)
C(n03)−C(n04)	1·490(17)	1·499(19)	C(n18)−C(n19)	1·515(20)	1·541(18)	Cl(3)−O(32)p		1·370(16)
C(n03)−O(n23)	1·431(14)	1·424(16)	C(n19)−C(n20)	1·469(21)	1·488(19)	Cl(3)−O(33)p		1·400(18)
C(n04)−O(n05)	1·439(14)	1·427(15)	C(n20)−C(n21)	1·500(21)	1·501(19)	Cl(3)−O(34)p		1·347(15)
O(n05)−C(n06)	1·427(13)	1·434(15)	C(n21)−C(n22)	1·548(18)	1·519(17)	Cl(4)−O(41)p		1·344(16)
C(n06)−C(n07)	1·523(18)	1·536(19)	O(n23)−C(n24)	1·434(15)	1·454(16)	Cl(4) O(42)p		1·302(17)
C(n06)−C(n11)	1·484(16)	1·450(18)	C(n24)−C(n25)	1·466(19)	1·444(20)	Cl(4)−O(43)p		1·288(19)
C(n07)−C(n08)	1·533(19)	1·543(23)	C(n25)−O(n26)	1·398(16)	1·441(16)	Cl(4)−O(44)p		1·346(18)
C(n08)−C(n09)	1·485(20)	1·515(27)	O(n26)−C(n27)	1·425(15)	1·407(16)			

	n = 1	n = 2		n = 1	n = 2		n = 1	n = 2
C(n22)−O(n01)−C(n02)−C(n03)	125	118	C(n06)−C(n07)−C(n08)−C(n09)	55	55	O(n16)−C(n17)−C(n18)−C(n19)	−172	−172
C(n17)−C(n22)−O(n01)−C(n02)	170	170	C(n07)−C(n08)−C(n09)−C(n10)	−55	−58	C(n22)−C(n17)−C(n18)−C(n19)	−53	−54
C(n21)−C(n22)−O(n01)−C(n02)	50	49	C(n08)−C(n09)−C(n10)−C(n11)	53	57	O(n16)−C(n17)−C(n22)−O(n01)	53	52
O(n01)−C(n02)−C(n03)−C(n04)	−70	−67	C(n09)−C(n10)−C(n11)−C(n06)	−52	−52	O(n16)−C(n17)−C(n22)−C(n21)	173	175
O(n01)−C(n02)−C(n03)−O(n23)	50	54	C(n09)−C(n10)−C(n11)−O(n12)	64	64	O(n01)−C(n22)−C(n17)−C(n18)	−70	−71
C(n02)−C(n03)−C(n04)−O(n05)	77	77	C(n06)−C(n11)−O(n12)−C(n13)	179	−179	C(n21)−C(n22)−C(n17)−C(n18)	50	52
O(n23)−C(n03)−C(n04)−O(n05)	−40	−42	C(n10)−C(n11)−O(n12)−C(n13)	59	61	C(n17)−C(n18)−C(n19)−C(n20)	54	56
C(n02)−C(n03)−O(n23)−C(n24)	170	171	C(n11)−O(n12)−C(n13)−C(n14)	146	140	C(n18)−C(n19)−C(n20)−C(n21)	−53	−57
C(n04)−C(n03)−O(n23)−C(n24)	−67	−65	O(n12)−C(n13)−C(n14)−C(n15)	−87	−84	C(n19)−C(n20)−C(n21)−C(n22)	52	57
C(n03)−C(n04)−O(n05)−C(n06)	168	170	O(n12)−C(n13)−C(n14)−O(n29)	32	31	C(n20)−C(n21)−C(n22)−O(n01)	68	64
C(n04)−O(n05)−C(n06)−C(n07)	−80	−78	C(n13)−C(n14)−C(n15)−O(n16)	65	62	C(n20)−C(n21)−C(n22)−C(n17)	−50	−53
C(n04)−O(n05)−C(n06)−C(n11)	156	157	O(n29)−C(n14)−C(n15)−O(n16)	−56	−56	C(n03)−O(n23)−C(n24)−C(n25)	164	169
O(n05)−C(n06)−C(n07)−C(n08)	−171	−175	C(n13)−C(n14)−O(n29)−C(n28)	66	69	O(n23)−C(n24)−C(n25)−O(n26)	−54	−58
C(n11)−C(n06)−C(n07)−C(n08)	−53	−55	C(n28)−O(n29)−C(n14)−C(n15)	−172	−171	C(n24)−C(n25)−O(n26)−C(n27)	174	175
O(n05)−C(n06)−C(n11)−C(n10)	173	175	C(n14)−C(n15)−O(n16)−C(n17)	−176	−171	C(n25)−O(n26)−C(n27)−C(n28)	−177	−178
O(n05)−C(n06)−C(n11)−O(n12)	53	55	C(n15)−O(n16)−C(n17)−C(n18)	−64	−61	O(n26)−C(n27)−C(n28)−O(n29)	53	54
C(n10)−C(n11)−C(n06)−C(n07)	51	52	C(n15)−O(n16)−C(n17)−C(n22)	172	176	C(n27)−C(n28)−O(n29)−C(n14)	−168	−171
C(n07)−C(n06)−C(n11)−O(n12)	−70	−68						

both molecules the Mg—O(axial) distances are longer than the Mg—O(equatorial) distances to the O atoms on the same cyclohexane ring. O(5) and O(12) are on one side of an approximate plane containing O(1), O(16), O(23), O(26), O(29), and the water O is on the other side. This compares with the $Mg(SCN)_2$ complex with benzo-15-crown-5 which contains the five ether O atoms in an approximate plane, and an N from the anions on each side. The deviations of the five O atoms and of the Mg atom from the plane are all slightly larger in the present structure.

This shape of the molecule with five O atoms in a plane and two below is similar to those found in the analogous molecule with benzene rings instead of the cyclohexanes here (Herbert & Truter, 1980; Owen, 1981).

Bond lengths and angles in the ligand (I) are as expected for this class of compound. The torsion angles are close to the expected ±*gauche* and *trans*, the largest deviations being O(5)—C(4)—C(3)—O(23), O(12)—C(13)—C(14)—O(29) and C(22)—O(1)—C(2)—C(3) (see Table 2).

Each water molecule is H-bonded to two perchlorate O atoms. Though the exact positions of the perchlorate O atoms must be in doubt in view of their large thermal parameters which indicate possible statistical disorder, the two molecules do seem to differ in their H-bonding schemes.* Molecule (1) shows fairly strong convention-

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* Hydrogen-bonding parameters have been deposited. See deposition footnote.

al single H bonds (O⋯O distances ~2·75 Å, angles at H ~173°). Molecule (2), however, has the perchlorates in different orientations (Fig. 2) and the H bonding is much weaker (O⋯O distances ~3·15 Å, angles at H again ~173°). There may be further interactions with a second O atom, though the geometry indicates this to be very weak.

The isomer of the ligand (I) is confirmed as that shown in Fig. 1, with both H atoms at the cyclohexane ring junctions on the same side of the molecule, and opposite to the O(23)—O(29) bridge.

There are no close contacts between molecules of note, and so the crystals are made up of equal numbers of discrete left- and right-handed molecules.

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Refinement of Potassium Tetrafluorophthalate, $2K^+.C_8F_4O_4^{2-}$

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Abstract. KTFP, $M_r = 314·3$, monoclinic, $P2/c$, $a = 7·133$ (2), $b = 10·825$ (3), $c = 7·114$ (2) Å, $\beta = 111·82$ (1)°, $V = 510·0$ Å^3, $Z = 2$, $D_x = 2·047$ Mgm^{-3}, Ni-filtered Cu K radiation, $\lambda K\alpha_1 = 1·54051$ Å, $\mu(Cu\ K\alpha) = 8·86$ mm^{-1}, $F(000) = 308$, 294 K, $R = 0·034$ for 836 reflections, starting with the positional parameters reported by Griffin, Yeung, LaPrade & Waugh [*J. Chem. Phys.* (1973), **59**, 777–783]. The average C—F intramolecular distance is 1·339 (3) Å, while the average intra- and shortest intermolecular F⋯F distances are 2·702 (3) and 2·713 (2) Å, respectively. This refinement of KTFP corroborated the original choice of space group (in which the molecules are constrained to lie on twofold rotation axes) rather than the acentric space group Pc. The K^+ ion is coordinated by one fluorine and six oxygen atoms; two of the oxygen atoms, from the same carboxyl group, are symmetrically arranged opposite the fluorine atom so as to form a pseudo-octahedral coordination.