

Short Communications

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The crystal structure of U_2IrC_2 .* By A. L. BOWMAN, G. P. ARNOLD and N. H. KRİKORIAN, *Los Alamos Scientific Laboratory, University of California, Los Alamos, New Mexico 87544, U.S.A.* and W. H. ZACHARIASEN, *Department of Physics, University of Chicago, Chicago, Illinois 60637, U.S.A.*

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The crystal structure of U_2IrC_2 has been determined as tetragonal, space group $I4/mmm$, U in 4(e), $z = 0.355$ (1), Ir in 2(a), C in 4(e), $z = 0.168$ (1), by neutron diffraction. The lattice parameters are $a_0 = 3.4804$ (1), $c_0 = 12.4821$ (8) Å.

The ternary compound uranium iridium carbide, U_2IrC_2 , was discovered as a product of the solid-state reaction between uranium carbide and iridium, and was subsequently prepared by arc-melting the elements (Krikorian, Wallace, Krupka & Radosevich, 1967; Farr & Bowman, 1964). The X-ray powder diffraction data were indexed on a tetragonal cell with dimensions $a_0 = 3.479$, $c_0 = 12.48$ Å. We have now determined the atom positions by neutron diffraction, fitting a tetragonal cell, space group $I4/mmm$, $a_0 = 3.4804$ (1), $c_0 = 12.4821$ (8) Å, with U in 4(e) (00z), $z \approx 0.35$, C in 4(e), $z \approx 0.16$, Ir in 2(a) (000).

Uranium iridium carbide was prepared by arc-melting the high-purity elements under argon. The arc-cast material was ground to <250 mesh and used without further heat treatment. Chemical analysis gives a composition $U_{2.01}IrC_{1.98}$. The diffraction patterns show UIr_2 and UC as impurity phases. Neutron-diffraction data were obtained at the Los Alamos Omega West Reactor, scanning from $2\theta = 5$ to 67.5° at an interval of 0.05° , at a wave-length of 1.3368 Å. The sample was contained in a parallel-sided holder made from a null-matrix alloy (Ti-Zr) (Sidhu, Heaton, Zaubers & Campos, 1956) and was placed in the symmetrical transmission position. An absorption correction, $\mu t = 0.541$, was measured. The diffraction pattern is shown in Fig. 1.

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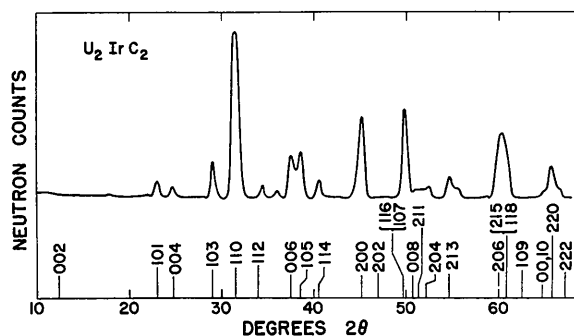


Fig. 1. Neutron diffraction pattern of U_2IrC_2 .

The observed intensities were determined by least-squares analysis of the diffraction data (Bowman, Wallace, Yarnell, Wenzel & Storms, 1965), and were fitted to the trial structure by least-squares solution of the equation (Bacon, 1962)

$$I = K \frac{\exp(-\mu t \sec \theta)}{\sin^2 2\theta} \exp\left(-2B \frac{\sin^2 \theta}{\lambda^2}\right) jF^2,$$

with $K = 0.102 \pm 0.002$, $B = 0.39 \pm 0.14$, $z_U = 0.3548 \pm 0.0008$, $z_C = 0.1677 \pm 0.0012$, $R = 0.037$, where $R = \sum w|I_o - I_c| / \sum wI_o$. Neutron scattering lengths, in units of 10^{-12} cm, of 0.85 for uranium, 1.06 for iridium and 0.665 for carbon were used. The observed and calculated neutron diffraction intensities are compared in Table 1.

Table 1. Neutron diffraction data

<i>h</i>	<i>k</i>	<i>l</i>	<i>d</i>	<i>I</i> (obs)	<i>I</i> (calc)
0	0	2	6.24	0.2	0.0
1	0	1	3.35	5.3	5.6
0	0	4	3.12	2.8	3.0
1	0	3	2.67	12.6	13.0
1	1	0	2.46	56.8	55.3
1	1	2	2.29	0.3	0.0
0	0	6	2.08	15.4	15.2
1	0	5	2.03	18.3	18.4
1	1	4	1.93	6.1	4.7
2	0	0	1.74	31.1	28.4
2	0	2	1.68	0.0	0.0
1	1	6	1.588	36.3	36.3
1	0	7			
0	0	8	1.560	0.7	1.1
2	1	1	1.545	2.2	2.5
2	0	4	1.520	2.9	3.0
2	1	3	1.458	8.4	8.2
2	0	6	1.335	26.0	26.5
2	1	5	1.320	18.1	19.6
1	1	8			
1	0	9	1.288	0.1	0.2
0	0	10	1.248	0.8	0.6
2	2	0	1.231	14.7	15.2
2	2	2	1.207	0.0	0.0

The U_2IrC_2 unit cell is shown pictorially in Fig. 2. This structure is closely related to that of UC (NaCl-type), as seen by referring the UC structure to a tetragonal cell containing six formula units. The U_2IrC_2 structure is derived

by replacing 2U atoms with 2Ir atoms and removing 2C atoms. The structure parameters are compared in Table 2. Interatomic distances between nearest neighbors are listed in Table 3.

Table 2. Comparison of UC and U_2IrC_2

Space group $I4/mmm$	
6UC	$2U_2IrC_2$
$a = a_c/\sqrt{2} = 3.51$	$a = 3.480$
$c = 3a_c = 14.88$	$c = 12.482$
2U in 2(a) (000)	2Ir in 2(a)
4U in 4(e) (00z)	4U in 4(e)
$z = \frac{1}{2}$	$z = 0.355$
2C in 2(b) (00 $\frac{1}{2}$)	—
4C in 4(e) (00z)	4C in 4(e)
$z = \frac{1}{2}$	$z = 0.168$

Table 3. Interatomic distances

U-1C	2.335 (27)
U-4C	2.477 (3)
U-4Ir	3.056 (9)
U-4U	3.480 (1)
Ir-2C	2.093 (22)
Ir-8U	3.056 (9)
C-1Ir	2.093 (22)
C-1U	2.335 (27)
C-4U	2.477 (3)

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The structure of *p*-azoxyanisole. By C. H. CARLISLE and C. H. SMITH, *Department of Crystallography, Birkbeck College, Malet Street, London WC1E 7HX, England*

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p-Azoxyanisole crystallizes in the monoclinic system. The space group is $P2_1/c$ with $a = 1.09$, $b = 0.81$, $c = 1.56$ nm, $\beta = 114^\circ$, and $Z = 4$.

It was learned shortly before the International Union of Crystallography meeting in 1969 at which we presented a paper on the crystal structure determination of the liquid crystal *p*-azoxyanisole (Carlisle & Smith, 1969) that Krigbaum, Chatani & Barber (1970), Braun, Hornstra & Leenhouts (1969), and Bednowitz (1970) had also independently and unknown to each other successfully determined the structure of this crystal. The results from all four laboratories are in agreement and this communication sets out very briefly the results obtained by us.

The crystals of *p*-azoxyanisole are monoclinic with $a = 1.09$, $b = 0.81$, $c = 1.56$ nm and $\beta = 114^\circ$, space group $P2_1/c$ and $Z = 4$.

Data using filtered Cu $K\alpha$ radiation were collected by photographic means in contrast with the other three groups who used diffractometer methods. The intensities of 1006 independent reflexions out of a (theoretically) possible 3000

were visually estimated and used in the investigation. The structure was solved by the symbolic addition method of Karle & Karle (1966) and was refined to an R value of 18.6% using anisotropic temperature factors and no weighting scheme. Fig. 1 shows the projected view of the structure on (010) and Fig. 2 shows the view down the a axis. This R value, which excludes the use of H atoms, compares not unfavourably with the lower R values of 9.1% obtained by Krigbaum *et al.* (1970), of 6.7% obtained by Braun *et al.* (1969) and of 8.1% obtained by Bednowitz (1970), all of whom included the scattering contribution of hydrogen atoms in their final results. A suitable weighting scheme would almost certainly have reduced the R value (18.6%) obtained by us.

The detailed characteristics of the four structures are essentially the same. The atomic coordinates, after transforming axes as necessary, have been compared and the differ-

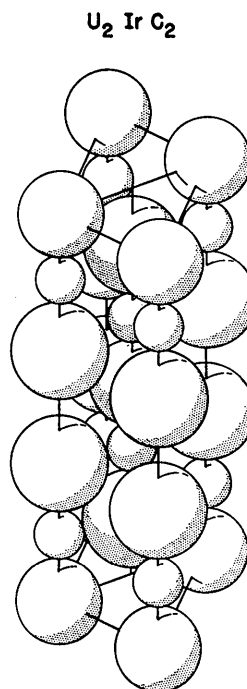


Fig. 2. Pictorial representation of U_2IrC_2 .