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The atomic position parameter in alpha uranium.*

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The crystal structure of the room temperature, or alpha phase of uranium, has been studied by Wilson (1933) and Jacob & Warren (1937) using powder methods, and by Lukesh (1949) who used single crystal techniques. Wilson reported that uranium crystallizes in the monoclinic system; Jacob & Warren indexed the powder data on the basis of the correct, orthorhombic unit cell and reported that the space group is D_{2h}^{17}-*Cmcm* with four atoms per unit cell. Their analysis of the relative intensities of the powder diffraction lines indicated that the value of y, the one variable positional parameter in the structure, was 0·105 ± 0·005. Lukesh confirmed the space group reported by Jacob & Warren but was unable to compute a meaningful y parameter because of absorption. In the present work a precise value of y has been obtained by a single crystal diffractometric technique for which the absorption is constant, i.e., independent of the Bragg angle.

A large face of a single crystal of α uranium (about 0·3 cm. in diameter was ground and polished parallel to (010). Diffraction intensities of (0k0) reflections were measured with a G.E. single crystal goniometer, using a scintillation counter as radiation detector. Filtered Mo K radiation was used. (0k0) reflections with k ranging from 2 to 16 (k even) were recorded.

The positional parameter was determined by locating the minimum in plots of R (the discrepancy factor) versus different assumed values of y in the range between 0·1000 and 0·1050. A preliminary analysis indicated that the correct value was close to 0·1025 and a temperature factor of $B=0{\cdot}4$ was computed in the usual way by plotting $\ln F_o/F_c$ versus $\sin^2\theta/\lambda^2$ using this value of y in the calculation of F_c. Thomas–Fermi atomic scattering factors were used and corrected for dispersion by the expression

$$f=|[(f_0+\Delta f')^2+(\Delta f'')^2]^{1/2}|\,,$$

where $\Delta f'=-5$ and $\Delta f''=15$ (Dauben & Templeton, 1955). We may note that inclusion of the dispersion factor in the calculations had minor effects on the indicated values of R and the temperature factor but altered the calculated y parameter by less than one part in the fourth significant figure.

Intensities were determined both by planimetering the areas under the peaks on the strip chart records of the diffracted beams and by direct scaler counting of the integrated intensities. The former appeared to give somewhat more reliable results, probably because of the greater ease with which the background could be located. A plot of R versus y is shown in Fig. 1. At $y=0{\cdot}1024$ the curve shows a sharp minimum; at this point the value of R is 1·5%. A similar plot of R versus y, using intensities determined by the second method gave a considerably less sharp minimum at $y=0{\cdot}10275$ at which point $R=3{\cdot}8\%$.

The best value of y was taken to be 0·1025 ± 0·0003, where the limits of error are for the 95% confidence interval of the mean.

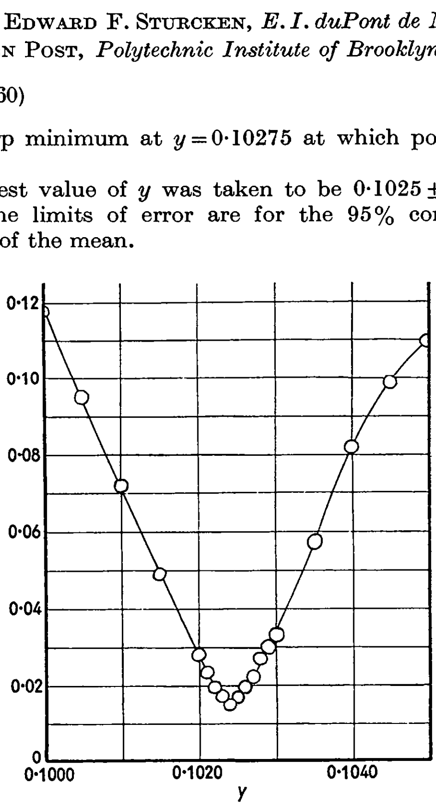

Fig. 1. R versus y for α-uranium.

Unit-cell dimensions were redetermined on the basis of diffractometric measurements using high purity annealed specimens of polycrystalline uranium and found to be:

$$a=2{\cdot}854,\; b=5{\cdot}869,\; c=4{\cdot}955\text{ Å},\;\text{all } \pm 0{\cdot}001\text{ Å}\,.$$

The cell parameters were calculated using a Hess (1951) modification of the Cohen (1935, 1936) least-square technique adapted to an IBM 650 computer.

The new parameter places the atom at 0·015 Å from the position given by Jacob & Warren. The twelve smallest interatomic distances are: two at 2·754, two at 2·854, four at 3·263, and four at 3·342 Å. Calculated diffraction intensities differ appreciably from those obtained with the previous y parameter.

References

Cohen, M. U. (1935). *Rev. Sci. Instrum.* **6**, 68.

Cohen, M. U. (1936). *Rev. Sci. Instrum.* **7**, 155.

Dauben, C. H. & Templeton, D. H. (1955). *Acta Cryst.* **8**, 841.

Hess, J. B. (1951). *Acta Cryst.* **4**, 209.

Jacob, C. W. & Warren, B. E. (1937). *Amer. Chem. Soc.* **59**, 2588.

Lukesh, J. S. (1949). *Acta Cryst.* **2**, 420.

Wilson, T. A. (1933). *Physics*, **4**, 148–52.

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