

Fig. 1. Projection of the structure along the *a* axis.

arrangement along the *a* axis. The distribution of anionic groups is very regular; Te(OH)<sub>6</sub> groups are located in planes *z* = 0 and 0.5, and P<sub>3</sub>O<sub>9</sub> anions in planes *z* = 0.25 and 0.75. The cohesions between these planes are assumed by the hydrogen bonds and the CsO<sub>*n*</sub> coordination polyhedra, in a three-dimensional way. Each Te(OH)<sub>6</sub> group is linked to six different P<sub>3</sub>O<sub>9</sub> anions. The H atoms of the water molecules connect two neighbouring phosphoric anions. The interatomic distances and bond angles found for the P<sub>3</sub>O<sub>9</sub> groups (Table 2) are those generally encountered in inorganic

cyclo-tri-phosphates. P—O mean distances are 1.538, 1.543 and 1.541 Å in P(1)O<sub>4</sub>, P(2)O<sub>4</sub> and P(3)O<sub>4</sub> tetrahedra respectively, the general mean being 1.543 Å for these compounds.

The TeO<sub>6</sub> group forms a very regular octahedron with 1.905 < Te—O < 1.915 Å and 86.9 < O—Te—O < 93.0°. One can remark, considering the atomic coordinates, that the Te atom is practically a non-crystallographic inversion centre for the TeO<sub>6</sub> octahedron.

The Cs—O distances in the different caesium coordination polyhedra are given in Table 2. Within a range of 3.5 Å Cs(1) has eight neighbours, Cs(2) and Cs(3) only seven.

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## Structure of Potassium Magnesium Hydrogenbiscarbonate Tetrahydrate\*

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**Abstract.** KMgH(CO<sub>3</sub>)<sub>2</sub>·4H<sub>2</sub>O, *M<sub>r</sub>* = 256.5, triclinic, *P* $\bar{1}$ , *Z* = 1,  $\lambda(\text{Mo } K\alpha)$  = 0.71069 Å,  $\mu$  = 0.725 mm<sup>-1</sup>, *F*(000) = 132: (1) *T* = 143 K, *a* = 6.697 (1), *b* = 7.267 (1), *c* = 5.336 (1) Å,  $\alpha$  = 107.04 (2),  $\beta$  = 107.83 (2),  $\gamma$  = 59.88 (2)°, *V* = 210.6 Å<sup>3</sup>, *D<sub>x</sub>* =

2.02 Mg m<sup>-3</sup>, *R* = 0.0230, 2616 unique reflections; (2) *T* = 295 K, *a* = 6.7125 (7), *b* = 7.3439 (9), *c* = 5.3677 (8) Å,  $\alpha$  = 107.267 (8),  $\beta$  = 107.965 (7),  $\gamma$  = 59.546 (8)°, *V* = 213.77 Å<sup>3</sup>, *D<sub>x</sub>* = 1.99 Mg m<sup>-3</sup>, *R* = 0.0331, 1215 unique reflections. Two carbonate groups are linked by a hydrogen bond to form a slightly twisted anion, H(CO<sub>3</sub>)<sub>2</sub><sup>3-</sup>, with an O...O distance of 2.447 (1) Å at 143 K and 2.461 (3) Å at 295 K; the distance between the planes of the carbonate groups is 0.512 (1) Å at 143 K and 0.503 (2) Å at 295 K.

\* Hydrogen Bond Studies. 154. Part 153: Olovsson & Jaskólski (1986).

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**Introduction.** This work forms part of a project involving studies of short hydrogen bonds in compounds containing carbonate or carboxylate groups. In particular, attention is focused on the influence of the environment on the electron and proton distribution in these bonds. In an early structure determination of the present compound by Stephan, MacGillavry & Koch (1972), a very short O...O distance was found (2.42 Å at room temperature) but difference Fourier syntheses gave no indication of the hydrogen positions. They suggested that the H atoms might not conform to the space group  $P\bar{1}$ . The structure has now been redetermined as an initial step in the project to clarify this point.

**Experimental.** Crystals were obtained according to the Berzelius method (Mellor, 1923), by adding one volume of an aqueous solution of magnesium nitrate, 20% (w), drop by drop, to seven volumes of potassium hydrogen-carbonate, 20% (w), at room temperature followed by slow evaporation. Several crystals had to be examined by polarizing microscope and Weissenberg technique before one was found which was not twinned. Cell parameters at room temperature determined by powder photograph taken with a Guinier-Hägg focusing camera, Ni filter,  $\lambda(\text{Cu K}\alpha) = 1.5418 \text{ \AA}$ , Si ( $a = 5.4309 \text{ \AA}$ ) used as internal standard. The  $\alpha$  and  $\beta$  angles are respectively 0.91 and 0.95° less than those from earlier work (Stephan, MacGillavry & Koch, 1972); the largest difference is  $30\sigma(\text{comb.})$ .

Details of the data collection and refinement are given in Table 1. The structure was solved from room-temperature data using Patterson and Fourier syntheses. All H atoms located from difference map. At low temperature, starting values from 295 K. Scattering factors,  $f'$  and  $f''$  from *International Tables for X-ray Crystallography* (1974). Isotropic extinction correction according to Becker & Coppens (1974*a,b*, 1975) formalism. Gaussian and Lorentzian distribution of mosaic blocks gave approximately the same  $R$  values. All programs described by Lundgren (1982). In the last cycle of refinement, one scale factor, 30 positional parameters, 48 anisotropic temperature factors, five isotropic temperature factors (hydrogen) and one isotropic extinction factor. The final positional parameters are given in Table 2.\*

**Discussion.** Interatomic distances and bond angles are listed in Table 3. They agree with earlier results within  $2\sigma(\text{comb.})$  (Stephan, MacGillavry & Koch, 1972), except for the shortest hydrogen bond. Fig. 1 displays

Table 1. *Details of the data collection and structure refinement*

Temperature (K)	143	295
Diffractometer	Enraf-Nonius	Stoe-Philips
Wavelength (Å), graphite monochromator	CAD-4	four circle
Crystal volume (mm <sup>3</sup> )	0.71069	0.71069
Number and range (°) of reflections in cell-parameters calculation	$1.53 \times 10^{-2}$	$6.58 \times 10^{-3}$
Transmission factor (minimum and maximum values)	25	20
Max. value of $(\sin\theta)/\lambda$ (Å <sup>-1</sup> )	$10 \leq \theta \leq 15$	$10 \leq \theta \leq 15$
Range of $h$	0.812, 0.889	0.812, 0.950
$k$	0.953	0.714
$l$	-12, 12	0, 9
Number of standard reflections and their variation with time (%)	-10, 13	-8, 10
	-11, 9	-7, 7
Number of reflections measured, $\omega$ -2 $\theta$ step scan mode	5	3
Number of unique reflections	4	7
$R(\text{intensity}) = \sum  I - \langle I \rangle  / \sum I$	4371	1329
Number of unobserved reflections, $I < 3\sigma$	2616	1215
$R = \sum  F_o - F_c  / \sum  F_o $	0.020	0.039
$R = \sum  F_o - F_c  / \sum  F_o $	161	156
$wR = [ \sum w(F_o^2 - F_c^2)^2 / \sum w F_o^4 ]^{1/2}$	0.230	0.0331
$S = [ \sum w(F_o^2 - F_c^2)^2 / (n - p) ]^{1/2}$	0.0392	0.0449
Ratio of maximum least-squares shift to e.s.d. in final refinement cycle $(\Delta/\sigma)_{\text{max}}$	0.014	0.031
Minimum and maximum heights in difference map (e Å <sup>-3</sup> )	0.0759	0.0904
Isotropic extinction coefficient, Gaussian mosaic-spread distribution, Type I	1.96	1.57
Maximum extinction correction ( $\gamma$ ), where $F^2(\text{corr.}) = \gamma F^2(\text{uncorr.})$	0.005	0.019
	-0.26, 0.45	-0.32, 0.34
	$0.287 \times 10^4$	$0.828 \times 10^4$
	1.49	1.84

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>)*

The upper row refers to the 143 K data, the lower row to the 295 K data. For non-H atoms,

$$\langle u^2 \rangle = \frac{1}{3} (1/2\pi^2) \sum_i \sum_j \beta_{ij} (\mathbf{a}_i \cdot \mathbf{a}_j).$$

	$x$	$y$	$z$	$\langle u^2 \rangle \times 10^2$
K	0.00000	0.00000	0.00000	1.51 (1)
	0.00000	0.00000	0.00000	3.06 (2)
Mg	0.00000	0.50000	0.50000	0.69 (1)
	0.00000	0.50000	0.50000	1.39 (3)
O1	0.01750 (8)	0.76440 (7)	0.46275 (10)	1.11 (1)
	0.01796 (27)	0.76145 (23)	0.46025 (32)	2.27 (4)
O2	0.76303 (8)	0.50848 (7)	0.13598 (9)	1.11 (1)
	0.76283 (26)	0.50808 (23)	0.13759 (27)	2.21 (4)
O3	0.26907 (7)	0.29703 (7)	0.29454 (9)	1.03 (1)
	0.26926 (23)	0.29539 (22)	0.29597 (25)	2.07 (4)
O4	0.59682 (7)	0.24438 (7)	0.60899 (9)	1.20 (1)
	0.59691 (24)	0.24362 (24)	0.60656 (26)	2.36 (4)
O5	0.62108 (7)	0.05402 (7)	0.20039 (9)	1.04 (1)
	0.62034 (23)	0.05479 (22)	0.20124 (26)	2.10 (4)
C	0.49156 (9)	0.20294 (8)	0.37448 (11)	0.79 (1)
	0.49123 (31)	0.20274 (28)	0.37462 (35)	1.55 (5)
H1	0.130 (2)	0.760 (2)	0.421 (2)	2.5 (3)
	0.156 (7)	0.741 (5)	0.419 (7)	7.3 (12)
H2	-0.097 (2)	0.856 (2)	0.385 (2)	2.4 (3)
	-0.090 (5)	0.846 (4)	0.380 (5)	3.5 (7)
H3	0.759 (2)	0.568 (2)	0.023 (3)	4.0 (4)
	0.749 (7)	0.566 (6)	0.006 (8)	8.7 (13)
H4	0.616 (2)	0.597 (2)	0.190 (3)	2.9 (3)
	0.625 (7)	0.591 (5)	0.181 (7)	6.2 (10)
H5	0.500	0.000	0.000	6.0 (7)
	0.500	0.000	0.000	9.0 (18)

the unit cell. K<sup>+</sup> has twelve O neighbours in a nearly close-packed arrangement. Six O atoms around Mg<sup>2+</sup> form an octahedral environment [see Fig. 1 in Stephan, MacGillavry & Koch (1972)]. In the water molecules, the apparent O-H distances (0.8–0.9 Å) are reasonable for an X-ray study, *i.e.* 0.1–0.2 Å

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44798 (73 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Interatomic distances (Å) and angles (°)*

The upper row refers to 143 K, the lower row to 295 K.

## (a) Potassium ion

K—O1	2.879 (1)	K—O3	3.249 (1)
	2.910 (2)		3.264 (1)
K—O1'	3.338 (1)	K—O4	2.950 (1)
	3.370 (2)		2.965 (1)
K—O2	3.154 (1)	K—O5	2.854 (1)
	3.173 (2)		2.868 (1)
O1—K—O1'	61.94 (2)	O1'—K—O5	50.93 (2)
	62.72 (5)		50.77 (4)
O1—K—O2	56.88 (2)	O2—K—O3	53.58 (2)
	56.59 (4)		53.30 (4)
O1—K—O3	55.60 (2)	O2—K—O4	61.93 (2)
	55.27 (4)		62.20 (4)
O1—K—O4	67.88 (3)	O2—K—O5	83.14 (2)
	67.72 (4)		83.45 (4)
O1—K—O5	66.85 (2)	O3—K—O4	73.80 (2)
	67.97 (4)		73.92 (4)
O1'—K—O2	66.03 (2)	O3—K—O5	59.83 (2)
	65.54 (4)		59.94 (4)
O1'—K—O3	72.75 (2)	O4—K—O5	79.97 (2)
	72.37 (4)		80.58 (4)
O1'—K—O4	50.21 (2)		
	49.91 (4)		

## (b) Magnesium ion

Mg—O1	2.052 (1)	O1—Mg—O2	88.17 (3)
	2.059 (1)		88.29 (6)
Mg—O2	2.092 (1)	O1—Mg—O3	88.98 (2)
	2.094 (1)		88.95 (6)
Mg—O3	2.053 (1)	O2—Mg—O3	88.26 (3)
	2.053 (1)		88.28 (5)

## (c) Hydrogenbiscarbonate ion, water molecules and hydrogen bonds

C—O3	1.276 (1)	O2—H3	0.83 (2)
	1.271 (2)		0.89 (4)
C—O4	1.267 (1)	O2—H4	0.95 (1)
	1.262 (2)		0.87 (4)
C—O5	1.322 (1)	O1...O4	2.691 (1)
	1.319 (2)		2.698 (2)
O5...H5	1.224 (1)	O1...O5	2.698 (1)
	1.231 (1)		2.713 (2)
O5...O5'	2.447 (1)	O2...O3	2.935 (1)
	2.461 (3)		2.986 (2)
O1—H1	0.83 (1)	O2...O4	2.667 (1)
	0.95 (4)		2.672 (2)
O1—H2	0.81 (1)		
	0.78 (3)		
O3—C—O4	124.31 (5)	H3—O2—H4	105. (1)
	124.55 (17)		101. (3)
O3—C—O5	117.86 (5)	O1—H1...O4	170. (1)
	117.77 (16)		167. (3)
O4—C—O5	117.83 (5)	O1—H2...O5	175. (1)
	117.69 (16)		174. (3)
C—O5—H5	111.47 (4)	O2—H3...O3	174. (1)
	111.99 (12)		176. (4)
H1—O1—H2	104. (1)	O2—H4...O4	162. (1)
	108. (3)		159. (3)

shorter than the true internuclear distances. Oxygen O2 has a tetrahedral coordination. On the other hand, oxygen O1 has an unusual coordination with five neighbours; this coordination number also occurs in another potassium compound,  $\text{KH}(\text{HCOO})_2$  (Hermansson, Tellgren & Lehmann, 1983). In Fig. 2 is shown the coordination of the water molecules.

The anion may be described as  $\text{H}(\text{CO}_3)_2^{3-}$ , where two carbonate groups are linked by a hydrogen bond with

an O...O distance of 2.447 (1) Å at 143 K and 2.461 (3) Å at room temperature. This anion forms a three-dimensional network of hydrogen bonds with water molecules, as shown in Fig. 1. The atoms in the carbonate group deviate by less than 0.0040 (6) Å at 143 K and 0.004 (2) Å at room temperature from a least-squares plane through the atoms.

Including all atoms, except H5, difference Fourier maps were calculated in order to study the behaviour of H5, the hydrogen atom in the short hydrogen bond and located at a centre of symmetry. The difference map calculated from low-temperature data (Fig. 3a) gives only one peak, a little elongated, but its maximum located at the centre of symmetry. However, at room temperature (Fig. 3b) there are two peaks separated by 0.71 Å and which slightly deviate from the straight line between O5 and O5'. The presence of two peaks may be an indication that H5 is disordered at room temperature. These double peaks closely resemble those found in the crystallographically symmetric O...O bond in potassium hydrogen *meso*-tartrate (Kroon & Kanters, 1972) as well as in hydrazinium hydrogen oxalate (Thomas, 1973). The neutron diffraction study on potassium hydrogen *meso*-tartrate (Currie, Speakman, Kanters & Kroon, 1975) supports some

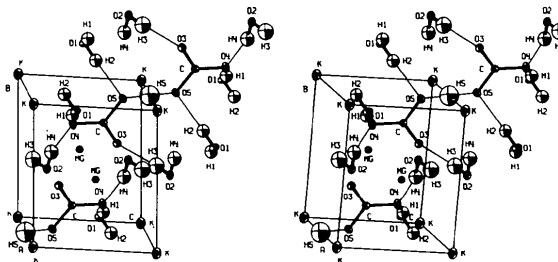


Fig. 1. The unit cell and the network of hydrogen bonds involving the anion,  $\text{H}(\text{CO}_3)_2^{3-}$ , and water molecules. Here and in the figure which follows the thermal vibration ellipsoids are drawn to include 50% probability (low-temperature data).

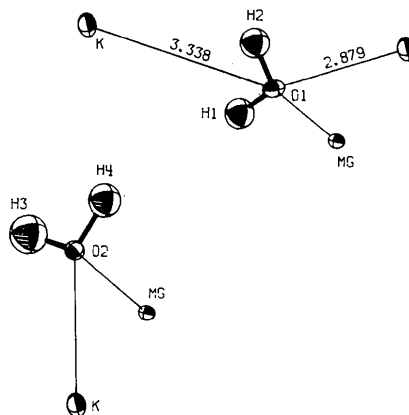


Fig. 2. The coordination of metal ions around the water molecules in  $\text{KMgH}(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$ .

form of proton disorder but with a much shorter distance between the two alternative locations of the proton nucleus (in an X-ray study the electron density maxima associated with the two alternative nuclear positions of H5 will be shifted towards the oxygen atoms leading to a more pronounced double maximum in this case). The most likely conclusion from this work is that if any disorder exists at room temperature, the distance between the two nuclear sites shall be less than 0.71 Å. At low temperature the H atom appears to be located at the centre, but with a very pronounced vibration along the direction of the O...O bond.

Considering the anion separately, with a centre of symmetry at the middle of O5...O5', the two carbonate groups in  $\text{H}(\text{CO}_3)_2^{3-}$  must be parallel. The anion can then have any conformation between the following extremes: (1) O3, O4 and C in a plane perpendicular to the plane defined by C, O5, O5' and C'; (2) all atoms of

the anion in the same plane. In the first case the carbonate planes are maximally displaced with respect to each other (2.280 Å). Least-squares-plane calculations show that the conformation of  $\text{H}(\text{CO}_3)_2^{3-}$  in the crystal is closer to the second. The angle between the O3, C, O4 plane and the C, O5, O5', C' plane is 13.1° at 143 K and 12.9° at 295 K. The distance between the planes of two carbonate groups is then 0.512 (1) Å at low temperature and 0.503 (2) Å at room temperature.

Few structures of hydrogencarbonates are known. In  $\text{NaHCO}_3$  (Sass & Scheurman, 1962; Sharma, 1965), the anions,  $\text{HCO}_3^-$ , form an infinite planar chain. In guanidinium bicarbonate,  $\text{C}(\text{NH}_2)_3\text{HCO}_3$  (Baldwin, Denner, Egan & Markwell, 1986), and in  $\text{KHCO}_3$  (Thomas, Tellgren & Olovsson, 1974), the anion is a planar cyclic dimer,  $(\text{HCO}_3)_2^{2-}$ . In the last compound, neutron studies clearly showed that the H atom is dynamically disordered in the protonated as well as in the deuterated compound with different fractional occupations; their results were recently confirmed by NMR single-crystal studies on  $\text{KDCO}_3$  (Benz, Haebleren & Tegenfeldt, 1986). The feature common to all these compounds is the type of hydrogen bonding: asymmetric and with O...O distance around 2.6 Å. In  $\text{Na}_2\text{CO}_3 \cdot \text{NaHCO}_3 \cdot 2\text{H}_2\text{O}$  (Choi & Mighell, 1982), the anion is identical to that found in the present work,  $\text{H}(\text{CO}_3)_2^{3-}$ , with a similar conformation. However, the network of hydrogen bonds with water molecules is approximately planar. From the analysis of the thermal-ellipsoid orientation in relation to the O-H bond (neutron data), they concluded that the H atom in the symmetric hydrogen bond is dynamically disordered between two sites separated by 0.211 Å. A careful investigation of the proton distribution in hydrogencarbonates has only been carried out in few cases (e.g. Thomas, Tellgren & Olovsson, 1974). The present study will be followed by a structure determination of  $\text{KNiH}(\text{CO}_3)_2 \cdot 4\text{H}_2\text{O}$ , which is in progress at our institute.

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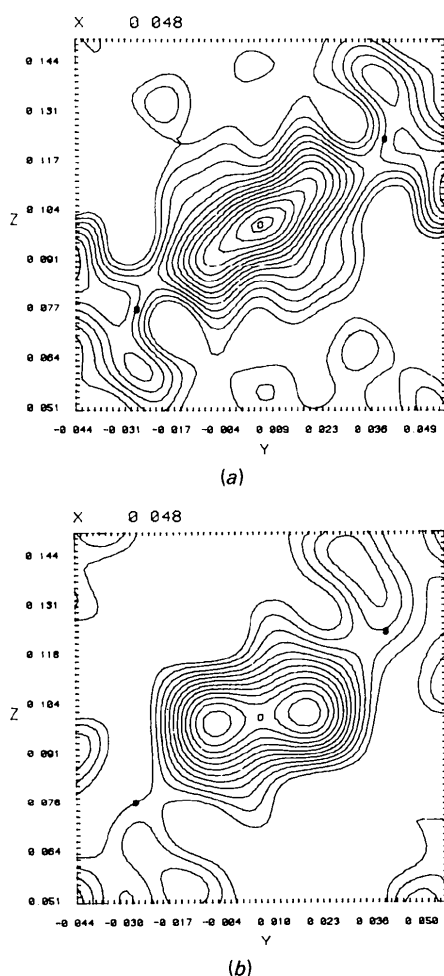


Fig. 3. The difference Fourier maps through O5...O5' bond, without H5. The positions of O5 and O5' are marked with filled circles. The inversion centre is represented by an open circle. Levels at  $0.05 \text{ e} \text{ \AA}^{-3}$ : (a) 143 K; (b) 295 K. The coordinates are for a transformed unit cell.

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## Structure of a Cubic Sodium Strontium Magnesium Tricarbonatodioxouranate(VI) Hydrate

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**Abstract.**  $\text{Na}_{0.87}\text{Sr}_{1.40}\text{Mg}_{0.17}[\text{UO}_2(\text{CO}_3)_3]_4 \cdot 66\text{H}_2\text{O}$ ,  $M_r = 680.7$ , cubic,  $Pa\bar{3}$ ,  $a = 20.290(3) \text{ \AA}$ ,  $V = 8353(4) \text{ \AA}^3$ ,  $Z = 24$ ,  $D_x = 3.25 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71069 \text{ \AA}$ ,  $\mu = 163 \text{ cm}^{-1}$ ,  $F(000) = 7425$ ,  $T = 295 \text{ K}$ , final  $R = 0.041$  for 1588 observed reflections. Five different cation coordination polyhedra and a  $[\text{UO}_2(\text{CO}_3)_3]$  unit build up a framework with the structural chemical formula  $\text{Na}^{[6]}(\text{Na}, \square)_2^{[7]}\text{Sr}_8^{[8]}(\text{Sr}, \text{Na}, \square)_6^{[8]}\text{Mg}^{[6]}\text{[U}^{[2+6]}\text{O}_2(\text{CO}_3)_3]_6 \cdot 28\text{H}_2\text{O}$ . Disorder in cation site occupation affects to some extent also the rest of the structure.

**Introduction.** In a study of uranyl carbonate minerals and related synthetic compounds, the preparation of monoclinic  $\text{SrMg}[\text{UO}_2(\text{CO}_3)_3] \cdot 12\text{H}_2\text{O}$  (Mereiter, 1986a) was attempted from solutions which also contained Na. Instead of the expected prismatic crystals large isometric crystals of a novel uranyl carbonate were obtained. The structure of this salt is reported here.

**Experimental.** Yellow cubes with corners truncated by small  $\{111\}$  faces, size  $\leq 5 \text{ mm}$ , stable in air, grown by room-temperature evaporation of solutions made from 3.18 g  $\text{Na}_2\text{CO}_3$ , 5.02 g  $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ , 1.06 g  $\text{Sr}(\text{NO}_3)_2$  and 1.02 g  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  in 70 g  $\text{H}_2\text{O}$ . Bright yellow-green fluorescence, optically isotropic to weakly birefringent with sector zoning, refractive index  $n$

$= 1.559$  at  $\lambda = 589 \text{ nm}$ . Optical anisotropy is probably connected to the observed disorder of two cation sites (see below). Chemical analysis gave on average 4%  $\text{Na}_2\text{O}$ , 21.2%  $\text{SrO}$ , 0.9%  $\text{MgO}$ , 42.2%  $\text{UO}_3$ , 19.5%  $\text{CO}_2$  and 12.1%  $\text{H}_2\text{O}$ , sum 99.9%, and evidence for reciprocal variations in Na and Sr content; calculated values for the chemical formula given in the *Abstract* are 3.95, 21.31, 0.99, 42.02, 19.39 and 12.35%, respectively. Preliminary cell and space-group information from X-ray powder patterns and precession photographs. Further work on a Siemens AED2 diffractometer using graphite-monochromatized  $\text{Mo K}\alpha$  radiation. Unit-cell dimensions from 24 reflections,  $16 \leq \theta \leq 18^\circ$ . Data collected with a crystal sphere of 0.18 mm diameter,  $\theta$ - $2\theta$  scans, scan width  $1.08 + 0.35\tan\theta$ , three standard reflections with  $\pm 2\%$  variation. Two sets each of 2706 independent reflections measured in two different octants of the reciprocal lattice,  $\theta$ : 2 to  $25^\circ$ ,  $h$ :  $-24$  to  $24$ ,  $k$ : 0 to  $24$ ,  $l$ : 0 to 18. Spherical absorption correction with transmission factors from 0.13 to 0.15; data merged to 1588 independent reflections with  $F_o \geq 4\sigma(F_o)$  which were subsequently used,  $R_{\text{merge}} = 0.033$  for these reflections.

The structure was solved by direct and Fourier methods in space group  $Pa\bar{3}$ , which is unambiguously defined by the observed Laue symmetry and systematic extinctions. H atoms could not be located. Structure refinement with anisotropic temperature factors for all