THE CRYSTAL STRUCTURE OF URANIUM TETRAFLUORIDE

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ABSTRACT

Uranium tetrafluoride has been analyzed by Fourier refinement of the U parameters reported by Zachariasen (6), followed by determination of the F parameters by crystal chemical and packing considerations. The structure is an ionic one in which each U atom is surrounded by eight F atoms, and each F atom is situated between two U atoms. There are two types of U-8F coordination groups which are geometrically similar to the arrangement in the \([\text{Mo(CN)}_8]^{4-}\) ion. The array of U-8F groups in space is related to the structures of ZrSiO$_4$, ThCl$_4$, and UC$_4$. The compounds UF$_4$, U$_4$F$_{17}$ and U$_2$F$_9$ have a number of structural and chemical similarities.
Zachariasen (6) determined the unit cell dimensions and space group for the compounds ThF₄, UF₄, NpF₄, PuF₄ and CeF₄ and observed that they formed an isomorphous series with the compounds ZrF₄ and HfF₄ whose cell constants and space group had previously been reported (5). In addition, Zachariasen deduced the approximate positions of the U atoms in UF₄.

When single crystals of UF₄ became available to this laboratory, it seemed quite desirable to determine the complete structure since it is representative of at least seven compounds.

**ORIENTATION OF SPACE GROUP C₆²h**

The isomorphous series of tetrafluorides contains twelve MF₄ units in the space group C₆²h which Zachariasen oriented with a base-centered translation according to the International symbol of C2/c. For the present investigation this setting is not a natural one and it was changed to the body-centered orientation of I2/c. This yields the following positions:

(000; 1/2 1/2 1/2) +
4: (a) 000; 00 1/2. (b) 0 1/2 0; 0 1/2 1/2. (c) 1/4 1/4 1/4; 3/4 1/4 1/4.
(d) 1/4 1/4 3/4; 3/4 1/4 3/4. (e) ±(0,y,1/4).
8: (f) ±(x, y, z); (x, y, 1/2 + z).

The relations between the body centered axes aₜ, cₜ and the C-centered axes aₐ, cₐ are aₜ = -aₐ - cₐ and cₜ = cₐ. The relations between the parameters xₜ, zₜ based on a body centered cell and the parameters xₐ, zₐ based on the C-centered cell are xₜ = 1-xₐ and zₜ = zₐ-xₐ.

**EXPERIMENTAL**

Needle-like crystals with dimensions less than 0.1 mm. in cross section were used for x-ray examination. The c axis proved to be parallel to the needle axis. This meant that with orientation I2/c the c* axis was inclined at about 4° to the needle axis whereas with orientation C2/c the c* axis was inclined at about 36° to the needle axis.

From precession photographs, using MoKα radiation, λ = 0.7107 Å, the unit cell constants for I2/c were found to be

\[
\begin{align*}
a &= 10.40 \pm 0.01 \text{ Å} \\
b &= 10.84 \pm 0.01 \text{ Å} \\
c &= 8.39 \pm 0.01 \text{ Å} \\
\beta &= 94° \pm 10' 
\end{align*}
\]
Intensities were recorded by multiple exposure with the precession camera and by multiple film technique with the Weissenberg camera. Intensities were measured by visual comparison and corrected for the Lorentz and polarization factors. The resulting structure factors are believed to be accurate to ± 10%, exclusive of errors due to absorption.

FOURIER ANALYSIS

The U positions were refined by two-dimensional Fourier syntheses on the three principal projections. Table 1 compares the present U parameters with Zachariasen's data, both referred to I2/c.

Table 1

<table>
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<tr>
<th>Atom</th>
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<th>This Paper</th>
<th>Zachariasen (6)</th>
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<tr>
<td></td>
<td></td>
<td>x  y  z</td>
<td>x  y  z</td>
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<tr>
<td>U₁</td>
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<td>0.200</td>
</tr>
<tr>
<td>U₂</td>
<td>(f)</td>
<td>0.296 0.927 0.125</td>
<td>0.292 0.937 ≅ 0.12</td>
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</table>

It was hoped that the electron density maps would give an indication of the F positions but this was not the case. The background fluctuations were dominated by diffraction effects from the U peaks. No detail of fluorine peaks could be retained when the diffraction peaks were removed by temperature corrections. A synthesis which included the observed coefficients plus coefficients calculated from the U positions for values of \( \sin \theta / \lambda \) greater than the limit of the sphere of reflection was unsatisfactory. Syntheses were also made in which the coefficients calculated from the U positions were subtracted from the observed coefficients. The maps from these difference syntheses yielded too many peaks to be accounted for by the F atoms, probably because of absorption errors in the experimental data. Fig. 4 illustrates the only reasonable difference synthesis obtained which was interpreted after the final structure was known.

DETERMINATION OF FLUORINE PARAMETERS

When the distances between U atoms were computed, using the parameters of table 1, the key to the structure was quickly obtained. Each \( U₁ \) atom is surrounded by eight \( U₂ \) atoms at the following distances:

\[
\begin{align*}
U₁ - 2U₂ &= 4.50 \text{ Å}.
U₁ - 2U₂ &= 4.51 \text{ Å}.
U₁ - 2U₂ &= 4.55 \text{ Å}.
U₁ - 2U₂ &= 4.57 \text{ Å}.
\end{align*}
\]
Each U₂ atom is surrounded by four U₁ atoms at the above distances, and in addition by four U₂ atoms at the following distances:

\[
\begin{align*}
U₂ - 2U₂ &= 4.48 \text{ Å} \\
U₂ - U₂ &= 4.51 \text{ Å} \\
U₂ - U₂ &= 4.59 \text{ Å} \\
\end{align*}
\]

These distances are all about double the value to be expected for a U-F distance, indicating that as a first approximation the F atoms lie at the mid-points between U atoms. Then each U is coordinated by eight F atoms and each F is located between two U atoms. The ionic charges are all balanced locally.

The structure is not satisfactory with the F ions at the exact mid-points between U atoms with regard to F-F distances, several of which are too short. However, the F atoms can be moved along certain directions in which the F-F distances vary more rapidly than the U-F distances. Thus by trial and error it was possible to achieve satisfactory packing of the F atoms without disturbing the U-F coordination scheme. The final F parameters obtained in this fashion are collected in Table 2 along with the U parameters.

Table II

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<td>(f)</td>
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<td>0.025</td>
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</table>

DESCRIPTION OF THE STRUCTURE

Fig. 1 illustrates the structure as seen along the c axis. The U₁-8F groups lie on 2-fold axes and are fairly symmetrical in appearance. The U₂-8F groups are in general positions and appear quite irregular in form. Each U₁-8F group shares its F atoms with eight neighboring U₂-8F groups. Each U₂-8F group shares its F atoms with four neighboring U₁-8F groups and with four neighboring U₂-8F groups.
STRUCTURE OF UF₄ PROJECTED ALONG THE C AXIS

FIGURE 1
In Fig. 2(a), a \( U_2-8F \) group is shown isolated from its surroundings. In Fig. 2(b) the \( U_2-8F \) group has been re-oriented for comparison with the \( U_1-8F \) group illustrated in Fig. 2(c). It becomes apparent that the \( U_2-8F \) group is more symmetrical than projections along the unit cell axes would indicate. The \( U_1-8F \) group has a true 2-fold axis of symmetry and the \( U_2-8F \) group makes a good approximation to the same symmetry.

Each \( U_1 \) atom has the following bond distances:

\[
\begin{align*}
U_1-2F_3 &= 2.29 \text{ Å} \\
U_1-2F_4 &= 2.35 \text{ Å} \\
U_1-2F_6 &= 2.29 \text{ Å} \\
U_1-2F_7 &= 2.31 \text{ Å} \\
\text{Ave.} &= 2.305 \text{Å}.
\end{align*}
\]

while each \( U_2 \) atom has

\[
\begin{align*}
U_2-F_1 &= 2.26 \text{ Å} \\
U_2-F_2 &= 2.31 \text{ Å} \\
U_2-F_3 &= 2.30 \text{ Å} \\
U_2-F_4 &= 2.33 \text{ Å} \\
U_2-F_5 &= 2.24 \text{ Å} \\
U_2-F_6 &= 2.27 \text{ Å} \\
U_2-F_7 &= 2.28 \text{ Å} \\
\text{Ave.} &= 2.28 \text{Å}.
\end{align*}
\]

The \( U-F \) bond distances and the geometry of the two types of \( U-8F \) groups are so similar that the \( U_1 \) and \( U_2 \) atoms appear to be equivalent chemically.

The closest \( F-F \) distances in a \( U_1-8F \) group are

\[
\begin{align*}
F_3-F_4 &= 2.88 \text{ Å} \\
F_3-F_6 &= 2.55 \text{ Å} \\
F_3-F_6' &= 2.57 \text{ Å} \\
F_5-F_7 &= 2.79 \text{ Å} \\
F_4-F_7' &= 2.81 \text{ Å} \\
F_4-F_7 &= 2.97 \text{ Å} \\
F_6-F_7 &= 2.78 \text{ Å}.
\end{align*}
\]

while the closest distances in a \( U_2-8F \) group are

\[
\begin{align*}
F_1-F_5' &= 2.76 \text{ Å} \\
F_1-F_7 &= 2.85 \text{ Å} \\
F_2-F_3 &= 2.64 \text{ Å} \\
F_2-F_5' &= 2.70 \text{ Å} \\
F_2-F_6 &= 2.86 \text{ Å} \\
F_3-F_5 &= 2.63 \text{ Å} \\
F_4-F_5' &= 2.75 \text{ Å} \\
F_4-F_5 &= 2.80 \text{ Å} \\
F_4-F_6 &= 2.51 \text{ Å} \\
F_4-F_7 &= 2.69 \text{ Å} \\
F_5'-F_6 &= 2.59 \text{ Å} \\
F_5'-F_6 &= 2.69 \text{ Å} \\
F_5'-F_7 &= 2.65 \text{ Å} \\
\end{align*}
\]

* also between atoms of different coordination groups.
(A) $U_2$–8F GROUP PROJECTED ALONG THE C AXIS
(B) $U_2$–8F GROUP RE-ORIENTED FOR COMPARISON WITH $U_1$–8F GROUP
(C) $U_1$–8F GROUP PROJECTED ALONG THE C AXIS

FIGURE 2
DISCUSSION

The geometry of the $U_1$-8F and $U_2$-8F groups of Fig. 2(c) and (b) is similar to that of the $\text{Mo(CN)}_8^{4-}$ ion (2). The idealized configuration has the point group symmetry 42m and is obtained by superimposing two tetrahedra, one elongated, the other flattened along the 4 axis as indicated in the stereogram of Fig. 3. The eight points lie at the apices of a dodecahedron.

The ensemble of U-8F groups is more closely related to the structures of ZrSiO$_4$, ThCl$_4$ and UCl$_4$. In ZrSiO$_4$ there are Zr-80 groups with Zr-40 = 2.05 Å, and Zr-40 = 2.41 Å. In ThCl$_4$ there are Th-8Cl groups with Th-4Cl = 2.46 Å, and Th-4Cl = 3.11 Å., while in UCl$_4$ there are U-8Cl groups with U-4Cl = 2.41 Å and U-4Cl = 3.09 Å (4). The space group for ZrSiO$_4$, ThCl$_4$ and UCl$_4$ is I4/amd with c/a = 0.901, 0.881 and 0.902. The body-centered tetragonal lattice generates coordination groups with the symmetry of Fig. 3. When UF$_4$ is oriented as I2/c the cell is body-centered pseudo-tetragonal with c/a $\neq$ 0.79.

The uranium fluorides can be divided into two categories depending on whether or not all the F atoms are bonded to two U atoms. In the first classification are UF$_6$ in which all F atoms are bonded to only one U atom, leading to discrete molecules (3), and $\alpha$- and $\beta$-UF$_5$ in which some of the F atoms are bonded to only one U atom (7). In the second group it appears quite certain that we have the series UF$_4$, U$_4$F$_{17}$ and U$_2$F$_9$.

The unusual compound U$_4$F$_{17}$ was identified by Agron, Grenall, Kunin and Weller (1). It yields a complex powder pattern which was not indexed but which was referred to as "distorted UF$_4$" because of its similarities to the UF$_4$ pattern. The UF$_4$ structure has 12 "holes" in it at the special positions (a), (b) and (c) of space group I2/c, some of which are apparent in Fig. 1. The void space at these points is not actually large enough to accommodate F atoms but distortions of the structure should make this possible. By adding three F atoms to the UF$_4$ unit cell, say one in (a) or (c) and two in (b), we change the composition from UF$_4$ to U$_4$F$_{17}$, there is an equal number of U-8F groups and U-9F groups, every F is bonded to two U atoms and there are nine "holes" left in the unit cell. The numerical ratio of U atoms to "holes" becomes 12/9 = 4/3.

In UF$_6$ (8), the U atoms resonate between valence states 4 and 5, there are 6-9F groups and every F is bonded to two U atoms. There are six "holes" in the structure at special position (b) of space group I4/m. The numerical ratio of U atoms to "holes" is 8/6 = 4/3. By adding six F atoms to the UF$_4$ unit cell, say two in (a) or (c) and four in (b), we change the composition from UF$_4$ to U$_2$F$_9$, every U atom forms U-9F groups and every F is bonded to two atoms. However, with only six "holes" left in the unit cell, the numerical ratio of U atoms to "holes" is 12/6.

It is quite likely that U$_4$F$_{17}$ is formed by adding three F atoms to the UF$_4$ unit cell, leading to a structure based on a body centered space lattice with axial ratios intermediate to those of UF$_4$ and U$_2$F$_9$. The new structure
STEREOGRAPHIC PROJECTION OF 8 MEMBERED COORDINATION GROUP WITH THE POINT GROUP SYMMETRY $\bar{4}2m$

FIGURE 3
is presumably of such a nature that additional F atoms can be assimilated to reach the \( \text{U}_2\text{F}_9 \) composition and cubic symmetry without changing the \( \frac{4}{3} \) ratio of U atoms to "holes". Half the U atoms in \( \text{U}_4\text{F}_{17} \) should be in valence state 4 while the remaining U atoms resonate between valence states 4 and 5. The color of both \( \text{U}_2\text{F}_9 \) and \( \text{U}_4\text{F}_{17} \) is black, indicating that a similar resonance between states 4 and 5 occurs in both structures.

The mechanism of adding F atoms, first to the \( \text{UF}_4 \) structure, and then to the \( \text{U}_4\text{F}_{17} \) structure, implies that there will be a progressive increase in density and this is born out by experiment. Using the \( \text{UF}_4 \) cell volume and successive contents of \( 12\text{UF}_4, 3\text{U}_4\text{F}_{17} \) and \( 6\text{U}_2\text{F}_9 \) one calculates densities of 6.63, 6.73, and 6.84 g./cc. The observed densities are higher, being 6.94 g./cc (liquid displacement) for \( \text{U}_4\text{F}_{17} \) and 7.08 g./cc (x-ray value) for \( \text{U}_2\text{F}_9 \). The successive increase in experimental values should be a measure of the distortion which the structures undergo in order to accommodate additional F atoms and it appears that \( \text{UF}_4 \) must be distorted more than \( \text{U}_4\text{F}_{17} \). This would mean that the \( \text{U}_4\text{F}_{17} \) structure resembles the \( \text{U}_2\text{F}_9 \) structure more closely than the \( \text{UF}_4 \) structure. This is reasonable since both \( \text{U}_4\text{F}_{17} \) and \( \text{U}_2\text{F}_9 \) contain U atoms in a resonating valence state.

**COMPARISON WITH EXPERIMENT**

The contribution of F atoms to the calculated structure factors is of the same order of magnitude as the experimental errors in the observed structure factors. A comparison of F obs. vs. F cal. for 200 zonal reflections gives a correlation of 18% for either U atoms alone or for both U and F atoms. The average ratio of the F contribution to the U contribution is about 12%. However, for the hk0 reflections where a correlation of 15% was obtained, with or without the F contribution, the difference synthesis referred to under Fourier Analysis seemed to have some significance. Fig. 4 illustrates the asymmetric unit of the \( \text{F}_0\text{F}_c \) synthesis projected along the c axis. The F positions obtained by other considerations than diffraction data are indicated by black dots. The F positions fall on or near maxima in the electron density even though experimental errors have introduced a number of other peaks.

Table III lists observed and calculated structure factors for the hk0, h0l and Okl reflections. Both U and F contributions were included in the calculated structure factors.

**ACKNOWLEDGMENT**

The writer is greatly indebted to Mr. Frank N. Bensey who carried out much of the x-ray photography and gave valuable assistance with computations as did Miss Virginia F. Feaster. Other computations were handled on I. B. M equipment by Mr. E. C. Witt.
ASYMMETRIC UNIT OF DIFFERENCE SYNTHESIS PROJECTED ALONG C AXIS. CONTOURS AT INTERVALS OF $3e^-/A^2$ WITH BROKEN CONTOUR AT $12e^-$ LEVEL. F POSITIONS IN FINAL STRUCTURE INDICATED BY BLACK DOTS.

FIGURE 4
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<th>( F_{\text{calc}} )</th>
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TABLE III (Continued)

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REFERENCES


