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Crystal Chemical Studies of the 5*f*-Series of Elements. III. A Study of the Disorder in the Crystal Structure of Anhydrous Uranyl Fluoride

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The ideal structure of UO_2F_2 is rhombohedral with one molecule per unit cell. The unit rhombohedron has dimensions $a=5{\cdot}755\pm0{\cdot}003$ kX., $\alpha=42^\circ\,47'\pm3'$. The space group is $R\bar{3}m$ and the atomic positions are: 1 U in (0, 0, 0), 2 O in $\pm(u, u, u)$ with $u=0{\cdot}122$, 2 F in $\pm(v, v, v)$ with $v=0{\cdot}294$.

Each uranium atom is bonded to two oxygen atoms with U–O = 1·91 A. and to six fluorine atoms with U–F = 2·50 A. The structure is of layer-structure type. Planes of uranium atoms are 5·22 A. apart and the axis of the uranyl group is normal to these planes. The fluorine atoms are 0·61 A. above and below the plane containing the uranium atoms. Weak O–O and O–F bonds hold the layers together.

All actual samples of UO_2F_2 are found to show stacking disorder, the order increasing with the heat treatment. The stacking disorder gives rise to anomalous features in the diffraction pattern. It is shown that the theory of X-ray diffraction in crystals with stacking disorder is capable of explaining all observed anomalies.

1. Introduction

The crystal structure of anhydrous uranyl fluoride, UO_2F_2, was investigated as part of the extensive crystal chemical studies of uranium compounds which the writer carried out within the Manhattan Project during the years 1943–6. Most of the results given in this paper were presented in a report issued within the Manhattan Project in January 1944.

A great many uranyl fluoride preparations of various origins were examined. These preparations were all in powder form and so finely divided that single crystals could not be isolated.

The anhydrous compound is hygroscopic. Some of the samples were obtained by evaporating a uranyl fluoride solution to dryness at 120° C., but most of the samples had been prepared by methods of 'dry chemistry'.

Except for minor but significant differences which will be discussed in detail, all investigated samples of anhydrous uranyl fluoride gave the same X-ray diffraction pattern.

2. The ideal structure of UO_2F_2

All X-ray observations were made with Cu *K* radiation filtered through nickel foil. Powder photographs were taken of all samples, using cameras of 9 or 19 cm. diameter and cylindrical specimens. The X-ray diffraction patterns of a number of the samples were also investigated with the aid of the Norelco Spectrometer.

A characteristic feature of the diffraction patterns is the co-existence of sharp and diffuse diffraction peaks. The intensity distribution in the diffuse peaks shows considerable variation from one sample to the next. It will be shown in a later section of this paper that the diffuseness of some of the diffraction peaks is due to structural disorder, and that the detailed intensity distribution can be quantitatively explained.

Table 1 shows the diffraction lines as observed in a powder photograph of a well heat-treated sample of UO_2F_2, and Table 2 gives the spectrometer data for the same sample. It is indicated in the second column of Table 1 and in the third column of Table 2 whether the diffraction peak is sharp (*S*) or diffuse (*D*).

Table 1. *Data from powder photograph of* UO_2F_2

Int.	Sharp or diffuse	$\sin^2\theta$ obs.	$H_1H_2H_3$	$\sin^2\theta$ calc.
ms	*S*	0·0218	003	0·0217
m	*D*	0·0477	101	0·0472
w	*D*	0·0546	102	0·0545
Trace	*D*	0·0841	104	0·0834
vw+	*S*	0·0877	006	0·0869
s	*S*	0·1353	110	0·1344
s	*S*	0·1570	113	0·1561
w	*D*	0·1829	201	0·1816
vw	*D*	0·1898	202	0·1889
vw	*S*	0·1967	009	0·1956
ms	*S*	0·2220	116	0·2213
w+	*D*	0·3169	211	0·3160
vw	*D*	0·3234	212	0·3233
w+	*S*	0·3306	119	0·3300
w+	*S*	0·4041	300	0·4032
m−	*S*	0·4256	303	0·4249
vw	*S*	0·4827	1.1.12	0·4820
w	*S*	0·4909	306	0·4901
w	*S*	0·5384	220	0·5376
m−	*S*	0·5597	223	0·5593
w−	*D*	0·5860	311	0·5848
vw	*D*	0·5916	312	0·5921
w−	*S*	0·5988	309	0·5988
w	*S*	0·6247	226	0·6245
vw	*D*	0·7204	401	0·7192
w−	*S*	0·7337	229	0·7332
vw	*S*	0·7514	3.0.12	0·7508
w	*D*	0·8559	321	0·8536
m−	*S*	0·9406	410	0·9408
s	*S*	0·9619	411	0·9625

Table 2. *Spectrometer data for* UO_2F_2

Observed intensity		Sharp or			$\sin^2\theta$
Area	Peak	diffuse	$H_1H_2H_3$	$K_1K_2K_3$	obs.
200	172	*S*	003	111	0·0221
179	101	*D*	101	100	0·0483
105	45	*D*	102	110	0·0541
40	21	*D*	104	211	0·0845
40	38	*S*	006	221	0·0874
25	9	*D*	105	$10\bar{1}$	0·1049
(100)	(100)	*S*	110	$1\bar{1}0$	0·1349
91	91	*S*	113	210	0·1565
36	10	*D*	107	322	0·1638
44	29	*D*	201	$11\bar{1}$	0·1820
29	20	*D*	202	200	0·1881
15	12	*S*	009	333	0·1956
60	48	*S*	116	321	0·2211

In Table 2 are given both the intensities at the diffraction maxima and the area under the various diffraction peaks. It was, however, difficult to measure the areas under the diffuse peaks.

The positions of the diffraction maxima correspond to a hexagonal cell of dimensions

$$a_1 = 4{\cdot}198 \pm 0{\cdot}001 \text{ kX.}, \quad a_3 = 15{\cdot}661 \pm 0{\cdot}01 \text{ kX.}$$

The Miller indices $H_1H_2H_3$ given in Tables 1 and 2 refer to this cell. The absent reflections show, however, that the true unit cell is a rhombohedron with

$$a = 5{\cdot}755 \pm 0{\cdot}003 \text{ kX.}, \quad \alpha = 42^\circ\, 47' \pm 3'.$$

The rhombohedral Miller indices are denoted by triplets $K_1K_2K_3$.

A direct density determination, carried out by Dr A. Novick, gave $\rho = 6{\cdot}24$ g.cm.$^{-3}$. Thus the rhombohedral unit cell contains one molecule UO_2F_2, the calculated density being $\rho = 6{\cdot}38$ g.cm.$^{-3}$.

Reference to Tables 1 and 2 shows that the sharp diffraction peaks have Miller indices $H_1H_2H_3$ such that $H_1 - H_2 = 3n$ and $H_3 = 3n$. Since reflections H_1H_20 and $00H_3$ are sharp, it is clear that the width of the diffuse lines cannot be ascribed to crystallite size.

If equivalent oxygen atoms and equivalent fluorine atoms are assumed, the space-group symmetry becomes $R\bar{3}m - D_{3d}^5$ with atomic positions as follows:

$$1\,\text{U in } (0, 0, 0),\ 2\,\text{O in } \pm(u, u, u),\ 2\,\text{F in } \pm(v, v, v).$$

The uranyl group is thus collinear as observed in other uranyl compounds (Fankuchen, 1935). In the structure of $Ca(UO_2)O_2$ and $Sr(UO_2)O_2$ the writer found a value U–O $= 1{\cdot}91 \pm 0{\cdot}10$ A. for the dimensions of the uranyl group. We shall adopt this value and accordingly we must set $u = 0{\cdot}122$.

If it is assumed that the closest distance of approach between two fluorine atoms or between an oxygen and a fluorine atom is 2·65 A., we find two possible values for the parameter v, namely, $v = 0{\cdot}294 \pm 0{\cdot}003$ or $v = 0{\cdot}376 \pm 0{\cdot}008$.

The sharp reflections cannot be used to distinguish between the two possible values of v, since $v = 0{\cdot}294$ and $v = 0{\cdot}372$ lead to identical structure factors for these reflections. In Table 3 are given the observed intensities for the sharp reflections and calculated intensities without and with oxygen and fluorine contributions. It is seen that the agreement between observed and calculated intensities is greatly improved when the effect of oxygen and fluorine atoms is taken into account. The parameter values $u = 0{\cdot}122$ and $v = 0{\cdot}294$ or 0·372 arrived at by spatial considerations are thus confirmed by the intensity data.

Table 3. *Intensities of sharp reflections*

		$I_{\text{calc.}}$	
$H_1H_2H_3$	$I_{\text{obs.}}$	Uranium only	All atoms
003	200	362	226
006	40	60	36
110	(100)	(100)	(100)
113	91	152	95
009	15	18	11
116	60	88	54

The intensities were calculated using the usual formula

$$I_{\text{calc.}} \propto |F|^2 p \frac{1 + \cos^2 2\theta}{\sin^2\theta \cos\theta}, \tag{1}$$

where p is the multiplicity of the reflecting planes.

The observed intensities of the diffuse reflections must be used to distinguish between the two values $v = 0{\cdot}294$ and $v = 0{\cdot}372$. As mentioned above, the great width of these reflections makes it difficult to measure their integrated intensities with accuracy. However, the observations on the integrated intensities, as shown in Table 4, definitely decide in favor of the value $v = 0{\cdot}294$.

Table 4. *Integrated intensities of diffuse reflections*

		$I_{\text{calc.}}$	
$H_1H_2H_3$	$I_{\text{obs.}}$	$v = 0{\cdot}294$	$v = 0{\cdot}373$
101	(270)	270	240
102	158	146	200
104	60	103	55
105	39	47	84
107	54	66	44

The intensity at the diffraction peak of a diffuse reflection can be more accurately measured than the area under the peak. Let I_{H_3} represent the intensity at the diffraction peak for a sequence of diffuse reflections $(H_1H_2H_3)$. It is shown in § 4 of this paper that

$$|F_H|^2 \propto \frac{H_3 \sin\theta}{1 + \cos^2 2\theta} I_{H_3}. \tag{2}$$

Hence we can use the peak intensities as well as the integrated intensities to distinguish between the values $v = 0{\cdot}294$ and $v = 0{\cdot}372$. The results given in Table 5 shows that this second method also leads to $v = 0{\cdot}294$.

Table 5. $|F_H|^2$ *from heights of diffuse peaks*

		Calculated	
$H_1H_2H_3$	Observed	$v = 0{\cdot}294$	$v = 0{\cdot}373$
101	(100)	(100)	(100)
102	83	63	98
104	111	73	43
105	53	43	86
107	144	100	75

3. Discussion of the ideal structure

The UO_2F_2 structure deduced in the preceding section can be regarded as a stack of identical, hexagonal layers, the distance between consecutive layers being 5·22 A. Each layer is electrically neutral and hence the layers are held together by secondary bonds.

The structure of a layer is shown in Fig. 1. The uranyl groups are normal to the layer, the oxygen atoms being 1·91 A. above and below the plane defined by the uranium atoms. The fluorine atoms are 0·61 A. above and below this plane.

Each uranium atom is bonded (by covalent bonds) to two oxygen atoms, thus forming the uranyl group, and in addition uranium forms six bonds to fluorine atoms with U–F = 2·50 A. The closest distance of approach between fluorine atoms belonging to the same layer is 2·71 A., while 2·75 A. is the closest distance between a fluorine and an oxygen atom of the same layer.

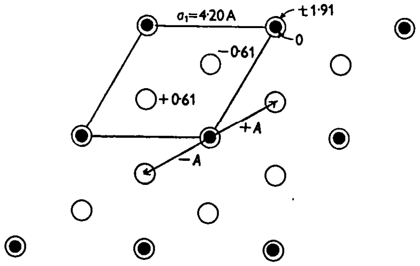

Fig. 1. The structure of a layer in UO_2F_2. Circles with filled centers represent uranyl groups with the uranium atoms in the plane of the paper and oxygen atoms 1·91 A. above and below this plane. The large open circles represent fluorine atoms which are 0·61 A. above or below the plane of the uranium atoms.

The vectors A and $-A$ represent the two possible horizontal displacements between consecutive layers in a stack.

The UO_2F_2 layer has the same structure as the UO_2O_2 layer in $Ca(UO_2)O_2$ and $Sr(UO_2)O_2$.

Because the uranyl groups are normal to the layer it is not possible to stack UO_2F_2 layers directly on top of one another. The next layer on top of the one shown in Fig. 1 is actually horizontally displaced by the amount indicated by the vector A shown in the figure. If the next layer had been displaced horizontally by the amount $-A$, a different structure would result, namely the one corresponding to $v = 0{\cdot}373$ which had to be rejected from intensity considerations. In the following we shall refer to the correct structure ($v = 0{\cdot}294$) as the A structure and to the incorrect structure ($v = 0{\cdot}373$) as the $\bar{A}$ structure.

The A structure and the $\bar{A}$ structure differ only in the binding between layers as shown below.

Bonds between layers

	A structure	$\bar{A}$ structure
O–3 O	2·80 A.	2·80 A.
O–1 F	2·70 A.	3·92 A.

The fact that the A structure is the correct one shows that there is an attractive force between oxygen atoms of one layer and fluorine atoms of adjacent layers. The main cohesive force between adjacent layers is, however, due to O–O interaction. It is not surprising that these O–O and O–F bonds between layers exist because both oxygen and fluorine atoms are strongly polarized as a result of their one-sided binding to uranium atoms.

4. The stacking disorder in the UO_2F_2 structure

In an earlier section it was pointed out that the diffuseness of some of the reflections could not be attributed to crystallite size. It was also mentioned that the intensity distribution in the diffuse peaks varied from sample to sample depending upon thermal history. This variation is illustrated in Fig. 2, which shows the diffuse peak at $2\theta \approx 26°$ for a well heat-treated sample and for a sample obtained by evaporating a UO_2F_2 solution to dryness at 120° C.

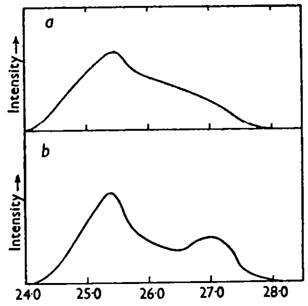

Fig. 2.

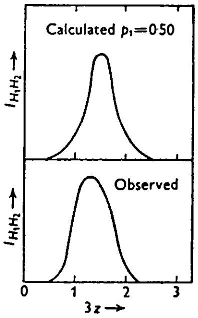

Fig. 3.

Fig. 2. The diffuse diffraction peak at $2\theta \approx 25\frac{1}{2}°$. Curve a was obtained for a sample which had received little heat treatment. Curve b shows how the diffraction is sharpened by further heat treatment.

Fig. 3. The calculated diffuse diffraction maximum in the $I_{H_1H_2}$ curve for $p_1 = 0{\cdot}50$, and the experimentally measured curve for a sample which has had little heat treatment.

We have shown that UO_2F_2 has a typical layer structure. It seems reasonable therefore to proceed on the assumption that the diffuseness of some of the peaks is associated with stacking disorder in the structure. We shall assume that the layers are equidistantly spaced and that the disorder is caused by variations in the relative horizontal displacement of consecutive layers.

The theory of X-ray diffraction from such a disordered layer structure was developed in a recent paper (Zachariasen, 1947).

The stacking disorder was characterized by means of functions $W_M(\delta)$ which give the probability of finding a relative horizontal displacement δ between layers M spacings apart. It was shown that the intensity of scattering, when plotted in the reciprocal lattice, is restricted to the lattice rows (H_1H_2z). The variation of intensity of scattering $I_{H_1H_2}(z)$ along such a lattice row was found to be given by the expression

$$I_{H_1H_2}(z) = K \mid \psi_{H_1H_2} \mid^2 \sum_M W^M_{H_1H_2} e^{i2\pi Mz}, \tag{3}$$

where K is a constant, $\psi_{H_1H_2}$ the structure factor for the layer, and $W^M_{H_1H_2}$ the Fourier coefficients for the function W_M.*

From (3) we have

$$W^M_{H_1H_2}=\frac{1}{K}\int_0^1 \frac{I_{H_1H_2}}{|\psi_{H_1H_2}|^2}e^{-i2\pi Mz}\,dz, \tag{4}$$

and hence it is possible to synthesize the functions W_M if $I_{H_1H_2}$ is experimentally measured and if the structure of the layer is known.

In the present case of UO_2F_2 efforts to prepare single crystals were unsuccessful, and the experimental functions $I_{H_1H_2}$ must be deduced from powder diffraction data. It is possible to do this, but the accuracy of the functions $I_{H_1H_2}$ obtained in this manner is not sufficiently great to justify the use of (4). Instead we shall make reasonable assumptions concerning the nature of the disorder, calculate the scattering function with the aid of (3) and compare the results with observations.

We shall assume that the stacking disorder in UO_2F_2 corresponds to an intermixture of the A and $\bar{A}$ structures. On this assumption the probability functions W_M will be discrete, and they will be different from zero only for $\delta=0$, A and $-A$.

Let P^0_M, P^+_M and P^-_M represent the probabilities of finding a relative displacement $\delta=0$, A and $-A$ respectively between layers M spacings apart. On the basis of the assumed disorder we shall then have

$$W^M_{H_1H_2}=P^0_M=P^+_M\,e^{-i2\pi(H_1-H_2)/3}+P^-_M\,e^{i2\pi(H_1-H_2)/3}. \tag{5}$$

Substitution of (5) in (3) yields, for $H_1-H_2=3n$,

$$I_{H_1H_2}(z)=K\,|\,\psi_{H_1H_2}\,|^2\sum_M e^{i2\pi Mz}; \tag{6a}$$

and, for $H_1-H_2=3n\pm1$,

$$I_{H_1H_2}(z)=\frac{K}{2}\,|\,\psi_{H_1H_2}\,|^2\left\{\sum_M(3P_M-1)\cos 2\pi Mz \pm\sqrt{3}\sum_M(P^+_M-P^-_M)\sin 2\pi Mz\right\}. \tag{6b}$$

According to (6*a*) $I_{H_1H_2}\neq0$ only at the reciprocal lattice points corresponding to integral values of z. The hexagonal period a_3 of § 2 being three times the layer separation, integral values of z imply $H_3=0, 3, 6\ldots$. Thus the assumed disorder leads to a correct prediction of the set of sharp reflections.

Unless the structure is completely ordered P^0_M, P^+_M, P^-_M approach the value $\frac{1}{3}$ as M increases. According to (6*b*) the scattering maxima $I_{H_1H_2}$ must therefore be diffuse for $H_1-H_2=3n\pm1$, as indeed is observed.

The quantities P^0_M, P^+_M, P^-_M can all be expressed in terms of the probability p_1 that a layer in the stack shows the correct displacement $+A$ with respect to the preceding layer. Hence $p_2=1-p_1$ is the corresponding probability of finding the anomalous displacement $-A$. Table 6 gives the expressions for small values of M. The general formulas for P^0_M, P^+_M, P^-_M are readily written down in terms of the binomial formula for $(p_1+p_2)^M$.

Table 6. *Quantities* P^0_M, P^+_M *and* P^-_M.

M	P^0_M	P^+_M	P^-_M
0	1	0	0
1	0	p_1	p_2
2	$2p_1p_2$	p_2^2	p_1^2
3	$p_1^3+p_2^3$	$3p_1^2p_2$	$3p_1p_2^2$
4	$6p_1^2p_2^2$	$p_1^4+4p_1p_2^3$	$4p_1^3p_2+p_2^4$
5	$5p_1^4p_2+5p_1p_2^4$	$10p_1^3p_2^2+p_2^5$	$p_1^5+10p_1^2p_2^3$
6	$p_1^6+20p_1^3p_2^3+p_2^6$	$6p_1^5p_2+15p_1^2p_2^4$	$15p_1^4p_2^2+6p_1p_2^5$

We are now in position to calculate the functions $I_{H_1H_2}(z)$ for any value of the probability p_1. $I_{H_1H_2}$ curves corresponding to $p_1=0{\cdot}50$ and to $p_1=0{\cdot}75$ are shown in Figs. 3 and 4.

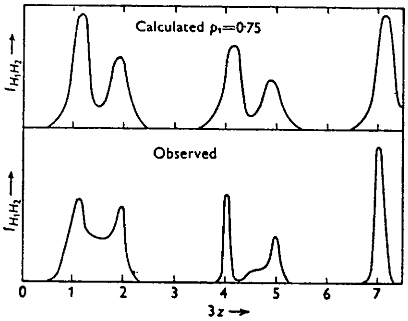

Fig. 4. The calculated and observed $I_{H_1H_2}$ curves, specifically $\frac{1}{2}\{I_{10}+I_{01}\}$. The calculated curve corresponds to $p_1=0{\cdot}75$. The experimental curve is for a well heat-treated sample.

The functions $I_{H_1H_2}(z)$ can be found experimentally. In powder diffraction patterns one measures the power $J(\theta)\,d\theta$ associated with unit length of a Debye circle. In terms of the intensity distribution $I_{H_1H_2}(z)\,dz$ of the reciprocal lattice one has

$$J_{H_1H_2}(\theta)\propto\frac{1+\cos^2 2\theta}{\sin^2\theta\cos\theta}\,I_{H_1H_2}\frac{dz}{d\theta}. \tag{7}$$

If θ_0 be the glancing angle corresponding to $z=0$ for given H_1 and H_2, the relationship between z and θ becomes

$$z=\frac{2a_3}{3\lambda}\sqrt{(\sin^2\theta-\sin^2\theta_0)}, \tag{8}$$

where a_3 is three times the layer separation. Accordingly one finds

$$I_{H_1H_2}(z)=C\,\frac{\sin\theta\sqrt{(\sin^2\theta-\sin^2\theta_0)}}{1+\cos^2 2\theta}\,J_{H_1H_2}, \tag{9}$$

where C is a constant.

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* In the paper (Zachariasen, 1947), it was assumed that the functions W_M were even functions. This assumption is not always correct. For greater generality the equations in the paper referred to above should accordingly be modified by replacing the cosine functions by the corresponding exponential functions.

It is obvious that experimental curves $I_{H_1H_2}(z)$ deduced from powder diffraction patterns will be rather inaccurate except in the regions corresponding to large values of $J_{H_1H_2}(\theta)$, i.e. in the immediate neighborhood of the prominent diffraction maxima. To a first approximation the height of the maxima $J_{H_1H_2}(\theta)$ is inversely proportional and the width directly proportional to the corresponding z values. Hence, the experimental error in the $I_{H_1H_2}$ curves increases with z. It should further be noted that powder diffraction patterns will give only mean curves $\frac{1}{2}\{I_{H_1H_2}(z)+I_{H_2H_1}(z)\}$.

Experimental curves $\frac{1}{2}\{I_{10}(z)+I_{01}(z)\}$ for two typical specimens of UO_2F_2 are shown in Figs. 3 and 4. The curve of Fig. 3 obviously corresponds to the sample with the greater, that of Fig. 4 to the sample with the smaller amount of stacking disorder. In the former sample one would estimate $p_1=0{\cdot}60$–$0{\cdot}65$ and in the latter $p_1=0{\cdot}90$–$0{\cdot}95$.

Considering the inaccuracy of the experimental data the agreement between observed and calculated $I_{H_1H_2}$ curves must be characterized as surprisingly good. The postulated nature of the stacking disorder can thus be said to be completely confirmed by the observations. The results outlined above have further demonstrated the value and the usefulness of the general theory of X-ray diffraction in crystals with stacking disorder.

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Crystal Chemical Studies of the 5*f*-Series of Elements. IV. The Crystal Structure of $Ca(UO_2)O_2$ and $Sr(UO_2)O_2$

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$Ca(UO_2)O_2$ and $Sr(UO_2)O_2$ are rhombohedral and isostructural. The unit cell containing one molecule has dimensions:

$$Ca(UO_2)O_2:\quad a=6{\cdot}254\pm0{\cdot}001\text{ kX.},\quad \alpha=36^\circ\ 2'\pm1',$$
$$Sr(UO_2)O_2:\quad a=6{\cdot}53\pm0{\cdot}03\text{ kX.},\quad \alpha=35^\circ\ 32'\pm20'.$$

The calculated density is 7·45 g.cm.$^{-3}$ for the calcium compound and 7·62 g.cm.$^{-3}$ for the strontium compound. The space group is $R\bar{3}m$ with 1 U in (0, 0, 0), 1 Ca (or Sr) in $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, $2O_I$ in $\pm(u, u, u)$, $2O_{II}$ in $\pm(v, v, v)$. For the calcium compound $u=0{\cdot}109\pm0{\cdot}006$, $v=0{\cdot}361\pm0{\cdot}006$.

The structure contains collinear uranyl groups with $U\text{–}O_I=1{\cdot}91\pm0{\cdot}10$ A. Each uranium atom forms six weaker bonds with $U\text{–}O_{II}=2{\cdot}29$ A. A calcium or strontium atom is bonded to eight oxygen atoms with Ca–O = 2·45 A., Sr–O = 2·58 A.

Introduction

While working in the Manhattan Project during the war the writer prepared a number of uranates and studied their crystal structures in order to get information on the crystal chemistry of hexavalent uranium compounds. This article gives the results of the crystal structure investigation of calcium uranate and of the isostructural strontium uranate. These results were obtained in 1944 and presented in the classified reports of the Manhattan Projects. The structure and dimensions of the uranyl group in calcium uranate given in this paper were reported at the International Crystallographic Conference in London in 1946; but the details of the investigation have only recently been declassified. The crystal structure of the uranates was determined on the basis of the photographic data of Table 4. The spectrometer data of Tables 1–3 were taken later and led to a complete confirmation of the structure originally found.

The experimental data

The compounds were prepared by the writer by adding U_3O_8 or UO_3 to an excess of molten $CaCl_2$ or $SrCl_2$. The melt was held at 1000° C. for half an hour, and the uranate formed as a surface crust. Both uranate preparations were of yellow-green color and consisted of microscopic crystals. These were very thin flakes with hexagonal outline and optically uniaxial character. Because of the pressure of war work it was not at the time possible to get a direct chemical analysis of the preparations. The chemical identity was therefore determined from the X-ray diffraction results as described below.

The microscopic crystals were too small to permit the use of single-crystal X-ray diffraction patterns. As a consequence crystal powder specimens were used. The